National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material 2670

Toxic Metals in Freeze-Dried Urine

This Standard Reference Material (SRM) is intended primarily for use in the determination of toxic metals in human urine. It may also be used in the determination of metals that are of biological interest in other matrices. SRM 2670 consists of four bottles of freeze-dried urine, two bottles each at low and elevated levels. The low level is normal human urine that was prepared from pooled samples. The pooled normal urine is not intended to represent a urine pool from normal human subjects. It is intended to provide a urine matrix that contains low concentration levels of trace metals. The elevated level is normal human urine that was spiked with selected metals. The normal and elevated levels are provided to permit construction of a typical 2-point calibration curve.

Certified Values of Constituents: The certified values are given in Table 1 and are based upon the analysis of samples, randomly selected from the lot, by methods that were selected to provide the most accurate values. These certified values apply only to the reconstituted urine (see section under "Use" for procedures for reconstituting). Noncertified values (in parentheses) are also given in Table 1 as additional information on the matrix. The noncertified values are for information only. Zinc, an important element, could not be certified in SRM 2670 due to contamination from the stopper used in the packaging. On reconstituted samples, the zinc concentration varied from 0.5 to 1.5 μg/mL. When SRM 2670 is renewed, an appropriate packaging material will be selected so that this important element can be certified.

Use: This SRM should be reconstituted by the addition of 20.0 mL of pure water to each bottle. If the water contains quantities of the analytes to be measured at levels which interfere with the analytical measurements, appropriate blank corrections should be made. The reconstituted material may be considered as fresh urine and should be handled under the same conditions as fresh urine. When reconstituted as recommended, the specific gravity of SRM 2670 is 1.0117 $\pm$ 0.0001 g/mL at 23 °C.

Storage and Stability: SRM 2670, unreconstituted, should be stored at a temperature between 2 and 8 °C. If properly stored it is expected to be stable for at least one year from date of purchase. The stability of the reconstituted material has not been rigorously assessed, therefore the reconstituted SRM should be used immediately. NIST will continue to monitor this SRM and, if evidence indicates a change in any of the certified values, purchasers will be notified. Please return the attached registration form to facilitate notification.

Statistical consultation was provided by K. Eberhardt of the Statistical Engineering Division.

The overall direction and coordination of the technical measurements leading to the certification of this SRM were performed under the chairmanship of B.I. Diamondstone of the Inorganic Analytical Research Division.

The technical and support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the Standard Reference Materials Program by T.E. Gills.

Gaithersburg, MD 20899
August 29, 1994
(Revision of certificate dated 4-23-93)

Thomas E. Gills, Chief
Standard Reference Materials Program

(over)

Source and Preparation of Material: The base material for SRM 2670 was procured and processed under a contract with Technimed Corp., Fort Lauderdale, FL (formerly Leon Laboratories, a Division of Leon Industries, Inc., St. Louis, MO). All processes were conducted under sterile conditions and continuously monitored to ensure sample consistency. Two lots of normal urine were collected (pooled into polyethylene tanks), filtered and homogenized. One lot was spiked with selected metals to form the elevated level.

The processed urine was pipetted into serum bottles, 20 mL units, and freeze-dried. To verify precise filling, random samples taken during the bottling process were weighed. The variability (2 sigma) of the weighed samples was less than 1% relative.

Homogeneity Assessment: Randomly selected bottles of SRM 2670 were sampled and tested for homogeneity. After allowing for measurement imprecision, the apparent inhomogeneity of all elements was less than 1% (relative standard deviation). The stated uncertainties include an allowance of $\pm$ 3% relative for possible bottle-to-bottle variation of the concentrations in reconstituted urine.

Table 1. Certified Values of Constituent Elements in SRM 2670

Note: The values in parentheses are not certified because they are not based on the results of either a reference method of known accuracy or two or more independent methods. They are included for information only.

Element	Conc. Units	Normal Level	Elevated Level	Analytical Methods Used
Aluminum	μg/mL	(0.18)	(0.18)*	a
Arsenic	μg/mL	(0.06)	0.48 ± 0.10	a,g
Beryllium	μg/mL	(≤0.0005)	(0.033)	a
Cadmium	μg/mL	(0.00040)	0.088 ± 0.003	a,f
Calcium	mg/mL	0.105 ± 0.005	0.105 ± 0.005*	c,h
Chloride	μg/mL	(4.4)	(4.4)*	e
Chromium	μg/mL	(0.013)	0.085 ± 0.006	a,f
Copper	μg/mL	0.13 ± 0.02	0.37 ± 0.03	a,h
Gold	μg/mL	(0.008 ng/mL)	(0.24)	j
Lead	μg/mL	(0.01)	0.109 ± 0.004	f
Magnesium	mg/mL	0.063 ± 0.003	0.063 ± 0.003*	b,d
Manganese	μg/mL	(0.03)	(0.33)	b,h
Mercury	μg/mL	(0.002)	0.105 ± 0.008	a,g
Nickel	μg/mL	(0.07)	(0.30)	h
Platinum	μg/mL	(0.008 ng/mL)	(0.12)	a,j
Potassium	mg/mL	(1.5)	(1.5)*	c,h
Selenium	μg/mL	0.030 ± 0.008	0.46 ± 0.03	a,i
Sodium	mg/mL	2.62 ± 0.14	2.62 ± 0.14	c,d
Sulfate	mg/mL	(1.3)	(1.3)*	e
Vanadium	μg/mL	--	(0.12)	f

*These levels are not spiked, but are endogenous to the matrix.

The uncertainties of the certified values are the statistical tolerance intervals at the 95% confidence level for coverage of 99% of the samples of SRM 2670. With 95% confidence, the analyte concentrations in at least 99% of the samples of this SRM should be included in the listed interval.[1]

[1]The concept of tolerance limits is discussed in Chapter 2, Experimental Statistics, NBS Handbook 91, 1966, and page 14, The Role of Standard Reference Materials in Measurement Systems, NBS Monograph, 148, 1975.

Analytical Methods

a. Electrothermal Atomization Atomic Absorption Spectrometry
b. Flame Atomic Absorption Spectrometry
c. Flame Emission Spectrometry
d. Inductively Coupled Plasma Spectrometry
e. Ion Chromatography
f. Isotope Dilution Thermal Source Mass Spectrometry
g. Instrumental Neutron Activation Analysis
h. DC Plasma Emission Spectrometry
i. Isotope Dilution Spark Source Mass Spectrometry
j. Radiochemical Neutron Activation Analysis

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