National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material 1598

Inorganic Constituents in Bovine Serum

This Standard Reference Material (SRM) is intended primarily for use in calibrating instrumentation and evaluating the accuracy of analytical methods for selected elements in blood serum, plasma, and similar biological fluids. SRM 1598 consists of two capped polypropylene vials, each containing 5 to 6 mL of bovine serum.

Certified and Noncertified Concentrations of Constituent Elements: The certified concentrations of the constituent elements are shown in Table 1. These concentrations are based on the agreement of results by at least two independent analytical methods. Noncertified concentrations, which are given for information only, appear in Table 2. Methods used for the determination and certification of the certified elements are shown in Table 3.

Notice and Warnings to Users:

Expiration of Certification: This certification will be invalid after one year from the date of shipment. Should it be invalidated before then, users will be notified by National Institute of Standards & Technology (NIST). Please return the attached registration card to facilitate notification.

Storage: The material should be stored in its original containers at -20 °C or below. A "frost-free" type freezer should not be used. SRM 1598 should not be exposed to intense sources of radiation, including ultraviolet lamps or sunlight. For long term storage, we recommend sealing in a plastic bag containing ice cubes to maintain 100% humidity.

Use: The frozen serum should be thawed at room temperature and mixed thoroughly by inverting the vial several times before use. Vials should be opened only in a clean area with precautions taken against contamination during sampling. We recommend weighing rather than pipetting samples for analysis because of the viscous nature of the serum. The minimum recommended sample size is 250 mg (see section on Homogeneity).

SRM 1598 IS INTENDED FOR "IN VITRO" DIAGNOSTIC USE ONLY!

Coordination of the technical measurements by members of NIST Inorganic Analytical Research Division was performed by H.M. Kingston of that Division; coordination of the technical measurements by cooperating analysts was performed by C. Veillon of the U.S. Department of Agriculture, Beltsville, MD.

Statistical analysis of the experimental data was performed by S.B. Schiller of NIST Statistical Engineering Division.

The technical and support aspects involved in the certification and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by Robert Alvarez.

Gaithersburg, MD 20899
January 31, 1990
(Revision of certificate dated 1-10-89)

William P. Reed, Acting Chief
Office of Standard Reference Materials

(over)

Table 1. Certified Concentrations of Constitutent Elements

Element[a]	Concentration, µg/g[b]
Copper	0.72 ± 0.04
Iron	2.55 ± 0.10
Magnesium	20.0 ± 0.4
Potassiumc	196 ± 5
Rubidium	0.17 ± 0.02
Zinc	0.89 ± 0.06

Element[a]	Concentration, ng/g[b]
Aluminum	3.7 ± 0.9
Cadmium	0.089 ± 0.016
Chromium	0.14 ± 0.08
Cobalt	1.24 ± 0.18
Manganese	3.78 ± 0.32
Molybdenum	11.5 ± 1.1
Selenium	42.4 ± 3.5

a Methods used for the determination and certification of each element are shown in Table 3.
b The certified concentrations are equally weighted means of results from two or more analytical methods. To convert concentrations from µg/g or ng/g, to µg/mL or ng/mL, respectively, multiply by the density of the serum, 1.029 g/mL at 23 °C.
c The stated uncertainty of the K concentration is based on results obtained in three laboratories by two different methods, neither of which is either the reference method or the definitive method as defined by the National Committee for Clinical Laboratory Standards. (See table 3). K is certified with a smaller uncertainty in freeze-dried Human Serum, SRM 909, based on the "definitive method" isotope dilution mass spectrometry.

The stated uncertainty includes allowances for measurement imprecision, material inhomogeneity, and differences among analytical methods. Each uncertainty is computed from a 95% prediction interval and an allowance for systematic error among the methods used, combined as the square root of the sum of their squares. In the absence of systematic error, the prediction intervals used have the following statistical property: 95% is the statistical expectation of the percentage of all samples of this SRM having concentrations within the stated uncertainty limits.

Table 2. Noncertified Concentrations of Constituent Elements

Elements other than those certified are present in this material. Those that were determined but not certified are given as additional information on the composition.

Element	Concentration, mg/g	Element	Concentration, ng/g
Calcium	(0.09)	Arsenic	(0.2)
Sodium	(3)	Cesium	(0.1)
		Lead	(0.6)
		Mercury	(0.2)
		Nickel	(0.7)
		Thallium	(0.4)
		Vanadium	(0.06)

Analysts, NIST Center for Analytical Chemistry
- Inorganic Analytical Research Division

1. E.S. Beary
2. T.A. Butler
3. M.S. Epstein
4. R.R. Greenberg
5. H.M. Kingston
6. P.J. Paulsen
*7. P.A. Pella
8. K.W. Pratt
9. T.C. Rains
10. T.M. Sullivan
11. G.C. Turk
12. L.J. Yu (Guest Scientist)
13. R. Zeisler

* Gas and Particulate Science Division

Cooperating Analysts

14. N.J. Miller-Ihli and S.A. Lewis, Beltsville Human Nutrition Research Center, U.S. Department of Agriculture, Beltsville, MD.

15. E. Pruszkowski, ICP-Mass Spectrometry Applications Laboratory, Perkin-Elmer Corporation, Norwalk, CT.

16. M. Stoeppler, Nuclear Research Institute, Julich, Federal Republic of Germany.

17. C. Veillon, and K. Patterson, Beltsville Human Nutrition Research Center, U.S. Department of Agriculture, Beltsville, MD.

18. J. Versieck, L. Vanballenberghe, and G.A. DeKesel, Department of Internal Medicine, Division of Gastroenterology, University Hospital, Ghent, Belgium; and Laboratory for Analytical Chemistry, University Institute for Nuclear Sciences, Ghent, Belgium.

Analytical Methods and Codes Used for the Determination of Certified Elements (See Table 3)

Code	Method
	Atomic Absorption Spectrometry
EAAS	Electrothermal
FAAS	Flame
HAAS	Hydride Generation
	Atomic Emission Spectrometry
DCPAES	Direct Current Plasma
FAES	Flame
	Electrochemical
DPASV	Differential Pulse Anodic Stripping Voltametry
POLAR	Polarography
	Ionization Spectrometry
LEIS	Laser-Enhanced (Chelation Separation)
	Mass Spectrometry
IDGCMS	Gas Chromatography, Quadrupole, Isotope Dilution
ICPMS	Inductively Coupled Plasma
IDICPMS	Inductively Coupled Plasma, Isotope Dilution
	Neutron Activation Analysis
PNAA	Preirradiation Chelation Separation
INAA	Instrumental
RNAA	Radiochemical

Preparation of Material: The bovine serum was prepared under the direction of C. Veillon, Beltsville Human Nutrition Research Center, U.S. Department of Agriculture, Beltsville, MD., as described previously (1,2).

In this procedure, blood was collected from four unconscious dairy cows under carefully controlled conditions to minimize contamination. The blood was centrifuged and the separated serum was combined in a single container and mixed continuously while aliquoting into individual polypropylene vials (5-6 mL/vial). The vials were capped, labeled, bagged with ice cubes to maintain 100% humidity, and stored at -20 °C. Vials were selected at random for analysis.

All operations, except the initial collection and centrifugation in sealed bottles, were carried out in a Class-100 area of a clean room. Prior to filling with serum, the vials had been soaked for two weeks in 5% nitric acid,

rinsed with deionized water, and dried by lyophilization in a plastic chamber known to be free of trace element contamination.

Homogeneity: Homogeneity of SRM 1598 was examined as part of the analytical program for certification by analyzing randomly selected vials. For the determination of Co and Se by electrothermal atomic absorption spectrometry, approximately 200 μg was used. No evidence of material inhomogeneity was found for these determinations or for other elemental determinations where larger samples were used. A minimum sample size of 250 mg is recommended.

References

(1.) Veillon, C.; Patterson, K.Y.; and Reamer, D.C. "Biological Reference Materials", Wolf, W.R. Ed.; Wiley, New York, 1985.

(2.) Veillon, C.; Lewis, S.A.; Patterson, K.Y.; Wolf, W.R.; Harnly, J.M.; Versieck, J.; Vanballenberghe, L.; Cornelis, R.; and O'Haver, T.C. Characterization of a Bovine Serum Reference Material for Major, Minor, and Trace Elements. Anal. Chem. **57**: 2106-2109; 1985.

Table 3. Determination of Certified Elements --Analytical Methods and Analysts

	Al	Cd	Co	Cr	Cu	Fe	K	Mg	Mn	Mo	Rb	*Se	Zn
Method													
EAAS	2,9. 12,14	16	2,9, 12	14,17					14	14		2,9, 12	
FAAS					14	14		14					14
HAAS												16	
DCPAES								3					
FAES							3,14				2,9, 12		
DPASV		16			16								16
POLAR													8
LEIS									5,11				
IDGCMS				17								17	
ICPMS							15						
IDICPMS								1,6		1,6			
PNAA	4,5				4,5				4,5				
INAA			13,18		18	13,18			18		13,18	13,18	13,18
RNAA		4,10		4,13	4,10					4,10,18			

* Single value (42 ng/g) by total reflection x-ray fluorescence spectrometry (P.A. Pella) agreed with results by other methods.