

# Standard Reference Material<sup>®</sup> 3669

## Arsenic Species in Frozen Human Urine (Elevated Levels)

### CERTIFICATE OF ANALYSIS

**Purpose:** This Standard Reference Material (SRM) is intended primarily for validating analytical methods and measurements for the determination of arsenic species in human urine.

**Description:** A unit of SRM 3669 consists of five pouches each containing a vial of arsenic species in frozen human urine at elevated levels. Each vial contains nominally 1.5 mL of urine.

The development of SRM 3669 was a collaboration between NIST and the Centers for Disease Control and Prevention (CDC), National Centers for Environmental Health, Division of Laboratory Sciences (Atlanta, GA).

**Certified Values:** Table 1 lists the certified values and expanded uncertainties for arsenic species in SRM 3669. The structural formulas of the arsenic species are shown in the Appendix. A NIST certified value is a value for which NIST has the highest confidence in its accuracy in that all known or suspected sources of bias have been investigated or taken into account [1].

The reported certified values are the weighted means of the individual sets of measurements made by NIST and CDC, estimated using a Gaussian random effects model [2] and the DerSimonian-Laird procedure [3,4]. The associated measurement uncertainty was evaluated by the application of the parametric statistical bootstrap, consistent with the ISO/JCGM Guides and its Supplement 1 [5–7]. The expanded uncertainty,  $U$ , is calculated as  $U = ku_c$ , where  $u_c$  represents, at the level of one standard deviation, the combined effects of between-laboratory, within-laboratory, and inhomogeneity components of uncertainty. The coverage factor,  $k$ , corresponds to an approximately 95 % level of confidence.

Table 1. Certified Values for Arsenic and Arsenic Species in SRM 3669<sup>(a)</sup>

Species	Arsenic ( $\mu\text{g/L}$ )	$k$ -factor
Arsenous acid (AsIII)	14.28 $\pm$ 0.65	1.99
Arsenic acid (AsV)	17.8 $\pm$ 1.0	2.00
Monomethylarsonic acid (MMA)	21.6 $\pm$ 1.0	1.98
Dimethylarsinic acid (DMA)	77.1 $\pm$ 1.6	2.00
Arsenobetaine (AB)	51.0 $\pm$ 2.2	1.99
Arsenic (As)	185.1 $\pm$ 9.9	1.98

<sup>(a)</sup> The measurand is the total concentration of arsenic or each arsenic species listed, and the certified value is metrologically traceable to the SI derived unit for mass concentration (expressed as micrograms per liter).

**Additional Information:** Additional information is provided in Appendix A.

**Period of Validity:** The certified values delivered by **SRM 3669** are valid within the measurement uncertainty specified until **31 March 2034**. The certified values are nullified if the material is stored or used improperly, damaged, contaminated, or otherwise modified.

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**Maintenance of Certified Values:** NIST will monitor this SRM over the period of its validity. If substantive technical changes occur that affect the certification, NIST will issue an amended certificate through the NIST SRM website (<https://www.nist.gov/srm>) and notify registered users. SRM users can register online from a link available on the NIST SRM website or fill out the user registration form that is supplied with the SRM. Registration will facilitate notification. Before making use of any of the values delivered by this material, users should verify they have the most recent version of this documentation, available through the NIST SRM website (<https://www.nist.gov/srm>).

**Safety:** SRM 3669 IS INTENDED FOR RESEARCH USE. This is a human-source material (human urine). SRM 3669 is a Biosafety Level 2 material and should be handled according to applicable federal, state, and/or local regulations and according to policies and procedures of recipient's organization.

**Storage:** The SRM should be stored at  $-80\text{ }^{\circ}\text{C}$  in the original unopened package. The certification does not apply to contents of previously opened pouches as the stability of all species has not been investigated under such conditions.

**Use:** Unopened pouches of SRM 3669 should be thawed at room temperature. The material should be used within 4 h after being thawed. Unused or remaining material should be discarded after the specified time. Once the pouches are cut open, each vial of the SRM should be homogenized by gently inverting the vial several times before a test portion is removed.

To determine arsenic species in SRM 3669, particulates in the subsample should be removed. Recommended procedures for removal of particulates are: (1) extracting supernatant after centrifuging at  $2 \times 10^4\text{ }g_n$  for 5 min, or (2) filtration using a  $0.45\text{ }\mu\text{m}$  syringe filter. The recommended minimum sample size for speciation measurement is 0.2 mL.

To determine the total arsenic in SRM 3669, the entire subsample, including particulates, should be used. The recommended minimum sample size for total arsenic measurement is 1 mL.

## REFERENCES

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*Certain commercial equipment, instruments, or materials may be identified in this Certificate of Analysis to adequately specify the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.*

*Users of this SRM should ensure that the Certificate of Analysis in their possession is current. This can be accomplished by contacting the Office of Reference Materials 100 Bureau Drive, Stop 2300, Gaithersburg, MD 20899-2300; telephone (301) 975-2200; e-mail [srminfo@nist.gov](mailto:srminfo@nist.gov); or the Internet at <https://www.nist.gov/srm>.*

**\* \* \* \* \* End of Certificate of Analysis \* \* \* \* \***



Table A2. Methods of Analysis for SRM 3669

Analyte	Methods <sup>(a)</sup>	Laboratory
Arsenous acid (AsIII)	Anion exchange LC – (H <sub>2</sub> dynamic reaction) ICPMS	CDC [9]
	Cation exchange LC – ICPMS	NIST [10]
	Anion exchange IC – (H <sub>2</sub> /He collision) ICPMS	NIST [10]
Arsenic acid (AsV)	Anion exchange LC – (H <sub>2</sub> dynamic reaction) ICPMS	CDC [9]
	Anion exchange LC – ICPMS	NIST [10]
	Anion exchange IC – (H <sub>2</sub> /He collision) ICPMS	NIST [10]
Monomethylarsonic acid (MMA)	Anion exchange LC – (H <sub>2</sub> dynamic reaction) ICPMS	CDC [9]
	Cation exchange LC – ICPMS	NIST [10]
	Anion exchange IC – (H <sub>2</sub> /He collision) ICPMS	NIST [10]
Dimethylarsinic acid (DMA)	Anion exchange LC – (H <sub>2</sub> dynamic reaction) ICPMS	CDC [9]
	Cation exchange LC – ICPMS	NIST [10]
	Anion exchange IC – (H <sub>2</sub> /He collision) ICPMS	NIST [10]
Arsenobetaine (AB)	Anion exchange LC – (H <sub>2</sub> dynamic reaction) ICPMS	CDC [9]
	Cation exchange LC – ICPMS	NIST [10]
	Anion exchange IC – (H <sub>2</sub> /He collision) ICPMS	NIST [10]
Total arsenic	Radiochemical neutron activation analysis	NIST [11]
	(H <sub>2</sub> /He collision) ICPMS after complete digestion of urine	NIST [12]

<sup>(a)</sup> LC: Liquid Chromatography; IC: Ion Chromatography; ICPMS: Inductively Coupled Plasma Mass Spectrometry.

Coordination of the technical measurements leading to the certification was under the direction of L.L. Yu of the NIST Chemical Sciences Division.

Analytical measurements for certification of this SRM were performed by W.C. Davis, R.L. Paul, and L.L. Yu of the NIST Chemical Sciences Division; N.D. Hilliard and C.D. Ward of the CDC Inorganic and Radiation Analytical Toxicology Branch.

Partial support for the development of this SRM was provided under the direction of R.L. Jones of the CDC Inorganic and Radiation Analytical Toxicology Branch.

Statistical consultation for this SRM was provided by D.D. Leber of the NIST Statistical Engineering Division.

\*\*\*\*\* End of Appendix A \*\*\*\*\*