National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 1549

Non-Fat Milk Powder

This Standard Reference Material (SRM) is intended primarily for use in calibrating instrumentation and evaluating the reliability of analytical methods for the determination of constituents in milk, milk powders, and other biological matrices. A unit of SRM 1549 consists of 100 g of material.

Certified Values of Constituents: The certified concentrations of the constituent elements are shown in Table 1. Certified values are based on results obtained by definitive methods of known accuracy; or alternatively, from concordant results by two or more independent analytical methods [1].

Information Concentration Values: Information concentration values for additional constituent elements are provided in Table 2. Information values for lactose are provided in Table 3. These are non-certified values with no reported uncertainties as there is insufficient information to assess uncertainties [1]. The information values are given to provide additional characterization of the material. The non-certified concentrations of lactose and ascorbic acid were determined by high performance liquid chromatography (HPLC); and for lactose only, by nuclear magnetic resonance (NMR). Use of this SRM to quantitatively monitor method performance for analytes other than those with certified or reference concentration values in Tables 1 is not warranted.

Expiration of Certification: The certification of **SRM 1549** is valid, within the measurement uncertainty specified, until **25 January 2013**, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see "Instructions for Use"). The certification is nullified if the SRM is damaged, contaminated, or otherwise modified.

Maintenance of SRM Certification: NIST will monitor this SRM over the period of its certification. If substantive technical changes occur that affect the certified values before the expiration of this certificate, NIST will notify the purchaser. Registration (see attached sheet) will facilitate notification.

The overall direction and coordination of the analyses were under the chairmanship of E.L. Garner, Chief of the Inorganic Analytical Research Division and W.E. May, Chief of the Organic Analytical Research Division.

Analytical measurements at NIST were performed by E.S. Beary; J. Brown Thomas; T.A. Butler; B. Coxon; M.S. Epstein; J.D. Fasett; J.W. Gramlich; R.R. Greenberg; W.R. Kelly; H.M. Kingston; W.F. Koch; G.M. Lambert; G.J. Lutz; J.R. Moody; T.J. Murphy; P.J. Paulsen; T.C. Rains; T.A. Rush; M.E. Watson; R.L. Watters, Jr.; and L. Watts.

Additional elemental analyses were performed by R.W. Dabeka, Food Research Division, Health Protection Branch, Tunney's Pasture, Ottawa, Ontario, Canada.; L. Kosta, A.R. Byrne, M. Dermelj, Institute "Jôsef Stefan", Ljubljana, Yugoslavia; and C. Vernon and K. Patterson, Beltsville Human Nutrition Research Center, U.S. Department of Agriculture, Beltsville, MD.

Statistical consultation was provided by L.R. Eberhardt of the NIST Statistical Engineering Division.

Support aspects involved in the issuance of this SRM were coordinated through the NIST Measurement Services Division.

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Gaithersburg, MD 20899
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Robert L. Watters, Jr., Chief
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INSTRUCTIONS FOR USE

Stability: The material should be kept in its original bottle and stored at temperatures between 10 °C and 30 °C. It should not be exposed to intense sources of radiation. The bottle should be kept tightly closed and stored in a desiccator in the dark.

SRM Preparation: A minimum sample of 500 mg of the dried material (see "Instructions for Drying") should be used for any analytical determination to be related to the certified values of this certificate. Dissolution procedures should be designed to effect complete dissolution, but without losses of volatile elements, such as mercury. Dissolution for these determinations should be carried out in a closed system.

Instructions for Drying: Samples of this SRM must be dried before weighing according to the following procedure: dry for 48 h at 20 °C to 25 °C in a vacuum oven at a pressure not greater than 30 Pa (0.2 mmHg).

Table 1. Certified Values of Constituent Elements[a,b]

Element	Concentration Mass Fraction (%)	Element	Concentration Mass Fraction (%)
Calcium	1.30 ± 0.05	Potassium	1.69 ± 0.03
Chlorine	1.09 ± 0.02	Sodium	0.497 ± 0.010
Magnesium	0.120 ± 0.003	Sulfur	0.351 ± 0.005
Phosphorus	1.06 ± 0.02		

Element	Concentration Mass Fraction (mg/kg)	Element	Concentration Mass Fraction (mg/kg)
Cadmium	0.0005 ± 0.0002	Lead	0.019 ± 0.003
Chromium	0.0026 ± 0.0007	Manganese	0.26 ± 0.06
Copper	0.7 ± 0.1	Mercury	0.0003 ± 0.0002
Iodine	3.38 ± 0.02	Selenium	0.11 ± 0.01
Iron	1.78 ± 0.10	Zinc	46.1 ± 2.2

[a] Analytical values are based on the "dry-weight" of material (see "Instructions for Drying").

[b] Except for Fe, the stated uncertainty includes the union of 95 % confidence intervals computed separately for each analytical method. It includes the effects of measurement error, possible effects of known systematic errors, and between-method differences. The uncertainty for Fe is given as a 95 % confidence interval for the weighted mean of the mass spectrometric and neutron activation values, and includes an allowance (added linearly) for systematic error in the methods. The weights were chosen to minimize the estimated mean squared error of the weighted mean [2].

Table 2. Information Values of Constituent Elements

Element	Concentration (mg/kg)	Element	Concentration (mg/kg)
Aluminum	2	Molybdenum	0.34
Antimony	0.00027	Rubidium	11
Arsenic	0.0019	Silicon	<50
Bromine	12	Silver	<0.0003
Cobalt	0.0041	Tin	<0.02
Fluorine	0.20		

Table 3. Information Values of Lactose

Compound	Number of Determinations	Concentration Mass Fraction (%)	Method
Lactose	5	49	HPLC
	5	45	Proton NMR

REFERENCES

[1] May, W.; Parris, R.; Beck II, C.; Fassett, J.; Greenberg, R.; Guenther, F.; Kramer, G.; Wise, S.; Gills, T.; Colbert, J.; Gettings, R.; MacDonald, B.; *Definition of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurements*; NIST Special Publication 260-136; U.S. Government Printing Office: Washington, DC (2000); available at http://www.nist.gov/srm/upload/SP260-136.PDF (accessed Oct 2012).

[2] Sacks, J; Ylvisaker, D.; *Approximately Linear Models*; Annals of Statistics, Vol. 6, pp. 1122–1137 (1978).

Certificate Revision History: 02 October 2012 (Editorial changes); 23 February 2009 (Removed information value for ascorbic acid); 20 May 2003 (Expiration period extended and editorial changes); 29 July 1985 (Certified value for iron added); 14 January 1985 (Certified value for phosphorus added); 17 April 1984 (Original certificate date).

APPENDIX A

Methods Used in Elemental Determinations

Element	Method Code[a]
Cadmium	ETAAS, RNAA
Calcium	ICP-OES, INAA
Chlorine	IC, INAA
Chromium	ID-EIMS, RNAA
Copper	ETAAS, DCP-OES, RNAA
Iodine	IDTIMS, IPAA
Iron	IDTIMS, RNAA
Lead	ETAAS, IDTIMS
Magnesium	ICP-OES
Manganese	ETAAS, DCP-OES, INAA
Mercury	CVAAS, RNAA
Phosphorus	DCP-OES, ICP-OES
Potassium	FES, INAA
Selenium	HGAAS, ID-SSMS, INAA, RNAA
Sodium	ICP-OES, INAA
Sulfur	IC, IDTIMS
Zinc	FAAS, ICP-OES, ID-SSMS, INAA

[a] **Acronyms for Analytical Methods:**

CVAAS	Cold-Vapor Atomic Absorption Spectrometry
DCP-OES	Direct Current Plasma Optical Emission Spectrometry
ETAAS	Electrothermal Atomic Absorption Spectrometry
FES	Flame Emission Spectrometry
FAAS	Flame Atomic Absorption Spectrometry
HGAAS	Hydride Generation Atomic Absorption Spectrometry
HPLC	High Pressure (Performance) Liquid Chromatography
IC	Ion Chromatography
ICP-OES	Inductively Coupled Plasma Optical Emission Spectrometry
IDTIMS	Isotope Dilution, Thermal Ionization Mass Spectrometry
ID-SSMS	Isotope Dilution Spark Source Mass Spectrometry
ID-EIMS	Isotope Dilution Electron Impact Mass Spectrometry
INAA	Instrumental Neutron Activation Analysis
IPAA	Instrumental Photon Activity analysis
Proton NMR	Proton Nuclear Magnetic Resonance
RNAA	Radiochemical Neutron Activation Analysis