

Certificate of measurement



4005

Glycine solutions – Absolute carbon isotope ratio

Certified Reference Material LGC171-KT

Certified values

Solution name	$n(^{13}\text{C})/n(^{12}\text{C})$ ¹ (ratio)	Uncertainty ² (ratio)
LGC1711	0.010642	0.000030
LGC1712	0.010821	0.000030
LGC1713	0.011227	0.000032

Notes:

1. The certified value is the absolute carbon isotope ratio $n(^{13}\text{C})/n(^{12}\text{C})$ determined at LGC using multi-collector inductively coupled plasma - mass spectrometry (MC-ICP-MS) calibrated with synthetic isotope mixtures, and is traceable to the SI through calibration standards of known purity.
2. The quoted uncertainty is the half-width of the expanded uncertainty interval calculated using a coverage factor (k) of 2, providing a level of confidence of approximately 95 %.

Date of Issue: March 2018
Last certificate update: May 2019

Signed: _____
Gill Holcombe (Mrs)
for the Government Chemist



Indicative values

Solution name	$\delta^{13}\text{C}_{\text{VPDB-LSVEC}}$ ² (‰)	Uncertainty ³ (‰)	Coverage factor (<i>k</i>)
LGC1711 ¹	-42.13	0.26	2
LGC1712 ¹	-24.62	0.23	2
LGC1713 ¹	+12.55	0.21	4.3

1) Not within LGC's scope of accreditation to ISO 17034.

2) The $\delta^{13}\text{C}$ values are expressed as parts per thousand (‰) difference from the Vienna PDB (VPDB-LSVEC) isotope scale^[1]. These values were determined by FIA-CO-IRMS (flow injection analysis – chemical oxidation – isotope ratio mass spectrometry) at LGC.

3) The quoted uncertainty is the half-width of the expanded uncertainty interval, which gives a level of confidence of approximately 95 %.

Material Preparation

The materials were prepared using commercially available high purity glycines dissolved in 0.08 M phosphoric acid. For each solution the glycine mass concentration is approximately 150 g/L.

Each solution was dispensed as 2 mL portions into amber glass ampoules. After flushing with argon, the ampoules were flame sealed and then stored at (18 ± 6) °C. Five hundred and fifty units of each solution were produced and each kit contains one ampoule of each solution.

Homogeneity Assessment

The solutions were tested for between-unit homogeneity by analysing randomly selected units for $\delta^{13}\text{C}_{\text{VPDB-LSVEC}}$ using FIA-CO-IRMS, as described for the confirmation data. The level of homogeneity was fit for purpose.

Stability

The nature of the material is such that deterioration is not anticipated over its lifetime when stored under the recommended conditions. The uncertainty associated with long-term instability was considered to be negligible compared with the uncertainty contributions from characterisation and possible inhomogeneity, and was therefore assigned a value of zero. The material will be monitored at LGC and customers will be notified of any changes in the certified values.

Certification

Certification was based on measurements carried out at LGC.

Absolute carbon isotope ratios were determined using gravimetrically prepared primary calibration standards with known $^{13}\text{C}/^{12}\text{C}$ isotope ratios. These measurement standards were prepared by the gravimetric mixing of two chemically identical glycine materials (sourced from Isotech Inc., USA), which had been made into solution using 0.3 M HNO_3 . One of the standards was highly enriched in the ^{13}C isotope, and the other depleted in the ^{13}C isotope. Measurements were performed using MC-ICP-MS calibrated with the synthetic isotope mixtures and the resulting $^{13}\text{C}/^{12}\text{C}$ isotope ratio is fully traceable to the SI unit of the kg. Full details of the method, which has ISO/IEC 17025 accreditation, can be found in two recent publications^{[2],[3]}. The calibration standards were checked for elemental impurities by ICP-MS, water content by Karl Fischer titration and organic carbon impurities by ^1H NMR.

Confirmatory measurements were performed by FIA-CO-IRMS using synthetic isotope mixtures.

In addition, delta values for $^{13}\text{C}/^{12}\text{C}$ on the VPDB scale were determined for each glycine and then converted to a $^{13}\text{C}/^{12}\text{C}$ isotope amount ratio using literature values for the $^{13}\text{C}/^{12}\text{C}$ isotope amount ratio of the VPDB standard. The $^{13}\text{C}/^{12}\text{C}$ isotope amount ratios obtained by EA-IRMS using two calibration strategies, are in agreement with each other and with the characterisation value obtained by MC-ICP-MS.

Accreditation

Property values on this certificate are within LGC's scope of accreditation to ISO 17034 unless otherwise indicated in the tables of values.

Intended Use

This reference material kit is primarily intended for use in the calibration of instruments for the determination of bulk absolute carbon isotope ratios. The kit can also be used for validation of new methods, monitoring of the performance of methods and for the training and evaluation of staff.

Please note that LGC1713 was prepared by mixing together a glycine material highly enriched in ^{13}C in both positions and a glycine material with a natural isotopic composition. For some techniques, e.g. mass spectrometry, an unusual proportion of ^{13}C doubly-labelled glycine will be detected. This should not present a problem for techniques which decompose the glycine molecule before the measurement of the isotope ratio. Provided that reference materials and unknown samples are quantitatively decomposed, both inorganic and organic samples can be calibrated with this kit.

Instructions for Use

Carefully open each ampoule. The minimum amount of solution to be used is 5 μL , which is based on the sample size taken for the homogeneity assessment.

Storage and Shelf Life

The sealed ampoules should be stored at $(20 \pm 5)^\circ\text{C}$ until required for analysis.

Based on measurements carried out at LGC, once ampoules are opened, any remaining sample can be stored in a screw-top amber glass vial at $(20 \pm 5)^\circ\text{C}$ for up to one year.

If stored under the recommended conditions the certified values will remain valid for 12 months from the date of shipment (see page 5 for shipment date).

Metrological Traceability

The certified values are traceable to the SI through the use of calibration standards prepared in house by the gravimetric mixing of two chemically identical glycines, one highly enriched in the ^{13}C isotope, and the other depleted in the ^{13}C isotope. Weighing operations are traceable to the SI through the use of calibrated weights traceable to the National Primary Standard of mass via the UK National Physical Laboratory (NPL).

The purity of the glycine materials was established at LGC with appropriate calibration and control of the instrumentation used in accordance with the requirements of ISO/IEC 17025. LGC's capability to produce SI traceable measurements of purity is verified by participation in international key comparison studies organised by the Organic Analysis Working Group of the Consultative Committee for Amount of Substance (CCQM, <http://www.bipm.org/metrology/chemistry-biology/>).

The indicative values are traceable to the $\delta^{13}\text{C}_{\text{VPDB-LSVEC}}$ scale through the use of the following calibrants (uncertainties quoted here are standard uncertainties):

USGS40 l-glutamic acid $\delta^{13}\text{C}_{\text{VPDB-LSVEC}} = (-26.39 \pm 0.04) \text{‰}$ (USGS, Virginia, USA)

IAEA-CH-6 sucrose $\delta^{13}\text{C}_{\text{VPDB-LSVEC}} = (-10.45 \pm 0.03) \text{‰}$ (IAEA, Vienna, Austria)

USGS41 l-glutamic acid $\delta^{13}\text{C}_{\text{VPDB-LSVEC}} = (+37.63 \pm 0.05) \text{‰}$ (USGS, Virginia, USA)

References

- [1] Coplen TB, Brand WA, Gehre M, Gröning M, Meijer HAJ, Toman B, Verkouteren RM (2006) Anal. Chem. 78:2439–2441
- [2] Malinovsky D, Dunn PJH, Goenaga-Infante H (2013) J. Anal. At. Spectrom. 28:1760-1772
- [3] Dunn PJH, Malinovsky D, Goenaga-Infante H (2015) Anal. Bioanal. Chem. 407: 3169-3180

Certificate Revision

This certificate was revised in September 2018 to clarify the advice for users regarding opened units, and provide further advice in the instructions for use section.

This certificate was revised in May 2019 to make minor corrections to the indicative values table and update the references from ISO Guide 34 to ISO 17034.

SPECIMEN

Unit Number

Date of Shipment

Legal Notice

The values quoted in this certificate are the best estimate of the true values within the stated uncertainties and based on the techniques described herein. No warranty or representation, express or implied, is made that the use of the product or any information, material, apparatus, method or process which is the subject of or referred to in this certificate does not infringe any third party rights. Further, save to the extent: (a) prohibited by law; or (b) caused by a party's negligence; no party shall be liable for the use made of the product, any information, material, apparatus, method or process which is the subject of or referred to in this certificate. In no event shall the liability of any party exceed whichever is the lower of: (i) the value of the product; or (ii) £500,000; and any liability for loss of profit, loss of business or revenue, loss of anticipated savings, depletion of goodwill, any third-party claims or any indirect or consequential loss or damage in connection herewith is expressly excluded.

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