National Measurement System

Setting the standards

LGC has produced the world's first reference material certified for carbon isotope ratios traceable to the SI (International System of Units).

LGC

The requirement

With the relative abundance of isotopes such as ^{13}C and ^{12}C fixed when the earth was formed – and on a global scale not changing since – the isotopic composition of a material can be affected subtly by its geographical origin, or how it has been processed or manufactured. This makes isotope ratio analysis a vital tool for determining food authenticity, for use in identifying counterfeit drugs or forensic drug analysis.

Traditionally, results of carbon isotope ratio measurements are not traceable to the SI. Instead they are reported using the Vienna Pee Dee Belemnite (VPDB) scale – defined using a sample of fossilised shells of an extinct organism called a belemnite (something like a shelled squid) collected decades ago from the banks of the Pee Dee River in South Carolina.

Existing reference materials are certified only in terms of $\delta_{13}C$ values on the VPDB scale. Results are reported as $\delta_{13}C$ values (expressed in parts per thousand, ‰), relative to the VPDB standard which, by definition, has a $\delta_{13}C$ of 0 ‰. As the availability of this material reduced, other reference standards were calibrated to the original sample and data normalised to the values of that standard.

However, this posed further problems with traceability, as every time the reference standard needs replacing, the new material requires recalibration to the original sample, and the uncertainty increases.

The solution

To solve this problem, LGC scientists developed a material with carbon isotope ratios fully traceable to the SI unit of mass (the kilogram) through calibration standards of known purity. These reference materials are easier to replace as the certified ratio does not depend on any particular material. Absolute carbon isotope ratios rather than relative isotope ratios provide a link between the VPDB and the SI making the production and maintenance of matrix-reference materials sustainable.

Glycine was selected as a reference material due to its relevance to both food and forensic sectors. Glycine is often used as a sweetener/taste enhancer in foods and can also be used in certain drug formulations to improve gastric absorption of the drug.

In order to develop a reference material certified for absolute carbon isotope ratios, LGC scientists first had to develop a novel calibration strategy based on the use of gravimetrically prepared synthetic isotope mixtures from enriched carbon isotopes.

Characterisation measurements of the glycine candidate reference material by multicollector-inductively coupled plasma- mass spectrometry (MC-ICPMS) were completed to obtain its $^{13}C/^{12}C$ isotope amount ratio value with known uncertainty value.

Confirmatory isotope ratio data by elemental analysis isotope ratio mass spectrometry (EA-IRMS) was obtained for the glycine candidate reference material for certification purposes. This was shown to be consistent with data obtained by MC-ICPMS. Stability and homogeneity measurements required for certification of the material were also completed using EA-IRMS, and the reference material was certified for absolute carbon isotope ratios traceable to the SI.

Impact

Glycine with certified $^{13}C/^{12}C$ isotope amount ratio traceable to the SI has been approved by the European Reference Materials (ERM®) consortium and is the first reference material produced with SI traceable absolute carbon isotope ratios. The material, ERM®-AE672a, is available from LGC's Standards division.

LGC's scope of accreditation for the production of reference materials to ISO Guide 34[i] has been extended to include the production of glycine characterised for the $^{13}C/^{12}C$ isotope ratio, and ISO/IEC 17025[ii] accreditation for the measurement step has also been achieved.

LGC is the only laboratory in the world to be accredited to ISO Guide 34 for production of reference materials for absolute carbon isotope ratio, and also as a testing laboratory for carbon isotope ratios by MC-ICPMS.

Dmitriy Malinovskiy, Science Leader in isotope ratio analysis, says:

> *"Measurements using a reference material certified for absolute isotope ratios have particular benefit in that mass balance calculations for measurement results are significantly more accurate. Use of the reference material will therefore enhance the quality of measurements in laboratories, enabling analysts to produce traceable, comparable and reliable results."*

[i] ISO Guide 34:2009. General requirements for the competence of reference material producers

[ii] ISO/IEC 17025:2005. General requirements for the competence of testing and calibration laboratories

For further information, contact:
LGC, Queens Road, Teddington, Middlesex TW11 0LY, UK

Tel: **+44 (0)20 8943 7393** Email: **nmshelp@lgcgroup.com** Web: **www.lgcgroup.com/nmi**

National Measurement System

The National Measurement System delivers world-class measurement for science and technology through these organisations

National Measurement Office

NPL National Physical Laboratory

LGC

nel technology for life

4251/LB/0215