



# National Bureau of Standards

## Certificate

### Standard Reference Material 485

#### Austenite in Ferrite

This standard is provided for the calibration of x-ray diffraction equipment used in determining the amount of retained austenite in ferrous materials. X-ray diffraction procedures require accurate measurements of the integrated intensity for a number of selected peaks; however, as the amount of retained austenite decreases, the accuracy of these measurements deteriorates. This nominally four percent austenite standard should serve the calibration needs at the low levels.

<u>SRM No.</u>	<u>Description</u>	<u>Austenite, percent</u>
485	Austenite in Ferrite <sup>a</sup>	4 (Nominal) <sup>b</sup>

<sup>a</sup>The austenite standard is available in disk form, 20.6 mm (13/16 in) in diameter by 2.5 mm (0.10 in) thick.

<sup>b</sup>Only one face on each disk is certified; the opposite face is labeled with the determined austenite content to the nearest 0.1%. The certified value is believed accurate to  $\pm 0.2\%$ . CAUTION: Damage to the certified face renders the certification of that disk void.

The preparation of specimens and the technical measurements leading to certification were directed and coordinated by G. E. Hicho.

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. E. Michaelis.

Technical details concerning the preparation and evaluation of this standard are given in NBS Special Publication 260-25, Standard Reference Materials: A Standard Reference Material Containing Nominally Four Percent Austenite (Revised 1972).

Washington, D.C. 20234  
August 23, 1972  
(Supersedes Certificate dated July 30, 1970)

J. Paul Cali, Chief  
Office of Standard Reference Materials

(over)

## SUPPLEMENTARY INFORMATION

The austenite content of this SRM is directly related to the nickel content as a result of blending austenitic stainless steel powder (20.4% Ni) with ferritic stainless steel powder (<0.1% Ni). Therefore, accurate and rapid x-ray fluorescence analysis could be used for the determination of the nickel content, and hence the austenite content, of each sintered and pressed compact.

An x-ray fluorescence calibration curve relating Ni K $\alpha$  count rate to austenite content was established using five specially prepared compacts which were characterized by the following two methods:

1. Quantitative Television Microscopy: Eighty fields were observed on each of the five compacts containing 5, 10, 15, 20, and 25 percent austenite, respectively. Observation covered three different time intervals. (D. E. Harne and G. E. Hicho)
2. Electro-Mechanical Drum Scanner: Five fields were observed on four of the five compacts, containing 10, 15, 20, and 25 percent austenite. Observations were made at the midpoints of four orthogonal radius lines and at the center. (D. E. Harne, G. E. Hicho, and G. A. Moore)

Methods 1 and 2 were given equal weight in establishing the curve. The calibration curve was least-square fitted by a third order polynomial using statistical procedures contained in OMNITAB routines. The x-ray fluorescence intensity data were collected at two different times for each of the five compacts; the calibration curve was virtually the same in both cases. Total Ni counts of 1 to  $1.5 \times 10^5$  were collected in each determination, leading to a typical standard deviation of  $\pm 0.05$  percent austenite (S. D. Rasberry and H. Yakowitz).

The certified value for each disk of SRM 485 is based on the results of three determinations of the Ni K $\alpha$  count rate converted to austenite content. Possible sources of bias that arose in establishing the calibration curve included drift of the quantitative television microscope and sampling procedures of areas observed with the drum scanner. An analysis of all these factors indicates that the stated austenite content is probably accurate within  $\pm 0.2$  percent austenite.

In an independent test, one compact was studied by several commercial quantitative microscopy devices in laboratories outside NBS. An analysis of the results indicated a mean value of 4.93 percent austenite. The value obtained at NBS using the established x-ray fluorescence calibration curve was 4.90 percent austenite.

This standard has been established to provide a means for calibrating x-ray diffraction equipment. SRM 485, in special cases, may be used as an x-ray fluorescence standard for determining the nickel content in nickel-iron or nickel-chromium-iron composites.

The powder for SRM 485 was prepared at the Federal Mogul Corporation, Detroit, Michigan, and the compacts were prepared at SKC/PM Engineering of Hawthorne, New Jersey, under the direction of G. E. Hicho.