

National Bureau of Standards

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.

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.

Additional Information on Composition: Noncertified concentrations of additional constituent elements are given for information only in Table 2. Noncertified concentrations of lactose and ascorbic acid were determined by high performance liquid chromatography; and for lactose only, by nuclear magnetic resonance.

Notice and Warnings to Users:

Expiration of Certification: This certification is invalid after 3 years from the date of shipping. Should it become invalid before then, purchasers will be notified by NBS.

Stability: The material should be kept in its original bottle and stored at temperatures between 10-30 °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.

Use: 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.

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

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.

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

Gaithersburg, MD 20899
July 29, 1985
(Revision of Certificates
dated 4-17-84 and
1-14-85)

(over)

Stanley D. Rasberry, Chief
Office of Standard Reference Materials

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

Analysts:

Center for Analytical Chemistry, National Bureau of Standards:

- | | | |
|----------------------|-------------------|-----------------------|
| 1. E.S. Beary | 8. R.R. Greenberg | 15. T.J. Murphy |
| 2. J.M. Brown-Thomas | 9. W.R. Kelly | 16. P.J. Paulsen |
| 3. T.A. Butler | 10. H.M. Kingston | 17. T.C. Rains |
| 4. B. Coxon | 11. W.F. Koch | 18. T.A. Rush |
| 5. M.S. Epstein | 12. G.M. Lambert | 19. M.E. Watson |
| 6. J.D. Fassett | 13. G.J. Lutz | 20. R.L. Watters, Jr. |
| 7. J.W. Gramlich | 14. J.R. Moody | 21. L. Watts |

Cooperating Analysts:

22. R.W. Dabeka, Food Research Division, Health Protection Branch, Tunney's Pasture, Ottawa, Ontario, Canada.
23. L. Kosta, A.R. Byrne, M. Dermelj, Institute "Josef Stefan", Ljubljana, Yugoslavia.
24. C. Veillon and K. Patterson, Beltsville Human Nutrition Research Center, U.S. Department of Agriculture, Beltsville, MD.

Table 1. Certified Concentrations of Constituent Elements

| <u>Element</u> | <u>Concentration, weight, %</u> | <u>Element</u> | <u>Concentration, weight, %</u> |
|------------------------------|-------------------------------------|------------------------------------|-------------------------------------|
| Calcium ^{2c, 5a} | 1.30 ± 0.05 | Potassium ^{2b, 5a} | 1.69 ± 0.03 |
| Chlorine ^{3, 5a} | 1.09 ± .02 | Sodium ^{2c, 5a} | 0.497 ± .010 |
| Magnesium ^{2c, 5a} | 0.120 ± .003 | Sulfur ^{3, 4a} | .351 ± .005 |
| Phosphorus ^{2a, 2c} | 1.06 ± .02 | | |
| <u>Element</u> | <u>Concentration, µg/g</u> | <u>Element</u> | <u>Concentration, µg/g</u> |
| Cadmium ^{1b, 5b} | 0.0005 ± 0.0002 | Lead ^{1b, 4a} | 0.019 ± 0.003 |
| Chromium ^{4c, 5b} | .0026 ± .0007 | Manganese ^{1b, 2a, 5a} | .26 ± .06 |
| Copper ^{1b, 2a, 5b} | .7 ± .1 | Mercury ^{1a, 5b} | .0003 ± .0002 |
| Iodine ^{4a, 6} | 3.38 ± .02 | Selenium ^{1d, 4b, 5a, 5b} | .11 ± .01 |
| Iron ^{4a, 5a} | 1.78 ± .10 | Zinc ^{1c, 2c, 4b, 5a} | 46.1 ± 2.2 |

1. Atomic absorption spectrometry

- a. cold vapor
- b. electrothermal
- c. flame
- d. hydride generation

2. Atomic emission spectrometry

- a. dc plasma
- b. flame
- c. inductively coupled plasma

3. Ion chromatography

4. Isotope dilution mass spectrometry

- a. thermal ionization
- b. spark source
- c. electron impact

5. Neutron activation

- a. instrumental
- b. radiochemical

6. Photon Activation

Notes: (1.) Analytical values are based on the "dry-weight" of material (see Instructions for Drying).

(2.) 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, as described in "Approximately Linear Models," by J. Sacks and D. Ylvisaker, *Annals of Statistics*, 6, pp. 1122-1137, 1978.

Table 2. Noncertified Concentrations of Constituent Elements

| <u>Element</u> | <u>Concentration,</u> <u>μg/g</u> | <u>Element</u> | <u>Concentration,</u> <u>μg/g</u> |
|----------------|--------------------------------------|----------------|--------------------------------------|
| Aluminum | (2) | Molybdenum | (0.34) |
| Antimony | (0.00027) | Rubidium | (11) |
| Arsenic | (.0019) | Silicon | (<50) |
| Bromine | (12) | Silver | (<0.0003) |
| Cobalt | (0.0041) | Tin | (<0.02) |
| Fluorine | (.20) | | |

Table 3. Noncertified Concentrations of Organic Constituents

| <u>Compound</u> | <u>Number of</u> <u>Determinations</u> | <u>Concentration,^a</u> <u>weight %</u> | <u>Method</u> |
|-----------------|---|--|---|
| Lactose | 5 | 49 ± 3 | High Performance Liquid Chromatography |
| | 5 | 45 ± 2 | Proton Nuclear Magnetic Resonance |
| <u>Compound</u> | <u>Number of</u> <u>Determinations</u> | <u>Concentration,^a</u> <u>μg/g</u> | <u>Methods</u> |
| Ascorbic Acid | 10 | 53 ± 5 | High Performance Liquid Chromatography |

^aUncertainties represent one standard deviation.