Fall 9-14-2021

SILVER BOW CREEK/BUTTE AREA NPL SITE BUTTE PRIORITY SOILS OPERABLE UNIT

Pioneer Technical Services, Inc.

Atlantic Richfield Company

Mike Mc Anulty
Liability Manager

317 Anaconda Road
Butte MT 59701
Direct (406) 782-9964
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September 14, 2021

Nikia Greene
Remedial Project Manager
US EPA – Montana Office
Baucus Federal Building
10 West 15th Street, Suite 3200
Helena, Montana 59626

Erin Agee
Senior Assistant Regional Counsel
US EPA Region 8 Office of Regional Counsel
CERCLA Enforcement Section
1595 Wynkoop Street
Denver, CO 80202
Mail Code: 8ORC-C

Daryl Reed
DEQ Project Officer
P.O. Box 200901
Helena, Montana 59620-0901

Jonathan Morgan, Esq.
DEQ, Legal Counsel
P.O. Box 200901
Helena, Montana 59620-0901

RE: Butte Priority Soils Operable Unit (BPSOU) Final Reclaimed Areas Maintenance and Monitoring Quality Assurance Project Plan – 2021

Agency Representatives:

On behalf of Atlantic Richfield Company and Butte-Silver Bow (BSB) the Butte Priority Soils Operable Unit (BPSOU) Final Reclaimed Areas Maintenance and Monitoring Quality Assurance Project Plan – 2021 and crosswalk is being submitted to address Agency comments provided September 7, 2021.

Pages that changed per Agency comments are provided along with the full PDF and crosswalk at the link below:

https://pioneertechnicalservices.sharepoint.com/:f:/s/submitted/EmN1LFF9ADFAoqNlFy0w3X4BQa63rzJXznC4i5IgcYyrAg

If you have any questions or comments, please call me at (907) 355-3914.

Sincerely,

Mike McAnulty

Mike Mc Anulty
Liability Manager
Remediation Management Services Company
An affiliate of **Atlantic Richfield Company**

Eric Hassler Program Director
Department of Reclamation and
Environmental Services
Butte-Silver Bow

Cc: Patricia Gallery / Atlantic Richfield - email
Chris Greco / Atlantic Richfield – email
Josh Bryson / Atlantic Richfield - email
Mike Mc Anulty / Atlantic Richfield - email
Loren Burmeister / Atlantic Richfield – email
Dave Griffis / Atlantic Richfield - email
Jean Martin / Atlantic Richfield - email
Irene Montero / Atlantic Richfield - email
David A. Gratson / Environmental Standards / email
Mave Gasaway / DGS - email
John Davis / PRR - email
Joe Vranka / EPA - email
David Shanight / CDM - email
Curt Coover / CDM - email
James Freeman / DOJ - email
John Sither / DOJ - email
Jenny Chambers / DEQ - email
Dave Bowers / DEQ - email
Carolina Balliew / DEQ - email
Matthew Dorrington / DEQ - email
Jim Ford / NRDP - email
Ray Vinkey / NRDP - email
Harley Harris / NRDP - email
Katherine Hausrath / NRDP - email
Meranda Flugge / NRDP - email
Ted Duaime / MBMG - email
Gary Icopini / MBMG - email
Becky Summerville / MR - email
Kristen Stevens / UP - email

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Matthew Mavrinac / RARUS - email
Harrison Roughton / RARUS - email
Brad Gordon / RARUS - email
Mark Neary / BSB - email
Eric Hassler / BSB - email
Julia Crain / BSB - email
Chad Anderson / BSB - email
Brandon Warner / BSB – email
Abigail Peltomaa / BSB - email
Eileen Joyce / BSB – email
Sean Peterson/BSB – email
Gordon Hart / BSB – email
Jeremy Grotbo / BSB – email
Josh Vincent / WET - email
Craig Deeney / TREC - email
Scott Bradshaw / TREC - email
Brad Archibald / Pioneer - email
Pat Sampson / Pioneer - email
Mike Borduin / Pioneer - email
Joe McElroy / Pioneer – email
Andy Dare / Pioneer – email
Karen Helfrich / Pioneer - email
Leesla Jonart / Pioneer - email
Connie Logan/ Pioneer – email
Ian Magruder/ CTEC- email
CTEC of Butte – email
Scott Juskiewicz / Montana Tech – email

File: MiningSharePoint@bp.com - email
BPSOU SharePoint - upload

SILVER BOW CREEK/BUTTE AREA NPL SITE
BUTTE PRIORITY SOILS OPERABLE UNIT

2021

Final

Reclaimed Areas
Maintenance and Monitoring
Quality Assurance Project Plan (QAPP)

Butte Silver Bow

and

Atlantic Richfield Company

September 2021

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 8, MONTANA OFFICE
FEDERAL BUILDING, 10 West 15TH Street, Suite 3200
Helena, MT 59626-0096
Phone 866-457-2690
www.epa.gov/region8

Ref: 8MO

September 7, 2021

Mr. Mike McAnulty
Liability Manager
Atlantic Richfield Company
317 Anaconda Road
Butte, Montana 59701

Eric Hassler
Superfund Program Manager
Butte-Silver Bow
155 W. Granite St., Room 108
Butte, Montana 59701

Re: Comment letter for the Butte Priority Soils Operable Unit (BPSOU) 2021 Final Reclaimed Areas Maintenance and Monitoring Quality Assurance Project Plan (QAPP) (dated June 25, 2021)

Dear Mike and Eric:

The U. S. Environmental Protection Agency (EPA), in consultation with the Montana Department of Environmental Quality (DEQ), is providing comments on the *Final Reclaimed Areas Maintenance and Monitoring Quality Assurance Project Plan (QAPP)*. Please see comments provided on the attached crosswalk and incorporate these comments and submit the final version of the plan for review.

If you have any questions or concerns, please call me at (406) 457-5019.

Sincerely,

NIKIA GREENE

Digitally signed by NIKIA GREENE
Date: 2021.09.07 10:30:41 -06'00'

Nikia Greene
Remedial Project Manager

cc: (email only)
Butte File
Jenny Chambers; DEQ
Matt Dorrington, DEQ

Daryl Reed; DEQ
Jon Morgan; DEQ counsel
Carolina Balliew; DEQ
Harley Harris; NRDP
Katherine Hausrath; NRDP
Jim Ford; NRDP
Ray Vinkey; NRDP
John Gallagher; BSBC
Eileen Joyce; BSBC
Sean Peterson; BSBC
Eric Hassler; BSBC
Brandon Warner; BSBC
Chad Anderson; BSBC
Karen Sullivan; BSBC
Julia Crain; BSBC
Abby Peltomaa; BSBC
Jeremy Grotbo; BSBC
Anne Walsh; UP
Robert Bylsma; UP counsel
Leo Berry; BNSF and UP counsel
Mark Engdahl; BNSF
Brooke Kuhl; BNSF counsel
Jeremie Maehr; Kennedy Jenks for BNSF and UP
Annika Silverman; Kennedy Jenks for BNSF and UP
Bob Andreoli; Patroit/RARUS
Becky Summerville; counsel for Inland Properties Inc.
Robert Lowry, BNSF counsel
Loren Burmeister; AR
Josh Bryson; AR
Mike Mcanulty; AR
Dave Griffis; AR
Jean Martin; Counsel AR
Mave Gasaway; attorney for AR
Adam Cohen; Counsel for AR
Pat Sampson; Pioneer for AR
Scott Bradshaw; TREC
Mike Borduin; Pioneer for AR
Karen Helfrich; Pioneer for AR
Andy Dare; Pioneer for AR
Scott Sampson; Pioneer for AR
Brad Archibald; Pioneer for AR
Andy Dare; Pioneer for AR
Tina Donovan; Woodardcurran for AR
Don Booth; AR consultant
Ted Duaime; MBMG
Gary Icopini; MBMG
David Shanight, CDM Smith

Curt Coover, CDM Smith
Chapin Storrar; CDM Smith
Erin Agee, EPA
Joe Vranka; EPA
Chris Wardell; EPA
Dana Barnicoat; EPA
Charlie Partridge; EPA
Jean Belille; EPA
Ian Magruder; CTEC (Tech Advisor)
Janice Hogan; CTEC
Kristi Carroll; Montana Tech Library

EPA REGION 8 QA DOCUMENT REVIEW CROSSWALK

QAPP/FSP/SAP for: *(check appropriate box)* ☐ **GRANTEE** ☐ **CONTRACTOR** ☐ **EPA** ☐ **Other**	**Entity** *(grantee, contract, EPA AO, EPA Program, Other)* AR and BSB County	**Regulatory Authority** **and/or** **Funding Mechanism**	**___ 2 CFR 1500 for Grantee/Cooperative Agreements** **___ 48 CFR 46 for Contracts** **___ Interagency Agreement (FFA, USGS,)** **___ EPA/Court Order** **___ EPA Program Funding** **___ EPA Program Regulation** **___ EPA CIO 2105**
Document Title ***[Note: Title will be repeated in Header]***	BPSOU Final Reclaimed Areas Maintenance and Monitoring QAPP - 2021		
QAPP/FSP/SAP Preparer	AR and BSB County		
Period of Performance *(of **QAPP**/FSP/SAP)*	**2021-2022**	**Date Submitted for Review**	6/25/2021
EPA Project Officer **EPA Project Manager**	Nikia Greene	**PO Phone #** **PM Phone #**	
QA Program Reviewer or Approving Official	Nikia Greene	**Date of Review**	8/16/2021

Documents Submitted for QAPP Review* (QA Reviewer must complete)*:

1. QA Document(s) submitted for review:

QA Document	**Document Date**	**Document Stand-alone**	**Document with QAPP**
QAPP	**6/25/2021**	**Yes** / No	
FSP		Yes / No	Yes / No
SAP		Yes / No	Yes / No
SOP(s)	**(attached)**		**Yes** / No

2. WP/SOW/TO/PP/RP Date ___________
WP/SOW/TO/RP Performance Period _____________

3. QA document consistent with the:
WP/SOW/PP for grants? Yes / No
SOW/TO for contracts? Yes / No

4. QARF signed by R8 QAM Yes / No / NA
Funding Mechanism IA / contract / grant / NA
Amount _____________

Notes for Document Submittals:

1. A QAPP written by a Grantee, EPA, or Federal Partner must include for review: Work Plan(WP) / Statement of Work (SOW) / Program Plan (PP) / Research Proposal (RP) and funding mechanism
2. A QAPP written by Contractor must include for review:
   - **a)** Copy of Task Order Work Assignment/SOW
   - **b)** Reference to a hard or electronic copy of the contractor's approved QMP
   - **c)** Copy of Contract SOW if no QMP has been approved
   - **d)** Copy of EPA/Court Order, if applicable
   - **e)** The QA Review must determine (with the EPA CO or PO) if a QARF was completed for the environmental data activity described in the QAPP.
3. **a**. Field Sampling Plan (FSP) and/or Sampling & Analyses Plan (SAP) must include the Project QAPP ***or*** must be a stand-alone QA document that contain all QAPP required elements (Project Management, Data Generation/Acquisition, Assessment and Oversight, and Data Validation and Usability).
   b. SOPs must be submitted with a QA document that contains all QAPP required elements.

Summary of Comments *(highlight significant concerns/issues)***:**

1. **The** AR and BSB County **must address the comments in the Summary of Comments, as well as those identified in the Comment section(s) that includes a "Response (date)" and Resolved (date)".**

2. AR and BSB submitted an older version of the QAPP crosswalk, therefore this crosswalk was revised to include the most updated version (Update #6 7-2017).
3. Please provide a clear reference for the BPSOU Monitoring and Maintenance Plan (M&M Plan) referred to in Section 1.0 Introduction and where the document is located.

   Some Page/Section location references were revised to include correct locations of required text/information.

Element	**Acceptable** *Yes/No/NA*	**Page/ Section**	**Comments**
A. Project Management			
A1. Title and Approval Sheet			
a. Contains project title	Yes	Title page and page i	EPA: No comments
b. Date and revision number line (for when needed)	Yes	Title page and page i	EPA: No comments
c. Indicates organization's name	Yes	Title page	EPA: No comments
d. Date and signature line for organization's project manager	Yes	Page i	EPA: No comments
e. Date and signature line for organization's QA manager	No	Page i	EPA: 2018 Crosswalk lists an EPA comment stating to add "Quality Assurance Approval Official" to Nikia Greene's signature line. For this 2021 QAPP please add "Delegated Approving Officer" to the signature line. **Atlantic Richfield Response: The requested text has been added.**
f. Other date and signatures lines, as needed	Yes	Page i	EPA: No comments
A2. Table of Contents			
a. Lists QA Project Plan information sections	Yes	Pages v to vii	EPA: No comments
b. Document control information indicated	Yes	Page vii	EPA: No comments
A3. Distribution List			
Includes all individuals who are to receive a copy of the QA Project Plan and identifies their organization	Yes	Pages ii to iv	EPA: No comments
A4. Project/Task Organization			
a. Identifies key individuals involved in all major aspects of the project, including contractors	Yes	Sections 2.0 through 2.3	EPA: No comments
b. Discusses their responsibilities	Yes	Sections 2.0 through 2.3	EPA: No comments
c. Project QA Manager position indicates independence from unit generating data	Yes	Section 2.2, Figure 2	EPA: No comments
d. Identifies individual responsible for maintaining the official, approved QA Project Plan	Yes	Section 2.3	EPA: No comments

e. Organizational chart shows lines of authority and reporting responsibilities	No	Figure 2	EPA: Please add text “Figure 2” to BPSOU Reclaimed Areas Program Organization and Communication Structure **Atlantic Richfield Response: The requested text has been added.**
A5. Problem Definition/Background			
a. States decision(s) to be made, actions to be taken, or outcomes expected from the information to be obtained	Yes	Sections 1.0 and 2.4	EPA: No comments
b. Clearly explains the reason (site background or historical context) for initiating this project	Yes	Sections 2.4 and 2.5	EPA: No comments
c. Identifies regulatory information, applicable criteria, action limits, etc. necessary to the project	Yes	Sections 2.4 and 2.5	EPA: No comments
A6. Project/Task Description			
a. Summarizes work to be performed, for example, measurements to be made, data files to be obtained, etc., that support the project=s goals	Yes	Section 2.5	EPA: No comments
b. Provides work schedule indicating critical project points, e.g., start and completion dates for activities such as sampling, analysis, data or file reviews, and assessments	Yes	Section 2.5	EPA: No comments
c. Details geographical locations to be studied, including maps where possible	Yes	Section 2.5	EPA: No comments
d. Discusses resource and time constraints, if applicable	Yes	Section 2.5	EPA: No comments
A7. Quality Objectives and Criteria			
a. Identifies - performance/measurement criteria for all information to be collected and acceptance criteria for information obtained from previous studies, - including project action limits and laboratory detection limits and - range of anticipated concentrations of each parameter of interest	Yes	Section 2.6	EPA: No comments
b. Discusses precision	Yes	Section 2.6.2	EPA: No comments
c. Addresses bias	Yes	Section 2.6.2	EPA: No comments
d. Discusses representativeness	Yes	Section 2.6.2	EPA: No comments
e. Identifies the need for completeness	Yes	Section 2.6.2	EPA: No comments
f. Describes the need for comparability	Yes	Section 2.6.2	EPA: No comments
g. Discusses desired method sensitivity	Yes	Section 2.6.2	EPA: No comments

A8. Special Training/Certifications			
a. Identifies any project personnel specialized training or certifications	Yes	Section 2.7	EPA: No comments
b. Discusses how this training will be provided	Yes	Section 2.7	EPA: No comments
c. Indicates personnel responsible for assuring training/certifications are satisfied	Yes	Section 2.7	EPA: No comments
d. identifies where this information is documented	Yes	Section 2.7	EPA: No comments
A9. Documentation and Records			
a. Identifies report format and summarizes all data report package information	Yes	Section 2.8	EPA: No comments
b. Lists all other project documents, records, and electronic files that will be produced	Yes	Section 2.8	EPA: No comments
c. Identifies where project information should be kept and for how long	Yes	Section 2.8	EPA: No comments
d. Discusses back up plans for records stored electronically	Yes	Section 2.8	EPA: No comments
e. States how individuals identified in A3 will receive the most current copy of the approved QA Project Plan, identifying the individual responsible for this	Yes	Section 2.8	EPA: No comments
B. Data Generation/Acquisition			
B1. Sampling Process Design (Experimental Design)			
a. Describes and justifies design strategy, indicating size of the area, volume, or time period to be represented by a sample	Yes	Section 3.3	EPA: No comments
b. Details the type and total number of sample types/matrix or test runs/trials expected and needed	Yes	Section 3.3	EPA: No comments
c. Indicates where samples should be taken, how sites will be identified/located	Yes	Section 3.3.1, Attachment 1	EPA: No comments
d. Discusses what to do if sampling sites become inaccessible	NA	NA	EPA: This is not an anticipated issue.
e. Identifies project activity schedules such as each sampling event, times samples should be sent to the laboratory, etc.	Yes	Section 2.5.1 and 2.5.2.1	EPA: No comments
f. Specifies what information is critical and what is for informational purposes only	Yes	Section 3.2	EPA: No comments
g. Identifies sources of variability and how this variability should be reconciled with project information	Yes	Step 6	EPA: No comments
B2. Sampling Methods			

a. Identifies all sampling SOPs by number, date, and regulatory citation, indicating sampling options or modifications to be taken	Yes	Section 3.3	EPA: No comments
b. Indicates how each sample/matrix type should be collected	Yes	Section 3.3.1	EPA: No comments
c. If in situ monitoring, indicates how instruments should be deployed and operated to avoid contamination and ensure maintenance of proper data	NA	NA	EPA: No in-situ instruments will be deployed
d. If continuous monitoring, indicates averaging time and how instruments should store and maintain raw data, or data averages	NA	NA	EPA: No continuous monitoring instruments will be deployed
e. Indicates how samples are to be homogenized, composited, split, or filtered, if needed	Yes	Section 3.4.1	EPA: No comments
f. Indicates what sample containers and sample volumes should be used	Yes	Section 3.3.1 and 3.6.1	EPA: No comments
g. Identifies whether samples should be preserved and indicates methods that should be followed	Yes	Section 3.3.1	EPA: No comments
h. Indicates whether sampling equipment and samplers should be cleaned and/or decontaminated, identifying how this should be done and by-products disposed of	Yes	Section 3.4.41	EPA: No comments
i. Identifies any equipment and support facilities needed	Yes	Section 3.5.1	EPA: No comments
j. Addresses actions to be taken when problems occur, identifying individual(s) responsible for corrective action and how this should be documented	Yes	Section 3.11	EPA: No comments
B3. Sample Handling and Custody			
a. States maximum holding times allowed from sample collection to extraction and/or analysis for each sample type and, for in-situ or continuous monitoring, the maximum time before retrieval of information	Yes	Section 3.4.2	EPA: No comments
b. Identifies how samples or information should be physically handled, transported, and then received and held in the laboratory or office (including temperature upon receipt)	Yes	Section 3.4.2	EPA: No comments
c. Indicates how sample or information handling and custody information should be documented, such as in field notebooks and forms, identifying individual responsible	Yes	Section 3.4.2	EPA: No comments

d. Discusses system for identifying samples, for example, numbering system, sample tags and labels, and attaches forms to the plan	Yes	Section 3.4	EPA: No comments
e. Identifies chain-of-custody procedures and includes form to track custody	Yes	Section 3.4.2	EPA: No comments
B4. Analytical Methods			
a. Identifies all analytical SOPs (field, laboratory and/or office) that should be followed by number, date, and regulatory citation, indicating options or modifications to be taken, such as sub-sampling and extraction procedures	Yes	Section 3.3.1 and 3.5	EPA: No comments
b. Identifies equipment or instrumentation needed	Yes	Section 3.3.2	EPA: No comments
c. Specifies any specific method performance criteria	Yes	Section ~~3.4.3 and~~ 3.5	EPA: No comments
d. Identifies procedures to follow when failures occur, identifying individual responsible for corrective action and appropriate documentation	Yes	Section 3.5	EPA: No comments
e. Identifies sample disposal procedures	Yes	Section 3.4.3	EPA: No comments
f. Specifies laboratory turnaround times needed	Yes	Section 3.5	EPA: No comments
g. Provides method validation information and SOPs for nonstandard methods	Yes	Section 5.0	EPA: No comments
B5. Quality Control			
a. For each type of sampling, analysis, or measurement technique, identifies QC activities which should be used, for example, blanks, spikes, duplicates, etc., and at what frequency	Yes	Section 3.3.1 and 3.7	EPA: No comments
b. Details what should be done when control limits are exceeded, and how effectiveness of control actions will be determined and documented	Yes	Section 3.7.2	EPA: No comments
c. Identifies procedures and formulas for calculating applicable QC statistics, for example, for precision, bias, outliers and missing data	Yes	Section 2.8.7	EPA: No comments
B6. Instrument/Equipment Testing, Inspection, and Maintenance			
a. Identifies field and laboratory equipment needing periodic maintenance, and the schedule for this	Yes	Section 3.8	EPA: No comments
b. Identifies testing criteria	Yes	Section 3.8	EPA: No comments
c. Notes availability and location of spare parts	Yes	Section 3.8	EPA: No comments

d. Indicates procedures in place for inspecting equipment before usage	Yes	Section 3.8	EPA: No comments
e. Identifies individual(s) responsible for testing, inspection and maintenance	Yes	Section 3.8	EPA: No comments
f. Indicates how deficiencies found should be resolved, re-inspections performed, and effectiveness of corrective action determined and documented	Yes	Section 3.8	EPA: No comments
B7. Instrument/Equipment Calibration and Frequency			
a. Identifies equipment, tools, and instruments that should be calibrated and the frequency for this calibration	Yes	Section 3.7.5	EPA: No comments
b. Describes how calibrations should be performed and documented, indicating test criteria and standards or certified equipment	Yes	Section 3.7	EPA: No comments
c. Identifies how deficiencies should be resolved and documented	Yes	Section 3.7.2	EPA: No comments
B8. Inspection/Acceptance for Supplies and Consumables			
a. Identifies critical supplies and consumables for field and laboratory, noting supply source, acceptance criteria, and procedures for tracking, storing and retrieving these materials	Yes	Section 3.9	EPA: No comments
b. Identifies the individual(s) responsible for this	Yes	Section 3.9	EPA: No comments
B9. Use of Existing Data (Non-direct Measurements)			
a. Identifies data sources, for example, computer databases or literature files, or models that should be accessed and used	Yes	Section 3.10	EPA: No comments
b. Describes the intended use of this information and the rationale for their selection, i.e., its relevance to project	Yes	Section 3.10	EPA: No comments
c. Indicates the acceptance criteria for these data sources and/or models	Yes	Section 3.10	EPA: No comments
d. Identifies key resources/support facilities needed	Yes	Section 3.10	EPA: No comments
e. Describes how limits to validity and operating conditions should be determined, for example, internal checks of the program and Beta testing	Yes	Section 3.10	EPA: No comments
B10. Data Management			
a. Describes data management scheme from field to final use and storage	Yes	Section 3.11	EPA: No comments

b. Discusses standard record-keeping and tracking practices, and the document control system or cites other written documentation such as SOPs	Yes	Section 3.11, Attachment 3.51	EPA: No comments
c. Identifies data handling equipment/procedures that should be used to process, compile, analyze, and transmit data reliably and accurately	Yes	Section 3.11, Attachment 3.51	EPA: No comments
d. Identifies individual(s) responsible for this	Yes	Section 3.11	EPA: No comments
e. Describes the process for data archival and retrieval	Yes	Section 3.11, Attachment 3.51	EPA: No comments
f. Describes procedures to demonstrate acceptability of hardware and software configurations	Yes	Section 3.11	EPA: No comments
g. Attaches checklists and forms that should be used	Yes	Section 3.11, Attachment 3.51	EPA: No comments
C. Assessment and Oversight			
C1. Assessments and Response Actions			
a. Lists the number, frequency, and type of assessment activities that should be conducted, with the approximate dates	Yes	Section 4.0	EPA: No comments
b. Identifies individual(s) responsible for conducting assessments, indicating their authority to issue stop work orders, and any other possible participants in the assessment process	Yes	Section 4.0	EPA: No comments
c. Describes how and to whom assessment information should be reported	Yes	Section 4.1 and 4.2	EPA: No comments
d. Identifies how corrective actions should be addressed and by whom, and how they should be verified and documented	Yes	Section 4.1 and 4.2	EPA: No comments
C2. Reports to Management			
a. Identifies what project QA status reports are needed and how frequently	Yes	Section 4.3	EPA: No comments
b. Identifies who should write these reports and who should receive this information	Yes	Section 4.3	EPA: No comments
D. Data Validation and Usability			
D1. Data Review, Verification, and Validation			
Describes criteria that should be used for accepting, rejecting, or qualifying project data	Yes	Section 5.0	EPA: No comments

D2. Verification and Validation Methods			
a. Describes process for data verification and validation, providing SOPs and indicating what data validation software should be used, if any	No	Section 5.0	EPA: Please correct reference for the *National Functional Guidelines for Inorganic Superfund Methods Data Review (EPA, 2016b).* The most current version should be dated as November 2020. **Atlantic Richfield Response: The requested update has been made.**
b. Identifies who is responsible for verifying and validating different components of the project data/information, for example, chain-of-custody forms, receipt logs, calibration information, etc.	Yes	Section 5.0	EPA: No comments
c. Identifies issue resolution process, and method and individual responsible for conveying these results to data users	Yes	Section 5.0	EPA: No comments
d. Attaches checklists, forms, and calculations	Yes	Section 5.0	EPA: No comments
D3. Reconciliation with User Requirements			
a. Describes procedures to evaluate the uncertainty of the validated data	Yes	Section 5.0	EPA: No comments
b. Describes how limitations on data use should be reported to the data users	Yes	Section 5.0	EPA: No comments

SILVER BOW CREEK/BUTTE AREA NPL SITE
BUTTE PRIORITY SOILS OPERABLE UNIT

2021

Final

Reclaimed Areas Maintenance and Monitoring Quality Assurance Project Plan (QAPP)

Prepared for:

Butte Silver Bow
Superfund Division
155 W. Granite
Butte, MT 59701

and

Atlantic Richfield Company
317 Anaconda Road
Butte, Montana 59701

Prepared by:

Pioneer Technical Services, Inc.
1101 S. Montana Street
Butte, Montana 59701

September 2021

APPROVAL PAGE

BPSOU Reclaimed Areas Maintenance and Monitoring
Quality Assurance Project Plan
Butte Area NPL Site

Approved: ______________________________ Date: __________
Nikia Greene, Delegated Approval Officer, EPA, Region 8

Approved: ______________________________ Date: __________
Daryl Reed, Project Officer, Montana DEQ

Approved: Mike McAnulty Date: 9/14/2021
Mike Mc Anulty, Liability Manager
Atlantic Richfield Company

Approved: Laura Moon Date: 9/14/2021

Laura Moon, Quality Assurance Manager
Pioneer Technical Services
for Atlantic Richfield Company

2021
Plan is effective on date of approval.

DOCUMENT REVISION SUMMARY

Revision No.	Author	Description	Date
	Pioneer Technical Services, Inc.	Annual Update	September 2021

DISTRIBUTION LIST

BPSOU Reclaimed Areas Monitoring and Maintenance
Quality Assurance Project Plan
Butte Area NPL Site

Key Personnel QAPP Recipients	Title	Organization	Telephone Number	E-mail Address
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TABLE OF CONTENTS

LIST OF FIGURES

LIST OF TABLES

LIST OF ATTACHMENTS

LIST OF ACRONYMS

Acronym	Definition	Acronym	Definition
°C	degrees Celsius	**MS**	matrix spike
ASA	American Society of Agronomy	**MSD**	matrix spike duplicate
bgs	Below Ground Surface	**NIST**	National Institute of Standards and Testing
BPSOU	BPSOU Butte Priority Soils Operable Unit	**NPL**	National Priorities List
BHRS	Butte Hill Revegetation Specifications	**O&M**	Operation and Maintenance
BRES	Butte Reclamation Evaluation System	**PDF**	Portable Document Format
BSB	Butte-Silver Bow	**QA**	Quality Assurance
CAP	Corrective action plan	**QAM**	Quality Assurance Manager
CAR	Corrective Action Report	**QAO**	Quality Assurance Officer
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act	**QAPP**	Quality Assurance Project Plan
CLP	Contract Laboratory Program	**QC**	Quality Control
CoC	Chain of custody	**QMP**	Quality Management Plan
DEQ	Montana Department of Environmental Quality	**RCRA**	Resource Conservation and Recovery Act
DMP	Data Management Plan	**RI**	Reclamation improvement
DQO	Data Quality Objectives	**ROD**	Record of Decision
EC	Electrical conductivity	**RPD**	relative percent difference
EDD	Electronic Data Deliverable		
EPA	U.S. Environmental Protection Agency	**SAR**	Sodium adsorption ratio
FSP	Field Sampling Plan	**SOP**	Standard Operating Procedure
ESD	Explanation of Significant Differences	**SOW**	Statement of Work
GIS	Geographical Information System	**SMP**	standard maintenance procedure
GPS	Global Positioning System	**SRM**	standard reference material
HAZWOPER	Hazardous Waste Operations and Emergency Response	**SSHASP**	Site-Specific Health and Safety Plan
ICP-MS	Inducted Coupled Plasma-Mass Spectrometry	**SSSA**	Soil Science Society of America
LAP	laboratory analytical protocol	**VI**	vegetative improvements
LCS	laboratory control spike	**USDA**	United States Department of Agriculture
M&M	Maintenance and Monitoring	**USGS**	U.S. Geological Survey
mg/kg	milligram per kilogram	**XRF**	X-ray fluorescence

1.0 INTRODUCTION

To ensure performance standards achieved through remedial action are upheld, reclaimed areas (shown in Figure 1 and listed in Attachment 1) are monitored according to the Butte Reclamation Evaluation System (BRES), which is attached to the U.S. Environmental Protection Agency (EPA) Record of Decision (ROD) as Appendix E (EPA, 2006a), and referred to in this document as BRES; and maintained as described in the Butte Priority Soils Operable Unit (BPSOU) Butte Reclaimed Areas Maintenance and Monitoring (M&M) Plan (M&M Plan) (Atlantic Richfield, 2018a)[1]. The BRES is the governing guidance document that all reclaimed areas in the BPSOU must follow as described in the EPA ROD. The BRES sets the methodology for evaluating the reclaimed areas and provides guidelines for corrective actions. The M&M Plan details the means and methods necessary to maintain reclaimed areas consistently to ensure the stability and integrity of those areas. Standard maintenance procedures (SMPs) provided in the M&M Plan provide assurance that maintenance performed on reclaimed areas is completed to a sufficient level that will continue to protect human health and the environment over the long term.

Individual site monitoring is performed by an independent third party in accordance with BRES, and the corresponding report provided to Butte-Silver Bow (BSB) (Appendix E) for review. As appropriate, BSB will initiate corrective action if necessary. Institutional control programs related to remedial activities are described in the latest version of the Atlantic Richfield BPSOU *Institutional Controls Implementation and Assurance Plan* (Atlantic Richfield, 2019).

1.1 Purpose

The BPSOU *Quality Management Plan* (QMP) (Atlantic Richfield, 2016) provides the overarching guidance to ensure collection of environmental data for the BPSOU meets requirements mandated by the EPA. This Quality Assurance Project Plan (QAPP) provides guidance for monitoring and maintenance activities and limited sampling and analyses and describes the quality assurance/quality control (QA/QC) policies and procedures to be implemented during routine data collection and analyses specific to BRES evaluations and maintenance of reclaimed areas. This QAPP has been developed in accordance with the EPA Requirements for QAPPs, EPA QA/R-5 (EPA, 2001), and the *Guidance on Systematic Planning Using the Data Quality Objectives (DQO) Process,* EPA QA/G4 (EPA, 2006b). This QAPP includes the following four key elements:

- Program management and objectives (Section 2.0).
- Measurement and data acquisition (Section 3.0).
- Assessment and oversight (Section 4.0). and
- Data review and usability (Section 5.0).

The sections below describe these key elements and detail any required planning, monitoring, sampling, and analyses. Sections in this QAPP expand on or reference information in other site-wide documents to comply with the Uniform Federal Policy for QAPPs (EPA, 2005) and to present project-specific requirements.

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[1] A Revised Draft Final Butte Reclaimed Areas Maintenance and Monitoring Plan is scheduled to be submitted as an appendix to the Solid Media Management Program Plan in 2021.

1.2 Databases

Within the program, there are a variety of databases that store reclaimed area information, Geographical Information System (GIS) locations, soil sample results, and other project data. Various individuals, from field personnel to operations personnel to data administrators, enter and manage the data (details are listed in sections 3-6). The database names used in this report are generalized as the program or project database, GIS database, reclamation database, or soils database. For specific information on the databases, refer to the current BPSOU Data Management Plan (DMP) (Atlantic Richfield, 2018b), referred to in this report as the BPSOU DMP.

2.0 PROGRAM MANAGEMENT

This section addresses the BPSOU reclaimed areas program (Program) and project administrative functions as well as project background, objectives, and documentation requirements for maintenance, monitoring, sampling, and analysis activities on each project site. Figure 2 shows the program organization and communication structure.

2.1 Agency Oversight

The EPA and Montana Department of Environmental Quality (DEQ) (the *Agencies*) are responsible for project oversight, review, and approval of site-specific remediation plans. The Agencies also review sampling results and review and approve project reports described in Section 2.5.3.

2.2 Atlantic Richfield Company

Atlantic Richfield Company (Atlantic Richfield) confirms conformance to the BRES and Reclaimed Areas M&M Plan (Figure 2).

Atlantic Richfield Operations Liability Manager

The Atlantic Richfield Liability Manager, Mike Mc Anulty, monitors the performance of the contractor(s), consults with the Contractor Project Manager(s) and QA officer(s) on deficiencies and aids in finalizing resolution actions, and reviews all reclamation activities under the Program. An Atlantic Richfield project representative, or designated alternate, can perform a site walk-through and assist with preparation of a site-specific work plan prior to implementation, or provide confirmation of all reclamation performed.

Atlantic Richfield Operations Quality Assurance Manager

The Atlantic Richfield QA Manager, David Gratson, (QAM) interfaces with the Liability Manager on company policies regarding quality. The QAM has the authority and responsibility to approve specific QA documents including this QAPP.

2.3 Butte Silver Bow Department of Reclamation and Environmental Services Organization and Responsibilities

The BSB Department of Reclamation and Environmental Services performs all associated maintenance and monitoring required to ensure reclaimed areas in BPSOU remain protective of human health and the environment. Key individuals comprising the BSB Department of Reclamation and Environmental Services are shown on Figure 2 and responsibilities are described below.

Program Director

The Program Director, Eric Hassler, oversees all activities and implementation of remedial actions throughout the department related to Superfund.

Assistant Program Director

The Assistant Program Director, Julia Crain, assumes the role of QAM and is responsible for assuring the quality of all field data, completing QC activities, reviewing and maintaining laboratory data packages, compiling an Annual Summary Report, maintaining quality records and managing program data (as described in Section 2.8.7), and reporting final remediated property requirements to the Agencies. The Annual Summary Report will include figures displaying site sample location, analytical results, and copies of all field data.

Environmental and Operation and Maintenance Manager

The Environmental and Operation and Maintenance (O&M) Division Manager (O&M Manager), Brandon Warner, assumes the role of Project Manager for reclaimed areas monitoring, maintenance, and end-use compliance. The Project Manager is responsible for maintaining the official approved QAPP, scheduling all work to be completed, and ensuring that the work is performed in accordance with the requirements contained herein. The O&M Manager/Project Manager is also responsible for consulting with the BSB Program Director and/or Assistant Program Director regarding any project deficiencies and resolutions.

Data Management Division Manager

The Data Management Division Manager, Abby Peltomaa, assumes the role of QA Officer (QAO) and ensures data quality, verification, and validation is completed per the project QAPP. The QAO may also be part of the review team for project final reports. The QAO is responsible for evaluating information from instances of nonconformance, inspection reports, surveillance reports, audit and assessment reports, quality system reviews (QSRs), corrective action reports (CARs), and other sources. The QAO, in support of the QAM, may review assigned project QAPPs and associated Standard Operating Procedure (SOPs) annually and verify that the current project QAPP and SOPs are available.

GIS Data Specialist

The GIS Data Specialist, Jeremy Grotbo, will coordinate with the QAM to ensure up to date GIS data are verified and maintained in the project database. The GIS Data Specialist will be responsible for maintaining GIS data such as site boundaries, updating proposed changes to site boundaries as described within standard procedures, and providing notification of updates to team members.

Field Evaluation Teams

Field evaluation teams perform annual evaluations of reclaimed areas as described in the BRES. The field evaluation team is responsible for completing training modules lead by BSB prior to performing field evaluations and performing field evaluations as prescribed in the BRES. Field evaluators provide an independent third-party perspective of reclaimed area performance.

Contractor

Atlantic Richfield and/or BSB may assign a Contractor to be responsible for completing individual site investigations.

Contractor Project Manager (CPM)

The CPM is responsible for scheduling all sampling work to be completed and ensuring that the work is performed in accordance with the requirements contained herein. The CPM is also responsible for consulting with the specific project QA personnel regarding any deficiencies and finalizing resolution actions. The CPM for each project will be listed in the supporting documents for each project area under this QAPP.

Field Team Leader

The Field Team Leader ensures that the QAPP for each project area has been reviewed by all members of the field team and that the QAPP is properly followed during field activities. The Field Team Leader will conduct daily safety meetings, assist in field activities, and document activities in the logbook.

The Field Team Leader is responsible for equipment, problem solving and decision making in the field, and for addressing technical aspects of the project. The Field Team Leader will provide "on-the-ground" overviews of project implementation by observing site activities to ensure compliance with technical project requirements, Health Safety Security and Environment (HSSE) requirements, and the Site-Specific Health and Safety Plan (SSHASP). Finally, the Field Team Leader is responsible for identifying potential Integrity Management (IM) issues, as appropriate, and preparing required project documentation.

Contractor Quality Assurance Officer (QAO)

The Contractor QAO is responsible for verifying effective implementation of QAPP requirements and procedures. This includes reviewing field and laboratory data and evaluating data quality. The Contractor QAO for each project will be listed in the supporting documents created for each project area under this QAPP and will be independent from the unit generating the data.

Safety and Health Manager

Where applicable the Safety and Health Manager is responsible for developing the SSHASP and reviewing it with all members of the field team. The Safety and Health Manager will lead applicable Task Risk Assessments and conduct the initial safety meeting prior to starting fieldwork. The Safety and Health Manager will ensure that work crews comply with all site safety and health requirements and will revise the SSHASP, if necessary.

Contract Laboratory

Any laboratory contracted to complete analyze samples must ensure that the laboratory's QA personnel are familiar with this QAPP and will perform the analytical and QC work as specified per laboratory methods and this QAPP. Contract laboratory QA personnel are responsible for reviewing final analytical reports produced by the laboratory, coordinating the laboratory analyses schedule, and supervising in-house chain-of-custody (CoC) procedures.

2.4 Problem Definition and Background

Land reclamation is a vital component of Superfund remedy across the BPSOU. The BRES sets the performance standards that all reclaimed areas in the BPSOU must achieve, evaluation methodology, and corrective action guidelines. Remediated source areas within BPSOU are monitored and evaluated as described in the BRES to confirm protectiveness of the remedy. The BRES is the evaluation tool used to ensure the integrity of the reclaimed lands is maintained over the long term.

This QAPP applies to monitoring and maintenance activities and limited sampling activities applicable to reclaimed areas (including insufficiently reclaimed areas) within the BPSOU Figure 1, and it was developed in response to the 2006 ROD (EPA, 2006a) and Explanation of Significant Differences (ESD) to the ROD (EPA, 2011).

2.5 Project Description and Schedule

Source areas associated with historic mining operations across BPSOU have been reclaimed to ensure protection of human health and the environment. Reclamation remedy includes removing waste material, placing fill material, adding cover soil, and adding growth media, or a combination thereof. Reclaimed areas must be monitored and evaluated to ensure the remedy performs as designed and remains protective of human health and the environment. As previously stated, the BRES is the evaluation tool for reclaimed areas across BPSOU.

The BRES evaluation method is specifically designed for the upland environment in Butte, Montana, and designed to address the diverse reclaimed land types and uses within the area. This tool provides methods and performance standards to accurately evaluate the integrity, stability, and protectiveness of reclaimed areas. The M&M Plan outlines the means and methods necessary to maintain reclaimed areas consistently.

Components of the BRES include monitoring, evaluation trigger items, and a long-term tracking and data management program. The long-term tracking and data management program ensures reclaimed areas are evaluated according to the rotating schedule, and issues are documented for corrective action, if necessary. The M&M Plan outlines appropriate guidance to perform maintenance and corrective actions.

2.5.1 Project Schedule

The BRES is used to continuously evaluate and prescribe corrective action of reclaimed sites within BPSOU in perpetuity. Reclaimed areas have been separated into quadrants representing approximately equivalent acreage to provide efficiency in performing M&M. Reclaimed sites within each quadrant are evaluated on a rotating schedule once every four years. Any corrective actions performed on reclaimed sites to address findings documented during BRES field evaluations are typically completed within a year of the evaluation, as weather and site conditions allow. Table 1 lists key application dates related to reclamation activities. Site evaluations and maintenance will continue in perpetuity, or as long as the EPA determines the program is required. The annual BRES evaluation, corrective action, and reporting cycle is illustrated in Figure 3.

Table 1. Project Tasks Performance Periods.

Task	*Application/Field Work	No Work Performed
Field Evaluations	May 1 - June 30	July 1 - April 30
Field Verification	June 1 - October 30	November 1 - May 31
Monitoring	March 2- November 30	December 1 - March 1
Capping	March 2- November 30	December 1 - March 1
Seeding	October 16 - 30; March 1 - June 14	June 15 - October 15
Fertilizing	October 16 - 30; March 1 - June 14	June 15 - October 15
Weed Spraying	March 2 - November 30	December 1 - March 1
	*subject to site and weather conditions, schedule may vary slightly	

2.5.2 Project Tasks

Project tasks are divided into site evaluations, maintenance fieldwork, and reporting as described in the section below. Performance periods for typical classifications of field work categories are provided in Table 1.

Site Evaluations

Annual BRES evaluations of reclaimed areas are conducted by an independent third party. Typically, the evaluations are performed during the spring period (as weather and site conditions allow) and are completed within 10-15 days of initiation, depending on the site conditions. Evaluators will have undergone program-specific training, led by BSB and EPA. The training will be held annually prior to conducting evaluations, and typically will take 1 standard workday (8 hours) to complete.

Site evaluations are performed to monitor and verify performance of site remedy and identify trigger items that require attention to ensure site protectiveness. Field personnel completing the evaluations enter the information into tablets linked to the project databases where it can be evaluated in real-time by BSB personnel (Section 3.11).

Site Maintenance
Maintenance performed as a result of annual field evaluations may include additional sampling, standard maintenance procedures, or implementation of site-specific corrective action plans (CAPs) to address trigger items. Personnel from BSB will perform or oversee all maintenance activities for reclaimed areas. Maintenance is typically completed within one year of the evaluation, as site accessibility and weather conditions allow.

2.5.3 Project Reporting

Personnel from BSB will perform all reporting activities described in this QAPP, and compile and submit the reports listed below to the Agencies by the dates listed in Table 2.

Table 2. Project Reporting Periods.

Reporting Periods	BSB Preparation	Review - Final Approval
Summary Report	May 1 - September 30	October 1 – April 30
Site-Specific Sampling and Analysis Plan	May 1 - September 30	October 1 – April 30
Corrective Actions Plan	September 1 - December 30	February 1 - March 1
Annual Operation and Maintenance Report	December 1 - March 30	March 30

Recommendations Summary Report – *Submitted by September 30*
A recommendation summary report will provide a summary of the annual BRES field evaluations and identify additional site-specific engineering evaluation or site sampling and analysis requirements. The report will provide a data summary of trigger items identified, erosion and vegetation scores, and proposed type of corrective action for each site evaluated and identify additional sampling or engineering evaluations as applicable. Summary reports will be in a tabular format for ease of review and quantification of findings. The reports will be submitted to the Agencies for review, comment, and approval.

Site-Specific Field Sampling Plan – *Submitted by September 30*
A site-specific field sampling plan (FSP) will be prepared to identify the sampling locations and required analysis. The specific FSP will be submitted to the Agencies for review, comment, and approval prior to commencement of field activities.

Corrective Action Plan – *Submitted by December 30*
The CAP will provide a detailed approach to correct trigger items identified in the field evaluation reports for sites requiring vegetative improvements (VIs), or reclamation improvements (RIs). The reports should also provide additional monitoring requirements to verify the effectiveness of the recommended corrective measures. Site-specific data and sampling results (historic and current data) will be included with report to support proposed corrective action. The reports will be submitted to the Agencies for review, comment, and approval.

Annual Summary Report – ***Submitted by March 30 (following year)***
The annual summary report will provide a final summary of work completed in the previous year. The report will include summaries of annual BRES evaluations, including site-specific trigger items, approved CAPs, and details of work completed for each site. Each annual report will include work completion summaries and typically include documentation of the materials used, their source, quantity, and final site condition. The report will describe specific field activities performed during implementation of the QAPP and the characteristics of the remedial action completed. The annual summary report will also serve as the construction completion report for field maintenance activities. The final approved report will be stored on the GIS server and the associated data stored in the soils database and the Program database tracking system (refer to the BPSOU DMP).

2.6 Quality Objectives and Criteria

This section discusses the internal QC and review procedures used to ensure that all data collected for this project are of sufficient quality and are reputable. The Data Quality Objectives (DQOs) were developed in accordance with the EPA's Guidance on Systematic Planning Using the DQO Process (EPA, 2006b). The DQOs are statements that define the type, quality, quantity, purpose, and use of data to be collected. The EPA developed a seven-step process to establish DQOs to help ensure that data collected during a field event are adequate to support reliable site-specific decision making (EPA, 2001 and EPA, 2006b). The sections below outline the QAPP DQOs.

2.6.1 Data Quality Objectives

The DQO process specifies project decisions, the data quality required to support those decisions, specific data types needed, data collection requirements, and analytical techniques necessary to generate the specified data quality. The process also ensures justification of the resources required to generate the data. The DQO process consists of seven steps, of which the output from each step influences the choices that will be made later in the process:

- Step 1: State the Problem.
- Step 2: Identify the Decision.
- Step 3: Identify the Inputs to the Decision.
- Step 4: Define the Study Boundaries.
- Step 5: Develop a Decision Rule.
- Step 6: Specify Tolerable Limits on Decision Errors.
- Step 7: Optimize the Design.

During the first six steps of the process, the planning team develops decision performance criteria that will be used to develop the data collection design. The final step of the process involves developing the data collection design based on the information from the other steps. The following provides a brief discussion of these steps and their application to this sampling effort.

Step 1: State the Problem - *The purpose of this step is to describe the problem to be studied so that the focus of the investigation will not be ambiguous.*

Remedial action preformed under the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) response actions within the BPSOU included storm water controls, waste removal, and engineered caps installed over mine waste left in place or in contaminated areas. Reclaimed areas must be maintained to achieve performance standards established in the BRES, which is attached to the ROD (EPA, 2006a) as Appendix E. Periodic monitoring, inspection, and maintenance tasks are required to ensure waste is not exposed, and the performance standards achieved through the remedial action are upheld.

Step 2: Identify the Decision - *This step identifies what questions the study will attempt to resolve and what actions may result.*

Routine evaluations are performed on an annual rotating schedule (by quadrant) to assess the trigger items listed below:

- Condition and diversity of vegetative cover.
- Presence of erosion.
- Condition of site edges.
- Presence of exposed mine waste material.
- Presence of bulk soil failure or mass instability.
- Presence of barren areas or gullies.

Step 3: Identify the Inputs to the Decision - *The purpose of this step is to identify the informational variables that will be required to resolve the decision statements and determine which variables require environmental measurements.*

Annual field evaluations are performed to determine if reclaimed areas achieve the performance standards described in the BRES. Independent, third-party entities complete field evaluations and provide the results to BSB. Results for each site are entered into the BRES Evaluation database form (Attachment 2) on a field-capable device. The data is directly uploaded to the project database as described in the BPSOU Reclaimed Areas User's Guide (included in the BPSOU Solid Media Management Plan). Trigger items identified on the field form that do not meet the BRES performance standards are assigned corrective actions, which are addressed as described in the M&M Plan.

Step 4: Define the Study Boundaries - *The purpose of this step is to define the spatial and temporal boundaries of the problem.*

The BRES evaluation does not include residential yards or playgrounds. Reclaimed areas within BPSOU in BSB are identified in Figure 1. Site-specific boundaries are available in GIS format to provide precise spatial location of reclaimed area boundaries being evaluated. The GIS files are stored on the GIS database (refer to BPSOU DMP) and BSB personnel can upload them to field tablets.

Step 5: Develop a Decision Rule - *The purpose of this step is to define the parameters of interest and integrate any previous DQO inputs into a single statement that describes a logical basis for choosing among alternative actions.*

Reclaimed areas will be evaluated as described in the BRES. The field evaluations identify specific trigger items with deficiencies that require corrective action and monitoring. These trigger items are identified and scored individually on the specific field evaluation form (Attachment 2). The BRES identifies decision logic diagrams for each trigger item to determine if additional action is required. Trigger items that do not meet the BRES requirements will be maintained as described in the M&M Plan. The specific condition of the following features at a site could trigger a corrective action requirement:

- Vegetation.
- Erosion.
- Exposed mine waste.
- Bulk soil failure or land slumps.
- Barren areas.
- Gullies.

Using the Butte Hill Revegetation Specifications (BHRS) as a guide (the BHRS is part of the BRES document in the ROD [EPA, 2006a]), trigger items that require VIs or RIs require an Agency-approved CAP. The CAP will provide detailed actions to correct any specific deficiencies. If sampling is required at any site to determine soil growth characteristics, samples will be collected from the 0-to-6 inch depth and analyzed for the following parameters to meet the requirements of cover soil in the BHRS.

- pH.
- Sodium Adsorption Ratio (SAR).
- Electrical Conductivity (EC).
- Soil textural classification.
- Organic Matter.
- Soil Nutrients.
- Herbicides or pesticides may be tested as needed.

If a CAP specifies that additional soil sampling is required to determine the presence of contaminants of concern above action levels provided in Table 3, field XRF analysis will be used to provide instantaneous data to characterize the site. Per BHRS, 18 inches is considered the minimum thickness required for long-term vegetation success. Sampling to this depth may be required to confirm if previous remediation meets BHRS and support decisions related to soil quality to promote vegetative growth.

Table 3. Solid Media Action Levels

Contaminant	Solid Media	Action Levels	Land Use
Lead [1]	Soil	2,300 mg/kg	Commercial
	Soil	2,300 mg/kg	Recreational
Arsenic [1]	Soil	500 mg/kg	Commercial
	Soil	1,000 mg/kg	Recreational
Cadmium [2]	Soil	20 mg/kg	Recreational
Copper [2]	Soil	1,000 mg/kg	Recreational
Zinc [2]	Soil	1,000 mg/kg	Recreational

1 From ROD Solid Media, Table D-1 (EPA, 2006a).
2 From Stream Side Tailings Operable Unit removal action levels.
mg/kg: milligrams per kilograms

The proposed location and depth of the samples will be provided in the site-specific FSP, verified in the field, and locations saved by the sampling team. All sample locations will be saved using a Global Positioning System (GPS). Additional samples may be collected if determined necessary. Samples will be labeled and handled according to the labeling and custody procedures described in this QAPP.

The M&M Plan contains SMPs that describe specific instructions to perform routine tasks to address triggers items.

Step 6: Specify Tolerable Limits on Decision Error - *The purpose of this step is to identify baseline conditions, limits, and ranges for decisions and consequences of decision errors.*

Site baseline conditions were documented in the development of original site boundaries, and sources of variability are described in the BRES.

Decision errors occur when information misleads the site managers into choosing an inappropriate response, including no action. The potential for decision errors exists because field evaluations are conducted by personnel who can be subjective in scoring performance criteria or may inadvertently enter information incorrectly into the database. Additionally, all analytical measurements inherently contain sampling and measurement errors. Sampling design errors occur when the data collection scheme does not adequately address the inherent variability of the matrix being sampled (e.g., discrete versus composite samples).

Field evaluation errors will be minimized by 1) ensuring field evaluators receive assessment training annually prior to conducting field evaluations, 2) ensuring all personnel use standard forms maintained in a cloud-based database, and 3) ensuring all data are downloaded to a dedicated computer to allow real-time QA processing by the BSB Data Management Division Manager.

Step 7: Optimize the Design - *The purpose of this step is to develop an optimized plan to complete the task.*

> The site evaluation and data collection scheme are designed to ensure that the information will be of sufficient quality to appropriately assess the condition of the site and identify trigger items. Site data will be maintained in and accessed through a secure database to ensure field personnel have current site data (boundaries, imagery, forms, etc.) (refer to BPSOU DMP).

2.6.2 Measurement Performance Criteria for Data

Measurement performance criteria are established by defining acceptance criteria and quantitative or qualitative goals (e.g., control limits) for accuracy, precision, representativeness, comparability, and completeness of measurement data. The definitions of precision, accuracy, representativeness, comparability, and completeness are provided below along with any acceptance criteria for data collected.

Precision

Precision related to site boundaries is the degree to which readings can be made. Field personnel are able to track site boundaries by physically walking along an established path and comparing what they see to site boundaries shown on aerial imagery.

Precision related to sampling is the amount of scatter or variance that occurs in repeated measurements of a particular analyte. Acceptance or rejection of precision measurements is based on the relative percent difference (RPD) of the laboratory and field duplicates. For example, perfect precision would be a 0% RPD between duplicate samples (both samples have the same analytical result). For soils analysis, acceptable precision is an RPD of plus or minus 35% in soil samples. This precision requirement is derived from the Contract Laboratory Program (CLP) Statement of Work (SOW) (EPA, 2016).

Precision related to BRES evaluations will be provided by ensuring all personnel complete training prior to conducting evaluations. Training will be provided through an annual standard BRES training program. Training will include classroom instruction related to procedures and evaluation principles along with field exercises to apply classroom training to provide reproducible data. Additional information regarding BRES evaluation training is in Section 2.7.

The mandatory annual training session will include vegetative cover identification, vegetative cover estimation method, erosional assessment, trigger item identification, and using field-compatible tablet devices to record and report data. Field evaluation teams will be trained to visually estimate vegetation cover using a modified point intercept method that uses frames of 0.25 square meters (m^2) with a 10-point grid system to quantitatively measure cover. Laser pointers will be used in conjunction with a grid of 10 points on a frame. The type of material intercepted by the lasers will be identified and recorded to determine percent live plant cover, litter, rocks, and bare ground. The field team's experience will be tested; the field team will make a visual estimate of cover on an area, then quantitatively measure cover on the same area.

Vegetation training will be complete once the field team members can reliably estimate vegetation cover to within plus or minus 10% of field team consensus scores.

Accuracy/Bias

Accuracy is the degree of agreement of a measured value with the true or expected value. For this QAPP, accuracy will be measured in two different ways. Related to site boundaries, accuracy will be assessed by field personnel equipped with a GPS-enabled device to track their position against the known map and boundary stored in the database. Accuracy can typically be obtained to within 2-5 feet using a GPS-enabled device.

For laboratory data, accuracy will be assessed by measuring recovery of laboratory control standards, specifically National Institute of Standards and Testing (NIST) for the metals. Recovery will be calculated and bias evaluated.

As previously discussed, evaluation personnel will receive mandatory training. The field team's training and experience will be tested; the field team will make a visual estimate of vegetative cover in an area, then quantitatively measure vegetative cover in the same area. Vegetation training will be complete once the field team can reliably estimate vegetation cover to within plus or minus 10% of the measured scores.

Representativeness

Representativeness is the degree to which sample data represent a characteristic of a population, parameter, or environmental condition. Representativeness is a qualitative parameter that is most concerned with proper design of the sampling and analytical schemes. Representativeness is achieved by determining the number and locations of samples and the appropriate sampling techniques needed to depict, as accurately and precisely as necessary, the conditions being measured. Representativeness deals with using protocols for sample storage, preservation, and transportation; analyzing samples with appropriate methods, techniques, and instrumentation; and using methods to document these protocols. Representativeness will be achieved through judicious selection of sampling locations and methods. Both in-place soils and backfill material will be analyzed by X-ray fluorescence (XRF) as described in SOP-05 (Attachment 3). The standard reference material for metals analysis will be NIST reference material No. 2711 (Moderately Elevated Trace Element Concentrations, available from the NIST website at https://www.nist.gov/) or another reference material with similar matrix conditions if the 2711 material is not available.

This QAPP requires that samples be representative of the medium being sampled and that the number of samples is sufficient to meet the project DQOs and satisfy the project remedial action design elements.

The BRES evaluation uses protocols for data retrieval, upload, QC, and storage via a central database maintained by BSB to ensure representative data are obtained. Sites are evaluated on a rotating four-year frequency. Protocol in area delineation ensures vegetation placement is representative across the site being evaluated. Specific areas are excluded (i.e., residential yards, playgrounds, and engineered covers) from BRES vegetative field evaluations to provide representative sample areas for evaluators. Application of techniques described in BRES evaluator training also ensure field evaluations are representative to the site. Field crews will obtain representative samples from a site using the point intercept grid method randomly across the site. Multiple samples will be evaluated to generate an overall site score.

Comparability

Data comparability is defined as the measure of the confidence with which one data set can be compared to another. Comparability is a qualitative parameter but must be considered in the design of the sampling plan and selection of analytical methods, QC protocols, and data reporting requirements. Comparability will be achieved by analyzing samples obtained in accordance with this QAPP and applicable contract laboratory SOPs, as well as the Program SOPs, which are comparable to the sampling methods used during previous investigations at the site. All data will be reported in units consistent with standard reporting procedures so that the results of the analyses can be compared with results from previous investigations.

Completeness

Completeness refers to the amount of usable data produced during field evaluations and a site-specific sampling and analysis program. The procedures established in this QAPP are designed to ensure, to the extent possible, that data will be valid and usable. The procedures also ensure that appropriate corrective action can be implemented. To achieve this objective, every effort will be made to ensure site evaluations are completed per this QAPP and as described in the BRES, and that the required samples are collected (avoiding sample loss) as described in site-specific sampling plans.

Sensitivity

Sensitivity describes how the uncertainty in an output can be apportioned to sources of uncertainty in its inputs. Sensitivity of BRES evaluations can be attributed to variability in environmental and site conditions. Evaluations will be constrained to be completed annually in the month of May, after evaluators complete standard training.

2.7 Special Training

Various training requirements are required depending on the task being completed. This section describes the training required to complete site evaluations, field sampling and analysis, and analytical laboratory tasks.

The BSB Environmental and Operations Division Manager will verify training has been completed by all personnel prior to conducting field evaluations and collecting samples. All training records will be maintained by the BSB Data Management Division Manager and summarized in the annual completion report.

2.7.1 Site Evaluation Training

All field personnel conducting site evaluations will be trained annually by stakeholder representatives and third-party personnel to perform field evaluations as described in the BRES document. This site evaluation training will provide field personnel with specific instructions related to field evaluation methodology, spatial data tools, calibration techniques, field evaluation principles, and applying the requirements of this QAPP prior to initiating fieldwork. Site evaluation training in a classroom will include specific instructions related to using a field tablet device (i.e., iPad) to access and populate the evaluation form (Attachment 2) and using the ESRI Collector for ArcGIS application to create geographic shapefiles. Site evaluation training completed in the field will include evaluation procedures and practice sessions to measure percent vegetative cover, classify erosion, and recognize trigger items. Field personnel will use the user's guide included with the ESRI Collector for ArcGIS application to help them use the application.

Evaluators will learn to apply the modified point-intercept QC method and complete QA visual estimates. The entire site evaluation training will encompass a test that includes vegetation measurements, erosion evaluation, trigger item identification, and plant identification pertinent to the BRES classification system, such as identifying acceptable species, undesirable weedy species, noxious weeds, etc., to verify proof of site evaluation training and readiness to conduct BRES evaluations.

2.7.2 Field Sampling and Analysis

For personnel who will collect samples in the field, prior to sampling personnel will review sampling procedures and requirements to ensure sample collection and handling methods follow QAPP requirements. Field sampling personnel will receive Hazardous Waste Operations and Emergency Response (HAZWOPER) training prior to conducting sampling in areas of impacted soils. Personnel will also be trained in proper use of field equipment and procedures according to relevant field data collection SOPs and methods described in any site-specific FSPs.

One hard copy of the current approved version of this QAPP will be maintained for reference in the field vehicle and/or field office. All field team personnel will have access to a portable document format (PDF) version of the complete QAPP. The Field Team Leader will assure that each member of the sampling team is familiar with the QAPP, will maintain signatures of each team member who has read the QAPP (including reviews and addenda, as necessary), and make sure each team member has been trained in the appropriate sample collection methods.

The Field Team Leader will review the site-specific health and safety plan (SSHASP) with all field personnel prior to fieldwork to assess the specific hazards and the control measurements that have been put in place to mitigate these hazards. The SSHASP review will also cover all other safety aspects of the site including personnel responsibilities and contact information, additional safety requirements and procedures, and the emergency response plan.

Field sample analysis will be performed as described in the specific equipment's user manuals. Field team members performing field XRF analysis will review the XRF unit's user manual or guide (Niton XL3 Analyzer User's Guide, or approved equivalent) prior to performing field analysis. The user guide for the Niton XL3 series of analyzers is in Attachment 3.3. At a minimum, field personnel will review the manual annually, before initiating sampling, to become familiar with the device. Personnel will follow the manual and specific SOPs when using the device including initial set-up, calibration, and field analysis. The XRF samples will be prepared and analyzed in a dedicated sample preparation area within the offices of the BSB Reclamation Division.

2.7.3 Analytical Laboratory

For laboratory analysis related training, required elements of laboratory training and QC are found in the SOPs (Attachment 3) along with laboratory analytical protocol (LAP) for standard wet chemistry analyses and individual contract laboratory protocols for Inducted Coupled Plasma-Mass Spectrometry (ICP-MS) analysis. These documents outline the specific laboratory QC samples, the frequency of analyses, control windows, and corrective action to be taken when windows are exceeded. Within laboratories, analytical instruments are initially calibrated using standards and blanks, and the calibration is routinely verified. The calibration is checked using an independent reference and instrument performance is monitored using method-specified QC check samples. Matrix spikes and laboratory duplicates measure method performance.

2.8 Project Data and Records

Data collection and management requirements for reclaimed areas and BRES sites were initiated in 2007. The information is used specifically to evaluate and maintain cap integrity and is stored within the BPSOU reclamation database maintained by BSB. This section describes procedures for documentation management and record keeping from initial record generation through final data formatting and storage. Geospatial data is stored in a Geodatabase, and non-geospatial data is stored in a Microsoft Access database maintained by BSB that can be accessed via a secure on-line portal. Refer to the BPSOU DMP for additional information on the databases mentioned in this section.

2.8.1 Property Access Agreements

Where applicable, BSB or Atlantic Richfield will request that property owners grant access to their properties to perform site evaluations, sampling, and remedial action as necessary. The Environmental and Operation and Maintenance Manager will manage requests for access, track the status of access requests and maintain copies of completed access agreements received from property owners. Completed access agreements will be photocopied and scanned with the electronic version stored on a server. Photocopied access agreements will also be copied to the project database.

2.8.2 Field Documentation/Data Forms

The reclamation database exists on a web-based server. Field personnel can enter the data and administrative personnel can track site evaluations and maintenance work performed on reclaimed sites. Personnel from BSB maintain the database. Field personnel enter the data into forms on a field tablet connected to the database and linked to ArcGIS Online. The BSB Data Management staff will pull the field data from the database to complete real-time quality checks (refer to Section 3.11).

Field personnel will use a field-capable tablet (i.e., iPad) to record specific real-time field data such as spatial boundaries and locations where evaluations or maintenance work is performed, capture the work with photographs, and document team members on the site, project duration, and equipment used, and material quantities used, removed, and applied to the site. Field documentation must be detailed to provide a description of site conditions during field evaluations and provide a permanent record of all field activities including sampling and maintenance activities.

BRES Evaluation Documentation

The BRES evaluation field documentation will include the data input form, accessed via Microsoft Office 365 through a secure web-based server, and site maps accessed via ArcGis Online. Field personnel will enter data directly to the form and upload the form to the database. Field personnel will take site photographs using the field device (iPad), geotag the photographs to the site, and upload them to the database. Hard copy field forms will not be used.

Documentation for each site will include the information required on the BRES Field Form (Attachment 2) and listed below:

- Site identification – site name, number, and description.
- Evaluation team members.
- Date of evaluation.
- Vegetation score and trigger criteria.
- Erosion score and triggers criteria.
- Additional trigger items scores
  - Site edges
  - Evidence of exposed mine waste
  - Barren areas
- Additional comments.

Reclaimed Areas Sampling Documentation

Additional sampling for any reclaimed areas will be performed as described in an approved CAP/FSP, and this QAPP. The following documentation is typical for any sampling event to be conducted on reclaimed areas in BPSOU. For any field sampling work, the sample container will be marked with the following:

- Sample location and ID number.
- Sample type collected.
- Date and time of sample collection.
- Samples taken by other parties (note the type of sample, sample location, time/date, sampler’s name, sampler’s company, and any other pertinent information).
- Sampling method, particularly any deviations from the field SOPs (Attachment 3).

- Documentation or reference of preparation procedures for reagents or supplies that will become an integral part of the sample (if any used in the field), specifically if sample bottles/preservatives are not provided by the laboratory and certified as cleaned.
- Sample preservation (if used).

Sufficient information should be recorded to allow the sampling event to be reconstructed without having to rely on the sampler's memory. Samples collected for laboratory analysis will be accompanied with a CoC record described in Section 2.8.4. Sampling data will be saved to the soils database and referenced to the reclaimed database via site field identification number (refer to BPSOU DMP).

Reclaimed Areas Maintenance Documentation
Field personnel will use the field-capable tablet to record maintenance information. The information recorded will be specific to the maintenance performed, but typically will consist of the following:

- Identification of site and date of maintenance performed.
- List of field crew members.
- Description of maintenance performed
- Quantity of material removed.
- Quantity and type of material imported.
- Standard maintenance procedures referenced.
- Equipment used.

The above-listed information will be incorporated into the Annual Summary Report and distributed according to Section 2.5.3.

2.8.3 Field Photographs

Field personnel will use a digital device (iPad or similar phone), with access to the Program database, to take photographs at the site. Field personnel will take photographs of sampling locations, field activities, and anything else to document site conditions, as necessary. Field photographs will be used to chronical the before, during, and after maintenance task activities. Photographs will be geotagged and uploaded to the project database.

2.8.4 Chain of Custody Records

Each sample collected will be assigned a unique sample number (described in Section 3.4) and the sample container labeled with sample number, address, and location. Then the information will be logged into the project sample logbook. The CoC records ensure that samples are traceable from the time of collection until final disposition. After samples have been collected, they will be maintained under strict protocols in accordance with the SOPs (Attachment 3). A CoC record will be initiated by the individual physically in charge of the sample collection. The CoC form may be completed concurrently with the field sampling or before shipping samples to the laboratory. The sampler is personally responsible for the care and custody of the samples until they are shipped. When transferring the sample possession, the individual relinquishing and receiving the sample will sign and record the date and time of day on the CoC record.

A copy of each as-transmitted form will be scanned and stored on a hard drive and hard copies will be included in project record files (refer to Section 3.11).

2.8.5 Analytical Laboratory Records

Results received from the laboratories will be documented both in report form and in an electronic format. Laboratory documentation will include laboratory confirmation reports such as information on how samples were batched and the analyses requested, data packages containing the laboratory report and the electronic data deliverable (EDD), and any change requests or corrective action requests. Section 5.1.4 lists the laboratory reporting requirements in detail. The deliverable (data package or report) issued by the laboratory must include data necessary to complete validation of laboratory results. Original reports and electronic files received from laboratories will be maintained with the project quality records. Refer to the BPSOU DMP for additional requirements. Sampling data will be forwarded to the Agencies in an annual summary report (refer to Section 2.3).

2.8.6 Project Reports

Required project reports provide a record of site evaluations, allow Agency review and approval, and provide a historical record of the activities at the site. The main required reports include a recommendations summary report, CAP, site-specific FSPs, and annual summary report. Refer to Section 2.5.3 for details on the reports.

2.8.7 Quality Records

Quality records are required to provide objective evidence that activities were performed and documented in a manner consistent with this QAPP and that the data are useful for their intended purpose. Records include legible and complete documentation related to evaluations, sampling, corrective action, and conventional maintenance tasks completed at reclaimed areas. These records will be organized and managed by the BSB Data Management Division Manager (or designee) and will include the following at a minimum:

- This QAPP and any approved revisions or addenda.
- Approved versions of the SSHASP and any addenda.
- Copies of field SMPs for field data collection, with any updates, revisions or addenda to those SMPs.
- Incoming and outgoing project correspondence (letters, telephone conversation records, and faxes).
- Individual property maps including any field drawings and field photographs.
- Field documentation forms.
- Copies of all field data sheets.
- Copies of all sample CoC forms.

- Copies of all laboratory agreements and amendments.
- Laboratory data packages (printed report and electronic version).
- Documentation of field and/or laboratory audit findings and any corrective actions.
- Draft and final delivered versions of all reports and supporting procedures such as statistical analyses, numerical models, etc.
- XRF correlative statistics.

All project information will be maintained indefinitely in the GIS database.

As described throughout this document, this QAPP will be maintained in the project database and updated annually, at a minimum. The BSB Environmental and Operation and Maintenance Manager, or designated alternate as appropriate, will maintain the document and perform updates as necessary. Hard copies will be distributed to field team leaders and the information accessible using field devices.

Any addendums or revisions to this QAPP will be electronically distributed to all parties identified on the distribution list by the BSB Environmental and Operation and Maintenance Manager, or designated alternate as appropriate. All records will be maintained and archived electronically for future reference. Updates will be provided, communicated, and implemented in a manner consistent with BSB Reclamation Division internal protocol.

2.9 Data Storage and Backup

Digital copies of documents will be stored in the appropriate database, as described previously. The document will be assigned a numeric code based on what area the document pertains to followed by a sequential number.

Data storage and backup will be maintained as described in the BPSOU DMP. Generally, a regular backup of component databases provides security against damage or loss of data. The BPSOU reclamation database is the electronic data repository used to store all reclamation data related to source areas that have been reclaimed and evaluated. The BRES data collection and management system was initiated in 2007 and is maintained by BSB. Within the system, electronic data is backed up daily. Daily backups are retained for a minimum of two weeks, weekly backups for two months, monthly backups for one year, and yearly backups for five years. Electronic data are stored long-term in a secure location using appropriate technology. At the discretion of a database administrator/data coordinator, additional backups may be performed after large quantities of data are imported or after significant manipulation or evaluation of the data in the database. Details regarding data management are provided in the BPSOU DMP.

3.0 MEASUREMENT AND DATA ACQUISITION

This section addresses all aspects of generating and acquiring data for a specific site. Adhering to these procedures ensures that the field team uses the appropriate methods to conduct field evaluations, collect samples, handle samples, specific laboratory analyses, complete field and laboratory QC, inspect/test/calibrate instruments and equipment, maintain equipment, manage data management, and follow established data security protocols.

3.1 Site Access

Reclaimed areas are evaluated on a four-year rotating schedule as prescribed in the BRES. Site access approval to privately owned properties must be obtained prior to performing any site evaluations. Requests for site access that are not granted will require EPA authority intervention to access the site, or the site will be removed from the reclaimed areas evaluation cycle.

3.2 Site Evaluations

Evaluators will use a field-capable tablet with ArcGIS Collector software to record specific real-time field data. The tablet will have access to electronic field evaluation forms that will be pre-populated for each specific site being evaluated. Once an evaluation is complete, the evaluation form will be saved to the cloud-based database (refer to BPSOU DMP).

Field crews will perform site evaluations to review the parameters listed on the evaluation forms and identify trigger items. Percent live cover is considered the most critical performance parameter in the BRES. The vegetative cover will be estimated using the modified point intercept method. A 10-point grid will be used in conjunction with a laser pointer indication system to determine percent live cover, litter, rocks, and bare ground.

3.2.1 Reclaimed Areas Site Identification Numbering

Site identification for all reclaimed areas will include the quadrant number in which the area is located, site reclaimed status, and sequential number within the quadrant. Additional sites may be added to the quadrant after appropriate reclamation within BPSOU is completed to ensure the site remedy is monitored for protectiveness.

3.3 Site Sampling

Sampling performed in reclaimed areas to address specific trigger items (i.e., exposed mine waste, barren areas, etc.) or support related maintenance tasks (cover soil characterization) will follow site-specific FSPs and applicable SOPs. The FSPs will provide detailed sampling requirements such as specific site maps, sample collection locations, and depth of sample collection. Field personnel will use the FSPs to further characterize sites or areas as needed and develop appropriate CAPs. A list of SOPs for sampling and related tasks is provided in Table 4. Attachment 3 contains the SOPs.

Table 4. Standard Operating Procedures

SOP Title	Organization	Date
Project Documentation	BSB	Dec-17
Completing Chain of Custody Forms	BSB	Dec-17
Equipment Decontamination	BSB	Dec-17
Composite Soil Sampling	BSB	Dec-17
Operating XL3 X-Ray Analyzer	BSB	Dec-17
Soil pH Testing	BSB	Dec-17
Personal Decontamination	BSB	Dec-17
Sample Packaging	BSB	May-18
Boundary Revisions	BSB	May-19

The following activities are typical for any sampling event conducted on reclaimed areas in BPSOU to characterize a site or area to address an RI deficiency within reclaimed source areas. Composite soil samples will come from two depth intervals. Composite samples will be collected at a frequency of not less than 1 sample per 5 acres, and no more than 1 sample per 100 square feet (ft^2). One composite sample, comprised of 3 composited subsamples, will be collected from 0 to 6 inches from the top of the surface to be analyzed for organic compounds (Walkley-Black), nitrogen, phosphorus, and potassium. One composite sample, comprised of 3 composited subsamples, will be collected from 6 to 18 inches from the top of the surface to be analyzed for metals (Table 3). Field personnel will use an XRF unit (Niton XL3t XRF Analyzer, or approved equivalent) for the XRF field analysis. A sample stand, which allows the samples to be analyzed in the plastic bags, will be used during analysis to ensure consistent exposure times and position of the XRF aperture for each sample. Results for will be recorded on field data sheets.

A field duplicate sample and field blank will be collected at a minimum frequency of 1 for every 20 samples. Disposable sampling equipment will be used to collect soil samples; therefore, equipment blanks will not be collected.

Field personnel will record all information in the field logbook. The procedures for sampling are summarized below.

3.3.1 Soil Sample Collection

The collection of soil samples performed under this QAPP will be completed as described in the steps provided below. Field personnel/samplers will record all information in the field logbook. The decision to collect additional "opportunistic" samples will be made in the field by the sampling crew personnel and/or Agency personnel during the time of sampling. The BPSOU confirmation soil samples will be shipped to a certified lab for analyses. General collection procedures are listed below and also provided in Composite Soil Sampling procedure in Attachment 3.1.

1. **Visually inspect the site** to determine the number of sections needed for composite sampling.

2. **Take photographs to create a record** to document the condition of the specific areas identified for investigation. Note the areas of concern from which soils may be removed, and document any physical structures or features (e.g., structures, fences, trees, etc.) that may be impacted.

3. **For each composite sample,** place the sample in the designated sealable plastic bag and label the bag with the address and the location of the sample. Equal amounts of soil from each designated subsample location will be composited into one sample.

4. **Collect the samples from depth intervals (placing each sample in the corresponding bag).** Collect up to 5 subsamples in an X pattern (if possible) for each composite sample section (see *Areas with Dense Vegetation* below). The first composite sample will consist of subsamples from the 0-to-6 inch depth interval, and the second composite sample will consist of subsamples from the 6-to-18 inch depth interval, as needed. Thoroughly homogenize each sample in the bag (or stainless steel bowl) by kneading the soil. Discard any particles greater than 0.5 inches in diameter. Preservation of soil samples is not necessary for the designated metals analysis. Sample volumes will consist of approximately 500 to 800 grams of material.

   Areas with Dense Vegetation
   For areas covered with dense vegetation or grass mat, collect the initial composite subsample from immediately beneath the vegetation. The sampler will separate it from the soil surface with a stainless steel knife, or equivalent, and shake and scrape the removed vegetative mat over the sample collection bowl to dislodge any mineral soil particles. Include all dislodged soil particles in the composite sample. Collect the remaining subsamples as described above.

5. **For metals analysis, field analyze the sample** using the XRF unit (Niton XL3t XRF, or approved alternate). Place a portion of the homogenized sample from the 6-to-18 inch depth interval into an additional quart-sized re-sealable plastic bag for placement in the analyzer. Compact the material so that there is a flat surface on the area to be analyzed, then visually inspect this area to ensure that only fines will be present in the XRF aperture.

6. **Record the results** for the selected metals on the XRF field data sheet (downloaded to the field tablet). Complete duplicate and replicate XRF analyses on at least 5 percent (5%) of the samples analyzed in the XRF unit.

7. **For laboratory-analyzed samples,** prepare and follow CoC procedures.

8. **Ensure all sampling identification information** is entered into the database tracking system.

3.3.2 Sample Collection Equipment

Soil samples will be collected using primarily hand tools, which will be limited to readily available products. If field supplies run out, replacement supplies will be purchased at nearby retailers. Field equipment that could be used for the soil sampling will include the following:

- Copy of the QAPP and FSP.
- Maps of sample locations.
- GPS unit.
- Sharpshooter® type shovels and breaker bars.
- Sterile single-use disposable plastic scoops.
- #10 (2 mm) stainless steel screens, or disposable mesh screen.
- 1-quart resealable plastic bags.
- XRF unit (Niton XL3t XRF analyzer, or approved alternate).
- Equipment and deionized water for decontamination.
- Sample coolers, ice, and tape.

3.4 Sample Identification

An alphanumeric coding system will be used to uniquely identify each sample collected. Sample identifiers will begin with the site ID (which will include the quadrant number) and include composite sample number (1, 2, 3, etc.) from the location, sample date, and depth interval codes.

Depth Intervals: (1) 0 to 6 inches below ground surface (bgs)
(2) 6 to 18 inches bgs

The example sample identification number Q1-RC12-1-08022017-1 indicates Reclaimed Site 12 located in Quadrant 1, composite sample 1 was obtained on August 2, 2017, from a depth interval of 0 to 6 inches below ground surface.

Sample identifiers will be documented in field logbooks and on the CoC forms, as required by the BPSOU field data sheets located in Attachment 3.

3.4.1 Soil Sieving Methods

All soil samples must be sieved using a No. 10 sieve (stainless steel or disposable mesh). Sieved fraction passing the No. 10 sieve will be placed in a new plastic bag labeled with the original sample number and date of sieving. The sieved fraction will be used for XRF analysis, and the coarse fraction will be properly discarded as described in Section 3.4.3.

3.4.2 Sample Handling and Chain of Custody

After collection and labeling, the samples will be maintained under strict CoC protocols, in accordance with SOP-02 (Attachment 3). The field sampling personnel will complete a CoC form for each shipment/delivery (i.e., batch of coolers) of samples to be delivered to the laboratory for analysis prior to expiration of the 28-day holding time. The sampler is responsible for initiating and filling out the form. The CoC for a shipment/delivery will list only those samples in that shipment/delivery. Any documentation, including CoCs, will be placed inside a re-sealable plastic bag and placed within the shipment/delivery container. Coolers that are to be shipped will be custody sealed, securely taped shut, and have a shipping label securely adhered to the cooler. Sample containers hand delivered to the laboratory do not need to be prepared for shipping.

The sampling personnel whose signature appears on the CoC form will be responsible for the custody of the samples from the time of sample collection until custody of the samples is transferred to a designated laboratory, a courier, or to another project employee for the purpose of transporting the samples to the designated laboratory. Custody is transferred when both parties to the transfer complete the portion of the CoC under "Relinquished by" and "Received by.” Signatures, printed names, company names, dates and times are required. Upon transfer of custody, the sampling personnel who relinquished the samples will retain the third sheet (pink copy), photocopy, or electronic copy of the CoC. When the samples are shipped by a common carrier, a Bill of Lading supplied by the carrier will be used to document the sample custody, and its identification number will be entered on the CoC. Copies, receipts, and carbons of Bills of Lading will be retained as part of the permanent documentation in the project file. It is not necessary for courier personnel to sign the CoC.

Upon receipt by the laboratory, the samples will be inspected for sample integrity. The CoC will be immediately signed, dated, and reviewed by laboratory personnel to verify completeness. Any discrepancies between the CoC and sample labels and any problems or questions noted upon sample receipt will be communicated immediately to the Field Team Leader. The laboratory will provide the Field Team Leader and Contractor QAO with a copy of the CoC and associated sample-receipt information within two working days of receipt of samples. The sample-receipt information routinely provided will include sample receipt date, sample IDs transcribed from the CoCs, sample matrix type, and list of analyses to be performed for each sample. Broken custody seals, damaged sample containers, sample labeling discrepancies between container labels and the CoC form, and analytical request discrepancies shall be noted on the CoC form. The Field Team Leader and Contractor QAO will be notified of any such problems; discrepancies or non-conformances will be resolved and addressed before the samples are analyzed.

The laboratory will be responsible for following their internal custody procedures from the time of sample receipt until sample disposal. Samples and extracts will be stored in a secure area controlled by the laboratory’s designated sample custodian. Samples will be removed from the shipping container and stored in their original containers unless damaged. Damaged samples will be disposed of in an appropriate manner after notifying the Field Team Leader and Contractor QAO, and authorization to dispose is received and documented. In addition, samples will be stored after completion of analyses in accordance with contractual requirements.

3.4.3 Sample Disposal

Soil samples shipped to the laboratory for analysis will be held until the laboratory analysis has been completed and then be disposed according to internal laboratory procedures or returned for proper disposal. Samples that exceed the action levels listed in Table 3 will be disposed of at the Butte Mine Waste Repository.

3.4.4 Decontamination

Field personnel will decontaminate all non-disposable sampling equipment after use at each sampling location. Disposable equipment and personal protective equipment intended for one-time use will not be decontaminated but will be packaged for appropriate disposal as a solid waste in the local landfill. Additional soil removed from holes during excavation, and not collected as samples, will be returned to the sample holes.

Field personnel will decontaminate reusable sampling equipment within the site boundaries at a centralized location. Sampling equipment will be decontaminated using the procedure below (and any related SOPs). All equipment will also be decontaminated before leaving the site to prevent off-site transport of contaminants.

- Rinse with water.
- Wash with non-phosphate detergent.
- Rinse three times with deionized water.
- Air dry.

For safety, all personnel will undergo decontamination procedures when leaving a contaminated area. Personnel decontamination includes routine practices as well as emergency decontamination. All personnel will take every measure possible to prevent the spread of potentially contaminated materials to clean areas.

3.5 Analytical Methods

This section describes the field and laboratory analytical methods used to analyze soil samples.

3.5.1 Field Analysis

Samples tested for pH in the field will be completed as described in SOP-6 (Attachment 3) using a Hanna Instruments, HI 99121 Soil pH Meter (or approved alternate). Field personnel will collect samples for XRF analysis in the field and take them to a dedicated sample preparation area within the sample site. To perform XRF analysis, field personnel will follow the process described in SOP-05 using an XRF unit (Niton XL3t XRF, or approved alternate). The sampler will place the sample on the XRF unit’s sample stand, which allows the samples to be analyzed in plastic bags, during analysis to ensure consistent exposure times and position of the XRF aperture for each sample. The sampler will record the results for the analytes (listed in Table 3) on the field data sheets.

3.5.2 Laboratory Metals Analysis

Field samples within plus or minus 25% of action levels will be submitted for laboratory analysis to confirm and expand on field XRF results. Samples will be prepared for metals analysis according to the appropriate SOPs (Attachment 3). If ICP-MS methods are necessary, a laboratory complying with EPA CLP protocol (EPA, 2016) will conduct the analysis in accordance with EPA test methods for evaluating Solid Waste, Physical/Chemical Methods, also known as SW-846, 6020A, Metals Analysis by ICP-MS. This method is typically able to provide analytical results within 10 working days of sample receipt at the laboratory. Laboratories will provide analytical results within 28 working days from receipt of samples.

3.6 Additional Analyses Methods

The subsections below describe the analytical methods an approved laboratory must use to analyze the soils for non-metals analysis. Non-metals analysis may be deployed to characterize sites or areas that require VI. Analysis must be completed prior to expiration of a 28-day sample holding time.

3.6.1 Non-Metals Analyses Methods for Soils

All soil samples submitted for non-metals analysis will be obtained from a 0-6 inch depth and will be analyzed by an approved laboratory for the following parameters: texture class and particle size, pH, saturation percent, EC in millimhos per centimeter (mmhos/cm), organic matter percent, nitrogen (NO_3), available phosphorus (P), and available potassium (K). The above parameters will be analyzed using U.S. Department of Agriculture (USDA) classification and test methods as described in the American Society of Agronomy (ASA)/Soil Science Society of America (SSSA) Monograph No. 9, Methods of Soil Analysis, Parts 1-2, most recent edition (ASA/SSSA, 1982, 1986). Approximately 500 to 800 grams of material will be collected in a single resealable (ZipLoc® type), quart-sized plastic bag (as described in Section 3.3.1 Steps 1-4, 7-8) and placed in a cooler with ice to maintain a 4-degrees Celsius (°C) temperature.

3.6.2 Cover Soil Analyses

All proposed cover soil sources must be approved by Atlantic Richfield and the Agencies prior to placement activities and the sources must meet Butte Hill Cover Soil specifications (EPA, 2006a). Cover soil approval requires submitting samples of the cover soil to an approved laboratory for analysis according to the conditions below.

- Three soil samples from the source site location will be submitted to the laboratory along with details on the area and depth to be excavated at the source site location.
- Each of the three soil samples will be analyzed by the approved laboratory for the following parameters to meet the requirements of the Butte Hill Cover Soil specifications:

    - Texture class and particle size.
    - pH.
    - Saturation percent.
    - EC in mmhos/cm.
    - Organic matter percent.
    - Nitrogen (NO_3), available phosphorus, and available potassium.
    - Analysis for soil metals parameters will include arsenic, cadmium, copper, lead, and zinc.

The above parameters will be analyzed using USDA classification and test methods as described in ASA/SSSA Monograph No. 9, Methods of Soil Analysis, Parts 1-2, most recent edition (ASA/SSSA 1982, 1986).

To the extent possible, the cover soil source should also be free of any noxious weeds.

3.7 Laboratory Quality Control Samples

Laboratory QC samples are introduced into the measurement process to evaluate laboratory performance and sample measurement bias. Laboratory QC samples may be prepared from environmental samples or generated from standard materials in the laboratory per the internal laboratory SOPs. All analyses will be governed by the appropriate calibration procedures and frequencies that are specified in the laboratory's SOPs.

3.7.1 Laboratory Blanks

Method blanks will be used to monitor laboratory processes and performance. A method blank is a volume of deionized water or a specified weight of inert material for solid samples that is carried through the entire sample preparation and analyses procedures. The method blank volume or weight will be approximately equal to the sample volumes or sample weights being processed. Method blanks are used to monitor interference caused by constituents in solvents and reagents and on glassware and other sampling equipment. Blank results outside of specified control limits will be re-run and/or flagged by the laboratory per the QC requirements of the analytical method.

3.7.2 Laboratory Control Samples

A laboratory control sample (LCS), or a blank spike, is an aqueous or solid control sample of known composition that is analyzed using the same sample preparation, reagents, and analytical methods employed for the program samples. The LCS is obtained from an outside source or is prepared in the laboratory by spiking reagent water or a clean solid matrix from a stock solution that is different from that used for the calibration standards. The LCS is the primary indicator of process control used to demonstrate whether the sample preparation and analytical steps are in control, apart from sample matrix effects. If the LCS recovery falls outside the specified control limits, the samples will be re-run and/or flagged by the laboratory per the QC requirements of the analytical method.

3.7.3 Analytical Duplicates

Analytical duplicates are samples that are split in the laboratory at some step in the measurement process and then carried through the remaining steps of the process. Duplicate analyses provide information on the precision of the operations involved. Analytical duplicates are a pair of subsamples from a field sample that are taken through the entire preparation and analyses procedure; any difference between the results indicates the precision of the entire method in the given matrix. Analyses of analytical duplicates and matrix spike (MS) duplicates monitor the precision of the analytical process. The frequency of analyses, precision goals, and corrective action information pertaining to analytical duplicates are available in the laboratory SOPs. If the analytical duplicate precision falls outside the specified control limits, the samples will be re-run and/or flagged by the laboratory per the QC requirements of the analytical method.

3.7.4 Matrix Spikes

Laboratory MS samples are used to evaluate potential sample matrix effects on the accurate quantitation of an analyte using the prescribed analytical method. The MS and matrix spike duplicate (MSD) are prepared by adding an analyte to a subsample of a field sample before sample preparation and analyses. A percent recovery is calculated from the concentrations of the analyte in the spiked and unspiked samples. If the percent recovery for the MS and MSD falls outside the control limits, the results are flagged by the laboratory as outside acceptance criteria along with the parent sample.

3.7.5 Field XRF Quality Control Samples

Field QC samples are used to identify any biases from transportation, storage, and field handling processes during sample collection and to determine sampling precision. All field QC samples will be delivered with field samples to the laboratory. This section includes brief descriptions of the QC samples to be collected during sampling activities along with frequency, collection, and analytical instructions. The measured values of a standard will be compared to the expected results, and if a measured value falls outside this range then the check sample will be reanalyzed. If the value continues to fall outside the acceptance range, the sampler will note this information on the XRF log. If any of the check sample results indicate that the XRF unit is not analyzing accurately, the XRF unit will be turned off, cleaned, and the energy calibration rerun. This information will be noted in the logbook and on the XRF field data sheet. The batch of samples analyzed prior to the unacceptable calibration verification check samples will be reanalyzed. Specific XRF unit QA/QC tasks are included with the specific XRF SOP (Attachment 3).

3.7.5.1 Blank Samples

The silicon dioxide sample, as provided by the XRF manufacturer (Niton or approved alternate), is a "clean" quartz or silicon dioxide matrix that contains concentrations of selected analytes near or below the XRF unit’s lower limit of detection. These samples are used to monitor for cross contamination. Field personnel will analyze this sample at the beginning of each day, once per every 20 samples, and at the end of each day’s analysis. The sample information will be recorded as “SIO2” on the XRF field data sheets. This sample will also be analyzed whenever field personnel suspect contamination of the XRF aperture. Any elements with concentrations above the established lower limit of detection will be evaluated for potential contamination. If it is determined that the concentration is higher than that recorded at the start of the day, the probe window and the silicon dioxide sample will be checked for contamination. If it is determined that contamination is not a problem, and the concentration is significantly above the limit of detection, sample results will be qualified by the XRF operator as ‘J’ estimated, and the problem recorded on the XRF field data sheet and in the logbook. If the problem persists, the XRF will be returned to the manufacturer for calibration.

3.7.5.2 Calibration Verification Check Samples

Calibration verification check samples help check the accuracy of the XRF unit and assess the stability and consistency of the analysis for the analytes of interest. A check sample will be analyzed as one of the initial samples, once per every 20 samples and as the last analysis. Results for the check sample (standard reference material [SRM]) will be recorded on the individual site XRF field data sheets and identified as a check sample. There will be 3 SRM check samples for the project (provided by the XRF unit manufacturer, Niton or approved alternate): NIST 2709a-Joaquin Soil, USGS SdAR-M2 (an SRM created by the U.S. Geological Survey), and a Resource Conservation and Recovery Act (RCRA) sample. There will also be XRF unit manufacturer-provided machine-specific expected results for several elements for the check samples. The measured values of a standard will be compared to the expected results, and if a measured value falls outside this range then the check sample will be reanalyzed. If the value continues to fall outside the acceptance range, this information will be noted on the XRF log. If any of the check sample results indicate that the XRF is not analyzing accurately, the XRF will be turned off, cleaned, and the energy calibration rerun. This information will be noted in the logbook and on the XRF field data sheet. The batch of samples analyzed prior to the unacceptable calibration verification check samples will be reanalyzed.

3.7.5.3 Duplicate Samples

The XRF duplicate samples will be analyzed to assess reproducibility of field procedures and soil heterogeneity. To run a duplicate sample on the XRF unit, field personnel will remove the sample bag from the analytical stand, knead it repeatedly, and replace it in the stand to be analyzed a second time. Duplicate samples will be recorded on the XRF field data form with a D designator in the sample identification number. One duplicate sample will be analyzed at the rate of 1 per 20 samples.

3.7.5.4 Replicate Samples

Field personnel will analyze a replicate sample at the rate of 1 per 20 XRF samples. When a replicate sample is required, the XRF will be restarted, after the primary sample analysis has been completed, to analyze the same sample a second time with the same soil in the XRF aperture. Replicate samples help to assess the stability and consistency of the XRF analysis. Replicate sample results will be recorded on the XRF field data form and designated with an R in the sample identification number.

3.8 Instrument/Equipment Testing, Inspection and Maintenance

To ensure continual quality performance of any instruments or equipment, the testing, inspection, and maintenance activities listed in this section will be performed and recorded.

3.8.1 Field Equipment

Field equipment used for evaluations is limited to handheld tablet devices, a laser pointer used to complete field evaluations, and sample collection tools described in Section 3.3.2. Disposable sampling equipment will be used to collect soil samples.

Field equipment used for sampling and analysis will be examined to certify that it is in proper operating order prior to its first use and at intermittent intervals during the day. Equipment, instruments, tools, and other items requiring preventative maintenance will be serviced in accordance with the manufacturer’s specified recommendations. Any routine maintenance recommended by the equipment manufacturer will also be performed and documented in field logbooks or appropriate data sheets. Equipment will be inspected, and the calibration checked if applicable, before it is used. Should equipment deficiencies be found, including calibration failures, the equipment will be immediately removed from service and repaired. Once equipment failures have been resolved and testing/calibration demonstrates proper equipment function, it will be returned to service. The field team leader, or their designee, will be responsible for field equipment checks and maintaining the Equipment Log.

Field equipment used for remediation and maintenance activities will be maintained as described in the manufacturer’s recommendations. Equipment will be inspected and replaced, if needed, before it is transported to a field setting.

3.8.2 Laboratory Equipment

Instruments used by the laboratories will be maintained in accordance with each laboratory’s QA plan and analytical method requirements. All analytical measurement instruments and equipment used by the laboratory will be controlled by a formal calibration and preventive maintenance program.

The laboratories will keep maintenance records and make them available for review, if requested, during laboratory audits. Laboratory preventive maintenance will include routine equipment inspections and calibrations at the beginning of each day or each analytical batch, per the laboratory internal SOPs and method requirements.

3.9 Inspection/Acceptance of Supplies and Consumables

All supplies and consumables received for the project (e.g., sampling equipment, supplies, etc.) will be checked for damage and other deficiencies that would affect their performance. The types of equipment needed to complete sampling activities are described in the relevant SOPs (Attachment 3 and specific site plans). The Field Team Leader or field team members will inspect all field supplies. The personnel at each laboratory will be responsible for inspecting laboratory supplies in accordance with their QA plan.

3.10 Boundary Adjustments

Boundary adjustments or additions are anticipated to include newly reclaimed areas, or boundary adjustments to incorporate newly reclaimed land adjacent to previously reclaimed areas at an existing BRES site. Initial boundary adjustment or definition will be made when remediation is complete. Aerial imagery will be reviewed along with completed construction data. Using GIS software, preliminary boundaries will be established on the aerial imagery. Field verification will then be completed by BSB or contractor field personnel. The entire site should be evaluated to note differences in land use, vegetation, slopes, etc. Final site boundaries and individual site polygons will be established accordingly. Specific boundary adjustment protocol is described in SOP SMP-10
(Attachment 3).

Newly reclaimed sites will be assigned a site identification number (site ID) and appropriate quadrant. The site ID, quadrant, remedial status, and attribute information will be saved to the reclamation database and the site included on the BRES evaluation list.

3.11 Data Management Procedures

This section describes how the project information will be managed including field evaluation, corrective actions, maintenance documentation, and laboratory data. The database used to track the site evaluations and maintenance work performed on reclaimed sites is housed on a cloud-based server. Field personnel enter field evaluation information into the field tablet, which populates the database. The BSB Data Management staff pull the information from the database for real-time QA checks (using Microsoft Access). Refer to the BPSOU DMP for specifics about the database.

As the information is used to make decisions specifically related to vegetative cover, BSB personnel will complete quality checks at various stages to verify the transfer of field data. The process includes these main steps:

- Field personnel enter site data directly from the field into a database.
- The BSB Data Management Division Manager reviews and verifies the data in real-time.
- Corrections are made, as necessary, to capture completion of work accurately, minor adjustments to boundary mapping information are made to match existing topography or boundary delineations, and material quantities are reported.

The QAM, see Section 2.3, will make necessary and appropriate corrections to field data and report the changes to the BSB Environmental and Operations Division Manager and field team leader as appropriate. Quality records will be maintained as described in the BPSOU DMP. These records, either electronic or hard copy in form, may include the following:

- Individual property maps (hard copy or scanned field drawings and electronic files).
- Project QAPP, including this QAPP, with any approved modifications, updates, addenda, and corrective or preventative actions.
- Field documentation.

- CoC records.
- Laboratory documentation (results received from the laboratory will be documented both in report form and in an electronic format).
- Recommendation summary report.
- CAPs with any approved modifications, updates, and addenda.
- Annual summary report.

Hard copy field and laboratory records will be maintained in the project's central data file, where original field and laboratory documents will be filed chronologically for future reference. These records will be scanned to produce electronic copies. The electronic versions of these records will be maintained on a central server system with backup scheduled on a daily basis.

Laboratory EDDs and related data packages will be reviewed as part of the internal data review process. Following these review steps, field and laboratory electronic data files will be imported to the project database.

Standardized data import formats and procedures will be used to upload both field and laboratory data into the database. Standardized parameter names and numerical formats and units of measure may be applied to the original information to facilitate comparability across all datasets and within the database.

4.0 ASSESSMENT AND OVERSIGHT

Assessment and oversight of data collection and reporting activities are designed to verify that field evaluations, sampling, and analyses are performed in accordance with the procedures established in this QAPP. Field and laboratory audit activities include two independent parts: internal and external audits. Internal audits may be performed by Atlantic Richfield or their approved representative, BSB, their contractor(s), or a contracted laboratory if deemed necessary. Internal audits are anticipated to be completed once per field evaluation cycle per quadrant, or once per every four years. External audits may be performed by the Agencies as necessary and will be scheduled as appropriate by the Agencies.

4.1 Quality Corrective Actions

Corrective action related to program quality is the process of identifying, recommending, approving and implementing measures to counter unacceptable procedures or out-of-QC performance, which can affect data quality. Corrective action can occur during field activities, analyses, and data assessment. A quality corrective action report template is provided in Attachment 3.

Non-conforming equipment, activities, conditions, and unusual incidents that could affect data quality and attainment of the project's quality objectives will be identified, controlled, and reported in a timely manner. For the purpose of this QAPP, a non-conformance is defined as a *malfunction, failure, deficiency, or deviation that renders the quality of an item unacceptable or indeterminate in meeting the project's quality objectives*.

Corrective action in the laboratory may occur prior to, during, and after initial analyses. A number of conditions such as broken sample containers, preservation or holding-time issues, and potentially high-concentration samples may be identified during sample log-in or just prior to analyses. Corrective actions to address these conditions will be taken in consultation with the key project personnel such as BSB Environmental and Operations Division Manager, QAM, and Liability Manager. In the event that corrective action requests are not in complete accordance with approved project planning documents, the Agencies will be consulted and concurrence will be obtained before the change is implemented, or new samples may be obtained.

All corrective actions taken by the laboratory will be documented in writing by the laboratory project manager and reported to the BSB QA Officer and Liability Manager. In the event that corrective action requests are not in complete accordance with approved project planning documents, the EPA will be consulted and concurrence will be obtained before the change is implemented. All corrective action records will be included in the QAPP's quality records.

4.2 Corrective Action During Data Assessment

The need for corrective action may be identified by any member of the project team during data assessment. Potential types of corrective action may include reassessment by the field team, re-analyses of samples by the laboratory, or re-submittal of data packages with corrected clerical errors. The appropriate and feasible corrective actions are dependent on the ability to mobilize the field team and whether the data to be collected is necessary to meet the required QA objectives (e.g., the holding time for samples is not exceeded). If corrective action requests are not in complete accordance with approved project planning documents, the EPA will be consulted and concurrence will be obtained before the change is implemented. Corrective actions of this type will be documented by the BSB QA Officer on a Corrective Action Report (Attachment 3) and will be included in any subsequent reports.

4.3 Reports to Management

Reports will be submitted according to the project schedule in Section 2.5.3. After field evaluations are complete, a recommendation summary report will be prepared to provide a summary of trigger items identified, erosion and vegetation score, and proposed type of corrective action for each site evaluated. Summary reports are provided in a tabular format for ease of review and quantification of findings (Section 2.5.3).

An annual summary report of all maintenance performed to reclaimed areas will be prepared by BSB. As detailed in Section 2.5.3, the report will include summaries of annual BRES evaluations, trigger items identified and implemented on a site, where CAPs are implemented, approved CAPs, and details of work completed at each site. Each annual report will include work completion summaries and typically include documentation of the materials used, their source, quantity, and final site condition. The report will describe specific field activities performed during implementation of the QAPP and the characteristics of the remedial action completed.

4.4 Annual Revision to QAPP

The BSB Environmental and Operations Division Manager, or designated alternate, will review this QAPP annually and make pertinent revisions. Updates will include pertinent data listed below, will be included in Attachment 4, and will be stored in the program database (refer to BPSOU DMP).

- Sample data – sites and specific sample locations and results.
- Field evaluations – completed field evaluations.
- Corrective action – description of construction activities and corrective action implemented to existing sites.
- Reclaimed sites boundary adjustments – any proposed site boundary adjustments to existing sites based on field evaluation data or proposed new boundaries.
- Any deviations from the approved QAPP.

5.0 DATA REVIEW AND USABILITY

This section lists the final project checks conducted after the data collection phase of the project is complete to confirm that the data obtained meet the project objectives and to estimate the effect of any deviations on data usability. The data review/validation process under this QAPP is streamlined to support the post-ROD (EPA, 2006a) decision-making process. Collection, analysis, and validation of data will be completed in accordance with the BPSOU QMP (Atlantic Richfield, 2016) and any project-specific maintenance and monitoring plan.

5.1 Data Review and Verification

This section lists the process to review and verify field data and complete internal laboratory data reporting.

5.1.1 Field Data Review

All GIS field information will be saved to ArcGIS Online as well as the project database to accurately track and manage completion of maintenance work, materials used, equipment, and daily logs. Field personnel will enter raw field data directly to the field tablets linked to the online database, where the data will be reviewed for accuracy and completeness by the BSB Database Manager before the records become final. Refer to the BPSOU DMP for details on the database. The overall quality of the field data from each assessment will be further evaluated during the data reporting. The field data will be reviewed periodically by the QAM, or designated alternate. Database entries will be reviewed for accuracy and completeness. Electronic files of field measurement data will be maintained as part of the project's quality records.

5.1.2 Field Data Quality Control

Personnel from BSB will complete QC check on field data at various stages. The QC process is instituted to ensure integrity of data used to make decisions specifically related to vegetative cover and transfer of field data. Field data QC protocol consists of reviewing field data and comparing quantitative field measurements of ground cover to visual estimates for comparability. Field personnel enter the data directly from the field on field tablets linked to the database. This will allow office staff access to the same data in real time. The information will be reviewed and verified by the database administrator; ensuring field information is reviewed by a person other than the person who entered the data. This step ensures the information is free of transcription errors and allows corrections before the information is finalized. Data from field evaluations will be cross referenced with spatial data collected in the field to ensure field evaluations capture both quantitative findings and specify the precise location where the trigger item occurs on the site.

At the completion of the field evaluation cycle, 10% of the site boundary polygons will be randomly selected for field verification using the modified point intercept method (described in the BRES) by personnel other than the previous evaluator. If the precision target is not met, the sites will be reevaluated.

The QAM will determine whether the DQOs have been met and determine the data completeness for the project. Data quality review related to site evaluations is a process to determine if the data meet project-specific DQOs. The data quality review will include verification of the following:

- Compliance with the QAPP.
- Completeness of field evaluations.
- Completed construction activities in accordance with CAPs.

Corrections may be made to accurately capture completion of work. Corrections may include minor adjustments of boundary mapping information to match existing topography or boundary delineations, and material quantities reported. Corrections will be reported to the BSB Data Management Division Manager and included in the annual reporting (Section 2.5.3).

5.1.3 Laboratory Data Review

Internal laboratory data review procedures will follow each laboratory’s quality management plan. At a minimum, paper records will be maintained by the analysts to document sample identification number and the sample tag number with sample results and other details, such as the analytical method used (e.g., method SOP number), name of analyst, the date of analysis, matrix sampled, reagent concentrations, instrument settings and the raw data. These records will be signed and dated by the analyst. Secondary review of these records by the Laboratory Supervisor (or designee) will take place prior to final data reporting. The laboratory is responsible for assigning appropriate flags/qualifiers in accordance with the analytical method and internal laboratory SOPs.

5.1.4 Laboratory Data Reporting Requirements

The laboratory will prepare hard copy data packages for transmittal of results. At a minimum, the data packages will include the case narrative, sample results, units, and QC sample results. Standard data packages will be transmitted to BSB within 14 days of laboratory sample receipt.

The laboratory will prepare electronic data packages for transmittal of results and associated QC information to Atlantic Richfield, or their designee, in general accordance with the EPA CLP SOW (EPA, 2016). Deviations from these specifications may be acceptable provided the report presents all the requested types of information in an organized, consistent and readily reviewable format.

An additional responsibility of the BSB Data Management Division Manager will be to determine whether the DQOs have been met and determine the data completeness for the project.

The data quality review, to determine if the data meet project-specific DQOs, will include verification of the following:

- Proper sample collection and handling procedures.
- Field QC results.
- Laboratory blank analysis.
- Detection limits.
- Laboratory duplicates.
- Laboratory data package.
- Data completeness and format
- Data qualifiers assigned by the laboratory.

Qualifiers that may be applied to the data include the following:

U	The analyte was analyzed for but was not detected above the reporting limit.
J	The analyte was positively identified; the associated numerical value is an estimate of the concentration of the analyte in the sample.

5.2 Data Validation

Analytical data will be validated by an independent third-party person not involved with the data generation or sample collection and the validation will follow EPA National Functional Guidelines (EPA, 2020). Level 2 validation packages will be provided at a rate of 1 data package per every 10 data packages received. Field data will be reviewed and validated using the Level A/B validation checklist (Attachment 3).

6.0 REFERENCES

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FIGURES

Figure 1. BRES Evaluation Reclaimed, Unreclaimed, and Insufficiently Reclaimed Areas [Boundaries]

Figure 2. BPSOU Reclaimed Areas Program Organization and Communication Structure

Figure 3. Reclaimed Areas Evaluation, Corrective Action, and Reporting Cycle

LEGEND

▨ UNRECLAIMED SITES

▤ INSUFFICIENTLY RECLAIMED SITES

🟦 BRES SITES

□ BPSOU BOUNDARY

N

DISPLAYED AS:
PROJECTION/ZONE: MSP
DATUM: NAD 83
UNITS: INTN'L FEET
SOURCE: PIONEER/BSB

0 400 800 1,600 2,400 3,200
Feet

FIGURE 1

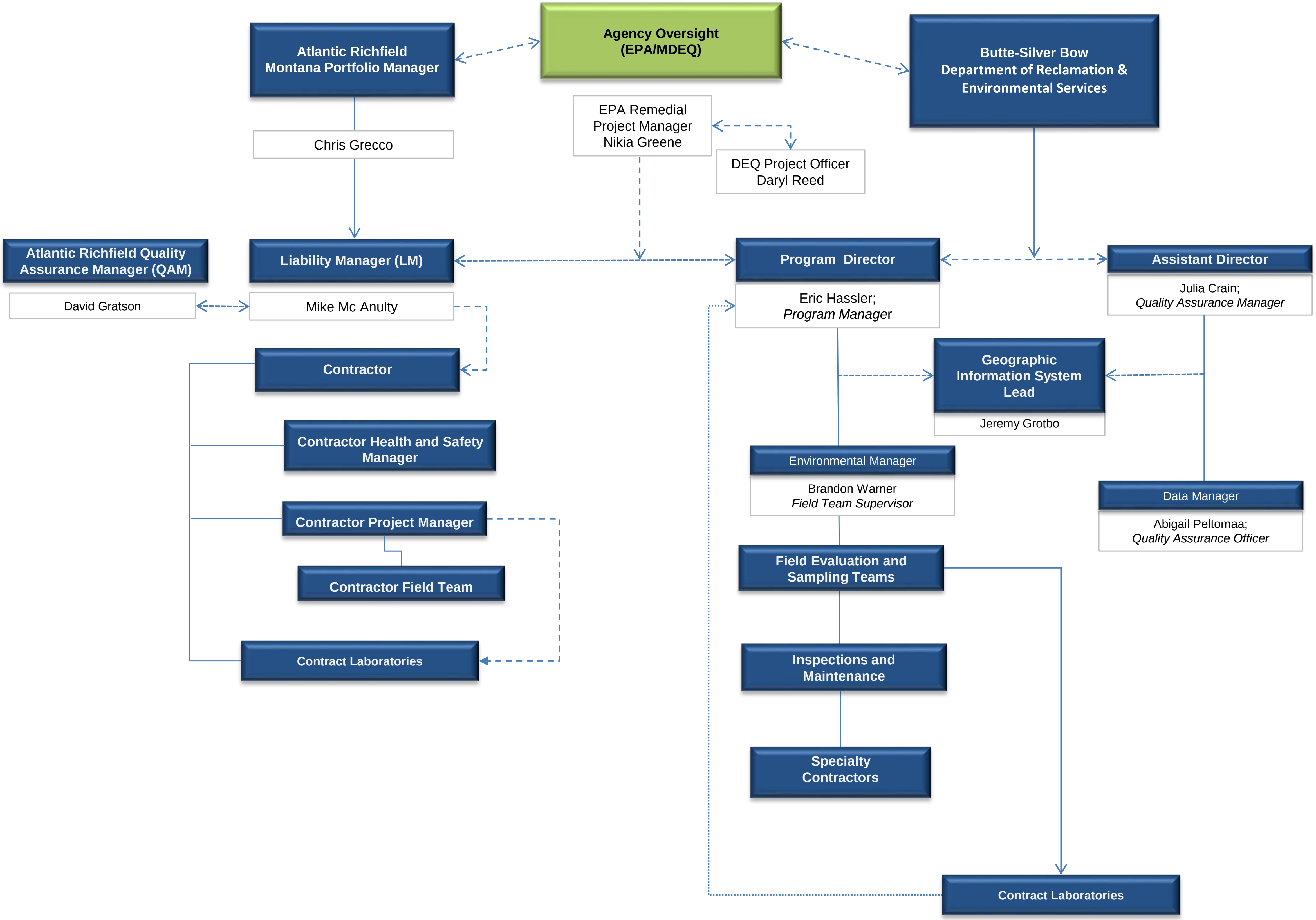
PIONEER
TECHNICAL SERVICES, INC.

DATE: 8/10/2018

BRES EVALUATION
RECLAIMED,
UNRECLAIMED, AND
INSUFFICIENTLY
RECLAIMED AREAS

Path: P:\ARCO\BPSOU\2018\Reclaimed Areas\GIS\Project\Reclaimed_Unreclaimed_Insufficient_7_2018.mxd

Figure 2. BPSOU Reclaimed Areas Program Organization and Communication Structure



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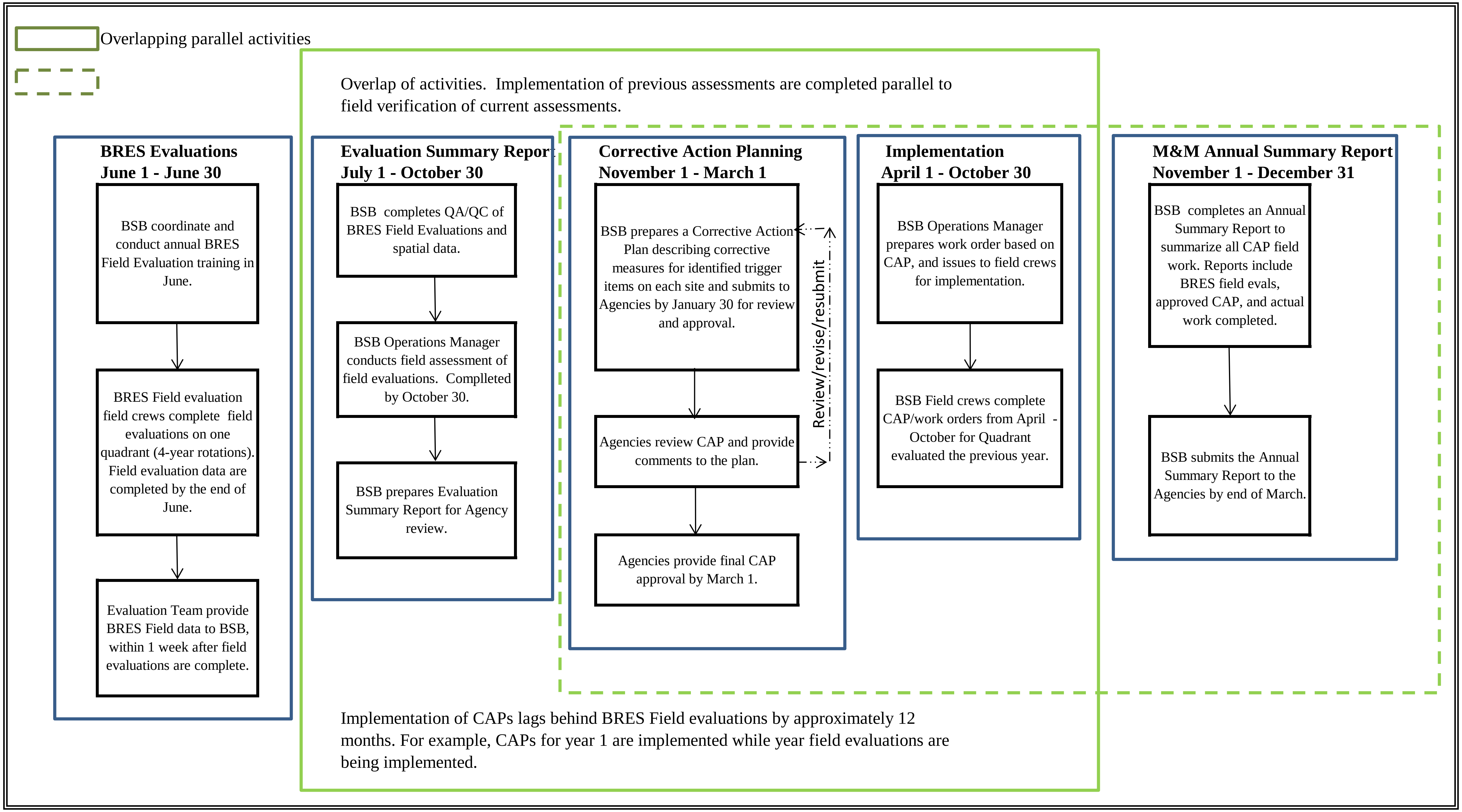
Specialty Contractors

Contract Laboratories



Overlapping parallel activities

Overlap of activities. Implementation of previous assessments are completed parallel to field verification of current assessments.

BRES Evaluations
June 1 - June 30

BSB coordinate and conduct annual BRES Field Evaluation training in June.

BRES Field evaluation field crews complete field evaluations on one quadrant (4-year rotations). Field evaluation data are completed by the end of June.

Evaluation Team provide BRES Field data to BSB, within 1 week after field evaluations are complete.

Evaluation Summary Report
July 1 - October 30

BSB completes QA/QC of BRES Field Evaluations and spatial data.

BSB Operations Manager conducts field assessment of field evaluations. Compllected by October 30.

BSB prepares Evaluation Summary Report for Agency review.

Corrective Action Planning
November 1 - March 1

BSB prepares a Corrective Action Plan describing corrective measures for identified trigger items on each site and submits to Agencies by January 30 for review and approval.

Agencies review CAP and provide comments to the plan.

Agencies provide final CAP approval by March 1.

Implementation
April 1 - October 30

BSB Operations Manager prepares work order based on CAP, and issues to field crews for implementation.

BSB Field crews complete CAP/work orders from April - October for Quadrant evaluated the previous year.

M&M Annual Summary Report
November 1 - December 31

BSB completes an Annual Summary Report to summarize all CAP field work. Reports include BRES field evals, approved CAP, and actual work completed.

BSB submits the Annual Summary Report to the Agencies by end of March.

Implementation of CAPs lags behind BRES Field evaluations by approximately 12 months. For example, CAPs for year 1 are implemented while year field evaluations are being implemented.

Figure 3. Reclaimed Areas Evaluation, Corrective Action, and Reporting Cycle.

Attachment 1
Reclaimed Areas List by Quadrant

Reclaimed, Unreclaimed, and Insufficiently Reclaimed Areas List in BPSOU.

Reclaimed Areas - Quadrant 1

Site Name	Reclaimed ID	BRES No.
Atlantic-1	Q1-RC3	30
Belle of Butte	Q1-RC4	8
Clark Street Dump	Q1-RC6	9
Corra 2 Dump	Q1-RC7	32
Curry	Q1-RC8	16
East Gray Rock	Q1-RC9	40, 41, 42, 43
GMMIA Phase I - North	Q1-RC10	NA
GMMIA Phase I - South	Q1-RC11	NA
GMMIA Repository	Q1-RC12	NA
Goldsmith Dumps - East	Q1-RC13	161
Goldsmith Dumps - West	Q1-RC14	161
Josephine Shaft	Q1-RC15	37
Lexington Dump	Q1-RC16	29
Lexington Dump North	Q1-RC17	29N
Magna Charta Lessee Dumps	Q1-RC18	11
Moose Dump	Q1-RC19	12
North Alice Culvert	Q1-RC20	177
Paymaster	Q1-RC21	17
Sister Dump	Q1-RC22	38
South Corra 2 and Rock Island Dumps	Q1-RC23	32S, 39
Twilight East	Q1-RC24	24
Venus Dump	Q1-RC25	25
Walkerville Playground	Q1-RC27	20
Waste Dump #5	Q1-RC28	31
West Gray Rock	Q1-RC29	40, 41, 42, 43
Blue Wing Dump (Engineered Cover)	Q1-RC30	19

Quadrant 1 Third Party Ownership

NAME	Reclaimed ID	BRES_ID
Eveline Dump	Q1-TP1	34
Leathers Property	Q1-TP2	178
Leathers Property West	Q1-TP3	178W
Ralph Sr	Q1-TP4	179
Rising Star Dumps West	Q1-TP5	15W
Rising Star Dumps East	Q1-TP6	15E

Reclaimed Areas - Quadrant 2

Site Name	Reclaimed ID	BRES No.
Blaine Center	Q2-RC1	171
Buffalo South and Silver Hill Dump	Q2-RC2	67,174
Buffalo, Kennedy, Poulin, and Spence Dumps	Q2-RC3	53,54,55,56
Del Monte & Garfield	Q2-RC4	35,45
La Platta Gulch	Q2-RC5	36
Little Mina	Q2-RC6	59
Little Mina-1	Q2-RC7	57
Little Mina-2	Q2-RC8	68
Missoula Mine	Q2-RC9	46
Missoula Mine East	Q2-RC10	46E
Moscow Dump	Q2-RC11	52
Mountain Con Mine - North	Q2-RC12	58, 60, 60A, 60B, 60C, 60D, 60E, 60F, 61N, 61E, 61W, 61S, 181
Old Glory	Q2-RC13	49
Old Glory West (Incl.)	Q2-RC14	48
Ravin	Q2-RC15	47
Upper Missoula Gulch	Q2-RC16	175
Wappello Dump	Q2-RC17	27
Mountain Con Mine - South	Q2-RC18	58, 60, 60A, 60B, 60C, 60D, 60E, 60F, 61N, 61E, 61W, 61S, 181

Quadrant 2 Third Party Ownership

Site Name	Reclaimed ID	BRES No.
Jennie Dell	Q2-TP1	33
Cripple Dump	Q2-TP2	26

Reclaimed Areas - Quadrant 4

Site Name	Reclaimed ID	BRES No.
Bonanza Dump	Q4-RC1	120, 120E
Catch Basin 08	Q4-RC2	CB08
Catch Basin 09	Q4-RC3	CB09
Clark Mill Tailings	Q4-RC4	155, 155E
Colorado Smelter	Q4-RC5	150
Colorado Smelter North	Q4-RC6	150N
Emma Dump	Q4-RC7	132
Kaw at Casey	Q4-RC8	1796
Metro Storm Drain	Q4-RC9	2310
Ophir Dump	Q4-RC10	136
Timber Butte Mill	Q4-RC11	156
Washoe Sampling Works	Q4-RC12	135
Charlie Judd Park (Engineered Cover)	Q4-RC13	142

Quadrant 4 Third Party Ownership

Site Name	Reclaimed ID	BRES No.
Maryland Ave & Iron St	Q4-TP1	1656
Star West Dump	Q4-TP2	134
Dexter Mill	Q4-TP3	133
Timber Butte Mill	Q4-TP4	156

Reclaimed Areas - Quadrant 3

Site Name	Reclaimed ID	BRES No.
Anselmo-Timber Yard Slope	Q3-RC1	71N
Anselmo Dump	Q3-RC2	70
Anselmo Mineyard	Q3-RC3	71
BA&P Trail Section A (1)	Q3-RC4	2330
BA&P Trail Section A (2)	Q3-RC5	2330
BA&P Trail Section A (3)	Q3-RC6	2330
BA&P Trail Section B	Q3-RC7	2340
BA&P Trail Section C	Q3-RC8	2350
BA&P Trail Section D	Q3-RC9	2360
BA&P Trail Section E	Q3-RC10	2370
BA&P Trail Section F	Q3-RC11	2380
BA&P Trail Section G	Q3-RC12	2390
Belmont Hoist	Q3-RC13	116N
Blue Jay Mine	Q3-RC14	101
Butte New England	Q3-RC15	115
Caledonia Triangle	Q3-RC16	172
Cellar Dirt Dump	Q3-RC17	82
Clear Grit Dump	Q3-RC18	81
Donkey Hill	Q3-RC19	72S
Hornet Addition	Q3-RC20	1503
Jasper Dump	Q3-RC21	73
Late Acquisition	Q3-RC22	79
Mandan Park Play Area	Q3-RC23	84
National Dump	Q3-RC24	75
New Era 1&2 – Downey Shafts	Q3-RC25	72
North Syndicate Pit	Q3-RC26	159, 160
Original Mine Yard	Q3-RC27	78
PA020 Dump	Q3-RC28	77
Parrot Shop, Dump, & Mine	Q3-RC29	97, 97S, 97S2, 97S3
Rialto Dump	Q3-RC30	94
Steward Mine Yard	Q3-RC31	83, 83A, 83B, 83C, 83D, 83F
Syndicate and Tullamore Dumps	Q3-RC32	180, 160S
Syndicate Pit	Q3-RC33	51
Travona Dump	Q3-RC34	121
Washoe Dump	Q3-RC35	96
Waste Dump #20	Q3-RC36	76
Waste Dump #37	Q3-RC37	90
West Gagnon Dump	Q3-RC38	74
West Ruby Dump	Q3-RC39	66
West Steward Parking Lot	Q3-RC40	80
Anderson Shaft (Engineered Cover)	Q3-RC41	117
Belmont Mine Yard (Engineered Cover)	Q3-RC42	116
Emma Shaft (Engineered Cover)	Q3-RC44	114
PA009 Dump (Engineered Cover)	Q3-RC43	110, 110N, 110S, 110E

Quadrant 3 Third Party Ownership

NAME	Reclaimed ID	BRES No.
Alliance Dump	Q3-TP1	128
Anderson Shaft - NE	Q3-TP2	117E
Capri Motel - Arctic Dump	Q3-TP3	100
Christmas	Q3-TP4	1501
Lizzie Shaft	Q3-TP5	105
Colorado Dump (Shaft)	Q3-TP6	104
Hoy-Hickey Shafts	Q3-TP7	106
Hesperus (Mercury St)	Q3-TP8	1542
Henriett	Q3-TP9	1539
Garden Street Area	Q3-TP10	173
Green Copper Dump	Q3-TP11	126
Child Harold-2 Dump	Q3-TP12	125
Heaney Dump	Q3-TP13	129
Soudan - Gold Hill	Q3-TP14	93
Robert Emmett Dumps	Q3-TP15	91
Tension Dump	Q3-TP16	127

Unreclaimed Sites for Evaluation

Ref No.	Site Name	Description
UR-1	Between Ryan Rd. and Alice St.	Apparent mine waste located near the Mini Irvine BRES No. 2.
UR-2	East of Scrap H Point Rd. near Moose Dump.	Apparent mine waste located in the surrounding areas of Moose Dump BRES No. 12.
UR-3	South of Dewey Point Rd. and Rising Star	Apparent mine waste located near the Surprise Dump BRES No. 14.
UR-4	Northwest corner of Center St. and Idaho St.	Apparent mine waste.
UR-5	Northwest corner of N Montana St. and Ruby St.	Apparent mine waste located in the surrounding areas of Moscow Dump BRES No. 52.
UR-6	Northwest corner of E Granite St. and Arizona St. - Capri Motel parking lot	Apparent mine waste located in the parking lot of the Capri Motel of BRES No. 100.
UR-7	Southwest corner of E Granite St. and Covert St.	Apparent mine waste located near the Blue Jay BRES No. 101.
UR-8	Southwest corner of Madison St. and S Warren St.	Apparent mine waste located near the Anderson Shaft BRES No. 117.
UR-9	West of S Excelsior Ave. North of I-15	Apparent mine waste located near the Bonanza Dump BRES No. 120.
UR-10	East end of E. Iron St.	Apparent mine waste located near the Otisco Dump BRES No. 123.
UR-11	Northwest corner of Atlantic St. and E. 2nd St.	Apparent mine waste located near the Child Harold BRES No. 125.
UR-12	West end of Munich St. and South of I-15	Apparent mine waste located near the Un-Named Dump BRES No. 148.
UR-13	North of I-15 and west of Colorado Smelter North	Apparent mine waste located west of the Colorado Smelter North BRES No. 150N.
UR-14	East of Copper Mountain Complex	Apparent mine waste located in the surrounding areas of Clark Tailings East BRES No. 155E.
UR-15	South of Ryan Rd and West of 4th St.	Apparent mine waste located near the Gold Smith Dumps BRES No. 161.
UR-16	Jefferson St and S Warren Ave.	Apparent mine waste located near the Garden Street BRES No. 173.
UR-17	Surrounding Areas of Upper Missoula Gulch	Apparent mine waste located in the surrounding areas of Upper Missoula Gulch BRES No. 175.
UR-18	Southwest corner of Hornet St. and Alabama St.	Apparent mine waste located near the Hornet Addition BRES No. 1503.
UR-19	RARUS/Patriot railroad from Montana Street to S Arizona St.	Identified by BSB for evaluation.
UR-20	Park and Covert Streets (NW Corner)	Site identified by BSB as areas for evaluation.
UR-21	East Galena St. (300 Block)	Site identified by BSB as areas for evaluation.
UR-22	N. Arizona and E. Granite St. (NE Corner)	Site identified by BSB as areas for evaluation.
UR-23	New and Mahoney St. – Remaining areas	Storm water site identified in ROD; Portion reclaimed under UAO.
UR-24	Clark Mill and adjacent mill tailings	Site identified by BSB as areas for evaluation.
UR-25	Scrap H Point Rd. – South Ryan Rd. embankment	Site identified by BSB as areas for evaluation.
UR-26	Grove Creek from Hanson to Rowe Rd.	Site identified by BSB as areas for evaluation.
UR-27	W. Copper and N. Washington St.(400 Block)	Site identified by BSB as areas for evaluation.
UR-28	Waukesha St. (800 Block)	Site identified by BSB as areas for evaluation.
UR-29	Greens Apts. – Surrounding areas	Site identified by BSB as areas for evaluation.
UR-30	N. Henry Ave. and West Zarelda St. – SW Corner	Site identified by BSB as areas for evaluation.
UR-31	Big Butte VFD – Surrounding areas	Site identified by BSB as areas for evaluation.
UR-32	S. Colorado St. and W. Mercury St. – SE Corner	Site identified by BSB as areas for evaluation.
UR-33	I-15 and Excelsior St.	Site identified by BSB as areas for evaluation.
UR-34	Desperation Air Shaft – east of site	Site identified by BSB as areas for evaluation.
UR-35	Steward Parking Lot – South of site	Site identified by BSB as areas for evaluation.
UR-36	South Parrott Slope – unreclaimed areas	Site identified by BSB as areas for evaluation.
UR-37	Main St. and Mullen St. – NE Corner	Site identified by BSB as areas for evaluation.
UR-38	Isele	Site requested by Agencies as areas for evaluation.Source Area (FSUA-132.)
UR-39	Bell of Butte; Surrounding Areas	Area surrounding Belle of Butte, north and east identifed for evaluation

Legend

Reclaimed Areas / BRES

nsufficiently Reclaimed Sites for Further Evaluatio

Unreclaimed Sites

Insufficiently Reclaimed Sites for Further Evaluation

Site Name	BRES No	Year Reclaimed	Description	Site Name	BRES No	Year Reclaimed	Description
Belle of Butte	8	1987	Recontours above the shaft, capped and revegetated.	Washoe Dump	96	1985; 1998	Waste removal, regraded, capped, and revegetated. 1998 a 4-foot walking trail was installed. More revegetation.
Magna Carta Lessee Dump	11	1998	Waste removed, constructed storm water contro ditches, recontoured, capped and revegetated.	Colorado Dump	104	1986	Waste removed, recontoured, and revegetated.
Curry	16	1991	Waste removal, regraded, capped, and revegetated.	Lizzie Shaft	105	1980-82	NA
Lexington Dump	29	1988	Regraded, applied lime rock, capped and revegetated.	Travona Dump	121	1991	Waste removal, regraded, capped, and revegetated.
Atlantic 1	30	1991	Recontoured, recapped, revegetated.	Tension Dump	127	1990/91	Waste removed, recontoured, storm water control ditch installed, capped and revegetated.
Corra 2 Dump	32	1991	Regraded, applied lime rock, capped and revegetated.	Heaney Dump	129	1990/91	Waste removal, regraded, capped, and paved with asphalt.
Eveline	34	1991	Waste removal, regraded, capped, and revegetated.	Dexter Mill	133	1990/91	Waste removal, regraded, capped, and revegetated.
Laplatta Gulch	36	1988	Waste removal, regraded, capped, and revegetated.	Star West Dump	134	1991	Recontoured, capped and revegetated.
Missoula Mine	46	1994	Regraded and seeded.	Washoe Sampling Works	135	1991	Waste and debris removal, recontoured, closed shaft, capped and seeded.
Zella	50	1991	Waste removal, regraded, capped, and revegetated.	Timber Butte Mill	156	1989	Waste removed, recontoured, storm water control ditch installed, capped and revegetated.
Poulin	53	1985	Graded, capped and revegetated.	Waste Rock Dump	158	Unknown	No site summary
Soudan Dump	93	1995	Parking lot development. Recontoured, installed a retaining wall, covered, and revegetated.	Black Bird	1625	1998	Regraded, capped and revegetated.

Attachment 2
BRES Evaluation Field Form

BRES FIELD FORM

Site Name: ____________________ **Date:** __________

Team Members (Circle your name): ____

Number of Polygons: _____ Slope: __________ Aspect: __________ Area Description: ___________

Polygon Evaluation	**1**	**2**	**3**
Vegetation (% live)			
Erosion (BLM score)			
% live weedy species			

Vegetation: % of ground covered by:	**POLYGON** 1	2	3
Live (desirable) species			
*Live (undesirable weedy) species			
*Noxious weeds			
TOTAL % LIVE			
Litter			
Rocks > 2"			

*Up to 5% of undesirable species and 0% of noxious weeds may count toward live cover.

1. Percent live: please check appropriate category:

1 ☐ **0-20** ☐ **21-39** ☐ **40-100**
2 ☐ **0-20** ☐ **21-39** ☐ **40-100**
3 ☐ **0-20** ☐ **21-39** ☐ **40-100**

Species Present:	Dominant	Frequent	Infreq
Sheep fescue			
Crested wheatgrass			
Slender wheatgrass			
Yellow sweetclover			
Alfalfa			
Other:			
____________			
____________			
____________			
____________			

Use polygon number in boxes

Erosion (BLM Form)	**POLYGON** 1	2	3
Surface Litter			
Surface Rock Movement			
Pedestalling			
Flow Patterns			
Rills			
Gullies			
Soil Movement			

2. Total BLM score 1____, 2____, 3____. Please check appropriate category.

1 ☐ **0-55** ☐ **56-100**
2 ☐ **0-55** ☐ **56-100**
3 ☐ **0-55** ☐ **56-100**

Weeds Present:	Dominant	Frequent	Infreq.
Spotted knapweed			
Dalmation toadflax			
Cheatgrass			
Baby's breath			
Kochia			
Thistle			
Other:			
____________			
____________			
____________			

Use polygon number in boxes

Other BRES Trigger Items
Identify trigger areas (using #) on air photo

3. Site Edges: Are polygon edges (outer edges of site only) significantly different than remainder of the polygon? Y_____ N_____ (check applicable items)

☐ **lime rock barrier** ☐ **depositional area**
☐ **more weeds** ☐ **steeper slope**
☐ **increased erosion** ☐ **less vegetation**
☐ **gullies** ☐ **other**________________________

Estimate width of affected edge ________

4. Exposed Waste Material? Y_____ N_____

- Estimated pH_____
- Approximate area _____
- Number of areas with exposed waste _____

5. Is there evidence of: Y_____ N_____

☐ **bulk soil failure** ☐ **land slumps**
☐ **subsidence**

6. Barren Areas: Y_____ N_____

- At Least 75 ft² • Not a rock outcrop
- Less than 10 % total cover (live & litter)

Number of barren areas ______
Do barren areas cover over 25% of polygon? Y____N____
Polygon barren area(s) located in (circle) 1 2 3

7. Gullies (over 6" in depth):
Y_____ N_____
Are any gullies actively eroding?
Y_____ N_____
Number of gullies ______

Comments.

Additional Vegetation:

Species	Dominant	Frequent	Infreq

LABORATORY NAME

LABORATORY ADDRESS

CHAIN OF CUSTODY

WORK ORDER #

Company Name:	Project Manager:
Address:	Project Name and Number:
City: State: Zip:	Email Address:
Phone:	Purchase Order #:
Fax:	Sampler Name and Phone #:

ANALYSIS REQUESTED

REMARKS

Turnaround Time (TAT) / Reporting

- ☐ Standard
- ☐ Next Day*
- ☐ 2^{nd} Day*
- ☐ Other* ____________

*All rush order requests must have prior approval

- ☐ Phone
- ☐ Mail
- ☐ Fax
- ☐ Email

SAMPLE ID	LAB ID	DATE	TIME								
				☐	☐	☐	☐	☐	☐	☐	*All reporting will be via email unless otherwise specified*
				☐	☐	☐	☐	☐	☐	☐	
				☐	☐	☐	☐	☐	☐	☐	
				☐	☐	☐	☐	☐	☐	☐	
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				☐	☐	☐	☐	☐	☐	☐	
				☐	☐	☐	☐	☐	☐	☐	
				☐	☐	☐	☐	☐	☐	☐	

Inspection Checklist

Received Intact?	☐ Y	☐ N
Labels & Chains Agree?	☐ Y	☐ N
Containers Sealed?	☐ Y	☐ N
Cooler Sealed?	☐ Y	☐ N

Delivery Method: ______________________

Temperature (°C): ______________________

Preservative: ______________________

____

Date & Time: ______________________

Inspected By: ______________________

RELINQUISHED BY (Signature)	DATE	TIME	RECEIVED BY (Signature)	DATE	TIME
PRINTED NAME	COMPANY		PRINTED NAME	COMPANY	
RELINQUISHED BY (Signature)	DATE	TIME	RECEIVED BY (Signature)	DATE	TIME
PRINTED NAME	COMPANY		PRINTED NAME	COMPANY	
RELINQUISHED BY (Signature)	DATE	TIME	RECEIVED BY (Signature)	DATE	TIME
PRINTED NAME	COMPANY		PRINTED NAME	COMPANY	

Attachment 3
Standard Forms and Procedures

Standard Operating Procedures
Field Data Sheets
Manufacturer Instructions
Corrective Action Report Template
Product Documentation and User Guide - BRES

Attachment 3.1
Standard Operating Procedures

QAPP-SOP-01
COMPLETING PROJECT DOCUMENTATION

STATUS: Draft Final
DATE ISSUED:
12/2017
REVISION: 0
PAGE 1 of 3

PURPOSE	Establish a uniform procedure to safely, consistently, and effectively perform project field documentation tasks under the Reclaimed Areas within Butte Priority Soils Operating Unit (BPSOU).
SCOPE	This SOP establishes the requirements for documenting project data, records, field observations, etc. It is applicable for all field activities including sampling, field evaluations, scheduled and opportunistic maintenance activities.

WORK INSTRUCTIONS

The following instructions are intended to provide sufficient guidance to perform the task in a safe, accurate, and reliable manner. Should these instructions present information that is inaccurate or unsafe, operations personnel must bring the issue to the attention of the BSB Superfund Project Manager and the appropriate revisions made.

TASK	INSTRUCTIONS
1. Electronic Logbooks.	An electronic field device (iPad, electronic tablet, etc.) will be utilized by the Superfund PM, field team leader, or appropriate supervisor. The device will be used to record field activities, specific real-time field data such as spatial boundaries and locations where evaluations or maintenance work is performed, capture the work with photographs, and document team members on the site, project duration, and equipment used, and material quantities used, removed, and applied to the site. A summary of field activities is provided below. **Opportunistic Maintenance Observations:** Opportunistic maintenance observtions are noted by any member of the workforce to document any maintenance activity noted in the field. The **Scheduled Maintenance:** Maintenance work order will be completed electronically using the field device. Electronic photos will also be generated using the device to record appropriate stages of work through completion of work activities. **BRES Field Evaluations:** BRES field forms are downloaded from a central database to the device. Evaluators complete evaluations and note appropriate trigger items, evaluation scores, observations, etc. on the electronic form. Field personnel will enter data directly to the form and upload the form to the project database. **Field Sampling:** For any field sampling work, the following data should be recorded: 1. Sample location and number. 2. Sample type and amount collected. 3. Date and time of sample collection. 4. Type of sample preservation. 5. Note the type of sample, sample location, time/date, name of person for whom the split was collected, that person's company, and any other pertinent information. 6. Sampling method, particularly any deviations from the SOP.

	7. The laboratory where the samples will be sent. 8. Electronic copy of any Chain of Custody form.
2. Photographs.	Take photographs of field activities using the field device. Photographs should include a scale in the picture when practical. Telephoto or wide-angle shots will not be used, since they cannot be used in enforcement meetings. Field personnel will take photographs of sampling locations, field activities, and anything else to document site conditions, as necessary. Field photographs will be used to chronical the before, during, and after maintenance task activities. Photographs will be geotagged and uploaded to the project database. Field personnel will include additional notes and remarks to describe photos as appropriate. 1. The general direction faced taking the photo. 2. A brief description of the subject and/or the fieldwork portrayed in the picture. 3. Description of maintenance proposed.

DRAWINGS, DOCUMENTS, AND TOOLS/EQUIPMENT The following documents should be referenced to assist in completing the associated task.	
RELATED SOPs/PROCEDURES/ WORK PLANS	
TOOLS	Electronic field devices.
FORMS/CHECKLIST	

APPROVALS/CONCURRENCE By signing this document, all parties acknowledge the completeness and applicability of this SOP for its intended purpose. Also, by signing this document, it serves as acknowledgement that I have received training on the procedure and associated competency testing.	
MANAGER	**DATE**
LEAD OPERATOR	**DATE**
OPERATOR	**DATE**
OTHER	**DATE**

Revisions:

APPROVALS/CONCURRENCE

By signing this document, all parties acknowledge the completeness and applicability of this SOP for its intended purpose. Also, by signing this document, it serves as acknowledgement that I have received training on the procedure and associated competency testing.

Revision	Description	Date

QAPP-SOP-02

COMPLETING CHAIN OF CUSTODY FORMS FOR ENVIRONMENTAL SAMPLING

STATUS: Draft Final
DATE ISSUED: 12/2017
REVISION: 0
PAGE 1 of 3

PURPOSE	Establish a uniform procedure to safely, consistently, and effectively perform site sampling tasks at residences under the Reclaimed Areas within Butte Priority Soils Operating Unit (BPSOU).
SCOPE	This SOP establishes the requirements for documenting and maintaining environmental sample chain of custody from point of origin to receipt of sample at the analytical laboratory. It is applicable from the time of sample acquisition until custody of the sample is transferred to an analytical laboratory.

WORK INSTRUCTIONS

The following instructions are intended to provide sufficient guidance to perform the task in a safe, accurate, and reliable manner. Should these instructions present information that is inaccurate or unsafe, operations personnel must bring the issue to the attention of the Project Manager and the appropriate revisions made.

TASK	INSTRUCTIONS
1.Complete sample labeling on sample container(s).	1. Label sample containers with sample information including: • sample ID number and location; • sample date and time collected; • sample type; • sampler's name and organization; • preservatives added to sample; and • remarks, notes, observations as applicable. 2. Log sample information (list above) with the project field data records.
2. Initiate COC form.	1. The field team leader will initiate the Chain of Custody form. 2. Complete the top section of the form to include project contact information.
3. Complete list of samples being shipped.	1. Provide sample ID numbers, as provided on sample containers, date and time of collection, sample type, and analysis required. 2. Additional comments, material description, and analysis turnaround time can also be provided.
4. Complete Transfer of Custody.	1. The individual relinquishing custody of samples listed on the form must complete the "Relinquished By" line of the form. 2. The individual receiving the sample(s) will complete the "Received By" line of the form. 3. Signatures, printed names, company names, dates and times are required for all parties.
4. Shipping carrier custody.	Bill of Lading supplied by the carrier will be used to document the sample custody, and its identification number will be entered on the CoC. It is not necessary for courier personnel to sign the CoC.
6. Retain recordss	Upon transfer of custody, the sampling personnel who relinquished the samples will retain a copy (photocopy or electronic copy) of the signed CoC.

DRAWINGS, DOCUMENTS, AND TOOLS/EQUIPMENT

The following documents should be referenced to assist in completing the associated task.

RELATED SOP's / WORK PLANS	Project Documentation SOP Environmental Sampling SOP BPSOU Reclaimed Areas Quality Assurance Plan (QAPP)
FORMS/CHECKLIST	Chain of Custody Form

APPROVALS/CONCURRENCE By signing this document, all parties acknowledge the completeness and applicability of this SOP for its intended purpose. Also, by signing this document, it serves as acknowledgement that I have received training on the procedure and associated competency testing.	
MANAGER	**DATE**
LEAD OPERATOR	**DATE**
OPERATOR	**DATE**
OTHER	**DATE**
OTHER	**DATE**

Revisions:

Rev.	Description	Date	Approval

QAPP-SOP-03

FIELD EQUIPMENT DECONTAMINATION

PURPOSE	Establish a uniform procedure to safely, consistently, and effectively provide standard instructions for equipment decontamination for work performed under the Reclaimed Areas within Butte Priority Soils Operating Unit (BPSOU).
SCOPE	This practice has been prepared for the BSB Superfund personnel workforce and applies to decontamination of field equipment utilized during Superfund sampling, maintenance, or monitoring activities.

WORK INSTRUCTIONS

The following instructions are intended to provide sufficient guidance to perform the task in a safe, accurate, and reliable manner. Should these instructions present information that is inaccurate or unsafe, operations personnel must bring the issue to the attention of the BSB Superfund Project Manager and the appropriate revisions made.

TASK	INSTRUCTIONS
1. Setup decontamination station.	If pressurized or gravity flow water is available, attach a hose or piping to reach the decontamination area. If no water is available, four 5-gallon buckets can be used for cleaning most equipment. Label the buckets: gross wash; soap wash; DI rinse; final rinse. Lay out plastic or foil to place the cleaned equipment on to air dry. Pour approximately 2 ½ to 3 gallons of de-ionized (DI) water into each bucket. Add a **few** (1-3 drops) of Liquinox© soap to the bucket marked: soap wash.
2. Remove gross contamination.	Remove gross contamination using pressurized or gravity flow tap water, if available. If not, equipment will be scrubbed in the 5-gallon bucket of DI water marked: gross wash and a stiff brush (dedicated to the gross wash step).
3. Wash equipment.	Move the equipment to the 5-gallon bucket marked: soap wash. Wash equipment with a stiff brush (dedicated to the soap wash step).
4. Triple rinse equipment.	Triple rinse the equipment with DI water to remove any soap residue in the bucket marked: DI rinse.
5. Second Rinse with DI Water	Triple rinse the equipment again in the bucket marked: Final rinse.
6. Rinse equipment with chemicals.	In many cases, the tap water and de-ionized water rinses will be sufficient. Chemical rinses of the equipment may be required. For inorganic contaminants, a mixture of 10:1 nitric acid in distilled water (10 parts water to 1 part nitric acid) is commonly used. Spray bottles, clearly marked with the appropriate chemical name, are an acceptable means of rinsing most equipment. To perform the chemical rinse, hold the equipment over a collection container (5-gallon bucket or bowl) spray the piece of equipment inside and out starting at the top and working down to the bottom. Make sure that all workers and vehicles are upwind of the spray.
7. Air dry equipment.	Place equipment on plastic sheeting or foil to air dry.
8. Transport/ store equipment.	Wrap equipment in foil or plastic wrap to transport or store.
9. Triple rinse decontamination equipment.	Triple rinse equipment (i.e., brushes, buckets, tubs, etc.) used in the decontamination process with water, preferably pressurized.

10. Wash decontamination equipment.	Agitate the equipment used in the decontamination process in the soap/DI water solution.
11. Triple rinse decontamination equipment.	Triple rinse equipment with DI-water.
12. Store and label decontamination equipment.	Place equipment in appropriate areas, so they are used only for decontamination purposes. Label the equipment, if necessary.
13. Dispose of decontamination solutions or solids.	Mine waste contaminated material will be disposed of at the Butte Treatment Lagoons BRW Area drying bed area, or Mine Waste repository.

DRAWINGS, DOCUMENTS, AND TOOLS/EQUIPMENT

The following documents should be referenced to assist in completing the associated task.

DRAWINGS	NA
RELATED SOPs/PROCEDURES/ WORK PLANS	
TOOLS	Five 5-gallon buckets, tap water, stiff brushes, soap, de-ionized or distilled water, chemicals for chemical rinse (if required), plastic sheeting or foil, tarps, and sprayers (if available). If additional items for decontamination are needed, they will be listed on the SAP.
FORMS/CHECKLIST	

APPROVALS/CONCURRENCE

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MANAGER	**DATE**
LEAD OPERATOR	**DATE**
OPERATOR	**DATE**
OTHER	**DATE**

Revisions:

APPROVALS/CONCURRENCE

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Revision	Description	Date

QAPP-SOP-04
COMPOSITE SOIL SAMPLING

STATUS: Draft Final
DATE ISSUED: 12/2017
REVISION: 0
PAGE 1 of 3

PURPOSE	Establish a uniform procedure to safely, consistently, and effectively perform site sampling tasks under the Reclaimed Areas within Butte Priority Soils Operating Unit (BPSOU).
SCOPE	Work described in this procedure includes visual assessment and site documentation, sampling collection and handling, and chain of custody protocol required to complete routine tasks.

WORK INSTRUCTIONS

The following instructions are intended to provide sufficient guidance to perform the task in a safe, accurate, and reliable manner. Should these instructions present information that is inaccurate or unsafe, operations personnel must bring the issue to the attention of the Project Manager and the appropriate revisions made.

TASK	INSTRUCTIONS
1.Visual inspection of site and map	1. **Visually inspect the area** to verify locations of composite sample collection. 2. **Photograph** and document the "existing" condition of the specific areas identified for soils removal.
2. Verify utility locates	Verify the utility locate information by identifying where natural gas pipes or other utilities enter any structures on or near the property. (Ex. if street lights are present with no overhead lines.)
3. Verify sample locations	Verify sample locations provided in the site-specific SAP. Use an appropriate method of survey to locate and mark sample locations for geo referencing.
4.Sampling	1. Prepare and label a sample bag with the address and the location of the sample for each composite sample. 2. **Collect the samples from 3 depth intervals (placing each sample in the corresponding bag).** Collect 5 subsamples in an X pattern (if possible) for each composite sample section. a. The first composite sample will consist of subsamples from the 0-to-2 inch depth interval, b. The second composite sample will consist of subsamples from the 2-to-6 inch depth interval, and c. The third composite sample will consist of subsamples from the 6-to-18 inch depth interval. d. For areas in a covered with grass, collect the initial composite sub-sample from immediately beneath the vegetative mat (sod), or in the absence of vegetation 0 to 2 inches below ground surface (bgs). 3. Show the location of each subsample on the map.
5. Sampling areas in a covered with grass	1. Collect the initial composite sub-sample from immediately beneath the vegetative mat (sod), or in the absence of vegetation 0 to 2 inches below ground surface (bgs). 2. If a vegetative mat is present, separate it from the soil surface with a stainless steel knife or equivalent, shake and scrape the removed

	vegetative mat over the sample collection bowl to dislodge any mineral soil particles. Include all dislodged soil particles in the composite sample. 3. Collect the remaining subsamples as described above
6. Chain of Custody	**Prepare a chain-of-custody** for samples transferred to laboratory for analysis. Follow procedures outlined in *Completing Chain of Custody Forms SOP*.

DRAWINGS, DOCUMENTS, AND TOOLS/EQUIPMENT The following documents should be referenced to assist in completing the associated task.	
DRAWINGS	NA
RELATED SOP's / WORK PLANS	Completing Chain of Custody SOP Project Documentation SOP Operating XL3 X-Ray Analyzer SOP Equipment Decontamination SOP BPSOU Reclaimed Areas Quality Assurance Plan (QAPP)
FORMS/CHECKLIST	

APPROVALS/CONCURRENCE By signing this document, all parties acknowledge the completeness and applicability of this SOP for its intended purpose. Also, by signing this document, it serves as acknowledgement that I have received training on the procedure and associated competency testing.	
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OTHER	**DATE**

Revisions:

Rev.	Description	Date	Approval

QAPP-SOP-05

FIELD ANALYSIS USING
XL3 X-RAY FLUORESCENCE (XRF) ANALYZER

STATUS: Draft Final
DATE ISSUED: 12/2017
REVISION: 0
PAGE 1 of 4

PURPOSE	Establish a uniform procedure to safely, consistently, and effectively operating XL3 X-Ray Fluorescence (XRF) analyzer under the Reclaimed Areas within Butte Priority Soils Operating Unit (BPSOU).
SCOPE	This procedure has been prepared for the Butte Silver Bow Superfund personnel. All personnel conducting field XRF sampling shall be trained and competent in work described below.

WORK INSTRUCTIONS

The following instructions are intended to provide sufficient guidance to perform the task in a safe, accurate, and reliable manner. Should these instructions present information that is inaccurate or unsafe, operations personnel must bring the issue to the attention of the BSB Superfund Project Manager and the appropriate revisions made.

TASK	INSTRUCTIONS
1. Assemble XRF stand.	a. Open the case containing the stand and insert 4 legs into base of stand. b. Place stand on a solid, level surface.
2. Prep XRF sample for analysis.	a. Wearing latex or nitrile gloves, remove any large aggregate from the sample and place in a separate bag for disposal. For gravel or rocky soils, a sieve can be used to remove the large aggregates. If a sieve is used, it needs to be decontaminated between samples. Equipment Decontamination SOP for instructions. b. Consolidate the sample into the bottom of the baggie. c. Open the lid to the XRF stand and place sample inside, making sure that sample is flush against the opening on the inside of the XRF stand. d. Close the lid to the XRF stand.
3. Turn on XRF analyzer.	a. Open the XRF case and remove XRF gun from case. b. Slide XRF battery onto bottom of XRF gun handle. c. Press and hold power button (⏻) until XRF gun turns on and wait for system to start. d. Press where it says ‘press to logon.’ A warning message appears asking to verify that the user is aware of the radiation source in the XRF unit. e. Press ‘Yes’ to continue.
4. Log in and calibrate detector.	a. Type password (1234) when prompted. b. Click ‘E’ to log in. After logging in, a screen appears with 7 icons appears, this is the Main Menu screen. c. Tap the ‘System Check’ icon. d. Tap ‘Yes.’ e. The XRF unit will then go through an internal calibration. f. When the calibration is done, tap ‘CLOSE’ on the XRF gun to return to the Main Menu screen. **The detector should be calibrated at the start of each day of operation.**
5. Set up XRF run test.	a. Set parameters (e.g., analysis types, time, and analytes) required for the analysis as detailed in the XL3 user’s manual, Sampling and Analysis Plan (SAP), or Work Plan (WP). b. Once logged into XRF system, tap the ‘Analyze’ icon on XRF screen. A screen

	appears. c. On the next screen tap ‘Soils.’ d. On the next screen tap ‘Data Entry.’ A Data Entry screen appears showing several options (Sample Name, Sampler, Date, etc.). e. In the upper right hand corner, next to the ‘Sample Name’ icon, click the symbol that looks like a miniature keyboard to display a keyboard on the screen. f. Type in the sample name (do not press return yet). g. Insert XRF gun into the bottom of the XRF stand with the XRF gun handle pointing away from you. Be sure that the XRF gun is securely in place in the bottom of the stand. h. Press ‘return’ in the lower right corner of the keyboard screen. i. To activate the unit, pull the trigger on the gun handle. The analysis will take approximately 2 minutes to complete.
6. Record data.	a. After the XRF analysis is complete, results from the analysis will appear on the screen. b. Record the results and Test Number displayed on the screen; use the up and down arrows on the XRF gun to scroll through data. c. Open the lid on the XRF stand and remove the sample. d. Mark the sample baggie as “RAN” so that sample does not get analyzed twice. Place ran samples in a labeled box for storage and record keeping.
7. Run additional samples.	a. With the XRF gun still in the XRF stand, press the return button (↵) on the XRF gun. This will display the ‘Data Entry’ screen. b. On the Data Entry Screen, press the keyboard symbol located to the right of ‘Sample Name’ to display the keyboard. c. Type the next sample name (do not press return yet). d. Place the sample into the XRF stand and close the lid to the stand (as discussed in Task 2). e. Repeat the steps in Task 5 to activate the XRF unit. f. Repeat Tasks 6 and 7 until all samples are analyzed.
8. Turn off XRF.	a. After all samples have been analyzed, remove the XRF gun from the bottom of the stand (press and hold buttons on the side of the stand to allow XRF gun to be removed from stand). b. Press the return button (↵) on the XRF gun until the Main Menu screen appears. c. Press and hold the power button (⏻) until the XRF turns off. d. Remove the battery from the gun and place these items back into the appropriate case. e. Disassemble the XRF stand and place back into the appropriate case.
Quality Assurance/Quality Control (QA/QC) Requirements.	Required QA/QC tasks: 1. Run the Niton-supplied XRF blanks and NIST standards at the start of each day. 2. Record the results in the field logbook or on the XRF field datasheet or equivalents. If the results are not within the ranges supplied by NITON in the user manual, initiate troubleshooting tasks on the analyzer (refer to the user’s manual).

3. Run the blank and one standard QA/QC samples during sample analysis at the rate of 1 for every 20 samples analyzed. QA/QC includes analyzing a replicate sample every 20 samples and a duplicate sample (see the steps below).

Analyze a replicate sample (1 for every 20 samples analyzed)

1. After recording the initial reading for a sample, DO NOT remove the sample from the holder.
2. Restart the XRF gun and rerun the sample.
3. Record the information on the field data form or logbook as a replicate (or R sample). Replicates samples help track the precision of the XRF.

Analyze a duplicate sample (after every 20 samples analyzed)

1. After every 20 samples, analyze a duplicate sample by recording the results of the 20th sample.
2. Remove the sample bag from the XRF stand, remix the sample, and replace it in the XRF stand.
3. Reanalyze the sample.
4. Record the results as a duplicate (or D sample). Duplicates help to determine the precision of the XRF analysis as well as the homogeneity of the sample matrix.
5. Run a NITON-supplied blank or NIST standard after the replicate/duplicate QA/QC samples to monitor the accuracy of the XRF results.

DRAWINGS, DOCUMENTS, AND TOOLS/EQUIPMENT

The following documents should be referenced to assist in completing the associated task.

DRAWINGS	
RELATED SOPs/PROCEDURES/ WORK PLANS	Equipment Decontamination SOP.
TOOLS	XRF and hand tools.
FORMS/CHECKLIST	

APPROVALS/CONCURRENCE

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MANAGER	**DATE**
LEAD OPERATOR	**DATE**
OPERATOR	**DATE**

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OTHER	**DATE**

Revisions:

Revision	Description	Date

QAPP – SOP-6

SOIL pH FIELD TESTING

STATUS: Final
DATE ISSUED: 5/2018
REVISION: 0
PAGE 1 of 3

PURPOSE	To establish a uniform procedure to safely, consistently, and effectively perform in-soil pH testing at Reclaimed Areas within the BPSOU.
SCOPE	Work described in this procedure includes soil sampling using a hand-held soil pH meter. Associated steps workers are required to implement include: dig a shallow hole using hand tools, calibration, operate the meter, rinse, and record data.

WORK INSTRUCTIONS

The following instructions are intended to provide sufficient guidance to perform the task in a safe, accurate, and reliable manner. Should these instructions present information that is inaccurate or unsafe, operations personnel must bring the issue to the attention of the Project Manager and the appropriate revisions made.

TASK	INSTRUCTIONS
1. Sample Size	a. A minimum of one complete pH sample is recommended per ¼ acre.
2. Device operation	a. To turn the device on or off Press: On/Off button. b. To Freeze the device Press: Set/Hold.
3. Device Calibration	a. Connect the PH probe to the meter. b. Hold the On/Off button until Calibration is visible on the screen. c. Put the probe in 7.01 calibration solution. d. The meter will recognize the solution and calibrate. e. Once the calibration is recognized and stable, press: On/Off
4. Prepare to take a pH measurement	a. Connect the probe when the device is off. b. Remove the protective cap from the probe. c. Insert the probe into the sample. d. Wait until the "not stable" read out has turned off; and e. Record the measurement.
5. Dig hole and prepare for sampling	a. Determine location from engineering assessment. b. Using a hand trowel, dig a hole approximately 2 - 4 inches deep, discarding the top 2 inches of soil. c. Remove foreign, non-organic material, and break-up large material. d. Perforate the soil with the included soil drill to a depth of at least 8 inches. e. If the soil is dry, moisten with a small amount of distilled water. f. Rinse the probe with **tap water** (not distilled). g. Insert the probe slightly into the soil, making sure that it is in contact with the soil surfaces.
6. Record measurement; rinse and repeat.	a. Once the readings have stabilized record the measurement. b. Remove the probe from the hole, gently **clean off loose soil with your fingers**, then rinse the probe with **tap water;** **Caution: Avoid using a rag or cloth to prevent damage to the probe.** c. Repeat this procedure in several locations; then d. Average the results
7. Documentation	a. Prepare a map showing sampling location(s) and sample results. b. A field GPS device may be used to record sample locations.

DRAWINGS, DOCUMENTS, AND TOOLS/EQUIPMENT The following documents should be referenced to assist in completing the associated task.	
DRAWINGS	NA
RELATED SOP's / WORK PLANS	Manufacturer's Instructions
FORMS/CHECKLIST	

APPROVALS/CONCURRENCE By signing this document, all parties acknowledge the completeness and applicability of this SOP for its intended purpose. Also, by signing this document, it serves as acknowledgement that I have received training on the procedure and associated competency testing.	
MANAGER	**DATE**
LEAD OPERATOR	**DATE**
OPERATOR	**DATE**
OTHER	**DATE**
OTHER	**DATE**

Revisions:

Rev.	Description	Date	Approval

APPROVALS/CONCURRENCE By signing this document, all parties acknowledge the completeness and applicability of this SOP for its intended purpose. Also, by signing this document, it serves as acknowledgement that I have received training on the procedure and associated competency testing.			

PURPOSE	Establish a uniform procedure to safely, consistently, and effectively provide standard instructions for personal decontamination for work performed under the Reclaimed Areas within Butte Priority Soils Operating Unit (BPSOU).
SCOPE	This practice has been prepared for the BSB Superfund personnel workforce and applies to decontamination of field equipment utilized during Superfund sampling, maintenance, or monitoring activities.

WORK INSTRUCTIONS

The following instructions are intended to provide sufficient guidance to perform the task in a safe, accurate, and reliable manner. Should these instructions present information that is inaccurate or unsafe, operations personnel must bring the issue to the attention of the BSB Superfund Project Manager and the appropriate revisions made.

TASK	INSTRUCTIONS
1. **Wash/ Remove outer contaminated items.**	Remove nitrile or latex gloves by grasping the outside of the opposite glove near the wrist. Pull and peel the glove away from the hand, turning the glove inside out with the contaminated side now on the inside. Hold the removed glove in the opposite gloved hand. Slide one or two fingers of the ungloved hand under the wrist of the remaining glove. Peel glove off from the inside, creating a bag for both gloves. If wearing protective coveralls such as Tyvec suites, brush built up material off the suit, only if in designated decontamination zone. Unzip the coverall and begin rolling that outwards, rolling it down over your shoulders. Place both hands behind your back and pull down each arm until completely removed. Sit down and remove each shoe then roll the coveralls down (ensuring the contaminated side is not touched or comes into contact with clothing) over your knees until completely removed. If there is not a designated decontamination zone, remove personal protective equipment (PPE) carefully to contain material and place it in the appropriate disposal container. For instructions to remove additional PPE not described in this document, refer to the project's HASP. Wash with soap (nonphosphate) and tap water the outer, more heavily contaminated items, such as boots. Rinse the items in tap water.
2. **Wash inner contaminated items.**	If necessary, wash with soap (nonphosphate) and tap water the inner, less contaminated items. Rinse the items in tap water.
3. **Store/ transport items.**	Store/transport contaminated items in a separate designated area to prevent cross contamination prior to disposal.
4. **Dispose of contaminated items.**	Dispose of contaminated clothing and equipment in accordance with site/project, client, and/or federal and state requirements.

DRAWINGS, DOCUMENTS, AND TOOLS/EQUIPMENT The following documents should be referenced to assist in completing the associated task.	
DRAWINGS	NA
RELATED SOPs/PROCEDURES/ WORK PLANS	Equipment Decontamination
MATERIALS AND SUPPLIES	The BSB Superfund Office is the primary decontamination location and contains lavatory facilities (running water, soap, etc.). In general, the following items will be needed for decontamination in the field: soap, tap water, tarps, decontamination tubs, brushes, and sprayer. Additional items needed for decontamination, will be identified as required.
FORMS/CHECKLIST	NA

APPROVALS/CONCURRENCE By signing this document, all parties acknowledge the completeness and applicability of this SOP for its intended purpose. Also, by signing this document, it serves as acknowledgement that I have received training on the procedure and associated competency testing.	
MANAGER	**DATE**
LEAD OPERATOR	**DATE**
OPERATOR	**DATE**
OTHER	**DATE**

Revisions:

Revision	Description	Date

QAPP-SOP-08

SOIL SAMPLE PACKAGING

STATUS: Draft Final
DATE ISSUED: 5/2018
REVISION: 0
PAGE 1 of 3

PURPOSE	Establish a uniform procedure to consistently, and effectively perform soil and water sample packaging and shipping for samples obtained under Reclaimed Areas within Butte Priority Soils Operating Unit (BPSOU).
SCOPE	This SOP establishes the requirements for properly preparing soil samples for shipment to the analytical laboratory. It is applicable from the time of sample acquisition until custody of the sample is transferred to an analytical laboratory.

WORK INSTRUCTIONS

The following instructions are intended to provide sufficient guidance to perform the task in a safe, accurate, and reliable manner. Should these instructions present information that is inaccurate or unsafe, operations personnel must bring the issue to the attention of the Project Manager and the appropriate revisions made.

TASK	INSTRUCTIONS
1. **Place the sample containers in Ziploc bags.**	Based on the analytes requested (e.g., low level mercury, low level chromium, etc.), it may be necessary to place each filled sample container in separate Ziploc bags to prevent cross contamination; keep the container clean, dry, and isolated; and protect the sample label. In most cases, all sample containers collected from a specific sample location are placed in a large Ziploc bag and shipped together.
2. **Package the samples.**	Place samples in a cooler, which has been previously lined with a plastic bag. Surround the samples with non-contaminating packaging materials to reduce movement and absorb any leakage. Double bag the ice and place it in the cooler. Seal the plastic bag in the cooler to contain the samples, packing material, and ice.
3. **Review and sign Chain of Custody forms.**	The Field Team Leader or their designated representative will double check the Chain-of-Custody (CoC) forms to assure those samples recorded on the CoC forms are in the cooler. The Field Team Leader or the designated representative will then sign the CoC form to relinquish custody. One copy of the signed CoC form will remain with the Field Team Leader. Make a photocopy of the completed forms if there are no carbon copies available.
4. **Tape paperwork to cooler.**	Place paperwork in a sealed Ziploc bag and tape it to the inside of the cooler lid.
5. **Bag samples for separate analytical batches.**	If the shipping cooler contains more samples than can be analyzed in one analytical batch, the laboratory may request that the samples in the cooler be bagged for separate analytical batches. This may be necessary so that the appropriate Quality Control/ Quality Assurance samples are included in each analytical batch. In this case, fill out separate COC forms for each batch and include the forms in the appropriate plastic bags. Place the COC forms for each batch in a sealed Ziploc bag. The COC forms for each batch should be placed at the top of the plastic bag so that they are clearly visible to laboratory personnel when they open the plastic bags.
6. **Label the cooler.**	Label the cooler with the appropriate labels to describe the content of the cooler. Close the cooler and place the appropriate shipping labels (e.g., overnight shipping from Federal Express, UPS, or the U.S. Postal Service or equivalent) on the lid of the cooler.

DRAWINGS, DOCUMENTS, AND TOOLS/EQUIPMENT The following documents should be referenced to assist in completing the associated task.	
RELATED SOP's / WORK PLANS	Project Documentation SOP Environmental Sampling SOP Completing Chain of Custody SOP BPSOU Reclaimed Areas Quality Assurance Plan (QAPP)
FORMS/CHECKLIST	Chain of Custody Form

APPROVALS/CONCURRENCE By signing this document, all parties acknowledge the completeness and applicability of this SOP for its intended purpose. Also, by signing this document, it serves as acknowledgement that I have received training on the procedure and associated competency testing.	
MANAGER	**DATE**
LEAD OPERATOR	**DATE**
OPERATOR	**DATE**
OTHER	**DATE**
OTHER	**DATE**

Revisions:

Rev.	Description	Date	Approval

APPROVALS/CONCURRENCE

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BPSOU RECLAIMED AREAS M&M SMP-10

BOUNDARY REVISION/CREATION

STATUS: Final
DATE ISSUED: 11/16
REVISION: 1
PAGE 1 of 2

PURPOSE	To establish a uniform procedure to safely and effectively perform inspection and maintenance tasks at sites listed under the BPSOU Reclaimed Areas.
SCOPE	Work described in this procedure includes review of existing aerial photos and GIS data, site investigations and verification, and final boundary revisions.

WORK INSTRUCTIONS

The following instructions are intended to provide sufficient guidance to perform the task in a safe, accurate, and reliable manner. Should these instructions present information that is inaccurate or unsafe, operations personnel must bring the issue to the attention of the Project Manager and the appropriate revisions made.

TASK	INSTRUCTIONS
1.Aerial Photo Review	a. Evaluate aerial photo documentation to identify obvious areas that do not appear to be remediated, or remediation does not match existing boundaries. b. Review and verify existing boundary .shp files.
2.GIS Data Review	a. Perform verification of .shp boundaries by comparison with relevant documents related to boundary determination, site features, landmarks, etc. b. Make preliminary boundary adjustments as needed.
3. Site Visit	a. Perform field verifications utilizing GPS enabled devices. b. Physically walk the boundary while possessing the GPS enabled device (mobile phone, tablet, computer, etc.) to create log file of the boundary path.
4. Desktop comparison	a. Perform desktop comparison of proposed and field generated boundaries and match discrepancies. Submit boundary revisions to the QAM for review and approval.
5. Polygon and Boundary Revision	a. Finalize boundary delineations, and submit to EPA/DEQ for approval b. After EPA/DEQ approval of the boundary revision, upload BRES Quadrant Boundary to BSB database.
6. New boundary designation	a. Newly reclaimed areas require a newly created boundary. b. Assign new boundaries with a BRES Site Number, and quadrant number. c. Upload the .shp file to the BRES maintenance database and prepare for four-year review cycle.

DRAWINGS, DOCUMENTS, AND TOOLS/EQUIPMENT

The following documents should be referenced to assist in completing the associated task.

DRAWINGS	BRES Quadrant Maps, Aerials

RELATED SOP's / WORK PLANS	
FORMS/CHECKLIST	

APPROVALS/CONCURRENCE

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MANAGER	**DATE**
LEAD OPERATOR	**DATE**
OPERATOR	**DATE**
OTHER	**DATE**
OTHER	**DATE**

Revisions:

Rev.	Description	Date	Approval
1	Updated per Agency comments 4/5/2016	5/15/2019	

METHOD 6020A

INDUCTIVELY COUPLED PLASMA - MASS SPECTROMETRY

1.0 SCOPE AND APPLICATION

1.1 Inductively coupled plasma-mass spectrometry (ICP-MS) is applicable to the determination of sub-µg/L concentrations of a large number of elements in water samples and in waste extracts or digests (References 1 and 2). When dissolved constituents are required, samples must be filtered and acid-preserved prior to analysis. No digestion is required prior to analysis for dissolved elements in water samples. Acid digestion prior to filtration and analysis is required for groundwater, aqueous samples, industrial wastes, soils, sludges, sediments, and other solid wastes for which total (acid-leachable) elements are required.

1.2 ICP-MS has been applied to the determination of over 60 elements in various matrices. Analytes for which EPA has demonstrated the acceptability of Method 6020 in a multi-laboratory study on solid and aqueous wastes are listed below.

Element		CASRN[a]
Aluminum	(Al)	7429-90-5
Antimony	(Sb)	7440-36-0
Arsenic	(As)	7440-38-2
Barium	(Ba)	7440-39-3
Beryllium	(Be)	7440-41-7
Cadmium	(Cd)	7440-43-9
Calcium	(Ca)	7440-70-2
Chromium	(Cr)	7440-47-3
Cobalt	(Co)	7440-48-4
Copper	(Cu)	7440-50-8
Iron	(Fe)	7439-89-6
Lead	(Pb)	7439-92-1
Magnesium	(Mg)	7439-95-4
Manganese	(Mn)	7439-96-5
Mercury	(Hg)	7439-97-6
Nickel	(Ni)	7440-02-0
Potassium	(K)	7440-09-7
Selenium	(Se)	7782-49-2
Silver	(Ag)	7440-22-4
Sodium	(Na)	7440-23-5
Thallium	(Tl)	7440-28-0
Vanadium	(V)	7440-62-2
Zinc	(Zn)	7440-66-6

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[a] Chemical Abstract Service Registry Number

Acceptability of the method for an element was based upon the multi-laboratory performance compared with that of either furnace atomic absorption spectrophotometry or inductively coupled plasma-atomic emission spectrometry. It should be noted that one multi-laboratory study was conducted in 1988 and advances in ICP-MS instrumentation and software have been made since that time and additional studies have been added with validation and improvements in performance of the method. Performance, in general, exceeds the multi-laboratory performance data for the listed elements. It is expected that current performance will exceed the multi-laboratory performance data for the listed elements (and others) that are provided in Section 13.0. Instrument detection limits, sensitivities, and linear ranges will vary with the matrices, instrumentation, and operating conditions. In relatively simple matrices, detection limits will generally be below 0.1 µg/L. Less sensitive elements (like Se and As) and desensitized major elements may be 1.0 µg/L or higher.

1.3 If Method 6020 is used to determine any analyte not listed in Section 1.2, it is the responsibility of the analyst to demonstrate the accuracy and precision of the method in the waste to be analyzed. The analyst is always required to monitor potential sources of interferences and take appropriate action to ensure data of known quality (see Section 9.4). Other elements and matrices may be analyzed by this method if performance is demonstrated for the analyte of interest, in the matrices of interest, at the concentration levels of interest in the same manner as the listed elements and matrices (see Sec. 9.0).

1.4 Use of this method should be relegated to spectroscopists who are knowledgeable in the recognition and in the correction of spectral, chemical, and physical interferences in ICP-MS.

1.5 An appropriate internal standard is required for each analyte determined by ICP-MS. Recommended internal standards are ^{6}Li, ^{45}Sc, ^{89}Y, ^{103}Rh, ^{115}In, ^{159}Tb, ^{165}Ho, and ^{209}Bi. The lithium internal standard should have an enriched abundance of ^{6}Li, so that interference from lithium native to the sample is minimized. Other elements may need to be used as internal standards when samples contain significant native amounts of the recommended internal standards.

2.0 SUMMARY OF METHOD

2.1 Prior to analysis, samples which require total ("acid-leachable") values must be digested using appropriate sample preparation methods (such as Methods 3005 - 3052).

2.2 Method 6020 describes the multi-elemental determination of analytes by ICP-MS in environmental samples. The method measures ions produced by a radio-frequency inductively coupled plasma. Analyte species originating in a liquid are nebulized and the resulting aerosol is transported by argon gas into the plasma torch. The ions produced by high temperatures are entrained in the plasma gas and introduced, by means of an interface, into a mass spectrometer. The ions produced in the plasma are sorted according to their mass-to-charge ratios and quantified with a channel electron multiplier. Interferences must be assessed and valid corrections applied or the data flagged to indicate problems. Interference correction must include compensation for background ions contributed by the plasma gas, reagents, and constituents of the sample matrix.

3.0 DEFINITIONS

Refer to Chapter One and Chapter Three for a listing of applicable definitions.

4.0 INTERFERENCES

4.1 Isobaric elemental interferences in ICP-MS are caused by isotopes of different elements forming atomic ions with the same nominal mass-to-charge ratio (m/z). A data system must be used to correct for these interferences. This involves determining the signal for another isotope of the interfering element and subtracting the appropriate signal from the analyte isotope signal. Since commercial ICP-MS instruments nominally provide unit resolution at 10% of the peak height, very high ion currents at adjacent masses can also contribute to ion signals at the mass of interest. Although this type of interference is uncommon, it is not easily corrected, and samples exhibiting a significant problem of this type could require resolution improvement, matrix separation, or analysis using another verified and documented isotope, or use of another method.

4.2 Isobaric molecular and doubly-charged ion interferences in ICP-MS are caused by ions consisting of more than one atom or charge, respectively. Most isobaric interferences that could affect ICP-MS determinations have been identified in the literature (References 3 and 4). Examples include $^{75}ArCl^+$ ion on the ^{75}As signal and MoO^+ ions on the cadmium isotopes. While the approach used to correct for molecular isobaric interferences is demonstrated below using the natural isotope abundances from the literature (Reference 5), the most precise coefficients for an instrument can be determined from the ratio of the net isotope signals observed for a standard solution at a concentration providing suitable (<1 percent) counting statistics. Because the ^{35}Cl natural abundance of 75.77 percent is 3.13 times the ^{37}Cl abundance of 24.23 percent, the chloride correction for arsenic can be calculated (approximately) as follows (where the $^{38}Ar^{37}Cl^+$ contribution at m/z 75 is a negligible 0.06 percent of the $^{40}Ar^{35}Cl^+$ signal):

Corrected arsenic signal (using natural isotopes abundances for coefficient approximations) =
(m/z 75 signal) - (3.13) (m/z 77 signal) + (2.73) (m/z 82 signal),

where the final term adjusts for any selenium contribution at 77 m/z,

NOTE: Arsenic values can be biased high by this type of equation when the net signal at m/z 82 is caused by ions other than $^{82}Se^+$, (e.g., $^{81}BrH^+$ from bromine wastes [Reference 6]).

Similarly,

Corrected cadmium signal (using natural isotopes abundances for coefficient approximations) =
(m/z 114 signal) - (0.027)(m/z 118 signal) - (1.63)(m/z 108 signal),

where last 2 terms adjust for any $^{114}Sn^+$ or $^{114}MoO^+$ contributions at m/z 114.

NOTE: Cadmium values will be biased low by this type of equation when $^{92}ZrO^+$ ions contribute at m/z 108, but use of m/z 111 for Cd is even subject to direct ($^{94}ZrOH^+$) and indirect ($^{90}ZrO^+$) additive interferences when Zr is present.

NOTE: As for the arsenic equation above, the coefficients could be improved. The most appropriate coefficients for a particular instrument can be determined from the ratio of the net isotope signals observed for a standard solution at a concentration providing suitable (<1 percent) counting precision.

The accuracy of these types of equations is based upon the constancy of the OBSERVED isotopic ratios for the interfering species. Corrections that presume a constant fraction of a molecular ion relative to the "parent" ion have not been found (Ref. 7) to be reliable, e.g., oxide levels can vary with operating conditions. If a correction for an oxide ion is based upon the ratio of parent-to-oxide ion intensities, the correction must be adjusted for the degree of oxide formation by the use of an appropriate oxide internal standard previously demonstrated to form a similar level of oxide as the interferent. For example, this type of correction has been reported (Ref. 7) for oxide-ion corrections using ThO^+/Th^+ for the determination of rare earth elements. The use of aerosol desolvation and/or mixed gas plasmas have been shown to greatly reduce molecular interferences (Ref. 8). These techniques can be used provided that method detection limits, accuracy, and precision requirements for analysis of the samples can be met.

4.3 Additionally, solid phase chelation may be used to eliminate isobaric interferences from both element and molecular sources. An on-line method has been demonstrated for environmental waters such as sea water, drinking water and acid decomposed samples. Acid decomposed samples refer to samples decomposed by methods similar to methods 3052, 3051, 3050 or 3015. Samples with percent levels of iron and aluminum should be avoided. The method also provides a method for preconcentration to enhance detection limits simultaneously with elimination of isobaric interferences. The method relies on chelating resins such as imminodiacetate or other appropriate resins and selectively concentrates the elements of interest while eliminating interfering elements from the sample matrix. By eliminating the elements that are direct isobaric interferences or those that form isobaric interfering molecular masses, the mass region is simplified and these interferences can not occur. The method has been proven effective for the certification of standard reference materials and validated using SRMs (References 13-15). The method has the potential to be used on-line or off-line as an effective sample preparation method specifically designed to address interference problems.

4.4 Physical interferences are associated with the sample nebulization and transport processes as well as with ion-transmission efficiencies. Nebulization and transport processes can be affected if a matrix component causes a change in surface tension or viscosity. Changes in matrix composition can cause significant signal suppression or enhancement (Ref. 9). Dissolved solids can deposit on the nebulizer tip of a pneumatic nebulizer and on the interface skimmers (reducing the orifice size and the instrument performance). Total solid levels below 0.2% (2,000 mg/L) have been currently recommended (Ref. 10) to minimize solid deposition. An internal standard can be used to correct for physical interferences, if it is carefully matched to the analyte so that the two elements are similarly affected by matrix changes (Ref. 11). When intolerable physical interferences are present in a sample, a significant suppression of the internal standard signals (to less than 30 % of the signals in the calibrations standard) will be observed. Dilution of the sample fivefold (1+4) will usually eliminate the problem (see Sec. 9.3).

4.5 Memory interferences or carry-over can occur when there are large concentration differences between samples or standards which are analyzed sequentially. Sample deposition on the sampler and skimmer cones, spray chamber design, and the type of nebulizer affect the extent of the memory interferences which are observed. The rinse period between samples must be long enough to eliminate significant memory interference.

5.0 SAFETY

Refer to Chapter Three for a discussion on safety related references and issues.

6.0 EQUIPMENT AND SUPPLIES

6.1 Inductively coupled plasma-mass spectrometer:

6.1.1 A system capable of providing resolution, better than or equal to 1.0 amu at 10% peak height is required. The system must have a mass range from at least 6 to 240 amu and a data system that allows corrections for isobaric interferences and the application of the internal standard technique. Use of a mass-flow controller for the nebulizer argon and a peristaltic pump for the sample solution are recommended.

6.1.2 Argon gas supply: high-purity grade (99.99%).

7.0 REAGENTS AND STANDARDS

7.1 Acids used in the preparation of standards and for sample processing must be of high purity. Redistilled acids are recommended because of the high sensitivity of ICP-MS. Nitric acid at less than 2 per cent (v/v) is required for ICP-MS to minimize damage to the interface and to minimize isobaric molecular-ion interferences with the analytes. Many more molecular-ion interferences are observed when hydrochloric and sulfuric acids are used (References 3 and 4). Concentrations of antimony and silver between 50-500 µg/L require 1% (v/v) HCl for stability; for concentrations above 500 µg/L Ag, additional HCl will be needed. Consequently, accuracy of analytes requiring significant chloride molecular ion corrections (such as As and V) will degrade.

7.2 Reagent water: All references to water in the method refer to reagent water unless otherwise specified. Refer to Chapter One for a definition of reagent water.

7.3 Standard stock solutions for each analyte may be purchased or prepared from ultra-high purity grade chemicals or metals (99.99 or greater purity). See Method 6010 for instructions on preparing standard solutions from solids.

7.3.1 Bismuth internal standard stock solution (1 mL = 100 µg Bi): Dissolve 0.1115 g Bi_2O_3 in a minimum amount of dilute HNO_3. Add 10 mL conc. HNO_3 and dilute to 1,000 mL with reagent water.

7.3.2 Holmium internal standard stock solution (1 mL = 100 µg Ho): Dissolve 0.1757 g $Ho_2(CO_3)_2 \cdot 5H_2O$ in 10 mL reagent water and 10 mL HNO_3. After dissolution is complete, warm the solution to degas. Add 10 mL conc. HNO_3 and dilute to 1,000 mL with reagent water.

7.3.3 Indium internal standard stock solution (1 mL = 100 µg In): Dissolve 0.1000 g indium metal in 10 mL conc. HNO_3. Dilute to 1,000 mL with reagent water.

7.3.4 Lithium internal standard stock solution (1 mL = 100 µg ^6Li): Dissolve 0.6312 g 95-atom-% ^6Li, Li_2CO_3 in 10 mL of reagent water and 10 mL HNO_3. After dissolution is complete, warm the solution to degas. Add 10 mL conc. HNO_3 and dilute to 1,000 mL with reagent water.

7.3.5 Rhodium internal standard stock solution (1 mL = 100 µg Rh): Dissolve 0.3593 g ammonium hexachlororhodate (III) $(NH_4)_3RhCl_6$ in 10 mL reagent water. Add 100 mL conc. HCl and dilute to 1,000 mL with reagent water.

7.3.6 Scandium internal standard stock solution (1 mL = 100 µg Sc): Dissolve 0.15343 g Sc_2O_3 in 10 mL (1+1) hot HNO_3. Add 5 mL conc. HNO_3 and dilute to 1,000 mL with reagent water.

7.3.7 Terbium internal standard stock solution (1 mL = 100 µg Tb): Dissolve 0.1828 g $Tb_2(CO_3)_3{\cdot}5H_2O$ in 10 mL (1+1) HNO_3. After dissolution is complete, warm the solution to degas. Add 5 mL conc. HNO_3 and dilute to 1,000 mL with reagent water.

7.3.8 Yttrium internal standard stock solution (1 mL = 100 µg Y): Dissolve 0.2316 g $Y_2(CO_3)_{3}.3H_2O$ in 10 mL (1+1) HNO_3. Add 5 mL conc. HNO_3 and dilute to 1,000 mL with reagent water.

7.3.9 Titanium interference stock solution (1 mL = 100 µg Ti): Dissolve 0.4133 g $(NH_4)_2TiF_6$ in reagent water. Add 2 drops conc. HF and dilute to 1,000 mL with reagent water.

7.3.10 Molybdenum interference stock solution (1 mL = 100 µg Mo): Dissolve 0.2043 g $(NH_4)_2MoO_4$ in reagent water. Dilute to 1,000 mL with reagent water.

7.3.11 Gold preservative stock solution for mercury (1 mL = 100 µg): Recommend purchasing as high purity prepared solution of $AuCl_3$ in dilute hydrochloric acid matrix.

7.4 Mixed calibration standard solutions are prepared by diluting the stock-standard solutions to levels in the linear range for the instrument in a solvent consisting of 1 percent (v/v) HNO_3 in reagent water. The calibration standard solutions must contain a suitable concentration of an appropriate internal standard for each analyte. Internal standards may be added on-line at the time of analysis using a second channel of the peristaltic pump and an appropriate mixing manifold. Generally, an internal standard should be no more than 50 amu removed from the analyte. Recommended internal standards include ^{6}Li, ^{45}Sc, ^{89}Y, ^{103}Rh, ^{115}In, ^{159}Tb, ^{169}Ho, and ^{209}Bi. Prior to preparing the mixed standards, each stock solution must be analyzed separately to determine possible spectral interferences or the presence of impurities. Care must be taken when preparing the mixed standards that the elements are compatible and stable. Transfer the mixed standard solutions to freshly acid-cleaned FEP fluorocarbon bottles for storage. Fresh mixed standards must be prepared as needed with the realization that concentrations can change on aging. Calibration standards must be initially verified using a quality control standard (see Section 7.7).

7.5 Blanks: Three types of blanks are required for the analysis. The calibration blank is used in establishing the calibration curve. The preparation blank is used to monitor for possible contamination resulting from the sample preparation procedure. The rinse blank is used to flush the system between all samples and standards.

7.5.1 The calibration blank consists of the same concentration(s) of the same acid(s) used to prepare the final dilution of the calibrating solutions of the analytes [often 1 percent HNO_3 (v/v) in reagent water] along with the selected concentrations of internal standards such that there is an appropriate internal standard element for each of the analytes. Use of HCl for antimony and silver is cited in Section 7.1.

7.5.2 The preparation (or reagent) blank must be carried through the complete preparation procedure and contain the same volumes of reagents as the sample solutions.

7.5.3 The rinse blank consists of 1 to 2 percent HNO_3 (v/v) in reagent water. Prepare a sufficient quantity to flush the system between standards and samples. If mercury is to be analyzed, the rinse blank should also contain 2 µg/mL (ppm) $AuCl_3$ solution.

7.6 The interference check solution (ICS) is prepared to contain known concentrations of interfering elements that will demonstrate the magnitude of interferences and provide an adequate test of any corrections. Chloride in the ICS provides a means to evaluate software corrections for chloride-related interferences such as $^{35}Cl^{16}O^+$ on $^{51}V^+$ and $^{40}Ar^{35}Cl^+$ on $^{75}As^+$. Iron is used to demonstrate adequate resolution of the spectrometer for the determination of manganese. Molybdenum serves to indicate oxide effects on cadmium isotopes. The other components are present to evaluate the ability of the measurement system to correct for various molecular-ion isobaric interferences. The ICS is used to verify that the interference levels are corrected by the data system within quality control limits.

NOTE: The final ICS solution concentrations in Table 1 are intended to evaluate corrections for known interferences on only the analytes in Sec. 1.2. If Method 6020 is used to determine an element not listed in Sec. 1.2, it is the responsibility of the analyst to modify the ICS solutions, or prepare an alternative ICS solution, to allow adequate verification of correction of interferences on the unlisted element (see Section 9.4).

7.6.1 These solutions must be prepared from ultra-pure reagents. They can be obtained commercially or prepared by the following procedure.

7.6.1.1 Mixed ICS solution I may be prepared by adding 13.903 g $Al(NO_3)_3 \cdot 9H_2O$, 2.498 g $CaCO_3$ (dried at 180 °C for 1 hour before weighing), 1.000 g Fe, 1.658 g MgO, 2.305 g Na_2CO_3, and 1.767 g K_2CO_3 to 25 mL of reagent water. Slowly add 40 mL of (1+1) HNO_3. After dissolution is complete, warm the solution to degas. Cool and dilute to 1,000 mL with reagent water.

7.6.1.2 Mixed ICS solution II may be prepared by slowly adding 7.444 g 85 % H_3PO_4, 6.373 g 96% H_2SO_4, 40.024 g 37% HCl, and 10.664 g citric acid $C_6O_7H_8$ to 100 mL of reagent water. Dilute to 1,000 mL with reagent water.

7.6.1.3 Mixed ICS solution III may be prepared by adding 1.00 mL each of 100-µg/mL arsenic, cadmium, selenium, chromium, cobalt, copper, manganese, nickel, silver, vanadium, and zinc stock solutions to about 50 mL reagent water. Add 2.0 mL concentrated HNO_3, and dilute to 100.0 mL with reagent water.

7.6.1.4 Working ICS Solutions

7.6.1.4.1 ICS-A may be prepared by adding 10.0 mL of mixed ICS solution I (Sec. 7.6.1.1), 2.0 mL each of 100-µg/mL titanium stock solution (Sec. 7.3.9) and molybdenum stock solution (Sec. 7.3.10), and 5.0 mL of mixed ICS solution II (Sec. 7.6.1.2). Dilute to 100 mL with reagent water. ICS solution A must be prepared fresh weekly.

7.6.1.4.2 ICS-AB may be prepared by adding 10.0 mL of mixed ICS solution I (Sec. 7.6.1.1), 2.0 mL each of 100-µg/mL titanium stock solution (Sec. 7.3.9) and molybdenum stock solution (Sec. 7.3.10), 5.0 mL of mixed

ICS solution II (Sec. 7.6.1.2), and 2.0 mL of Mixed ICS solution III (Sec. 7.6.1.3). Dilute to 100 mL with reagent water. Although the ICS solution AB must be prepared fresh weekly, the analyst should be aware that the solution may precipitate silver more quickly.

7.7 The quality control standard is the initial calibration verification solution (ICV), which must be prepared in the same acid matrix as the calibration standards. This solution must be an independent standard near the midpoint of the linear range at a concentration other than that used for instrument calibration. An independent standard is defined as a standard composed of the analytes from a source different from those used in the standards for instrument calibration.

7.8 Mass spectrometer tuning solution. A solution containing elements representing all of the mass regions of interest (for example, 10 µg/L of Li, Co, In, and Tl) must be prepared to verify that the resolution and mass calibration of the instrument are within the required specifications (see Section 10.1). This solution is also used to verify that the instrument has reached thermal stability (see Section 11.4).

8.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1 Sample collection procedures should address the considerations described in Chapter Nine.

8.2 See the introductory material in Chapter Three, Inorganic Analytes, for information on sample handling, storage, holding times and preservation. Only polyethylene or fluorocarbon (TFE or PFA) containers are recommended for use in this method.

9.0 QUALITY CONTROL

9.1 All quality control data should be maintained and be available for easy reference or inspection.

9.2 Instrument detection limits (IDLs) in µg/L can be estimated by calculating the average of the standard deviations of three runs on three non-consecutive days from the analysis of a reagent blank solution with seven consecutive measurements per day. Each measurement must be performed as though it were a separate analytical sample (i.e., each measurement must be followed by a rinse and/or any other procedure normally performed between the analysis of separate samples). IDLs must be determined at least every three months and kept with the instrument log book. Refer to Chapter One for additional guidance.

9.3 The intensities of all internal standards must be monitored for every analysis. If the intensity of any internal standard in a sample falls below 30 percent of the intensity of that internal standard in the initial calibration standard, a significant matrix effect must be suspected. Under these conditions, the detection limit has degraded and the correction ability of the internal standardization technique becomes questionable. The following procedure is followed: First, make sure the instrument has not just drifted by observing the internal standard intensities in the nearest clean matrix (calibration blank, Section 7.5.1). If the low internal standard intensities are also seen in the nearest calibration blank, terminate the analysis, correct the problem, recalibrate, verify the new calibration, and reanalyze the affected samples. If drift has not occurred, matrix effects need to be removed by dilution of the affected sample. The sample must be diluted fivefold (1+4) and reanalyzed with the addition of appropriate amounts of internal standards. If the first dilution does not eliminate the problem, this procedure must be repeated until the internal-standard intensities rise above the 30 percent limit. Reported results must be corrected for all dilutions.

9.4 To obtain analyte data of known quality, it is necessary to measure more than the analytes of interest in order to apply corrections or to determine whether interference corrections are necessary. For example, tungsten oxide moleculars can be very difficult to distinguish from mercury isotopes. If the concentrations of interference sources (such as C, Cl, Mo, Zr, W) are such that, at the correction factor, the analyte is less than the limit of quantification and the concentration of interferents are insignificant, then the data may go uncorrected. Note that monitoring the interference sources does not necessarily require monitoring the interferant itself, but that a molecular species may be monitored to indicate the presence of the interferent. When correction equations are used, all QC criteria must also be met. Extensive QC for interference corrections are required at all times. The monitored masses must include those elements whose hydrogen, oxygen, hydroxyl, chlorine, nitrogen, carbon and sulfur molecular ions could impact the analytes of interest. Unsuspected interferences may be detected by adding pure major matrix components to a sample to observe any impact on the analyte signals. When an interference source is present, the sample elements impacted must be flagged to indicate (a) the percentage interference correction applied to the data or (b) an uncorrected interference by virtue of the elemental equation used for quantitation. The isotope proportions for an element or molecular-ion cluster provide information useful for quality assurance.

> NOTE: Only isobaric elemental, molecular, and doubly charged interference corrections which use the observed isotopic-response ratios or parent-to-oxide ratios (provided an oxide internal standard is used as described in Section 4.2) for each instrument system are acceptable corrections for use in Method 6020.

9.5 Dilution test (serial dilution): If the analyte concentration is within the linear dynamic range of the instrument and sufficiently high (minimally, a factor of at least 100 times greater than the concentration in the reagent blank, refer to Section 7.5.2), an analysis of a fivefold (1+4) dilution must agree within ± 10% of the original determination. If not, an interference effect must be suspected. One dilution test must be included for each twenty samples (or less) of each matrix in a batch.

9.6 Post-digestion spike addition: An analyte spike added to a portion of a prepared sample, or its dilution, should be recovered to within 75 to 125 percent of the known value or within the laboratory derived acceptance criteria. The spike addition should be based on the indigenous concentration of each element of interest in the sample. If the spike is not recovered within the specified limits, the sample must be diluted and reanalyzed to compensate for the matrix effect. Results must agree to within 10% of the original determination. The use of a standard-addition analysis procedure may also be used to compensate for this effect (refer to Method 7000).

9.7 A laboratory control sample (LCS) should be analyzed for each analyte using the same sample preparations, analytical methods and QA/QC procedures employed for the test samples. One LCS should be prepared and analyzed for each sample batch at a frequency of one LCS for each 20 samples or less.

9.8 Check the instrument calibration by analyzing appropriate quality control solutions as follows:

9.8.1 Check instrument calibration using a calibration blank (Section 7.5.1) and the initial calibration verification solution (Sections 7.7 and 11.6).

9.8.2 Verify calibration at a frequency of every 10 analytical samples with the instrument check standard (Section 7.6) and the calibration blank (Section 7.5.1). These solutions must also be analyzed for each analyte at the beginning of the analysis and after the last sample.

9.8.3 The results of the initial calibration verification solution and the instrument check standard must agree within ± 10% of the expected value. If not, terminate the analysis, correct the problem, and recalibrate the instrument. Any sample analyzed under an out-of-control calibration must be reanalyzed .

9.8.4 The results of the calibration blank must be less than 3 times the current IDL for each element. If this is not the case, the reason for the out-of-control condition must be found and corrected, and affected samples must be reanalyzed. If the laboratory consistently has concentrations greater than 3 times the IDL, the IDL may be indicative of an estimated IDL and should be re-evaluated.

9.9 Verify the magnitude of elemental and molecular-ion isobaric interferences and the adequacy of any corrections at the beginning of an analytical run or once every 12 hours, whichever is more frequent. Do this by analyzing the interference check solutions A and AB. The analyst should be aware that precipitation from solution AB may occur with some elements, specifically silver. Refer to Section 4.0 for a discussion on interferences and potential solutions to those interferences if additional guidance is needed.

9.10 Analyze one duplicate sample for every matrix in a batch at a frequency of one matrix duplicate for every 20 samples.

9.10.1 The relative percent difference (RPD) between duplicate determinations must be calculated as follows:

$$\text{RPD} = \frac{|D_1 - D_2|}{(D_1 + D_2)/2} \times 100$$

where:

RPD = relative percent difference.
D_1 = first sample value.
D_2 = second sample value (duplicate)

A control limit of 20% RPD should not be exceeded for analyte values greater than 100 times the instrumental detection limit. If this limit is exceeded, the reason for the out-of-control situation must be found and corrected, and any samples analyzed during the out-of-control condition must be reanalyzed.

9.11 Ultra-trace analysis requires the use of clean chemistry. Several suggestions for reduction on the analytical blank are provided in Chapter Three.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Conduct mass calibration and resolution checks in the mass regions of interest. The mass calibration and resolution parameters are required criteria which must be met prior to any samples being analyzed. If the mass calibration differs more than 0.1 amu from the true value, then the mass calibration must be adjusted to the correct value. The resolution must also be verified to be less than 0.9 amu full width at 10 percent peak height.

10.2 Calibrate the instrument for the analytes of interest (recommended isotopes for the analytes in Sec. 1.2 are provided in Table 2), using the calibration blank and at least a single initial calibration standard according to the instrument manufacturer's procedure. Flush the system with the rinse blank (Sec. 7.5.3) between each standard solution. Use the average of at least three integrations for both calibration and sample analyses.

> NOTE: Analysts have noted improved performance in calibration stability if the instrument is exposed to the interference check solution after cleaning sampler and skimmer cones. Improved performance is also realized if the instrument is allowed to rinse for 5 or 10 minutes before the calibration blank is run.

10.3 All masses which could affect data quality should be monitored to determine potential effects from matrix components on the analyte peaks. The recommended isotopes to be monitored are listed in Table 2.

10.4 Immediately after the calibration has been established, the calibration must be verified and documented for every analyte by the analysis of the calibration verification solution (Section 7.7). When measurements exceed ± 10% of the accepted value, the analyses must be terminated, the problem corrected, the instrument recalibrated, and the new calibration verified. Any samples analyzed under an out-of-control calibration must be reanalyzed. During the course of an analytical run, the instrument may be "resloped" or recalibrated to correct for instrument drift but resloping must not be used as an alternative to reanalyzing samples following an unacceptable QC sample, such as a CCV. A recalibration must then be followed immediately by a new analysis of a CCV and CCB before any further samples may be analyzed.

11.0 PROCEDURE

11.1 Solubilization and digestion procedures are presented in Chapter Three (e.g., Methods 3005 - 3052).

> NOTE: If mercury is to be analyzed, the digestion procedure must use mixed nitric and hydrochloric acids through all steps of the digestion. Mercury will be lost if the sample is digested when hydrochloric acid is not present. If it has not already been added to the sample as a preservative, Au should be added to give a final concentration of 2 mg/L (use 2.0 mL of 5.3.11 per 100 mL of sample) to preserve the mercury and to prevent it from plating out in the sample introduction system.

11.2 Initiate appropriate operating configuration of the instruments computer according to the instrument manufacturer's instructions.

11.3 Set up the instrument with the proper operating parameters according to the instrument manufacturer's instructions.

11.4 Operating conditions: The analyst should follow the instructions provided by the instrument manufacturer. Allow at least 30 minutes for the instrument to equilibrate before analyzing any samples. This must be verified by analyzing a tuning solution (Section 7.8) at least four times with relative standard deviations of $\leq$ 5% for the analytes contained in the tuning solution.

> NOTE: The instrument should have features that protect itself from high ion currents. If not, precautions must be taken to protect the detector from high ion currents. A channel electron multiplier or active film multiplier suffer from fatigue after being exposed to high ion currents. This fatigue can last from several seconds to hours depending on the extent of exposure. During this time period, response factors are constantly changing, which invalidates the calibration curve, causes instability, and invalidates sample analyses.

11.5 Calibrate the instrument following the procedure outlined in Section 10.0.

11.6 Flush the system with the rinse blank solution (Sec. 7.5.3) until the signal levels return to the DQO or method's levels of quantitation (usually about 30 seconds) before the analysis of each sample (see Section 10.3). Nebulize each sample until a steady-state signal is achieved (usually about 30 seconds) prior to collecting data. Analyze the calibration verification solution (Section 7.6) and the calibration blank (Section 7.5.1) at a frequency of at least once every 10 analytical samples. Flow-injection systems may be used as long as they can meet the performance criteria of this method.

11.7 Dilute and reanalyze samples that are more concentrated than the linear range for an analyte (or species needed for a correction) or measure an alternate but less-abundant isotope. The linearity at the alternate mass must be confirmed by appropriate calibration (see Sec. 10.2 and 10.4). Alternatively apply solid phase chelation chromatography to eliminate the matrix as described in Sec. 4.3.

12.0 DATA ANALYSIS AND CALCULATIONS

12.1 The quantitative values shall be reported in appropriate units, such as micrograms per liter (µg/L) for aqueous samples and milligrams per kilogram (mg/kg) for solid samples. If dilutions were performed, the appropriate corrections must be applied to the sample values.

12.1.1 If appropriate, or required, calculate results for solids on a dry-weight basis as follows:

(1) A separate determination of percent solids must be performed.

(2) The concentrations determined in the digest are to be reported on the basis of the dry weight of the sample.

$$\text{Concentration (dry weight)(mg/kg)} = \frac{C \times V}{W \times S}$$

Where,

C = Digest Concentration (mg/L)
V = Final volume in liters after sample preparation
W = Weight in kg of wet sample

$$S = \frac{\%\ \text{Solids}}{100}$$

Calculations must include appropriate interference corrections (see Section 4.2 for examples), internal-standard normalization, and the summation of signals at 206, 207, and 208 m/z for lead (to compensate for any differences in the abundances of these isotopes between samples and standards).

13.0 METHOD PERFORMANCE

13.1 In an EPA multi-laboratory study (Ref. 12), twelve laboratories applied the ICP-MS technique to both aqueous and solid samples. Table 3 summarizes the method performance data for aqueous samples. Performance data for solid samples are provided in Table 4.

13.2 Table 5 summarizes the method performance data for aqueous and sea water samples with interfering elements removed and samples preconcentrated prior to analysis. Table 6 summarizes the performance data for a simulated drinking water standard.

14.0 POLLUTION PREVENTION

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity and/or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best option.

14.2 For information about pollution prevention that may be applicable to laboratories and research institutions consult *Less is Better: Laboratory Chemical management for Waste Reduction* available from the American Chemical Society's Department of Government Relations and Science Policy, 1155 16th St., N.W. Washington, D.C. 20036, (202) 872-4477.

15.0 WASTE MANAGEMENT

The Environmental Protection Agency requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. The Agency urges laboratories to protect the air, water, and land by minimizing and controlling all releases from hoods and bench operations, complying with the letter and spirit of any sewer discharge permits and regulations, and by complying with all solid and hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions. For further information on waste management, consult *The Waste Management Manual for Laboratory Personnel* available from the American Chemical Society at the address listed in Sec. 14.2.

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17.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

The pages to follow contain Tables 1 through 7, and a flow diagram of the method procedure.

TABLE 1

RECOMMENDED INTERFERENCE CHECK SAMPLE COMPONENTS AND CONCENTRATIONS

Solution Component	Solution A Concentration (mg/L)	Solution AB Concentration (mg/L)
Al	100.0	100.0
Ca	300.0	300.0
Fe	250.0	250.0
Mg	100.0	100.0
Na	250.0	250.0
P	100.0	100.0
K	100.0	100.0
S	100.0	100.0
C	200.0	200.0
Cl	2000.0	2000.0
Mo	2.0	2.0
Ti	2.0	2.0
As	0.0	0.100
Cd	0.0	0.100
Cr	0.0	0.200
Co	0.0	0.200
Cu	0.0	0.200
Mn	0.0	0.200
Hg	0.0	0.020
Ni	0.0	0.200
Se	0.0	0.100
Ag	0.0	0.050
V	0.0	0.200
Zn	0.0	0.100

TABLE 2
RECOMMENDED ISOTOPES FOR SELECTED ELEMENTS

Element of interest	Mass
Aluminum	**27**
Antimony	121, **123**
Arsenic	**75**
Barium	138, 137, 136, **135**, 134
Beryllium	**9**
Bismuth (IS)	209
Cadmium	**114**, 112, **111**, 110, 113, 116, 106
Calcium (I)	42, 43, **44**, 46, 48
Chlorine (I)	35, 37, (77, 82)[a]
Chromium	**52**, **53**, **50**, 54
Cobalt	**59**
Copper	**63**, **65**
Holmium (IS)	165
Indium (IS)	**115**, 113
Iron (I)	**56**, **54**, **57**, 58
Lanthanum (I)	139
Lead	**208**, **207**, **206**, 204
Lithium (IS)	6[b], 7
Magnesium (I)	24, **25**, **26**
Manganese	**55**
Mercury	202, **200**, 199, 201
Molybdenum (I)	98, 96, 92, **97**, 94, (108)[a]
Nickel	58, **60**, 62, **61**, 64
Potassium (I)	**39**
Rhodium (IS)	103
Scandium (IS)	45
Selenium	80, **78**, **82**, **76**, **77**, 74
Silver	**107**, **109**
Sodium (I)	**23**
Terbium (IS)	159
Thallium	**205**, 203
Vanadium	**51**, **50**
Tin (I)	120, **118**
Yttrium (IS)	89
Zinc	64, **66**, **68**, **67**, 70

NOTE: Method 6020 is recommended for only those analytes listed in Sec.1.2. Other elements are included in this table because they are potential interferents (labeled I) in the determination of recommended analytes, or because they are commonly used internal standards (labeled IS). Isotopes are listed in descending order of natural abundance. The most generally useful isotopes are underlined and in boldface, although certain matrices may require the use of alternative isotopes.

[a] These masses are also useful for interference correction (Section 4.2).

[b] Internal standard must be enriched in the ^{6}Li isotope. This minimizes interference from indigenous lithium.

TABLE 3

ICP-MS MULTI-LABORATORY PRECISION AND ACCURACY DATA FOR AQUEOUS SOLUTIONS

Element	Comparability[a] Range	%RSD Range	N[b]	S[c]
Aluminum	95 - 100	11 - 14	14 - 14	4
Antimony	d	5.0 - 7.6	16 - 16	3
Arsenic	97 - 114	7.1 - 48	12 - 14	4
Barium	91 - 99	4.3 - 9.0	16 - 16	5
Beryllium	103 - 107	8.6 - 14	13 - 14	3
Cadmium	98 - 102	4.6 - 7.2	18 - 20	3
Calcium	99 - 107	5.7 - 23	17 - 18	5
Chromium	95 - 105	13 - 27	16 - 18	4
Cobalt	101 - 104	8.2 - 8.5	18 - 18	3
Copper	85 - 101	6.1 - 27	17 - 18	5
Iron	91 - 900	11 - 150*	10 - 12	5
Lead	71 - 137	11 - 23	17 - 18	6
Magnesium	98 - 102	10 - 15	16 - 16	5
Manganese	95 - 101	8.8 - 15	18 - 18	4
Nickel	98 - 101	6.1 - 6.7	18 - 18	2
Potassium	101 - 114	9.9 - 19	11 - 12	5
Selenium	102 - 107	15 - 25	12 - 12	3
Silver	104 - 105	5.2 - 7.7	13 - 16	2
Sodium	82 - 104	24 - 43	9 - 10	5
Thallium	88 - 97	9.7 - 12	18 - 18	3
Vanadium	107 - 142	23 - 68	8 - 13	3
Zinc	93 - 102	6.8 - 17	16 - 18	5

Data obtained from reference 12.

[a] Comparability refers to the percent agreement of mean ICP-MS values to those of the reference technique (ICP-AES or GFAA).
[b] N is the range of the number of ICP-MS measurements where the analyte values exceed the limit of quantitation (3.3 times the average IDL value). A larger number gives a more reliable comparison. [c] S is the number of samples with results greater than the limit of quantitation.
[d] No comparability values are provided for antimony because of evidence that the reference data is affected by an interference.

TABLE 4

ICP-MS MULTI-LABORATORY PRECISION AND ACCURACY DATA FOR SOLID MATRICES

Element	Comparability[a] Range	%RSD Range	N[b]	S[c]
Aluminum	83 - 101	11 - 39	13 - 14	7
Antimony	d	12 - 21	15 - 16	2
Arsenic	79 - 102	12 - 23	16 - 16	7
Barium	100 - 102	4.3 - 17	15 - 16	7
Beryllium	50 - 87	19 - 34	12 - 14	5
Cadmium	93 - 100	6.2 - 25	19 - 20	5
Calcium	95 - 109	4.1 - 27	15 - 17	7
Chromium	77 - 98	11 - 32	17 - 18	7
Cobalt	43 - 102	15 - 30	17 - 18	6
Copper	90 - 109	9.0 - 25	18 - 18	7
Iron	87 - 99	6.7 - 21	12 - 12	7
Lead	90 - 104	5.9 - 28	15 - 18	7
Magnesium	89 - 111	7.6 - 37	15 - 16	7
Manganese	80 - 108	11 - 40	16 - 18	7
Nickel	87 - 117	9.2 - 29	16 - 18	7
Potassium	97 - 137	11 - 62	10 - 12	5
Selenium	81	39	12	1
Silver	43 - 112	12 - 33	15 - 15	3
Sodium	100 - 146	14 - 77	8 - 10	5
Thallium	91	33	18	1
Vanadium	83 - 147	20 - 70	6 - 14	7
Zinc	84 - 124	14 - 42	18 - 18	7

Data obtained from reference 12.

[a] Comparability refers to the percent agreement of mean ICP-MS values to those of the reference technique.

[b] N is the range of the number of ICP-MS measurements where the analyte values exceed the limit of quantitation (3.3 times the average IDL value).

[c] S is the number of samples with results greater than the limit of quantitation.

[d] No comparability values are provided for antimony because of evidence that the reference data is affected by an interference.

TABLE 5

METHOD PERFORMANCE DATA FOR AQUEOUS AND SEA WATER SAMPLES[A] WITH INTERFERING ELEMENTS REMOVED AND SAMPLES PRECONCENTRATED PRIOR TO ANALYSIS

ELEMENT	ISOTOPE	CONCENTRATION (ng/mL)[B]		CERTFIED
		9.0 mL	27.0 mL	
Manganese	55	1.8±0.05	1.9±0.2	1.99±0.15
Nickel	58	0.32±0.018	0.32±0.04	0.30±0.04
Cobalt	59	0.033±0.002	0.028±0.003	0.025±0.006
Copper	63	0.68±0.03	0.63±0.03	0.68±0.04
Zinc	64	1.6±0.05	1.8±0.15	1.97±0.12
Copper	65	0.67±0.03	0.6±0.05	0.68±0.04
Zinc	66	1.6±0.06	1.8±0.2	1.97±0.12
Cadmium	112	0.020±0.0015	0.019±0.0018	0.019±0.004
Cadmium	114	0.020±0.0009	0.019±0.002	0.019±0.004
Lead	206	0.013±0.0009	0.019±0.0011	0.019±0.006
Lead	207	0.014±0.0005	0.019±0.004	0.019±0.006
Lead	208	0.014±0.0006	0.019±0.002	0.019±0.006

Data obtained from reference 12.

[A] The dilution of the sea-water during the adjustment of pH produced 10 mL samples containing 9 mL of sea-water and 30 mL samples containing 27 mL of sea-water. Samples containing 9.0 mL of CASS-2, n=5; samples containing 27.0 mL of CASS-2, n=3.

[B] Concentration (ng/mL) ± 95% confidence limits.

TABLE 6

ANALYSIS OF NIST SRM 1643b, TRACE METALS IN WATER[A]

ELEMENT	ISOTOPE	CONCENTRATION (ng/mL)[B] DETERMINED	CERTFIED
Manganese	55	30±1.3	28±2
Nickel	58	50±2	49±3
Cobalt	59	27±1.3	26±1
Nickel	60	51±2	49±3
Copper	63	23±1.0	21.9±0.4
Zinc	64	67±1.4	66±2
Copper	65	22±0.9	21.9±0.4
Zinc	66	67±1.8	66±2
Cadmium	111	20±0.5	20±1
Cadmium	112	19.9±0.3	20±1
Cadmium	114	19.8±0.4	20±1
Lead	206	23±0.5	23.7±0.7
Lead	207	23.9±0.4	23.7±0.7
Lead	208	24.2±0.4	23.7±0.7

Data obtained from reference 12.

[A] 5.0 mL samples, n=5.
[B] Concentration (ng/mL) ± 95% confidence limits.

TABLE 7

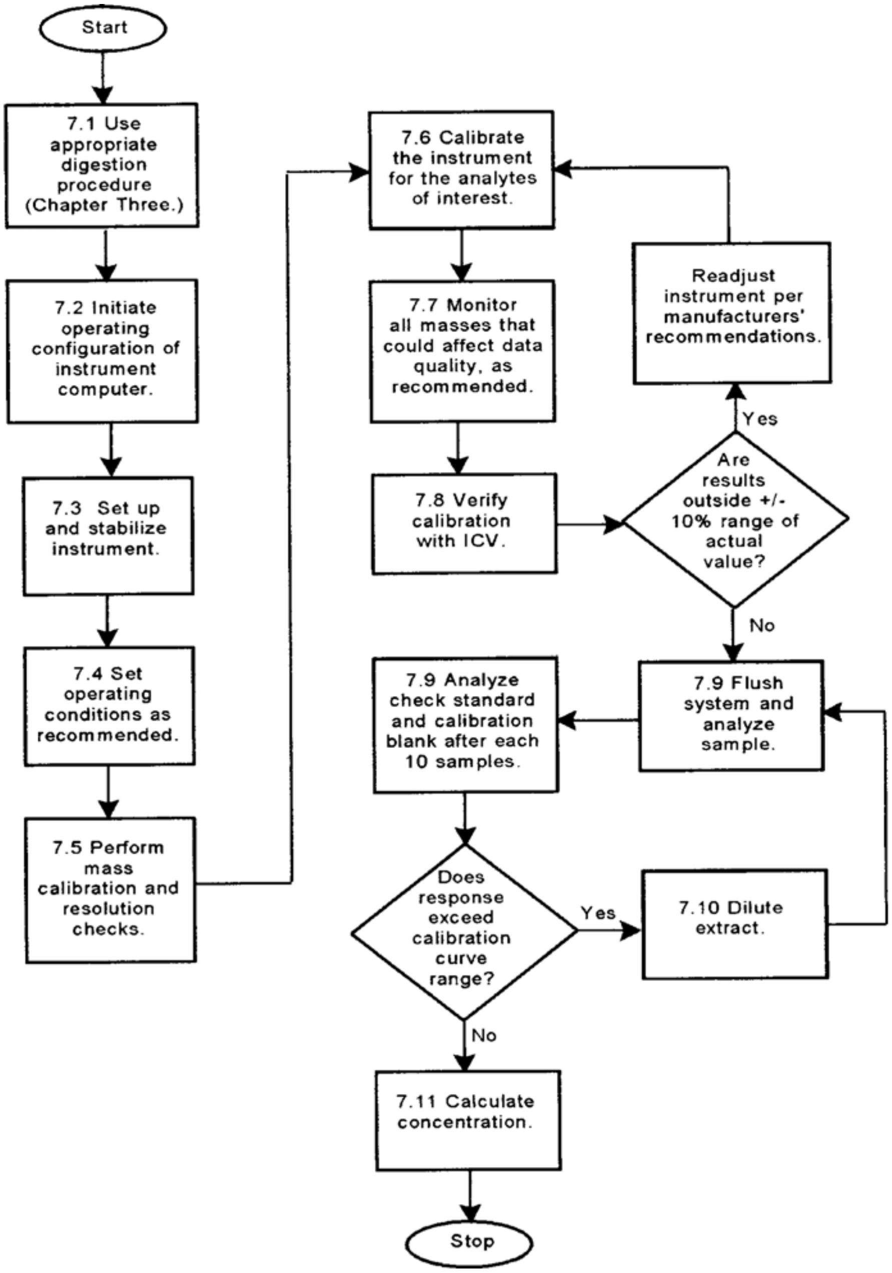
COMPARISON OF TOTAL MERCURY RESULTS IN HEAVILY CONTAMINATED SOILS

Soil Sample	Mercury in µg/g	
	ICP-MS	CVAA
1	27.8	29.2
2	442	376
3	64.7	58.2
4	339	589
5	281	454
6	23.8	21.4
7	217	183
8	157	129
9	1670	1360
10	73.5	64.8
11	2090	1830
12	96.4	85.8
13	1080	1190
14	294	258
15	3300	2850
16	301	281
17	2130	2020
18	247	226
19	2630	2080

Source: Reference 16.

METHOD 6020A

INDUCTIVELY COUPLED PLASMA - MASS SPECTROMETRY



CLARK FORK RIVER

SUPERFUND SITE INVESTIGATIONS

LABORATORY ANALYTICAL PROCEDURE
for
X-RAY FLUORESCENCE ANALYSIS
of
SOLID MEDIA:
I. LABORATORY-GRADE
INSTRUMENTATION METHOD

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October 1995

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TABLE OF CONTENTS

TABLES

ACRONYMS AND ABBREVIATIONS

C	Celsius
CLP	contract laboratory program
g	gram
IDL	instrument detection limit
LCS	laboratory control sample
LAB DUP	laboratory duplicate
LAB REP	laboratory replicate
LAP	laboratory analytical procedure
LCS	laboratory control sample
LOQ	Limit of quantification
MDL	method detection limit
mL	milliliter
NIST	National Institute of Science and Technology
NRCC	National Research Council of Canada
QA/QC	quality assurance/quality control
QAPP	quality assurance project plan
ROI	regions of interest
RPD	relative percent difference $= \frac{\lvert \text{Sample 1 - Sample 2} \rvert}{\text{Sample 1 + Sample 2)/2}} \times 100$
SAP	sampling and analysis plan
SB	sand blank
SRM	standard reference material
XCS	cross-contamination blank
XRF	x-ray fluorescence
%R	% recovery $= \frac{\text{analyzed value}}{\text{reference value}} \times 100$

LABORATORY ANALYTICAL PROCEDURE FOR X-RAY FLUORESCENCE ANALYSIS OF CLARK FORK BASIN SOLID MEDIA

1.0 SCOPE AND APPLICATION

1.1 This Laboratory Analytical Procedure (LAP) covers the use of energy-dispersive x-ray fluorescence (XRF) spectroscopy for the determination of trace and minor constituent elements in solid matrix samples. The method presented here will produce data of unrestricted use quality ("enforcement quality") with minimal sample preparation and rapid analysis turnaround.

1.2 The method has been demonstrated for arsenic, cadmium, copper, lead, and zinc (ARCO 1991b,c). With appropriate protocol modifications, the general method can be extended to elements above sodium on the periodic chart and to matrices other than granular solids. The instrument detection limits (IDLs) are defined as 3 times the average standard deviation of a set of measurements on a silica matrix lightly spiked with the analyte being measured, but with no interfering analytes. The following IDLs are representative of the IDLs expected by this method. The indicated range is the range of concentration (IDL to maximum) over which the technique is linear. Method detection limits (MDLs) and limits of quantification (LOQs) are matrix-dependent and must be determined on a project-specific basis. The MDL and LOQ are defined as 3 and 10 times, respectively, the standard deviation of replicate measurements on low concentration samples. They are normally determined with appropriate standard reference materials (SRMs).

TABLE 1-1. INSTRUMENT DETECTION LIMITS AND LINEAR RANGES

Analyte	Emission Series Measured	IDL (mg/kg)	Linear Range (mg/kg)
Arsenic	K	4	4–50,000
Cadmium	K	1.5	1.5–50,000
Calcium	K	40	40–150,000
Copper	K	9	9–50,000
Iron	K	20	20–150,000
Lead	L	7	7–50,000
Manganese	K	12	12–50,000
Zinc	K	5	5–50,000

1.3 Detection limits will vary with measurement times, choice of x-ray spectroscopic equipment and x-ray parameters, and sample matrix. This method was developed specifically for Spectrace® laboratory-grade instruments.

1.4 Use of this method is restricted to analyses supervised by x-ray spectroscopists or chemists knowledgeable in correction techniques for spectral, chemical, and physical interferences.

2.0 SUMMARY OF METHOD

2.1 The basis of XRF spectrometry is the detection and measurement of x-rays emitted from the atoms of an irradiated sample. A beam of x-rays is directed into a sample, exciting some of the atoms in the sample to energy levels above their ground state. In this unstable atomic state, a vacancy exists in one of the lower energy level electron orbitals. Electrons from higher energy states fill these vacancies in the lower energy levels, releasing energy in the process. Some of the energy is emitted as x-rays with energies characteristic of their element of origin. The intensity of the fluorescent radiation depends on several factors, but is related to the concentration of the element in the sample. A solid-state lithium drifted silicon detector measures the spectral distribution of x-rays. The measured spectrum is then processed with the aid of an instrument calibration to determine elemental concentrations of the analytes.

2.2 Prior to analysis, samples are conditioned by oven drying, sieving, and grinding to a uniform fine-grained material with approximately 95 percent (weight) less than 74 microns in size (200 mesh).

3.0 INTERFERENCES

3.1 Matrix interference effects include absorption of the incident exciting radiation and the emitted fluorescent radiation and the excitation of the sample by the absorbed fluorescent x-rays. These effects can result in increasing or decreasing the apparent concentration of an analyte. Spectral interferences result from the overlap of fluorescent lines from different elements of the sample which may interfere with, and artificially increase, apparent analyte intensities. Other interferences are caused by the detection of scattered primary radiation and by the escape of energy from the detector following the absorption of a characteristic x-ray. Refer to Bertin (1975) for a complete discussion of interferences.

The Spectrace 5000® uses a two-step process to correct for interferences. The detected spectrum is corrected for the effects of scattered radiation and for spectral line overlaps (using a library of pure element line shapes) through a multiparameter least-squares regression, and then corrected for absorption and enhancement interferences through a "Fundamental Parameters" technique. This is an iterative computational method using the measured x-ray intensities and three Fundamental Parameters (primary spectral distribution, absorption coefficients, and fluorescent yields) to calculate interelement influences and analyte compositions. In theory, no standards are required, but in practice some standards are used to account for unknown or poorly-known parameters.

4.0 APPARATUS AND MATERIALS

4.1 Use the Spectrace Instruments Model TX-5000® energy dispersive XRF spectrometer with Si(Li) x-ray detector; 50 kV, 17 watt Rh target x-ray tube; provision for x-ray beam filtering; and Fundamental Parameters computational software installed in an IBM-compatible computer of the AT or higher class. A minimum configuration includes a 286 microprocessor with 640 kilobytes of available memory, a hard disc, and a compatible math coprocessor.

4.2 For grinding, use a rotary swing mill sample grinder, such as the SPEX INDUSTRIES Model 8510® or equivalent, and hardened steel grinding containers. NOTE: Hardened steel grinding vessels will lose iron and chromium to the sample during grinding. If this contamination will interfere with analysis, choose another grinding vessel.

4.3 Use a drying oven and desiccator for holding dried samples during cool-down.

4.4 Sieve samples with U.S. Standard No. 10 and stainless steel mesh sieves with pans.

4.5 Contain samples in SPEX Model 3529® or equivalent x-ray sample cups, and x-ray grade polypropylene foil, such as SPEX Catalog No. 3520® or equivalent.

4.6 Use silica sand with concentrations less than the LOQ of analytes of concern.

4.7 Use disposable wipes, Kimwipes®, or equivalent for wiping grinding vessels and other equipment.

5.0 REAGENTS

5.1 Reagents should be pure or low atomic number compound forms of all elements with atomic number greater than 15 likely to be present in any samples to be analyzed as a major or minor constituent or as an analyte. Those required in the laboratory for instrument calibration include, as a minimum, potassium, calcium, titanium, chromium, manganese, iron, cobalt, nickel, copper, zinc, arsenic, selenium, bromine, rubidium, strontium, zirconium, molybdenum, silver, cadmium, tin, antimony, barium, mercury, and lead.

5.2 A suite of three to five project-specific reference standards will be used in calibration to generate "alpha coefficients," which are used in the Fundamental Parameters technique to correct for matrix effects. Reference standards should be chosen to be representative of sample matrices of a project area, and such that, for each analyte, at least one of the reference standards contains elemental concentrations two to four orders of magnitude above the IDL. Where practical, reference standards will be selected from Standard Reference Materials certified by National Institute of Standards and Technology or a similar standardizing organization. Sample preparation methods for reference standards will be identical to those for field samples.

5.3 Laboratory control samples (LCSs) will be chosen from the suite of project-specific reference standards described above. An LCS should generally contain elemental concentrations in the range of 100 to 10,000 mg/kg of each analyte of interest. Precision of the LCS measurement improves with higher analyte concentrations, so samples with higher elemental concentration of the analytes should be given preferential consideration in choosing the LCS. Alternately, LCSs with analyte concentrations near the expected level of the natural samples for the project can be used to gain additional precision information at project-specific levels. If analyte concentrations are low, laboratory control data will be less effective.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 All sample handling is carried out under strict chain-of-custody procedures in accordance with Clark Fork River Superfund Site Investigations quality assurance project plan (QAPP) and LAP (ARCO 1991d,e).

6.2 The analysis described in this procedure begins with an approximately 500 g sample delivered from the field. Field sample collection is specified in the *Clark Fork River Superfund Site Investigation Standard Operating Procedures* (ARCO 1991f) and the project sampling and analysis plan (SAP).

6.3 Holding times for solid matrix inorganic analysis (except for mercury and cyanide) of Clark Fork Basin samples are not restricted, nor are there special preservation or temperature requirements. Samples will normally be analyzed within 2 weeks of receipt at the laboratory.

7.0 PROCEDURE

7.1 Sample Preparation

7.1.1 Drying and Sieving: Project-specific SAPs or work plans should be referred to for sampling protocol. The following procedure is typical. Remove foreign objects such as rocks, twigs, etc., from the field sample. Homogenize the sample and place approximately 40 cm^3 of sample in a glass or aluminum vessel and hold at 105 ±5 °C for 4 hours. Cool to room temperature in a desiccator. Disaggregate the sample, using a mortar if necessary, and sieve through a U.S. Standard No. 10 sieve. Retain the −10 mesh fraction either in a desiccator or in an airtight container. Decontaminate sieves and reusable drying dishes.

7.1.2 Grinding: Place a 12- to 22-g representative subsample of homogenized 10-mesh material into a decontaminated grinding vessel and grind for a time period which achieves comminution of approximately 95 percent passing 200-mesh. Ninety seconds is sufficient for most soils. Remove the ground sample and store in a desiccator or airtight container. Wipe the grinding vessel clean with a disposable wipe. Place approximately 15 g of clean sand in the grinding vessel, grind for a similar time period, and discard the sand. Wipe the vessel clean with a disposable wipe and repeat the procedure for the next sample.

7.1.3 Filling the Sample Cup: Fill a sample cup with 6–8 cm^3 of the homogenized ground material (approximately 8–10 g) and cover the window with polypropylene foil. The window should be visibly wrinkle-free. Tap the cup with the window side down to eliminate bridging of the material. The sample is now ready for analysis. Note that if samples have been stored for over 12 months, they should be redried before analysis.

7.2 Instrument Calibration

7.2.1 Calibration Instructions: Detailed calibration instructions can be found in the operator's manual for the Spectrace 5000® spectrometer. This section summarizes the major steps in calibrating the instrument using the Fundamental Parameters technique.

7.2.2 Reference Spectra: Reference spectra are used in the first analysis step to correct for spectral interferences. Measure all reference spectra, using the pure element form if available, or compounds with no component within Z = 3 of the target element. Reference spectra should be measured at the x-ray voltage and filter setting to be used during sample analysis. Accumulate at least 30,000 counts in the peak counting channel.

Set regions of interest (ROI) boundaries about all spectral lines to be used to quantitate the analytes of interest and on all lines that will influence the quantitation of the analytes. See the discussion on interferences (Section 3.0) for guidance. A maximum of 20 ROIs can be set for any analysis protocol. If additional ROIs are required for adequate analysis, multiple analysis protocols must be established.

7.2.3 Calibration Standards: Project-specific reference standards are used to derive alpha coefficients for the correction of interelement matrix effects. Enter referee analyses for each standard. One hundred percent of each standard's composition must be specified. For analytes above calcium on the periodic chart, low atomic number components of soil samples can be assumed to be silicon and oxygen without compromising analytical accuracy.

Measure each standard using the x-ray protocol (tube current, tube voltage, beam filter) to be used during analysis. Specify calibration technique to be Fundamental Parameters. Upon completion of measurement of all standards, the instrument will calculate interelement influence coefficients and store in an "alpha-coefficients" file.

7.3 Measurement of Unknowns

7.3.1 Maintain all excitation parameters the same as used during the calibration. Set measurement time such that the desired analytical precision can be met. Place the sample cups on the instrument's carousel and initiate automatic counting sequence. Instrument will proceed through the sample measurement and will calculate concentration levels for each analyte. Data are filed by sample number in the computer memory. Elemental concentration levels will be archived for a period of 12 months after submittal of the formal data report as the primary analysis data. These data may be augmented with intermediate files and raw pulse height spectra for each sample.

8.0 LABORATORY QUALITY ASSURANCE/QUALITY CONTROL

8.1 Documentation: All quality assurance and quality control (QA/QC) data will be maintained and available for easy reference and inspection in accordance with the Clark Fork River Superfund Site Investigations QAPP and LAP (ARCO 1991d,e). Laboratory QA/QC requirements are summarized in Table 8-1.

TABLE 8-1. SUMMARY OF LABORATORY QUALITY ASSURANCE/QUALITY CONTROL CHECKS

Action	Frequency[a]	Control Limit	Corrective Action[b]
Energy calibration	Daily	Set by instrument	Service instrument
Initial calibration verification (LCS)	Daily	Recoveries per Table 8-2	1. Repeat LCS. 2. Repeat energy calibration, LCS. 3. Service instrument.
Calibration verification/ continuing calibration (LCS)	1 per 20 and last sample of the day	Recoveries per Table 8-2	1. Re-run LCS and affected samples. 2. Energy calibrate; re-run LCS, affected samples. 3. Recalibrate.
Laboratory duplicate	1 per 20	RPDs per Table 8.3	1. Re-run sample pair. 2. Prepare second pair of samples; repeat.
Laboratory replicate	1 per 20	RPDs per Table 8.4	1. Re-run sample pair. 2. Prepare second pair of samples; repeat.
Cross-contamination check sample (XCS)	1 per 50	<MDL	If consistently out-of-control, modify sample prep.
Sand blank	1 per 50	<MDL	If consistently out-of-control, modify sample prep.
	Each new sand batch	<MDL; all analytes	Reject sand

[a] Daily means each day that samples are analyzed under this LAP.

[b] Corrective actions are sequential for cases indicating multiple corrective actions. If the first corrective action is not sufficient to bring analysis back into control, the second action, if noted, will be implemented. If the second corrective action also fails, the third action, if noted, will be implemented.

TABLE 8.2 (REVISED) PERMISSIBLE RECOVERIES FOR NIST LCS AND ICV SAMPLES: QA/QC ACCEPTANCE WINDOWS

ANALYTE	SAMPLE I.D.	Min Recovery	Max Recovery	Recovery Range
Arsenic	NIST 2710a	1232	1848	80% - 120%
Cadmium	NIST 2710a	9.2	15.4	75% - 125%
Copper	NIST 2710a	3146	3694	92% - 108%
Lead	NIST 2710a	5189	5851	94% - 106%
Zinc	NIST 2710a	3929	4431	94% - 106%

TABLE 8-4. MAXIMUM ACCEPTABLE LAB REP RPDs FOR VARIOUS LEVELS OF CONTAMINATION

Concentration	RPD Control Limit for Laboratory Replicate
<2 × MDL	No Limits
2 × MDL to 2 × LOQ	35%
>2 × LOQ	20%

Note 1: If the two samples fall in different concentration windows, the wider control limit applies.

8.2 Energy Calibration/Gain Check: A calibration/gain check is to be performed at the beginning of each day that samples are run. In the gain check mode, the Spectrace 5000® automatically adjusts gain to within acceptable limits by monitoring the shape and location of the Cu K alpha peak, or reports the inability to gain adjust. In case of failure, analysis is interrupted until gain control is established. Sample analysis is repeated from the last in-control LCS measurement.

8.3 Initial Calibration Verification: Calibration verification is to be performed after the energy calibration/gain check is completed at the beginning of each day that samples are run by measuring one of the LCSs. Table 8-2 gives acceptable recovery ranges for selected analytes. Analysis cannot proceed until the initial calibration meets recovery requirements. See Table 8.1 for corrective actions.

8.4 Calibration Verification/Continuing Calibration: Calibration verification is to be performed every 20 analyses, and at the end of the last analytical run of each day, by analyzing an LCS and comparing the results to the established values. Table 8-2 gives acceptable recovery ranges for selected analytes. Failure (any recovery outside the acceptable recovery range) will trigger corrective action and reanalysis of samples since the last in-control LCS measurement.

8.5 Laboratory Duplicates: Laboratory duplicate (LAB DUP) samples are sample splits taken after completion of sample preparation. A LAB DUP is to be prepared and analyzed for every 20 samples analyzed. Two sample cups are filled from the same ground sample preparation batch so that the LAB DUP will be a measure of analysis precision on homogeneous samples. Control limits, stated in terms of RPDs, are indicated in Table 8-3. LAB DUPs out of control after corrective action steps indicated in Table 8.1 will be reported as reanalyzed and a comment reporting the out-of-control measurement will be included in the cover letter. The initial results and the reanalyses will both be reported in the data package.

8.6 Laboratory Replicates: Laboratory replicates (LAB REP) are sample splits taken before any laboratory sample preparation steps are taken. A LAB REP is to be prepared and analyzed for every 20 samples analyzed. Since the sample split occurs as

TABLE 8-2. PERMISSIBLE RECOVERIES FOR NIST LCS SAMPLES—QA/QC ACCEPTANCE WINDOWS

Analyte	Sample I.D.	Min Recovery (mg/kg)	Max Recovery (mg/kg)	Recovery Range
Arsenic	NIST 2710	501	751	80–120%
Copper	NIST 2710	2,714	3,190	92–108%
Iron	NIST 2710	32,100	35,500	95–105%
Lead	NIST 2710	5,260	5,810	95–105%
Manganese	NIST 2710	9,600	10,600	95–105%
Zinc	NIST 2710	6,600	7,300	95–105%
Arsenic	NIST 2711	68.3	142	65–135%
Copper	NIST 2711	91.2	137	80–120%
Iron	NIST 2711	27,500	30,300	95–105%
Lead	NIST 2711	1,070	1,255	92–108%
Manganese	NIST 2711	510	766	80–120%
Zinc	NIST 2711	315	385	90–110%

TABLE 8-3. MAXIMUM ACCEPTABLE LAB DUP RPDs FOR VARIOUS LEVELS OF CONTAMINATION

Concentration	RPD Control Limit for Laboratory Duplicate
<2 × MDL	No Limits
2 × MDL to 2 × LOQ	35%
2 × LOQ to 5 × LOQ	20%
5 × LOQ to 10 × LOQ	10%
>10 × LOQ	8%

Note 1: Stated in terms of MDL and LOQ, limits are analyte-dependent.

Note 2: If the two samples fall in different concentration windows, the wider control limit applies.

the first step in the process, the LAB REP will be a measure of total laboratory precision. Control limits, stated in terms of RPDs, are indicated in Table 8.4. LAB REPs out of control after corrective action steps indicated in Table 8.1 will be reported as reanalyzed and a comment reporting the out-of-control measurement will be included in the cover letter. The initial results and the reanalyses will both be reported in the data package.

8.7 Cross-Contamination Check Sample (XCS): Cross-contamination blanks are samples prepared by grinding and recovering clean sand after the grinding vessel has been decontaminated per paragraph 7.1.2. This gives a measure of cross-contamination from the prior sample, since any measurable analyte concentration must have come from the grinding vessel. Cross-contamination blanks will be prepared and analyzed at a frequency of one for every 50 natural samples. These samples will be used to monitor the expected level of cross-contamination established in the statistical summary of performance (ARCO 1994). XCS blanks with contamination above the control limits (less than or equal to the MDLs of each analyte) will be used to refine the estimate of maximum contamination and, if appropriate, modify the requirements of paragraph 8.9 for repreparation and reanalysis of potentially contaminated samples.

8.8 Sand Blanks: Prior to acceptance of a new batch of cleaning sand, one sample will be prepared and analyzed to evaluate potential contamination. Sand with elemental concentrations above the MDL for any analyte will not be used. Sand blanks will be prepared on a 1-per-50-natural sample frequency. Normally, the sand blank will follow immediately after the XCS and will use the same grinding vessel. Control limits are the MDL for each analyte except iron. The control limit for iron, due to contamination from the grinding vessel, is 500 mg/kg.

8.9 Cross-Contamination Reanalysis: Any sample with an indicated analyte concentration above the MDL must be re-prepared and reanalyzed if the preceding sample prepared in the same grinding vessel has any analyte concentration greater than 1,000 times the measured concentration in the sample. The results of the reanalysis will be reported as the analyzed value. The reanalysis will be noted in the report remarks. Examination of the historic XCS sample results show a possibility of contamination remaining in the grinding vessels following the preparation of particularly highly contaminated samples. This reanalysis requirement should eliminate the possibility of significant cross-contamination caused by residual contamination in the grinding vessels.

9.0 REPORTING REQUIREMENTS

9.1 As required by the *Clark Fork River Superfund Site Investigations Data Management/Data Validation Plan* (ARCO 1991a), the formal analytical report will include, as a minimum, the following elements:

- Cover letter that includes case narrative describing any problems encountered during sample analysis (including any required reanalyses), sample storage information sufficient to satisfy Level B data requirements, and reference to the analytical method (this revised LAP);

- Table of analytical results with results rounded to correct significant figures and undetected results reported at the MDLs using a *U* qualifier; the table of analytical results will identify the sample preparation groups;
- Laboratory quality control results (blanks, LAB DUPs, LAB REPs, LCSs, calibration verification results) with RPDs and percent recoveries, as applicable; QA/QC samples will be keyed to the appropriate sample preparation groups;
- MDLs and LOQs for each analyte and date of determination of such limits;
- Raw data printouts with date of analysis from Spectrace® spectrometer;
- Sample log for incoming samples that provides the correlation between laboratory sample identification numbers and field sample numbers;
- Sample preparation records (e.g., grinding vessel ID number, sample weight, laboratory ID);
- Electronic copy of analytical data in spreadsheet format; the laboratory currently can provide data under Microsoft's Works® (DOS Version 2.0) or Borland's Quattro Pro® (DOS Version 5.1);
- Chain-of-custody forms for all samples in the data package; and
- Sample analysis request forms, if different from the chain-of-custody form.

10.0 METHOD PERFORMANCE

10.1 This method has been demonstrated to be capable of producing unrestricted use quality ("enforcement quality") data for arsenic, cadmium, copper, lead, and zinc. Precision above the LOQ for each analyte ranges from about 10–15 percent near the LOQ to less than 5 percent well above the LOQ. Accuracy, as measured by the slope of a regression between the XRF values and the certified values of NIST- or NRCC-certified SRMs is better than 5 percent. Correlation with a single laboratory using Contract Laboratory Program (CLP) methodology is generally in the ±5- to 10-percent range. More detailed method performance data are presented in *Investigation of X-ray Fluorescence Analysis Accuracy and Precision on Smelter Hill Samples* (ARCO 1991c), *X-ray Fluorescence Analysis of Streamside Tailings Samples: Accuracy and Precision with Laboratory-Grade Instrumentation* (ARCO 1991d), and *XRF Performance Review for the period February 1992 Through August 1993* (ARCO 1994).

10.2 With appropriate modifications to sample preparation and x-ray excitation parameters, it should be possible to produce data of similar quality for other inorganic analytes above magnesium on the periodic table of the elements.

11.0 REFERENCES

ARCO. 1991a. Clark Fork River Superfund site investigations data management/data validation plan. January 1992. Prepared by PTI Environmental Services, Bellevue, WA. ARCO, Anaconda, MT.

ARCO. 1991b. Summary Report: Investigation of x-ray fluorescence analysis accuracy and precision on Smelter Hill samples. July 1991. Prepared by Ashe Analytics, Inc., Butte, MT. ARCO, Anaconda, MT.

ARCO. 1991c. X-ray fluorescence analysis of streamside tailings samples: accuracy and precision with laboratory-grade instrumentation. September 1991. Prepared by Ashe Analytics, Inc., Butte, MT. ARCO, Anaconda, MT.

ARCO. 1991d. Clark Fork River Superfund site investigations quality assurance project plan. June 1991. Prepared by PTI Environmental Services, Bellevue, WA. ARCO, Anaconda, MT.

ARCO. 1991e. Clark Fork River Superfund site investigations laboratory analytical protocol. January 1992. Prepared by PTI Environmental Services, Bellevue, WA. ARCO, Anaconda, MT.

ARCO. 1991f. Clark Fork River Superfund site investigations standard operating procedures. January 1992. ARCO, Anaconda, MT.

ARCO. 1994. XRF performance review for the period February, 1992 through August 1993. Prepared by Ashe Analytics, Inc., Butte, MT. ARCO, Anaconda, MT.

Bertin, E.P. 1975. Principles and practices of x-ray spectrometric analysis. Second Edition. Plenum Press, NY.

Spectrace Corp. Operator's manual for the Spectrace EX-5000® x-ray fluorescence spectrometer. Spectrace Corporation, Mountain View, CA.

Attachment 3.2
Field Data Sheets

BUTTE HILL COVER SOIL APPROVAL SUBMITTAL

6/14/2017

Source:
Sample #:

				Specification Met	
Description		**Specification**	**Sample**	**Yes**	**No**
Chemical (mg/kg)					
Arsenic (As)	<	97			
Cadmium (Cd)	<	4			
Copper (Cu)	<	250			
Lead (Pb)	<	100			
Zinc (Zn)	<	250			
pH (s.u.)					
	>	5.5			
	<	8.5			
SAR					
	<	12			
Saturation (%)					
	<	85			
	>	25			
EC (mmhos/cm)					
	<	4			
Textural Classification (USDA) <2.0 mm					
Loam					
Sandy loam					
Sandy clay loam					
Sandy clay					
Clay loam					
Silty clay					
Silty clay loam					
Silt loam					
Silt					
*Per EPA Approval (Loamy sand)					
Rock Content (%) (by volume)	<	45			

Other Information Requested

Organic Matter (%)		
WB	1.63	
Soil Nutrients		
	NO_3 (ug/g)	23.3
	P (ug/g)	30.3
	K (ug/g)	191
Particle Size		
	Sand (%)	80
	Silt (%)	10
	Clay (%)	10

Legend:
🟩 # Value - Criteria met
🟥 **# Value** - Does not meet Criteria

B-SB Representative ________________ Date: ________________

EPA Representative: ________________ Date: ________________

Level 2 Data Validation Checklist for Sample Analysis

Site:
Project:
Sample Date(s):
Data Validator:

Case No:
Sample Matrix:
Analysis Date(s):
Validation Date(s):

Laboratory:
Analyses:

1. Holding Times

Analyte	**Laboratory**	**Matrix**	**Method**	**Holding Times**	**Collection Date(s):**	**Analysis Date(s)**	**Holding Time Met (Y/N)**	**Affected Data Flagged (Y/N)**

Were any data flagged because of holding time? Y ☐ N ☐
Were any data flagged because of preservation problems? Y ☐ N ☐

Describe Any Actions Taken:

Comments:

2. Blanks

Were Method Blanks (MBs) analyzed at the frequency of 1 per analytical batch? Y ☐ N ☐
Were MBs within the control window? Y ☐ N ☐
Were any data flagged because of blank problems? Y ☐ N ☐

Describe Any Actions Taken:

Comments:

3. Laboratory Control Samples

Were Laboratory Control Samples (LCS) analyzed at the frequency of 1 per batch? Y ☐ N ☐
Were LCS results within the control window? Y ☐ N ☐
Were any data flagged because of LCS problems? Y ☐ N ☐

Describe Any Actions Taken:

Comments:

4. Duplicate Sample Results

Were Laboratory Duplicate Samples (LDS) analyzed at the frequency of 1 per batch? Y ☐ N ☐
Were LDS results within the control window? Y ☐ N ☐
Were any data flagged because of LDS problems? Y ☐ N ☐

Describe Any Actions Taken:

Comments:

5. Matrix Spike Sample Results

Were Laboratory Matrix Spike Samples (LMS) analyzed at the frequency of 1 per batch? Y ☐ N ☐
Were LMS results within the control window? Y ☐ N ☐
Were any data flagged because of LMS problems? Y ☐ N ☐

Describe Any Actions Taken:

Comments:

6. Field Blanks

Were field blanks submitted as specified in the QAPP? Y ☐ N ☐ N/A ☐
Were field blanks within the control window? Y ☐ N ☐ N/A ☐
Were any data qualified because of field blank problems? Y ☐ N ☐ N/A ☐

Describe Any Actions Taken:

Comments:

7. Field Duplicates

Were field duplicates submitted as specified in the QAPP? Y ☐ N ☐ N/A ☐
Were results for field duplicates within the control window? Y ☐ N ☐ N/A ☐
Were any data qualified because of field duplicate problems? Y ☐ N ☐ N/A ☐

Describe Any Actions Taken:

Comments:

8. Overall Assessment

Are there analytical limitations of the data that users should be aware of? Y ☐ N ☐

If so, explain:

Comments:

9. Authorization of Data Validation

	Data Validator	
Name:		**Reviewed by:**
Signature:	______________________________	______________________________
Date:	______________________________	______________________________

Level 2 Data Validation Checklist XRF Sample Analysis

Site:
Project:
Sample Date(s):
Data Validator:

Case No:
Sample Matrix:
Analysis Date(s):
Validation Date(s):

Laboratory:
Analyses:

1. Holding Times

Analyte	Laboratory	Matrix	Method	Holding Times	Collection Date(s)	Analysis Date(s)	Holding Time Met (Y/N)	Affected Data Flagged (Y/N)

*Reference for Holding Times –

Were any data flagged because of holding time? Y ☐ N ☒
What sample preparation steps were performed (i.e. drying, sieving etc.)?
Were the samples prepped according to the SAP/QAPP? Y ☒ N ☐

Describe Any Actions Taken:

Comments:

2. Energy Calibration (System Check)

Was the energy calibration performed at the frequency of once per day? Y ☐ N ☐
Was the energy calibration Resolution below 195? Y ☐ N ☐
Did the energy calibration run for at least 50 seconds? Y ☐ N ☐

Describe Any Actions Taken:

Comments:

3. SiO_2 Standards

Was the SiO_2 Standard analyzed at the beginning of analysis? Y ☐ N ☐
Was the SiO_2 Standard analyzed at the frequency of 1 per 20 natural samples? Y ☐ N ☐
Were the SiO_2 Standard results within the control limits? Y ☐ N ☐
Were any data flagged because of the SiO_2 Standard results? Y ☐ N ☐

Describe Any Actions Taken:

Comments:

4. Calibration Check Samples

Were the appropriate Calibration Check Samples (CCS) analyzed at the beginning of analysis? Y ☐ N ☐
Were the appropriate CCS analyzed at the frequency of 1 per 20 natural samples? Y ☐ N ☐
Were CCS results within the control limits? Y ☐ N ☐
Were any data flagged because of CCS problems? Y ☐ N ☐

Describe Any Actions Taken:

Comments:

5. Duplicate Sample Results

Were Duplicate Samples analyzed at the frequency of 1 per 20 natural samples?	Y ☐	N ☐
Were Duplicate Sample results within the control window?	Y ☐	N ☐
Were any data flagged because of duplicate sample results?	Y ☐	N ☐

Describe Any Actions Taken:

Comments:

6. Replicate Sample Results

Were Replicate Samples analyzed at the frequency of 1 per 20 natural samples?	Y ☐	N ☐
Were replicate sample results within the control window?	Y ☐	N ☐
Were any data flagged because of replicate sample results?	Y ☐	N ☐

Describe Any Actions Taken:

Comments:

7. Overall Assessment

Are there analytical limitations of the data that users should be aware of? Y ☐ N ☐

If so, explain:

Comments:

8. Authorization of Data Validation

Data Validator
Name:

Reviewed by:

Signature: ______________________________ ______________________________

Date: ______________________________ ______________________________

Level A/B Assessment Checklist

1. General Information

Site:
Project:
Client:
Sample Matrix:

2. Screening Result

Data are:
1. Unusable ____
2. Level A ____
3. Level B ____

I. **Level A**

Criteria – The following must be fully documented.	**Yes/No**	**Comments**
1. Sampling date		
2. Sampling team or leader		
3. Physical description of sampling location		
4. Sample depth (soils)		
5. Sample collection technique		
6. Field preparation technique		
7. Sample preservation technique		
8. Sample shipping records		

II. **Level B**

Criteria – The following must be fully documented.	**Yes/No**	**Comments**
1. Field instrumentation methods and standardization complete		
2. Sample container preparation		
3. Collection of field replicates (1/20 minimum)		
4. Proper and decontaminated sampling equipment		
6. Field custody documentation		
7. Shipping custody documentation		
8. Traceable sample designation number		
9. Field notebook(s), custody records in secure repository		
10. Completed field forms		

Attachment 3.3
Manufacturer Instructions

Instruction Manual

HI 99121
Soil pH
Test Kit

HANNA instruments

ISO 9000 Certified
Company since 1992

http://www.hannainst.com

Dear Customer,

Thank you for choosing a Hanna Product. Please read the instruction manual carefully prior to use. It will provide you with necessary information for the correct use of the kit.

Remove the test kit from the packing material and examine it carefully to make sure that no damage has occurred during shipping. If there is any noticeable damage, notify your Dealer or the nearest Hanna office immediately.

Each kit is supplied with:

• HI 991000	portable pH meter
• HI 1292D	pH electrode
• HI 721319	soil drill
• HI 7051M	soil preparation solution
• HI 70004	buffer solution pH 4.01 (1 pc.)
• HI 70007	buffer solution pH 7.01 (1 pc.)
• HI 721312	hard carrying case
• HI 740036	100 ml plastic beaker (1 pc.)

Note: Any damaged or defective item must be returned in its original packing materials.

Note: Read the HI 991000 instruction manual to ensure correct use of the meter.

SOIL pH

pH is the measure of the hydrogen ion concentration $[H^+]$. Soil can be acid, neutral or alkaline, according to its pH value.

Fig. 1 shows the relationship between the scale of pH and types of soil. Most plants prefer a pH range from 5.5 to 7.5; but some species prefer more acid or alkaline soils. Nevertheless, every plant requires a particular range of pH, for optimum growth.

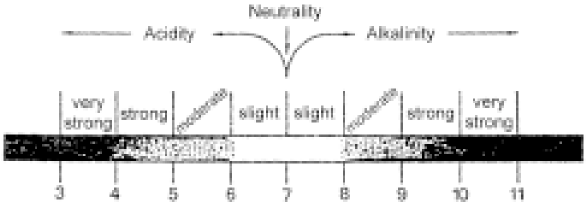

Fig. 1. Types of soil according to the pH value

pH strongly influences the availability of nutrients and the presence of micro-organisms and plants in the soil.

For example, fungi prefer acidic conditions whereas most bacteria, especially those supplying nutrients to the plants, have a preference for moderately acidic or slightly alkaline soils. In fact, in strongly acidic conditions, nitrogen fixing and the mineralization of vegetable residual is reduced.

Plants absorb the nutrients dissolved in the soil water and the nutrient solubility depends largely on the pH value. Hence, the availability of elements is different at different pH levels (Fig. 2).

Each plant needs elements in different quantities and this is the reason why each plant requires a particular range of pH to optimize its growth.

For example, iron, copper and manganese are not soluble in an alkaline environment. This means that plants needing these elements should theoretically be in an acidic type of soil. Nitrogen, phosphorus, potassium and sulfur, on the other hand, are readily available in a pH range close to neutrality.

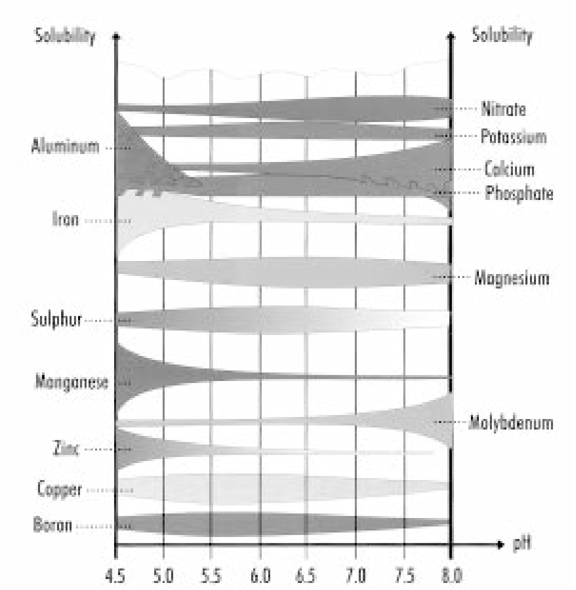

Fig. 2. Solubility of the elements according to varying pH values

Furthermore, abnormal pH values, increase the concentration of toxic elements for plants. For example, in acid conditions, there can be an excess of aluminum ions in such quantities that the plant can not tolerate.

Negative effects on chemical and physical structure are also present when pH values are too far from neutral conditions (break up of aggregates, a less permeable and more compact soil).

Management of the soil in relation to the pH value

Once the pH value is known, it is advisable to choose crops that are suitable for this range (e.g. in an acid soil, cultivate rice, potato, strawberry).

Add fertilizers that do not increase acidity (for example urea, calcium nitrate, ammonium nitrate and superphosphate) or lower alkalinity (e.g. ammonium sulfate).

It is recommended that a cost evaluation is made prior to commencement of the soil pH modification. Corrective substances can be added to modify the soil pH, however, the effects are generally slow and not persistent. For example, by adding lime, the effects in clay soil can last for as long as 10 years, but only 2-3 years in a sandy soil.

For an acid soil, we can use substances such as lime, dolomitic, limestone and marl, according to the nature of the soil (Tab.1).

Soil Ameliorants	Clay soil	Silty soil	Sandy soil
CaO	30-50	20-30	10-20
$Ca(OH)_2$	39-66	26-39	13-26
$CaMg(CO_3)_2$	49-82	33-49	16-33
Ca CO_3	54-90	36-54	18-36

Tab.1. Quantity (q/ha) of pure compound necessary to increase 1 unit of pH

High pH levels can depend on different elements, hence, there are different methods for its correction.

- Soils rich with limestone :

Add organic matter (this is due to the fact that non-organic ameliorants such as sulfur and sulfuric acid might not make economic sense due to the large quantities needed).

- Alkaline-saline soils :

Alkalinity is due to the presence of salts (in particular a high concentration of sodium can be harmful).

Irrigation washes away salts, hence, an appropriate use of irrigation can provide positive results (drop-irrigation being the most recommended).

If alkalinity is caused by sodium, it is recommended to add substances such as gypsum (calcium sulfate), sulfur or other sulfuric compounds (Tab.2). Also in this case, a cost evaluation is necessary.

Soil ameliorants (pure compounds)	Quantity (Kg)
Calcium chloride: $CaCl_2 \cdot 2H_2O$	85
Sulfuric acid: H_2SO_4	57
Sulfur: S	19
Iron sulfate: $Fe_2(SO_4)_3 \cdot 7H_2O$	162
Aluminum sulfate: $Al_2(SO_4)_3$	129

Tab.2. Quantities provide the same result as 100 Kg of gypsum

Procedure for direct ground measurement

1) Dig, discarding 5 cm of topsoil
2) Perforate the soil (with HI 721319 soil drill) to a depth of about 20 cm or more
3) If the soil is dry, moisten it with a small amount of distilled water

4) Wash the electrode with tap water (not distilled)
5) Insert the electrode pushing it slightly into the soil to ensure proper contact
6) Observe the measurement
7) Wash the electrode with tap water (not distilled) and (using a finger) gently remove any soil remaining on the electrode (avoid using a rag or cloth)
8) Repeat the procedure in different locations in the field
9) Consider the average of the measured data

For best result, it is advisable to measure the pH of a soil solution, using a sample of soil and soil preparation solution HI 7051; it is better to use this procedure if you have to test a stony field in which you risk damaging the electrode.

Procedure for the measurement of soil solution (1:2,5)

A) Sampling
1) Extracting Soil Sample
   Take 1 sample per 1000 m^2 (0.25 acre) of homogeneous area
   Even for small areas, 2 samples are recommended (the more the samples, the better the end-results, because the result is more representative)
2) Avoid extracting samples from soil presenting obvious anomalies and consider them separately
3) Sample quantity:
   Take the same quantity of soil for each sample. For example, use bags with similar dimensions (1 bag per sample)
4) Depth of extraction:
   General: dig and discard 5 cm (2") of topsoil
   Herbaceous crops: from 20 to 40 cm of depth (8" to 16")
   Orchards: from 20 to 60 cm of depth (8" to 24")
5) Spread the soil samples on the pages of a newspaper and let the soil dry in a shady place or put it in an oven at 40° C
6) Crumble the dried soil and mix all the samples together to obtain a homogeneous mixture, discarding stones and vegetable residues
7) From this mixture, take the soil sample for analysis

B) Soil solution preparation and measurement
1) Sift the soil at 2 mm
2) Weigh 10 gr of soil and put it in 25 ml of soil preparation solution HI 7051 (utilize the apposite beaker) or 20 gr of soil per 50 ml of soil preparation solution HI 7051
3) Mix for 30 seconds
4) Wait 5 minutes
5) Mix again and measure the pH of the solution

ORGANIC SUBSTRATE (PEAT AND MOULD)

pH measurement of organic substrates is important in greenhouses and nursery growing pots. pH should be checked at the outset to make sure that the pH of the substrate bought is that desired (pH can change if too much time elapses from the date of packaging to the moment of utilization).

A) Direct Measurement in pot

If the substrate is not wet, add a little distilled water. Insert the electrode into the soil and measure.

B) Measurement of the Organic Substrate Solution (1: 2 ratio).

Let the substrate dry;
Discard the coarse vegetable residues and pebbles;
In a beaker, prepare a solution composed of 1 part mould and 2 parts HI 7051 solution (for example: fill the beaker with the substrate up to 50 ml, press it gently, empty the content in an other container and add 100 ml of HI 7051 solution);
Mix for 30 seconds and then wait for 5 minutes;
Mix again and measure the pH of the solution.

IRRIGATION WATER

The quality of irrigation water is a very important factor to consider. If the pH value is very far from neutral (pH = 7), probably, other anomalies are present.
Ranges for evaluation of water quality:
- good from 6 to 8,5
it can be utilized without problems
- sufficient from 5 to 6 and from 8,5 to 9
sensible crops could have problems
- scarce from 4 to 5 and from 9 to 10
utilize it carefully, avoid wetting the vegetation
- very scarce with pH < 4 and pH > 10

There are other anomalies that have to be identified via chemical analysis.

NUTRIENT SOLUTION

A rational fertilization is needed for optimum growth of plants in greenhouses; the pH of a nutrient solution (water + fertilizer) has to meet the plants need. If you have a fertirrigation system with automatic pH control, ensure that it is functioning properly.

Check the pH of the solution distributed to the plants as well as any recycled solutions.

ORCHARD PLANTS

	Preferred pH Range		Preferred pH Range
Apple	5-6.5	Orange	5-7
Apricot	6-7	Peach	6-7.5
Cherry	6-7.5	Pear	6-7.5
Grapefruit	6-7.5	Plum	6-7.5
Grapevine	6-7	Pomegranate	5.5-6.5
Lemon	6-7	Walnut	6-8
Nectarine	6-7.5		

VEGETABLES AND HERBACEOUS CULTIVATIONS

	Preferred pH Range		Preferred pH Range
Artichoke	6.5-7.5	Pepper	6-7
Asparagus	6-8	Early Potato	4.5-6
Barley	6-7	Late Potato	4.5-6
Bean	6-7.5	Sweet Potato	5.5-6
Brussels Sprout	6-7.5	Pumpkin	5.5-7.5
Early carrot	5.5-7	Rice	5-6.5
Late carrot	5.5-7	Soybean	5.5-6.5
Cucumber	5.5-7.5	Spinach	6-7.5
Egg Plant	5.5-7	Strawberry	5-7.5
Lettuce	6-7	String	6-7.5
Maize	6-7.5	Sugar beet	6-7
Melon	5.5-6.5	Sunflower	6-7.5
Oat	6-7	Tomato	5.5-6.5
Onion	6-7	Watermelon	5.5-6.5
Pea	6-7.5	Wheat	6-7

LAWN

	Preferred pH Range
Lawn	6-7.5

GARDEN PLANTS AND FLOWERS

	Preferred pH Range		Preferred pH Range
Acacia	6-8	Ligustrum	5-7.5
Acanthus	6-7	Magnolia	5-6
Amaranth	6-6.5	Narcissus	6-8,5
Bougainvillea	5.5-7.5	Oleander	6-7.5
Dahlia	6-7.5	Oleander	6-7.5
Erica	4.5-6	Paulownia	6-8
Euphorbia	6-7	Portulaca	5.5-7.5
Fuchsia	5.5-7.5	Primula	6-7.5
Gentian	5-7.5	Rhododendron	4.5-6
Gladiolus	6-7	Roses	5.5-7
Hellebore	6-7.5	Sedum	6-7.5
Hyacinth	6.5-7.5	Sunflower	5-7
Iris	5-6.5	Tulip	6-7
Juniper	5-6.5	Viola	5.5-6.5

HOUSEPLANTS

	Preferred pH Range
Abutilon	5.5-6.5
African violet	6-7
Anthurium	5-6
Araucaria	5-6
Azalea	4.5-6
Begonia	5.5-7.5
Camellia	4.5-5.5
Croton	5-6
Cyclamen	6-7
Dieffenbachia	5-6
Dracaena	5-6
Freesia	6-7.5
Gardenia	5-6
Geranium	6-8
Hibiscus	6-8
Jasmine	5.5-7
Kalanchoe	6-7.5
Mimosa	5-7
Orchid	4.5-5.5
Palms	6-7.5
Peperomia	5-6
Philodendron	5-6
Yucca	6-7.5

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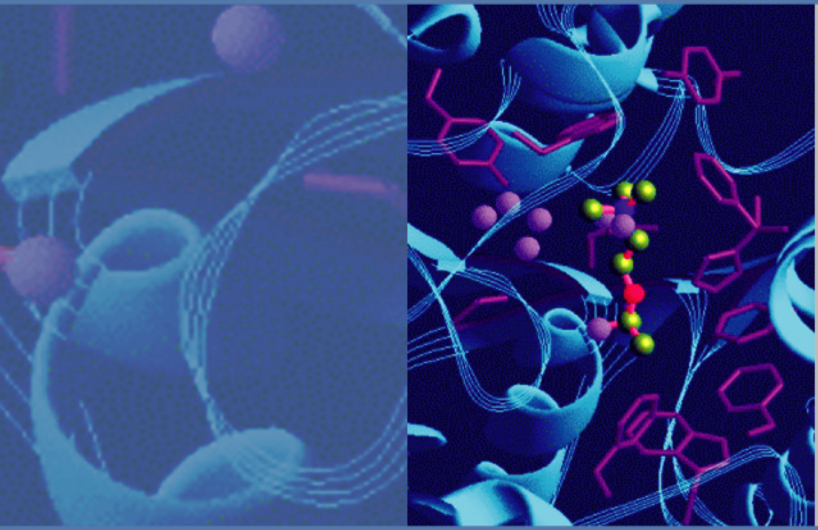

Thermo Fisher Scientific Niton Analyzers

XL3 Analyzer

Version 8.0

User's Guide (Abridged)

Refer to NITON XL3 Resource Guide for complete information

Revision A **October 2011**

Part of Thermo Fisher Scientific

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Manual Overview

Warnings, Cautions, and Notes

Warnings

Warnings are extremely important recommendations, violating which may result in either injury to yourself or others, or damage to your analyzer and/or data. Warnings will always be identified as Warnings in the text, and will always be visually presented as follows:
WARNING This is a Warning.

Example Warning:

WARNING Tampering with the 5,500 ppm (Lead high) lead-in-soil standard may cause exposure to lead dust. Keep all standards out of reach of children.

Cautions

Cautions are important recommendations. Cautions will always be identified as Cautions in the text, and will always be visually presented as follows:
CAUTION This is a Caution.

Example Caution:

CAUTION Never tamper with Test Standards. They should not be used unless they are completely intact

Notes

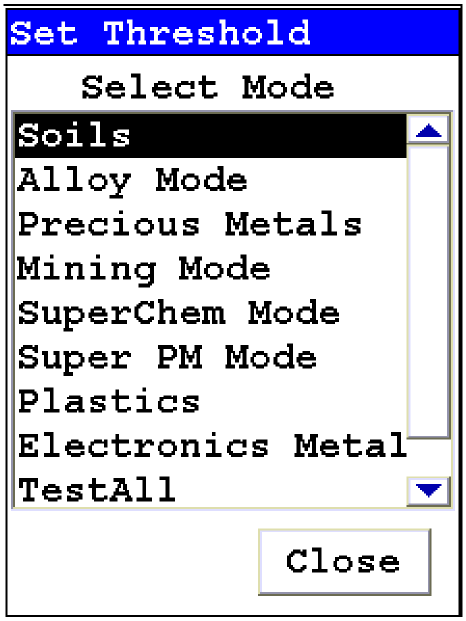
Notes are informational asides which may help you with your analyses. Notes will always be identified as Notes in the text, and will always be visually presented as follows:
Note This is a Note.

Example Note:

Note For defensible Quality Control, keep a record of the time and precision of every calibration

Figures

Figures are illustrations used to show what something looks like. Figures will always be labelled and identified as Figures directly below the Figure itself, and will always be visually presented as follows:



Set Threshold

Select Mode

Soils
Alloy Mode
Precious Metals
Mining Mode
SuperChem Mode
Super PM Mode
Plastics
Electronics Metal
TestAll

Figure 1. **This is a Figure**

Physical Buttons

Physical Buttons are actual buttons on the analyzer which must be pushed to activate their function. Physical Buttons will always be identified as Buttons in the text, and will always be visually presented as follows:

This is a Physical Button.

Example Physical Buttons:

On/Off/Escape Button, Clear/Enter Button, Interlock Button, and Trigger Button.

Other Hardware

Other Hardware refers to any physical part of the analyzer which performs a necessary function. Other Hardware will always be visually presented as follows:

This is an example of Other Hardware.

Example Other Hardware:

Battery, Touch Screen Display, Measurement Window, and USB Cable

Using Your Analyzer

This section discusses the basics of using your analyzer, no matter the specific type of analysis you wish to perform. First we go over analyzer safety, particularly radiation safety. Using an X-ray based analyzer safely is very important, and not difficult, provided you read, understand, and follow these guidelines. Secondly, we outline the startup procedure we recommend for daily use to ensure that your analyzer is performing properly and at its most efficient level.

Safely and Effectively Using Your Analyzer

CAUTION Niton analyzers are not intrinsically safe analyzers. All pertinent Hot Work procedures should be followed in areas of concern.

Radiation and General Safety

WARNING Always treat radiation with respect. Do not hold your analyzer near the measurement window during testing. Never point your analyzer at yourself or anyone else when the shutter is open.

Radiation and General Safety

This section covers topics related to radiation safety and general safety when using a Thermo Scientific Niton XL3 analyzer. At a minimum all operators of the analyzer should be familiar with the instructions provided in this chapter in order to handle the analyzer in a safe manner. In addition to reading the information presented on the following pages, Thermo Fisher Scientific recommends that instrument users participate in a radiation safety and operational training class.

Radiation Protection Basics

The Niton Model XL3t analyzer contains an x-ray tube which emits radiation only when the user turns the x-ray tube on. When the x-ray tube is on and the shutter is open, as during a measurement, the analyzer emits a directed radiation beam - see Figures 1 and 2. Reasonable effort should be made to maintain exposures to radiation as far below dose limits as is practical. This is known as the ALARA (As Low as Reasonably Achievable) principle. For any given source of radiation, three factors will help minimize your radiation exposure: Time, Distance, and Shielding.

The Niton Model XL3p analyzer contains a radioactive sealed source. Radiation from this source is fully contained within the device when not in use and allowed to escape through the measurement window only while the user is analyzing a sample. Radiation emission is controlled by a shutter. The analyzer emits a directed radiation beam (See Figure 1 and Figure

2) when the shutter is open during a measurement. Reasonable effort should be made to maintain exposures to radiation as far below dose limits as is practical. This is known as the ALARA (As Low as Reasonably Achievable) principle. For any given source of radiation, three factors will help minimize your radiation exposure: Time, Distance, and Shielding.

Time

The longer you are exposed to a source of radiation the longer the radiation is able to interact in your body and the greater the dose you receive. Dose increases in direct proportion to length of exposure.

Distance

The closer you are to a source of radiation, the more radiation strikes you. Based on geometry alone, dose increases and decreases with an inverse-squared relation to your distance from the source of radiation (additional dose rate reduction comes from air attenuation). For example, the radiation dose one foot from a source is nine times greater than the dose three feet from the source. Remember to keep your hands and all body parts away from the front end of the analyzer when the shutter is open to minimize your exposure.

Shielding

Shielding is any material that is placed between you and the radiation source. The more material between you and the source, or the denser the material, the less you will be exposed to that radiation. Supplied or optional test stands are an additional source of shielding for analysis. A backscatter shield accessory is also available and may be appropriate in some applications.

Exposure to Radiation

Human dose to radiation is typically measured in rem, or in one-thousandths of a rem, called millirem (mrem), 1 rem = 1000 mrem. Another unit of dose is the Sievert (Sv), 1 Sv = 100 rem. The allowable limit for occupational exposure in the U.S (and many other countries) is 5,000 mrem/year (50 mSv/year) for deep (penetrating) dose and 50,000 mrem/year (500 mSv/year) for shallow (i.e., skin) dose or dose to extremities. Deep, shallow, and extremity exposure from a properly used Niton XL3t analyzer should be less than 200 mrem per year, (2.0 mSv per year) even if the analyzer is used as much as 2,000 hours per year, with the shutter open continuously. The only anticipated exceptions to the 200 mrem maximum annual dose are: 1) routine and frequent analysis of plastic samples without use of a test stand, backscatter shield, or similar additional protective measures, or 2) improper use where a part of the body is in the primary beam path.

Note NEVER OPERATE THE DEVICE WITH A PART OF YOUR BODY IN THE PRIMARY BEAM PATH OR WITH THE PRIMARY BEAM PATH DIRECTED AT ANYONE ELSE.

Also, consider the use of protective accessories such as a shielded test stand or backscatter shield (or equivalent) when performing routine and/or frequent analysis of any of the following:

- plastic (or similarly low density) samples,
- thin samples (such as foils, circuit boards, and wires)
- samples that are smaller than the analysis window.

Shown in Table 1are the typical background radiation doses received by the average member of the public. The radiation dose limits for radiation workers in the US are also shown in Table 2.

Table 1. Typical Radiation Doses Received (Source: NCRP 1987)

Category	Dose in mrem	Dose in mSv
Average total dose in US (annual)	360	3.6
Average worker exposure (annual)	210	2.1
Average exposure for an underground miner	400	4.0
Exposure for airline crew (1,000 hours at 35,000 ft)	500	5.0
Additional from living in Denver at 5300’ (annual)	25	.25
Additional from 4 pCi/l radon in home	1,000	10.0
Typical Chest X-Ray	6	0.06
Typical Head or Neck X-Ray	20	0.2
Typical pelvis/hip x-ray	65	0.65
Typical lumbar spine x-ray	30	0.3
Typical Upper G.I. x-ray	245	2.45
Typical Barium enema x-ray	405	4.05
Typical CAT scan	110	1.10

Table 2. Annual Occupational Dose Limits for Radiation Workers
(Source: Code of Federal Regulations Title 10, Part 20)

Category	Dose in mrem	Dose in mSv
Whole Body	5000	50
Pregnant Worker (during gestation period)	500	5
Eye Dose Equivalent	15,000	150
Shallow dose equivalent to the skin or any extremity or organ	50,000	500
Maximum allowable dose for the general public (annual)	100	1.0
For a Minor	500	5.0

Monitoring your radiation exposure

Individuals can be monitored for the radiation dose they receive by use of radiation dosimetry devices (dosimeters). Monitoring dose using a dosimeter can be a way of identifying improper use and at the same time demonstrating proper use. In some locations, dosimetry is required by regulations and in others it is optional. It is normally required when the user could reasonably be expected to receive in excess of 10% of the annual dose limit. Thermo Fisher Scientific recommends that you determine and obey the local regulatory requirements concerning radiation monitoring of occupational workers.

Two common types of dosimeters are whole-body badges and ring badges. Whole body badges are often attached to the user's torso (e.g., clipped to the collar, shirt pocket, or waist as appropriate). A ring badge is worn on the finger as a measure of maximum extremity dose. When worn, the specific location of the dosimeter should be that part of the body that is expected to receive the highest dose. This location will depend on how the analyzer is used and so it may not be the same for all users. Dosimetry services are offered by many companies. Two companies offering dosimetry services in the USA and much of the world are:

Table 3. Dosimeters

Company	Global Dosimetry Solutions	Landauer, Inc.
Address	2652 McGaw Avenue	2 Science Road
City and State	Irvine, CA 92614	Glenwood, IL 60425-9979
Website	www.dosimetry.com	www.landauerinc.com
Phone Number	(800) 251-3331	(800) 323-8830

Note Wearing a dosimeter badge does not protect you against radiation exposure. A dosimeter badge only measures your exposure (at the dosimeter location).

Pregnancy and Radiation Exposure

International guidance documents (e.g., ICRP Publication 60 and NCRP Publication 116*) recommend that the radiation dose to the embryo/fetus of a pregnant woman should not exceed a total of 500 mrem (10% of normal radiation worker limit) during the gestation period. While this dose limit exceeds the dose limit to a trained operator, pregnant workers may want to take special precautions to reduce their exposure to radiation. For more information see the U.S. NRC Regulatory Guide 8.13 "Instruction Concerning Prenatal Radiation Exposure" which can be found on the resource CD.

* The International Commission on Radiological Protection, ICRP, is an independent Registered Charity, established to advance for the public benefit the science of radiological protection, in particular by providing recommendations and guidance on all aspects of protection against ionizing radiation.

* The National Council on Radiation Protection and Measurements (NCRP) was chartered by the U.S. Congress in 1964 as the National Council on Radiation Protection and Measurements.

How to Use the Niton XL3t Analyzer Safely

The Niton XL3t analyzer is designed to be safe to operate provided that it is used in accordance with manufacturer's instructions. Under conditions of normal use, monitored operators seldom receive a measurable dose and have not been known to receive in excess of 10% of the annual occupational dose limits (a criteria that would require monitoring under regulation in the U.S.). In addition to proper use of the XL3t, it is recommended that you follow these precautions to ensure your safety and the safety of those around you.

Know where the beam is

The primary beam is a directed beam out of the front of the analyzer that can have high dose rates. The secondary beam, or scattered beam, has much lower dose rates.

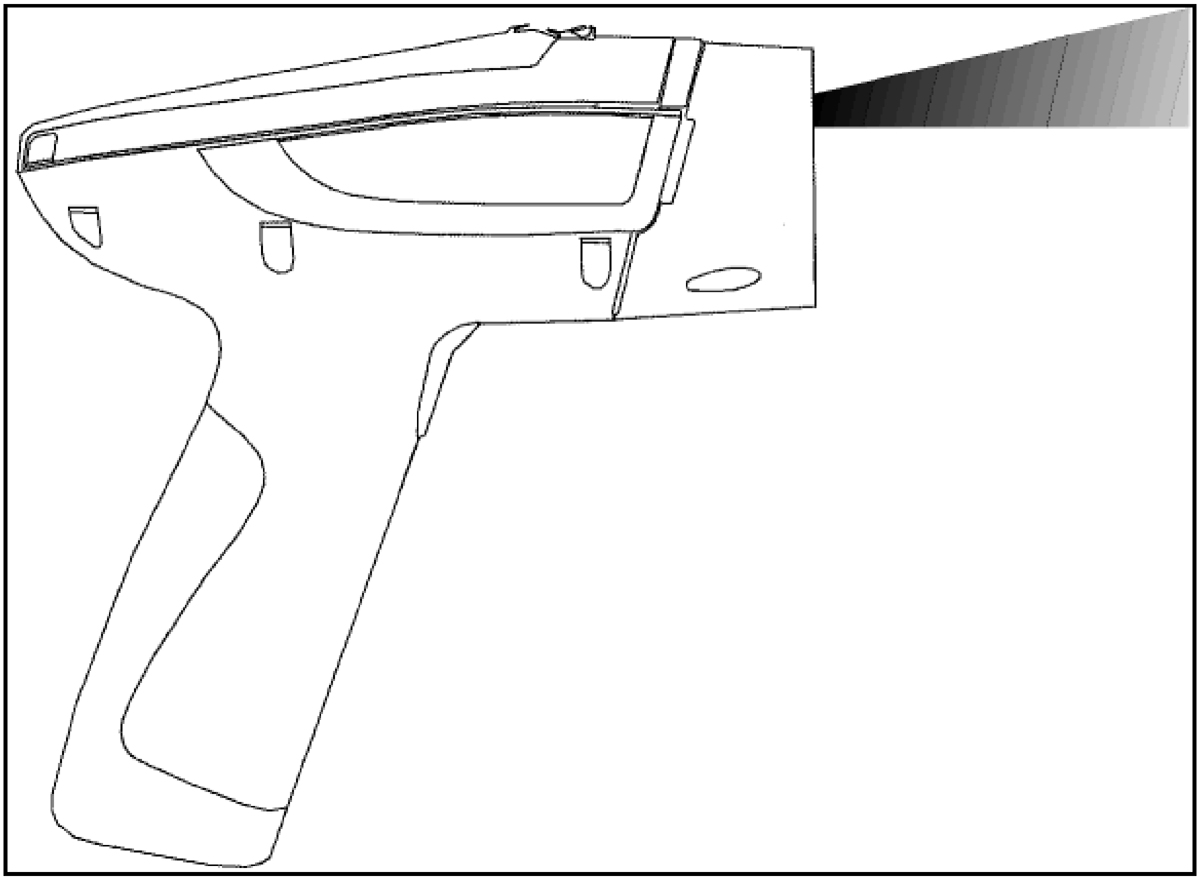


Figure 2. Primary Beam

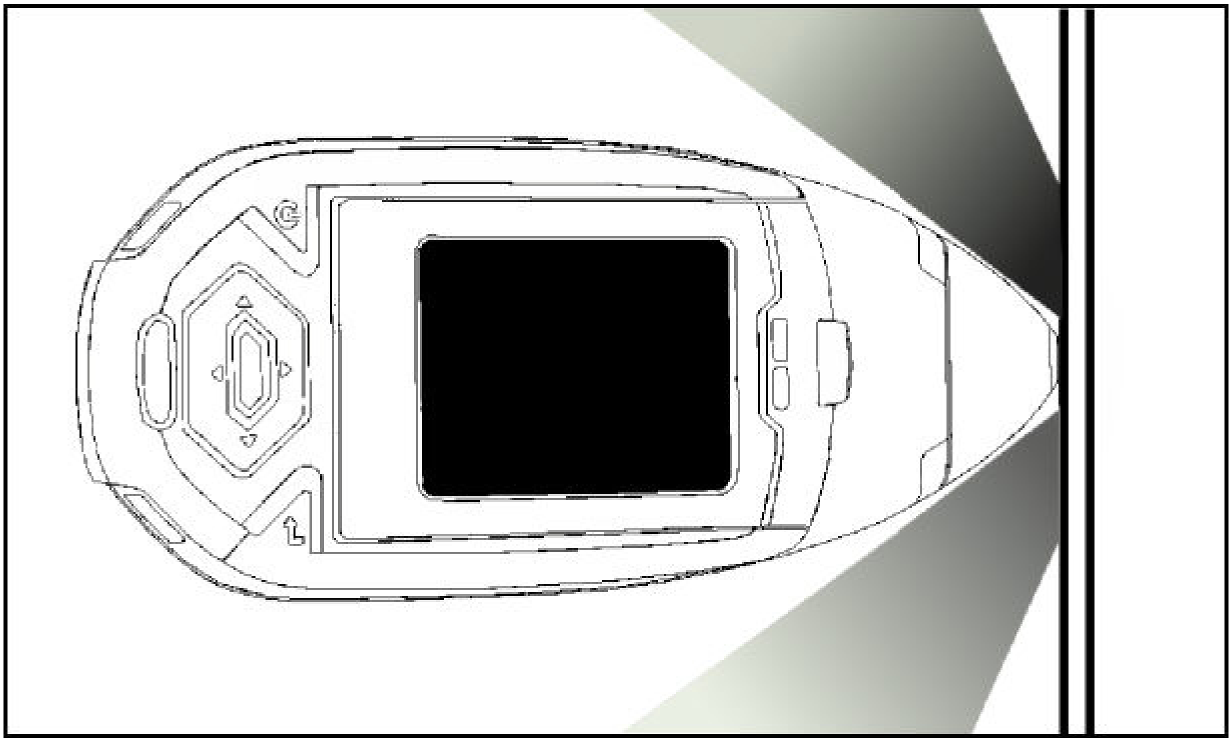


Figure 3. Secondary (Scattered) Beam

The Shutter-Open Indicator Lights

When the lights are flashing, the primary beam is on, and radiation is being emitted from the front of the analyzer.

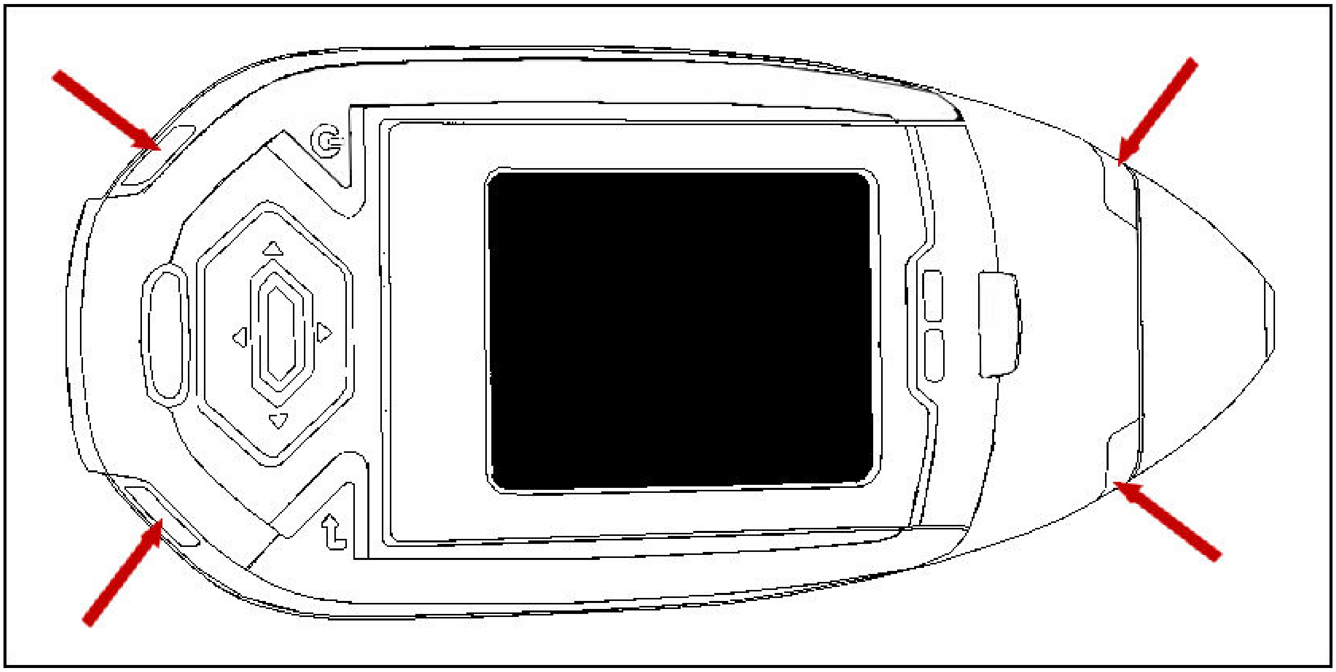


Figure 4. The X-ray Beam Indicator Lights

Handle and Use with Respect

Avoid holding the front of the analyzer when the x-ray tube is energized and the shutter is open. Never point the instrument at yourself or anyone else when the shutter is open and the x-ray tube is energized. Never look into the path of the primary beam.

Follow a Radiation Protection Program

Your organization should establish, document, and follow a Radiation Protection Program. An example of such a program can be found on the resource CD (provided with the instrument).

Take Proper Care of your Niton XL3

Keeping your analyzer maintained in good condition will help minimize the risk of accidental exposure. Mechanical malfunction of the shutter can be avoided by maintaining the measurement window, as described in the User Guide. This prevents foreign objects from entering your analyzer

Avoid Over-Exposures

Direct contact with the window could result in overexposures in the times indicated inTable 4 below.

Table 4. Potential Exposure Limit Times

Location of Dose	Limit	Time to Reach Limit
Deep Dose / Whole Body	5 rem (50 mSv)	2.1 minutes
Shallow Dose / Extremities	50 rem (500 mSv)	0.95 minutes
Member of Public (i.e. untrained operator)	0.1 rem (1 mSv)	2.5 seconds*

Extremity is defined by the NRC as the hand, elbow, arm below the elbow, foot, knee, or leg below the knee. Whole Body is defined by the NRC as the head, trunk (including male gonads), arms above the elbow, or legs above the knee.

*Based on maximum deep dose rate and US exposure limit.

Safe Handling of Samples

As mentioned many times in this chapter, never place any part of your body in the path of the x-ray beam. There is always a safe way to handle samples whether they are small, irregularly shaped, or of low density. Never look into the path of the primary beam.

Small Samples

A small sample would be any sample that is smaller than the measurement window. Small samples present a unique risk because they don't block the entire beam path. The difficulty with placing small samples down on a work surface to analyze them is that you may get readings from the work surface that interfere with analytical results. A test stand is an effective way of analyzing small samples accurately and safely. Never hold samples during analysis or look into the path of the primary beam.

Irregularly Shaped Samples

Irregularly shaped samples may not allow the proximity button to be depressed, or they may not entirely cover the primary beam and cause additional scattering. A back scatter shield is a safe way of reducing your radiation exposure while effectively analyzing an irregularly shaped sample.

Low Density Materials (such as plastics).

X-rays are attenuated more through denser materials and less through low density materials such as plastic. This causes higher dose rates in the scattered radiation. If you are frequently handling low density samples, you should consider the use of test stands, backscatter shields, or the equivalent.

Niton XL3t Radiation Profile

Radiation Meter Information

Model: Bicron MicroRem

SN: 2057

Cal Due: 10/10/2009

Background Radiation Level

<0.01 mr/hr

Table 1-4 - Niton XL3t Radiation Profile - Scatter Measurements - mRem/hr

kV	uA	Range	Substrate	Max @ 5cm	Max @ 30 cm	Max @ Trigger
50	40	EM, GM, S, T, IP, PM, DA, M, E, P (Main Filter) S, T, M, E (High Filter)	Plastic	40	3.5	2
50	40	EM, GM, S, T, IP, PM, DA, M, E, P (Main Filter) S, T, M, E (High Filter)	Soil	8	0.4	0.07
20	100	S, P, T, M, E (Low Filter)	Aluminum	0.015	0.01	0.01
20	100	S, P, T, M, E (Low Filter)	Stainless	0.015	0.01	0.01
20	100	S, P, T, M, E (Low Filter)	Plastic	0.13	0.015	0.015
20	100	S, P, T, M, E (Low Filter)	Soil	0.015	0.015	0.015
15	100	IP, EM (Low Filter)	Aluminum	0.015	0.015	0.015
15	100	IP, EM (Low Filter)	Stainless	0.015	0.015	0.015

* GM = General Metals, EM = Electronics Metals, DA = Dental Alloy, PM = Precious Metals, M = Mining, S = Soil, E = Exploration, IP = Industrial Paint, T = Thin Sample, P = Plastic

Scatter Measurements off various substrates - Dose Rates in mRem/hr

Table 1-5 - Niton XL3t Radiation Profile - Scatter Measurements - µSv/hr

kV	uA	Range	Substrate	Max @ 5cm	Max @ 30 cm	Max @ Trigger
50	40	EM, GM, S, T, IP, PM, DA, M, E (P (Main Filter) S, T, M, E (High Filter)	Plastic	400	35	20
50	40	EM, GM, S, T, IP, PM, DA, M, E (P (Main Filter) S, T, M, E (High Filter)	Soil	80	4	0.7
20	100	S, P, T, M, E (Low Filter)	Aluminum	0.15	0.1	0.1
20	100	S, P, T, M, E (Low Filter)	Stainless	0.15	0.1	0.1
20	100	S, P, T, M, E (Low Filter)	Plastic	1.3	0.15	0.15
20	100	S, P, T, M, E (Low Filter)	Soil	0.15	0.15	0.15
15	100	IP, EM (Low Filter)	Aluminum	0.15	0.15	0.15
15	100	IP, EM (Low Filter)	Stainless	0.15	0.15	0.15

Notes:

Scatter measurements were taken at a radius of 5 or 30 cm around the nose of the analyzer with the highest scatter dose rate being recorded.

Scatter Measurements off various substrates - Dose Rates in µSv/hr

* GM = General Metals, EM = Electronics Metals, DA = Dental Alloy, PM = Precious Metals, M = Mining, S = Soil, E = Exploration, IP = Industrial Paint, T = Thin Sample, P = Plastic

Table 1-6 Niton XL3t Radiation Profile - In Beam Measurements - Rem/hr

kV	uA	Range	Contact Deep	Contact Shallow	5cm Deep	30 cm Shallow
50	40	EM, GM, S, T, IP, PM, DA, M, E, P (Main Filter) S, T, M, E (High Filter)	110	410	8.4	1.3
20	100	S, P, T, M, E (Low Filter)	150	3200	0.52	0.05
15	100	IP, EM (Low Filter)	14.0	1100	0.43	0.042

In Beam Measurements - Dose Rates in Rem/hr

* GM = General Metals, EM = Electronics Metals, DA = Dental Alloy, PM = Precious Metals, M = Mining, S = Soil,

E = Exploration, IP = Industrial Paint, T = Thin Sample, P = Plastic

Reported results are based on measurement results that have been reduced to 2 significant digits by rounding up. For example, a measurement result of 1441 would be reported as 1500.

Table 1-7 Niton XL3t Radiation Profile - In Beam Measurements - mSv/hr

kV	uA	Range	Contact Deep	Contact Shallow	5cm Deep	30cm Shallow
50	40	EM, GM, S, T, IP, PM, DA, M, E, P (Main Filter) S, T, M, E (High Filter)	1100	4,100	84.0	13
20	100	S, P, T, M, E (Low Filter)	1500	32000	5.2	0.50
15	100	IP, EM (Low Filter)	140	11000	4.3	0.42

Notes:

In beam dose rates were measured using thermoluminescent dosimeters (TLDs) or Optically Stimulated Luminescent Dosimeters (OSL).

In Beam Measurements - Dose Rates in mSv/hr

* GM = General Metals, EM = Electronics Metals, DA = Dental Alloy, PM = Precious Metals, M = Mining, S = Soil,

E = Exploration, IP = Industrial Paint, T = Thin Sample, P = Plastic

Reported results are based on measurement results that have been reduced to 2 significant digits by rounding up. For example, a measurement result of 1441 would be reported as 1500.

Niton XL3t GOLDD Plus Radiation Profile

Table 1-8 - Niton XL3t GOLDD Plus Radiation Profile - In Beam Measurements - mSv/hr

kV	uA	Range	Contact Deep	Contact Shallow	5cm Deep	30cm Deep
50	40	EM, MC, P, TA, PP (Main) M, S, T (High)	440	1300	74	5.3
50	40	GM, PM, M, S, IP (Main)	1500	3800	360	22
20	100	EM, P, TA, M, S, T (Low)	690	19000	190	9.2
15	133.3	GM (Low)	240	13000	50	2.3
8	200	GM, P, M (Light)	0.30	17000	0.10	<0.003

Notes:
*SAMPLE TYPES (MODES)
GM=General Metals, M=Mining, EM=Electronics Metals, S=Soils, PM=Precious Metals, IP=Industrial Paint (Action lead Paint & Quantify lead Paint), MC=Metal Coatings, PP=Painted Products, P=Plastics, TG=Test All Geo (soil and mining), TA=Test All (consumer products), T=Thin
Reported results are based on measurement results that have been reduced to 2 significant digits by rounding up. For example, a measurement result of 1441 would be reported as 1500.

Table 1-9 - Niton XL3t GOLDD Plus Radiation Profile - In Beam Measurements - Rem/hr

kV	uA	Range	Contact Deep	Contact Shallow	5cm Deep	30cm Deep
50	40	EM, MC, P, TA, PP (Main) M, S, T (High)	44	130	7.4	0.53
50	40	GM, PM, M, S, IP (Main)	150	380	36	2.2
20	100	EM, P, TA, M, S, T (Low)	69	1900	19	0.92
15	133.3	GM (Low)	24	1300	5.0	0.23
8	200	GM, P, M (Light)	0.030	1700	0.010	<0.0003

Notes:
*SAMPLE TYPES (MODES)
GM=General Metals, M=Mining, EM=Electronics Metals, S=Soils, PM=Precious Metals, IP=Industrial Paint (Action lead Paint & Quantify lead Paint), MC=Metal Coatings, PP=Painted Products, P=Plastics, TG=Test All Geo (soil and mining), TA=Test All (consumer products), T=Thin
Reported results are based on measurement results that have been reduced to 2 significant digits by rounding up. For example, a measurement result of 1441 would be reported as 1500.

Table 1-10 - Niton XL3t GOLDD Plus Radiation Profile - Scatter Measurements - mRem/hr

kV	uA	Range	Substrate	Max @ 5cm	Max @ 30 cm	Max @ Trigger
50	40	EM, MC, P, TA, PP (Main) M, S, T (High)	Steel	0.14	<0.01	<0.01
50	40	EM, MC, P, TA, PP (Main) M, S, T (High)	Aluminum	2	<0.01	<0.01
50	40	EM, MC, P, TA, PP (Main) M, S, T (High)	Soil	2	0.04	<0.01
50	40	EM, MC, P, TA, PP (Main) M, S, T (High)	Plastic	10	0.45	6
50	40	GM, PM, M, S, IP (Main)	Steel	0.3	<0.01	<0.01
50	40	GM, PM, M, S, IP (Main)	Aluminum	4	0.01	0.01
50	40	GM, PM, M, S, IP (Main)	Soil	4	0.09	<0.01
20	100	EM, P, TA, M, S, T (Low)	Soil	<0.01	<0.01	<0.01
20	100	EM, P, TA, M, S, T (Low)	Plastic	0.07	<0.01	<0.01
15	133.3	GM (Low)	Steel	<0.01	<0.01	<0.01

Table 1-10 - Niton XL3t GOLDD Plus Radiation Profile - Scatter Measurements - mRem/hr

15	133.3	GM (Low)	Aluminum	<0.01	<0.01	<0.01
8	200	GM, P, M (Light)	<0.01 (no detectable scatter radiation) at any location for steel, aluminum, soil, or plastic sample types			

Notes:
*SAMPLE TYPES (MODES)
GM=General Metals, M=Mining, EM=Electronics Metals, S=Soils, PM=Precious Metals, IP=Industrial Paint (Action lead Paint & Quantify lead Paint), MC=Metal Coatings, PP=Painted Products, P=Plastics, TG=Test All Geo (soil and mining), TA=Test All (consumer products), T=Thin

Table 1-11 - Niton XL3t GOLDD Plus Radiation Profile - Scatter Measurements - μSv/hr

kV	**uA**	**Range**	**Substrate**	**Max @ 5cm**	**Max @ 30 cm**	**Max @ Trigger**
50	40	EM, MC, P, TA, PP (Main) M, S, T (High)	Steel	1.4	<0.1	<0.1
50	40	EM, MC, P, TA, PP (Main) M, S, T (High)	Aluminum	20	<0.1	<0.1
50	40	EM, MC, P, TA, PP (Main) M, S, T (High)	Soil	20	0.4	<0.1
50	40	EM, MC, P, TA, PP (Main) M, S, T (High)	Plastic	100	4.5	60
50	40	GM, PM, M, S, IP (Main)	Steel	3	<0.1	<0.1
50	40	GM, PM, M, S, IP (Main)	Aluminum	40	0.1	0.1

Table 1-11 - Niton XL3t GOLDD Plus Radiation Profile - Scatter Measurements - µSv/hr

50	40	GM, PM, M, S, IP (Main)	Soil	40	0.9	<0.1
20	100	EM, P, TA, M, S, T (Low)	Soil	<0.1	<0.1	<0.1
20	100	EM, P, TA, M, S, T (Low)	Plastic	0.7	<0.1	<0.1
15	133.3	GM (Low)	Steel	<0.1	<0.1	<0.1
15	133.3	GM (Low)	Aluminum	<0.1	<0.1	<0.1
8	200	GM, P, M (Light)	<0.1 (no detectable scatter radiation) at any location for steel, aluminum, soil, or plastic sample types			

Note *SAMPLE TYPES (MODES)

GM=General Metals, M=Mining, EM=Electronics Metals, S=Soils, PM=Precious Metals, IP=Industrial Paint (Action lead Paint & Quantify lead Paint), MC=Metal Coatings, PP=Painted Products, P=Plastics, TG=Test All Geo (soil and mining), TA=Test All (consumer products), T=Thin

Niton XL3p Radiation Profile

Table 1-12 - Niton XL3p Radiation Profile - In Beam Measurements

Distance From Window	Dose Rate (mSv/hr)	Dose Rate (mrem/hr)
5 cm	0.45	45
30 cm	0.03	3.0
100 cm	0.003	0.3

Table 1-13 - Niton XL3p Radiation Profile - Scatter Measurements - mSv/hr

Location	Plastic Substrate	Wood Substrate	Soil Substrate	Aluminum Substrate	Steel Substrate
Max Scatter @ 5cm from Snout (A’)	0.06	0.03	0.018	0.01	0.0042
Max Scatter @ Trigger (B)	0.0038	0.002	0.0015	0.0048	0.0003

Table 1-14 - Niton XL3p Radiation Profile - Scatter Measurements - mRem/hr

Location	Plastic Substrate	Wood Substrate	Soil Substrate	Aluminum Substrate	Steel Substrate
Max Scatter @ 5cm from Snout (A’)	6.0	3.0	1.8	1.0	0.42
Max Scatter @ Trigger (B)	0.38	0.2	0.15	0.48	0.03

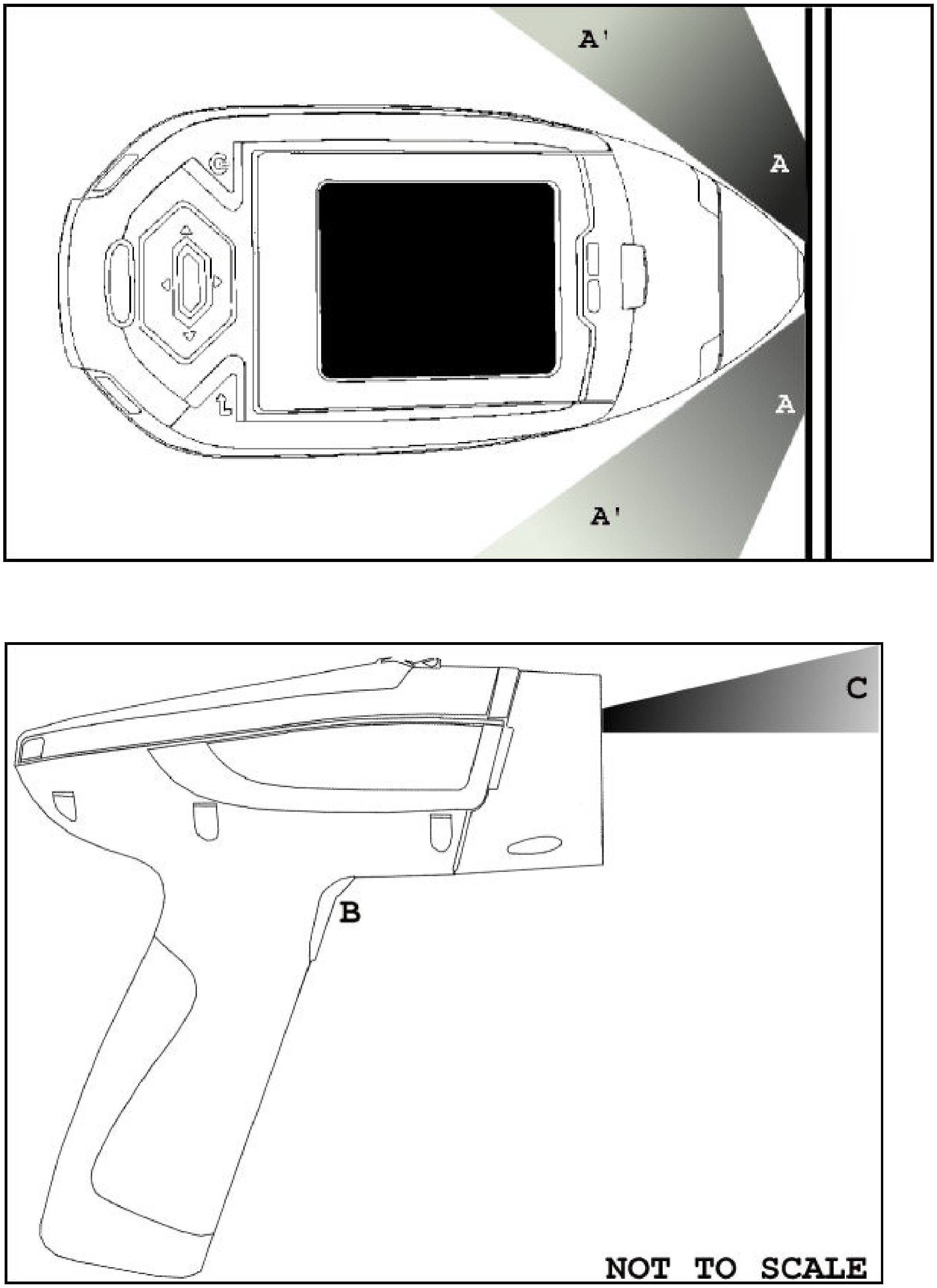


Figure 5. Primary and Secondary Dose Locations (Not to Scale)

Primary Radiation

Niton XL3t and XL3t GOLDD

Primary radiation is radiation that is produced by the analyzer and emitted out through the measurement window. Individuals should never place any part of their body in the primary beam path when the x-ray tube is on. There should always be a sample in contact with the measurement window when the x-ray tube is on. The sample will absorb most of the primary-beam radiation unless it is smaller than the instrument's measurement window or of low density and/or thickness. Caution should be taken when analyzing samples that are small, thin, and/or low in density as they may allow much more of the primary beam to escape. In-beam primary radiation dose rates are listed in Table 1-6, 1-7, 1-8, 1-9, and their location identified relative to the analyzer in Figure 4 as Dose Point C.

Niton XL3p

Primary radiation is radiation that is produced by the analyzer and emitted out through the measurement window. Individuals should never place any part of their body in the primary beam path when the shutter is open. There should always be a sample in contact with the measurement window when the shutter is open. The sample will absorb most of the primary-beam radiation unless it is smaller than the instrument's measurement window or of low density and/or thickness. Caution should be taken when analyzing samples that are small, thin, and/or low in density as they may allow much more of the primary beam to escape. In-beam primary radiation dose rates are listed in Table 1-6, 1-7, 1-8, 1-9, and their location identified relative to the analyzer inFigure 4 as Dose Point C.

Secondary Radiation

Under conditions of normal and proper use, individuals can be exposed to secondary (or "scattered") radiation. Secondary radiation is low-level radiation that emanates from the sample being analyzed as a result of primary beam radiation scattering in the sample or primary beam radiation inducing fluorescent x-rays in the sample. Dose points A, A’ and B in Figure 4 are examples of where you can encounter secondary radiation. The magnitude of this secondary radiation is sample dependent. Higher density samples such as steel will emit the lowest levels as they absorb most primary and secondary radiations. Lower density samples such as aluminum, wood, and especially plastic, will produce higher levels of secondary radiation.

Niton XL3t and XL3t GOLDD

Secondary radiation dose rates are listed in Tables 1-4, 1-5, 1-10, and 1-11, for a few common sample types over a wide range of densities.

Niton XL3p

Secondary radiation dose rates are listed in Tables 1-13 and 1-14 for a few common sample types over a wide range of densities.

Holding Samples

The operator is reminded that one should never hold samples during analysis, doing so will result in higher than necessary exposure to secondary radiation and could expose the operator directly to the much higher primary-beam dose rates.

Deep and Shallow Dose

You will find in the tables that shallow dose rates are listed for some dose points. All dose rates listed in the In-Beam Primary Radiation Tables are deep dose unless they are specifically identified as shallow dose. Deep dose is dose from penetrating radiation that is delivered to both skin and underlying tissues and organs and is the type most commonly referred to when describing external radiation hazards. Occupational deep dose is limited to a maximum of 5 rem (50 mSv) per year in the United States and most countries internationally. Deep dose is measured at 1.0 cm below the skin surface.

Shallow dose is often referred to as "skin dose" because it is a result of low penetrating radiation that only interacts with the skin. Shallow dose is limited to a maximum of 50 rem (500 mSv) per year in the United States and most countries internationally. Shallow dose is listed for primary in-beam dose points only because the low penetrating radiation that causes shallow dose is nearly all absorbed by a sample and does not produce any significant secondary radiation. Shallow dose is measured at a point 0.007 cm below the surface.

Proper and Improper Operation

Storage and Transportation

Storage

Regulations in nearly all locations will require that you store your analyzer locked in a secured area to prevent access, use, and/or removal by unauthorized individuals. Storage requirements will vary by location, particularly with regard to storage at temporary job sites or away from your primary storage location such as hotels and motels and in vehicles. You should contact your local Radiation Control Authority to identify the specific storage requirements in your jurisdiction.

Transportation

Niton XL3t and XL3t GOLDD

There are no X-ray tube specific US Department of Transportation (DOT) or International Air Transport Association (IATA) radiation regulations regarding shipping the Niton XL3t analyzer. It is recommended that you ship the analyzert in its carrying case and an over-pack to protect the sensitive measuring equipment inside the analyzer. Do NOT ship the analyzer with the battery pack connected to the analyzer.

Niton XL3p

For Thermo Fisher Scientific, Niton Analyzers (Niton Analyzers), in the United States, the government agency that has primary authority and regulations which apply to transportation is the Department of Transportation (DOT) (Code of Federal Regulations 49 Parts 100 to 185).

In addition, the EPA, OSHA, and the NRC also have regulations that touch on the transportation of hazardous substances.

The International Air Transport Association (IATA) has recommendations that every major air carrier has incorporated into their policies for the air transport of hazardous substances.

The enforcement of DOTregulations is carried out by the Department of Transportation, the Federal Aviation Administration, the Federal Highway Administration, the Federal Railroad Administration, the Coast Guard, and Customs and Border Protection. State agencies may also enforce state DOT regulations.

The major aspects of the regulations include:

- "training to recognize hazards and how to safely deal with hazardous substances,

- "classification and identification of packages to inform of hazards,

- "protective packaging to safely transport hazardous materials,

- "hazard communication to inform personnel of hazards in a package,

and

- "incident reporting to inform regulators of incidents.

As part of shipping hazardous substances, you should be trained in:

- "a general awareness and a familiarity with the general provisions of the DOT and hazardous materials regulations,

- "training that is function specific and be applicable to the daily work performed,

- "the recognition and identification of hazardous substances,

• "the specific requirements for functions performed,

• "security measures to keep a package secure.

• "safety issues as related to safe handling and hazard communication.

• "knowledge of emergency response information, self-protective procedures, and accident prevention procedures.

Employers are responsible for providing the proper training (every three years for DOT) to employees, testing employee knowledge, and record keeping.

The DOT Hazardous Material Regulations set the criteria for determining the hazard class and the proper shipping name for hazardous materials. The Hazard Classes as follows:

Class

1. Explosives

2. Gases

3. Flammable and Combustible Liquids

4. Flammable solids, combustible materials, and dangerous when wet materials

5. Oxidizers and organic peroxides

6. Toxic materials and infectious substances

7. Radioactive Materials

8. Corrosive Materials

9. Miscellaneous dangerous goods

Niton analyzers with radioisotopes are Hazard Class 7. The Hazard Class of the material being transported tells you which Parts of the regulations are required.

A UN number is assigned to each type of hazardous material. It is the letters "UN" followed by a four digit numerical code, which allows emergency responders to identify the chemical being shipped. The UN number for Niton analyzers is "UN 2911".

There is also an official name designation for Niton Analyzers called the Proper Shipping Name. The proper Shipping Name for the Niton Analyzers is "Radioactive material, excepted package-instruments and articles".

A reportable quantity (RQ) is listed in DOT regulations for each hazardous material (e.g., each radioactive isotope). If you are shipping more than the reportable quantity in a package, that package must be marked clear and legibly with the letters "RQ". The Niton XL3p analyzer contains a 30 mCi (1.11 GBq) Am-241 source and is therefore considered a reportable quantity requiring the RQ marking when being transported.

Also, the quantity of Am-241 that is used in a Model XL3p Analyzer requires a special form certification for transport as an excepted package. This Special Form certificate must accompany the instrument during shipment. Thermo Fisher Scientific will provide the Special Form certificate for any analyzer with Am-241."

The type of protective packaging used is dependent on the nature of the material to be packaged. All packaging must be designed to prevent a release of hazardous material during normal transportation or storage of the material. The classification of package used for Niton Analyzers is designated as an "Excepted Package". Always ship the analyzer in its original plastic case to ensure that the packaging used meets the regulatory requirements for an Excepted Package.

Shipments of radioactive materials must have proper Labeling and Marking.

Niton analyzers have a Marking requirement (i.e., UN number and RQ if applicable), but not a Labeling requirement (i.e., diamond shaped hazmat labels), and vehicles transporting these analyzers are not required to have "Placards".

When reading the DOT regulations, you will find the following information useful.

Thermo Fisher Scientific, Niton Analyzers are shipped:

- "Under the proper shipping name "Radioactive material, excepted package-instruments or articles" in accordance with 49 CFR 173.424,

- "with the radiation level at 10 cm from the unpacked instrument surface less than 10 mrem/hr (0.1 mSv/hr) "
Note A Niton Analyzer in proper condition will be less than 0.5 mrem/hr (0.005 mSv/hr) at 10 cm,

- "with the radiation level at the package surface less than 0.5 mrem/hr (0.005 mSv/hr)"
Note A Niton Analyzer in proper condition will be less than 0.05 mrem/hr (0.0005 mSv/hr) at the surface of the case,

- "with all radioactive sources as "solid", "sealed sources"

- "Am-241 listed in A1 column of 173.435 (270 Ci) (Special Form capsule)

- "with the package design meeting the requirements of 173.410

- "package marked with "UN2911"

- "with the Am-241 source, the package is marked with "RQ"

- "meeting the 173.424 criteria for labeling and marking requirements

For any shipment: Include in the package a current copy of the instrument Leak Test.

Include a list of emergency numbers in the package.

For Am-241, include the Special Form Certificate in the package.

Always ship in supplied plastic case, with the case secured against accidental opening. Always ship with the battery disconnected.

When shipping by air: Ship with the proper IATA marking (See IATA Dangerous Goods regulations Figure 10.7.8.A), UN 2911, and proper shipping name.

"RQ" marking and "dangerous goods declaration" are required.

When shipping by Ground: Ship under proper marking "UN2911"

"RQ" marking and shipping papers are required (Note: a dangerous goods declaration form can be used to meet the shipping paper requirement).

A "Dangerous Goods Declaration" can be obtained by the air carrier that you will be using. Instructions can also be obtained from the same source.

Carefully follow the directions given by the air carrier. Several typed copies will be required.

Shipping papers contain all of the same information as a Dangerous Goods Declaration, but do not have a specified format for that information.

At a minimum, a properly prepared shipping paper clearly identifies the hazardous substance by its proper shipping name, hazard class and division, UN identification number, packing group (if applicable), and total quantity. It also has consigner information, consignee information, and a shipper's declaration that the package is in compliance with the DOT regulations.

The elements of hazard warning information are communicated through shipping documents, packaging markings, and written emergency response information.

The DOT & FAA Hazardous Materials Regulations require the carrier to report all incidents involving hazardous materials.

An "incident" involves the unintended release of hazardous materials (Am-241), suspected radioactive contamination, if the general public is evacuated for an hour or more, or the flight pattern or routine of an aircraft is altered.

For any "incident", contact the Company Radiation Safety Officer or Responsible Party and the state radiation control program.

Any "incident" needs to reported to the:

Hazardous Materials Information Center

1-800-467-4922

Mon-Fri 9AM-5PM Eastern

Leak Tests (Niton XL3p Only)

The Niton XL3p Series analyzer contains a radioactive source that must be periodically leak tested. The purpose of leak testing is to verify the integrity of the source encapsulation. A leak test sample is obtained by wiping exterior surfaces of the device with moderate pressure using a cotton swab, filter paper, or whichever wiping media is supplied by the analysis laboratory. Leak test samples are then typically analyzed at a laboratory, although some device users have the equipment and licensed authority to perform this analysis

Unless specified otherwise by your local authority or radioactive material license, the gauge must be leak tested at intervals not to exceed 6 months. In the US, leak test samples may be acquired by any end-user, however the analysis of the sample must be performed by an organization licensed to do so. If you are using a vendor to perform the laboratory analysis of the leak test sample, they will send you a leak test kit which comes with complete instructions for performing the test. These vendors will also typically send you a reminder when it is time to perform the next leak test on your instrument. Please follow the test kit instructions carefully, and promptly mail the test samples to the laboratory. They will send you a leak test certificate soon after. Keep one copy of the leak test certificate with the device at all times (i.e., in the case) and another copy safely on file.

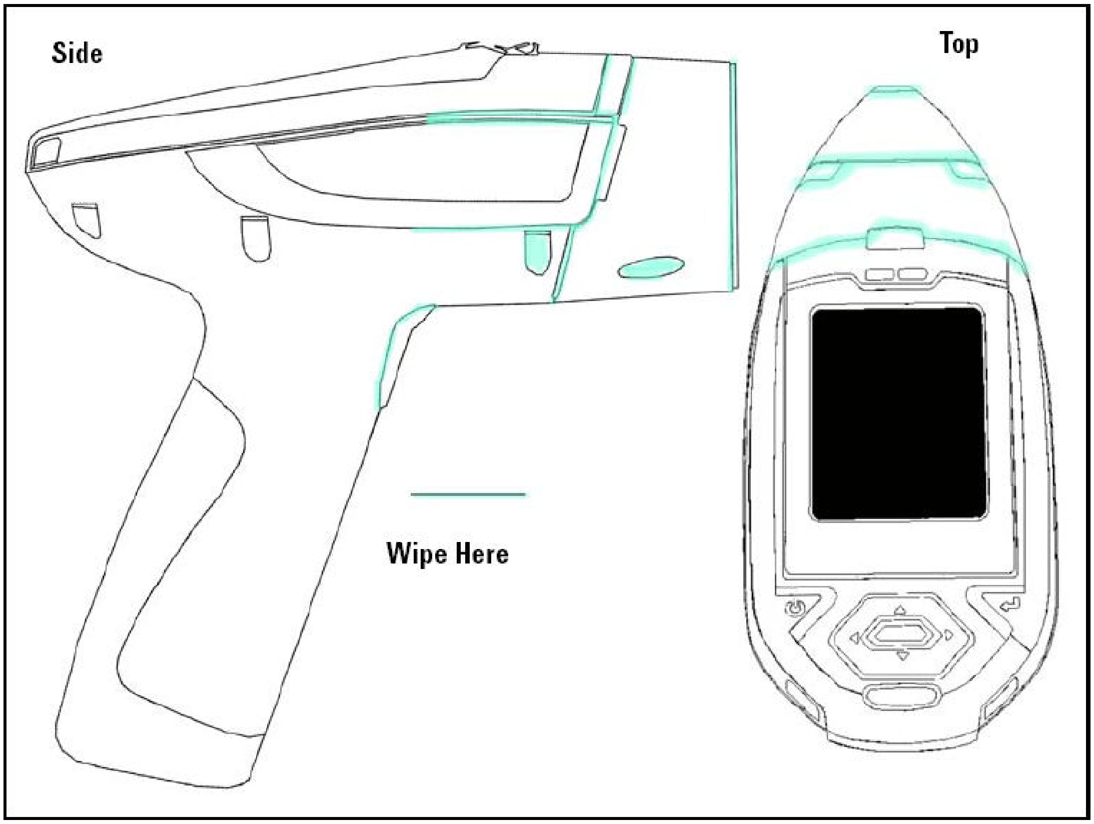


Figure 6. Wipe Test Locations

CAUTION REMOVE THE BATTERY WHILE PERFORMING A WIPE TEST TO BE SURE THAT THE SHUTTERS ARE NOT OPEN DURING THIS PROCEDURE!

Lost or Stolen Instrument

THIS PAGE CONTAINS EMERGENCY CONTACT INFORMATION THAT SHOULD BE AVAILABLE TO THE OPERATOR AT ALL TIMES.

If the Niton XL3t analyzer is lost or stolen, notify your Radiation Safety Officer (RSO) or the equivalent responsible individual at your company or institution immediately. Your company's RSO, as well as other important emergency contacts, are listed below. Your company RSO may need to notify the x-ray tube regulatory authority and the local police. It is also recommended that a notification is made to Thermo Fisher Scientific.

Damaged Instrument

Minor Damage

If the instrument is intact but there is indication of an unsafe condition such as a cracked case, a shutter mechanism failure, or the lights remain flashing after a measurement is terminated, follow these steps:

1. Stop using the instrument

2. Remove the battery. The x-ray tube can not produce radiation when the battery is disconnected. The instrument is now safe to handle.

3. Place the instrument securely in the holster.

4. Place the instrument in the carrying case that came with the instrument.

5. Notify your Radiation Safety Officer (RSO) or the equivalent responsible individual at your company or institution immediately.

6. You or your RSO should call Thermo Fisher Scientific at one of their contact numbers listed below for additional instructions and guidance.

Major Damage

If the instrument is severely damaged:

1. Perform the same steps as described above for minor damage. There will be no radiation hazard as long as the battery is removed from the instrument.

2. Place all components in a plastic bag and contact Thermo Fisher Scientific.

Emergency Response Information

Please Complete the Following Emergency Response Information and Keep with the Analyzer at All Times

NITON ANALYZER EMERGENCY CONTACT INFORMATION

The Company RSO is:____

RSO Telephone Number:______________________________________

Regulatory Agency Emergency Number:_________________________

Local Fire Department:____

Local or State Police Department:______________________________

Thermo Fisher Scientific's Niton Analyzer Contact Numbers

Main Number (USA): (800) 875-1578

Additional Radiation Emergency #'s: (978) 790-8269 or (617) 901-3125

Outside the USA - Local Niton Service Center:____________________

Europe

Niton Analyzers Europe

Munich, Germany

Phone: +49 89 3681 380

Fax: +49 89 3681 3830

Email: niton.eur@thermofisher.com

Asia

Niton Analyzers Asia

Hong Kong

Phone: +852 2869-6669

Fax: +852 2869-6665

Email: niton.asia@thermofisher.com

Registration and Licensing

As a user of a Niton XL3 analyzer, you may be required to register or obtain a license with your local radiation control authority. In the US, if you intend to do work with your analyzer in states other than your own, you may be required to register there as well. See the Safety and Compliance Web Hub for much more information.

Regarding Safety Devices for the Open Beam Configuration:

In the US, you may be required to file for an exemption, "variance letter", with your state if there is a requirement for a safety device that would prevent entry of an extremity into the primary beam. If you need assistance with the exemption letter, you may contact the radiation safety group.

Registration and Licensing FAQ

Startup Procedure

Unpacking and Preparing Your Niton XRF Analyzer

WARNING The nose of the analyzer should not be touched during sample testing and calibration. If an ESD event occurs during measurement, the instrument may terminate the testing in progress and automatically reset to Log On screen. Any test data collected prior to reset will be lost and the testing may have to be repeated.

Figure 7. Populated Analyzer Case

- Inspect the shipping carton for signs of damage such as crushed or water damaged packaging. Immediately notify the shipping company and Thermo Fisher Scientific, in the United States, toll free, at (800) 875-1578, or outside the United States, at +1-978-670-7460, if there is any visible damage to the shipping container or any of its contents.

- Open the packing carton. If your analyzer is not packed in its carrying case, please call Thermo Fisher Scientific immediately, in the United States, toll free, at (800) 875-1578, or outside the United States, at +1-978-670-7460.

- Verify the contents of the shipping container against the enclosed packing list. If there are any discrepancies between the actual contents of the shipping container and the enclosed packing list, please notify Thermo Fisher Scientific immediately, in the United States, toll free, at (800) 875-1578, or outside the United States, at +1-978-670-7460.

- Open the carrying case and visually inspect the analyzer for damage before removing it from the case. Contact the freight carrier and Thermo Fisher Scientific if you find any damage to the case or its contents.

- Save the shipping carton and all packing materials. Store them in a safe, dry area for reuse the next time that you ship the analyzer.

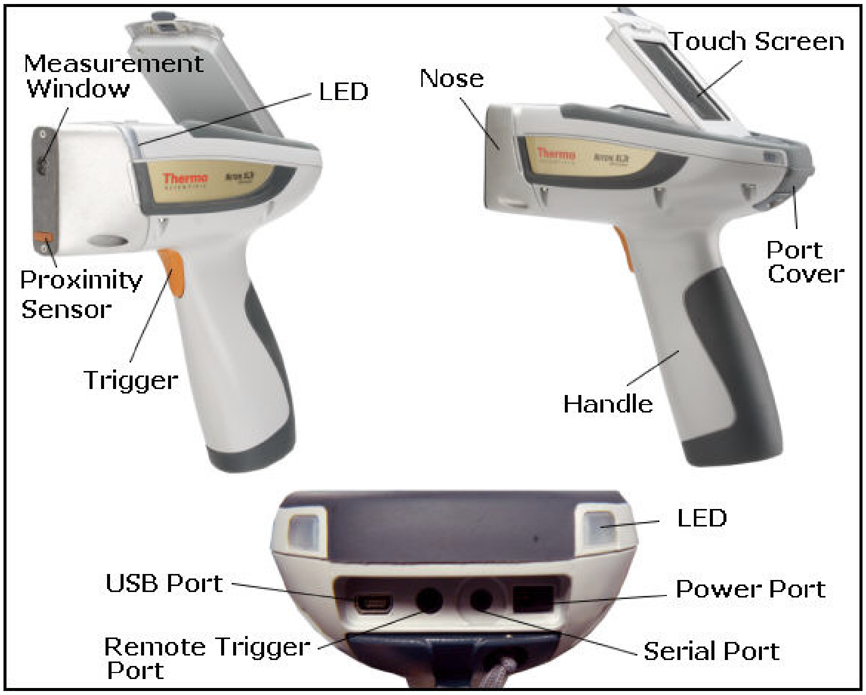


Figure 8. **The Thermo Scientific Niton XL3 Analyzer Overview**

Battery Installation and Charging

Installing or Replacing The Battery Pack

1. Slide back the catch on the bottom of your analyzer's pistol grip and drop the battery out into your hand.
2. Place the old battery aside and slide the new battery up into the cavity in the bottom of the pistol grip. The battery is keyed, and will only insert fully one way.

Figure 9. Rear and Side of Battery Pack Showing Key

CAUTION Do not force the battery into the cavity!

3. Press in until the latch resets.

Recharging The Battery Pack

Fully recharging a battery pack takes approximately 2 hours.

1. Remove the battery pack from the analyzer.
2. Place the battery pack upside down into the charger. The battery pack is keyed, and will only fit into the charger fully one way. If your battery pack is resting on the back of the back of the charger rather than sliding all the way to the bottom, remove the battery pack, turn it around, and re-insert it into the charger.

CAUTION Do not force the battery into the charger!

3. The red light is on when the charger is plugged in. This is the power indicator light.

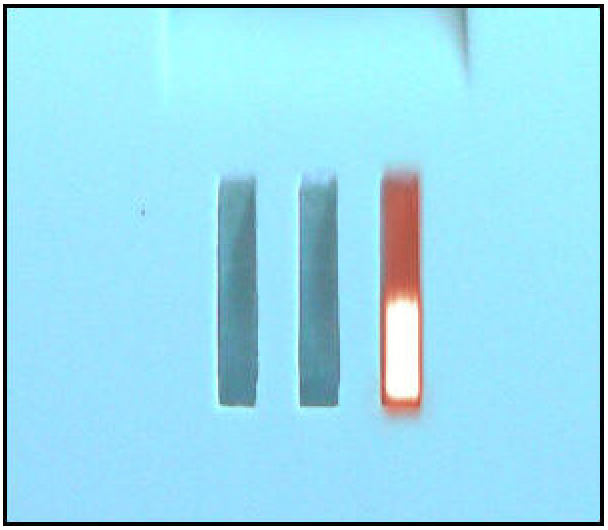


Figure 10. Power On

4. The yellow light indicates that the battery pack is currently being charged.

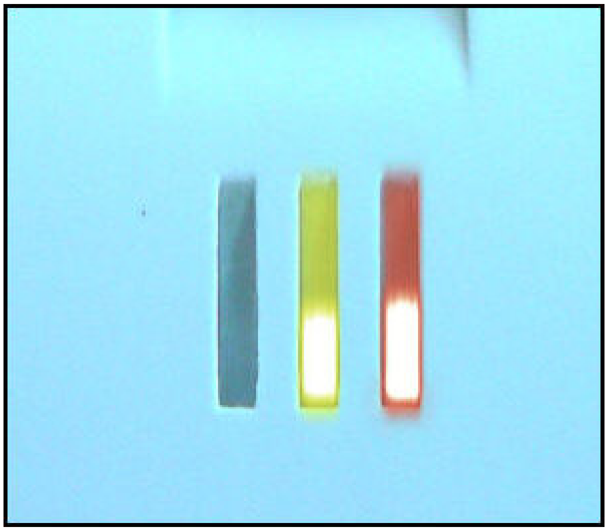


Figure 11. Charging

5. The green light indicates that the battery pack has finished charging and is ready for use.

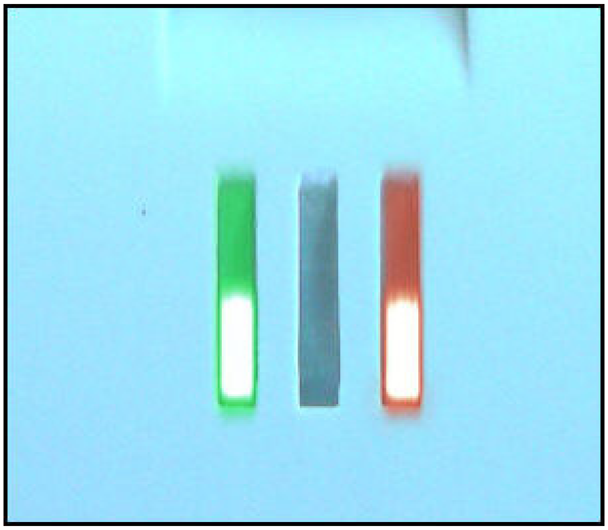


Figure 12. Charge Completed

Note If there is a fully seated battery pack in the charger and only the red light is on, there is a fault with the battery pack or charger.

Figure 13. Battery Pack in the Charger

CAUTION Do not store battery packs or charger in direct sunlight.

CAUTION Do not let the battery pack recharge for excessive periods of time.

The Control Panel

The control panel is located on the analyzer's top housing, directly below the Touch Screen. The control panel consists of a 4 Way Touch Pad, an Interlock Button, and two Control Buttons, one on each side. Using either the control panel or the touch screen you may navigate through all of the analyzer's screens and menus. You can control the movement of the screen cursor by pressing the 4 Way Touch Pad in one of four directions to highlight each of the menu options. The Clear/Enter button to the right of the 4 Way Touch Pad is used to select highlighted menu options. The On/Off/Escape Button both controls the power to the analyzer and serves as an "escape" button. When the On/Off/Escape Button is pushed and immediately released, it functions as an "escape", and brings you back to the Main Menu from the current screen in the menu system.

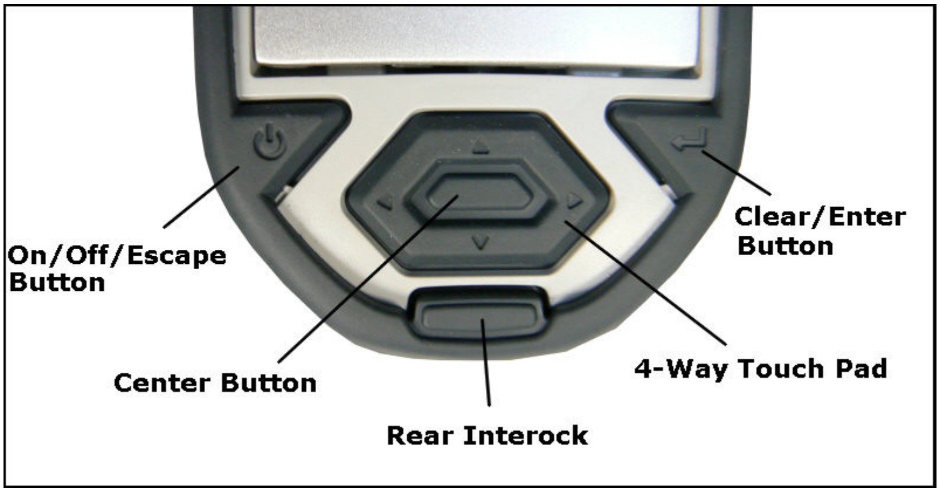


Figure 14. The Control Panel

To turn your analyzer on, push and hold the On/Off/Escape Button until the Touch Screen comes on. To turn off your analyzer, push the On/Off/Escape Button and hold it down until the Touch Screen shuts off.
CAUTION If for any reason your analyzer does not shut off normally, removing the battery will shut it down safely.

You also have the option of operating the analyzer, including navigating the menu system, by using the built in Touch Screen. To select a menu option, tap on the icon once. The touch screen icons have the same functionality as the 4 Way Touch Pad, the On/Off/Escape Button, and the Enter Button. This User's Guide will refer to the process of choosing a course of action by selecting an icon from a menu, either using the Touch Screen or using the control panel buttons, as “selecting.”

Selecting the Return Icon works everywhere throughout the User Interface to bring you back to the previous menu from the current menu in the menu system. Use the On/off/escape button to return to the Main Menu.

The LCD Touch Screen

The LCD Touch Screen on your Niton XL3 Analyzer is designed to swing up and down to different angles for ease in viewing and interacting with your analyzer. The LCD Touch Screen is connected to your analyzer along the base of the screen, right above the Control panel. The screen is not designed to separate from the analyzer, but can be adjusted to any arbitrary angle between zero degrees - that is, flush with the analyzer - and 85 degrees, which is almost perpendicular. The LCD Touch Screen will stay at any given angle between these extremes until moved to a different angle. When in closed position, the screen is secured by a catch at the top center of the screen housing.

Figure 15. Niton XL3 Analyzer Showing LCD Screen Tilted.

To raise the LCD Touch Screen, disengage the catch at the top-center of the LCD Touch Screen housing and gently pull the screen towards you until it is at the best angle for your use.

To close the LCD Touch Screen, gently push away from you along the top edge of the screen housing. The screen will swing down until the catch solidly engages with an audible click.
Note The LCD Touch Screen cannot be removed from your Niton XL3 analyzer. Removing or attempting to remove the LCD Touch Screen will damage your analyzer and void your warranty.
Note Always close your LCD Touch Screen before storing or transporting your Niton XL3 analyzer.

Startup Procedure

To turn on the analyzer, depress the On/off/escape button on the control panel until the Touch Screen comes on.

On startup, the screen will show by a Start Screen which will automatically count down from 4 to 0 in increments of one second.

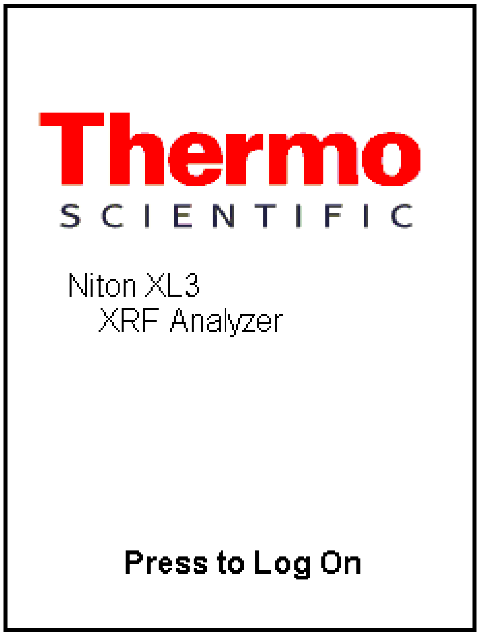
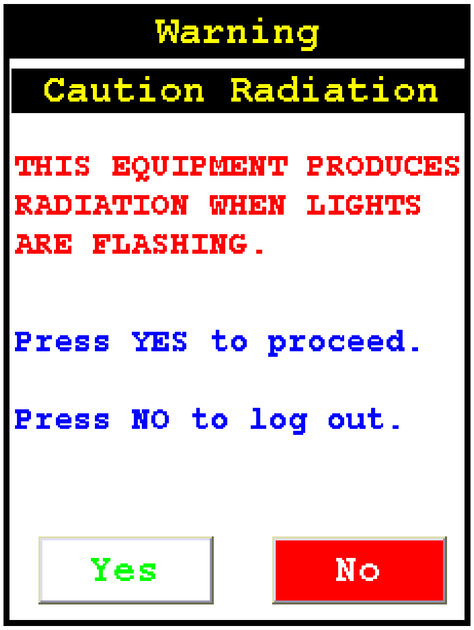


Figure 16. Logon Screen

When the startup is complete, the Start Screen will be replaced by the Logon Screen (see Figure 15). Tap anywhere on this screen to continue.

The Logon Screen will be replaced by a Warning Screen, see Figure 16, advising you that this analyzer produces radiation when the lights are flashing. You must acknowledge this warning by selecting the Yes button before logging on. Selecting the No button will return you to the Logon Screen.



Warning

Caution Radiation

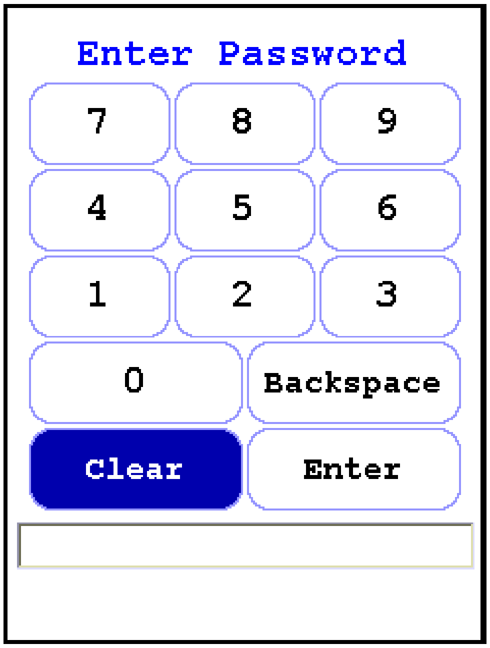
THIS EQUIPMENT PRODUCES RADIATION WHEN LIGHTS ARE FLASHING.

Press YES to proceed.

Press NO to log out.

Figure 17. Warning Screen

After selecting the Yes button, the Virtual Numeric Keypad becomes available for you to log onto the analyzer.



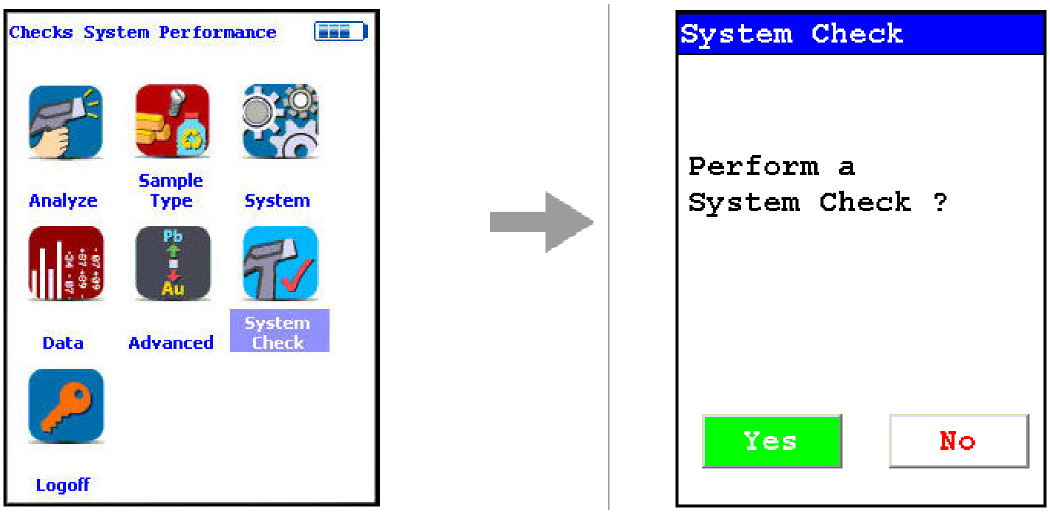
Enter Password

Figure 18. Virtual Numeric Keypad

Select your 4 digit security code, followed by the Enter button. The default password is 1-2-3-4, followed by the Enter button. If you enter an incorrect number, you can use the Backspace button to backspace over it, or use the Clear button to clear the field. After you have completed the log on procedure, the word "USER" will appear on the bottom of the screen, then the Main Menu will appear. Note that security codes are editable. Please see Passwords and User Privileges for instructions on creating user-definable passwords.

Check the date/time. The time should be set correctly for accurate and verifiable record keeping (Setting the Date and Time).
Note Your analyzer can be stored and operated safely in temperatures from minus 5° C (23° F) to 50° C (122° F). You will not be able to take a measurement if the analyzer overheats. If it is hot to the touch, you should allow it to cool before testing.

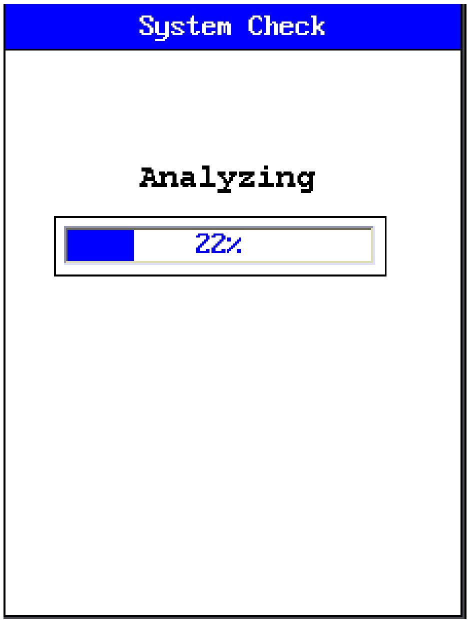
Performing a System Check



Perform a System Check ?

Figure 19. System Check Menu Path

Select the System Check Icon on the Main Menu to perform a system check. Thermo Scientific recommends that you perform a system check once every working day, as part of your normal startup procedure, after allowing a minute or so for the analyzer to warm up.



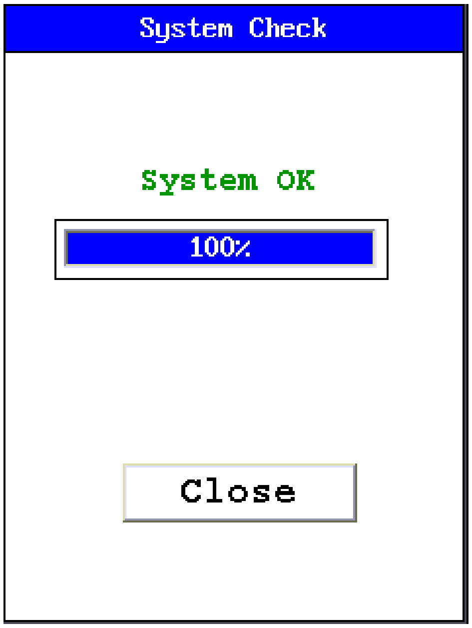
System Check

Analyzing

22%

Figure 20. System Check in Progress

While performing the system check, your screen will show a progress bar indicating the progress of the check. When it is done, the screen will show a 100% completion as in Figure 20 below.



System Check

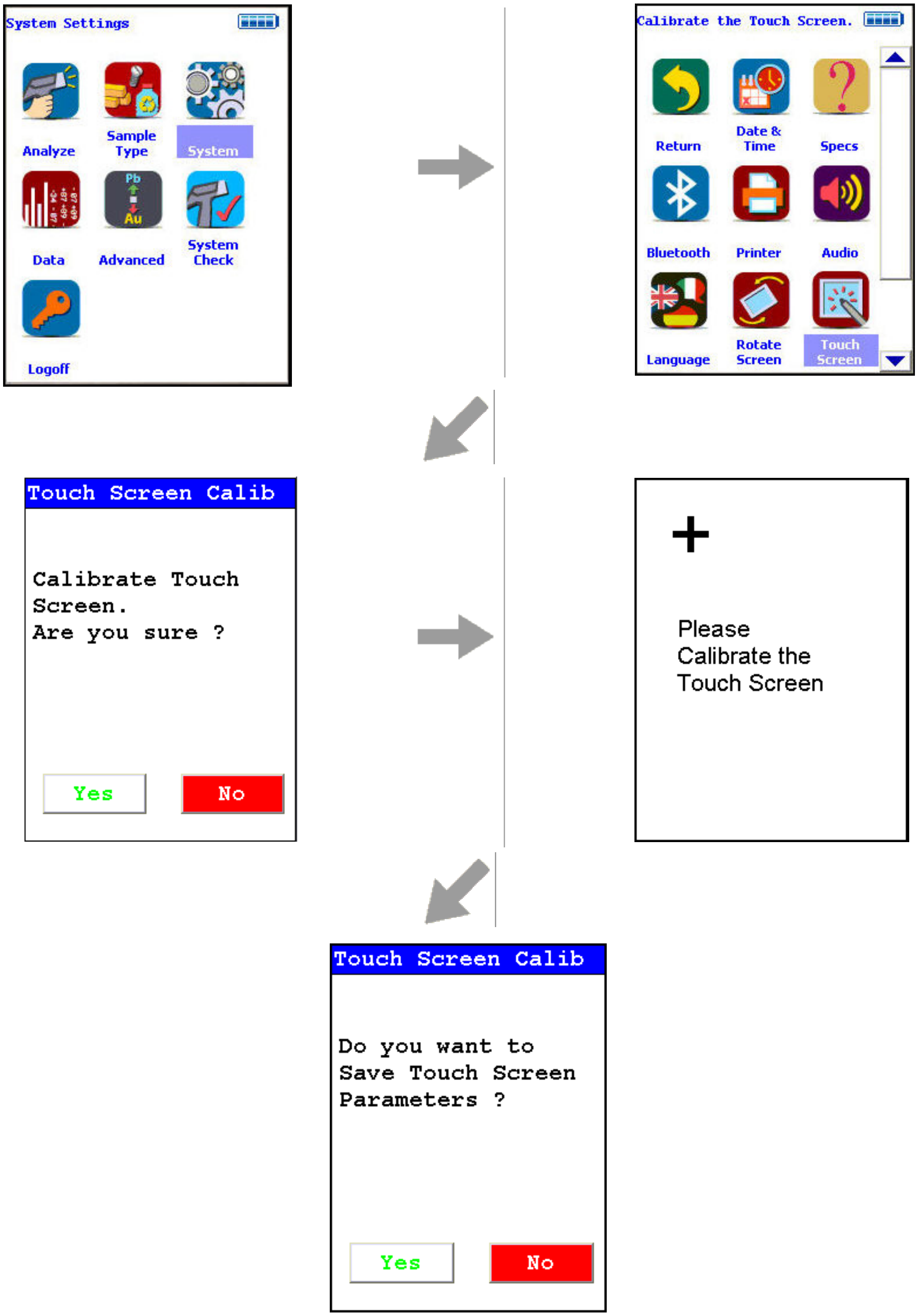
System OK

100%

Figure 21. System Check Completed

If you see any result other than "System OK", perform another system check. If the result is still not "System OK", please notify Thermo Scientific Service at 800-875-1578.

Calibrating the Touch Screen



Calibrate Touch Screen.
Are you sure ?

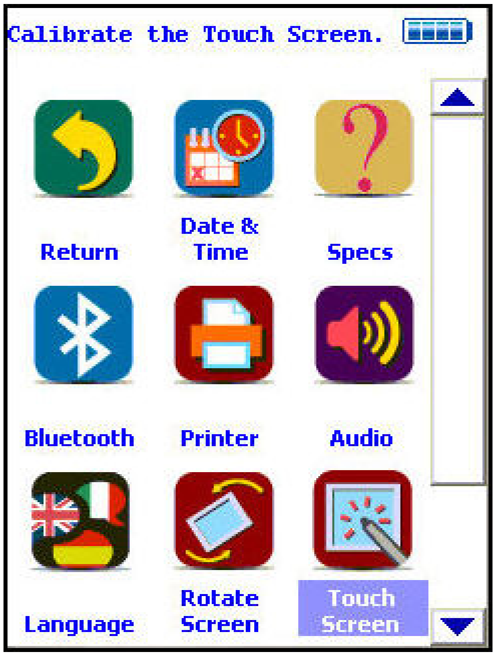
Please Calibrate the Touch Screen

Do you want to Save Touch Screen Parameters ?

Figure 22. Touch Screen Calibration Menu Path

Select the Calibrate Touch Screen icon to re-calibrate the analyzer's touch screen display. This procedure establishes the display boundaries for the touch screen interface.

1. Select the Touch Screen icon.
2. The display will show a message asking you to confirm whether or not you want to calibrate your Touch Screen. Select the Yes button.
3. The display will show the message: "Calibrate Touch Screen". There will be a small cross in the upper left-hand corner of the display.
4. Tap on this cross with the stylus, and the cross will disappear and reappear in the upper right-hand corner of the screen.
5. Tap on the cross again, and it will reappear in the lower right-hand corner of the screen.
6. Tap on the cross again and it will reappear in the lower left-hand corner of the screen.
7. Tap on the cross once more, and you will be presented with a Confirmation Screen.
8. Select the Yes Button to confirm that the parameters are good. Select the No Button to start the process again.
9. Once you have confirmed the parameters, the System Menu will be displayed. The screen is now calibrated.



Calibrate the Touch Screen.

Figure 23. The System Menu

Calibrating the Touch Screen Without Using the Touch Screen

If the touch screen becomes misaligned, you may need to use the buttons below the screen to complete this process. There are 2 single buttons and a 4 way switch located to the rear of the display screen. The button at the left is the On/Off/Escape button. The button to the right is the Enter button and the center keypad is a 4 Way Touch Pad.

The 4 Way Touch Pad has 4 positions, Up, Down, Left and Right. The Interlock Button is not used in this procedure.

Figure 24. The Control Buttons for the Niton XL3

1. Please, turn on your XRF analyzer using the On/Off button.

Note From this point please DO NOT touch the touch screen.

2. Press the Enter button. You are now at the Radiation Warning screen.
3. Using the 4 way touch pad on the on the cover of the instrument, move the cursor around the screen by pressing the appropriate up, down, left, or right button. Please move the cursor such that the "Yes" option is highlighted in green.
4. Press the Enter button. You are now at the Enter Password Screen.
5. Move the cursor on the Virtual Numeric Keypad using the 4-way Touch Pad to the appropriate first number in your password. Press the Enter button on the right - it has the arrow/enter key symbol on it. The first number of your password should appear in the lower left of the screen.
6. Repeat step 5 until you have entered the entire password. Then move the cursor to the Enter Key on the Virtual Numeric Keypad and press the Enter button to enter it.

7. You will now be at the Main Menu.
8. Again using the 4 Way Touch Pad, move the cursor to highlight the System icon, and press the Enter button to select it.
9. You will now be at the System Menu.
10. From the System Menu screen, move the cursor to highlight the Touch Screen icon and press the Enter button.
11. Now move the cursor to highlight the Yes Button and press the Enter button.
12. You are now at the Touch Screen Calibration screen.

Note You must now use the touch screen for the balance of this procedure

13. In the upper left hand corner you will see a crosshair - like this: “+”. Using the stylus or a pen, tap the center of the “+”.
14. Repeat this for each “+” sign that appears, there should be one for each of the 4 corners.
15. Select the Yes Button to confirm that the parameters are good. Select the No Button to start the process again.
16. Once you have confirmed the parameters, the System Menu will be displayed. The screen is now calibrated.

Your touch screen should work properly after this and you may use normally it from this point forward. If it does not, please repeat the process.

The Data Ports

Figure 25. The Data Ports

USB Port

The USB Port is a communications and control port, for uploading and downloading data, configuration files, and software to the analyzer.

Remote Trigger Port

The Remote Trigger Port controls the analyzer's trigger function, for use with accessories and test stands.

Serial Port

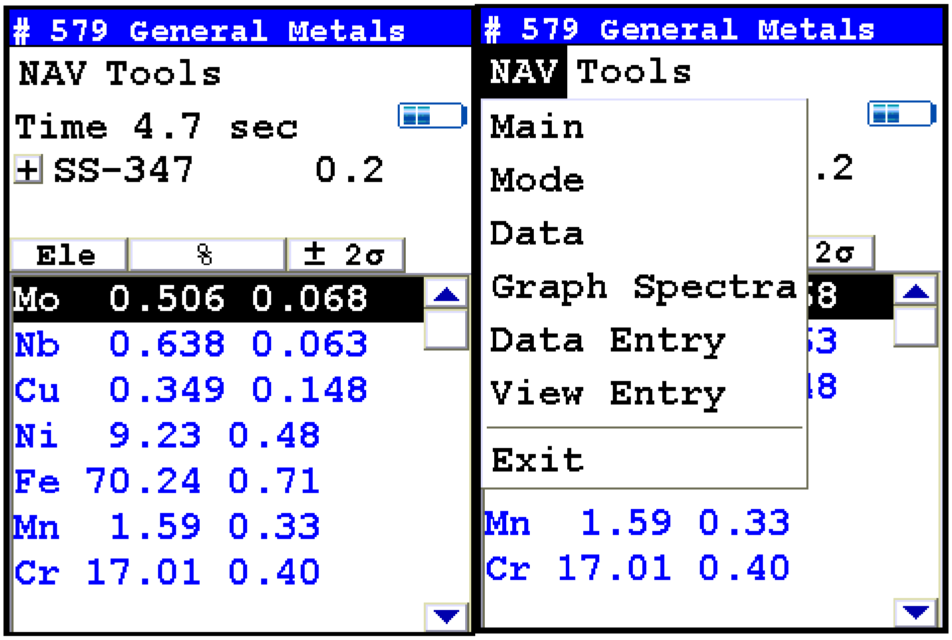
The Serial Port is a communications and control port, for uploading and downloading data, configuration files, and software to the analyzer.

Power Port

The Power Port is used to run the analyzer under external power.

Using the Navigation (NAV) Menu

The NAV Menu enables you to move between various menus and screens directly, without going through the intervening screens. Select a destination from the drop down menu and you will be brought directly to that menu or screen.



```
# 579 General Metals
NAV Tools
Time 4.7 sec
+ SS-347        0.2
Ele    %    ± 2σ
Mo   0.506 0.068
Nb   0.638 0.063
Cu   0.349 0.148
Ni   9.23 0.48
Fe  70.24 0.71
Mn   1.59 0.33
Cr  17.01 0.40
```

Figure 26. Accessing the NAV Menu

Using the Tools Menu

The Tools Menu enables you to perform common data-related tasks such as printing and averaging readings. Select a task from the menu to initiate that task. The options available can vary depending on the selected mode.

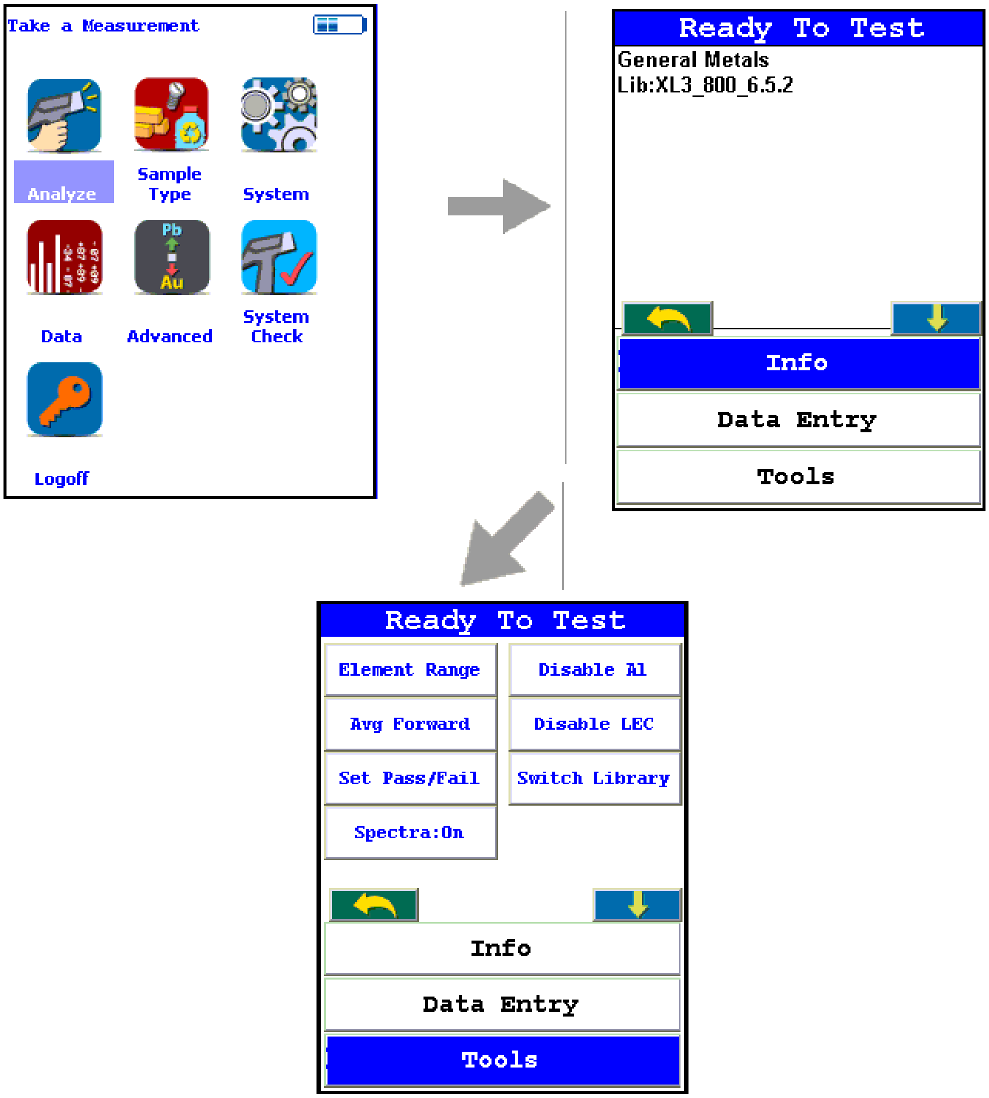
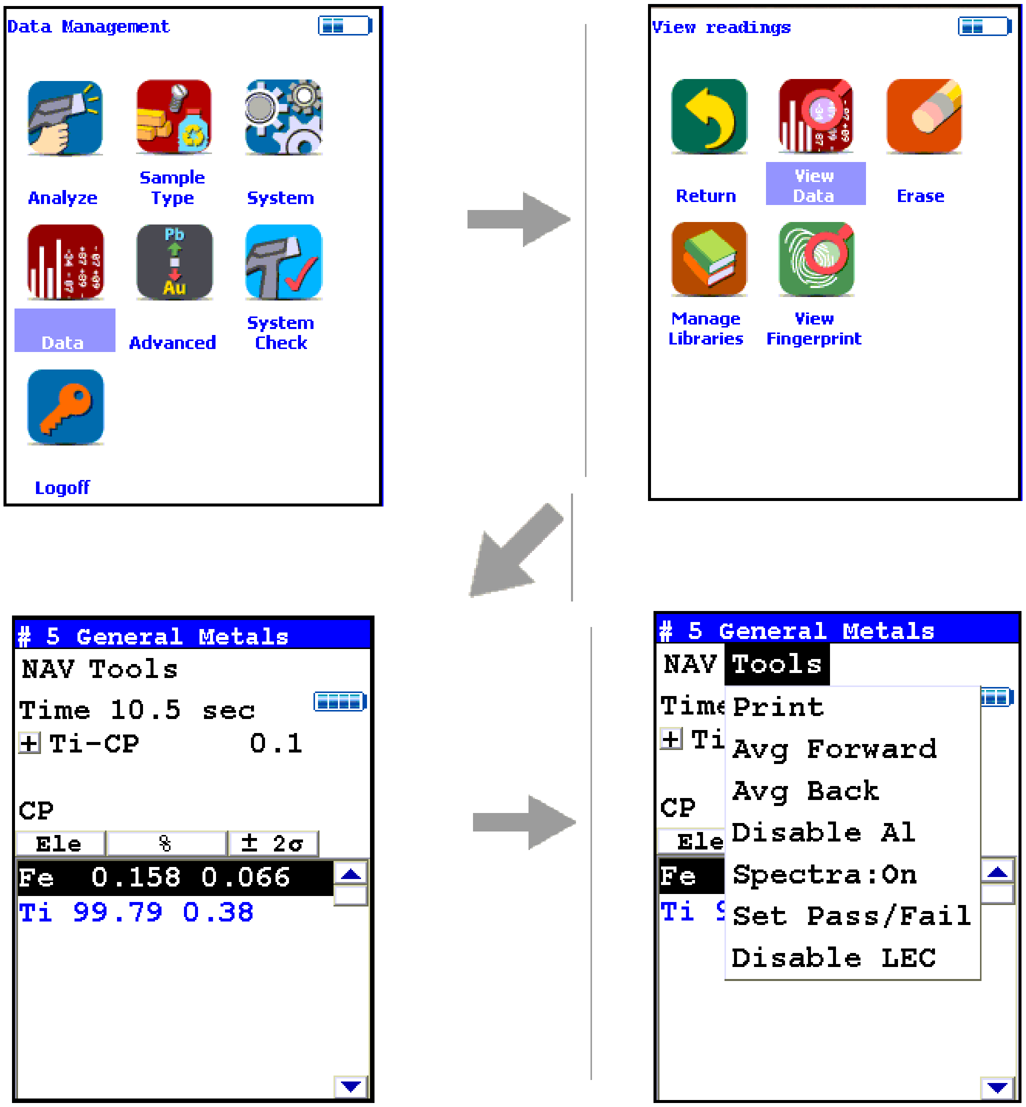


Figure 27. The Tools Menu Path (Main)

The Tools Menu can be accessed in two ways, and the options are different depending on the way you access it. The main Tools Menu is accessed by selecting the Analyze Icon from the Main Menu, then selecting the Tools button from the slide down window on the Ready to Analyze screen.



```
# 5 General Metals
NAV Tools
Time 10.5 sec
+ Ti-CP        0.1

CP
 Ele     %    ± 2σ
Fe   0.158 0.066
Ti  99.79  0.38
```

Figure 28. The Tools Menu Path (Alt)

The alternate Tools Menu can be accessed by selecting the Data Icon from the Main Menu, selecting the View Data Icon from the Data Menu, then selecting the drop down Tools Menu from the View Data screen. It can also be accessed from the Analysis Screen, after the reading is saved.

Toggling Buttons and Options

Most of the buttons and options on the Tools Menu are toggles. These buttons and options on the Tools Menu change to their opposites when selected. To stop averaging, for example, select Stop Avg Fwd or Stop Avg Back from the Tools Menu as appropriate.

Tools Menu Options (Mode Dependent)

Averaging Results

You will find the two types of result averaging here: Avg Forward

Spectra: On/Spectra: Off

You will find how to use Live Spectra here: Spectrum:On/Spectrum:Off

Alloy Pass-Fail

You will find how to use Alloy Pass/Fail here: Set Pass/Faill

Editing and Switching Alloy Libraries

You will find how to use Alloy Libraries here: Switch Library (Main)

Enable/Disable Al

You will find how to use the Enable/Diable Al option here: Enable/Disable Al

Coatings Method

You will find how to use Coatings Method here: Coatings Method

Thickness Correction

You will find how to use Thickness Correction here: Thickness Correction

Enable Paint

You will find how to use Enable Paint here: Enable/Disable Paint

Action Level

You will find how to use Action Level here: Action Level

Print Data

You will find how to use Print Data here: Print Data

Element Ranges

You will find how to use Element Ranges here: Adjusting the Element Range

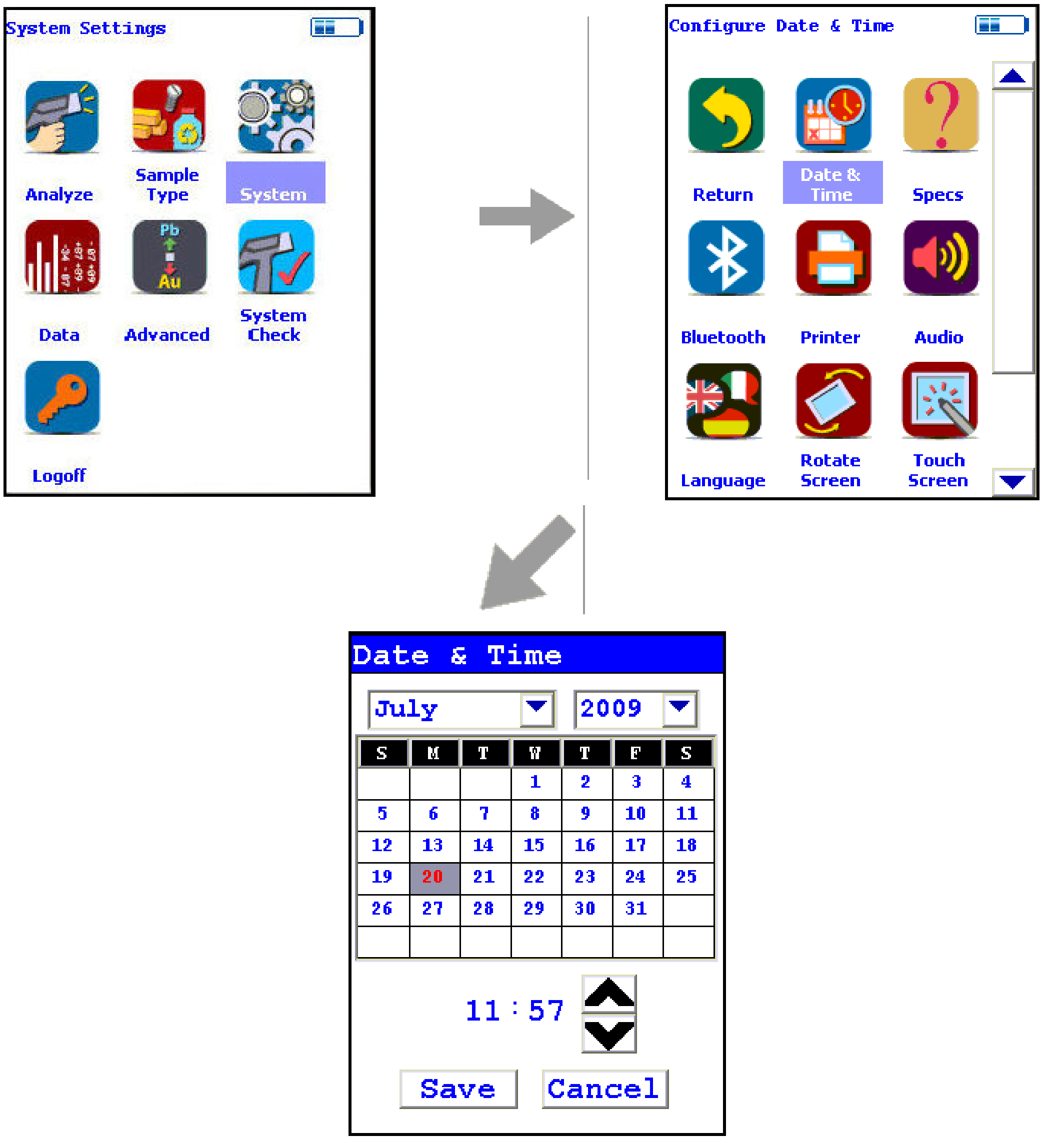
Toggle He

You will find how to use Toggle He here: Enable/Disable Al

Toggle Spot

You will find how to use Small Spot here: Calibrating the Spot

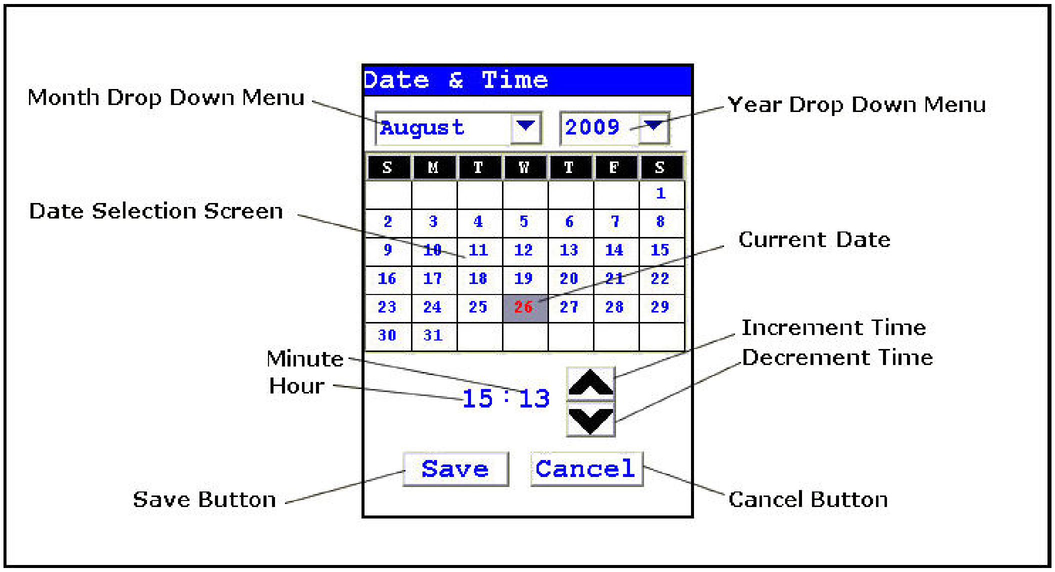
Setting the Date and Time



S	M	T	W	T	F	S
			1	2	3	4
5	6	7	8	9	10	11
12	13	14	15	16	17	18
19	20	21	22	23	24	25
26	27	28	29	30	31	

Figure 29. The Date and Time Menu Path

From the System Menu, select the Date & Time icon to set the date and time as needed for different time zones, daylight savings time, or any other reason. The date and time are factory preset prior to shipping. The clock is a 24 hour clock, so add 12 to PM hours - i.e. 1:13 PM would be 13:13.



August 2009

S	M	T	W	T	F	S
						1
2	3	4	5	6	7	8
9	10	11	12	13	14	15
16	17	18	19	20	21	22
23	24	25	26	27	28	29
30	31					

15 : 13

Figure 30. Setting the Date & Time

When the Date & Time icon is selected, the Date & Time Screen comes up on your analyzer's LCD Screen. You may change the Month, Year, Date, Hour, and Minute on your analyzer.

Changing the Month

To change the month, select the downward pointing triangle button next to the month displayed. A drop down menu will appear, listing the months of the year in order of appearance.

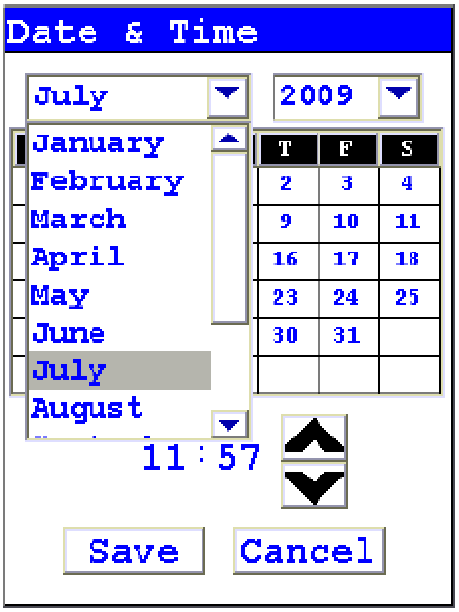


Figure 31. Month Drop Down Menu

Select the month you want from the drop down menu, using the vertical slider button to display hidden months. The display will change to show the month you selected.

Changing the Year

To change the year, select the downward pointing triangle button next to the year displayed. A drop down menu will appear, listing the years in order of appearance.

Figure 32. Changing the Year

Select the year you want from the drop down menu, using the vertical slider button to display hidden years. The display will change to show the year you selected.

Changing the Date

To change the date, select the date you want from the Date Selection Screen. The date you selected will be highlighted in red, while the old date will be shown in red numbers.

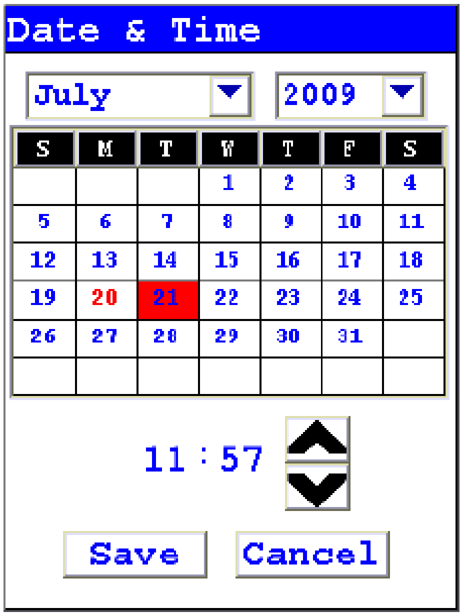


Figure 33. Selecting the Date

Changing the Hour and Minute

To change the hour, select the hour numbers. The hour numbers will be highlighted in gray. Then select the Upwards Pointing Chevron Button to increment (increase) the hour, or the Downward Pointing Chevron Button to decrement (decrease) the hour.

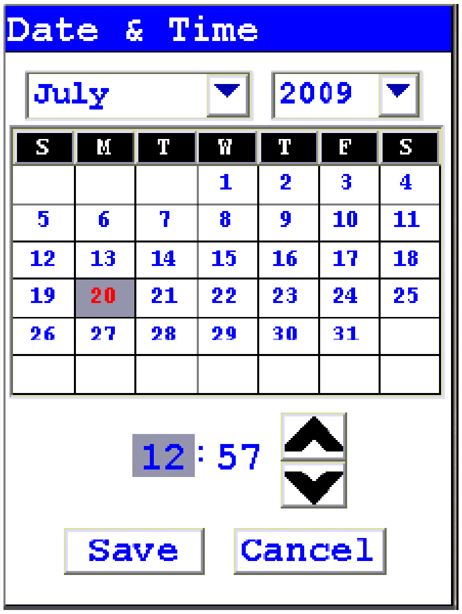


Figure 34. Changing the Hour

To change the minute, select the minute numbers. The minute numbers will be highlighted in gray. Then select the Upwards Pointing Chevron Button to increment (increase) the minute, or the Downward Pointing Chevron Button to decrement (decrease) the minute.

Figure 35. Changing the Minute

Saving Your Changes

To save your changes, select the "Save" screen Button. The display will return to the previous screen and the Date and Time will be saved.

Exiting Without Saving

To exit the screen without saving changes, select the "Cancel" Screen Button. The display will return to the previous screen and the Date and Time will not be saved.

Data Entry

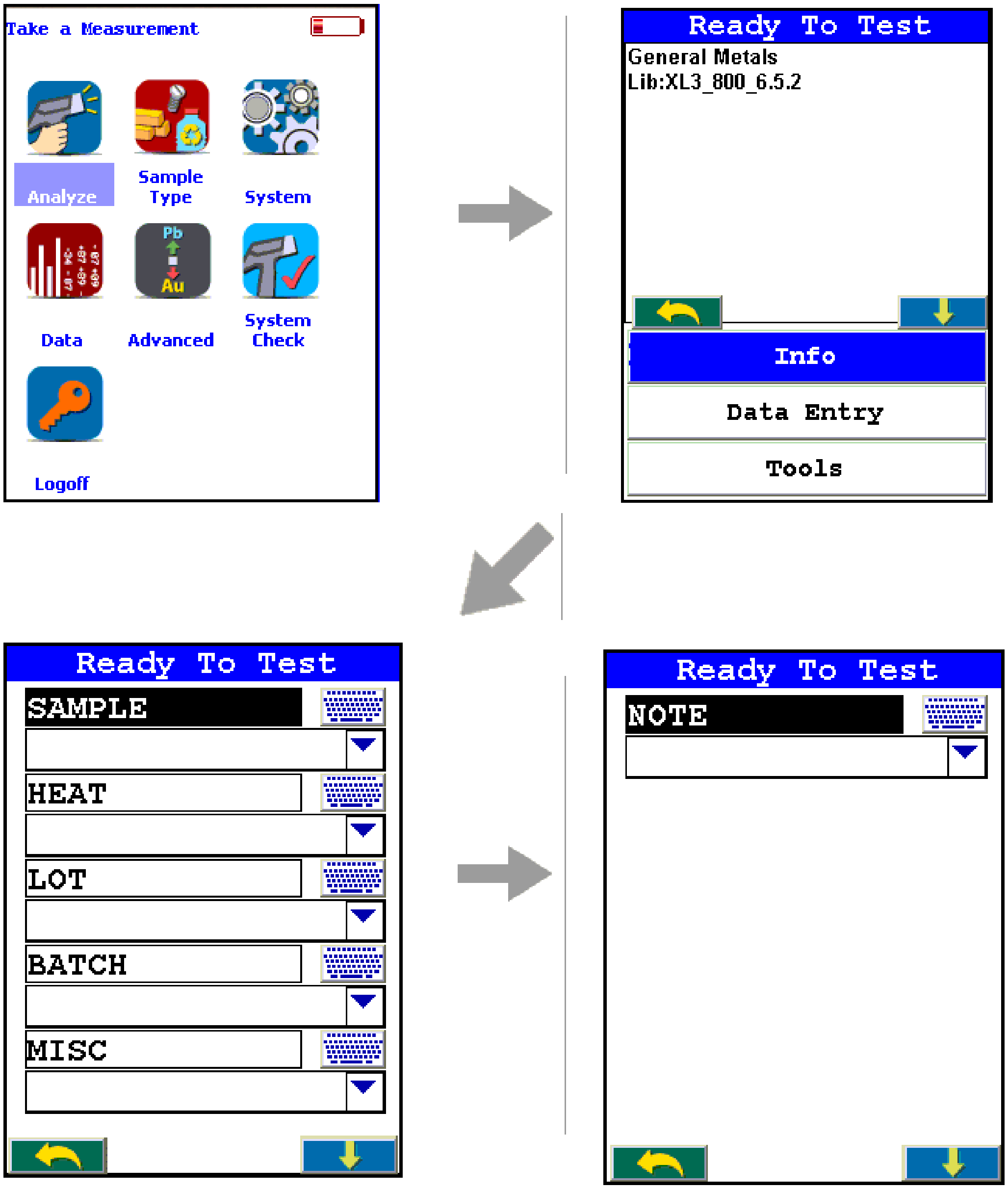


Figure 36. The Data Entry Menu Path

The Data Entry Menu can be accessed from the Ready to Test Screen, then selecting the Data Entry button from the slide down window on the Ready to Test screen. The Data Entry Menu is composed of several data fields with associated buttons. How many and exactly which fields are displayed depends on the Mode the analyzer is currently in.

Entering data into these fields enables you to keep track of details about the reading, for use in later analysis and enhancing the defensibility of the readings.

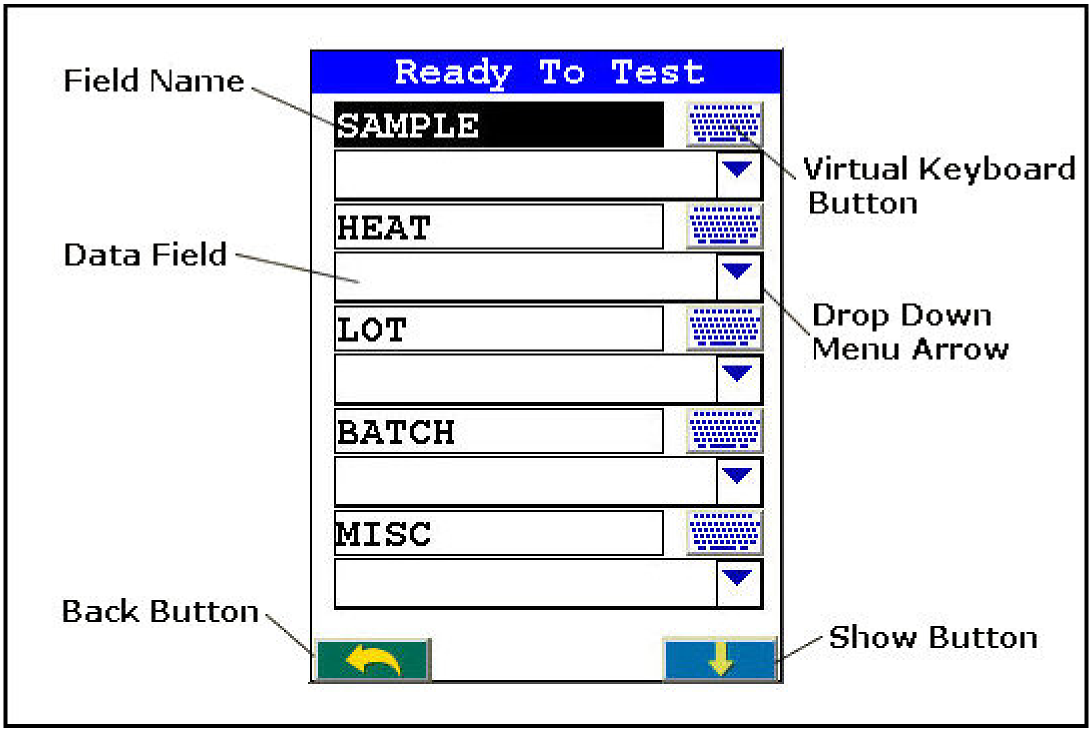


Figure 37. The Data Entry Menu Buttons

The Data Entry Menu Buttons

The Data Entry Menu Buttons each enable you to enter data into the fields in different ways.

Field Name

The Field Name is the designation of the type of data to be entered into the field. Selecting the Field Name enables you to enter data into that field.

Virtual Keyboard Button

Selecting the Virtual Keyboard Button will bring up the Virtual Keyboard, which you can use to type data into the field.

Drop Down Menu Arrow

Selecting the Drop Down Menu Arrow enables you to select data from a list you have previously entered into the analyzer.

Data Field

Your data will appear in the Data Field.

Back Button

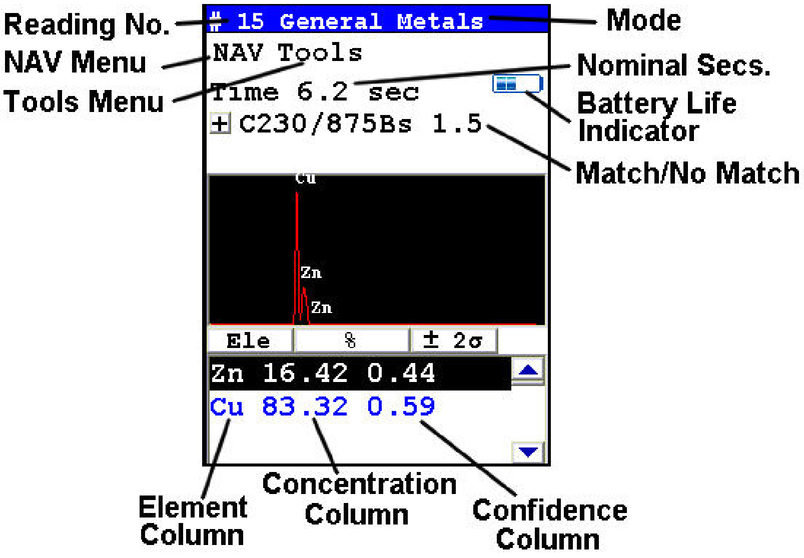
Selecting the Back Button will return you to the Main Menu.

Show Button

Selecting the Show Button will bring the slide down window up into view for access to the Tools Button or Info Button.

The Results Screen

Your analyzer will display the Results Screen throughout the duration of each reading. The Results Screen is updated regularly throughout the reading. When the reading is complete, a final screen update will appear, and your analyzer will display the final results of the measurement which has just been completed.



Time 6.2 sec

+ C230/875Bs 1.5

Ele	%	± 2σ
Zn	16.42	0.44
Cu	83.32	0.59

Figure 38. The Results Screen

The Results Screen displays the following information:

The Reading Number shows a number sequentially assigned by your analyzer in order to uniquely identify each reading. The reading number automatically increments with each successive reading.

The Nominal Seconds Test Duration line shows the number of nominal (source) seconds elapsing since the initiation of the reading. Nominal seconds are instrument time designed to compensate for the electronic dead-time that may occur when your analyzer is taking a measurement, and therefore tend to be a bit slower than real time.

The Mode displays the test mode in use during the measurement.

The Match/No Match line indicates whether your analyzer has found a matching alloy in it's library for the sample you have measured, and displays the name(s) of any matching alloys and the Match Number (chi squared deviation).

The Element (left) column shows the elements that have been detected in the sample.

The Concentration Level (central) column shows the concentration levels of the corresponding elements in percentages.

The Confidence (right) column displays the 2 sigma (95%) confidence interval for the corresponding elements.

If there are too many elements detected to fit onto a single screen, you can see the balance of the elements and their results (as in figure 3-16) by pressing the down arrow of the 4-way touch pad. To see previous results, use the left arrow of the 4-way touch pad. To go forward to later readings, use the right arrow of the 4-way touch pad.

Match Number

The Match Number is a rating of confidence in the identification of the alloy. The Match number ranges from 0.0 to 10.0, with 0.0 being a perfect match. Any number greater than 4.0 gives a result of No Match by default, although you can change this match threshold. Any number less than 2.0 is considered a Good Match, and can usually be brought closer to 0.0 with longer testing times. Numbers between 2.0 and 3.0 can be considered Probable Matches, and numbers between 3.0 and 4.0 as Possible Matches, often having one or more elements out of spec.

How to Analyze

To analyze samples, from the main menu select sample type, and then click on the appropriate Mode icon. Once in the Selection Screen you have a number of sub-modes to select from. depending on how your instrument is calibrated. See the Example Path below.

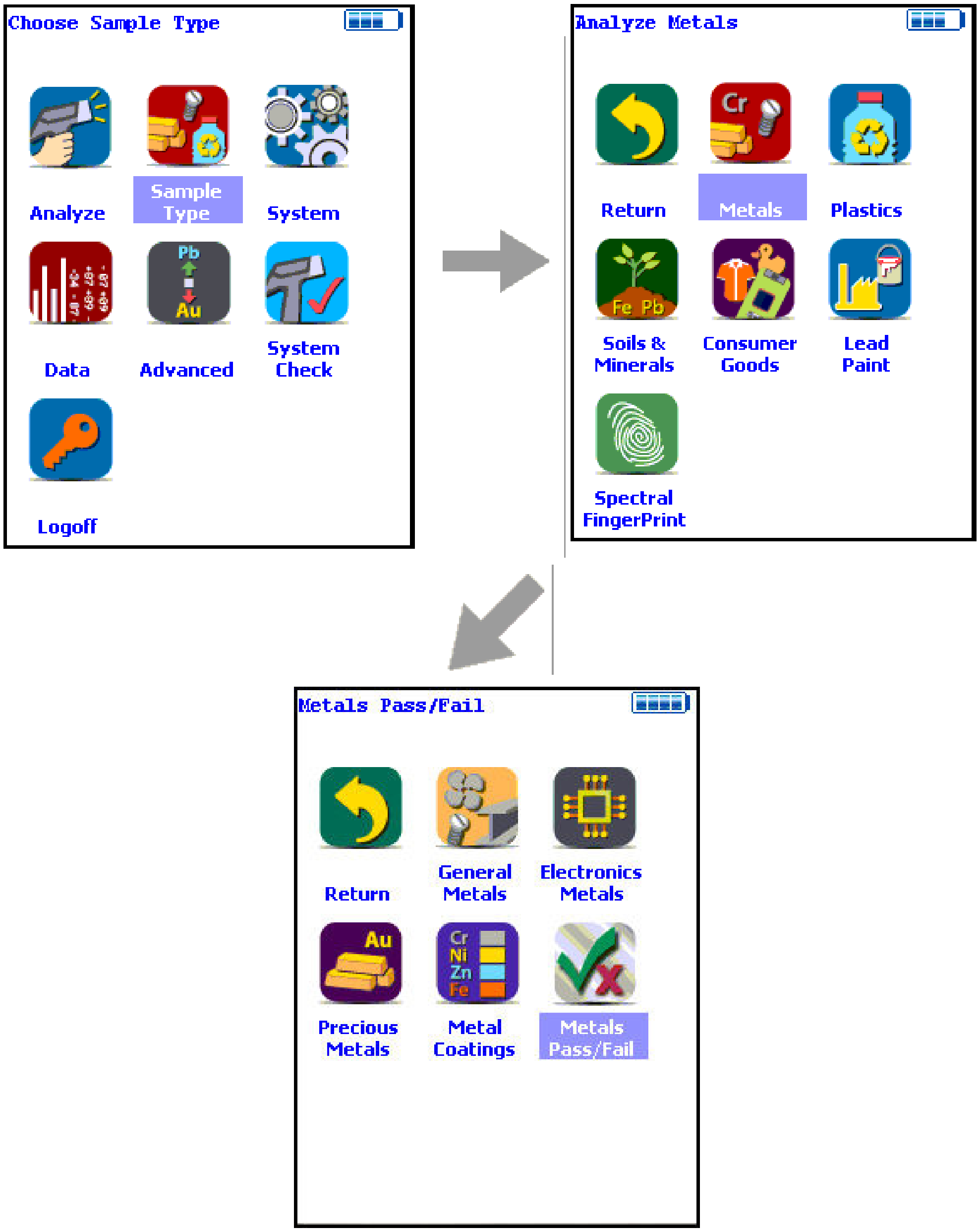
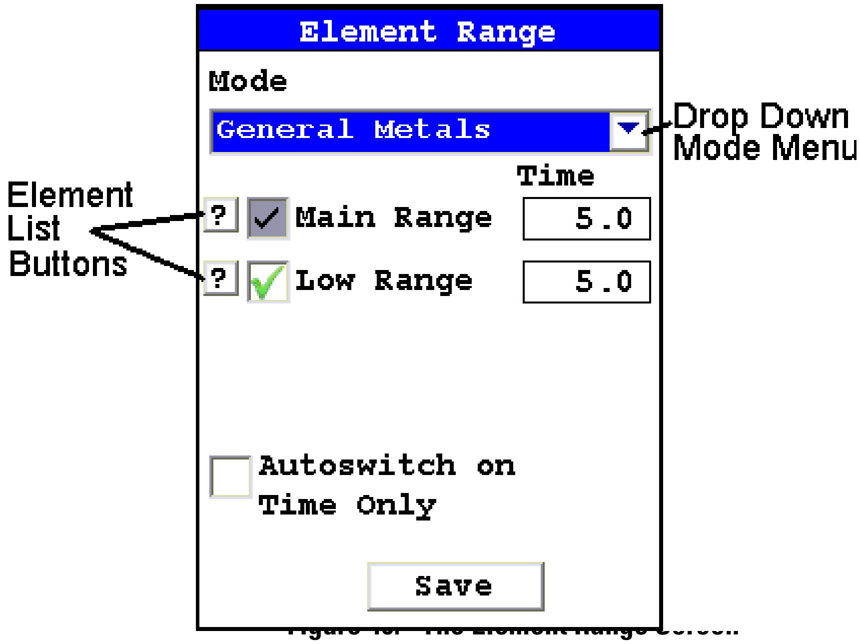


Figure 39. The Metals Analysis Menu Path (Example)

Element Ranges and Lists

From the Element Range Screen, select the Element List Button to display the Element List for the Range you want to use. This list shows the elements that the Range is best designed to detect. See Adjusting the Element Range for details.



Mode
General Metals

		Time
?	☑ Main Range	5.0
?	☑ Low Range	5.0

☐ Autoswitch on Time Only

Figure [illegible]. The Element Range Screen

General Analysis

Note Each user should read the Thermo Scientific Niton XL3 User’s Guide carefully before initiating measurements with the system. Users are strongly urged to attend the Thermo Scientific Niton XRF Analyzer Radiation Safety and Operations Training courses offered regularly, or the web-based trainings. For more information, visit www.thermo.com/niton.

PREPARATORY TASKS

Attach a charged battery to the analyzer and turn it on. Follow the screen instructions and “Log On” as the operator using either the default password or a custom one as designated by the user in an NDU file.

Wait five (5) minutes before using the analyzer, allowing the instrument electronics to stabilize.

Verify that the date is set properly for data tracking purposes.

From the Main Menu, select the System icon, then the Specs icon. The date will be displayed for verification. If the date is incorrect, correct it prior to proceeding. This can be done by "Closing" out of the Specs screen and selecting the Date & Time icon. Detailed information on this procedure is available in Setting the Date and Time.

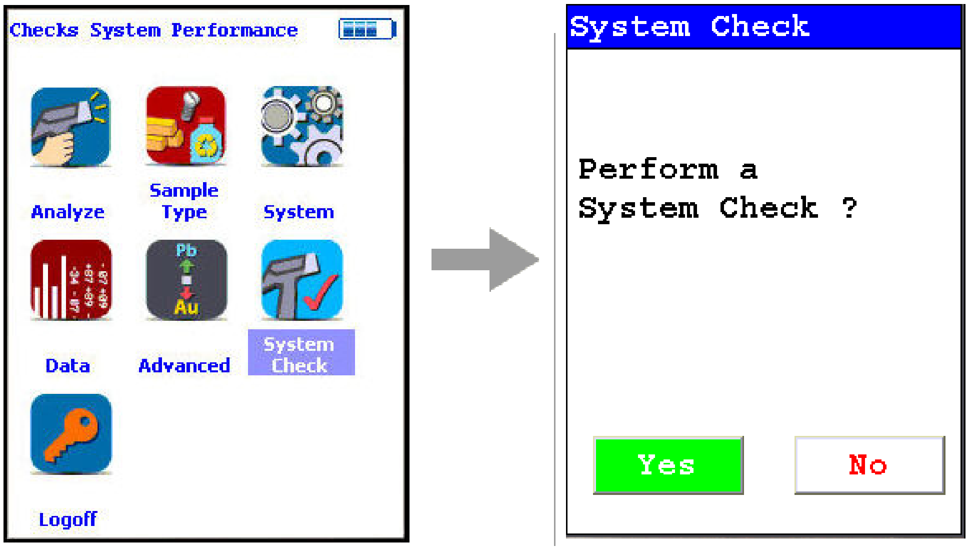
(Optional) Connect the analyzer to a computer via the included serial cable, USB cable, or Bluetooth™ wireless module. (Consult "Using Your Analyzer With Your PC" on page 151for details, if necessary.)

During analysis and detector calibrations, it is important to ensure that the analyzer is not exposed to strong electromagnetic fields, including those produced by computer monitors, hard drives, cellular telephones, walkie talkies, etc. Keep a minimum two (2) feet (0.7 meters) distance between the analyzer and electronic devices.

From the Main Menu, select System Check icon then the Yes button. (Figure 1.)

System Check calibrates the detector and verifies it is operating to specifications. After starting the process, no further user interaction is required during this operation. When the instrument is finished performing the check, the unit will show either "System OK" or one of the failure errors.

If the unit shows a failure error, then perform a second System Check by clicking Recheck. If the unit still does not show a "System OK," please contact Thermo Scientific Niton Analyzers toll-free in the USA at (800) 875-1578, +1 978 670-7460, niton@thermofisher.com, or contact your local Niton Analyzers representative for assistance.



Checks System Performance

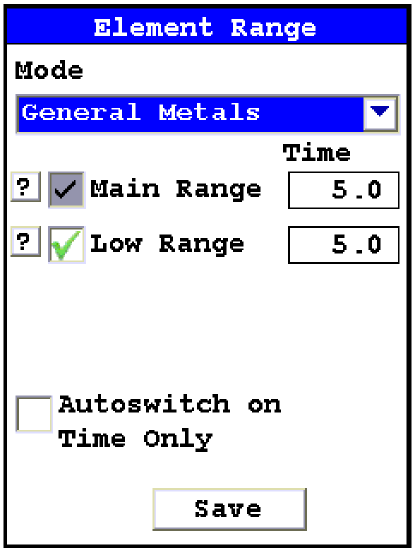
System Check

Perform a System Check ?

Figure 41. System Check Menu Path

Thermo Scientific Niton XL3 analyzers are equipped with excitation filters that optimize the analyzers' sensitivity for various elements. The "Main Range" filter provides optimum sensitivity for the elements manganese (Mn) through bismuth (Bi). The "Low Range" filter is used to optimize the sensitivity for the elements from titanium (Ti) through chromium (Cr).

Note that the main range filter can be used to analyze Ti, V and Cr, but the sensitivity is not as good as when using the low filter. The "Light Range" filter is available only with He-purged and GOLDD technology analyzers, and is typically used in light element analysis. The amount of time that the analyzer spends in each filter position is user definable, but the default settings should be used unless there is reason to change them. Please note that the analyzer will continue alternating excitation filters until the user selectable maximum analysis time is reached or the operator terminates the measurement.



Element Range

Mode: General Metals

		Time
?	☑ Main Range	5.0
?	☑ Low Range	5.0

☐ Autoswitch on Time Only

Figure 42. Setting Element Ranges

Verify instrument measurement accuracy using the supplied reference material (RM) supplied with the analyzer.

Test the factory-supplied reference standard (or other approved check sample) based on a 30s measurement using main range filter only. If the sample is correctly identified and all major elements read within calculated acceptance limits (within the low and high values of factory readings found on the QC sheet, proceed to General Testing Protocol section

If the analyzer reports values outside the acceptance tolerance ranges specified in the tables, repeat the detector calibration then repeat the reference sample analysis.

If the analyzer again fails to meet the acceptance tolerance ranges specified in the tables, please contact Thermo Scientific Niton Analyzers or your local representative for assistance.

GENERAL TESTING PROTOCOL

Good surface preparation is essential for obtaining accurate test results. All non- representative material (e.g., paint, coating, scale) must be removed prior to testing. An approximately 2-inch-square section of surface should be cleaned down to the material to be analyzed. See the Resource Guide for information on Sample Preparation.

The analyzer will often display a correct alloy identification and/or accurate chemistry result before the specified time interval. If the accuracy meets the user's requirements, it is not necessary to measure for the full time.

Longer measurements might be necessary if low concentrations of elements must be determined.

INSTRUMENT QC

Measure the supplied reference calibration check sample AT LEAST once a shift. If correct, continue work. If incorrect, redo System Check and re-take the past 2 hours of results.

UNDERSIZED OR NON-CONTACT SAMPLES

(Samples that do not make contact with or that do not fully cover the measurement aperture)

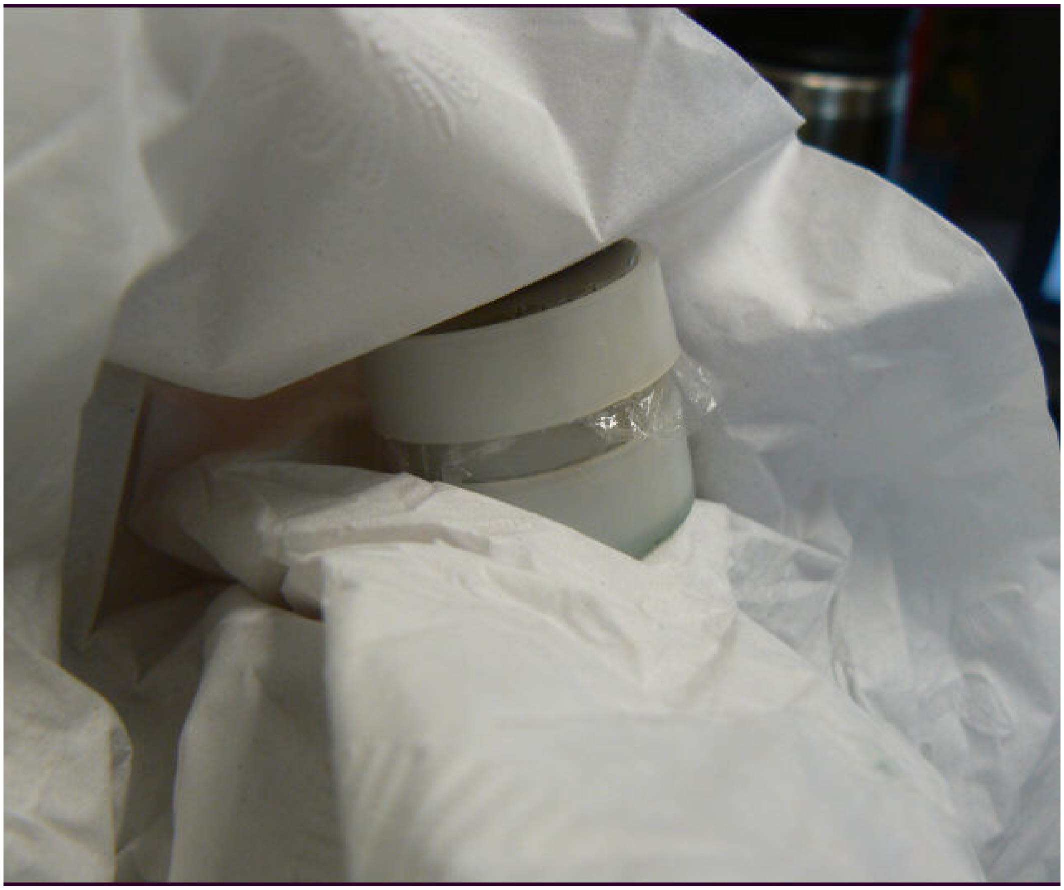
For samples that do not fully cover the measurement aperture, increase the testing time by increasing the time in inverse proportion to the decrease in percentage of aperture covered. For example: a rod only covers ½ of the aperture, so increase the measurement time by two (e.g., from 10 to 20 seconds per filter for alloy chemistry).

The best procedure to measure undersized samples is to use the Thermo Scientific Niton portable test stand (optional), which is shielded to prevent radiation exposure to the operator.

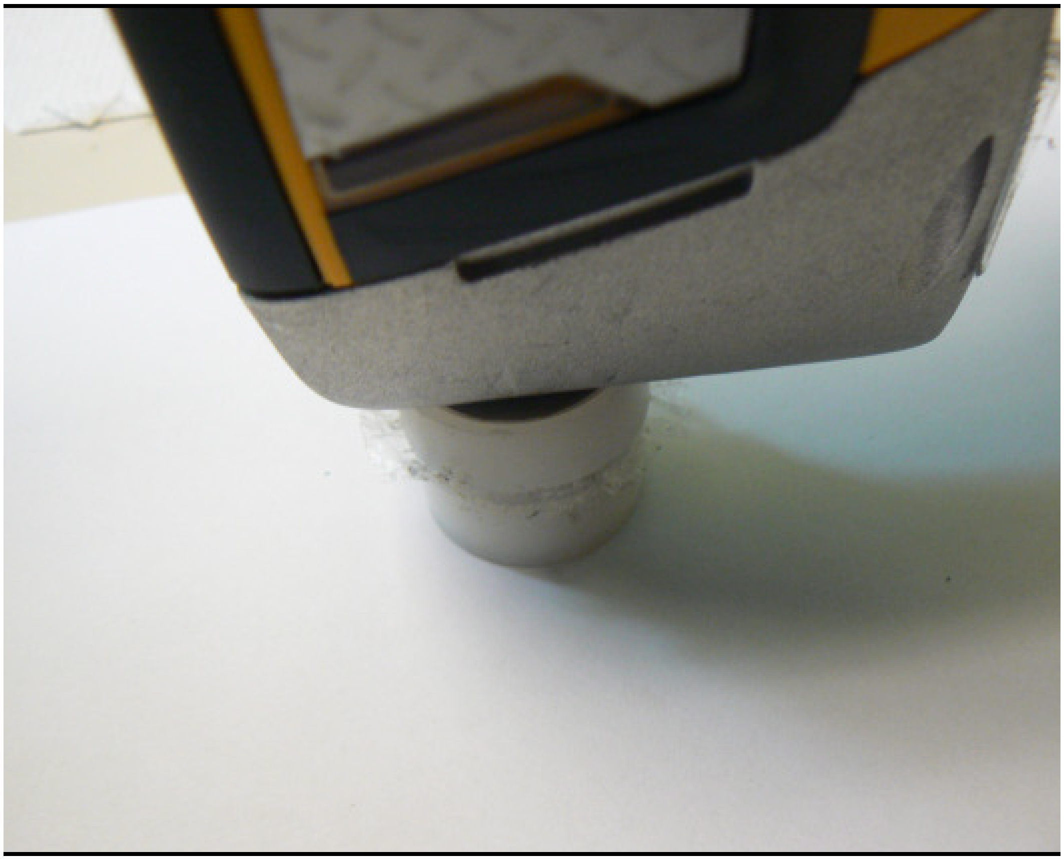
An undersized sample may alternately be measured while lying on another material. Results may be affected by the signal coming from the underlying material itself. Use only pure aluminum, pure plastic, or clean wood and employ the Disable Al feature. Use the Tools Menu, then select Disable Al, and check the underlying surface itself to be sure no metals are present. Be sure to use the Tools Menu and select Enable Al before testing aluminum alloys.

Basic Operation

Taking a Sample Analysis

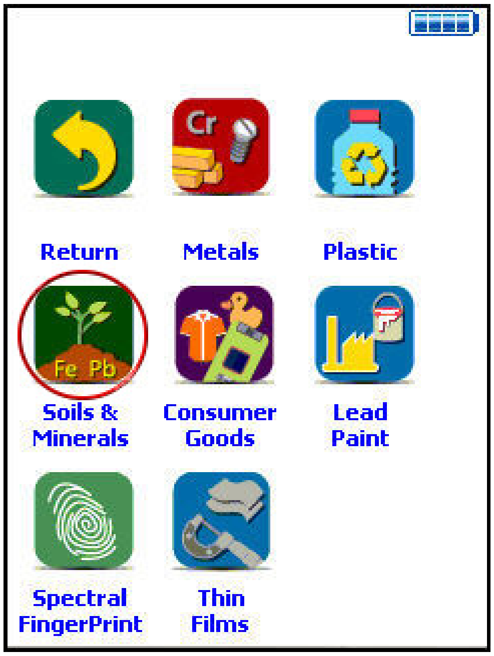


1. Clean the sample to be analyzed so it is free of all surface contamination.



2. Place the analyzer so the sample is covered by the analysis window.

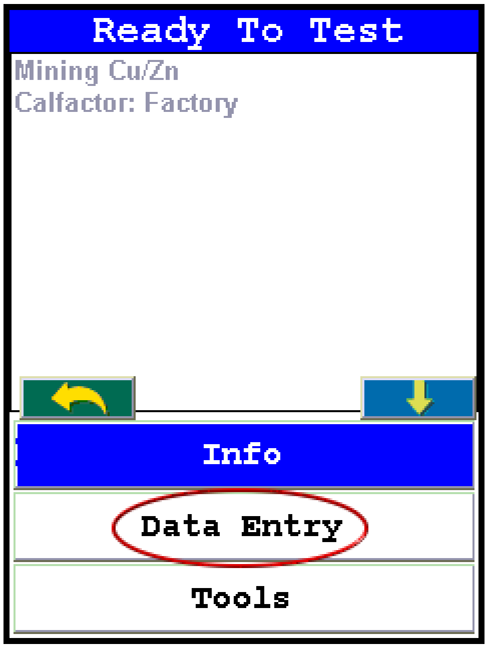
3. Select the Sample Type Icon.



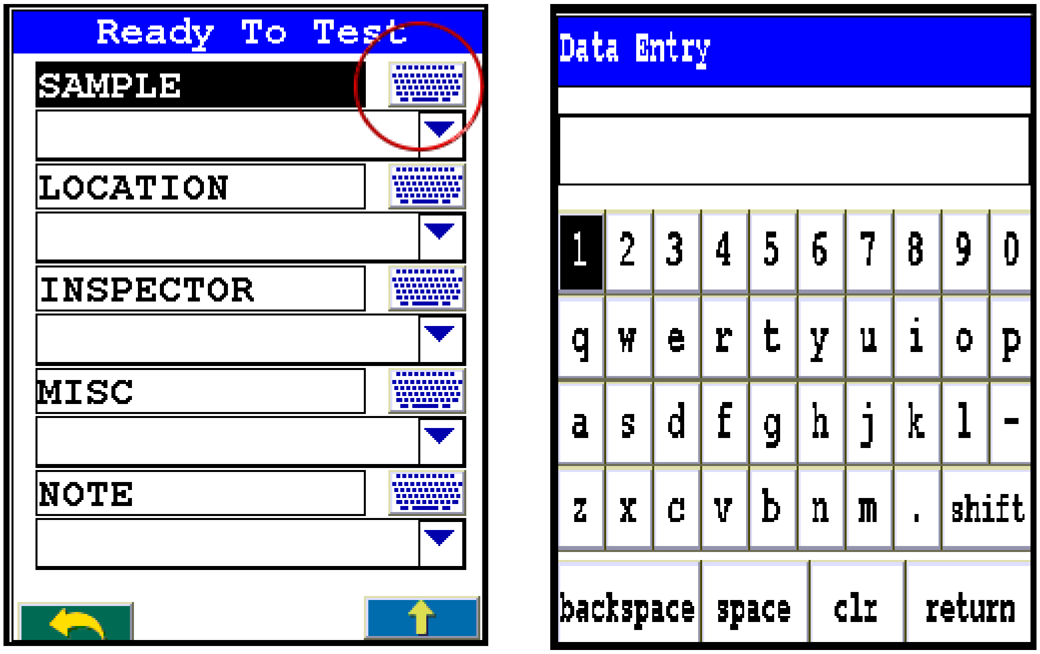
4. Select the proper Mode (in this case Mining Cu/Zn) from the Mode Menu.

Note See “Analysis Modes” on page 79. for more information on the Modes available.

5. Select the Analyze Icon.



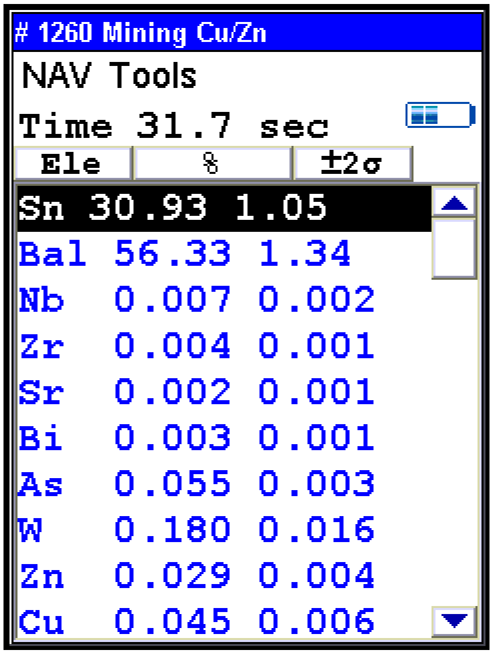
5a. Select Data Entryif you wish todo any data entry.



5b and 5c. Enter the data on the sample using the Virtual Keyboard.

6. Initiate a Reading by pressing the trigger.

7. When the sample has been sufficiently analyzed, release the trigger.



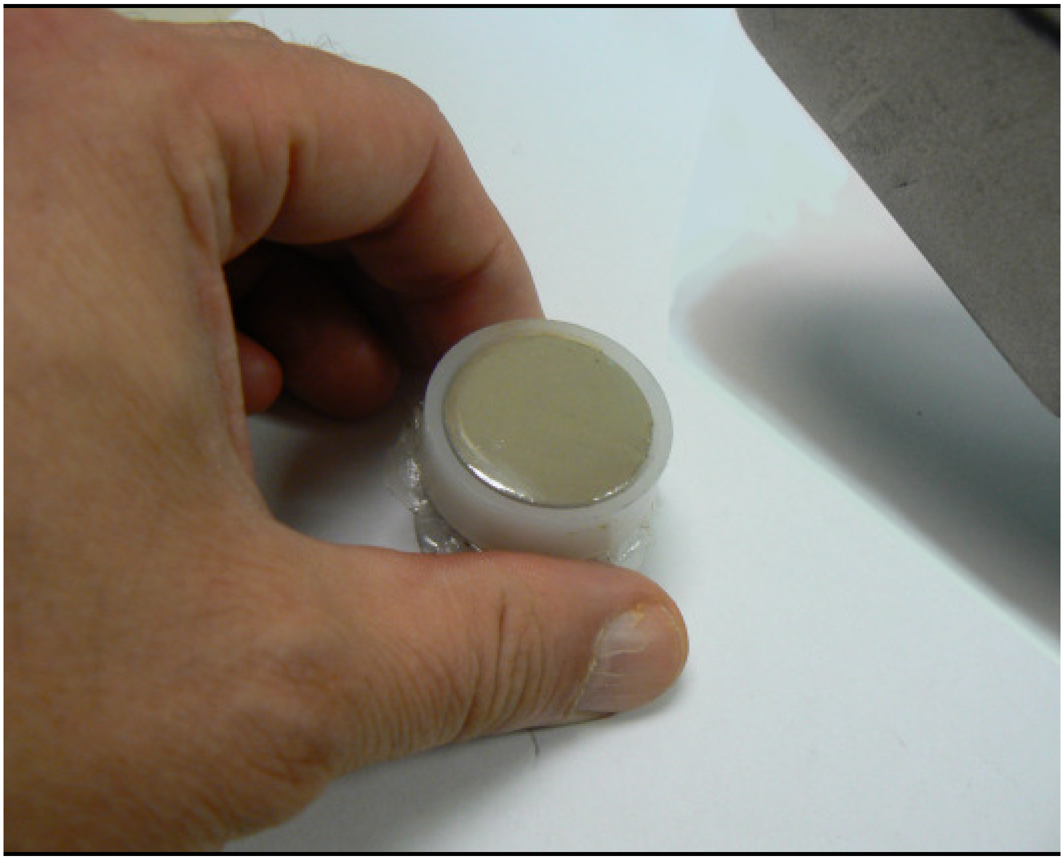
1260 Mining Cu/Zn

NAV Tools

Time 31.7 sec

Ele	%	±2σ
Sn	30.93	1.05
Bal	56.33	1.34
Nb	0.007	0.002
Zr	0.004	0.001
Sr	0.002	0.001
Bi	0.003	0.001
As	0.055	0.003
W	0.180	0.016
Zn	0.029	0.004
Cu	0.045	0.006

8. View the composition returned.



9. Remove the sample.

Analysis Modes

Your analyzer has several Analysis Modes. Which Analysis Mode you should use depends on the nature of the sample you are attempting to analyze.

General Metals Mode

Use this mode to analyze samples entirely composed of metal alloys. This mode will attempt to return an Alloy Grade Identification by matching the analyzed composition of the sample with the nominal composition of alloys in the analyzer's Alloy Grade Library. It will also return an elemental composition of the alloy as analyzed. Alloy Composition is output by default in terms of percent of composition by weight.

See “Using General Metals Mode” on page 81.

Electronic Metals Mode

Use this mode to analyze electronic component samples - circuit boards, chips, etc. This mode will attempt to return an Alloy Grade Identification by matching the analyzed composition of the sample with the nominal composition of electronic alloys in the analyzer's Alloy Grade Library. It will also return an elemental composition of the electronic alloy as analyzed. Electronic Metal Composition is output by default in terms of percent of composition by weight.

See “Using Electronic Metals Mode” on page 82.

Precious Metals Mode

Use this mode to analyze samples composed primarily of precious metals. This mode will attempt to return an Alloy Grade Identification by matching the analyzed composition of the sample with the nominal composition of alloys in the analyzer's Precious Alloy Grade Library. It will also return an elemental composition of the precious metal sample as analyzed. Precious Alloy Composition is output by default in terms of parts per million.

See “Using Precious Metals Mode” on page 82.

Plastics Mode

Use this mode to analyze samples composed primarily of plastic. This mode will return an elemental composition of the plastic sample as analyzed. Plastic Composition is output by default in terms of parts per million.

See “Using Plastics Mode” on page 84.

Soils Mode

Use this mode to analyze samples composed primarily of soil and rock. This mode will return an elemental composition of the soil sample as analyzed. Soil Composition is output by default in terms of parts per million.

See “Using Soils Mode” on page 84.

Mining Cu/Zn Mode

Use this mode to analyze samples composed of potential metal ore - rock containing high proportions of metal - and containing Cu and/or Zn. This mode will return an elemental composition of the ore sample as analyzed. Ore Composition is output by default in terms of percent of composition by weight.

See “Using Mining Cu/Zn Mode” on page 85.

Mining Ta/Hf Mode

Use this mode to analyze samples composed of potential metal ore - rock containing high proportions of metal - and containing Ta and/or Hf. This mode will return an elemental composition of the ore sample as analyzed. Ore Composition is output by default in terms of percent of composition by weight.

See “Using Mining Ta/Hf Mode” on page 86.

TestAll Mode

Use this mode to analyze samples composed of unknown and/or mixed composition, such as toys and consumer products. This mode will attempt to return a general Material Identification by comparing the analysis with other general types of materials. It will select the proper sub-mode for analysis and return an elemental composition of the sample as analyzed. Material Elemental Composition is output by default in terms of parts per million.

See “Using TestAll Mode” on page 86.

TestAll Geo Mode

Use this mode to analyze powder, mineral, and ore samples without first determining whether the samples would best be analyzed with Mining or Soil Mode. This mode uses both the Compton Normalization calibration (Soil) and the Fundamental Parameters calibration (Mining) to determine whether the soil calibration is acceptable or whether the total metal content is too high for Compton mode. It will then return an elemental composition of the sample as analyzed. If the sample can be analyzed via soil mode, then the analyzer will display results from both Soil and Mining Modes in one unified list. If both calibrations contain the same element, then the mode that has the lower detection limit will be displayed. Material Elemental Composition is output by default in terms of both parts per million (mg/kg) and percent of composition by weight, with 0.10% being the cutoff point.

Note Due to the nature of this mode, your analyzer will only use factory calibrations. User modified Cal Factors will not be available.

See “Using TestAll Geo Mode” on page 86.

Using General Metals Mode

1. Clean the sample to be analyzed so it is free of all surface contamination, grinding the surface if appropriate.

2. Place the analyzer so that the sample covers the analysis window.

3. Select the Mode icon.

a. Select General Metals from the Mode Menu.

4. Select the Analyze icon.

a. Select the Data Button if you wish to do any data entry.

b. Enter the data on the sample using the Virtual Keyboard.

5. Initiate the analysis.

6. When the sample has been sufficiently analyzed, stop the analysis.

7. View the composition returned.

8. Remove the sample.

Using Electronic Metals Mode

1. Clean the sample to be analyzed so it is free of all surface contamination.

2. Place the analyzer so that the sample covers the analysis window.

3. Select the Mode icon.

a. Select Electronic Metals from the Mode Menu.

4. Select the Analyze icon.

a. Select the Data Button if you wish to do any data entry.

b. Enter the data on the sample using the Virtual Keyboard.

5. Initiate the analysis.

6. When the sample has been sufficiently analyzed, stop the analysis.

7. View the composition returned.

8. Remove the sample.

Using Precious Metals Mode

1. Clean the sample to be analyzed so it is free of all surface contamination.

2. Place the analyzer so that the sample covers the analysis window.

3. Select the Mode icon.

a. Select Precious Metals from the Mode Menu.

4. Select the Analyze icon.

a. Select the Data Button if you wish to do any data entry.

b. Enter the data on the sample using the Virtual Keyboard.

5. Initiate the analysis.

6. When the sample has been sufficiently analyzed, stop the analysis.

7. View the composition returned.

8. Remove the sample.

Using Plastics Mode

1. Clean the sample to be analyzed so it is free of all surface contamination.

2. Place the analyzer so that the sample covers the analysis window.

3. Select the Mode icon.

a. Select Plastics from the Mode Menu.

4. Select the Analyze icon.

a. Select the Data Button if you wish to do any data entry.

b. Enter the data on the sample using the Virtual Keyboard.

5. Initiate the analysis.

6. When the sample has been sufficiently analyzed, stop the analysis.

7. View the composition returned.

8. Remove the sample.

Using Soils Mode

1. Pack the sample into a Sample Cup.

a. Clean the sample to be analyzed so it is free of all surface contamination.

2. Place the analyzer so that the sample covers the analysis window.

3. Select the Mode icon.

a. Select Soils from the Mode Menu.

4. Select the Analyze icon.

a. Select the Data Button if you wish to do any data entry.

b. Enter the data on the sample using the Virtual Keyboard.

5. Initiate the analysis.

6. When the sample has been sufficiently analyzed, stop the analysis.

7. View the composition returned.

8. Remove the sample.

Using Mining Cu/Zn Mode

1. Clean the sample to be analyzed so it is free of all surface contamination.

2. Place the analyzer so that the sample covers the analysis window.

3. Select the Mode icon.

a. Select Mining Cu/Zn from the Mode Menu.

4. Select the Analyze icon.

a. Select the Data Button if you wish to do any data entry.

b. Enter the data on the sample using the Virtual Keyboard.

5. Initiate the analysis.

6. When the sample has been sufficiently analyzed, stop the analysis.

7. View the composition returned.

8. Remove the sample.

Using Mining Ta/Hf Mode

1. Clean the sample to be analyzed so it is free of all surface contamination.

2. Place the analyzer so that the sample covers the analysis window.

3. Select the Mode icon.

a. Select Mining Ta/Hf from the Mode Menu.

4. Select the Analyze icon.

a. Select the Data Button if you wish to do any data entry.

b. Enter the data on the sample using the Virtual Keyboard.

5. Initiate the analysis.

6. When the sample has been sufficiently analyzed, stop the analysis.

7. View the composition returned.

8. Remove the sample.

Using TestAll Mode

1. Clean the sample to be analyzed so it is free of all surface contamination.

2. Place the analyzer so that the sample covers the analysis window.

3. Select the Mode icon.

a. Select TestAll from the Mode Menu.

4. Select the Analyze icon.

a. Select the Data Button if you wish to do any data entry.

b. Enter the data on the sample using the Virtual Keyboard.

5. Initiate the analysis.

6. When the sample has been sufficiently analyzed, stop the analysis.

7. View the composition returned.

8. Remove the sample.

Using TestAll Geo Mode

1. Clean the sample to be analyzed so it is free of all surface contamination.

2. Place the analyzer so that the sample covers the analysis window.

3. Select the Mode icon.

a. Select TestAll Geo from the Mode Menu.

4. Select the Analyze icon.

a. Select the Data Button if you wish to do any data entry.

b. Enter the data on the sample using the Virtual Keyboard.

5. Initiate the analysis.

6. When the sample has been sufficiently analyzed, stop the analysis.

7. View the composition returned.

8. Remove the sample.

Common Operations

Metal Sample Prep

Up until recently, sample preparation was not a big worry for XRF metals analysis, as the LOD of the analyzer was seldom low enough for any but the most heavy contamination to be intrusive; but recent developments such as He-purged analysis have brought analysis to a level where even light surface contamination can skew an analysis.

You should always prepare your samples before analysis, especially when using He-purged analysis, as these analyzers will see even trace amounts of contaminants. Oils from fingerprints and other body contact, lint, oxidation materials, and abrasive materials used in cleaning can all skew readings if not removed. Sample preparation is simple and not time consuming, and usually well worth the effort.

The following is a list of problems that need correction before testing:

- Oxidation or Rust may produce an increase or decrease in one or more element test values unless we remove the rust or oxidation and expose the raw metal.
- Paint may contain several elements which need to be tested at lower levels within metal alloys (Ti & Zn in white paint, Fe in red paint, Cr in green paint).
- Oil, grease or lubricates may contain high levels of the following elements: lithium, aluminum, barium, strontium, molybdenum or calcium.

Plated surfaces may have high levels of the following elements: zinc, chromium, nickel, or copper.

CAUTION Anything on the metal surface will become part of your test results!

Sample Analysis Preparation

You need to clear the surface of your samples of any paint, plating, or any oxidation such as rust or verdigris before analysis. In order to accomplish this, you need the following:

- Isopropyl alcohol - not rubbing alcohol, which contains oils.
- Lint-free paper.
- Diamond paper - P/N 179-1202- cut into 1 inch/2.5 cm squares. Never re-use this paper, as it may transfer contaminants to the surface of the sample from previous cleanings. Depending on the state of the sample, several squares may be needed per sample.

- A Sample Grinder for removing deeper surface contamination. Choice of grinding wheel media also may be important, depending on what you are testing for. Never re-use grinding media, as contaminants can be transferred from sample to sample on the media itself.

For light contamination on hard metal reference standards, remove the oxidation by scrubbing the dry sample lightly with the diamond paper square, using the fingers to maintain pressure. If the diamond paper begins to load up with material, discard it and use a fresh square. When the oxidation is removed, wipe the sample with lint-free paper soaked with isopropyl alcohol to remove any oils or dust. Let the sample dry before attempting analysis.

For soft metal reference standards, wipe the sample with lint-free paper soaked with isopropyl alcohol, then remove the oxidation by scrubbing the wet sample lightly with the diamond paper square, using the fingers to maintain pressure. If the diamond paper begins to load up with material, discard it and use a fresh square. When the oxidation is removed, wipe the sample again with lint-free paper soaked with isopropyl alcohol to remove any oils or dust. Let the sample dry before attempting analysis.

Oils, lint and dust can be removed by wiping the sample with lint-free paper soaked with isopropyl alcohol. Let the sample dry before attempting analysis.

Surface Oxidation

With the exception of a limited number of metal types, most metal alloys form an oxide covering on the surface when exposed to oxygen or air. This oxide covering is visible in carbon and low alloy steel as a red colored substance called rust. Other metal alloys form oxidation which is not always visible, but that does not mean that it is not present. If the test results for low concentration elements are higher or lower than expected, remove the oxide coating by grinding and retest. Follow proper safety procedures when changing discs or grinding materials.

During a recent case study the effects of sample preparation became apparent. A customer asked for low detection limits of nickel, chromium and copper in carbon steel pipe. The reported chemistry of the purchased material is listed on the first line in the chart below. The test results of a hand held Niton XL3t 900S GOLDD instrument appears in the second line of the chart. The results from a test on the unground surface appear in the bottom line of the chart. Note the values for nickel and copper in this carbon steel alloy in the chart below. The oxidation on the surface of this pipe was not visibly egregious. We need to always be wary of the presence of even low levels of oxidation and their possible effects on analytic accuracy.

Table 1. Comparative test results with and without grinding

Sample	% Mn	% Ni	% Cr	% Mo	% Cu
Reported Chemistry	0.650	0.090	0.070	0.030	0.040

Table 1. Comparative test results with and without grinding

Test Results with Ground Surface	0.67	0.089	0.070	0.033	0.039
Test Results with Unground Surface	0.61	0.178	0.081	0.033	0.514

Painted Surfaces

Paint is a mixture of several items that are combined into a liquid which is applied to the surface of materials such as metal. Once applied this liquid dries with time and adheres to the surface of metal. Paint is used to protect or decorate the metal item. Paint can also be used to identify or mark the metal during the manufacturing process.

Components of paint are divided into classifications of pigments, binders, solvents, additives and fillers. The inorganic elements in pigments will contribute to increases in displayed values for those elements if paint on the metal surface is not removed prior to testing. Be especially careful of the presence of heavy elements, which can also act to shield x-rays from lighter elements in the metal sample.

The following is a list of some of the most common components of paint:

White Paint

- Antimony (Sb)
- Lead (Pb)
- Titanium (Ti)
- Zinc (Zn)
- Cobalt (Co)

Red Paint

- Iron (Fe)
- Lead (Pb)
- Green Paint
- Chromium (Cr)

An experiment was conducted to determine the effect and severity of surface problems on XRF results. Results from analyses of a 1541 alloy steel sample are shown below, before and after surface grinding. The sample had painted markings, of light to medium thickness, on the surface, as well as light rust. Note the change in titanium, zinc and cobalt levels after surface grinding.

Table 2. Prepped and unprepped painted metal analysis

Sample	Mn	Ni	Cr	Mo	Ti	Zn	Co
Ground Surface	1.49	0.04	0.03	0.004	0.011	0.0001	0.03
Unground Surface	1.34	0.01	0.04	0.011	2.507	1.751	0.21

Oil, Grease & Cutting Oils

Oil and grease contain a number of elements combined into a viscous substance and applied to moving parts in order to reduce friction. Grease coatings can remain on component surfaces after it has been removed from service. Grease can also be applied to a metal's surface by accidental contact with other materials coated in heavy grease. Metals can also be coated in oil as a result of cutting and machining processes in manufacturing.

Grease and oil may contain the following elements:

- Aluminum (Al)
- Zinc (Zn)
- Molybdenum (Mo)
- Sodium (Na)
- Calcium (Ca)

An experiment was performed to show how grease on metal surfaces affects XRF results. A carbon steel sample was cleaned and ground as a control surface for the experiment. XRF tests were performed on the control surface, and again after light and heavier layers of automotive wheel bearing grease were applied to the surface of the steel sample. Results are shown below. Note the elevated levels of molybdenum, cobalt and zinc from the grease.

Table 3. Clean and greased sample metal analysis

Sample	Mn	Ni	Cr	Mo	Cu	Co	Zn
Clean Surface	1.18	0.001	0.041	0.004	0.001	0.001	0.019
Light Grease	1.07	0.001	0.001	0.067	0.033	0.322	0.416
Heavy Grease	0.96	0.001	0.001	0.500	0.062	1.760	3.430

If a sample's surface contains lubricants or cutting oil, use a solvent and a clean towel or rag to remove them before analysis. You may then need to grind the surface to insure good results. Clean first, grind second, test last.

Remember to follow safe techniques for handling and disposing of solvents and cleaning rags

Anodized, Plated and Galvanized Surfaces

Anodizing is the process of polarizing the metal surface into a passive state which protects it against corrosion. This process is most often applied to aluminum alloys.

Galvanized steel is one of the most common of the coated surfaces. In this process, steel is passed through a molten bath of a zinc alloy. Zinc reacts with the steel metal to form a bonding layer on the steel surface. The zinc layer does not separate from the steel and forms a protective layer that protects the steel from oxidation.

Galvanized layers are relatively thick compared to other plating elements and methods. When grinding to remove the zinc coating, you will find increased zinc values even when you can see the steel surface. Grind a little further and zinc values will disappear. Zinc clings to the surface of the sanding disc, so you will need to frequently change discs.

Electroplating is another common practice of applying a coating which not only protects the surface from oxidation, but also improves the base material's wear resistance, lubricity and improves the overall aesthetics of the product. The electroplated coating is generally thinner and more evenly applied than galvanizing. Electroplating has a wide range of elements and in some situations there may be two or more different coatings on the same part.

The following is a partial list of elements that are used to plate the surface of base metals:

Ni, Cr, Cadmium (Cd), Tin (Sn), Zn, Al

Cordless Right Angle Drill

This style of drill is recommended for most surface preparation in the field because it gives the operator the greatest amount of control, and thus safety, when grinding samples. When moving a sanding disc on a conventional drill over a sample, forces tend to produce movement the operator may find difficult to control. Control and stability are important in grinding from effectiveness and safety perspectives.

A cordless right angle drill similar to the one pictured below is recommended for light to medium surface removal. For materials with heavy oxidation such as carbon and low alloy steel, an angle grinder, explained in the next section, is recommended. A kit with the drill, batteries and charging units, can be purchased from ThermoFisher, or companies such as DeWalt, Hitachi, Makita, Milwaukee or Ryobi.

Figure 1. Example of Right Angle Drill

A disc holder is needed with the drill to hold the sanding disc. (In the US, we recommend a 3.0 inch disc holder. It has a 0.25 inch shank to insert into the chuck of the drill.) If sanding discs are ordered from a local supplier, attention should be paid to the method of attaching the sanding disc to the disc holder. There are three types of connections: metal snap-on, plastic twist and plastic snap-on.

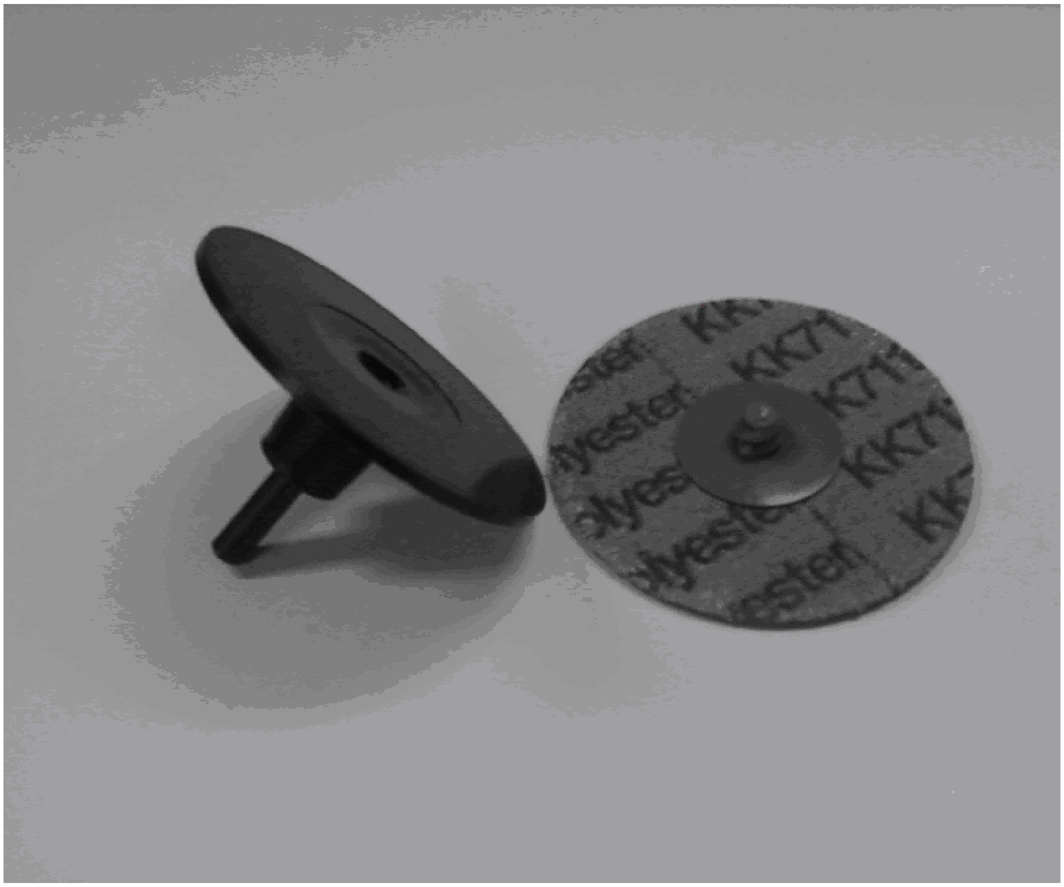


Figure 2. **Sanding Disc**

Before attaching the grinder and sanding disc as pictured below, first remove the battery to disable the grinder. Then insert the shaft of the disc holder into the drill and securely tighten the chuck. Next, attach the appropriate sanding disc. The method of attachment will vary depending upon the type of fastener on the sanding disc (snap-on or twist connectors). Reinstall the battery and prepare for use.

Figure 3. Attaching the Sanding Disc 1

Figure 4. Attaching the Sanding Disc 2

Cordless Angle Grinder

A cordless angle grinder similar to the one pictured below will successfully remove medium to heavy oxidation or paint. This grinder (which uses a 4.5 inch sanding disc with a rubber backup pad) can be purchased from ThermoFisher or industrial tool manufactures like DeWalt, Makita or Milwaukee.

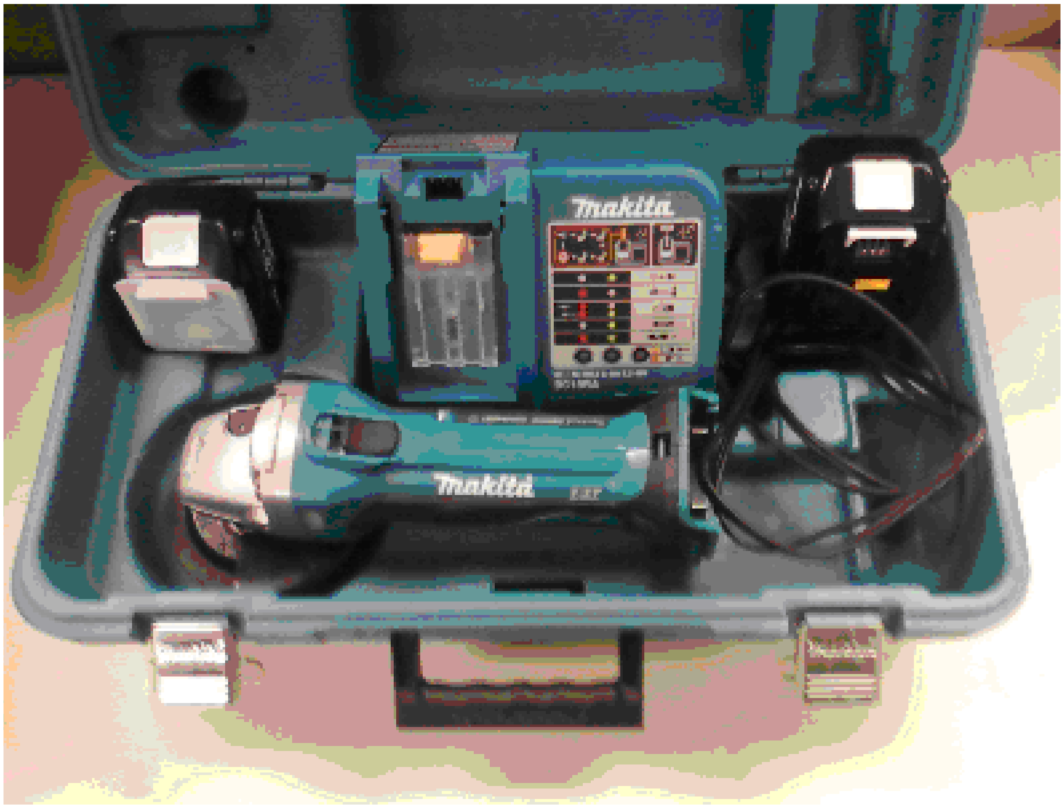


Figure 5. Cordless Angle Grinder Kit

A grinder kit typically contains the grinder, a battery, and charging unit. If the kit contains a grinding stone wheel, remove and dispose of it. Grinding stones are not to be used for XRF sample preparation. A rubber backup pad and a retaining nut are needed to use with sanding discs. (See picture below).

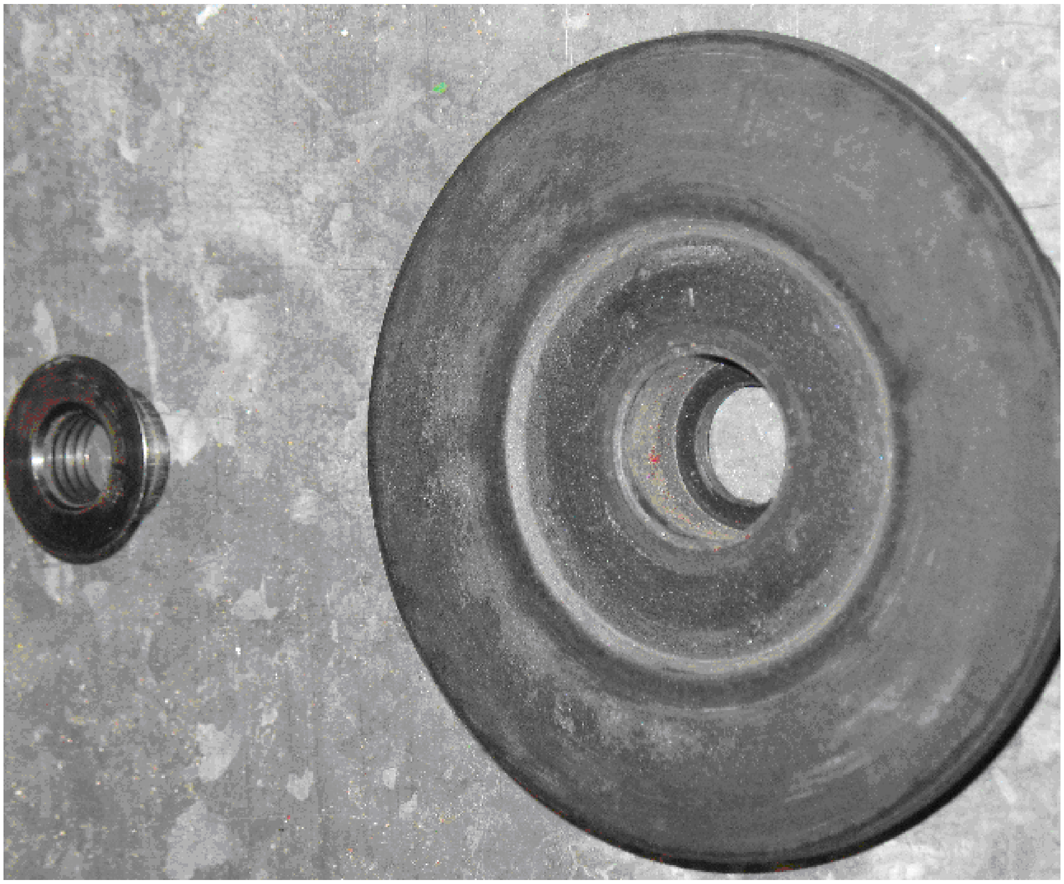


Figure 6. Rubber Backing Pad and Nut

In the US, sanding discs are 4.5 inch diameter and can be purchased in various grit sizes of 36 to 120. The surface abrasive can be one of the following materials: aluminum oxide, silicon carbide or zirconia alumina. The selection of sanding discs is covered in the next section.

Figure 7. **Assembling the Grinder**

Remove the battery before assembling the grinder, backup pad and sanding disc. Start by installing the backup pad onto the drive shaft of the grinder, or, with some backup pads. You will need to screw it onto the threaded shaft.

Next place the sanding disc over the drive shaft onto the backup pad. Hold the locking button on the reverse side of the grinder while tightening the retaining nut into the hole of the sanding disc.

Once the backup pad, sanding disc and locking nut are secured, reinstall the battery. The grinder is now ready for use.

Sanding Discs

It has been well tested and determined that samples can be easily contaminated by the abrasive material contained in and on a sanding disc. An example would be the increase in aluminum content of carbon steel after grinding the sample with a new aluminum oxide sanding disc. Aluminum from the aluminum oxide disc imbeds itself in the surface of the steel sample and an XRF would show an unusually high aluminum concentration.

Aluminum oxide is the most common abrasive surface used today. For most applications it will be safe to use aluminum oxide discs. But if test results for aluminum in any metal alloy are significantly higher than expected, switch to another type of abrasive disc. Also, when grinding aluminum, aluminum oxide discs tend to trap aluminum from the metal surface into the disc surface. Once this happens, the disc looses its efficiency and cross contaminates the next sample.

Silicon Carbide

Silicon carbide discs are a good alternative for aluminum oxide and the cost of a disc is only slightly higher than aluminum oxide. This adhesive type is best for grinding aluminum, copper and other soft metals.

Zirconia Alumina

Zirconia alumina discs are more expensive than aluminum oxide or silicon carbide but they last much longer and so may be the best investment. Few metal alloys have low additive levels of zirconium, so it is one of the safest abrasive types for general use.

One exception is the Aluminum alloy Al 7050 which is a near twin to alloy Al 7075 except for the ~0.1% Zr in 7050. Therefore, if 7075 is ground with Zr grinding paper it may be erroneously identified as Al 7050 due to the Zr transferred from the grinding disk to the surface of the Al 7075. s

Diamond Sanding Paper

Do not use diamond sanding paper for surface preparation in the field. Even after extensive and aggressive sanding with diamond paper, a metal surface will not be prepared properly. Diamond sanding paper is only recommended for removal of very light oxide coatings on flat surfaces such as analytical reference standards.

- Nickel, cobalt, and steel alloys should be ground using 36, 40, 50 or 60 grit discs. The selection of a grit size of 100 or la
- inum, copper alloys, and other softer metals should be ground using 60 or 80 grit discs.
- Grinding stones are not recommended because they will absorb surface material and transfer them onto the next surface ground.

Safety Rules

When using a grinder, follow these safety rules:

- When changing sanding discs, always remove the grinder battery to prevent accidental activation of the grinder.
- Allow the grinder to stop spinning before placing it on a flat surface.

- Replace any damaged or torn sanding discs immediately.
- Always wear impact eye protection to prevent eye damage from flying debris.
- Place small samples or standards in a clamping device when grinding to prevent accidental contact between the spinning disc and your hand.
- Use proper techniques and safety precautions when grinding beryllium, beryllium copper, lead, or titanium alloys.
- Always follow the safety instructions outlined by the grinder's manufacture as mentioned in the instruction manual..

Soil Sample Prep

Examine the site for differences in surface characteristics before sampling. Valid results depend on a sufficient and appropriate selection of sites to sample. Incorrect sample collection may give rise to misleading or meaningless results, regardless of the analysis method. Delineate sections with different characteristics and treat them as different areas. It may be desirable to subdivide larger areas even if they have the same characteristics to ensure a thorough examination. Make certain to label each bag thoroughly. Common information included on each bag includes the person and/or the company who collected the sample, the location and area where the sample was taken, and the date the sample was collected.

Prepared sample analysis is the most accurate method for determining the concentration of elements in a bulk medium using the instrument. Sample preparation will minimize the effects of moisture, large particle size, variations in particle size and sample non-homogeneity.

Note More sample preparation (drying, milling and sifting) will yield greater accuracy. The drier, finer, and more homogeneous the particles, the better the measurements.

Preparing Bulk Soil Samples

We recommends establishing a specific sample protocol. Following this protocol for preparing and testing samples is vital for achieving a level of accuracy comparable with laboratory results. The equipment you need to prepare samples is included in your kit. Among these are a mortar and pestle, several different sized metal sieves, and cups to hold the samples

.

CAUTION All test equipment must be kept clean to prevent contaminationof samples.

Cleaning Your Equipment:

The mortar, pestle, and grinding mill may be cleaned with dry paper towels. You can also clean the mortar, pestle, and the mill's container with water, but be sure each is absolutely dry before using them on another sample. The mortar and pestle may be cleaned by grinding clean, dry sand in the mortar. Use the short bristle brushes (included in your Soil Testing Kit) to clean the sieves. If you have an electric soil grinder in your kit, when the soil grinder blades wear out, unbolt the worn blades and replace them. Call the Thermo Sales Department at 1-800-875-1578 for replacement blades.

Note Using the soil grinder may artificially increase the amount of Fe in soil samples.

Sample Preparation

Prior to analysis, the material should be dry and well homogenized. Ideally, the entire sample should be dried to constant weight, sifted to remove gravel and debris, and ground or milled to a fine powder. Dry the sample if it is moist and cohesive. The sample can be dried in any of several ways. Choose one of the following:

- Oven dry the sample for approximately 2 hours at 150° C, until the sample reaches a constant weight. Note: Oven drying is inappropriate when volatile compounds may be present in the sample. For example, lead present as tetraethyl lead would be driven off by the heat of drying. Some forms of mercury and arsenic are volatile. Air drying will preserve more of these volatile substances.
- Air dry the sample overnight at room temperature in a shallow pan.
- Stir gently and warm the sample in a pan over a hot plate or burner.

Coning and Quartering

You may need to divide your sample at various times during preparation. Coning and quartering is a method for dividing the sample into homogenous quarters.

- Pour the dry material slowly and carefully onto a flat sheet or pan, forming a symmetrical cone. Divide the cone into equal piles using a flat thin-bladed tool, such as a knife or ruler. Divide these in half again.
- Now you have four samples, each one-quarter the size of the original and each more homogenous than the original.
- Grind the sample to break up dirt clods and/or paint chips.

WARNING Grinding and sifting dried samples produces dust. Even clean soil contains silica, which may be hazardous when airborne. Prepare all samples in a ventilated area; wear a mask, gloves, and an apron; and spread a drop cloth.

Sift using the #10 (2mm) mesh and separate out the larger pieces (stones, organic matter, metallic objects, etc. Examine the larger particles by eye but do not include in the sample. Grind the sample again so its particles will be finer and more homogenous. Use mortar and pestle, or an electrically powered grinding mill. Sift at least 10 grams of the sample through #60 (250 ?m) and #120 (125 ?m) mesh. Re-grind the un-passed material until the entire fraction is able to pass. Mix the resulting sample.

Placing the Sample in an XRF Sample Cup

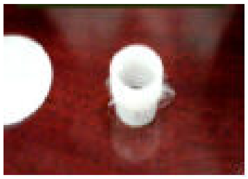
Note The sample container should be a sample cup of a type that can be filled from the rear; that is, the side opposite the window (e.g. Thermo NITON Part Number 187-466). Thermo recommends using a 1/4 mil Polypropylene film (e.g. Thermo NITON Part Number 187-461). A supply of cups and films are included.

The container used to hold the sample will affect the accuracy of the measurement. Use a container with as thin-walled a window as is convenient and use the same kind of container and window for each sample. Consistency and careful attention to detail are keys to accurate measurement.

PLACE FILM

Place a circle of polypropylene film on top of an XRF sample cup. This film goes on the end of the cup with the indented ring. Thermo recommends preparing the cup ahead of time, if possible.

SECURE FILM



Secure the film with the collar. The flange inside the collar faces down and snaps into the indented ring of the cup. Inspect the installed film window for continuity and smooth, taut appearance.

FILL CUP

Set the cup on a flat surface film-window-side down. Fill it with at least five grams of the prepared sample, making sure that no voids or uneven layers.

TAMP SAMPLE

Lightly tamp the sample into the cup. The end of the pestle makes a convenient tamper.

PLACE FILTER

Place a filter-paper disk on the sample after tamping it.

STUFF CUP

Fill the rest of the cup with polyester fiber stuffing to prevent sample movement. Use aquarium filter or pillow filling as stuffing. A small supply of stuffing comes with your bulk sample kit.

CAP CUP

Place a cap on your cup.

LABEL CUP

Place a label on teh cup. Using a pen with indelible ink, write identifying information on the cup. Keep a record of the sample designation, the site and location, the date of the sample, and any other relevant comments.

Cup is ready for testing.

Preparing Liquids and Sludge

Liquids

Fill an XRF sample cup with the liquid to be tested (do not pad the sample with cotton). The cup must be full so it is best if some liquid is allowed to overflow when the cap is put on.

Sludge

Sludge can be placed directly into an XRF cup for screening. This is considered in-situ testing because no attempt has been made to prepare the sample. For more accuracy, the sludge can be dried, sieved, and ground. Prepare in an XRF sample cup and test the same way you would with a soil sample. For risk analysis, it is advisable to use a 60-mesh sieve to isolate and test only fine particles.

Preparing Mining Samples

Examine the site for differences in surface characteristics before sampling. Valid results depend on a sufficient and appropriate selection of sites to sample. Incorrect sample collection may give rise to misleading or meaningless results, regardless of the analysis method. Delineate sections with different characteristics and treat them as different areas. It may be desirable to subdivide larger areas even if they have the same characteristics to ensure a thorough examination. Make certain to label each bag thoroughly. Common information included on each bag includes the person and/or the company who collected the sample, the location and area where the sample was taken, and the date the sample was collected.

Prepared sample analysis is the most accurate method for determining the concentration of elements in a bulk medium using the instrument. Sample preparation will minimize the effects of moisture, large particle size, variations in particle size and sample non-homogeneity.

Note More sample preparation (drying, milling and sifting) will yield greater accuracy. The drier, finer, and more homogeneous the particles, the better the measurements.

Specimen Preparation - Fused Glass Disk

The samples need to be predried for 2-6 hours in 105°C depending on the moisture content.

1. Grind the dried samples to ~200mesh (74 ?m).

2. Calcination (Ashing) the sample

a. About 4-6 g of dry pulverized sample is calcinated in an alumina or platinum crucible in a muffle furnace at 1000°C for 1 hour.

b. The sample is cooled in a dedicator and loss on ignition (LOD) is calculated from weight difference before and after Calcination.

3. Weight 1.0g of calcinated sample into fusion crucible add 5.0 g of lithium tetraborate and 0.3 lithium fluoride, and 10-20 mg lithium bromide as a nonstick agent.

4. Fuse in a fluxer for at least 4 min in the flame.

5. The resulting disk is released from the mold, cooled, then presented to the spectrometer.

Specimen Preparation - Pressed Powder Briquette Preparation

1. Thoroughly remix the sample in its jar by rotating in a figure-eight motion with two hands

2. Weight 7.0g of sample into weighting boat by taking several separate gram-size portions then fine grind sample using a swing mill.

3. Add 2 small drops of propylene glycol on the top of the powder sample in the mill as a grinding aid, grind 4min at 1000rpm to obtain 10 ?m particle size.

4. Add 0.5g binder to the sample and continue grinding for 30sec more.

5. Brush the finely grounded samples into 31 mm aluminum sample cap and press at 50,000psi for 1 min.

CAUTION All test equipment must be kept clean to prevent contamination of samples.

Setting Up Beep Times

Selecting the Measurement Parameters icon allows you to set up Beep Times, enabling changes to the beep settings for various modes. This option allows you to change the beep settings for different modes independently. Select Mode you want to change, then the Measurement Parameters icon to set up your preferred beep times.

First Beep

This option allows you to change the seconds of delay before the First Beep. Select the screen button labeled with the number of seconds of delay for the First Beep. The Beep One Time editor will open. Clear the current number of seconds with the "C" button, then select the E button to enter the information.

Second Beep

This option allows you to change the seconds of delay before the Second Beep. Select the screen button labeled with the number of seconds of delay for the Second Beep. The Beep Two Time editor will open. Clear the current number of seconds with the "C" button, then select the E button to enter the information.

Third Beep

This option allows you to change the seconds of delay before the Third Beep. Select the screen button labeled with the number of seconds of delay for the Third Beep. The Beep Three Time editor will open. Clear the current number of seconds with the "C" button, then select the E button to enter the information.

Beep on Grade Match

Selecting this option will enable a special beep when the reading chemistry matches an alloy grade, and put a check mark in the box. Selecting the box again will remove the check mark and turn the beep off

Sorting the Custom Element Display

Select the Custom Element Display icon to configure sorting criteria used for analysis display. Select the mode you wish to change, then selecting the Custom Element Display icon opens up the Custom Element Display Screen.

On the left of the display are elements, each with its currently selected display option beside it to the right. The element list is ranked by importance, with the most important element on top, and each one lower down of less importance than the one above it.

By selecting an element and using the arrow buttons to the right of the list, you can change its ranking. Use the Up Button to move an element one rank closer to the top with each click. Use the Dn Arrow Button to move an element one rank closer to the bottom with each click.

Display Options

The Display Options Drop Down Menu allows you to change the display status of any element to one of three states:

- Normal - The standard state. Element displays only when the elemental value is greater than the limit of detection.
- Always - Always display the results for this element. Use this state for elements critical to all of your analyses.
- Never - Never display the results for this element. Use this state for elements which are unimportant to your work. This makes your instrument display less complex.

Select the element you want to change, then select the menu option corresponding to your choice of display status. The currently selected element is displayed in white on black.

Select the Save Button to save your current status as the new default. Select the Reset button to reset the settings to the previously saved state. Select the Close button to exit the screen without saving.

Report Settings

Under Electronics Metals, Plastics, and Test All Modes, A field called Report Settings is available. Selecting the triangle next to the Report Settings Field will open a pop up menu allowing you to choose between the three Report Settings Modes. Select the mode you wish to edit.

Changing the settings for one analysis mode will not affect the settings for other modes, and the configurations can be saved independently.

RoHS Option

When the RoHS Option is selected, clicking on the Pass and Fail values works as it does in any other Mode.

Detection Option

When the Detection Option is selected, Selecting the Pass/Fail field for that element acts as an On/Off Toggle, which will switch Pass/Fail mode between On and Off for the selected element. Selecting it again will reverse the toggle.

Consumer Products Option

When the Consumer Products Option is selected, clicking on the Pass and Fail values works as it does in any other Mode. In addition, the total of Cl+Br is also calculated and used for Pass/Fail Testing.

Max Measure Time

Under the Method Setup -> Measurement Parameters option is a field called Max Measure Time. Here you can set up the maximum time your analyzer will continue to analyze the sample. Select the Max Measure Time field, and a Virtual Numeric Keypad will pop up, allowing you to input a new Maximum Measurement Time in seconds. The default Max Measure Time is set to 300 seconds.

Minumum Test Time

Under the Method Setup -> Consumer Goods option is a field called Minimum Test Time. Here you can set up the minimum time your analyzer will continue to analyze the sample when using the Detection Option only. Select the Minimum Test Time field, and a Virtual Numeric Keypad will pop up, allowing you to input a new Minimum Test Time in seconds. The default Minimum Test Time is set to 60 seconds.

Virtual Keyboard

Whenever you see the Keyboard Icon, you can select it to bring up a Virtual Keyboard on your touch screen. Generally, selecting the keys on the Virtual Keyboard will type the corresponding character into the field. The exceptions are the meta-keys Del, Clear, Left, Right, Shift, Backspace, Cancel, and Enter.

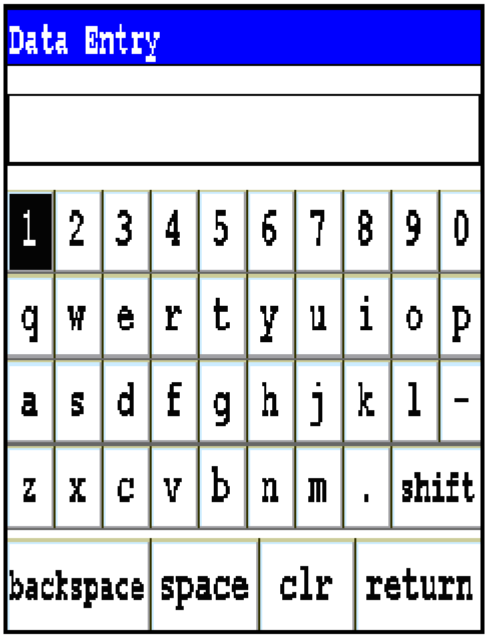


Figure 8. Virtual Keyboard

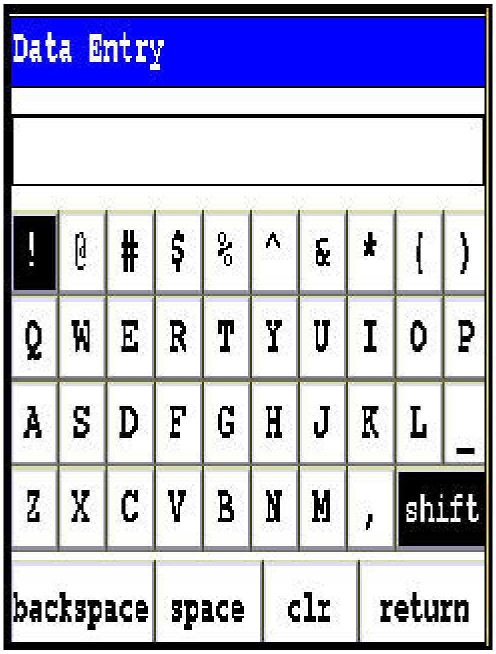


Figure 9. Shifted Virtual Keyboard

Del

Del is the Delete Key. Selecting this key will delete the character to the left of the cursor.

Clear

Clear is the Clear Key. Selecting this key will clear all characters from the field.

Left

Left is the Left Cursor Key. Selecting this key will move the cursor one space to the left.

Right

Right is the Right Cursor Key. Selecting this key will move the cursor one space to the right.

Shift

Shift is the Shift Key. Selecting this key will bring up the alternate, shifted keyboard. See Figure 1-1B. Selecting the Shift Key on the shifted keyboard will bring up the normal keyboard. See Figure 1-1A.

Backspace

Backspace is the Backspace Key. Selecting this key will delete the character to the right of the cursor.

Cancel

Cancel is the Cancel Key. Selecting this key will return you to the normal screen without inputting your changes into the field.

Enter

Enter is the Enter Key. Selecting this key will return you to the normal screen, replacing the former contents of the field with the changes you have made.

Setting Display Units

Select the Display Units radio buttons on the Set Display Units page to choose between ppm (parts per million) and percentage (hundredths of whole) displays when taking readings, and to change the Sigma value you want for the reading.

In the Display Units area, you can select between Percent composition and Parts per Million as the units displayed in a measurement, and you can change this setting independently for any mode. You can also change the Sigma for each of these modes independently. When you have changed the display units to the appropriate values, select the Close button to save these settings for use.

Changing Precision (Sigma Value)

Sigma is the symbol used for Standard Deviation, a measure of how much a set of numbers deviates from the mean. For example, each of the three data sets {0, 0, 14, and 14}, {0, 6, 8, and 14} and {6, 6, 8, 8} has a mean of 7. Their standard deviations are 7, 5, and 1, respectively. The third set has a much smaller standard deviation than the other two because its values are all close to 7. In a loose sense, the standard deviation tells us how far from the mean the data points tend to be. The number of standard deviations between the process mean and the nearest specification limit is given in sigmas. As process standard deviation goes up, or the mean of the process moves away from the center of the tolerance, the sigma number goes down, because fewer standard deviations will then fit between the mean and the nearest specification limit.

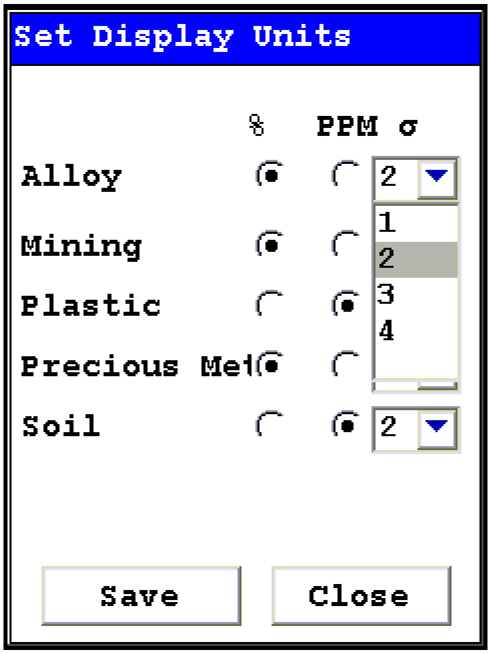
Confidence Intervals

Confidence intervals assume that the data are from an approximately normally distributed population - generally, sums of many independent, identically distributed random variables tend towards the normal distribution as a limit. Using this assumption, about 68 % of the values must be within 1 standard deviation of the mean, about 95 % of the values must be within two standard deviations, about 99.7 % must lie within 3 standard deviations, and about 99.99% of the values must lie within 4 standard deviations.

The greater the sigma value of the test, the more confident you can be that the sample is as it appears, but the more difficult and time consuming the testing must be to verify this. That's why it's important to use the most appropriate sigma value for the test. By adjusting the sigma value for each type of test, you can optimize the process for your needs.

Adjusting the Sigma Values

The sigma values are listed in the column headed with the Greek letter "sigma". The default value is 2 sigma. You can change this value by selecting the down arrow next to the value, which opens up a drop-down menu from which you can select the desired sigma value by clicking on it.



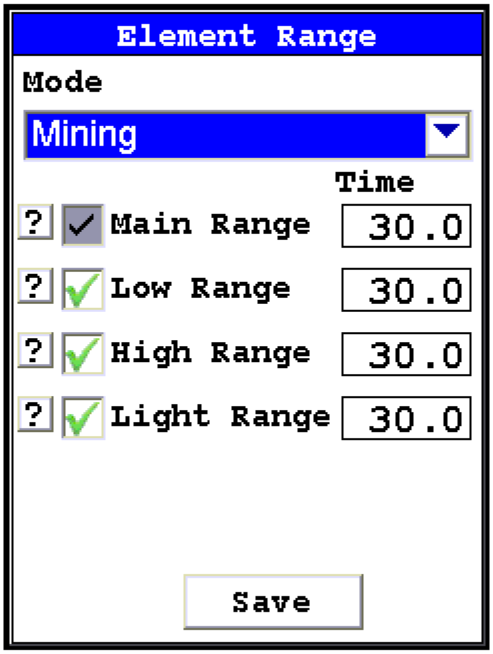
Set Display Units

	%	PPM	σ
Alloy	●	○	2
Mining	●	○	
Plastic	○	●	
Precious Met	●	○	
Soil	○	●	2

Figure 10. Selecting the Sigma Value

When you have changed the sigma values to the appropriate number, select the Close button to save these settings for use.

Adjusting the Element Range



Mode: Mining

			Time
?	☑	Main Range	30.0
?	☑	Low Range	30.0
?	☑	High Range	30.0
?	☑	Light Range	30.0

Figure 11. Adjusting the Element Range

Multi-Range tests are used to either preferentially excite specific elements for increased sensitivity, or to cover a wider element range than one Range alone can provide. Most modes, when enabled, will use several Ranges in sequence to produce a combined analysis result. In typical Metals analysis applications, Main Range is used for the analysis of most elements, Low Range is utilized for the subsequent high sensitivity analysis of V, Ti, and Cr, High Range is is used to optimize the sensitivity for the elements from Palladium (Pd) through Barium (Ba), and Light Range is typically used in light element analysis. Multi-Range switching can be set to activate off time alone, or, when time switching is disabled, off settings in the General Metals grade library. In most modes, Low and Light Range add the capability to analyze light elements which cannot be efficiently excited by Mid Range.

Select the mode you wish to configure from the Mode Menu. You can set different configurations for different modes.

The Element Range Screen enables you to directly enable or disable any Range, or control the time that a Range alters the irradiation of the sample before auto-switching to another Range.

Select the checkbox next to the Range you want to use to determine exactly which of the Ranges contained in your Analyzer is used for sample testing. Selecting an empty checkbox will enable that range and place a check into the box as an indicator. Selecting a checked box will disable the Range and clear the box.

In typical metals analysis applications, Main Range is used for the analysis of most elements. You cannot deselect the Main Range in metals analysis.

Low Range is utilized for the subsequent high sensitivity analysis of V, Ti, and Cr.

Select the Element List Button - labeled with a question mark - to display the Element List for that Range. This list shows the elements that the Range is best designed to detect.

Select the Range Time field for the intended range to change the switch time for that range. The Range Time Editor will appear. This enables you to set the number of seconds each enabled range is allotted before auto-switching will occur when needed during sample testing. Your analyzer will auto-switch from one range to another when the testing time for that range is greater than or equal to the time you have chosen, and the identified alloy is flagged as needing the switch in the Niton Alloy Library.

Select the C button to clear the current time, then from the virtual numeric key pad, select each digit you want to input, then select the E button to enter.

Setting the Date and Time

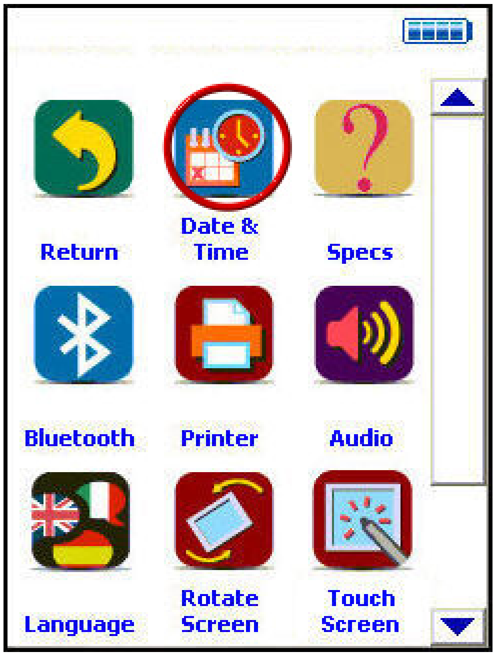
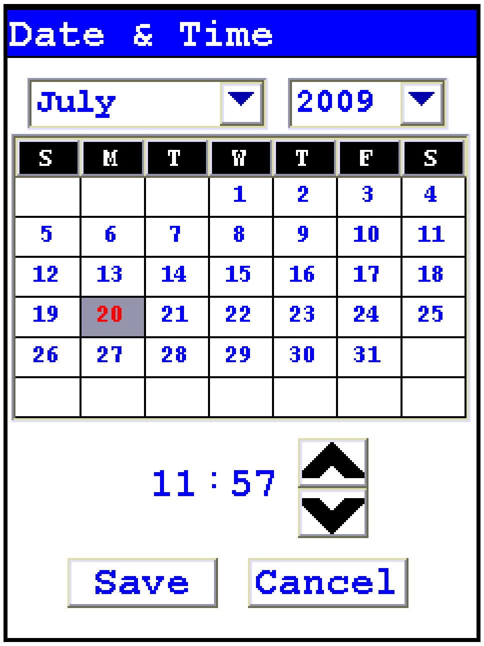


Figure 12. Setting the Date and Time

From the System Menu, select the Date & Time icon from the System Screen to set the date and time as needed for different time zones, daylight savings time, or any other reason. The date and time are factory preset prior to shipping. The clock is a 24 hour clock, so add 12 to PM hours - i.e. 1:13 PM would be 13:13.



Date & Time

July

2009

S	M	T	W	T	F	S
			1	2	3	4
5	6	7	8	9	10	11
12	13	14	15	16	17	18
19	20	21	22	23	24	25
26	27	28	29	30	31	

11 : 57

Figure 13. The Date & Time Screen

When the Date & Time button is selected, the Date & Time Screen comes up on your analyzer's LCD Screen. You may change the Month, Year, Date, Hour, and Minute on your analyzer.

Changing the Month

To change the month, select the downward pointing triangle button next to the month displayed. A drop down menu will appear, listing the months of the year in order of appearance.

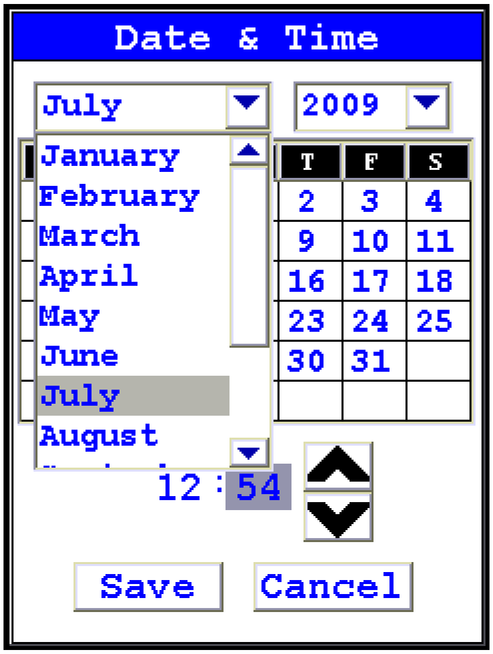


Figure 14. Month Drop Down Menu

Select the month you want from the drop down menu, using the vertical slider button to display hidden months. The display will change to show the month you selected.

Changing the Year

To change the year, select the downward pointing triangle button next to the year displayed. A drop down menu will appear, listing the years in order of appearance.

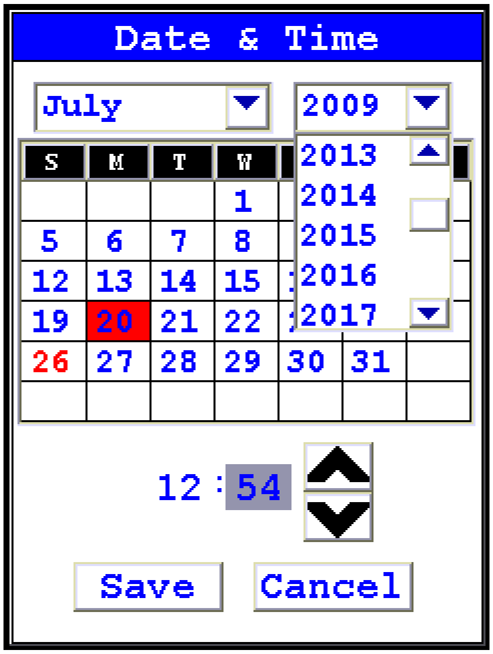
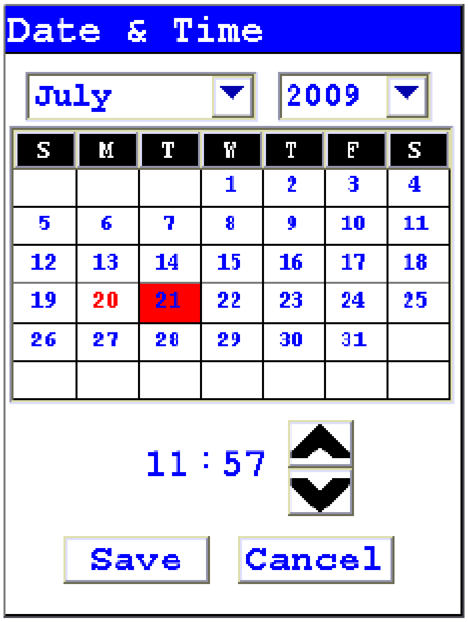


Figure 15. Changing the Year

Select the year you want from the drop down menu, using the vertical slider button to display hidden years. The display will change to show the year you selected.

Changing the Date

To change the date, select the date you want from the Date Selection Screen. The date you selected will be highlighted in red, while the old date will be shown in red numbers.



July 2009

S	M	T	W	T	F	S
			1	2	3	4
5	6	7	8	9	10	11
12	13	14	15	16	17	18
19	20	21	22	23	24	25
26	27	28	29	30	31	

11 : 57

Figure 16. Selecting the Date

Changing the Hour and Minute

To change the hour, select the hour numbers. The hour numbers will be highlighted in gray. Then select the Upwards Pointing Chevron Button to increment (increase) the hour, or the Downward Pointing Chevron Button to decrement (decrease) the hour.

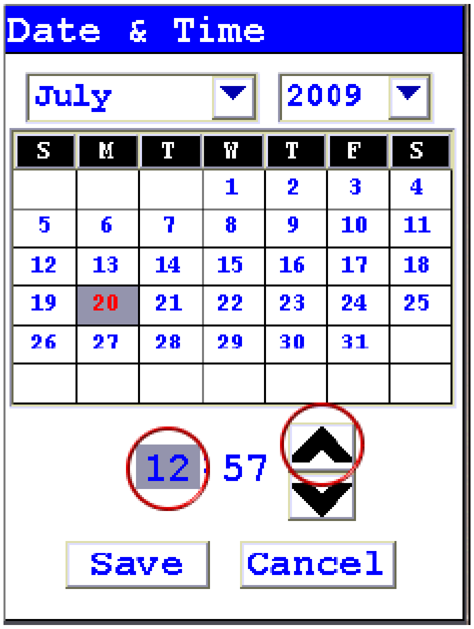


Figure 17. Changing the Hour

To change the minute, select the minute numbers. The minute numbers will be highlighted in gray. Then select the Upwards Pointing Chevron Button to increment (increase) the minute, or the Downward Pointing Chevron Button to decrement (decrease) the minute.

Figure 18. Changing the Minute

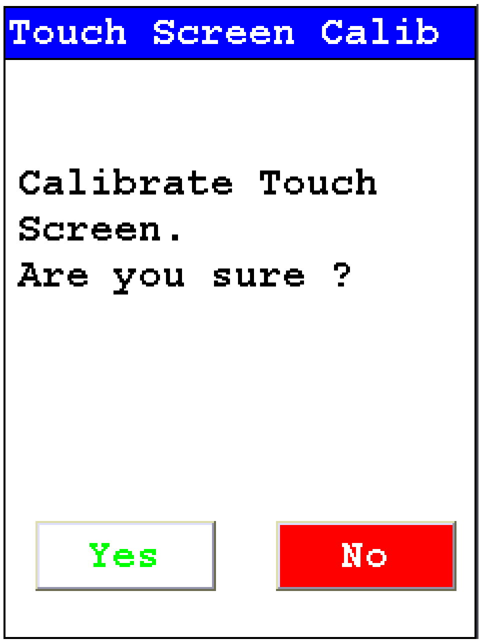
Saving Your Changes

To save your changes, select the "Save" screen Button. The display will return to the previous screen and the Date and Time will be saved.

Exiting Without Saving

To exit the screen without saving changes, select the "Cancel" Screen Button. The display will return to the previous screen and the Date and Time will not be saved.

Calibrating the Touch Screen



Calibrate Touch Screen.
Are you sure ?

Figure 19. Initiating Touch Screen Calibration

Select the Calibrate Touch Screen button from the System Screen to re-calibrate the analyzer's touch screen display. This procedure establishes the display boundaries for the touch screen interface.

1. Select the Touch Screen icon.

2. The display will show a message asking you to confirm whether or not you want to calibrate your Touch Screen. Select the Yes button.

3. The display will show the message: "Calibrate Touch Screen". There will be a small cross in the upper left-hand corner of the display.

4. Tap on this cross with the stylus, and the cross will disappear and reappear in the upper right-hand corner of the screen.

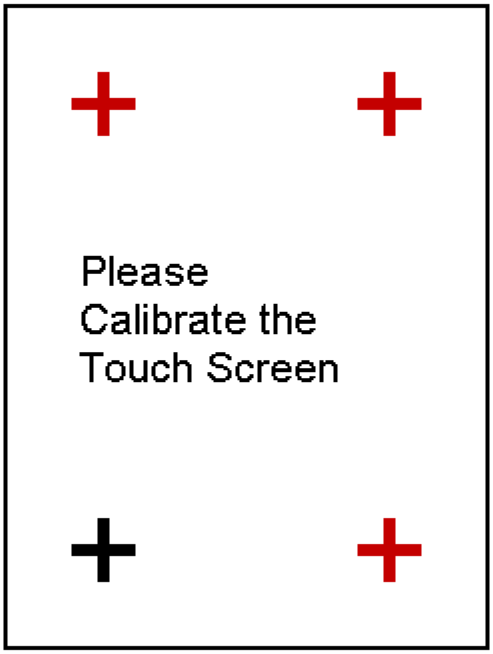
5. Tap on the cross again, and it will reappear in the lower right-hand corner of the screen.

6. Tap on the cross again and it will reappear in the lower left-hand corner of the screen.

7. Tap on the cross once more, and you will be presented with a Confirmation Screen.

8. Select the Yes Button to confirm that the parameters are good. Select the No Button to start the process again.

9. Once you have confirmed the parameters, the System Menu will be displayed. The screen is now calibrated.



Please
Calibrate the
Touch Screen

Figure 20.   Touch Screen Calibration Crosses

The Touch Screen can be calibrated - and the system operated - with a USB mouse plugged into the USB ports in the rear of teh analyzer.

Data Management

Viewing Data

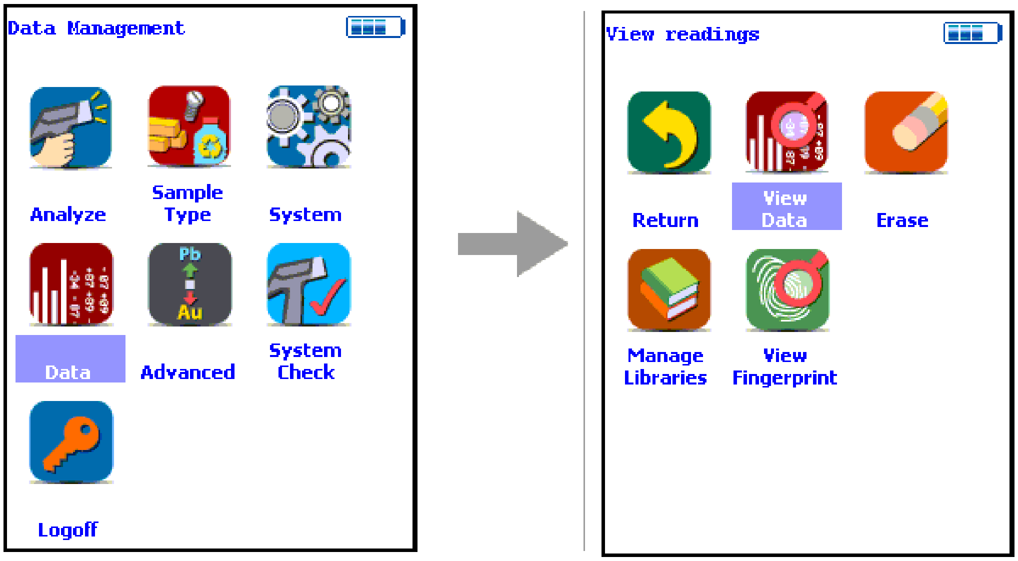
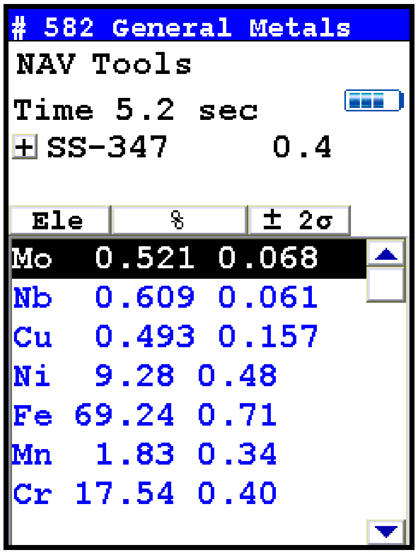


Figure 21. The View Data Menu Path

Use the Data Screen to view previously taken test result readings. When the View Data icon is selected, the Results screen of your most recent test is shown on the Touch Screen.



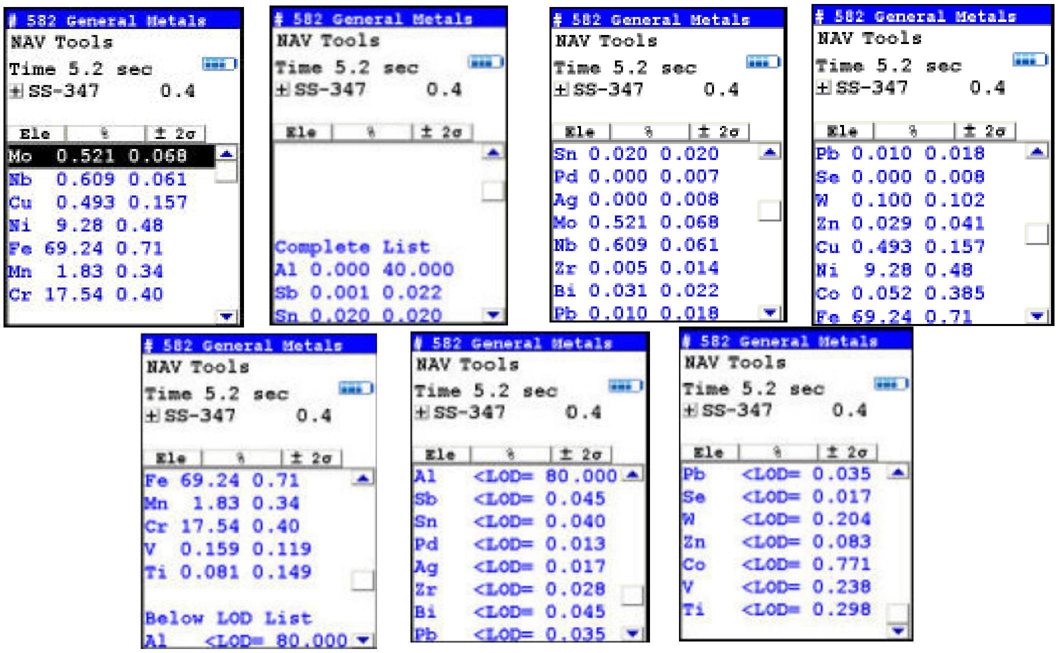
582 General Metals
NAV Tools
Time 5.2 sec
SS-347 0.4

Ele	%	± 2σ
Mo	0.521	0.068
Nb	0.609	0.061
Cu	0.493	0.157
Ni	9.28	0.48
Fe	69.24	0.71
Mn	1.83	0.34
Cr	17.54	0.40

Figure 22. The View Data Screen

Using the buttons on the control panel, you may view different readings or additional data for individual readings. Your analyzer will display the standard screen analysis. Pressing the Down Button on the 4-way touch pad will display a complete scrolling elemental chemistry listing. Each press of the Down Button scrolls the screen down to the next element. You can also use the scroll bar along the right side to scroll or page through the elements.

Scrolling Down Through the Complete Listing of Elements



```
# 582 General Metals
NAV Tools
Time 5.2 sec
+ SS-347      0.4
Ele   %    ± 2σ
Mo  0.521 0.068
Nb  0.609 0.061
Cu  0.493 0.157
Ni   9.28 0.48
Fe 69.24 0.71
Mn   1.83 0.34
Cr 17.54 0.40
```

```
# 582 General Metals
NAV Tools
Time 5.2 sec
+ SS-347      0.4
Ele   %    ± 2σ
Complete List
Al 0.000 40.000
Sb 0.001 0.022
Sn 0.020 0.020
```

```
# 582 General Metals
NAV Tools
Time 5.2 sec
+ SS-347      0.4
Ele   %    ± 2σ
Sn 0.020 0.020
Pd 0.000 0.007
Ag 0.000 0.008
Mo 0.521 0.068
Nb 0.609 0.061
Zr 0.005 0.014
Bi 0.031 0.022
Pb 0.010 0.018
```

```
# 582 General Metals
NAV Tools
Time 5.2 sec
+ SS-347      0.4
Ele   %    ± 2σ
Pb 0.010 0.018
Se 0.000 0.008
W  0.100 0.102
Zn 0.029 0.041
Cu 0.493 0.157
Ni   9.28 0.48
Co 0.052 0.385
Fe 69.24 0.71
```

```
# 582 General Metals
NAV Tools
Time 5.2 sec
+ SS-347      0.4
Ele   %    ± 2σ
Fe 69.24 0.71
Mn  1.83 0.34
Cr 17.54 0.40
V  0.159 0.119
Ti 0.081 0.149

Below LOD List
Al    <LOD= 80.000
```

```
# 582 General Metals
NAV Tools
Time 5.2 sec
+ SS-347      0.4
Ele     %     ± 2σ
Al    <LOD= 80.000
Sb    <LOD= 0.045
Sn    <LOD= 0.040
Pd    <LOD= 0.013
Ag    <LOD= 0.017
Zr    <LOD= 0.028
Bi    <LOD= 0.045
Pb    <LOD= 0.035
```

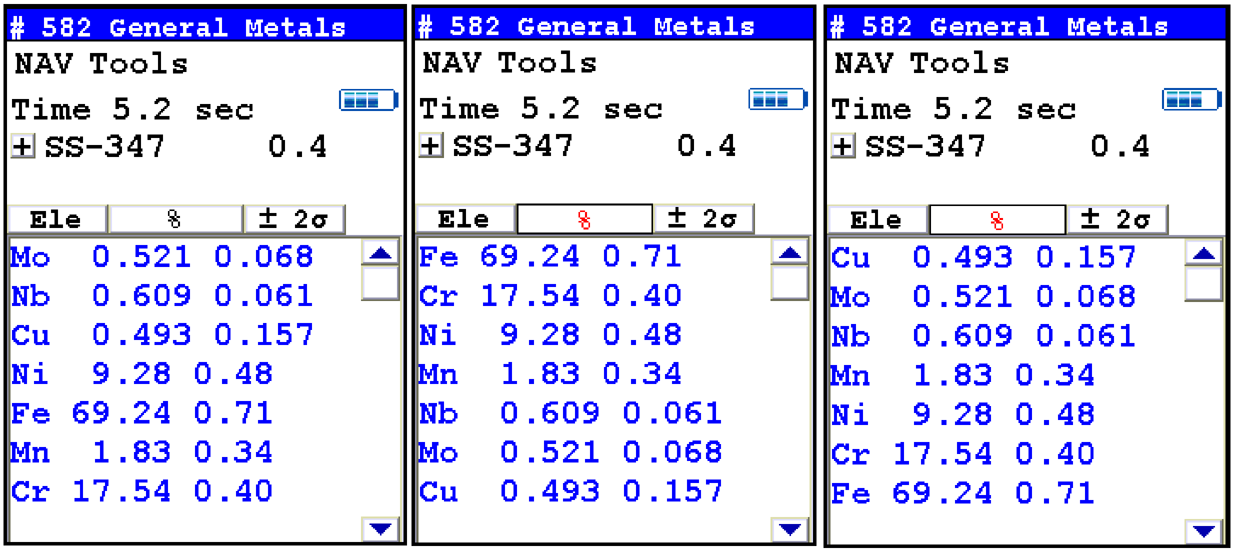
```
# 582 General Metals
NAV Tools
Time 5.2 sec
+ SS-347      0.4
Ele     %     ± 2σ
Pb    <LOD= 0.035
Se    <LOD= 0.017
W     <LOD= 0.204
Zn    <LOD= 0.083
Co    <LOD= 0.771
V     <LOD= 0.238
Ti    <LOD= 0.298
```

Figure 23. Complete Listing of Elements

Pressing the Left Button on the 4-way touch pad of your analyzer will display the previous reading, or if the first reading is currently displayed, the last reading. Pressing the Right Button on the 4-way touch pad will display the next reading, or if the last reading is currently displayed, the first reading in memory. Your analyzer can store up to 10,000 readings. You can also look at the complete x-ray spectra for each reading stored in the analyzer's memory.

Sorting Elements

You can sort element rows by various criteria in order to view your data in the manner you prefer. The Sort Buttons, which double as column headings, can be used to re-sort the data in different ways. The Data Screen always begins as a Standard Sort, as you have defined it. Selecting the appropriate Sort Button once toggles the sort order to High-to-Low. Selecting the Sort Button again toggles the sort order to Low-to-High. To return to the Standard Sort, select the Sort Button a third time.



```
# 582 General Metals
NAV Tools
Time 5.2 sec
+ SS-347        0.4
Ele    %    ± 2σ
Mo   0.521 0.068
Nb   0.609 0.061
Cu   0.493 0.157
Ni   9.28 0.48
Fe  69.24 0.71
Mn   1.83 0.34
Cr  17.54 0.40
```

```
# 582 General Metals
NAV Tools
Time 5.2 sec
+ SS-347        0.4
Ele    %    ± 2σ
Fe  69.24 0.71
Cr  17.54 0.40
Ni    9.28 0.48
Mn    1.83 0.34
Nb    0.609 0.061
Mo    0.521 0.068
Cu    0.493 0.157
```

```
# 582 General Metals
NAV Tools
Time 5.2 sec
+ SS-347        0.4
Ele    %    ± 2σ
Cu   0.493 0.157
Mo   0.521 0.068
Nb   0.609 0.061
Mn   1.83 0.34
Ni   9.28 0.48
Cr  17.54 0.40
Fe  69.24 0.71
```

Figure 24. Standard, High-to-Low, and Low-to-High Composition Sorts

Element Sorts

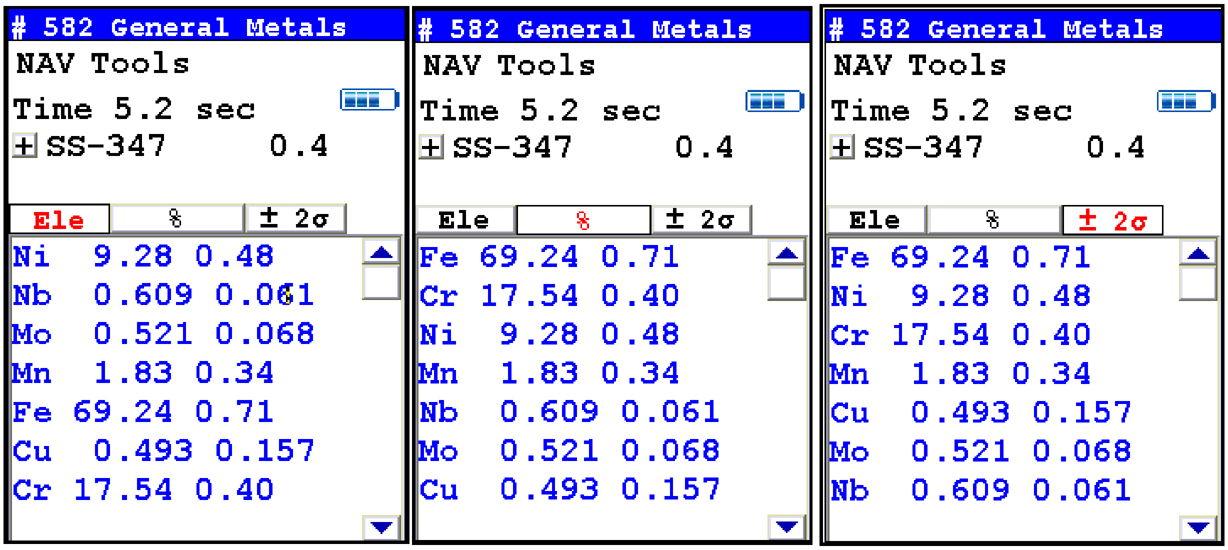
Element sorts are performed alphabetically based on the element symbol.

Composition Sorts

Composition sorts are performed numerically based on the percentage of composition, i.e. from lowest to highest concentration, or by toggling again, from highest to lowest.

Error Sorts

Error sorts are performed based on the size of the error in the reading, i.e. from largest to smallest error, or by toggling again, from smallest to largest.



```
# 582 General Metals
NAV Tools
Time 5.2 sec
+ SS-347       0.4
Ele    %    ± 2σ
Ni   9.28 0.48
Nb   0.609 0.061
Mo   0.521 0.068
Mn   1.83 0.34
Fe  69.24 0.71
Cu   0.493 0.157
Cr  17.54 0.40
```

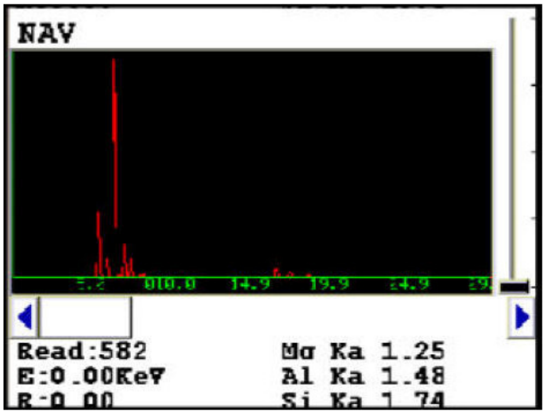
```
# 582 General Metals
NAV Tools
Time 5.2 sec
+ SS-347       0.4
Ele    %    ± 2σ
Fe  69.24 0.71
Cr  17.54 0.40
Ni   9.28 0.48
Mn   1.83 0.34
Nb   0.609 0.061
Mo   0.521 0.068
Cu   0.493 0.157
```

```
# 582 General Metals
NAV Tools
Time 5.2 sec
+ SS-347       0.4
Ele    %    ± 2σ
Fe  69.24 0.71
Ni   9.28 0.48
Cr  17.54 0.40
Mn   1.83 0.34
Cu   0.493 0.157
Mo   0.521 0.068
Nb   0.609 0.061
```

Figure 25. Element, Composition, and Error Sorts

Spectrum Graph

For any reading result, simply use the NAV Menu to gain access to the reading's spectrum graph. Selecting Spectra will show a graphed spectrum of this reading, called SpectraView. SpectraView can be a useful tool for rapid, qualitative analysis of a sample. See Viewing the Spectrum for details.



```
Read:582          Mg Ka 1.25
E:0.00KeV         Al Ka 1.48
R:0.00            Si Ka 1.74
```

Figure 26. The SpectraView Screen

Viewing the Spectrum

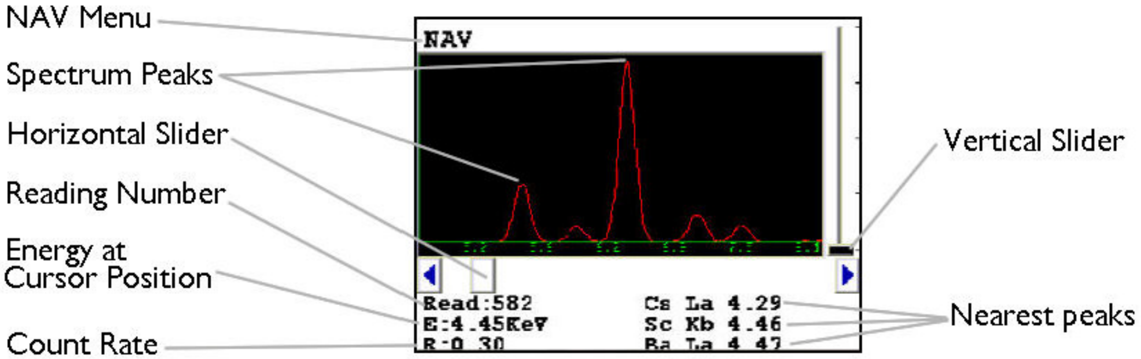
SpectraView

SpectraView enables you to to visually inspect the fluorescent x-ray peaks obtained from any sample and qualitatively identify them using the on-board software. In SpectraView Mode, the spectrum is displayed using a linear energy scale along the x-axis, with the count rate autoscaled logarithmically on the y-axis so that the highest peak on the screen reaches the top of the scale.

How to Use SpectraView

You can access the SpectraView screen after taking a measurement in any mode, or while viewing a previous measurement, by selecting Spectra from the NAV Menu. Once you are in SpectraView, you can use the up and down positions of the 4-way touch pad to scroll through the spectrum, or you can tap on the spectrum display with the stylus to place the cursor at the point you tapped. The vertical cursor line indicates the current position along the spectrum.

Viewing the Information in SpectraView Mode



```
Read:582        Cs La 4.29
E:4.45KeV       Sc Kb 4.46
R:0.30          Ba La 4.47
```

Figure 27. The SpectraView Screen

By default, the following information is shown along with the spectrum:

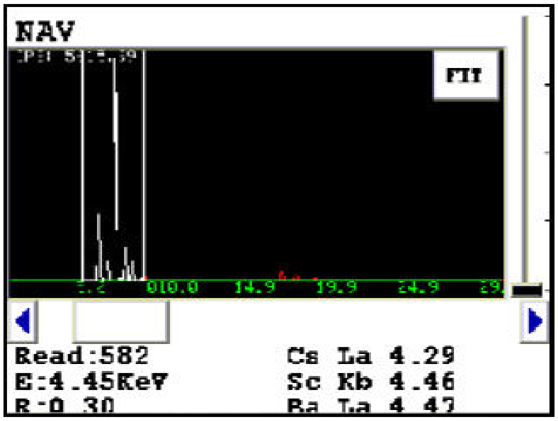
- The Reading number (Bottom Left) in the form "Read:x", where x is the Reading number.
- The position of the cursor on the energy scale (Bottom Left, under the Reading number), in the form "E: x.xx KeV", where KeV is kilo electron volts.
- The count rate (Bottom Left, under the energy position), in the form “R:x.xx”.

- Ka, Kb, La, Lb, and/or Lg peaks of the three elements closest to where your cursor is positioned on the energy scale (Bottom Right). This information is written with the element symbol first, followed by either Ka (K shell alpha peak), Kb (K shell beta peak), La (L shell alpha peak), La (L shell beta peak), or Lg (L shell gamma peak). An example would be "Al Ka 1.48." To determine if a given element is present, look at the count rate at that cursor position.

Note SpectraView cannot be used to determine exact element percentages in a sample.

Fitting the Spectrum

By using the touch screen, you can select parts of the displayed spectrum and zoom in. Touch and hold the stylus to the screen immediately before the area of the spectrum you wish to enhance, then - still holding the stylus to the screen - sweep it across the area of the spectrum you wish to see closer, lifting the stylus from the screen when you pass the end of the area of interest. The screen will display vertical lines to either side of the area of interest, delineating the boundaries of the area.

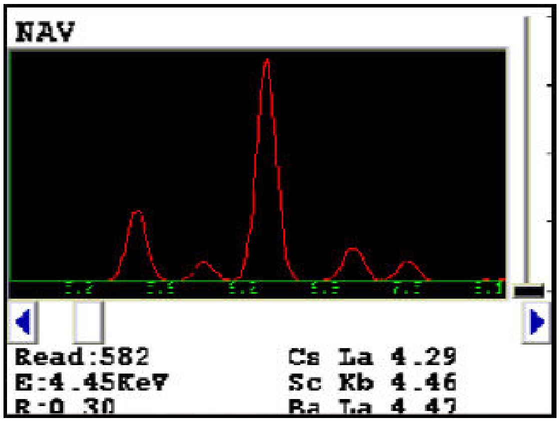


Read:582
E:4.45KeV
R:0.30

Cs La 4.29
Sc Kb 4.46
Ba La 4.47

Figure 28. **Delineating the Area of Interest**

Select the FIT button in the upper right hand corner of the Spectrum to fit the area of interest to the display area.



Read:582
E:4.45KeV
R:0.30

Cs La 4.29
Sc Kb 4.46
Ba La 4.47

Figure 29. Area of Interest Fit to the Display

The view of the spectrum will change to show only the area of interest.

Multiple Ranges

SpectraView can display any individual spectra, including those obtained from multiple Ranges (filters) if you are using more than one Range. Use the NAV Menu to select which spectrum to view.

The Spectra1 choice will display the spectrum produced by the first Range.

The Spectra2 choice will display the spectrum produced by the second Range.

SpectraView Navigation

Use the left button on the 4-way touch pad to expand the spectrum, centered on the position of the cursor.

Use the right button on the 4-way touch pad to contract the spectrum, centered on the position of the cursor.

Viewing Fingerprints

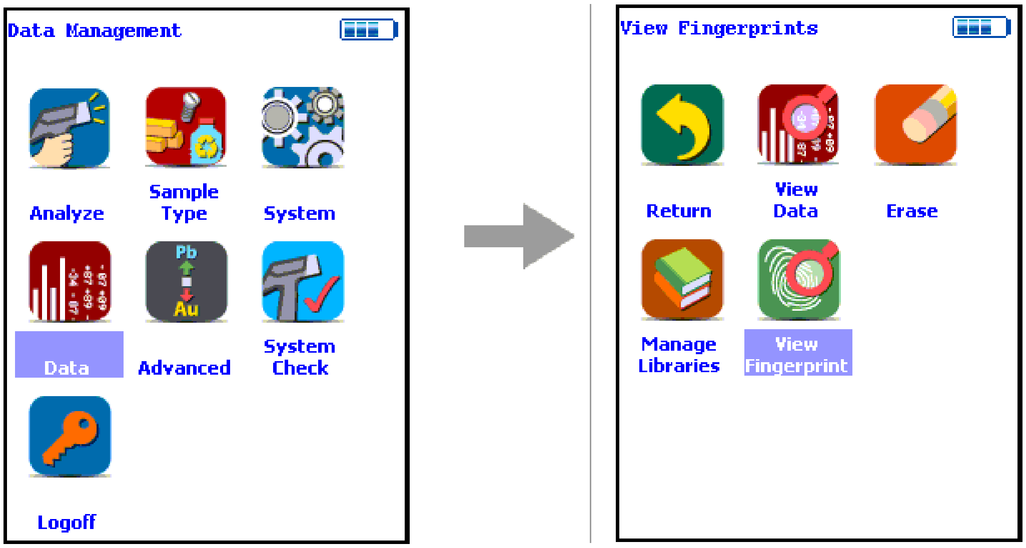
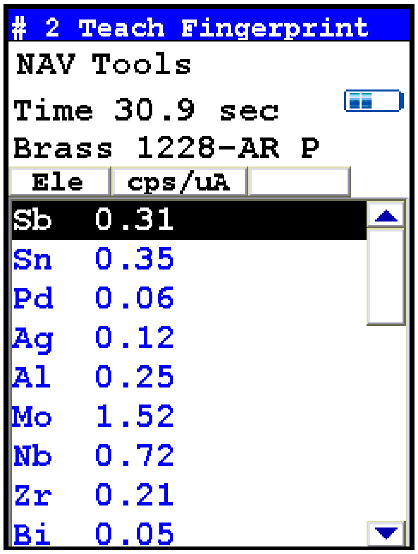


Figure 30. The View Fingerprints Menu Path

Select the View Fingerprints icon to view data saved as reference sample Fingerprints in Teach Fingerprint Mode. When the View Fingerprints icon is selected, the Results Screen of your most recent Teach Fingerprint is shown on the Touch Screen display.



```
# 2 Teach Fingerprint
NAV Tools
Time 30.9 sec
Brass 1228-AR P
 Ele   cps/uA
Sb   0.31
Sn   0.35
Pd   0.06
Ag   0.12
Al   0.25
Mo   1.52
Nb   0.72
Zr   0.21
Bi   0.05
```

Figure 31. The View Fingerprints Screen

Erasing Data

The Erase All Data Screen

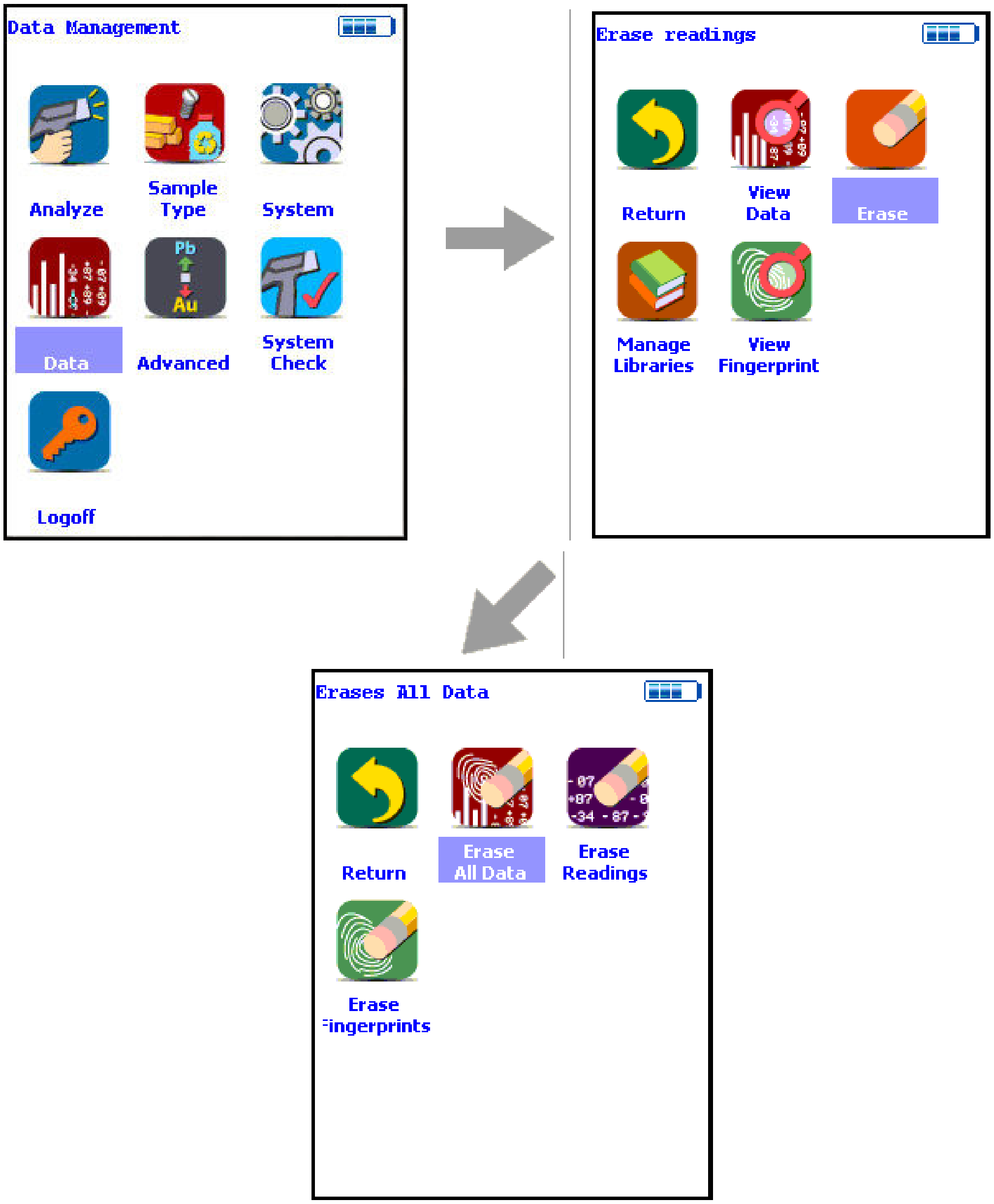


Figure 32. The Erase All Data Menu Path

Select the Erase All Data icon to erase all data, including signatures and readings, from your analyzer. Selecting the Erase All Data icon will bring up a confirmation screen asking you “Are you sure?” with options to select “YES” or “NO”. Selecting the Yes Button will erase all data from your analyzer. Selecting the No Button will return you to the Erase Menu.

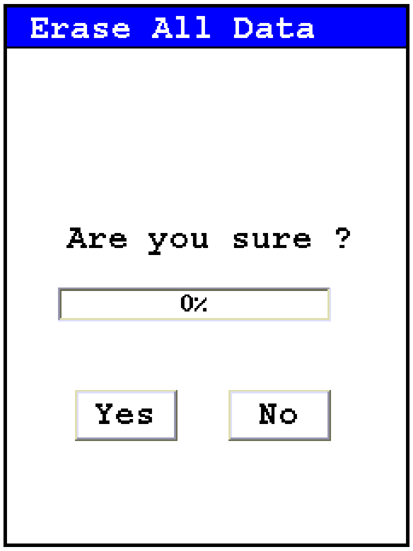


Figure 33. The Erase All Data Confirmation Screen

CAUTION Never turn off the analyzer while data is being erased!

WARNING Do not attempt to take measurements while downloading readings! This will generate an error requiring a system reset, and may corrupt your stored readings, requiring all stored readings to be erased.

The Erase Readings Screen

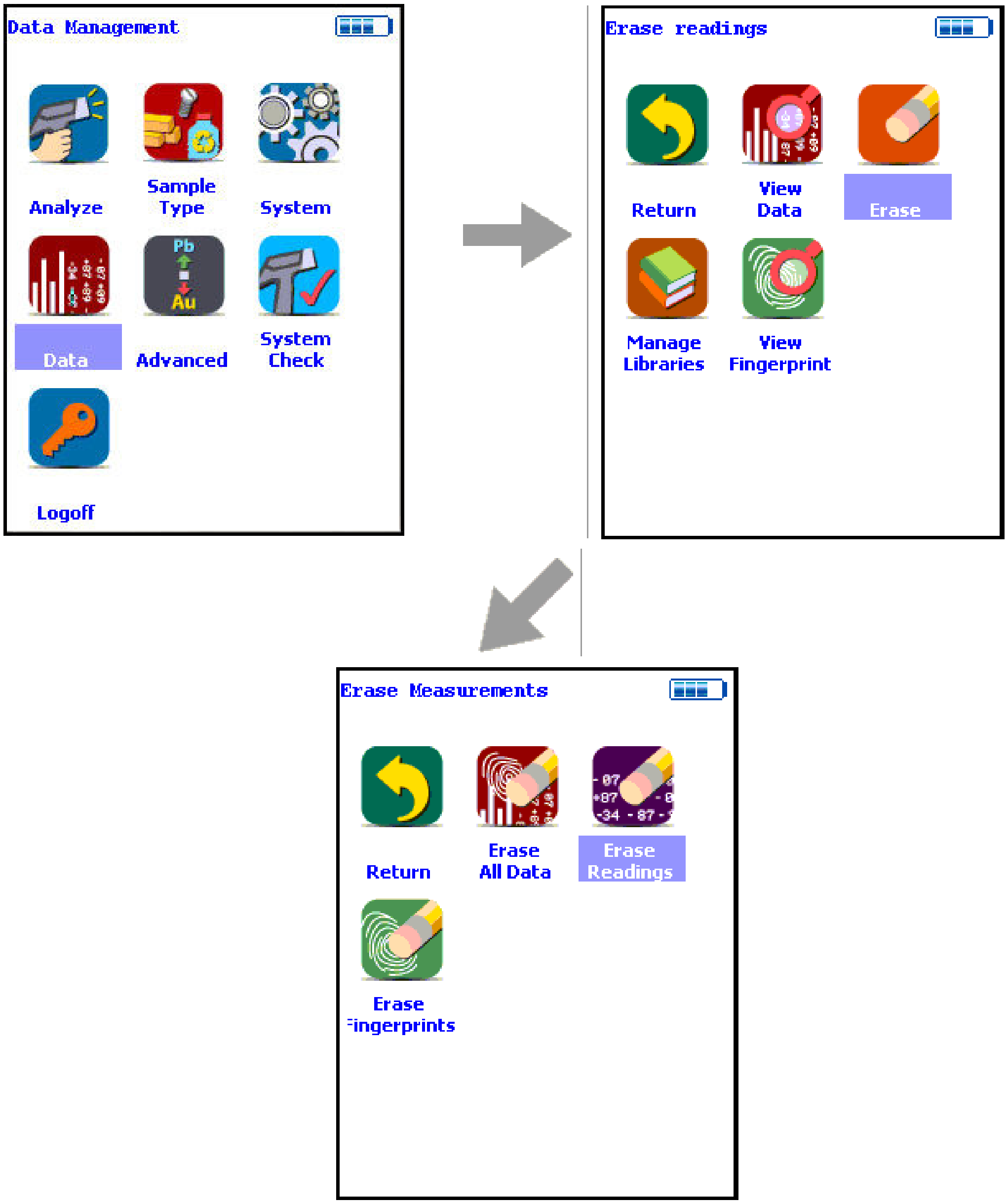


Figure 34. The Erase Readings Menu Path

Select the Erase Readings icon to erase all accumulated test readings from your analyzer. Selecting the Erase Readings icon will bring up a confirmation screen asking you “Are you sure?” with options to select “YES” or “NO”. Selecting the Yes Button will erase all test reading data from your analyzer. Selecting the No Button will return you to the Erase Menu.

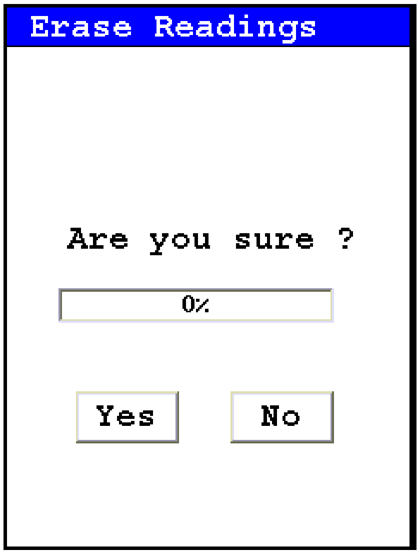


Figure 35. The Erase Readings Confirmation Screen

Note - We recommend that you download all your readings into an NDT file for recording purposes before erasing all data.

The Erase Fingerprints Screen

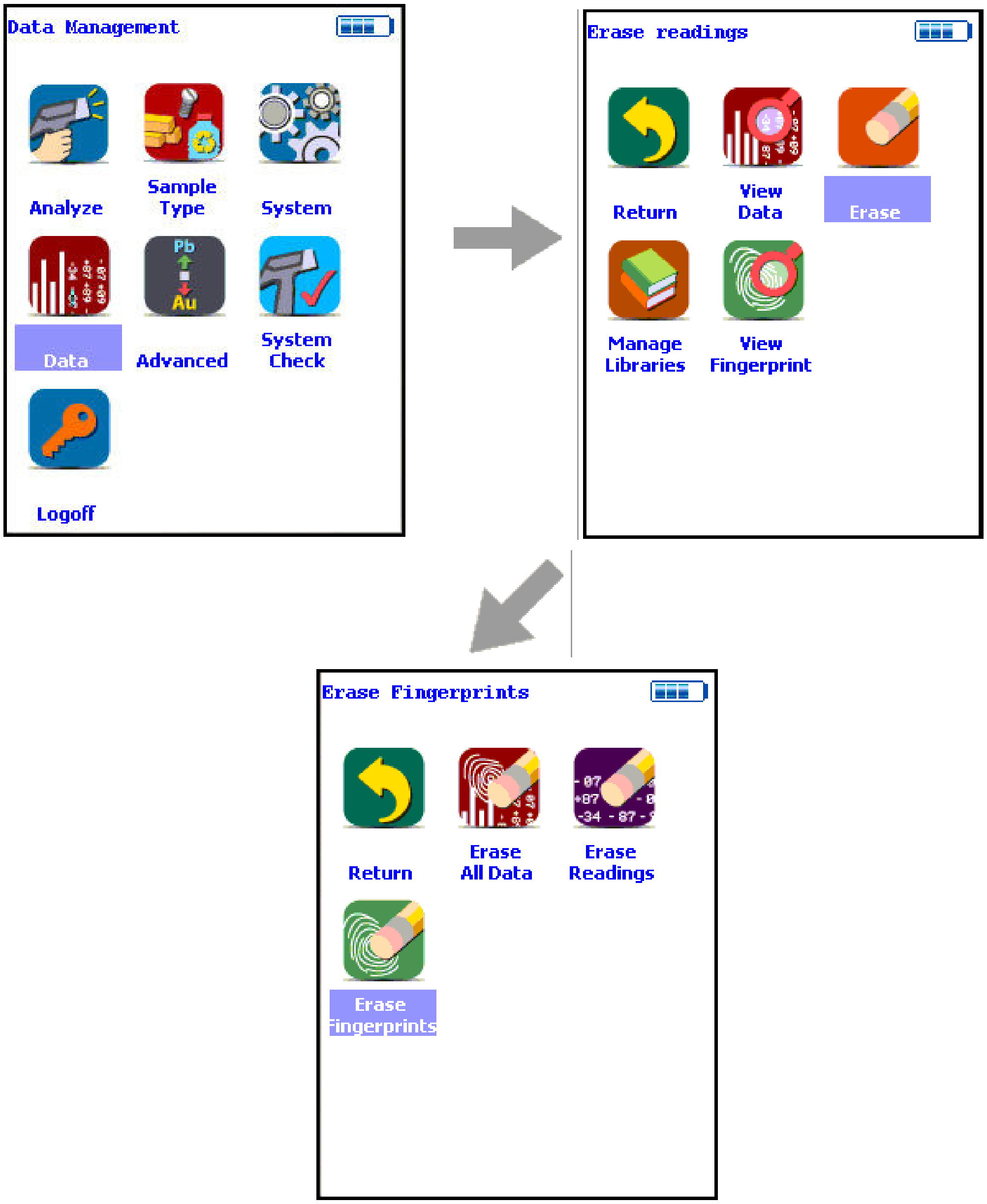


Figure 36. The Erase Fingerprints Menu Path

Select the Erase Fingerprints icon to erase all accumulated alloy fingerprints from your analyzer. Selecting the Erase Fingerprints icon will bring up a confirmation screen asking you “Are you sure?” with options to select “YES” or “NO”. Selecting the Yes Button will erase all fingerprint data from your analyzer. Selecting the No Button will return you to the Erase Menu.

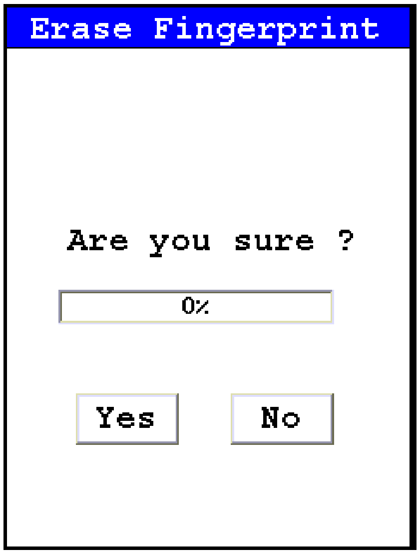


Figure 37. The Erase Fingerprints Confirmation Screen

Managing Libraries

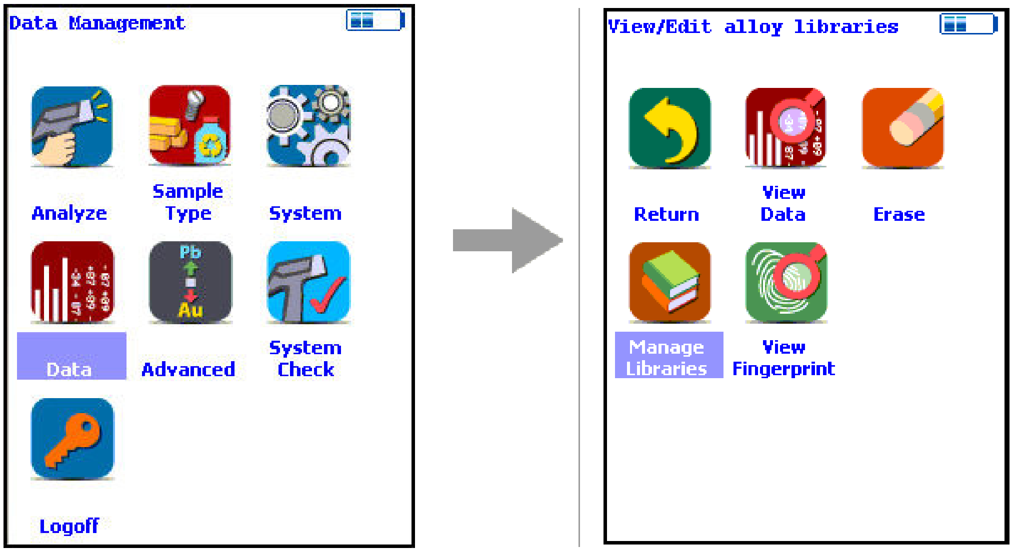


Figure 38. The Manage Libraries Menu Path

Select the Manage Libraries icon to access the Library Management Menu. The Library Management Menu allows you to view and modify data in the Primary Library as well as the currently loaded alternate libraries. Just select the library you wish to view or editfrom the list on screen.

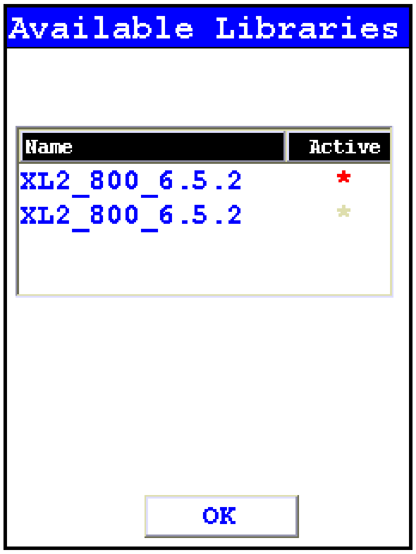


Figure 39. Viewing the Libraries

The entries in the Grade Library serve as a reference for chemistry based analysis. The library entries allow the analyzer to work properly “out of the box” without needing time-consuming pre-analysis.

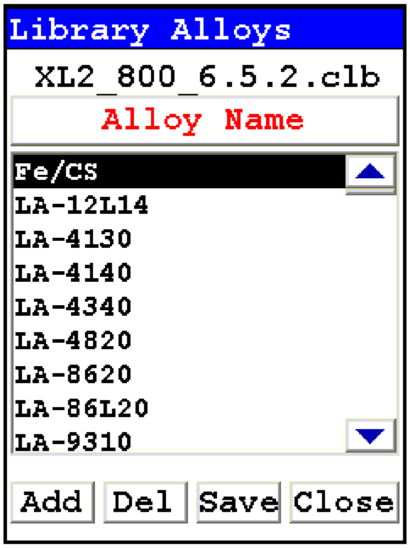


Figure 40. The Library Editor

Using the Library Editor

The Library Editor enables you to edit any library to conform to your specifications.

Alloy Name Button

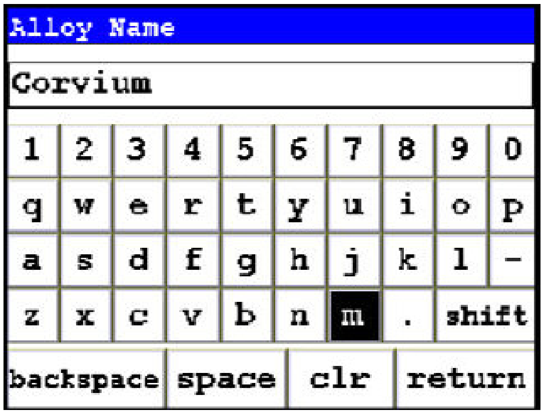
Selecting the Alloy Name Button sorts the alloy list alphanumerically.

(Name in List)

Selecting the actual name of the alloy - i.e. “Fe/CS” - will bring up the Element Specification Screen.

Add Button

Selecting the Add Button will add a new alloy to the Library. First the Alloy Name Editor will appear, enabling you to enter the name of the new alloy.



Alloy Name

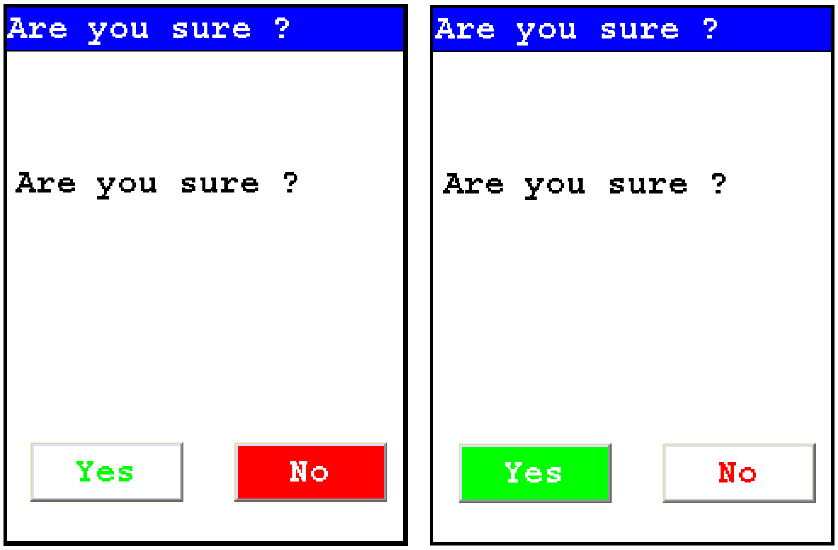
Corvium

Figure 41. The Alloy Name Editor

The Alloy Name Editor is a standard Virtual Keyboard. Use it as you would any Virtual Keyboard. Hitting the return key enters the name into the alloy list. Select the name of the new alloy to bring up the Element Specification Screen and enter the specification of the alloy.

Del Button

Selecting the Del Button will delete the currently selected alloy. First a confirmation screen appears.



Are you sure ?

Are you sure ?

Are you sure ?

Are you sure ?

Figure 42. Confirmation Screen

Selecting the Yes Button will delete the alloy from the list. Selecting the No Button will return you to the Alloy List.

Save Button

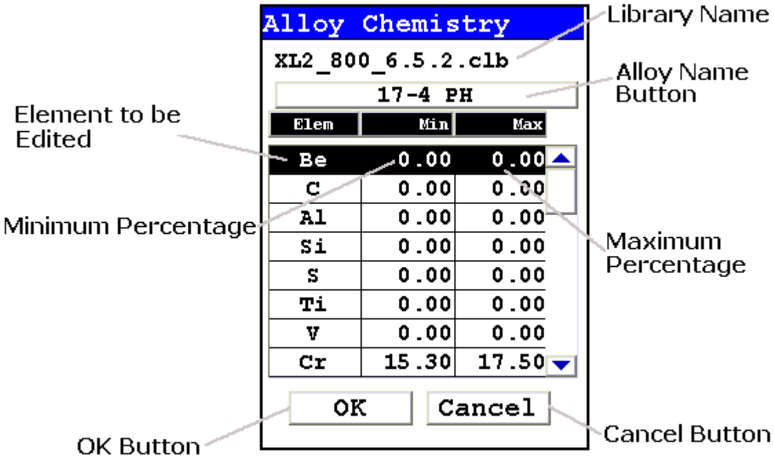
Selecting the Save button will save the current Library.

Close Button

Selecting the Close button will close the current Library without saving it.

The Element Specification Screen

The Element Specification Screen allows you to edit the elemental content of any alloy.



Alloy Chemistry

XL2_800_6.5.2.clb

17-4 PH

Elem	Min	Max
Be	0.00	0.00
C	0.00	0.00
Al	0.00	0.00
Si	0.00	0.00
S	0.00	0.00
Ti	0.00	0.00
V	0.00	0.00
Cr	15.30	17.50

Figure 43. The Element Specification Screen

Library Name

This is the name of the library you are editing. Make sure you are editing the correct library before proceeding further.

Alloy Name

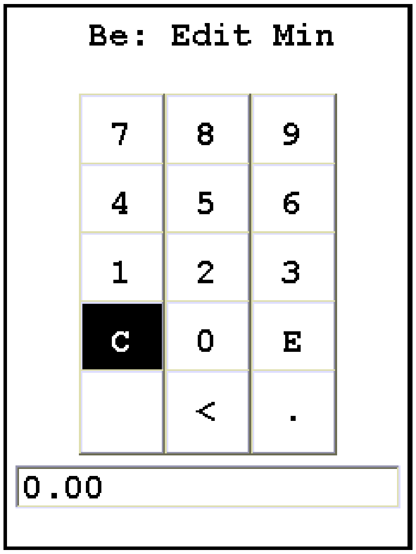
This is the name of the alloy you are editing. Make sure you are editing the correct alloy before proceeding further.

Element to be Edited

This is the element you need to edit for this alloy.

Minimum Percentage

This is the lowest amount of the element in question you want to be in the alloy. If the element in the analyzed sample is any lower, the sample will not be recognized as this alloy. Selecting the element minimum will open the Minimum Editor.



Be: Edit Min

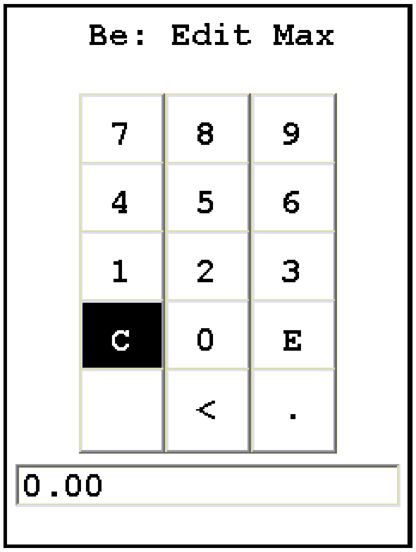
0.00

Figure 44. The Minimum Editor

This is a standard Virtual Numerical Keypad. The C Button will clear the current display, The < Button will backspace one space, and the E Button will enter this number as the minimum. After selecting the E Button, you will be returned to the Element Specification Screen.

Maximum Percentage

This is the highest amount of the element in question you want to be in the alloy. If the element in the analyzed sample is any higher, the sample will not be recognized as this alloy. Selecting the element maximum will open the Maximum Editor.



Be: Edit Max

0.00

Figure 45. The Maximum Editor

This is a standard Virtual Numerical Keypad. The C Button will clear the current display, The < Button will backspace one space, and the E Button will enter this number as the minimum. After selecting the E Button, you will be returned to the Element Specification Screen.

OK Button

Selecting the OK button will save the edited library.

Cancel Button

Selecting the Cancel button will exit the Element Specification Screen for this alloy, returning you to the Library Editor.

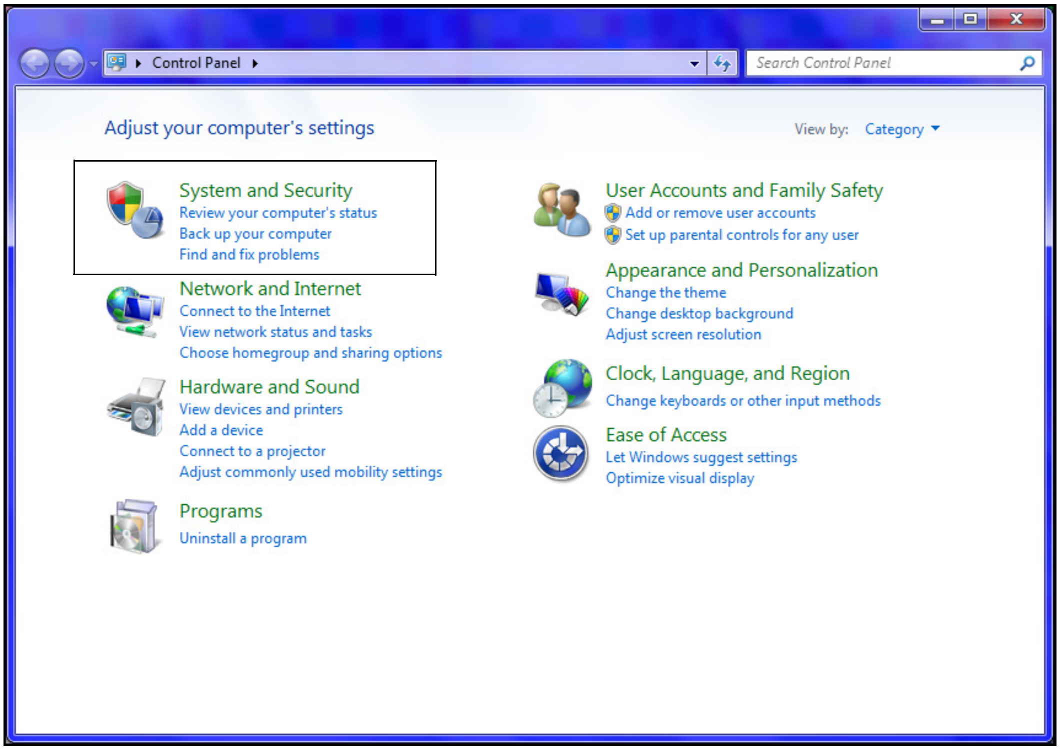
Connectivity

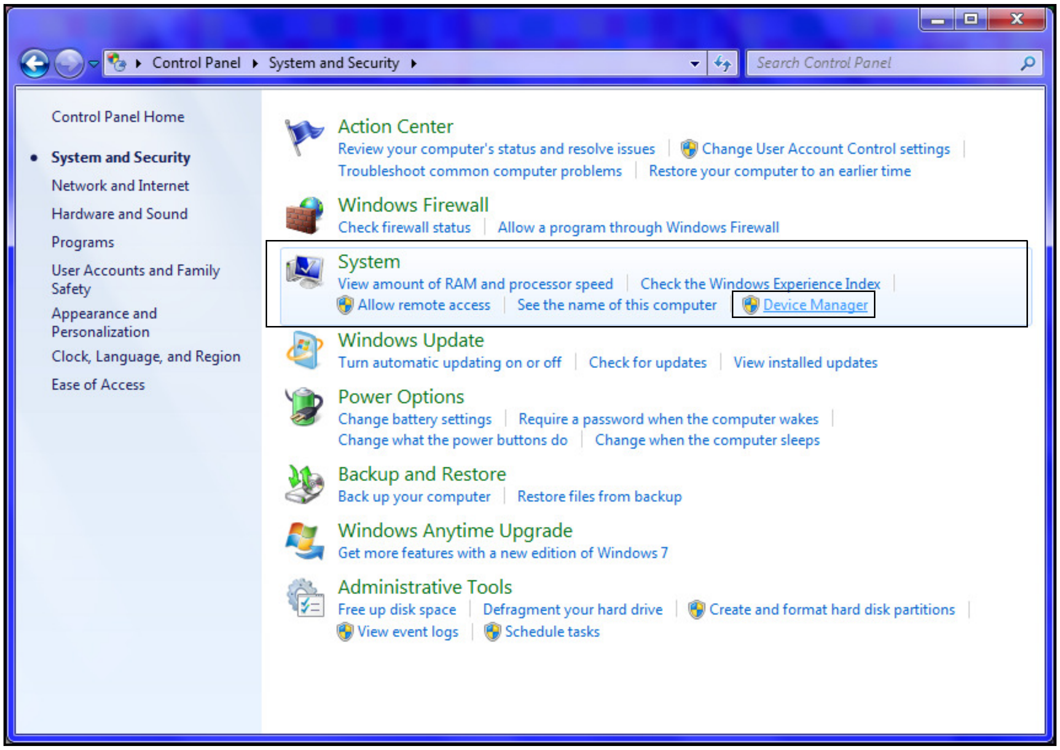
This section discusses how to connect your computer and your analyzer, for data transfer and integration, translation to other formats, data storage and security, as well as controlling your analyzer remotely through your computer. Connection can be achieved via USB, serial, and/or Bluetooth wireless means. Your analyzer comes with software which facilitates these uses, and works together with your analyzer to increase your productivity and efficiency.

Installing the Windows 7 USB Driver

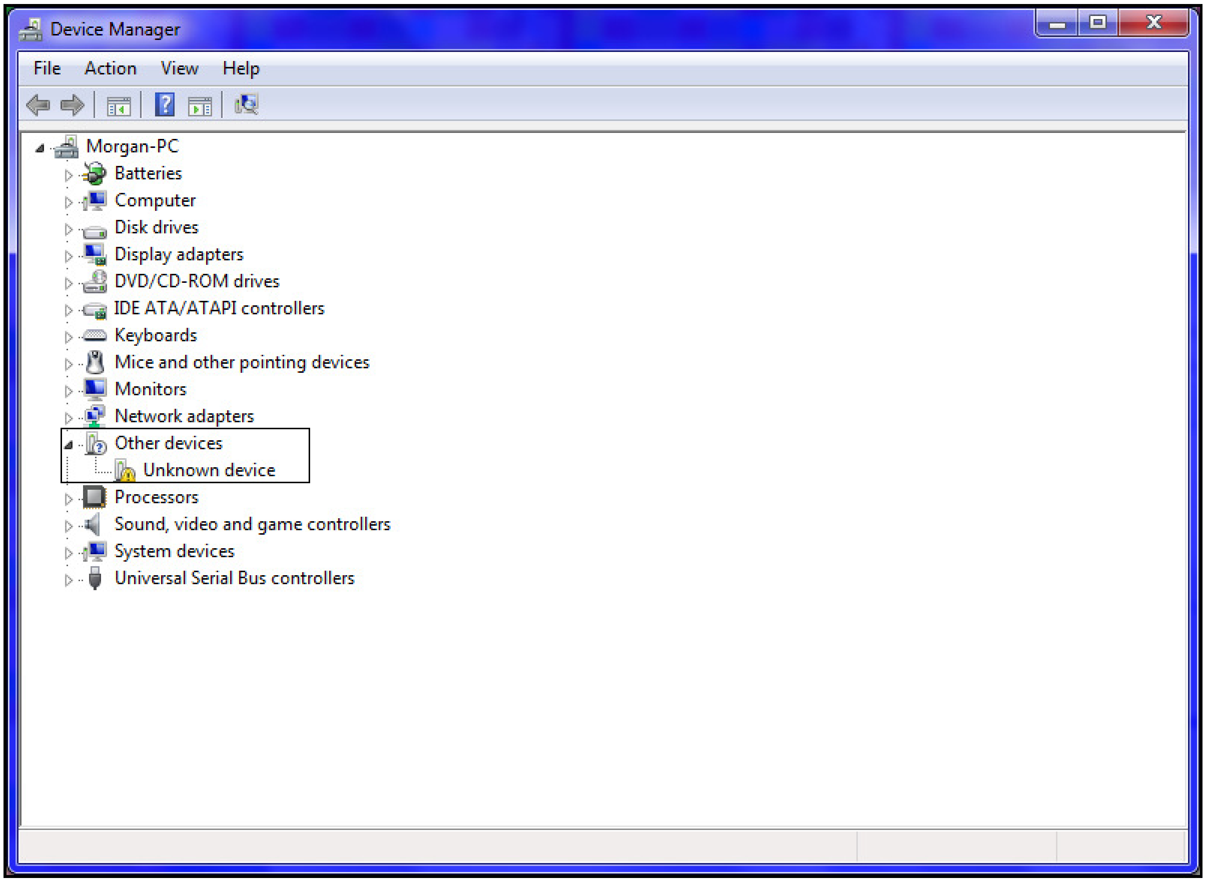
1. Insert the NDT CD and close out any dialogue box that pops up. The driver is located on this disk.

2. Click on “Control Panel” and locate the “Device Manager”. If it is not available directly under “Control Panel”, look under “System and Security” then “System”.



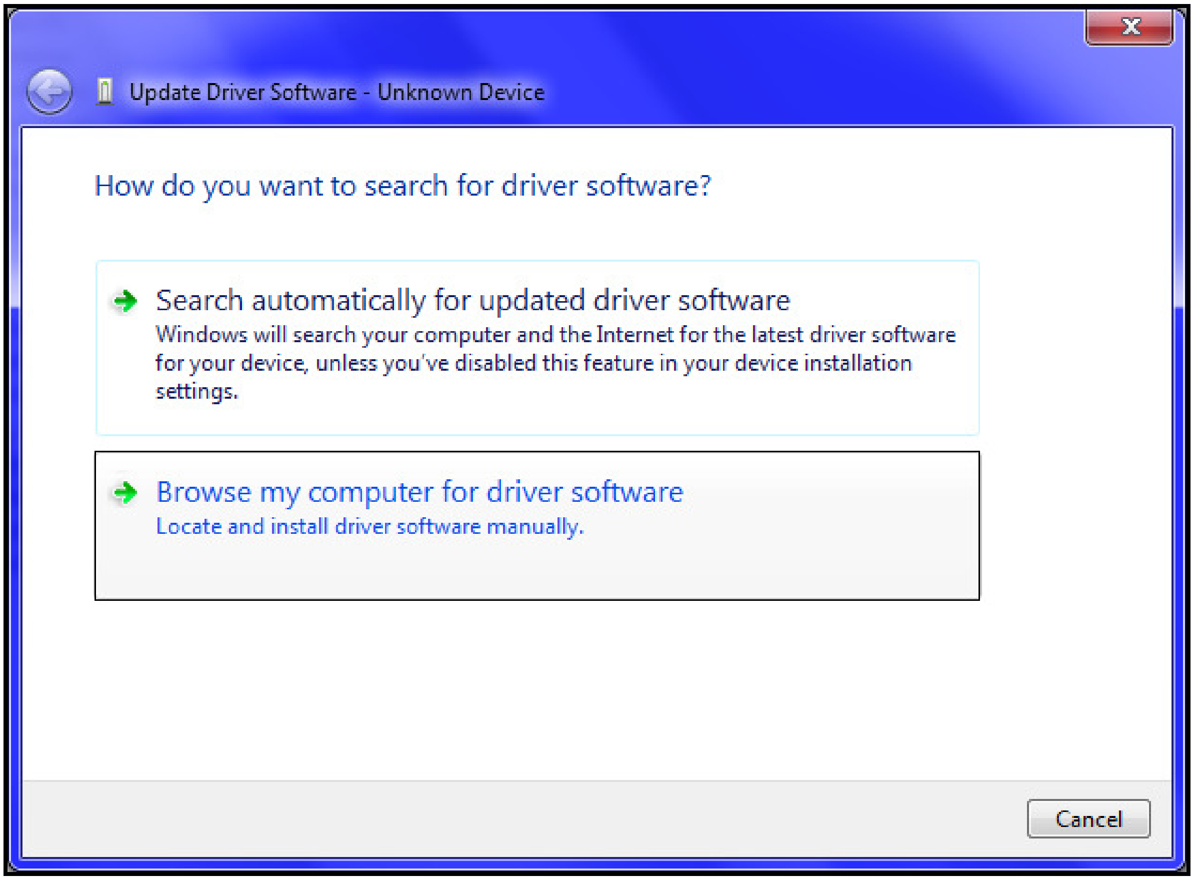


3. Open “Device Manager”
4. Plug in instrument using the USB cable provided
5. Message will appear “Device Driver Software Not Successfully Installed”
6. In “Device Manager”, “Unknown Device” will appear under “Other Devices”



7. Right click on “Unknown Devices”; select “Update Driver Software”

8. Click on “Browse My Computer for Driver Software”

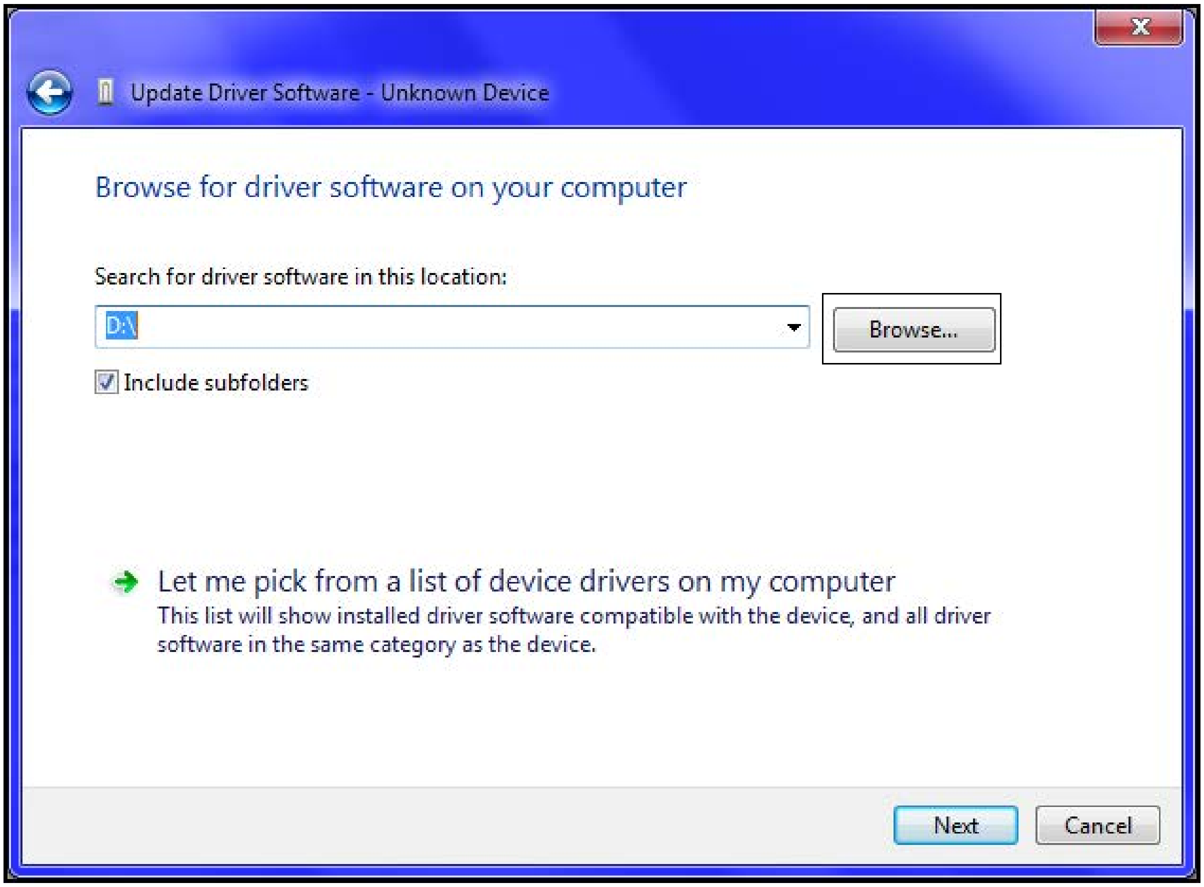


How do you want to search for driver software?

Search automatically for updated driver software
Windows will search your computer and the Internet for the latest driver software for your device, unless you've disabled this feature in your device installation settings.

Browse my computer for driver software
Locate and install driver software manually.

9. Click “Browse” button; select CD drive or the location of the driver if you are not installing from the NDT CD (recommended).



Update Driver Software - Unknown Device

Browse for driver software on your computer

Search for driver software in this location: D:\

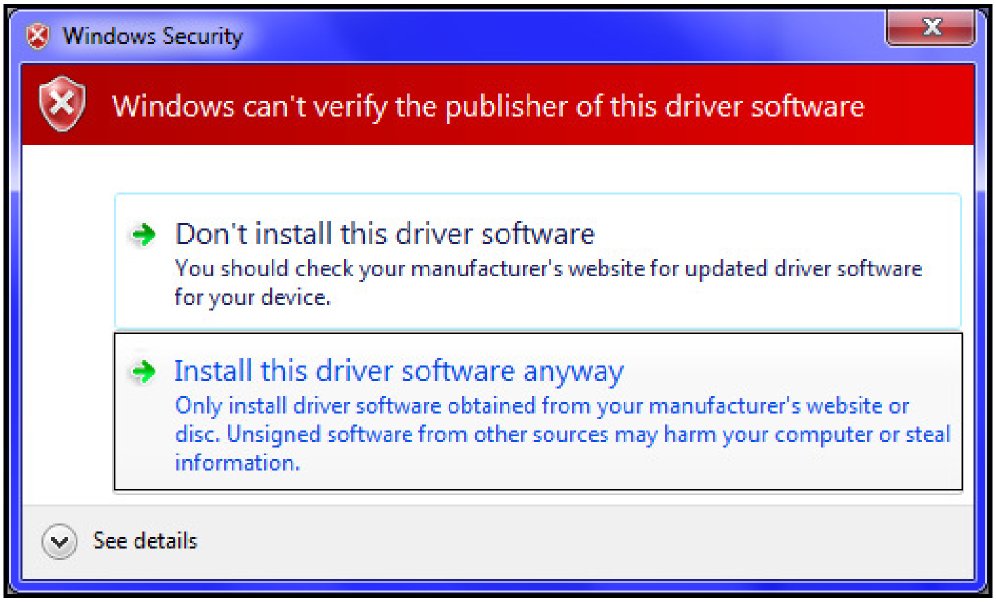
☑ Include subfolders

Let me pick from a list of device drivers on my computer

This list will show installed driver software compatible with the device, and all driver software in the same category as the device.

10. Click “OK”
11. Click “Next”

12. A Security Dialog Box will appear. Select “Install This Driver Software Anyway?”



Windows can't verify the publisher of this driver software

Don't install this driver software
You should check your manufacturer's website for updated driver software for your device.

Install this driver software anyway
Only install driver software obtained from your manufacturer's website or disc. Unsigned software from other sources may harm your computer or steal information.

13. Driver will install; close out.

Using Your Analyzer With Your PC

Using the Wireless (Bluetooth) USB Adapter to Connect the XRF Analyzer

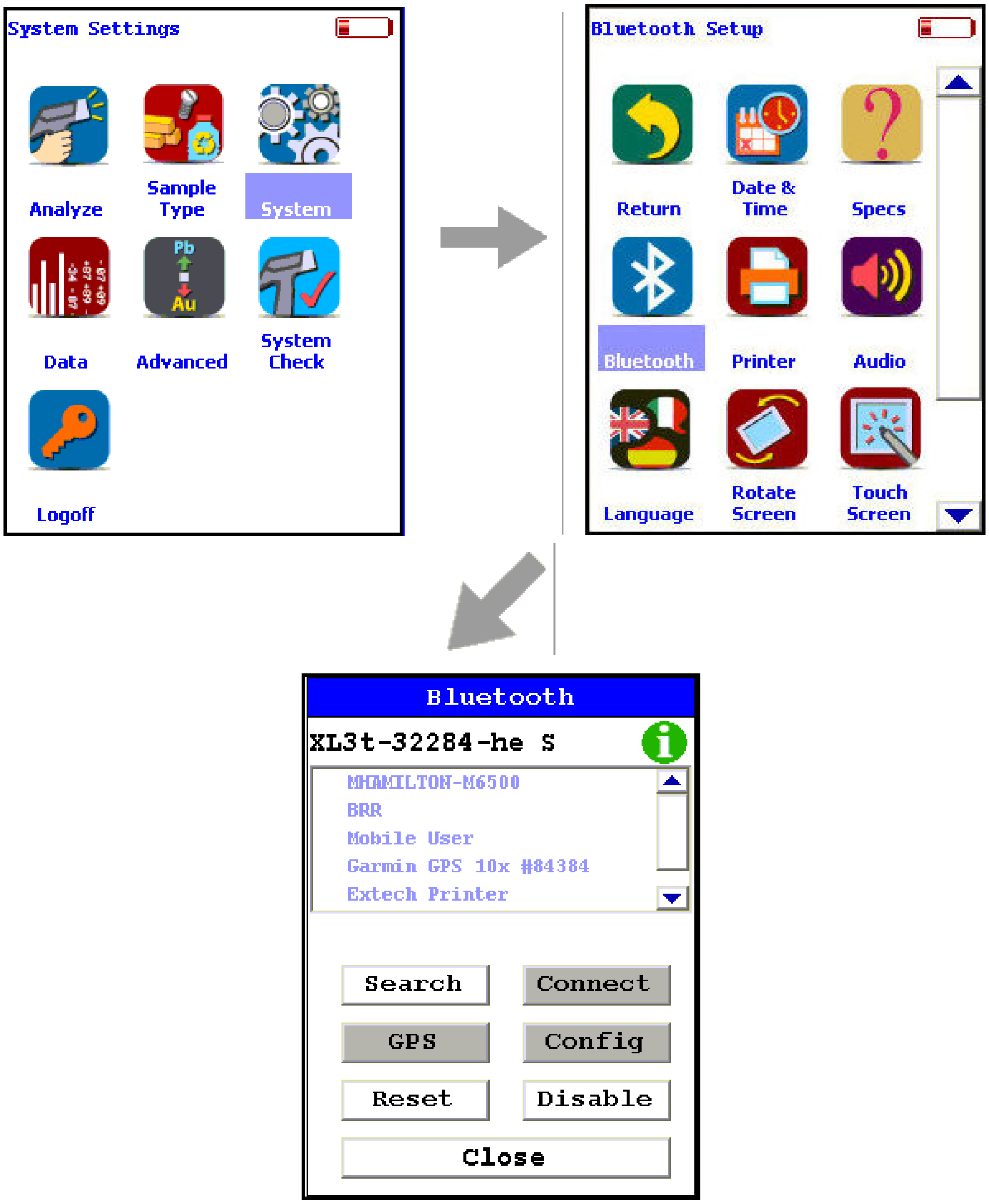
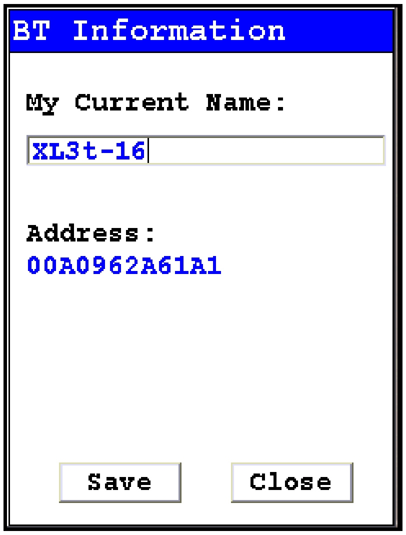


Figure 46. The Bluetooth Setup Menu Path

The USB adapter provided by Niton uses Bluetooth wireless technology. See Setting up Bluetooth to set up Bluetooth.

Select the Bluetooth icon from the System Screen to set up your analyzer for Bluetooth wireless connection.

i Icon


BT Information

My Current Name:

XL3t-16

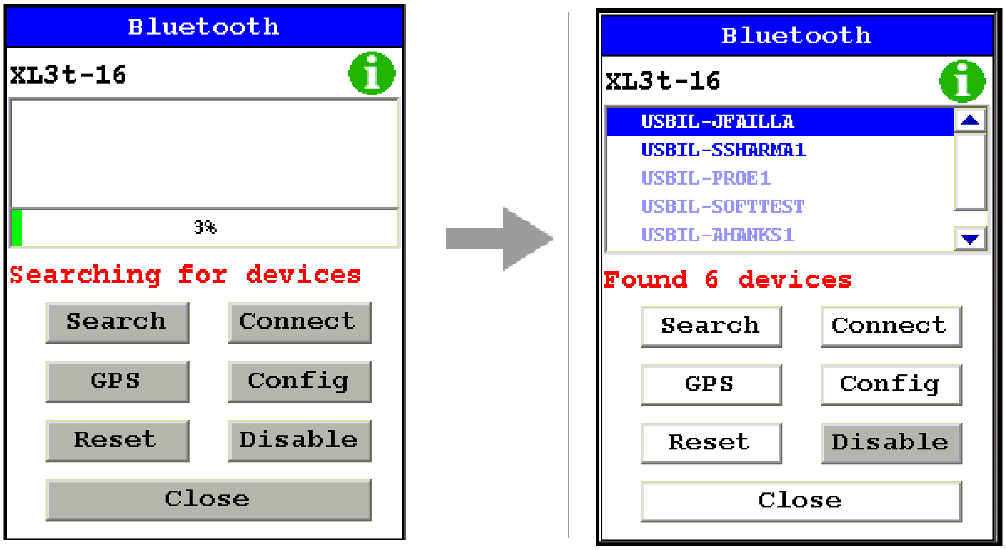
Address:
00A0962A61A1

Figure 47. BT Information Screen

Selecting the i Icon in the top right of the Bluetooth Setup Screen will open the Bluetooth Information Screen. The Bluetooth Information Screen will supply the current name as well as the MAC address of your analyzer

Search Button

Selecting the Search Button will initiate a search for currently available Bluetooth devices in the area.



Searching for devices

USBIL-JFAILLA
USBIL-SSHARMA1
USBIL-PROE1
USBIL-SOFTTEST
USBIL-AHANKS1

Found 6 devices

Figure 48. Bluetooth Searching

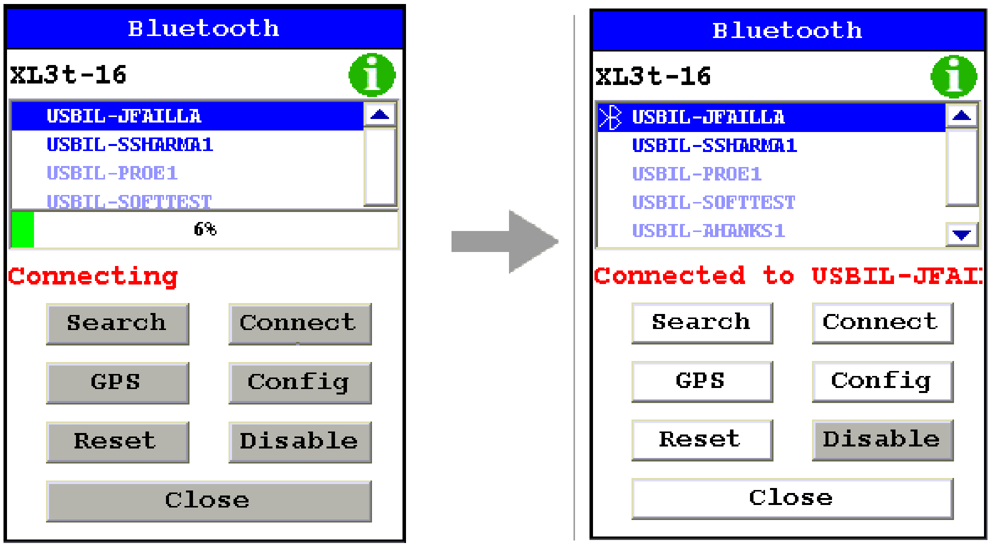
After the search, your analyzer will report which Bluetooth devices it has found in the main window of the screen.

You can connect your PC and analyzer two different ways, from the analyzer to the PC, and from the PC to the analyzer.

Connecting From Your Analyzer to Your PC

After searching, select the PC you would like to connect to from the main window of the screen.

Select the Connect Button. Your analyzer's screen will show connection progress.



Bluetooth

XL3t-16

USBIL-JFAILLA
USBIL-SSHARMA1
USBIL-PROE1
USBIL-SOFTTEST

6%

Connecting

Bluetooth

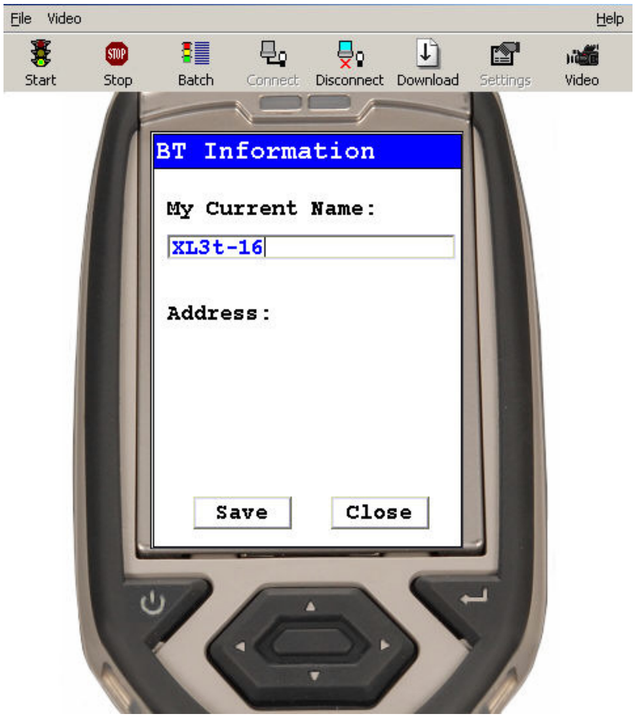
XL3t-16

USBIL-JFAILLA
USBIL-SSHARMA1
USBIL-PROE1
USBIL-SOFTTEST
USBIL-AHANKS1

Connected to USBIL-JFAI

Figure 49. Connecting Via Bluetooth

Open the program you are attempting to connect to. Here we are connected to NDTr, running the analyzer remotely over COM 5



BT Information

My Current Name: XL3t-16

Address:

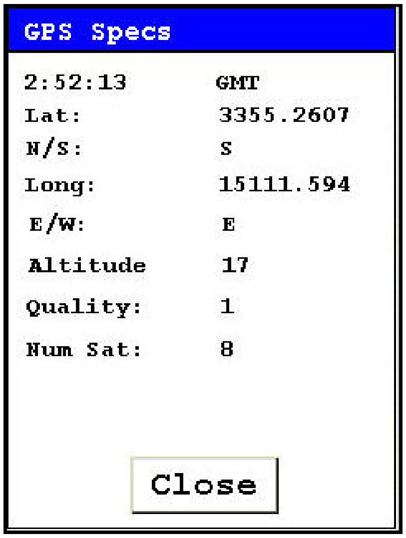
Figure 50. Connecting Via Bluetooth

Connect Button

Select a located Bluetooth device from the Search List, then select the Connect Button to connect to that device.

GPS Button

Select the GPS Button to download GPS data from a connected GPS Device.



GPS Specs

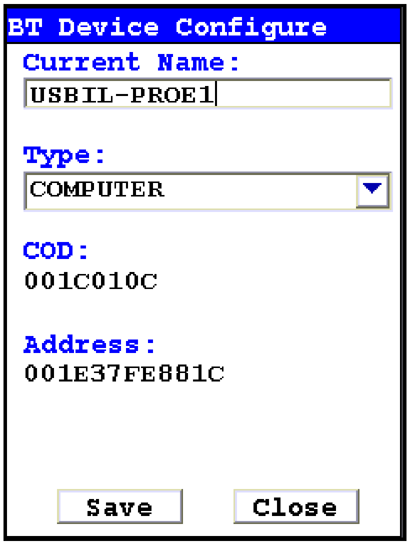
2:52:13	GMT
Lat:	3355.2607
N/S:	S
Long:	15111.594
E/W:	E
Altitude	17
Quality:	1
Num Sat:	8

Figure 51. Bluetooth GPS Data Screen

Reset Button

Selecting the Reset Button initiates a Bluetooth reset.

Config Button



BT Device Configure

Current Name:
USBIL-PROE1

Type:
COMPUTER

COD:
001C010C

Address:
001E37FE881C

Figure 52. Bluetooth Config Screen

Selecting the Config Button will load the Config Screen. This screen enables you to se the name of the currently selected Bluetooth device, change the type of device, see that device's COD Number, and see that device's MAC Address.

Changing the Bluetooth Device Type

Select the Down Arrow button next to the Type field to change the type of device connected. Select the proper type from the drop down menu. Select the Save button to save this configuration. Select the Close Button to exit without saving.

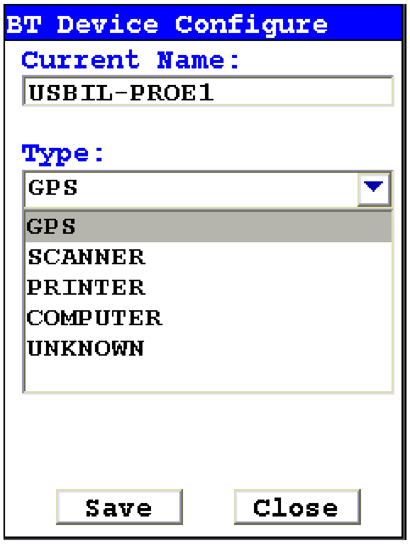


Figure 53. Changing Bluetooth Device Type

Disable/Enable Button

Selecting the Disable Button will disable Bluetooth neetworking and change the button to Enable. Selecting the Enable Button will restart Bluetooth networking and change the button back to Disable.

Close Button

Select the close button to exit from Bluetooth setup.

Using a USB Cable to Connect Your Analyzer

To connect the XL3 XRF Analyzer to your PC using the USB cable:

1. Insert the Standard USB connector on the USB cable into a USB port on your computer.

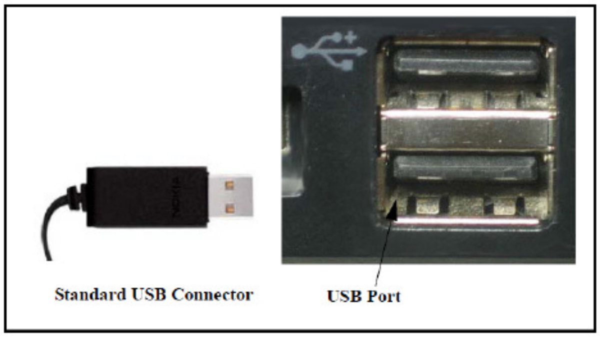


Figure 54. Standard USB Connector

2. Open the Port Cover on the XRF Analyzer.
3. Turn on the analyzer and insert the mini USB connector on the USB cable into the USB port in the handle of the XRF Analyzer.
4. Upon initial installation, insert the NDT disk located behind the foam in your case. Follow the prompts and install the USB driver located on the disk.

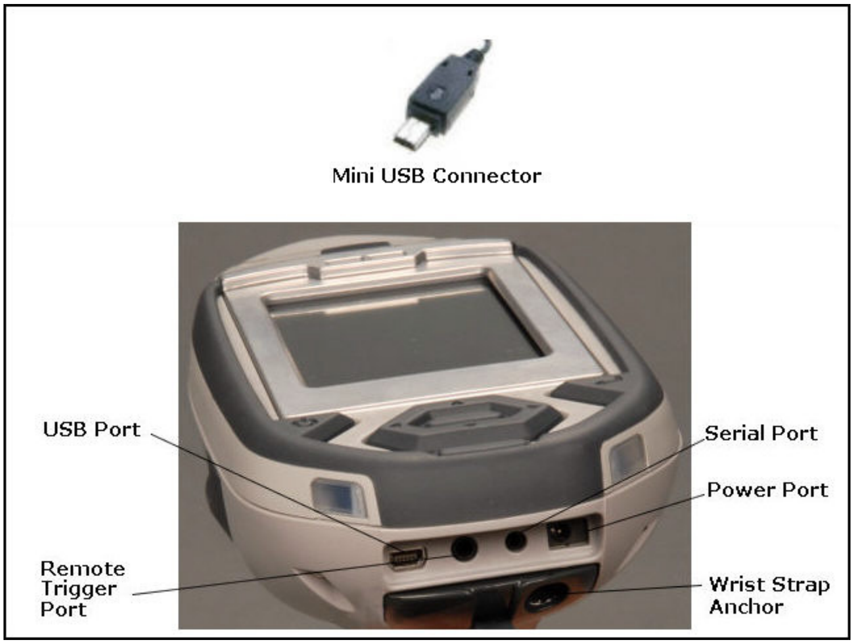


Figure 55. Mini USB Connector

Downloading Data

Standard Download

To download data you have collected offline:

1. Make sure that the XRF Analyzer is connected to your computer. See "Using Your Analyzer With Your PC" on page 151for more information.

2. Turn on the XRF Analyzer. See the manual for the XRF Analyzer for more information.

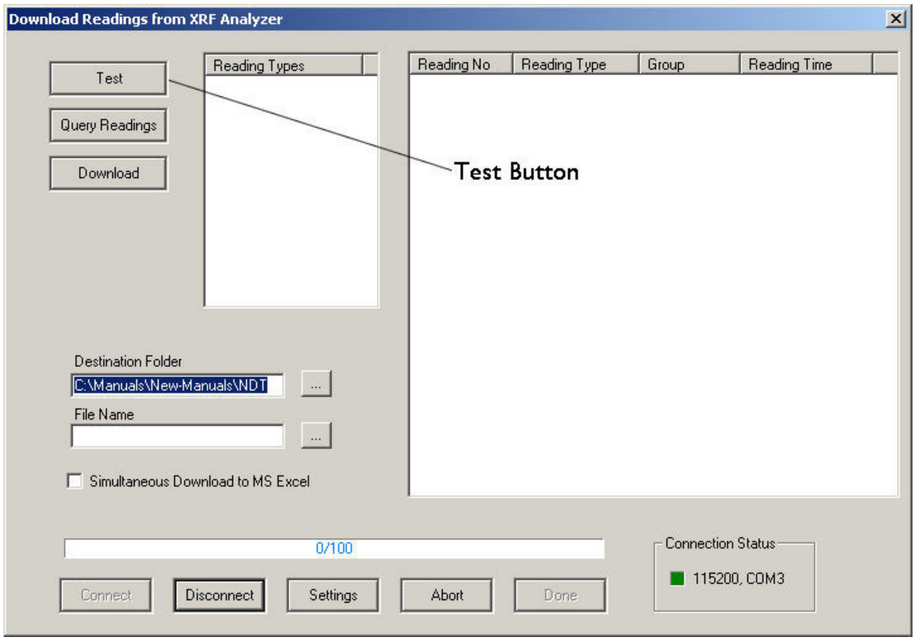
Note Wait at least 30 seconds after turning on the XRF Analyzer to begin downloading files. The System Start screens do not allow downloading.

3. Start Niton Data Transfer.

4. Click the Download button. The Download dialog box will open.

Figure 56. The Download Button

5. In the Download dialog box, Select the Test button to test the serial connection to the Analyzer.



Destination Folder: C:\Manuals\New-Manuals\NDT

0/100

Connection Status: 🟩 115200, COM3

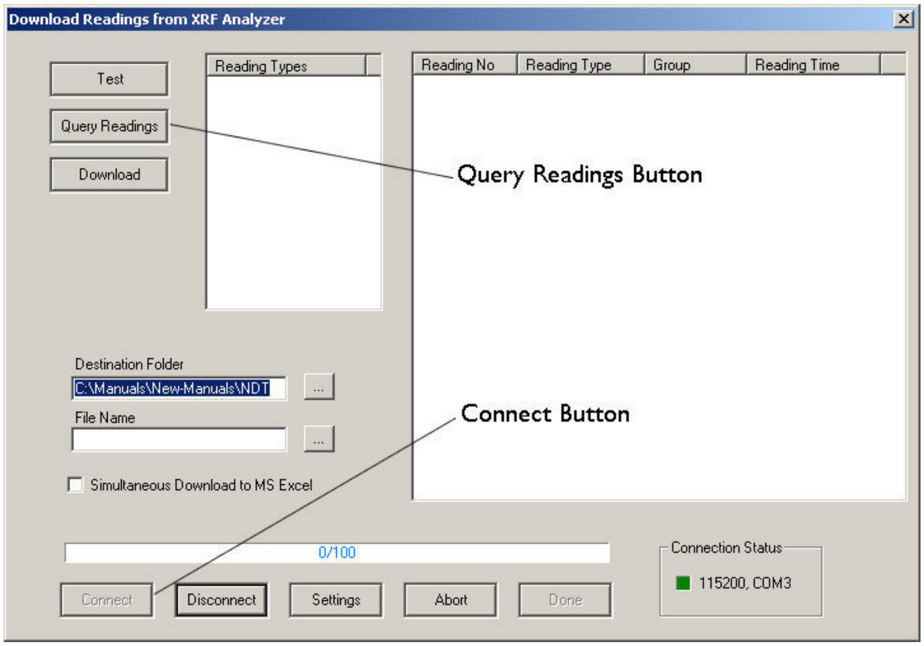
Figure 57. The Test Button

6. You should get a pop-up window informing you that the connection tested successfully. If the test fails, there is a problem with your serial port setup.

Hardware successfully communicating

Figure 58. Connection Success Window

7. In the Download dialog box, click the Connect button.



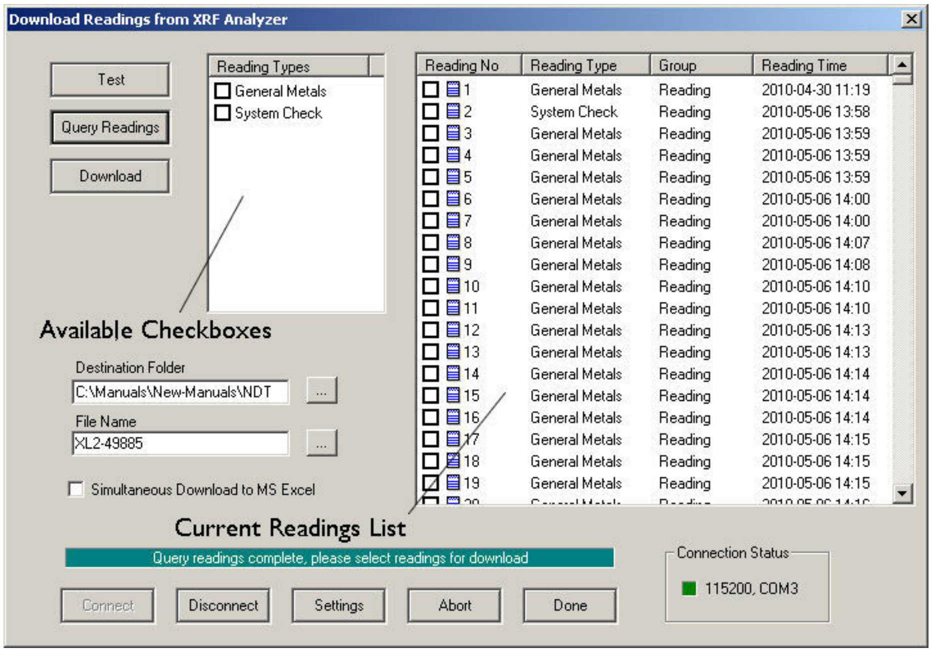
Destination Folder: C:\Manuals\New-Manuals\NDT

0/100

Connection Status: 🟩 115200, COM3

Figure 59. The Connect Button

8. Click the Query Readings button. This will return a list of all current readings on your analyzer. The list appears in the large white box in the Download dialog box.



Reading Types:

☐ General Metals
☐ System Check

Reading No	Reading Type	Group	Reading Time
☐ 1	General Metals	Reading	2010-04-30 11:19
☐ 2	System Check	Reading	2010-05-06 13:58
☐ 3	General Metals	Reading	2010-05-06 13:59
☐ 4	General Metals	Reading	2010-05-06 13:59
☐ 5	General Metals	Reading	2010-05-06 13:59
☐ 6	General Metals	Reading	2010-05-06 14:00
☐ 7	General Metals	Reading	2010-05-06 14:00
☐ 8	General Metals	Reading	2010-05-06 14:07
☐ 9	General Metals	Reading	2010-05-06 14:08
☐ 10	General Metals	Reading	2010-05-06 14:10
☐ 11	General Metals	Reading	2010-05-06 14:10
☐ 12	General Metals	Reading	2010-05-06 14:13
☐ 13	General Metals	Reading	2010-05-06 14:13
☐ 14	General Metals	Reading	2010-05-06 14:14
☐ 15	General Metals	Reading	2010-05-06 14:14
☐ 16	General Metals	Reading	2010-05-06 14:14
☐ 17	General Metals	Reading	2010-05-06 14:15
☐ 18	General Metals	Reading	2010-05-06 14:15
☐ 19	General Metals	Reading	2010-05-06 14:15

Destination Folder: C:\Manuals\New-Manuals\NDT

File Name: XL2-49885

☐ Simultaneous Download to MS Excel

Query readings complete, please select readings for download

Connection Status: 🟩 115200, COM3

Figure 60. Current Reading List

9. Select the readings that you want to download. There are two ways to do this.

a. Click the boxes next to each of the reading numbers to select or de-select individual readings. You can select a range of readings by pressing the shift key, then selecting the first and last reading in the range. All readings from the first reading selected to the last will then be selected.

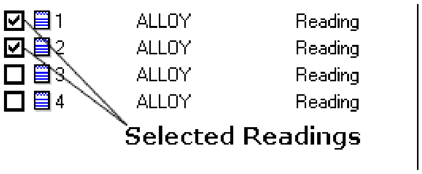


Figure 61. Selecting Readings

b. Click the boxes on the left to select or de-select all the readings of a specific type. You can also use the Shift-Click method of selecting a range of readings as described above.

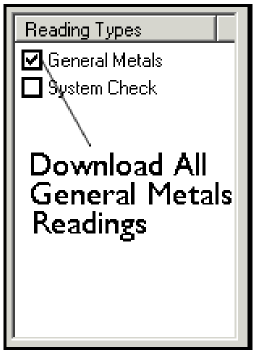
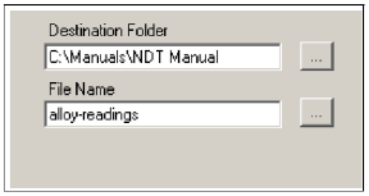


Figure 62. Using Check Boxes

10. The download generates a data file containing the selected readings. To save the file for later use:

c. Enter the path for the file in the Destination Folder field. You can use the ... button to browse.



Destination Folder: C:\Manuals\NDT Manual

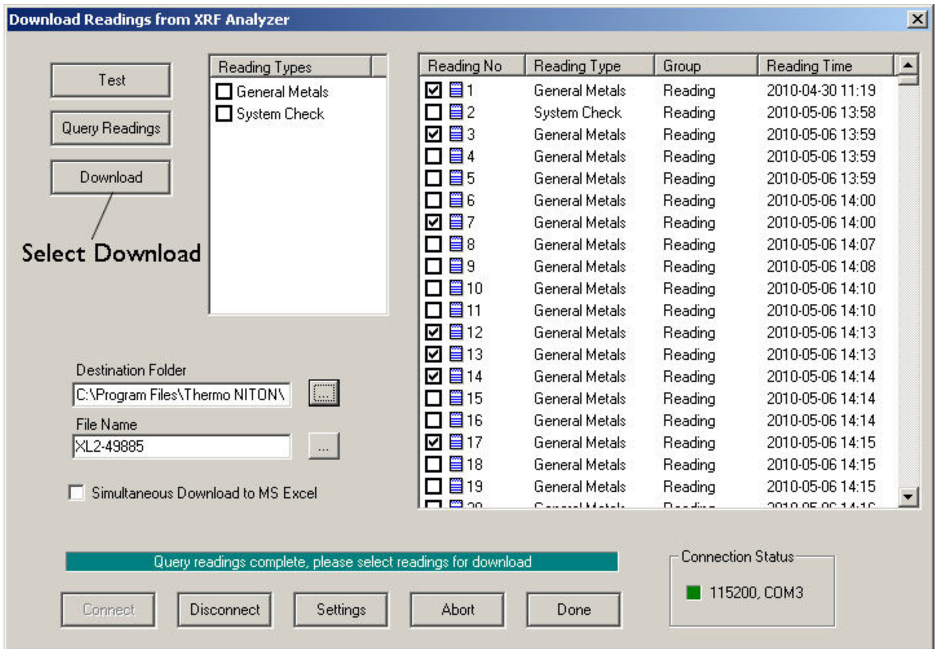
File Name: alloy-readings

Figure 63. The Browse Button

d. Enter a name for the file in the File Name field.

CAUTION Some characters are not allowed in the file name. Characters such as the "#" sign will cause an error. Niton recommends using only alphanumeric characters "-", "_" and the space character when naming a file.

e. Click the Download button.



Destination Folder: C:\Program Files\Thermo NITON\

File Name: XL2-49885

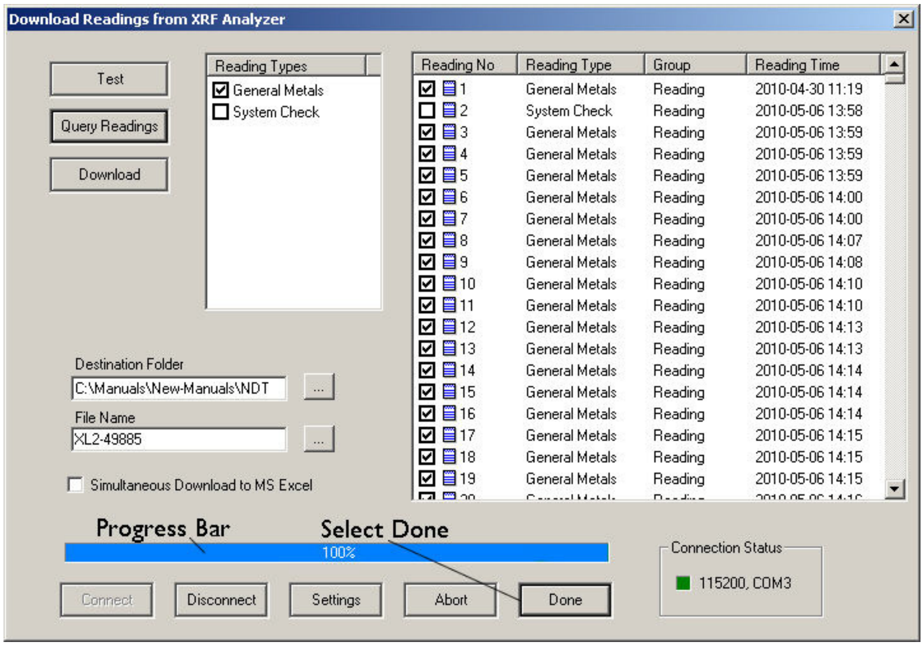
Reading No	Reading Type	Group	Reading Time
☑ 1	General Metals	Reading	2010-04-30 11:19
☐ 2	System Check	Reading	2010-05-06 13:58
☑ 3	General Metals	Reading	2010-05-06 13:59
☐ 4	General Metals	Reading	2010-05-06 13:59
☐ 5	General Metals	Reading	2010-05-06 13:59
☐ 6	General Metals	Reading	2010-05-06 14:00
☑ 7	General Metals	Reading	2010-05-06 14:00
☐ 8	General Metals	Reading	2010-05-06 14:07
☐ 9	General Metals	Reading	2010-05-06 14:08
☐ 10	General Metals	Reading	2010-05-06 14:10
☐ 11	General Metals	Reading	2010-05-06 14:10
☑ 12	General Metals	Reading	2010-05-06 14:13
☑ 13	General Metals	Reading	2010-05-06 14:13
☑ 14	General Metals	Reading	2010-05-06 14:14
☐ 15	General Metals	Reading	2010-05-06 14:14
☐ 16	General Metals	Reading	2010-05-06 14:14
☑ 17	General Metals	Reading	2010-05-06 14:15
☐ 18	General Metals	Reading	2010-05-06 14:15
☐ 19	General Metals	Reading	2010-05-06 14:15

Query readings complete, please select readings for download

Connection Status: 🟩 115200, COM3

Figure 64. The Download Button

When the progress bar shows that all the readings are downloaded, click the Done button.



Reading Types
- ☑ General Metals
- ☐ System Check

Destination Folder: C:\Manuals\New-Manuals\NDT

File Name: XL2-49885

☐ Simultaneous Download to MS Excel

100%

Connection Status: 🟩 115200, COM3

Reading No	Reading Type	Group	Reading Time
☑ 1	General Metals	Reading	2010-04-30 11:19
☐ 2	System Check	Reading	2010-05-06 13:58
☑ 3	General Metals	Reading	2010-05-06 13:59
☑ 4	General Metals	Reading	2010-05-06 13:59
☑ 5	General Metals	Reading	2010-05-06 13:59
☑ 6	General Metals	Reading	2010-05-06 14:00
☑ 7	General Metals	Reading	2010-05-06 14:00
☑ 8	General Metals	Reading	2010-05-06 14:07
☑ 9	General Metals	Reading	2010-05-06 14:08
☑ 10	General Metals	Reading	2010-05-06 14:10
☑ 11	General Metals	Reading	2010-05-06 14:10
☑ 12	General Metals	Reading	2010-05-06 14:13
☑ 13	General Metals	Reading	2010-05-06 14:13
☑ 14	General Metals	Reading	2010-05-06 14:14
☑ 15	General Metals	Reading	2010-05-06 14:14
☑ 16	General Metals	Reading	2010-05-06 14:14
☑ 17	General Metals	Reading	2010-05-06 14:15
☑ 18	General Metals	Reading	2010-05-06 14:15
☑ 19	General Metals	Reading	2010-05-06 14:15

Figure 65. The Progress Bar

You should now see the readings you selected for download displayed, one reading per horizontal line. The data has been saved to the folder and filename you indicated prior to downloading. If an error message has appeared, see the following section.

You can also automatically save reports in .csv format for importing into Excel or other programs.Simultaneous Save as CSV File

Table 4: Error Messages while Downloading

Error Message	Action
Couldn't open \\.\COM7 Error Code: 2	Select another COM port.
The port \\.\COM2 is in use	Select another COM port.
Please Open the Port	Click the Connect button.

Table 4: Error Messages while Downloading

Hardware Not Responding or Hardware Not Ready	Turn on the XRF Analyzer. If you are using a serial cable, check that the cable is inserted snugly. If you are using a serial cable, select the other COM port. If you are using the wireless USB adapter, connect the serial port. See the "Installing and Using Bluetooth" manual for complete instructions on setting up the Bluetooth adapter to work with your analyzer. Check that the spare battery is fully charged.
The Serial Port connection failed: RFCOMM connection failed	Check that the battery is fully charged.
WARNING: 38400 baud rate not supported.	This indicates a potential problem. Test the serial port. If there is a problem connecting, switch baud rate on both the analyzer and the NDT software to 115200.
Incorrect Data in reading # XXX. Reading will be skipped. Error code: BOUNDARY_ERROR1.	This indicates a version mismatch between your instrument code and the NDT code running on your computer. Use a version of NDT that matches the ver?sion number of the software on your analyzer.
Incorrect Data in reading # XXX. Reading will be skipped. Error code: BOUNDARY_ERROR2.	This indicates a version mismatch between your instrument code and the NDT code running on your computer. Use a version of NDT that matches the ver?sion number of the software on your analyzer.
Incorrect Data in reading # XXX. Reading will be skipped. Error code: BOUNDARY_ERROR3.	This indicates a version mismatch between your instrument code and the NDT code running on your computer. Use a version of NDT that matches the ver?sion number of the software on your analyzer.
WARNING: 115200 baud rate not supported.	This indicates a potential problem. Test the serial port. If there is a problem connecting, switch baud rate on both the analyzer and the NDT software to 38400.
SH4 Successfully Communicating Result: SUCCESS	This indicates a normal connection.

Note When using the wireless USB adapter, if the serial port repeatedly disconnects, check that the battery is fully charged.

Live Download (Automatic Save)

If desired, your Niton XL3 analyzer has the capability to download and store each reading to the PC in real time to a file of your choice. To enable this feature, you must do the following:

- Your Niton analyzer must be turned on and connected to the PC. See Using Your Analyzer With Your PC.

- The NDTr program module must be running and connected to your analyzer. See Operating Your Analyzer Remotely.

- The Download icon in the NDTr toolbar must be selected.

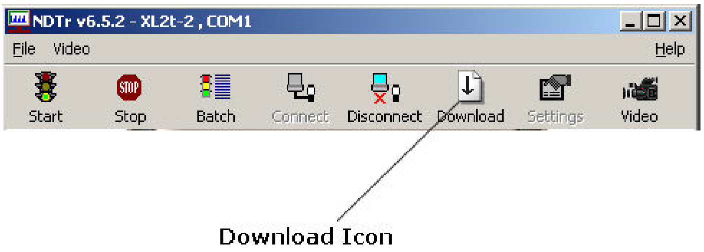


Figure 66. Live Download Icon

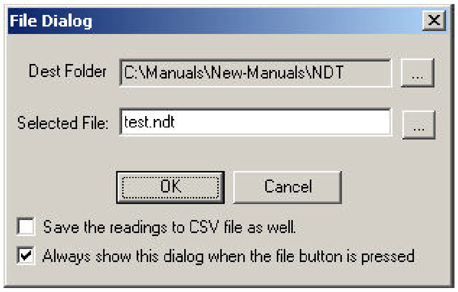
The file created is in a format readable by the NDT program module, has an extension of .ndt, and looks identical to a file of manually downloaded readings - see Standard Download. It can also create a simultaneous .CSV file. Simultaneous Save as CSV File .

Please note the following:

1. When the instrument is unplugged, selecting the Download icon does nothing.
2. When you disconnect, then reconnect, your analyzer, Download appends future readings to same file.
3. Live Download does not overwrite any previous readings in the file. If you want to do this, you must first explicitly erase the file before initiating Live Download.
4. Live Download does not retroactively add any readings taken while your analyzer was disconnected.

Changing the Filename for Live Download

Once you have selected the Download icon, a dialog box appears as shown below:



C:\Manuals\New-Manuals\NDT

test.ndt

☐ Save the readings to CSV file as well.

☑ Always show this dialog when the file button is pressed

Figure 67. File Dialog Box

You can change the destination file or folder by clicking in the appropriate text box and typing in the new file name, or by clicking on the browse button (...) to the right of the text box and selecting a different pre-existing filename. To implement these changes, click the OK button.

Your instrument serial number is associated with the file. If a different instrument is connected and Live Download is started, a message will appear saying that the connected instrument and file instrument do not match, and Live Download will not start. Saving the session as a new file will alleviate this issue

Simultaneous Save as CSV File

By clicking on the checkbox box labeled "Save the readings to CSV file as well" you can enable simultaneously saving the data as a standard CSV (Comma Separated Value) file for use with other programs.

Controlling Your Analyzer From Your PC

The NDTr program allows you to completely control your Niton analyzer remotely, from your computer. It works over serial or USB connection over the supplied connector, or Bluetooth wireless communication. See Using Your Analyzer With Your PC for details on how to connect your Analyzer and PC.

The NDTr Toolbar

The NDTr Toolbar is a string of icons along the top of the NDTr window. It looks like this:

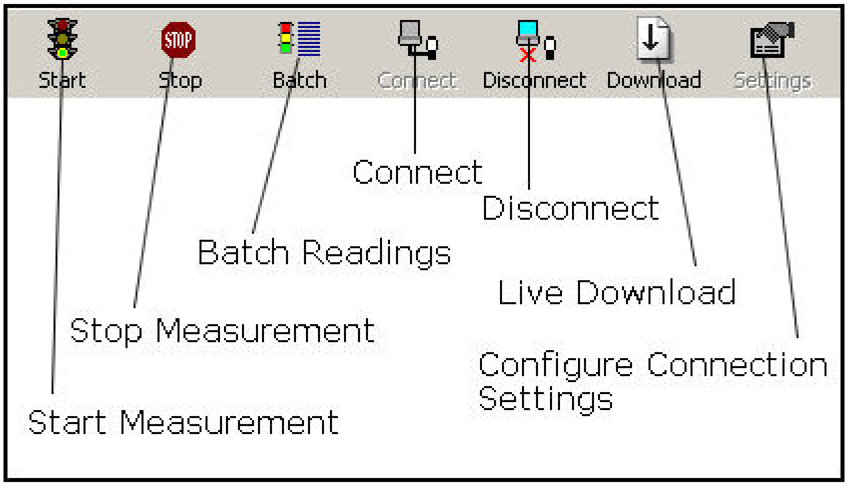


Figure 68. The NDTr Toolbar

Start Measurement

Clicking this icon will initiate a measurement in whatever mode the analyzer is in currently.

Stop Measurement

Clicking this icon will halt any ongoing measurement on the analyzer.

Configure Connection Settings

Clicking this icon will allow you to change your configuration settings.

Connect

Clicking this icon will attempt to establish a connection between your computer and your analyzer.

Disconnect

Clicking this icon will disconnect your computer from your analyzer.

Live Download

See Live Download from NDTr

Configure Connection Settings

Clicking on the Configure Connection Settings icon allows you to change the Com Port for connecting your computer to your analyzer. Once you click on the icon, a settings dialog box will appear.

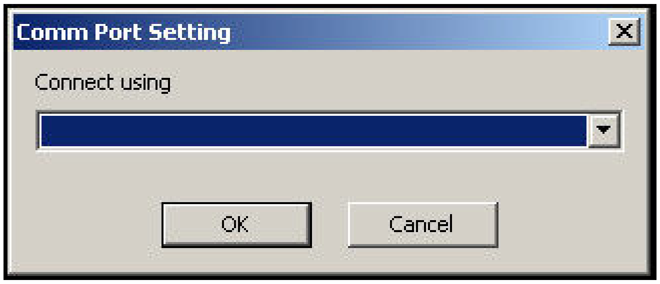
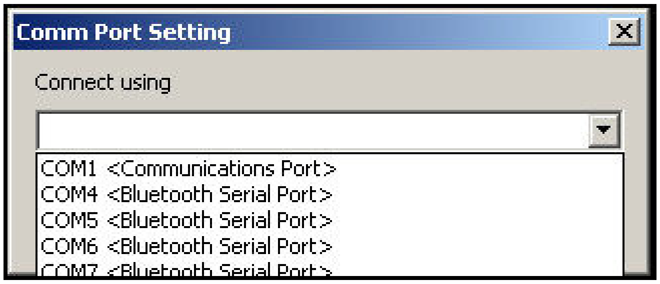


Figure 69. Connection Settings Dialog Box

Selecting the Com Port

Selecting the down arrow in the "Connect Using" field will display the various Com Ports on the computer that the analyzer can connect through.



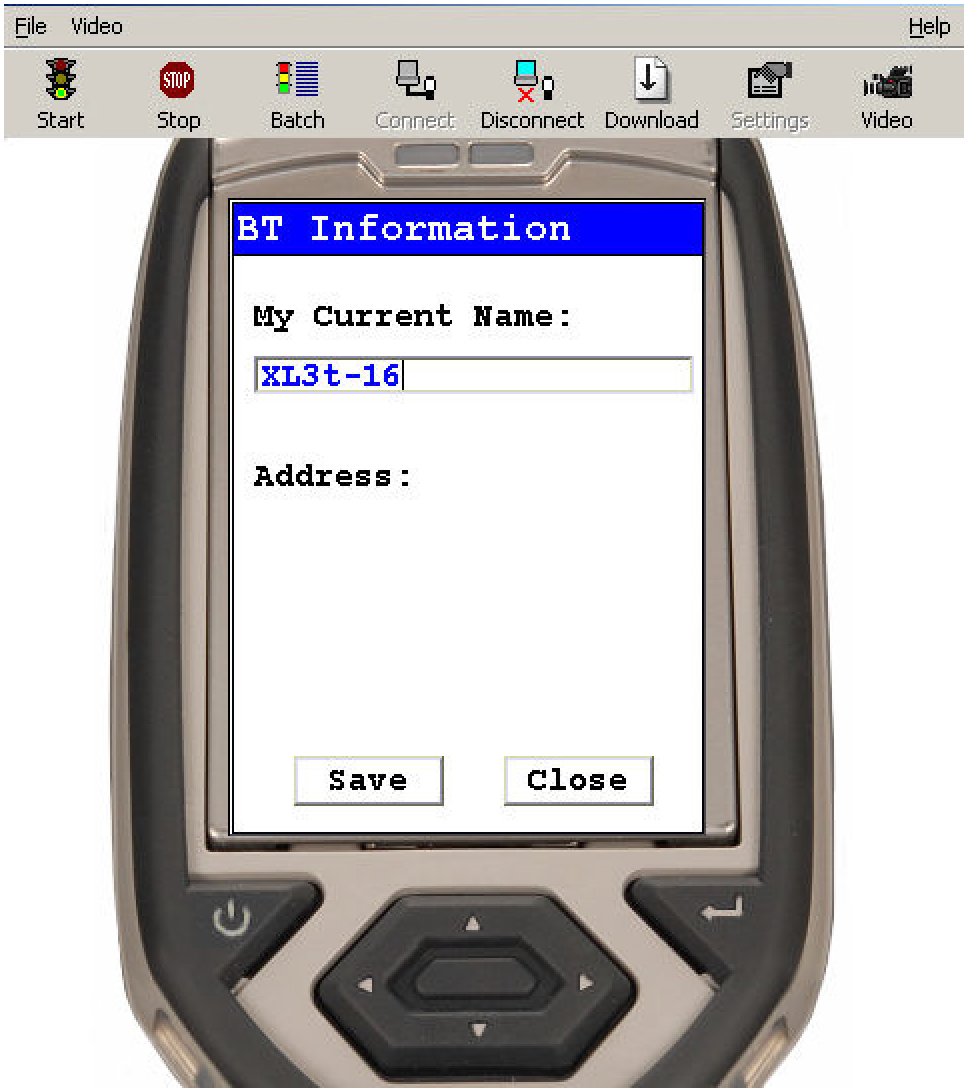
COM1 <Communications Port>
COM4 <Bluetooth Serial Port>
COM5 <Bluetooth Serial Port>
COM6 <Bluetooth Serial Port>
COM7 <Bluetooth Serial Port>

Figure 70. Selecting the Com Port

Select the proper com port from the list, then select the OK Button.

Operating Your Analyzer Remotely

NDTr version 7 and above will automatically select the proper virtual interface for you, whether you have a Thermo Scientific Niton XL2 or XL3. The virtual interface operates exactly as the analyzer would. Selecting the buttons, icons and menus with your mouse works exactly as if you were selecting them with your finger or stylus on the real analyzer.



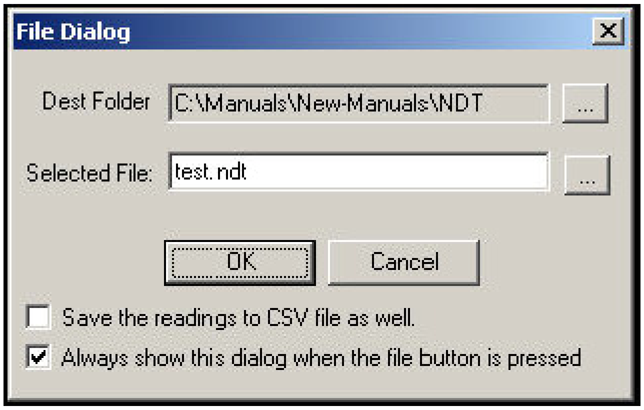
My Current Name: XL3t-16

Address:

Figure 71. Niton XL3 Virtual Interface

Live Download from NDTr

Once you have connected to your analyzer using NDTr, click on the Download icon on the NDTr toolbar. When you click the Download icon, a Download dialog box will appear.



Dest Folder: C:\Manuals\New-Manuals\NDT

Selected File: test.ndt

☐ Save the readings to CSV file as well.

☑ Always show this dialog when the file button is pressed

Figure 72. File Dialog Box

Dest Folder

This field shows the last used save folder, defaulting to the folder where NDT is installed.

... (Browse Folders) Button

Selecting this button enables you to select a different folder for saving the file. This will change the Dest Folder Field.

Selected File

This shows the filename the reading will be saved to unless you change it.

... (Browse Files) Button

Selecting this button enables you to select a different file name by browsing the file listing. The file extension “.ndt” will be appended to the name - i.e. File name “file” will be saved as “file.ndt” and the file will be in the NDT format.

Save the Reading to CSV File as Well Checkbox

Selecting this checkbox enables you to create a second autosave file with CSV format for importing into spreadsheets such as Excel. This file will have the same name as the NDT file above, but with the file extension “.csv” instead - i.e. “test.ndt” will be saved as “test.csv” as well. The checkbox is selected when there is a check in it, and deselected when it is empty.

Always Show this Dialog Box when the File button is Pressed Checkbox

Selecting this checkbox will enable you to change the filename whenever you want. Deselecting this checkbox will save the file under the same name in the same folder every time. The checkbox is selected when there is a check in it, and deselected when it is empty.

Learning More, Service, and Support

This section of the Resource guide is about getting the most out of your analyzer. We cover troubleshooting your analyzer by using the Specs screen. We also cover advanced topics like setting thresholds, using the Tools menu, correcting for light elements in the sample composition, setting up pass/fail analysis, changing safety and start/stop parameters, and many other special situations you may need. We have also included a number of documents for reference, so you can learn more about XRF analysis if you are so inclined.

Replacing the Measurement Window

WARNING Before you begin, remove the battery from your analyzer!

- Remove the two screws holding the Measurement Window Bracket to the nose of your analyzer.

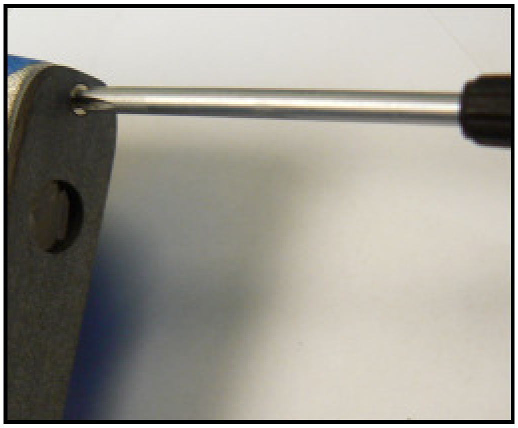


Figure 1. Removing the Window Bracket Screws

- Remove the Measurement Window Bracket from the analyzer, and turn it over, exposing the back with seal and Measurement Window.
- Remove the old Measurement Window from the bracket.
- Clean the Window area thoroughly, using a clean, guaranteed lint-free cloth and isopropyl alcohol.

Figure 2. Removing the Old Window

- Measurement Windows come in two types - Prolene (P.N 187-1454) for 900 Series analyzers, and Polypropylene (P/N 187-1555) for all other analyzers.

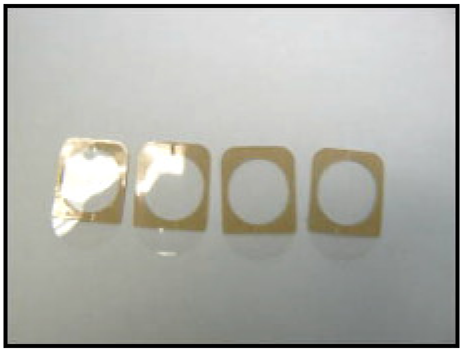


Figure 3. Polyethylene Window P/N 187-1555

Figure 4. Prolene Window PN 187-1454

- When the bracket is clean, remove the backing from the Measurement Window. Place the window on the Bracket gently. Make sure the opaque portions of the window do not intrude over the large measurement hole in the Bracket.

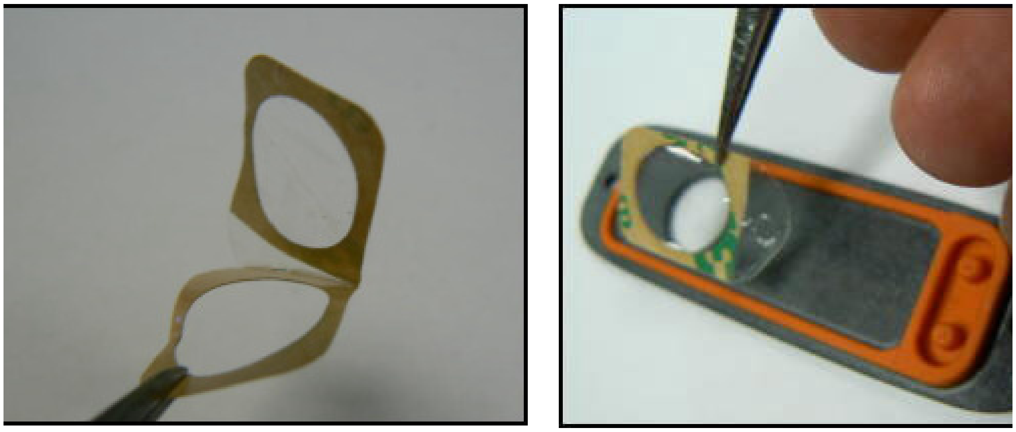


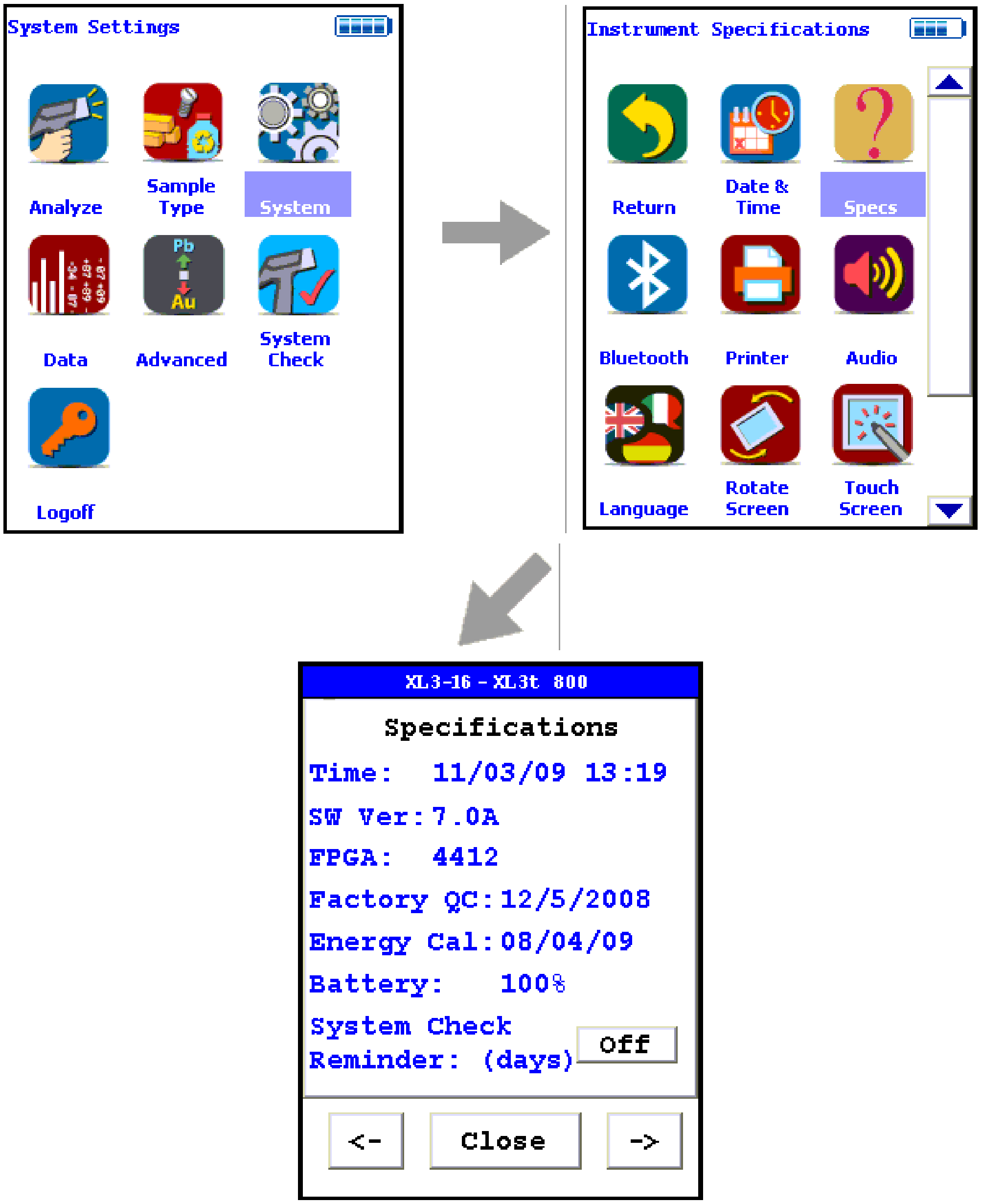
Figure 5. Removing the Backing from Prolene Window (Left) and Applying Window to Bracket (Right)

CAUTION Do not use fingers to press window into place! Use a smooth, hard surface such as back of tweezers.

Figure 6. Measurement Window Replaced

- Replace Window Bracket on the front of your analyzer, then insert screws.

The Specs Screen



XL3-16 - XL3t 800

Specifications

Time: 11/03/09 13:19

SW Ver: 7.0A

FPGA: 4412

Factory QC: 12/5/2008

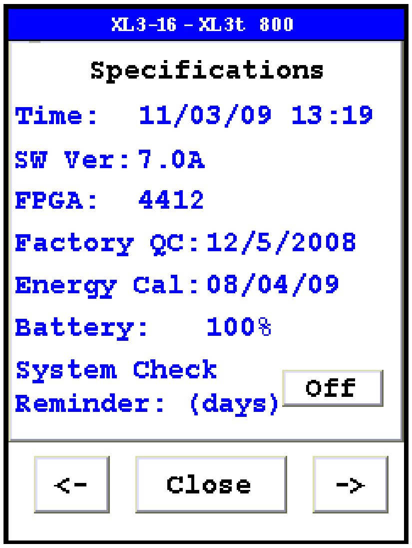
Energy Cal: 08/04/09

Battery: 100%

System Check Reminder: (days) Off

Figure 7. The Specs Screen Menu Path

Select the Specs icon from the System Menu to display the analyzer's specifications. These specifications include your analyzer's serial number, software and firmware versions, and other data. Press the Close Screen Button to return to the previous menu. Press the ">-" Screen Button to go to the Diagnostic Menu, and press the "<-" Screen Button to return to the Specifications Screen.



Specifications

Time: 11/03/09 13:19

SW Ver: 7.0A

FPGA: 4412

Factory QC: 12/5/2008

Energy Cal: 08/04/09

Battery: 100%

System Check Reminder: (days) Off

Figure 8. The Specifications Screen

On the Specs Screen, standard information on the state of your analyzer is shown for your reference. This information should be reported to Service if there is a problem.

Specs Information

The following is the information supplied on the Specs Screen:

Instrument Specific Serial Number

This is located in the left part of the blue band at the top of the screen.

Model Number

This is located in the right part of the blue band at the top of the screen.

Date And Time

This is the current Date and Time. This is particularly important for date stamping.

SW Version

This is the currently loaded software version, which should be reported to Service if there is any problem.

FPGA

This is the currently loaded FPGA software version, which should be reported to Service if there is any problem. FPGA versions are always a four digit number. Any other number of digits may be a sign of a problem in the FPGA code.

Factory QC

This is the date that the machine was QCed at the factory.

Energy Cal

This line notes the last time a System Check was performed.

Battery

This line gives the proportional charge remaining to the battery.

System Check Reminder

Select the OFF Screen Button after "System Check Reminder" to set a reminder to calibrate your analyzer. Selecting the button will open the Cal. Reminder Editor. Select the number of days you want between reminders with the numeric keys. Of the other screen buttons, C = Clear All, E = Enter, and OFF shuts off the Reminder Function. Selecting the E button will enter the current value as the reminder interval and return to the Specs Screen.

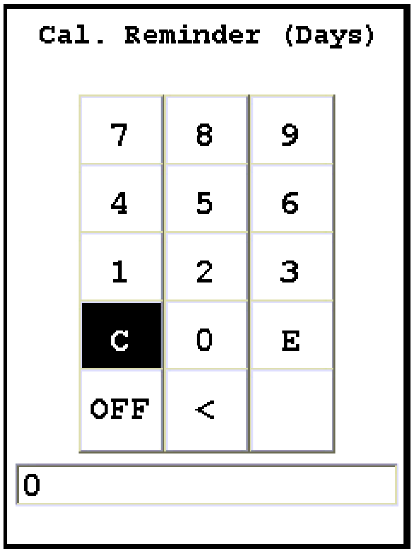
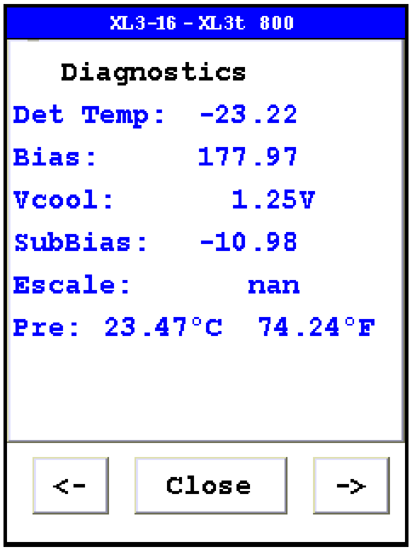


Figure 9. Cal Reminder Editor Screen

Diagnostics

Select the "->" Screen Button to load the Diagnostics Screen. The Diagnostics Screen shows Detector Temperature, Bias, Cooler Voltage, SubBias, Energy Scale, and Temperature in C and F scales.

The Diagnostics Screen can be of great utility in assuring proper operation of your analyzer.



Diagnostics

Det Temp: -23.22

Bias: 177.97

Vcool: 1.25V

SubBias: -10.98

Escale: nan

Pre: 23.47°C 74.24°F

Figure 10. The Diagnostics Screen

The proper ranges of operational values on the Diagnostics Screen follow.

Det Temp:

Detector Temperature should be within this range:

- 25 + or - 5 degrees

Bias:

Bias should be within this range:

175 + or - 10

VCool:

VCool will vary with the ambient temperature.

SubBias:

SubBias should be within this range:

-11 + or - 3

Escale:

Escale should be within this range:

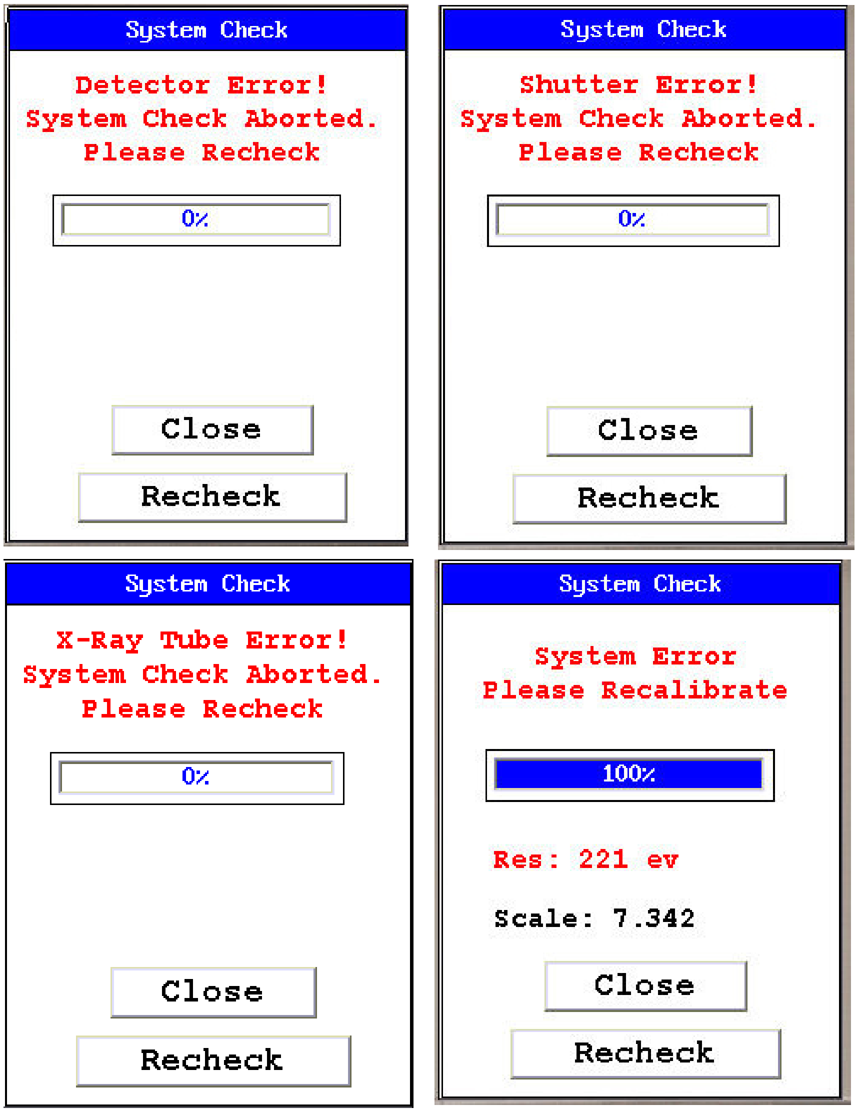
6.6 through 9.0

Preamp:

Preamp value should only be noted, and reported to Service if there is a problem.

Error Messages

The analyzer will auto-detect many perceived hardware problems and may display the following error messages:



Detector Error!
System Check Aborted.
Please Recheck

Shutter Error!
System Check Aborted.
Please Recheck

X-Ray Tube Error!
System Check Aborted.
Please Recheck

System Error
Please Recalibrate

Res: 221 ev

Scale: 7.342

Figure 11. The Specs Screen Menu Path

Please re-run the System Check and if the problem persists, contact customer service.

Registration and Licensing FAQ

Niton XL3t and XL3t GOLDD

As a user of a Niton XL3t analyzer, you may be required to register or obtain a license with your local radiation control authority. In the US, if you intend to do work with your analyzer in states other than your own, you may be required to register there as well. Below is a list of commonly asked questions that come up when filling out registration forms.

FAQ

Q: What is the max mA, max kVp, and max power?

A: Maximum mA is 0.1 mA

Maximum kVp is 50 kVp

Maximum power: 2 watts

Q: What is the accelerator voltage or MeV?

A: This should be filled out as not applicable N/A as it does not apply to Niton XL3t analyzers.

Q: What is the radioisotope?

A: There are no radioactive isotopes in Niton XL3t analyzers.

Q: What category is the Niton XL3t?

A: States differ greatly in their categories; the following is a list of common categories:

- X-Ray Fluorescence
- Analytical or Analytical XRF
- Open Beam or Open Beam Analytical
- Portable Gauge or Portable XRF
- Industrial Analytical or Non-Destructive Testing

When selecting the category make sure that you don't select medical or radiographic.

Q: How many tubes are in the Niton XL3t?

A: One.

Q: What is the analyzer serial number?

A: The serial number is a 5 digit number located on the yellow sticker on the underside of your analyzer.

Q: What is the tube serial number?

A: The serial number of the tube can be found on the Calibration Certificate.

Q: What is the type of X-Ray Processing?

A: None. Niton XL3t analyzers do not use film.

Q: How often do I need to perform leak tests on the Niton XL3t?

A: Never. Leak tests are only required for analyzers with radioactive isotopes. Niton XL3t analyzers do not have radioactive isotopes.

Niton XL3p

Routine Maintenance Guidelines

Maintenance, Cleaning and Repairs

To ensure the reliability, durability, and performance of your Niton Analyzer, keep it clean—especially the transparent measurement window covering the analysis window. Clean the measurement window <u>gently</u> with a cotton swab. Clean the body of the analyzer with a soft cloth. <u>Never</u> use detergents, or solvents on your analyzer, or immerse your analyzer in water. If the measurement window becomes frayed, ripped, or contaminated with metal particulates, replace it with a new window. measurement windows (Standard Window Niton P/N 187-1555 or Helium Purge Window Niton P/N 187-1454) may be ordered from Thermo Fisher Scientific's Service Department in the United States, toll free, at (800) 875-1578, or outside the United States, at +1-978-670-7460 or from your local Authorized Niton Analyzers Service Center.

From time to time, your touch screen will need cleaning. Niton recommends that you use a lens cleaning solution with a soft cloth. Do not use water to clean your Niton Analyzer.

WARNING All Service, except exterior cleaning and measurement window replacement, must be performed by Thermo Scientific or an Authorized Niton Analyzers Service Center. Do not attempt to make repairs yourself. Opening the case of your Niton will void the analyzer Warranty in its entirety.

CAUTION Always obtain a Return Authorization (RA) number from Thermo Fisher Scientific's Service Department in the United States, toll free, at (800) 875-1578, or outside the United States, at +1-978-670-7460 before returning your analyzer to the Niton Service Department or local Authorized Niton Analyzers Service Center.

Storing and Transporting Your Niton XL3 Analyzer

All Niton Analyzers are transported in waterproof, drop-resistant, fully padded carrying cases with padlocks. In most countries, Niton XRF analyzers may be transported by car or plane or shipped as an *ordinary* package. For most courier services, no special labels are required on the outside of the Niton analyzer case or on additional packaging.

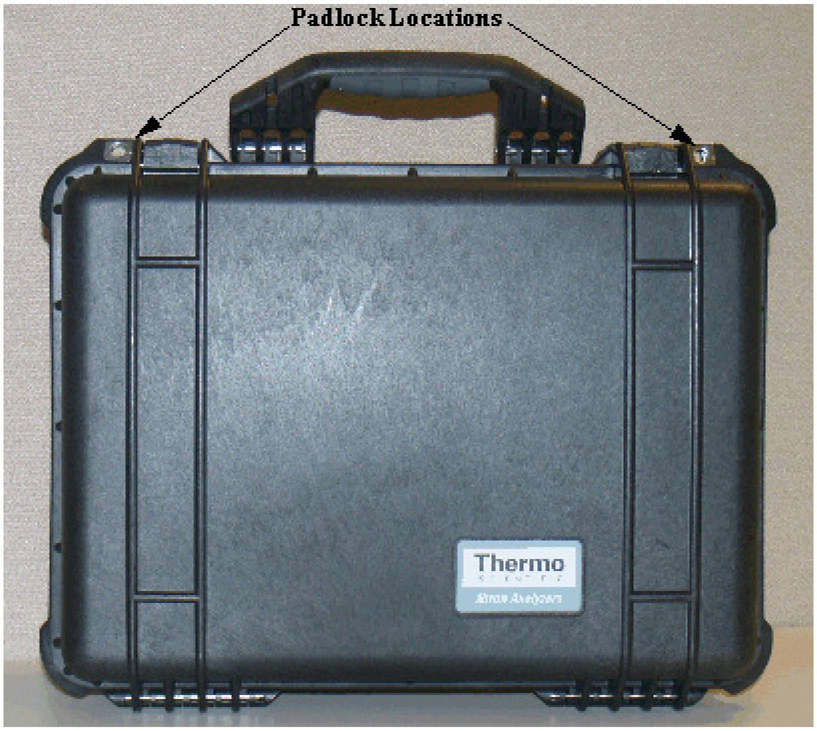


Figure 12. The Niton Carrying Case

All padlocks are shipped with a default combination of "0-0-0". If you change this combination, please inform Thermo of the new combination if you return the unit for service.

To change the combination:

1. Dial the default combination to open the lock, and pull out the shackle.
2. Rotate the shackle 180 degrees and push it down as far as it can go.
3. While holding the shackle down, rotate it 90 degrees back in either direction and release shackle.
4. Change the dial settings to the desired combination, record the combination, and without disturbing the dials, rotate the shackle back 90 degrees to the position it had in step 2.
5. Pull shackle out and rotate it 180 degrees and secure it. Your lock now has its own secret combination.

CAUTION Always transport the unit in its padded carrying case, and store the Niton Analyzer in its case whenever it is not being used.

CAUTION In most cases, no notification is required if transporting within state boundaries. This may not be the case when entering federal properties.

CAUTION Within the United States, always keep a copy of the US DOT compliance statement in your Niton analyzer case at all times. A copy is included with your analyzer.

CAUTION Always follow all pertinent local and national regulations and guidelines, wherever your analyzer is transported or used.

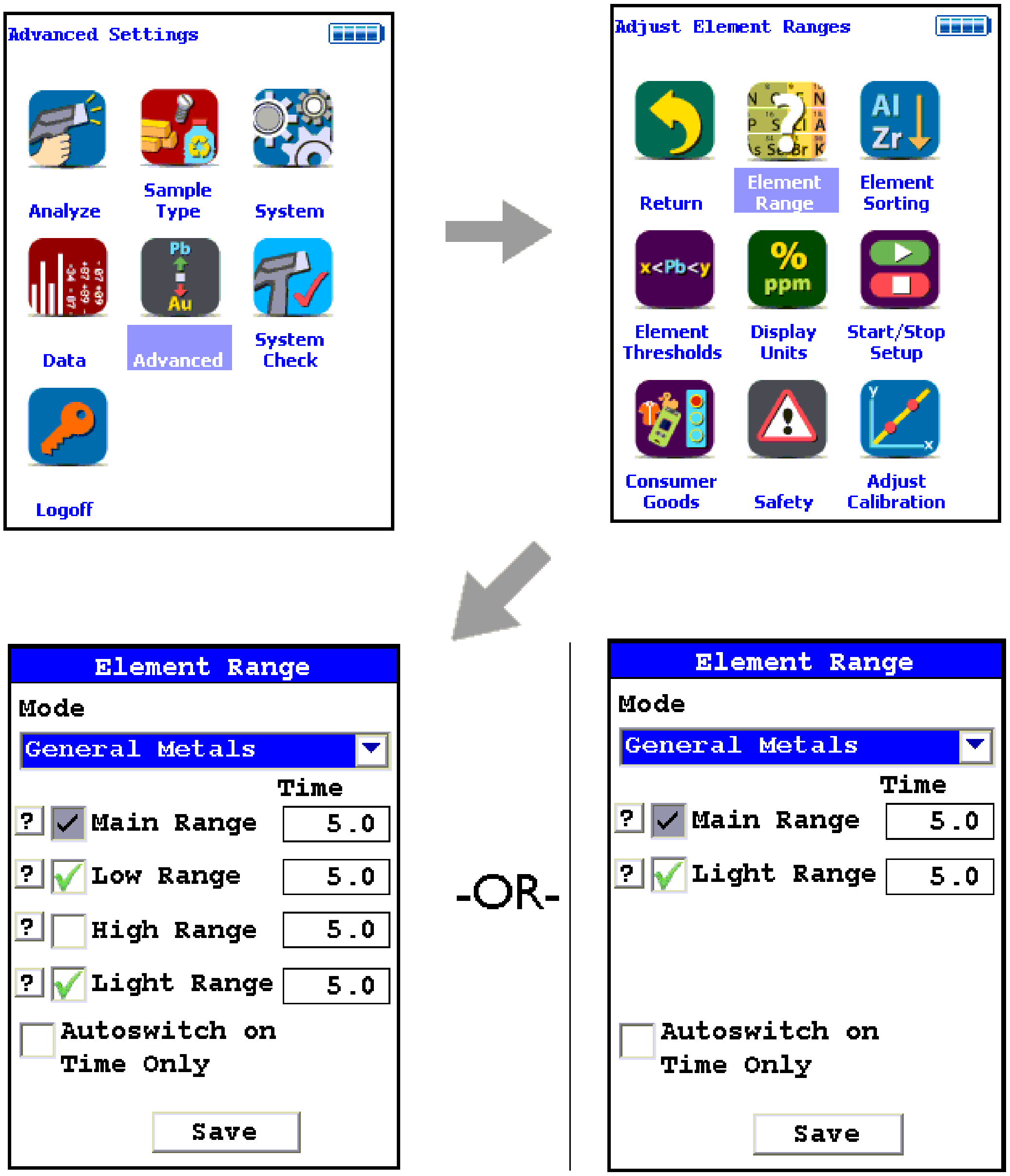
CAUTION Always obtain a Return Authorization (RA) number from Thermo Fisher Scientific's Service Department in the United States, toll free, at (800) 875-1578, or outside the United States, at +1-978-670-7460 before returning your analyzer to the Service Department or to your local Authorized Niton Analyzers Service Center.

CAUTION If you return your Niton analyzer without the carrying case, you will void your warranty in its entirety. You will be billed for a replacement case plus any repairs resulting from improper shipping.

CAUTION CAUTION Always remove the battery pack when transporting or storing your analyzer.

Advanced Settings

Adjusting the Element Range



Element Range

Mode: General Metals

?		Time
?	☑ Main Range	5.0
?	☑ Low Range	5.0
?	☐ High Range	5.0
?	☑ Light Range	5.0

☐ Autoswitch on Time Only

Save

-OR-

Element Range

Mode: General Metals

?		Time
?	☑ Main Range	5.0
?	☑ Light Range	5.0

☐ Autoswitch on Time Only

Save

Figure 1. **The Range Menu Path (Main)**

Multi-Range tests are used to either preferentially excite specific elements for increased sensitivity, or to cover a wider element range than one Range alone can provide. Most modes, when enabled, will use two Ranges in sequence to produce a combined analysis result. In typical alloy analysis applications, Main Range is used for the analysis of most elements, Low Range is utilized for the subsequent high sensitivity analysis of V, Ti, and Cr, and Light Range is available only with He-purged and 900 series GOLDD technology analyzers, and is typically used in light element analysis. Multi-Range switching can be set to activate off time alone, or, when time switching is disabled, off settings in the General Metals grade library. In most modes, Low and Light Range add the capability to analyze light elements which cannot be efficiently excited by Mid Range.

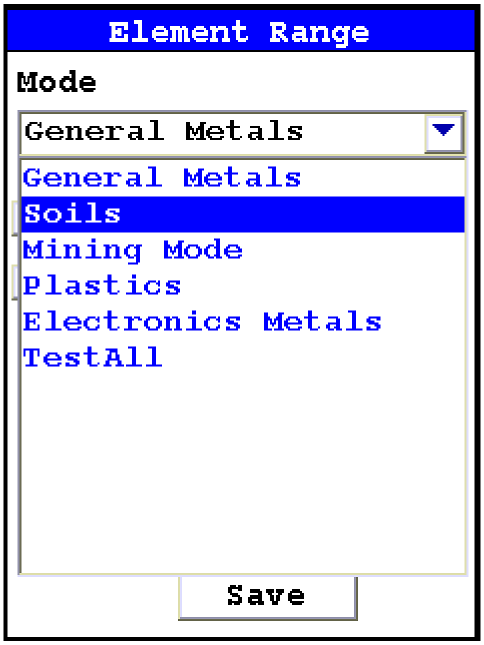


Figure 2. **Selecting the Mode**

Select the mode you wish to configure. You can set different configurations for different modes.

The Element Range Screen enables you to directly enable or disable any Range, or control the time that a Range alters the irradiation of the sample before auto-switching to another Range.

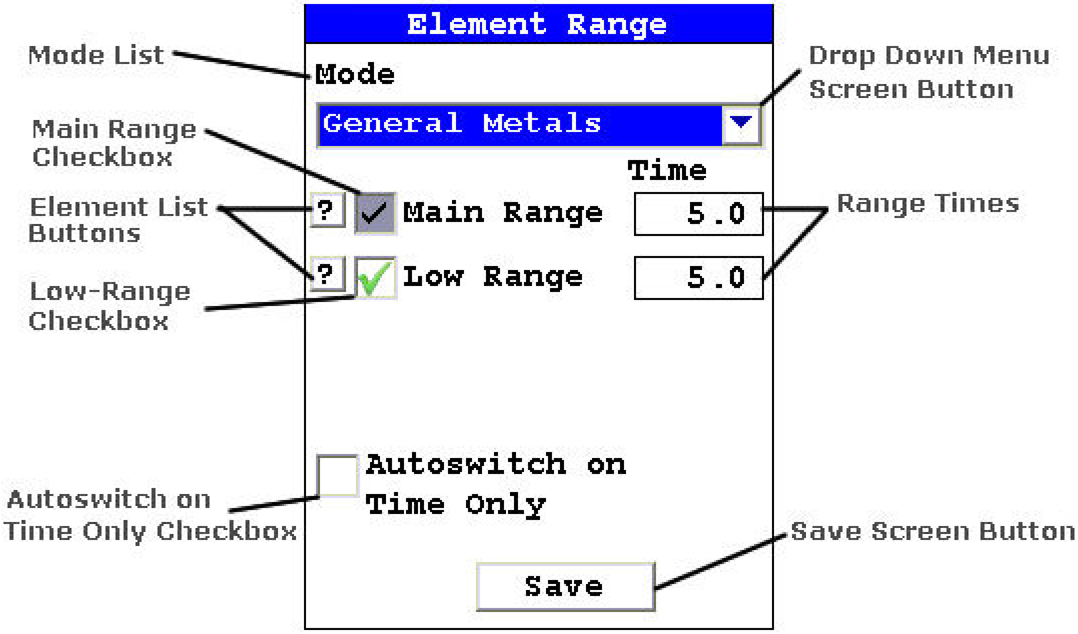
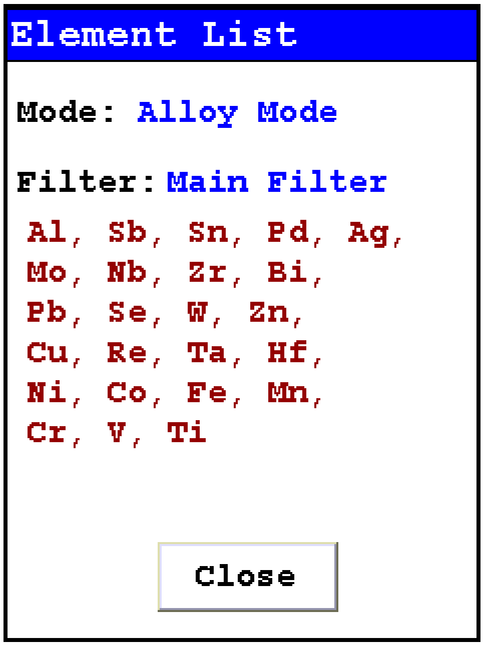


Figure 3. The Element Checkboxes

Select the checkbox next to the Range you want to use to determine exactly which of the Ranges contained in your Analyzer is used for sample testing. Selecting an empty checkbox will enable that range and place a check into the box as an indicator. Selecting a checked box will disable the Range and clear the box.

In typical alloy analysis applications, Main Range is used for the analysis of most elements. You cannot deselect the Main Range in alloy analysis

Low Range is utilized for the subsequent high sensitivity analysis of V, Ti, and Cr.

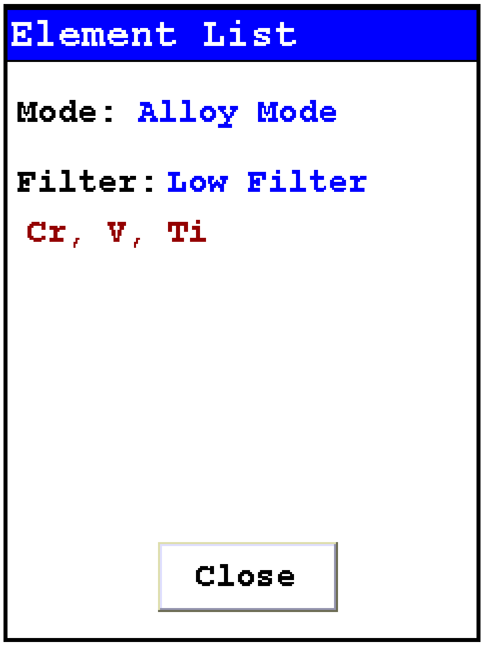


Element List

Mode: Alloy Mode

Filter: Main Filter

Al, Sb, Sn, Pd, Ag,
Mo, Nb, Zr, Bi,
Pb, Se, W, Zn,
Cu, Re, Ta, Hf,
Ni, Co, Fe, Mn,
Cr, V, Ti

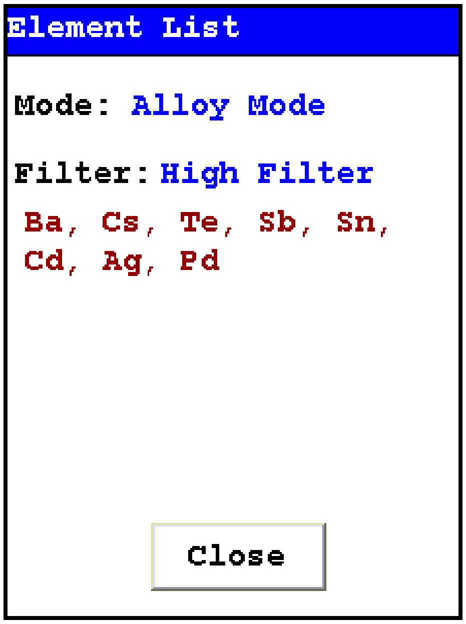


Element List

Mode: Alloy Mode

Filter: Low Filter

Cr, V, Ti

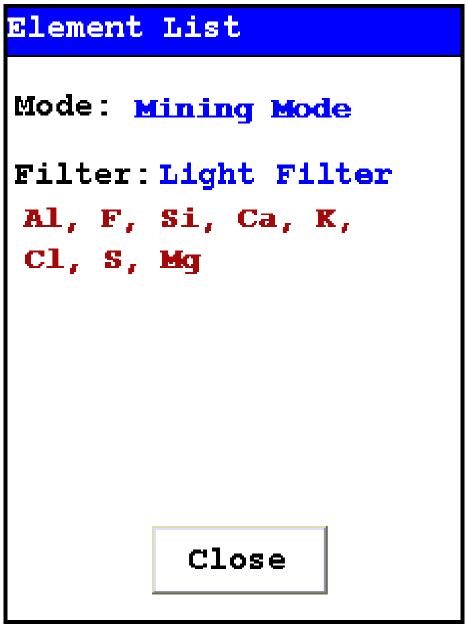


Element List

Mode: Alloy Mode

Filter: High Filter

Ba, Cs, Te, Sb, Sn,
Cd, Ag, Pd



Element List

Mode: Mining Mode

Filter: Light Filter

Al, F, Si, Ca, K,
Cl, S, Mg

Figure 4. The Range Element Lists

Select the Element List Button to display the Element List for that Range. This list shows the elements that the Range is best designed to detect.

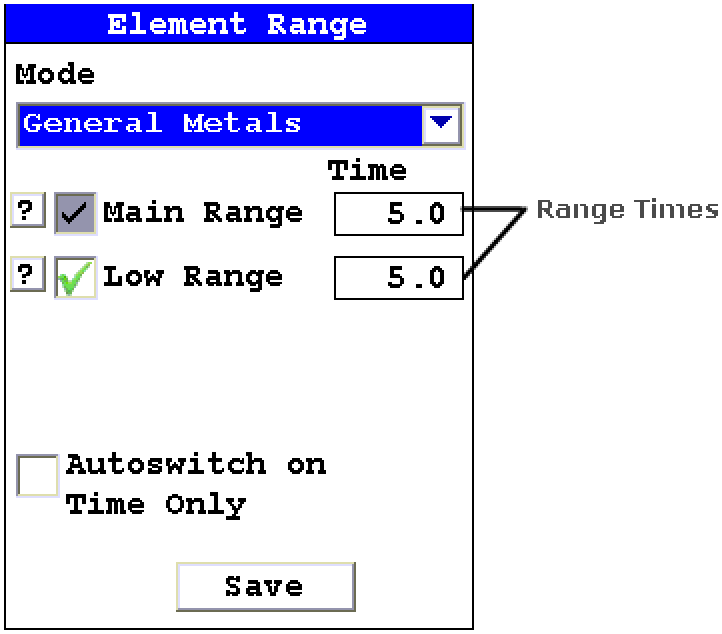
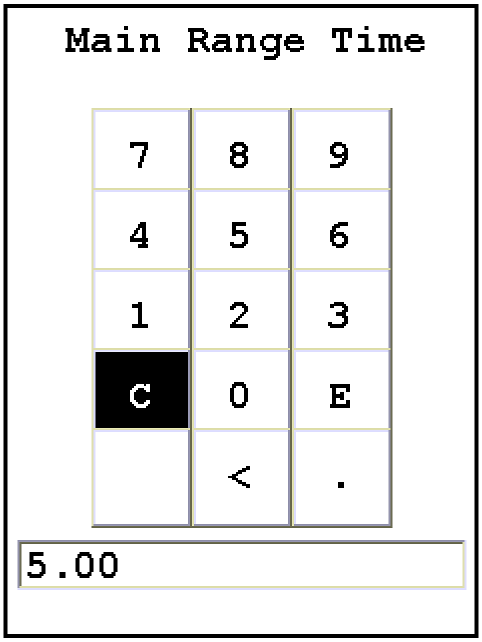


Figure 5. The Range Time Fields

Select the Range Time field for the intended range to change the switch time for that range. The Range Time Editor will appear. This enables you to set the number of seconds each enabled range is allotted before auto-switching will occur when needed during sample testing. Your analyzer will auto-switch from one range to another when the testing time for that range is greater than or equal to the time you have chosen, and the identified alloy is flagged as needing the switch in the Niton Alloy Library.



5.00

Figure 6. The Range Time Editor

Select the C button to clear the current time, then from the virtual numeric key pad, select each digit you want to input, then select the E button to enter.

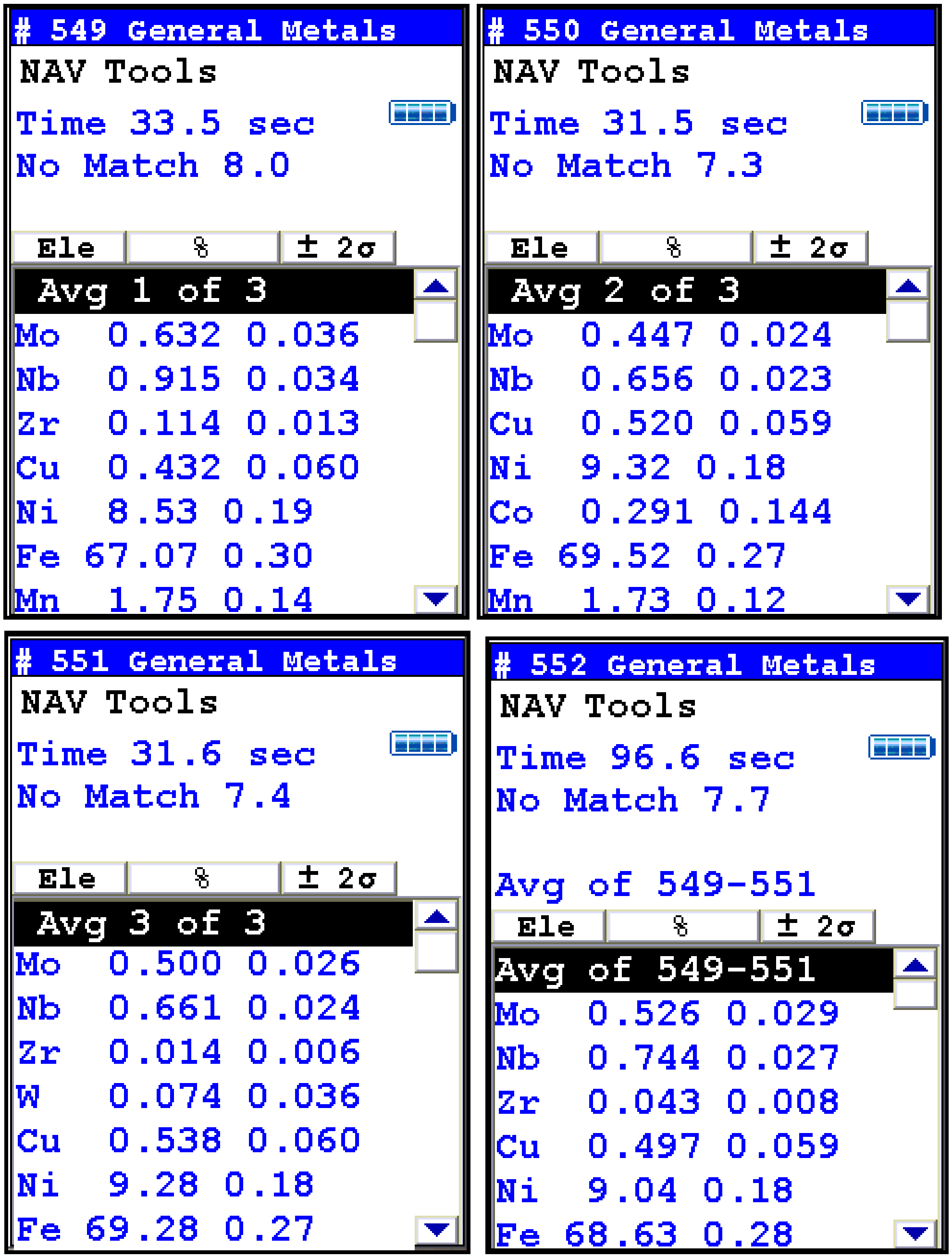
Tools Menu Options

The following options can be performed from the Tools Menu. Options which are only on the main Tools Menu are listed as (Main). Those which can be found only on the alternate Tools Menu are listed as (Alt).

Avg Forward

Enables you to average different readings together from this analysis forward. Select the Avg Forward button to initiate future sample averaging. Avg Forward will set up an automatic personal averaging protocol to be followed until your analyzer is shut down, or this feature is disabled. To begin, select the number of readings you want to average from the virtual numeric keypad. Your analyzer will calculate an average reading after that number of tests, and continue this pattern until stopped. For example, if you select 3 on the virtual keypad, the analyzer will automatically calculate, average, and store a reading for every three tests you take, storing the individual readings along the way.

The range number is selected using a virtual numeric keypad on your analyzer similar to the keypad used for login. Select the digits in the range number from the keypad, then select the E button to enter the number. The C button will clear all, and the "<" button will clear the last digit entered. The average will automatically be displayed.



```
# 549 General Metals
NAV Tools
Time 33.5 sec
No Match 8.0

Ele     %       ± 2σ
 Avg 1 of 3
Mo   0.632 0.036
Nb   0.915 0.034
Zr   0.114 0.013
Cu   0.432 0.060
Ni   8.53 0.19
Fe 67.07 0.30
Mn   1.75 0.14
```

```
# 550 General Metals
NAV Tools
Time 31.5 sec
No Match 7.3

Ele     %       ± 2σ
 Avg 2 of 3
Mo   0.447 0.024
Nb   0.656 0.023
Cu   0.520 0.059
Ni   9.32 0.18
Co   0.291 0.144
Fe 69.52 0.27
Mn   1.73 0.12
```

```
# 551 General Metals
NAV Tools
Time 31.6 sec
No Match 7.4

Ele     %       ± 2σ
 Avg 3 of 3
Mo   0.500 0.026
Nb   0.661 0.024
Zr   0.014 0.006
W    0.074 0.036
Cu   0.538 0.060
Ni   9.28 0.18
Fe 69.28 0.27
```

```
# 552 General Metals
NAV Tools
Time 96.6 sec
No Match 7.7

Avg of 549-551
Ele     %       ± 2σ
Avg of 549-551
Mo   0.526 0.029
Nb   0.744 0.027
Zr   0.043 0.008
Cu   0.497 0.059
Ni   9.04 0.18
Fe 68.63 0.28
```

Figure 7. Example Averaging Screens

Avg Back (Alt)

Note The alternate Tools Menu is only available when viewing readings, and the menu is only accessible through the touch screen interface or NDTr.

Enables you to average different readings together from this analysis backward. Select the Avg Back option to initiate backwards sample averaging. Avg Back will take the number of readings you select and average their analytical results. The range is counted from the last reading backward by the number of readings selected. If your last reading was #15, selecting 3 would average readings #13, 14, and 15. The average is calculated, displayed, and stored into memory as the next sequential reading number, in this case, #16.

The range number is selected using a virtual numeric keypad on your analyzer similar to the keypad used for login. Select the digits in the range number from the keypad, then select the E button to enter the number. The C button will clear all, and the “<“ button will clear the last digit entered. The average will automatically be displayed.

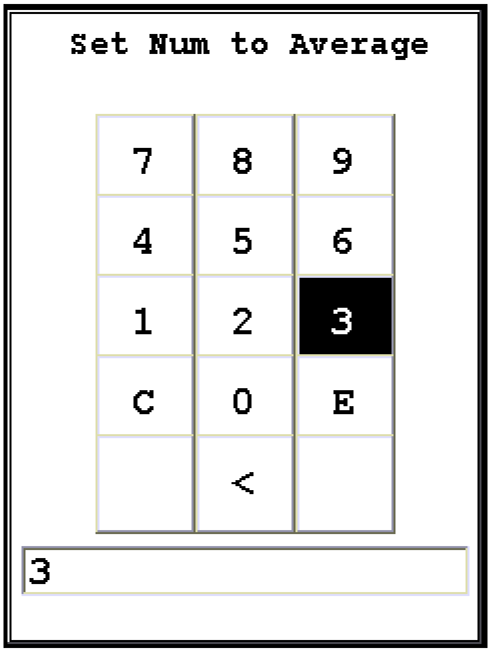


Figure 8. The Virtual Numeric Keypad

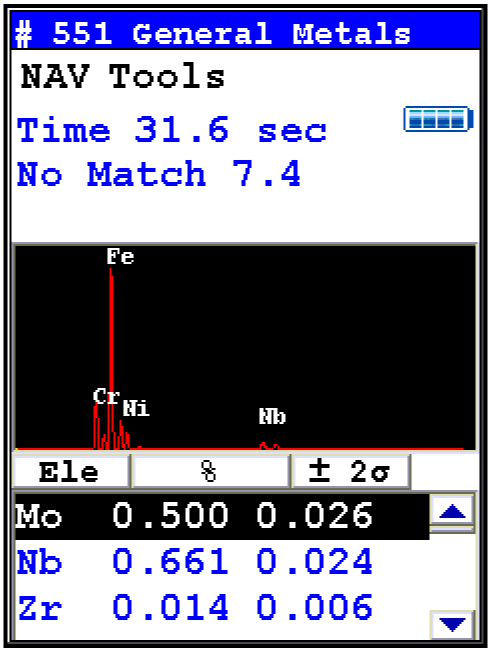
Note You cannot average readings taken in different modes. Doing this will generate an error.

Spectrum:On/Spectrum:Off

The Tools Menu contains a toggle option to display live spectra as sample analysis occurs.

Activating and Deactivating the Live Spectrum

From the Tools Menu, select the Spectra:On button to turn the Spectrum feed on. Once the spectrum is displayed, selecting Spectra:Off from the Tools Menu will stop the live spectrum display.



551 General Metals

NAV Tools

Time 31.6 sec

No Match 7.4

Ele	%	± 2σ
Mo	0.500	0.026
Nb	0.661	0.024
Zr	0.014	0.006

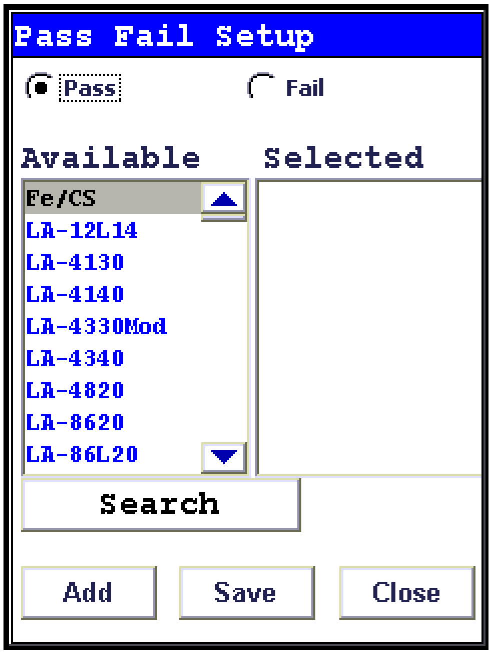
Figure 9. Example Analysis Screen Showing Live Spectrum

Print (Alt)

Select the Print option from the Tools Menu to print the current analysis screen to any attached portable printer. If you do not have a portable printer attached to your analyzer, nothing will happen.

Set Pass/Fail

You can set up your analyzer to sort on a pass/fail basis. Pass/Fail uses the chemistry of a user-generated list of alloys in the library as a basis for comparison. If the sample analysis is entirely within the specifications for one of these alloys, a PASS result is given, otherwise a FAIL result is returned. To turn on Pass/Fail, select the Tools Menu and select Set Pass/Fail from the menu. The Pass/Fail Setup Screen will come up.



● Pass ○ Fail

Figure 10. Set Pass/Fail Screen

Add/Remove (Toggle)

Select alloys from the Available list and then the Add Button to move the alloy to the Selected List. Select alloys from the Selected list and then the Remove Button to remove the alloys from the Selected List.

Save

Select the Save Button to save these criteria.

Close

Select the Close Button to exit without saving.

Pass

Select the Pass Single button to initiate Pass Mode. Use Pass Mode when you have a desirable match. If the alloy being analyzed matches one of the alloys in the selected list, the alloy will Pass the analysis.

Fail

Select the Fail button to initiate Fail Mode. Use Fail Single Mode when you have an undesirable match. If the alloy being analyzed matches one of the alloys in the selected list, the alloy will Fail the analysis.

Setting the Reference Alloys for Pass or Fail

Before you use Pass or Fail mode, you need to set the Reference Alloys. Select the alooy or alloys from the slide down menu on the Pass Fail Setup Screen, then select the Add button.

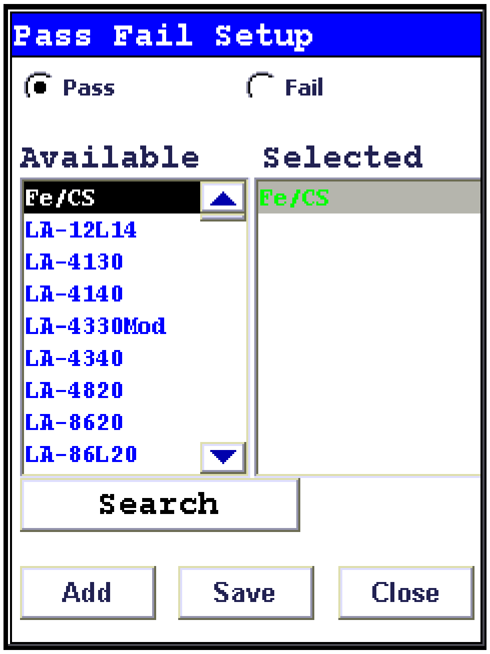
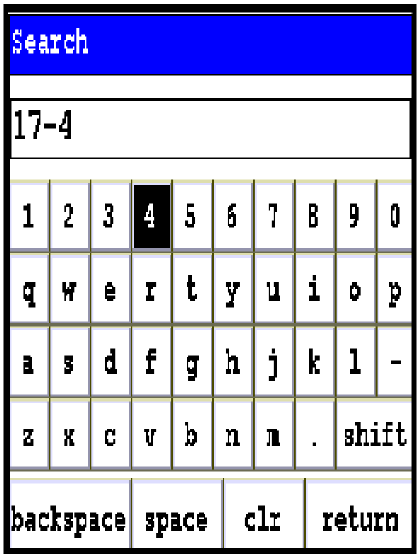


Figure 11. . The Pass Fail Setup Screen

Searching for Reference Alloys

Select the Search button to search the library for the alloy you want as your Reference Alloy.



17-4

Figure 12. Using the Search Function

Type the name of your reference alloy into the Virtual Keyboard, and the left column will display any matches. Select the match you want and the Add button to make it your reference alloy.

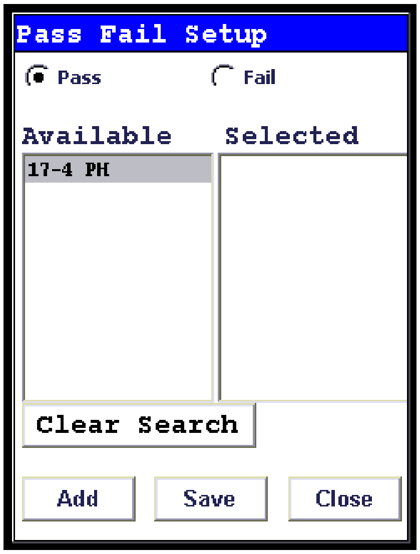


Figure 13. Search Results

UHow Pass/Fail Mode Works

Pass/Fail Mode compares the chemistry to that of the alloy(s) selected, using the cutoff you selected. When the sample analysis reaches a match with the chemistry of any one of the alloys on the Selected list, a PASS or FAIL notice is generated as appropriate.

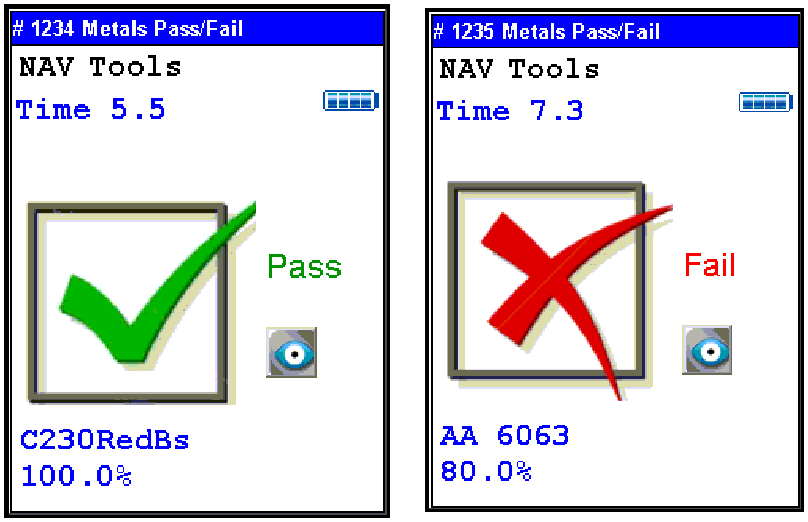


Figure 14. Metals Pass and Fail Screens

Switch Library (Main)

Selecting the Switch Library button from the Tools Menu will swap the currently loaded library with the other library on the analyzer. Selecting Switch Library again will switch them back.

Enable/Disable Al

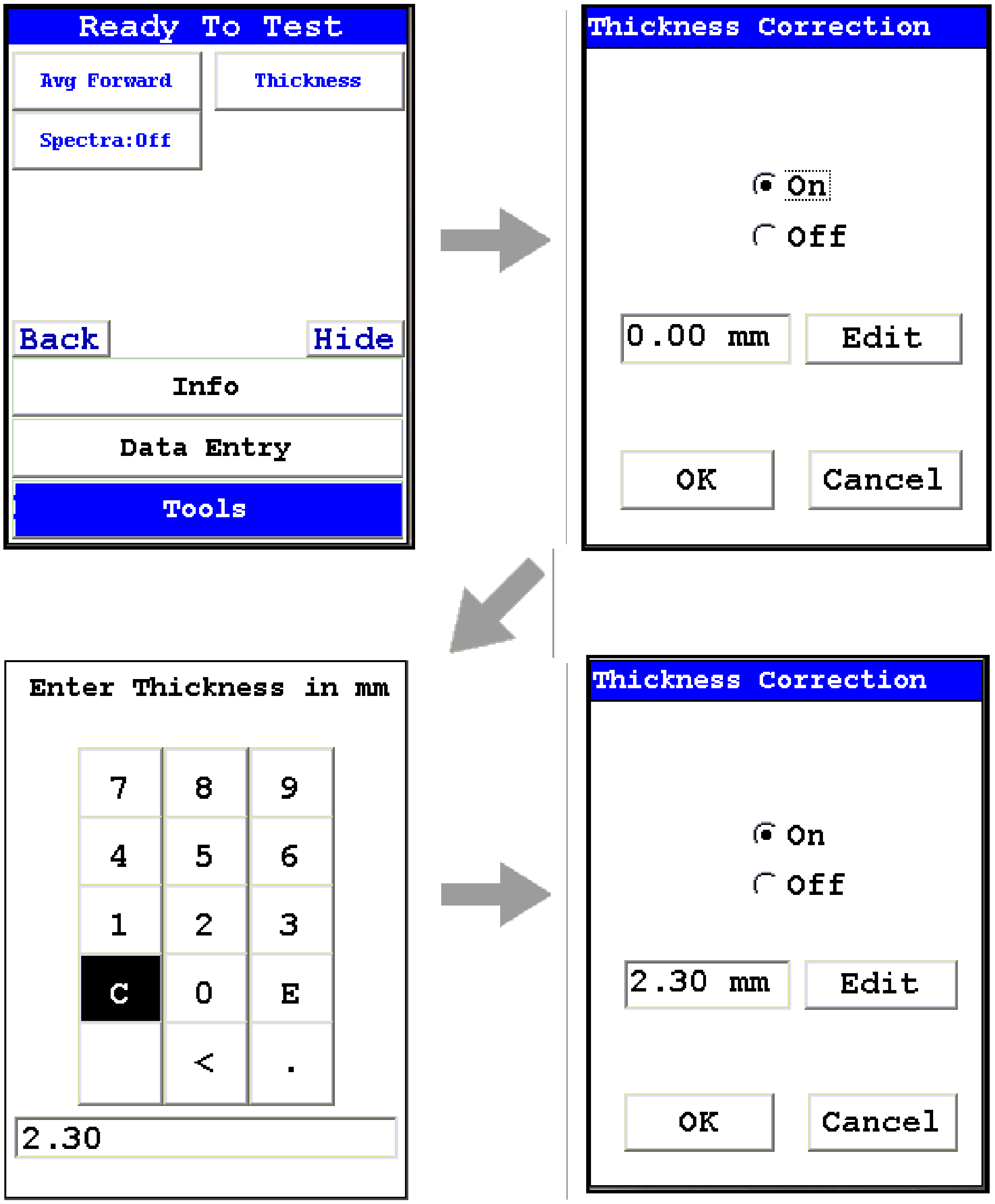
Normally, the collective amount of unquantifiable light elements in alloy analysis - the "balance" - is assumed to be aluminum and labeled as such in the analysis. Selecting the Disable Al button from the Tools Menu will delete this "aluminum" from the analysis results, showing only the quantified elements. Selecting the Enable Al button, the default state, will label this "balance" as "aluminum".

Thickness Correction

Plastics, and polymers in general, unlike metals or soil, are very weak absorbers of X rays. This is because polymers are composed mainly of very light elements such as carbon and hydrogen. While just half a millimeter of steel will completely stop 23.1 keV energy X rays of cadmium, for example, it takes at least 10mm of plasticized PVC and as much as 100mm of polyethylene (PE) to do so. Fortunately, polymers that may contain cadmium (Cd), lead (Pb) and other restricted elements would also contain considerable quantity of elements such as antimony (Sb), bromine (Br), titanium (Ti), etc. Their presence results in much stronger absorption of X rays which means that, instead of 100mm, it takes only about 15mm of compounded PE to achieve saturation thickness for these X rays. If the thickness of analyzed polymer sample is less than 5mm for PVC or less than about 9mm for a “typical” PE, the measured intensity of X rays will be a function of both analyte concentration and sample thickness. This is why measurements performed on thin samples (less than saturation thickness) need to be corrected for thickness.

How to apply Thickness Correction.

In order for the instrument to apply thickness correction to the measured concentration results, the user must be using the Thickness Correction screen and enter the thickness of the analyzed plastic object expressed in [mm] before the measurement is initiated. The thickness may be entered with precision to the second decimal place, although in practice only one decimal place is sufficient for effective correction.



● On
○ Off
0.00 mm

2.30

● On
○ Off
2.30 mm

Figure 15. How to Enable and Adjust Thickness Correction for Plastics Analysis

When to use Thickness Correction

Thickness Correction should only be used during the analysis of plastic (polymer) objects. It has been experimentally verified that the correction algorithm will yield satisfactory results, for a 60 second minimum testing time, for samples as thin as 0.3mm. Nevertheless, the recommended range of use of the correction is from 1mm upwards. It is imperative that this correction is not used for thin films such as single foils and plastic membranes; analysis of thin films is performed using the Thin Sample Mode. (Contact Contact Thermo Scientific or your local Niton Analyzers representative for information on this testing mode.)

Whenever possible, one should analyze as thick a sample as available. For example, if the analyzed object is a piece of heatshrink tubing with wall thickness of 0.3mm, the best way to analyze it is to obtain several pieces of the tubing (four for example) and stack them like a flat sandwich, with the thickness correction set to 1.2mm. Doing so makes for faster and more precise analyses. While it would be possible to analyze just a single layer of the tubing with correction at 0.3 mm, by stacking several layers we reduce the relative error of measurement (by a factor approximately equal to the square root of the number of layers). Conversely, when analyzing thinner samples, we need to extend the measurement time fourfold (by the number of layers) in order to maintain the same relative error of measurement. We can see how quickly measurement time would escalate to impractical levels for thinner samples.

Examples: The most frequent instances in which thickness correction would be called for are analyses of plastic sheeting or plastic insulation on wires and/or cables and heat shrink tubing. Flat plastic sheeting or plastic enclosures pose no problems. We can either analyze an object “as is”, or stack several layers of it before analysis. Plastic insulation such as that on wiring or cables requires a little more sophisticated approach. First, the wire must be removed so that only insulation is analyzed. Then, the insulation should be flattened for analysis, and a thickness correction should be applied that is equal to double the wall thickness. Alternatively, if the insulation is stiff, it should be cut lengthwise into strands which are placed on the indtrument for analysis. The applied thickness correction should be equal to the wall thickness of the sleeve. Both operations are shown in Figure 37 and Figure 38.

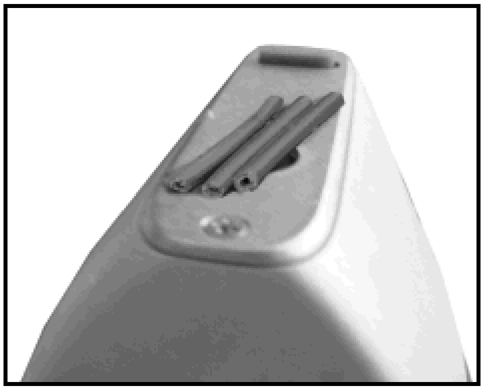


Figure 16. Wire Insulation Cut Into Strands

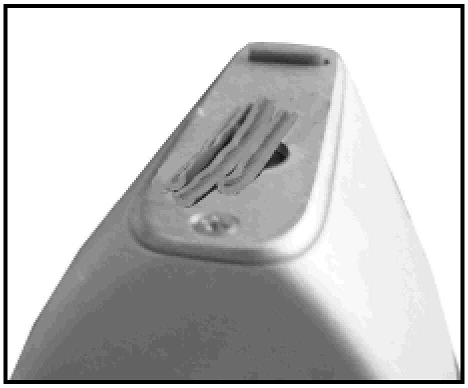


Figure 17. PVC Wire Insulation With Conductor Removed

A piece of large diameter heat shrink tubing presents an interesting case. It is tempting to analyze this object as is - see Figure 39. However, one needs to know that while lead or bromine or chromium X-rays from the upper wall of tubing will not contribute to the signal measured, X rays of such elements as cadmium, antimony, tin or barium in the upper wall will significantly contribute to overall signal. It is therefore imperative to either flatten the tubing for analysis or cut it in pieces and then analyze as shown in Figure 40.

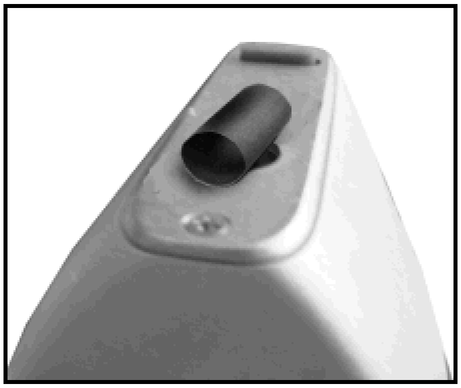


Figure 18. . Incorrect Way to Measure Heat Shrink Tubing

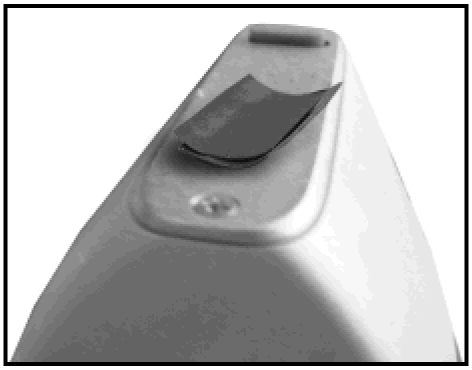


Figure 19. . Correct Way to Measure Heat Shrink Tubing

WARNING Thickness correction is only for use with plastic/polymer samples.

Enable/Disable Paint

Selecting the Enable Paint option from the Tools Menu will enable the Painted Products mode and toggle the option to Disable Paint. Selecting the Disable Paint option will disable Painted Products mode and toggle the option to Enable Paint.

Action Level

Selecting the Action Level option from the Tools Menu will anable you to change the action level used for qualitative testing.

Print Data

Selecting the Print Data option from the Tools Menu will print the currently displayed data to the optional attached printer. See Setting Up the Printer for details.

Coatings Method

Metals are sometimes coated with various materials. If you wish to analyze the coating, select the Coatings Method.

Passwords and User Privileges

1. Install the latest version of Niton software (NDT) on your PC, if possible. You may obtain the latest version of NDT by contacting service at 800-875-1578.

2. You can check the version number by opening NDT, selecting the Help menu, then selecting "About Niton Data Transfer"

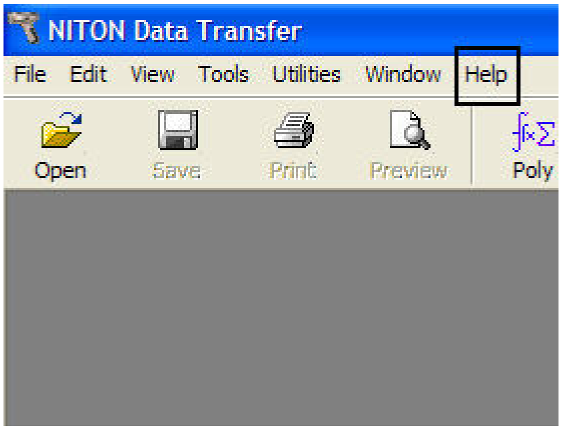


Figure 20. . Selecting Help

3. Select the File menu

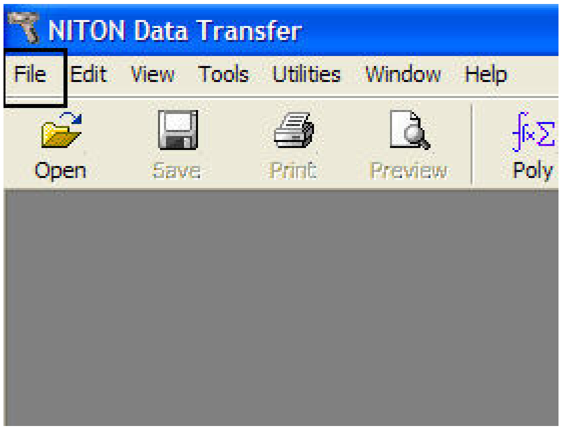
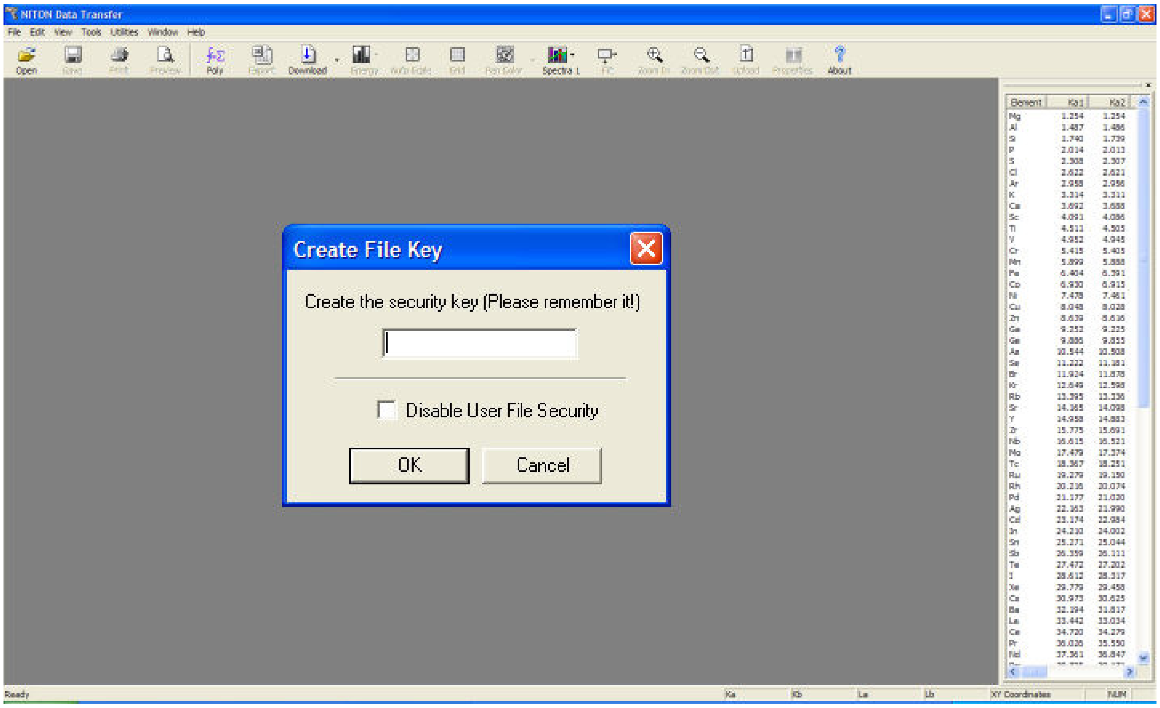


Figure 21. Selecting File

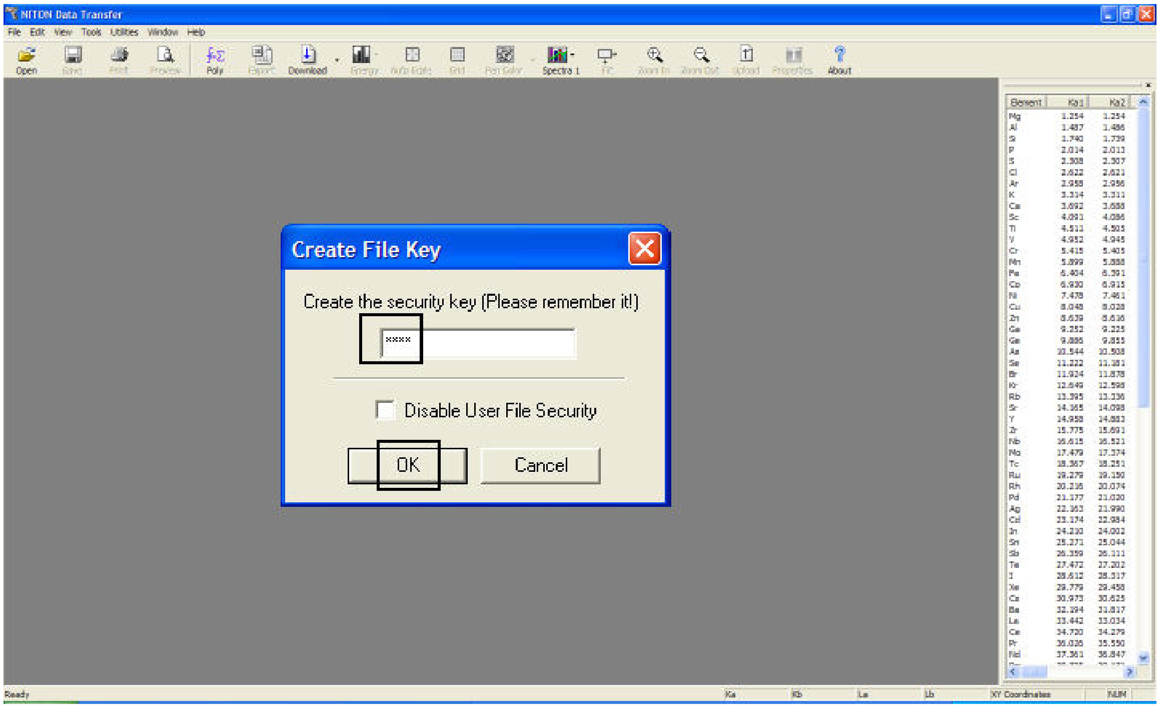
4. Select "New" then "New Password File". Your screen should look like this:



Create the security key (Please remember it!)

Figure 22. Creating the Security Key

5. Create a unique security key, then select the OK Button



Create the security key (Please remember it!)

****

Figure 23. Security Key

6. Your screen should look like this:

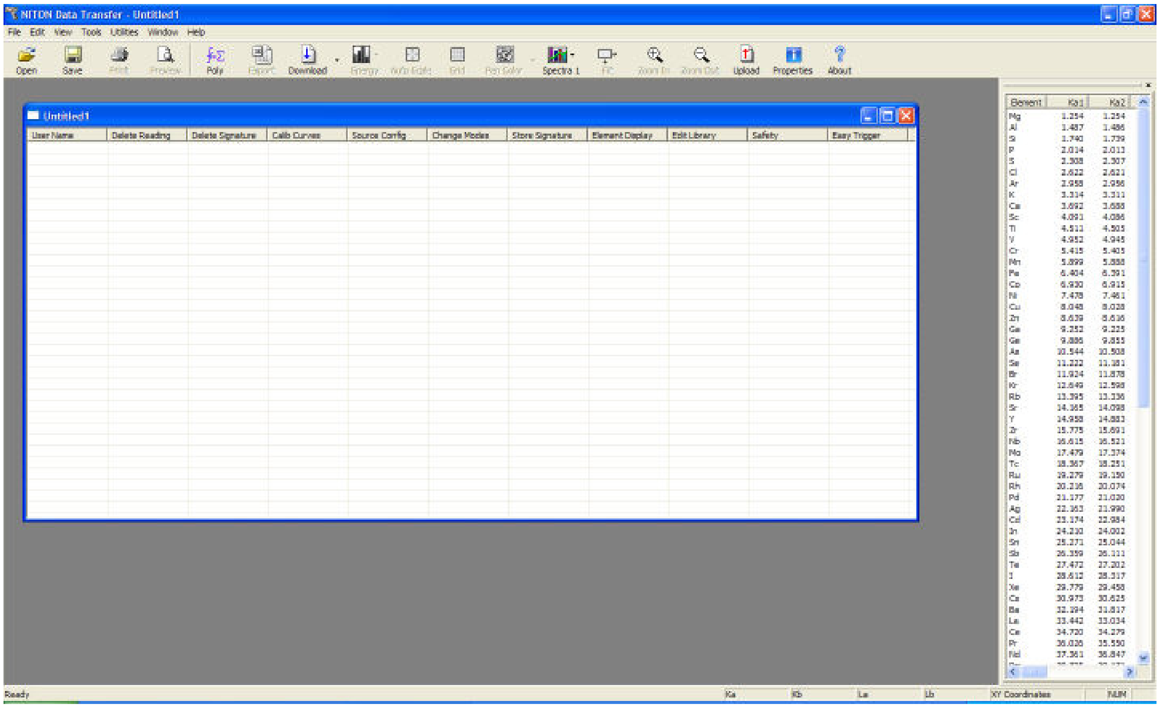
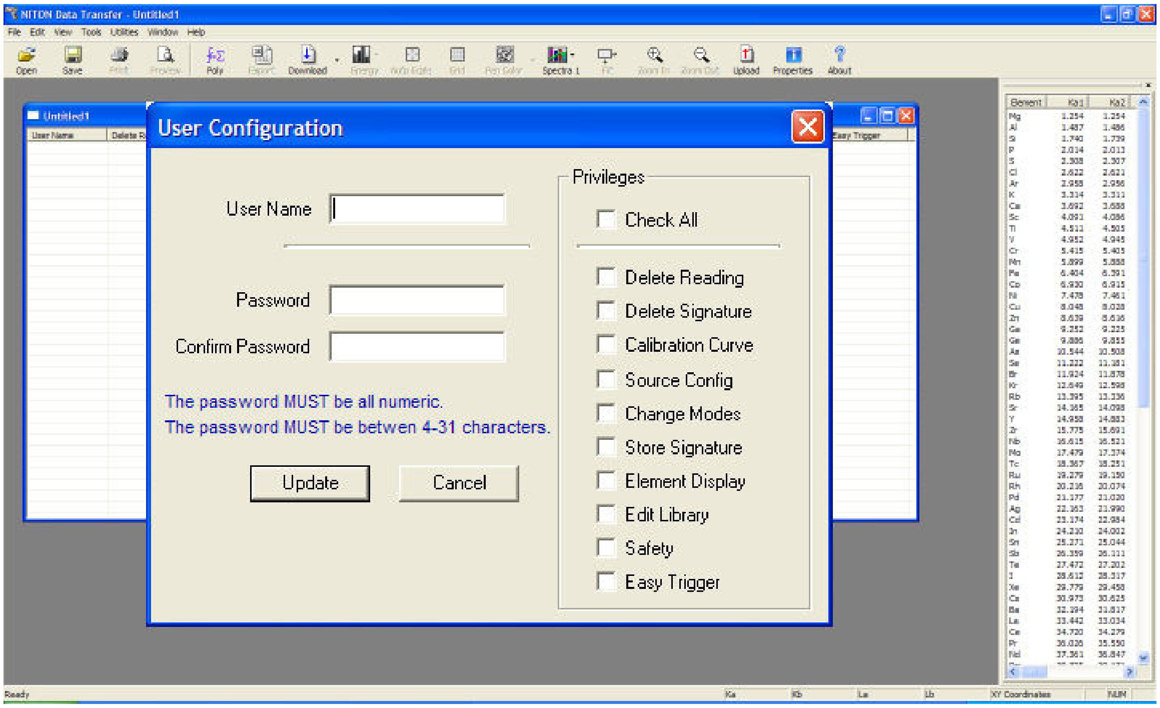


Figure 24. User Account Creation Screen

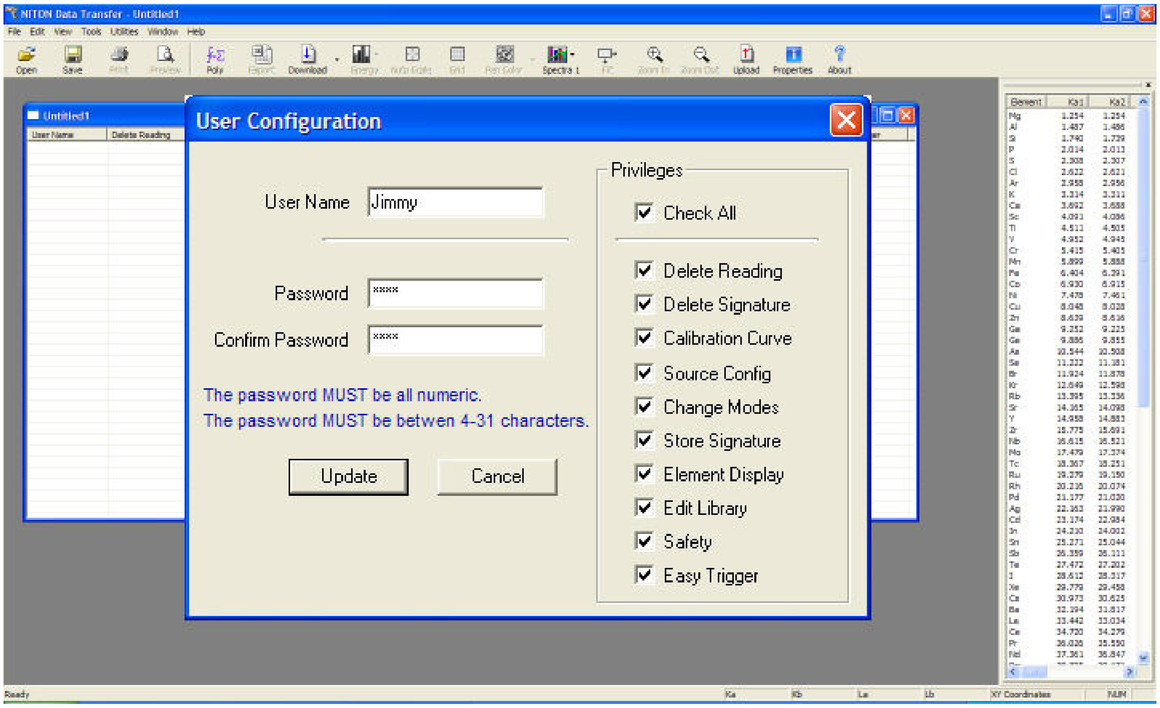
7. Right click, then select “New User”



The password MUST be all numeric.
The password MUST be betwen 4-31 characters.

Figure 25. User Creation Dialog Box

8. Enter a user name and password, then select the privileges assigned to this user. Selecting the Check All check box will result in enabling all features.



User Configuration

User Name: Jimmy

Password: ****

Confirm Password: ****

The password MUST be all numeric.
The password MUST be betwen 4-31 characters.

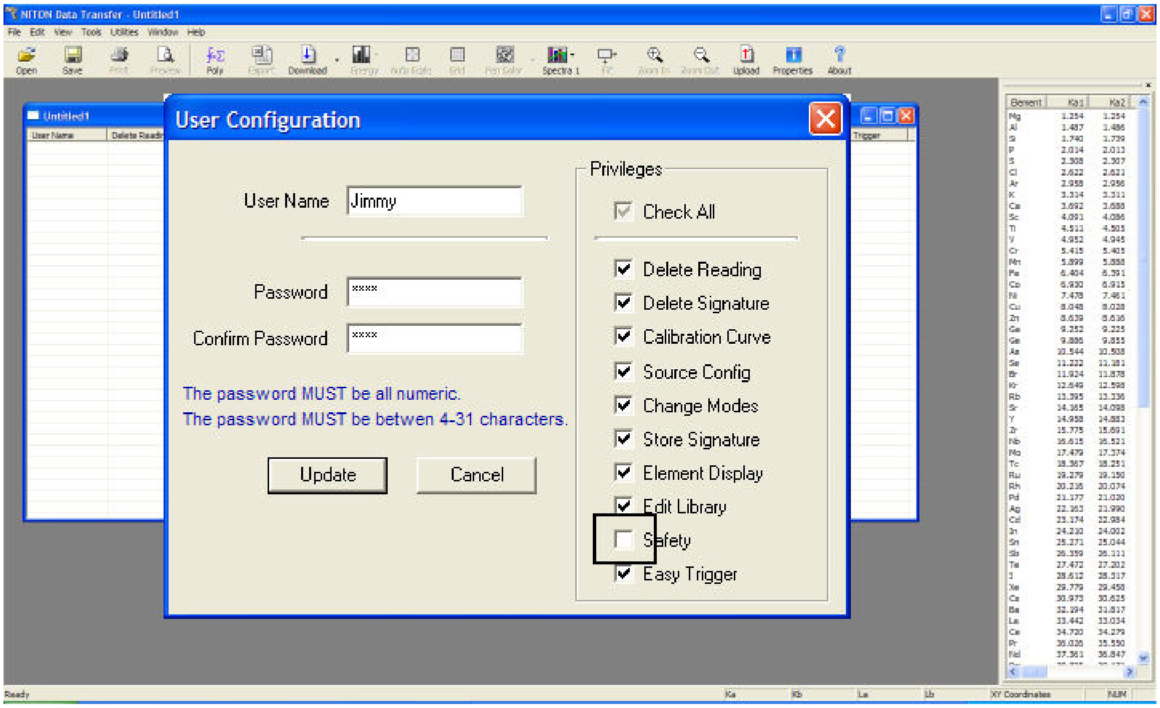
Privileges

☑ Check All

☑ Delete Reading
☑ Delete Signature
☑ Calibration Curve
☑ Source Config
☑ Change Modes
☑ Store Signature
☑ Element Display
☑ Edit Library
☑ Safety
☑ Easy Trigger

Figure 26. . Creating a User

WARNING it is recommended that only users at the highest level have access to the “Safety” feature. This should be unchecked for all other operators.



User Configuration

User Name: Jimmy

Password: ****

Confirm Password: ****

The password MUST be all numeric.
The password MUST be betwen 4-31 characters.

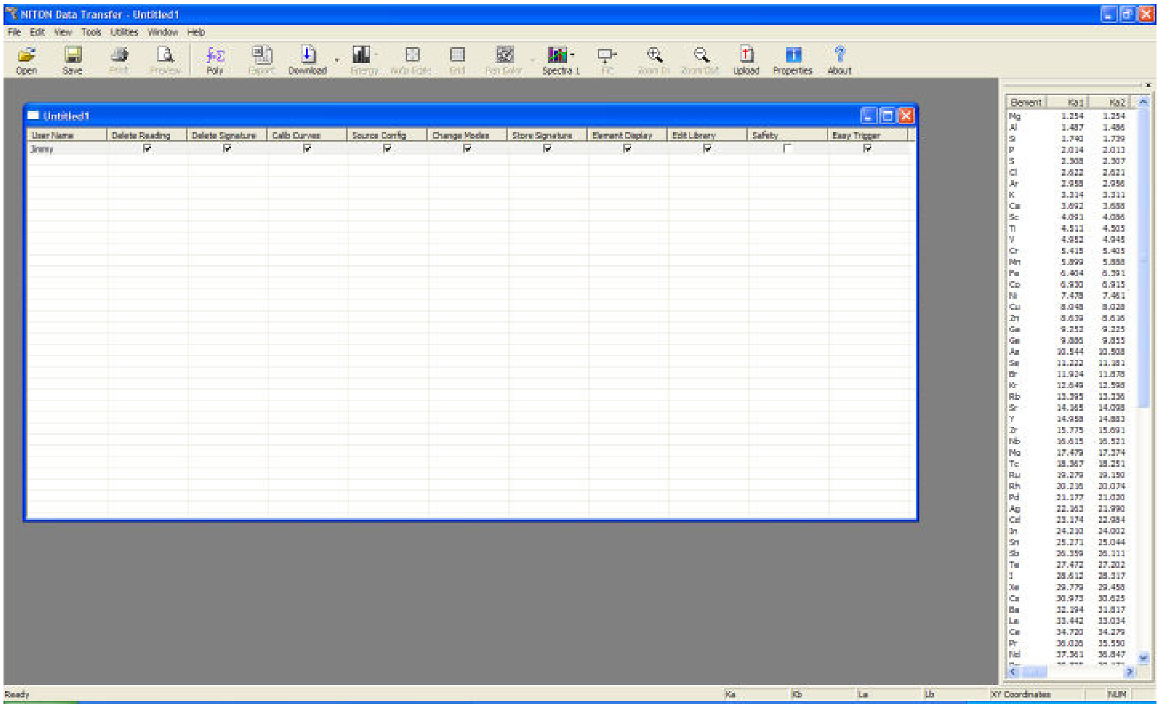
Privileges

☑ Check All

- ☑ Delete Reading
- ☑ Delete Signature
- ☑ Calibration Curve
- ☑ Source Config
- ☑ Change Modes
- ☑ Store Signature
- ☑ Element Display
- ☑ Edit Library
- ☐ Safety
- ☑ Easy Trigger

Figure 27. Unchecking Safety

9. Select the Update Button



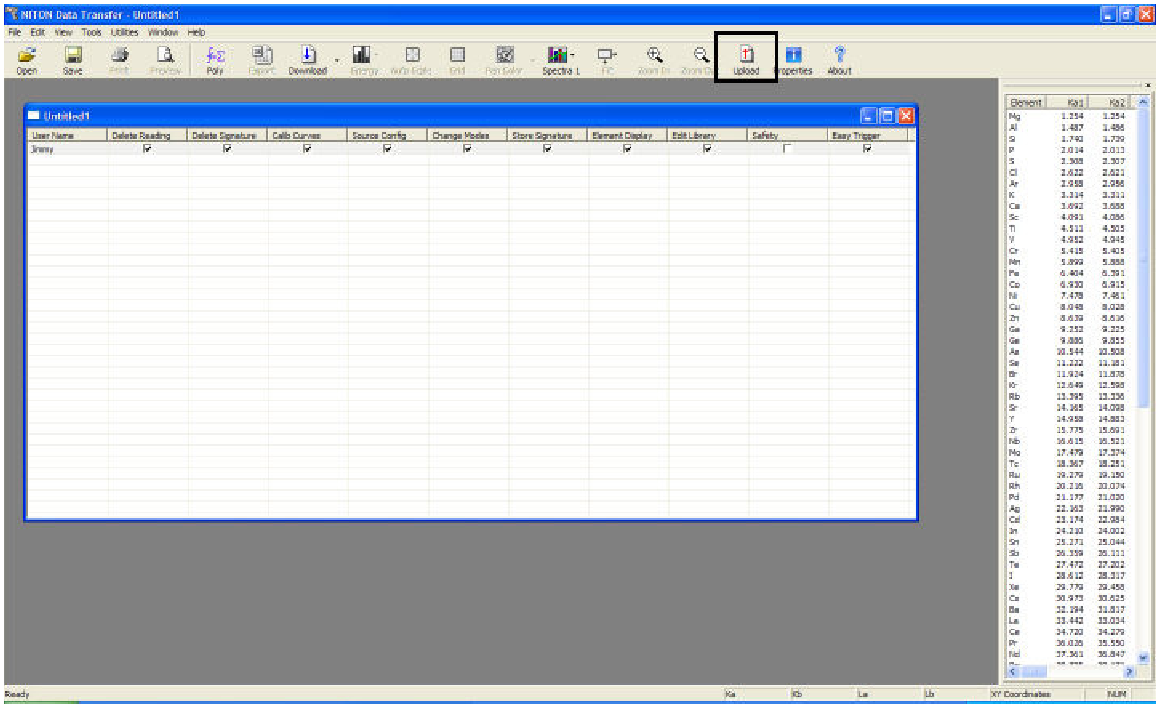
User Name	Delete Reading	Delete Signature	Calib Curves	Source Config	Change Modes	Store Signature	Element Display	Edit Library	Safety	Easy Trigger
Jimmy	☑	☑	☑	☑	☑	☑	☑	☑	☐	☑

Figure 28. User is Created

a. You are now ready to upload your password file to the analyzer.

10. Be sure the analyzer is switched on; connect the analyzer using USB or serial connection.

11. Select the Upload icon.

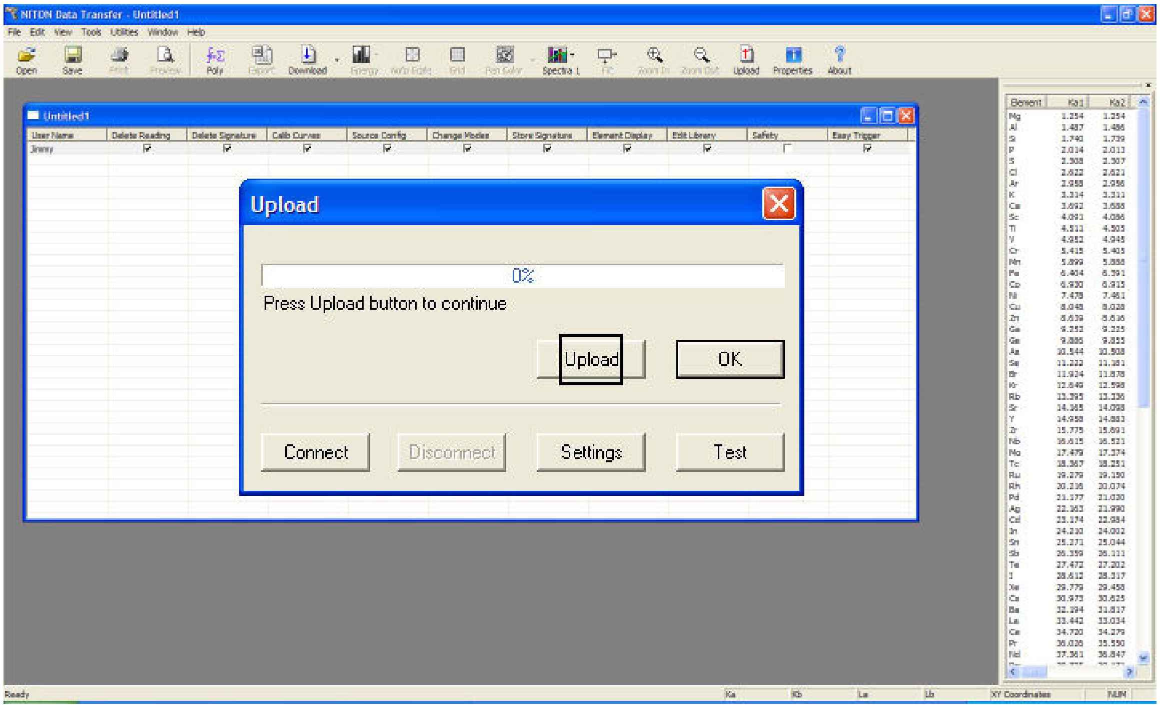


User Name	Delete Reading	Delete Signature	Calib Curves	Source Config	Change Modes	Store Signature	Element Display	Edit Library	Safety	Easy Trigger
Jimmy	☑	☑	☑	☑	☑	☑	☑	☑	☐	☑

Element	Ka1	Ka2
Mg	1.254	1.254
Al	1.487	1.486
Si	1.740	1.739
P	2.014	2.013
S	2.308	2.307
Cl	2.622	2.621
Ar	2.958	2.956
K	3.314	3.311
Ca	3.692	3.688
Sc	4.091	4.086
Ti	4.511	4.505
V	4.952	4.945
Cr	5.415	5.405
Mn	5.899	5.888
Fe	6.404	6.391
Co	6.930	6.915
Ni	7.478	7.461
Cu	8.048	8.028
Zn	8.639	8.616
Ga	9.252	9.225
Ge	9.886	9.855
As	10.544	10.508
Se	11.222	11.181
Br	11.924	11.878
Kr	12.649	12.598
Rb	13.395	13.336
Sr	14.165	14.098
Y	14.958	14.883
Zr	15.775	15.691
Nb	16.615	16.521
Mo	17.479	17.374
Tc	18.367	18.251
Ru	19.279	19.150
Rh	20.216	20.074
Pd	21.177	21.020
Ag	22.163	21.990
Cd	23.174	22.984
In	24.210	24.002
Sn	25.271	25.044
Sb	26.359	26.111
Te	27.472	27.202
I	28.612	28.317
Xe	29.779	29.458
Cs	30.973	30.625
Ba	32.194	31.817
La	33.442	33.034
Ce	34.720	34.279
Pr	36.026	35.550
Nd	37.361	36.847

Figure 29. Selecting Upload

12. Your screen should look like this:



0%

Press Upload button to continue

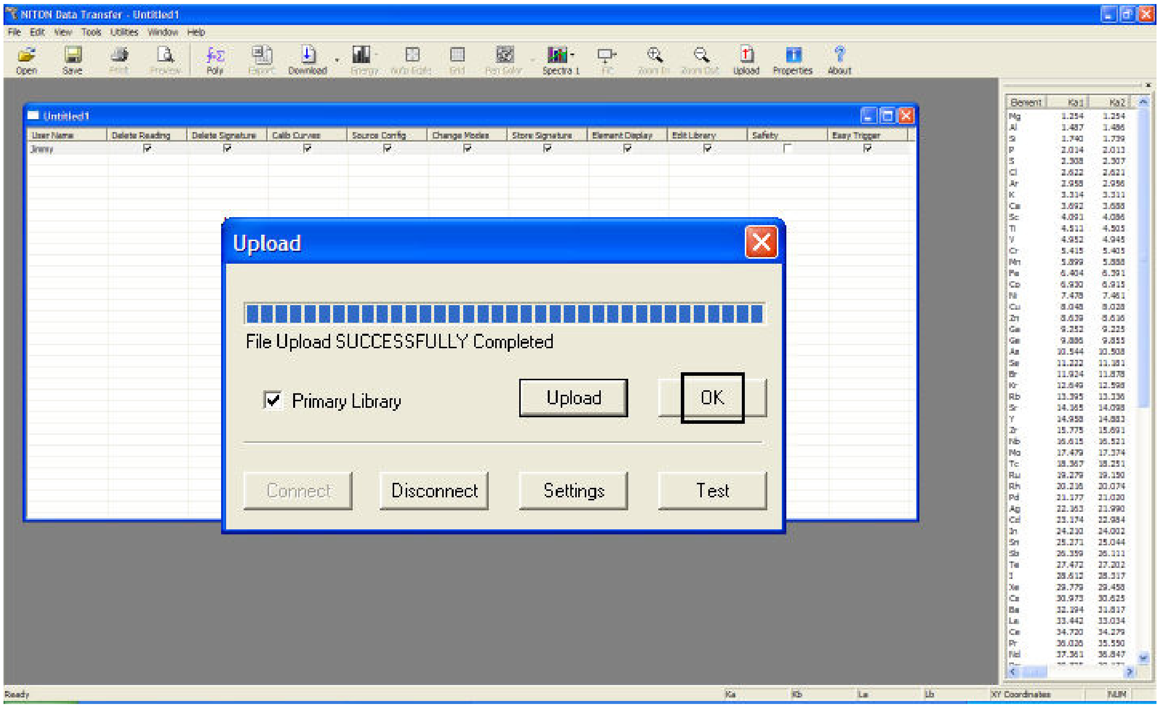
Figure 30. **Selecting Upload**

13. Select the Settings Button and choose the comm port that your analyzer is connect to.

14. Select the Connect Button, then the Upload Button.

15. Upon completion, you will receive a “File Upload Successfully Completed” message.

16. Click the OK Button; save your password file at this time by selecting the File icon then “Save As.



User Name	Delete Reading	Delete Signature	Calib Curves	Source Config	Change Modes	Store Signature	Element Display	Edit Library	Safety	Easy Trigger
Jimmy	☑	☑	☑	☑	☑	☑	☑	☑	☐	☑

File Upload SUCCESSFULLY Completed

☑ Primary Library

Element	Ka1	Ka2
Mg	1.254	1.254
Al	1.487	1.486
Si	1.740	1.739
P	2.014	2.013
S	2.308	2.307
Cl	2.622	2.621
Ar	2.958	2.956
K	3.314	3.311
Ca	3.692	3.688
Sc	4.091	4.086
Ti	4.511	4.505
V	4.952	4.945
Cr	5.415	5.405
Mn	5.899	5.888
Fe	6.404	6.391
Co	6.930	6.915
Ni	7.478	7.461
Cu	8.048	8.028
Zn	8.639	8.616
Ga	9.252	9.225
Ge	9.886	9.855
As	10.544	10.508
Se	11.222	11.181
Br	11.924	11.878
Kr	12.649	12.598
Rb	13.395	13.336
Sr	14.165	14.098
Y	14.958	14.883
Zr	15.775	15.691
Nb	16.615	16.521
Mo	17.479	17.374
Tc	18.367	18.251
Ru	19.279	19.150
Rh	20.216	20.074
Pd	21.177	21.020
Ag	22.163	21.990
Cd	23.174	22.984
In	24.210	24.002
Sn	25.271	25.044
Sb	26.359	26.111
Te	27.472	27.202
I	28.612	28.317
Xe	29.779	29.458
Cs	30.973	30.625
Ba	32.194	31.817
La	33.442	33.034
Ce	34.720	34.279
Pr	36.026	35.550
Nd	37.361	36.847

Figure 31. Successful Installation Message

17. Restart your analyzer; your password file should be successfully installed.

NDF Files: User Data Structuring

Creating New User-Defined Fields

You can create your own data entry fields for your Niton analyzer customized to your own needs and usage. These fields are saved in a special format called an NDF (Niton Data File) file. To create a new NDF file, select the File menu, then select New, then select New NDF File.

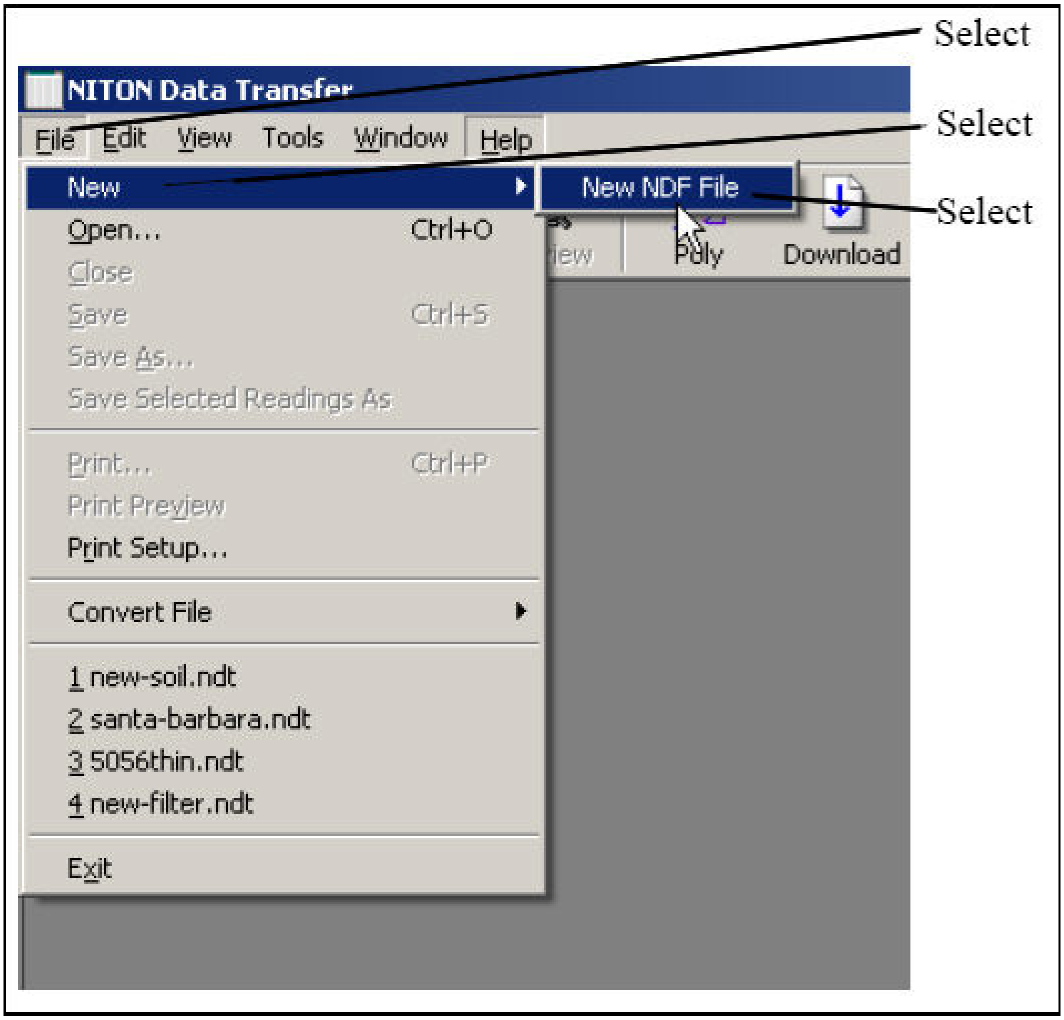
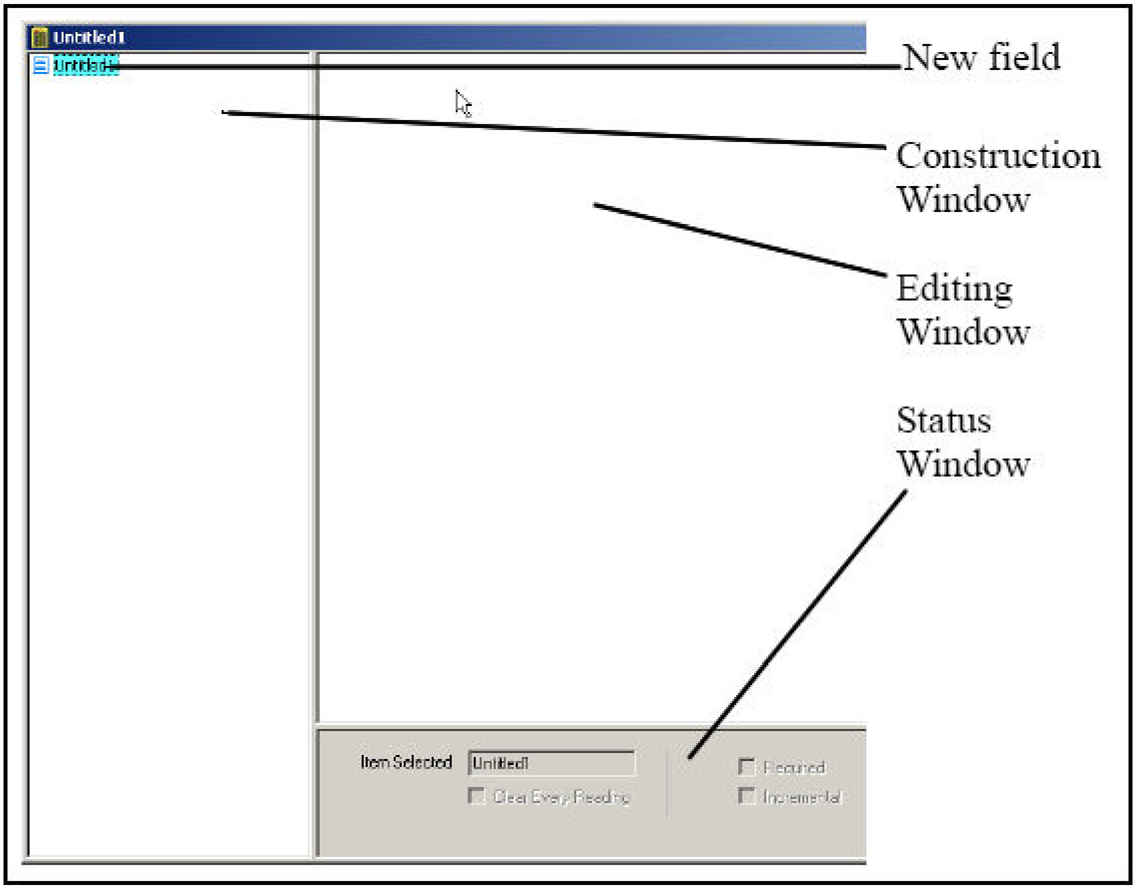


Figure 32. Creating a New NDF File

This will create a new window in which you can create your own fields, and specify their structure and parameters. The new window will appear with a single box, called “Untitled.”



Item Selected: Untitled1

Figure 33. NDF File Work Area

By right-clicking on this box, you can access a pop-up menu allowing you to set the mode of the new data fields. Select New Mode to access the menu.

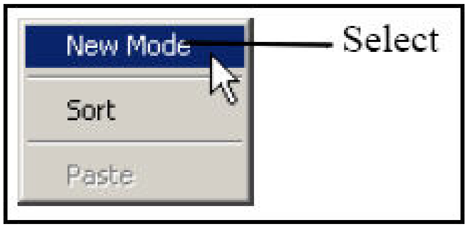


Figure 34. Selecting New Mode

The Mode you select will be the Mode within which the new data entry fields will appear. If you have multiple Modes enabled on your analyzer, the new fields will only be available from the Mode you select. Only the default fields will be available from the other Mode or Modes.

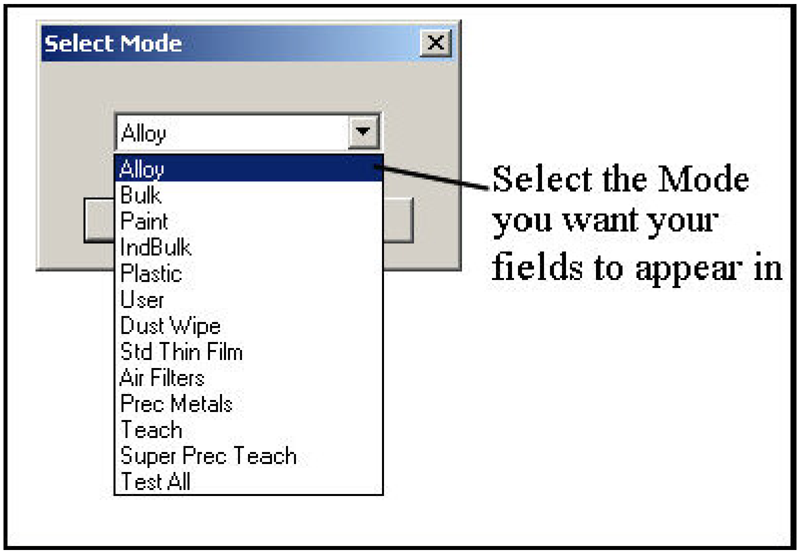


Figure 35. Selecting Mode

When you select the Mode for the new data fields, the Construction Window will change to look like this:

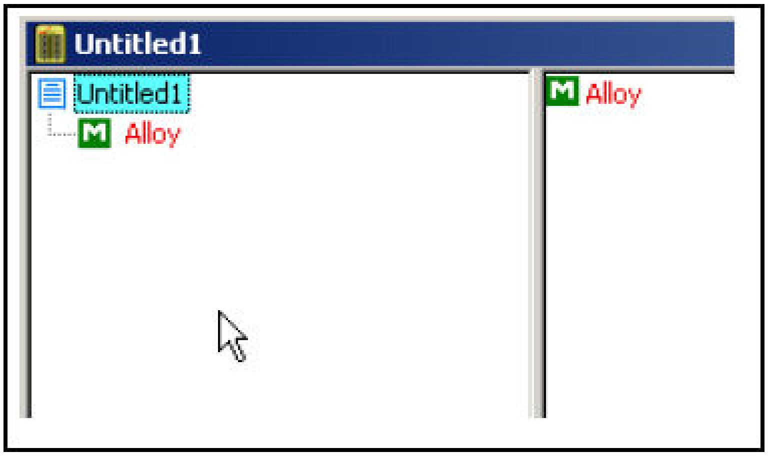


Figure 36. Working within a Mode

The “M” indicates the mode you have chosen - in this case Alloy Mode. Right click on the Mode name to access a pop-up menu.

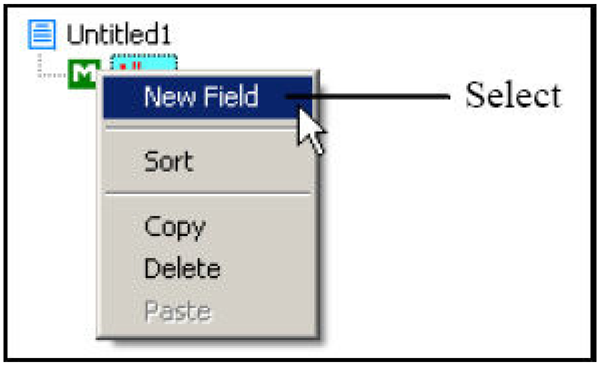


Figure 37. Mode Pop-Up Menu

Select New Field from the menu, and a blank new field will appear in the construction window.

Figure 38. Adding a New Field

Right clicking on the New Field box will bring up another pop-up menu. This menu gives you various options for using the field in your operations.

Selecting Required makes it mandatory that the new field be filled in prior to taking a measurement. This is very useful for necessary descriptors which vary from measurement to measurement, such as lot numbers, condi-tion descriptors, locations, etc.

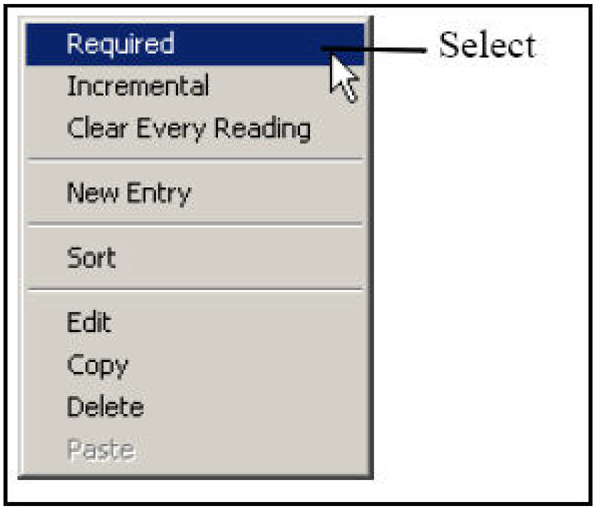


Figure 39. Making Fields Required

Selecting the Incremental option sets up a field which increments the field descriptor by one for each measurement taken. This option is handy for measuring several items with identical descriptors, such as samples within a single lot, or several instances of the same part number, because it appends the incremental number to the descriptor.

For example: P/N 455A2-1, P/N 455A2-2, P/N 455A2-3.

Another Example: Impeller-1, Impeller-2, Impeller-3.

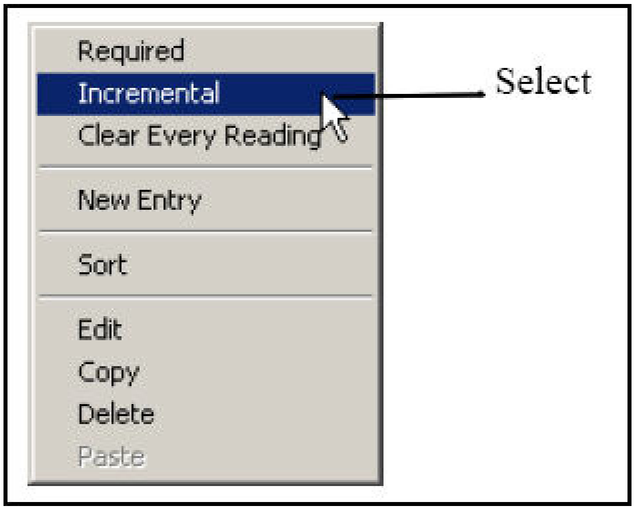


Figure 40. Making Fields Incremental

Selecting Clear Every Reading will toggle between two states. By default, the field will fill with the data which was input during the last reading. By selecting Clear Every Reading, you tell the instrument to clear the data from the field for each new reading, insuring that the person taking the reading must input new data each time. This is very useful for times when the data descriptor is expected to vary widely between readings.

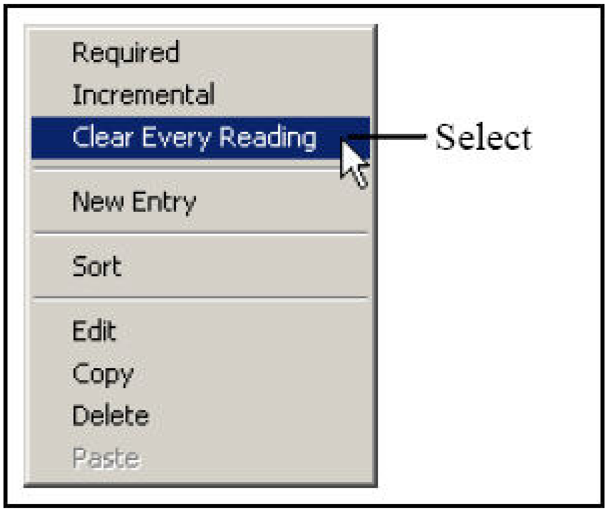
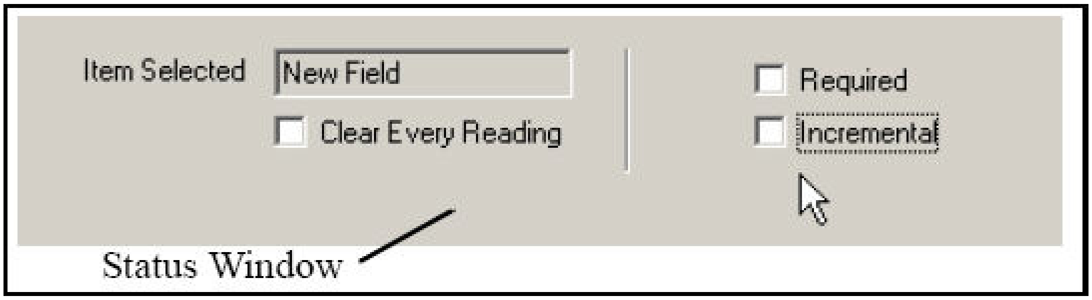


Figure 41. Clearing Data for New Readings

The state of each of these options can be seen in the Field Status Window at the bottom of the Construction Window. All options in effect for the field selected are checked.


Item Selected: New Field

☐ Clear Every Reading

☐ Required

☐ Incremental

Figure 42. Field Status Window - Default

This shows a field with no options in effect, the default configuration. This is a field that will present the previous reading's data for this field - which may be changed by the user - without incrementing it, but does not require the user to input any data if there is none already there from a previous reading.

Item Selected: New Field

☑ Clear Every Reading

☑ Required

☐ Incremental

Figure 43. Field Status Window - Options Enabled

This shows a field with both Required and Clear Every Reading options in effect. This presents a field that is cleared for each reading, and must be filled in by the user before a reading is taken.

Selecting Edit from the pop-up menu allows you to edit the name of the field in the Editing Window to the right of the Construction Window.

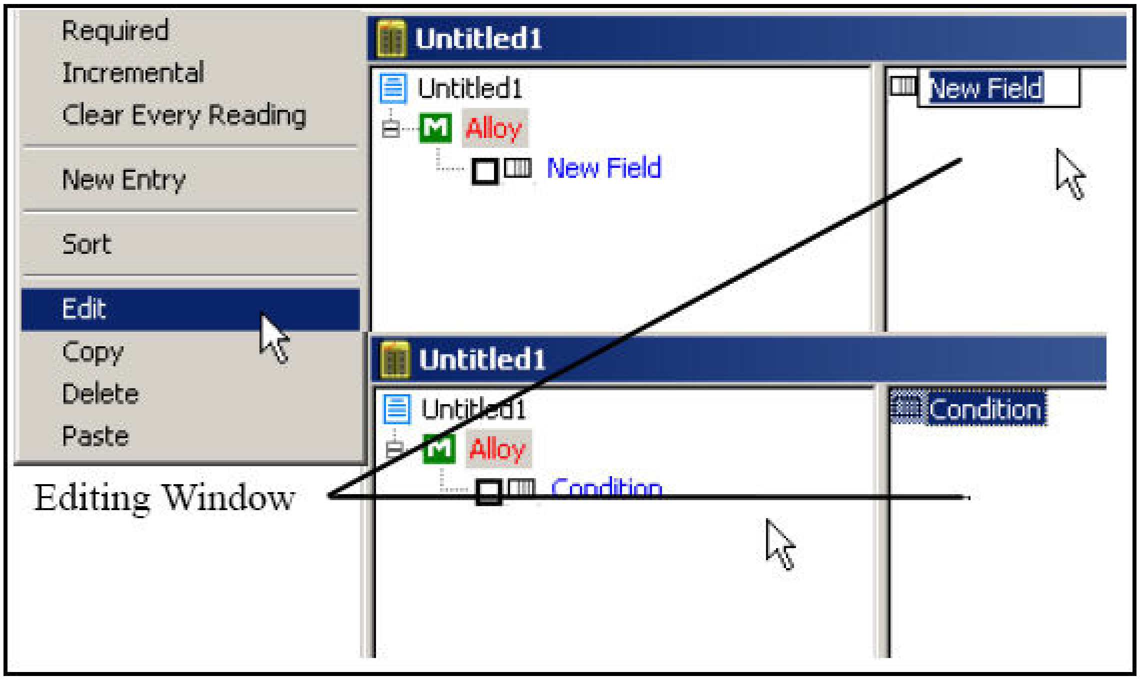
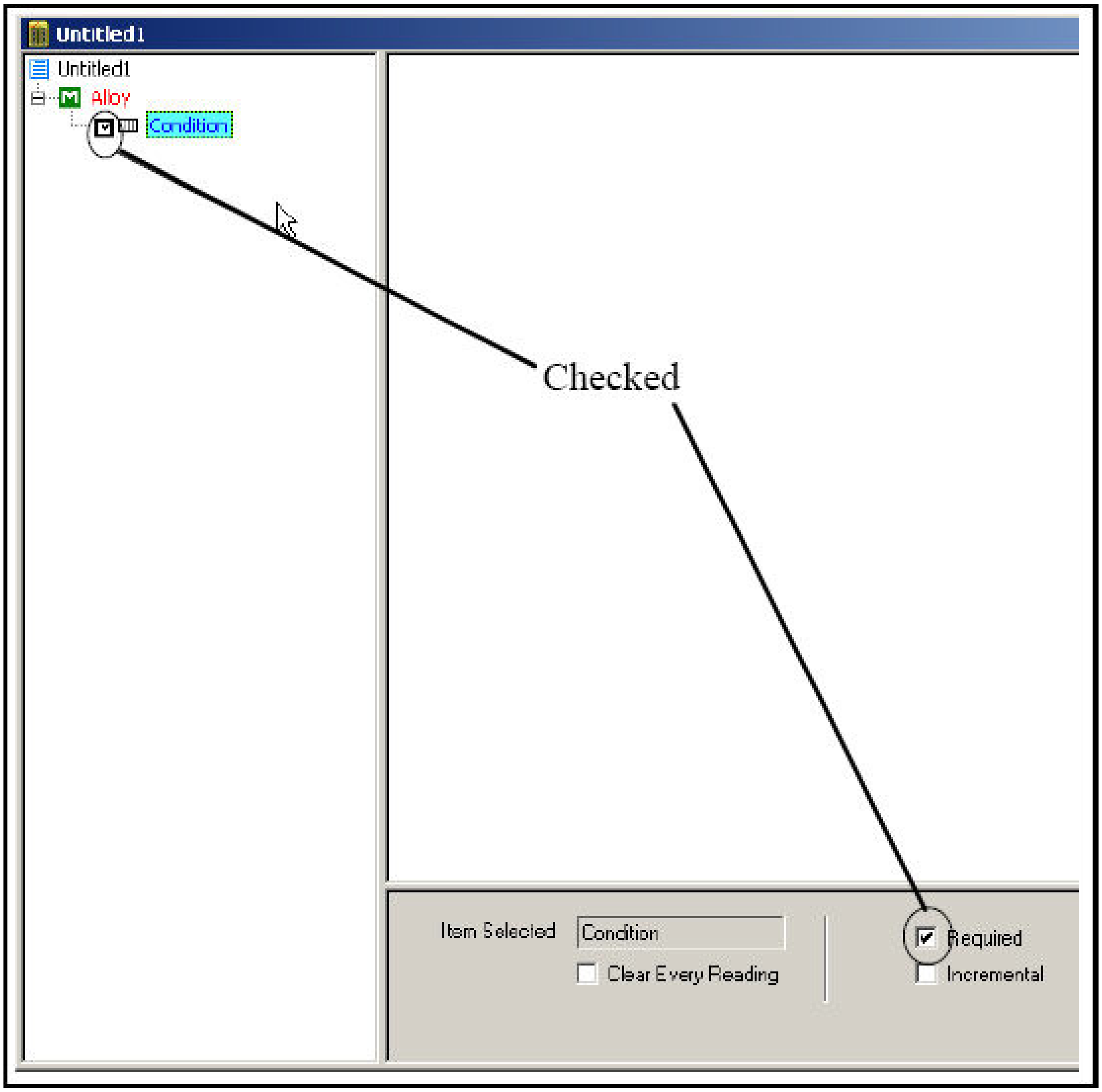


Figure 44. Editing the Field Name

Selecting the box to the left of the field toggles the Required option on or off.



Item Selected: Condition

☐ Clear Every Reading

☑ Required

☐ Incremental

Figure 45. Toggling the Required Option

Selecting Copy from the pop-up window allows you to copy the currently selected field.

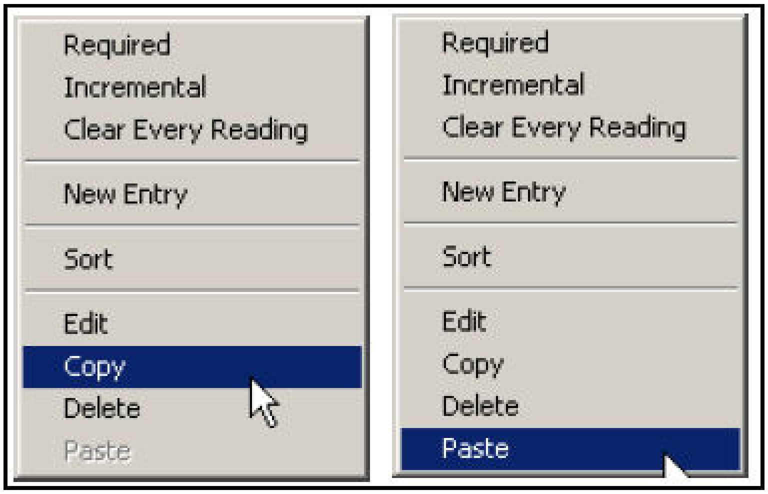


Figure 46. Copying the Current Field

Once you copy a field, the Paste option can be selected to paste the copied field into the Construction Window.

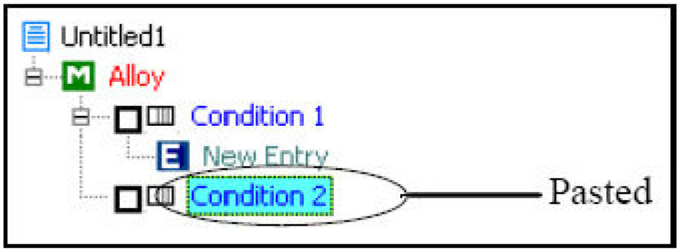


Figure 47. Pasting a Copied Field

Selecting the New Entry option from the pop-up menu allows you to define a choice for the user for this field.

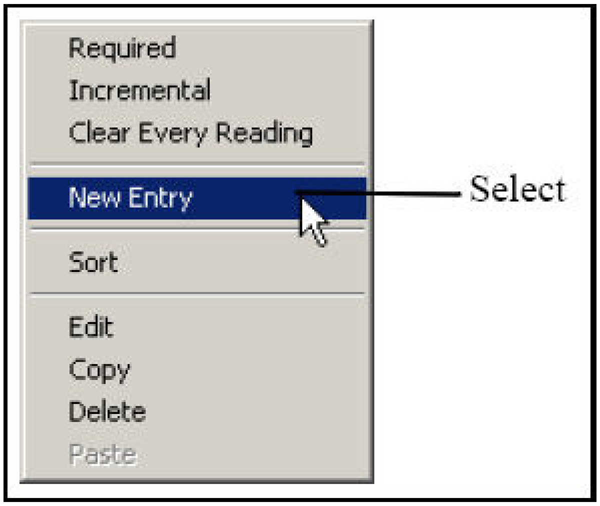


Figure 48. Creating a New Entry

This is a New Entry in the Construction Window.

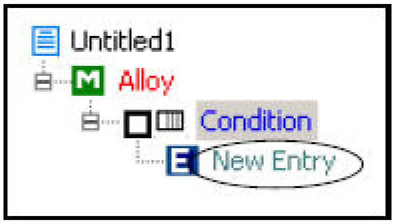


Figure 49. New Entry in the Construction Window

The “E” is for “Entry.” You can edit the entry once it is created, the same way as you edit the field name. Right click on the entry name, and choose Edit from the pop-up menu.

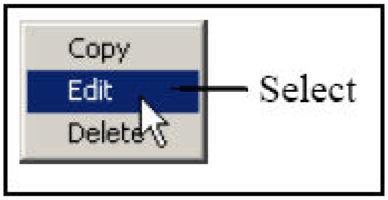


Figure 50. Editing the New Entry

You can sort your entries by name, alphanumerically, by right clicking on the field and selecting “Sort” from the pop-up menu.

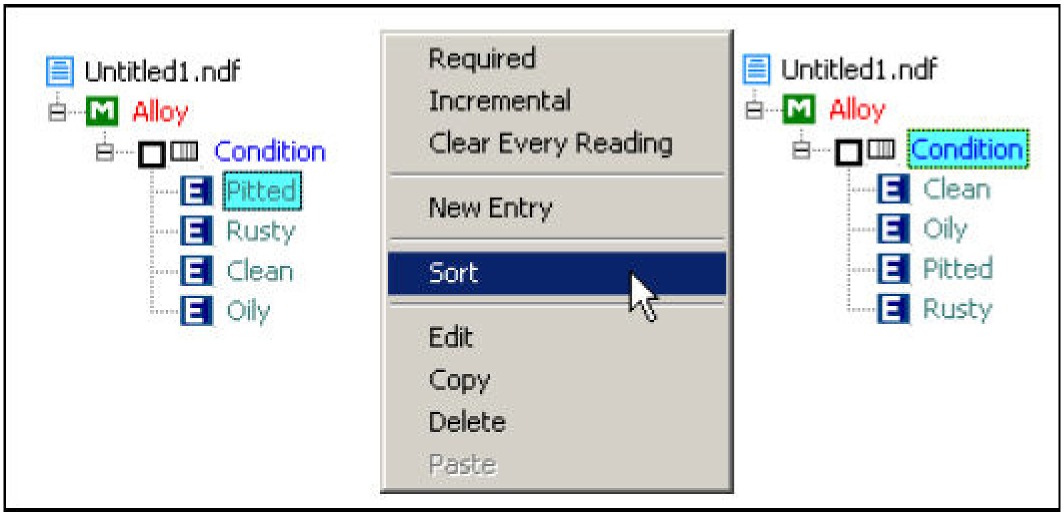


Figure 51. Sorting Entries

To delete a field or entry, just right click on the item you wish to delete, and select Delete From the pop-up menu.

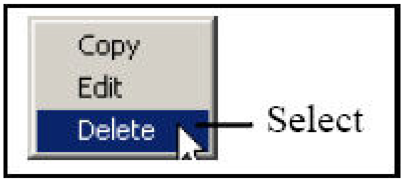
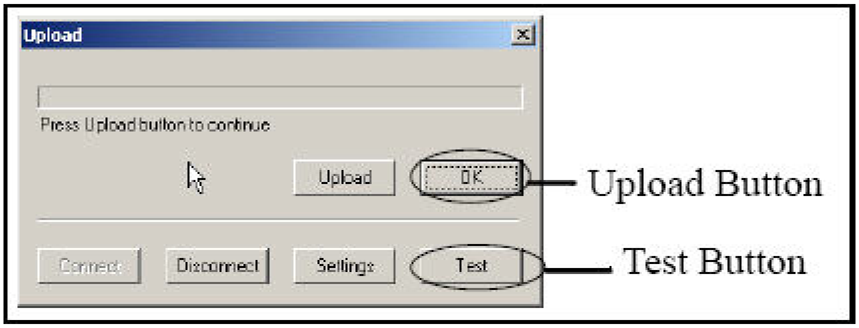


Figure 52. Deleting Fields and Entries

When you are finished creating your new NDF file, Upload it to your instrument using the Upload icon.

Figure 53. Uploading the NDF File

Make sure the instrument is connected to your computer by testing the con-nection first. Use the Test button on the Upload Window.

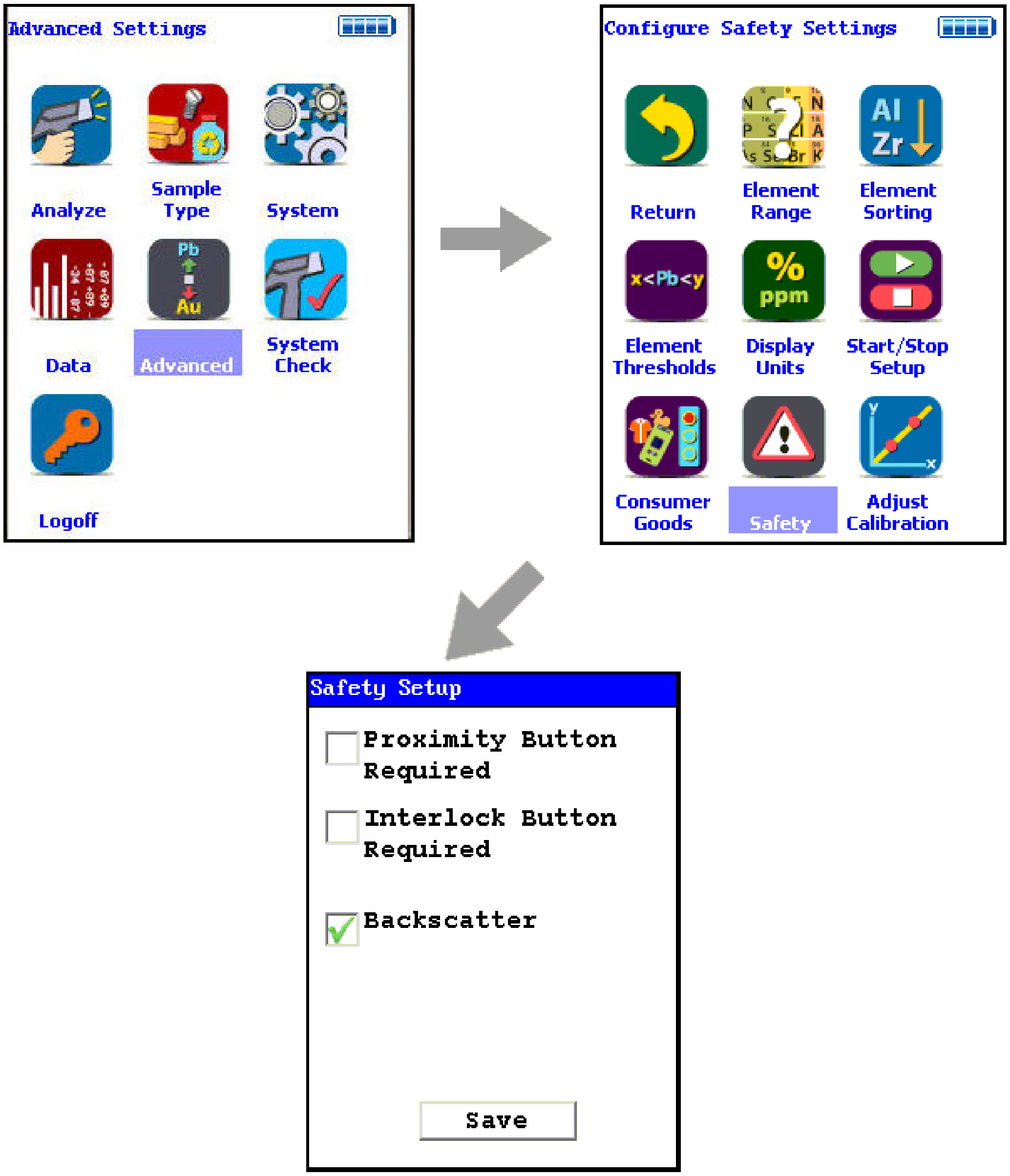


Press Upload button to continue

Figure 54. Testing the Upload Connection

Safety Settings

Access to the Safety Settings Screen is blocked unless the user logging in has explicitly been granted Safety access. The default login of 1234 does not have Safety access. See Passwords and User Privileges.



☐ Proximity Button Required

☐ Interlock Button Required

☑ Backscatter

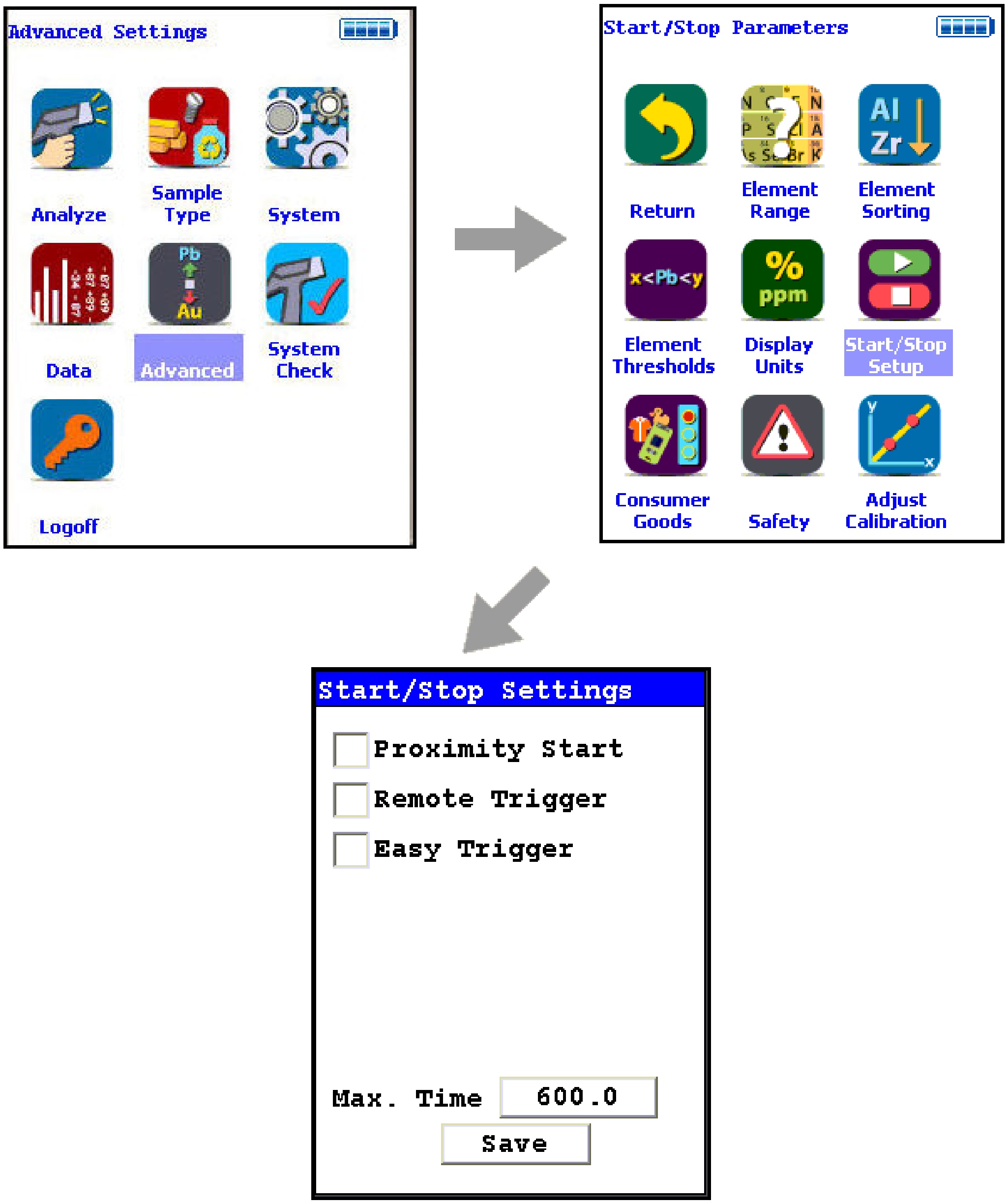
Figure 55. . Safety Settings Menu Path

The Safety Settings Screen enables you to change the Method of Operation for your analyzer. Each checkbox on the screen enables or disables the safety device named for purposes of the preconditions for operation. For example, checking the Proximity Button Required checkbox sets the engagement of the Proximity Sensor as a precondition for operation. Checking the Proximity Button Required checkbox and the Interlock Button Required checkbox sets the engagement of both the Proximity Button and the Interlock Button as preconditions for operation.

Safety settings always override start-stop settings. If your safety setting requires the use of the Proximity Button, you cannot set start-stop settings which ignore the Proximity Button. For example, the Easy Trigger start-stop setting must have the Backscatter safety setting enabled. While using Easy Trigger, you cannot disable Backscatter.

WARNING The backscatter sensor is enabled by default and acts as a recommended safety feature for most applications. Some sample types, however, cannot be analyzed when this feature is enabled. Samples that present very little mass to the analysis window, such as certain thin films, thin layers of plastic, and very thin wires, may not be of sufficient mass to allow the analysis to continue while backscatter is enabled. One should disable the backscatter feature only when necessary to analyze such low mass samples, and re-enable the feature when finished with these sample types. These samples also provide very little absorption of the primary x-ray beam so it is typically most appropriate to analyze these samples in a test stand when possible.

Start/Stop Setup



Start/Stop Settings

☐ Proximity Start

☐ Remote Trigger

☐ Easy Trigger

Max. Time 600.0

Save

Figure 56. The Start/Stop Settings Menu Path

The Start/Stop Setting Screen enables you to change the preconditions for operation at a lower level than the Safety level. See Safety Settings for more information. Start/Stop settings cannot contradict Safety settings. If your safety setting requires the use of the Proximity Button, you cannot set start-stop settings which ignore the Proximity Button. For example, the Easy Trigger start-stop setting must have the Backscatter safety setting enabled. While using Easy Trigger, you cannot disable Backscatter.

The Start/Stop parameter options are Proximity Start, Remote Trigger, and Easy Trigger. There is also a field to set the maximum time for sample analysis before the analysis stops.

Proximity Start

Select the Proximity Start checkbox to use the Proximity Start parameters. Using Proximity Start, once the reading has been started, release of the Proximity Button will immediately stop the analysis. You cannot use Proximity Start with Easy Trigger.

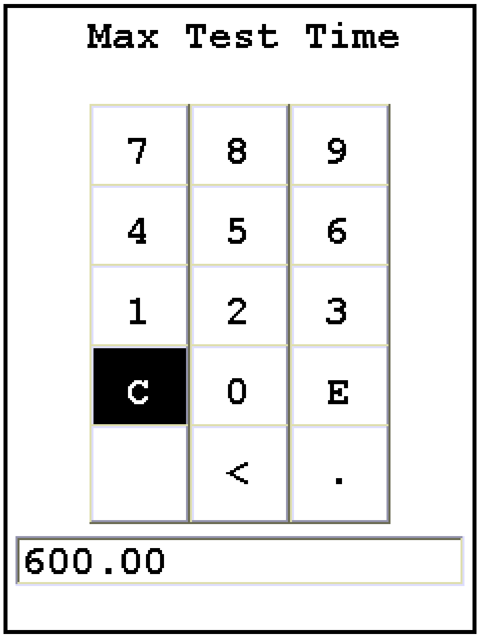
Remote Trigger

Select the Remote Trigger checkbox to use the Remote Trigger parameters. Remote Tigger is used with the Extend-a-Pole accessory to control the analysis. With the Extend-a-Pole's input cable connected to the analyzer's Remote Trigger port, you can initiate and stop analysis remotely from the Extend-a-Pole's handle trigger. You can use Remote Trigger with either Proximity Start or with Easy Trigger.

Easy Trigger

Select the Easy Trigger checkbox to use the Easy Trigger parameters. Easy Trigger uses a single press and release of the trigger to initiate analysis, and a second press to stop analysis. Selecting Easy Trigger will immediately disable Proximity Start.

Max Time Field



600.00

Figure 57. The Max Test Time Editor

Select the Max Time field to change the maximum analysis time parameter. Selecting the Max Time Field brings up a Virtual Numeric Keypad. To input the maximum number of seconds before automatic shutoff, select the C button to clear the current time, then from the Virtual Numeric Keypad, select each digit you want to input, then select the E button to enter. Of the non-numeric screen buttons, C = Clear All, E = Enter, and ">" will backspace over the current value. Selecting the E button will enter the current value as the Max Time, and return to the Start/Stop Settings Screen.

Save Button

Selecting the Save Button will save your current settings.

Methods of Operation

CAUTION After being powered on, your Niton Analyzer will perform an internal re-calibration before an analysis is initiated. It is recommended that you let your analyzer warm up for ten minutes after start up, before testing is begun.

There are six different methods of operation for taking a sample measurement, and your analyzer will be configured to use one of those methods for alloy samples, depending on the regulatory requirements of your locality. These methods are:

- Trigger-Only method. With the Trigger-Only method, you only need to place the measurement window flush with the sample to be analyzed and pull the trigger for sample analysis to be initiated.
- Trigger-and-Proximity-Sensor method. With the Trigger-and-Proximity-Sensor method, you must place the measurement window against the sample to be analyzed to engage the proximity sensor on the front of the analyzer, then pull the trigger for sample analysis to be initiated.
- Momentary-Trigger-Touch-and-Proximity-Sensor method. With the Momentary-Trigger-Touch-and-Proximity-Sensor method, you must place the measurement window against the surface to be analyzed to engage the proximity sensor on the front of the analyzer, then pull the trigger. The trigger may be released and the reading will continue until you release the proximity button, or other criteria (such as Max Time) are reached.
- Trigger-and-Interlock method. With the Trigger-and-Interlock method, you need to place the measurement window close to the sample to be analyzed, press and keep pressing the interlock button at the rear of the analyzer with your free hand, then pull the trigger for sample analysis to be initiated.
- Trigger-Interlock-and-Proximity-Sensor method. With the Trigger-Interlock-and-Proximity-Sensor method, you must place the measurement window against the sample to be analyzed to engage the proximity sensor on the front of the analyzer, press and keep pressing the interlock button at the rear of the analyzer with your free hand, then pull the trigger for sample analysis to be initiated.
- Easy Trigger method. With the Easy trigger method, you need only place the measurement window against the sample area and pull the trigger once to initiate a sample analysis. Your analyzer will continuously sample the backscatter, using a complex internal algorithm, to determine if the measurement window is against a sample or pointing to the empty air. If it finds that there is no sample directly against the measurement window, the analyzer will stop directing radiation through the window as soon as this determination is made.

The analyzer is constantly checking the backscatter characteristics to determine if a sample is against the measurement window, whether or not the Easy Trigger method is being used, and will shut off any radiation directed through the window if it determines that there is no sample present.

With any of these methods, analysis will stop if any one of the preconditions are violated. For example, with the Trigger-Interlock-and-Proximity-Sensor method, if the trigger or the Proximity Sensor or the Interlock is released, the reading will stop immediately, and the X-ray tube will shut down.

After your analyzer is calibrated, initiate a sample reading using the appropriate method. If you attempt to initiate a sample reading using a different method, the analyzer will inform you that one or more of the preconditions need to be met in order for sample analysis to begin.

Note The LED lights will blink whenever the x-ray tube is on.

WARNING The nose should not be touched during sample testing and calibration. If an ESD event occurs during measurement, the instrument may terminate the testing in progress and automatically reset to LogOn screen. Any test data collected prior to reset will be lost and the testing may have to be repeated.

WARNING The preconditions for operation must be continued for the duration of the reading. If the preconditions are violated, the x-ray tube will turn off, the calibration shutter will close, and the measurement will end. The LED lights will stop blinking when the measurement is ended. The flashing of the LED lights is not synchronized to minimize power consumption.

To end the test, simply release the trigger mechanism, or any other applicable preconditions.

Camera and Small Spot Video

The Camera feature is only usable with properly configured analyzers, and the Small Spot feature is only available on Small Spot analyzers.

If your analyzer is equipped with an internal video camera, you can turn that camera on and off, and turn the saving of images with the readings on and off through an interface. When the camera is on, the image will show in the Ready to Test screen, as in Figure 123. If the camera is off, saving of images will also be off. If the camera is on and the image saving function is also on, the images will automatically be saved with the reading. Saving images will curtail the maximum number of readings stored.

How to Use the Camera

When a Camera equipped analyzer is in the Ready to Test screen, the video feed appears live on the analyzer’s touch screen. This is the image that can be saved with the sample analysis. When you take a measurement, if you choose to do so, the bitmap image will be saved on the analyzer along with the analysis results. The interface is accessible through the System menu, as in Figure 120.

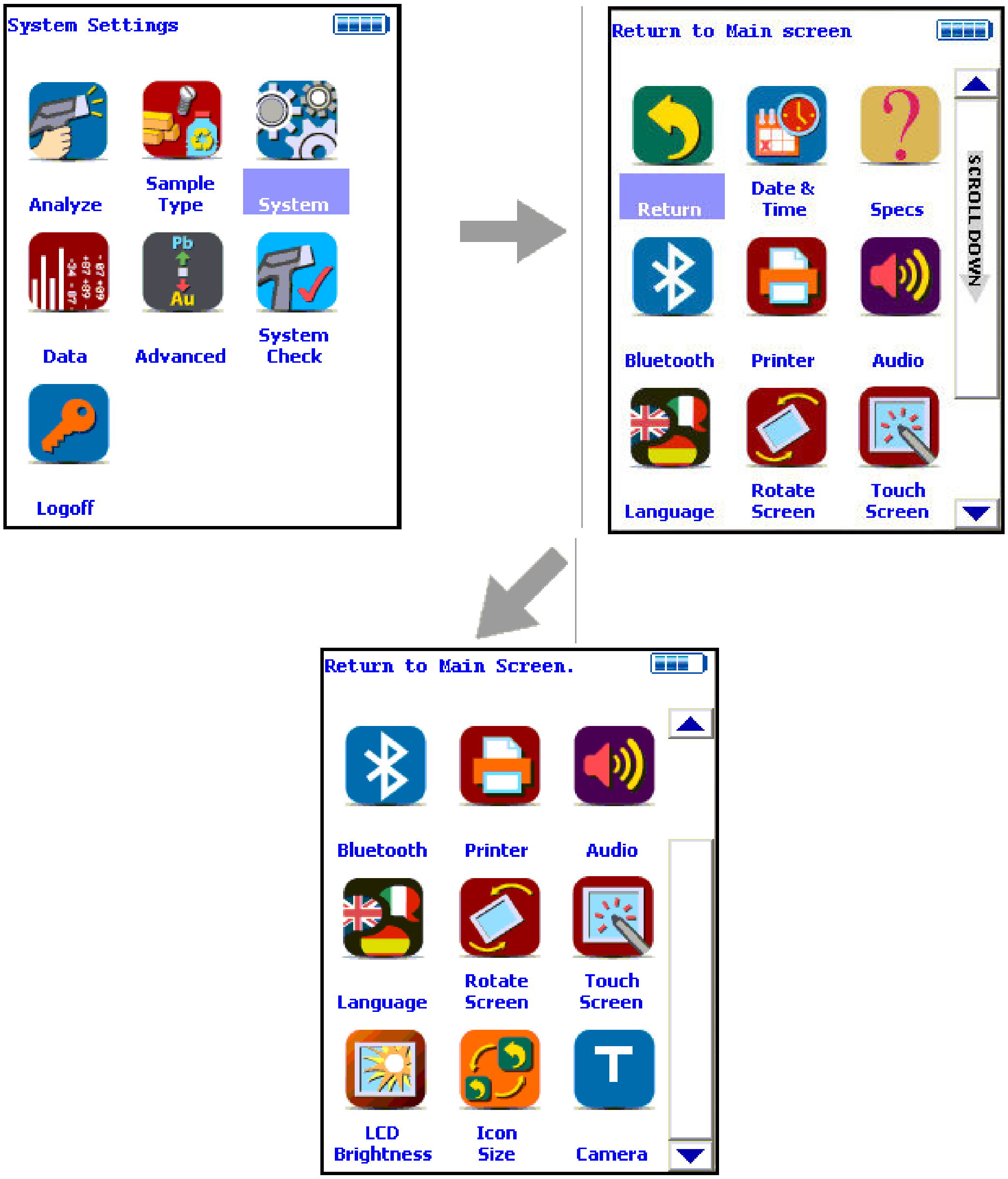


Figure 58. The Camera Menu Path

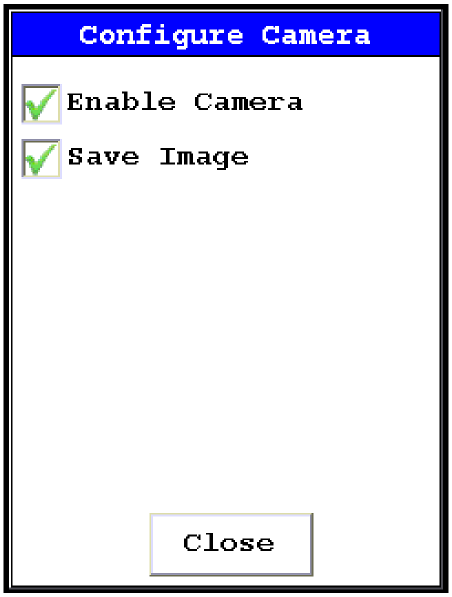


Figure 59. Setting Up the Camera View and Image Saving

Selecting the empty checkbox next to Enable Camera will turn the internal camera on, displaying the camera view in the Ready to Test screen. Selecting the checkbox again turns the camera off. Enable Camera is enabled by default.

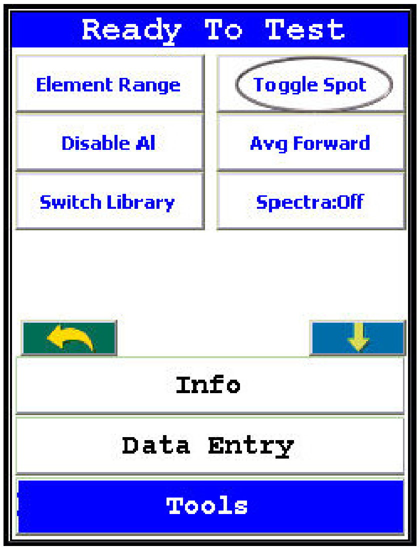
Selecting the empty checkbox next to Save Image will enable image saving with the analysis. Selecting the checkbox again will disable automatic saving of image data. Save Image is enabled by default.

Stored camera images from previous measurements can be viewed on the analyzer.

How to Use the Small Spot Technology

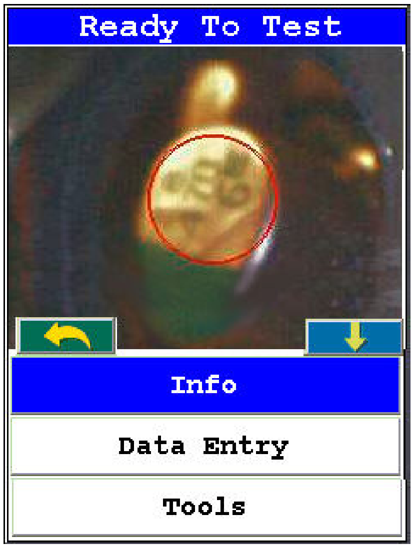
With a properly equipped Small Spot analyzer, you can restrict the analysis to a small spot within the camera view. You can toggle the Spot on and off from the Tools Menu as in Figure 122.

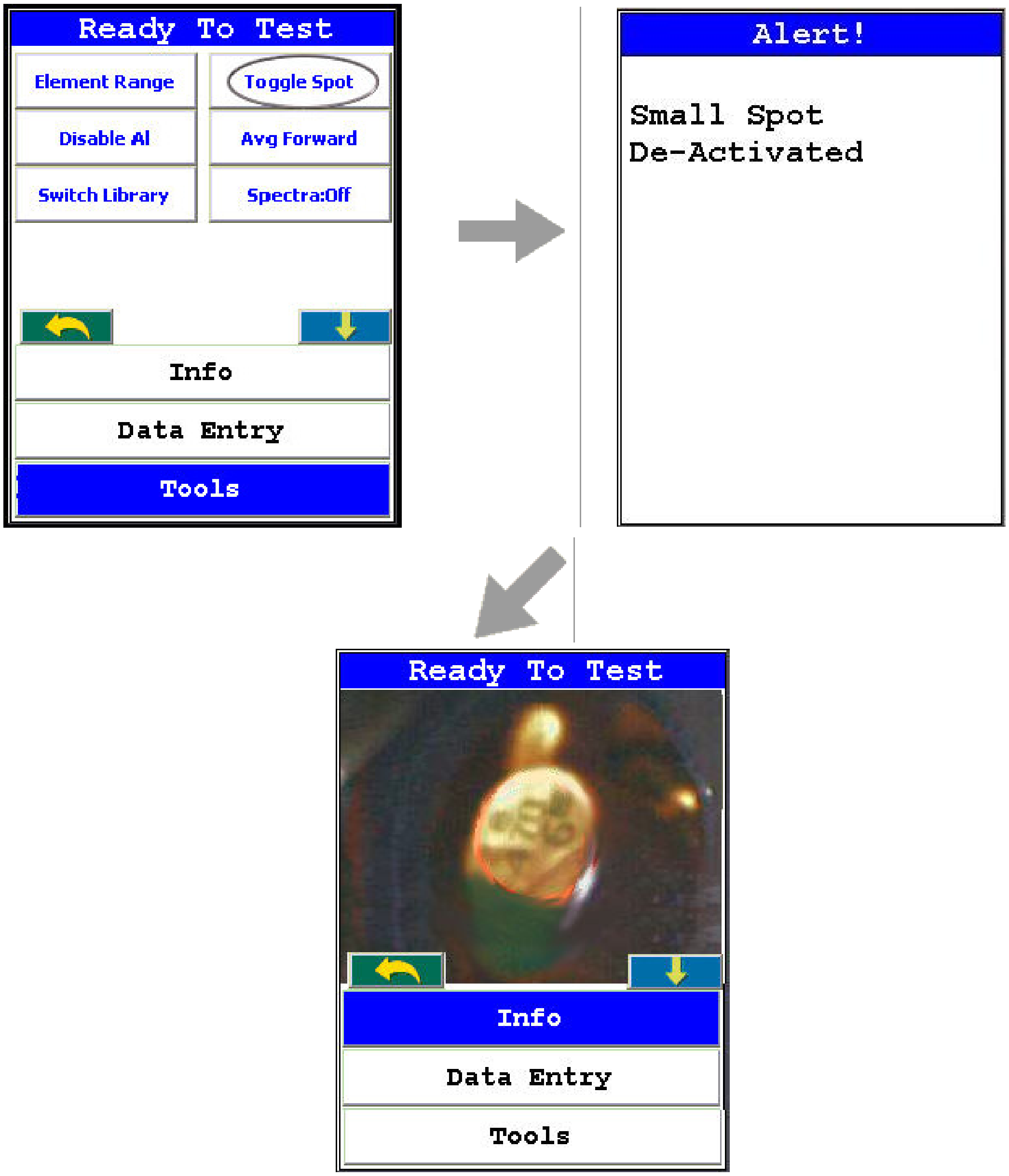
A red circle will appear on the display. The circle marks the area analyzed.




Alert!

Small Spot
Activated





Small Spot
De-Activated

Figure 60. Toggling the Small Spot from the TOOLS Menu

Using the Small Spot

The Small Spot can be used in several different modes.

Mining

- 3mm spot size allows analysis of veins and inclusions in mineral samples
- Initiate from Tools - Toggle Spot

He Purged

- Helium purge allows analysis of light elements (Mg, V and Ti)
- Small spot enables analysis of light elements in welds and inclusions
- Helium and Small Spot modes can be used simultaneously for Metals and Mining modes

Calibrating the Spot

If your instrument has small spot capability, you may calibrate the spot to optimize performance. Please follow the following procedure to calibrate the spot.

Requirements: CPU code 7.0A or higher, Small Spot QC Calibration Sample part #180-607.

1. Connect the instrument to NDTr. It is strongly recommended that this procedure be done in a test stand whenever possible. See Operating Your Analyzer Remotely for details.
2. Log on (Default "1234")
3. Enter a mode that has small spot capability. It is recommended that the you use either Metals or Electronics Alloy if possible. Then exit out to the main menu.
4. Select the Small Spot Calibration icon from the Advanced Menu

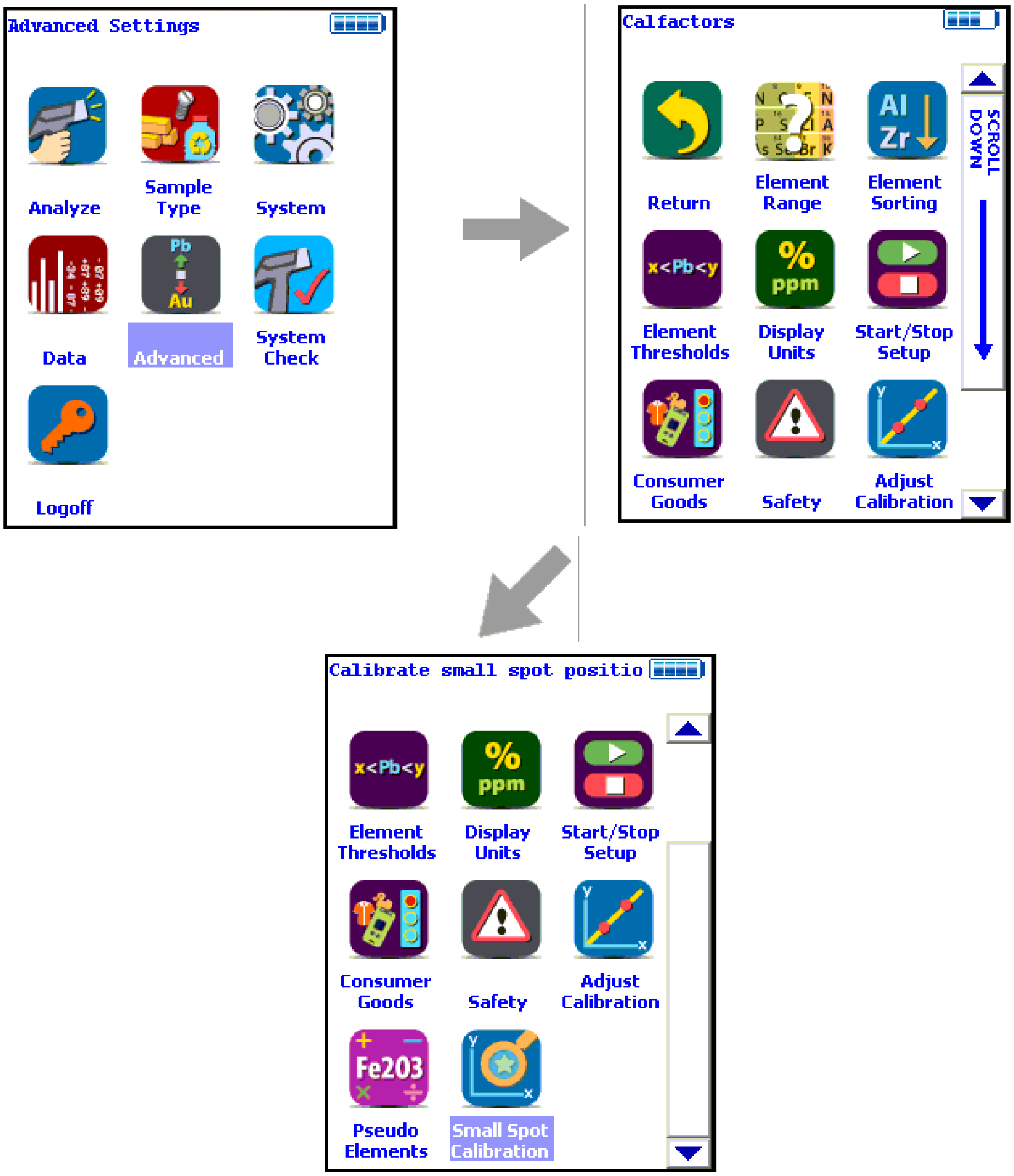
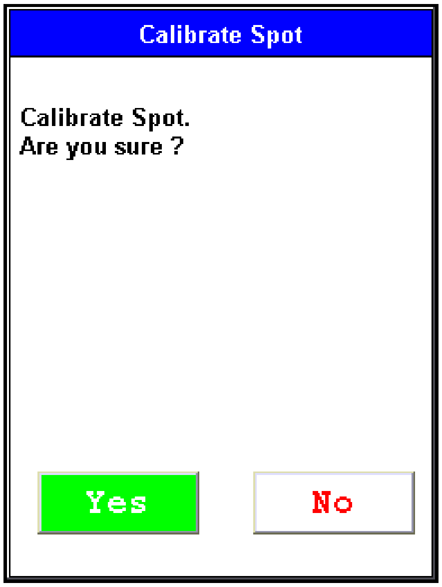


Figure 61. The Small Spot Menu Path

5. From the Calibrate Spot Screen, select the Yes Button.

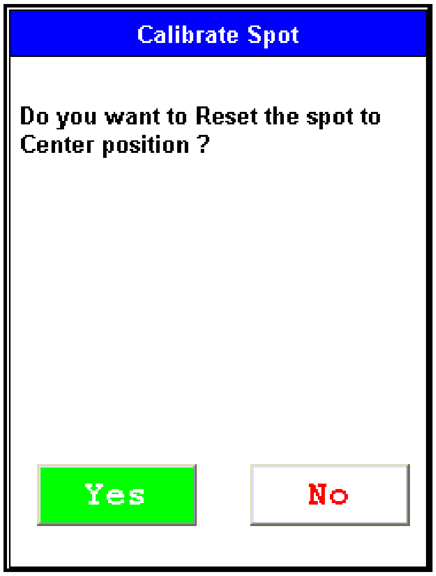


Calibrate Spot

Calibrate Spot.
Are you sure ?

Figure 62. Selecting the Yes Button

6. You will then receive a prompt asking if you want to reset the spot to the center position. It is recommended that you do this upon initial calibration. Saying "Yes" changes the position of the spot circle on the first calibration iteration to the calculated center. Saying "No" leaves this spot where it currently is for the first calibration iteration.



Calibrate Spot

Do you want to Reset the spot to
Center position ?

Figure 63. Centering the Spot

7. You will then see the small spot target on your instrument. Place the small spot calibration standard on the nose of analyzer so that the copper is in the center of the circle. If the copper is not properly centered for each of the five calibration shots, your calibration will not be valid.

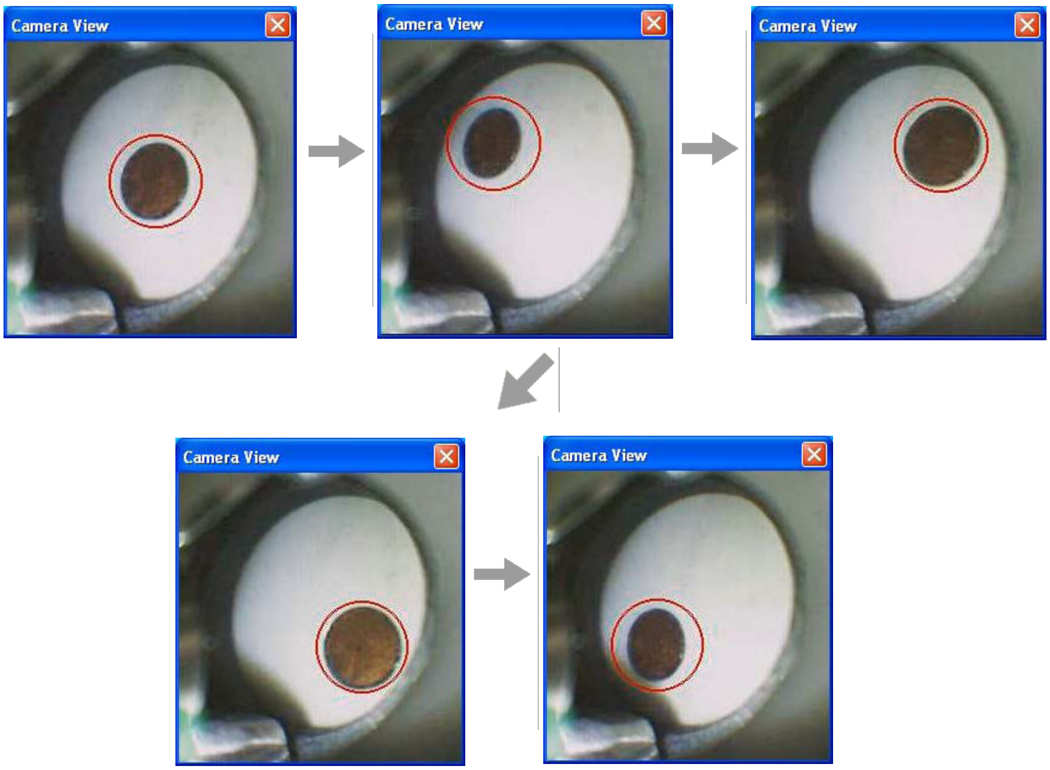
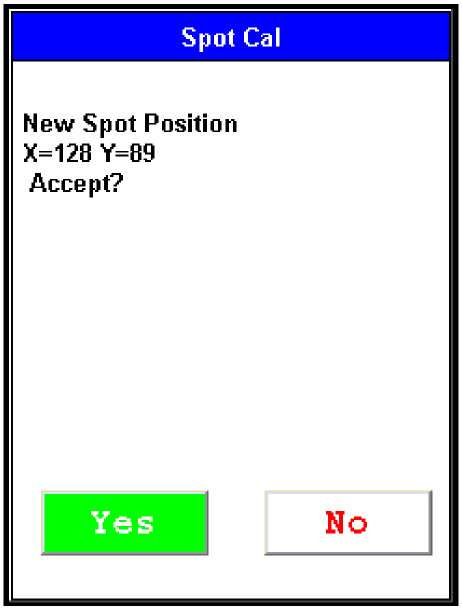


Figure 64. Centering the Spot

8. After centering the standard, take a reading for 30 seconds. After the reading time ends, you will notice that the spot has changed orientation. Please follow the previous step and re-center your standard. You will be asked to do this a total of five times.

9. You will then receive a prompt informing you of the detected X and Y position of the spot. If you made an error along the way, please select “No” and begin again. If you would like to accept the calibration, please select “Yes”.



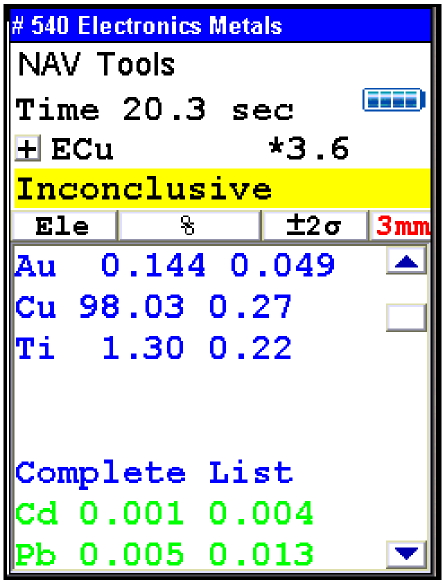
New Spot Position
X=128 Y=89
Accept?

Figure 65. Spot Coordinates

10. Position the Small Spot Reference Sample over the calibrated spot and take a measurement. You should see >95% Cu in Metals or Electronics Alloy mode.

Note The 180-607 standard is unique and should only be used for spot calibration.

Figure 66. Positioning the Small Spot Reference Sample



540 Electronics Metals

NAV Tools

Time 20.3 sec

ECu *3.6

Inconclusive

Ele	%	±2σ
Au	0.144	0.049
Cu	98.03	0.27
Ti	1.30	0.22

Complete List

Cd	0.001	0.004
Pb	0.005	0.013

Figure 67. Small Spot Check Analysis

Helium Purged Analysis

Attaching the Helium Supply to Your Analyzer

Make sure before you start that you are using a clean Prolyne Measurement Window intended for use with He-purged analysis, rather than the standard Polypropylene Measurement Window. Securely attach the regulator accompanying your analyzer to the helium tank. This custom regulator governs the helium flow to 70 cc/min flow rate. Confirm that the helium tank contains a sufficient supply for your testing purposes.

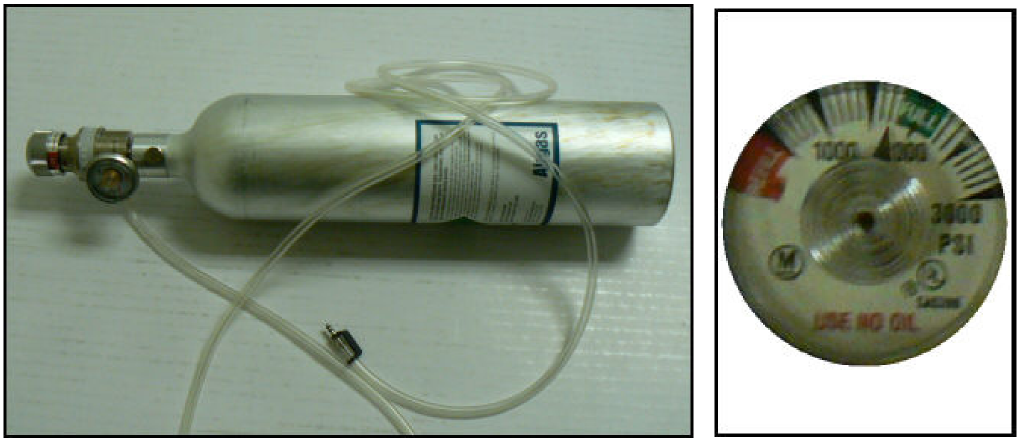


Figure 68. The Helium Tank Setup - Inset Showing Regulator Dial

1. Check the line for kinks, knots, or other obstructions before turning on helium flow.
2. Attach the ferrule, shown in Figure 2, to the Helium Input Port, shown in Figure 3.

Figure 69. The Helium Ferrule

Figure 70. The Helium Input Port

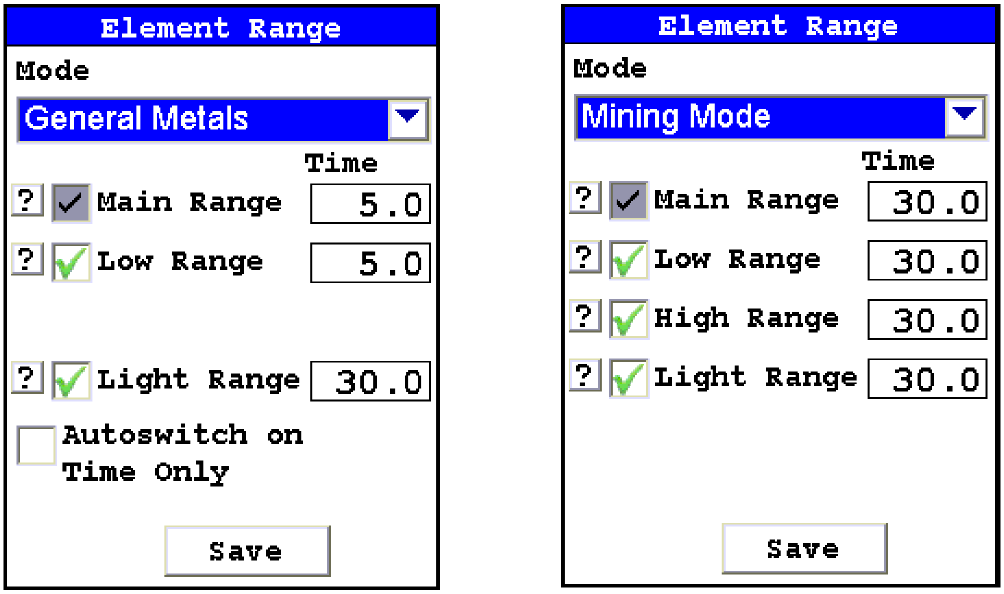
3. Snap the Locking Clip into the Locking Groove, securing the ferrule to the instrument, as in Figure 4.

Figure 71. Ferrule Attached and Locked

4. Turn on helium flow. Allow the helium to purge the instrument's front end for a minimum of five minutes before starting analysis. Helium purging can be done at the same time as instrument warm-up.

He-Purged Analysis

Helium purge is required for optimum detection of light elements such as Mg, Al, Si and P. For analyses using helium, we recommend using the default Range setup, as shown below in Figures 5 and 6. As with other energy dispersive XRF analyzers, the longer the testing time, the better the statistical precision of the reading. See Adjusting the Element Range for more on setting Ranges.



Element Range

Mode: General Metals

	Time
? ☑ Main Range	5.0
? ☑ Low Range	5.0
? ☑ Light Range	30.0

☐ Autoswitch on Time Only

Element Range

Mode: Mining Mode

	Time
? ☑ Main Range	30.0
? ☑ Low Range	30.0
? ☑ High Range	30.0
? ☑ Light Range	30.0

Figure 72. The Default Range Setups for Metals and Mining

The user can see the precision improving with analysis time by watching the third column on the analysis display – the 2-sigma error. This value decreases as reading time increases. Knowledge of this relationship is important, especially for light element work.

See Metals Analysis and/or Mining Analysis for more information on sample analysis. See also Avg Forward, Spectrum:On/Spectrum:Off, Enable LEC/Disable LEC, and Set Pass/Fail for more information on specific aspects and methods of analysis.

Table 1 shows the approximate limits of detection of the light elements in various matrices. If testing time is shortened, the limits of detection (which are directly based on statistical precision) will increase.

Table 1. Approximate LODs for Light Elements in Fe and Al Matrices

Element	Fe Matrix (concentration %)	Al Matrix (concentration %)
Zn	0.015	0.010
Cu	0.030	0.030
Ni	0.050	0.010
Cr	0.015	0.015
V	0.010	0.010
Ti	0.010	0.010

Table 1. Approximate LODs for Light Elements in Fe and Al Matrices

Al	0.031	-
Si	0.075	0.150
Mg	1.300	0.650

The above LODs were calculated with 60 sec per filter measurement times

Sample Requirements

1. Must be flat, sit directly on top of and cover the entire measurement window.
2. Surface must be clean and free of oxidation or other surface films.

See Metal Sample Prep for more information for metal sample preparation, and the Mining Analysis SOP for more information on preparing mining samples.

Alloy Grade Libraries

Using a library of alloy grade specifications, your alloy analyzer will attempt to identify an alloy sample being analyzed. It does this by comparing the chemistry analysis results against the minimum and maximum specifications in the library. The library is an .alb or .clb file, and is viewable, editable, and upload-able via the included NDT program. Your analyzer can store two alloy grade libraries in its memory, and automatically chooses one of the two for use while testing. See Managing Libraries for information on how to manage your Libraries.

Selecting the He Library

The 900 library will be auto loaded when the Light Range is selected. When the Light Range is de-selected, the standard 800 library will autoload. Selecting Switch Library (Main) from the Tools Menu will not work. It is disabled in favor of the autoloading. Alternately, you can select Toggle Helium from the Tools Menu. In non-GOLDD analyzers, this will select the Light Range and change to the 900 Library. Selecting it again will de-select the Light Range and change to the 800 Library.

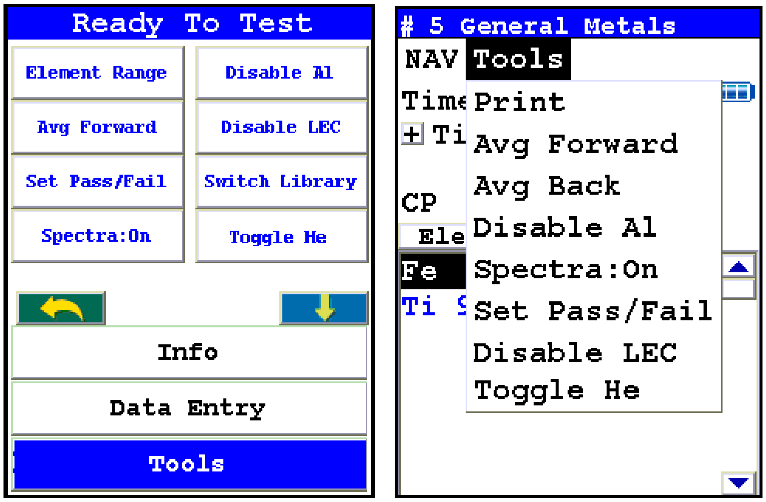
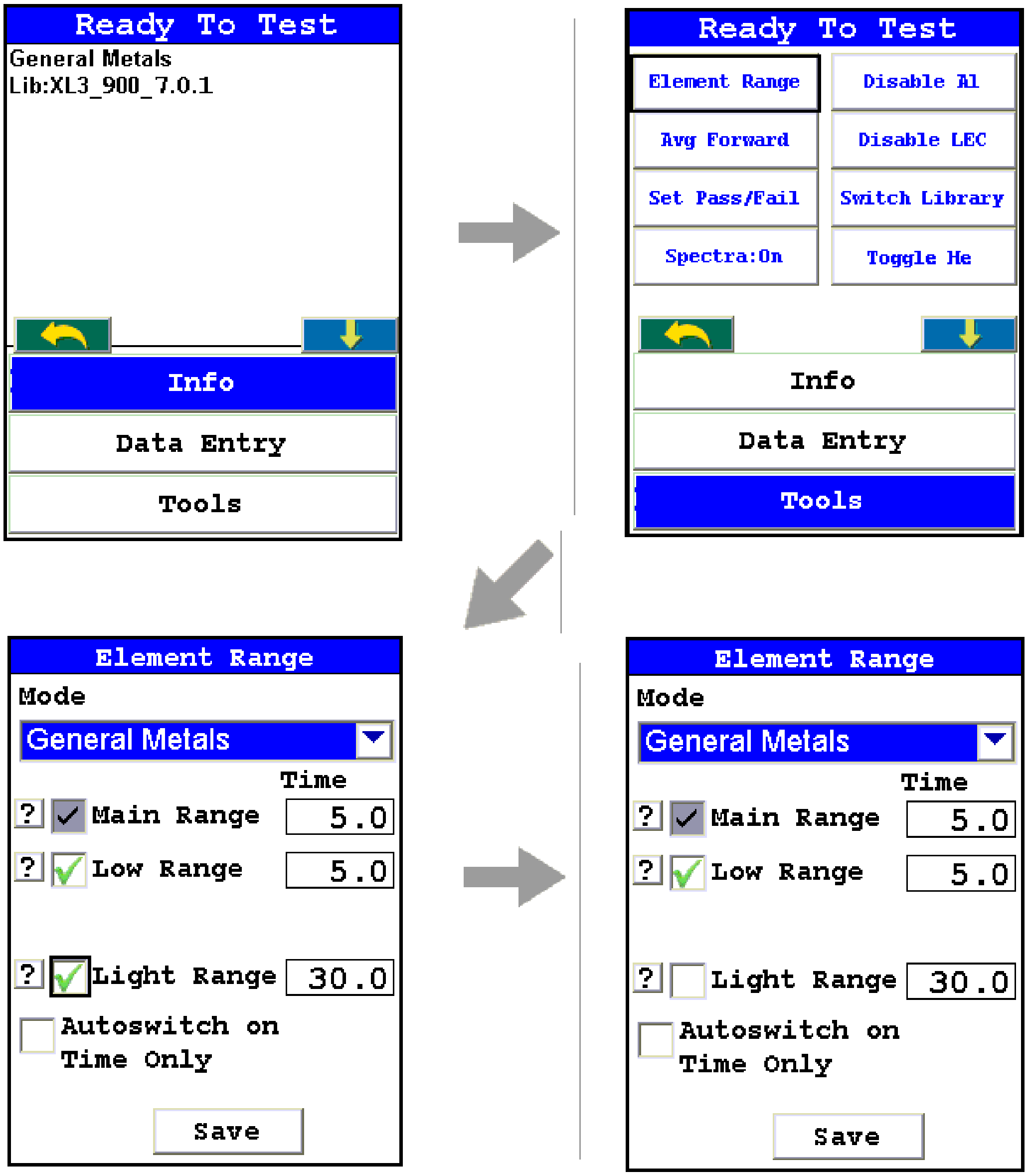


Figure 73. Selecting Toggle He from the Tools Menu and Alt Tools Menu

In GOLDD analyzers equipped with Helium Purge, selecting the Toggle Helium function from the Tools Menu will not deselect the Light Range. Instead it switches between Helium and Non-Helium applications - the Light Range remains enabled. You may also wish to use the analyzer without the Light Range activated. To do this, deselect the light range via the Tools Menu, as shown in Figure 1-7. To use the analyzer this way, be sure helium has been completely purged from the system.

Note When switching between Helium and Non-Helium applications on a GOLDD analyzer, allow the helium to dissipate out of system for at least ten minutes before beginning non-helium analysis. To expedite this process, you may remove the Measurement Window bracket for three or four minutes instead.



General Metals
Lib:XL3_900_7.0.1

Mode: General Metals

		Time
?	☑ Main Range	5.0
?	☑ Low Range	5.0
?	☑ Light Range	30.0

☐ Autoswitch on Time Only

Mode: General Metals

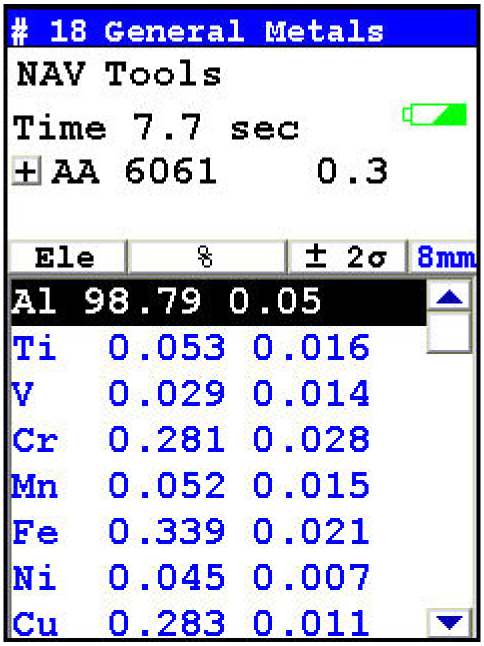
		Time
?	☑ Main Range	5.0
?	☑ Low Range	5.0
?	☐ Light Range	30.0

☐ Autoswitch on Time Only

Figure 74. Menu Path for De-Selecting the Light Range

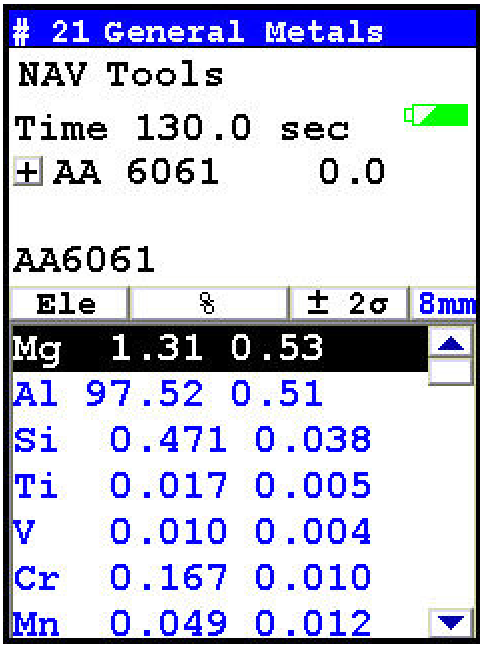
GOLDD and He-Purged Analysis Results

When using GOLDD technology analyzers and/or optional He-Purged analyzers, your results screen may look very different from the examples in this User’s guide, due to the general lack of elements below the LOD. Here are two analysis screens showing GOLDD analysis, for comparison.



```
# 18 General Metals
NAV Tools
Time 7.7 sec
+ AA 6061     0.3

 Ele      %      ± 2σ  8mm
Al 98.79 0.05
Ti   0.053 0.016
V    0.029 0.014
Cr   0.281 0.028
Mn   0.052 0.015
Fe   0.339 0.021
Ni   0.045 0.007
Cu   0.283 0.011
```

Figure 75. All Alloy Analysis of Al 6061, Showing Fast ID of Al Alloys (Light Range Disabled)



```
# 21 General Metals
NAV Tools
Time 130.0 sec
+ AA 6061     0.0

AA6061
 Ele      %      ± 2σ  8mm
Mg  1.31 0.53
Al 97.52 0.51
Si   0.471 0.038
Ti   0.017 0.005
V    0.010 0.004
Cr   0.167 0.010
Mn   0.049 0.012
```

Figure 76. All Alloy Analysis of Al 6061, Showing Long Analysis of Composition (Light Range Enabled)

Service

See “Contact Us” on page 1 to find your nearest Service facility.

Warranty

Warranty statement

Seller warrants that the Products will operate or perform substantially in conformance with Seller's published specifications and be free from defects in material and workmanship, when subjected to normal, proper and intended usage by properly trained personnel, for the period of time set forth in the product documentation, published specifications or package inserts. If a period of time is not specified in Seller’s product documentation, published specifications or package inserts, the warranty period shall be one (1) year (unless otherwise agreed upon at time of purchase) from the date of shipment to Buyer in the country of purchase. Any part replaced on an instrument, covered by the original factory warranty, will be warranted for the remainder of the instrument's factory warranty. Seller agrees during the Warranty Period, to repair or replace, at Seller's option, defective Products so as to cause the same to operate in substantial conformance with said published specifications; provided that Buyer shall (a) promptly notify Seller in writing upon the discovery of any defect, which notice shall include the product model and serial number (if applicable) and details of the warranty claim; and (b) after Seller’s review, Seller will provide Buyer with service data and/or a Return Material Authorization (“RMA”), which may include biohazard or other Radiation safety decontamination procedures and other product-specific handling instructions, then, if applicable, Buyer may return the defective Products to Seller with all costs prepaid by Buyer. Replacement parts may be new or refurbished, at the election of Seller, the warranty of these parts expire with the instrument warranty. All replaced parts shall become the property of Seller. Shipment to Buyer of repaired or replacement Products shall be made in accordance with the Delivery provisions of the Seller’s Terms and Conditions of Sale. Accessories and Consumables are expressly excluded from this warranty (see list A for details).

Notwithstanding the foregoing, Products supplied by Seller that are obtained by Seller from an original manufacturer or third party supplier are not warranted by Seller, but Seller agrees to assign to Buyer any warranty rights in such Product that Seller may have from the original manufacturer or third party supplier, to the extent such assignment is allowed by such original manufacturer or third party supplier.

In no event shall Seller have any obligation to make repairs, replacements or corrections required, in whole or in part, as the result of (i) normal wear and tear, (ii) accident, disaster or event of force majeure, (iii) misuse, fault or negligence of or by Buyer, (iv) use of the Products in a manner for which they were not designed, (v) causes external to the Products such as, but not limited to, power failure or electrical power surges, (vi) improper storage and handling of the Products, (vii) use of the Products in combination with equipment or software not supplied by Seller, (viii) Moderately heavy or excessive impact against any object, including but not limited to floors, walls, furniture, sample or other objects. A shock sensor is fitted inside of the instrumentation; warranty is void if this shock sensor is activated, (ix) Excessive water, moisture or condensing humidity that breaches the instrument seals, (X) Excessive or extreme ambient or direct temperature or (vi) Heavy vibrations directly to the instrument for extended periods of time. If Seller determines that Products for which Buyer has requested warranty services are not covered by the warranty hereunder, Buyer shall pay or reimburse Seller for all costs of investigating and responding to such request at Seller's then prevailing time and materials rates. If Seller provides repair services or replacement parts that are not covered by this warranty, Buyer shall pay Seller therefore at Seller's then prevailing time and materials rates.

ANY INSTALLATION, MAINTENANCE, REPAIR, SERVICE, RELOCATION OR ALTERATION TO OR OF, OR OTHER TAMPERING WITH, THE PRODUCTS PERFORMED BY ANY PERSON OR ENTITY OTHER THAN SELLER WITHOUT SELLER'S PRIOR WRITTEN APPROVAL, OR ANY USE OF REPLACEMENT PARTS NOT SUPPLIED BY SELLER, SHALL IMMEDIATELY VOID AND CANCEL ALL WARRANTIES WITH RESPECT TO THE AFFECTED PRODUCTS.

THE OBLIGATIONS CREATED BY THIS WARRANTY STATEMENT TO REPAIR OR REPLACE A DEFECTIVE PRODUCT SHALL BE THE SOLE REMEDY OF BUYER IN THE EVENT OF A DEFECTIVE PRODUCT. EXCEPT AS EXPRESSLY PROVIDED IN THIS WARRANTY STATEMENT, SELLER DISCLAIMS ALL OTHER WARRANTIES, WHETHER EXPRESS OR IMPLIED, ORAL OR WRITTEN, WITH RESPECT TO THE PRODUCTS AND INCLUDING WITHOUT LIMITATION ALL IMPLIED WARRANTIES OF MERCHANTABILITY OR FITNESS FOR ANY PARTICULAR PURPOSE. SELLER DOES NOT WARRANT THAT THE PRODUCTS ARE ERROR-FREE OR WILL ACCOMPLISH ANY PARTICULAR RESULT.

Accessories, Spares and Consumables - exclusions

(List A)

Specific warranties of some common accessories:

- Battery Charger and batteries - 12 months
- Instrument accessories - 12 months
- Consumable - no warranty

- Soil Grinder - no warranty
- Single-stage or two stage helium tank regulator - 12 months
- Test stands, extend-a-poles and docking stations – 12 months
- Parts or spares sold, installed or supplied outside of the product warranty period and not listed above – 12 months

Attachment 3.4
Corrective Action Report Template

Corrective Action Report/ Corrective Action Plan

Project ID	Project Name	Document ID

Preparer's Signature/Submit Date	Submitted to:

Description of the requirement or specification	
Reason for the Corrective Action	
Location, affected sample, affected equipment, etc. requiring corrective action	
Suggested Corrective Action	(Continue on Back)
Corrective Action Plan	(Continue on Back) ☐ Approval signature/date:____________________ Approval of corrective actions required by EPA? ☐ Yes ☐ No ☐ EPA approval name/date:____________________ ☐ Corrective actions completed name/date:____________________
Preventative Action Plan	(Continue on Back) ☐ Preventative actions completed name/date:____________________

Corrective Action Report/
Corrective Action Plan

Suggested Corrective Action (Continued)	
Corrective Action Plan (Continued)	
Preventative Action Plan (Continued)	

Attachment 3.5

Product Documentation and User Guide – Butte Reclamation Evaluation System

The City and County of
Butte-Silver Bow Montana

Product Documentation and User Guide

Butte Reclamation Evaluation System (BRES)

Produced By

Butte-Silver Bow
County

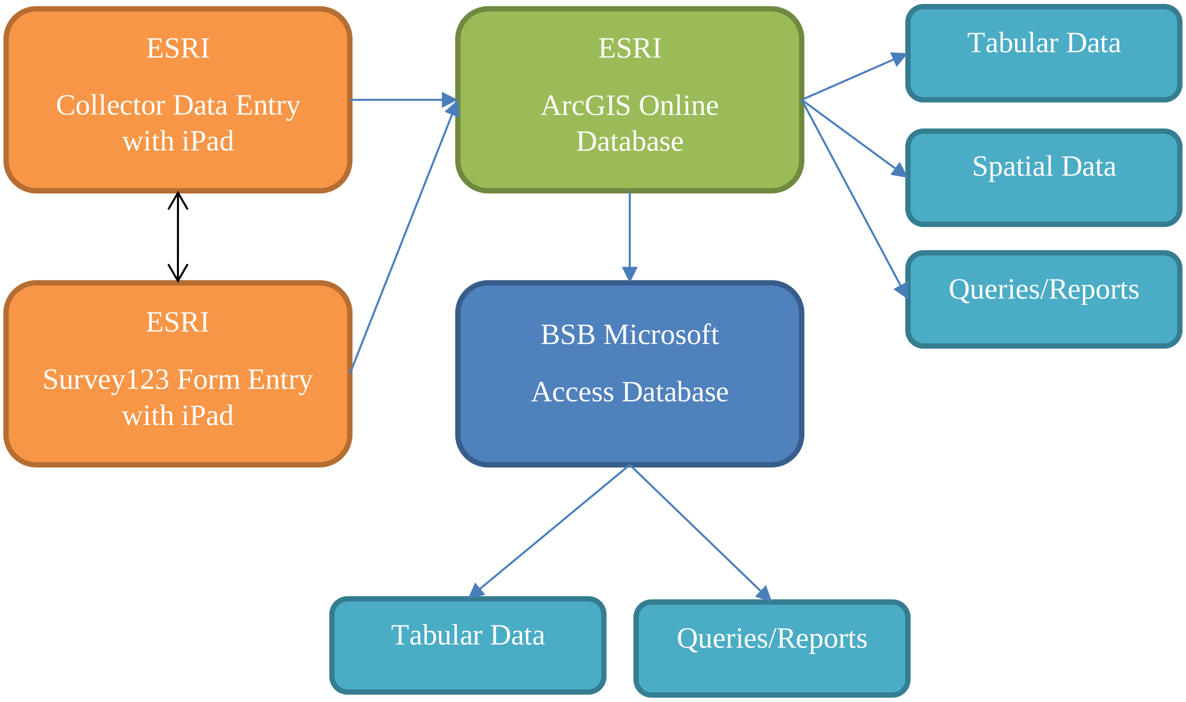
http://co.silverbow.mt.us/

Table of Contents

Contents

Product Overview

The Butte Reclamation and Evaluation System (BRES) system is an integrated data collection, management and storage system for BRES data. The system utilizes of tablets running ESRI's Survey123 and Collector applications for data collection which is seamlessly stored in BSB's ArcGIS Online database, and a Microsoft Access database front end application for querying and reporting purposes. By leveraging ESRI's ArcGIS online storage, data can be accessed using the ArcGIS Online maps (https://www.arcgis.com/index.html), Survey123's web portal (https://survey123.arcgis.com/) or from a variety of applications by utilizing ESRI's REST API. BSB's Microsoft Access Database utilizes the REST API to sync data from ArcGIS online to a local database. The flowchart below gives an overview of how the system works.



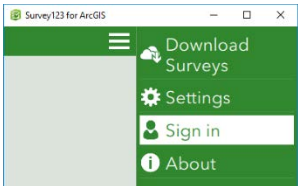
System Requirements

The BRES system requires use of ArcGIS Online, ESRI's Survey123 and ESRI Collector software, this software is included with the counties annual ESRI maintenance. Survey123 and Collector applications can run on IOS or Android devices, the county currently utilizes iPad's for field data collection. Microsoft Access is used as a front-end application for querying and reporting, this software is included with the counties current Microsoft software package. It is also important to note that it's preferable to have a tablet with cellular service for real time updating and collection, BSB utilizes Verizon in their IPad's for this currently.

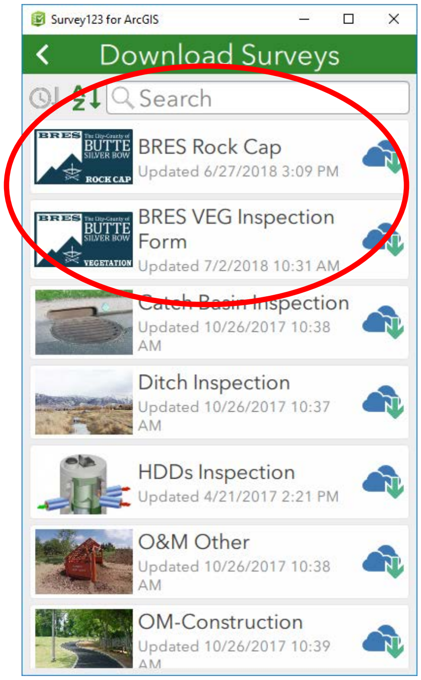
Installation Instructions

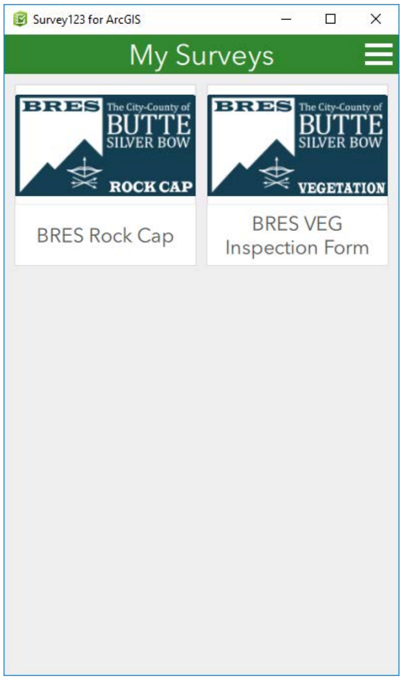
Suvey123 Installation

1. Visit the appropriate app store on your device
    - Google play for Android
    - App store for IOS
2. Search for Survey123 for ArcGIS, click install and launch the app.
    a. In the upper right corner click the three horizontal lines and choose Sign in
        - Sign in credentials are provided by BSB's GIS department through ArcGIS Online



    b. Once signed in, click the three horizontal lines again and choose Download Surveys
    c. Click the Download button to install the BRES Rock Cap and BRES VEG Inspection forms.



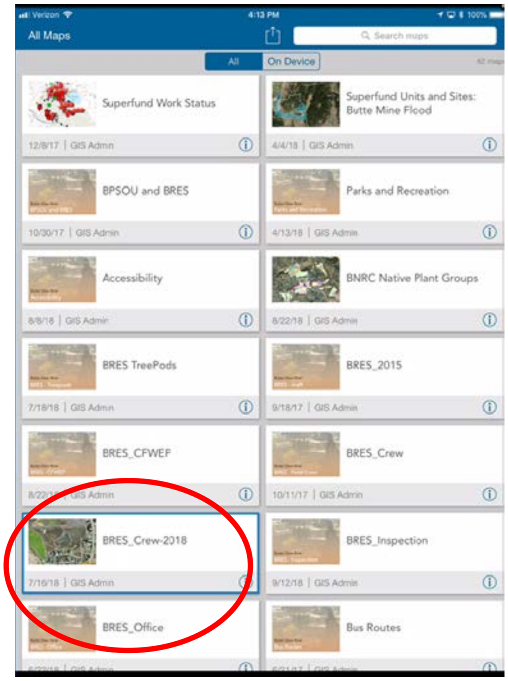


    d. The surveys are now ready to use on the device.

ArcGIS Collector Installation

1. Visit the appropriate app store on your device
    - Google play for Android
    - App store for IOS
2. Search for Collector for ArcGIS, click install and launch the App

3. Choose to sign into ArcGIS Online and enter the sign in credentials provided by BSB GIS
4. Open the appropriate map to collect Data



Microsoft Access Database Installation

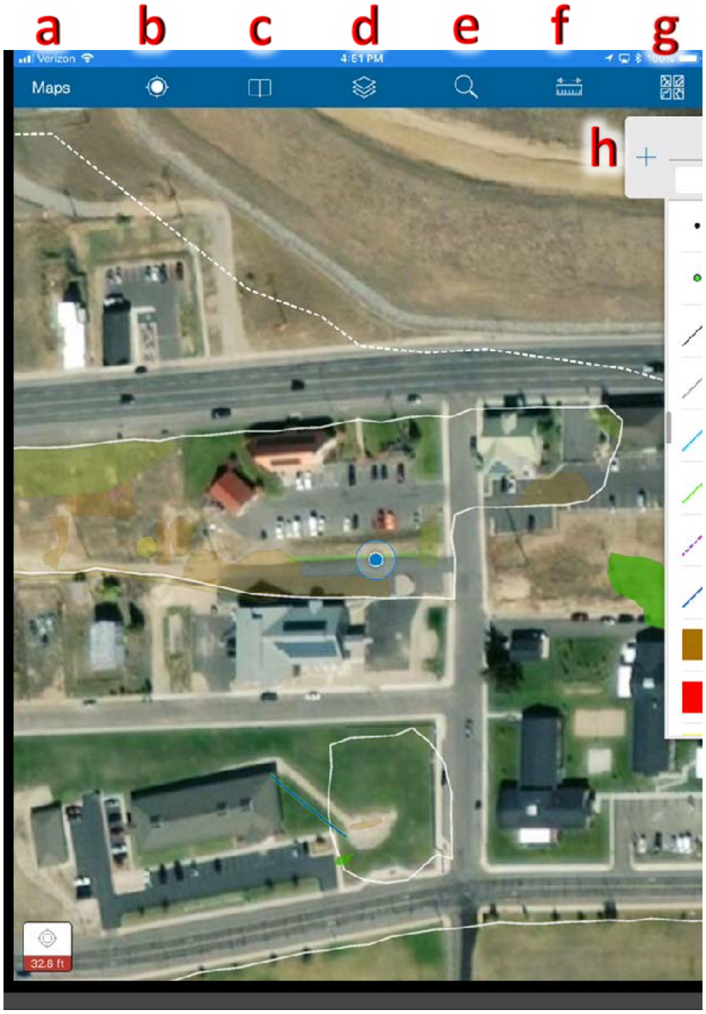
1. The Microsoft Access database requires that Microsoft Access 2013 or greater is installed. The custom database is installed by copying the file to a network or local file directory. Data is automatically synced when the database is open.

Operational Instructions

ESRI Collector Application

Application Overview

1. On the iPad launch the collector application, sign in and open the BRES mapping application. See installation instructions for installing ESRI Collector for information on installing the application.
2. Once opened the application zooms to your current GPS location as indicated by blue dot on the screen as shown below.



3. Below is a explination of the collector app’s icons. Please see coorisponding letter from image above for information on it’s use
   a. Tap the Maps icon in collector to select a different map to use for BRES data collection
   b. The location icon turns on or off location services (GPS), when the icon is filled it is using the current GPS location from your device. If it’s hollow no location services are being utilized.

c. This is the bookmarks icon which allows the user to bookmark frequent places or map extents.
d. Tap the layers icon to view a list of layers and optionally turn them on and off.
e. The search icon allows you to search for a location and optionally bookmark for later use.
f. The measure icon allows the user to mesaure distance or areas by tapping on the map or using your current GPS location.
g. The basemap icon allows users to choose between several different basemaps, for example aerial or topographic.
h. Tap the plus (+) symbol to open up the data collection menu.

Collecting Feaures

Tap the plus (+) arrow on the upper right-hand side of the screen to open the data collection menu.

Collecting Point Features

1. To collect a point feature, tap the feature type (Manhole in this example)
2. Collector begins collecting a point at your current GPS location. Fill out attribute information in the right-hand menu and hit Submit to create a feature at the current GPS location
3. To digitize a point feature, click on the screen at the desired location, fill out attribute information and tap Submit

Collecting Line Features

1. To collect a line feature, tap the feature type (Pipe_Cluvert) in this example.

2. To collect using current GPS location tap the Start Streaming button at the top of the screen and start walking the route.
3. Attribute information can be entered before starting streaming or streaming can be paused at any time for data entry.
4. To finish data collection, click the Submit button in the top right corner of the screen. Alternatively tap the Cancel button to discard changes

Identifying Features Launching Survey123 application

1. To identify a feature, make sure the layer is turned on in the Layers menu, zoom to a location and click a feature to display the pop-up menu.

Vegetative_improvement: Lots of bar...

BRES_sites: 115 Butte New England

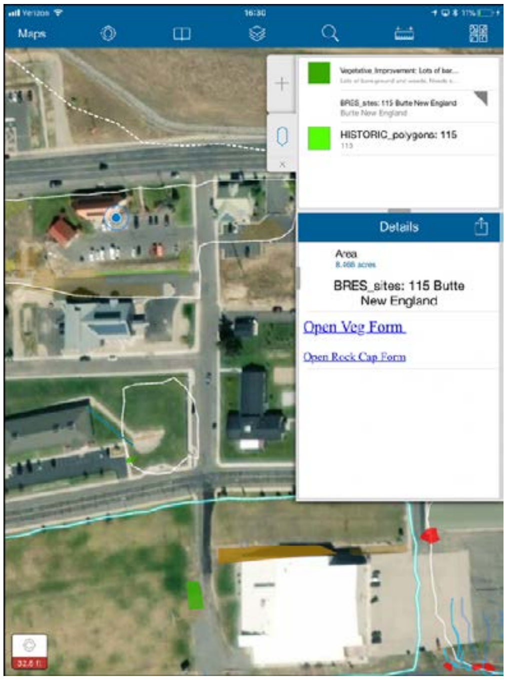
HISTORIC_polygons: 115

Area

Vegetative_Improvement: Lots of bare ground and weeds. Needs some vegetation improvement.

Date
July 3, 2018

Comments
Lots of bare ground and weeds. Needs some vegetation improvement.



Vegetative_Improvement: Lots of bar...

BRES_sites: 115 Butte New England
Butte New England

HISTORIC_polygons: 115

Area

BRES_sites: 115 Butte New England

Open Veg Form

Open Rock Cap Form

2. The identify menu appears on the upper left portion of the screen, as you can see the top menu shows how many layers have been identified (3 in this example). To switch between layers, tap the layer name.
3. When identifying a BRES polygon feature you will see options to launch the Veg inspection and Rock Cap inspection forms, clicking the appropriate hyperlink launches the appropriate form and links the identified BRES polygon information. For more information on using these forms see ESRI Survey123 Application in this manual.

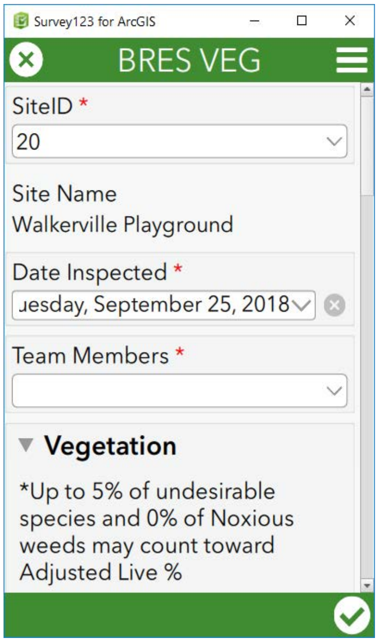
ESRI Survey123 Application

Vegetation Inspection Form

1. Vegetation Inspection Form can be launched in the following ways:
    a. The preferred way is to launch the program directly from ESRI collector application using the hyperlink by identifying a BRES polygon. This method automatically fills in the location information (SiteID and Site Name) on the Vegetation inspection form.
    b. To launch the application directly tap the Survey123 application on your device.

c. Sign in with credentials provided by BSB GIS department

d. Tap BRES VEG Inspection Form, then tap Collect to start collecting data.



SiteID *
20

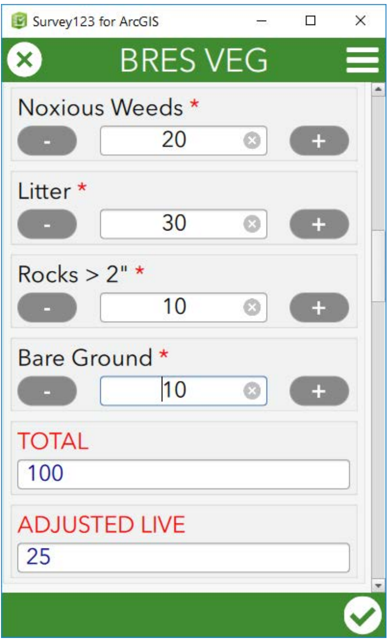
Site Name
Walkerville Playground

Date Inspected *
uesday, September 25, 2018

Team Members *

▼ Vegetation

*Up to 5% of undesirable species and 0% of Noxious weeds may count toward Adjusted Live %



Noxious Weeds * 20

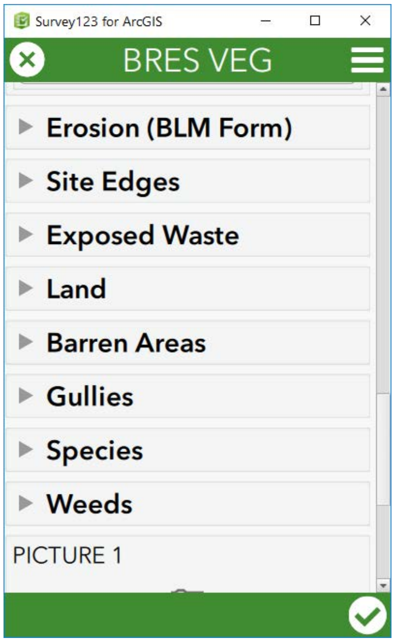
Litter * 30

Rocks > 2" * 10

Bare Ground * 10

TOTAL
100

ADJUSTED LIVE
25



▶ Erosion (BLM Form)
▶ Site Edges
▶ Exposed Waste
▶ Land
▶ Barren Areas
▶ Gullies
▶ Species
▶ Weeds
PICTURE 1

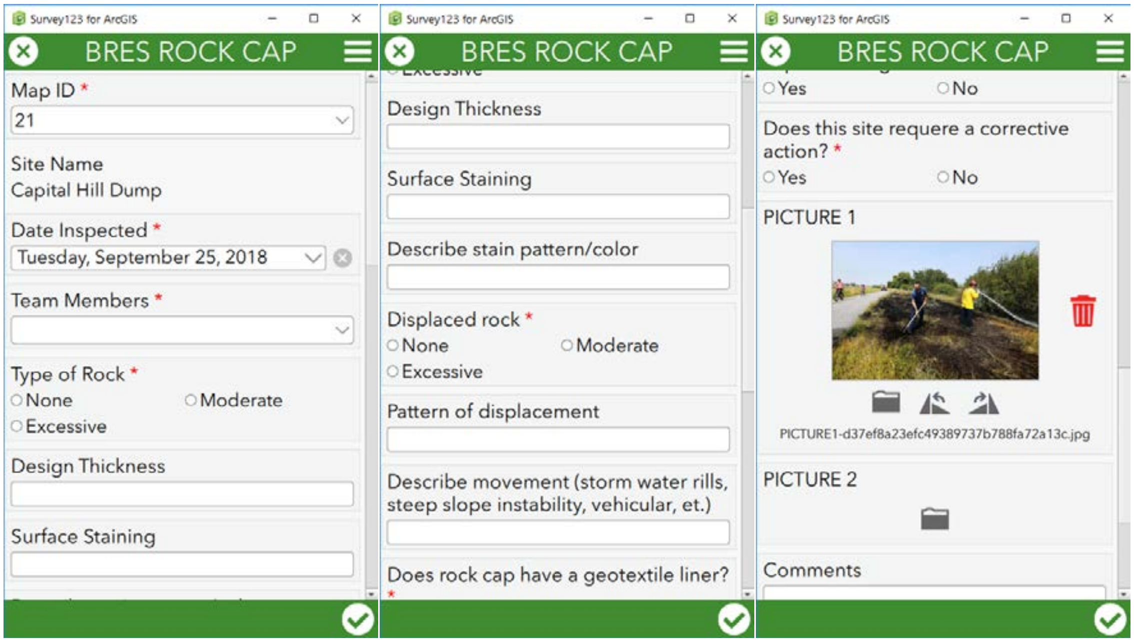
2. If app was launched from ESRI Collector, the SiteID and Site Name are automatically filled in. Selecting a SiteID from the drop-down list automatically selects a site name.
3. Fields with an asterisk (*) beside them are required fields which require an entry. The TOTAL for Vegetation and the Erosion (BLM Form) are automatically calculated. Once the required TOTAL for the Vegetation section equals 100 and the calculated ADJUSTED LIVE score is shown
4. Tapping the arrow next to each section expands it for required data entry.
5. Up to three pictures can be taken with each inspection. To capture a picture, tap the icon in the picture section, take a picture and the app embeds a thumbnail into the form, once

picture 1 has been taken the picture 2 sections becomes visible, picture 3 is visible when pictures 1 and 2 have been taken.

6. When all data has been entered tap the check mark in the bottom right hand corner to submit the data.

Rock Cap Inspection Form

1. The Rock Cap Inspection Form can be launched in the following ways:
    a. The preferred way is to launch the program directly from ESRI collector application using the hyperlink by identifying a BRES polygon. This method automatically fills in the location information (SiteID and Site Name) on the Rock Cap Inspection form.
    b. To launch the application directly tap the Survey123 application on your device.
    c. Sign in with credentials provided by BSB GIS department
    d. Tap Rock Cap Inspection form, then tap Collect to start collecting data.



Map ID *
21
Site Name
Capital Hill Dump
Date Inspected *
Tuesday, September 25, 2018
Team Members *
Type of Rock *
○None ○Moderate
○Excessive
Design Thickness
Surface Staining

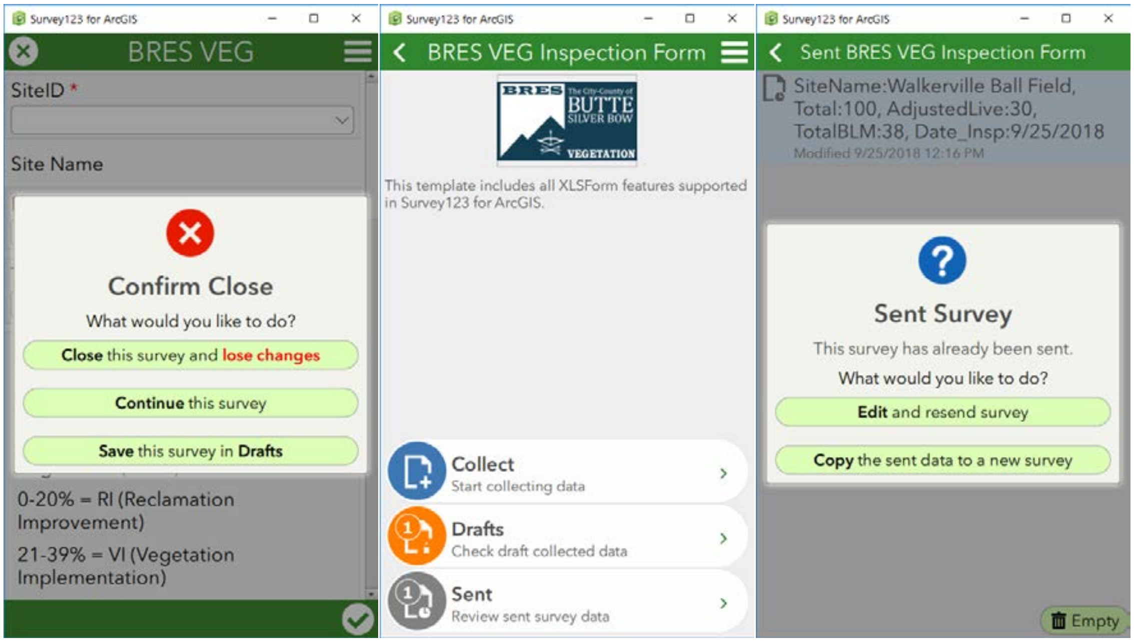
Design Thickness
Surface Staining
Describe stain pattern/color
Displaced rock *
○None ○Moderate
○Excessive
Pattern of displacement
Describe movement (storm water rills, steep slope instability, vehicular, et.)
Does rock cap have a geotextile liner? *

○Yes ○No
Does this site requere a corrective action? *
○Yes ○No
PICTURE 1
PICTURE1-d37ef8a23efc49389737b788fa72a13c.jpg
PICTURE 2
Comments

2. If app was launched from ESRI Collector, the SiteID and Site Name are automatically filled in. Selecting a SiteID from the drop-down list automatically selects a site name.
3. Fields with an asterisk (*) beside them are required fields which require an entry.
4. Up to three pictures can be taken with each inspection. To capture a picture, tap the icon in the picture section, take a picture and the app embeds a thumbnail into the form, once picture 1 has been taken the picture 2 sections becomes visible, picture 3 is visible when pictures 1 and 2 have been taken.
5. When all data has been entered tap the check mark in the bottom right hand corner to submit the data.

Survey123 tips and tricks

- To update a survey from the My Surveys screen, tap the three horizontal lines in the upper right-hand corner of the app, choose the refresh icon to update the survey.
- Setting favorite answers is an excellent way for speed up data entry. With a survey open fill out all of the information in the survey to save as favorites, tap the three horizontal lines in the top right corner of the screen and choose set as favorite answers. To use favorite answers on a new survey, tap the three horizontal lines and choose paste answers from favorite to fill in the appropriate fields.
- To edit a sent survey tap Sent on the main survey screen to Review sent survey data, this button shows a list of submitted surveys. Tapping as sent survey gives the user the option to edit and resend the survey or copy the sent data to a new survey.
- To cancel a survey, tap the X in the upper left portion of the screen, this gives you the option to save the survey as a draft that can be opened later from the main screen.



Confirm Close

What would you like to do?

0-20% = RI (Reclamation Improvement)

21-39% = VI (Vegetation Implementation)

This template includes all XLSForm features supported in Survey123 for ArcGIS.

SiteName:Walkerville Ball Field, Total:100, AdjustedLive:30, TotalBLM:38, Date_Insp:9/25/2018

Modified 9/25/2018 12:16 PM

Sent Survey

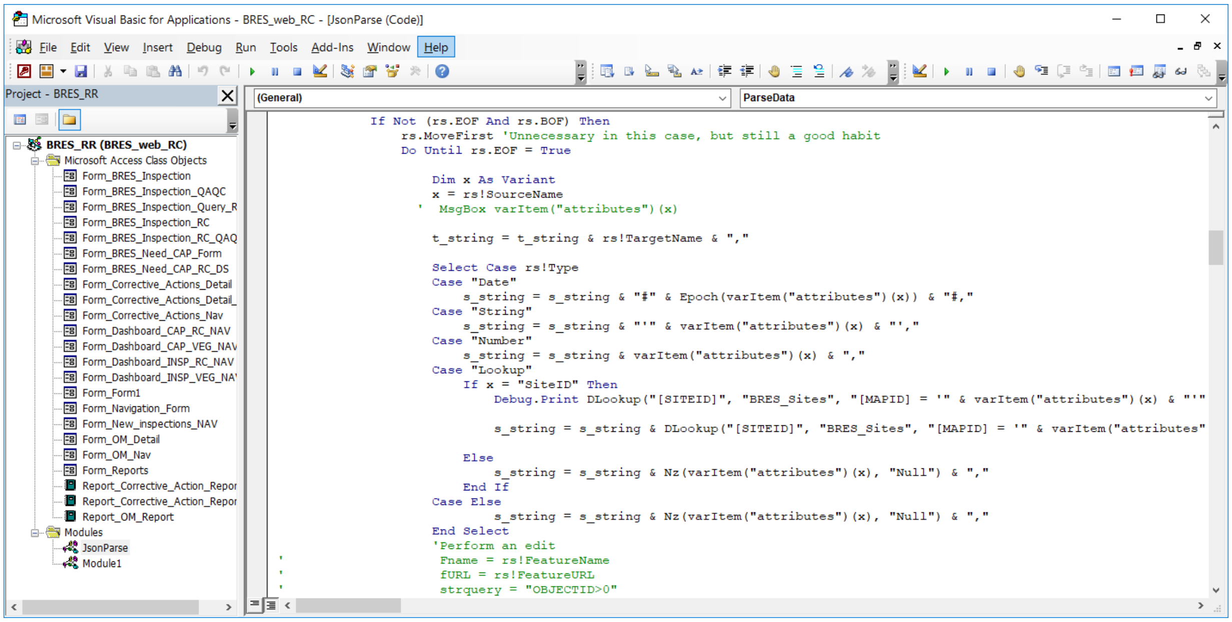
This survey has already been sent.

What would you like to do?

Microsoft Access Application

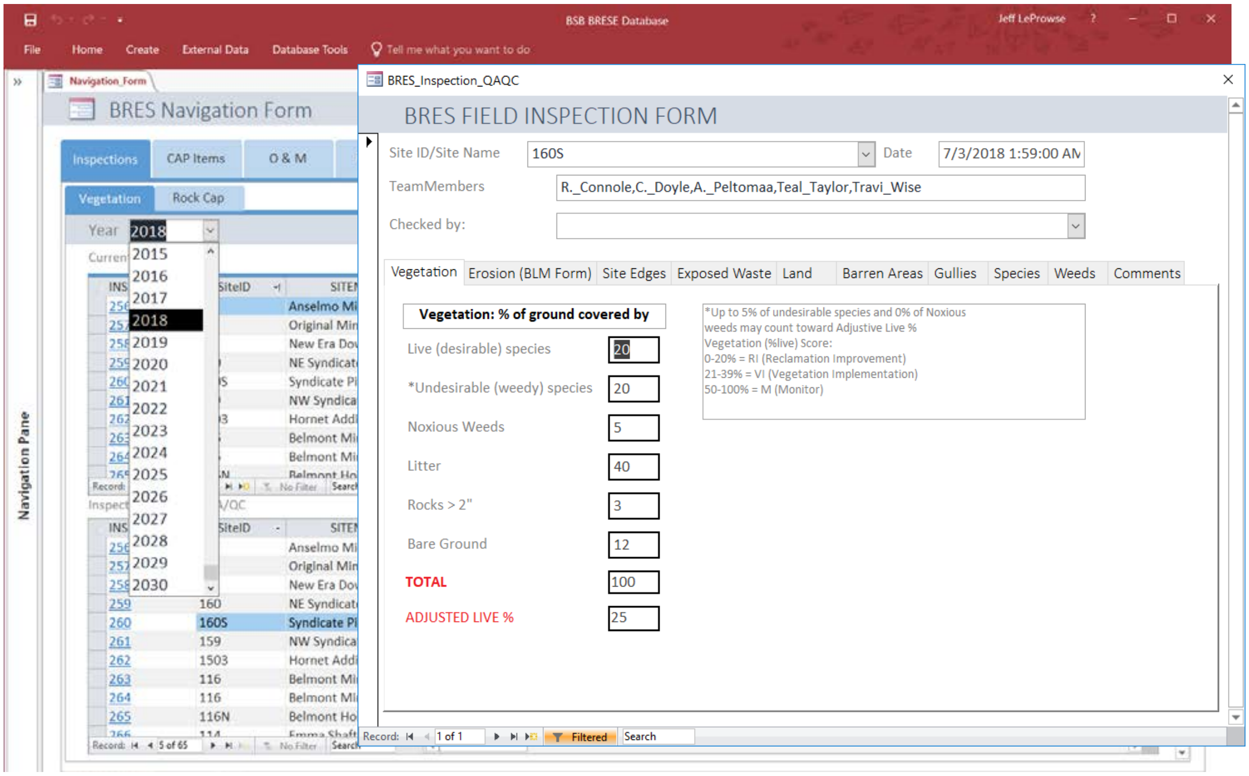
The Microsoft Access application provides a dashboard style view which automatically syncs with ArcGIS online (AGOL). This application leverages ESRI's REST API to retrieve data from BSB's ArcGIS online server. It's important to note this is a one-way sync from ArcGIS online. If a record is deleted in the Access app but not in AGOL the record will be retrieved again on the load event of the navigation form.

The code uses Visual Basic for Applications and can be viewed and edited from within the application by using the Alt+F11 buttons. A screen snap of the code is shown below.



```
            If Not (rs.EOF And rs.BOF) Then
                rs.MoveFirst 'Unnecessary in this case, but still a good habit
                Do Until rs.EOF = True

                    Dim x As Variant
                    x = rs!SourceName
                  '  MsgBox varItem("attributes")(x)

                    t_string = t_string & rs!TargetName & ","

                    Select Case rs!Type
                    Case "Date"
                        s_string = s_string & "#" & Epoch(varItem("attributes")(x)) & "#,"
                    Case "String"
                        s_string = s_string & "'" & varItem("attributes")(x) & "',"
                    Case "Number"
                        s_string = s_string & varItem("attributes")(x) & ","
                    Case "Lookup"
                        If x = "SiteID" Then
                            Debug.Print DLookup("[SITEID]", "BRES_Sites", "[MAPID] = '" & varItem("attributes")(x) & "'"

                            s_string = s_string & DLookup("[SITEID]", "BRES_Sites", "[MAPID] = '" & varItem("attributes"

                        Else
                            s_string = s_string & Nz(varItem("attributes")(x), "Null") & ","
                        End If
                    Case Else
                            s_string = s_string & Nz(varItem("attributes")(x), "Null") & ","
                    End Select
                    'Perform an edit
'                    Fname = rs!FeatureName
'                    fURL = rs!FeatureURL
'                    strquery = "OBJECTID>0"
```

The dashboard style form automatically opens and syncs with AGOL on when the database is opened. As shown in the screen snap below this navigation form allows users to quickly query data by year by selecting a year from the drop-down list and clicking through the Vegetation, Rock Cap inspections or the Corrective Action Plans (CAP Items). Clicking the hyperlink for individual INSPECTIONID displays details about each item. The first tab of the navigation form displays inspections by year and inspections that need to be QA/QC'd in the bottom screen. Once inspections have been QA/QC'd any items requiring a corrective action plan will appear in the CAP Items tab.



BRES FIELD INSPECTION FORM

Site ID/Site Name: 160S
Date: 7/3/2018 1:59:00 AM
TeamMembers: R._Connole,C._Doyle,A._Peltomaa,Teal_Taylor,Travi_Wise
Checked by:

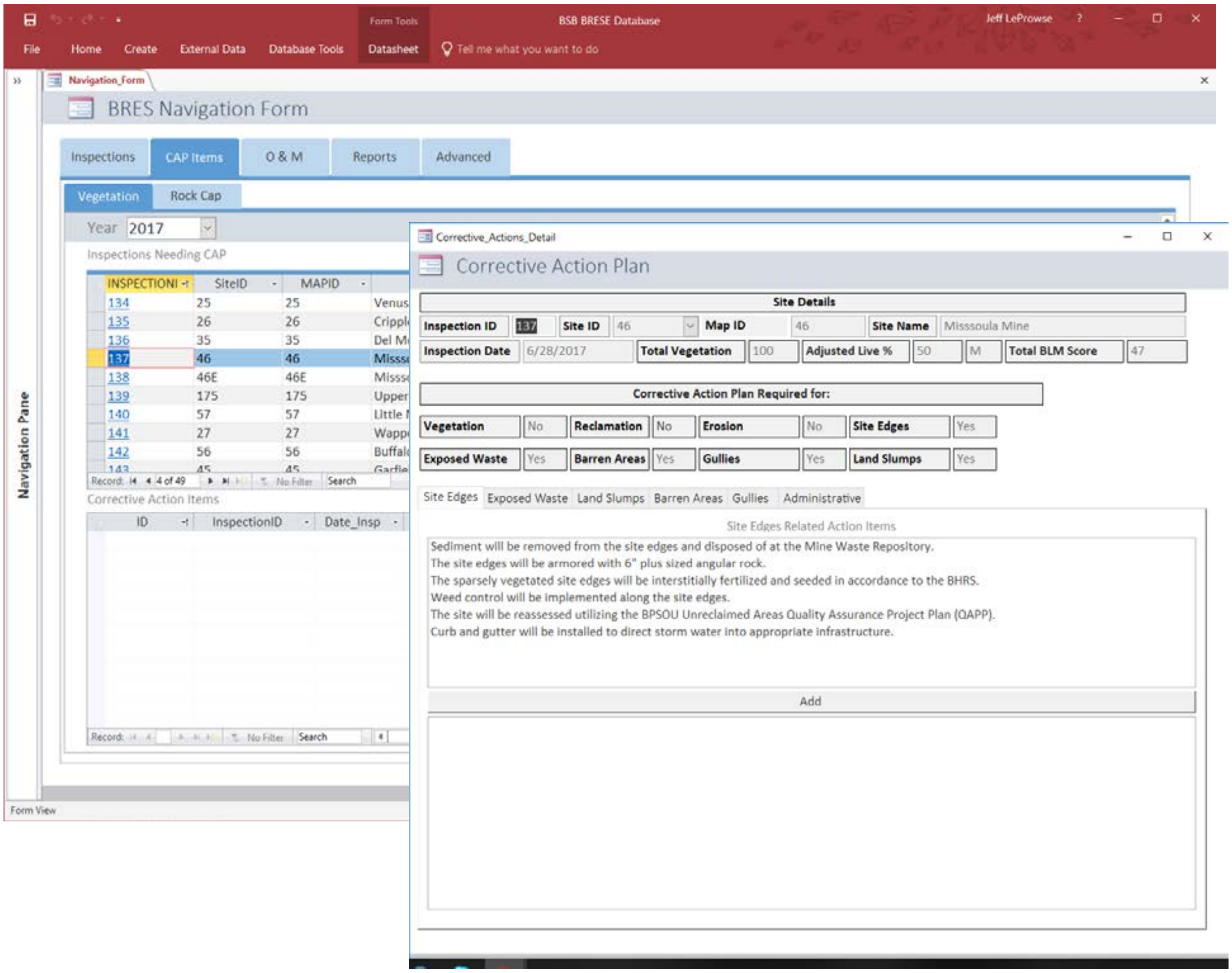
Vegetation | Erosion (BLM Form) | Site Edges | Exposed Waste | Land | Barren Areas | Gullies | Species | Weeds | Comments

Vegetation: % of ground covered by

Live (desirable) species	20
*Undesirable (weedy) species	20
Noxious Weeds	5
Litter	40
Rocks > 2"	3
Bare Ground	12
TOTAL	100
ADJUSTED LIVE %	25

*Up to 5% of undesirable species and 0% of Noxious weeds may count toward Adjustive Live %
Vegetation (%live) Score:
0-20% = RI (Reclamation Improvement)
21-39% = VI (Vegetation Implementation)
50-100% = M (Monitor)

The CAP Items tab shown below displays Inspections needing a corrective action plan (CAP) in the top section of the form, clicking the hyperlink for the INSPECTIONID column displays a pop up form to apply corrective actions for the inspection which moves the item to the corrective Action Items list.



Year 2017

Inspections Needing CAP

INSPECTIONI	SiteID	MAPID	
134	25	25	Venus
135	26	26	Crippl
136	35	35	Del M
137	46	46	Missso
138	46E	46E	Missso
139	175	175	Upper
140	57	57	Little
141	27	27	Wapp
142	56	56	Buffal
143	45	45	Garfie

Corrective Action Items

ID	InspectionID	Date_Insp

Corrective Action Plan

Site Details

Inspection ID	137	**Site ID**	46	**Map ID**	46	**Site Name**	Misssoula Mine
Inspection Date	6/28/2017	**Total Vegetation**	100	**Adjusted Live %**	50	M	**Total BLM Score** 47

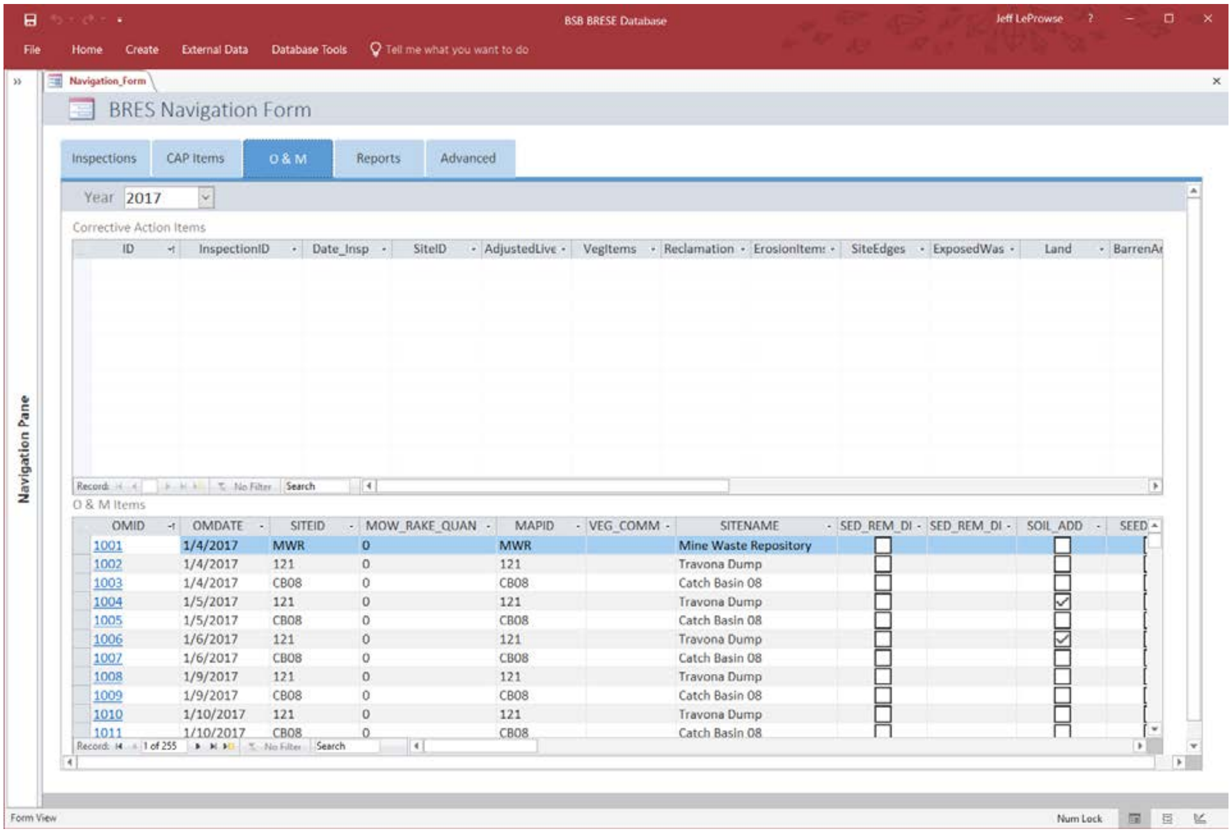
Corrective Action Plan Required for:

Vegetation	No	**Reclamation**	No	**Erosion**	No	**Site Edges**	Yes
Exposed Waste	Yes	**Barren Areas**	Yes	**Gullies**	Yes	**Land Slumps**	Yes

Site Edges Related Action Items

Sediment will be removed from the site edges and disposed of at the Mine Waste Repository.
The site edges will be armored with 6" plus sized angular rock.
The sparsely vegetated site edges will be interstitially fertilized and seeded in accordance to the BHRS.
Weed control will be implemented along the site edges.
The site will be reassessed utilizing the BPSOU Unreclaimed Areas Quality Assurance Project Plan (QAPP).
Curb and gutter will be installed to direct storm water into appropriate infrastructure.

The O&M tab allows for recording Operating and Maintenance activities per year and shows a list of Corrective action items in the top window. The O&M section has been replaced by a different application BSB started utilizing in 2018 to have field crews record this information on iPad's with Survey123.



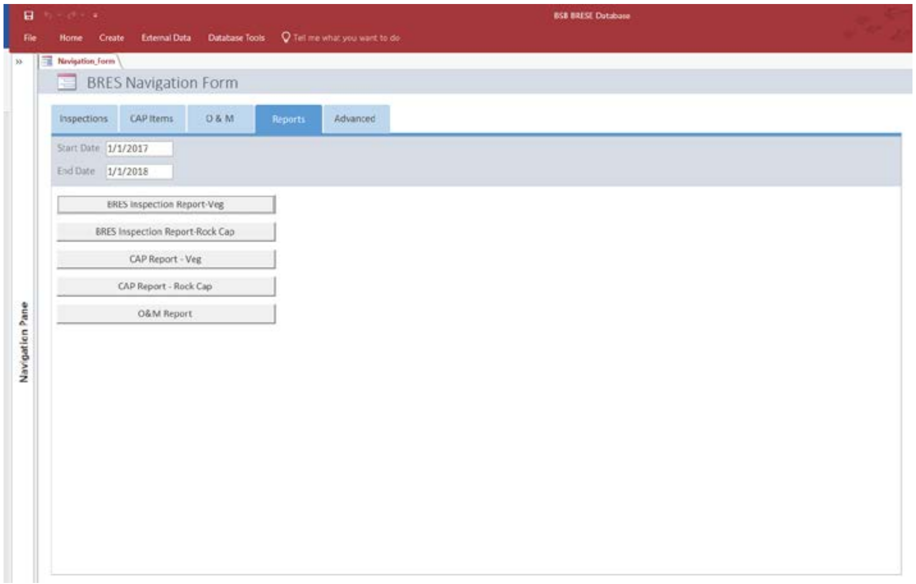
Year 2017

O & M Items

OMID	OMDATE	SITEID	MOW_RAKE_QUAN	MAPID	VEG_COMM	SITENAME	SED_REM_DI	SED_REM_DI	SOIL_ADD	SEED
1001	1/4/2017	MWR	0	MWR		Mine Waste Repository	☐		☐	
1002	1/4/2017	121	0	121		Travona Dump	☐		☐	
1003	1/4/2017	CB08	0	CB08		Catch Basin 08	☐		☐	
1004	1/5/2017	121	0	121		Travona Dump	☐		☑	
1005	1/5/2017	CB08	0	CB08		Catch Basin 08	☐		☐	
1006	1/6/2017	121	0	121		Travona Dump	☐		☑	
1007	1/6/2017	CB08	0	CB08		Catch Basin 08	☐		☐	
1008	1/9/2017	121	0	121		Travona Dump	☐		☐	
1009	1/9/2017	CB08	0	CB08		Catch Basin 08	☐		☐	
1010	1/10/2017	121	0	121		Travona Dump	☐		☐	
1011	1/10/2017	CB08	0	CB08		Catch Basin 08	☐		☐	

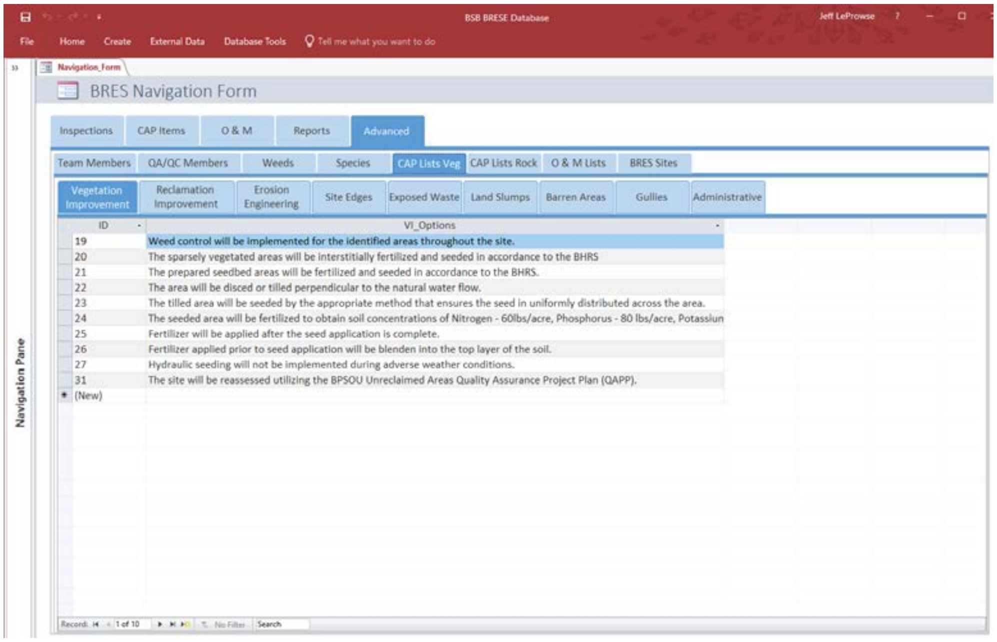
Record: 1 of 255

The reports tab allows easy generation of reports between two dates as shown below. To generate a report, enter start and end date can click the appropriate report to run and print or save as a PDF.



Start Date 1/1/2017
End Date 1/1/2018

The Advanced tab allows users to add and edit information on the various list that appear in the database.



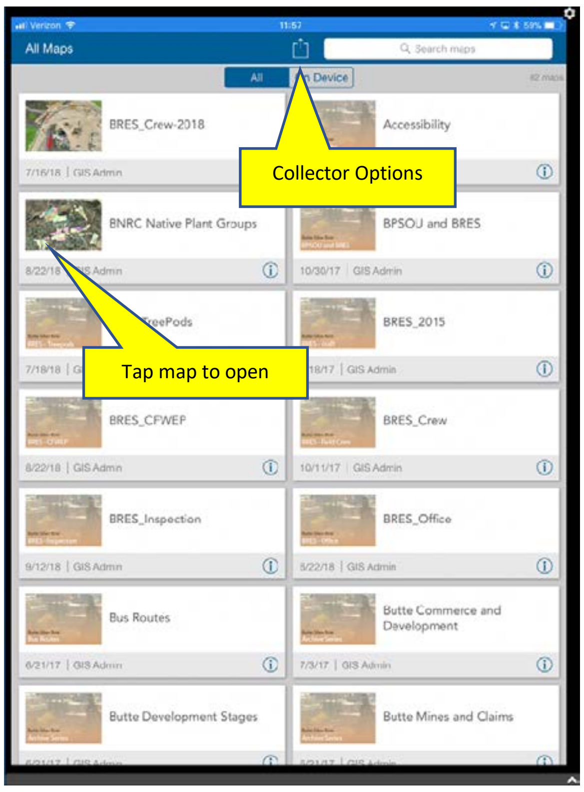
ID	VI_Options
19	Weed control will be implemented for the identified areas throughout the site.
20	The sparsely vegetated areas will be interstitially fertilized and seeded in accordance to the BHRS
21	The prepared seedbed areas will be fertilized and seeded in accordance to the BHRS.
22	The area will be disced or tilled perpendicular to the natural water flow.
23	The tilled area will be seeded by the appropriate method that ensures the seed in uniformly distributed across the area.
24	The seeded area will be fertilized to obtain soil concentrations of Nitrogen - 60lbs/acre, Phosphorus - 80 lbs/acre, Potassiun
25	Fertilizer will be applied after the seed application is complete.
26	Fertilizer applied prior to seed application will be blenden into the top layer of the soil.
27	Hydraulic seeding will not be implemented during adverse weather conditions.
31	The site will be reassessed utilizing the BPSOU Unreclaimed Areas Quality Assurance Project Plan (QAPP).
(New)	

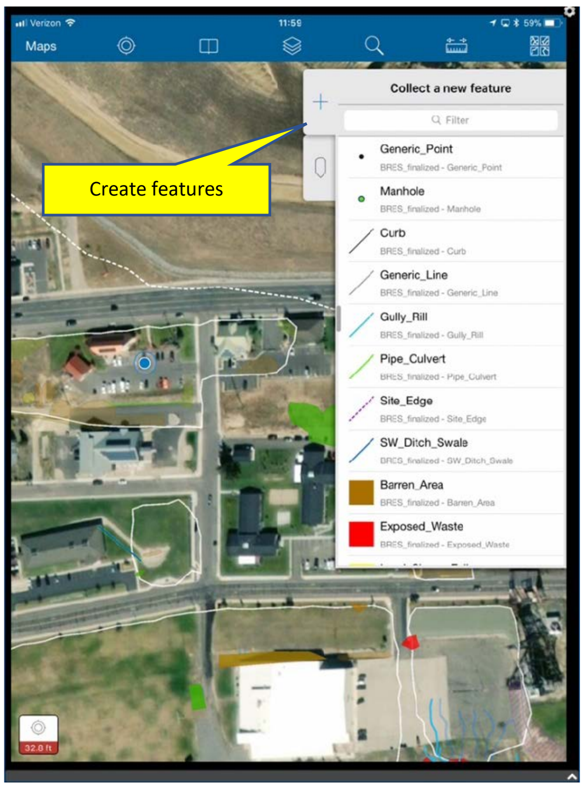
Appendix A

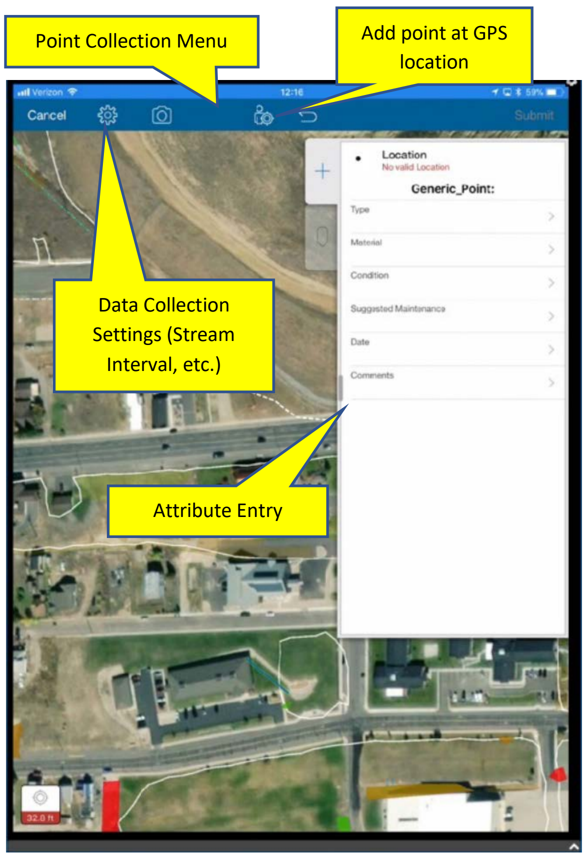
Quick Reference Guides

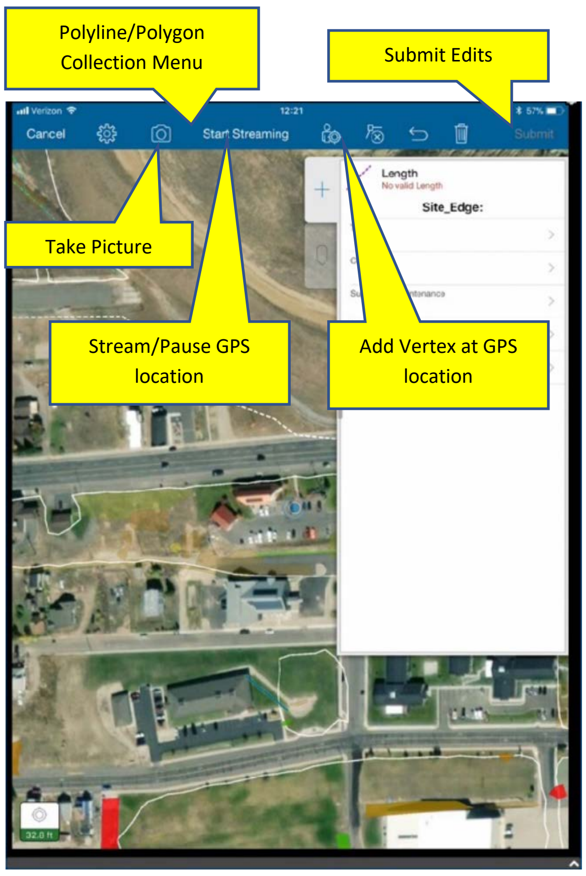
Appendix A – Quick Reference Guides

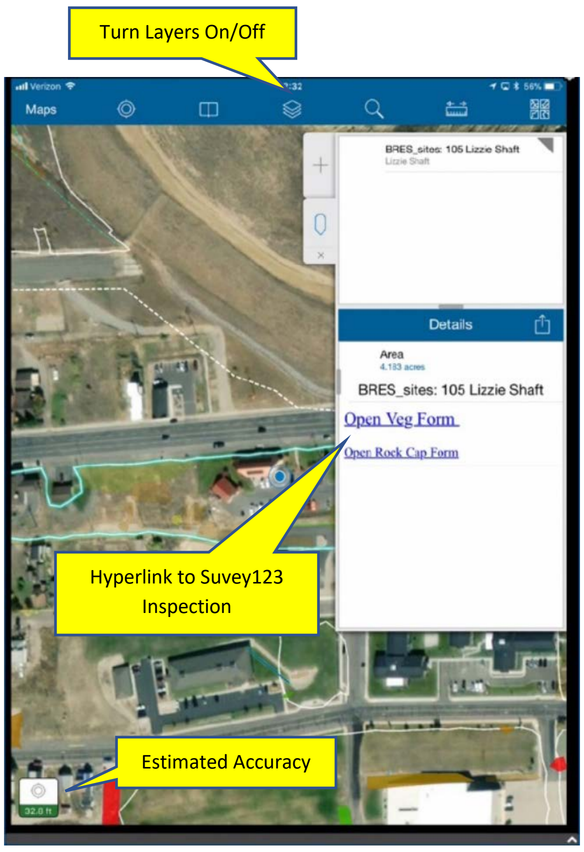
ESRI Collector

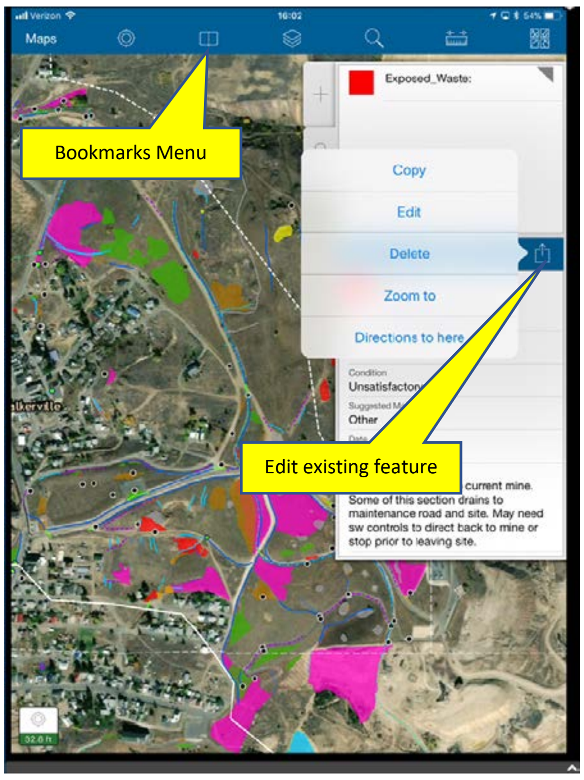


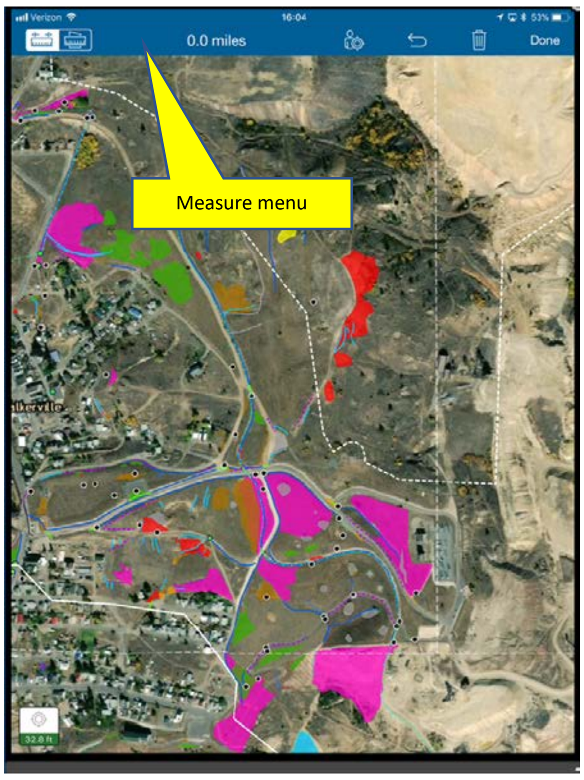




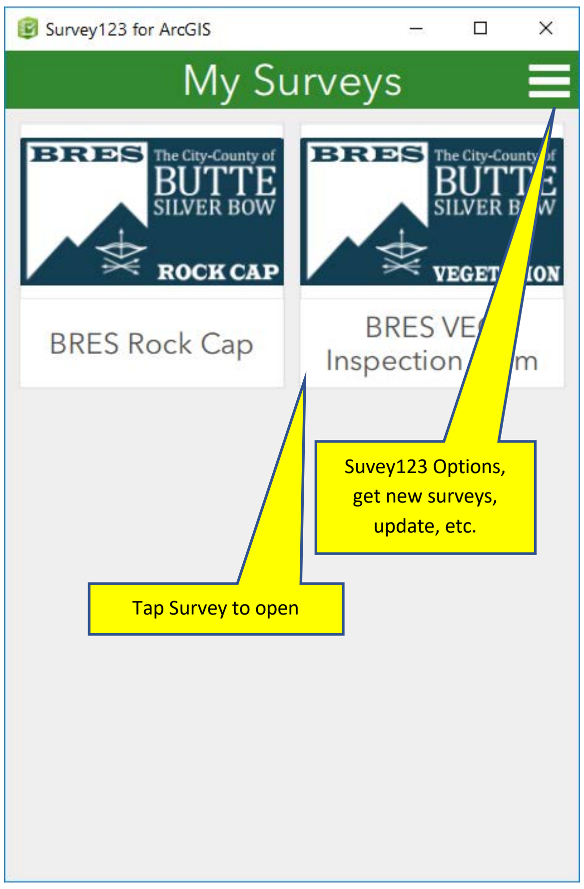






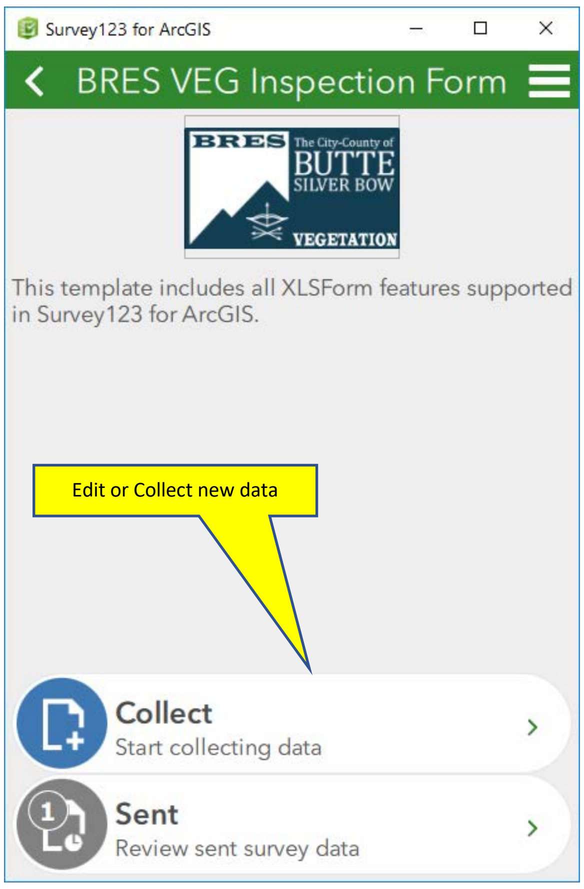


Survey123 Vegetation Inspection



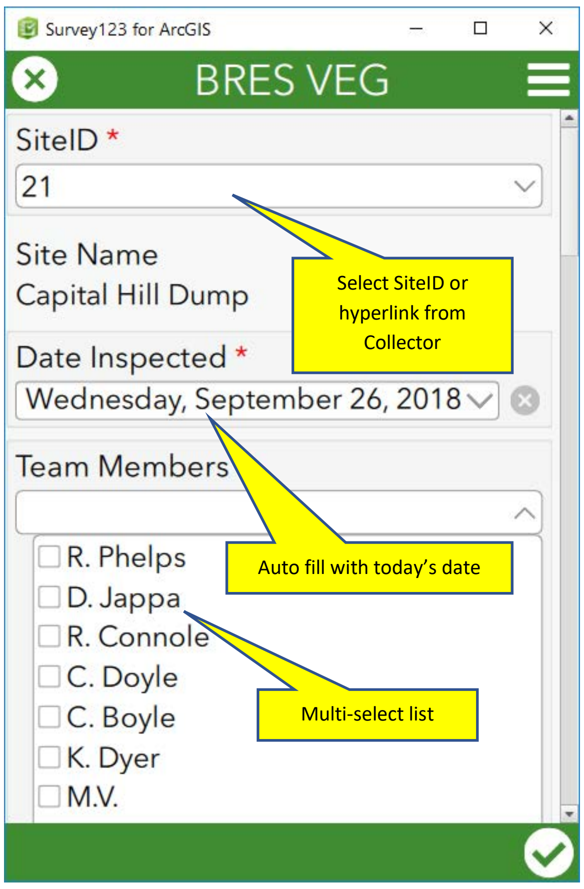
Survey123 Options, get new surveys, update, etc.

Tap Survey to open



This template includes all XLSForm features supported in Survey123 for ArcGIS.

Edit or Collect new data



SiteID *
21

Site Name
Capital Hill Dump

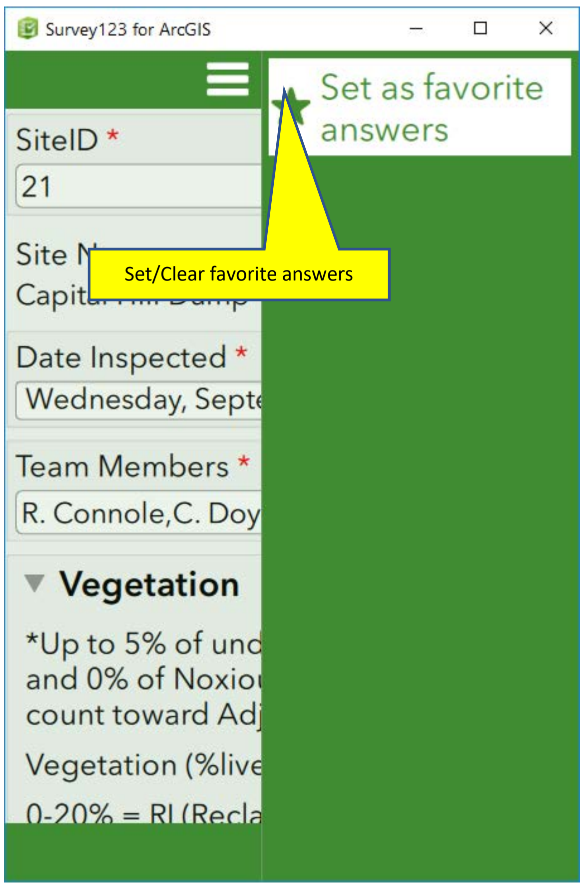
Date Inspected *
Wednesday, September 26, 2018

Team Members *
☐ R. Phelps
☐ D. Jappa
☐ R. Connole
☐ C. Doyle
☐ C. Boyle
☐ K. Dyer
☐ M.V.

Select SiteID or hyperlink from Collector

Auto fill with today's date

Multi-select list



SiteID *
21

Site N…
Capit…

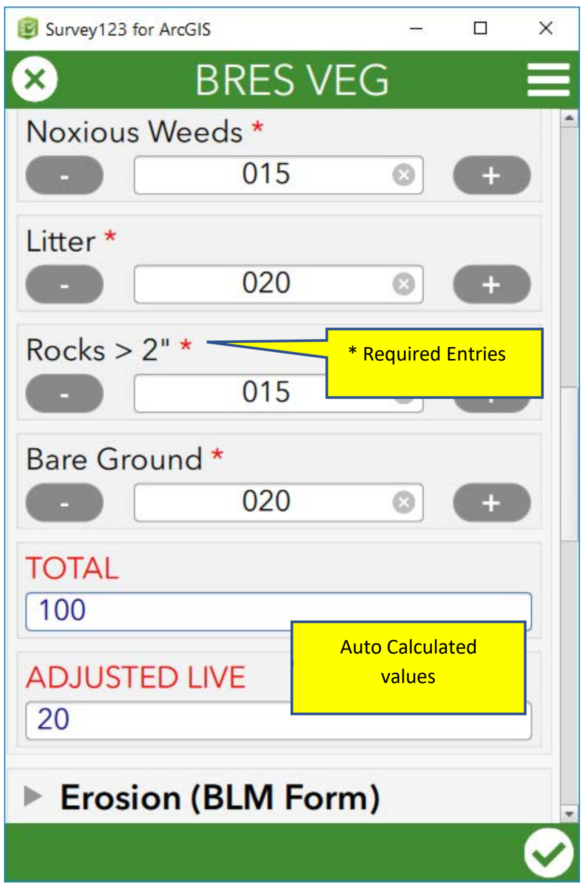
Date Inspected *
Wednesday, Septe…

Team Members *
R. Connole,C. Doy…

Vegetation

*Up to 5% of und… and 0% of Noxio… count toward Adj…
Vegetation (%live…
0-20% = RI (Recla…

Set/Clear favorite answers



Noxious Weeds *
015

Litter *
020

Rocks > 2" *
015

Bare Ground *
020

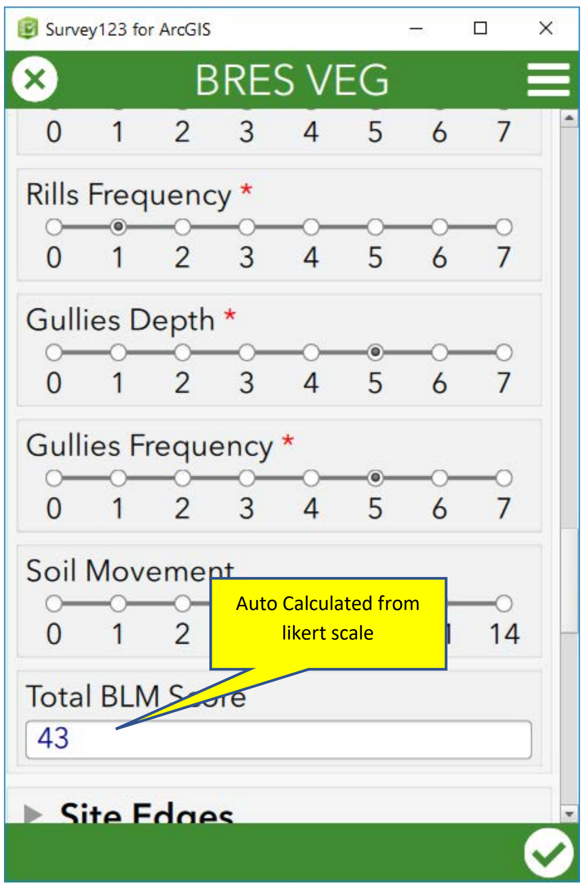
TOTAL
100

ADJUSTED LIVE
20

Erosion (BLM Form)

* Required Entries

Auto Calculated values



Rills Frequency *
● 1

Gullies Depth *
● 5

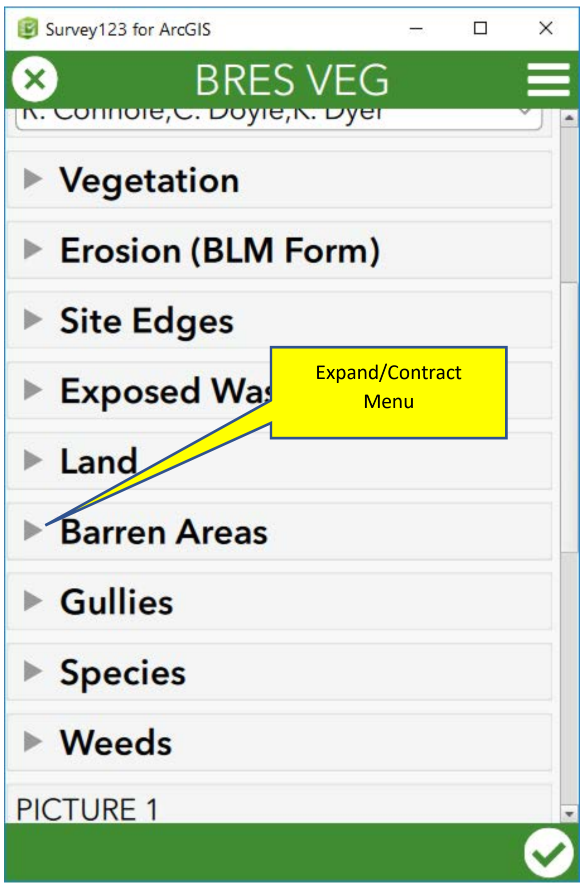
Gullies Frequency *
● 5

Soil Movement
0 1 2 … 14

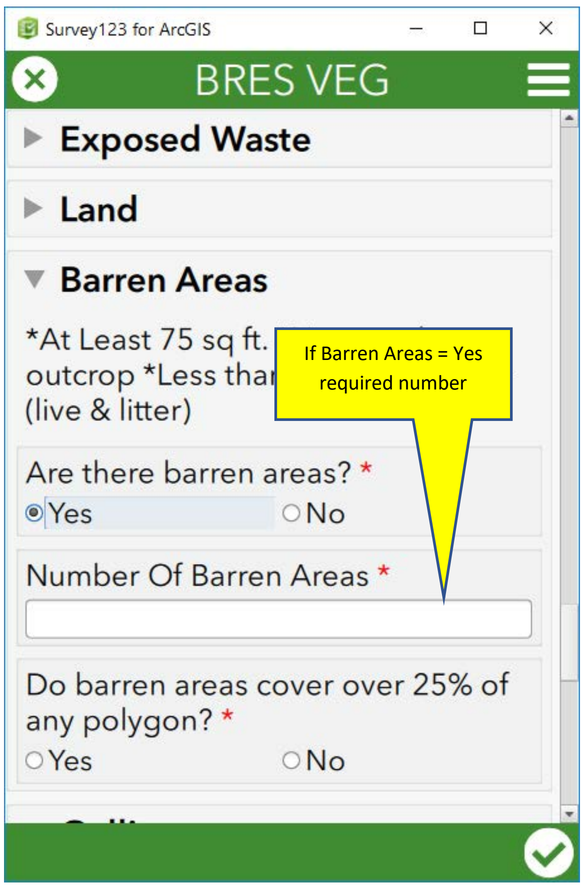
Total BLM Score
43

Site Edges

Auto Calculated from likert scale



Expand/Contract Menu



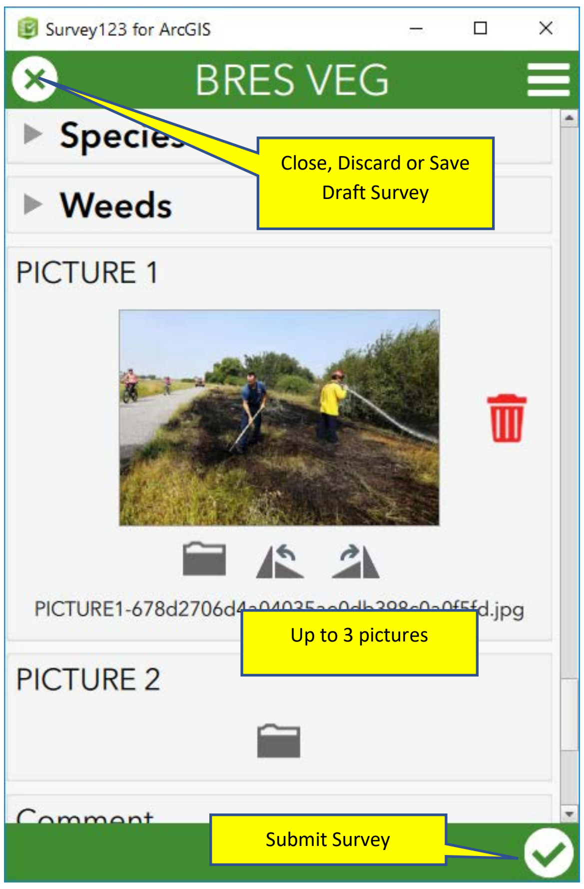
*At Least 75 sq ft. … outcrop *Less tha… (live & litter)

Are there barren areas? *
● Yes ○ No

Number Of Barren Areas *

Do barren areas cover over 25% of any polygon? *
○ Yes ○ No

If Barren Areas = Yes required number

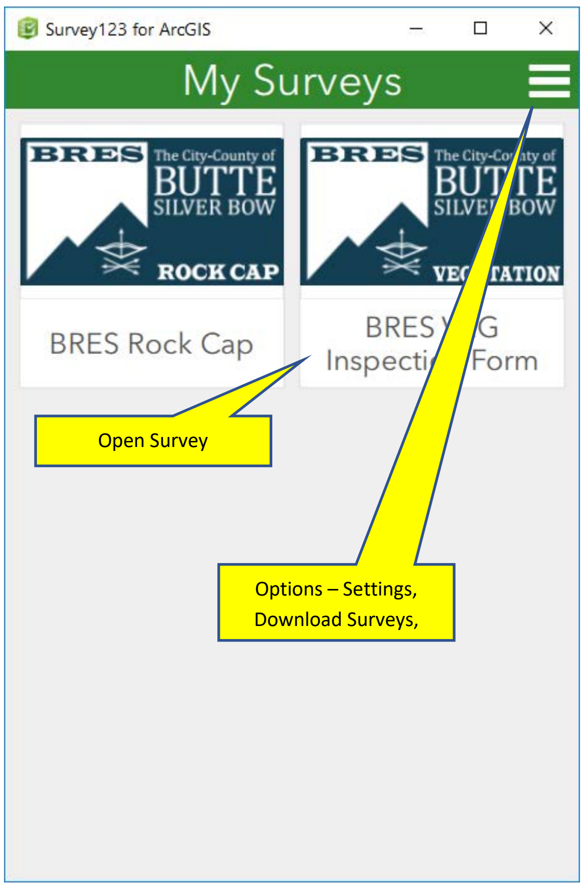


Close, Discard or Save Draft Survey

Up to 3 pictures

Submit Survey

Survey123 Rock Cap Inspection



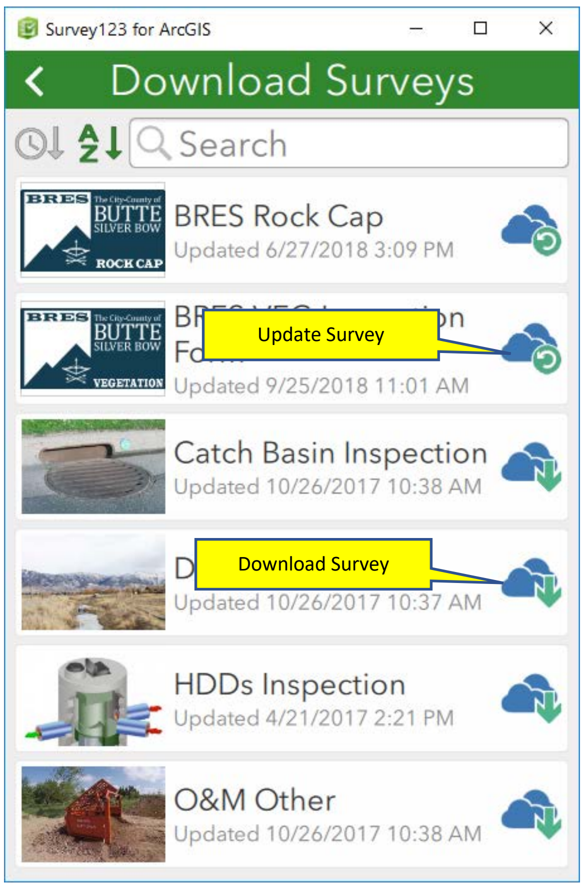
My Surveys

BRES Rock Cap

BRES VEG Inspection Form

Open Survey

Options – Settings, Download Surveys,



Download Surveys

BRES Rock Cap
Updated 6/27/2018 3:09 PM

BRES VEG Inspection Form
Updated 9/25/2018 11:01 AM

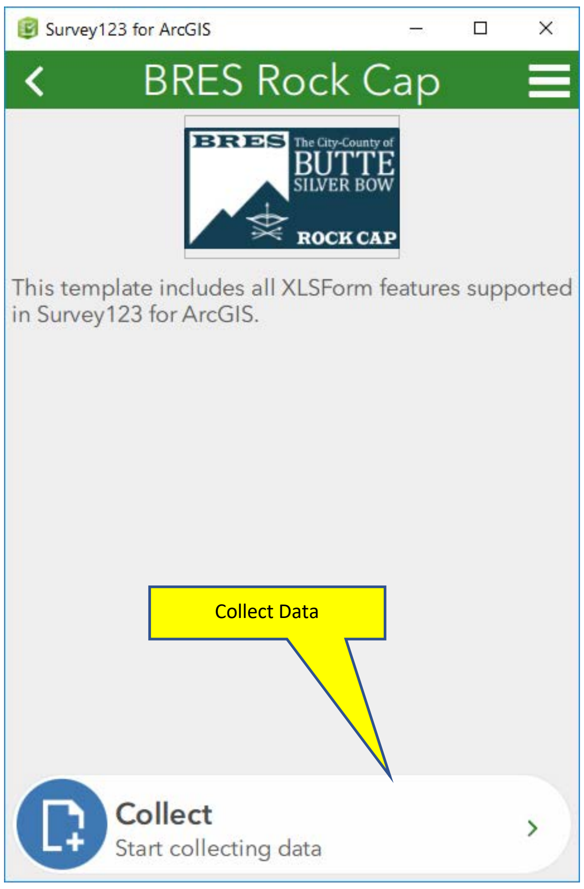
Update Survey

Catch Basin Inspection
Updated 10/26/2017 10:38 AM

Download Survey
Updated 10/26/2017 10:37 AM

HDDs Inspection
Updated 4/21/2017 2:21 PM

O&M Other
Updated 10/26/2017 10:38 AM

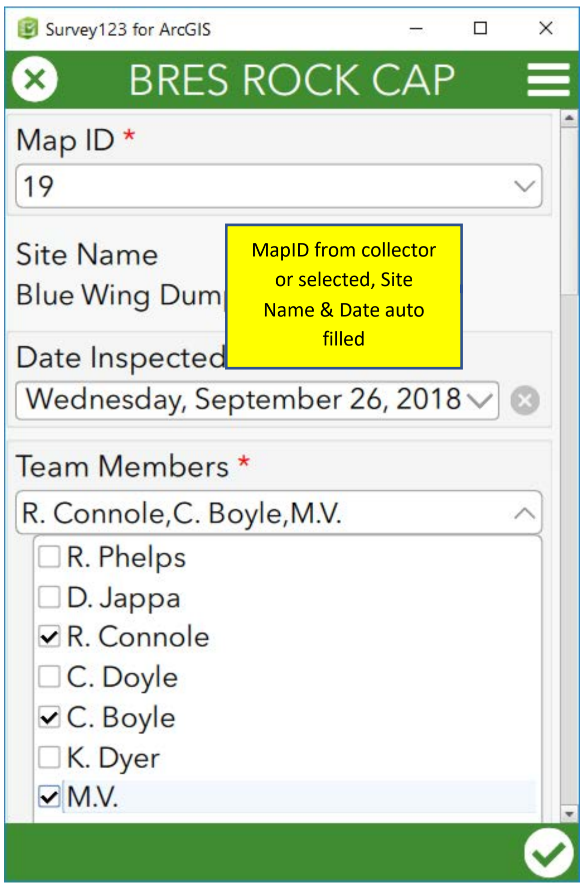


BRES Rock Cap

This template includes all XLSForm features supported in Survey123 for ArcGIS.

Collect Data

Collect
Start collecting data



BRES ROCK CAP

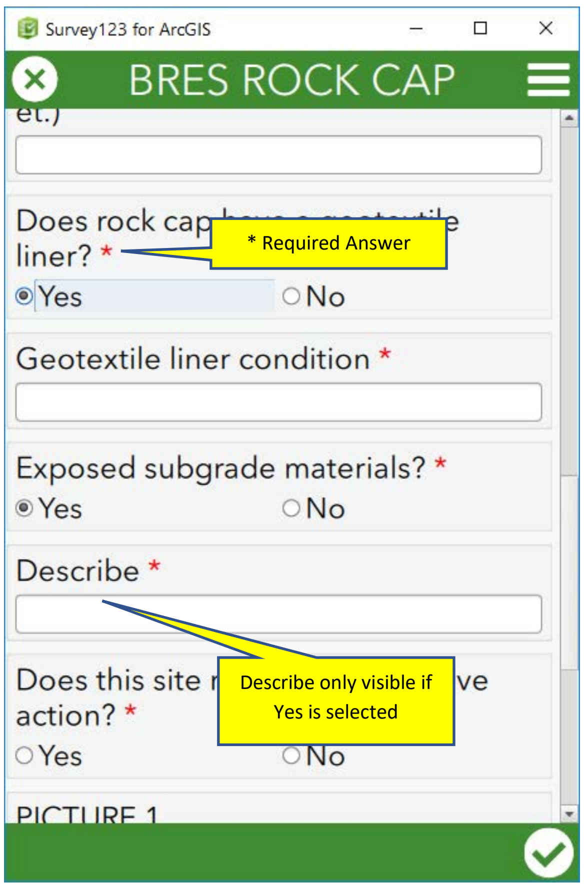
Map ID *
19

Site Name
Blue Wing Dump

MapID from collector or selected, Site Name & Date auto filled

Date Inspected
Wednesday, September 26, 2018

Team Members *
R. Connole,C. Boyle,M.V.
☐ R. Phelps
☐ D. Jappa
☑ R. Connole
☐ C. Doyle
☑ C. Boyle
☐ K. Dyer
☑ M.V.



BRES ROCK CAP

Does rock cap have a geotextile liner? *
● Yes ○ No

* Required Answer

Geotextile liner condition *

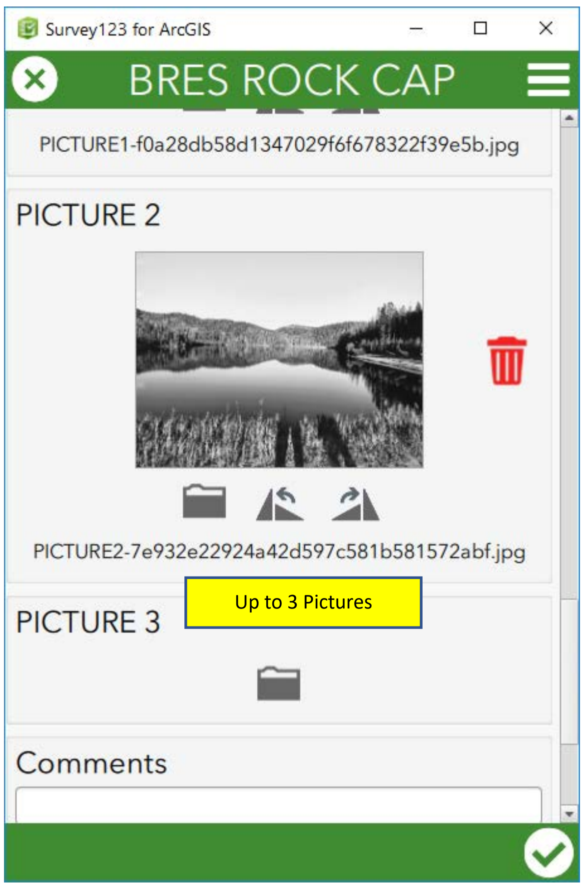
Exposed subgrade materials? *
● Yes ○ No

Describe *

Describe only visible if Yes is selected

Does this site require action? *
○ Yes ○ No

PICTURE 1



BRES ROCK CAP

PICTURE1-f0a28db58d1347029f6f678322f39e5b.jpg

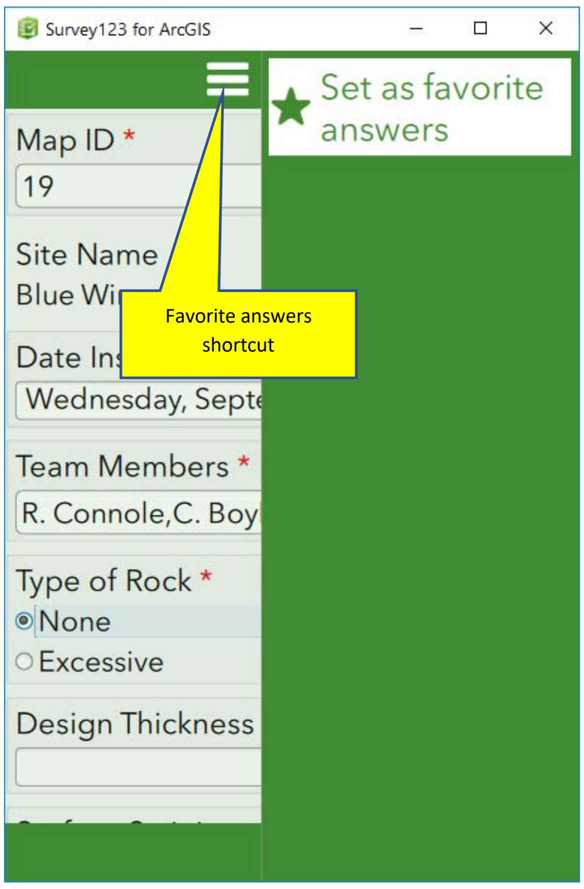
PICTURE 2

PICTURE2-7e932e22924a42d597c581b581572abf.jpg

Up to 3 Pictures

PICTURE 3

Comments



Set as favorite answers

Map ID *
19

Site Name
Blue Wi

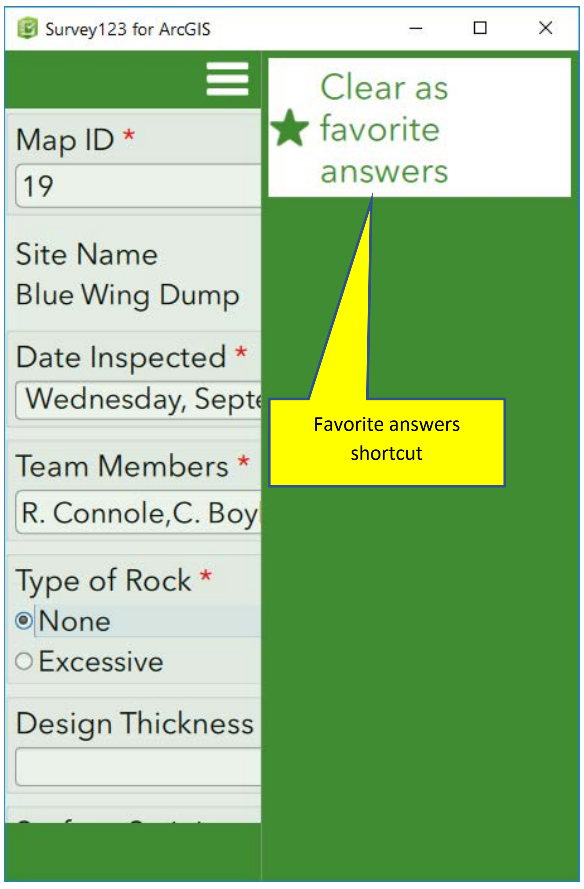
Date Ins
Wednesday, Septe

Team Members *
R. Connole,C. Boy

Type of Rock *
● None
○ Excessive

Design Thickness

Favorite answers shortcut



Clear as favorite answers

Map ID *
19

Site Name
Blue Wing Dump

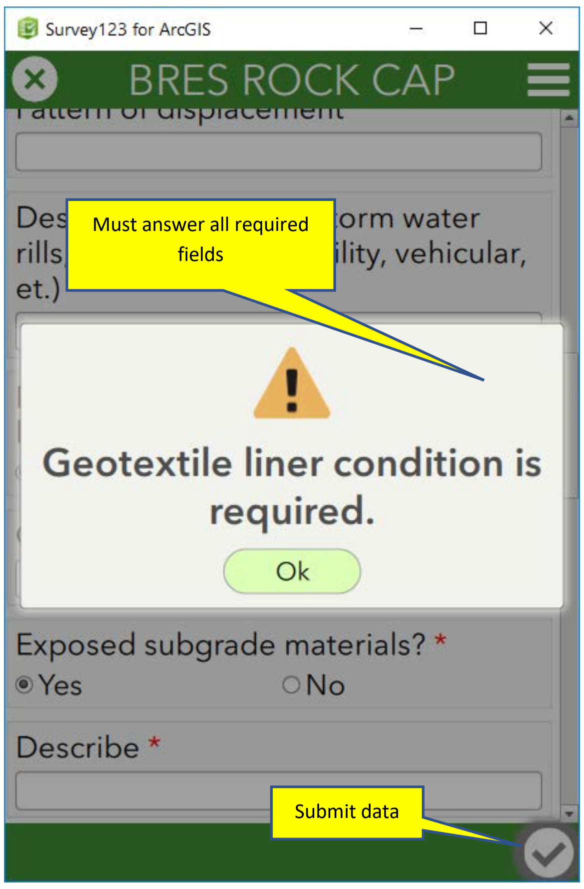
Date Inspected *
Wednesday, Septe

Team Members *
R. Connole,C. Boy

Type of Rock *
● None
○ Excessive

Design Thickness

Favorite answers shortcut



BRES ROCK CAP

Pattern of displacement

Describe (storm water rills, utility, vehicular, etc.)

Must answer all required fields

Geotextile liner condition is required.

Exposed subgrade materials? *
● Yes ○ No

Describe *

Submit data

Appendix B

Schema Design of Feature Classes

Feature = Vegetation Inspection			
Name	**Field_Type**	**Alias**	**Length**
objectid	OID	ObjectID	
globalid	GlobalID	GlobalID	38
SiteID	String	SiteID	9
sitecal	String	sitecal	255
SiteName	String	Site Name	255
Date_Insp	Date	Date Inspected	255
TeamM	String	Team Members	255
LiveDesirableSpecies	Integer	Live (desirable species)	
LiveUndesirableWeedySpecies	Integer	Undesirable (weedy species)	
NoxiousWeeds	Integer	Noxious Weeds	
LitterIncMoss	Integer	Litter	
RocksGT2Inches	Integer	Rocks > 2"	
BareGround	Integer	Bare Ground	
Total	Integer	TOTAL	
AdjustedLive	Integer	ADJUSTED LIVE	
blmnote	String	BLM Score 0-55 = M (Monitor) 56-100 = EV (Engineering Evaluation)	255
SurfaceLitter	String	Surface Litter	2
SurfaceRockMovement	String	Surface Rock Movement	2
Pedestalling	String	Pedestalling	2
FlowPatterns	String	Flow Patterns	2
RillsDepth	String	Rills Depth	1
RillsFrequenct	String	Rills Frequency	1
GulliesDepth	String	Gullies Depth	1
GulliesFrequency	String	Gullies Frequency	1
SoilMovement	String	Soil Movement	2
TotalBLM	Integer	Total BLM Score	
SiteEdgesYN	String	Are outer edges of the site significantly different than the remainder of the site?	3
LimeRockBarrier	String	Lime Rock Barrier	3
DepositionalArea	String	Depositional Area	3
MoreWeeds	String	More Weeds	3
SteeperSlope	String	Steeper Slope	3
IncreasedErosion	String	Increased Erosion	3
LessVegetation	String	Less Vegetation	3
Gullies	String	Gullies	3
ExposedWasteMaterial	String	Exposed Waste Material?	3
NumberOfAreasWithExposedWaste	Integer	Number of Areas with Exposed Waste	
BulkSoilFailure	String	Bulk Soil Failure	3
Subsidence	String	Subsidence	3
LandSlumps	String	Land Slumps	3
atleast	String	*At Least 75 sq ft. *Not a rock outcrop *Less than 10% total cover (live & litter	255
BarrenAreasYN	String	Are there barren areas?	3
NumberOfBarrenAreas	Integer	Number Of Barren Areas	
DoBarrenAreasCoverOver25	String	Do barren areas cover over 25% of any polygon?	3
GulliesOver6InchesYN	String	Are there any gullies over 6" in depth	3
AreAnyGulliesActivelyEroding	String	Are any gullies actively eroding	3
DSpecies	String	Dominant	255
FSpecies	String	Frequent	255
ifSpecies	String	Infrequent	255

Dweeds	String	Dominant	255
Fweeds	String	Frequent	255
ifweeds	String	Infrequent	255
Comment	String	Comment	255
CreationDate	Date	CreationDate	8
Creator	String	Creator	128
EditDate	Date	EditDate	8
Editor	String	Editor	128

Feature = Rock Cap Inspection

Name	**Field_Type**	**Alias**	**Length**
objectid	OID	ObjectID	
globalid	GlobalID	GlobalID	38
SiteID	String	Map ID	9
sitecal	String	sitecal	255
SName	String	Site Name	255
Date_Insp	Date	Date Inspected	255
TeamM	String	Team Members	255
ROCK_TYPE	String	Type of Rock	9
DESIGN_THICK	String	Design Thickness	255
SUR_STAIN	String	Surface Staining	255
SUR_STAIN_COMMENT	String	Describe stain pattern/color	255
DISP_ROCK	String	Displaced rock	9
DISP_ROCK_PATT	String	Pattern of displacement	255
MOVEMENT	String	Describe movement (storm water rills	255
GEOTEX_LINER	String	Does rock cap have a geotextile liner?	3
GEOTEX_LINER_COND	String	Geotextile liner condition	255
EXP_SUBGRADE	String	Exposed subgrade materials?	3
EXP_SUBGRADE_DETAIL	String	Describe	255
REQUIRE_CAP	String	Does this site requere a corrective action?	3
COMMENTS	String	Comments	255
CreationDate	Date	CreationDate	8
Creator	String	Creator	128
EditDate	Date	EditDate	8
Editor	String	Editor	128

Attachment 4
Annual Updates

Rev. No.	Year	Description
1	2021	Updated recipient lists to reflect current distribution list. Updated Section 2 Project Organization and Responsibilities and Figure 2 to reflect Atlantic Richfield roles and title changes. Updated citations to reference the most recent 2018 Atlantic Richfield Company Data Management Plan (DMP) and the 2019 Institutional Controls Implementation and Assurance Plan. Attachment 3.1 Updated SMP-10. Attachment 3.2 Updated data validation checklists. Attachment 3.5 Added Product Documentation and User Guide – Butte Reclamation Evaluation System