National Research Council Canada | Conseil national de recherches Canada

Certificate of Analysis

Certified Reference Material

MOOS-3

Seawater Certified Reference Material for Nutrients

The following table lists the measurands for which certified values have been established along with their expanded uncertainty, $U_{CRM} = ku_c$, where u_c is the combined standard uncertainty calculated according to the JCGM Guide [1] and k is the coverage factor. A coverage factor of two (2) was applied which corresponds to approx. 95 % confidence. It is intended that U_{CRM} encompasses every aspect that reasonably contributes to the uncertainty in amount content.

Table 1: Certified values and expanded uncertainties (*k*=2) for MOOS-3

Nutrient	Form	Molar concentration, µmol/L	Mass concentration, mg/L	Mass fraction, mg/kg	International recognition of measurement capability (CMC)
phosphate (a,b)	P	1.60 ± 0.15	0.050 ± 0.005	0.048 ± 0.004	TEW40
	PO_4^{3-}	1.60 ± 0.15	0.152 ± 0.014	0.147 ± 0.013	
silicate (a,c,d)	Si	30.4 ± 0.7	0.85 ± 0.02	0.83 ± 0.02	TEW41
	SiO_2	30.4 ± 0.7	1.83 ± 0.04	1.77 ± 0.04	
nitrite (e)	N	3.54 ± 0.05	0.0496 ± 0.0007	0.0481 ± 0.0007	TEW39
	NO_2^-	3.54 ± 0.05	0.1630 ± 0.0024	0.1581 ± 0.0023	
nitrate (e)	N	23.0 ± 0.2	0.322 ± 0.003	0.313 ± 0.003	–
	NO_3^-	23.0 ± 0.2	1.427 ± 0.015	1.384 ± 0.015	
nitrite + nitrate (f)	N	26.6 ± 0.3	0.372 ± 0.004	0.361 ± 0.003	TEW38

Additional information

Density of MOOS-3 (at 20 °C) as determined by pycnometry: $\rho_{MOOS\text{-}3}$ = 1.0307(8) kg/L

Mass concentration of nitrite as determined by colorimetry:

$$\gamma(NO_2^-) = 0.157(15) \text{ mg/L or } \gamma(N) = 0.048(5) \text{ mg/L}$$

Standard atomic weights used:

$$A_r(N) = 14.0069(2),\ A_r(O) = 15.9994(2),\ A_r(P) = 30.973\,762(2),\ A_r(Si) = 28.0850(6)$$

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Coding

The coding refers to the instrumental method of analyte determination.

- **a** Spectrophotometry
- **b** Ion exchange chromatography inductively coupled plasma mass spectrometry (ICP-MS)
- **c** Isotope dilution ICP-MS
- **d** Inductively coupled plasma atomic emission spectrometry (ICP-AES)
- **e** Isotope dilution gas chromatography mass spectrometry
- **f** Calculated from nitrite and nitrate content

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 National Research Council Canada (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 (Table 1).

Intended use

This certified reference material is primarily intended for use in the calibration of procedures and the development of methods for the analysis of nutrients in seawater. A minimum sample amount of 3 mL is recommended.

Storage and sampling

This material should be stored in the original bottle in a refrigerator (at nominal temperature of +4 °C) and dark location, although samples stored at +37 °C for two week periods have not undergone detectable changes. Material shall not be frozen.

Once opened, contamination may induce changes in the measurands. Care should be taken if it is intended to remove only a portion of the sample and store the remainder for use at a later date. The bottle should be opened for the minimal period in a clean area and otherwise remain tightly closed. Please note that the CRM unit may contain small amount of debris of biological or inorganic origin. The presence of such material does not affect the validity of the certified values.

Preparation of material

MOOS-3 was collected at latitude 47.062833 °N and longitude 59.982333 °W, off the northern tip of Cape Breton Island, Nova Scotia (Canada). The water was sampled from a depth of about 200 m using a rosette containing 22 Niskins, each of about 10 L volume. The contents of each Niskin were transferred, using a peristaltic pump, through a 0.05 µm cartridge filter into 50 L carboys. The water was collected 24 June 1996, irradiated 16 July 1996, and stored in a cold room at NRC. On December 2011, the water was homogenized and bottled.

Analytical methods

Methods based on traditional colorimetric procedures were used for phosphate and silicate. Independent methods based on ion exclusion chromatography inductively coupled plasma mass spectrometry for silicate (isotope dilution) and phosphate were developed at NRC to augment the colorimetric results. Silicate was also determined using ICP atomic emission spectrometry. Isotope dilution gas chromatography mass spectrometry method was developed at NRC for nitrite and nitrate [2]. All measurements contributing to this certificate were conducted by Chemical Metrology personnel at the NRC in Ottawa.

Stability

The predecessor CRM, MOOS-2, has been periodically analyzed for more than ten years and found to be both physically and chemically stable over this time interval. We expect similar results for MOOS-3. Uncertainty components for long and short term stability were considered negligible and are thus not included in the uncertainty budget.

Homogeneity

The material was tested for homogeneity at NRC. Results from sub-samples were evaluated using ANOVA and included in the calculation of the certified values.

Uncertainty

Included in the overall combined uncertainty estimate (u_c) are uncertainties in the batch characterization (u_{char}) and uncertainties related to possible between-bottle variation (u_{hom}). Expressed as standard uncertainties relative to the combined uncertainty, these components are listed in Table 2.

Table 2: Relative uncertainty components for MOOS-3

Nutrient	u_c, relative	u_{char}, relative	u_{hom}, relative
phosphate (PO_4^{3-})	100%	66%	34%
silicate (SiO_3^{2-})	100%	94%	6%
nitrite (NO_2^-)	100%	95%	5%
nitrate (NO_3^-)	100%	88%	12%

Metrological traceability

Results presented in this certificate are traceable to the SI through gravimetrically prepared standards of established purity, CRMs and international measurement intercomparisons. As such, they serve as suitable reference materials 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 (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 *Comité international des poids et mesures* (CIPM) Mutual Recognition Arrangement. The SIM approval is available upon request.

Updates

Users should ensure that the certificate they have is current. Our website at www.nrc.gc.ca/crm will contain any new information.

References

1. Evaluation of measurement data – Guide to the expression of uncertainty in measurement JCGM 100:2008.
2. Pagliano E, Meija J, Sturgeon RE, Mester Z, D'Ulivo A. Negative chemical ionization GC/MS determination of nitrite and nitrate in seawater using exact matching double spike isotope dilution and derivatization with triethyloxonium tetrafluoroborate. *Anal Chem.* 2012, 84, 2592-2596.

Authorship

The following NRC staff members contributed to the production and certification of MOOS-3: V.P. Clancy, I.P. Gedara, P. Grinberg, J. Meija, Z. Mester, E. Pagliano, S. Willie and L. Yang.

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MOOS-3

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Approved by: _____

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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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