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Office of
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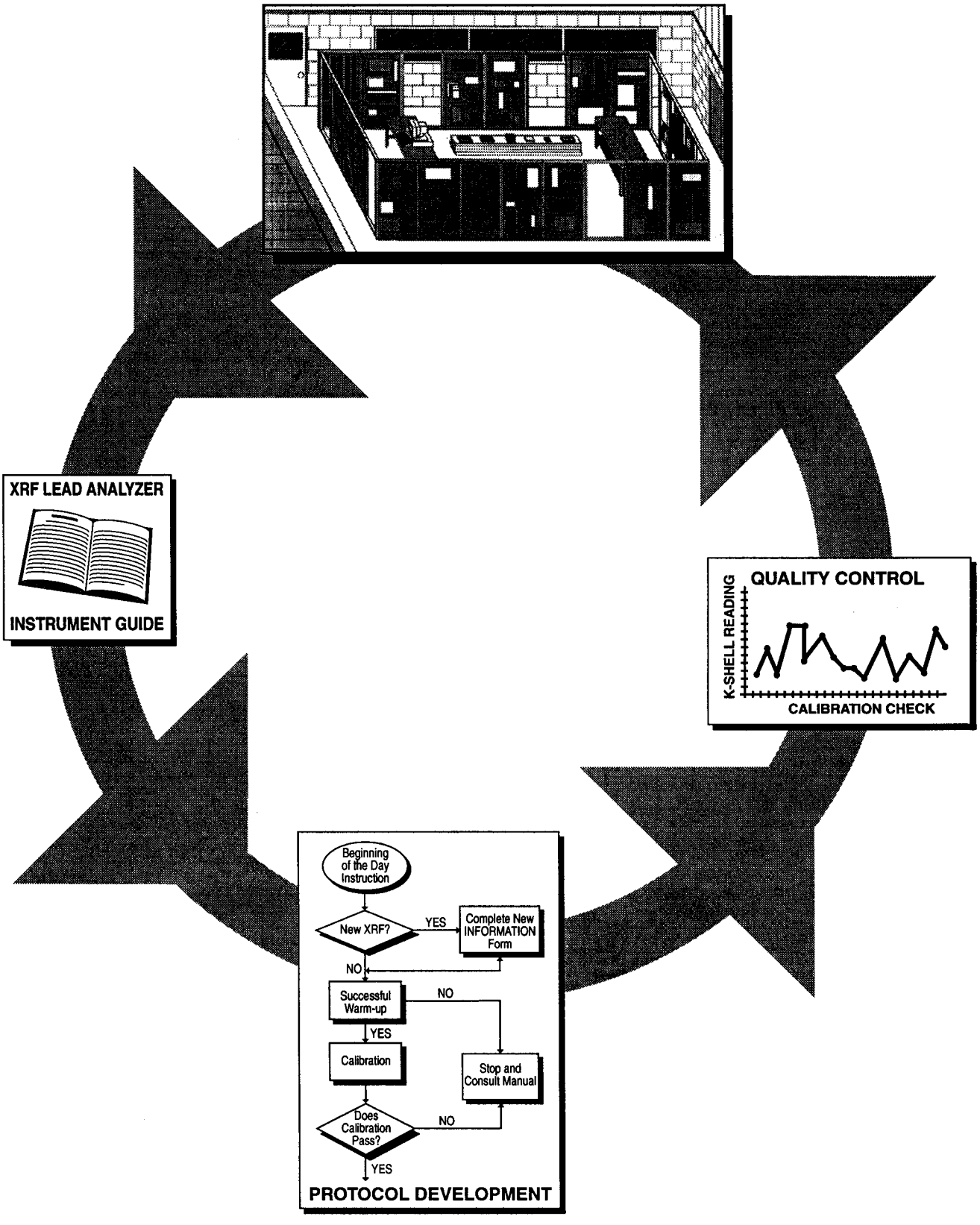
Archive Operations and Protocols



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Archive Operations and Protocols

Technical Branch
National Program Chemicals Division
Office of Pollution Prevention and Toxics
Office of Prevention, Pesticides, and Toxic Substances
U.S. Environmental Protection Agency
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CONTRIBUTING ORGANIZATIONS

The methodology described in this Operations Manual is part of a task funded by the U.S. Environmental Protection Agency (EPA) and the U.S. Department of Housing and Urban Development. The task was managed by EPA and was conducted collaboratively by three organizations under contract to EPA: Midwest Research Institute, Battelle Memorial Institute, and QuanTech. Each organization's responsibilities are listed below.

Battelle Memorial Institute

Battelle Memorial Institute (Battelle) was responsible for oversight of archive sample maintenance and archive testing.

Midwest Research Institute

Midwest Research Institute (MRI) was responsible for the development of this operations manual, sample maintenance, collection of paint samples, laboratory analysis, and supervision of testing.

QuanTech

QuanTech (formerly David C. Cox & Associates) was responsible for testing design, data management, development of statistical methods, and the writing of the methodology report and the associated XRF Performance Characteristic Sheets.

U.S. Environmental Protection Agency

The U.S. Environmental Protection Agency (EPA) co-funded the task, managed the task, reviewed task documents, and managed the peer review of this report. The EPA Project Leader was John Schwemberger. The EPA Work Assignment Managers were Sam Brown, John Scalera, and John Schwemberger. The EPA Project Officers were Sam Brown, Jill Hacker, Phil Robinson, and Sineta Wooten.

U.S. Department of Housing and Development

The Department of Housing and Urban Development (HUD) co-funded the task and was responsible for reviewing the *XRF Performance Characteristic Sheets* and for contacts with the manufacturers of lead-based paint testing technologies. The key HUD staff member was Bill Wisner.

ACKNOWLEDGMENTS

Special thanks are due to Mary McKnight of the National Institute of Standards and Technology (NIST) for operating XRF instruments during archive testing and for her advice and comments.

CONTENTS

1.0 Background

Archive operations, which this manual addresses, were preceded by the U.S. Environmental Protection Agency (EPA) and U.S. Department of Housing and Urban Development (HUD) field study of portable X-ray fluorescence (XRF) instruments and chemical test kits.[1,2] The results of the EPA field study are presented in the reports *A Field Test of Lead-Based Paint Testing Technologies: Technical Report*, (EPA 747-R-95-0026, May 1995) and *A Field Test of Lead-Based Paint Testing Technologies: Summary Report*, (EPA 747-R-95-002a, May 1995). The predecessors to this document are the Quality Assurance Project Plans for the pilot[3] and full[4] EPA/HUD Field Study. The results of the Field Study, which was conducted at various housing units throughout several U.S. cities, were used to develop a comprehensive performance database of commercially available XRF instruments and field test kits.

In conjunction with the Field Study, samples of painted housing components were collected for two purposes: first, to perform data verification and quality assurance after completion of the Field Study and, second, to have a way to evaluate a new XRF instrument or test kit that might enter the market after the end of the Field Study.

The samples collected from the Field Study have become an archive of materials that is being used as the basis for an interim testing program for new portable XRFs, while a protocol for laboratory testing of new portable XRFs is being developed by National Institute of Standards and Technology (NIST) for HUD. At this point, no destructive technologies are being tested at the archive in order to preserve the integrity of the samples. The archive facility is maintained under secure and controlled conditions to assure the integrity of the samples.

This document contains the operating procedures for the archive for testing that took place between December 1994 and September 1996. This document also contains protocols for testing and sample collection that were used during this period of time. This report is being released to provide documentation of these operating procedures and protocols for the time period from December 1994 to September 1996.

1.1 Purpose of the EPA/HUD Lead-Based Paint Archive

The EPA/HUD Lead-Based Paint Archive was developed to evaluate new portable XRF instruments and to produce instrument-specific information necessary for the instruments to be used to conduct lead-based paint inspections as specified in the HUD Guidelines. The instrument-specific information is embodied in the XRF Performance Characteristic Sheets (PCSs), which are an extension of Chapter 7 in the HUD Guidelines.

PCSs are XRF instrument model-specific documents intended to provide up-to-date performance information on XRF instruments. Data from the EPA/HUD Field Test of Lead-Based Paint Testing Technology study[1] have been used to develop the first set of PCSs. The EPA/HUD Lead-Based Paint Archive is a collection of real-world samples that

were assembled to test XRF instruments not available for evaluation during this study. Results from analysis of data collected from XRF testing at the archive site are used to produce PCSs for these instruments.

The archive is not currently being used to test chemical test kits for lead because of the destructive nature of the analysis. Portable XRF instruments are essentially non-destructive.

1.2 Purpose of the Operations Manual

The purpose of this Operations Manual is to document the general procedures used to maintain and operate the facility and describe the quality assurance and specific test procedures for testing the XRF instruments.

1.3 History of the Archive Components

The components in the EPA/HUD Lead-Based Paint Archive consist of sections of painted building components that are commonly encountered in residential housing, such as doors, walls, baseboards, gutters, and window frames. Samples in the archive represent a subset of the 1,290 samples tested in the above-referenced study. During this study, selected testing locations on architectural components were targeted for collection from housing units in three different cities: 10 single-family units in Denver, Colorado; 4 multifamily units in Louisville, Kentucky; and 8 multifamily units in Philadelphia, Pennsylvania. In addition, four additional samples were sent by the EPA's Office of Research and Development and are included in the archives. Some of the samples that were collected in Louisville were obtained after they had been removed from units in the development and placed in receptacles for removal; therefore, the specific housing units that these samples came from are unknown. Building materials that contained the selected measurement locations were removed and later assembled into the Lead-Based Paint Archive.

1.4 Quality of Data

As with any environmental measurement project, the quality of the data is important to assure that the data are appropriate for the intended use. To assure that the data collected in the archive testing are of sufficient quality, a Quality Assurance Project Plan (QAPjP) Addendum (Appendix A) was prepared. The QAPjP Addendum addresses data quality objectives for the archive testing, the experimental design, and the data collection and analysis procedures and protocols. The QAPjP ensures that the data collected are accurate and of appropriate quality for the intended use.

1.5 Report Organization

This report presents the historical background of the archive facility, a physical description of the facility, and general operating procedures relative to staff

responsibilities, sample management, testing procedures, reporting of data, and quality assurance/quality control. The report has an appendices section, which includes the QAPjP, historical XRF Testing Protocols, and Supplemental Protocols used at the archive.

1.6 Peer Reviews

This report was reviewed by five subject area experts prior to its publication. Comments on the document were largely editorial. In response, changes were made to clarify certain points and to make the report easier to read.

One reviewer commented on the relationship between this report and the EPA report *Methodology for XRF Performance Characteristic Sheets*. This report describes the operation of the archive facility in the period December 1994 to September 1996 and includes detailed protocols that were used for archive testing and sample collection. Data collected from the archive were used to develop XRF Performance Characteristic Sheets that have been released to the public through the National Lead Information Center Clearinghouse. (The phone number of the Clearinghouse is 1-800-424-LEAD.) The report *Methodology for XRF Performance Characteristic Sheets* describes the statistical methodology used to develop the information in the XRF Performance Characteristic Sheets. In effect, this report describes how data for the XRF Performance Characteristic Sheets were collected, and the report *Methodology for XRF Performance Characteristic Sheets* describes how those data were analyzed to produce the XRF Performance Characteristic Sheets.

Other reviewer comments concerned the representativeness of the archived samples and maintaining the integrity of samples over time. Most of the samples came from an EPA/HUD field study conducted in three separate cities. The selection of samples for the archives were selected on a random basis within specific substrate and lead concentration-range categories. The archived samples are regularly inspected and the storage conditions are controlled as described in this report to help extend the integrity of the samples for testing as long as possible.

Another recurring comment involved the purpose and intent of the appended protocols in this report. The XRF testing protocols represent a historical record of testing, and the supplemental protocols provide detailed instructions that were used for operation of the archive and for sample characterization purposes.

2.0 Description of Facility

Building No. 1, the facility housing the archive samples, is a 1,380 ft^2 concrete block building accommodating the design and layout used for archive materials.

2.1 Location

The building used for the EPA/HUD Lead-Based Paint Archive is located at the MRI Deramus Field Station in Grandview, Missouri. Deramus Field Station is a rural 78-acre site with 17,500 ft^2 of laboratory space in six special-purpose buildings.

2.2 Archive Design/Layout

The archive components consist of building materials, with the selected measurement locations indicated, vertically mounted into testing walls that are constructed from sheets of plywood bolted to a wood frame. The walls are arranged in a rectangular configuration approximately 32 feet long by 20 feet wide by 8 feet high.

The archive testing facility (see Figure 1) consists of 158 archive components. The components are mounted in a manner to minimize interferences during XRF testing. Mounting characteristics include the following:

- Separation of at least 4 feet between the testing wall containing the components and any objects behind the targeted area.
- Removal of plywood from the back side of most sample locations. For a few components, including all brick and concrete components, the components are mounted within a plywood box that is attached to the wall.
- Exclusion of materials used for mounting, with the exception of plywood or Styrofoam for a few components, from positions lying directly behind XRF test areas on the components.

In a few cases, the statistical test design includes XRF measurements on both the front- and backside of an individual component. The backside measurements are treated as separate and unique measurements and do not coincide with the front-side measurements of the archived sample during the XRF test sequence.

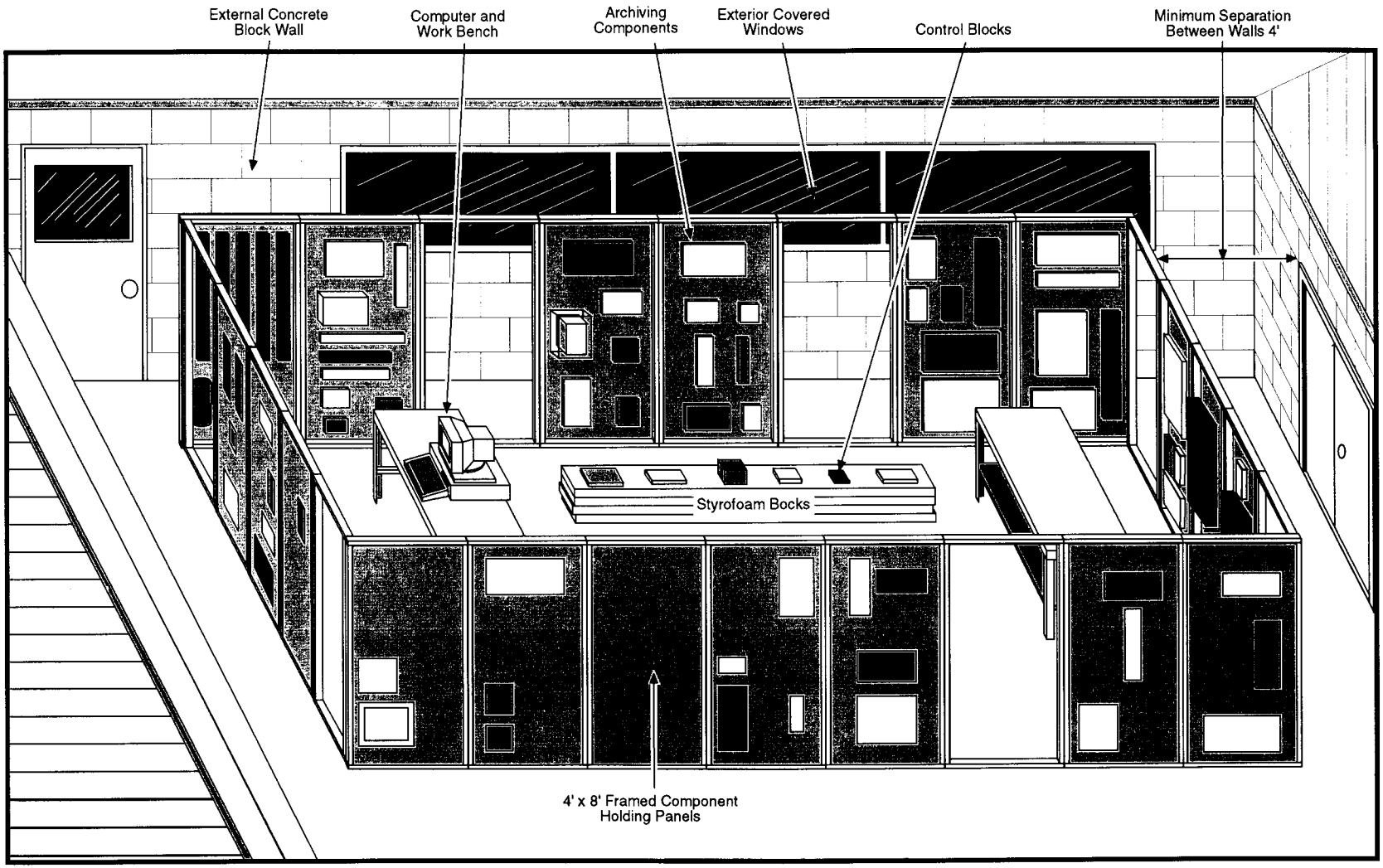


Figure 1. Archive Testing Facility

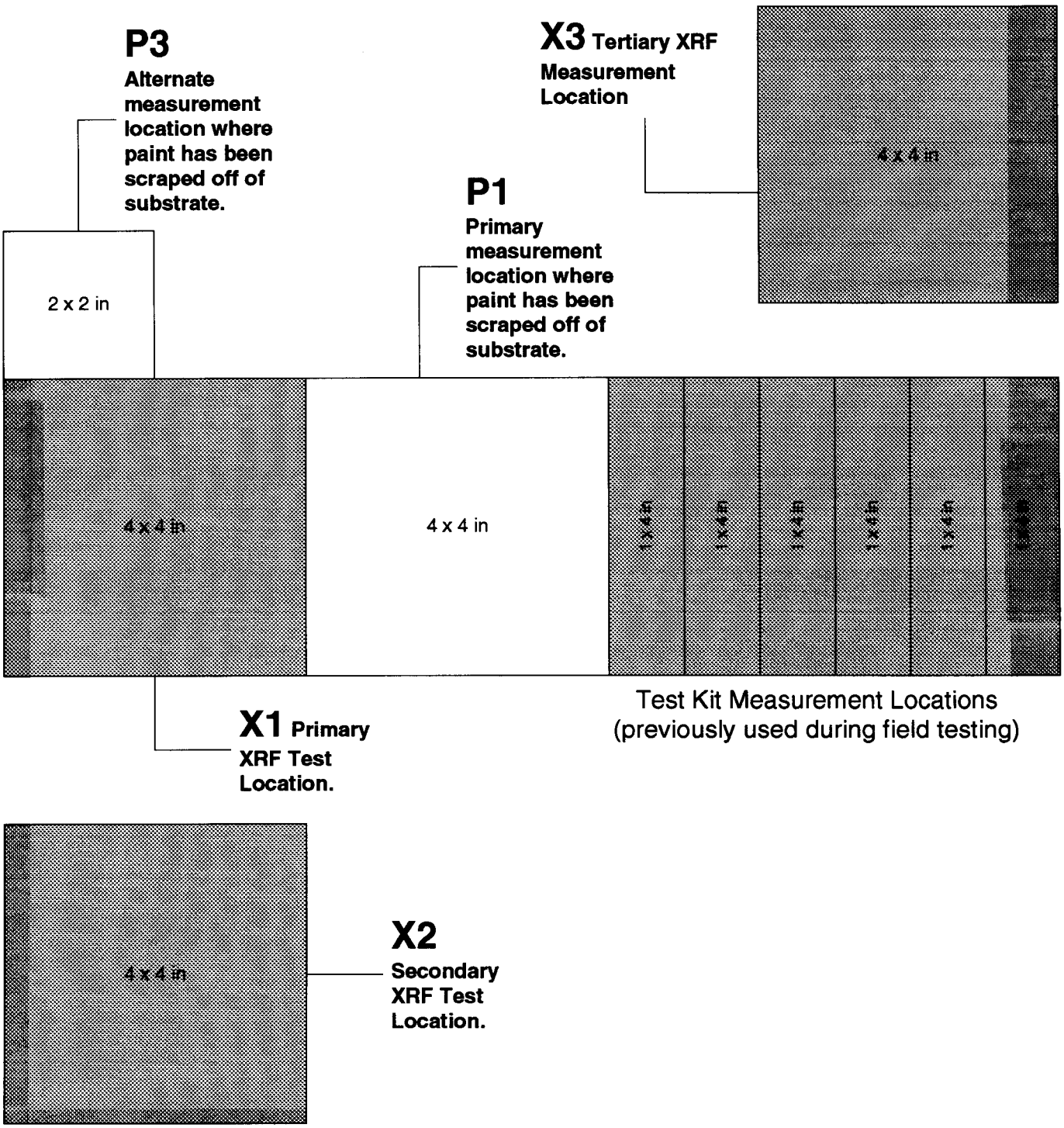


Figure 2. Example Template Used for Testing Archived Components

2.3 Security

The Deramus Field Station is within a limited access/entry area that is controlled by locked gates. Each of the laboratory buildings are under separate locks and can be accessed only by authorized MRI personnel. All of the keys for the locks at the field station are in the custody of MRI Security, which issues keys to personnel that are authorized access to specific buildings.

The archive samples are secured by double lock entry on the building. Access is controlled by keys issued by MRI. Only the MRI Program Manager, Task Leader, and QA Officer have been issued keys to the building. MRI Buildings and Grounds has a key to the building for access in cases of structural maintenance, emergencies, and general safety inspections. Subcontractors, visitors, or non-project personnel are not allowed access to this facility without being escorted by the MRI Program Manager, Task Leader, or QA Officer.

2.4 Component Description

The 158 archive components are normally tested once in a random order. XRF measurement locations have been marked on each archive sample using a dark colored marking pen. The markings are in the form of squares and rectangles labeled with the letters "**X**" or "**P**," followed immediately by the numbers "**1**", "**2**", or "**3**." An example of the template used for testing the components is shown in Figure 2. The template is modified for some samples due to space limitations, etc. **X1** is the primary painted XRF testing location, and **X2** and **X3** are secondary and tertiary painted testing locations, respectively. **P1** is a testing location on each component that has been scraped of its paint; the **P1** area is used in measurements involving "bare" or substrate-only testing. Some samples have **P2**, **P3**, or **P4** areas, which are secondary testing locations that have also been scraped of paint, but these secondary locations are not typically used during XRF testing.

XRF testing areas on samples mounted on the testing wall are identified according to the study protocols. Each testing location has an assigned unique number that is used for identification purposes. Lead levels have been determined from laboratory analysis of paint chip samples collected from at least two areas at each sampling location. Up to three separate painted XRF testing areas are identified on each sampling location using designations X1, X2, and X3 (as noted above). The testing walls contain 158 archive components from various locations and a variety of substrates. A summary of the type of component substrates is presented in the table below.

The development of the PCSs are based on XRF measurement data from the X1 primary painted XRF testing location and the P1 nonpainted substrate of each component. The secondary test locations on each component (e.g., X2, P2, etc.) were not used for developing the PCSs.

Summary of Samples in the EPA/HUD Lead-Based Paint Archive

Substrate Type	No. of Samples	Range of Lead Levels (mg/cm^2)
Wood	63	0.0003 - 30.11
Plaster	38	0.0024 - 16.07
Metal	38	0.0032 - 3.97
Drywall	14	0.0002 - 0.90
Brick	3	0.0035 - 17.50
Concrete	2	0.0009 - 0.10

2.5 Control Block Information

Control substrate blocks are part of the test design to record instrument variability relative to changes in substrates and sensitivity "drift" with time. The control blocks are a series of six quality control (QC) blocks consisting of different materials (metal, wood, brick, drywall, concrete, and plaster). Each of the QC blocks has been tested by chemical analysis (ICP-AES) of representative core samples to characterize the concentration of lead and potential interfering elements.

At the beginning and end of each day, a series of three quality control measurements will be performed on each of the six control blocks. The three QC measurements that will be taken on each control block are with the yellow NIST standard film (3.53 mg/cm^2) covering the QC block, followed by the red NIST standard film (1.02 mg/cm^2), followed by one reading taken without a NIST film. This set of measurements will be repeated at the end of the day.

Two sets of continuing QC measurements are performed after the standard readings are completed on every fifteenth archive component. The first set of continuing control readings is performed on the QC block that is composed of material similar to the last archive component tested. The second set of continuing control readings is performed on the QC block that is composed of material similar to the next archive component to be tested.

In a few cases, archive components were added to the archive measurement sequence and designated by the letter "A" followed by the number of the preceding component. In these cases, the inserted archive component was not counted as one of the 15 readings for the purpose of the continuing QC measurement in order to keep continuity with previous tests. At a later date, the entire archive component series was statistically re-randomized and renumbered so that all inserted components were fully integrated into the test sequence.

For the wood control block only, two additional consecutive readings are taken with the red NIST standard film (1.02 mg/cm^2) over the bare wood substrate during the beginning of day QC measurement series, continuing calibration QC checks during archive

testing, and at the end-of-the-day QC checks. The purpose of these replicate measurements on wood is to establish precision of the instrument throughout each testing period and for subsequent calculation of the calibration check values for the PCS documents.

3.0 Staff and Responsibilities

For this study, the staffing necessary to conduct the operations are the Program Manager, Testing Supervisor, Testing Monitor, and XRF Contractor/Operator.

The Program Manager will:

- Ensure that all necessary resources are available.
- Ensure that all personnel are informed of the project QA requirements.
- Ensure that all personnel conduct the work in a safe manner.
- Ensure that the project and financial status are reported to the OPPT Work Assignment Manager.

The Testing Supervisor will:

- Verify ID assignments on sample locations on the archive components.
- Assure that the data are collected as described in the protocol.
- Collect all forms and electronic data and make appropriate distributions to MRI, NIST, and QuanTech.
- Assure that resources are available to achieve planned testing.
- Oversee testing activities.
- Provide beginning-of-day and end-of-day instructions.
- Make primary decisions regarding any testing difficulties that may arise.
- Complete a new set of SAMPLE LOCATION CONDITION forms for:
    - Locations on the components previously identified as being in poor condition, which already have completed records from the spring 1995 testing.
    - Any additional sample locations that are observed to be in poor condition.
- Provide the following forms:
    - Archive XRF Information
    - Source Age Adjustment Table
    - Testing Order List
    - Control Readings
    - Standard Readings
    - Sample Location Condition
- Take radiation emission measurements on the XRF instrument.
- Assure that everyone who enters the facility during XRF testing is wearing a dosimeter badge and collect the badges at the end of each day.
- Arrange for testing of the dosimeter badges at the appropriate time interval.

The Testing Monitor will:

- Record all relevant data on the appropriate data forms or field notebooks.
- Work with the XRF Contractor/Operator to help assure the protocols are followed.
- Work with the Testing Supervisor to help assure that protocols are followed and any deviations are properly documented.

The XRF Contractor/Operator will:

- Handle the instruments and make the measurements.
- Work with the Testing Monitor to see that the appropriate data are recorded.
- Work with the Testing Supervisor to help assure that protocols are followed and any deviations are properly documented.
- Download data to a computer and verify that the transfer was successful.
- Report to the Testing Supervisor any indications of deteriorating samples observed during testing.

4.0 Archive Sample Management

The integrity of the components are ensured by maintaining the archived samples within controlled environment and by protecting the samples against damage from the XRF instruments during testing.

4.1 Maintaining Archive Samples

The archive facility at MRI's Deramus Field Station has been set up to provide an environment that will help preserve the components as long as possible to assure the collection of comparable data. To ensure this, the building is temperature controlled throughout the year.

The conditions of the archive components are monitored during and after each XRF activity to assure that no damage has been caused by the instrument. If any damage is observed either to the component or the painted surface, the damage will be documented on the sample condition records and reported to the Program Manager and the EPA Work Assignment Manager. A joint decision will be made as to the corrective action to be taken. The repairs to the archive samples are generally performed by the Testing Supervisor.

4.2 Securing Archive Samples

The building itself is located on MRI property, which is fenced and posted as restricted access. The front gate is locked and restricted to MRI personnel only. This MRI property also has a custodian living on-site for security and maintenance purposes. The Archive facility is equipped with its own phone line with direct access to MRI Security and Safety officers.

4.3 Managing Testing with Archive Samples

All testing activities on the archived samples are carried out using approved protocols under supervision of the MRI Testing Supervisor. Normally, the Project Task Leader performs these duties. The specific responsibilities of the Testing Supervisor was described in Section 3 of this document and these responsibilities are carried out each time the archives are used for testing.

XRF testing using subcontracted, independent operators is performed in compliance with the appropriate protocol. Subcontractors are restricted to the operation of the instruments only and are not involved with technical or procedural decisions during the tests.

5.0 Facility Operations

All testing of the archives must be done using pre-approved protocols specific for the XRF instrumentation being used. The protocol provides the calibration requirements (usually cited from the manufacturer's manual), the number and sequence of QC samples, and the sequence for the archive components. In addition, the specific protocols specify the length of timed readings, if appropriate, or the test mode (which usually varies in time based on the lead concentration of the paint). The testing for most XRF operations should be accomplished in two to two-and-a-half days. The measurements taken for XRF testing on the archive components are:

- Initial (beginning of the day) control measurements
- Standard measurements
- Continuing control measurements
- Ending (end of the day) control measurements

5.1 Order of Testing

Randomization of the testing order was assured by the following steps in the design of the Archive test facility. First, the placement and order of the components on the boards was assigned in a randomized order. Second, the sequential order of testing was also randomized to make the XRF operators walk and move about the facility, as they would during a lead-based paint inspection.

Four additional plaster samples were added to the sample test series after the archive facility was built. The samples were physically added to the facility by mounting all four on a separate board. In order to maintain the randomness of the sequential testing order, the four additional samples were statistically "remixed" with the pool of samples and assigned random numbers in the test series. The samples were inserted into the original sequential order of testing by adding the letter "A" after the component number it followed; for example, if one of the added samples came after 100, it would be given the identification of 100A and the test sequence would become: 99, 100, 100A, 101, 102, etc.

Continuing calibration checks on the XRF instruments were performed at the normal 15 sample intervals, except when the added plaster samples were encountered. For consistency, the "A" samples did not count as one of the 15 samples separating the continuing calibration checks; this allowed the QC checks to be performed after the same samples as before the 4 plaster samples were added. At a later date, the entire archive component series was statistically rerandomized and renumbered so that all inserted components were fully integrated into the test sequence.

When it is necessary to change the Archive Testing Order, the procedure starting on page C-39 of the Supplemental Protocols is used to renumber the samples.

5.2 Personnel and Radiation Safety

Although safety and operational instructions are the responsibility of the study Supervisor, safety is the collective responsibility of everyone and requires the unqualified support and cooperation of each person involved with the archive operations. In conducting tests at the MRI archives, each person must be aware and participate in general safe work and radiation safety practices. Since many accidents are the result of indifference, failure to use common sense, or failure to follow instructions, those involved in the study must always be aware of what coworkers are doing in the immediate area, especially with the XRF instrument.

Since the effects of radiation exposure are not immediately apparent, all XRF instrumentation will be monitored during use with a survey meter (Geiger-Mueller counter) equipped with a beta-gamma detector. Any XRF instrument having counts (mR/hr) above the manufacturer radiation safety specifications will not be tested. In addition to the monitoring of the instruments, each person will have and wear, during the archive testing process, a personal film dosimeter to measure and evaluate his or her exposure.

5.3 Testing Protocols

Testing protocols (Appendix B) for each XRF instrument under evaluation are developed from manufacturer-supplied documents, such as the manufacturers' instrument operating manuals. Efforts are exerted to perform testing in a manner consistent with manufacturer guidance subject to the restriction that no destructive testing can be permitted on samples within the archive. Sampling locations are tested using extensive before, during, and after QC checks on NIST Standard Reference Material paint films (SRM 2579) placed over blocks of representative substrates (control blocks). Testing at each sampling location includes measurements on each painted area (X1, X2, and X3) and measurements on a bare substrate area both with and without being covered by the 1.02 mg/cm^2 NIST SRM.

Other testing protocols developed for this project that are not a part of XRF testing but are part of the overall project requirements are found in Appendix C.

5.4 XRF Contractor/Operator Responsibilities and Orientation

The XRF Contractor/Operator is responsible for his or her training on the equipment being tested. The contracted instrument operators are required to have a backup instrument on-site so that testing could be continued with the backup instrument if necessary. Both the primary and backup instrument should preferably have sources no more than 6 months old. At the end of the test day, all test data are downloaded to the facility computer and backup diskettes; then the electronic data files are purged from the operator's instrument before the instrument is taken off-site. The XRF Contractor/Operator also is responsible for following all federal and state licensing requirements in handling and using radioactive material. The contractor/operator is responsible for self monitoring of radiation exposure using an appropriate film dosimeter.

If the full test round cannot be completed using the primary instrument, the backup instrument may be used to continue the testing only after demonstrating that manufacturer and protocol calibration/QC checks are acceptable on the backup instrument. If both the primary and backup instruments fail before completing the full test series, the testing is stopped, all causes and actions are documented, and all recoverable data are collected as normal. Testing does not resume until instruments that can pass all applicable warm-up, calibration, and quality control checks are available.

5.5 Archive Testing

The general procedure for performing the test is given below.

1. Receive beginning-of-day instructions from Testing Supervisor.

2. Perform manufacturer's initial calibration checks. Perform additional manufacturer's calibration checks at intervals as required by the manufacturer's specifications.

3. Perform initial control block measurements on all six blocks. Record temperature and humidity data.

4. Perform standard set measurements (15 archive components).

5. After the completion of the first 15 archive components, and following every 15 archive components thereafter, perform continuing control readings on the two control blocks with the same substrates as the last and next substrates tested. Intermittent delays (such as lunch breaks, etc.) are taken at the continuing calibration check points.

6. Record temperature and humidity data approximately halfway through the day.

7. At various times during the day, record in the field notebook the time required by the XRF instrument to actually display the lead measurement result. The data recorded during the day for this purpose should include the start and end times for 20 measurements. The start time is the time of day when the XRF probe face-plate is placed on the surface to be tested and testing commences. The end time is the time of day when the result is displayed on the XRF readout area.

8. At the end of the first day of testing, perform ending control block measurements on all six blocks. Record temperature and humidity data.

9. Perform end-of-day activities, which include: performing final manufacturer-specified calibration checks, reviewing data forms for completeness, downloading electronically stored data to the on-site computer, and ensuring that all electronically stored data is erased from the XRF instrument.

6.0 Statistical Analysis of Data

Data collected from the archive are analyzed using statistical methods similar to those that were developed in the above-referenced study. A model is used to estimate the bias and precision of an XRF instrument as a function of the amount of lead present in paint. Estimates are also derived for substrate-corrected XRF results, in cases where such correction is demonstrated to improve performance. Inconclusive ranges and thresholds for the XRF readings are derived using bias and precision estimates.

Detail on the methodology for developing the XRF Performance Characteristic Sheets is available in the report *Methodology for XRF Performance Characteristic Sheets*.[5]

7.0 Deliverables and Reports

Deliverables and reports for this project include:

- Testing Protocols
- Revisions to the XRF Operations Manual
- Revisions to the Quality Assurance Project Plan
- XRF Data Reports
- Radiation Monitoring Report
- Component Monitoring Report
- Component Repair Report
- Performance Characteristic Sheets

8.0 Audits

Quality assurance activities associated with the Archive Operations include audits performed on work in progress and on completed XRF test data sets. Because the quality and completeness of the data set are vital to accurately evaluate the individual XRF instruments, QA audits are focused on data collection, transfer, and verification.

Prior to XRF testing, protocols and data forms are reviewed by QA staff to assure that the data set will include all available data produced by a particular instrument. For example, the forms are "customized" to assure that all appropriate modes of operation, time displays, precision indicators, and other readouts are included in the data recording process.

During the XRF testing, instrument displays are randomly checked by visual verification (in addition to voice communication between the instrument operator and data recorder) by either the recorder himself, the supervisor, or the QA officer. These verification checks are noted on the forms. For any situation in which the monitor's reading differs from the operator's reading, the testing will be temporarily stopped until the cause of the discrepancy is determined and the actions taken will be noted on the data collection forms.

Throughout the test day and after each day's recordings are complete, the data forms are checked for completeness and clarity by either the supervisor or the QA officer. The forms are checked for proper page numbering, clarity of notes, appropriate strike-outs of nonusable data, signatures, dates, and instrument identifications.

After electronic data transfers from the instrument to the facility computer and backup diskettes, the electronic files are audited by verifying correct dates and file names, checking consistency of byte numbers between original and copied files, and inspecting the data files to see if beginning and ending data (sequence numbers, lead concentrations, etc.) are consistent with the recorded forms.

Care is taken to ensure adequate backups of the data exist in case of accidental loss. Originals of the completed data forms are photocopied and electronic files are copied onto the facility computer, then backed up on two sets of diskettes. Exact copies or the originals are provided for the statistical analysis study, and the originals are retained or transferred back to the project files for archiving.

QA audits during statistical analysis are conducted to assure the accuracy of derived results. These audits include double-entry checks during transfer of the recorded data into the database files and cross-checks between the hand-recorded (primary) data set and the electronically captured (secondary) instrument data files.

9.0 References

1. *A Field Test of Lead-Based Paint Testing Technologies: Technical Report*, EPA 747-R-95-002b, May 1995.

2. *A Field Test of Lead-Based Paint Testing Technologies: Summary Report*, EPA 747-R-95-002a, May 1995.

3. MRI Report, *Quality Assurance Project Plan for the Pilot Study: Comprehensive Field Study of XRFs, Lead Paint Test Kits, and Laboratory Analyzed Paint Chip Samples*, EPA Contract No. 68-D0-0137, MRI Project 9801-A-57, March 15, 1993.

4. *Quality Assurance Project Plan for Comparative Field Study of Methodologies Used to Detect Lead in Paint*, dated November 30, 1993.

5. *Methodology for XRF Performance Characteristic Sheets*, EPA 747-R-95-008, September 1997.

Appendix A

QAPjP Addendum

Contents

QUALITY ASSURANCE PROJECT PLAN (QAPjP) ADDENDUM FOR COMPARATIVE FIELD STUDY OF METHODOLOGIES USED TO DETECT LEAD IN PAINT

Work Assignment No. 5-24
Contract No. 68-DO-0137

Work Assignment No. 3-02
Contract No. 68-D3-0004

This document is an addendum to the QAPjP of July 8, 1993, titled "Quality Assurance Project Plan for Comparative Field Study of Methodologies Used to Detect Lead in Paint."

1.0 SUMMARY

In 1993, the Environmental Protection Agency (EPA) initiated a field evaluation of XRF instruments and lead paint test kits. These two technologies were tested on samples of painted components in vacant housing units. As stated in the QAPjP (section 3.3.3), samples from certain (specific) locations were collected and archived for future use.

Since there are XRF instrument manufacturers whose instruments were not in the study, these manufacturers are requesting an evaluation of their products. The Department of Housing and Urban Development (HUD) has been the recipient of these requests, and HUD has asked EPA to provide a limited evaluation based on the archive materials from the field evaluation. In addition, two questions that have arisen from the study results should be resolved. One of these involves spatial variation in painted building components and the other involves the order of substrates when taking XRF measurements.

EPA proposes to initiate its testing of the new instruments and other XRF instruments in January 1995. The funding will be derived from money earmarked for the completion of the field evaluation.

2.0 DESIGN

2.1 GENERAL OBJECTIVES AND DESIGN

Approximately 150 samples of painted housing components are available for testing of new instruments. The range of lead levels in these samples is approximately as follows: 50% less than 0.4 mg/cm^2, 25% between 0.4 and 1.7 mg/cm^2, and 25% greater than 1.7 mg/cm^2. Most of the samples consist of metal, plaster, wood, and drywall. Brick and concrete substrates have only token representation due to physical limitations in collecting samples of such substrates.

The first and primary objective is to provide a limited evaluation of new XRF instruments that were not in the full study and to report the results to HUD.

The second objective is to deal with spatial variability in the paint samples. This will be done by taking a second sample for ICP analysis so as to better characterize the lead in the paint in the XRF test area, by using this second sample to validate interpolation models,

and by taking XRF readings at additional locations on the archived material to determine the impact of additional areas on XRF measurement.

The third and last objective is to determine if the order of substrate materials has an effect on XRF readings.

Methods and approaches will be the same as in the field evaluation study, except where results of the field study indicated a change was desirable.

For the XRF instruments in this study addendum, a source that is no more than 6 months old is required, as was the case in the full field study. Testing will be done by personnel from XRF testing firms under subcontract to MRI or by personnel from the National Institute of Standards and Technology (NIST). New instruments will be included that are being sold and delivered to testers in this addendum study. The new instruments that are expected to be included in this study are: the revised XL by Niton Corporation, the SEFA manufactured by HNU Corporation, the LPA-1 manufactured by Radiation Monitoring Devices, and the LeadStar manufactured by Xsirius Corporation. In general, a reading with one of these instruments will be nominally 15 seconds long, but manufacturer instructions will be examined to determine each instrument's standard reading mode, and a reading will be redefined to fit the manufacturer's standard reading mode unless this is not practical.

The other instrument to be used in this addendum to the study is the Lead Analyzer. The Lead Analyzer will be used to determine the impact of laboratory-setting versus field measurements, providing a common element between the field and archive phases of the study, and to pilot test the archive layout. The ML-1, the XK-3, or the MAP-3 will be considered for use if a suitable Lead Analyzer is not available for this study.

For each of the new instruments, two instruments will be used: one by a testing firm under subcontract to MRI and one owned and operated by NIST. For the Lead Analyzer (or the ML-1, the XK-3, or the MAP-3), a single instrument will be used, either from a testing firm under subcontract to MRI or from NIST. However, each subcontractor will be requested to bring a back-up instrument in case the primary instrument fails to operate. NIST will be requested to ensure that their instruments are operating properly before traveling to the testing site.

2.2 STUDY OBJECTIVES

The study objectives for the first objective are:

1) Estimate the bias of the new XRF instruments at 0 and 1 milligrams per square centimeter to within plus or minus 0.2 with 95% confidence, overall and to within plus or minus 0.4 with 95% confidence on wood, metal, and plaster.

2) Estimate the precision of the new XRF instruments at 0 and 1.0 milligrams per square centimeter to within plus or minus 3 times what was in the full study (with 95% confidence), overall and on wood, metal, and plaster.

3) Develop an operating characteristic curve over all substrates and on wood, metal, and plaster for each new XRF. Estimate the threshold probability and 50% point with precision no more than three times the standard errors of similar parameter estimates in the full study. [Note: The 50% point is defined as the lead level at which the probability of receiving a positive indication for lead is 50%.]

4) Estimate the inconclusive region for each XRF using order statistics, overall and on wood, metal, and plaster. Compute misclassification rates and inconclusive rates overall and for wood, metal, and plaster substrates.

5) Estimate the bias and precision of each new XRF on the NIST SRMs and develop control limits for usage of these instruments in the field.

6) Estimate time for a nominal reading using the standard operating protocol for each new instrument.

7) Determine the influence of paint thickness on XRF measurements.

8) Assess the performance of auxiliary indicators of XRF performance, such as the "absorption index" of the Niton XL.

The study objectives for the second objective are:

1) Estimate the lead in the paint in the primary XRF testing area using an average of samples near the primary XRF testing area.

2) Investigate interpolation models for spatial variation in paint using paint chip samples at varying distances.

3) Characterize differences between XRF testing at one area versus XRF testing at three areas on a sample.

The study objective for the third objective is to determine if substrate order has an impact on XRF readings.

2.3 DATA QUALITY OBJECTIVES

[NOTE: The ICP data refers to the chemical analyses performed in the characterization and lead-level determination of the archived test samples.]

For the ICP data generated, the data quality objectives (DQOs) for the archive phase are the same as for the full study, as stated in Chapter 4 of the "Quality Assurance Plan for Comparative Study of Methodologies Used to Detect Lead in Paint" (July 8, 1993).

For the archive phase, NIST Powdered Paint SRM 2582, if available, will be used in each batch in addition to the NIST SRM 1579 used in the full study (1 per batch) and the AIHA materials. The recovery accuracy for reference materials will remain $\pm$ 25 percent. Based on the full study data, it is anticipated laboratory split subsamples will demonstrate a standard deviation of less than 15 percent.

The DQOs for representativeness, comparability, and completeness are relevant to sampling activities and the layout of the archive testing room.

Efforts to obtain representativeness include the use of the exact same testing locations identified in the field for the archive samples that were also tested in the full study. These test locations were distinctly marked with a template as to where the XRF was to be positioned for testing. Samples selected to be archived were taken to obtain desired levels of lead over all substrates; however, because of the difficulty and resulting damage associated with the removal of certain substrates, the archive consists of a limited number of samples, such as brick and concrete.

In an effort to demonstrate correlation of the field study with the archive phase, initial instrument testing of the archive will include, if possible, one of the exact same instruments used in the field, a model of the Lead Analyzer manufactured by TN Technologies. An effort is also being made to obtain the same Lead Analyzer operator as was contracted for in the full study.

All of the paint chip sample data generated from the archive phase will be provided in mass/mass as well as mass per area units for direct comparison to the full study data. All XRF readings will be reported in mass per area units unless another type of unit is peculiar

to an XRF. Where applicable, both L-shell and K-shell readings will be recorded. Important auxiliary variables, such as the "absorption index" of the Niton XL that reflect instrument performance will also be recorded.

As to completeness, every XRF instrument being evaluated must generate measurements for each primary XRF test location. The total number of primary testing locations is anticipated to be approximately 150.

As a part of the evaluation, 100% of the archive test samples must be tested by XRFs on the bare substrate without the NIST Red Film (1.02 mg/cm^2). The goal for measurement of bare substrate with the NIST Red Film is also 100%.

Micrometer readings to determine the thickness of paint will be taken whenever possible by measuring a paint chip.

3.0 SAMPLE COLLECTION

Sample collection will be discussed in chronological order.

The first step will be the marking of sample locations. There are three types of samples in the archive: those marked with the full study template, those marked with the pilot template, and those marked by a partial template from the full study.

For samples marked by the full study template (completely or partially), the second paint chip sample will be located adjacent to the left side of the template or the top of the template, just outside the XRF testing area. A measured area will be marked off, using lead-free markers. Whenever possible, the measured area will be 2" by 2". The measured area will be placed catty-corner to the primary paint chip sample, if possible. The distance from the center of the second paint chip sample to the center of the primary paint chip sample will be approximately 6" or 8". Records will be kept to determine which samples have 6" samples and which have 8" samples. If circumstances dictate a different distance, records will be kept to reflect that.

For samples marked by the pilot template, there were two paint chip samples taken inside the template. In this case the centroid of the two initial samples will be computed. An additional area, 2" by 2", for paint chip collection will be marked off near the primary XRF testing area and approximately catty-corner to the centroid, but not in an area where duct tape was previously applied. Records will be kept so the location of the three areas for paint chip collection are known and distances between them can be computed.

Two additional XRF testing locations will be marked on as many samples as possible. Their centers will be located and marked at distances which are as far apart as possible, under the constraint that newly marked locations will be on a line with the original XRF testing area. Records will be kept of the coordinates.

After all marking has been completed, primary paint chip XRF test components will be collected from samples for which there is no current primary paint chip sample. Bare substrate areas for XRF testing will also be cleared for these samples. [NOTE: These bare areas will be provided for these samples by scraping the paint from the substrate.] Extra paint chips from the bare areas will be stored in labeled containers and saved for possible future use. The paint chips will be stored in their recovered state, with no homogenization, and labeled as to where collection took place by sample number and location. Records will be kept of the size of the area from which the paint chips were collected. Collection of secondary paint chip samples will take place after XRF testing is completed if there is a concern over loss of samples. [NOTE: The concern is loss or deterioration of lead-based paint from the sample surface.] If there is no such concern and if adherence to schedule is important, secondary paint chip samples can be collected when primary paint chip samples are collected.

The next step will be collection of XRF data.

QuanTech will develop a standard order of testing samples. The order will be random and designed to force the operator to walk around the room during the course of testing. The testing order will *not* be identical to the order the samples are mounted in the facility.

The standard collection procedure for the Lead Analyzer and the new instruments is as follows.

Follow the manufacturer's procedures for start-up and quality control. Start each day's testing by testing the NIST films on the control blocks, using the same procedures as used in the full study, with the exception that only a single reading is to be made with the 3.52 mg/cm^2 NIST SRM (yellow) and on a bare block. After completion of control block testing, start testing the archive samples. Follow the order of testing samples developed by QuanTech. At each sample, take three readings on the primary XRF paint area. Then take three readings with the NIST 1.02 mg/cm^2 red standard over the bare area. Next take one reading over the bare area (no NIST standard). Finally take a single reading at each of the two additional XRF areas marked as associated with that sample. This completes the testing at the sample. Move on to the next sample and continue to test until all sample locations in the archive have been completed.

Take a continuing control reading every 15 samples. Take a standard set of control readings on the last substrate tested, and then a standard set of control readings on the next substrate to be tested. Use procedures followed in the full study for the continuing control block readings, with the exception that only a single reading is to be taken on the yellow NIST SRM and a bare block.

After completion of all the samples in the archive, test the "comparison set" by taking a single nominal 15 second reading on each sample in the comparison set in the order specified. The "comparison set" will be selected by QuanTech to test the effect of substrate changes on XRF readings. Since the standard order will be random, the "comparison set" order will be grouping by substrate, with a suitable ordering within substrate groups. Comparisons will be made between the readings on the standard order set and the "comparison set" to determine the effect of taking readings on randomly ordered substrates and grouped substrates. Do not take continuing control readings during "comparison set" testing unless specified in the QuanTech design.

At the end of each day's testing, take readings on the control blocks as was done in the full study, with the exception that only a single reading is to be taken on the yellow NIST SRM and a bare block.

See Attachment I to this addendum for an outline of the XRF testing plan that is described above.

The Lead Analyzer would ideally be used first to test out the archive in early January 1995. The Lead Analyzer will go through the standard testing procedure described above. The new instruments would ideally be tested next and would go through the same standard testing procedure. One of the three older instruments would be tested if a Lead Analyzer could not be obtained in a timely fashion.

After all XRF testing is completed, secondary paint chip samples will be collected if not already collected. Micrometer readings to determine thickness of paint will be collected where possible.

4.0 ANALYSES AND MEASUREMENTS

Analyses and measurements will be conducted using the methods and approaches in Chapter 4 of the QAPjP.

NIST paint SRM 2582, if available, will be included as a QC sample in the batches for laboratory analysis.

5.0 DATA PROCESSING AND STATISTICAL ANALYSIS PROCEDURES

All XRF operators will be assigned a monitor who will record all data on forms. Data will be recorded on forms developed by MRI in consultation with QuanTech and EPA. Forms from the full study will be used where possible. Attention will be paid to recording the correct serial number and other identifying numbers so as to be able to identify the instrument that was tested in the archive phase.

A set of identification numbers that uniquely identify each XRF reading will be developed. These identification numbers will be recorded by the monitors on the forms to accurately identify each XRF reading. The XRF operators of instruments that have an electronic data capture capability will be given instructions in the insertion of identification codes. These codes will be designed to provide enough information to ease matching between data on forms and electronically captured data without impending XRF testing.

All data will be double key entered as the primary quality assurance step in data management. The data forms from the monitors will be the primary source of XRF data. However, comparisons will be made to electronic data from the XRFs where possible. Obvious data collection errors will be corrected. To the extent possible, examples of listings from the instruments will be obtained prior to their use in the archive to aid planning.

For the first study objective, statistical approaches that were used in the full study to estimate bias and precision of XRF instruments, to estimate operating characteristic curves and their key parameters, and to estimate bias, precision, and control limits for the NIST SRMs will be used for the archive testing. Inconclusive regions will be estimated by order statistics. Misclassification and inconclusive rates will be calculated. Average time of a nominal reading will be computed, as well as other descriptive statistics of reading time. The results of the thickness of paint [NOTE: the thickness values] from the micrometer readings will be brought into the regression models as a covariate, and the significance of the covariate will be estimated. Paint thickness variability in the samples will be assessed using methods developed in the full study. Auxiliary variables will be examined by scatterplots, correlation estimates, and, where applicable, regression approaches.

For the second study objective, averages of paint chip sample results will be computed to serve as a more accurate baseline for testing on a sample. Ratios and differences of secondary to primary paint chip sample results will be examined and characterized by distributional approaches to determine if there are any unusual pairs of results. Paint chip sample results from different distances will be compared, and interpolation approaches will be examined in light of the new data. XRF results from testing a single area will be

compared to XRF results from testing three areas by using approaches developed in the full study for analyzing results from laboratory and field duplicates.

For the third objective, comparisons between readings from the standard order and the "comparison set" will be made by scatterplots, control chart plots, non-parametric tests, and (paired) t-tests.

6.0 AUDITS

MRI's QA unit will audit all data that is sent to QuanTech following practices that were used in the full study.

QuanTech will identify sources of error in data and estimate the error rate from each source using methods that were used in the full study. This includes reconciliation of handwritten forms and electronically captured data.

EPA will audit the archive facility, the data at MRI and QuanTech, and the statistical software at QuanTech.

7.0 REPORTS

MRI will report data from laboratory batches following practices used in the full study. MRI will report XRF data on data collection forms to QuanTech and, if requested, to EPA no later than six working days after collection of the data. Where applicable, MRI will furnish to QuanTech data disks with electronically captured data from the XRF testing.

A standard analysis procedure will be set up. The archive data from the Lead Analyzer will be used to test out the standard analysis procedure.

QuanTech will submit a report on the analysis of the data and an XRF Performance Characteristic Sheet no later than four weeks from the receipt of the data for a particular instrument, given that the laboratory analysis has been completed. When data from a second instrument from the same manufacturer is received, the analysis and XRF Performance Characteristic Sheet will be updated.

8.0 REFERENCES

Quality Assurance Project Plan for the Comparative Field Study of Methodologies Used to Detect Lead in Paint, July 8, 1994, Midwest Research Institute, EPA Contract Number 68-DO-0137.

9.0 HEALTH AND SAFETY

Health and safety steps in Chapter 9 of the QAPjP of July 8, 1993, will be followed.

Only one XRF instrument at a time will be allowed in the archive testing facility.

Manufacturers will be required to have proper licensing of instruments completed before testing is carried out. Operators of the instruments must have completed appropriate safety training from the manufacturer.

QAPJP ADDENDUM ATTACHMENT I
XRF Testing Plan as Proposed in QAPjP Addendum Revision Number 1
Dated 12/14/1994

Note: A "15-sec reading" refers to a single reading from an XRF instrument following the manufacturer's standard protocol. Based on past experience, such a reading for new instruments is expected to be, on average, about 15 seconds in length with a fresh source.

I. Beginning of Day Control Block Testing

1. On each of the six substrate control blocks, using order of substrates from the full study (metal, wood, brick, drywall, concrete, and plaster):

    A. One 15-sec reading with Yellow NIST Film overlay;

    B. Three 15-sec readings using Red NIST Film overlay;

    C. One 15-sec reading on bare substrate control block.

II. XRF Readings Per Sample Location

1. Three 15-sec readings on X1;

2. Bare Substrate Area:

    A. Three 15-sec Using Red NIST Film overlay;

    B. One 15-sec on Bare Substrate Area;

3. One 15-sec reading on X2;

4. One 15-sec reading on X3;

III. Continuing Control Block Check

1. Every 15 Sample Locations

    A. On Substrate Control Block Type Reflective of Most Recent Location Tested:

        a. One 15-sec using Yellow NIST Film overlay;

b. Three 15-sec using Red NIST Film overlay;

c. One 15-sec on bare substrate control block.

B. On Substrate Control Block Type Reflective of Next Location to be Tested:

a. One 15-sec using Yellow NIST Film overlay;

b. Three 15-sec using Red NIST Film overlay;

c. One 15-sec on bare substrate control block.

NOTE: B is optional in situations where there is no change in substrate at the continuing control block check.

IV. Comparison Set (30 Test Locations Grouped By Substrate)

1. Take one 15-sec reading for:

A. X1

V. End of Day Control Block Testing

1. On each of the 6 substrate control blocks, using the order from the full study (metal, wood, brick, drywall, concrete, and plaster):

A. One 15-sec reading with Yellow NIST Film overlay;

B. Three 15-sec readings using Red NIST Film overlay;

C. One 15-sec reading on bare substrate control block.

VI. XRF data base download as necessary and hard copy review.

QAPjP CLARIFICATIONS AND ADDITIONS

Section 2.1 Definition of ICP Analysis

"ICP Analysis" refers to inductively coupled plasma atomic emission spectroscopy (ICP-AES) analysis for lead. For archive paint samples, a method similar to the EPA SW-846 Method 6010 was used. The method for the archive paint samples is described in detail in Appendix G of the report "A Field Test of Lead-Based Paint Testing Technologies: Technical Report (EPA 747-R-95-002b)."

Section 3.0 Testing Order Used After Addition of Four Additional Components to the Archives

Four additional plaster samples were added to the sample test series after the archive facility was built. The samples were physically added to the facility by mounting all four on a separate board. In order to maintain the randomness of the sequential testing order, the four additional samples were statistically "remixed" with the pool of samples and assigned random numbers in the test series. The samples were inserted into the original sequential order of testing by adding the letter "A" after the component number it followed; for example, if one of the added samples came after 100, it would be given the identification of 100A and the test sequence would become: 99, 100, 100A, 101, 102, etc. Continuing calibration checks on the XRF instruments were performed at the normal 15-sample intervals, except when the added plaster samples were encountered. For consistency, the "A" samples did not count as one of the 15-samples separating the continuing calibration checks; this allowed the QC checks to be performed after the same samples as before the 4 plaster samples were added. At a later date, the entire archive component series was statistically rerandomized and renumbered so that all inserted components were fully integrated into the test sequence.

Section 4.0 Time Measurements

The time of day each component is measured by XRF test is recorded from a clock or watch to ±1 min precision from local standard time. Actual durations of the XRF measurement are recorded to the nearest second and are generally recorded directly from the instrument display, if available.

Section 4.0 Performance Criteria for Temperature and Relative Humidity Measurements

The digital meter used to record the relative humidity and temperature at the time of the XRF test measurements is calibrated to be traceable to NIST standards within accuracies of ±1°C for temperature and ±5% RH for relative humidity.

Section 7.0 Disk Formatting for XRF Test Data

In general, no special formatting of computer disks is required for the XRF testing. File formats and downloading procedures vary by XRF instrument and are described on the data forms at the time of the testing.

Section 9.0 Radiation Safety Certification of XRF Operators

Before any XRF testing is performed, the XRF operator is required to show evidence of current certification and applicable licenses in radiation safety and the operation of XRF test equipment.

Appendix B

XRF Testing Protocols

Contents

GENERAL XRF MEASUREMENT PROTOCOLS

1.0 SUMMARY

This document describes the standard protocol for collecting XRF measurement data on painted surfaces and corresponding substrate surfaces of the archived samples. This document also includes instructions for recording the measurements and making quality control (QC) measurements during this study. In general, XRF operators will be requested to make measurements and to electronically store and download testing results according to the manufacturer's general operating procedures. It is the responsibility of the data monitor to record as much information as possible about the operation of each XRF instrument participating in this study.

For purposes of this study, an XRF reading is defined as a single measurement event that generates a single lead measurement value. Furthermore, XRF instruments are classified into one of the following two categories:

1) XRF instruments with an operator adjustable measurement time where the reading time, with a new source, is to be set to one of the following:

    a. Reading time with a new source is to be set to 15 seconds. Fifteen seconds is the minimum reading time to be used for this study. If the XRF instrument cannot automatically increase this 15 second nominal reading time as the source ages then manual reading time adjustments are to be made taking into account the half-life of the source.

    b. Reading time to be set to manufacturer's specifications as indicated in SOP's provided by the manufacturer or set to 15 seconds, whichever is longer.

2) XRF instruments that do not permit measurement time to be adjusted by the operator. The reading time is to be that which is programmed by the manufacturer into the XRF instrument.

In situations where the study protocol given herein differs dramatically from the manufacturer's protocol, or when this study protocol cannot be followed because of operational limitations, the XRF operator is required to discuss the situation with the field supervisor to resolve the problems. Operators should not contact the manufacturer of the XRF instrument unless approved by the field supervisor. Any deviations from this

protocol must be agreed to by the field supervisor and fully documented **before** implementing the deviation. In any case, each XRF must be operated in a safe and consistent manner throughout this study.

2.0 MATERIALS AND EQUIPMENT

- One primary and one back-up portable field XRF instrument with any extra required supporting equipment. (To be provided by XRF contractor.)
- One set of NIST paint films (SRM 2579); contains five films of different lead levels. (To be provided by XRF contractor.)
- Dosimeter badges; one for each XRF operator and one for each individual working within the same room where XRF testing takes place. (Operator badges will be provided by XRF contractor, badges for QuanTech personnel will be provided by QuanTech, and badges for MRI personnel will be provided by MRI).
- Forms for recording data and the "Archive Testing Order" sheet; see exemplars in this protocol. (To be provided by MRI; will be available at site.)
- Waterproof (indelible) permanent marking pen. (To be provided by MRI; will be available at site.)
- Watch, clock, or other equivalent timepiece for reporting the testing times on the data forms. (To be provided by MRI; will be available at site.)
- Device(s) to measure temperature and relative humidity. (To be provided by MRI; will be available at site.)
- Bound field notebooks. (To be provided by MRI; will be available at site.)
- Pre-moistened wipes for cleaning of tools, hands, etc. (To be provided by MRI; will be available at site.)
- Quality control (QC) blocks, each approximately 4 inch by 4 inch. The thicknesses given are approximate: ¾ inch wood (pine), 2 inch concrete (with aggregate), ½ inch drywall, 20 to 25 gauge metal, 2½ inch brick, and 1 inch plaster. (To be provided by MRI; will be available at site)
- One 12-inch thick styrofoam block for supporting QC control blocks under measurement. (To be provided by MRI; will be available at site.)

- Computer equipment including an IBM compatible CPU (preferably with an INTEL 386 or 486 processor), monitor, printer and a supply of 3½ inch formatted diskettes. (To be provided by MRI; will be available at site.)

- Radioactive decay tables that list the half-life of the radioactive sources used by the XRF instruments participating in this study. (To be provided by MRI; will be available at the site.)

3.0 MEASUREMENT PROCEDURES

The archive testing program utilizes 158 archive samples. These 158 archive samples will be tested in random order. The same order of testing is to be used for all instruments, for all test rounds. (With two exceptions, the order for testing has been the same for all testing of instruments. The two exceptions are the changes resulting from adding additional plaster samples to the archive and the occurrence of a single test round with an ordering based on grouping similar substrates. The testing based on the similar substrates grouping has not been used for PCS development.) The marking is in the form of squares and rectangles labeled with the letters "**X**" or "**P**" followed immediately by the numbers "**1**", "**2**", or "**3**". **X1** is the primary painted XRF testing location, **X2** and **X3** are secondary painted testing locations. **P1** is a secondary testing location that has been scraped of its paint.

All testing should be accomplished in a two day period. If in the event that testing is not completed after the second day, perform the testing protocol on the third day as described for day two. XRF testing on the archived samples can be summarized into the following:

- INITIAL control measurements, as described in Section 3.3.
- STANDARD measurements as described in Section 3.4.
- CONTINUING control measurements as described in Section 3.5.
- COMPARISON SET measurements as described in Section 3.6.
- ENDING control measurements as described in Section 3.3.

The two day general work plan is given below.

Day 1 General Work Plan:

1. Receive beginning-of-day instructions from field supervisor as described in Section 3.1.

2. Perform manufacturer's initial calibration checks. Perform additional manufacturer's calibration checks at intervals as required by the manufacturer's specifications. Both are discussed in Section 3.2.

3. Perform initial control block measurements on all six blocks as described in Section 3.3. Record temperature and humidity data.

4. Perform standard set measurements as described in Section 3.4.

5. After the completion of the first fifteen archive samples, and following every fifteen archive sample thereafter, perform continuing control readings on the two control blocks with the same substrates as the last and next substrates tested as described in Section 3.5.

6. Record temperature and humidity data approximately half-way through the day.

7. At various times during the day, record in the field notebook the time required by the XRF instrument to actually display the lead measurement result. The data recorded during the day for this purpose should include the start and end times for twenty measurements. A start time is the time of day when the XRF probe face-plate is placed on the surface to be tested and testing commences. The end time is the time of day when the result is displayed on the XRF readout area. These data should be collected for each reading time.

8. At the end of the first day of testing, perform ending control block measurements on all six blocks as described in Section 3.3. Record temperature and humidity data.

9. Perform end-of-day activities as described in Section 3.7 which includes review of data forms for completeness, downloading electronically stored data, and transferring data forms to the field supervisor.

Day 2 General Work Plan:

10. At the start of the second day, receive beginning-of-day instructions from field supervisor as described in Section 3.2.

11. Perform manufacturer's initial calibration checks. Perform additional manufacturer's calibration checks at intervals as required by the manufacturer's specifications. Both are discussed in Section 3.2.

12. Perform initial control block measurements on all six blocks as described in Section 3.3. Record temperature and humidity data.

13. Perform the standard measurements (Section 3.4) not completed on the previous day of testing. Begin with the testing location immediately following the last tested location from the day before.

14. Record temperature and humidity data approximately half-way through the day.

15. After the completion of the first fifteen archive samples on this day, and following every fifteen archive sample thereafter, perform continuing control readings on the two control blocks with the same substrates as the last and next substrates tested as described in Section 3.5.

16. Upon completion of all 154 standard testing locations, re-test the locations as described in Section 3.6. This testing is known as the "comparison set" testing.

17. At every change of substrate, perform continuing control measurements on the two control blocks with the same substrate as the last and next substrate tested as described in Section 3.5.

18. At the end of this day of testing, perform ending control block measurements on all six blocks. Record temperature and humidity data as described in Section 3.3.

19. Perform end-of-day activities as described in Section 3.7 which includes review of data forms for completeness, download electronically stored data, and transfer data forms to the field supervisor.

3.1 INITIAL XRF TESTING PROCEDURE

XRF operators and monitors will receive detailed overview instructions from the field supervisor on the first day of XRF testing that will include the following topics:

- General safety instructions.

- Definitions of testing locations, measurements, testing time, and types of data.

- Completion of the "Archive XRF Instrument Information" form.
- Specific site issues and description of marked locations and what markings signify.
- Order of performing measurements.
- Use of each data form and placement of data on forms.
- Responsibilities of the XRF operator to inform the field supervisor when the determination of the duration of the reading time is being made.
- Responsibilities of the XRF operator to enter all information necessary for the electronic storage of testing results.
- Responsibilities of the XRF operator to call out all readings real-time.
- Responsibilities of the monitor to record information about the manufacturer's calibration or warm-up protocols in the field notebooks.
- Responsibilities of the monitor to record **all** data real-time and use verbal feedback to verify data. (No reading is to be discarded; however additional data can be taken if insisted on by the XRF operator. Additional readings should be recorded in the "Comments" column of the appropriate form.)
- Responsibilities of the monitor to periodically observe the actual instrument readout (particularly for recording both K- and L-shell readings). The monitor should observe about 10% of the readings and should place a check mark on the data form next to each observed reading.
- Responsibilities of the monitor to record temperature and humidity data in the field notebook three times a day.

3.2 BEGINNING OF EACH DAY ON-SITE PROCEDURES

The XRF operator and monitor will receive initial instructions from the acting field supervisor at the beginning of each testing day. Items will generally include a brief overview of those listed under Section 3.1 plus any additional items that are dictated by variable field conditions.

At the beginning of each day, the XRF operator will perform tests and instrument checks that are required by the manufacturer of the XRF to prepare the instrument for taking lead measurements. The operator is to enter "99999" into the XRF instrument for the location identification during these procedures. The XRF operator must inform the monitor that a manufacturer-recommended procedure is being performed and the nature of the procedure.

The monitor will record the time and nature of all such manufacturer-recommended procedures in the field notebook. This information should include but not be limited to: 1) what is being done, 2) the displayed result if any, and 3) the consequence, representation, or definition of the result. The monitor will also record temperature and humidity data in the field notebook.

3.3 CONTROL BLOCK READINGS — BEGINNING AND ENDING

At the beginning and end of each day, each XRF operator will perform a set of measurements on control blocks covered separately with two NIST SRM 2579 standards (red, 1.02 mg/cm^2; and yellow, 3.53 mg/cm^2) and without any NIST film covering. These QC readings will be taken on six substrate blocks: metal, wood, brick, drywall, concrete, and plaster. Before the start of each testing day five readings will be taken on each control block. The first reading will be taken through a yellow NIST (3.53 mg/cm^2) film covering, followed by three readings taken through a red NIST (1.02 mg/cm^2) film covering, followed by one reading taken without any NIST film covering. This same procedure will be repeated at the end of the day. Data from these beginning and end control readings will be recorded on the "ARCHIVE XRF TESTING DATA -- CONTROL READINGS" form. A step-by-step description is provided below:

At the beginning of each testing day, perform the following procedures:

1. For each new "ARCHIVE XRF TESTING DATA -- CONTROL READINGS" form needed, the monitor will complete the header of the form. Be sure to indicate in the appropriate space if the beginning control block measurements precede STANDARD set or COMPARISON set readings. Likewise, indicate in the appropriate space if the ending control block measurements follow STANDARD set or COMPARISON set readings.

2. If not already done, perform whatever normal instrument checks are required by the XRF manufacturer to prepare the instrument for taking lead measurements. The entered location identification number for these readings should be "99999".

The XRF operator will inform the monitor what the procedure is and why it is being done. The monitor will write this information in the field notebook.

3. For each control block, the monitor will fill in the "QC Type", "Block Type", "Time of Measurement," and "XRF Shell" columns. The location identification will be a two character code: a number and a letter. For the beginning of the day control block readings, the number will always be "1". The letter depends on the substrate of the control block and is the block type code shown in the table below. XRF instruments incapable of storing alpha characters should use the number codes given in the table below.

CONTROL BLOCK TYPE	BLOCK TYPE CODE	NUMBER CODE
metal	M	1
wood	W	2
brick	B	3
drywall	D	4
concrete	C	5
plaster	P	6

4. Perform control block measurements for all six control blocks using the procedure outlined below.

    a. Perform the measurements on the control blocks in the following substrate order: metal, wood, brick, drywall, concrete, and plaster.

    b. Place the control block within the marked area on the styrofoam support.

    c. Enter into the XRF instrument the location identification information, as described above.

    d. Center the yellow NIST film on each control block. Take one reading by placing the XRF probe face-plate on the NIST film and taking readings through the NIST film into the center of the control block. The XRF operator will call out the reading(s) from the instrument's display. The monitor will record each reading on the "ARCHIVE XRF TEST DATA -- CONTROL READINGS" form, verbally verifying the value written.

    e. Repeat this procedure with the red NIST film and take three readings. When the three readings are being taken, any movement of the XRF

probe face-plate should be avoided so that the three readings are taken on the same exact spot.

f. Repeat this procedure on bare substrate. Place the XRF probe face-plate on the control block without any NIST film covering and take one reading into the center of the control block.

g. The monitor will record any other information in the "Comments" column as necessary.

At the end of the testing day perform all of the above control measurements exactly as they were performed at the beginning of the day with one exception: the first character of the two character identification code to be entered into the XRF instrument and to be recorded on the form is the number "3" for control block readings taken at the end of the day.

3.4 PROCEDURE FOR STANDARD MEASUREMENTS AT EACH TESTING LOCATION

On each archive sample, standard measurements are to be taken. The order in which standard measurements are to be taken is the sequential order of the numbers on the white cards located in the plastic sleeve near each archive sample. The monitor may use the "Archive Testing Order Sheet" to locate archive samples. Standard measurements consist of nine XRF readings and are summarized as follows:

- One XRF reading taken on the painted surface of the primary area labeled **X1**.
- One XRF reading on the bare substrate area covered with the red NIST standard (1.02 mg/cm^2) labeled **P1**.
- One XRF reading taken on the surface of the bare substrate area without any NIST standard covering labeled **P1**.
- One XRF reading taken on the surface of the area labeled **X2**. Note that on a few archive samples, the **X2** area is not present.
- One XRF reading taken on the surface of the area labeled **X3**. Similar to above, on a few archive samples, the **X3** area is not present.

A step-by-step description of taking standard set XRF measurements follows:

1. For each new "ARCHIVE XRF TEST DATA -- STANDARD READINGS" form needed, the monitor will complete the header of the form.

2. For each archive sample, the monitor will record the testing location identification number located on the white card in the "Location ID" column on the "ARCHIVE XRF TEST DATA -- STANDARD READINGS" and record the "Time of Measurement" and "XRF Shell". The XRF operator will enter the white card location identification number into the instrument.

3. Perform measurements on the painted and bare substrate surfaces as follows:

    a. Take one reading on the painted surface marked as **X1**. The three readings should be taken on the same exact spot, so any movement of the XRF probe face-plate should be avoided when taking these readings. The XRF operator will call out the reading(s) from the instrument's display. The monitor will record each reading on the "ARCHIVE XRF TEST DATA - STANDARD READINGS," verbally verifying the value written. The monitor will also record other information in the "Comments" column as necessary.

    b. Take one reading on the bare substrate surface covered with the 1.02 mg/cm^2 NIST standard red film at the testing location labeled **P1**. This area is adjacent to the **X1** testing area described above and has approximate dimensions of either four inches by four inches or four inches by two inches. The three readings should be taken on the same exact spot, so any movement of the XRF probe face-plate should be avoided when taking these readings. If difficulties are encountered holding the NIST film against the substrate surface, use a small piece of masking tape to hold it in place. Be sure the tape is placed such that it adheres only to areas outside the marked location. The XRF operator will call out the reading(s) from the instrument's display. The monitor will write each reading value on the "ARCHIVE XRF TEST DATA - STANDARD READINGS," verbally verifying the value written. The monitor will record any other information in the "Comments" column as necessary.

    c. Take one reading on the bare substrate surface without any NIST film covering. The XRF operator will call out the reading(s) from the instrument's display. The monitor will write each reading value on the "ARCHIVE XRF TEST DATA - STANDARD READINGS," verbally

verifying the value written. The monitor will record any other information in the "Comments" column as necessary.

d. Take one reading at the painted surface marked as **X2**. The XRF operator will call out the reading(s). The monitor will write this value on the "ARCHIVE XRF TEST DATA - STANDARD READINGS," verbally verifying the value written. The monitor will record any other information in the "Comments" column as necessary. Note that in a small number of cases, the **X2** area will not be present.

e. Take one reading at the painted surface marked as **X3**. The XRF operator will call out the reading(s). The monitor will write this value on the "ARCHIVE XRF TEST DATA - STANDARD READINGS," verbally verifying the value written. The monitor will record any other information in the "Comments" column as necessary. Like the **X2** area, the **X3** area will not be present on every archive sample.

4. Continuing control readings are performed after every fifteenth archive sample as outlined in Section 3.5.

3.5 PROCEDURES FOR CONTINUING CONTROL READINGS

Two sets of continuing control readings are performed after every fifteenth archive sample for standard readings and at every substrate change when taking "comparison set" readings. The procedure for continuing control readings is the same for either standard testing or "comparison set" testing. The first set of continuing control readings is performed on the control block that is composed of similar material as the last archive sample tested. The second set of continuing control readings is performed on the control block that is composed of similar material as the next archive sample to be tested. For each set of control readings, use the following steps:

1. For each new "ARCHIVE XRF TESTING DATA -- CONTROL READINGS" form needed, the monitor will complete the header of the form. Be sure to indicate in the appropriate space if these are STANDARD or COMPARISON control readings.

2. For each control block, the monitor will fill in the "QC Type", "Block Type", "Time of Measurement," and "XRF Shell" columns and the XRF operator will enter the location identification into the instrument. The location identification will be a two character code: a number and a letter. For continuing control block readings the number will always be "2". The second character, a letter, depends

on the substrate of the control block and is the block type code shown in the table below. XRF instruments incapable of storing alpha characters should use the number codes given in the table below.

CONTROL BLOCK TYPE	BLOCK TYPE CODE	NUMBER CODE
metal	M	1
wood	W	2
brick	B	3
drywall	D	4
concrete	C	5
plaster	P	6

3. Take the continuing control readings using two NIST films and the bare control block without any NIST film covering as follows:

    a. Place the control block within the marked area on the styrofoam support block.

    b. Center the yellow NIST film on the control block. Take one reading through the NIST film and into the center of the control block.

    c. Repeat the procedure using the red NIST film and take three readings. The three readings should be taken on the same exact spot, so any movement of the XRF probe face-plate should be avoided when taking these readings.

    d. Take one reading on the bare control block without any NIST covering. The XRF operator will call out the reading(s) from the instrument's display. The monitor will write each reading on the "ARCHIVE XRF TEST DATA -- CONTROL READINGS" form, verbally verifying the value written. The monitor will record any other information in the "Comments" column as necessary.

3.6 PROCEDURE FOR COMPARISON SET READINGS

After completion of all the archive samples in the standard set, XRF testing of the "comparison set" follows. Comparison set readings are to be taken on all 158 archive samples. The comparison set is ordered such that the archive samples are grouped by

substrate in the following order: metal, wood, brick, drywall, concrete, and plaster. The monitor should exchange the relative positions of the white and pink cards located in the plastic sleeve near each archive sample so that the pink card is visible prior to comparison set testing. Comparison set readings are to be taken in the sequential order of the numbers on the pink cards. The monitor may use the "Archive Testing Order Sheet" to locate archive samples. Comparison set readings are taken on the surface of the area labeled **X1**; readings are not taken at any of the other marked areas. At each testing location labeled **X1** perform the following steps:

1. For each new "ARCHIVE XRF TEST DATA -- COMPARISON READINGS" form needed, the monitor will complete the header of the form.

2. For each archive sample, the monitor will record the pink card location identification number in the "Location ID" column on the "ARCHIVE XRF TEST DATA -- COMPARISON READINGS" and will also record the "Time of Measurement" and the "XRF Shell". The XRF operator will enter the pink card location identification number into the XRF instrument.

3. The XRF operator will take one XRF reading on the painted surface of the area labeled **X1** and call out the reading(s) from the instrument's display. The monitor will write each reading value on the "ARCHIVE XRF TEST DATA - COMPARISON READINGS," verbally verifying the value written. The monitor will record other information in the "Comments" column as necessary.

4. Continuing control readings are performed at every substrate change as described in Section 3.5. Five different pairs of continuing control measurements will be made during XRF testing of the comparison set.

Note: This "comparison set" with substrate-by-substrate ordering was used once during the course of archive testing and was subsequently dropped from future testing, as testing with random ordering met the primary goals of the project.

3.7 END-OF-DAY ACTIVITIES

- The XRF operator and monitor will ascertain that all form headers are completed, including the appropriate page numbering so that the forms of the same type are numbered in chronological order for that day of testing only, starting with page 1. The last page number should also be written on every page.

- XRF operator and monitor will verify that all required XRF readings were taken at each testing location as specified in this protocol. Verification will be performed

by reviewing the data forms and checking the appropriate box in the "Archive End of Day Data Checklist" form.

- The XRF operator will download (transfer) electronically stored data and purge the data that is currently stored in the instrument after the download is completed. The electronically stored data should be downloaded at the test site to the $3^{1}/_{2}$ inch diskettes and backup copies on a second set of diskettes should be made. The computer equipment provided at the site may be used for this purpose. The monitor should verify that the download was successful prior to instructing the XRF operator to purge the data stored in the XRF instrument. The monitor will record the procedure used to download and purge the electronically stored data in the field notebook.

- XRF data forms will be transferred to the acting field supervisor at the end of each day. The acting field supervisor will check the data forms for completeness and conduct other end-of-day activities before releasing workers for the day.

CLARIFICATIONS AND ADDITIONS TO GENERAL XRF MEASUREMENT PROTOCOLS

Section 3.0 Order of Testing

Sample testing on the archived lead-based painted components is performed in the same sequence for all testing rounds and for all XRF instruments. Briefly, the testing order was established by random spatial distribution of the samples throughout the facility and by predetermining a random test sequence by random number generation from all substrate types and lead concentration levels.

Section 3.1 Reading Verifications by Monitor

For any situation in which the monitor's reading differs from the operator's reading, the testing will be temporarily stopped until the cause of the discrepancy is determined and the actions taken will be noted on the data collection forms.

With the following exception, the sequence order is the same for all XRF testing. Four additional plaster samples were added to the sample test series after the archive facility was built. The samples were physically added to the facility by mounting all four on a separate board. In order to maintain the randomness of the sequential testing order, the four additional samples were statistically "remixed" with the pool of samples and assigned random numbers in the test series. The samples were inserted into the original sequential order of testing by adding the letter "A" after the component number it followed; for example, if one of the added samples came after 100, it would be given the identification of 100A and the test sequence would become: 99, 100, 100A, 101, 102, etc. Continuing calibration checks on the XRF instruments were performed at the normal 15 sample intervals, except when the added plaster samples were encountered. For consistency, the "A" samples did not count as one of the 15 samples separating the continuing calibration checks; this allowed the QC checks to be performed after the same samples as before the 4 plaster samples were added.

Section 3.6 Comparison Set Readings

The procedure indicated in Section 3.6 was discontinued early in the program and replaced with a single unified ordering system.

TESTING PROTOCOLS FOR THE LPA-1

General Responsibilities

The XRF Operator will:

- Handle the instruments and make the measurements.
- Work with the Testing Monitor to see that the appropriate data are recorded.
- Work with the Testing Supervisor to help assure that protocols are followed and any deviations are properly documented.
- Download data to a computer and verify that the transfer was successful.
- Report to the Testing Supervisor any indications of deteriorating samples observed during testing.

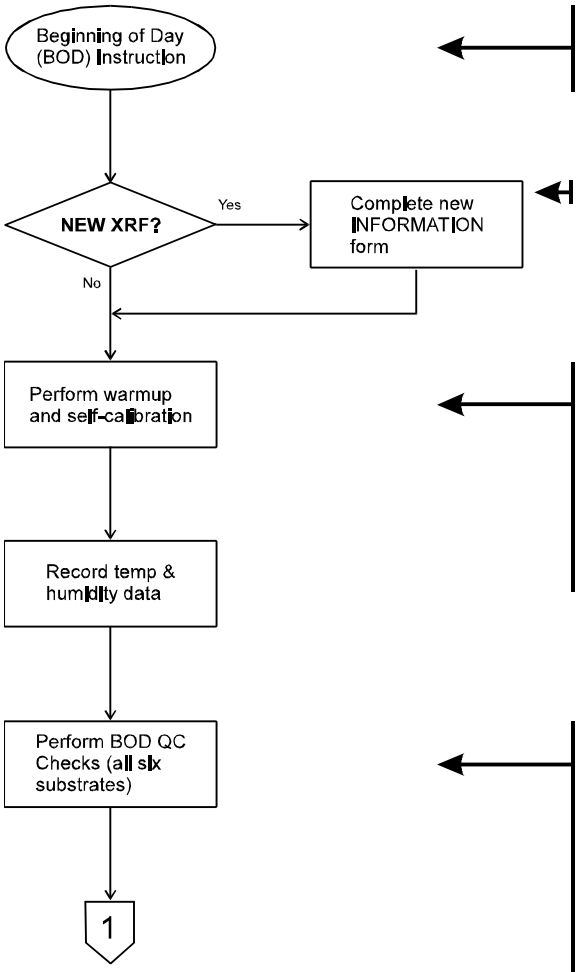
The Monitor will:

- Record all relevant data on the appropriate data forms or field notebooks.
- Work with the XRF Operator to help assure the protocols are followed.
- Work with the Testing Supervisor to help assure that protocols are followed and any deviations are properly documented.

The Supervisor will:

- Verify ID assignments on sample locations on the archive components.
- Assure that the data are collected as described in the protocol.
- Collect all forms and electronic data and make appropriate distributions to MRI, NIST, and QuanTech.
- Assure that resources are available to achieve planned testing.
- Oversee testing activities.
- Provide beginning-of-day and end-of-day instructions.
- Make primary decisions regarding any testing difficulties that may arise.
- Complete a new set of SAMPLE LOCATION CONDITION forms for:
  a. Sample locations previously identified as being in poor condition which already have completed records from the early spring 1995 testing.
  b. Any additional sample locations that are observed to be in poor condition.
- Provide the following forms:
  - Testing order list
  - Sample location condition
  - Standard readings
  - Control readings
  - Archive XRF information
  - Source age adjustment table

FLOW CHART SUMMARY | **DESCRIPTION DETAILS FOR RMD**



Instructions to include: safety issues, field form use, archive layout and testing order, Sample ID numbering for electronic data capture, and voice-call back verification of data recording by monitor.

See form for needed information.

1. Enter date and time.
2. Allow to warmup at least 20 minutes to assure that the auto self-calibration occurs (automatically done every 15 minutes).
3. Perform calibration check using STD Mode set to 20 seconds: Test both bare and painted sides of factory supplied wood substrate. Record results on INFORMATION form.
4. Set Quick Mode Testing using an action level of 1.0 mg/cm^2.

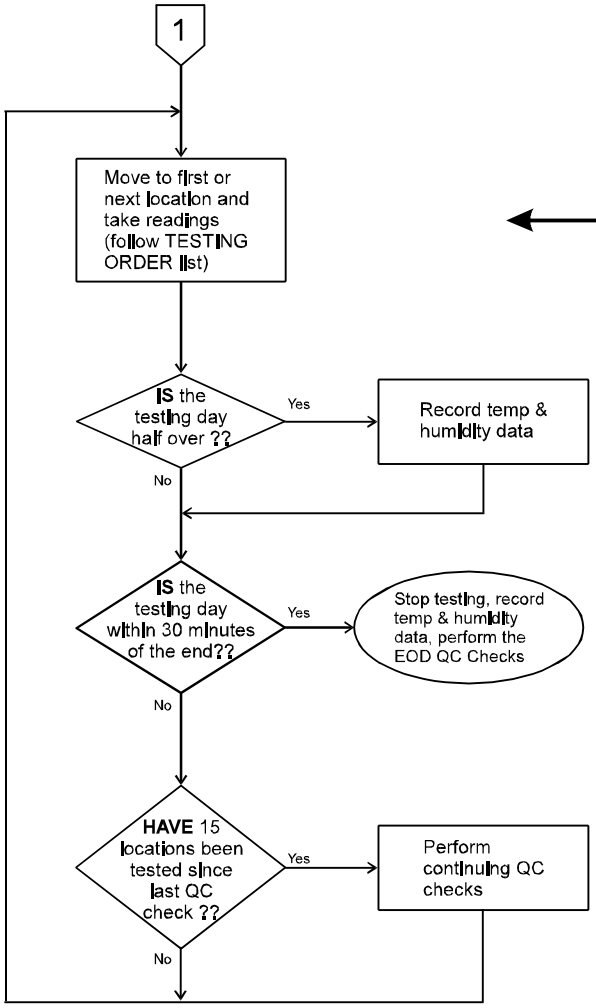
BOD (or EOD) QC checks: test a total of 6 pre-placed blocks on a minimum of 12 inches of styrofoam support. **Please do not move blocks.**

For each block, using a 20-sec STD Mode reading, test in the following order:

a. 1 reading, yellow NIST on block
b. 1 reading, red NIST on block
c. 1 reading, no NIST on block

Record the data on the CONTROL READINGS form (RMD). If possible key in ID code as indicated on the form for electronic data capture.

FLOW CHART SUMMARY



DESCRIPTION DETAILS FOR RMD

Test all of the samples at each testing location first in the Quick mode, then repeat using the STD Mode set to 20 seconds.

For each testing location, in each mode, test in the following order:

a. 1 reading, on X1
b. 1 reading, on bare (P1) + red NIST
c. 1 reading, on bare (P1)
d. 1 reading, on X2
e. 1 reading, on X3.

Record data on the STANDARD READINGS form (RMD).

Periodically measure the time required to take a reading from start to data display and record it in the field notebook.

EOD QC checks are the same as BOD QC checks: follow same procedure as for BOD QC Checks.

Record temp and humidity in a field notebook.

Download any electronically stored data, verify successful transfer before clearing the memory to make room for more data.

Start next day at top of flow chart using the next sample to be tested as the first location.

Continuing QC checks, total of 2 blocks tested on a minimum of 12 inches styrofoam support. Use blocks matching same type as the last and next locations (if same type for both, test same block twice).

Follow same testing order and reading times as that used for BOD testing.

Record on CONTROL READINGS form (RMD). If possible, key in ID code as indicated on the form for electronic data capture for these readings.

Archive XRF Instrument Information

Date _____

Testing Site _____

Testing Dates _____

Contractor _____

Manufacturer _____

Model No. _____

Serial No. _____

XRF Operator (Printed name) _____

XRF Operator (Signature) _____

Source Material _____

Source Serial No. _____

Source Age or Date _____

Detector Type _____

Operating Parameters Used

Open shutter sampling time? (fixed or variable) _____

If fixed, what is the duration time? _____

Daily warm-up and calibration check used? (Briefly discuss) _____

_____

_____

Regulatory level value used for setting the XRF instrument? (Yes or No) _____

If yes, enter the value used _____

Other XRF parameters under operator control used? (Briefly discuss) _____

_____

_____

☐ Check here to indicate the presence of additional comments on the back of this form.

Archive XRF Test Data -- Standard Readings

Page _____ of _____

Date ______________ XRF Device **RMD** ______________ Serial Number ______________

XRF Operator (Printed Name) ______________ XRF Field Monitor (Printed name) ______________

Location ID	Time of Measurement	Data Item	Paint Surface Reading: X1	Substrate + NIST Red, 1.02 mg/cm² Reading	Substrate Only Reading	Paint Surface Reading: X2	Paint Surface Reading: X3	Comments
		K-shell (mg/cm²) Quick Mode						
		K-shell (mg/cm²) STD Mode						
		K-shell (mg/cm²) Quick Mode						
		K-shell (mg/cm²) STD Mode						
		K-shell (mg/cm²) Quick Mode						
		K-shell (mg/cm²) STD Mode						
		K-shell (mg/cm²) Quick Mode						
		K-shell (mg/cm²) STD Mode						
		K-shell (mg/cm²) Quick Mode						
		K-shell (mg/cm²) STD Mode						
		K-shell (mg/cm²) Quick Mode						
		K-shell (mg/cm²) STD Mode						
		K-shell (mg/cm²) Quick Mode						
		K-shell (mg/cm²) STD Mode						
		K-shell (mg/cm²) Quick Mode						
		K-shell (mg/cm²) STD Mode						
		K-shell (mg/cm²) Quick Mode						
		K-shell (mg/cm²) STD Mode						
		K-shell (mg/cm²) Quick Mode						
		K-shell (mg/cm²) STD Mode						

Archive XRF Test Data -- Control Readings

Page _____ of _____

Date ______________ XRF Device **RMD** ______________ Serial Number ______________

XRF Operator (Printed Name) ______________ XRF Field Monitor (Printed name) ______________

QC Type: 1 = Initial Control Readings 2 = Continuing Control Readings 3 = Ending Control Readings Block Type: M = Metal, W = Wood, B=Brick, D=Drywall, C=Concrete, P=Plaster

Identification		Time of Measurement	Data Item	Readings			Comments
QC Type	Block Type			Yellow, 3.53 mg/cm²	Red, 1.02 mg/cm²	No NIST SRM	
			K-shell (mg/cm²) STD Mode				
			K-shell (mg/cm²) STD Mode				
			K-shell (mg/cm²) STD Mode				
			K-shell (mg/cm²) STD Mode				
			K-shell (mg/cm²) STD Mode				
			K-shell (mg/cm²) STD Mode				
			K-shell (mg/cm²) STD Mode				
			K-shell (mg/cm²) STD Mode				
			K-shell (mg/cm²) STD Mode				
			K-shell (mg/cm²) STD Mode				

TESTING PROTOCOLS FOR THE XL

General Responsibilities

The XRF Operator will:

- Handle the instruments and make the measurements.
- Work with the Testing Monitor to see that the appropriate data are recorded.
- Work with the Testing Supervisor to help assure that protocols are followed and any deviations are properly documented.
- Download data to a computer and verify that the transfer was successful.
- Report to the Testing Supervisor any indications of deteriorating samples observed during testing.

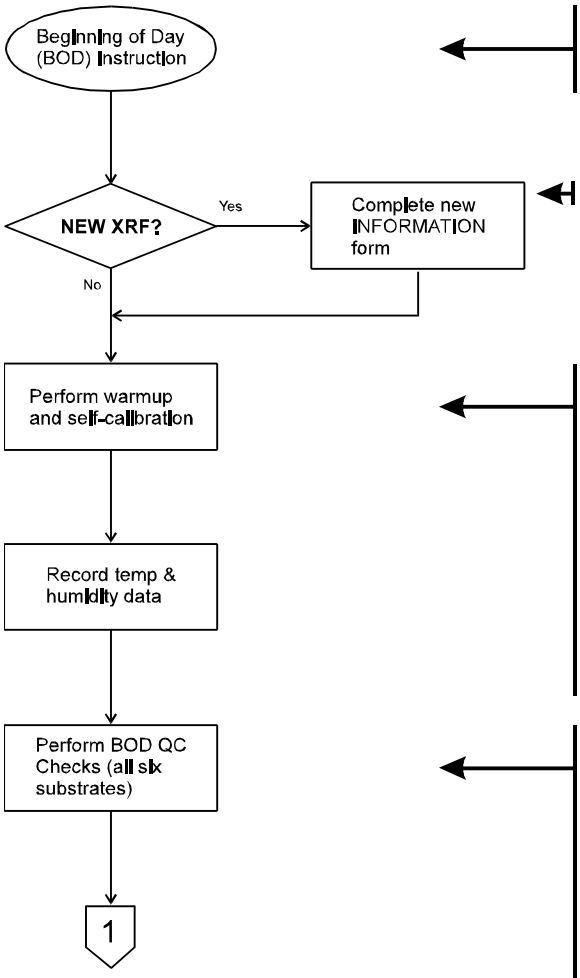
The Monitor will:

- Record all relevant data on the appropriate data forms or field notebooks.
- Work with the XRF Operator to help assure the protocols are followed.
- Work with the Testing Supervisor to help assure that protocols are followed and any deviations are properly documented.

The Supervisor will:

- Verify ID assignments on sample locations on the archive components.
- Assure that the data are collected as described in the protocol.
- Collect all forms and electronic data and make appropriate distributions to MRI, NIST, and QuanTech.
- Assure that resources are available to achieve planned testing.
- Oversee testing activities.
- Provide beginning-of-day and end-of-day instructions.
- Make primary decisions regarding any testing difficulties that may arise.
- Complete a new set of SAMPLE LOCATION CONDITION forms for:
    a. Sample locations previously identified as being in poor condition which already have completed records from the early spring 1995 testing.
    b. Any additional sample locations that are observed to be in poor condition.
- Provide the following forms:
    - Testing order list
    - Sample location condition
    - Standard readings
    - Control readings
    - Archive XRF information
    - Source age adjustment table

FLOW CHART SUMMARY | **DESCRIPTION DETAILS FOR NITON**



Instructions to include: safety issues, field form use, archive layout and testing order, Sample ID numbering for electronic data capture, and voice-call back verification of data recording by monitor.

See form for needed information.

1. Perform self-calibration.
2. Allow to warm-up for 20 minutes.
3. Perform self-calibration - perform every 4 hours. Record any results on the XRF INSTRUMENT INFORMATION form.
4. Perform calibration check using a 20 second reading (2nd beep):
   Test both sides on the supplied 5 standard films against wood substrate.
   IF lead value is outside displayed window by greater than 10% or 0.1 mg/cm^2 (whichever is higher),
   THEN, repeat calibration check using a 1 minute check,
   IF OK, then go back to step 2.
   IF not, contact THE SUPERVISOR TO ADVISE.

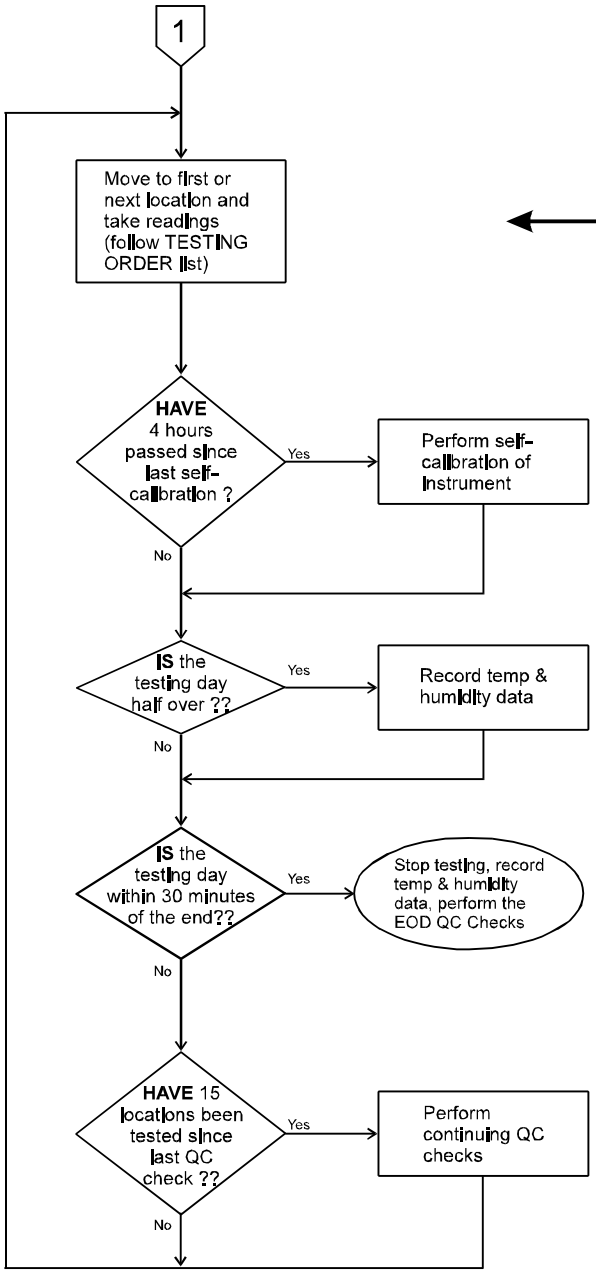
BOD (or EOD) QC checks: test a total of 6 pre-placed blocks on a minimum of 12 inches of styrofoam support. **Please do not move blocks.**

For each block, using a 20-sec mode, test in the following order:

a. 1 reading, yellow NIST on block
b. 1 reading, red NIST on block
c. 1 reading, no NIST on block

Record the data on the CONTROL READINGS form (NITON). If possible key in ID code as indicated on the form for electronic data capture.

FLOW CHART SUMMARY



DESCRIPTION DETAILS FOR NITON

For each testing location, using 20-second mode test in the following order:

a. 1 reading, on X1
b. 1 reading, on bare (P1) + red NIST
c. 1 reading, on bare (P1)
d. 1 reading, on X2
e. 1 reading, on X3.

Record data on the STANDARD READINGS form.

For first testing day, at least, determine using X1 data, whether a longer testing time is needed based on the attached NITON truth table. If indicated, collect a 120 second reading on X1 after completion of a-e using the 20 second mode.

Periodically measure the time required to take a reading from start to data display, and record it in the field notebook.

EOD QC checks are the same as BOD QC checks: follow same procedure as for BOD QC Checks.

Record temp and humidity in a field notebook.

Download any electronically stored data, verify successful transfer before clearing the memory to make room for more data.

Start next day at top of flow chart using the next sample to be tested as the first location.

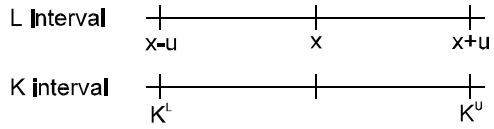
Continuing QC checks, total of 2 blocks tested on a minimum of 12 inches styrofoam support. Use blocks matching same type as the last and next locations (if same type for both, test same block twice).

Follow same testing order and reading times as that used for BOD testing.

Record on CONTROL READINGS form (NITON). If possible key in ID codes as indicated on the form for electronic data capture for these readings.

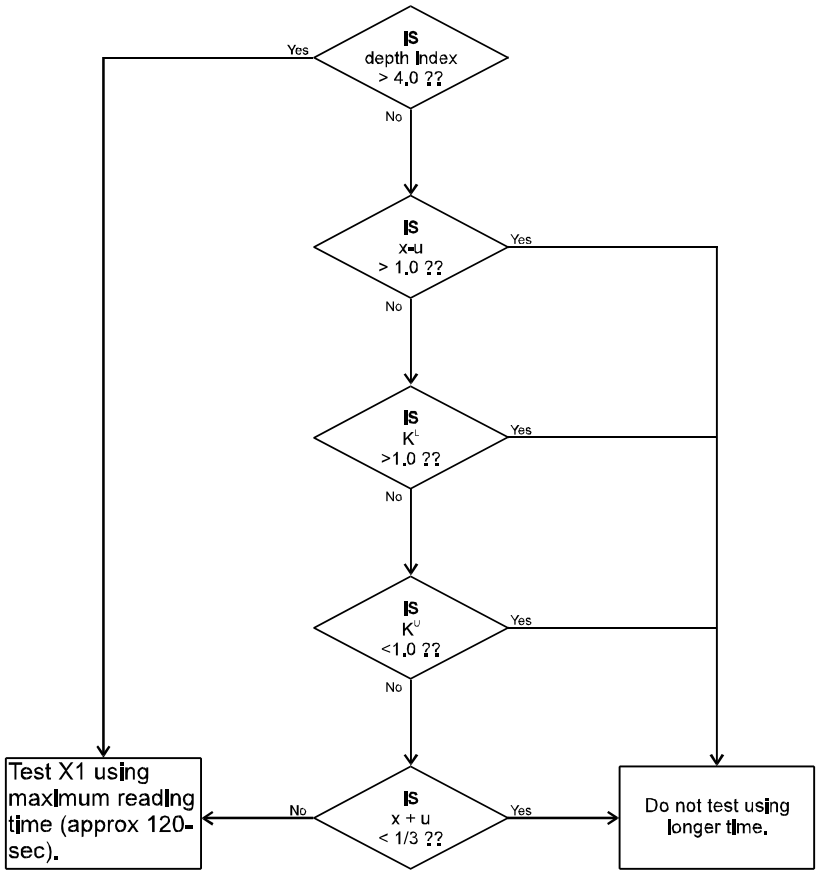
NITON TRUTH TABLES FOR DETERMINATION OF LONGER TESTING TIMES
Summarized from NITON operating procedures update dated 12/21/94

DEFINITION OF DISPLAYED LEAD RESULTS:

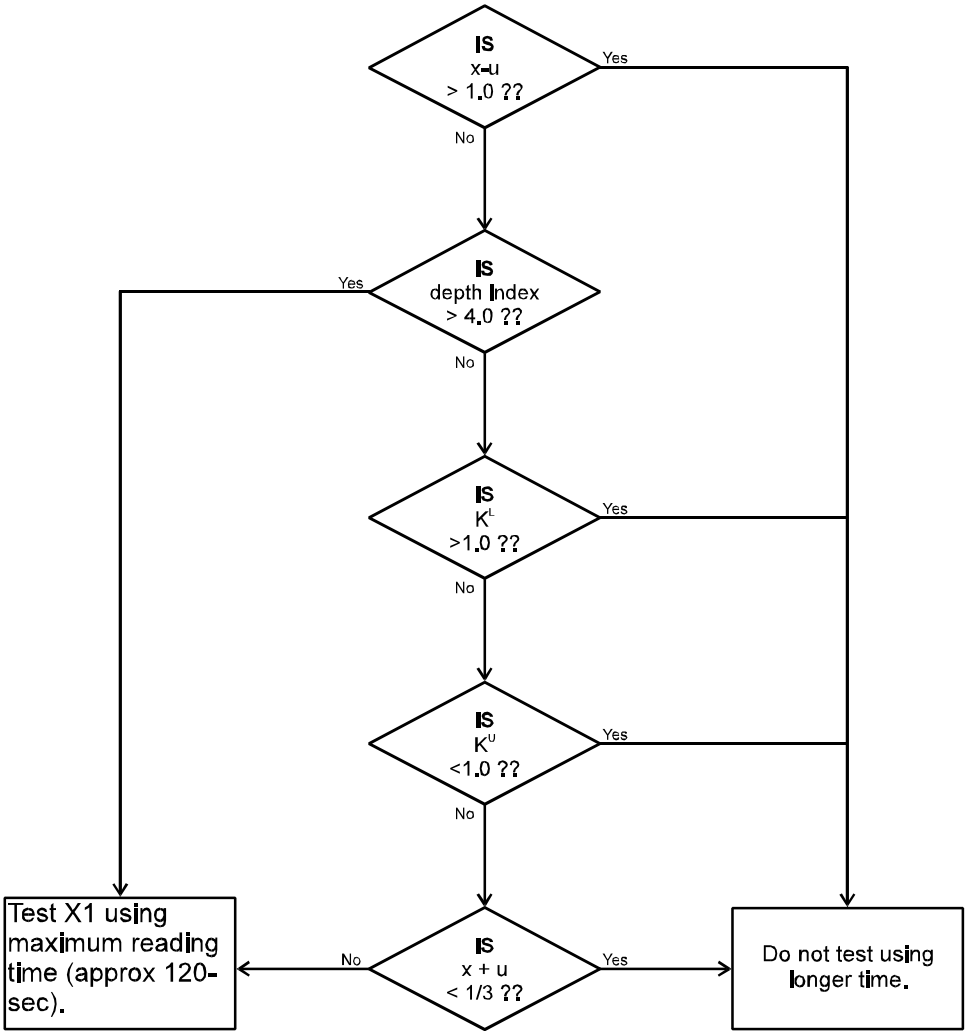


where: x = mean L shell result
u = L shell uncertainty
K^L = K shell lower result
K^U = K shell upper result

DECISION FLOWCHART (version A):



DECISION FLOWCHART (version B):



NOTE: Truth tables are based on supplemental documentation received from NITON (12/21/94). Testing duration decisions based on the depth index were missing from the supplemental documentation. However, decisions based on the depth index were the only clearly identified directive in the XL manual. Therefore, this conditional branch was included into the truth table flow-charts. Differences between truth table versions reflect only the placement of this depth index check within the flow-chart. All samples were tested using version B.

Archive XRF Instrument Information

Date _____

Testing Site _____

Testing Dates _____

Contractor _____

Manufacturer _____

Model No. _____

Serial No. _____

XRF Operator (Printed name) _____

XRF Operator (Signature) _____

Source Material _____

Source Serial No. _____

Source Age or Date _____

Detector Type _____

Operating Parameters Used

Open shutter sampling time? (fixed or variable) _____

If fixed, what is the duration time? _____

Daily warm-up and calibration check used? (Briefly discuss) _____

_____

_____

Regulatory level value used for setting the XRF instrument? (Yes or No) _____

If yes, enter the value used _____

Other XRF parameters under operator control used? (Briefly discuss) _____

_____

_____

☐ Check here to indicate the presence of additional comments on the back of this form.

Archive XRF Test Data -- Standard Readings

Page _____ of _____

Date ______________ XRF Device **NITON XL** ______________ Serial Number ______________

XRF Operator (Printed Name) ______________ XRF Field Monitor (Printed name) ______________

Location ID	Time of Measurement	Data Item	Paint Surface Reading: X1	Substrate + NIST Red, 1.02 mg/cm² Reading	Substrate Only Reading	Paint Surface Reading: X2	Paint Surface Reading: X3	Comments
		Testing Time (sec)						
		L-shell (mg/cm²)						
		L-shell (mg/cm²) Uncertainty						
		Lower K-shell (mg/cm²)						
		Upper K-shell (mg/cm²)						
		Depth Index						
		Testing Time (sec)						
		L-shell (mg/cm²)						
		L-shell (mg/cm²) Uncertainty						
		Lower K-shell (mg/cm²)						
		Upper K-shell (mg/cm²)						
		Depth Index						
		Testing Time (sec)						
		L-shell (mg/cm²)						
		L-shell (mg/cm²) Uncertainty						
		Lower K-shell (mg/cm²)						
		Upper K-shell (mg/cm²)						
		Depth Index						

Archive XRF Test Data -- Control Readings

Page _____ of _____

Date ______________ XRF Device **NITON XL** ______________ Serial Number ____________________

XRF Operator (Printed Name) ____________________ XRF Field Monitor (Printed name) ____________________

QC Type: 1 = Initial Control Readings 2 = Continuing Control Readings 3 = Ending Control Readings Block Type: M = Metal, W = Wood, B=Brick, D=Drywall, C=Concrete, P=Plaster

Identification		Time of Measurement	Data Item	Readings			Comments
QC Type	Block Type			Yellow, 3.53 mg/cm²	Red, 1.02 mg/cm²	No NIST SRM	
			Testing Time (sec)				
			L-shell (mg/cm²)				
			L-shell (mg/cm²) Uncertainty				
			Lower K-shell (mg/cm²)				
			Upper K-shell (mg/cm²)				
			Depth Index				
			Testing Time (sec)				
			L-shell (mg/cm²)				
			L-shell (mg/cm²) Uncertainty				
			Lower K-shell (mg/cm²)				
			Upper K-shell (mg/cm²)				
			Depth Index				
			Testing Time (sec)				
			L-shell (mg/cm²)				
			L-shell (mg/cm²) Uncertainty				
			Lower K-shell (mg/cm²)				
			Upper K-shell (mg/cm²)				
			Depth Index				

TESTING PROTOCOLS FOR THE LEADSTAR

General Responsibilities

The XRF Operator will:

- Handle the instruments and make the measurements.
- Work with the Testing Monitor to see that the appropriate data are recorded.
- Work with the Testing Supervisor to help assure that protocols are followed and any deviations are properly documented.
- Download data to a computer and verify that the transfer was successful.
- Report to the Testing Supervisor any indications of deteriorating samples observed during testing.

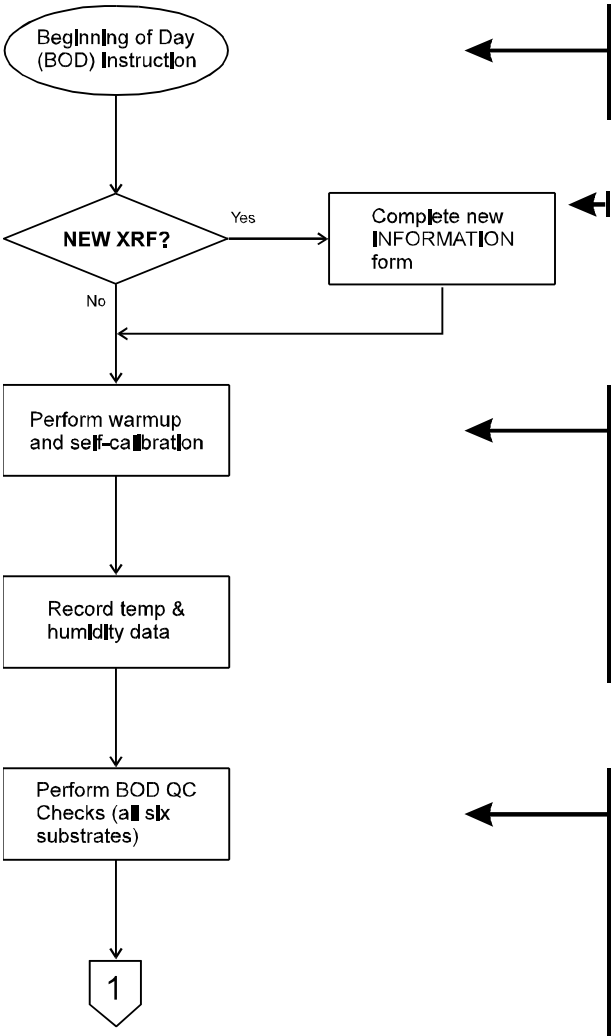
The Monitor will:

- Record all relevant data on the appropriate data forms or field notebooks.
- Work with the XRF Operator to help assure the protocols are followed.
- Work with the Testing Supervisor to help assure that protocols are followed and any deviations are properly documented.

The Supervisor will:

- Verify ID assignments on sample locations on the archive components.
- Assure that the data are collected as described in the protocol.
- Collect all forms and electronic data and make appropriate distributions to MRI, NIST, and QuanTech.
- Assure that resources are available to achieve planned testing.
- Oversee testing activities.
- Provide beginning-of-day and end-of-day instructions.
- Make primary decisions regarding any testing difficulties that may arise.
- Complete a new set of SAMPLE LOCATION CONDITION forms for:
    a. Sample locations previously identified as being in poor condition which already have completed records from the early spring 1995 testing.
    b. Any additional sample locations that are observed to be in poor condition.
- Provide the following forms:
    - Testing order list
    - Sample location condition
    - Standard readings
    - Control readings
    - Archive XRF information
    - Source age adjustment table

FLOW CHART SUMMARY | **DESCRIPTION DETAILS FOR XSIRIUS**



Instructions to Include: safety issues, field form use, archive layout and testing order, Sample ID numbering for electronic data capture, voice-call back verification of data recording by monitor, a blank display for negative lead results, and the use of a 30-sec reading adjusted for source age.

See form for needed information.

1. Allow to warm-up for 5 minutes.
2. Enter or verify:
   a. the selected file names to store the data
   b. the action level is set to 1.0 mg/cm^2
   c. any other user entered parameters.
3. Perform self-calibration as per the manual and manufacturer training directives using the manufacturer supplied calibration sample(s).

Summarize any user entered information and the calibration procedure on the instrument INFORMATION form.

BOD (or EOD) QC checks: test a total of 6 pre-placed blocks on a minimum of 12 inches of styrofoam support. **Please do not move blocks.**

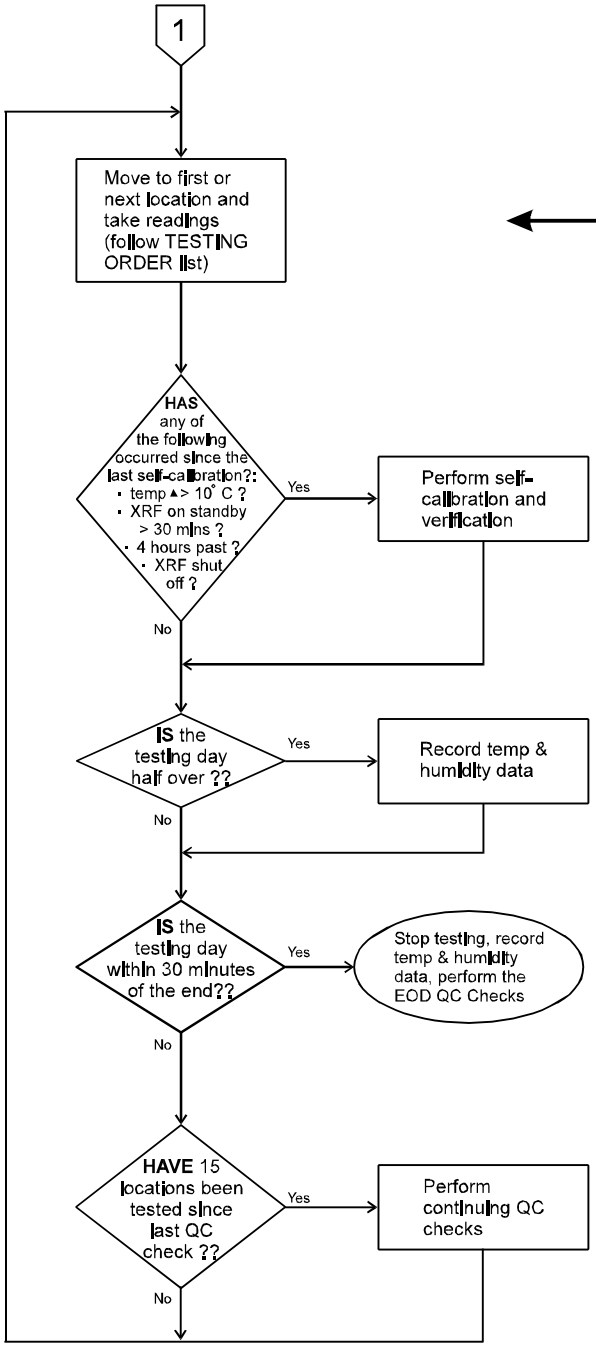
For each block, using a nominal 15-sec reading, test in the following order:

a. 1 reading, yellow NIST on block
b. 1 reading, red NIST on block. **IF** the block is wood, **THEN** collect 2 more readings using the red NIST on the block. Record extra readings on subsequent lines of the data form placing a diagonal line through the unused yellow and no NIST data blocks.
c. 1 reading, no NIST on block

Before moving to the next control block repeat a-c above using a nominal 30-sec reading adjusted for source age.

Record the data on the CONTROL READINGS form.

FLOW CHART SUMMARY



DESCRIPTION DETAILS FOR XSIRIUS

For each testing location, using a nominal 15-sec reading, test in the following order:

a. 1 reading, on X1
b. 1 reading, on bare (P1) + red NIST
c. 1 reading, on bare (P1)
d. 1 reading, on X2
e. 1 reading, on X3.

Record data on the STANDARD READINGS form. **IF** during any of the a-e testing above, the density is ≥ 4 **THEN:**

- complete all a-e testing using the nominal 15-sec reading time
- Change the testing mode to a nominal 30-sec reading adjusted for source age and repeat a-e testing above recording the data on the STANDARD READINGS form using a new row.

1. **IF** XRF off, allow to warmup for 5 minutes.
2. Enter or verify:
   a. the selected file names to store the data
   b. the action level is set to 1.0 mg/cm^2
   c. any other user entered parameters.
3. Perform self-calibration as per the manual and manufacturer training directives using the manufacturer supplied calibration sample(s).

Summarize changes to any user entered information on the Instrument INFORMATION form.

EOD QC checks are the same as BOD QC checks: follow same procedure as for BOD QC Checks.

Record temp and humidity in a field notebook.

Download any electronically stored data, verify successful transfer before clearing the memory to make room for more data.

Start next day at top of flow chart using the next sample to be tested as the first location.

Continuing QC checks, total of 2 blocks tested on a minimum of 12 inches styrofoam support. Use blocks matching same type as the last and next locations (if same type for both, test same block twice).

Follow same testing order and reading times as that used for BOD testing (i.e.: **First**, use the nominal 15-sec reading for testing a-c. **Second**, before moving to the next control block, repeat a-c testing using a nominal 30-sec reading.)

Archive XRF Instrument Information

Date _____ Testing Site _____

Contractor _____ Manufacturer _____

Model No. _____ Serial No. _____

XRF Operator (Printed name) _____

XRF Field Monitor (Printed name) _____

Source Material _____ Source Age or Date _____

Source Serial No. _____ Detector Type _____

Operating Parameters Used

Open shutter sampling time(s)? (fixed or variable) _____

If fixed, what is the duration time(s)? _____

Daily warm-up and calibration check used? (Briefly discuss) _____

_____

_____

Regulatory level value used for setting the XRF instrument? (Yes or No) _____

If yes, enter the value used _____

Other _____

_____

_____

Data File Names and Descriptions _____

_____

_____

_____

_____

☐ Check here to indicate the presence of additional comments on the back of this form.

Archive XRF Test Data -- Standard Readings

Page ____ of ____

Date ____________ XRF Device **XSIRIUS** ____________ Serial Number ____________

Location ID	Time of Measurement	Nominal Testing Time	Data Item	Paint Surface Reading: X1	Substrate + NIST Red, 1.02 mg/cm² Reading	Substrate Only Reading	Paint Surface Reading: X2	Paint Surface Reading: X3	Comments
			L-shell (mg/cm²)						
			L-shell (mg/cm²) Uncertainty						
			K-shell (mg/cm²)						
			K-shell (mg/cm²) Uncertainty						
			Density						
			Result (+, -, or ?)						
			Sequence Number						
			L-shell (mg/cm²)						
			L-shell (mg/cm²) Uncertainty						
			K-shell (mg/cm²)						
			K-shell (mg/cm²) Uncertainty						
			Density						
			Result (+, -, or ?)						
			Sequence Number						
			L-shell (mg/cm²)						
			L-shell (mg/cm²) Uncertainty						
			K-shell (mg/cm²)						
			K-shell (mg/cm²) Uncertainty						
			Density						
			Result (+, -, or ?)						
			Sequence Number						

Archive XRF Test Data -- Control Readings

Page _____ of _____

Date ______________ XRF Device **XSIRIUS** Serial Number ______________

QC Type: 1 = Initial Control Readings 2 = Continuing Control Readings 3 = Ending Control Readings

Block Type: M = Metal, W = Wood, B=Brick, D=Drywall, C=Concrete, P=Plaster

Identification		Time of Measurement	Nominal Testing Time	Data Item	Readings			Comments
QC Type	Block Type				Yellow, 3.53 mg/cm²	Red, 1.02 mg/cm²	No NIST SRM	
				L-shell (mg/cm²)				
				L-shell (mg/cm²) Uncertainty				
				K-shell (mg/cm²)				
				K-shell (mg/cm²) Uncertainty				
				Density				
				Result (+,-, or ?)				
				Sequence Number				
				L-shell (mg/cm²)				
				L-shell (mg/cm²) Uncertainty				
				K-shell (mg/cm²)				
				K-shell (mg/cm²) Uncertainty				
				Density				
				Result (+,-, or ?)				
				Sequence Number				
				L-shell (mg/cm²)				
				L-shell (mg/cm²) Uncertainty				
				K-shell (mg/cm²)				
				K-shell (mg/cm²) Uncertainty				
				Density				
				Result (+,-, or ?)				
				Sequence Number				

SUPPLEMENTAL PROTOCOLS FOR THE XL: PLASTER SAMPLE ADDITIONS

The following protocols are to be used for testing of plaster samples additions assigned to testing ID positions of 155-158.

NOTE: Record data on relevant data forms (see attached).

1. Warmup for 20 minutes.
2. Calibration checks, using 20-second mode, test both sides of factory samples (5). If value differs by 10% or 0.1 (whichever is greater, then go back to 1). Record results on back of data form.
3. **Testing (Use 20-second mode for QCs, follow truth table for samples)**
   a. QC-Control Blocks:
      a1. Wood — 3-red over bare; 3-bare
      a2. Plaster — 3-red over bare; 3-bare
   b. Sample 155 — 1-X1; 1-red over bare; 1-bare; 1-X2; 1-X3
   c. Sample 156 — 1-X1; 1-red over bare; 1-bare; 1-X2
   d. Sample 157 — 1-X1; 1-red over bare; 1-bare; 1-X2; 1-X3
   e. Sample 158 — 1-X1; 1-red over bare; 1-bare; 1-X2; 1-X3
   f. QC-Control Blocks
      a1. Plaster — 3-red over bare; 3-bare
      a2. Wood — 3-red over bare; 3-bare

Archive XRF Test Data -- Control Readings

Page _____ of _____

Date ______________ XRF Device **NITON XL** Serial Number ______________

XRF Operator (Printed Name) ______________ XRF Field Monitor (Printed name) ______________

QC Type: 1 = Initial Control Readings 2 = Continuing Control Readings 3 = Ending Control Readings Block Type: M = Metal, W = Wood, B=Brick, D=Drywall, C=Concrete, P=Plaster

Identification		Time of Measurement	Data Item	Readings						Comments
QC Type	Block Type			Red, 1.02 mg/cm²	Red, 1.02 mg/cm²	Red, 1.02 mg/cm²	No NIST SRM	No NIST SRM	No NIST SRM	
			Testing Time (sec)							
			L-shell (mg/cm²)							
			L-shell (mg/cm²) Uncertainty							
			Lower K-shell (mg/cm²)							
			Upper K-shell (mg/cm²)							
			Depth Index							
			Testing Time (sec)							
			L-shell (mg/cm²)							
			L-shell (mg/cm²) Uncertainty							
			Lower K-shell (mg/cm²)							
			Upper K-shell (mg/cm²)							
			Depth Index							
			Testing Time (sec)							
			L-shell (mg/cm²)							
			L-shell (mg/cm²) Uncertainty							
			Lower K-shell (mg/cm²)							
			Upper K-shell (mg/cm²)							
			Depth Index							

Archive XRF Test Data -- Standard Readings

Page _____ of _____

Date ______________ XRF Device **NITON XL** ______________ Serial Number ______________

XRF Operator (Printed Name) ______________ XRF Field Monitor (Printed name) ______________

Location ID	Time of Measurement	Data Item	Paint Surface Reading: X1	Substrate + NIST Red, 1.02 mg/cm² Reading	Substrate Only Reading	Paint Surface Reading: X2	Paint Surface Reading: X3	Comments
		Testing Time (sec)						
		L-shell (mg/cm²)						
		L-shell (mg/cm²) Uncertainty						
		Lower K-shell (mg/cm²)						
		Upper K-shell (mg/cm²)						
		Depth Index						
		Testing Time (sec)						
		L-shell (mg/cm²)						
		L-shell (mg/cm²) Uncertainty						
		Lower K-shell (mg/cm²)						
		Upper K-shell (mg/cm²)						
		Depth Index						
		Testing Time (sec)						
		L-shell (mg/cm²)						
		L-shell (mg/cm²) Uncertainty						
		Lower K-shell (mg/cm²)						
		Upper K-shell (mg/cm²)						
		Depth Index						

SUPPLEMENTAL PROTOCOLS FOR THE LPA-1: PLASTER SAMPLE ADDITIONS

The following protocols are to be used for testing of plaster samples additions assigned to testing ID positions of 155-158.

NOTE: Record data on relevant data forms (see attached).

1. Warmup for 20 minutes to assure self calibration occurs.
2. Calibration checks, using 20-second mode, test both sides of factory block. Record results on back of data form.
3. **Testing: <u>First</u> use Quick Mode at a single block or sample,**
   <u>then</u> use STD mode (20 seconds)
   a. QC-Control Blocks
      a1. Wood — 3-red over bare; 3-bare
      a2. Plaster — 3-red over bare; 3-bare
   b. Sample 155 — 1-X1; 1-red over bare; 1-bare; 1-X2; 1-X3
   c. Sample 156 — 1-X1; 1-red over bare; 1-bare; 1-X2
   d. Sample 157 — 1-X1; 1-red over bare; 1-bare; 1-X2; 1-X3
   e. Sample 158 — 1-X1; 1-red over bare; 1-bare; 1-X2; 1-X3
   f. QC-Control Blocks
      a1. Plaster — 3-red over bare; 3-bare
      a2. Wood — 3-red over bare; 3-bare

Supplemental QC Testing Data -- RMD

Page _____ of _____

XRF Model **LPA-1** Serial Number ____________ Source Serial Number ____________

XRF Operator (Printed Name) ____________ XRF Field Monitor (Printed name) ____________

Block Type: M = Metal, W = Wood, B=Brick, D=Drywall, C=Concrete, P=Plaster

Date	Block Type	Time of Measurement	Data Item	Readings						Comments
				Red, 1.02 mg/cm²	Red, 1.02 mg/cm²	Red, 1.02 mg/cm²	No NIST SRM	No NIST SRM	No NIST SRM	
			K-shell (mg/cm²) Quick Mode							
			K-shell (mg/cm²) STD Mode							
			K-shell (mg/cm²) Quick Mode							
			K-shell (mg/cm²) STD Mode							
			K-shell (mg/cm²) Quick Mode							
			K-shell (mg/cm²) STD Mode							
			K-shell (mg/cm²) Quick Mode							
			K-shell (mg/cm²) STD Mode							
			K-shell (mg/cm²) Quick Mode							
			K-shell (mg/cm²) STD Mode							
			K-shell (mg/cm²) Quick Mode							
			K-shell (mg/cm²) STD Mode							
			K-shell (mg/cm²) Quick Mode							
			K-shell (mg/cm²) STD Mode							
			K-shell (mg/cm²) Quick Mode							
			K-shell (mg/cm²) STD Mode							
			K-shell (mg/cm²) Quick Mode							
			K-shell (mg/cm²) STD Mode							
			K-shell (mg/cm²) Quick Mode							
			K-shell (mg/cm²) STD Mode							

Archive XRF Test Data -- Standard Readings

Page _____ of _____

Date ______________ XRF Device **RMD** ______________ Serial Number ______________

XRF Monitor (Printed Name) ______________________________

Location ID	Time of Measurement	Data Item	Paint Surface Reading: X1	Substrate + NIST Red, 1.02 mg/cm² Reading	Substrate Only Reading	Paint Surface Reading: X2	Paint Surface Reading: X3	Comments
		K-shell (mg/cm²) Quick Mode						
		K-shell (mg/cm²) STD Mode						
		Sequence Number						
		K-shell (mg/cm²) Quick Mode						
		K-shell (mg/cm²) STD Mode						
		Sequence Number						
		K-shell (mg/cm²) Quick Mode						
		K-shell (mg/cm²) STD Mode						
		Sequence Number						
		K-shell (mg/cm²) Quick Mode						
		K-shell (mg/cm²) STD Mode						
		Sequence Number						
		K-shell (mg/cm²) Quick Mode						
		K-shell (mg/cm²) STD Mode						
		Sequence Number						
		K-shell (mg/cm²) Quick Mode						
		K-shell (mg/cm²) STD Mode						
		Sequence Number						
		K-shell (mg/cm²) Quick Mode						
		K-shell (mg/cm²) STD Mode						
		Sequence Number						

SUPPLEMENTAL PROTOCOLS FOR THE LPA-1 AND XL: CONTROL BLOCKS

The following protocols are to be used for gathering independent sets of additional wood control block data on these instruments. Record data on Supplemental QC data form (see attached). Perform supplemental testing for each instrument using the indicated protocols for a total of 9 days.

Supplemental QC Testing - LPA-1

1. Warmup for 20 minutes to assure self calibration occurs. Record results on back of data form.

2. Calibration checks, using 20-second mode, test both sides of factory block.

3. **Testing: First use Quick Mode at a single block, then use STD mode (20 seconds) on that block**

   QC-Control Blocks

a1.	Wood	3-red over bare
		3-bare
a2.	Plaster	3-red over bare
		3-bare

Supplemental QC Testing - XL

1. Warmup for 20 minutes.

2. Calibration checks, using 20-second mode, test both sides of factory samples (5). If value differs by 10% or 0.1 (whichever is greater, then go back to 1). Record results on back of data form.

3. **Testing**: **Use 20-second mode**

   QC-Control Blocks:

a1.	Wood	3-red over bare
		3-bare
a2.	Plaster	3-red over bare
		3-bare

Supplemental QC Testing Data -- RMD

Page _____ of _____

XRF Model **LEADSTAR** Serial Number __________ Source Serial Number __________

XRF Operator (Printed Name) __________ XRF Field Monitor (Printed name) __________

Block Type: M = Metal, W = Wood, B=Brick, D=Drywall, C=Concrete, P=Plaster

Date	Block Type	Time of Measurement	Data Item	Readings						Comments
				Red, 1.02 mg/cm²	Red, 1.02 mg/cm²	Red, 1.02 mg/cm²	No NIST SRM	No NIST SRM	No NIST SRM	
			K-shell (mg/cm²) Quick Mode							
			K-shell (mg/cm²) STD Mode							
			K-shell (mg/cm²) Quick Mode							
			K-shell (mg/cm²) STD Mode							
			K-shell (mg/cm²) Quick Mode							
			K-shell (mg/cm²) STD Mode							
			K-shell (mg/cm²) Quick Mode							
			K-shell (mg/cm²) STD Mode							
			K-shell (mg/cm²) Quick Mode							
			K-shell (mg/cm²) STD Mode							
			K-shell (mg/cm²) Quick Mode							
			K-shell (mg/cm²) STD Mode							
			K-shell (mg/cm²) Quick Mode							
			K-shell (mg/cm²) STD Mode							
			K-shell (mg/cm²) Quick Mode							
			K-shell (mg/cm²) STD Mode							
			K-shell (mg/cm²) Quick Mode							
			K-shell (mg/cm²) STD Mode							
			K-shell (mg/cm²) Quick Mode							
			K-shell (mg/cm²) STD Mode							

Supplemental QC Testing Data -- Niton

Page _____ of _____

XRF Model **XL** ____________ Serial Number ____________ Source Serial Number ____________

XRF Operator (Printed Name) ____________ XRF Field Monitor (Printed name) ____________

Block Type: M = Metal, W = Wood, B=Brick, D=Drywall, C=Concrete, P=Plaster

Date	Block Type	Time of Measurement	Data Item	Readings						Comments
				Red, 1.02 mg/cm²	Red, 1.02 mg/cm²	Red, 1.02 mg/cm²	No NIST SRM	No NIST SRM	No NIST SRM	
			Testing Time (sec)							
			L-shell (mg/cm²)							
			L-shell (mg/cm²) Uncertainty							
			Lower K-shell (mg/cm²)							
			Upper K-shell (mg/cm²)							
			Depth Index							
			Testing Time (sec)							
			L-shell (mg/cm²)							
			L-shell (mg/cm²) Uncertainty							
			Lower K-shell (mg/cm²)							
			Upper K-shell (mg/cm²)							
			Depth Index							
			Testing Time (sec)							
			L-shell (mg/cm²)							
			L-shell (mg/cm²) Uncertainty							
			Lower K-shell (mg/cm²)							
			Upper K-shell (mg/cm²)							
			Depth Index							

SUBCONTRACTER TESTING PROTOCOLS FOR THE LPA-1

General Responsibilities

The XRF Operator will:

- Handle the instruments and make the measurements.
- Work with the Testing Monitor to see that the appropriate data are recorded.
- Work with the Testing Supervisor to help assure that protocols are followed and any deviations are properly documented.
- Download data to a computer, verify that the transfer was successful, and delete all data files containing archive data from the XRF electronic memory.
- Report to the Testing Supervisor any indications of deteriorating samples observed during testing.
- Set the STD mode of operation to a nominal 30-second measurement time when using this mode.

The Monitor will:

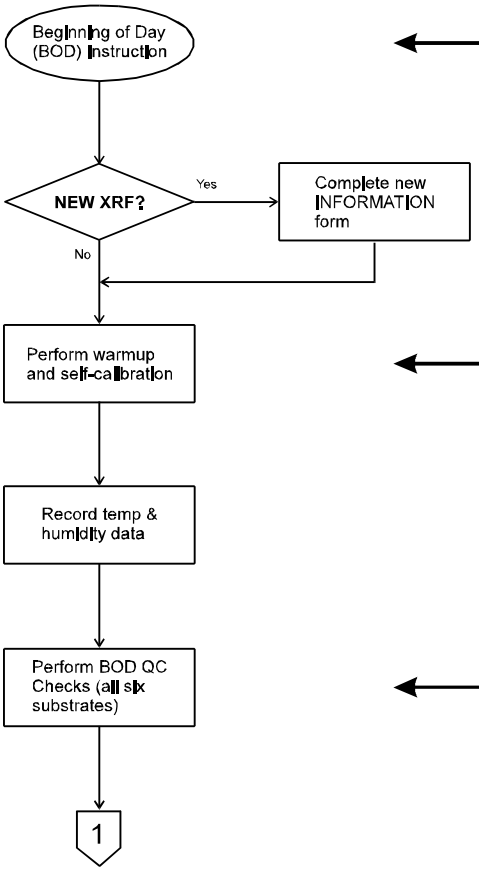
- Record all relevant data on the appropriate data forms or field notebooks.
- Work with the XRF Operator to help assure the protocols are followed.
- Work with the Testing Supervisor to help assure that protocols are followed and any deviations are properly documented.

The Supervisor will:

- Verify ID assignments on sample locations on the archive components.
- Assure that the data are collected as described in the protocol.
- Collect all forms and electronic data and make appropriate distributions to MRI, and QuanTech.
- Assure that resources are available to achieve planned testing.
- Oversee testing activities.
- Provide beginning-of-day and end-of-day instructions.
- Make primary decisions regarding any testing difficulties that may arise.
- Provide the following forms:

- Testing order list
- Standard readings
- Control readings
- Archive XRF information
- Source age adjustment table

FLOW CHART SUMMARY | **DESCRIPTION DETAILS FOR RMD**



Instructions to include: safety issues, field form use, archive layout and testing order, sequence numbering for electronic data capture, and voice-call back verification of data recording by monitor.

See form for needed information.

1. Enter date and time.
2. Allow to warmup at least 20 minutes to assure that the auto self-calibration occurs (automatically done every 15 minutes).
3. Perform calibration check using STD Mode set to a nominal 30 seconds: Test both bare and painted sides of factory supplied wood substrate. Record results on INFORMATION form.
4. Set Quick Mode Testing using an action level of 1.0 mg/cm^2.

BOD (or EOD) QC checks: test a total of 6 pre-placed blocks on a minimum of 12 inches of styrofoam support. Perform testing in order 1-6 as indicated on the styrofoam support using a 30-sec STD Mode reading, and record the sequence number for each reading on the form to identify electronic data entries.

For each block, excluding wood, test in the following order and record data in the appropriate columns using a single row of the Control Readings form:

a. 1 reading, yellow NIST on block
b. 1 reading, red NIST on block
c. 1 reading, no NIST on block

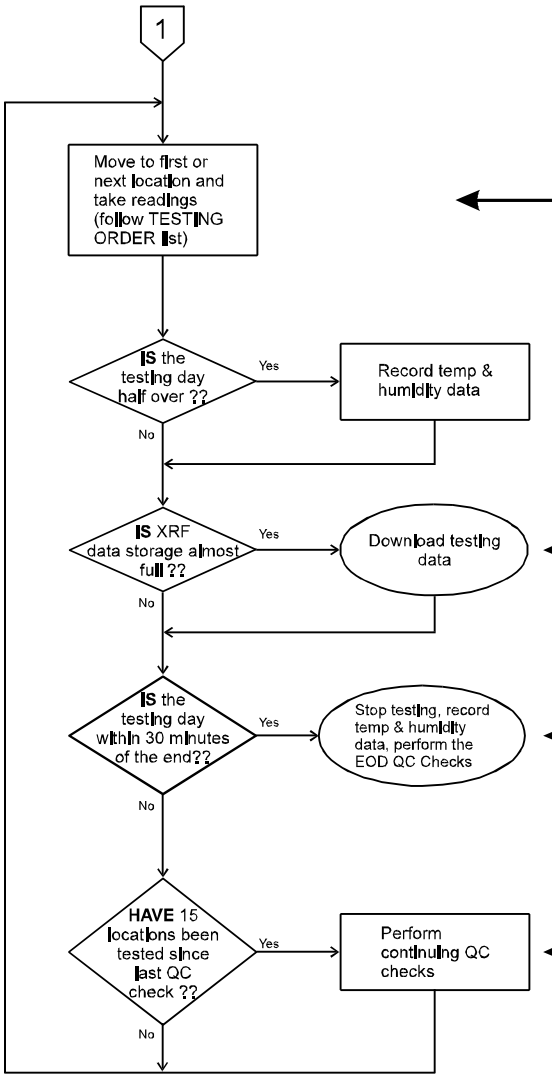
NOTE: For all control blocks except wood, fill in all three data entries in each row. The wood exception is explained below.

For the wood substrate, test in the following order and record data in the appropriate columns using three rows of the Control Readings form:

a. 1 reading, yellow NIST on the control block
b. 3 consecutive readings, red NIST on the control block
c. 1 reading, no NIST on the control block

NOTE: since 3 readings with red NIST on wood control blocks are taken consecutively, it is necessary to blank out 4 data entries in the 3 rows of the form as follows: for row 1 blank out the "No NIST SRM" entry, for row 2 blank out the "yellow NIST" and the "No NIST SRM" entries, and for row 3, blank out the "yellow NIST" entry.

FLOW CHART SUMMARY **DESCRIPTION DETAILS FOR RMD**



Test all of the samples at each testing location first in the Quick mode, then repeat using the STD Mode set to a nominal 30 seconds.

For each testing location, in each mode, test in the following order:

a. 1 reading, on X1
b. 1 reading, on bare (P1) + red NIST
c. 1 reading, on bare (P1)
d. 1 reading, on X2
e. 1 reading, on X3.

Record data on the STANDARD READINGS form.

Periodically measure the time required to take a reading from start to data display and record it in the field notebook.

Download all electronically stored data and verify successful transfer before clearing memory to make room for more data.

EOD QC checks are the same as BOD QC checks: follow same procedure as for BOD QC Checks.

Record temp and humidity in a field notebook.

Download any electronically stored data, verify successful transfer before clearing memory.

Start next day at top of flow chart using the next sample to be tested as the first location.

Continuing QC checks, total of 2 blocks tested on a minimum of 12 inches styrofoam support. Use blocks matching same type as the last and next locations (if same type for both, test same block twice).

Follow the same testing order, reading times and data recording directives as described for BOD QC checks.

Archive XRF Instrument Information

Date _____ Testing Site _____

Contractor _____ Manufacturer _____

Model No. _____ Serial No. _____

XRF Operator (Printed Name) _____

Source Material _____ Source Age or Date _____

Source Serial No. _____ Detector Type _____

Operating Parameters Used

Open shutter sampling time(s)? (fixed or variable) _____

If fixed, what is the duration time(s)? _____

Daily warm-up and calibration check used? (Briefly discuss) _____

_____

_____

Regulatory level value used for setting the XRF instrument? (Yes or No) _____

If yes, enter the value used _____

Other _____

_____

_____

Data File Names and Descriptions _____

_____

_____

_____

_____

_____

☐ Check here to indicate the presence of additional comments on the back of this form.

Archive XRF Test Data -- Standard Readings

Page _____ of _____

Date ______________ XRF Device RMD, LPA-1 ______________ Serial Number ______________

XRF Monitor (Printed Name) ______________________________

Location ID	Time of Measurement	Data Item	Paint Surface Reading: X1	Substrate + NIST Red, 1.02 mg/cm² Reading	Substrate Only Reading	Paint Surface Reading: X2	Paint Surface Reading: X3	Comments
		K-shell (mg/cm²) Quick Mode						
		Sequence Number						
		K-shell (mg/cm²) STD Mode						
		Sequence Number						
		K-shell (mg/cm²) Quick Mode						
		Sequence Number						
		K-shell (mg/cm²) STD Mode						
		Sequence Number						
		K-shell (mg/cm²) Quick Mode						
		Sequence Number						
		K-shell (mg/cm²) STD Mode						
		Sequence Number						
		K-shell (mg/cm²) Quick Mode						
		Sequence Number						
		K-shell (mg/cm²) STD Mode						
		Sequence Number						
		K-shell (mg/cm²) Quick Mode						
		Sequence Number						
		K-shell (mg/cm²) STD Mode						
		Sequence Number						

Archive XRF Test Data -- Control Readings

Page _____ of _____

Date ______________ XRF Device RMD, LPA-1 ______________ Serial Number ______________________

XRF Monitor (Printed Name) ____

QC Type: 1 = Initial Control Readings 2 = Continuing Control Readings 3 = Ending Control Readings

Block Type: M = Metal, W = Wood, B=Brick, D=Drywall, C=Concrete, P=Plaster

Identification		Time of Measurement	Data Item	Readings			Comments
QC Type	Block Type			Yellow, 3.53 mg/cm²	Red, 1.02 mg/cm²	No NIST SRM	
			K-shell (mg/cm²) STD Mode				
			Sequence Number				
			K-shell (mg/cm²) STD Mode				
			Sequence Number				
			K-shell (mg/cm²) STD Mode				
			Sequence Number				
			K-shell (mg/cm²) STD Mode				
			Sequence Number				
			K-shell (mg/cm²) STD Mode				
			Sequence Number				
			K-shell (mg/cm²) STD Mode				
			Sequence Number				
			K-shell (mg/cm²) STD Mode				
			Sequence Number				
			K-shell (mg/cm²) STD Mode				
			Sequence Number				
			K-shell (mg/cm²) STD Mode				
			Sequence Number				
			K-shell (mg/cm²) STD Mode				
			Sequence Number				

SUBCONTRACTOR TESTING PROTOCOLS FOR THE LEADSTAR

General Responsibilities

The XRF Operator will:

- Handle the instruments and make the measurements.
- Work with the Testing Monitor to see that the appropriate data are recorded.
- Work with the Testing Supervisor to help assure that protocols are followed and any deviations are properly documented.
- Download data to a computer and verify that the transfer was successful.
- Report to the Testing Supervisor any indications of deteriorating samples observed during testing.

The Monitor will:

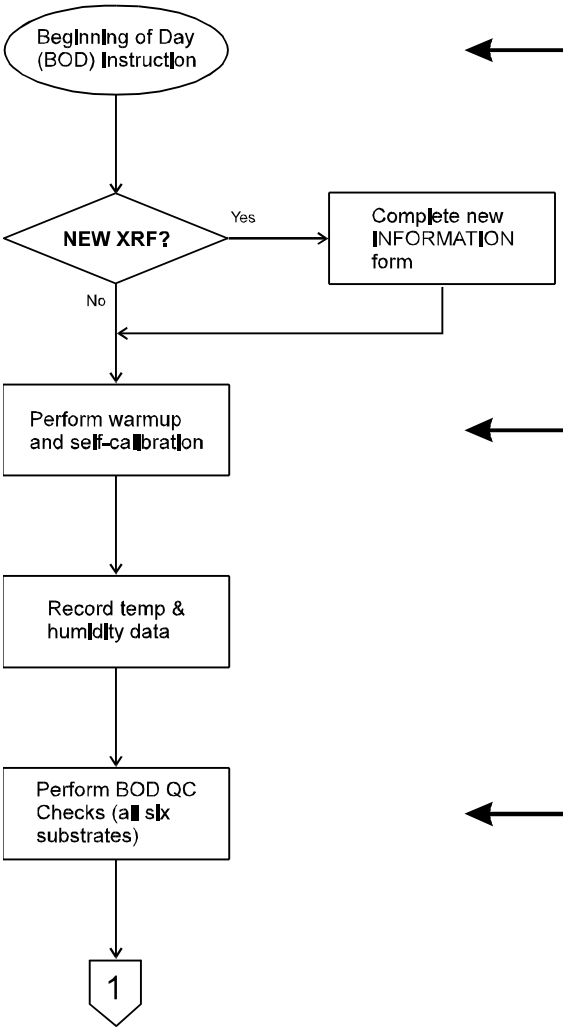
- Record all relevant data on the appropriate data forms or field notebooks.
- Work with the XRF Operator to help assure the protocols are followed.
- Work with the Testing Supervisor to help assure that protocols are followed and any deviations are properly documented.

The Supervisor will:

- Verify ID assignments on sample locations on the archive components.
- Assure that the data are collected as described in the protocol.
- Collect all forms and electronic data and make appropriate distributions to MRI and QuanTech.
- Assure that resources are available to achieve planned testing.
- Oversee testing activities.
- Provide beginning-of-day and end-of-day instructions.
- Make primary decisions regarding any testing difficulties that may arise.
- Provide the following forms:

  - Testing order list
  - Archive XRF information
  - Standard readings
  - Control readings

FLOW CHART SUMMARY | **DESCRIPTION DETAILS FOR XSIRIUS**



Instructions to include: safety issues, field form use, archive layout and testing order, Sample ID numbering for electronic data capture, voice-call back verification of data recording by monitor, a blank display for negative lead results, and the use of a 30-sec reading adjusted for source age.

See form for needed information.

1. Allow to warm-up for 5 minutes.
2. Enter or verify:
   a. the selected file names to store the data
   b. the action level is set to 1.0 mg/cm^2
   c. any other user entered parameters.
3. Perform self-calibration as per the manual and manufacturer training directives using the manufacturer supplied calibration sample(s).

Summarize any user entered information and the calibration procedure on the Instrument INFORMATION form.

BOD (or EOD) QC checks: test a total of 6 pre-placed blocks on a minimum of 12 inches of styrofoam support. **Please do not move blocks.**

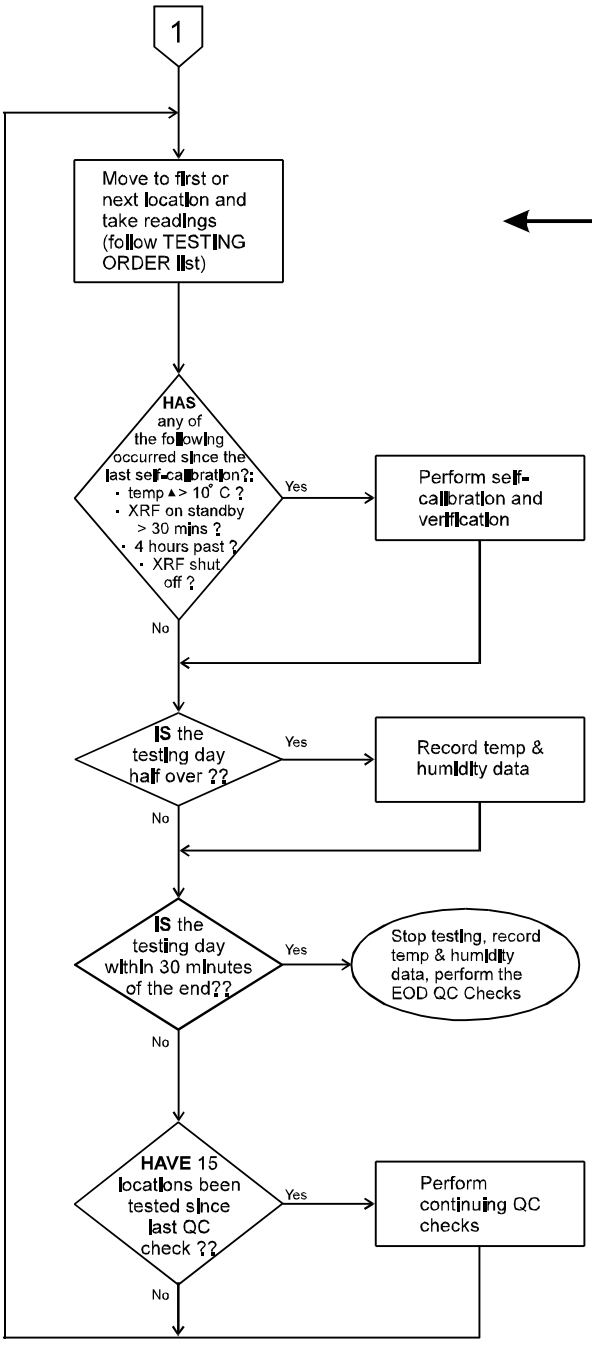
For each block, using a nominal 15-sec reading, test in the following order:

a. 1 reading, yellow NIST on block
b. 1 reading, red NIST on block. **IF** the block is wood, **THEN** collect 2 more readings using the red NIST on the block. Record extra readings on subsequent lines of the data form placing a diagonal line through the unused yellow and no NIST data blocks.
c. 1 reading, no NIST on block

Before moving to the next control block repeat a-c above using a nominal 30-sec reading adjusted for source age.

Record the data on the CONTROL READINGS form.

FLOW CHART SUMMARY



DESCRIPTION DETAILS FOR XSIRIUS

For each testing location, using a nominal 15-sec reading, test in the following order:

a. 1 reading, on X1
b. 1 reading, on bare (P1) + red NIST
c. 1 reading, on bare (P1)
d. 1 reading, on X2
e. 1 reading, on X3.

Record data on the STANDARD READINGS form. **IF** during any of the a-e testing above, the density is ≥ 4 **THEN:**

- complete all a-e testing using the nominal 15-sec reading time
- Change the testing mode to a nominal 30-sec reading adjusted for source age and repeat a-e testing above recording the data on the STANDARD READINGS form using a new row.

1. **IF** XRF off, allow to warmup for 5 minutes.
2. Enter or verify:
   a. the selected file names to store the data
   b. the action level is set to 1.0 mg/cm^2
   c. any other user entered parameters.
3. Perform self-calibration as per the manual and manufacturer training directives using the manufacturer supplied calibration sample(s).

Summarize changes to any user entered information on the Instrument INFORMATION form.

EOD QC checks are the same as BOD QC checks: follow same procedure as for BOD QC Checks.

Record temp and humidity in a field notebook.

Download any electronically stored data, verify successful transfer before clearing the memory to make room for more data.

Start next day at top of flow chart using the next sample to be tested as the first location.

Continuing QC checks, total of 2 blocks tested on a minimum of 12 inches styrofoam support. Use blocks matching same type as the last and next locations (if same type for both, test same block twice).

Follow same testing order and reading times as that used for BOD testing (**i.e.: First**, use the nominal 15-sec reading for testing a-c. **Second**, before moving to the next control block, repeat a-c testing using a nominal 30-sec reading.)

Archive XRF Instrument Information

Date _____ Testing Site _____

Contractor _____ Manufacturer _____

Model No. _____ Serial No. _____

XRF Operator (Printed Name) _____

Source Material _____ Source Age or Date _____

Source Serial No. _____ Detector Type _____

Operating Parameters Used

Open shutter sampling time(s)? (fixed or variable) _____

If fixed, what is the duration time(s)? _____

Daily warm-up and calibration check used? (Briefly discuss) _____

_____

_____

Regulatory level value used for setting the XRF instrument? (Yes or No) _____

If yes, enter the value used _____

Other _____

_____

_____

Data File Names and Descriptions _____

_____

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_____

☐ Check here to indicate the presence of additional comments on the back of this form.

Archive XRF Test Data -- Standard Readings

Page ____ of ____

Date ____________ XRF Device XSIRIUS, LeadStar ____________ Serial Number ____________

Location ID	Time of Measurement	Nominal Testing Time	Data Item	Paint Surface Reading: X1	Substrate + NIST Red. 1.02 mg/cm² Reading	Substrate Only Reading	Paint Surface Reading: X2	Paint Surface Reading: X3	Comments
			L-shell (mg/cm²)						
			L-shell (mg/cm²) Uncertainty						
			K-shell (mg/cm²)						
			K-shell (mg/cm²) Uncertainty						
			Density						
			Result (OK, NG, or ?)						
			Sequence Number						
			L-shell (mg/cm²)						
			L-shell (mg/cm²) Uncertainty						
			K-shell (mg/cm²)						
			K-shell (mg/cm²) Uncertainty						
			Density						
			Result (OK, NG, or ?)						
			Sequence Number						
			L-shell (mg/cm²)						
			L-shell (mg/cm²) Uncertainty						
			K-shell (mg/cm²)						
			K-shell (mg/cm²) Uncertainty						
			Density						
			Result (OK, NG, or ?)						
			Sequence Number						

Archive XRF Test Data -- Control Readings

Page _____ of _____

Date ______________ XRF Device XSIRIUS, LeadStar Serial Number ______________

QC Type: 1 = Initial Control Readings 2 = Continuing Control Readings 3 = Ending Control Readings

Block Type: M = Metal, W = Wood, B=Brick, D=Drywall, C=Concrete, P=Plaster

Identification		Time of Measurement	Nominal Testing Time	Data Item	Readings			Comments
QC Type	Block Type				Yellow, 3.53 mg/cm²	Red, 1.02 mg/cm²	No NIST SRM	
				L-shell (mg/cm²)				
				L-shell (mg/cm²) Uncertainty				
				K-shell (mg/cm²)				
				K-shell (mg/cm²) Uncertainty				
				Density				
				Result (OK, NG, or ?)				
				Sequence Number				
				L-shell (mg/cm²)				
				L-shell (mg/cm²) Uncertainty				
				K-shell (mg/cm²)				
				K-shell (mg/cm²) Uncertainty				
				Density				
				Result (OK, NG, or ?)				
				Sequence Number				
				L-shell (mg/cm²)				
				L-shell (mg/cm²) Uncertainty				
				K-shell (mg/cm²)				
				K-shell (mg/cm²) Uncertainty				
				Density				
				Result (OK, NG, or ?)				
				Sequence Number				

General Responsibilities

The XRF Operator will:

- Perform all manufacturer-recommended warm-up procedures and quality control checks, and work with the Testing Monitor and the Testing Supervisor to document that the procedures and checks were done, what the outcomes were, and, if appropriate, what actions were taken.
- Handle the instruments and make the measurements.
- Work with the Testing Monitor to see that the appropriate data are recorded.
- Work with the Testing Supervisor to help assure that protocols are followed and any deviations are properly documented.
- Download data to a computer and verify that the transfer was successful.
- Report to the Testing Supervisor any indications of deteriorating samples observed.

The Monitor will:

- Record all relevant data on the appropriate data forms or field notebooks.
- Work with the XRF Operator to help assure the protocols are followed.
- Work with the Testing Supervisor to help assure that protocols are followed and any deviations are properly documented.

The Supervisor will:

- Verify ID assignments on sample locations on the archive components.
- Assure that the data are collected as described in the protocol.
- Collect all forms and electronic data and make appropriate distributions to MRI, QuanTech, and NIST.
- Assure that resources are available to achieve planned testing.
- Oversee testing activities.
- Provide beginning-of-day and end-of-day instructions.
- Make primary decisions regarding any testing difficulties that may arise.
- Make sure that all testing is done safely, that all personnel on site wear dosimeter badges during testing, that dosimeter badges are collected and analyzed after testing is completed, and that results of dosimeter badge analysis are relayed to the badge wearers.
- Take radiation measurements periodically around each instrument being used for testing.
- Provide blank forms to the Testing Monitor (blank forms to be delivered to the Testing Supervisor by QuanTech) and review forms filled out by the Testing Monitor for completeness. The forms for the following will be used during testing:
    - Testing order list
    - Archive XRF information
    - Standard readings
    - Control readings

Beginning of Work Session

At the beginning of the work session, be sure that there is a fresh battery pack in the Analyzer. It is good practice to transfer previous data to the personal computer before beginning the day's work. If the previous data have not been transferred, refer to the "Downloading" section below. If the data have been transferred, reset the Analyzer memory by pressing the RESET button. The SET button confirms the reset.

Verify Keypad Settings

Before taking any measurements, a few system configuration checks should be made:

1. Check that the Manual Shutter Lock and Keylock (if provided) are unlocked.
2. Check that any old data in the LPA-1 has been downloaded if a Memory Reset is to be performed.
3. Check that the Abatement Level is properly set. The Abatement Level setting can be checked by turning on the LPA-1 with the Trigger, then pressing the SET key. The LPA-1 will then display ABATE AT XX mg. If the setting is not correct, the value can be incremented with the SET key.
4. Verify that the LPA-1 is in the desired operating mode. When in Standard Mode, the display will show STD MODE XX Sec, where XX is the number of seconds previously programmed for the length of the measurement. If a Quick Mode measurement is desired, change operating modes by pressing the SELECT MODE button.
5. Verify the setting of the LPA-1 system clock by allowing the LPA-1 to power down, then pressing the NEW UNIT key for two seconds. Press the SET key to confirm. The display is formatted as month-day-year-time. The time should be accurate within 15 minutes. To exit this mode, press the NEW UNIT key again and allow the LPA-1 to shut off.

Check Calibration Sample

Before and after each job, it is good quality control practice to do a system performance check. It is recommended that this be done by taking three readings on the Calibration Test Block provided with the instrument and on another lead-free wood block. The tested value of the yellow test pad is recorded on the back of the block. A reading of appropriate length (see below) should be taken on both the Calibration Block and also on any block of unpainted wood or drywall that the user has at hand to provide a zero lead reference. *Note: The back side of the calibration is not a good zero reference because some amount of the lead on the front of the block is liable to be detected through the thin wood block.*

Use the Date of Receipt of the LPA-1 from the factory as a starting point to correct the Performance Test reading for half-life decay as follows:

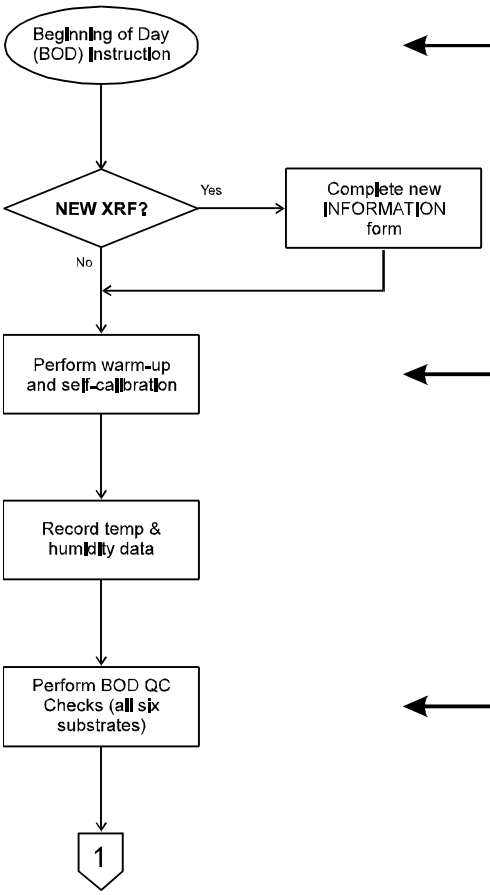
0 to 120 days (4 mos.)—Test at 30 seconds
121 to 175 days (6 mos.)—Test at 40 seconds
176 to 270 days (9 mos.)—Test at 50 seconds
270 to 455 days (15 mos.)—Test at 60 seconds
Beyond 15 months—Time to replace the Source

Ninety-five percent of the time, the value of any single calibration reading should be the value of the calibration block ±0.3 mg/cm^2.

Alternately the user may wish to perform 60-second measurements throughout the life of the source. This eliminates guesswork and keeps field procedures simple. In this case, the user should expect to see readings that are the value of the calibration block ±0.2 mg/cm^2.

If the Lead Readings were outside of the correct range, wait at least 5 minutes for the Analyzer's automatic Calibration Check to occur. After the Check is completed, reread the Standard Block. Should the readings again fall outside the acceptable range, call RMD before continuing with the inspection.

FLOW CHART SUMMARY | **DESCRIPTION DETAILS FOR LPA-1**



Instructions to include: safety issues, field form use, archive layout and testing order, sequence numbering for electronic data capture, and voice-call back verification of data recording by monitor.

See form for needed information.

1. Enter date and time.
2. Perform warm-up and calibration check procedure as specified by the manufacturer (see attachment).
3. Set Quick Mode Testing using an action level of 1.0 mg/cm^2.

BOD (or EOD) QC checks: test a total of 6 pre-placed blocks on a minimum of 12 inches of styrofoam support. Perform testing in order 1-6 as indicated on the styrofoam support. Record the sequence number for each reading on the form to identify electronic data entries as described below.

For each block, excluding wood, test using the nominal 30 second STD mode in the following order and record data in the appropriate columns using a single row of the Control Readings form:

a. 1 reading, yellow NIST on block
b. 1 reading, red NIST on block
c. 1 reading, no NIST on block

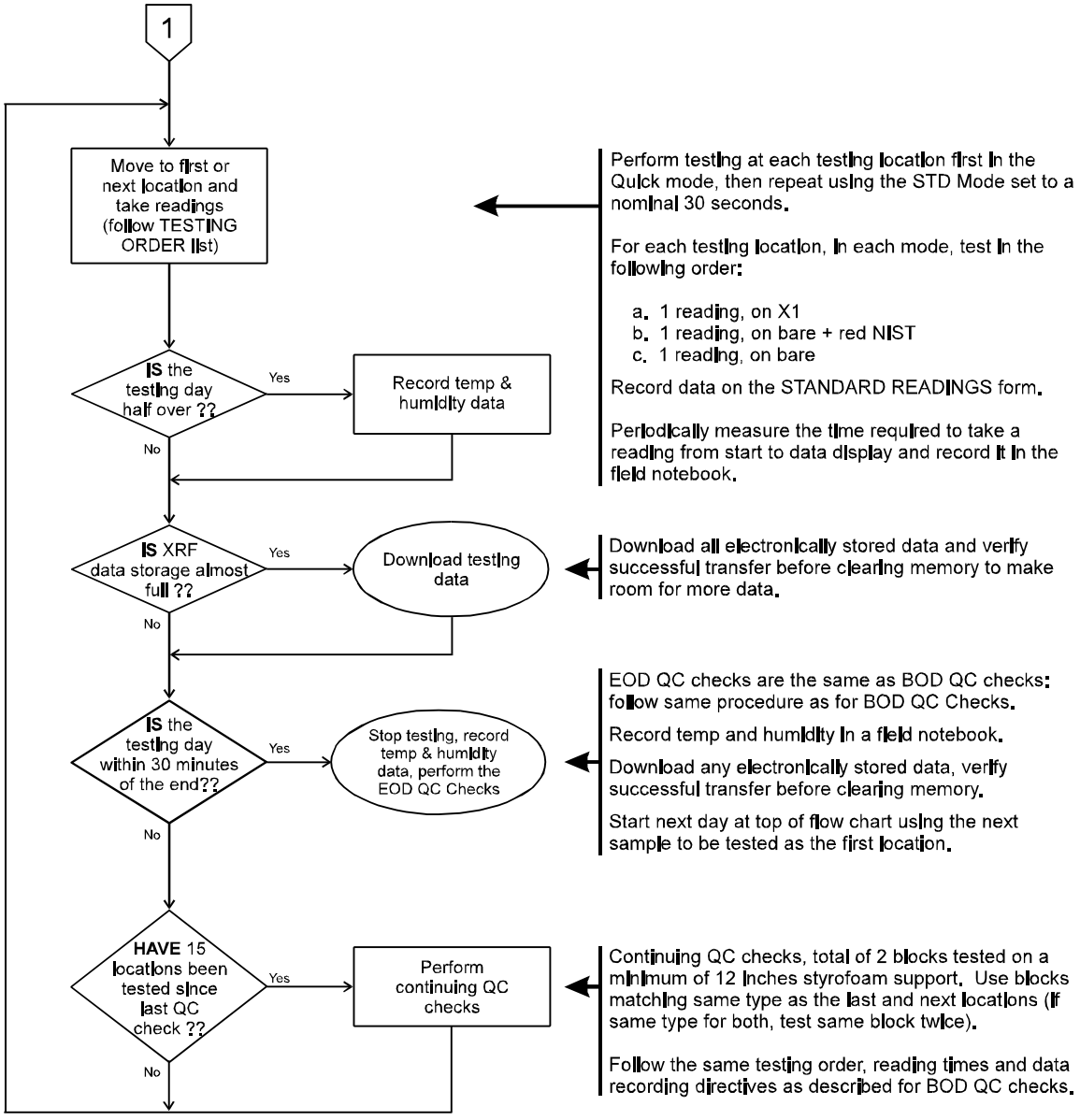
NOTE: For all control blocks except wood, fill in all three data entries in each row. The wood exception is explained below.

For the wood substrate, test using two modes in the order a-c below. First, test using the Quick mode. Second, test using a nominal 30 second mode. Record data in the appropriate columns using three rows of the Control Readings form for each test mode:

a. 1 reading, yellow NIST on the control block
b. 3 consecutive readings, red NIST on the control block
c. 1 reading, no NIST on the control block

NOTE FOR EACH MODE: since 3 readings with red NIST on wood control blocks are taken consecutively, it is necessary to blank out 4 data entry column blocks in 3 consecutive rows of the form as follows: for row 1, blank out the "No NIST SRM" entry block, for row 2, blank out the "yellow NIST" and the "No NIST SRM" entry blocks, and for row 3, blank out the "yellow NIST" entry block.

FLOW CHART SUMMARY **DESCRIPTION DETAILS FOR LPA-1**



Archive XRF Instrument Information

Date _____ Testing Site _____

Contractor _____ Manufacturer _____

Model No. _____ Serial No. _____

XRF Operator (Printed Name) _____

Source Material _____ Source Age or Date _____

Source Serial No. _____ Detector Type _____

Software Version No. _____

Operating Parameters Used

Open shutter sampling time(s)? (fixed or variable) _____

If fixed, what is the duration time(s)? _____

Daily warm-up and calibration check used? (Briefly discuss) _____

_____

_____

Regulatory level value used for setting the XRF Instrument? (Yes or No) _____

If yes, enter the value used _____

Other _____

_____

_____

Data File Names and Descriptions _____

_____

_____

_____

_____

_____

☐ Check here to indicate the presence of additional comments on the back of this form.

Archive XRF Test Data -- Standard Readings

Page _____ of _____

Date ______________ XRF Device LPA-1______________ Serial Number______________

XRF Operator (Printed Name) ______________ XRF Monitor (Printed Name) ______________

Location ID	Time of Measurement	Data Item	Paint Surface Reading: X1	Substrate + NIST Red, 1.02 mg/cm² Reading	Substrate Only Reading	Comments
		K-shell (mg/cm²) Quick Mode				
		Sequence Number				
		K-shell (mg/cm²) STD Mode				
		Sequence Number				
		K-shell (mg/cm²) Quick Mode				
		Sequence Number				
		K-shell (mg/cm²) STD Mode				
		Sequence Number				
		K-shell (mg/cm²) Quick Mode				
		Sequence Number				
		K-shell (mg/cm²) STD Mode				
		Sequence Number				
		K-shell (mg/cm²) Quick Mode				
		Sequence Number				
		K-shell (mg/cm²) STD Mode				
		Sequence Number				
		K-shell (mg/cm²) Quick Mode				
		Sequence Number				
		K-shell (mg/cm²) STD Mode				
		Sequence Number				

Archive XRF Test Data -- Control Readings

Page _____ of _____

Date ______________ XRF Device LPA-1 ______________ Serial Number ______________

XRF Monitor (Printed Name) ______________ XRF Operator (Printed Name) ______________

QC Type: 1 = Initial Control Readings 2 = Continuing Control Readings 3 = Ending Control Readings

Block Type: M = Metal, W = Wood, B=Brick, D=Drywall, C=Concrete, P=Plaster

Identification		Time of Measurement	Test Mode (STD or QUICK)	Data Item	Readings			Comments
QC Type	Block Type				Yellow, 3.53 mg/cm²	Red, 1.02 mg/cm²	No NIST SRM	
				K-shell (mg/cm²)				
				Sequence Number				
				K-shell (mg/cm²)				
				Sequence Number				
				K-shell (mg/cm²)				
				Sequence Number				
				K-shell (mg/cm²)				
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				K-shell (mg/cm²)				
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				K-shell (mg/cm²)				
				Sequence Number				
				K-shell (mg/cm²)				
				Sequence Number				
				K-shell (mg/cm²)				
				Sequence Number				
				K-shell (mg/cm²)				
				Sequence Number				
				K-shell (mg/cm²)				
				Sequence Number				

General Responsibilities

The XRF Operator will:

- Perform all manufacturer recommended warm-up procedures and quality control checks, and work with the Testing Monitor and the Testing Supervisor to document that the procedures and checks were done, what the outcomes were, and, if appropriate, what actions were taken.
- Handle the instruments and make the measurements.
- Work with the Testing Monitor to see that the appropriate data are recorded.
- Work with the Testing Supervisor to help assure that protocols are followed and any deviations are properly documented.
- Download data to a computer and verify that the transfer was successful.
- Report to the Testing Supervisor any indications of deteriorating samples observed.

The Monitor will:

- Record all relevant data on the appropriate data forms or field notebooks.
- Work with the XRF Operator to help assure the protocols are followed.
- Work with the Testing Supervisor to help assure that protocols are followed and any deviations are properly documented.

The Supervisor will:

- Verify ID assignments on sample locations on the archive components.
- Assure that the data are collected as described in the protocol.
- Collect all forms and electronic data and make appropriate distributions to MRI, QuanTech, and NIST.
- Assure that resources are available to achieve planned testing.
- Oversee testing activities.
- Provide beginning-of-day and end-of-day instructions.
- Make primary decisions regarding any testing difficulties that may arise.
- Make sure that all testing is done safely, that all personnel on-site wear dosimeter badges during testing, that dosimeter badges are collected and analyzed after testing is completed, and that results of dosimeter badge analysis are relayed to the badge wearers.
- Take radiation measurements periodically around each instrument being used for testing.
- Provide blank forms to the Testing Monitor (blank forms to be delivered to the Testing Supervisor by QuanTech) and review forms filled out by the Testing Monitor for completeness. The forms for the following will be used during testing:
    - Testing order list
    - Archive XRF information
    - Standard readings
    - Control readings

Energy Calibration and Standard Operating Check

Complete both of these procedures:

- at the beginning of each work day
- if the Analyzer has been off for more than one hour
- if the Analyzer has been exposed to a temperature change of > 10°C/20°F
- if the unit has been subjected to shock or been damaged

At the end of the day, perform the Standard Operating Check (without running the Energy Calibration) to verify that the Analyzer performed properly using that day's energy calibration.

Energy Calibration Procedure

Perform the Energy Calibration before the Operating Check (unless you are running the "end of work day" check). This procedure will automatically calibrate the spectrometer function.

Lay the flat safety cover on a level, stable surface and carefully set the probe in the cover.

1. Choose your application.
2. From the Ready screen press 5, "Options."
3. Press 1, "Energy Calibration."
4. Follow the screen prompts.

Standard Operating Check

The two-part standard operating check verifies the Analyzer's operation with respect to element X-ray response and analytical background. The operating check is discussed in detail in Chapter 2.

X-Ray Response

X-ray response is checked by making a 50-second measurement on a pure Pb sample, then viewing Pb intensity data (Pb intensity should be between 0.95 and 1.05). See Chapter 2 for detailed instructions on how to conduct the x-ray response check. If intensity is not > 0.95 and < 1.05, check the probe window for contamination and make sure the Pb sample is centered over the aperture, then rerun the check. If intensity is still off, turn to Chapter 8 of the Operator's Manual and run a spectrum energy calibration check.

Note: The x-ray response check may result in an "Algorithm did not converge" message in the Pb in Paint Chips and Pb in Soil applications. This should be expected, since the pure Pb intensity is very different from that produced by a "normal" paint chip or soil sample. For the x-ray response check, we are only interested in the intensity data—not the analysis result.

Analytical Background Check

The background check verifies proper operation of the spectrometer and can alert you to dirt or contamination on the probe window. Always protect the probe window since dirt, moisture, or contamination on it can compromise your analysis.

The check for Pb in paint chips is run on the "blank," which is a sample cup containing SiO_2. To conduct the check, set up the Uniblock in the lab configuration (steps 1-4) and present the sample as described. Analysis results on this sample should show a Pb value within ±5 standard deviations of zero (if your results are in units rather than standard deviations, press 5, "OPTS" on the Results screen and then 3, "Show STD"). If results are not within ±5 standard deviations of zero, you must run the "Acquire Background" procedure. If you don't see any results, you have Display Thresholds enabled. To disable thresholds, go to the Ready screen, select 5, "Options," then 6, "More Options," then 5, "Disable display thresholds."

Warning: During any analysis, there is a *minimal* risk of radiation exposure. While an analysis is in progress,

- Do not remove the probe from the Uniblock (or other measurement surface)
- Do not look at or touch the probe window.

To abort any analysis in progress, press CONT or the red button on the probe.

---

1. Lay the Uniblock flat-side up so that you can read the words "Aluminum" and "Steel."
2. Slide the probe clamp into the cutout end of the Uniblock.
3. Place the probe in the Uniblock with the probe window facing up and the probe handle cable pointing away from the probe clamp.
4. Carefully tighten the clamp knob until the probe is secure. Do not force the knob to turn since this can damage the clamp. Verify that the probe is secure.

5. Set the sample shield on the probe. The cutout should be centered over the probe window. If the cutout is off center, turn the shield around and recheck the position of the probe window.
6. Secure the sample shield on the probe with the bungee cord.
7. Place the 30mm sample positioning ring over the probe aperture.
8. Visually inspect the sample and verify that surfaces which will be presented to the probe are clean.
9. Place the SiO_2 sample in the positioning ring on the probe. Close the shield cover.
10. Measure the sample for at least 60 seconds (adjusted for source decay). Use Table 1 in Chapter 2 to determine adjusted analysis time.
11. Label and store the result. This is discussed in detail in Chapter 2.

When you accept the label (press CONT from the label screen), the analysis results will be displayed. Compare the Pb assay value with its standard deviation (STD) value. It must be < 5 standard deviations above or below "zero." If it is not, check for contamination on the probe window or the sample cup. If contamination does not appear to be the problem, run the "Acquire Background" procedure.

Acquire Background

If your analysis showed a Pb value of more than ±5 standard deviations from zero on the SiO_2 sample, you should acquire the background.

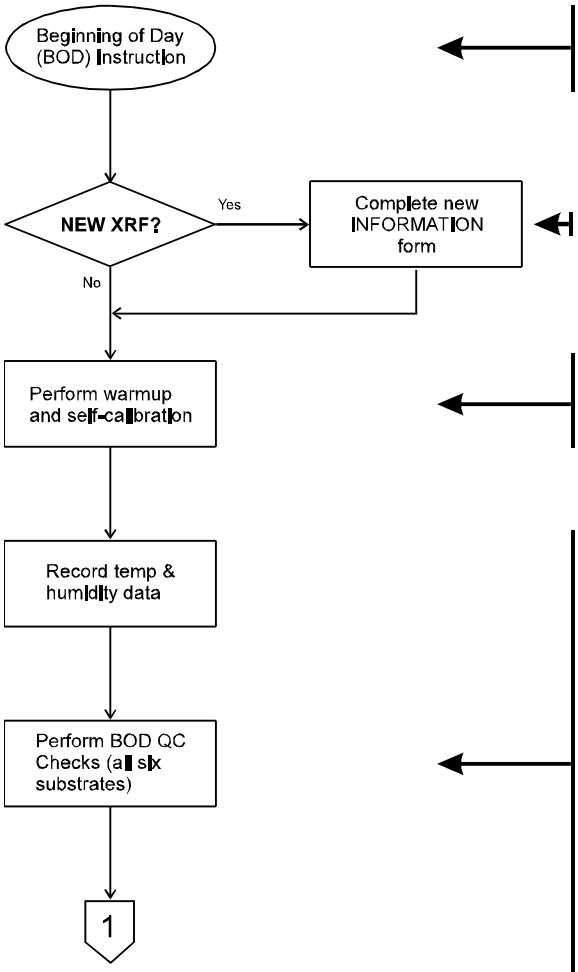
1. Go to the Ready screen
2. Press 5, "More."
3. Press 2, "Acquire a Background."
4. Place the SiO_2 and then the Teflon sample on the probe as requested.

After you have acquired the background, run the background check again to verify the acquisition was successful. If the Analyzer still does not check out, contact TN Technologies at 800-736-0801 (U.S. only) or 512-388-9100.

Known Standard Check

We suggest that you run (and store) at least one analysis on a known standard before beginning your actual sample measurement. For the Pb in Paint Chips application, this check should be run on the Fisher SRS 013 sample, for 200 live seconds, adjusted for source decay. Results should be .07% ± 0.01%.

FLOW CHART SUMMARY **DESCRIPTION DETAILS FOR Pb ANALYZER**



Instructions to include: safety issues, field form use, archive layout and testing order, sequence numbering for electronic data capture, and voice-call back verification of data recording by monitor.

See form for needed information.

1. Enter date and time.
2. Allow to warmup as specified by the manufacturer.
3. Perform calibration check as specified by the manufacturer (see attachment).

BOD (or EOD) QC checks: test a total of 6 pre-placed blocks on a minimum of 12 inches of styrofoam support. Perform testing in order 1-6 as indicated on the styrofoam support using a nominal 15 second real time reading, and record the sequence number for each reading on the form to identify electronic data entries.

For each block, excluding wood, test in the following order and record data in the appropriate columns using a single row of the Control Readings form:

a. 1 reading, yellow NIST on block
b. 1 reading, red NIST on block
c. 1 reading, no NIST on block

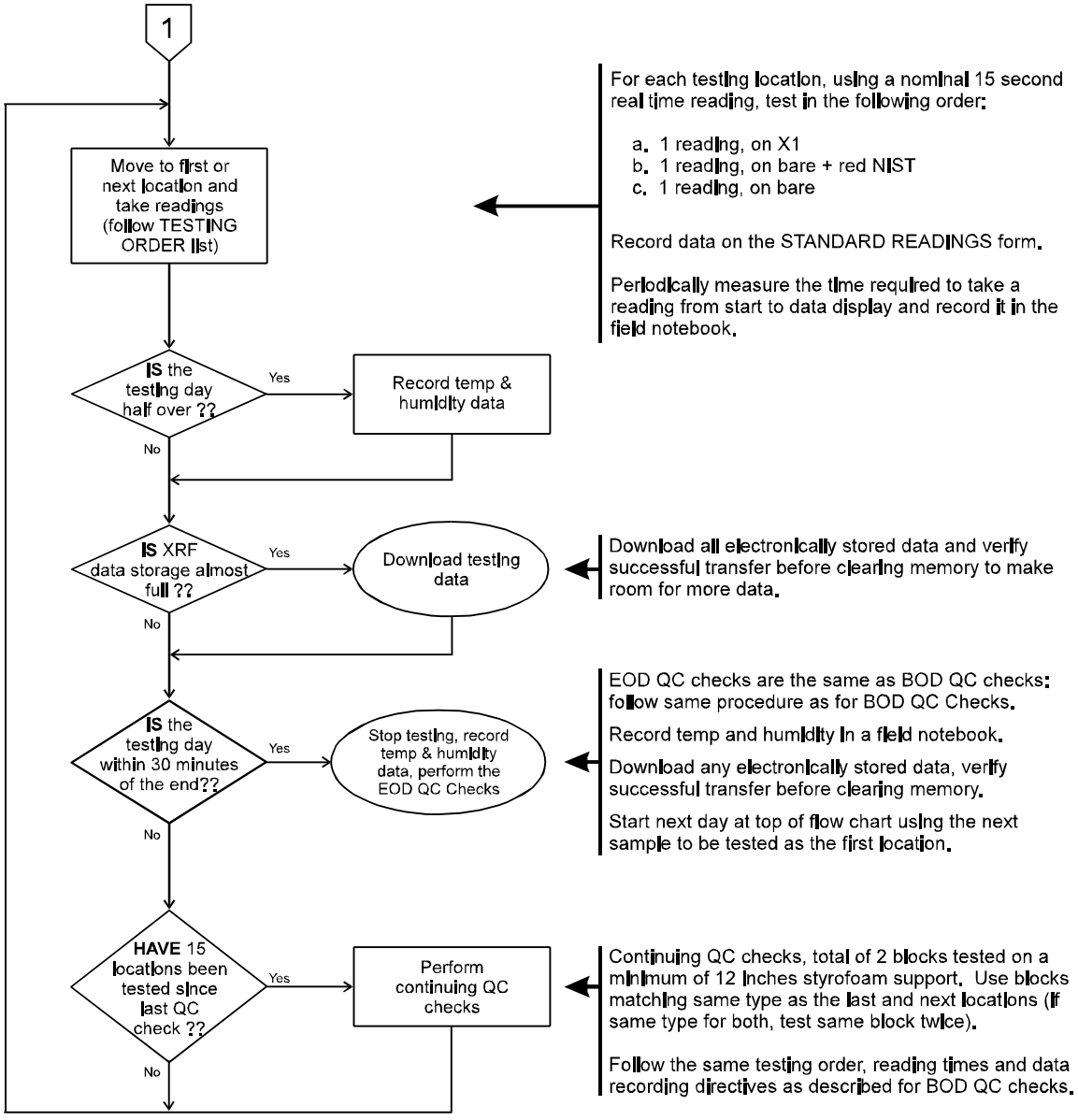
NOTE: For all control blocks except wood, fill in all three data entries in each row. The wood exception is explained below.

For the wood substrate, test in the following order and record data in the appropriate columns using three rows of the Control Readings form:

a. 1 reading, yellow NIST on the control block
b. 3 consecutive readings, red NIST on the control block
c. 1 reading, no NIST on the control block

NOTE: since 3 readings with red NIST on wood control blocks are taken consecutively, it is necessary to blank out 4 data entry column blocks in 3 consecutive rows of the form as follows: for row 1, blank out the "No NIST SRM" entry block, for row 2, blank out the "yellow NIST" and the "No NIST SRM" entry blocks, and for row 3, blank out the "yellow NIST" entry block.

FLOW CHART SUMMARY **DESCRIPTION DETAILS FOR Pb ANALYZER**



Archive XRF Instrument Information

Date _____ Testing Site _____

Contractor _____ Manufacturer _____

Model No. _____ Serial No. _____

XRF Operator (Printed Name) _____

Source Material _____ Source Age or Date _____

Source Serial No. _____ Detector Type _____

Software Version No. _____

Operating Parameters Used

Open shutter sampling time(s)? (fixed or variable) _____

If fixed, what is the duration time(s)? _____

Daily warm-up and calibration check used? (Briefly discuss) _____

_____

_____

Regulatory level value used for setting the XRF Instrument? (Yes or No) _____

If yes, enter the value used _____

Other _____

_____

_____

Data File Names and Descriptions _____

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☐ Check here to indicate the presence of additional comments on the back of this form.

Archive XRF Test Data -- Standard Readings

Page _____ of _____

Date ______________ XRF Device _Pb Analyzer___________ Serial Number____________________

XRF Operator (Printed Name) ______________________________ XRF Monitor (Printed Name) ______________________________

Location ID	Time of Measurement	Data Item	Paint Surface Reading: X1	Substrate + NIST Red, 1.02 mg/cm² Reading	Substrate Only Reading	Comments
		K-shell (mg/cm²)				
		L-shell (mg/cm²)				
		Sequence Number				
		K-shell (mg/cm²)				
		L-shell (mg/cm²)				
		Sequence Number				
		K-shell (mg/cm²)				
		L-shell (mg/cm²)				
		Sequence Number				
		K-shell (mg/cm²)				
		L-shell (mg/cm²)				
		Sequence Number				
		K-shell (mg/cm²)				
		L-shell (mg/cm²)				
		Sequence Number				
		K-shell (mg/cm²)				
		L-shell (mg/cm²)				
		Sequence Number				
		K-shell (mg/cm²)				
		L-shell (mg/cm²)				
		Sequence Number				

Archive XRF Test Data -- Control Readings

Page _____ of _____

Date _______________ XRF Device _Pb Analyzer___________ Serial Number ______________________

XRF Monitor (Printed Name) ________________________________ XRF Operator (Printed Name) ________________________________

QC Type: 1 = Initial Control Readings 2 = Continuing Control Readings 3 = Ending Control Readings

Block Type: M = Metal, W = Wood, B=Brick, D=Drywall, C=Concrete, P=Plaster

Identification		Time of Measurement	Test Mode (STD or QUICK)	Data Item	Readings			Comments
QC Type	Block Type				Yellow, 3.53 mg/cm²	Red, 1.02 mg/cm²	No NIST SRM	
				K-shell (mg/cm²)				
				L-shell (mg/cm²)				
				Sequence Number				
				K-shell (mg/cm²)				
				L-shell (mg/cm²)				
				Sequence Number				
				K-shell (mg/cm²)				
				L-shell (mg/cm²)				
				Sequence Number				
				K-shell (mg/cm²)				
				L-shell (mg/cm²)				
				Sequence Number				
				K-shell (mg/cm²)				
				L-shell (mg/cm²)				
				Sequence Number				
				K-shell (mg/cm²)				
				L-shell (mg/cm²)				
				Sequence Number				
				K-shell (mg/cm²)				
				L-shell (mg/cm²)				
				Sequence Number				

General Responsibilities

The XRF Operator will:

- Perform all manufacturer recommended warm-up procedures and quality control checks, and work with the Testing Monitor and the Testing Supervisor to document that the procedures and checks were done, what the outcomes were, and, if appropriate, what actions were taken.
- Handle the instruments and make the measurements.
- Work with the Testing Monitor to see that the appropriate data are recorded.
- Work with the Testing Supervisor to help assure that protocols are followed and any deviations are properly documented.
- Download data to a computer and verify that the transfer was successful.
- Report to the Testing Supervisor any indications of deteriorating samples observed.

The Monitor will:

- Record all relevant data on the appropriate data forms or field notebooks.
- Work with the XRF Operator to help assure the protocols are followed.
- Work with the Testing Supervisor to help assure that protocols are followed and any deviations are properly documented.

The Supervisor will:

- Verify ID assignments on sample locations on the archive components.
- Assure that the data are collected as described in the protocol.
- Collect all forms and electronic data and make appropriate distributions to MRI, QuanTech, and NIST.
- Assure that resources are available to achieve planned testing.
- Oversee testing activities.
- Provide beginning-of-day and end-of-day instructions.
- Make primary decisions regarding any testing difficulties that may arise.
- Make sure that all testing is done safely, that all personnel on site wear dosimeter badges during testing, that dosimeter badges are collected and analyzed after testing is completed, and that results of dosimeter badge analysis are relayed to the badge wearers.
- Take radiation measurements periodically around each instrument being used for testing.
- Provide blank forms to the Testing Monitor (blank forms to be delivered to the Testing Supervisor by QuanTech) and review forms filled out by the Testing Monitor for completeness. The forms for the following will be used during testing:
    - Testing order list
    - Archive XRF information
    - Standard readings
    - Control readings

Calibration Check Procedure for MicroLead I—Revision 4

I. Diagnostics and Source Dating

A. Press power switch to turn on analyzer. Verify that no FAULT codes are displayed after diagnostic countdown is completed.

B. Check TODAY'S DATE. Check SOURCE DATE. If you are going to store field readings, press the CLEAR MEMORY function key for 3 seconds to erase the operator's memory space.

II. Set Zero Reference to Remove Bias

A. Press the ZERO STANDARD function key (verify "PROBE" is shown on the display).

B. With the probe centered on the *Gypsum Zero Standard*, pull the probe trigger and hold until at least four Read Cycles are completed (nine is better). Verify that the display is showing 0.0 mg/cm^2.

III. Recording Calibration Check Data

A. Verify Accuracy (Bias) for Zero Lead Level.

1. With the probe centered on the *Gypsum Zero Standard*, take at least five replicate 1st Read Cycle measurements (ten is better). Record readings in logbook. Compute average ($\bar{x}$) and standard deviation (σ).

2. If $\bar{x}$ does not equal 0.0 mg/cm^2, ±0.1 mg/cm^2, start over at II.

B. Verify Precision for Zero Lead Level.

If σ does not equal 0.3 mg/cm^2 or less, take additional measurements to improve your statistical database. Recalculate $\bar{x}$ and σ. If σ still does not meet factory specification, call Warrington's service department at 512-251-7771.

Repeat step III at least every 2 hours throughout the day and at the very end of the day. Record calibration check readings in your logbook each time. Also, approximately twice per month verify that the "Radiation Density" of the (black) *Concrete Zero Standard* is reading "22".

C. Verify Accuracy (Bias) for Positive Lead Level.

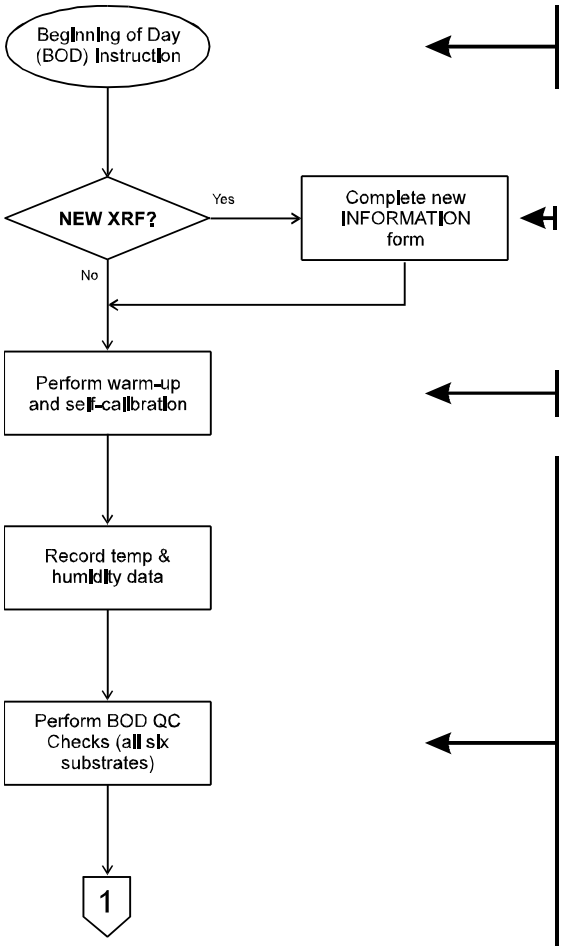
1. Place a calibrated Lead Standard on the *Gypsum Zero Standard*. With the probe centered, take five replicate 1st Read Cycle measurements (ten is better). Record readings in logbook. Compute $\bar{x}$ and σ.

2. If $\bar{x}$ does not equal the stated lead (Pb) value, ± 0.1 mg/cm^2, take additional measurements to improve your statistical database. Recalculate $\bar{x}$ and σ. If $\bar{x}$ still does not equal the stated lead (Pb) value, ± 0.1 mg/cm^2, follow manufacturer's instructions for LEAD STANDARD. **Note: Do not press the LEAD STANDARD function key unless you have received specific instructions on how to properly use this function.**

D. Verify Precision for Positive Lead Level.

If σ does not equal 0.3 mg/cm^2 or less, take additional measurements to improve your statistical database. Recalculate $\bar{x}$ and σ. If σ still does not meet factory specification, call Warrington's service department at 512-251-7771.

FLOW CHART SUMMARY | **DESCRIPTION DETAILS FOR MICROLEAD I**



Instructions to include: safety issues, field form use, archive layout and testing order, and voice-call back verification of data recording by monitor.

See form for needed information.

Perform warm-up and calibration check procedures as specified by the manufacturer (see attachment).

BOD (or EOD) QC checks: test a total of 6 pre-placed blocks on a minimum of 12 inches of styrofoam support. Perform testing in order 1-6 as indicated on the styrofoam support. Record the sequence number for each reading on the form to identify electronic data entries as described below.

For each block, excluding wood, test in the following order and record data in the appropriate columns using a single row of the Control Readings form:

a. 1 reading, yellow NIST on block
b. 1 reading, red NIST on block
c. 1 reading, no NIST on block

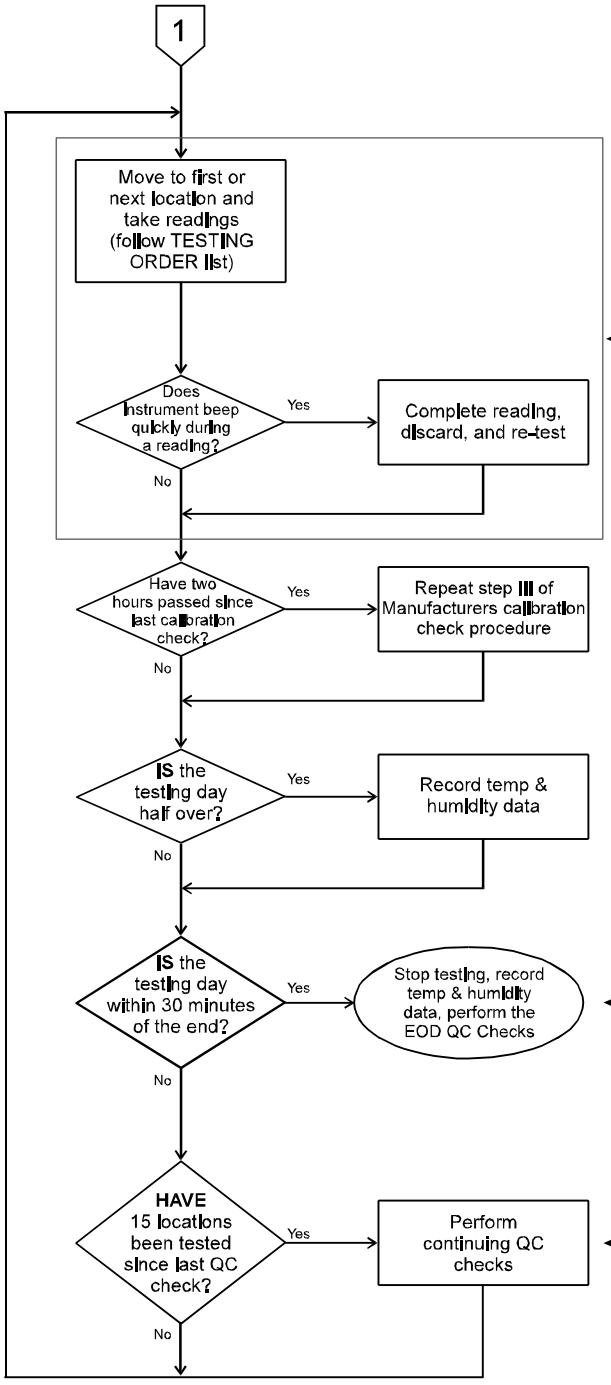
NOTE: For all control blocks except wood, fill in all three data entries in each row. The wood exception is explained below.

For the wood substrate, test in the order a-c below. Record data in the appropriate columns using three rows of the Control Readings form:

a. 1 reading, yellow NIST on the control block
b. 3 consecutive readings (3 independent trigger pull events), red NIST on the control block
c. 1 reading, no NIST on the control block

NOTE: since 3 readings with red NIST on wood control blocks are taken consecutively, it is necessary to blank out 4 data entry column blocks in 3 consecutive rows of the form as follows: for row 1, blank out the "No NIST SRM" entry block, for row 2, blank out the "yellow NIST" and the "No NIST SRM" entry blocks, and for row 3, blank out the "yellow NIST" entry block.

FLOW CHART SUMMARY **DESCRIPTION DETAILS FOR MICROLEAD I**



Perform testing at each testing location using a single nominal 15 second trigger pull event.

For each testing location, test in the following order:

a. 1 reading, on X1
b. 1 reading, on bare + red NIST
c. 1 reading, on bare

Record data on the STANDARD READINGS form.

Periodically measure the time required to take a reading from start to data display and record it in the field notebook.

If instrument beeps quickly during a reading, a major density change has been detected. Under these conditions, complete the reading and record the value in the comments column of the form (do not record in the normal data entry block). Then, re-test and record the result in the normal manner.

EOD QC checks are the same as BOD QC checks: follow same procedure as for BOD QC Checks.

Record temp and humidity in a field notebook.

Start next day at top of flow chart using the next sample to be tested as the first location.

Continuing QC checks, total of 2 blocks tested on a minimum of 12 inches styrofoam support. Use blocks matching same type as the last and next locations (if same type for both, test same block twice).

Follow the same testing order, reading times and data recording directives as described for BOD QC checks.

Archive XRF Instrument Information

Date _____ Testing Site _____

Contractor _____ Manufacturer _____

Model No. _____ Serial No. _____

XRF Operator (Printed Name) _____

Source Material _____ Source Age or Date _____

Source Serial No. _____ Detector Type _____

Software Version No. _____

Operating Parameters Used

Open shutter sampling time(s)? (fixed or variable) _____

If fixed, what is the duration time(s)? _____

Daily warm-up and calibration check used? (Briefly discuss) _____

_____

_____

Regulatory level value used for setting the XRF Instrument? (Yes or No) _____

If yes, enter the value used _____

Other _____

_____

_____

Data File Names and Descriptions _____

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☐ Check here to indicate the presence of additional comments on the back of this form.

Archive XRF Test Data -- Standard Readings

Page _____ of _____

Date ______________ XRF Device Microlead I ______________ Serial Number ____________________

XRF Operator (Printed Name) ______________________________ XRF Monitor (Printed Name) ______________________________

Location ID	Time of Measurement	Data Item	Paint Surface Reading: X1	Substrate + NIST Red, 1.02 mg/cm² Reading	Substrate Only Reading	Comments
		K-shell (mg/cm²)				
		K-shell (mg/cm²)				
		K-shell (mg/cm²)				
		K-shell (mg/cm²)				
		K-shell (mg/cm²)				
		K-shell (mg/cm²)				
		K-shell (mg/cm²)				
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		K-shell (mg/cm²)				
		K-shell (mg/cm²)				
		K-shell (mg/cm²)				
		K-shell (mg/cm²)				

Archive XRF Test Data -- Control Readings

Page _____ of _____

Date ______________ XRF Device Microlead I ______________ Serial Number ______________

XRF Monitor (Printed Name) ______________ XRF Operator (Printed Name) ______________

QC Type: 1 = Initial Control Readings 2 = Continuing Control Readings 3 = Ending Control Readings Block Type: M = Metal, W = Wood, B=Brick, D=Drywall, C=Concrete, P=Plaster

Identification		Time of Measurement	Data Item	Readings			Comments
QC Type	Block Type			Yellow, 3.53 mg/cm²	Red, 1.02 mg/cm²	No NIST SRM	
			K-shell (mg/cm²)				
			K-shell (mg/cm²)				
			K-shell (mg/cm²)				
			K-shell (mg/cm²)				
			K-shell (mg/cm²)				
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			K-shell (mg/cm²)				
			K-shell (mg/cm²)				
			K-shell (mg/cm²)				

TESTING PROTOCOLS FOR THE XK-3

General Responsibilities

The XRF Operator will:

- Handle the instruments and make the measurements.
- Work with the Testing Monitor to see that the appropriate data are recorded.
- Work with the Testing Supervisor to help ensure that protocols are followed and any deviations are properly documented.
- Download data to a computer and verify that the transfer was successful.
- Report to the Testing Supervisor any indications of deteriorating samples observed during testing.

The Monitor will:

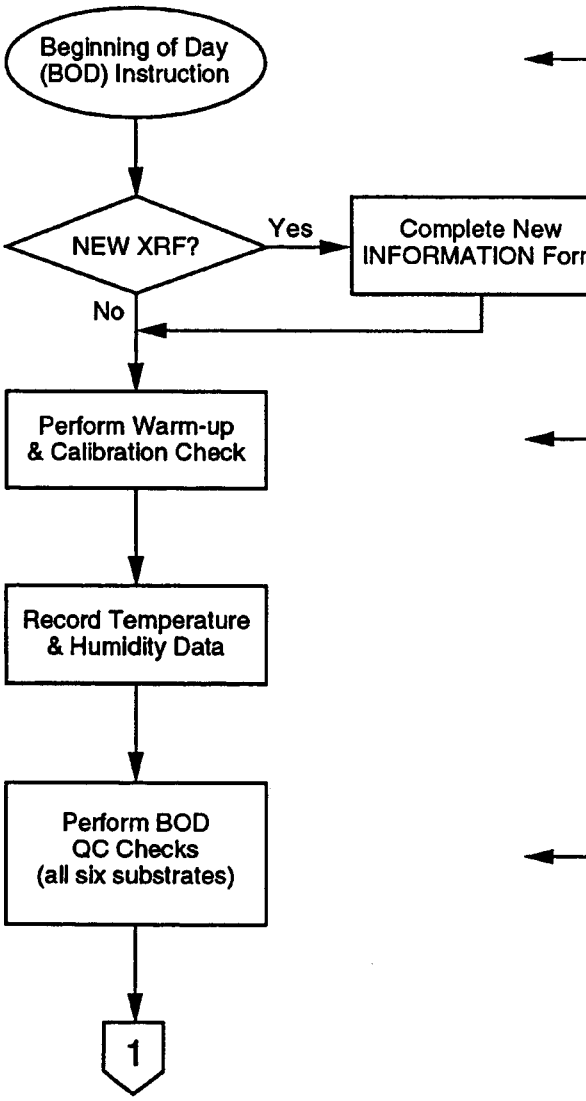
- Record all relevant data on the appropriate data forms or field notebooks.
- Work with the XRF Operator to help ensure the protocols are followed.
- Work with the Testing Supervisor to help ensure that protocols are followed and any deviations are properly documented.

The Supervisor will:

- Verify ID assignments on sample locations on the archive components.
- Ensure that the data are collected as described in the protocol.
- Collect all forms and electronic data and make appropriate distributions to MRI, NIST, and QuanTech.
- Ensure that resources are available to achieve planned testing.
- Oversee testing activities.
- Provide beginning-of-day and end-of-day instructions.
- Make primary decisions regarding any testing difficulties that may arise.
- Complete a new set of SAMPLE LOCATION CONDITION forms for:
    a. Sample locations previously identified as being in poor condition which already have completed records from the early spring 1995 testing.
    b. Any additional sample locations that are observed to be in poor condition.
- Provide the following forms:
    - Testing order list
    - Sample location condition
    - Standard readings
    - Control readings
    - Archive XRF information
    - Source age adjustment table

Testing Protocols for the PGT XK-3 Lead-In-Paint Analyzer

Flow Chart Summary



Description Details for PGT XK-3

Instructions to include: safety issues, field form use, archive layout and testing order, sample ID numbering for electronic data capture, voice-call back verification of data recording by monitor, and a blank display for negative lead results.

See form for needed information.

1. Connect the battery pack to the XK-3 unit.
2. Find the lock switch underneath the handle and push it forward.
3. Allow to warm-up (stabilize) for 10 minutes.

Note: The larger the temperature difference between where the XK-3 has been stored and where the testing is to be done, the longer it may take to stabilize.

4. Perform calibration check as per the manual and manufacturer training directives using the manufacturer supplied calibration sample(s).

Summarize any user entered information and the calibration procedure on the instrument INFORMATION form.

BOD (or EOD) QC checks: test a total of 6 pre-placed blocks on a minimum of 12 inches of Styrofoam™ support. Please do not move blocks.

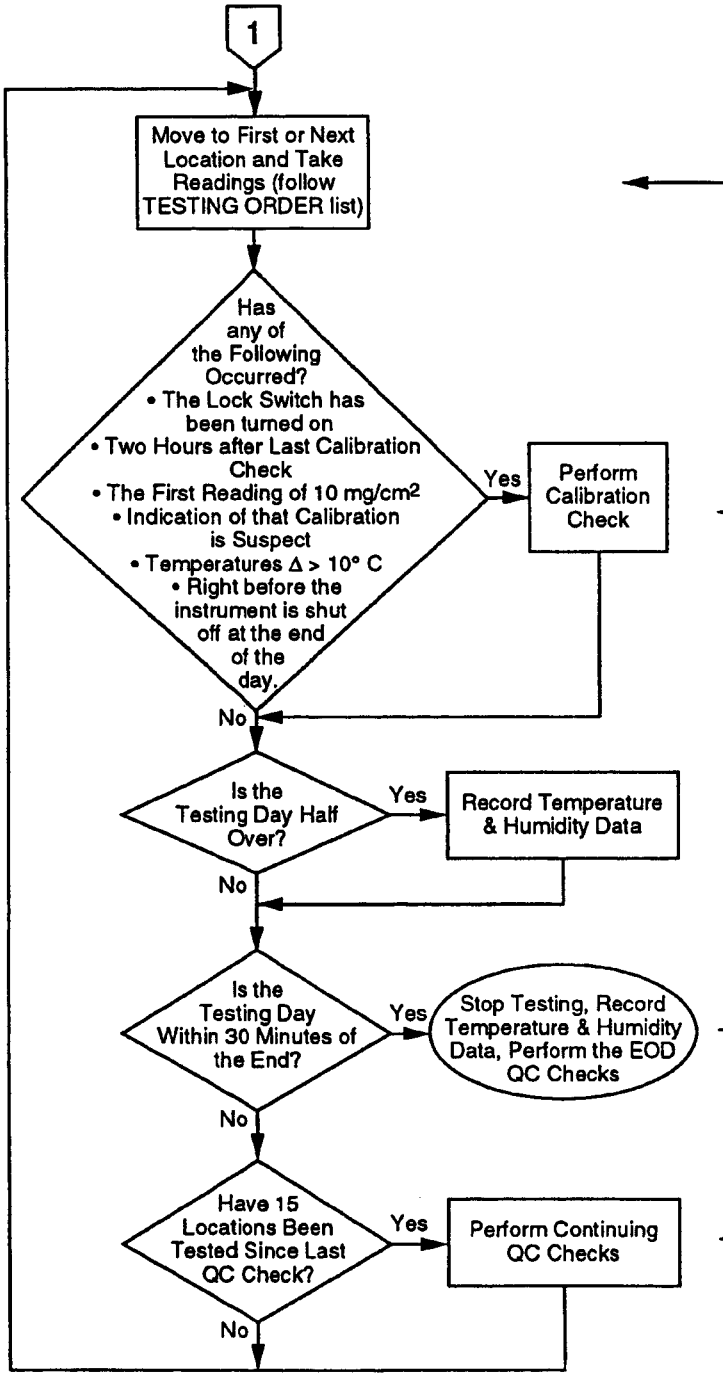
For each block test in the following order:

a. 1 reading, yellow NIST on block
b. 1 reading, red NIST on block. IF the block is wood, THEN collect 2 more readings using the red NIST on the block. Record extra readings on subsequent lines of the data form placing a diagonal line through the unused yellow and no NIST data blocks.
c. 1 reading, no NIST on block.

Record the data on the CONTROL READINGS form.

Testing Protocols for the PGT XK-3 Lead-In-Paint Analyzer

Flow Chart Summary



Description Details for PGT XK-3

For each testing location, test and record the measurements in the following order:

a. 1 reading, on X1
b. 1 reading, on bare (P1) + red NIST
c. 1 reading, on bare (P1)
d. 1 reading, on X2
e. 1 reading, on X3.

Note: If insufficient time is available during NIST testing, X2 and X3 readings may be deleted.

1. If XRF off, allow to warm up for 10 minutes.
2. Perform calibration checks as per the manual and manufacturer training directives using the manufacturer-supplied calibration sample(s).

Summarize changes to any user entered information on the instrument INFORMATION form.

Note: See item #8 of "Operating Instructions" in this protocol (p. 5) for specific instructions on calibration checks and use of Reset Button after "10.0 mg/cm^2" readings.

EOD QC checks are the same as BOD QC checks; follow same procedure as for BOD QC checks.

Record temperature and humidity in a field notebook.

Start next day after calibration at top of flow chart using the next sample to be tested as the first location.

Note: The PGT does not store or down load electronic data files.

Continuing QC checks, total of 2 blocks tested on a minimum of 12 inches Styrofoam™ support. Use blocks matching same type as the last and next locations (If same type for both, test same block twice).

Follow same testing order and reading times as that used for BOD testing.

USING THE XK-3

Operating Instructions

1. Remove the battery case, coiled cable, and XK-3 unit from the carrying case. Connect the battery pack to the XK-3 unit, using the coiled cable. Do not operate the XK-3 unless the battery pack is connected to the unit with the coiled cable, or a rapid power drain of the handle batteries will result.

2. Find the lock switch underneath the handle (toward the rear of the unit) and push it forward. A red light over the display will glow to indicate that the instrument is now ready to perform analysis as soon as the shutter is opened. The XK-3 may require approximately 10 minutes of operation to stabilize after first being turned on. The larger the temperature difference between where the XK-3 has been stored and where it's being used, the longer it will take for it to stabilize. The stabilization process can best be carried out and observed by using the Calibration Check procedures as described.

3. Depress the red reset button on the back plate of the unit (just above the coiled cable connection) for 10 seconds.

4. Grasping the rear of the wooden handle, position the faceplate of the instrument against the surface to be measured and press down firmly and quickly so that a mechanical click occurs. This action will open the shutter, and the red light over the window will blink to indicate the shutter is no longer completely closed. As soon as the shutter is completely open, the previous readout in the window vanishes, leaving the display blank except for a single decimal point.

5. When the new reading appears, an audible beep will be heard to indicate completion of the reading. Release the handle. The display window will retain the reading until the handle is again depressed to begin another measurement.

6. The XK-3 is self-correcting for the decay of the source. As the source ages, the instrument automatically extends its counting time.

7. Readings with a negative sign (–) may be normal. It depends on the statistical variations of the readings or could indicate a substrate with negative substrate contribution; that is substrate equivalent lead (SEL) as defined in the HUD lead abatement guidelines. It may be necessary to correct for the effect of different substrate materials on the readings. This subject is covered in detail in the XK-3 User Schools.

8. The first time a reading of 10.0 mg/cm^2 is seen during a testing sequence, the XK-3 should be reset and its calibration checked (see Calibration Check, below). Each subsequent reading of 10.0 mg/cm^2 should be followed by use of the reset button. Calibration should be checked only after the first reading of 10 mg/cm^2 in a series. If the XK-3 checks out, just reset after subsequent 10.0 mg/cm^2.

9. Push the lock switch to the LOCK position when testing has been completed.

NOTE: If the safety shutter is not completely closed, the red light flashes to show that radiation may be coming from the instrument. The red light will flash any time the shutter is not completely closed. If the red light is flashing with the lock switch ON and the operating handle in the UP position, it indicates a possibility of the shutter being open.

If the red power safety light is not lit after the locking switch in ON, and there is no indication of low battery level or absence of power, treat the unit according to the sticky shutter procedure (as described in the Operator's Manual).

Calibration Check

To perform the calibration check, follow this procedure:

1. Assemble the XK-3 and turn the lock switch ON, as outlined in the preceding section.

2. Place the calibration check board on a lead-free surface, yellow lead sample side up.

3. Depress the red reset button (on the back of the XK-3 just above the cable connection) all the way in with a ball-point pen and hold it in for 10 seconds. The reset button activates an internal circuit that resets the electronics.

4. Put the XK-3 on the check board with the yellow lead sample toward the heel of the XK-3. Align the XK-3 on the board with the white arrows on the sides of the XK-3 at least 1½ inches away from the yellow sample.

5. Depress the safety handle all the way in as described in the Operating Instructions until a new reading appears on the display.

6. Take at least 10 readings and average them. The average should fall within ±0.5 mg/cm^2 of 0.0 mg/cm^2. If the XK-3 has only just been turned ON, it may be necessary to repeat this test several times before it meets the requirements. This is

normal; the XK-3 is stabilizing, as mentioned in Section 1 of the Operating Instructions.

7. If the XK-3 still doesn't meet the ±0.5 mg/cm^2 criterion, use the reset button and repeat the test.

8. Turn the check board around so that the arrows on the XK-3 sides are aligned with the arrows on the yellow lead side.

9. Repeat Steps 5 through 7, using the value on the lead sample as the criterion, instead of 0.0 mg/cm^2. With new XK-3s, the check sample lead value is 1.5 mg/cm^2.

10. If the averages obtained in these tests are not within the ±0.5 mg/cm^2 of the value on the lead sample, the instrument should be returned for service.

ARCHIVE XRF INSTRUMENT INFORMATION

Date _____ Testing Site _____

Contractor _____ Manufacturer _____

Model No. _____ Serial No. _____

XRF Operator (Printed Name) _____

Source Material _____ Source Age or Date _____

Source Serial No. _____ Detector Type _____

Software Version No. _____

Operating Parameters Used

Open shutter sampling time(s)? (fixed or variable) _____

If fixed, what is the duration time(s)? _____

Daily warm-up and calibration check used? (Briefly discuss) _____

Regulatory level value used for setting the XRF Instrument ? (Yes or No) _____

If yes, enter the value used _____

Other _____

Data File Names and Descriptions NA—The PGT does not store or download electronic data files.

NOTE: The PGT instrument does not store or download electronic data files.

☐ Check here to indicate the presence of additional comments on the back of this form.

Archive XRF Test Data -- Standard Readings

Page _____ of _____

Date _____ XRF Device **PGT XK-3** Serial Number _____

XRF Operator (Printed Name) _____ XRF Field Monitor (Printed Name) _____

Location ID	Time of Measurement	Data Item	Paint Surface Reading: X1	Substrate + NIST Red, 1.02 mg/cm^2 Reading	Substrate Only Reading	Paint Surface Reading: X2	Paint Surface Reading: X3	Comments[1]
		K-shell (mg/cm^2)						
		K-shell (mg/cm^2)						
		K-shell (mg/cm^2)						
		K-shell (mg/cm^2)						
		K-shell (mg/cm^2)						
		K-shell (mg/cm^2)						
		K-shell (mg/cm^2)						
		K-shell (mg/cm^2)						
		K-shell (mg/cm^2)						
		K-shell (mg/cm^2)						

[1] Note every time the Reset (R) button is used during data collection. The Reset button is used every time a reading of 10.0 mg/cm^2 is observed during testing.

Archive XRF Test Data -- Control Readings

Date XRF Device **PGT XK-3** Serial Number _____

XRF Operator (Printed Name) . XRF Field Monitor (Printed Name) _____

QC Type: 1=Initial Control Readings 2=Continuing Control Readings 3=Ending Control Readings Bock Type: M=Metal W=Wood B=Brick D=Drywall C=Concrete P=Plaster

Identification		Time of Measurement	Data Item	Readings			Comments[1]
QC Type	Block Type			Yellow, 3.53 mg/cm^2	Red, 1.02 mg/cm^2	No NIST SRM	
			K-shell (mg/cm^2)				
			K-shell (mg/cm^2)				
			K-shell (mg/cm^2)				
			K-shell (mg/cm^2)				
			K-shell (mg/cm^2)				
			K-shell (mg/cm^2)				
			K-shell (mg/cm^2)				
			K-shell (mg/cm^2)				
			K-shell (mg/cm^2)				
			K-shell (mg/cm^2)				

[1] Note every time the Reset (R) button is used during data collection. The Reset button is used every time a reading of 10.0 mg/cm^2 is observed during testing.

General Responsibilities

The XRF Operator will:

- Perform all manufacturer recommended warm-up procedures and quality control checks, and work with the Testing Monitor and the Testing Supervisor to document that the procedures and checks were done, what the outcomes were, and, if appropriate, what actions were taken.
- Handle the instruments and make the measurements.
- Work with the Testing Monitor to see that the appropriate data are recorded.
- Work with the Testing Supervisor to help ensure that protocols are followed and any deviations are properly documented.
- Download data to a computer and verify that the transfer was successful.
- Report to the Testing Supervisor any indications of deteriorating samples observed.

The Monitor will:

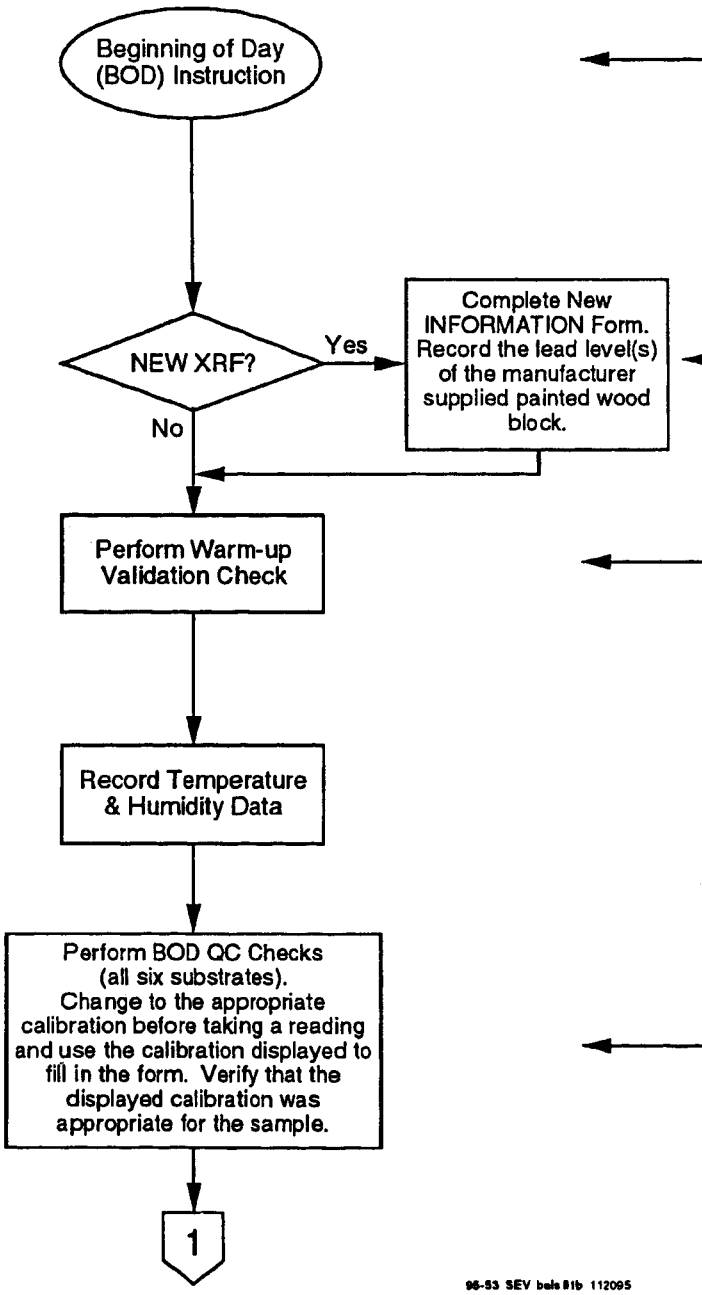
- Record all relevant data on the appropriate data forms or field notebooks.
- Work with the XRF Operator to help ensure the protocols are followed.
- Work with the Testing Supervisor to help ensure that protocols are followed and any deviations are properly documented.

The Supervisor will:

- Verify ID assignments on sample locations on the archive components.
- Ensure that the data are collected as described in the protocol.
- Collect all forms and electronic data.
- Ensure that resources are available to achieve planned testing.
- Oversee testing activities.
- Provide beginning-of-day and end-of-day instructions.
- Make primary decisions regarding any testing difficulties that may arise.
- Make sure that all testing is done safely, that all personnel on site wear dosimeter badges during testing, that dosimeter badges are collected and analyzed after testing is completed, and that results of dosimeter badge analysis are relayed to the badge wearers.
- Take radiation measurements periodically around each instrument being used for testing.
- Provide blank forms to the Testing Monitor (blank forms to be delivered to the Testing Supervisor by QuanTech) and review forms filled out by the Testing Monitor for completeness. The forms for the following will be used during testing:
    - Testing order list
    - Archive XRF information
    - Standard readings
    - Control readings

Testing Protocols for the SCITEC MAP 4 Spectrum Analyzer

Flow Chart Summary



Description Details for MAP 4

Instructions to include: safety issues, field form use, archive layout and testing order, sample ID numbering for electronic data capture, and voice-call back verification of data recording by monitor.

Modes of Operation

- Manufacturer Validation Checks: use TEST mode, use CONFIRM mode if called for by check procedure.
- Control Blocks (BOD, EOD, and CC): use TEST mode only.
- X1: use SCREEN and TEST modes, use CONFIRM if TEST is inconclusive,use UNLIMITED until a light goes on.
- BARE and RED NIST: TEST mode only
- BARE: TEST mode only
- X2: TEST mode only
- X3: TEST mode only

Note: "STOPPING RULE" for the unlimited mode is discussed in the Addendum on page 7 of this protocol.

See form for needed information.

Note: The manufacturer's supplied painted wood block will be used for all manufacturer's specified validation checks.

1. Allow to warm-up (stabilize) for 5 minutes.
2. Enter or verify:
   a. the selected file names to store the data
   b. the action level is set to 1.0 mg/cm^2
   c. any other user entered parameters.
3. Perform validation check as per the manual and manufacturer training directives using the manufacturer supplied calibration sample(s).

Summarize any user entered information and the calibration procedure on the instrument INFORMATION form.

BOD (or EOD) QC checks: test a total of 6 pre-placed blocks on a minimum of 12 inches of Styrofoam^{TM} support. Please do not move blocks.

Note: Per the manufacturer's instructions, the "Steel" calibration mode will be used for all metal substrates except aluminum. The "K & L Gen" calibration mode will be used for the other test substrates.

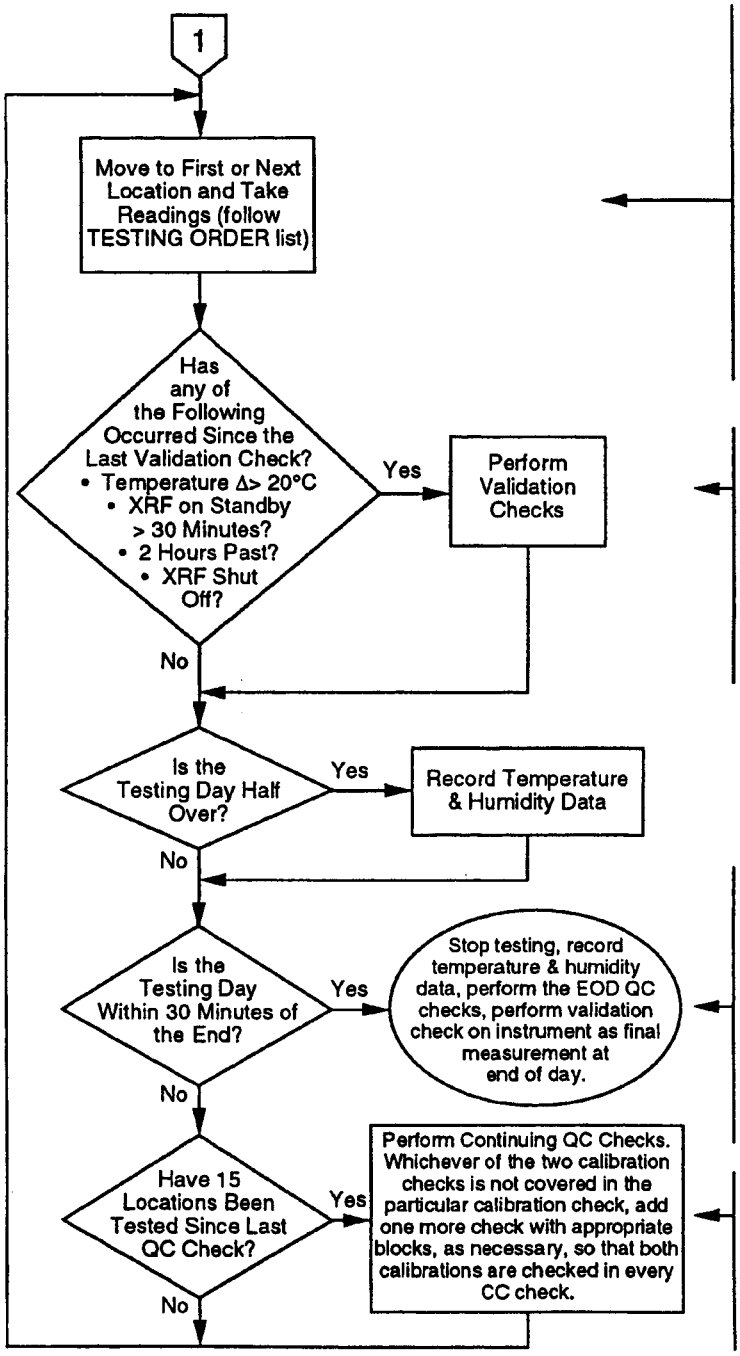
For each block, using the precision "Test" setting, test in the following order:

a. 1 reading, yellow NIST on block
b. 1 reading, red NIST on block. IF the block is wood, THEN collect 2 more readings using the red NIST on the block. Record extra readings on subsequent lines of the data form placing a diagonal line through the unused yellow and no NIST data blocks.
c. 1 reading, no NIST on block.

Record the data on the CONTROL READINGS form.

Testing Protocols for the SCITEC MAP 4 Spectrum Analyzer

Flow Chart Summary



Description Details for MAP 4

The calibration mode should be selected based on the apparent top substrate.

Modes of Operation

- X1: use SCREEN and TEST modes, use CONFIRM if TEST is inconclusive, use UNLIMITED until a light goes on.
- BARE and RED NIST: TEST mode only
- BARE: TEST mode only
- X2: TEST mode only
- X3: TEST mode only

Note: See forms for specific data recording instructions.

Note: If insufficient time is available during NIST testing, X2 and X3 readings may be deleted.

1. If XRF off, allow to warm up.
2. Enter or verify:
   a. the selected filenames to store the data
   b. the action level is set to 1.0 mg/cm²
   c. any other user entered parameters.
3. Perform validation check as per the manual and manufacturer training directives using the manufacturer-supplied calibration sample(s).

Summarize changes to any user entered information on the instrument INFORMATION form.

Perform and record a final validation check as the last measurement before leaving the site at end of day.

EOD QC checks are the same as BOD QC checks; follow same procedure as for BOD QC checks.

Record temperature and humidity in a field notebook.

Download any electronically stored data, verify successful transfer before clearing the memory to make room for more data.

Start next day at top of flow chart using the next sample to be tested as the first location.

Continuing QC checks, total of 2 blocks tested on a minimum of 12 inches Styrofoam™ support. Use blocks matching same type as the last and next locations (If same type for both, test same block twice).

Follow same testing order and reading times as that used for BOD testing.

85-53 SEV bals II2b 112095

Morning Validation Checks

Each morning, perform a validation check on your MAP 4 instrument following these steps. The morning validation check is performed at the test site. Record all specified calibration/validation checks on both paper and electronic data storage; also, note when the TEST setting was used and when the CONFIRM setting was used for all the manufacturer-required validation checks and record the results.

1. Using rubber bands, attach the painted block of wood included with your instrument to MAP 4's faceplate. Make sure the painted side faces the instrument. Make sure the orientation of the painted block to the faceplate is the same for all tests.

2. Position MAP 4 so that there are at least 3 feet open air between the instrument front and the nearest surface. Maintain this distance for the duration of each measurement. Do not point the instrument near another person or where a person is likely to approach.

3. Take **5** measurements at Test precision. Be sure to use appropriate codes to identify these as validation checks. This will be important for your records.

4. Using the forms supplied by SCITEC, record the results as follows:

    a. Record the results of each individual measurement, and so on. Also record the time and date the measurements were taken.

    b. Average together the results of the tests and record this number.

    c. On the *Calibration Check Graph* write the average on the first available line under *Recorded Averages*.

    d. On the graph, plot the location of the average as it compares to SCITEC's average (written on the *Average* line). For example, if your average is .3 higher than SCITEC's, place an “x” at .3 above the Average line.

5. Repeat these steps each morning and carefully monitor the location of the “x”'s on the graph. Any of the following trends could indicate that your instrument is not functioning properly:

    - A trend of eight or more readings above or below the Average line,

    - A trend going from high to low (or low to high).

- A consistent pattern of "too high" or "too low" readings, random, but widely scattered.

6. If you notice any of the above trends, contact SCITEC at 1-800-4NO-LEAD.

Continuing Validation Checks

Frequent validation checks should be made at the worksite while you are testing, following these steps:

1. Take **1** measurement at Test precision on a NIST standard backed by a sample of substrate material.

2. Write the results of each individual *Daily Calibration Check*.

3. If the result of any of these individual checks is off of SCITEC's average by more than ±.2 mg/cm^2:

   a. Take a Confirm measurement.

   b. If you're off by more than ±.1 of SCITEC's average, take one more Confirm measurement.

   c. If you're still off by more than ±.1, contact SCITEC.

Validation Check Codes

The code entered for a validation check measurement should indicate the date, time, who was using the instrument, and which instrument was used. We suggest the following coding system.

Date	Time	Operator No.	MAP 4 Serial No.
02255	0745	7240	177
↑	↑	↑	↑
Feb. 25, 1995	7:45 a.m.	From Operator's Certificate	Last 3 digits in your instrument serial No.

Date A five-digit date is used.

- The first two digits indicate the month, e.g., 02 = February.
- The second two digits indicate the day, e.g., 09 = the ninth.
- The final digit indicates the year, e.g., 5 = 1995.

Time Military time is used. Four digits must be entered.

- 0700 = 7.00 a.m.
- 1200 = noon
- 1345 = 1.45 p.m.

Operator Number This is the four-digit number on your Operator's Spectrum Analyzer Training Certificate

Serial Number These are the last three digits in the serial number assigned to your MAP 4 instrument.

Taking a Measurement

The standard assay command on the ASSAY menu allows you to take measurements using the selected precision, calibration, and action level. Before taking measurements be sure that these are set appropriately (see *Section 3: MAP 4 Operation's Manual*).

To Take a Measurement

1. Display the **Standard assay** command. You can do this by pressing **GO TO** from any command.

2. Press **ENTER/YES**. MAP 4 asks for a code to identify this measurement. The prompt varies depending on ARM mode. If ARM mode is ON, you are asked for an ARM code. If ARM mode is OFF, you are asked for an ID code.

3. Type a code and press **ENTER/YES**. If ARM mode is off you are also asked for a sequence number.

   Sequence number indicates the order of measurements under a code. #1 indicates this is the first measurement taken under code 1234567. To accept the sequence

number, press **ENTER/YES**. To change it, press **ESC NO** and enter the desired number.

4. Once you've entered a code, you receive a prompt similar to start.

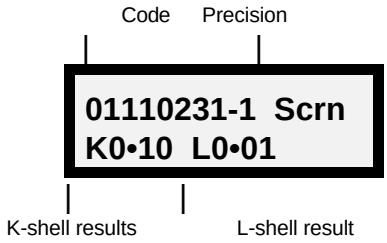
5. Place MAP 4's faceplate flat against the sample surface.

6. Insert the key into the lock located on the front left side of the instrument. Turn the key clockwise into the ON position (so that the arrow on the key points up).

7. Pull the trigger and hold MAP 4 in place. After the "beep," release the trigger. A light indicates the level of lead contamination (related to the K-Shell reading):

   - **Positive** = lead is **above** the selected level.
   - **Negative** = lead is **below** the selected level.
   - **Retest** = **inconclusive** results. Further testing is required.

   Your measurement results are displayed, similar to this:



Note: Assay values greater than 4.99 mg/cm^2 will be truncated to 5.00 mg/cm^2.

NOTE: Record the indicator readings on the data forms as:

P (positive),
N (negative), or
I (inconclusive)

8. Turn the key to OFF position, and remove MAP 4 from the surface.

9. To take another measurement press **ENTER/YES**.

   - If ARM mode is on, you are returned to the "Enter ARM code" prompt. You can either enter a new code or press Enter/Yes to bring up the last code used.

   - If ARM mode is off, you are returned to the "TRIGGER to strt" prompt. MAP 4 has automatically assigned an ID code to the next measurement using the last code entered and next sequence number (e.g., if the last measurement was 555-1, this measurement will be 555-2). To take a measurement under this code, simply position the instrument and pull the trigger. To enter a different ID start typing it; this takes you to the "Enter ID code" prompt.

Addendum: Measurement Time for the Unlimited Mode

The stopping rule for the **UNLIMITED** mode will be: Test until either a light goes on, or the displayed precision reaches ±0.1 mg/cm^2 or 2 minutes have elapsed since the trigger was pulled.

The 2-minute time limit is for a source of 11.5 millicuries (mCi). If the source strength of the instrument is less than 11.5 mCi, the 2-minute time limit will be increased proportionately to account for the lower source strength. For example, if an instrument with a source strength of 9 mCi is used, the time limit for that instrument would be 2 minutes and 30 seconds (120 seconds × 11.5/9 = 153 seconds, or approximately 2 minutes and 30 seconds). The time limit would never be less than 2 minutes, even if a source stronger than 11.5 mCi is used.

ARCHIVE XRF INSTRUMENT INFORMATION

Date _____ Testing Site _____

Contractor _____ Manufacturer _____

Model No. _____ Serial No. _____

XRF Operator (Printed Name) _____

Source Material _____ Source Age or Date _____
Is instrument automatically adjusted for source decay _____

Source Serial No. _____ Detector Type _____

Software Version No. _____

NOTE: Adjust clock time to Central Time Zone before starting tests.

Operating Parameters Used

Open shutter sampling time(s)? (fixed or variable) _____

If fixed, what is the duration time(s)? _____

Daily warm-up and calibration check used? (Briefly discuss) _____

_____

_____

Regulatory level value used for setting the XRF Instrument ? (Yes or No) _____

If yes, enter the value used _____

Other _____

_____

Manufacturer's supplied painted wood block lead level(s): _____

Data File Names and Descriptions _____

_____

_____

_____

_____

_____

☐ Check here to indicate the presence of additional comments on the back of this form.

Archive XRF Test Data -- Standard Readings

Page _____ of _____

Date ______________ XRF Device **SCITEC MAP 4** Serial Number ______________________

XRF Operator (Printed Name) ____________________ XRF Field Monitor (Printed Name) ________________

Location ID	Time of Measurement	Data Item	Paint Surface Reading: X1	Substrate + NIST Red, 1.02 mg/cm² Reading	Substrate Only Reading	Paint Surface Reading: X2	Paint Surface Reading: X3	Comments[3]
		K-shell (mg/cm²)						Calib. mode: G S
		Sequence Number						
		Indicator[1]	P N I	P N I	P N I	P N I	P N I	
		Mode[2]	Screen Test Confirm Unlimited	Test only	Test only	Test only	Test only	
		Reading time (sec)[4]						
		K-shell (mg/cm²)						Calib. mode: G S
		Sequence Number						
		Indicator[1]	P N I	P N I	P N I	P N I	P N I	
		Mode[2]	Screen Test Confirm Unlimited	Test only	Test only	Test only	Test only	
		Reading time (sec)[4]						
		K-shell (mg/cm²)						Calib. mode: G S
		Sequence Number						
		Indicator[1]	P N I	P N I	P N I	P N I	P N I	
		Mode[2]	Screen Test Confirm Unlimited	Test only	Test only	Test only	Test only	
		Reading time (sec)[4]						

1 Record lead reading indicator levels as P (positive or red light), N (negative or green light), or I (inconclusive or yellow light). Circle as appropriate.
2 Record test mode by circling the appropriate line item.
3 Indicate calibration mode used in G (K&L Gen), S (steel).
4 Record reading time, in seconds, for all mode readings on XI every 15th sample.

Archive XRF Test Data -- Control Readings

Page _____ of _____

Date ______________ XRF Device **SCITEC MAP 4** Serial Number ______________________

XRF Operator (Printed Name) ____________________ XRF Field Monitor (Printed Name) ______________

QC Type: 1=Initial Control Readings 2=Continuing Control Readings 3=Ending Control Readings Block Type: M=Metal W=Wood B=Brick D=Drywall C=Concrete P=Plaster

Identification		Time of Measurement	Data Item	Readings			Comments[3]
QC Type	Block Type			Yellow, 3.53 mg/cm^2	Red, 1.02 mg/cm^2	No NIST SRM	
			K-shell (mg/cm^2)				Calib. mode: G S
			Sequence Number				
			Indicator[1]	P N I	P N I	P N I	
			Mode[2]	Test only	Test only	Test only	
			Reading time (sec)[4]				
			K-shell (mg/cm^2)				Calib. mode: G S
			Sequence Number				
			Indicator[1]	P N I	P N I	P N I	
			Mode[2]	Test only	Test only	Test only	
			Reading time (sec)[4]				
			K-shell (mg/cm^2)				Calib. mode: G S
			Sequence Number				
			Indicator[1]	P N I	P N I	P N I	
			Mode[2]	Test only	Test only	Test only	
			Reading time (sec)[4]				

1 Record lead reading indicator levels as P (positive or red light), N (negative or green light), or I (inconclusive or yellow light). Circle as appropriate.
2 Record test mode by circling the appropriate line item.
3 Indicate calibration mode used in G (K&L Gen), S (steel).
4 Record time, in seconds, for all readings of RED NIST over wood.

SUBCONTRACTOR TESTING PROTOCOLS FOR THE LEADSTAR

General Responsibilities

The XRF Operator will:

- Provide a backup instrument, to be made available on site.
- Provide the necessary serial cable (supplied with the LeadStar) to connect the analyzer serial port on the LeadStar electronics module to the serial port on the host computer.
- Handle the instruments and make the measurements.
- Work with the Testing Monitor to see that the appropriate data are recorded.
- Work with the Testing Supervisor to help assure that protocols are followed and any deviations are properly documented.
- Download data to a computer provided by the Testing Supervisor, and verify that the transfer was successful.
- Report to the Testing Supervisor any indications of deteriorating samples observed during testing.
- Provide extra set of charged batteries to extend the testing day beyond 8 hr. Batteries should be replaced whenever the XRF instrument indicates that the "time remaining is less than 10%."
- Certify in writing that all test data are erased (non-accessible, non-recoverable) from the instrument memory (as directed by the supervisor) before taking the instrument off-site and after each day's testing.

The Monitor will:

- Record the results of the manufacturer's warmup and calibration procedures on the appropriate form.
- Record all relevant data on the appropriate data forms or field notebooks.
- Work with the XRF Operator to help ensure that the protocols are followed.
- Work with the Testing Supervisor to help assure that protocols are followed and any deviations are properly documented.

The Supervisor will:

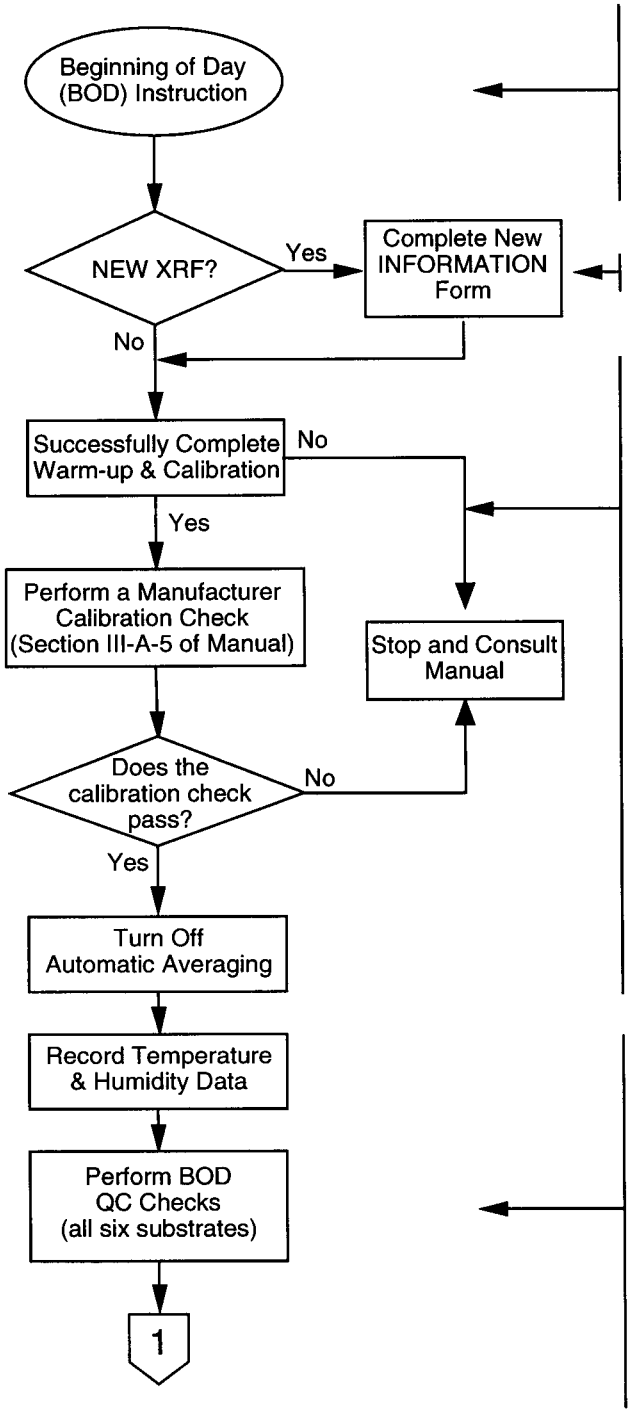
- Verify ID assignments on sample locations on the archive components.
- Assure that the data are collected as described in the protocol.
- Collect all forms and electronic data and make appropriate distributions to MRI and QuanTech.

- Ensure that resources are available to achieve planned testing.
- Oversee testing activities.
- Provide beginning-of-day and end-of-day instructions.
- Ensure that host computer is configured to automatically load the interlink interface by including the following line in the config.sys file: *device=c:\dos\interlink.exe /drives:2/noprinter/com:1*
- Make primary decisions regarding any testing difficulties that may arise.
- Provide the following forms:

  a. Testing order list
  b. Standard readings
  c. Archive XRF information
  d. Control readings
  e. Instrument calibration/calibration check data
  f. General observations and other field data

- Provide the EPA Work Assignment Manager with a status report (via phone) at the end of each day of testing.
- Provide the EPA Work Assignment Manager with an oral report of deviations from the testing protocol as soon as possible during the course of the testing, with a written report to follow no later than one working day after the completion of testing.
- Ensure that all personnel who enter the archive facility during XRF testing wear dosimeter badges, and will arrange for the testing of the badges and reporting of results after completion of testing.
- Take radiation measurements near the instrument periodically during the course of the testing, compare the measured values to the corresponding values in the instrument Operator Manual. If radiation levels significantly exceed the values listed in the operator's manual, testing will be stopped and the EPA Work Assignment Manager will be notified. The radiation measurement instrument will be calibrated for the energy range of ^{57}Co and appropriate window thickness.

Flow Chart Summary



Description Details for Advanced Detectors

Instructions to include: safety issues, field form use, archive layout and testing order, Sample ID numbering for electronic data capture, voice-call back verification of data recording by monitor. The Fixed Mode will be used with a setting of 15 (nominal) seconds, and the Brief Mode will be used to test X1 only with a maximum setting of 30 (nominal) seconds. Both modes make adjustments for source decay.

See form for needed information.

1. Allow to warm-up for 5 minutes.
2. Enter or verify:
   a. the selected file names to store the data
   b. the action level is set to 1.0 mg/cm^2
   c. any other user entered parameters
3. Perform calibration as per the manual and manufacturer training directives using the manufacturer supplied calibration sample(s). (Instrument Operation Instructions, Section III-A, of the LeadStar Operator Manual, dated March 1996, specifically: Subsection 4 – calibration.)
4. Perform calibration check as per the manual (Section III-A, Subsection 5). The calibration checks are to be performed on the RED NIST (1.02 mg/cm^2) standard film over the wood control block over styrofoam.

Summarize any user entered information and the calibration procedure on the instrument INFORMATION form.

Note: The instrument calibration and calibration check readings are separate and distinct form the test series QC controls – Record each on separate forms provided. Determinations as to whether the instrument is in calibration will be made based on the results of the calibration and calibration checks. Results of the BOD, EOD, and continuing QC control checks will not in themselves be used to determine if an instrument is or is not in calibration, but may be used as grounds to call for a calibration check by the operator.

BOD (or EOD) QC checks: test a total of 6 pre-placed blocks on a minimum of 12 inches of Styrofoam™ support. **Please do not move blocks.**

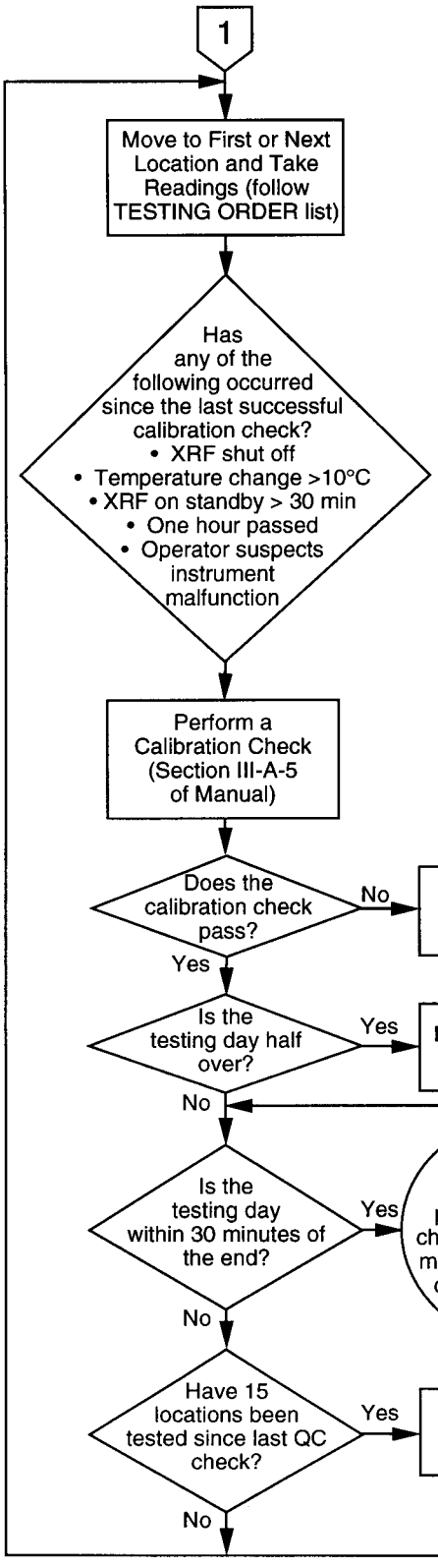
For each block, using a nominal 15-second reading (Fixed Mode), test in the following order:

a. 1 reading, yellow NIST on block
b. 1 reading, red NIST on block. **IF** the block is wood, **THEN** collect 2 more readings using the red NIST on the block. Record extra readings on subsequent lines of the data form placing a diagonal line through the unused yellow and no NIST data blocks.
c. 1 reading, no NIST on block.

Record the data on the CONTROL READINGS form.

96-38 hoot/bais 080996

Flow Chart Summary



Description Details for Advanced Detectors

For each testing location (158 samples), using a nominal 15-second (Fixed Mode) reading, test in the following order:

a. 1 reading, on X1
b. 1 reading, on bare (P1) + red NIST
c. 1 reading, on bare (P1)
d. 1 reading, on X2
e. 1 reading, on X3.
f. Set nominal (max.) time to 30 seconds and read x1 only in Brief mode.

Record the length of time it takes to measure X1 in the Brief mode only.

1. **IF** XRF off, allow to warm up for 5 minutes.
2. Enter or verify:
   a. the selected filenames to store the data
   b. the action level is set to 1.0 mg/cm^2
   c. any other user-entered parameters

Summarize changes to any user-entered information on the instrument INFORMATION form.

Note: Sample data must be preceded by a successful calibration and bracketed by successful calibration checks. If there is a failure in a calibration check (failure means two successive tries at a calibration check both of which are outside the interval of 0.82 to 1.12), sample measurements from the point of the last successful calibration check must be redone with a recalibrated instrument.

EOD QC checks are the same as BOD QC checks; follow same procedure as for BOD QC checks.

Record temperature and humidity in a field notebook.

Download any electronically stored data, verify successful transfer before clearing the memory to make room for more data.

Start next day at top of flow chart using the next sample to be tested as the first location.

Continuing QC checks, total of 2 blocks tested on a minimum of 12 inches Styrofoam™ support. Use blocks matching same type as the last and next locations (If same type for both, test same block twice).

Follow same testing order and reading times as that used for BOD testing (i.e., use the nominal 15-second reading for testing a-c).

Archive XRF Instrument Information

Date _____ Testing Site _____

Contractor _____ Manufacturer _____

Model No. _____ Serial No. _____

XRF Operator (Printed Name) _____

Source Material _____ Source Age or Date _____

Source Serial No. _____ Detector Type _____

Operating Parameters Used

Open shutter sampling time(s)? (fixed or variable) _____

If fixed, what is the duration time(s)? _____

Daily warm-up, calibration, and calibration check use? (Briefly discuss) _____

_____

_____

Regulatory level value used for setting the XRF instrument? (Yes or No) _____

If yes, enter the value used _____

Other _____

_____

_____

Data File Names and Descriptions _____

_____

_____

_____

_____

_____

☐ Check here to indicate the presence of additional comments on the back of this form.

Supervisor _____ Date _____

96-34 schel frm 072596

GENERAL OBSERVATIONS AND OTHER FIELD DATA

Date of testing: _____

XRF instrument: _____

Recorded by (name and date): _____

Temperature and Humidity Data

	Time of measurement	Temperature	% RH
Instrument ID:			
Start of testing			
Mid-test			
End of testing			

Radiation Measurements

Time of measurement	Orientation	Reading
Instrument ID:		

GENERAL OBSERVATIONS AND OTHER FIELD DATA, cont.

General comments and observations on XRF instrument performance during testing, exceptions to the protocol, etc.:

_____
_____
_____
_____
_____

Describe the data file downloading and XRF instrument file deletion procedures used:

_____
_____
_____
_____
_____

☐ Statement of Confidentiality by operator attached.

STATEMENT OF DATA CONFIDENTIALITY
(to be signed by the XRF Instrument operator)

It is understood and agreed by _____, representing
(printed name)

_____, that for the XRF tests performed on
(company name)

_____, the data are not to be disclosed or discussed
(dates of testing)

with any manufacturer or vendor or any outside party other than the U.S. EPA or its representatives.

Furthermore, by my signature below, I certify that all the data files generated by these tests and recorded in the XRF instrument's electronic memory have been deleted, purged, and made non-accessible prior to removing the instrument off-site from the archive test facility. I understand that I and the company I represent will be liable for any fraudulent use or disclosure of these data.

Signed _____

Date _____

Archive XRF Test Data
Calibration Data for LeadStar XRF Instrument

Date _____ Standard used _____ Recorder_____

XRF Device Advanced Detectors, Inc., LeadStar Serial number _____

Time of measurement	K-shell (mg/cm^2)	K-shell (mg/cm^2) uncertainty	L-shell (mg/cm^2)	L-shell (mg/cm^2) uncertainty	Density	Seq No.

Was the instrument warm-up successful? ☐ yes ☐ no If no, stop and consult manual.

Does the measurement meet the K-shell tolerance range? ☐ yes ☐ no If no, stop and consult manual.

Nominal value = _____ and tolerance range = _____ for the calibration standard.

General observations or exceptions to the protocol:

_____

_____

_____

NOTES:

[a] The calibration procedure is specified in Section III-4 of the instrument Operator manual, dated March 1996. Calibration and calibration checks are performed using a nominal 15-sec fixed mode.

[b] Per Section III-A-2,3 of the Operator's Manual: The "warm-up" period of ~ 5 min is automatically performed when the instrument is turned on; after this step is completed, the instrument prompts the operator to run the calibration.

[c] Record K-shell data on control charts during testing; **the tolerance range for calibration is specified on back of the calibration standard**.

Archive XRF Test Data
Calibration Check Data for LeadStar XRF Instrument

Date _____ Standard used _____ Recorder_____

XRF Device Advanced Detectors, Inc., LeadStar Serial number _____

Time of measure-ment	AV3 K-shell (mg/cm^2)	K-shell (mg/cm^2) uncertainty	L-shell (mg/cm^2)	L-shell (mg/cm^2) uncertainty	Density	Seq No.	Indicate Pass (P) or Fail (F)

General observations or exceptions to the protocol:

NOTES:

[a] The calibration procedure is specified in Section III-5 of the instrument Operator manual, dated March 1996. Automatic averaging is turned on, 3 readings are taken, and the average of the 3 readings should be within or equal to 1.12 to 0.82 mg/cm^2 for the calibration check to be successful. (Use the AV3 reading for this determination.)

[b] Per Section III-A-2,3 of the Operator's Manual: The "warm-up" period of ~ 5 min is automatically performed when the instrument is turned on; after this step is completed, the instrument prompts the operator to run the calibration.

[c] Record K-shell data on control charts during testing; **the tolerance for the calibration check is +0.1 to –0.2mg/cm^2 of the theoretical standard level, that is, 1.12 to 0.82 for the 1.02 mg/cm^2 NIST standard**.

[d] Calibration and calibration checks are performed using a nominal 15-sec fixed mode.

[e] If calibration check fails criteria, stop and consult protocol and manual for corrective actions.

Page ____ of ____

Archive XRF Test Data—Control Readings

Date _____ Recorder _____

XRF Device Advanced Detectors, Inc., LeadStar Serial No. _____

QC type: 1=BOD readings 2=Continuing Control readings 3=EOD readings

Block type: M=Metal W=Wood B=Brick D=Drywall C=Concrete P=Plaster

Identification		Time of measure-ment	Data item	Readings (Mode = R[a])			Comments (Note autocorrection if it occurs)[b]
QC type	Block type			Yellow, 3.53 mg/cm^2	Red, 1.02 mg/cm^2	No NIST SRM	
			K-shell (mg/cm^2)				
			K-shell (mg/cm^2) Uncertainty				
			L-shell (mg/cm^2)				
			L-shell (mg/cm^2) Uncertainty				
			Density				
			Instrument condition (ok / not ok)				
			Sequence number				
			K-shell (mg/cm^2)				
			K-shell (mg/cm^2) Uncertainty				
			L-shell (mg/cm^2)				
			L-shell (mg/cm^2) Uncertainty				
			Density				
			Instrument condition (ok / not ok)				
			Sequence number				

a Modes: R = Normal (15-sec Fixed). This mode used for all QC checks.

b If autocorrection occurs, the last reading immediately before autocorrection will be retaken and recorded. Indication the form which reading was taken before autocorrection and the reading taken after autocorrection.

Archive XRF Test Data—Standard Readings

Page ____ of ____

Date _____ Recorder _____

XRF Device Advanced Detectors, Inc., LeadStar Serial No. _____

Location ID	Time of measure-ment	Data item	Paint surface reading: X1	Substrate + NIST Red, 1.02 mg/cm^2 reading	Substrate only reading	Paint surface reading: X2	Paint surface reading: X3	X1 @ 30 sec	Comments [Autocorrection(b)]
		K-shell (mg/cm ^{2})							
		K-shell (mg/cm ^{2}) Uncertainty							
		L-shell (mg/cm ^{2})							
		L-shell (mg/cm ^{2}) Uncertainty							
		Density							
		Instrument condition							
		Mode[a]							
		Brief mode-time of measurement (sec)[c]	NA	NA	NA	NA	NA		
		Sequence number							
		K-shell (mg/cm ^{2})							
		K-shell (mg/cm ^{2}) Uncertainty							
		L-shell (mg/cm ^{2})							
		L-shell (mg/cm ^{2}) Uncertainty							
		Density							
		Instrument condition							
		Mode[a]							
		Brief mode-time of measurement (sec)	NA	NA	NA	NA	NA		
		Sequence number							

a Modes: R = Normal or Fixed time, B = Brief. Take all readings in Normal Mode, except "x1@30 sec" in Brief Mode.

b Indicate with a notation in the comments section whenever XRF instrument performs an automatic drift adjustment (autocorrection). When this occurs (-- every 4 hours), repeat the last reading. Clearly indicate which reading was taken before and after the autocorrection.

c For the X1, 30 second, Brief Mode only, record the length of time it takes to complete a reading from trigger pull to the time it takes for the reading to be displayed. This can be measured with a stopwatch using the audible tones of the LeadStar which occur when the measurement begins and when it ends.

References:

The following table lists XRF operation manuals used for protocol development.

List of XRF Operator Manuals Used in Protocol Development

Company name	Title	Date
Advanced Detectors, Inc. 1220 Avenida Acaso Camarillo, CA 93012	Lead Star Operator Manual (Software Version 4.1 or higher)	March 1996
NITON Corp. Bedford, MA 01730	Operating Procedures for the NITON XL Lead Detector	December 21, 1994
NITON Corp. Bedford, MA 01730	User Manual, XL Spectrum Lead Detector	Not specified
Princeton Gamma-Tech, Inc. 1200 State Road Princeton, NJ 08540	PGT XK-3 Lead-in Paint Analyzer Instruction Manual	January 4, 1993
RMD, Inc. 44 Hunt Street Watertown, MA 01721	User's Guide to the LPA-1 Lead Paint Analysis System	June 1995 print date
SCITEC Corp. 415 N. Quay Kennewick, WA 99336	MAP 3—Standard Operating Procedures for Lead-based Paint Testing with the MAP XRF Specimen Analyzer	August 7, 1995
SCITEC Corp. 415 N. Quay Kennewick, WA 99336	MAP 4—Operations Manual The MAP XRF Specimen Analyzer	Not specified
TN Technologies, Inc. P.O. Box 800 Round Rock, TX 78680-0800	Pb Analyzer Users' Guide (Revision 1.01)	October 1994
Warrington, Inc. 2113 Wells Branch Pkwy Austin, TX 78728	Microlead I Instruction Manual (Revision 4)	Not specified
Xsirius, Inc. 1220 Avenida Acaso Camarillo, CA 93012	LeadStar Operator Manual	May 30, 1995

Appendix C

Supplemental Protocols for Sampling, Characterization, and Analysis of Lead-based Paint Samples

Contents

Collection of Paint Chip Samples from Archive Materials

COLLECTION OF PAINT CHIP SAMPLES FROM ARCHIVE MATERIALS

SUMMARY

This document describes the standard protocol for obtaining a single paint chip sample from a painted substrate. This standard also includes instructions for sample storage and transport requirements.

MATERIALS AND EQUIPMENT

ITEM	No. per pair of collectors
Safety goggles	2 + 1 extra
Leather gloves	2 pair
Disposable gloves	1 bag 100 pair
Respirator with organic vapor filters	2 - one fitted to each collector
Razor blade holder	2 + 1 extra
Razor blades	25
Wood chisel	2
Hammer	1
Tweezers	2
Micrometer	1 per team
White paper, 8.5 x 11	300 sheets
Masking tape	20 rolls, 1-inch
Duct tape	8 rolls, 2-inch
Marking pens	6
Clip board with timepiece	2 + 1 extra
“Paint Chip Collection” data forms	Enough for 200 samples
Sample containers (plastic centrifuge tubes, plastic resealable bags)	a minimum of 200 tubes
Resealable plastic bags	a minimum of 200 1 qt bags
Extra shipping container for paint chip samples	4
Trouble lights and spare bulbs or equivalent lighting	2
Extension cords	200 ft.

ITEM	No. per pair of collectors
Power	110V at site
Pocket knife	2 + 1 extra
Metal marking template	2
Heat gun	2
Replacement heat gun element	2
Tool pouch with belt	1 per tester
Fire extinguisher	1 at site

Note: Other items as needed.

COLLECTION PROCEDURE

At each sampling location, perform the following steps (See Note 1):

NOTE 1: A regular sample will be collected at all locations. Some locations will require collection of an additional sample called a side-by-side sample. Locations that require a side-by-side sample are identified by the presence of an individual 2 in x 2 in square placed at one end of the marked location. For locations having a side-by-side sample, follow steps 1 through 9 below for collection of the regular sample first. After completing this sample collection, collect the side-by-side sample using the same procedure using different bar code number as described in step 2.

1.0 FILL OUT FORM:

1.1 For each new "Paint Chip Collection Reporting" form needed (see attached), complete the header of the form.

1.2 Record the sampling location identification number in the appropriate column and row on the form.

2.0 LABEL PAINT COLLECTION CONTAINER:

Label a paint collection container with the sampling location number and the sample ID type separated by "-" (for example; 905507-P3).

3.0 SCORE SAMPLING AREA:

Using a metal template and a cutting tool, score the perimeter of the designated area to be removed. Collect nominal 2 inch by 2 inch samples whenever possible unless otherwise indicated on the sample. If it is impractical to use the template, the score can be made using the outside edge of the template as a guide. The area scored using the alternative method should be done in a manner that maintains right angle corners as close as possible. Avoid using pencil or pen to mark the sample outline.

4.0 SETUP COLLECTION TRAY:

Affix a closed bottom paper funnel (or other appropriate collection shape) made from a clean white sheet of paper or equivalent collection device directly below the sampling location. The collection device should be located as close as possible to the sampling site but should not interfere with the removal procedure. Avoid placing tape on any surface targeted for XRF testing.

5.0 REMOVE PAINT:

Remove paint from the scored areas using the methods described below:

5.1 **PRIMARY PAINT REMOVAL METHOD:** Using a heat gun, heat the sample area. Extreme caution should be exercised when using the heat gun. Be sure to have a fire extinguisher nearby during heat gun use. Do not overheat the sample area, heat only until the paint becomes soft and supple. If working in teams of two persons, have one collector heat the area while the other removes the sample with a paint scrapper. Remove all paint down to the bare substrate. If the paint does not become soft and supple in a minute or two, discontinue the use of heat and try the alternative paint removal method.

Avoid the inclusion of the substrate in the collection device. If substrate does fall into the collection device, remove only that substrate that can be easily removed without losing any of the paint sample. Do not remove any substrate that cannot be separated from the paint sample. The laboratory will remove extraneous substrate if possible, under laboratory conditions.

5.2 **ALTERNATIVE PAINT REMOVAL METHOD:** Using the appropriate cutting tool for a particular substrate or condition of the sample site, begin removing the paint from the substrate. If possible, peel the paint off of the substrate by sliding the blade along the score and underneath the paint. Remove all paint down to the bare substrate.

6.0 MEASURE ESTIMATED PAINT THICKNESS

After completing removal of all the paint within the collection area, examine the paint chips contained within the collection tray for appropriate intact pieces large enough to perform a check on the paint thickness using a micrometer. If possible, use a clean pair of tweezers to handle the paint chips. An appropriate intact piece is one that appears to contain all paint layers with little or no substrate and has a minimum of a 1/4 by 1/4 inch surface area. If possible, perform and record with units of measure on the form, up to three thickness measurements using appropriate intact pieces. If one large appropriate piece is available, all three measurements can be made on that piece in different spreadout locations. If not, attempt to use more that one appropriate intact piece. Make a relevant entry in the comments column describing how many chips were used to make the measurements. Care must be taken to perform the measurement directly over the collection tray to ensure that no loss of paint occurs.

7.0 TRANSFER THE PAINT TO THE LABELED SAMPLE CONTAINER

Transfer the collected paint sample from the collection tray to the sample container, seal the container, and place it in a plastic bag. Exercise care to ensure that <u>all</u> paint taken from the recorded area is placed into the sample container. Use the Styrofoam holder that comes with the sample containers to aid in holding the container during transfer.

8.0 MEASURE AND RECORD THE SAMPLING AREA

Carefully and accurately measure the sampling area dimensions. Do not attempt to calculate areas in the field. Record the data and dimensions including units used (e.g., 5.1 cm x 5.0 cm) on the data form using a permanent marker. Try to use only centimeters for recording data. Avoid making measurement in inches. Any irregularities or problems that arise in the process should be noted in the Comments column of the form.

9.0 PERFORM FINAL CHECKS AND STORE SAMPLE

Verify the record keeping on the sample just collected by examining the data form entries and sample container just filled. Place the collected sample into a designated box until shipment can be made back to the laboratory. Return all completed forms and samples by the end of each sampling day to the field supervisor.

SUBSTRATE CLEARING PROCEDURE

For sample locations that have no cleared XRF bare area, paint must be cleared down to the substrate adjacent to the P1 sample area. At these locations, after collection of the P1 sample, collect sample from the XRF bare area down to the substrate in the same manner as collecting other paint samples. If possible, collect paint to enlarge the exposed substrate area to approximately the same width as the adjacent painted XRF testing area. All locations are to be collected into one sample collection container and labeled as a "-P4" sample. More than one set of area dimensions may be required to identify the total area collected for "-P4" samples.

Archive Paint-Chip Collection Reporting Form

Page _____ of _____

Date ________________________ Paint thickness units of measure ____________________

Field Sampler (Printed name) ____

Sample Type: P1 = Original, P2 = Original Field Duplicate, P3 = New Field Replicate, P4 = P1 Complement

Location ID	Sample Type	Dimensions of Area Sampled (cm x cm)	Paint Thickness	Comments

The Measurement of Paint Thickness on the Archive Samples

The Measurement of Paint Thickness on the Archive Samples

Protocol

For U.S. Environmental Protection Agency
Office of Pollution Prevention and Toxics

Battelle Task Order 1-08
Subcontract No. 103639-G002421
MRI Work Assignment No. 5020-08

Protocol Approval

Protocol Title: The Measurement of Paint Thickness on the Archive Samples

Origin Location: Battelle Memorial Institute and Midwest Research Institute

WA Leader(s): Tom Kelly and Paul Constant

Organization WA Leaders(s) and Program Managers		**EPA/OPPT WA Manager(s)**	
Tom Kelly, Battelle	Date	Samuel Brown	Date
Bruce Buxton, Battelle	Date	John Schwemberger	Date
Jack Balsinger, MRI	Date		
Paul Constant, MRI	Date		
Richard Schmehl QuanTech	Date		

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NIST:	M. McKnight		P. Constant		

Protocol for the Measurement of Paint Thickness on the Archive Samples

Introduction

This document provides a testing protocol for measuring the paint thickness on the Lead-based Paint Archive samples using an OG202 Tooke Paint Inspection Gauge (Figure 1). This testing protocol is divided into three sections. Section 1 outlines the procedure to select the measurement locations; Section 2 provides the procedure for the calibration of the Tooke gauge, and Section 3 provides the procedure for the paint thickness measurements.

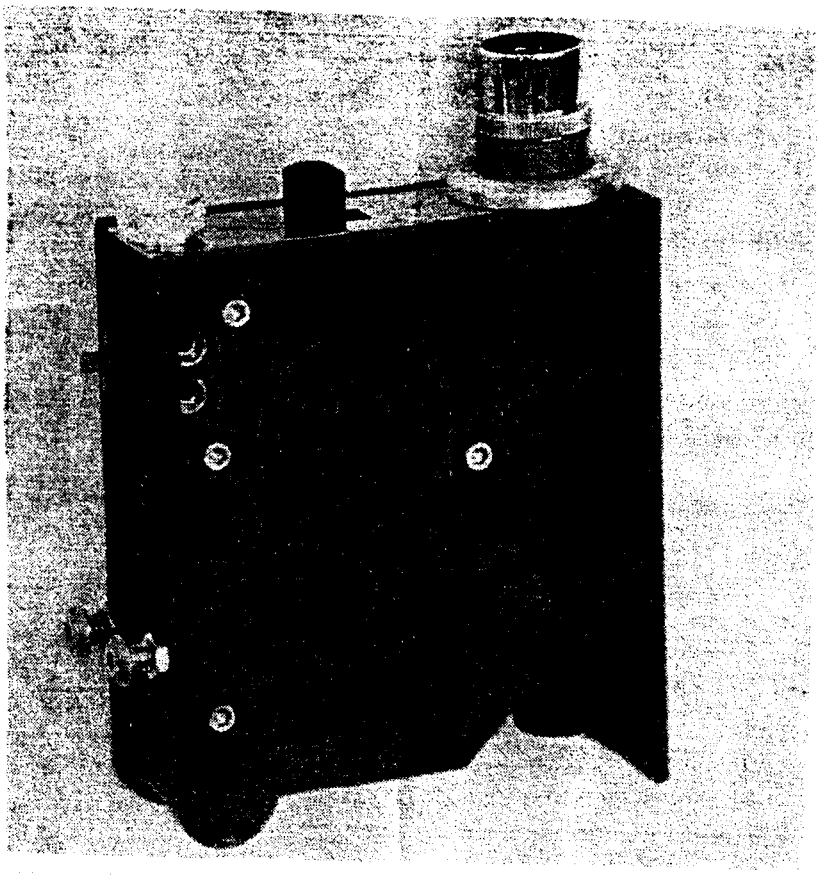


Figure 1. OG202 Tooke Paint Inspection Gauge (approximately actual size)

1 Measurement Location Selection Procedure

The measurement location selection procedure describes the selection, identification, and labeling of the two test locations used for performing the thickness measurements with a Tooke gauge. Section 1.1 provides the operational criteria, Section 1.2 provides procedures for classifying the archive sample test areas, and Section 1.3 gives the documentation requirements for the location.

1.1 Operational Criteria

Following area operational criteria for the Tooke gauge:

- Paint layers must be sufficiently intact to permit a small v-groove ($\approx {}^{1}/_{32}$ in wide by ${}^{1}/_{4}$ in long) to be cut through the paint down just into the underlying base substrate.

- A small mark called a "benchmark" must be drawn, using a contrasting color, across the surface of the paint prior to making the v-groove. This mark is used to identify the top of the paint surface when the paint layers are viewed with the microscopic portion of the Tooke gauge.

Following are the criteria for the archive project:

- Avoid potential damage to XRF areas or destabilization of any of the measurement areas of an archive sample.

- Do not measure where the paint is likely to be damaged in testing due to the presence of textured or paper surfaces.

- Collect normal spatial variability data by:

  a. Measuring the paint thickness on each sample at two (2) separate locations whenever possible. If more measurements are needed, the EPA WAM and the WAL/Supervisor will decide if there are enough painted areas on the sample to do more measurements without having a significant impact on the use of the sample for XRF measurements.

b. Measuring the paint thickness on each sample to ensure that the chosen area for measurement is representative of the normal paint thickness and not of a step-wise progression of different paint layers where one layer is partially peeled off and repainted leaving several visible layers of paint.

1.2 Archive Sample Substrate Considerations

When the Tooke gauge is used on different substrates, the nature of the substrate must be evaluated and the samples must be assessed for the possibility of causing excessive damage to painted surface. Therefore, before the archive sample is marked, the following must be considered:

- On wood or other directional material, incisions must be made in the grain or "machine" direction to avoid ragged cuts.

- With some coatings, improved cuts can be achieved by speeding or slowing the cutting rate.

- Coatings with poor adhesion qualities, even though not apparent during visual inspection, exhibit a ragged line at the substrate interface.

1.3 Selection of Measurement Location and Documentation

An undamaged surface in the test kit area should be used to perform thickness measurements because these areas are already partially damaged and cannot be used for other nondestructive type testing. The decision as to which location to select for performing thickness measurements depends on the test kit area and the type of classification.

Archive samples are classified by one of five types identified below and presented in Attachment 1 for individual archive samples.

- **Type A samples**: Archive samples, where the test kit area is in good condition and two (2) locations are selected, one in the test kit area closest to the XRF bare substrate area and the other farthest away from the XRF bare substrate area.

- **Type B samples**: Archive samples, where the test kit area is in poor condition and the usable areas must be carefully selected. Two locations are selected to maximize the distance between the two measurement locations, as indicated for

type A samples. In addition, care should be taken to ensure that all layers of paint are present and that paint adhesion is sufficient to measure the thickness.

- **Type C samples**: Archive samples, where the test kit area is in bad condition and one location can selected within the test kit block area. If necessary, a location could be placed just outside the test kit area, provided that the operational criteria are met.

- **Type D samples**: Archive samples, where the test kit area is unusable or does not exist, but ample area is available outside of the XRF areas for measurements. Two locations are selected, one each on opposite sides of the primary XRF test area (X1).

- **Type E samples**: Archive samples, where the test kit area is unusable, or does not exist, and there is little or no area available outside the XRF area. The archive sample is reviewed for possible measurement locations. If no area is acceptable for the measurements after the review, the samples are not measured for paint thickness.

The documentation for each measurement location includes:

- Applying operational criteria (Section 1.1) to the sample test area and evaluating, classifying, and documenting them in a project notebook.

- Identifying the thickness measurement location by:

  a. Writing a small number (1, 2, 3, etc.) next to the selected location using an indelible ink pen. Cartesian coordinates will be recorded, in inches, to the closest 1/4 in for each thickness measurement. Record these coordinates in appropriate columns of the thickness data form (Attachment 2). Cartesian coordinates will be measured starting from the 0,0 ordinate which is indicated on each archive samples location with a "0." This "0" is located on the lower, left-most corner of the archive template with a few exceptions that are generally limited to those samples that have no test kit areas. For all samples, the 0,0 ordinate is located at a corner of a testing area. Use the following procedure to measure the coordinates:

     (1) Using the inch grid plastic overlay sheet, place the sheet on the sample and line-up the 0,0 point on the sheet with the 0,0 ordinate on the sample.

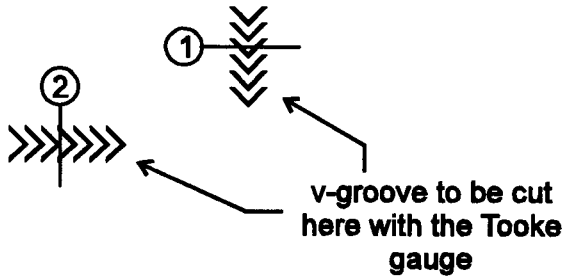
(2) Align the x and y axis on the overlay sheet to coincide with the edges of the template.

(3) It is important to record cartesian coordinates in the same manner as was done for the other testing areas to ensure consistency of the data within the database for this project. Therefore, using a copy of the originally completed cartesian coordinates data for the other testing areas as a tool, verify proper alignment of the overlay sheet by comparing the coordinates of center of the X1 testing area to the originally recorded coordinates. Shift the overlay sheet slightly, as needed, to ensure that the new x and y coordinates match the originals for the X1 testing area. Care should be taken when shifting the overlay sheet to maintain the axis alignments of the overlay sheet parallel with the template reference lines.

(4) Holding the template in place, record the x and y coordinates of the center of the thickness v-groove. Be sure to circle the negative signs for negative coordinate data entries to ensure accurate communication of the coordinates.

b. Circling the number.

c. Drawing a line from the circled number to the area where the thickness measurement is to be performed. This line is drawn so that it will serve as the "benchmark" line that is required to use the Tooke gauge.



- Recording data for each sample including: the archive location, the ID numbering convention of "Sample ID No."—"Thickness Location No." (for example: 81711-1), and the paint thickness reading.

2 Calibration and Check Procedure for the Tooke Gauge

The original calibration at KTA-Tator, Inc., was performed by setting the guide studs in a precise geometric alignment with the cutting tips. The calibration was verified using precision-applied film standards. The geometric alignment of the Tooke gauge will not change unless it is physically damaged; therefore, the manufacturer recommends that calibration be verified periodically using film thickness standards within the normal working range of the materials being tested.

Since a procedure for the preparation and calibration of the Tooke gauge is necessary to ensure reliable measurements, the following procedure for instrument preparation and calibration will be conducted. The use of the procedure will ensure that the gauge is ready to use, that the calibration has been verified, and that a system is in place to check the consistency of readings during the paint thickness measurements of the archive samples.

2.1 The Tooke gauge is prepared for use by checking that:

- there is no physical damage to the gauge
- the gauge is clean
- the batteries are working, and replacement batteries are available
- the light is operational
- the three cutting tips are in good condition and locked in place in the gauge

2.2 Initial and continuing calibration checks:

The calibration of the Tooke gauge is verified using the NIST Certified Coating Thickness Calibration Standards (Table 1). The range for the three sets of SRM 1362a (1362a-91.179, 1359-92.233, and 1363a-93.116) is from 1.57 mils (40.0 μm) to 30.80 mils (782 μm). Each of the NIST Certified Coating Thickness Calibration Standards consists of four (4) coupons (standards) on a card. The coupons measure 30 mm x 30 mm, which is enough area to accommodate 8 cuts in the practical usable area. Since change in the readings occurs only when the gauge or cutting tip is

damaged, new cuts are made in the SRMs only if the gauge or tip has been damaged or if the cutting tip shows significant wear.

Table 1. Certified Coating Thickness Calibration Standards

SRM No.	Film Thickness (μm)	Film Thickness (mils)	Calibration	
			initial/final	continue
1362a-91.179	40.0	1.57	X[a]	X
1359-92.233	48.3	1.90	[b]	[b]
1362a-91.179	77.9	3.07	[b]	[b]
1359-92.233	138.0	5.45	[b]	X
1362a-91.179	146.0	5.75	[b]	[b]
1362a-91.179	207.0	8.15	[b]	[b]
1363a-93.116	256	10.08	X	X
1363a-93.116	399.0	15.69	[b]	[b]
1363a-93.116	492.0	19.37	[b]	[b]
1359-92.233	513.0	20.20	[b]	X
1363a-93.116	617.0	24.30	[b]	[b]
1359-92.233	782.0	30.80	X	X

[a] SRMs used (X).
[b] These calibration standards will be reserved for future measurements.

When the archive samples are being measured for paint thickness, a minimum of three (3) initial calibration thickness measurements are made on the newly cut (grooved) NIST standards. A continuing calibration check is made on previously scribed grooves. Since different coupons are selected for the continuing calibration than for the initial calibration, there will be at least two new scribes out of the 10 continuing calibrations during the measurement process. After every 15 archive samples, a continuing calibration measurement is made that is the closest to the thickness of the last measured archive sample. These NIST SRMs encompass the expected range of paint thickness found in the archive samples. The three (3) cutting tips used to scribe the NIST SRM are given in Table 2. Newly cut grooves in the NIST SRMs for continuing calibration checks will be made after every 30 measurements of the archive samples and at the end of the measurement activity. Newly cut grooves also will be made, with the approval of the supervisor, if there is perceived damage to the cutting tip.

Table 2. Cutting Tip Selection Guide

Cutting tip	Maximum coating thickness	Uncertainty of thickness determination	1 Division on the reticle scale represents
1x	50 mil	±0.25 mil	1.0 mil
2x	20 mil	±0.13 mil	0.5 mil
10x	3 mil	±0.025 mil	0.1 mil

The procedure for the initial calibration is as follows:

- Select the NIST coupon card (4 standards) and place in a holder designed to assist in properly scribing the measurement groove.

- Using a black or blue marker, draw a short horizontal line (≈ ¼") on the selected NIST calibration standard (Table 1).

- Select a cutting tip that provides the lowest uncertainty, but for which the film thickness does not exceed the maximum coating thickness (Table 2).

- Place the Tooke gauge with the selected tip against the SRM surface slightly above the horizontal marked line to form a tripod with the cutting tip and the guide studs.

- Maintain moderate pressure but continuous three point contact and draw the gauge down against the SRM surface, starting with the cutting tip slightly above the horizontal marked line. Cut a short (≈ $^1/_4$ in) v-groove across the horizontal marked line.

- Using the microscopic portion of the gauge, view the intersection of the horizontal marked line and the v-groove. If the image of the groove is unclear, focus the image using the thumb screw located on the body of the gauge below the microscope. If the reticle (scale) image is unclear, sharpen the reticle image by adjusting the eyepiece of the microscope.

- Using the wall of the groove with an angle of ≥ 45°, align the top edge of the paint with one of the major divisions of the reticle image and count the number of units (marks) between the top and the bottom of the paint layer. Record the clock time, the cutting tip used, and the measurement reticle image on a copy of

the report form "Calibration Measurements for the Tooke Gauge" (Attachment 2). Also indicate in the comment column if a new scribe was used for the measurement.

- Calculate the paint film thickness by dividing the number of units read on the reticle image by the cutting tip designation.

  Example: The reading of 12 units using cutting tip 2x

  $$\text{mil} = \frac{12}{2} = 6 \text{ mils paint thickness}$$

  or for μm, the conversion is mils x 25.4

  $$\mu\text{m} = \frac{(12)(25.4)}{2} = 152.4 \ \mu\text{m}$$

NOTE: The precision of individual observations of a uniform coating on a smooth substrate has been determined to be within ± 10 % (ASTM D4138-94 "Standard Test Methods for Measurement of Dry Film Thickness of Protective Coating Systems by Destructive Means").

3 Paint Thickness Measurement Procedure

Following is a summary of the testing procedure for the thickness measurement activities. The order in which the measurements are taken is the same as the XRF measurement order.

3.1 Using a black or blue marker, draw a short horizontal line (≈ $^1/_4$ in) at the selected thickness measurement location.

3.2 Determine the cutting tip to be used according to Table 2. Select a cutting tip that provides the lowest uncertainty, but for which the likely paint thickness does not exceed the maximum coating thickness. If the paint thickness can not be estimated, use the 2x tip.

3.3 Place the Tooke gauge with the selected tip against the painted surface slightly above the horizontal marked line to form a tripod with the cutting tip and the guide studs.

3.4 Maintain moderate pressure, but continuous three point contact, and draw the gauge down against the painted surface starting with the cutting tip slightly above the

horizontal marked line. Cut a short (≈ $^1/_4$ in) v-groove across the horizontal marked line.

NOTE: Care must be taken to avoid damaging any of the painted surface areas when using the Tooke gauge in cutting the grooves. Avoid sliding the gauge across existing paint edges.

3.5 Using the microscopic portion of the gauge, view the intersection of the horizontal marked line and the v-groove. If the image of the groove is unclear, focus the image using the thumb screw located in the body of the gauge below the microscope. If the reticle (scale) image is unclear, sharpen the reticle image by adjusting the eyepiece of the microscope.

3.6 Using the wall of the groove with an angle of ≥ 45°, align the top edge of the paint with one of the major divisions of the reticle image and count the number of units (marks) between the top and the bottom of the paint layer. Record the measurement from the reticle image using a copy of the report form "Measurement of Paint Thickness" (Attachment 2).

3.7 The paint film thickness is calculated by dividing the number of units read on the reticle image by the cutting tip designation.

Example: The reading of 12 units using cutting tip 2x

$$\text{mil} = \frac{12}{2} = 6 \text{ mils paint thickness}$$

or for μm, the conversion is mils x 25.4

$$\mu\text{m} = \frac{(12)(25.4)}{2} = 152.4 \ \mu\text{m}$$

3.8 If the cutting tip is found to be inappropriate for the measured thickness, select a different cutting tip and mark a new area immediately adjacent to the first cut with the next number and identify the location (Section 3.1a). Make a new cut and measure the paint film thickness (3.3-3.7).

Attachment 1

Testing Order Numbers of Archive Samples for Each Board Classified by Type for Use for Paint Thickness Sample Selection

BOARD	TYPE (see page 3 of 10)				
	A	B	C	D	E
A	65, 91	80	75, 137		
B		62, 70, 103, 104, 150		51	66
C	127, 132, 140, 143	54, 112	22, 38		
D	15, 96, 153	92	9, 120, 125	115	101, 151
E	76, 138	40, 47, 121	3, 33, 87, 94	109	
F	44, 78, 99	12, 28, 45, 117			
G	93	49, 68	105, 128		
H	25	98, 124, 146	85, 100	24	
I	20				
J		11, 61, 131	13, 119		111, 147
K	6, 67, 90	23, 52, 106, 129, 142		56	
L	81	1, 8, 130, 144	34, 50, 64, 79, 152		
M	10, 16, 48, 149	42	88	63, 134	
N		17, 55	14, 41, 114, 133		
O	36, 83	82, 139	27, 35, 116		110
P	84, 123, 135, 145	46, 58, 113			57
Q		74, 95, 154	21, 26, 30, 37, 43, 102	7	
R	18, 29, 39, 108, 136	72, 122		5, 4	
S			73A, 113A, 115A		8A
Back Sides (All Boards)	60	2, 59, 107, 118	77	31, 32, 53, 69, 71, 73, 86, 89, 97, 126, 141, 148	19

Attachment 2

Date: _____

Measurements taken by: _____

Calibration Measurements for the Tooke Gauge

Cal. type*	Testing clock time	SRM ID	Cutting tip	Measurement (scale units)	Calculated thickness (mils)	Comments

* Calibration type—1 = beginning of the day; 2 = continuing; 3 = end of the day.

Pg ___ of ___

Date: _____

Measurements taken by: _____

Measurements of Paint Thickness

Testing order No.	Sample ID	Cart. coord.		Cutting tip	Measurement (scale units)	Calculated thickness (mils)	Comments
		x	y				

Pg ___ of ___

Sampling Procedure for Control Blocks
[Revised 5/17/95]

1. Background

There is need to determine the lead content of each of the six control blocks used in the test of portable XRF devices. The control blocks are of the following material: brick, concrete, plaster, metal, drywall, and wood. An important step to this end is to obtain a representative sample from each type control block. The principal assumption made is that the material of each block is homogenous throughout.

2. Equipment

1 Adjustable speed drill press
1 Drill press vise
1 High-speed, ½ metal drill bit for metal, wood, plaster, and drywall control blocks
1 Carbide-tip, ½ masonry drill bit for brick and concrete control blocks
1 8-in x 10-in plastic photo-developing tray
2 Wood blocks (2-in x 4-in x ¼-in) for keeping the jaws of the vise from damaging the control blocks
1 Funnel
Gloves and safety eye glasses
baby wipes and laboratory wipes
6 Centrifuge tubes
Labels for the centrifuge tubes

3. Sampling procedure

3.1 Place each control block in a clean Ziploc bag and seal.

3.2 Adjust the drill press so that its angular velocity is slow enough for the material drilled to stay on top of the control block.

3.3 Don safety glasses and blow down the drill press and the drill press vise. Don a pair of gloves, and then wipe down the drill press, vise, and drill bit, using baby wipes followed by laboratory wipes. Be sure all parts are dry. Place drill bit in chuck and tighten chuck.

3.4 Place the clean plaastic photo-developing tray on the drill press table.

3.5 Remove and dispose of gloves. Don a new pair of gloves.

3.6 Remove control block from plastic bag and place in vise. Place a wood block on either side of the block and secure the block in vise.

3.7 Place vise into photo-developing tray. Adjust drill press so that drill bit will penetrate the block but will not break the bottom surface of the block.

3.8 Drill at least three holes to the depth desired to obtain sufficient sample, carefully retracting drill bit from each hole.

3.9 Carefully remove block from vise and transfer drilled material from the top of the block into a clean centrifuge tube via a clean funnel. Secure top onto centrifuge tube. Label the tube using indelible ink with the control block set number, substrate type, date, and collector's name. Remove gloves and dispose of them.

3.10 Repeat steps 3.2 through 3.9 until samples required have been acquired.

3.11 When ready to digest sample, shake centrifuge tube to thoroughly mix the particulate material. Subsample to obtain the amount of material needed, and proceed with weighing and other necessary steps in preparing the sample for analysis.

Note: One of the peer reviews of the report commented that collected material should be ground before subsampling, as drilling may produce large particles or pieces. The protocol above was found to provide adequate samples; however, this step recommended by the reviewer will be added to the above protocol if this protocol is used in future work.

Density Measurement Procedure for Control Blocks [5/17/95]

Density measurements of each control block (brick, concrete, plaster, metal, drywall, and wood) will be made by the following procedure:

For metal, drywall, and wood control blocks -

1. Weigh the control block and record mass measurement in grams.

2. Measure the width, length, and depth of the control block and record each measurement in cm.

3. Calculate the volume of the control block in cm^3.

4. Calculate the density of the control block by dividing the mass by the volume. Record the density determined.

For brick, concrete, and plaster -

Since these control blocks are not of uniform shape, cut out a uniform sub-block, and then follow the following procedure:

1. Weigh the control block sub-block and record the mass measurement in grams.

2. Measure the width, length, and depth of the control block sub-block and record each measurement in cm.

3. Calculate the volume of the sub-block in cm^3.

4. Calculate the density of the subunit by dividing the mass by the volume. Record the density determined.

Microwave Assisted Acid Digestion of Environmental Samples (SW-846 Method 3052)

Title of Method: **Microwave Assisted Acid Digestion of Environmental Samples (SW-846 Method 3052)**

1 Summary of Method

1.1 A representative sample of up to 0.5 g is digested in 9 mL of concentrated nitric acid and 3 mL hydrofluoric acid for 15 min using microwave heating with a suitable laboratory microwave unit. The sample and acid are placed in a fluorocarbon heavy duty microwave vessel (HDV). The vessel is capped and heated in the microwave unit. After cooling, the vessel contents are transferred to volumetric flasks, diluted to volume, and analyzed by the appropriate SW-846 method (ASF-601, ASF-602). The method is based upon EPA draft Method 3052, Revision 0, January 1995.

1.2 This method is applicable to the microwave assisted acid digestion of ash and other siliceous wastes. Sludges, sediments, soils, and oil contaminated soils may be digested using this method if a total decomposition (relative to the target analyte list) analysis is required. This method is applicable for the following elements:

Aluminum	Bismuth	Gold	Molybdenum	Strontium
Antimony	Cadmium	Iron	Nickel	Thallium
Arsenic	Calcium	Lead	Potassium	Titanium
Boron	Chromium	Magnesium	Selenium	Vanadium
Barium	Cobalt	Manganese	Silver	Zinc
Beryllium	Copper	Mercury	Sodium	

1.3 This method is provided as a rapid multielement, total acid digestion prior to analysis protocol so that decisions can be made about the site or material. Digests and alternative procedures produced by the method are suitable for analysis by flame atomic absorption (FLAA), graphite furnace atomic absorption (GFAA), inductively coupled plasma optical emission spectroscopy (ICP-OES) and inductively coupled plasma mass spectrometry (ICP-MS) (see Appendix A, Table A-1 for estimated detection limits).

2 Required Information

To perform this procedure, the following minimum amount of information is required:

1. Project charge number
2. List of all samples to be processed
3. Sample matrix type

4. List of analytes

5. Assessment of sample hazard level and any special safety precautions

6. Required QC information including a list of samples to be spiked and/or prepared in duplicate, required spiking analytes, spike levels, and spiking procedure

7. Copies of relevant analysis plans, quality assurance plans, interoffice memos, etc.

3 Apparatus and Materials

All glassware and plasticware should be cleaned according to SOP ASF-201. Microwave vessels should be cleaned according to manufacturer's specifications.

3.1 Heavy duty microwave vessels (Figure 1)

3.2 Graduated cylinders: 100 mL

3.3 Analytical balance capable of weighing to an accuracy of 0.0001 g

3.4 Analytical balance capable of weighing 1 kg to an accuracy of 0.01 g

3.5 Volumetric flasks with stoppers: 100 mL, Class A or polypropylene if hydrofluoric acid is used.

3.6 Centrifuge

3.7 Centrifuge tubes: polyethylene with screw caps, 50-mL capacity

3.8 Food service towels

3.9 Kimwipes

3.10 Filter paper, Whatman 541, or equivalent

3.11 Plastic or glass funnels

3.12 Gloves: disposable, powderless, vinyl

3.13 Funnels: plastic or glass sized to fit into the 100-mL volumetric flasks and Whatman 541 filter paper

3.14 Centrifuge tubes: plastic, sized to fit available centrifuge, minimum volume of 20 mL

3.15 Microwave apparatus requirements (CEM Model MDS 2100)

3.15.1 The microwave unit provides programmable power with a minimum of 600 W, which can be programmed to within ±12 W of the required power. Typical units provide a nominal 600 to 1200 W of power. Temperature and pressure monitoring and controlling the microwave unit is desirable. The temperature performance requirements necessitate the microwave decomposition system to sense the temperature to within ±2.5°C and automatically adjust the microwave field output power within 2 seconds of sensing. Temperature sensors should be accurate to ±2°C; verification at two points > 50°C apart should be determined periodically. Temperature feedback control provides the primary control performance mechanism for the method.

3.15.2 The microwave unit cavity is corrosion resistant and well ventilated.

3.15.3 All electronics are protected against corrosion for safe operation.

3.15.4 The system requires fluorocarbon (HDV) lined digestion vessels (80- to 120-mL capacity) capable of withstanding pressures up to 40 atm (580 psi) and capable of controlled pressure relief at pressures exceeding 40 atm (580 psi).

3.15.5 A rotating turntable is employed to ensure homogeneous distribution of microwave radiation within the unit. The speed of the turntable should be a minimum of 4 rpm.

CAUTION: A safety concern relates to the use of sealed containers without pressure relief valves in the unit. Temperature is the important variable controlling the reaction. Pressure is needed to attain elevated temperatures but must be safely contained. However, many digestion vessels constructed from certain fluorocarbons may crack, burst, or explode in the unit under certain pressures. Only fluorocarbon (such as PFA or TFM) lined containers with pressure relief mechanisms or container with PFA-fluorocarbon liners and pressure relief mechanisms are considered acceptable at present.

4 Reagents

4.1 Milli-Q reagent water: minimum resistance of 16.67 MΩ-cm or equivalent

4.2 Concentrated nitric acid (HNO_3)—70% to 71%: Baker Instra-Analyzed or equivalent

4.3 Concentrated hydrochloric acid (HCl): 35% to 38%: Baker Instra-Analyzed or equivalent

4.4 Concentrated hydrofluoric acid (HF): 48% to 51%: Baker Instra-Analyzed or equivalent

5 Quality Control

Quality control samples to be prepared along with submitted samples are discussed in the following sections and summarized in Table 1. Not all of the QC samples listed may be required for every project. QC samples may be added or deleted from the suggested list for other projects. Project specific QC requirements with be outlined by the appropriate person: facility manager, project leader, or designee prior to starting this procedure.

Table 1. Quality Control Samples

QC samples	Definition	Typical frequency
Method blanks	Milli-Q reagent water—digest as a sample with addition of all reagents. Should reflect the maximum treatment given any one sample.	1 per 20 samples, a minimum of 1 per batch
Spiked samples	A portion of a sample is fortified with all the target analytes before preparation. Analytes, standards, and spike levels are specified by the facility manager, project leader, or designee.	1 per 20 samples per matrix type, a minimum of 1 per batch
Duplicates (either duplicate of samples or spikes)	Two equal portions of a homogenized sample are prepared and analyzed independently, or two spiked samples are prepared at the same spike level from the same sample.	1 per 20 samples per matrix type, a minimum of 1 per batch
Laboratory control spike (LCS) (spiked method blank)	A blank is fortified with all target analytes before preparation. Analytes, standards, and spike levels are specified by the facility manager, project leader, or designee.	2 per 20 samples, a minimum of 1 per batch
Reference material (standard reference)	A material of known composition, where the analyte levels are certified by the manufacturer (e.g., lead paint dust from NIST).	As required by project, usually 1 per batch of samples

A batch of samples is defined as a group of samples which are prepared together in a specified time period (see Appendix A, Table A-2 for a complete list of samples to be processed along with QC sample).

5.1 For each batch of samples, a method blank (Milli-Q water and reagents), a spike, a spike duplicate, and two LCS are typically carried throughout the entire sample preparation and analytical process. The inclusion of a reference material and the choice of either a duplicate sample or a duplicate spike is specified in the quality assurance project plan. Any questions must be directed to the Work Assignment Leader.

5.2 Spiked samples are processed on a routine basis with each sample batch to estimate method accuracy expressed as percent recovery, relative to the true spiked value. A spiked sample is a sample aliquot (split from an original sample) which is spiked with a known amount of analyte. All standard stock sources and concentrations used for spiking are recorded in the laboratory notebook. The facility manager, project leader, or designee will provide directions on the analytes and spiking levels to be used for each specific project. Spiked samples are typically processed at a frequency of 1 for every 20 actual samples per matrix type. A minimum of 1 spike is required for each batch processed.

5.3 Duplicates of samples and/or spikes will be prepared to provide precision data on sample batch processing. At least one duplicate should be processed at a frequency of 1 for every 20 samples per matrix type. A minimum of 1 duplicate and/or spike duplicate is required for each batch processed.

5.4 An LCS (a spiked method blank) is not required in SW-846 Method 3052; however, it is recommended. An LCS is a method blank which is spiked with the analytes of interest and is digested. Two LCSs should be processed with each batch of samples. The LCS is used to verify sample preparation efficiency in the absence of sample matrix effects. If spiked sample recovery data do not meet data quality objectives, then the LCS will be used to determine whether this is a result of losses during the sample digestion procedure or is likely due to sample matrix effects.

6 Interferences

6.1 Very reactive or volatile materials that may create high pressures when heated may cause venting of the vessels with potential loss of sample and analytes. The complete decomposition of either carbonates, or carbon-based samples, may cause enough pressure to vent the vessel if the sample size is greater than 0.25 g when used in the 80- to 120-mL vessels with a pressure relief device capable of withstanding pressures up to 40 atm (580 psi).

6.2 Most samples will be totally dissolved by this method with judicious choice of the acid combinations used. A few refractory compounds, such as TiO_2, alumina, and other oxides may not be totally dissolved.

7 Procedure

7.1 Preliminary Activities

Before samples are prepared, the following information must be provided:

1. Samples to be prepared

2. QC samples:

   - Number of blanks
   - Number of spikes
   - Number of duplicates
   - Number of LCSs
   - Number and type of reference materials

3. All spiking levels including:

   - Level in sample
   - Stock standards
   - Any dilutions of standards
   - Spike volumes

7.2 Temperature control of closed vessel microwave instruments provides the main feedback control performance mechanism for the method. Control requires a temperature sensor in one or more vessels during the entire decomposition. The microwave decomposition system should sense the temperature to within ±2.5°C and permit adjustment of the microwave output power within 2 seconds.

7.3 All volumetric ware must be carefully acid washed and rinsed with reagent water according to ASF-201. Microwave vessels are cleaned according to the manufacturer's specifications.

7.4 Sample Digestion

7.4.1 Balance Calibration Check

The balance calibration should be checked at a minimum of one level, equal to approximately the tare and actual weight of the sample or microwave vessel. Standard weights should be Class S.

- For sample weights, the observed mass of the calibration weight (not including the tare weight) must be within 0.5% of the reference mass. Since a 500-mg sample is to be weighed into a vessel: an empty vessel is tared, and then a 500-mg calibration weight is added, and it must give an observed mass of 500 ±2.5 mg. This check should be done before and after sample weighing.

- For vessel weights, the observed mass of the calibration weight must be within the tolerance of the balance. Since the balance tolerance is ±0.01 g, the observed mass of the weight should be within 0.01 g of a reference mass.

- If the balance calibration does not pass this test at the beginning of weighing, the balance should be repaired or another balance should be used.

- If the balance calibration does not pass this test at the end of weighing, the samples or standards should be reweighed using a balance that can meet these requirements.

- Balance calibration results and the MRI number of the calibration weights should be recorded in the project records documenting the preparation of samples.

7.4.2 Calibration of Microwave Equipment

Since the microwave unit uses temperature feedback control to follow performance specifications of the method, only the temperature probe is calibrated once every 6 months using the manufacturer's recommended procedure. The temperature of the system is checked by immersing a thermowell containing the fiber optic probe in a beaker of water at room temperature. The temperature is compared to a thermometer that is NIST traceable. If the temperature of the system and the NIST traceable thermometer drops by more than 1.5°C, recalibrate the system. Record in a notebook the system temperature and the thermometer readings.

7.4.3 Weigh a well-mixed sample to the nearest 0.001 g into the fluorocarbon sample vessel liner. For soils, ash, sediments, and sludges use no more than 0.5 g. For oil-contaminated soils use no more than 0.2 g.

7.4.4 Add 9 mL concentrated nitric acid and 4 mL concentrated hydrofluoric acid in a fume hood. Either a repipettor or Eppendorf pipet may be used. If a vigorous reaction occurs, allow the reaction to stop before continuing. The hydrofluoric acid content may be varied by ±2 mL as the method is a performance-based method and optimal concentrations of hydrofluoric acid depends on the silicon dioxide content of the sample. Samples with higher concentrations of silicon dioxide > 70% may require higher concentrations of hydrofluoric acid. Alternatively, samples with smaller concentrations of silicon dioxide < 10% may require much less hydrofluoric acid.

7.4.5 Add 4 mL concentrated hydrochloric acid in a fume hood If a vigorous reaction occurs, allow the reaction to stop before capping the vessel. Cap the vessel and torque the cap according to the unit manufacturer's directions. Weigh the closed vessels to the nearest 0.01 g. Place the vessels in the microwave carousel. This alternative acid addition is appropriate for the stabilization of Sb and Ag and high concentrations of Fe and Al. It will, however, limit the analysis to flame atomic absorption (FLAA), and inductively coupled plasma emission spectroscopy (ICP-ES) and eliminate the analysis by both graphite furnace atomic absorption (GFAA), and inductively coupled plasma mass spectrometry (ICP-MS).

7.4.6 OPTION 1. If the samples still retain undigested organic matter, hydrogen peroxide may be added prior to capping the vessels. Peroxide (30%) may be added in small and catalytic quantities 0.1 to 2 mL with an Eppendorf if the pressure capabilities and equipment specifications permit. Alternative and variable reagent concentrations

must be implemented on equipment capable of monitoring and controlling such reactions and demonstration of safe operation must precede its use.

CAUTION: Only one acid mixture may be used in a single batch in the microwave to permit monitoring to uniformly be representative.

CAUTION: Toxic nitrogen oxide fumes may be evolved, therefore all work must be performed in a properly operating ventilation system. The analyst should also be aware of the potential for a vigorous reaction. If a vigorous reaction occurs, allow to cool before capping the vessel.

CAUTION: Toxic hydrogen fluoride fumes may be evolved, therefore all work must be performed in a properly operating ventilation system. The analyst should wear protective gloves and face protection and must not at any time permit a solution containing hydrofluoric acid come in contact with skin or lungs.

CAUTION: When adding hydrochloric acid, toxic chlorine fumes may be evolved, therefore all work must be performed in a properly operating ventilation system. The addition of hydrochloric acid must be from concentrated acid and not from a premixed combination of acids as a buildup of chlorine gas will result from a premixed acid solution. The analyst should also be aware of the potential for a vigorous reaction. If a vigorous reaction occurs, allow to cool before capping the vessel.

CAUTION: When digesting samples containing volatile or easily oxidized organic compounds, initially weigh no more than 0.10 g and observe the reaction before capping the vessel. If a vigorous reaction occurs, allow the reaction to cease before capping the vessel. If no appreciable reaction occurs, a sample weight up to 0.25 g can be used.

CAUTION: The addition of peroxide should only be done when the reactive components of the sample are known. It may react rapidly and violently on easily oxidizable materials and should not be added if unknown organic constituents are present.

7.4.7 Properly place the carousel in the microwave unit and connect appropriate temperature and pressure sensors to the monitoring vessels. When temperature feedback control is being used, six vessels either with samples and reagents or just reagents are loaded in the carousel. The temperature of the monitoring vessel should rise to 180 ± 5°C in less than 5.5 min and remain between 180 ± 5°C for 9.5 min. The pressure should peak between 5 and 15 min for most soil, fly ash, sludge, and sediment samples.[7,8,5] The pressure may exceed these limits in the case of high concentrations of carbonate or organic compounds. In those cases the pressure will be limited by the relief pressure of the vessel. All vessels should be sealed according to the manufacturer's recommended specifications.

7.4.7.1 For reactive substances, the heating profile may be altered for safety purposes. The samples may be heated at a slower rate to prevent potential uncontrollable exothermic reactions. The time to reach 180 ± 5°C may be increased to 10 min provided that 180 ± 5°C is subsequently maintained for 9.5 min.

7.4.8 If calibration control is being used any vessels containing acids for analytical blank purposes are counted as sample vessels and when fewer than the recommended number of samples are to be digested, the remaining vessels should be filled with the same nitric and hydrofluoric acid mixture (with HCl also optional) to achieve the full complement of vessels. This provides an energy balance since the microwave power absorbed is proportional to the total mass in the cavity.[4] Irradiate each group of sample vessels.

7.4.9 If the hydrofluoric acid concentration is a consideration in the analysis technique such as with ICP methods, a hydrofluoric acid resistant torch may be used. Alternatively, boric acid at a concentration of 0.0125M H_3BO_3 may be added to permit the complexation of the fluoride to protect the quartz plasma torch.

7.4.10 At the end of the microwave program, allow the vessels to cool for a minimum of 5 min before removing them from the microwave unit. When the vessels have cooled to room temperature, weigh and record the weight of each vessel assembly. If the weight of acid plus sample has decreased by more than 1.0% from the original weight (≈ 0.25 g), discard the sample. Determine the reason for the weight loss. These are typically attributed to loss of vessel seal integrity, use of a digestion time longer than 15 min, too large a sample, or improper heating conditions. Once the source of the loss has been corrected, prepare a new sample or a set of samples for digestion beginning at 7.3.2.

Weight difference = (Initial–digested) vessel weight

7.4.11 Complete the preparation of the sample by carefully uncapping the venting each vessel in a fume hood. Vent the vessel using the procedure recommended by the vessel manufacturer. Transfer the sample into a 100-mL volumetric polypropylene flask and dilute the digest to volume with Milli-Q water. If the digested sample contains particulates which may clog nebulizers or interfere with injection of the sample into the instrument, the sample may be centrifuged, allowed to settle, or filtered.

7.4.11.1 Settling: If undissolved material remains such as TiO_2, or other refractory oxides, allow the sample to stand until the supernatant is clear. Allowing a sample to stand overnight will usually accomplish this. If it does not, centrifuge or filter the sample.

7.4.11.2 Centrifugation: Centrifugation at 2,000 to 3,000 rpm for 10 min is usually sufficient to clear the supernatant.

7.4.11.3 Filtering: If necessary the filtering apparatus must be thoroughly cleaned and prerinsed with dilute (approximately 10% v/v) nitric acid. Filter the sample through qualitative filter paper into a second acid-cleaned container. Samples may be acrodisced prior to analysis.

7.5 Transfer 50 mL of the diluted digest to a centrifuge tube and analyze by ICP or GFAA.

7.6 Obtain a printout of the digestion performance and place in the batch file for verification of the digestion parameters.

8 References

1. *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods*, 3rd Ed., U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, U.S. Government Printing Office, Washington, D.C., 1986; SW-846.

2. Kingston, H. M. and L. B. Jassie, "Safety Guidelines for Microwave Systems in the Analytical Laboratory," in *Introduction to Microwave Acid Decomposition: Theory and Practice*, Kingston, H. M. and Jassie L. B., eds., ACS Professional Reference Book Series, American Chemical Society, Washington, D.C., 1988.

3. *1985 Annual Book of ASTM Standards*, Vol. 11:01, "Standard Specification for Reagent Water, ASTM, Philadelphia, Pennsylvania, 1985, D1193-77."

4. *Introduction to Microwave Sample Preparation: Theory and Practice*, Kingston, H. M. and Jassie, L. B., eds., ACS Professional Reference Book Series, American Chemical Society, Washington, D.C., 1988.

5. Kingston, H. M., EPA IAG #DWI-393254-01-0 January 1-March 31, 1988, quarterly report.

6. Binstock, D. A., W. M. Yeager, P. M. Grohse, and A. Gaskill, *Validation of a Method for Determining Elements in Solid Waste by Microwave Digestion*, Research Triangle Institute Technical Report Draft, RTI Project Number 321U-3579-24, November 1989, prepared for the Office of Solid Waste, U.S. Environmental Protection Agency, Washington, D.C. 20460.

7. Kingston, H. M. and P. J. Walter, "Comparison of Microwave Versus Conventional Dissolution for Environmental Applications," *Spectroscopy*, Vol. 7, No. 9,20-27, 1992.

8. Kingston, H. M. and S. Haswell, *Microwave Enhanced Chemistry*, ACS Professional Reference Book Series, American Chemical Society, Washington, D.C., 1995 (in press).

9. Kingston, H. M., P. J. Walter, E. M. L. Lorentzen, and G. P. Lusnak, Report to NIST Office of Standard Reference Materials, "The Performance of Leaching Studies on Soil SRMs 2170 and 2711," Duquesne University, 1994.

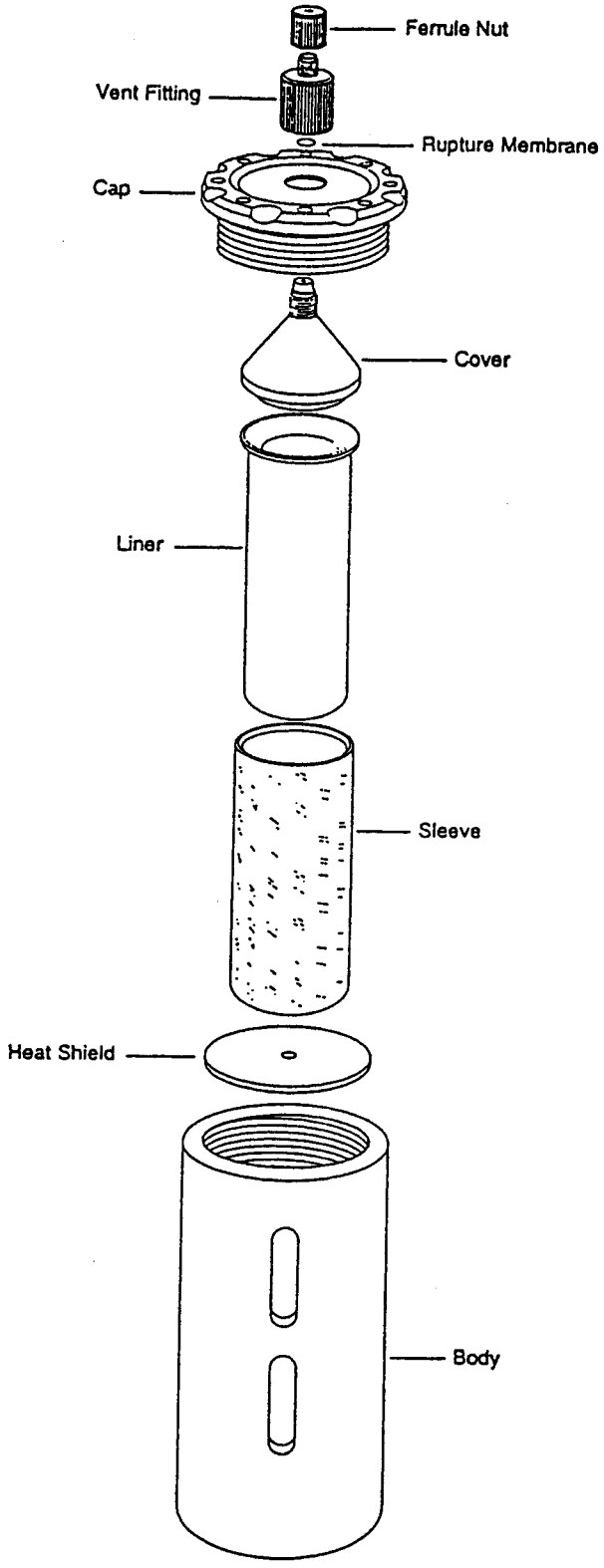


Figure 1. Standard Heavy Duty Vessel

Procedure for Changing the Archive Testing Order (August 12, 1996)

PROCEDURE FOR CHANGING THE ARCHIVE TESTING ORDER

NEEDED SUPPLIES and INTRODUCTION

- A copy of this procedure, which is presented in two steps. The first step (A) should be done by a different person than the second step (B).
- One set of *Archive Testing Order Change Forms* (hereafter referred to as **the Forms**).
- Cards: i) business size cards (2" x 3.5");
  ii) either pink or white (different in color from the cards with the current testing order numbers);
  iii) labeled 1 through 158 using large numbers so that the cards are placed vertically (i.e., long side up and down) in their holders.

Note 1: This procedure assumes that the current testing order cards are white.

STEP A: PLACE THE NEW ORDER NUMBER CARDS

A.1 Complete the appropriate lines in the header on each page of **the Forms**.

A.2 Starting with Board A and moving sequentially through Board S, perform the following at each test board:

A.2.1 Using **the Forms** as a worksheet, locate the first white card with the current order number without a corresponding entry in the ***1st CK*** column. Pull out a pink card with the new order number and place this card in-front-of the white card with the current order number. **Do not remove the white card.**

A.2.2 Using **the Forms**, place an "X" in the ***1st CK*** column on the appropriate row to indicate that a pink card has been placed over the white card with the current order number.

A.2.3 Repeat steps A.2.1 and A.2.2 until all samples on the board show only pink cards (no white cards showing). Before moving to then next board, verify that an "X" has been entered in the ***1st CK*** column on all rows of **the Forms** for this board letter. If not, then investigate, make corrections, and note any needed corrective action in the comments column of **the Forms** before moving on to A.3.

A.3 Go to the next board letter and repeat A.2 until all samples on all boards show only pink cards.

STEP B: VERIFY CARD PLACEMENT AGAINST FIELD SAMPLE ID NUMBERS

B.1 Obtain **the Forms** used during Step A. No entries should have been made under the ***2nd CK*** column.

B.2 Starting with Board A and moving sequentially through Board S, perform the following at each test board:

B.2.1 Using **the Forms**, identify the first new order number without an entry in the ***2nd CK*** column. On the board, locate the pink card with the matching new order number and lift the plastic sleeve holder enough to view the field sample identification number written on the board underneath the sleeve. To verify that the field identification number on the board matches that shown on **the Forms** do the following.

i) **If** the numbers match, **then** place an "X" in the *2nd CK* column on the appropriate row on **the Forms** to indicate that the numbers match.

ii) **If** the numbers do not match, then

a) **record all three numbers** in the comments column of the appropriate row: *Pink = xxx, White = xxx, Field ID = xxxxxx* (where x's are the actual numbers found at that test location);

b) **make corrections** as needed to fix the problem noting in the comments column what corrective actions were taken;

c) **place an "X"** in the *2nd CK* column on **the Forms**, after corrective action was taken.

B.2.2 Repeat B.2.1 until all numbers on the board have been verified and have "X" entries in the ***2nd CK*** column on all rows of **the Forms** for this board letter. If not, then investigate, make corrections, and note any needed corrective action in the comments column of **the Forms** before moving on to the next step.

B.3 Go to the next board letter and repeat B.2 until all samples on all boards have been verified.

ARCHIVES TESTING ORDER CHANGE FORM page 1 of 8

Name of person performing 1st check _____

Date _____

Name of person performing 2nd check _____

BOARD LETTER	CURRENT ORDER No.	NEW ORDER No.	1st CK	FIELD SAMPLE ID	2nd CK	COMMENTS

50272-101

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14.

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16. Abstract (Limit: 400 words)

An archive of selected housing components were collected during a multi-city field study on lead-based paint technologies (Report No. EPA 747-R-95-002a). The archived housing components represent a variety of substrates (drywall, concrete, brick, metal, plaster, and wood) which were found to have both high and low-level lead contamination in the paint as determined by chemical analysis of paint chip samples. The archive has been used to study the technical performance of different portable X-ray fluorescence (XRF) instruments by statistical evaluation and comparison to laboratory analysis data and quality control samples. This report documents the operation of the archive facility and the testing protocols used during its operation. Based on the archive testing, technical performance of the XRF instruments is measured in terms of bias and precision relative to the amount of lead contamination and category of substrate. The results of the XRF testing are published as XRF Performance Characteristic Sheets (PCS) which are available from the National Lead Information Center by calling 1-800-424-LEAD.

17. Document Analysis a. Descriptors

lead-based paint, lead-based paint testing, archive facility, evaluation testing, testing new instruments

b. Identifiers/Open-Ended Terms

X-ray fluorescence instruments, XRF instruments, portable XRF, XRF testing protocols, XRF Performance Characteristic Sheets, PCS

c. COSATI Field/Group

18. Availability Statement	19. Security Class (This Report) UNCLASSIFIED	21. No. of Pages 208
	20. Security Class (This Page) UNCLASSIFIED	22. Price

(See ANSI-Z39.18) See Instructions on Reverse OPTIONAL FORM 272 (4-77) (Formerly NTIS-35) Department of Commerce