



Certificate of Analysis

Certified Reference Material

SALT-1

Sea Salt Certified Reference Material for Nutrients in Seawater

SALT-1 is an artificial sea salt Certified Reference Material (CRM) from the National Research Council Canada (NRC) to be used as a proxy for seawater. A unit of SALT-1 consists of approximately 4.1 g of salt powder certified for phosphate, silicate and nitrate. The entire content of the SALT-1 unit should be transferred into a 100 mL plastic volumetric flask and reconstituted in nutrient free purified water. The SALT-1 reconstituted solution has typical properties of seawater and it can be used for calibration, method development, validation, and quality control for the analysis of nutrients in seawater, similar to the NRC MOOS-3.

Table 1 shows the certified values established for the SALT-1 reconstituted solution. The expanded uncertainties associated with the certified values were calculated according to the JCGM Guide [1] and correspond to approx. 95 % confidence ($k = 2$).

Table 1: Certified quantity values and expanded uncertainties ($k = 2$) for SALT-1 reconstituted in water to 100 mL solution

Analyte	Amount concentration (in the 100 mL solution) $\mu\text{mol/L}$	Mass concentration (in the 100 mL solution) mg/L	International recognition of measurement capability (CMC)
phosphate, PO_4^{3-} (a)	1.615 ± 0.030	0.1534 ± 0.0029	TEW40
silicate, as SiO_2 (a)	8.89 ± 0.31	0.534 ± 0.019	TEW41
nitrate, NO_3^- (b)	18.98 ± 0.45	1.177 ± 0.028	—

Coding

The coding refers to the instrumental method of analyte determination.

- a Matrix-matching external calibration spectrophotometry [2-3]
- b Exact-matching isotope dilution gas chromatography mass spectrometry [4]

International recognition of measurement capability

The measurement capabilities supporting these results are registered at the Calibration and Measurement Capabilities (CMC) database of the *Bureau international des poids et mesures* (BIPM) indicating recognition of the measurement certificates by National Metrology Institutes (NMIs) participating in the Mutual Recognition Arrangement (MRA) with the corresponding identifiers. List of all registered measurement capabilities in water matrix could be found in the BIPM database at <https://www.bipm.org/kcdb/>.

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

The SALT-1 reconstituted solution can be used for calibration, method development, validation, and quality control for the analysis of nutrients in seawater, similar to the NRC MOOS-3 [5]. The reconstitution yields a material commutable to seawater for the analysis of phosphate, silicate, and nitrate [6].

The SALT-1 material should be reconstituted as follows:

1. Transfer the entire content of a single CRM bottle into a plastic volumetric flask (100 ± 0.2 mL volume). No subsampling from the unit should be performed.
2. Rinse the SALT-1 bottle with nutrient free purified water (18.2 MΩ cm) and transfer it into the volumetric plastic flask.
3. Add 50-70 mL of nutrient free purified water into the volumetric plastic flask and solubilize the salt powder.
4. Bring the solution to 100 mL volume, mix it and store it in the dark at room temperature.
5. After reconstitution, phosphate and nitrate can be measured within 48 h. The dissolution of silicate requires 24 h, therefore the silicate should be measured only 24 h after reconstitution and up to 72 h. Outside these time intervals, the certified values provided in Table 1 are not valid.

The reconstituted SALT-1 solution has a pH of 9 which results in slight cloudiness. This could be prevented by adding 0.1 M HCl. However, many analytical methods use acid derivatization [3] which makes this adjustment unnecessary. Adding 1.5 mL of 0.1 M HCl during step 3 will result in a pH of 7.7-8.0, whereas adding 2.4 mL of 0.1 M HCl during step 3 will result in a pH of 7.2-7.3.

Storage

It is recommended that the material is stored at nominal temperature of +4 °C under typical refrigerator conditions. Each bottle is packaged in a trilaminate foil pouch which provide protection to atmospheric moisture. The CRM should be stored in the original pouch and opened just before use. The bottles should be opened only in a clean area with precautions taken against contamination.

Preparation of material

An artificial sea salt was spiked with a solution of nutrients and homogeneity was obtained using a high-energy planetary ball mill. The material was subsequently bottled into plastic bottles with ~4.1 g of salt powder. The mass fraction of the nutrients in the SALT-1 powder is as follows: $w(\text{phosphate, PO}_4^{3-}) = 3.744 \pm 0.049 \text{ } \mu\text{g/g}$, $w(\text{silicate as SiO}_2) = 13.04 \pm 0.42 \text{ } \mu\text{g/g}$ and $w(\text{nitrate, NO}_3^-) = 28.73 \pm 0.57 \text{ } \mu\text{g/g}$. Uncertainty is reported with a coverage factor $k = 2$.

Stability

The content of phosphate, silicate and nitrate was not affected when the material was subjected to typical transportation conditions. When SALT-1 is exposed to temperature above 4 °C, the pH of its reconstituted solutions can rise above 9, requiring pH adjustment to limit precipitation. Under long-term storage conditions, the material was proven to be stable for over three years. Monitoring of stability will continue throughout the lifecycle of the CRM.

Homogeneity

The homogeneity of SALT-1 was assessed from randomly selected units of CRMs using the analysis of variance (ANOVA).

Uncertainty

Evaluation of the uncertainty associated with certified values was carried out. Uncertainties associated with batch characterization and possible between bottle variations are included in the combined uncertainty estimate [1].

Metrological traceability

Results presented in this certificate are traceable to the International System of Units (SI) through CRMs produced by National Metrology Institutes and gravimetrically prepared standards of established purities. As such, SALT-1 serves as suitable reference material for laboratory quality assurance programs, as outlined in ISO/IEC 17025.

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/), 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 *Comité international des poids et mesures* (CIPM) Mutual Recognition Arrangement. The SIM approval is available upon request.

Updates

For updates please refer to doi.org/10.4224/crm.2022.salt-1.

References

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Cited by

A list of scientific publications citing SALT-1 CRM can be found at doi.org/10.4224/crm.2022.salt-1.

Authorship

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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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