

What is XRF?

X-ray fluorescence (XRF) employs the use of a primary X-ray beam hitting a material and causing each element in that material to produce (fluoresce) a characteristic X-ray. Since each characteristic X-ray has its own unique energy, a solid state detector in conjunction with a microchip can produce an energy spectrum.

How is portable XRF different?

A portable or handheld XRF is not significantly different from the 50-year-old laboratory versions, except its components have taken advantage of the improvements in electronic and solid state miniaturization.

How does the portable XRF determine the elemental composition in a sample?

Using the energy spectrum and a computer processor, the elements and the number of counts for each element can be classified. When this information is further processed through a mathematical algorithm, the spectral information can be turned into actual data, such as the percentage of each element in the material.

How is portable XRF used for screening for drywall types?

When performing drywall investigations, it is common to find more than one type of drywall. In some instances, over 5 different types of drywall in a single residence have been identified. It is also common to find different drywall types in a single wall.

Using a single filter, the instrument can be set up to view selected elements, and the data can be used to quickly screen residences for different types of drywall. See Figures 1a and 1b.

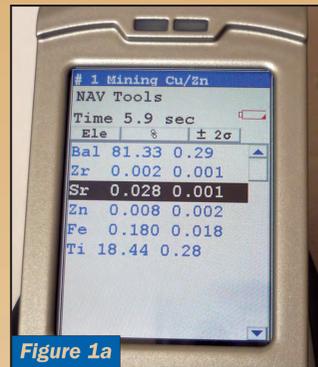


Figure 1a

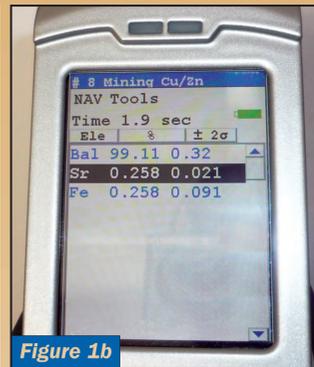


Figure 1b

Figure 1a. The instrument identifying elements through a wall finish. Note Strontium level at 0.028. Time to run the test was 5.9 seconds.

Figure 1b. The instrument identifying a different drywall through a wall finish. Note the Strontium level 0.258. Time to run this test was 1.9 seconds.

Advantages:

1. The analysis is nondestructive and results can typically be obtained within a few seconds per test.
2. The test can be performed through wall finish.
3. The data is logged and stored and can be downloaded at a later date.
4. The entire house, including ceilings, can be screened for similar elemental values that correspond to the different types of drywall.

Cautions:

1. When two drywalls with similar elemental signature are used, the result may not be conclusive.
2. The drywall finish may introduce significant variability into the screening.
3. The operator must evaluate the effect of each wall finish and also effect of finish depth variations, which typically requires taking additional readings.
4. The data is not conclusive until it can be further confirmed against sample of the drywall either through a direct reading at the edge of the drywall or through collection and preparation of a sample.

XRF Applications in Drywall Investigations

- Elemental composition
- Qualitative and quantitative
- Does not say if drywall will off-gas

Screening for Drywall Types

- Select elements to study
- Obtain qualitative and quantitative data for selected elements
- Use results to find like types of drywall within a residence

Elemental Fingerprinting

- Developing elemental fingerprint for drywall types
- Using the instrument to recognize fingerprint
- Compare fingerprint to known problematic drywalls



Figure 2

Figure 2. Library sample preparation tools.



Figure 3

Figure 3. Examples of prepared standard samples for the library.

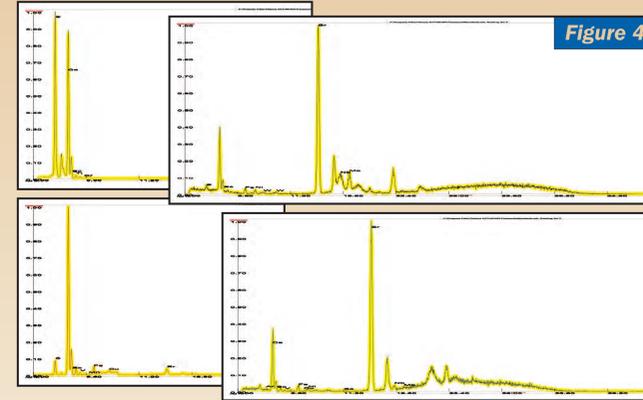


Figure 4

Figure 4. Example of spectral fingerprints.

The procedure for determining problematic drywall:

1. Verify if a residence meets the State of Florida Case definition criteria.
2. Use XRF or other methods to determine types of drywalls used in the residence.
3. Obtain samples of the different types of drywall.
4. Run copper exposure aging test or similar on the samples to verify sulfur attack in controlled conditions.
5. Run independent Carbon Disulfide verification test or equivalent.
6. Use data from steps 1, 2 and 3 to classify the drywall as problematic and prepare a standard XRF sample for spectral fingerprinting.

The procedure for developing spectral fingerprint:

1. Prepare standard XRF sample. EPA Method 6200 can be used as a reference. See Figures 2 and 3.
2. Run XRF analysis using four filters to create accurate spectral fingerprints. See Figure 4.
3. Add spectral fingerprints to the library.
4. Once the fingerprint is entered into the library, the instrument can be used to match unknown samples to samples in the library. See Figures 5a and 5b.
5. The Thermo Scientific Niton XRF analyzer will include the library with the instrument and provide a mechanism in which the library can be updated with more spectral fingerprints based on current research.

Figure 5a. The use of the instrument in a matching mode demonstrating a close match to standard sample FGD - 1.

Figure 5b. The use of the instrument in a matching mode demonstrating a close match to standard sample TIG - 1.

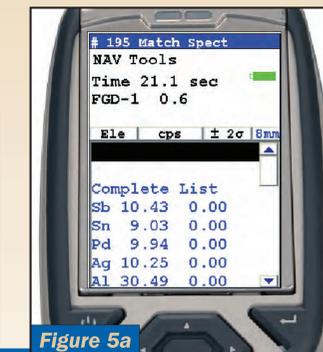


Figure 5a

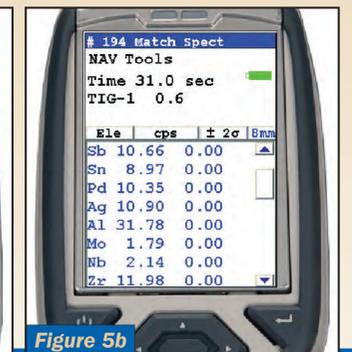


Figure 5b

The Use of XRF in Drywall Evaluation

XRF History

X-ray fluorescence (XRF) is a well-characterized and powerful analytical chemistry technique that was first commercialized more than 50 years ago [1] and which has been used in multiple laboratories for the quantitative analysis of elemental composition in many different sample types ever since. These sample types include (but are not limited to) environmental, biological, industrial, and geological materials.

As improvements in technology have allowed the gradual miniaturization of electronics, XRF manufacturers have seized upon these advances to create the first portable XRF, and subsequently, a handheld XRF. In fact, the latest handheld XRF analyzers replicate long-used XRF bench top technology, thereby eliminating the trip to the laboratory.

Government Backs XRF Development

During the "pioneering" years of XRF technology, the U.S. government supported the development of XRF uses.

- Niton Corporation (now part of Thermo Fisher Scientific) awarded Small Business Innovation Research (SBIR)

grants. (The U.S. Small Business Association Office of Technology administers the SBIR program.)

- Three Environmental Protection Agency (EPA) SBIR grants for "Improved Technology for Measuring Lead in Lead-based Paint" Phase I - 1991, Phase II - 1992, Phase III - 1995

- Two Department of Energy (DOE) SBIR grants for "A new Method for X-ray Fluorescence Analysis of Contaminated Materials." Phase I - 1995, Phase II - 1996

• EPA - Works with Niton in 1996 to develop an application to measure the elemental composition of material, in this case, soil, in real time.

- Results in the well-received and widely used procedure called Method 6200 - Field Portable X-ray Fluorescence Spectrometry for the Determination of Elemental Concentrations in Soil and Sediment.

- EPA Method 6200 is incorporated into Statement of Work (SOW) 846 under the Resource Conservation and Recovery Act (RCRA); allows the use of our analyzer to aid workers in making public health decisions about toxic elements in situations such as "Brown Fields" and in river sediments. Coupled with a protocol called Triad [2] the handheld XRF analyzer provides real time control of a remediation site.

XRF Applications & Thermo Scientific Niton Analyzers

Handheld Thermo Scientific Niton XRF analyzers deliver fast, accurate, nondestructive elemental analysis to a growing list of applications, many of which different government agencies have come to rely on and trust. Data results are tamperproof, thereby ensuring data integrity.

- **Lead-based paint** - At its inception, Niton manufactured and sold instruments for measuring lead in paint, a large public health issue around the world. The basic software allows for easy data entry for building testing protocols, such as house address, room number or type, building component like wall or ceiling. This, coupled with our encrypted data and tamperproof files, has given us the luxury of a legally defensible set of data that government agencies, like HUD, demand.
- **Environmental** - XRF analyzers provide near-instantaneous, in-situ analysis of soil, lead dust wipes, and air filter samples; accurate testing of all eight RCRA metals, 12 priority pollutants, and 19 EPA target analytes; near real-time delineation of contamination boundaries, with legally-defensible data. Our analyzers are used by regulators such as the EPA.
- **Alloy** - Niton analyzer introduced in 1998 with funda-

mental parameters (FP) software algorithm spurred by the alloy market and the need to evaluate spectral data and readout into direct elemental composition; enabled quantitative analysis of as many as 32 elements simultaneously.

- ASTM E1476-97 - Standard Guide for Metals Identification, Grade Verification, and Sorting, describes the method by which handheld XRF may be used to determine alloy composition.

- **Positive Material Identification (PMI)** - handheld XRF is one of the recognized technologies for PMI by the American Petroleum Industry (API RP 578). OSHA 1910 CFR requires the PMI testing of all critical materials used in this industry. Great loss of life and destruction of property have occurred from the use of incorrect material. In the extremely important testing application, handheld XRF is the dominant methodology for determining material composition.
- **Consumer Goods Screening** - The passage of the Consumer Product Safety Improvement Act of 2008 (CPSIA), reduces the permissible levels of lead in paint, restricts allowable levels of lead in all other materials, implements an alternative standard for measuring lead in surface coatings and permits the use of XRF analyzers for screening purposes. Manufacturers, importers, and

retailers all must comply with regulations that limit permissible levels of lead and other toxic materials in products ranging from toys and jewelry to clothing, furniture, and packaging. The Consumer Product Safety Commission (CPSA) has chosen and trusts Thermo Scientific Niton analyzers for its own screening needs.

- **Mining** - The mining industry is also a significant part of our business. With precious metals at such high prices, mining of low concentrations of elements such as gold are often below the detection limits of our handheld XRF analyzers. However, by using "tag" elements, i.e., elements that are usually present with gold such as selenium and arsenic, we are able to provide very useful information. In the same manner, if drywall is out gassing carbon disulfide or carbonyl sulfide, we cannot see the carbon or its state of matter, but we can see the tag elements that would identify the mine it came from.

Gypsum, Drywall, and XRF

For well over 20 years, XRF has played an important role in the testing of material constituents of gypsum. A Google search of "XRF and gypsum" produced over 45,000 hits. Many XRF instruments have been sold for this application by a wide variety of manufacturers. Originally, most of these XRF analyzers were benchtop instruments located in

laboratories, which, of course, required sending samples to the lab, costing both time and money.

Gypsum (calcium sulfate dihydrate, CaSO₄·2H₂O) is a common mineral found and mined around the globe. It is considered to be one of the oldest building materials in the world [3]. Gypsum undergoes a calcination (drying) process to produce the gypsum plaster (calcium sulfate hemihydrate, CaSO₄ · ½ H₂O), which is used in the manufacture of drywall.

The "Chi-square Method"

To ensure a robust elemental match that allows for the variances in alloy chemistry among various foundries, a statistical method called "Chi-square" was selected as the best possible mathematical tool to supply the user the information needed to make such important PMI confirmations. A Wikipedia reference [4] explains in detail how this statistical approach works. The Chi-square analysis provides what is known as a match number, which is a statistical probability of how this sample matches a known pool of data.

In other words, did the present sample come from the same pool of data as any given sample in a known library? The instrument gives the ID of the closest library item and its match number. A match number of 0.0 is perfect, while a number higher than 4.0 is a probable no match. The

another outcome of the Chi-square ability is spectral matching, which allows the analyzer to take a spectral picture of a material and later compare it to another material to see how it matches. This is very useful in determining the distribution of drywall goods in a home. If, for example, no match were found in the library (or a high match number obtained), the instrument could be used to determine how prevalent and where this type of drywall is in the house. If there were more than one type of drywall, the spectrum of each type could be loaded into the instrument and each sheet of drywall could be categorized. Using this method, a minimally invasive testing procedure could be employed to limit the sample confirmation size to the number of different spectra. Then, based upon the confirmation test, all the offending drywall could be identified.

The use of handheld XRF, and in particular the Thermo Scientific Niton XRF, for the evaluation of drywall was

mentioned in the first EPA drywall study published May 7, 2009, Drywall Sample Analysis. This study details the use of the XRF to identify the range of elements that were present in the drywall other than the expected elements. It can be concluded from this study that EPA uses our technology to not only investigate the unknowns in a material, but to verify that the expected elements are present in the proper proportions.

Our technology is trusted by EPA as a public health tool for such applications as soil safety analysis and lead paint. OSHA trusts this technology for worker safety in areas where workers are exposed to toxins in the air. The American Petroleum Institute recommends our technology to ensure that refineries don't endanger workers or the public from materials that aren't able to do the job. Consumer Product Safety Commission uses our analyzer to protect the children of our nation from dangerous imports. The drywall analysis is yet another application in which we can help.

References:

1. <http://www.learnxrf.com/History.htm>
2. <http://www.triadcentral.org/>
3. United States Geological Survey report <http://minerals.usgs.gov/minerals/pubs/commodity/gypsum/gpsmyb01.pdf>
4. http://en.wikipedia.org/wiki/Chi-square_distribution