National Institute of Standards and Technology
U.S. Department of Commerce

Date of Issue:
18 January 2023

Reference Material 8550
USGS25
(Nitrogen Isotopes in Ammonium Sulfate)

REFERENCE MATERIAL INFORMATION SHEET

Purpose: This Reference Material (RM) is a secondary reference material with known nitrogen stable isotope ratios [1,2]. It is intended to be a validation material for nitrogen stable isotope ratios of working standards that have been calibrated relative to atmospheric nitrogen as $\delta^{15}N_{AIR}$. The equivalent name used by the International Atomic Energy Agency (IAEA) and the U.S. Geological Survey (USGS) for this RM is USGS25.

Description: RM 8550 consists of one vial containing approximately 0.8 g of ammonium sulfate salt ($[NH_4]_2SO_4$).

Non-Certified Values: Although not certified, the assigned isotope-delta value for this RM, provided in Table 1 below, is at present the best estimate of the true value.

Table 1. Non-Certified Value for Nitrogen Stable Isotopes of RM 8550 (USGS25)

NIST RM Number	Name	Non-Certified Value $\delta^{15}N_{AIR}$	Combined Uncertainty $\delta^{15}N_{AIR}$ [(a)]	Expanded Uncertainty $\delta^{15}N_{AIR}$ [(a)]
8550	USGS25	–30.41 ‰	0.27 ‰	0.54 ‰

[(a)] RM 8550 is given with a combined standard uncertainty in addition to an expanded uncertainty value, $k = 2$. The expanded uncertainty is equal to $U = ku_c$, where u_c is the combined standard uncertainty and k is the coverage factor, as defined in the ISO/JCGM Guide [3]. The non-certified value and uncertainties are given in units of per mil (‰), which is equivalent to per thousand.

Metrological Traceability: RM 8550 is a secondary reference material with a known nitrogen stable isotope value. The nitrogen stable isotope value assigned for RM 8550 is traceable to atmospheric nitrogen (AIR) and RM 8558 (USGS32). While atmospheric nitrogen officially defines the zero delta point for the nitrogen δ-scale [4,5], RM 8547 is recommended as the scale anchor point for samples with a preparation requiring combustion [2].

Stable isotope values for nitrogen are not traceable to the International System of Units (SI) or other higher-order reference system [3,6]. A *Traceability Exception* has been approved by the Bureau International des Poids et Mesures (BIPM) International Committee for Weights and Measures (CIPM), which states non-SI traceable isotope values "should be made traceable to materials recognized as International Standards" [6,7].

Period of Validity: The non-certified value is valid within the measurement uncertainty specified until **31 December 2032.** The value assignments are nullified if the material is stored or used improperly, damaged, contaminated, or otherwise modified.

Maintenance of Non-Certified Value: NIST will monitor this material to the end of its period of validity. If substantive technical changes occur that affect the non-certified value during this period, NIST will update this Reference Material Information Sheet and notify registered users. RM users can register online from a link available on the NIST SRM website or fill out the user registration form that is supplied with the RM. Registration will facilitate notification. Before making use of any of the values delivered by this material, users should verify they have the most recent version of this documentation, available through the NIST SRM website (https://www.nist.gov/srm).

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Safety: Consult the Safety Data Sheet (SDS) for hazard information.

Storage: This RM should be kept in a dry environment as it will attract water when exposed to air. To minimize the potential for contamination, it is recommended that this RM be stored in the container in which it is supplied.

Additional Information: The distribution of RM 8550 is limited to one unit per customer per three-year period of time. Users are encouraged to prepare their own standards for daily use and calibrate those standards against international reference materials. Preparation, analysis, and reporting information can be found in Appendix A.

REFERENCES

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Information Sheet Revision History: 18 January 2023 (Revised expanded uncertainty value and added combined uncertainty value; changed period of validity; updated format; editorial changes); 30 January 2013 (Uncertainty updated to an expanded uncertainty for $\delta^{15}N_{AIR}$; expiration date assigned; editorial changes); 03 February 1993 (Updated value for RM 8549; added RM 8558 to report); 22 June 1992 (Original report issue date).

Certain commercial equipment, instruments, or materials may be identified in this Reference Material Information Sheet to adequately specify the experimental procedure. Such identification does not imply recommendation or *endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.*

Users of this RM should ensure that the Reference Material Information Sheet in their possession is current. This can be accomplished by contacting the Office of Reference Materials 100 Bureau Drive, Stop 2300, Gaithersburg, MD 20899-2300; telephone (301) 975-2200; e-mail srminfo@nist.gov; or the Internet at https://www.nist.gov/srm.

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APPENDIX A

PREPARATION AND ANALYSIS

Technical aspects involved in the issuance of this RM were coordinated through the NIST Chemical Sciences Division by R.A. Kraft.

Support aspects involved in the issuance of this RM were coordinated through the NIST Office of Reference Materials.

Sample Preparation: RM 8550 (USGS25) was prepared as a dried salt by dissolving and recrystallizing a mixture of normal reagent salt and ^{15}N-depleted salt by J.K. Böhlke (USGS, Reston, Virginia) [8,9].

Analytical Methods: The non-certified value for δ^{15}N in RM 8550 (USGS25) was derived from an inter-laboratory comparison test after elimination of outliers [9]. The δ^{15}N values were measured by mass spectrometry on N_2 gas that was quantitatively produced using variants of a buffered sample combustion method coupled with additional purification steps. The measured results were then normalized to yield a value of +180 ‰ for RM 8558 (USGS32). Working laboratory standards with a range of nitrogen stable isotope ratios can be produced by the methods described by Böhlke *et al.* [8,10].

Homogeneity: There is no evidence of isotopic heterogeneity in this RM for sample sizes in the range of 10 µmol to 100 µmol of nitrogen [9].

REPORTING

Terminology: The terminology used here is based on the guidance given by IUPAC for isotope terminology, where stable isotope-number ratio refers to the number of atoms of one isotope relative to the number of atoms of a second isotope in the same system [2]. This is often abbreviated to stable isotope ratio. Isotope-delta value refers to the stable isotope-number ratio of a measured sample relative to the stable isotope-number ratio of a reference material (see example below). Isotope-amount ratio is numerically the same as isotope-number ratio but refers specifically to the amount (moles) of an isotope relative to the amount (moles) of another isotope in the same system [11].

Isotope-delta Values: The nitrogen stable isotope-delta value of a measured sample reported on the AIR scale are defined as the difference in measured isotope-number ratio of nitrogen in a sample relative to the isotope-number ratio of nitrogen in AIR:

$$\delta^{15}N = \frac{\left[\dfrac{N_{sample}(^{15}N)}{N_{sample}(^{14}N)}\right] - \left[\dfrac{N_{AIR}(^{15}N)}{N_{AIR}(^{14}N)}\right]}{\left[\dfrac{N_{AIR}(^{15}N)}{N_{AIR}(^{14}N)}\right]}$$

Normalization: By convention AIR is the zero point of the nitrogen stable isotope δ-scale. AIR refers to N_2 of tropospheric air [4,5], for which $R(^{15}N/^{14}N)_{AIR} = 0.003677$ [12,13]. The δ-value for RM 8558 (USGS32) is also defined by convention and has a δ^{15}N value of +180 ‰. A formula for normalizing nitrogen isotope measurement results using two laboratory standards LS1 (AIR) and LS2 (USGS32) can be expressed as:

$$\delta^{15}N_{sample,cal} = \delta^{15}N_{LS1,cal} + \left(\delta^{15}N_{sample,WS} - \delta^{15}N_{LS1,WS}\right) \times f$$

where the normalization factor *f* is:

$$f = \frac{\left(\delta^{15}N_{LS2,cal} - \delta^{15}N_{LS1,cal}\right)}{\left(\delta^{15}N_{LS2,WS} - \delta^{15}N_{LS1,WS}\right)}$$

where *WS* denotes measurements made versus a transfer gas (working standard),
cal denotes calibrated measurements made versus the AIR scale, and
$\delta^{15}N_{LS1,cal}$ and $\delta^{15}N_{LS2,cal}$ are the conventionally fixed δ^{15}N values for AIR and RM 8558 (USGS32), or those of calibrated laboratory working standards.

The δ-definition above assumes $f = 1$, and does not account for scale compression.

The following recommendations are provided for reporting the relative difference of nitrogen stable isotope-number ratios using the δ-notation modified from Coplen [11], it is recommended that:

- $\delta^{15}N$ values should be reported with respect to air (atmospheric nitrogen gas) and normalized to RM 8558 (USGS32) [12].
- Authors should report δ values of internationally distributed (secondary) isotopic reference materials that were assumed for normalization of data for samples of similar chemical composition, as appropriate for the measurement method. In this manner, measurement results can be adjusted in the future as analytical methods improve and consensus values of internationally distributed isotopic reference materials change.

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