Reference Materials for Environmental Analysis: Rising to the Challenge

ENVIRONMENTAL ANALYSIS/STANDARDS

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Using reference materials to validate and control analytical procedures is not new: the first recorded use of what we would now recognise as a reference material was in 1824 by Gay-Lussac [1]. Just 100 years ago the US NBS released a reference material program, developed in partnership with the US iron and steel industry, to bring uniform quality to steel and iron produced in the USA. Some of those first "SRMs" are still available from NIST, for example SRM 4L, Cast Iron Chips Certified for Trace Elements is in its 12th lot since the early 1900s!

Over the last 100 years a number of measurement communities embraced the routine use of reference materials, including the pharmaceutical industry in the 1930s, the geological sector, through the work of the United States Geological Survey in the 1950s, the hospital pathology sector in the 1970s, the environmental market in the late 1980s and most recently the biotech sector at the turn of this Century.

In each case the driver that resulted in the widespread adoption of reference materials was, directly or indirectly, regulation.

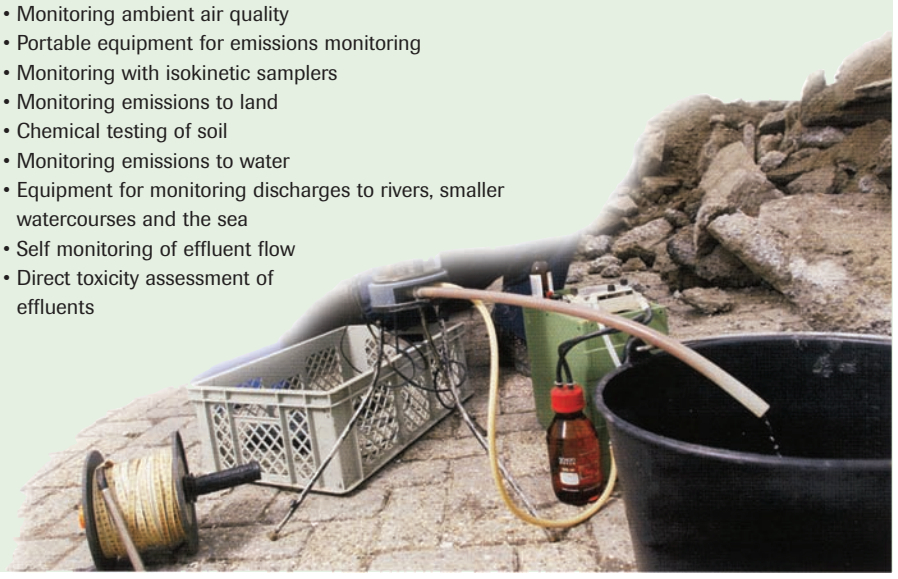
Although the environmental sector started using reference materials in the mid to late 1980s the first wave looked mostly at the levels of organic and inorganic determinands in flora and fauna, as society started to get to grips with the consequences of the liberal use of pesticides in the 1950s and some notable industrial accidents in the 1960's which resulted in gross environmental pollution. It has only been in the last 5 years, as western society enters a "post industrial, post cold war" phase has the systematic analysis of contaminated land on former industrial and military sites resulted in a significant growth of soil and sediment analysis.

In the United Kingdom the Environment Agency (EA) has long been a champion for the betterment of all aspects of environmental analysis, implementing a number of EU Directives and Regulations through the "MCERTs" Program.

In the UK all businesses that make emissions to air, land and water require a permit from the Environment Agency, which usually requires the structured monitoring of emissions. This can be done either continuously or at times defined in their permit, known as spot tests or periodic monitoring. In both cases testing must meet the EA's quality requirements.

The main testing categories are:

- Continuous monitoring of industrial chimneys, stacks and flues
- Emissions monitoring from chimney stacks - using accredited laboratories and certified staff
- Monitoring ambient air quality
- Portable equipment for emissions monitoring
- Monitoring with isokinetic samplers
- Monitoring emissions to land
- Chemical testing of soil
- Monitoring emissions to water
- Equipment for monitoring discharges to rivers, smaller watercourses and the sea
- Self monitoring of effluent flow
- Direct toxicity assessment of effluents



All pose analytical challenges, but the most recent, and from the perspective of the supply of reference materials, the most demanding is the chemical testing of soil.

In 2002 the Environment Agency issued a Performance Standard for Laboratories undertaking the Chemical Testing of Soil, [2] which following a lengthy process of consultation and discussion between the EA, the laboratories and the UK Accreditation Service (UKAS), finally came into force earlier this year. The standard requires that all chemical testing of soil, where results are to be submitted to the EA for regulatory purposes, must be carried out by laboratories accredited to the current version of the European and international standard, ISO/IEC 17025 for this MCERTS performance standard. The responsibility for accrediting testing laboratories is the responsibility of UKAS, and is normally achieved by an extension to the existing ISO 17025 Accreditation held by the laboratory as in reality the MCERTS performance standard is an application of ISO/IEC 17025 specifically for the chemical testing of soil. Its scope covers:

- performance targets;
- the selection and validation of methods;
- sampling pre-treatment and preparation;
- participation in proficiency testing schemes;
- the reporting of results and information.

What has this all meant for the providers of Certified Reference Materials?

Firstly, it is important to realise that the EA does not prescribe those analytical methods that a laboratory should use, it simply requires that the method used shall be appropriate for the matrix and parameter at the level of concentration being analysed. This means that methods must be properly and rigorously validated. The validation procedures should, where available, include the analyses of matrix certified reference materials relevant to the matrices, analytes and anticipated analyte levels under investigation. The method has to be validated for each parameter analysed on matrices likely to be analysed within the laboratory. This validation must include at least three different soil matrices – typically a sandy soil, a loam and a clayey soil. For the method, parameter and matrix, the performance characteristics shall be determined with a minimum of ten degrees of freedom which normally means analysing each certified reference material in duplicate in different analytical batches. Eleven batches will normally guarantee a minimum of ten degrees of freedom.

The bottom line is that the requirement for matrix certified reference material use is enormous: for every analyte three matrices and 11 samples, normally duplicated with the analyte close to the detection limit and in the upper part of the methods normal range need to be analysed. Unfortunately the reference material consequences of the accreditation of 40 plus labs for more than 60 determinands described in Schedule A of the MCERTs Standard was not fully appreciated by anyone in the EA or UKAS and the RM suppliers were not properly brought into the equation until the process was well underway. There are simply not enough matrix materials available to allow all the permutations of matrix, analyte and analyte level to be properly validated without resorting to using spiked clean soils..

At the start of 2006 there are two organisations trying very hard to rise to meet the challenge of providing sufficient matrix CRMs demanded by the labs seeking and maintaining MCERTs Accreditation. They are: RT Corporation Ltd [3] and LGC, [4] through the VAM program. RT

Corporation, as a result of having spent most of the last 15 years developing matrix CRMs for the US EPA's equivalent of MCERTs has provided the lions share of matrix CRMs Certified for organic determinands, whilst LGC, with the experience of RM production funded by the DTI's VAM Program, has been able to supply a part of the demand for matrix CRMs certified for trace elements. But both producers would have been better able to respond had they been brought into the process earlier and had the time to develop a suite of suitable matrix CRMs.

References

For anyone seeking more information about MCERTs, the proper use of Certified Reference Materials in Environmental analysis or the CRMs available, the following references may be useful.

1] Reference Materials for Chemical Analysis by Stoeppler, Wolf and Jenks. Wiley-VCH, Weinheim 2001, ISBN 3-527-30162-3

2] MCERTS Performance Standard for Laboratories undertaking the Chemical Testing of Soil 2nd Edition: download from www.mcerts.net

3] RT Corporation Ltd, www.rt-corp.com

4] LGC, www.lgc.co.uk