National Institute of Standards & Technology

Report of Investigation

Reference Materials 8559, 8560, 8561

8559: Natural Gas, Coal Origin (>80% methane)
8560: Natural Gas, Petroleum Origin (>50% methane)
8561: Natural Gas, Biogenic (>95% methane)

These Reference Materials (RMs) are intended to provide natural gas samples having compound specific values for isotopic composition and uncertainty with $^{13}C/^{12}C$ ratios expressed in parts per thousand relative difference ($\delta^{13}C$ as ‰) from Vienna Peedee Belemnite (VPDB) [1]. References to compound specific values determined outside National Institute of Standards and Technology (NIST) for $\delta^{13}C$, $\delta^{2}H$ and $\delta^{15}N$ are provided as supplemental information. RMs 8559–8561 are not certified, but the use allows comparability of stable carbon and hydrogen isotope ratio data obtained by investigators in different laboratories, and provides a means to standardize laboratory gases with measurement traceability to the internationally accepted isotope ratio reference scale. An RM unit consists of a 50 mL stainless steel cylinder sealed with an all-metal seat in a bellows valve. Each cylinder contains about 10 mmol (almost 2 g) of gas compressed to about 5 MPa (800 psia).

Reference Values: Compound-specific isotopic reference values and expanded uncertainties are in Table 1.

Table 1. $\delta^{13}C$ NIST Reference Values and Expanded Uncertainties (U)(†) vs. VPDB

Material	RM 8559 $\delta^{13}C$ vs. VPDB ± U (‰)			RM 8560 $\delta^{13}C$ vs. VPDB ± U (‰)			RM 8561 $\delta^{13}C$ vs. VPDB ± U (‰)		
Methane	-29.11(c)	±	0.09	-44.84(c)	±	0.09	-73.27(c)	±	0.09
Ethane	-25.9(b)	±	0.2	-31.8(b)	±	0.3	no data		
Propane	-22.12(b)	±	0.09	-25.34(b)	±	0.09	no data		
n-Butane	-21.8(a)	±		-22.67(b)	±	0.10	no data		
iso-Butane	-24.7(a)	±		-24.9(b)	±	0.2	no data		
n-Pentane	no data			-21.6(a)			no data		
iso-Pentane	no data			-22.8(a)			no data		
Carbon dioxide	-9.2(b)	±	0.8	-8.5(c)	±	0.3	no data		

(†) $U = ku_c$, where u_c is the combined standard uncertainty and $k = 2$ is the coverage factor [2]. Reference values are calculated on the basis of currently accepted oxygen isotope relationships, where $\lambda = 0.50$ and $K = 0.0083329582$ [3], and are traceable to VPDB through RM 8563 ($\delta^{13}C_{VPDB} = -41.56$ ‰ ± 0.09 ‰) [4].

(a) informational value from one laboratory – no uncertainty provided

(b) The reference value is a weighted mean of the results from two analytical methods. The uncertainty is calculated by combining a between-method variance, a pooled within-source variance, and the uncertainty in the RM 8563 value assignment, following the ISO and NIST Guides [2].

(c) The reference value is a weighted mean of the results from three analytical methods. The uncertainty is calculated by combining a between-method variance incorporating inter-method bias, a pooled within-source variance, and the uncertainty in the RM 8563 value assignment, following the ISO and NIST Guides [2].

Expiration of Reference Values: The reference values for the isotopic composition of RM 8559, RM 8560, RM 8561 are valid until **31 December 2017**, provided they are handled and stored in accordance with the instructions given in this report.

Maintenance of Reference Values: The isotopic compositions of these RMs are monitored on a yearly basis. If substantive changes occur that affect the certification before the expiration of this certificate, NIST will notify the purchaser. Registration (see attached sheet) will facilitate notification.

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See Report Revision History on Last Page

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The technical aspects involved in the issuance of this RM were coordinated through the NIST Analytical Chemistry Division by R.D. Vocke, Jr.

Consultation on the statistical evaluation of the data was provided by S.D. Leigh of the NIST Statistical Engineering Division.

Support aspects involved in the issuance of these RMs were coordinated through the NIST Measurement Services Division.

INSTRUCTIONS FOR USE

Storage and Handling: As these materials are classified as hazardous due to their flammability and compressed nature, the user should become familiar with information in the Material Safety Data Sheet (MSDS) that accompanies these RMs. Until use, it is recommended that the gases be stored in their original container at ambient temperature (20 ºC to 30 ºC). Sampling schemes should employ clean, leaktight vacuum practices, and avoid extended molecular flow conditions (< 25 hPa) that could lead to significant isotopic fractionation. It is not recommended to use the gas mixture after the pressure drops below 250 hPa. To minimize surface effects with the higher hydrocarbons, the RM units were prepared at 80 ºC. This temperature should be used when sampling the gas mixture for butanes and heavier compounds. To avoid valve damage and subsequent leakage, the valve should not be overtightened: applied torque should not exceed 1 N-m (9 in-lb). For further information regarding the valves or cylinders, please contact the appropriate manufacturer.

$$\delta^{m}_{\mathrm{LRM}.normalized} = \delta^{m}_{\mathrm{RM1}.assigned} + \left[\frac{\delta^{m}_{\mathrm{LRM}.measured} - \delta^{m}_{\mathrm{RM1}.measured}}{\delta^{m}_{\mathrm{RM2}.measured} - \delta^{m}_{\mathrm{RM1}.measured}}\right] \times \left[\delta^{m}_{\mathrm{RM2}.assigned} - \delta^{m}_{\mathrm{RM1}.assigned}\right] \qquad \text{Eq.1}$$

Use: These RMs may be used to determine normalized δ^{13}C and δ^{18}O values of compounds within a laboratory reference mixture (LRM) through interpolation using Equation 1, where δ^{m} values are measured or assigned δ^{13}C (m=13) values of a compound in two RMs (RM1 and RM2) that bracket the composition of the LRM. Upon establishing the normalized composition of at least two LRMs, these in turn may be used routinely to determine the normalized compositions of samples. When reporting normalized delta values of samples, the assigned values of the compounds in the RMs should also be reported. A website is available at http://www.nist.gov/widps-co2 to aid in data standardization, reduction, and normalization [5].

ORIGINS, PREPARATION AND CHARACTERIZATION[1]

Origins: The coal-related gas used for RM 8559 was collected by G. Hut (University of Groningen, The Netherlands) and compressed in two 50 L steel stock cylinders. The gas consists of more than 80 % CH_4 with a δ^{13}C value of approximately –29 ‰ versus VPDB. As an intercomparison material, this gas was known as NGS-1. The oil related gas used for RM 8560 was collected by T. Ricchiuto (Agip S.p.A., Milano, Italy) and compressed in two 50 L steel stock cylinders. This gas mixture contains more than 50 % CH_4 with a δ^{13}C value of approximately –45 ‰ versus VPDB. As an intercomparison material, this gas was known as NGS-2. The biogenic material used for RM 8561 was collected by T. Ricchiuto (Agip S.p.A., Milano, Italy) and compressed in two 50 L steel stock cylinders. The gas contains more than 95 % CH_4 with a δ^{13}C value of approximately –73 ‰ versus VPDB. As an intercomparison material, this gas was known as NGS-3.

Preparation: RMs 8559, 8560, and 8561 were prepared by R.D. Vocke, Jr. of the NIST Surface and Microanalysis Science Division, Chemical Science and Technology Laboratory, National Institute of Standards and Technology, Gaithersburg, MD, USA. For each RM, forty 50 mL cylinders were assembled at NIST and connected to a forty-port stainless steel manifold fitted with an oil-free molecular drag vacuum pump. The RM stock cylinder was connected to the manifold by one meter of stainless steel tubing. All connections were welded or employed all-metal compression fittings. The transfer line, manifold, and 50 mL cylinders were heated to 80 °C and helium leak-tested under vacuum and positive pressure conditions. The stock cylinder was slowly preheated to 80 °C using a controlled heating system described elsewhere [6]. The 50 mL cylinders were flushed with the gas mixture, vented and evacuated, then filled up to the pressure of the heated stock cylinder. After 10 minutes, the valve on each 50 mL cylinder was closed and the system allowed to cool to room temperature overnight. The next day, the cylinder valves were checked for tightness. The RM

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[1] Certain commercial equipment, instrumentation, or materials are identified in this certificate to specify adequately the experimental procedure. Such identification does not imply recommendation or endorsement by the NIST, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

units were scribed to record their position on the manifold, then removed, capped, labeled, and stored.

Isotopic Uniformity: At the high pressures and temperatures used in the production of the RM units, isotopic fractionation and variation across the units are unlikely. However, an estimate of unit-to-unit isotopic heterogeneity was performed at NIST using replicate samples from 10 % of the units prepared for RM 8561, and 7.5 % of the units prepared for RM 8559 and RM 8560. Samples were chosen from cylinders that had been positioned on opposite ends of the manifold during production. As the most abundant compound, the isotopic variability of methane was chosen to represent the isotopic variability of the RM units. The isotopic heterogeneities of compounds other than δ^{13}C in methane were untested.

At NIST, each sampling consisted of about 5 mmol of methane, which was expanded into a 6 L volume and diluted with helium until the pressure exceeded 0.13 MPa (1.3 atm). This gas mixture was regulated to flow at 2 mL/min through a 10 mL loop that was sampled on demand through an actuated 6-way valve. Each 10 microliter sample contained about 8 nmol of methane that was separated from other compounds by gas chromatography through a PLOT capillary column. Upon emerging from the column, the hydrocarbon peaks were converted to carbon dioxide in a ceramic combustion tube containing CuO and Pt at 960 °C. Water vapor was removed from the effluent with a Nafion drier, and the isotope voltage ratios of the CO_2 peaks were monitored through time-integrated high-precision isotope ratio mass spectrometry (IRMS). These measurements were standardized by introducing a sample of reference CO_2 to the effluent and determining relative differences between the reference and sample CO_2, i.e., $\delta^{45}CO_2$ and $\delta^{46}CO_2$ values. These delta values, in turn, were converted to δ^{13}C values on the VPDB isotope ratio scale using the standard algorithm accessible through the NIST website http:/www.nist.gov/widps-co2 [5].

Table 2. Isotopic Heterogeneity of Methane in Reference Material Units

Reference Material	Repeatability within a cylinder u_i of δ^{13}C [(a)] (‰)	Repeatability across cylinders u_i of δ^{13}C means [(a)] (‰)
RM 8559	0.054 (m=5) [(b)]	0.026 (n=3) [(b)]
RM 8560	0.048 (m=5) [(b)]	0.050 (n=3) [(b)]
RM 8561	0.050 (m=5) [(b)]	0.040 (n=4) [(b)]

[(a)] The symbol u_i denotes the standard uncertainty obtained from Type A evaluations only, and is synonymous with the experimental standard deviation [2].

[(b)] m = number of measurements on single sample; n = number of independent RM units sampled

Analytical repeatability averaged 0.051 ‰, which is equivalent or greater than the observed measurement reproducibility across RM units. This indicates that real differences in RM units, if they exist, are masked by measurement imprecision and are insignificant compared with other sources of uncertainty in the value assignments.

Analytical Methods: Standardized δ^{13}C compositions for methane and other carbonaceous compounds in the RM gas mixtures are based on measurements by two laboratories using different methods in collaboration with NIST [7]. Each participating laboratory received one of each candidate RM, and was requested to measure replicate samples of each RM and report the measurements as $\delta^{45}CO_2$ and $\delta^{46}CO_2$ against a laboratory working gas. Traceability of these measurements to the internationally accepted isotope ratio scale (VPDB) was established by various equivalent methods.

Table 3. Methods[(a)] Utilized at Participating Laboratories

Laboratory	Separation Method	Measurement Method	Traceability Method
Isotech Laboratories	Preparatory GC	Dual-inlet IRMS	RM 8563 and RM 8564 in measurement sequence
ExxonMobil Upstream Research Company	Capillary GC (temperature ramp)	GC-C-IRMS (Thermo Finnigan Delta+)	Laboratory reference gas measured at NIST by dual-inlet IRMS against RM 8563 and RM 8564
NIST	Capillary GC (isothermal)	GC-C-IRMS (ThermoFinnigan MAT 252)	RM 8563 diluted and sampled through GC

[(a)] Gas Chromatography (GC)
Gas Chromatography Combustion Isotope Ratio Mass Spectrometry (GC-C-IRMS)
Isotope Ratio Mass Spectrometry (IRMS)

The analytical methods utilized at NIST have been described in the previous section. Techniques used by the other laboratories are described in the open literature [8,9].

Participants in the Interlaboratory Comparison Exercise for RMs 8559-8561:

Dennis Coleman, Chao-Li Liu, Brian Grable, Nhung Le, Alan Langenfeld; Isotech Laboratories, Champaign, IL
Ngami Phan, John Guthrie; Exxon-Mobil Upstream Research Corporation, Houston, TX
Michael Verkouteren; NIST, Gaithersburg, MD.

Data Handling Procedures: At NIST, measurement data from all laboratories were converted to standardized $\delta^{13}C$ values (for each chemical compound) using the standard data reduction algorithm. To assign values, we calculated consensus means and uncertainties using two different methods: Maximum Likelihood Estimation (MLE) [10], and Type B on Bias Estimation (BOB) [11]. The MLE method calculates a weighted mean with an uncertainty based on between-laboratory variance incorporating inter-method bias with a pooled within-laboratory variance following the ISO/NIST Guide to the Expression of Uncertainty in Measurement. The BOB method calculates an unweighted mean with an uncertainty calculated by combining a between-method variance with a pooled within-method variance following the ISO/NIST Guide to the Expression of Uncertainty in Measurement. The BOB approach is considered appropriate for combining results from a small (< 5) number of laboratories, whereas the MLE approach is useful for combining results of laboratories having dissimilar uncertainties. Both approaches apply here. In all but two cases, the consensus means calculated by the two approaches were within 0.02 ‰. For ethane and iso-butane in RM 8560, the means differed by 0.03 ‰ and 0.05 ‰, respectively. In these two cases, uncertainties in the individual laboratory results differed by over 0.2 ‰ so we elected to use the consensus mean from the MLE method.

Uncertainties calculated using the BOB method tended to be somewhat larger than those determined through the MLE method, so we elected to use the larger uncertainties whenever two laboratories reported measurements. When three laboratories reported measurements, or when the uncertainties from two laboratories differed by more than 0.2 ‰, the MLE method was used to assign uncertainties. When only one laboratory reported measurements for a particular compound, no uncertainty was assigned to the "consensus" mean; rather, these data were reported as Informational Values.

The consensus means are reported in Table 1 as NIST Reference Values, with expanded uncertainties (U) as determined through Equation 2, where u_c is the uncertainty in the consensus mean determined through the MLE or BOB methods, and u_T is the uncertainty in the value assignment of RM 8563, the NIST Reference Material that was used to establish traceability with the VPDB isotope ratio scale (see following discussion of traceability). The current value of u_T is 0.040 ‰ [4].

$$U = 2 \cdot \left[u_c^2 + u_T^2 \right]^{1/2} \qquad \text{Eq.2}$$

Value assignments may be compared with the prior data from the IAEA intercomparison exercise (see Appendix). The results here are well within the 2-sigma interval of the IAEA data, with RM values generally representing more depleted compositions. The standard uncertainties of RM consensus means are 3 to 30 times smaller than the standard deviations from the IAEA exercise.

Traceability: VPDB is the accepted "stated reference" [12] of a proper $^{13}C/^{12}C$ delta measurement traceability chain, and VPDB is realized primarily and explicitly through the limestone RM 8544 (NBS19), where:

NBS19–limestone: $\delta^{13}C_{VPDB} \equiv +1.95$ ‰, $\delta^{18}O_{VPDB} \equiv -2.20$ ‰

For non-carbonate samples, the carbon dioxide generated from NBS19–limestone, using 100 % phosphoric acid and assuming an oxygen isotope fractionation factor of 1.01025 (at 25.0 ºC), is used to realize the VPDB scale, where:

NBS19–CO_2: $\delta^{13}C_{VPDB} \equiv +1.95$ ‰, $\delta^{18}O_{VPDB} = +8.03$ ‰

It is assumed that this chemical process, when quantitative, does not affect the carbon isotope ratio. Many reference materials are available that have been compared directly with NBS19–CO_2. Here, we used the CO_2 RM 8563 and compared measurements of this RM with the measurements of the CO_2 from the oxidation of the separated carbonaceous compounds. Our $\delta^{13}C_{VPDB}$ traceability chain for each value assignment therefore contains two direct comparisons, each of which has a stated uncertainty.

SUPPLEMENTAL INFORMATION

As NGS-1, NGS-2, and NGS-3, these materials have been used by researchers for many years. A literature search has revealed a number of publications that report values for these materials. These are cited below. Such identification does not imply endorsement by the National Institute of Standards and Technology, but is offered as a convenience and resource to the users of RMs 8559-8561.

Hut, 1987 [1]: δ^{13}C, δ^{2}H, and chemical compositions – **values reprinted in Appendix**
Dumke et al., 1989 [13]: δ^{13}C and δ^{2}H
Sohns et al., 1994 [14]: δ^{15}N
Morse et al., 1996 [15]: δ^{13}C and δ^{2}H

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Users of these RMs should ensure that the report of investigation in their possession is current. This can be accomplished by contacting the SRM Program at: telephone (301) 975-6776; fax (301) 926-4751: e-mail srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.

Appendix

Prior Intercomparison of Isotopic Measurements: $\delta^{13}C$ results in ‰
Relative to VPDB and using $\delta^{13}C_{NBS19/VPDB}$=+1.95‰

Institute Code	Person Responsible	NGS1 (CH_4)	NGS1 (C_2H_6)	NGS1 (C_3H_8)
F-6	J. Baradat	-28.9	-26.4	-19.9
I-4	T. Ricchiuto	-29.16	-26.02	
NL-1	W.G. Mook	-29.16	-25.42	
USA-15	M. Beeunas/M. Schoell	-28.91	-25.93	
USA-16	P.D. Jenden	-28.97	-26.09	-21.67
USA-17	D.C. Boatwright	-28.6	-26.3	

Institute Code	Person Responsible	NGS2 (CH_4)	NGS2 (C_2H_6)	NGS2 (C_3H_8)	NGS2 (CO_2)
F-6	J. Baradat	-43.5	-31.1	-24.3	-7.5
I-4	T. Ricchiuto	-44.92	-32.1	-25.9	-8.08
NL-1	W.G. Mook	-44.76	-31.17		-8.02
USA-15	M. Beeunas/M. Schoell	-45.57	-32.06	-25.70	
USA-16	P.D. Jenden	-44.58	-31.84	-25.01	-8.38
USA-17	D.C. Boatwright	-43.1	-31.7	-23.5	-8.9

Institute Code	Person Responsible	NGS3 (CH_4)	NGS3 (C_2H_6)
F-6	J. Baradat	-73.1	-59.8
I-4	T. Ricchiuto	-72.85	
NL-1	W.G. Mook	-72.67	
USA-15	M. Beeunas/M. Schoell	-72.77	
USA-16	P.D. Jenden	-72.55	-51.37
USA-17	D.C. Boatwright	-69.6	

Values of $\delta^{13}C$ from Hut 1987 [1]

Intercomparison of Isotopic Measurements: $\delta^{2}H$ results in ‰ normalized on the VSMOW/SLAP scale

Institute Code	Person Responsible	NGS1 (CH_4)	NGS2 (CH_4)	NGS2 (C_2H_6)	NGS3 (CH_4)
I-4	T. Ricchiuto		-173		
NL-1	W.G. Mook	-133.7	-169.4	-114.5	-175.6
USA-16	P.D. Jenden	-141.9	-175.2	-128.2	-176.3

Approximate composition of NGS1, NGS2 and NGS3 in volume %

	NGS1	NGS2	NGS3
CH_4	81.238	52.775	98.825
C_2H_6	2.832	2.650	0.042
C_3H_8	0.387	1.290	0.004
i-C_4H_{10}*	0.09	0.26	0.00
n-C_4H_{10}*	0.08	0.55	0.01
n-C_5H_{12}*	0.01	0.21	0.00
n-C_5H_{12}*	0.01	0.22	0.00
n-C_6H_{14}*	0.01	0.08	0.00
O_2	0.000	0.000	0.002
N_2	14.265	16.613	1.118
CO_2	0.999	25.143	0.009

Measurements by G.J. van Rossum; N.V. Nederlandse Gasunie; Groningen; The Netherlands.
*Measurements by J. Baradat; Société Nationale Elf Aquitaine; France.

Measurements by T. Ricchiuto; Agip S.p.A. Milano, Italy; G.F. van Rossum and J. Baradat on the same components show deviations from the quoted volume percentage of the components of less than 1 %.

Values of δ^2H and Chemical composition from Hut 1987 [1]