# Screening Trace Elements in Areas of Intensive Agriculture in Brazil with a Field-Portable XRF Analyzer

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## ABSTRACT

Portable XRF analyzers have been routinely used for environmental purposes, mainly in areas with high contents of trace elements (TE). However, their use in the analysis of TEs in soils of agricultural areas is still limited, especially in Brazil. Portable XRF might not replace atomic absorption spectroscopy when sub-parts per million levels are required. Yet it offers some advantages that are very important for field applications such as minimal sample preparation and fast identification of many TEs of interest. Also, portable XRF is a promising technique because it generates a minor amount of residues and has low cost for reagents. In order to check if such technique can provide accurate quantitative results for TEs, we tested surface soils samples (n = 68) collected under native vegetation and cultivated areas of the state of Mato Grosso (MT), which is the largest producer of soybeans, corn, and cotton in Brazil and also the greatest consumer of agricultural inputs that may represent a diffuse source of TEs to agricultural ecosystems. Results obtained by the portable XRF analyzer were compared with data using the USEPA 3051A procedure for TE extraction followed by quantification via atomic absorption spectroscopy, using soils standard reference materials for QA/QC protocols. Although we have obtained good recoveries (SRMs) for the analyzed TEs (As, Cu, Mn, Pb, and Zn) using both techniques, there was no correlation for data obtained between the two selected analytical methods. This can be explained by the low TE contents in the studied samples, which are uncontaminated soils and also by the fact that the portable XRF apparatus have much higher detection limits.

## INTRODUCTION

XRF has been shown to be a powerful tool for a large number of samples and analytical applications. XRF analyzers are simple to operate, field portable and provide fairly selective detection of elements ranging from Na to U with LODs in the parts per million range for many of these elements. Perhaps the most rewarding aspects of XRF are its minimal sample preparation requirements and nondestructive analysis capabilities. XRF can be used for in situ identification of contaminated areas or in components that may be too large to bring back to the laboratory [1].

Trace elements (TEs) in soil can be detected by several conventional analytical techniques such as spectroscopic techniques. Analysis by atomic absorption spectrophotometry (AAS) is a well-known method for the detection of metals in aqueous samples. However, extraction procedures for heavy metals from soil samples typically involve lengthy process that requires the use of harsh conditions. For example, the use of routine methods, like aqua regia, requires boiling of samples and the use of concentrated HCl and $HNO_3$ for pseudo total extraction of trace metals. Clearly, the capability to perform direct, in situ analysis of solid soil samples, without the need for digestion as is potentially available through portable XRF instruments would be a major step forward. Rapid pollution monitoring is especially important in instances of toxic dust, as timely on-site analysis and fast decision making are of the highest importance, in order to protect the health of local communities[2].

## OBJECTIVE

The aim of this study is to evaluate if a portable XRF can provide accurate quantitative results for selected TEs (As, Cu, Mn, Pb, Zn) in surface soils samples collected under native vegetation and cultivated areas in the State of Mato Grosso, Brazil's largest producer of grains and livestock.

## MATERIAL AND METHODS

- Soil samples of cultivated and native vegetation areas of Mato Grosso State in Brazil were collected at 0,0-0,2 m, resulting in 68 representative samples.

Native vegetation areas

Cultivated areas: soybeans, corn and cotton

- Trace elements (As, Cu, Mn, Pb, Zn) contents were determined by AAS after soil digestion following the USEPA 3051A protocol, as well as by XRF, using a portable analyzer

Soil preparation

Soil preparation

Soil digestion

AAS technique with flame and furnace graphite

## MATERIAL AND METHODS

Portable XRF technique

Soil preparation

XRF analyzer – 40W

XRF Turbo SD LE - Bruker® - 120s

- Certified reference materials were used in both techniques for quality assurance / quality control

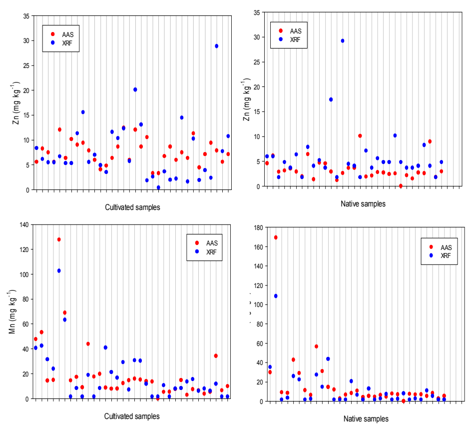
## RESULTS

Table 1. Recovery of trace elements by AAS and XRF

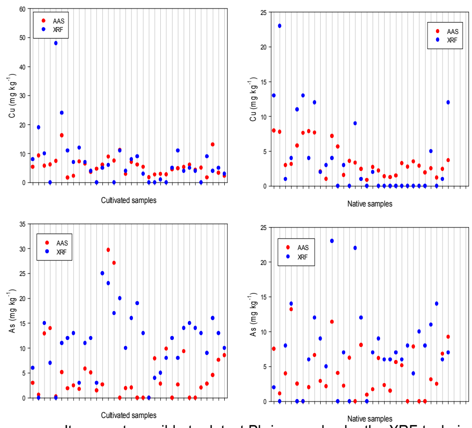
| Elements | AAS technique | | XRF technique | |
|---|---|---|---|---|
| | Reference material | ----- % ----- | Reference material | ----- % ----- |
| As | NIST 2710a | 94.0 | NIST 2709 | 101.7 |
| | | | NIST 2711 | 88.57 |
| Cu | NIST 2710a | 104.0 | NIST 2709 | 78.03 |
| | | | NIST 2711 | 78.95 |
| Mn | NIST 2710a | 75.0 | NIST 2709 | 100.6 |
| | | | NIST 2711 | 98.11 |
| Pb | NIST 2710a | 108.0 | NIST 2709 | nd |
| | | | NIST 2711 | 98.97 |
| Zn | NIST 2710a | 95.0 | NIST 2709 | 111.3 |
| | | | NIST 2711 | 95.03 |

nd = not detected

Pb could not be detected by the XRF technique in low-Pb samples. However, there was a good recovery for all evaluated TEs for both techniques in samples containing high contents of TEs.




## RESULTS




It was not possible to detect Pb in samples by the XRF technique, which explains the absence of data for this element.

## CONCLUSION

Portable XRF analyzers provides a rapid in situ detection of trace elements such as As, Cu, Mn and Zn in soil samples, but data obtained with this equipment yielded poor correlation with data obtained by the USEPA3051A method. This might be due to the low TE contents in the studied samples, which are uncontaminated soils and also by the fact that the portable XRF apparatus have much higher detection limits.

Portable XRF delivers a good qualitative result for TEs in soil samples, yet it can not substitute traditional techniques as acid digestion followed by AAS analyses in samples containing low contents of TEs (e.g., agricultural areas).

## REFERENCES

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