

Review of Affects of the Sample Preparation Procedure for MCERTs Metals in SAL East Kilbride

Introduction

Soil samples are prepared for metals determination by drying and grinding to a <425µm fraction. The extraction is then performed on the dry fraction and results reported on a dry weight basis. A validation study was performed to confirm that the drying and grinding process did not significantly alter the results obtained.

Method Summary

Three soil types are analysed '< 2mm, as received' and 'dried and ground':

'< 2mm as received' samples are prepared by sieving the selected soils through a 2mm sieve in their wet state. Portions of the '< 2mm as received' sample are removed for determination of loss on drying at < 40°C and subsequent grinding to <425µm to produce the 'dried and ground' sample.

Soil types are characterised on wet sample using the MCERTs protocol documented in SAL EK LP7 Issue 3.

The 'dried & ground' and '<2mm as received' are extracted for metals in sextuplicate using 1.00g and hotblock digestion (110°C for 1 hour using 10ml of concentrated hydrochloric acid and 3.5ml of concentrated nitric acid). After cooling all digests are made up to a final volume of 50ml using deionised water, mixed, filtered and then analysed in one analytical batch by ICP-OES.

Data obtained by SAL East Kilbride

The selected soil samples tested were real samples predominately classed as loam topsoil, sand/gravel and clay. These soil types represent the majority of samples analysed at the laboratory.

Topsoil : SAL Ref S22473/004

Sand/Gravel : SAL Ref S22200/003

Clay : SAL Ref S21901/004

The enclosed data were obtained in the SAL East Kilbride laboratory using the Spectro CIROS ICP-OES. Samples prepared by P. Geri 6th December 2005, Metals extractions performed by L M Cheung 9th December 2005 and ICP-OES analysis performed by M McGibbon 13th January 2006.

Results and Discussion

The results obtained are reported in the attached documents.

Note that the native results for Beryllium and Cadmium in sand/gravel and clay were below the reporting limit and consequently produced unusable data.

Topsoil

The only metal that produced a real statistical difference was Chromium at +7%.

The t-test for Cadmium indicated no significant difference and it should be noted that native value for this element is at the reporting limit.

Lead produced a difference of +11% however a t test shows that there is no significant difference in the results.

Sand/Gravel

Arsenic produced the highest difference in values, -17% however a t test shows that there is no significant difference in the results. All other differences are less than or equal to 10% and are statistically not significant.

Clay

The clay results show statistical real differences for Copper, Lead and Zinc with the dried and crushed results for these three metals all higher than the '<2mm as received'. This is not unexpected and is attributed to higher extraction efficiency due to the finer particle size in the dried and crushed portion.

Conclusion

Overall these data suggest that no significant losses occur in the sample preparation process for the elements of interest.

P. Geri
19th January 2005