# National Institute of Standards & Technology

# Certificate of Analysis

## Standard Reference Material 1548

### Total Diet

This Standard Reference Material (SRM) is intended primarily for use in evaluating the reliability of analytical methods used for the determination of major, minor and trace constituent elements; proximate content of fat, ash, protein (Kjeldahl nitrogen) and caloric content (bomb calorimetry) in mixed diets and similar foods and biological materials. A unit of SRM 1548 consists of a homogeneous mixture of freeze-dried foods packaged in two separate bottles of approximately 6.5 grams each.

This SRM was prepared from excess foods obtained from the U.S. Food and Drug Administration's Total Diet Study (FDA-TDS). The FDA-TDS is an on-going program which collects foods in various regions of the United States in order to monitor the food supply for pesticides, toxicants and some nutrients[1]. The foods used to prepare SRM 1548 were proportioned such that the material is representative of the United States adult dietary intake[2].

Material Application:

Together with other SRMs and RMs issued by NIST, SRM 1548 is expected to be useful for improving the accuracy of measurements used in evaluating the role of nutrient constituents in health and disease. These measurements will also be used for establishing dietary requirements for nutrients, accumulating accurate base line concentration data, and generating composition data for nutrient and proximate constituents in foods and related materials.

WARNING: For laboratory analysis and "in vitro" use only. Not for human consumption.

Use and transport of this material must meet all U.S. regulatory requirements and guidelines. This material has been $^{60}Co$ radiation sterilized at a dose of 2.5-5.0 mrad to prevent bacterial growth.

Storage:

SRM 1548 should be stored in a refrigerator at a temperature between 2 and 8 °C in its original container, and tightly capped. It should not be exposed to intense direct light or ultraviolet radiation. Under recommended storage conditions, this SRM is expected to be stable for at least two years from the date of shipment from NIST. Should evidence indicate degradation, purchasers will be notified by NIST. Please return the attached registration card to facilitate notification.

Recommended Procedures for Use:

Allow bottle to come to room temperature before opening. Exposure of sample to air should be minimized and any unused portion stored in a sealed bottle as described under "Storage". Before each use, the contents of the bottle should be well mixed by gently shaking and rolling the container. The recommended minimum sample weight for this diet material is 500 milligrams. Residual moisture content should be determined on a separate sample for conversion of analytical results to a dry weight basis. The recommended drying method is freeze-drying. If freeze-drying is not available, vacuum drying at a temperature not to exceed approximately 25 °C for 24 hours will be adequate. Due to the high fat content of this material, excessive drying times and temperatures will lead to loss of volatile lipid components and an incorrect estimation of dry weight.

Gaithersburg, MD 20899
November 14, 1991
(Revision of certificate dated 9-28-90)

William P. Reed, Chief
Standard Reference Materials Program

(over)

Table 1. Certified Elemental Concentration and Uncertainty

| Element | Certified Value[a] | Uncertainty[b] | Units | Analysts and Methods[d] |
|---|---|---|---|---|
| Nitrogen[c] | 3.44 | 0.14 | wt% | 9,14,20,21,22 |
| Chlorine | 0.87 | 0.04 | wt% | 1,12 |
| Sodium | 0.625 | 0.026 | wt% | 2,17 |
| Potassium | 0.606 | 0.028 | wt% | 2,17 |
| Phosphorus | 0.324 | 0.004 | wt% | 3,19 |
| Sulfur | 0.258 | 0.026 | wt% | 1,11,12 |
| Calcium | 0.174 | 0.007 | wt% | 3,17 |
| Magnesium | 556 | 27 | ug/g | 3,17 |
| Iron | 32.6 | 3.6 | ug/g | 6,17 |
| Zinc | 30.8 | 1.1 | ug/g | 6,17 |
| Manganese | 5.2 | 0.4 | ug/g | 4,7,17 |
| Copper | 2.6 | 0.3 | ug/g | 4,7,17 |
| Selenium | 0.245 | 0.005 | ug/g | 6,18 |
| Cadmium | 0.028 | 0.004 | ug/g | 5,7,15,16 |

[a] The certified values are equally weighted means of results from at least two analytical techniques.
[b] Each uncertainty is obtained from a 95% prediction interval plus an allowance for systematic error. The resulting uncertainty limits will cover the concentration of approximately 95% of samples of this SRM having a minimum sample size of 0.5 gm.
[c] Kjeldahl Nitrogen Method
[d] Analysts and Methods listed in Table 3.

Table 2. Certified Values for Matrix Components, Cholesterol and Caloric Content

| Constituent | Certified Value | Uncertainty | Units | Analysts and Methods[e] |
|---|---|---|---|---|
| Kjeldahl Nitrogen[a] | 3.44 | 0.14 | wt% | 9,14,20,21,22 |
| Fat[b] | 20.6 | 2.0 | wt% | 14,20,21,22 |
| Ash[b] | 3.53 | 0.17 | wt% | 11,14,20,21,22 |
| Dietary Fiber[c] | 3.69 | 0.11 | wt% | 20,21 |
| Caloric Content[d] | 5.22 | 0.02 | kcal/g | 11 |

[a] Certified Values and Uncertainty for Kjeldahl Nitrogen reflect values in Table 1. The conventional average protein conversion factor of 6.25 may be used to give an estimate of protein content of 21.5 % in this material.
[b] Certified Values and Uncertainty for Fat, Ash and Fiber are means and prediction intervals determined similarly as in Table 1.
[c] AOAC Official Method of Analysis, 15th Ed (1990), Method Number 985.29.
[d] Values for caloric content were determined by a single definitive method.
[e] Analysts and Methods listed in Table 3.

Table 3. Analysts, Methods and Analytes

| | Laboratory/Analyst(s) | Method(s) | Analyte(s) |
|---|---|---|---|
| NIST Center for Analytical Chemistry<br>- Inorganic Analytical Research Division | | | |
| 1. | L.A. Holland<br>W.F. Koch | Ion Chromatography<br>Oxygen Bomb Combustion | S, Cl |
| 2. | L.J. Wood<br>R.L. Watters<br>M.S. Epstein<br>L. Yu | Flame Emission<br>Spectrometry | K, Na |
| 3. | L.J. Wood<br>R.L. Watters | Inductively Coupled<br>Plasma Emission Spectroscopy | Ca, Mg, P |
| 4. | G.C. Turk<br>H.M. Kingston<br>C. Clements<br>L.B. Jassie | Laser-Enhanced<br>Ionization Spectroscopy<br>Automated Chelation Separation | Cu, Mn,Ni |
| 5. | K.W. Pratt | Anodic Stripping<br>Voltametry | Cd, Pb |
| 6. | R.R. Greenberg<br>T.M. Sullivan | Instrumental<br>Neutron Activation Analysis | Cr, Cs, Eu,Fe<br>Rb, Se,Sn,Zn |
| 7. | G.V. Iyengar | Radiochemical<br>Neutron Activation Analysis | Cd, Cu, Mn,Mo |
| 8. | R.G. Downing<br>G.V. Iyengar<br>W.B. Clarke(McMaster University) | Neutron Activation-<br>Mass Spectrometry | B, Li |
| - Gas and Particulate Science Division | | | |
| 9. | G.A. Sleater | Kjeldahl | Nitrogen |
| NIST, Center for Chemical Technology<br>- Chemical Thermodynamics Division | | | |
| 11. | J. C. Colbert<br>E. Diaz | Bomb Calorimetry<br>Gravimetry<br>ASTM | Caloric Content,<br>Sulfur<br>Ash |

Table 3 cont.

| | | | |
|---|---|---|---|
| Food and Drug Administration, Washington, D.C.<br>- Center for Food Safety and Nutrition | | | |
| 12. | D.L. Anderson | Neutron Capture Prompt Gamma Activation Analysis | B, S, Cl |
| 13. | W. Cunningham | Instrumental Neutron Activation Analysis | Al |
| 14. | J.T. Tanner<br>M. Bueno<br>G. Angyl<br>C. Weaver<br>E. Anderson | AOAC,<br>AOAC, Muffle<br>AOAC, Kjeldahl<br>AOAC, Microbiological | Fat<br>Ash<br>Protein<br>Vitamins |
| 15. | S.C. Hight | Anodic Stripping Voltametry | Cd, Pb |
| Nuclear Research Center, KFA, Julich, FRG | | | |
| 16. | M. Stoeppler | Anodic Stripping Voltametry | Cd, Pb |
| U.S. Department of Agriculture, Beltsville, MD<br>- Nutrient Composition Laboratory | | | |
| 17. | F.E. Greene<br>N.J. Miller-Ihli | Flame<br>Atomic Absorption Spectroscopy | Ca, Cu, Fe, K<br>Mg, Mn, Na, Zn |
| - Vitamin and Mineral Nutrition Laboratory | | | |
| 18. | N. Hardison<br>K. Patterson<br>C. Veillon | Isotope Dilution<br>Mass Spectrometry | Se |
| 19. | D. Hill<br>E.R. Morris | Colorimetric | P |
| Wageningen Agricultural University, The Netherlands | | | |
| 20. | P.v.d. Bovenkamp | AOAC,<br>Kjeldahl<br>Folch - $CH_3Cl/CH_3OH$<br>Muffle 550 °C | Dietary Fiber<br>N<br>Fat<br>Ash |
| RIKILT, Wageningen, The Netherlands | | | |
| 21. | J. Labrijn | AOAC,<br>Kjeldahl<br>Weibull-Soxlet, Petroleum Ether<br>Muffle 550 °C | Dietary Fiber<br>N<br>Fat<br>Ash |
| Inspection Health Protection, Maastricht, The Netherlands | | | |
| 22. | H. Roomans | Kjeldahl<br>Weibull-Soxhlet, Petroleum Ether<br>Muffle 550 °C | N<br>Fat<br>Ash |

Table 4. Non-Certified Constituents[a]

| Constituent | Value | Units | Analysts and Methods[b] |
|---|---|---|---|
| Al | (33) | ug/g | 13 |
| Rb | (4.8) | ug/g | 6 |
| Sn | (3.6) | ug/g | 6 |
| B | (2.5) | ug/g | 12,8[c] |
| Ni | (0.41) | ug/g | 4 |
| Mo | (0.27) | ug/g | 7 |
| Pb | (0.05) | ug/g | 5,15,16 |
| Li | (49) | ng/g | 8[c] |
| Cs | (14) | ng/g | 6 |
| Eu | (0.30) | ng/g | 6 |

[a] Values in this table are not certified and are listed for information only.
[b] Analysts and Methods listed in Table 3.
[c] Information from [7].

Preparation of Material.

The material for SRM-1548 was prepared as described elsewhere[2] from the 200 separate foods collected in the FDA-TDS[1] to be representative of the daily adult intake of the U.S. population. Excess food items from three collections of the FDA-TDS were shipped frozen to NIST, portioned and composited to produce six ten-kilogram batches of material. This was done similar to previously described collections[3]. Each batch was blended in a commercial food processor equipped with specially fabricated Ti blades and a nylon coated bowl to prevent contamination. Each of the blended batches was freeze-dried separately, and the six batches then combined and reblended under liquid nitrogen. After radiation sterilization in bulk, ($^{60}$Co, 2.5-5 mrad) the material was sieved in small portions thru a 30 mesh polythelyene sieve while still frozen. The resulting material was bottled by hand in a clean air hood, to yield a number of bottles containing approximately 6.5 grams each of the final material.

Characterization and Homogeneity:

During the bottling process, every 20th bottle was removed for homogeneity testing and analyses. Homogeneity was determined by repeat analysis of 10 bottles of the packaged material for a number of trace elements by instrumental neutron activation analysis. Acceptable homogeneity was demonstrated for all elements certified in Table 1, and the uncertainty values include a factor for minor inhomogeneities. One element of nutritional concern, chromium, was shown to be significantly inhomogeneous (See footnote 1). Other elements not mentioned in this certificate may be inhomogeneous and care should be taken when interpretating data on their determination in this material.

ANALYSIS AND CERTIFICATION:

Analysis of SRM 1548 was carried out by a number of individuals and groups both within NIST and outside collaborators using a variety of different analytical techniques. This material has been characterized for a wide variety of constituents, from elemental content to organic compounds.

Table 1 lists certified values for 14 elements. Final certified values were obtained from an equally weighted mean obtained from the individual data reported by each collaborator. The uncertainty values reflect best estimates of all sources of uncertainty including both inter-laboratory and inter-method variability and any uncertainties due to inhomogeneity.

Table 2 lists certified values for Matrix Components and Caloric Content.

Energy data were generated by the Chemical Thermodynamics Division, NIST using bomb calorimetry. This value reflects the total combustible energy content of SRM 1548, which may differ from the metabolizable caloric content of this diet material. This energy value should NOT be used to directly reflect the nutritional caloric content of this diet material.

The proximate(matrix) data were generated by a group of collaborators outside NIST, including laboratories in both North America and in Europe. In addition, a Kjeldahl nitrogen value (also included in Table 1) was obtained from the Gas and Particulate Research Division, NIST. Collection of the data from the European laboratories was coordinated by P.C.H. Hollman, RIKILT, Wageningen, The Netherlands. These data were generated together with certification analyses carried out in the European Economic Community program (Community Bureau of Reference, BCR, Brussels, Belgium) to produce individual food matrix Certified Reference Materials characterized for proximate content [4,5]. Thus the values for proximate content in SRM 1548 are consistent with those of the BCR materials and SRM 1548 may be used in conjunction with them.

Table 3 lists the individual methods used and the analysts and laboratories involved in generating the analytical data for each certified constituent. Coordination of the preparation of SRM 1548 was carried out by G.V. Iyengar, IARD, NIST and W.R. Wolf, SRMP, NIST and USDA, Beltsville, MD. Coordination of the data for elemental analyses was carried out by G.V. Iyengar and coordination of the data for proximate and organic constituents was carried out by W.R. Wolf.

Footnote 1. Repeated measurements by neutron activation analysis(Analyst 6, Table 3) did show significant inhomogeneity for chromium content in the bottled material. This inhomogeneity was confirmed in the bottled material by independent analysis using stable isotope dilution mass spectrometry(Analyst 18, Table 3). Due to the observed inhomgeneity in the bottled material, there is no value listed for chromium content, and no use of this material should be made for accurate determination of chromium content.

Statistical support and determination of certified values was done by S. B. Schiller of the Statistical Engineering Division, NIST.

Table 4 lists values for constituents that are not certified in SRM 1548. These values are from a limited number of analyses or by only one method and are given for information only. As additional information becomes available for this material, future certification of these and additional constituents in this material may be possible. The content of water soluble vitamins in this material can be estimated from published information on analyses of the IAEA USDIET composites [6](Analysts 14, Table 3).

Technical and support aspects involved in preparation, certification, and issuance of this SRM were coordinated through the Standard Reference Materials Program by W.R. Wolf, NIST Research Associate, U.S. Department of Agriculture and R. Alvarez, SRMP.

References:

1. J.A. Pennington, Revision of the Total Diet Study Food List and Diets, J. Am. Diet Assoc., 82 (1983) 166-173.

2. W.R. Wolf, G.V.Iyengar, J.T. Tanner, Mixed Diet Reference Materials for Nutrient Analysis of Foods: Preparation of SRM 1548 Total Diet, in Proceedings of 4th Symposium on Biological and Environmental Reference Materials (BERM-4), Fresenius J. Analytical Chemistry (1990), 338,473-475.

3. G.V. Iyengar, J.T. Tanner, W.R.Wolf, and R. Zeisler, Preparation of a Mixed Human Diet Material for the Determination of Nutrient Elements, Selected Toxic Elements and Organic Nutrients: A Preliminary Report. The Sci. of the Total Environment, 61, (1987), 235-252.

4. P. Hollman, P.J. Wagstaffe, Development of Food Reference Materials for Major Nutritional Properties, in Proceedings of the 4th BERM, Fresenius J. of Analytical Chemistry, (1990) 338, 430-434.

5. P.J. Wagstaffe, J.J. Belliardo,BCR Programm and Measurements For Food and Agriculture: Reference Values and Reference Materials, Fresenius J. of Analytical Chemistry, (1990) 338, 469-472.

6. J.T. Tanner, G.V. Iyengar, W.R. Wolf, Organic Nutrient Content of U.S. Food and Drug Administrations' Total Diet and Its Posssible Use as a Standard Reference Material, Fresenius J. of Analytical Chemistry, (1990) 338, 438-440.

7. G.V. Iyengar, W.B. Clarke, and R.G. Downing, Determination of Boron and Lithium in Diverse Biological Matrices Using Neutron Activation-Mass Spectrometry (NA-MS)., Fresenius J. of Analytical Chemistry (1990) 338, 562-566.