National Institute of Standards & Technology

Report of Investigation

Reference Material 8548

IAEA-N-2

(Nitrogen Isotopes in Ammonium Sulfate)

This Reference Material (RM) is intended for use in developing and validating methods for measuring relative differences in nitrogen (N) isotope-number ratios, $R(^{15}N/^{14}N)$ [1]. Even though the value for this RM is a reference value and not certified [2], its use will improve the comparability of data from different laboratories. The equivalent name used by the International Atomic Energy Agency (IAEA) and the U.S. Geological Survey (USGS) for this RM is IAEA-N-2. RM 8548 consists of one vial containing approximately 0.4 g of ammonium sulfate salt ($(NH_4)_2SO_4$).

Table 1. Reference Value[(a)] and Expanded Uncertainty
for the Relative N Isotope-Number Ratio Difference of RM 8548

RM Number	Name[(b)]	Reference Value $10^3\ \delta\ ^{15}N_{AIR}$[(c)]	Expanded Uncertainty $10^3\ \delta\ ^{15}N_{AIR}$[(c)]
8548	IAEA-N-2	+20.41	±0.07

(a) A reference value is a non-certified value that is the best estimate of the true value; however, the value may reflect only the measurement precision and may not include all sources of uncertainty [2].

(b) IAEA-N-2 is also known as IAEA-N2.

(c) The $\delta^{15}N_{AIR}$ value is expressed as a mean and an expanded uncertainty. The expanded uncertainty is equal to $U = ku_c$, where u_c is the combined standard uncertainty as defined by the ISO Guide [3] and k is the coverage factor. The combined standard uncertainty is intended to represent, at the level of one standard deviation, the effect of random errors on the reference value that were evaluated by statistical means (Type A). Any uncertainty due to biases in the methods is not included in the expanded uncertainty. The coverage factor, $k = 2.179$ (n=13), provides an expanded uncertainty interval that has about a 95 % probability of encompassing the mean. The values for RM 8548 are taken from reference 4. (AIR – N_2 of tropospheric air).

Reference Difference in Isotope-Number Ratio Values: The differences in measured isotope-number ratios of stable nitrogen isotopes in substance P, $R(^{15}N/^{14}N)_P = [N(^{15}N)_P / N(^{14}N)_P]$, are reported as $\delta^{15}N$ values [5]. The relative differences in isotope-number ratios for nitrogen are referenced to AIR where:

$$\delta^{15}N = [R(^{15}N/^{14}N)_{sample} / R(^{15}N/^{14}N)_{AIR}] - 1$$

AIR refers to N_2 of tropospheric air [6], for which $R(^{15}N/^{14}N)_{AIR} = 0.003677$ [5,7,8]. The reported $\delta^{15}N$ results have been normalized to yield a value of +180 ‰ for RM 8558 (USGS32) [4], where the symbol ‰ is part per thousand and is equal to 0.001.

Expiration of Value Assignment: RM 8548 is valid, within the measurement uncertainty specified, until **31 December 2020** provided the RM is handled in accordance with instructions given in this Report of Investigation (see "Instructions for Storage, Handling, and Use"). This report is nullified if the RM is damaged, contaminated, or otherwise modified.

Maintenance of RM: NIST will monitor this RM over the period of its validity. If substantive technical changes occur that affect the value assignment before the expiration of this report, NIST will notify the purchaser. Registration (see attached sheet) will facilitate notification.

Support and technical aspects of the preparation, analysis, and distribution of this RM was coordinated through the NIST Chemical Sciences Division by R.D. Vocke, Jr.

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Office of Reference Materials

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

INSTRUCTIONS FOR STORAGE, HANDLING, AND USE

Storage and Handling: 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.

Use: It is recommended that RM 8548 (IAEA-N-2) be used to calibrate or check working laboratory standards that are used routinely. Working laboratory standards with a range of N isotope-ratios can be produced by the methods described by Böhlke *et al.* [9,10].

Distribution: The distribution of RM 8548 (IAEA-N-2) is limited to one unit per customer per three-year period of time.

PREPARATION AND ANALYSIS

Preparation: RM 8547 (IAEA-N-2) was prepared as a relatively coarse dry salt by E. Salati (CENA, Piracicaba, Sao Paulo, Brazil) between 1978 and 1983 [11,12].

Analytical Methods: The reference value for $\delta^{15}N$ in RM 8548 (IAEA-N-2) was derived from an inter-laboratory comparison test after elimination of outliers [4]. The $\delta^{15}N$ 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).

The $\delta^{15}N$ value and expanded uncertainty reported in Table 1 are the values accepted by the Commission on Isotopic Abundances and Atomic Weights of the International Union of Pure and Applied Chemistry (IUPAC) (http://ciaaw.org/Nitrogen.htm) and the IAEA as of the date of this report.

Isotopic 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 [4].

Normalization of Data: The $\delta^{15}N$ values in samples should be normalized to the AIR-USGS32 scale by calibrating the measurement with respect to atmospheric N_2 [5] and the $\delta^{15}N$ value of the ^{15}N-enriched anchor, RM 8558 (USGS32) [4]. A general formula for normalizing nitrogen isotope measurement results using two laboratory standards LS1 (AIR) and LS2 (USGS32) can be expressed as:

$$\delta^{15}N_{sample,cal} = \delta^{15}N_{LS1,cal} + \left(\delta^{15}N_{sample,WS} - \delta^{15}N_{LS1,WS}\right) \times f \qquad (1)$$

where the normalization factor f is:

$$f = \left(\delta^{15}N_{LS2,cal} - \delta^{15}N_{LS1,cal}\right) \Big/ \left(\delta^{15}N_{LS2,WS} - \delta^{15}N_{LS1,WS}\right) \qquad (2)$$

Note: In the above formulas, cal denotes calibrated measurements made versus the AIR–USGS32 scale, and $\delta^{15}N_{LS1,cal}$ and $\delta^{15}N_{LS2,cal}$ are the conventionally fixed $\delta^{15}N$ values for AIR and USGS32. WS denotes measurements made versus a transfer gas (working standard), and $\delta^{15}N_{LS1,WS}$ and $\delta^{15}N_{LS2,WS}$ are the $\delta^{15}N$ values for calibrated laboratory working standards.

Reporting of Nitrogen Stable Isotope *δ*-values: The following recommendations from IUPAC are provided for reporting $\delta^{15}N$ values [5]. It is recommended that:

- the value of 272 be employed for $^{14}N/^{15}N$ of N_2 in air for the calculation of atom fraction ^{15}N from measured $\delta^{15}N$ values;
- all $\delta^{15}N$ values should be reported with respect to air (atmospheric nitrogen gas) and normalized to RM 8558 (USGS32).

In addition, researchers are encouraged to report the isotopic composition of RM 8548 (IAEA-N-2) and other internationally distributed nitrogen isotopic reference materials [13] in their publications, as appropriate to the method, as though they have been interspersed among unknowns.

Current Reports of Investigation (ROI) for all light stable isotopic Reference Materials mentioned in this report are available on the SRM web site [14].

REFERENCES

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Report Revision History: 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)

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