

# On finding the “right” reference material, Part 2

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In the last edition of *Spectroscopy Europe* this column considered why selecting the right reference material seems to have become more onerous in recent years, despite some large organisations such as Sigma Aldrich and LGC Promochem making the supply of certified reference materials a key part of their business and smaller specialist companies such as RT Corporation and Inorganic Ventures establishing European offices and distribution points.

In this article we look at the sources of information that a Quality Manager (QM) can use to help select a reference material that is “fit for purpose” and which will both meet the needs of the laboratory and satisfy the accreditation organisation’s auditor.

As an example of the sort of challenge faced by QMs a real world example has been used. On occasions the author is asked by laboratory QMs for advice on available RMs to meet a specific need. In this example the QM at BAE SYSTEMS Environmental in Chorley asked for three

different soil matrices; clay, sand and loam, that each contained a range of PAH at two levels.

The initial brief from BAE forced the elimination of most of the candidates: they were sediments, not soils. It transpired that UKAS do not accept that a sediment matrix is suitable for validating a method that is to be used for the analysis of terrestrial soil. In the absence of scientific evidence that proves sediments do behave as soils they take a safe, conservative view.

It became clear that there are, easily available from European suppliers, only a few soils that contain certified levels of PAHs: they are shown in the table below.

With just 11 suitable CRMs it was obvious that it simply was not possible to meet the UK MCERTS requirement of three matrices with each analyte at two levels. Of the 11 soil matrices available only five had information on soil characterisation clearly stated in the catalogue. The others have an indication

of the source of the soil, but not the type. Further delving into the certificates revealed more soil type information about the BAM CRMs, bringing the number of usable candidates to seven, but there was only one clay soil that fortunately contained all the analytes of interest.

The way complex organic analytes extract from a soil matrix does vary considerably with the type of soil: a clay soil is much more retentive than sand. So the use of a range of matrices is a realistic requirement by the accreditation body. The MCERTS Standard requires validation of every analyte at two levels in three soil types: in theory six CRMs, but with a need to use more than one CRM to cover all the matrices the total number of CRMs needed starts to climb. With CRM costs ranging from £1.00 to £3.00 per gram and with 2.5 g sample needed for every analysis it became clear that this method validation would be costly in reference

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CRM	Matrix	PAH (mg kg <sup>-1</sup> )				
		Acenaphthene	Acenaphthylene	Benzo(a)-anthracene	Benzo(b)-fluoranthene	Benzo(k)-fluoranthene
RTC CRM 115	Loamy Sand	4.6	not present	12.1	0.93	not present
RTC CRM 123	Silty Loam	7.52	7.24	8.38	not present	not present
RTC CRM 125	Loam	3.24	1.66	2.22	not present	not present
RTC CRM 131	Clay Loam	2.35	3.31	6.36	2.1	1.39
RTC CRM 134	Sandy Loam	2.56	1.16	4.9	(0.043)	4.07
BCR CRM 524	Industrial Soil	not present	not present	22.5	13.5	6.2
NIST SRM 1649a	Urban Dust	not present	not present	2.21	6.45	1.913
BAM CC 013	Industrial Soil (loamy sand)	1.59	not present	7.52	7.16	3.75
BAM CC 014	Industrial Soil (loamy sand)	0.92	not present	4.19	4.96	2.25
LGC 6138	Coke Works Soil	50.4	not present	181	not present	not present
LGC 6144	Gasworks Soil	not present	7.8	28	26	17.5

# NEW PRODUCTS

## ATOMIC

### AA autosampler

Hitachi has introduced an autosampler for the Z-2300 flame-only atomic absorption spectrophotometer which will accommodate up to 80 test tubes. It has a random access function which can be set for each analyte element, allowing automatic measurement of multiple elements. On completion, the burner chamber is cleaned and the flame is turned off automatically. Test tube racks are available for 80 × 15 mm or 56 mm × 18 mm diameter test tubes. A 50-tube rack is also available for flame emission photometry.

Hitachi High-Technologies

Circle 100

## FLUORESCENCE

### Quantum yields

Horiba Jobin Yvon has introduced the Quantum Yield Accessory for determining photoluminescence quantum yields for fluorescent samples. It can be used for research on OLEDs, DNA sequencing and detection, immunology, nanocrystals, green fluorescent protein, quantum dots and phosphors. Specially designed to slide into the sample compartments of their Fluolog-3 and FluoroMax-3 spectrofluorometers, the accessory includes a 4 inch (10 cm) integrating sphere, sample holders for liquids and thin solid films and special software for automatically determining quantum yields.

Horiba Jobin Yvon

Circle 101

## MASS SPEC

### GC/MS

Shimadzu recently introduced the CGMS-QP2010s gas chromatograph/mass spectrometer which features their patented constant linear velocity, 20 temperature ramps and both scan and SIM modes. The SIM mode enables monitoring of up to 64 sets of ions with up to 64 ions per set allowing the determination of many compounds simultaneously. Other features include post quadrupole focusing lenses, redesigned ion optics and front access for easy maintenance. The GCMSsolution Data Station also fully controls and processes data for any analysis. Accessories include Shimadzu's AOC020i auto injector, AOC-5000 injection system and PY2020 D pyrolysers.

Shimadzu

Circle 102

## CRM COLUMN

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materials, excluding the time needed to assemble the required information.

A careful log was kept of the time spent obtaining the information. After defining the requirements and establishing that sediments were not eligible, two databases were visited to see what was available. The best are:

VIRM: [www.virm.net](http://www.virm.net)

COMAR: [www.comar.bam.de](http://www.comar.bam.de)

But it does take some time to learn how to ask questions and refine the search criteria. This stage took about two hours to produce a short list of 13 possible materials. A couple of suppliers were eliminated because either the producer was not accredited in any way or there was no known European source.

From the short list of 11 CRMs, the web sites of the suppliers were visited to establish prices, information on the Certificate and shipping costs. This stage took 4.5 hours to complete.

RT Corporation Ltd: [www.rt-corp.com](http://www.rt-corp.com)

BAM: [www.bam.de](http://www.bam.de)

IRMM: [www.irmm.jrc.be](http://www.irmm.jrc.be)

NIST: [www.ts.nist.gov](http://www.ts.nist.gov)

LGC: Promochem [www.lgcpromochem.com](http://www.lgcpromochem.com)

It was found that not all the information needed to make an informed choice was presented on the web sites, in some cases it was necessary to refer to both the printed catalogue and copies of the Certificates to obtain all the information needed.

The final stage was to e-mail all the suppliers to check availability. In two cases the stocks of a particular CRM were found to be limited, so to ensure continuity of supply the QM was left with no choice but to reserve stock to be held at the supplier for call off over the next months. This job took an hour and a half.

Analysing the exercise: a knowledgeable specialist, with up-to-date copies of all the main catalogues and good contacts within the producers had spent a full working day, spread over three days, to compile the table shown above. In many working laboratories the costs associated with sourcing and managing the CRMs needed have become a significant and growing cost of ensuring the quality and validity of their product: good data.

### Data acquisition software

Thermo Electron has introduced improved LCQUAN 2.5 data acquisition software for the Finnigan TSQ Quantum series of triple quads. It features a bi-directional interface with Thermo's Watson LIMS. It also enables 21 CFR Part 11 compliance and adds several new features.

Thermo Electron

Circle 103

### Ion trap LC/MS

Varian has broadened its mass spectrometry range with the introduction of the Varian 500-MS LC ion trap, a new liquid chromatography/mass spectrometer designed for the analysis of thermally labile compounds, including pharmaceutical products, drug metabolites, pesticides and herbicides. The instrument features SelecTemp, which electronically controls the atmospheric pressure ionisation for drying gas during gradient LC separations by controlling temperature distribution throughout a complete analytical run and automatically records all parameters in data file. It also features Enhanced Charge Capacity which extends the number of ions that can be analysed simultaneously.

Varian

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