Reference materials for new measurement technologies

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Reference materials support measurement technologies: over the last 100 years or so the development and widespread use of reference materials has followed the introduction of a new analytical technology, regulatory imperative or market need.

History shows that, in general, reference materials provided by instrument suppliers are viewed by the market as a second best, only to be used if there is no third party alternative. In the mid 1970s "computerised analytical systems" were starting to make big inroads into the clinical chemistry market. Because many of the new systems were closed, the customer had no choice but to take the reagents, calibration systems and reference materials offered. Many potential customers wanted proof that these new systems would produce good data, so a team lead by Professor Tom Whitehead at the Woolfson Research Laboratories in Birmingham did what today most labs would consider to be a normal "ISO 17025" or GMP IQ–OQ–PQ validation on many of the systems, sometimes without the support of the instrument supplier. They used validated RMs in the containers the instrument used for calibration, after decoding the software codes that told the instrument what was in the container. They found that number of the systems relied on "factors" built into the software to give the "right" result!

Thirty years later much has changed: accreditation of laboratories to ISO 17025 and together with ISO REMCO Guide 34, of RM producers, ensures total transparency. Laboratory accreditation, instrument and method validation coupled with effective PT means it is impossible for suppliers to sell systems that don't deliver their promises. Today's challenge is that novel spectroscopy technologies arrive far more quickly than RM suppliers can respond to. A good example is surface analysis. The need to get good data quickly, often directly from a production site and in many cases without materially damaging the sample is driving much R&D work. I was particularly interested in John Watt's article on page 6 of this issue. His group are focussed on understanding how adhesion works: a key area of materials technology as when adhesion is as well understood and measured as can be presently be done with more traditional mechanical joining technologies then stronger, safer and lighter materials can be more widely used.

New measurement technologies demand RMs, but before RMs can be developed the analytical technology has to be properly understood. The last VAM programme, (2003–2006) started looking at "Measurement for Emerging Technologies" and this should continue in VAM 2006 to 2009. Even so, even very well understood analytical techniques, when applied to new areas, do not always work as expected, as has been discovered when X-ray fluorescence spectroscopy (XRF), dominant in the metallurgy sector for many years, has been applied to contaminated land analysis. New smaller, faster XRF analysers created interest and RM producers were asked to supply CRMs and PT samples to a number labs comparing XRF as an alternative to acid digestion and ICP. The results are interesting as data, in mg kg^{-1}, from a recent PT round shows.

The results for copper and selenium are "acceptable" by conventional PT performance assessment criteria. I'm told by the PT provider that in their experience, barium, manganese and vanadium XRF results, when compared to acid digestion, are generally high but there is no good explanation. In this study XRF may be seeing the total amount for Cr, Co, Sr and V. The true value is from total digestion with Aquaregia and Hydrofluoric and ICP-MS, but there is bias due to insoluble oxides in each analyte. Most routine labs use less rigorous extraction, so the mean value from a PT round is usually lower than the true value.

This example demonstrates that if good, reliable RMs are to be developed for the new technologies much robust R&D is needed. Experience shows that this is best done independently: the question remains: how will this work be funded?

Element	Conventional mean value	Std Deviation	True value	XRF value
Barium	130.4	8.6	108.5	738.7
Manganese	459.4	40.6	445.0	761.0
Vanadium	94.8	6.1	80.5	127.5
Chromium	106.9	86.1	100.7	194.8
Strontium	125.2	14.2	128.7	296.0
Copper	116.8	9.7	102.7	122.6
Selenium	143.8	14.0	169.1	180.4