



National Bureau of Standards

Certificate of Analysis

Standard Reference Material 1567a

Wheat Flour

This Standard Reference Material (SRM) is intended primarily for calibrating instruments and evaluating the reliability of analytical methods for the determination of minor and trace elements in wheat flour and similar agricultural food products.

Certified Concentrations of Constituent Elements: The certified concentrations of the constituent elements are shown in Table 1. Except for sulfur, the concentrations are based on results obtained by two or more independent, reliable analytical methods. Non-certified values which are given for information only, appear in Table 2. Sulfur is certified based on its determination by a definitive method, isotope dilution thermal ionization mass spectrometry. All values are based on a minimum sample size of 500 mg and are reported on a "dry weight" basis. (See "Instructions for Drying").

Notice and Warnings to Users:

Expiration of Certification: This certification will be invalid after 5 years from the date of shipping. Should it be invalidated before then, purchasers will be notified by NBS.

Storage: 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, including ultraviolet lamps or sunlight. Ideally, the bottle should be kept in a desiccator in the dark at the temperature indicated.

Use: The following procedures should be followed to relate the analytical determinations to the values reported in this certificate. The bottle should be shaken well before each use, and a minimum sample of 500 mg of the material should be used. Mercury should be determined without drying and the concentration values adjusted for the moisture content of the material using separate samples. Other elements may be determined either on samples without drying as indicated above or on samples vacuum-dried for 24 hours as indicated under "Instructions for Drying."

Coordination of some technical measurements leading to this certificate was performed by M.S. Epstein of the Inorganic Analytical Research Division.

Statistical analysis of the experimental data was performed by K.R. Eberhardt of the NBS Statistical Engineering 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
October 23, 1987

Stanley D. Rasberry, Chief
Office of Standard Reference Materials

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Table 1. Certified Concentrations of Constituent Elements^a

Minor Constituents

<u>Element^b</u>	<u>Concentration, Percent by Weight^c</u>
Calcium ^{1a,2b}	0.0191 ± 0.0004
Magnesium ^{1a,2a,4a}	0.040 ± 0.002
Phosphorus ^{2a,2c,5}	0.134 ± 0.006
Potassium ^{2b,4a}	0.133 ± 0.003
Sulfur ^{3b}	0.165 ± 0.002

Trace Constituents

<u>Element^b</u>	<u>Concentration, µg/g^c</u>
Aluminum ^{2a,4a}	5.7 ± 1.3
Cadmium ^{1b,4b}	0.026 ± 0.002
Copper ^{1a,4a,b}	2.1 ± 0.2
Iron ^{1a,3b,4a}	14.1 ± 0.5
Manganese ^{1a,4a}	9.4 ± 0.9
Molybdenum ^{2c,3a,4a}	0.48 ± 0.03
Selenium ^{1c,4a}	1.1 ± 0.2
Sodium ^{2b,4a}	6.1 ± 0.8
Zinc ^{1a,4a}	11.6 ± 0.4

^aAnalytical values are based on the "dry-weight" of material (see "Instructions for Drying"). Mercury should be determined on samples without drying and the results adjusted to a "dry-weight" basis by determining moisture on separate samples.

^bNumber and letter code, as superscripts, indicate methods used for certification. (See Analytical Methods).

^cThe certified concentration is the weighted mean computed according to the procedure described by R.C. Paule and J. Mandel (NBS Journal of Research, 87, 1982, pp. 377-385). The uncertainty is stated as a 95% confidence interval plus an additional allowance for systematic error among the methods used. The allowance for systematic error is the greatest difference between the weighted mean and the component means for the analytical methods used. For manganese, an additional allowance for material inhomogeneity is included, so that the uncertainty represents a 95% expected coverage statistical tolerance interval.

Analytical Methods

1. Atomic Absorption Spectrometry
 - a. Flame
 - b. Graphite Furnace
 - c. Hydride Generation
2. Atomic Emission Spectrometry
 - a. DC Plasma
 - b. Flame
 - c. Inductively Coupled Plasma
3. Mass Spectrometry
 - a. Isotope Dilution Inductively Coupled Plasma
 - b. Isotope Dilution Thermal Ionization
4. Neutron Activation Analysis
 - a. Instrumental
 - b. Radiochemical
5. Spectrophotometry

Table 2. Non-certified Concentrations of Constituent Elements^a

NOTE: The values shown in this table are not certified because they are not based on the results of either two or more independent reliable methods or a definitive method of known high accuracy. These values are included for information only and therefore no uncertainty limits are provided.

Trace Constituents

<u>Element</u>	<u>Content</u> <u>μg/g</u>	<u>Element</u>	<u>Content</u> <u>μg/g</u>
Arsenic	(0.006)	Mercury	(0.0005)
Bromine	(6)	Rubidium	(0.7)
Chlorine	(565)	Tin	(0.0033)
Cobalt	(0.006)	Tungsten	(0.0008)
Iodine	(0.0009)	Uranium	(0.0003)
Lead	(< 0.020)	Vanadium	(0.011)

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

Preparation of Material: The wheat flour for this Standard Reference Material was described by the supplier as milled from a blend of Hard Red Spring and Hard Red Winter wheat grown primarily in South Dakota. The flour was taken from the mill packer during the middle of a run to obtain homogeneous material. The flour had been bleached and brominated in accordance with standard treatments for commercial bakery use. At NBS, the material was passed through a sieve with openings of 425 μm (No. 40) and blended. The bottled material was then subjected to 2.5 megarads of Co-60 radiation for microbiological control at Neutron Products, Inc., Dickerson, Md.

Homogeneity Assessment: Homogeneity was evaluated by instrumental neutron activation using samples of approximately 500 mg and counting the radionuclidic activities. The uncertainties for the concentrations in Table 1 include these results.

Instructions for Drying: Except for mercury, elements should be determined on samples which have been dried as follows:

Vacuum-dry the material at approximately 25 °C for 24 hours at a pressure not greater than 70 Pa (0.5 mm Hg) with a cold trap at a temperature of about -30 °C or below.

Mercury should be determined on undried samples. However, because the certificate values are reported on a "dry-weight" basis, the elemental concentration determined on undried samples should be adjusted for the moisture content of the samples. The moisture content, which was approximately 9% when bottled, should be determined on separate samples by the vacuum-drying procedure described above. Samples for analysis should not be oven-dried lest elements be lost by volatilization.

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