

Review of Affects of the Sample Preparation Procedure for MCERTs Metals in Manchester

Introduction

Soil samples are prepared for metals determination by drying and grinding to a 425 u 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.

Procedure

The attached procedure was developed to allow validation of the process. In a number of cases no detectable amounts were present so a pair of spiked samples of each of the laboratory in house 'blank' materials were analysed. The spikes were performed with the independent standard solution, and spikes were allowed to equilibrate for more than an hour before starting the drying/grinding process. Aliquots of the dried and ground samples were also spiked immediately before digestion to allow a recovery determination to be made.

Results and Discussion

1. 'Native' soils

The results obtained are reported in the tables. In most cases the change in values between the original and the wetted/dried/ground samples is less than 10%. Significance testing has been applied to all results where the difference is greater than 5% (see significance testing table). A summary of the performance is listed below. Note that a significance test has been performed where a Y/N is included in the brackets after the numerical result.

Metal	Sand	Loam	Clay
Hg	N.D.	N.D.	N.D.
Mn	-3.5%	9.2%(N)	-12.7%(Y)
Mo	N.D.	35.3%(Y)	N.D.
Pb	-3.8%	26.9%(Y)	-7.6%(N)
Se	N.D.	7.7%** (N)	N.D.
Sn	2.3%	24.7%(Y)	N.D.
V	-4.5%	21.1%(Y)	-9.8%(N)

* the level of Se in this sample is below the reporting limit of 2 mg/kg (mean 1.7), so is very close to the LOD.

Therefore, the only cases where there is a significant positive bias is for the loamy soil, where there is a positive bias for the Mo, Pb, Sn and V. This suggests that for these elements the extraction is more efficient on the dried and ground sample, or the specific aliquot chosen from the wet sample was less contaminated than the remainder after drying and grinding. There is one example of a significant low bias, namely Mn in the clay. The difference is just outside the MCERTs target.

2. Spikes.

The data are reported in the second table. The mean of the paired samples are compared. In almost all cases the differences between the dried and ground versus 'wet' samples is less than 5%. Only in the case of Hg on the sand is the difference significantly more than this (9.4%), but the difference between the two results on the dry sample is quite high (nearly 20%), making the drawing of any conclusion unreliable.

Conclusions

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

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