

Accreditation: what is at the end of the journey?

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Where is the accreditation of analytical laboratories taking the reference material producer industry? I've recently been cogitating the long term impact of the growing spread of ISO 17025 accreditation on the development and supply of certified reference materials and I'm concerned that the "quality business" is driving laboratory accreditation into areas where reference material producers will be under increasing pressure.

Much of my concern comes from the use of ISO 17025 Accreditation by Government agencies to effectively privatise environmental testing work previously carried out by government laboratories. On the face of it this trend is good: accredit efficient commercial labs to carry out the testing: their accreditation body will ensure that, by virtue of properly validated methods regular within and between batch QA and participation in external PT, the lab continues to produce data that is reliable and fit for purpose.

So what is the problem? It is the demands that the accreditation process makes on CRMs and PT providers. Good practice suggests that method validation should be done using CRMs that are matrix matched to the sort of sample the lab will receive for analysis. Further, the use of reliable CRMs is accepted as the best way to validate a procedure. But real world matrix CRMs contain naturally accrued analytes, not including all the analytes the lab wants to analyse.

Consider the UK MCERTS Standard for Soil Analysis.¹ When accrediting a new procedure Para 5.4.5.3 says:

"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."

It goes on to say that: *"Recovery estimates shall be obtained using two significantly different but appropriate concentration levels, for example, at 20% and 80% of the expected range."*

So the lab must find CRMs that contain all the analytes of interest, e.g. the EPA 16 PAHs, one at a high level and one at a low level. So labs need to find, per matrix two CRMs, one at high level and one at low level in an amount to perform a minimum of 22 determinations.

But a real world matrix CRM does not contain 16 PAH at a uniform level. So what does the lab do and what can the producer do to meet the labs needs? I recently reviewed a catalogue offering of PAH in soil CRMs from a CRM supplier.²

Their catalogue lists 24 soil CRMs certified for "BNAs", a group of more than 30 analytes including PAHs. Their CRMs cover a range of soil types, from sediment, through clay and loam to sand. Their CRMs contain from one to 15 of 16 PAH, at levels ranging from 169 mg kg^{-1} to 0.03 mg kg^{-1} . They also list four PAH in soil CRMs that contain the full suite of PAH at typical levels.

But even with this large range of CRMs it isn't possible to select three soil types and then find all 16 PAH at two levels. To do so a lab might have to use more than 12 actual CRMs and still do their own spiking of a clean soil to fill in the gaps. With matrix CRMs at more than £130 per 50–100 g this is expensive.

Looking at the BNA in soil CRMs offered by this supplier it was clear that they evolved from totally naturally sourced matrices to semi-synthetic mate-

rials. Why? Because I suspect that only by starting with clean soils of defined type can the producer be reasonably certain of ending up with a soil containing the range of analytes at the levels the customers demand.

Over the last six months I've been talking to scientists from UK labs working towards MCERTs or ISO 17025 accreditation for organic analytes in soil. It is clear that the labs would really like a small range of CRMs containing all their accredited determinands in three matrices and at two levels, so they would all need just six CRMs. The CRMs would contain enough material to carry out 66 determinations. The argument was that if everyone used the same CRMs there would be even better comparability of data between labs, and (perhaps more to the point) the process of accreditation would be cheaper.

Whilst it may be technically possible to produce such CRMs they would be far removed from CRMs produced by national metrology institutes (NMIs) 10 or 15 years ago. The original idea of a NMI CRM was material that could show that a determination could be analysed and allow the proper demonstration of traceability to the mole with a clear and credible statement of uncertainty. In a past RM column³ I pointed out that the relationship between an analyte and a matrix was complex and that efforts to improve "production efficiency" in commercial environmental laboratories by the use of accelerated extraction systems had made achieving accreditation a difficult challenge. Some labs felt that because a natural matrix, but fortified CRM, would

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taught like in the mid-19th century using glass burettes, colour indicators and Erlenmeyer flasks. This contrasts considerably to the fact that market research puts the widely used automatic titrators to about 20,000 units per annum predominantly sold to quality control labs in many branches.

With pleasure I recall contacts almost throughout my entire professional life with the German Analytical Professor Georg Schwedt who worked in half a dozen Universities during his scientific career. He not only published books on how to do chemical experiments in a kitchen or with products from a local supermarket but he also introduced the comparative study of analytical methods in his teachings at the Technische Hochschule of Clausthal-Zellerfeld. Students had to analyse a given sample with an array

of methods, e.g. photometry, titration, UV-Vis etc. The results, cost and usability had to be compared and the best method was established for that given problem. I feel this is the kind of situation a chemist meets in many facets throughout their professional life. This approach minimises the risk of being biased by lack of exposition to a broader instrument spectrum in the orchestra of analytical instrumentation."

Jan Volker Geil, Vice President, Business Development Metrohm Group, Metrohm Ltd, International Headquarters, Oberdorfstrasse 68, CH9101 Herisau, Switzerland.

*"Tony,
Neither the e-mail address nor the website given in the Education Article are working.*

Article was spot on."

Geoff Dent
(Sorry Geoff, you were just too quick!
Tony)

"I mostly disagree with this article. Without the basics and understanding the limits of the different spectroscopic techniques no one will be able to decide which tool is the best to resolve a given issue or to find new areas. Why should everyone use the most expensive latest technique if a problem could be resolved using cheapest technology, may be even non spectroscopic. Advanced Spectroscopy should be taught at Doctorate level."

An Industrial Analyst

Thanks for your support in our campaigning and I hope you will continue to contact us with your praise and criticism! You can use the form at <http://www.alis-consult.com/education.html>

Reference

1. A.N. Davies, *Spectrosc. Europe* **21(1)**, 17 (2009).

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not allow them to achieve the required level of precision and bias there was something "wrong" with the CRM.

All of this is slowly leading me to have a real concern that accreditation of commercial service laboratories to ISO 17025 is actually compromising chemical metrology. This is because as labs struggle to "jump through the hoops" that are essential for their continued commercial viability they are losing sight of what good chemical analysis is supposed to be about. I'd be very interested to know what you think about this issue.

References

1. http://publications.environment-agency.gov.uk/pdf/GEHO0203BKAY-e-e.pdf?lang=_e
2. RT Corporation, www.rt-corp.com
3. P.J. Jenks, *Spectrosc. Europe* **19(1)**, 30 (2007).