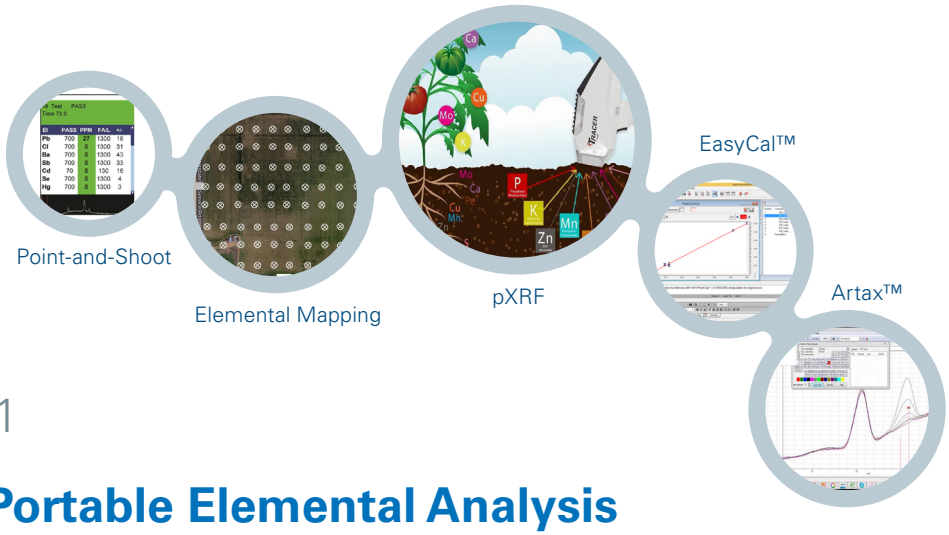


Technical Note #1701

Advancements in Portable Elemental Analysis for Healthy Soil and Crop Management

(As presented at the 2017 USDA National Cooperative Soil Survey Conference in Boise, Idaho)

Portable X-ray Fluorescence (pXRF) Spectrometry has been proven to be an effective elemental analysis field tool for monitoring heavy metal contamination in soils and sediments for over a decade. Two approved environmental methods for this are US EPA 2007 SW-846 Test Method 6200, "Field portable XRF for the determination of elemental concentrations in soil and sediment" and ISO 13196:2013 "Soil Quality: Screening soils for selected elements by EDXRF using a handheld or portable instrument". Additionally, the "USDA Soil Survey Field and Laboratory Methods Manual (2014, No. 51, V.2)" describes the pXRF method for major and trace elemental analysis in the "Soil Mineralogical Analyses" section.

Advancements in pXRF technology and data analysis software since those methods were published have improved the elemental range, detection limits, speed of measurement, breadth of data analysis and user experience significantly. These technological and data analysis advancements provide actionable value for effective assessment and management of soil and plant health.

Healthy Soil & Plants: Real-Time In-Field Testing

Portable XRF has been used to monitor heavy metals in soil for many years now. Additionally pXRF technology helps determine toxic metal concentrations in water, fertilizers, fertigants and crops with matrix-matched standards. It is also very helpful in characterizing the efficiency of bio- or phytoremediation species used for soil decontamination by testing the soil and toxic metal uptake in the biomass (e.g. willow tree for Cd, Cr, U or sorghum for Cd, Pb, As).

The US EPA published "Method 6200 Field Portable X-Ray Fluorescence Spectrometry for the Determination of Elemental Concentrations in Soil and Sediment" in 2005 to guide screening inspections and to perform compositional analysis of sites for regulatory clearance purposes. Other methods have been developed since then, including "ISO 13196:2013 Soil Quality: Screening Soils for Selected elements by EDXRF using a handheld or portable instrument".

As described in these methods, pXRF technology saves time and cost of elemental analysis. It is nondestructive which means samples can be saved or analyzed again by other technologies. Portable XRF not only provides immediate information to take action on, but also quickly

Elemental Mapping of Large Fields

Gridding of farm bordering old highway for 80 soil sampling points to take pXRF in-field measurements of lead (Pb).

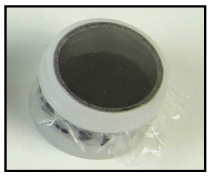
Handheld XRF extension pole with remote control are convenient for extensive in-situ soil testing.

Minimize required lab analysis time and costs with pXRF in-field testing.

Smart Sampling for Large Areas

Environmental SOP methods help guide with "smart sampling" by taking multiple measurements with in-field pXRF, and correlating with occasional lab ICP measurements.

1. Grid the sampling area of interest
2. Use pXRF for direct topsoil measurements
3. Map concentrations for elements of interest
4. Bag and mix a % of samples for homogenized pXRF tests
5. Fully prepare a % of bagged samples for highest accuracy & precision of pXRF results
6. Correlate % of fully prepped pXRF results with lab ICP-OES results
7. Use lab analysis for "Hot Spot" or regulatory clearance



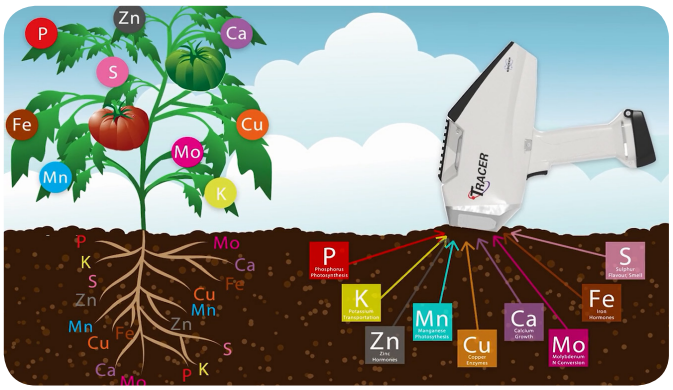
pXRF technology helps save time and costs for elemental analysis. It is non-destructive and can provide actionable information in the field.

helps define "hot spots" which may need lab analysis for regulatory directed final clearance.

These environmental SOP methods help guide specialists towards "smart sampling" by encouraging multiple measurements of topsoil in the field with pXRF, collecting a representative amount in baggies for more homogeneous sample pXRF measurements, and finally taking a portion of the bagged material for fully prepared sample measurements. These dried, ground, sieved and packed soil samples provide fully homogeneous, small particle and flat surface samples for the most precise and accurate pXRF measurements to correlate with lab ICP-OES measurements.

pXRF advancements for P, K, Mg, S, Ca, micro-nutrients and toxic metals

The "USDA Soil Survey Field and Laboratory Methods Manual (2014, No. 51, V.2)" describes the pXRF method for major and trace elemental analysis in the "Soil Mineralogical Analyses" section. This SOP describes the use of pXRF for soil applications beyond heavy metal contaminants, including the assessment of soil textural separates, gypsum quantification, enhanced horizonation, rapid onsite analysis of volcanic ash soils, and rapid and non-destructive plant nutrient analysis.



Advancements in pXRF technology enable measurements of elements much lighter in atomic weight than heavy metals. The capability of lighter element analysis means that not only can pXRF measure heavy metals in soils and plants, it can also now detect micro-nutrients, essential soil nutrients (P and K, but not N) and major soil elements (Mg, S, Ca) quickly, precisely and accurately.

Elemental Soil Chemistry Assessments with pXRF and ICP-OES

Technology	Elements and Concentration Ranges			Configurations		Elemental Measurements		
	Elements	Elemental Challenges	Detection	Handheld	Benchtop	Sequential	Simultaneous	Integrated GPS Elemental Mapping
ICP-OES	Li(3)–U(92)	C, N, H, O, low P, low S, halogens	PPTr – PPTh	No	Yes	Yes	Yes	No
pXRF	Na(11)–U(92)	Low Na, Mg, Al	PPM - 100%	Yes	Yes	Yes	Yes	Yes

Technology	Sample Presentation to the Analyzer								Additional Maintenance	
	Liquids	Solids/Powders Tissues	Pressed Pellet	Sample Cup	Bagged Sample	In-Situ/ As-Is	Reuse Sample	HAZMAT Disposal	Nebulizer	Torch
ICP-OES	Yes	Yes: Dissolve/Digest Liquefy/Dilute	No	No	No	No	No	Yes	Yes	Yes
pXRF	Yes	Yes	Yes	Yes	Yes	Yes	Yes	No	No	No

XRF technology and data analysis software advancements

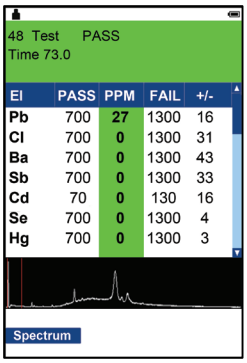
Advancements in pXRF tube, detector, geometry and electronics as well as data analysis software since early methods were published have not only improved the elemental range, detection limits and speed of measurement, but have also significantly expanded the breadth of data analysis and enhanced user experience in the field.

In addition to complying with instrument guidelines for these environmental SOP methods, Bruker's newest portable handheld XRF, the TRACER 5^{i}, provides the capability to measure a much wider range of elements including lower levels of nutrients and elements such as magnesium, potassium, calcium and even sodium. The TRACER 5^{i} is also unique in that the excitation conditions can be customized and controlled by the user. This includes power settings (kV and µA), filters (standard and custom), sample spot size and even the beam path environment – air, helium or vacuum. These capabilities are very important for agricultural applications since many of the samples and their matrices are so varied that simple point-and-shoot calibrated handhelds aren't always sufficient.

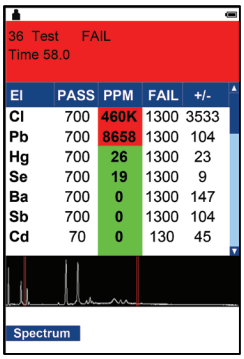
Both TITAN and TRACER 5^{i} handheld XRF users can also take advantage of data analysis software advancements. One new software module, EasyCal™ quantitative software, provides the ability for a user to create his or her own calibrations for unique applications. Users can also take advantage of the powerful and comprehensive Artax™ qualitative software for measuring the presence or relative abundance of elements in question. Artax™ software is also ideal for working with large data sets for trends and other types of semi-quantitative analysis.

Point-and-Shoot pXRF Results

Point-and Shoot Pass Results, Composition and Spectra



Point-and Shoot Fail Results, Composition and Spectra



Handheld XRF analyzers can be pre-calibrated with commercially available standard reference materials (SRMs). Unknown samples with similar matrices to the SRMs can be analyzed quantitatively.

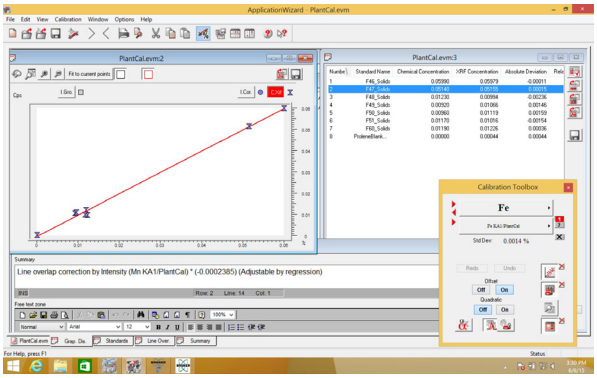
Thresholds, such as those regulated by US EPA for soils, can be set to provide Pass/Fail/Inconclusive or Yes/No/Inconclusive results on the display.

Specialists can also use their own SRMs to fine-tune the pre-set calibrations to better match their sample matrices.

Point-and-shoot pXRF calibrated testing

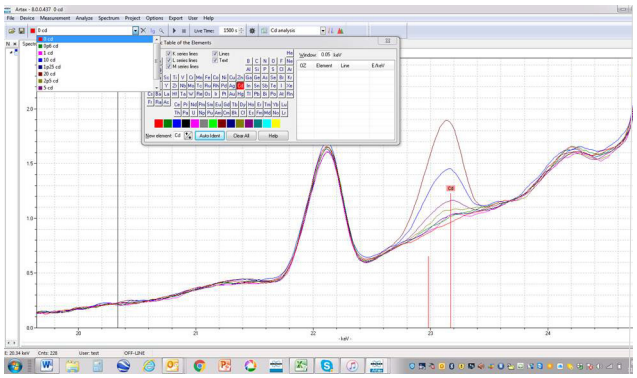
Portable XRF analyzers can be purchased with calibrations for a variety of applications, including those with preset pass/fail results based on pre-defined thresholds and ranges. Specialists can also use their own SRMs for fine-tuning the pre-set calibrations to better match their unique sample matrices. Additionally, users can develop their own methods and, with EasyCal™ software, create calibrations to provide point-and-shoot results for user-specific applications.

EasyCal™ Quantitative Data Analysis Software



Users can create their own calibrations for materials such as iron (Fe) in corn byproducts prior to use in the biofuel manufacturing process.

Artax™ Qualitative Measurement & Analysis Software



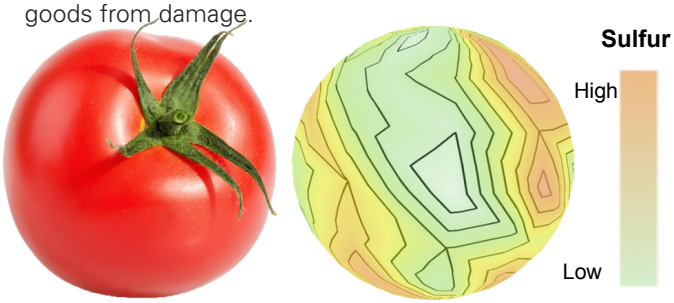
Users can develop methods with unique manual filters optimized for challenging measurements such as cadmium (Cd) uptake in plants.

Advancements in pXRF Technology with Bruker's TRACER 5^{i}

- Analyzes elements from Na - U in all types of samples in the lab or in the field
- Bruker patented SharpBeam™ geometry for high performance speed and sensitivity
- Battery or AC powered; Wi-Fi and USB enabled
- Sample spot size collimators: 3mm and 8 mm diameter
- Internal camera with adjustable LED, focal and reticle positioning, five image captures/data point
- Air, helium and vacuum path capable; pressure and temperature compensation
- Automatic filter wheel for standard filters and manual hatch for custom filters
- Advanced PC data analysis software – BIT, EasyCal™ and Artax™
- EasyAccess™ Rail for field tools and Relaxed Grip Strap™
- Portable, lightweight, collapsible desktop stand for use with sample cups, baggies or small samples

Mapping surface treatments of produce

Handheld XRF can even be used to assess chemical treatments on the surface of produce. 3D relative abundance plots, such as sulfur on a tomato, help farmers determine optimal surface treatment and placement to protect their goods from damage.



Summary

Two published methods for monitoring metal contamination in soils and sediments are US EPA 2007 SW-846 Test Method 6200, "Field portable XRF for the determination of elemental concentrations in soil and sediment" and ISO 13196:2013 "Soil Quality: Screening soils for selected elements by EDXRF using a handheld or portable instrument". The published "USDA Soil Survey Field and Laboratory Methods Manual (2014, No. 51, V.2)" also describes the pXRF method for major and trace elemental analysis in the "Soil Mineralogical Analyses" section.

Advancements in pXRF technology and data analysis software since those methods were published have improved the elemental range, detection limits, speed of measurement, breadth of data analysis and user experience significantly. These advancements provide real value for applications involving the effective assessment and management of soil and plant health.

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