**X-Ray Fluorescence (XRF) Spectrum Analyzer**

The NITON 700 Series Multi-Element Analyzer is a handheld portable XRF instrument for identifying and quantifying elemental concentrations in a variety of environmental samples. It is a non-destructive on-site assessment tool used in industrial hygiene, site assessment, remediation and compliance testing.

**XRF Principles**

A radioactive source, Cd-109 or Am-241, emits gamma rays with energies sufficient to eject K-shell and L-shell electrons from the target atom. An electron from a higher energy shell drops down to fill the vacancy created by the ejected electron. This electron gives off its excess energy in the form of X-rays, the fluorescence. Each element produces unique fluorescent X-rays. An energy-dispersive X-ray fluorescence spectrum is produced for the sample. The energies (KeV) of the emitted X-rays are characteristic of the target atom and are used for identification of the element. The intensity of the X-rays is proportional to the concentration of the element in the sample (Martin and Pesce, 1997).

**Elements detected**

|  |  |
| --- | --- |
| **Cd-109 source** | **Am-241 Source** |
| Arsenic | Antimony |
| Chromium | Barium |
| Cobalt | Cadmium |
| Copper | Indium |
| Iron | Iodine |
| Lead | Palladium |
| Mercury | Silver |
| Molybdenum | Tin |
| Nickel |  |
| Rubidium |  |
| Selenium |  |
| Strontium |  |
| Titanium |  |
| Zinc |  |
| Zirconium |  |

**XRF Applications**

* elements in paints and coatings (EPA/HUD, 1996)
* dust wipes for surface contamination (NITON, 2000a)
* lead in workplace air (Morley, 1997)
* air filters (NIOSH, 1998)
* soil samples (Clark et. al., 1999)

**Precautions**

* A beam of gamma rays is continuously emitted, but shielded by the instrument and protected by interlocks. When the shutter of the instrument is opened, gammas are directed to the sample and x-rays are emitted from materials absorbed by the gamma. Thus, the device should be used in a safe way to minimize the radiation exposure.
* Always be aware of the location of the instrument’s radioactive source and the direction of its gamma beam.
* Shutter should be open only to do a test.
* Instruments should not be pointed at yourself or anyone when the shutter is opened.

**Radiation measurement**

* Radiation is typically measured in rems, or in one thousandth of rem, called millirems (mR).
* The occupational exposure limit for whole body radiation is 5,000 mR/year.
* Exposure from properly handling the XRF should be less than 50 mR/year, assuming 2000 hours of instrument use.

In Texas, instrument users must undergo radiation training. The owner must have a radiation license and the sealed radiation source must be leak tested every six months. A copy of the leak test results and a copy of the radiation license must accompany the instrument. There are regulations that govern air transport, crossing state lines and international borders of radioactive materials, so its ease of use is limited.

**NITON XRF**

* The NITON XRF 700 is designed and calibrated to measure 14 elements simultaneously with the storage capacity of 3000 readings of complex x-ray spectra.
* Data can be downloaded to a personal computer using NITON XTRASTM software.

**Calibration**

* The device is factory calibrated and re-sourced.
* Field calibration should be done every time the device is turned on, every hour or so of operation or when reset to a new mode.

**Bulk Sample Mode**

Used to measure concentrations of contaminants in any fairly homogeneous, fine-grained medium such as soil, paint chips or liquid.

**Types of Bulk Samples**

Field Screening: A site may be quickly surveyed to determine whether gross contamination is present in the area. In the field screening mode 14 elements are qualitatively measured and displayed. This mode will give only approximate results.

Testing prepared samples: Soil samples are collected and returned to the lab for preparation and analysis.

* A nonmetallic spade is used to collect 50-100 grams of soil sample from the topsoil.
* Samples are stored in plastic bags, labeled with the date, the site and the location from where the samples are collected.
* Samples are grinded to make it as fine powder.
* If the sample is moist, it should be air dried overnight at room temperature.
* Prepared soil samples are placed in a ¼ mil Mylar film sample cup.
* Polyester fiber is used to closely tighten on the top of the cup to prevent the movement of the sample.
* A cap is placed at the top of the cup. The cup should be labeled with relevant information such as the sample location, date, and sample number.
* The cup with soil sample in it should then be placed on the soil-testing platform.
* Place the XRF on top of the platform for analysis.
* Measurement screen will display the readings of the elements.

**Thin Sample Modes**

Thin sample modes can be used to measure concentrations of contaminants on variety of thin layers, including dust wipes and filters.

* 37 mm CE Filter Mode
* TSP/PM Filter Mode
* Dust Wipe Mode
* Standard Thin Sample Mode
* User-Definable Thin Sample Mode

**Paint Mode**

* Standard Paint Mode: In standard mode, the instrument will take readings until 95% confidence reading of positive or negative level is achieved.
* Standard Mode + Spectra: This mode is similar to Standard paint mode except that the x-ray spectrum is displayed with each reading.
* K&L + Spectra Mode: The instrument will display the complete test information continuously, from the beginning of each reading.

**XRF Approvals**

* EPA method 6200 for testing soil.
* EPA and HUD (Housing and Urban Development) for testing Pb paint.
* HUD for testing dust samples.

**Barcode Data Entry**

* Barcode data can be used to enter test location information and other data electronically.
* NITON paint-testing barcode templates.
* User defined barcode templates.

**Battery Packs**

* Fully charged nickel metal hydride battery pack gives eight or more hours of continuous use.
* Battery packs can be recharged at least 500 times.
* Discharge batteries before charging.
* Charge batteries overnight, but do not overcharge.

**Studies using XRF**

* Comparison elemental analytical results of XRF [NIOSH Method 7702 (NIOSH, 1998)] to that of ICP [NIOSH Method 7300 (NIOSH, 1994)]. Air samples, collected on 37 mm, 0.8 µm, cellulose ester membrane filters, analyzed by XRF, then digested with concentrated nitric acid and analyzed by ICP, allowing direct comparison of the two instrumental methods.
* Comparative studies of XRF analysis of dust wipe samples for removable contamination to that of ICP. This procedure follows ASTM Method 1728-95 (NITON, 2000), where 100 cm2 of surface is wiped with a filter pad containing solvent. These wipes could also be acid digested and analyzed by ICP for comparative purposes.
* Use of XRF to analyze painted surfaces for lead and other elements (NITON, 2000b).
* Elements in playgrounds.
* Lead and other metals in pottery.

**Industries or trades for study**

* Small business operations that are essentially unprotected by health and safety regulations, and lack programs designed to protect worker health.
* Examples: auto repair, paint shops, metal works, print shops, wood working, electroplating, ceramics, photo processing, asphalt and tar works.

**Protocol**

A literature search and preliminary field survey would be conducted followed by sampling for elemental hazards hypothesized to be present using XRF. Since the XRF may detect and quantify 24 different elements, some previously unrecognized hazards may be identified. The data collected will be compared with health standards for evaluation of health significance. If multiple contaminants are simultaneously identified and quantified, a toxicity index can be used that considers the additive effects of all components found in the sample.

XRF complements ICP. They both simultaneously measure many elements, but the sample preparation and detection limits are quite different, hence they will have different applications.

## References

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**Questions**

1. What is XRF and how is it used?
2. What is the principle of operation of the XRF?
3. How are samples collected and prepared for analysis?
4. Describe the calibration procedure.
5. What are the interferences?
6. What precautions and regulations need to be adhered to safely and legally use the XRF?
7. What elements and what levels were observed on the pottery sample?