



# Report of Investigation

## Reference Material 8552

### NSVEC

#### (Nitrogen Isotopes in Gaseous Nitrogen)

This Reference Material (RM) is intended for use in developing and validating methods for measuring relative differences in nitrogen (N) isotope-number ratios,  $R(^{15}\text{N}/^{14}\text{N})$  [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 NSVEC (Nitrogen Svec: named for H. Svec, formerly of Ames Lab, IA). RM 8552 is supplied in a borosilicate tube containing approximately 300  $\mu\text{mol}$  of  $\text{N}_2$ .

Table 1. Reference Value<sup>(a)</sup> and Expanded Uncertainty for the Relative N Isotope-Number Ratio Difference of RM 8552.

RM Number	Name	Reference Value $10^3 \delta^{15}\text{N}_{\text{AIR}}^{(b)}$	Expanded Uncertainty $10^3 \delta^{15}\text{N}_{\text{AIR}}^{(b)}$
8552	NSVEC	-2.78	$\pm 0.03$

<sup>(a)</sup> 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].

<sup>(b)</sup> The  $\delta^{15}\text{N}_{\text{AIR}}$  value is expressed as a mean and an expanded uncertainty. The expanded uncertainty is equal to  $U = k u_c$ , 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,  $k = 2.365$  ( $n=8$ ), provides an expanded uncertainty interval that has about a 95 % probability of encompassing the mean. The values for RM 8552 are taken from reference 4 (AIR – tropospheric air).

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

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

AIR refers to  $\text{N}_2$  of tropospheric air [6], for which  $R(^{15}\text{N}/^{14}\text{N})_{\text{AIR}} = 0.003677$  [5,7,8]. The reported  $\delta^{15}\text{N}$  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 8552 is valid, within the measurement uncertainty specified, until **31 December 2020**, provided the RM is handled and stored 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.

Support aspects involved in the issuance of this RM were coordinated through the NIST Measurement Services Division.

Carlos A Gonzalez, Chief  
Chemical Sciences Division

Robert L. Watters, Jr., Director  
Office of Reference Materials

## INSTRUCTIONS FOR HANDLING, STORAGE, AND USE

**Handling and Storage:** Until use, it is recommended that this RM be stored in the original glass tube at ambient temperature (20 °C to 30 °C).

**Use:** Opening this RM requires the proper use of a suitable tube cracker [9–12], and after opening, the RM should be used immediately for calibration or standardization. It is recommended that it be used to calibrate or check working laboratory standards that are used routinely. Working laboratory standards with a range of  $\delta^{15}\text{N}$  values can be produced by the methods described by J.K. Böhlke et al. [13,14].

**Distribution:** Because the supply of RM 8552 (NSVEC) is limited, one unit may be purchased by a customer each three-year period of time.

### PREPARATION AND ANALYSIS

**Preparation:** RM 8552 (NSVEC) is a pure tank gas that is believed to be the same tank gas identified as “Matheson pre-purified  $\text{N}_2$ ” by Junk and Svec [4]. The high pressure  $\text{N}_2$  cylinder resides at the USGS, Reston, Virginia.

**Analytical Methods:** The reference value for  $\delta^{15}\text{N}$  of RM 8552 (NSVEC) was derived from results of an interlaboratory comparison test after elimination of outliers [4]. The  $\delta^{15}\text{N}$  values were measured by mass spectrometry on  $\text{N}_2$  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  $\delta^{15}\text{N}$  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 reference material [4].

**Normalization of Data:** The  $\delta^{15}\text{N}$  values in samples should be normalized to the AIR-USGS32 scale by calibrating the measurement with respect to atmospheric  $\text{N}_2$  [5] and the  $\delta^{15}\text{N}$  value of the  $^{15}\text{N}$ -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:

$$\delta^{15}\text{N}_{\text{sample,cal}} = \delta^{15}\text{N}_{\text{LS1,cal}} + (\delta^{15}\text{N}_{\text{sample,WS}} - \delta^{15}\text{N}_{\text{LS1,WS}}) \times f \quad (1)$$

where the normalization factor  $f$  is:

$$f = \frac{(\delta^{15}\text{N}_{\text{LS2,cal}} - \delta^{15}\text{N}_{\text{LS1,cal}})}{(\delta^{15}\text{N}_{\text{LS2,WS}} - \delta^{15}\text{N}_{\text{LS1,WS}})} \quad (2)$$

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

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

- the value of 272 be employed for  $^{14}\text{N}/^{15}\text{N}$  of  $\text{N}_2$  in air for the calculation of atom fraction  $^{15}\text{N}$  from measured  $\delta^{15}\text{N}$  values;
- all  $\delta^{15}\text{N}$  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 8552 (NSVEC) and other internationally distributed nitrogen isotopic reference materials [15] in their official 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 NIST Standard Reference Materials web site [16].

## REFERENCES

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<b>Report Revision History:</b> 30 January 2013 (Expanded uncertainty added for $\delta^{15}\text{N}_{\text{AIR}}$ value; 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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