

# Matrix dissolution: the wild card in soil analysis

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For more than 20 years there has been scientific debate about the impact the matrix makes on soil analysis and by how much this needs to be taken into consideration when considering method validation, ongoing QC and proficiency testing. There are many variables in soil analysis, not withstanding the matrix: they include extraction procedure and the associated chemistry, analytical technique, equipment and data manipulation. The effect of the type of dissolution, especially on the determination of heavy metals, has long been an issue. In 1996 I was involved with the development of some CRMs in a joint project involving a US CRM producer, RT Corporation, the Netherlands Government's RIZA Agency, CN Schmidt BV and Promochem GmbH (now LGC Promochem GmbH). One paper<sup>1</sup> produced from this cooperation, published in *Fresenius' Journal*, showed very clearly that certain heavy metals in sewage sludge showed a very differing extraction pattern when the analysis was preceded by a US EPA developed Nitric Acid digestion rather than the aqua regia digestion commonly used in Europe.

As environmental analysis has moved from research to the commercial and regulatory market the need to properly understand the relationship between the matrix and the digestion method on soil analysis has become more urgent. The paper by Micó and co-workers at University of Valencia on page 23 of this issue of *Spectroscopy Europe* describes a particularly interesting aspect of differences in digestion of calcareous soils.

The presence of chalk rich, or calcareous, soils are relatively limited, although from the office of *Spectroscopy Europe* nestling in the South Downs of England it is difficult to imagine any other soil type. Even so, their relative rarity and, possibly

the belief that they are benign samples that are easily digested, has meant that very little work has been done on their analysis. Other than BCR 141 there are no other top level certified reference materials based on such a matrix.

The many UK laboratories accredited by UKAS<sup>2</sup> to the UK Environment Agency's MCERTs Standard have had to follow a rigorous method validation programme.<sup>3</sup> The MCERTs Standard states that: "*Validation procedures include a number of operations. These shall include the analyses of, where available and appropriate, matrix certified reference materials relevant to the matrices, parameters and range of parameter concentrations under investigation. The method shall be validated for each parameter analysed on matrices likely to be analysed within the laboratory. This validation shall include at least three different soil matrices...*" The standard goes on to say that: "*For the method, parameter and matrix, the performance characteristics shall be determined with a minimum of ten degrees of freedom. This shall be carried out by analysing each certified reference material or spiked samples in duplicate in different analytical batches. 11 batches will guarantee a minimum of ten degrees of freedom.*" (Authors emphasis). This means that for each heavy metal the lab must analyse three CRMs 11 times, and to meet other requirements of the standard, at two levels. This is probably the most rigorous method validation regime required of any group of commercial laboratories anywhere.

Most of the method validation for MCERTs has been done using either available CRMs or spiked clean soils: the labs have little choice but to use what is available. This means that matrices for which CRMs or clean samples are not available,

including calcareous soil and made up ground, are under represented and most method validation has been completed using sandy soil, loam or clay soil because there is good availability of such CRMs and clean soils. In much of the southern half of the United Kingdom calcareous soils predominate and a large proportion of old, or "brown", land available for re-development cannot be easily classified as sandy soil, loam or clay soil. So how far does a laboratory have to go to validate its analytical methods? The paper by Micó and co-workers shows that calcareous matrices are just as difficult to digest as sewage sludge and other soil types, so it is perhaps the case that there are no easy soil matrices and all soil types pose a difficult analytical challenge, worthy of proper analytical validation. If this is so, how can a commercial laboratory ever justify the cost associated with a full method validation for every matrix type? It cannot. The real question is "is the data produced by the laboratory fit for purpose?" Just because it is possible to explore every possibility does not mean it is necessary to do so: it is much more important to understand what the data is to be used for and ensure that the data meets the needs of the customer. Chasing scientific purity may be intellectually stimulating, but at times it is necessary to ask if the cost can be justified. So how much method validation is really needed?

## References

1. J.F.N. Maaskant, A.H. Boekholt, P.J. Jenks and R.D. Rucinski, *Fresenius J. Anal. Chem.* **360**, 406–406 (1998).
2. <http://www.ukas.org/testing/single-search.asp>
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