



# Certificate of Analysis

## Certified Reference Material

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### VANA-1

#### Isotopic Certified Reference Material of vanillin

VANA-1 is a vanillin isotopic Certified Reference Material (CRM) from the National Research Council Canada (NRC). A unit of VANA-1 consists of approximately 0.75 g of vanillin. This certified reference material is intended to be used with at least one other reference material for calibrating carbon isotope delta measurements to the Vienna Pee Dee Belemnite (VPDB) scale.

The certified value for carbon isotope delta in VANA-1 was determined by combining the measurement results from three participating expert laboratories, including the NRC, using a random laboratory effects statistical model. The expanded uncertainty associated with the certified value was calculated according to the JCGM Guide [1] and corresponds to approximately 95 % confidence ( $k = 2$ ). The certified carbon isotope delta value and its expanded uncertainty is

$$-31.30 \text{ ‰} \pm 0.06 \text{ ‰}$$

expressed on the VPDB scale with a value of  $-46.6 \text{ ‰}$  assigned to LSVEC and  $+1.95 \text{ ‰}$  to NBS19 [2].

#### Certified values

Certified values are considered to be those for which the NRC has the highest confidence in accuracy and that all known and suspected sources of bias have been taken into account and are reflected in the stated expanded uncertainties. Certified values are the best estimate of the true value and uncertainty.

#### Intended use

This certified reference material is intended to be used with at least one other reference material for calibrating carbon isotope delta measurements to the VPDB scale. A minimum sample mass of 500  $\mu\text{g}$  is recommended. NRC is also providing VANB-1 CRM with a carbon isotope delta value of  $-25.85 \text{ ‰} \pm 0.05 \text{ ‰}$  on the VPDB scale [3].

#### Storage and sampling

Each CRM unit is packaged in a trilaminate foil pouch. Unopened units of VANA-1 may be stored at room temperature in a clean location out of direct sunlight. The CRM units should be opened in a clean area with precautions taken against contamination during sampling. Opened vials are recommended to be tightly capped and stored at room temperature in a dry box or desiccator out of direct sunlight.

## Preparation of material

VANA-1 was prepared from high purity vanillin. The 1 kg bottle was tumbled to homogenize the contents, sieved through approximately 600  $\mu\text{m}$  mesh, and tumbled for further homogenization before bottling. A single unit of VANA-1 contains approximately 0.75 g of vanillin.

## Stability

VANA-1 was found to be isotopically stable at room temperature, and temperatures typical of storage and transport ( $-20\text{ }^{\circ}\text{C}$  to  $+40\text{ }^{\circ}\text{C}$ ).

## Homogeneity

The homogeneity of carbon isotope delta values in VANA-1 has been assessed by analyzing 40 units of VANA-1. The uncertainty due to homogeneity was evaluated using Bayesian random effects model and was included in the uncertainty budget.

## Uncertainty

Included in the overall combined uncertainty estimate ( $u_c$ ) are the uncertainties from characterization by the NRC and two external laboratories ( $u_{\text{char}}$ ), and the uncertainty related to the between-bottle variation of isotope delta values ( $u_{\text{hom}}$ ). Expressed as standard uncertainties, these components are  $u_c = 0.03\text{ }^{\circ}\text{‰}$ ,  $u_{\text{char}} = 0.030\text{ }^{\circ}\text{‰}$ , and  $u_{\text{hom}} = 0.007\text{ }^{\circ}\text{‰}$ .

## Metrological traceability

The *Comité international des poids et mesures* (CIPM) has noted that isotope delta measurements that cannot presently be made traceable to the International System of Units (SI) should be made traceable. The carbon isotope delta value in VANA-1 is traceable to such internationally recognized reference materials [4] and serves as a suitable reference material for laboratory quality assurance programs, as outlined in ISO/IEC 17025.

Traceability of carbon isotope delta values in VANA-1 was established using internationally recognized reference materials. For uncertainty evaluation, we have revised the standard uncertainties for the five reference materials below by adding the uncertainty associated with the consistency between the reference materials,  $u = 0.029\text{ }^{\circ}\text{‰}$  [5], to their reported standard uncertainties [4,6-8]. The following certified isotope delta values and revised standard uncertainties were used:

IAEA-CH-6: $-10.450(49)\text{ }^{\circ}\text{‰}$	USGS65: $-20.290(49)\text{ }^{\circ}\text{‰}$
IAEA-600: $-27.770(49)\text{ }^{\circ}\text{‰}$	NBS 22: $-30.030(58)\text{ }^{\circ}\text{‰}$
USGS61: $-35.050(49)\text{ }^{\circ}\text{‰}$	

Additionally, IAEA-603, IAEA-610, IAEA-611, and IAEA-612 were used as calibrators. However, the isotope delta values for these materials are certified relative to the VPDB with no reference to LSVEC [9,10]. Consequently these values have to be converted to the VPDB scale as defined by NBS19 and LSVEC [11]. Thus, the following isotope delta values were used for these reference materials:

IAEA-603: $+2.474(23)\text{ }^{\circ}\text{‰}$	IAEA-610: $-9.145(19)\text{ }^{\circ}\text{‰}$
IAEA-611: $-30.925(21)\text{ }^{\circ}\text{‰}$	IAEA-612: $-36.878(26)\text{ }^{\circ}\text{‰}$

The reassessment of IAEA-603, IAEA-610, IAEA-611, and IAEA-612 did not include the aforementioned additional uncertainty due to lack of coherence [5] because this suite of materials were calibrated independent of the other internationally recognized reference materials.

## Quality Management System (ISO 17034, ISO/IEC 17025)

This material was produced in compliance with the NRC Metrology Quality Management System, which conforms to the requirements of ISO 17034 and ISO/IEC 17025. The Metrology Quality Management System supporting NRC Calibration and Measurement Capabilities, as listed in the *Bureau international des poids et mesures* (BIPM) Key Comparison Database ([kcdb.bipm.org/](http://kcdb.bipm.org/)), has been reviewed and approved under the authority of the Inter-American Metrology System (SIM) and found to be in compliance with the expectations of the CIPM Mutual Recognition Arrangement. The SIM approval is available upon request.

## Updates

For updates please refer to [doi.org/10.4224/crm.2022.vana-1](https://doi.org/10.4224/crm.2022.vana-1).

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## Cited by

A list of scientific publications citing VANA-1 can be found at [doi.org/10.4224/crm.2022.vana-1](https://doi.org/10.4224/crm.2022.vana-1).

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**This Certificate is only valid if the corresponding material was obtained directly from the NRC or an Authorized Reseller.**

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