

# Thermo Scientific Niton XRF Analyzers

## A Guide to ELPAT Proficiency for Portable Laboratories Using a Niton® XRF Analyzer for Lead in Soil and Lead on Dust Wipes

### What is ELPAT?

*Environmental Lead Proficiency Analytical Testing, ELPAT is the process of accrediting laboratories that analyze environmental samples for lead in soil, paint, air filters, and dust to help ensure that the lab's process and protocols will produce accurate, repeatable, reliable results.*

Environmental Lead Proficiency Analytical Testing (ELPAT) is a program created through a cooperative federal interagency group, comprising the American Industrial Hygiene Association (AIHA), the Centers for Disease Control and Prevention (CDC), National Institute for Occupational Safety and Health (NIOSH), and the United States Environmental Protection Agency (USEPA) Office of Pollution Prevention and Toxics (OPPT). The program's intent is to provide proficiency samples to, and audit results from, laboratories wishing to perform lead (Pb) analysis.

To qualify for the National Lead Laboratory Accreditation Program (NLLAP), which is a requisite for certifying analytical results, laboratories must demonstrate through the analysis of samples received through the ELPAT program that they can accurately analyze lead in samples typically associated with the abatement and control of lead-based paint. Samples obtained through ELPAT come in four different forms: paint chips, air filters, dust wipes, and soils. Laboratories may be certified as proficient in one, two, three, or all four of the sample types.

### **Lead in Paint Chips**

Handheld Thermo Scientific Niton XRF analyzers provide definitive results when operated in Lead Paint Mode and do not require NLLAP listing to provide approved results of paint *in-situ*. Lead paint analyzers operate under a Performance Characteristic Sheet (PCS), which specifies the operating conditions under which the instrument provides definitive results. Typically, Niton analyzers are not used for ELPAT Accredited testing of lead-based paint chips.

### **Lead in Air Filters**

Now, with the ability to provide immediate, real-time, lead-in-air analysis, industrial hygienists can adjust the level of personal protective equipment required for each task in the remodeling, repair, or renovation process. This can result in significant cost savings by eliminating the need to maintain respiratory protection programs. The ELPAT Accreditation process for lead in 37 mm mixed cellulose air filters is combined with the IHPAT Accreditation process for metals in air. Difficult-to-analyze gravimetrically loaded samples require excellent technique to attain proficiency. If you are interested in obtaining ELPAT Accreditation for air filters, please contact your local Niton Analyzers representative or contact Thermo Scientific Niton Analyzers directly for assistance.

### **Lead in Dust Wipes**

Dust wipe analysis has the potential for providing the most significant return to laboratories and their clients based on the immediacy of the data available to decision makers on Pb abatement projects. Prior to analyzing ELPAT dust samples, and to achieve results in good agreement with known values of the samples, users **must** follow the step-by-step process detailed below.

### **Lead in Soil**

Niton XRF analyzers can easily analyze the qualification samples for ELPAT lead in soil. Before analysis, the provided sample is homogenized, sieved, and placed in an XRF sample cup. We recommend measuring these samples for 60 seconds.

### **Why Consider Becoming ELPAT Accredited with a Niton Analyzer?**

For most federally funded residential renovation, repair, and remodeling projects in pre-1978 housing, HUD regulations require testing of the residence to identify the lead dust level that remains after the work

and cleaning are completed. These dust samples, collected as wipes from floors, window sills, and window wells are called "Clearance Wipes," and the testing is usually referred to as "Clearance Testing."

Historically, clearance samples were sent to traditional laboratories for analysis. This typically involved acid digestion of the sample and analysis on an atomic absorption spectrometer (AAS). This process, depending on shipping time and the laboratory backlog, could take several days or weeks. Residents were unable to return to their homes until the dwelling passed this clearance testing.

If any of the sample results came back above the threshold requirements (see Table 1), then it was necessary to recall the cleaning contractor and redo the whole process. Further, since the original test results could take a week to obtain, the contractor had usually started another project. Therefore, the result of failing the clearance test required pulling workers from the new project to re-clean the first project. Then, the clearance tests were repeated, the wait began again, and, meanwhile, the family remained out of their home.

Now, imagine having the results within minutes, instead of days, and identifying problem areas before the contractor leaves the job site. Though sample analysis savings may be realized when field XRF analysis is performed in favor of traditional laboratory analysis, the real savings occur in the reduction of temporary housing costs for the impacted tenants and their landlords, and the reduction in labor costs for contractors.

**Table 1: Lead Dust Re-occupancy Requirements (maximum allowable<sup>1</sup>).**

| <b>Location</b>         | <b>Threshold</b>       |
|-------------------------|------------------------|
| Floors                  | 40 µg/ft <sup>2</sup>  |
| Window sills (interior) | 250 µg/ft <sup>2</sup> |
| Window troughs          | 400 µg/ft <sup>2</sup> |

### **Can You Consider Your Niton Analyzer a Portable Laboratory?**

There are two accrediting authorities currently working under ELPAT, the AIHA and the American Association for Laboratory Accreditation (A2LA). At the time of this writing, the A2LA considers the Niton analyzer to be a field operation (a temporary operation using a portable testing technology, testing on-site) and eligible for accreditation. Currently, AIHA is considering the addition of XRF analysis to its list of eligible technologies.

### **How Do Niton Analyzer Results Compare to Traditional Digestion Techniques?**

Laboratories using acid digestion followed by AAS analysis usually report a limit of detection (LOD) of approximately 5 µg/wipe. A Thermo Scientific Niton XLi or XLp 300 Series analyzer, which is also capable of performing HUD Guidelines-compliant residential lead-based paint testing, offers an LOD of approximately 10 µg/wipe [**Note:** The Niton x-ray tube source offers LODs of approximately 5 µg/wipe, but it should not be used for residential Pb paint testing until HUD approves a Performance Characteristics Sheet (PCS) it.] Since the lowest action level is 40 µg/wipe on floor samples, the Niton analyzer has the ability to produce reliable and definitive results.

Niton analyzers have proven themselves in USEPA Environmental Technology Verification (ETV) programs for having LODs and linear dynamic ranges appropriate for the analysis of lead dust wipes. In fact, during the ETV program, Niton analyzers consistently reported fewer false negatives at the all-important 40 µg/ft<sup>2</sup> action level than the USEPA's reference laboratory!

### **Why Haven't My Competitors Already Become Accredited?**

Using a Niton XRF analyzer to produce clearance results takes some effort and commitment.

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<sup>1</sup> 40CFR Part 745 Lead: Identification of Dangerous Levels of Lead; Final Rule, January 5, 2001

First, for the analysis itself, samples must be collected carefully without tearing the wipe, folded properly and dried before being placed in a bag and labeled appropriately for the analysis. Then, the actual analysis takes approximately 4 minutes. As the source ages, the analytical time will increase.

Additionally, there are the associated costs of obtaining the instrument (purchase, lease, or rent), having appropriate staff, paying accreditation fees, spending the time required for documenting procedures and proficiency testing, and pursuing ongoing certification.

While becoming ELPAT proficient is not easy, it does offer a way to take your laboratory and all the associated protocols into the field, providing your clients with near instantaneous results, and all the advantages associated with real-time information.

### **What Are the Basic Requirements for ELPAT Accreditation?**

There are four key requirements for ELPAT accreditation.

#### ***Qualified Personnel***

Qualified personnel are defined as follows: a competent, degreed technical manager with several years of experience in metals analysis; a competent, degreed quality manager, with training in chemistry and statistics; a competent, experienced laboratory supervisor; and a competent analyst. While contract employees, consultants, or even the same person acting in multiple roles may fill some of these positions, the purpose of Accreditation is to ensure that quality results are produced consistently. Each accrediting agency sets specific requirements for positions, training, and experience, which are explained in the application requirements provided by that accrediting agency and are available on its website.

#### ***Procedures and Protocols***

Every step taken in the process must be defined and documented, including sample collection, preparation, chain of custody, instrument calibration and operation, operator and observer safety, quality control, testing supplies, data logging, results reporting, and all methodologies.

You should expect that the final document will run several hundred pages, and that it will take up to several months to collect and organize your procedures and protocols. However, the EPA and accrediting agencies will accept Procedures and Protocols prepared by consultants, which will shorten the preparation time significantly.

#### ***Proficiency Testing***

The ELPAT proficiency testing occurs quarterly, with spike samples sent out approximately a month before results are due. Results are reported online to the accrediting agency. Each round of samples includes four spikes and a blank. You have to produce acceptable results on 75 percent of the samples in four successive rounds to become accredited (acceptable results are within three standard deviations of the determined acceptable range). This means you have to prove the technology for a year before you can actually become accredited. Examples of reference values and their acceptable ranges from previous ELPAT accreditation rounds are included in Table 2.

**Table 2: Examples of Reference Values and Their Acceptable Ranges**

| <b>Ref Value (µg lead)</b> | <b>Lower Limit (µg)</b> | <b>Upper Limit (µg)</b> |
|----------------------------|-------------------------|-------------------------|
| 36.7507                    | 24.761                  | 48.7405                 |
| 44.4963                    | 29.6488                 | 59.6488                 |
| 58.7207                    | 39.3079                 | 78.1334                 |
| 79.2559                    | 56.9001                 | 101.6118                |
| 118.0425                   | 86.7594                 | 149.3256                |
| 164.2896                   | 124.1133                | 204.4658                |
| 295.1711                   | 205.5666                | 384.7756                |

Practice proficiency samples are available from the accrediting agencies and include a certificate of analysis with the correct result range. This will permit you to evaluate your sample preparation and testing procedures before beginning the process. Our applications scientists are experienced in helping our users prepare for and pass the qualifying accreditation rounds, and are available to assist you throughout the process.

### **On-Site Evaluation**

After your procedures and protocols have been accepted, and you have passed the requisite rounds of proficiency testing, the accrediting agency will perform an on-site audit.

### **Estimated Costs for ELPAT Accreditation for a Portable Laboratory Using a Niton Analyzer**

*Estimates for 2008 –*

- \$500 initial application fee
- \$1,250 Accreditation fee
- \$1,000 Site assessment fee (per day),
- \$500 Proficiency sample fee
- Time and labor to document protocols

### **Should I Consider Becoming Accredited?**

Yes Organizations should certainly think about becoming accredited, but also must realize that it is a serious commitment and may not be the right choice for everyone. ELPAT Accreditation is designed to ensure that laboratories meet the highest standards for proficiency and performance. It will take an investment of time, effort, and money, though the return on this investment is meaningful. For example, a Minnesota laboratory estimated that it costs \$5,000 annually to maintain their accreditation (for AAS analysis of samples), not counting the time spent in documentation. Based on an estimated 200 residential units tested for clearance each year, at an average of 9 samples per dwelling, the distributed cost is \$2.77 per sample. Note, however, that they also estimated that the ability to perform clearance testing in the field would save over \$15,000 annually in cleaning, retesting, and resident relocation costs.

### **How Do I Get Started?**

Visit the A2LA website at [www.a2la.org](http://www.a2la.org).

Visit the AIHA website at [www.aiha.org](http://www.aiha.org)

Contact us at [niton@thermofisher.com](mailto:niton@thermofisher.com)

### **Analyzing the ELPAT Wipes with a Thermo Scientific Niton XRF Analyzer**

The ELPAT qualifying wipes were designed for traditional wet lab digestion analysis and are not typically representative of a field-collected dust wipe. A clean wipe is weighed, a portion of lead-contaminated dust is placed on the wipe (see Figure 1), and the wipe is weighed again. Since the parts per million (ppm) of lead in the contaminated dust is known, the weight of the added dust provides an accurate determination of the amount of lead on the wipe. The wipe is folded and then placed into a centrifuge tube for shipment with the assumption that laboratories will digest the sample prior to AAS analysis. This deposition and folding usually creates a small area where the majority of the lead is concentrated.



**Figure 1: Uneven distribution of lead sample on ELPAT wipe**

With traditional wet lab acid digestion analysis, the wipe may be analyzed while it is crumpled in the centrifuge tube because the distribution of lead on the wipe is not important.

Our dust sample analysis method follows ASTM E 1728 and assumes a homogeneous distribution of the dust over the surface of the dust wipe. The first wipe is a vertical series of “S” curves. This first wipe collects the majority of the sample and is usually distributed as a handprint shape covering most of the wipe. The wipe is folded with the sample on the interior; particular attention must be paid to ensure that the wipe is neatly folded.

This folded wipe then is used to collect a second sample on the same area, this time in a horizontal series of “S” curves. This wipe collects the majority of the remaining sample and should now be folded again with the sample on the interior. Pay particular attention to neatly folding the wipe.

The now twice-folded wipe is used to collect a third sample on the same area, this time in a wipe around the perimeter of the sampling area. This wipe collects any small amounts of sample missed or pushed aside during the initial two sets of passes. The wipe is folded, again with the sample on the interior. As previously noted, the wipe must be neatly folded.

Next, the wipe is folded again, then folded again, for a total of five folds. The resulting wipe is now 32 layers thick, and measures 1 inch by 1.5 inches, and the sample is (statistically) evenly distributed across the sampling area.

**Note:** Folding to precise corners is important to ensure that the resulting 32 folds provide an even thickness

The folded wipe is now placed into a plastic bag meeting specific dimensional and compositional requirements. Even after folding, the wipe has a surface area four times the size of the XLp window.

Next, the wipe is dried to (approximately 9 percent) moisture and analyzed using a test frame in the environmental test stand. Each wipe is analyzed four times (once in each of the four quadrants of the wipe). These four results are averaged by the Niton XRF analyzer, and the final result is produced.

### **Preparing the ELPAT Qualifying Wipe for Analysis**

There are 16 steps to preparing the ELPAT qualifying wipe.

*Step 1* – Prepare a clean work surface. Any contamination on this work surface can become transferred to the ELPAT qualifying wipe and increase the reported lead content. Remember that you may be testing for a total lead content as low as 20 micrograms.



**Figure 2: ELPAT sample in sealed container**

*Step 2* – Using fresh gloves, carefully open the sample tube and extract the sample (some labs take Step 3 here). Carefully open the sample. Some labs use tweezers or forceps for this step (see Figures 2 and 3).



**Figure 3: Carefully remove ELPAT wipe from sample tube**

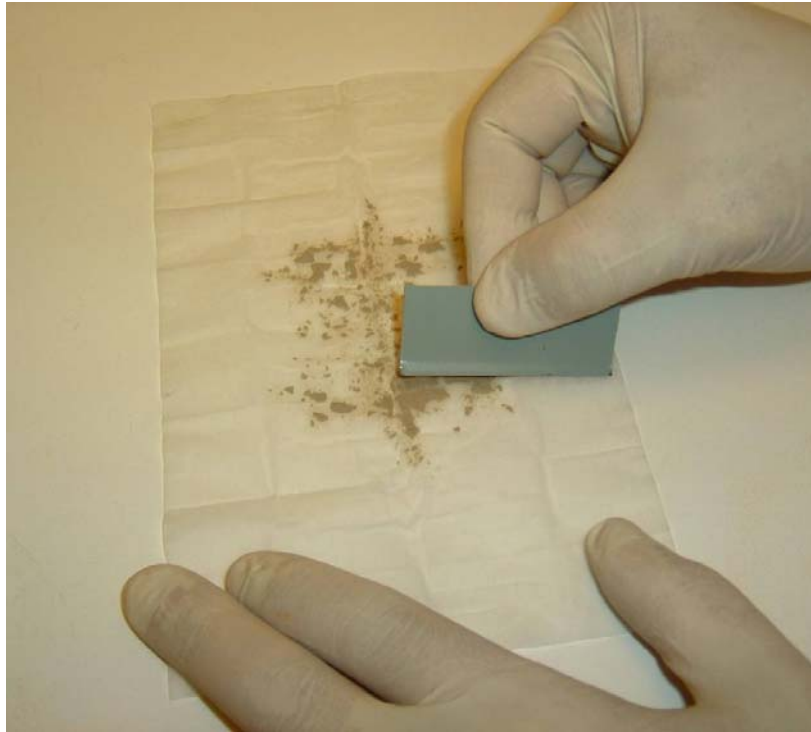
*Step 3* – The wipe should be carefully and slowly opened with the dust load on top. Opening the wipe slowly prevents any “spring” action that may lose sample material (see Figure 4).



**Figure 4: Open wipe flat on clean surface**

*Step 4* – Add distilled water to moisten the sample. An eye dropper works well.

*Step 5* – Spread the dust load carefully across the surface of the entire wipe using a clean spatula edge. A 3-inch section of a clear Lucite ruler (or equivalent) also works well for this procedure. Our applications scientists recommend using a four-corner method, spreading the dust load being from the center outward (see Figures 5 and 6). If the dust load is off center, estimate an approximately proportional amount to spread.



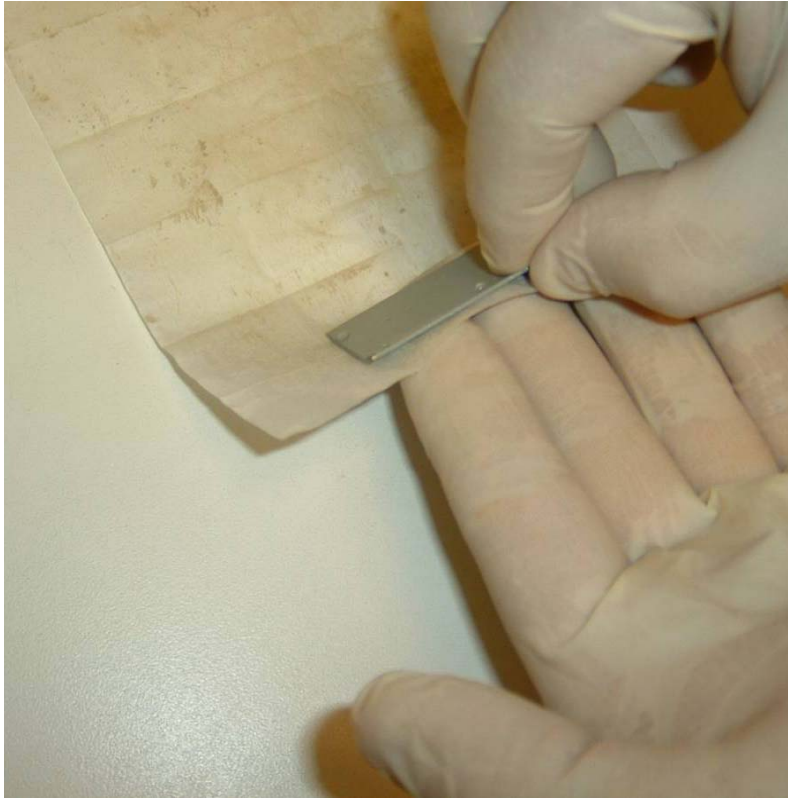
**Figure 5: Begin spreading from the center of wipe**



**Figure 6: Continue to spread lead as evenly as possible**



**Caution:** The edge used to spread the sample smear contains residual of the dust load. DO NOT SET THE EDGE DOWN YET!



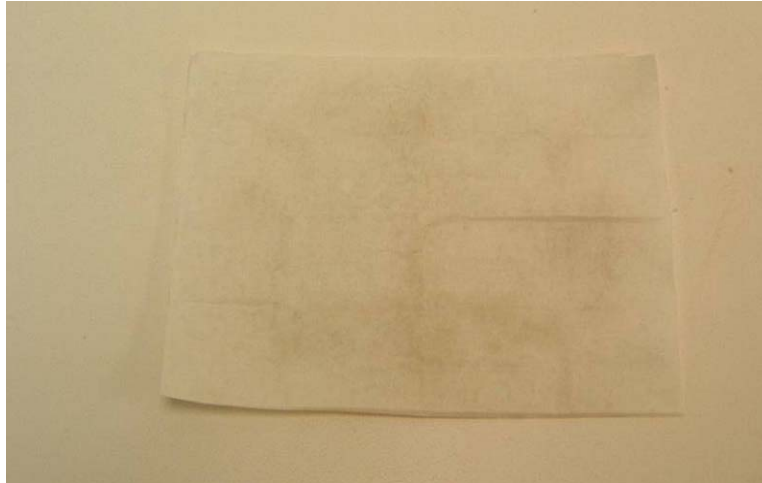
**Figure 7: Wiping off the spreading edge**



**Figure 8: Evenly distributed lead on wipe, ready to fold**

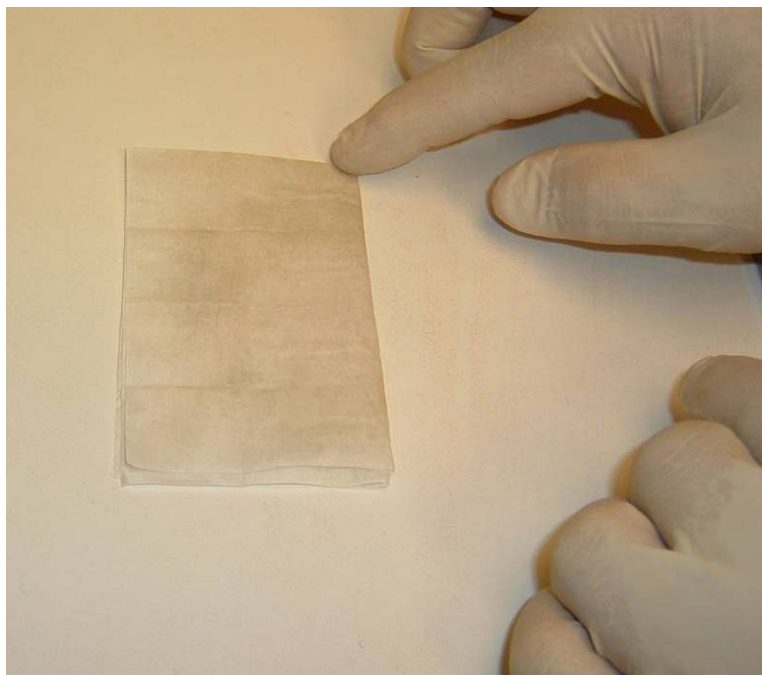
*Step 6* – Using tweezers or forceps, fold the wipe in half with the sample side inward, carefully aligning the corners. Wipe the spreading edge on the clean top surface of the wipe to remove residual dust load (see Figures 7, 8, and 9).

**Caution:** The edge used to spread the dust load still contains residual of the dust load. DO NOT SET THE EDGE DOWN YET!



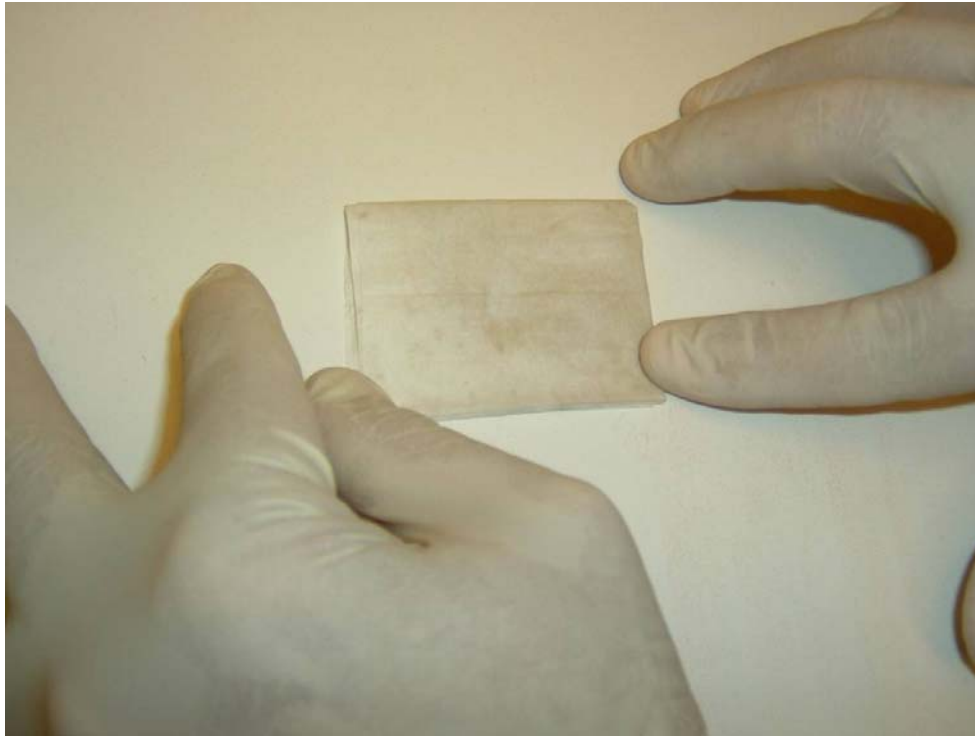
**Figure 9: Wipe after first fold; note the square corners**

*Step 7* – With tweezers or forceps, fold the wipe in half with the sample side inward, carefully aligning the corners. Wipe the spreading edge on the clean top surface of the wipe to remove residual dust load. This is the second fold (see Figure 10).



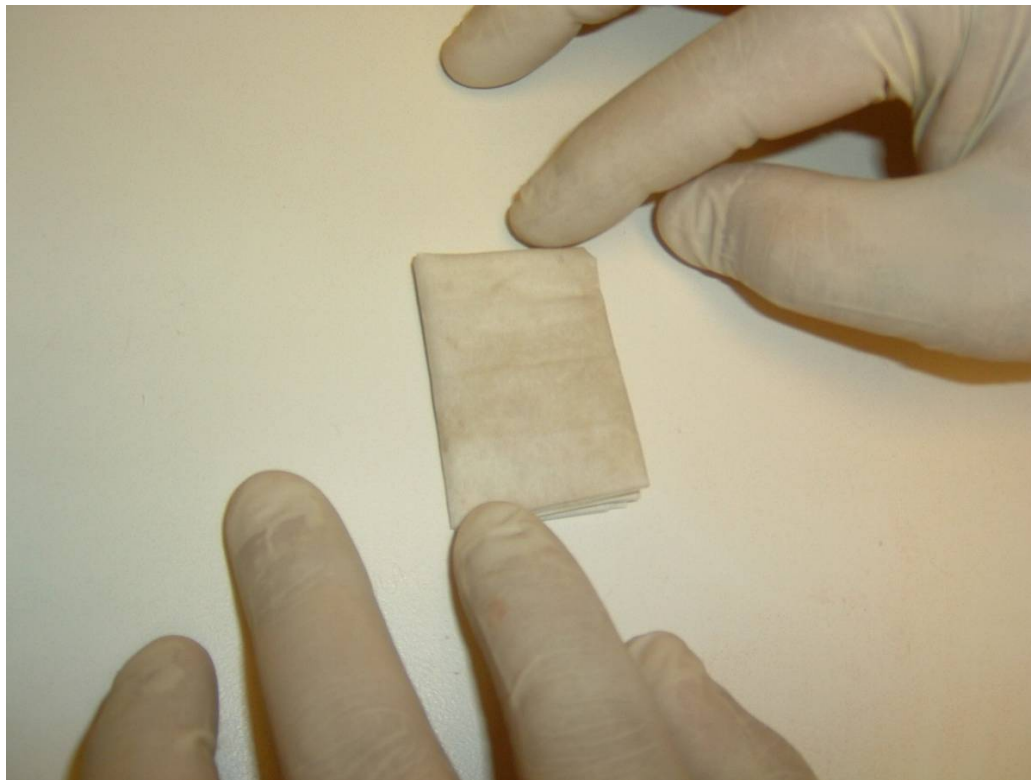
**Figure 10: Wipe after second fold; note the square corners**

*Step 8* – As before, use tweezers or forceps to fold the wipe in half with the sample side inward, carefully aligning the corners. Wipe the tweezers on the clean top surface of the wipe to remove any residual sample smear. This is the third fold (see Figure 11).



**Figure 11: Wipe after third fold; note the square corners**

*Step 9* – Again using tweezers or forceps, fold the wipe in half with the sample side inward, carefully aligning the corners. This is the fourth fold (see Figure 12).



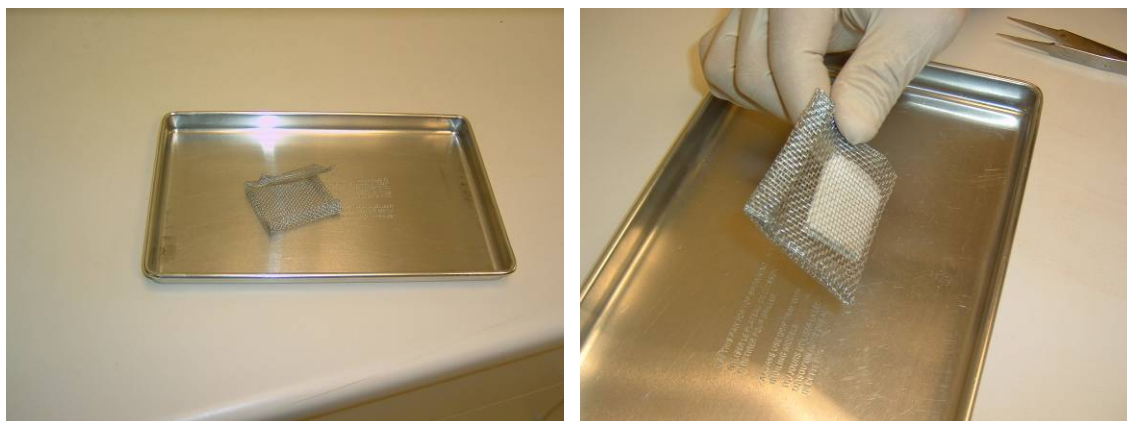
**Figure 12: Wipe after fourth fold; note the square corners**

*Step 10* – For the fifth and final fold, using tweezers or forceps, fold the wipe in half with the sample side inward, carefully aligning the corners (see Figure 13).

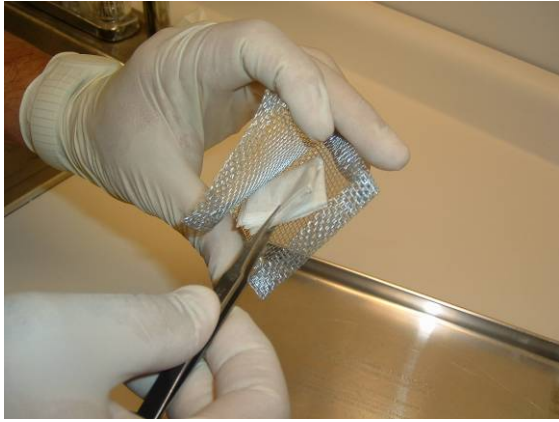


**Figure 13: Wipe after fifth fold; the wipe should measure 1 x 1.5 inches**

*Step 11* – To stabilize the sample, place the wipe into a piece of wire mesh aluminum screening and label the sample to maintain the chain of custody (see Figure 14). Place the sample into a toaster oven (or equivalent) and dry the sample at 250°F for 20 minutes; alternatively, the sample may be exposed to ambient conditions overnight. Remove the sample from the wire mesh and insert it into the dust wipe testing bag (see Figures 15 and 16). Label the bag to maintain the chain of custody over the samples.



**Figure 14: Wipe in drying screen**



**Figure 15: Clean drying screen and tray**



**Figure 16: Use clean tweezers to transfer wipe**

*Step 12* – Turn on your Niton XRF analyzer, let the electronics stabilize for several minutes, and perform a detector calibration. Be sure to note the resolution on your sample notes page.

*Step 13* – Thermo Scientific provides Calibration Check Dust Wipes with low and high lead content. Test the high lead content Calibration Check Dust Wipe. The reported results should fall within the result range listed on the high lead content Calibration Check Dust Wipe. Record the results.

Test the low lead concentration Calibration Check Dust Wipe three times. Record the results. The average of the reported results of the three tests should be within +/- 50 percent of the calculated midpoint of the result range listed on the low lead content Calibration Check Dust Wipe.

If the XRF fails to pass the calibration check, turn off the instrument and repeat from Step 12. In the event that the XRF analyzer fails a second calibration check, contact Thermo Scientific Niton Analyzers Customer Service for assistance.

Please note that Calibration Test Dust Wipes have an expiration date and must be replaced by this date; dispose properly.

*Step 14* – Remove the plastic tray from portable test stand (Figures 17a and 17b).



**Figure 17a: Portable test stand with tray**



**Figure 17b: Portable test stand without tray**

*Step 15* – Place the baggie containing the dried wipe into the metal frame for dust wipe analysis (Figure 18a), and place the frame into the portable test stand (see Figure 18b).



Figure 18a: Dust wipe in metal frame

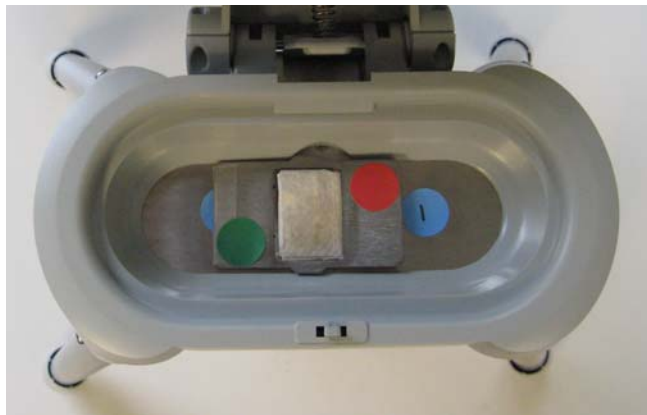


Figure 18b: Portable test stand with metal frame

*Step 16* – Analyze the sample four times, for 60 seconds each time, as detailed in the User's Guide. First, analyze the sample in positions 1 and 2, then rotate the frame 180 degrees and test the samples again in positions 1 and 2 (see Figures 19-22). In this way, all 4 quadrants have been tested. Be sure to record your results on your sample notes page.

**Important Notice:** When using the Portable test stand, manually slide metal frame from position 1 to position 2. Remove the frame from the test stand after the second 60-second reading and rotate the frame 180 degrees, do not turn over. Repeat Step 16 for positions 3 and 4. Due to variations in sample thickness, it is possible for the sample to slide out of position if the metal frame is not clamped while being inserted.

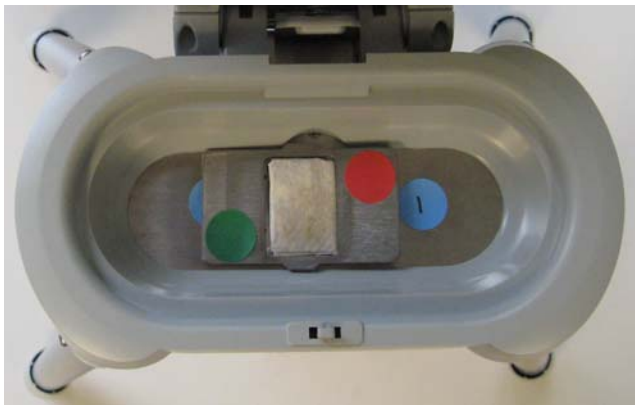


Figure 19: Position 1

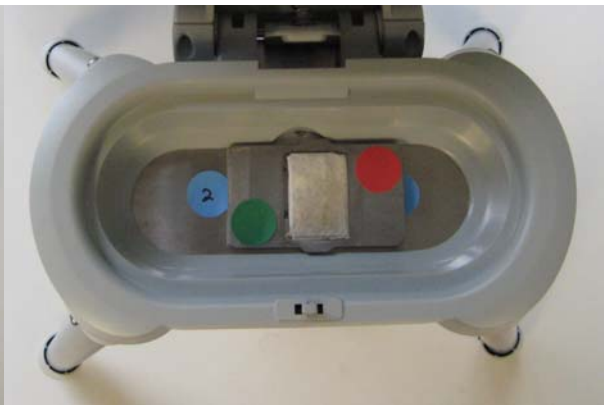


Figure 20: Position 2

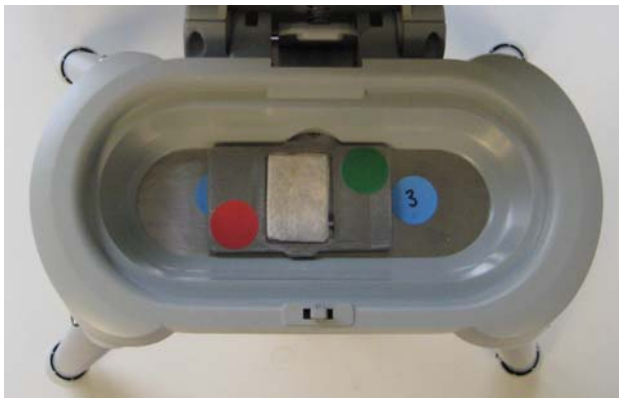


Figure 21: Rotated. Position 3

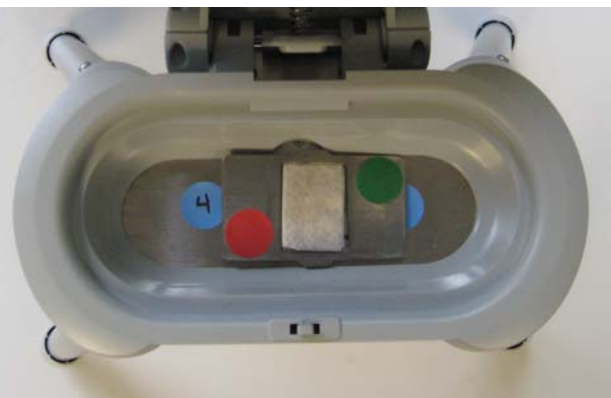


Figure 22: Rotated. Position 4

*Step 16* – Repeat the testing of the sample and record the results on your sample notes page.

Many Niton XRF users test their ELPAT qualifying samples several times, rotating the sample in the holder and testing both sides. The Niton XRF analyzer should produce very similar results regardless of the orientation or side of the sample analyzed. If your results vary, contact us for assistance.

### **Submitting Your Results**

Each ELPAT accrediting agency uses a slightly different method for reporting results of the qualifying rounds. Usually, the sample submission is done online, on a secure website. It is important to print the confirmation page to ensure that your results were submitted and accepted by the accrediting agency. Failing to submit samples in a round is regarded as a failure of that round.

For assistance or further information on how we can help your laboratory with its ELPAT process, please contact us at [niton@thermofisher.com](mailto:niton@thermofisher.com) or call +1 978-670-7460.

Appendix: EPA SITE Report, NIOSH Method 7702 pdf, *Environmental-Applications-Dust, and in Lead Paint-Applications-ELPAT*