

Soil analysis

On-site screening of heavy metals in soils with the Niton XL5 Plus handheld XRF analyzer

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Introduction

Heavy metal contamination of soils has become a significant environmental concern due to its adverse effects on food quality, groundwater, micro-organisms, and plant growth. The most consequential origins of heavy metals in the environment are human activities such as mining, smelting, traffic, agriculture, domestic activities, and industries including iron, steel, and chemical.

Environmental consciousness is growing globally with more knowledge that is increasingly available about the harmful effects of heavy metals. As a result, public pressure around the world has prompted additional regulation and laws:

- Restricting the release of heavy metals in industrial or mining activities¹
- Prescribing risk assessment and/or monitoring for potentially contaminated agricultural land or residential, commercial, and industrial areas

These statutes have led to the enforcement of legal limits for heavy metals. Now, sites must be screened quickly to ensure adherence to regulations. However, thanks to high-density in-situ testing, handheld x-ray fluorescence (XRF) analyzers are ideal for mapping a site's pollution, determining the extent and boundaries of contamination, and defining required remediation strategies. Handheld XRF can also be used to prescreen samples before sending them for confirmatory laboratory testing.



The Thermo Scientific™
Niton™ XL5 Plus
Handheld XRF Analyzer.





Figure 1. a) Niton XL5 Plus handheld XRF analyzer b) Display of pass/fail result and elemental analysis of soil sample.

The US EPA Method 6200^{2,3} and ISO 13196 norm^{4,5} have established handheld XRF as an acceptable method for soil analysis. For over twenty-five years, Niton handheld XRF analyzers have supported environmental professionals, universities, industries, and regulators in screening and quantifying heavy metals in soils and sediments.

Instrumentation & features

With its state-of-the-art proprietary miniaturized 5W/50kV-x-ray tube, latest silicon-drift-detector (SDD) technology, featherweight and compact form factor, the Niton XL5 Plus handheld XRF analyzer (Figure 1a) significantly enhances analytical performance and operation in soil analysis. The Niton XL5 Plus Soils mode can measure elements from sulfur to uranium by using up to three different combinations of filters and voltages; these combinations improve the signal-to-noise ratios of fluorescence lines across the entire range of energy. The Soils mode uses a proprietary Fundamental Parameter (FP) algorithm to deliver accurate results. This algorithm considers the high variability of sample composition by accounting for absorption and secondary excitation effects arising from the matrix. The Niton XL5 Plus analyzer displays the results in real-time on screen (Figure 1b).

Users can optimize the Soils mode with various customizations, including measurement time differences, pass/fail thresholds, data field sets, element display formats, pseudo-elements and matrix-specific calibration adjustments. Users can store the customized parameters under different Soils mode profiles. These profiles

support multiple configurations on the same instrument, enabling instant measurements with the appropriate settings. Furthermore, the Niton XL5 Plus analyzer uses integrated GPS to facilitate spatial data visualization, site mapping and modeling of heavy metal pollution. Users can also connect external GPS systems to the Niton XL5 Plus analyzer via Bluetooth. Measurement data can be transferred to PCs using USB or wireless connectivity, including Wi-Fi.

Sample presentation

Instrument users can execute soil analysis in either in-situ point-and-shoot mode or intrusive mode². In point-and-shoot mode, the user will aim the Niton XL5 Plus handheld XRF analyzer directly at the soil surface after removing debris, small stones, or vegetation. This analysis is suitable for screening and delivers qualitative and semi-quantitative results, enabling quick localization of pollution hot spots. For best results, a Soil Guard (Figure 2a) is recommended to keep the analyzer's window bracket clean. To enhance analyzer capabilities and improve the ergonomics of point-and-shoot measurements, users may operate the instrument upside down using a tripod (Figure 2b).

When operating in intrusive mode^{2,5}, users will remove a fraction of soil from the site, homogenize using a mortar or grinder, and sieve and dry if necessary. Then, the resulting powder is deposited into a sample bag or cup, which is introduced to the analyzer using a mini-test stand (Figure 2c). Intrusive analysis, including basic sample preparation, can be done on-site or in a laboratory.



Figure 2. Accessories for the Niton XL5 Plus handheld XRF analyzer: a) Soil guard to protect window bracket b) Tripod for in-situ measurements c) Mini test stand.

Analysis of sample cups containing homogenized powder is typically lengthier (30 seconds to several minutes). This analysis can detect elements at lower concentrations and yield higher accuracy than point-and-shoot measurements.

Analytical performance

Sensitivity

Over the past 25 years, considerable progress has been made regarding sensitivity for heavy metal detection in soils. Figure 3 shows various Niton XRF analyzer limits of detection (LOD) for elements Cr, Ni, Cu, Zn, Hg, As, Se, Pb, Cd, Sn, Sb, and Ba. Continuous technological development of x-ray sources, detectors, and electronics has enhanced LODs for these elements by one to two orders of magnitude.

The Niton XL5 Plus handheld XRF sensitivity achievement is unprecedented compared to previous generations. This sensitivity is especially beneficial to identify heavy metals present at low concentrations such as Hg or Cd. Users can now detect these heavy metals below ten parts per million (ppm) within reasonably short measurement times of 20 seconds or less per filter.

Accuracy

In-situ analysis in point-and-shoot mode will typically lead to qualitative or semi-quantitative results. These results may suffice to assess the presence/absence of heavy metal contamination or to delineate an area with high vs. low concentration.

However, in soil sample analysis, the most significant uncertainty does not originate from the analytical technology or method itself (laboratory-based techniques or handheld XRF). This uncertainty stems from the sampling process, which may inadequately represent the spatial distribution of contaminants⁶. Hence, thorough sample mixing, quartering, particle size reduction, and homogenization are preconditions to obtain accurate quantitative results.

When samples are homogeneous and representative, the analyzer's Soils mode delivers exceptionally accurate results of heavy metal content out-of-the-box. Figure 4 illustrates the positive correlation between certified and measured values for V, Cr, Cu, Zn, As, Cd, Hg, and Pb obtained in the set analysis of 28 commercially available standard reference materials for concentrations varying from a few to several thousand parts per million (ppm).

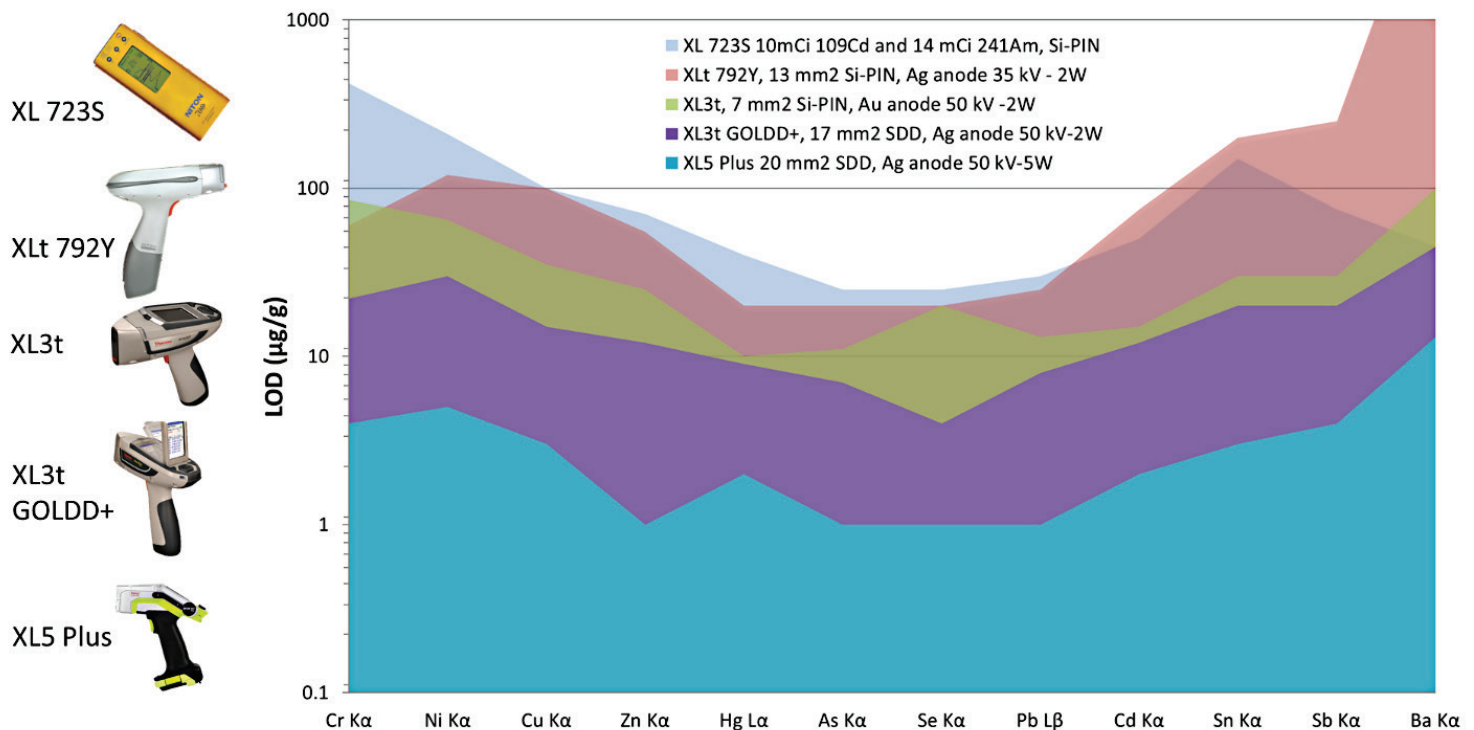


Figure 3. Limits of detection (60s per filter) in ppm in pure silica for a selection of heavy metals. Niton XL5 Plus handheld XRF analyzer (2021) vs legacy products: XL3t GOLDD+ (2010), XL3t (2007), XLt 792(2003) and XL 723S (1997).

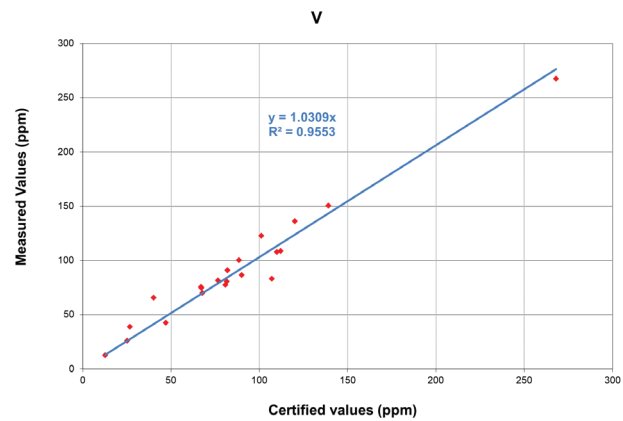
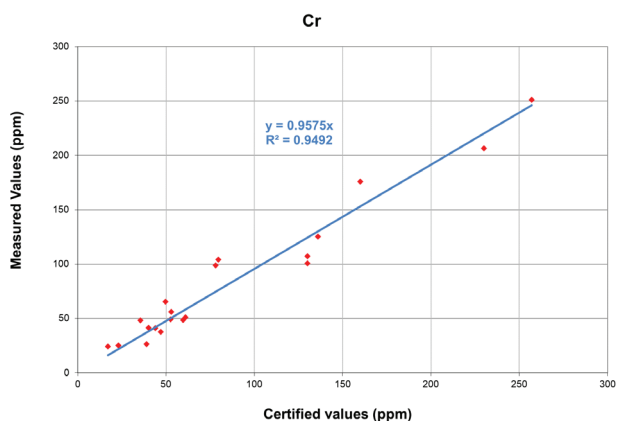
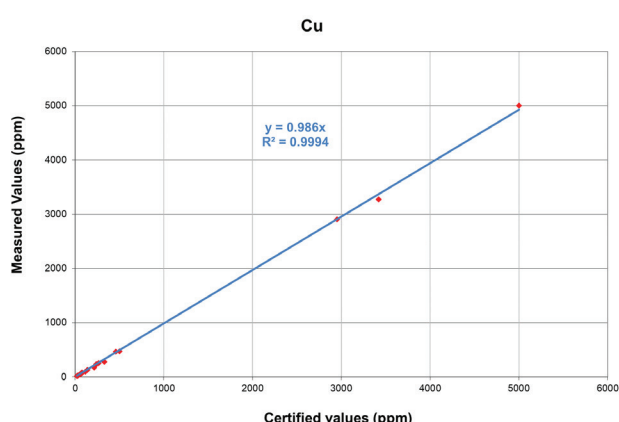
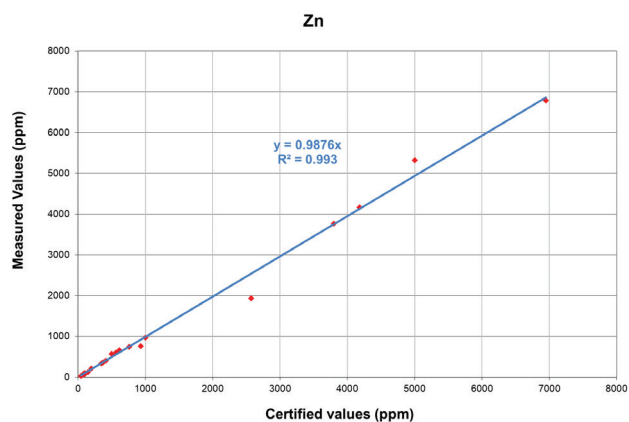
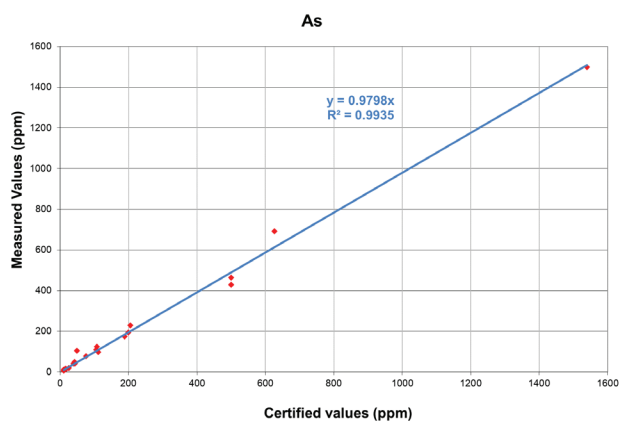
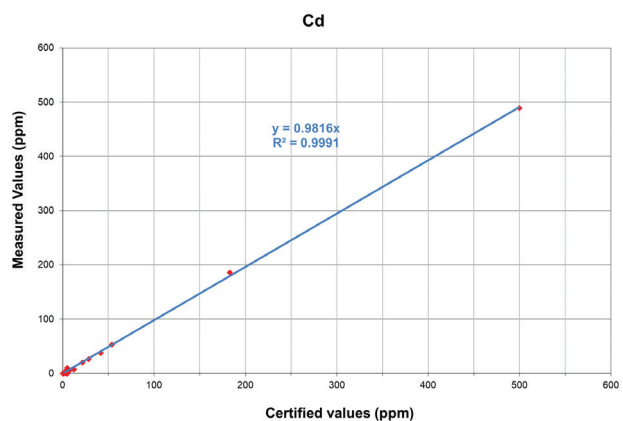
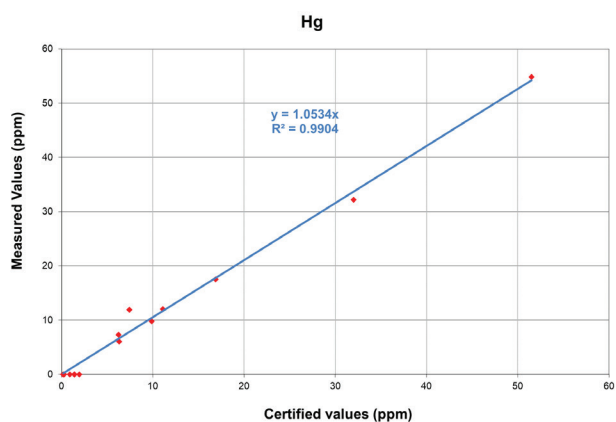
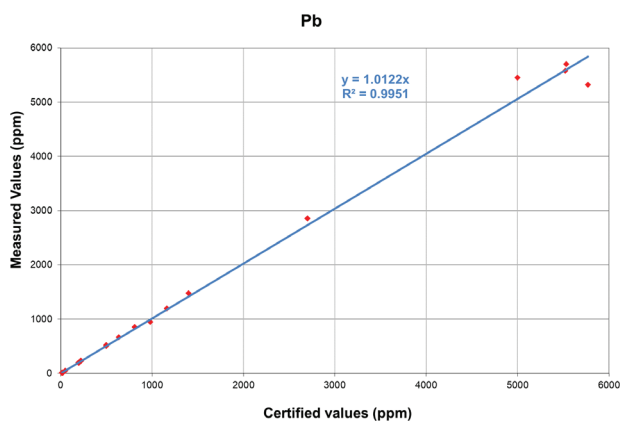


Figure 4. Correlation plots between certified and measured values using a Niton XL5 Plus handheld XRF analyzer (30s measurement time per filter) for V, Cr, Cu, Zn, As, Cd, Hg and Pb in a set of 28 soil powder standard reference materials. Samples were introduced into cups fitted with a 4 μ m polypropylene film.

Conclusion

Handheld XRF analysis allows users to optimize their time and efficiency by making real-time assessments and reducing the number of soil samples sent to a lab through prescreening. With dramatically improved sensitivity and portability, the Niton XL5 Plus handheld XRF analyzer makes next-level power and advancements available to users today by:

- Detecting unprecedented low levels of heavy metals in point-and-shoot mode
- Generating lab-quality analytical data in the field with minimal sample preparation
- Providing powerful elemental analysis in a small, lightweight instrument

Environmental professionals, regulatory agencies, and construction companies can transform their environmental hazard assessment into an efficient, on-site, and cost-effective process supporting faster return on investments with:

- Real-time decision-making through in-situ measurements and site characterization of metal pollutant hotspots according to EPA Method 6200 and ISO 13196
- Managing uncertainty with improved in-field actions using the Triad⁷ approach
- Reducing external lab costs by in-situ analysis and sample prescreening
- Ensuring contamination below threshold levels and reducing volume of excavated soil by continual measurement post-extraction
- Screening & clearance of construction sites to limit building delays
- Instant measurements of all 8 RCRA elements and 12 EPA priority pollutants
- Defensible field decisions reflecting site condition thanks to larger sample density
- Hazardous waste screening and image capturing using the on-board camera
- Brownfield remediation monitoring



References

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6. M. Ramsey, Contaminated Land: Cost-effective Investigation within Sampling Constraints, Chapter 3 in *Portable X-ray Fluorescence Spectrometry Capabilities for In Situ Analysis*, Edited by Philip Potts and Margaret West, RSC Publishing 2008
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