Reference Material for $\delta^{15}$N

REFERENCE SHEET

USGS25

NIST name: RM 8550

Ammonium Sulfate

Reference value of $\delta^{15}$N$_{\text{AIR}}$ of USGS25 expressed in per mille (‰) on the AIR scale$^{(a)}$ and its associated standard uncertainty ($k=1$).

<table>
<thead>
<tr>
<th>Stable isotope ratio</th>
<th>Reference value$^{(b)}$</th>
<th>Standard uncertainty with $k=1$$^{(b)}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\delta^{15}$N$_{\text{AIR}}$ (‰)</td>
<td>-30.41</td>
<td>0.27</td>
</tr>
</tbody>
</table>

$^{(a)}$ AIR refers to atmospheric nitrogen, which is homogeneous, universally constant and has by convention a $\delta^{15}$N of 0‰ [1, 2].

$^{(b)}$ Values are calculated as mean and standard deviation, after elimination of outliers [2], from the 13 accepted data sets, each being obtained by different laboratories and different methods of determination.
Origin and preparation of the material

USGS25 is a dried salt of ammonium sulfate \([(NH_4)_2SO_4]\) prepared by J.K. Böhlke (USGS, Reston, Virginia, USA) in 1992 [2, 3]. The material was prepared by dissolving and recrystallizing a mixture of Fisher ammonium sulfate (A938-500, lot #915021) and Cambridge Isotope Laboratories ammonium sulfate (99.99% 14N; lot BI 1328).

Homogeneity of the material

USGS25, when analysed in different laboratories during the characterisation study, was found homogeneous for sample sizes in the range of 1 - 10 mg [2, 3]. 15 aliquots of 1.3 mg from different bottles were analysed [3], using off-line combustion followed by dual-inlet IRMS, against the same aliquot of reference gas (NSVEC). The results indicate that USGS25 is isotopically homogeneous within the normal analytical precision of the mass spectrometer (k=1, 0.02-0.04 ‰).

Characterization study

The current characterisation was performed in 1993 based on the results of an inter-laboratory comparison test involving 15 laboratories [2]. The δ¹⁵N values were measured against Air N₂ by isotope ratio mass spectrometry on N₂ gas quantitatively obtained off-line using different combustion methods followed by purification steps [2]. The measured results were then normalized against USGS32 using the consensus δ¹⁵N value relative to Air N₂ of +180‰. The assigned value has been confirmed in two laboratories using both online combustion continuous flow and offline dual-inlet mass spectrometry in 2003 [4] and at the IAEA using an online combustion continuous flow system in 2021.

Assignment of values

The reference value and uncertainty have been determined by calculating arithmetic mean and standard deviation of the data of the inter-laboratory comparison test, after elimination of values that differ from the means by more than two standard deviations, followed by recalculation and elimination until all values were within two standard deviations.

The details concerning the reported results as well as the calculation may be found in reference [2].

The δ¹⁵N value and uncertainty are the values accepted by the Commission on Isotopic Abundances and Atomic Weights of the International Union of Pure and Applied Chemistry (IUPAC) in 2014 [5] and agreed with NIST and USGS, as of the date of this report. Moreover, they have been accepted by the Stable isotope experts participating to the Technical Meeting on the Development of IAEA Stable Isotope Reference Materials and Related Products, 30 August – 3 September 2021.

Statement on metrological traceability and uncertainty of assigned values

The value is traceable to the δ¹⁵Nₐir scale, normalised by assigning a consensus value of +180‰ to USGS32. For calibration against atmospheric N₂, different reference materials (air, salts, and tank gases) with δ¹⁵N values between approximately -3‰ and +3‰ were used.

The uncertainty of the assigned value is the standard deviation of the accepted results obtained during the characterisation study [2].
Absolute isotopic abundances
No absolute isotope abundance ratio determination was performed on this material.

Intended use
USGS25 is a secondary reference material. Its intended use is as calibrant for samples that need combustion, for obtaining traceability of the data to the $\delta^{15}$N_{AIR} scale.

Instructions for use, handling and storage
USGS25 is issued in units of 0.5 g.
As the material is hygroscopic, it is recommended that it is stored in cool and dry place in the tightly closed container in which it was supplied to the user.
Upon opening and taking a portion for analysis, the container must be rapidly and tightly closed.
The recommended minimum sample size to be used for analysis is 1 mg.

Limit of distribution
Only one unit per year per laboratory may be ordered. This strategy should ensure that material is available for international use for as long as possible.

Issue and expiry date
The issue date of USGS25 was January 1995 with its current characterisation published in 1995. The issue date of this reference sheet is provided in the footer of this document.
The reference value was confirmed in 2003 and in 2021. Based on this, the expiry date is February 2032, provided the original packages are handled and stored in accordance with the instructions given in this reference sheet (see “Instructions for use, handling and storage”).
IAEA is monitoring the long-term stability of the material and customers will be informed in case of any observed change.

Legal disclaimer
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Compliance with ISO Guide 31:2015
The content of this IAEA Reference Sheet is in compliance with ISO Guide 31:2015, Reference materials – Contents of certificates, labels and accompanying documentation [6].

Citation of this reference sheet
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Note

Reference values as stated in this reference sheet may be updated if more information becomes available. Users of this material should ensure that the reference sheet in their possession is current. The current version can be found in the IAEA’s Reference Materials online catalogue:

https://nucleus.iaea.org/sites/ReferenceMaterials/Pages/Stable-Isotopes.aspx

Further information:

For further information regarding this material, please contact the producer:

Terrestrial Environmental Radiochemistry Laboratory
Division of Physical and Chemical Sciences
Department of Nuclear Sciences and Applications
International Atomic Energy Agency
Vienna International Centre
PO Box 100
1400 Vienna
Austria
Tel.: +43 1-2600-28237
E-mail: NAEL-Reference-Materials.Contact-Point@iaea.org

References


The project officer is responsible for the content of this reference sheet. The Chair of the RM Certification Committee approves this reference sheet and authorizes its release on behalf of the Committee.