

National Bureau of Standards Certificate of Analysis

Standard Reference Material 1567

Wheat Flour

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

Certified Values of Constituent Elements: The certified values for the constituent elements are shown in Table 1. They 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.

All values are based on a minimum sample size of 400 mg and are reported on a "dry-weight" basis.

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 400 mg of the material should be used. Selenium and mercury should be determined in the material 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 hr as indicated under "Instructions for Drying."

The overall direction and coordination of the technical measurements leading to this Certificate were performed under the chairmanship of H. L. Rook.

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.

Washington, D.C. 20234
January 3, 1978

J. Paul Cali, Chief
Office of Standard Reference Materials

(over)

Table 1. Certified Values of Constituent Elements^a

Minor Constituents

<u>Element</u>	<u>Content</u> <u>Wt. Percent</u>
Potassium	0.136 ± 0.004 ^b
Calcium	0.019 ± 0.001

Trace Constituents

<u>Element</u>	<u>Content</u> <u>µg/g</u>	<u>Element</u>	<u>Content</u> <u>µg/g</u>
Iron	18.3 ± 1.0	Copper	2.0 ± 0.3
Zinc	10.6 ± 1.0	Selenium	1.1 ± 0.2
Manganese	8.5 ± 0.5	Cadmium	0.032 ± 0.007
Sodium	8.0 ± 1.5	Mercury	0.001 ± 0.0008

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

^bThe estimated uncertainty is based on judgment and represents an evaluation of the combined effects of method imprecision, possible systematic errors among methods, and material variability for samples 400 mg or more. (No attempt was made to derive exact statistical measures of imprecision because several methods were involved in the determination of the constituents).

Table 2. Non-certified Values for Constituent Elements^a

NOTE: The values shown in this table are not certified because they are not based on the results of two or more independent reliable methods. These values are included for information only.

Trace Constituents

<u>Element</u>	<u>Content</u> <u>µg/g</u>	<u>Element</u>	<u>Content</u> <u>µg/g</u>
Bromine	(9)	Nickel	(0.18)
Rubidium	(1)	Arsenic	(0.006)
Molybdenum	(0.4)	Tellurium	(≤0.002)

^aAnalytical 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: A preliminary evaluation of homogeneity was made by instrumental neutron activation using samples of 150 to 300 mg and counting the activities from radionuclides of Mn, K, Zn, Na, and Br. The homogeneity of other certified elements was evaluated using samples of 400 mg or less with the exception of mercury and calcium for which 500 mg and 1 g, respectively, were used. The uncertainties for the concentrations in Table 1 include these results.

Instructions for Drying: Except for selenium and mercury, elements may 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.

Se and Hg should be determined on undried samples; other elements may be so determined. However, because the Certificate values are reported on a "dry-weight" basis, the elemental concentrations 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 either the vacuum-drying procedure described above or drying the sample in air in an oven at 85 °C for 24 hours. Both of these procedures yielded the same loss in weight. Samples for analysis should not be oven-dried lest elements be lost by volatilization.

Analytical Methods Used and Analysts

Analytical Methods

- A. Atomic absorption spectrometry
- B. Flame emission spectrometry
- C. Isotope dilution spark source mass spectrometry
- D. Neutron activation
- E. Polarography

Analysts

Analytical Chemistry Division, National Bureau of Standards

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|--------------------|---------------------|
| 1. J. R. Baldwin | 10. G. J. Lutz |
| 2. T. J. Brady | 11. E. J. Maienthal |
| 3. M. G. Diaz | 12. R. Mavrodineanu |
| 4. L. P. Dunstan | 13. J. D. Messman |
| 5. M. S. Epstein | 14. R. M. Morris |
| 6. M. Gallorini | 15. P. J. Paulsen |
| 7. T. E. Gills | 16. T. C. Rains |
| 8. R. R. Greenberg | 17. P. A. Sleeth |
| 9. R. M. Lindstrom | |

Cooperating Analysts

18. W. R. Wolf and J. Holden, Nutrition Institute, U.S. Department of Agriculture, Beltsville, Md.

Addendum to
National Bureau of Standards
Certificate of Analysis
Standard Reference Material 1567
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Additional Certification

The following certified value is to be added to Table 1.

Table 1. Certified Values of Constituent Elements^a

<u>Element</u>	<u>Content, $\mu\text{g/g}$</u>
Lead	0.020 ± 0.010^b

^aLead should be determined on samples without drying and the results adjusted to a "dry-weight" basis by determining moisture on separate samples.

^bThe estimated uncertainty, based on judgment, is for samples 2 g or more.

Analytical Methods Used and Analysts

Inorganic Analytical Research Division, National Bureau of Standards.

Isotope dilution, mass spectrometry, I. L. Barnes and E. S. Beary;

Polarography, E. J. Maienthal.

Cooperating Analyst

R. W. Dabeka, Food Directorate, Health Protection Branch, Ottawa, Canada.