U. S. Department of Commerce
Maurice H. Stans
Secretary

National Bureau of Standards
L. M. Branscomb, Director

National Bureau of Standards
Certificate of Analysis
Standard Reference Material 479
Fe-Cr-Ni Alloy Microprobe Standard

This Standard Reference Material is characterized for chemical homogeneity of iron, chromium, and nickel at the micrometer level of spatial resolution and is satisfactory for use as a homogeneous material for electron probe microanalysis. It is issued in wafer form, 4.6 mm in diameter and 1 mm thick, in the as-cut condition after electrical discharge machining.

CERTIFIED CHEMICAL CHARACTERIZATION

SRM	Nickel[a]	Chromium[a]	Iron (by difference)[b]
		Weight, percent	
479	10.7	18.3	71.0

[a] Nickel was determined gravimetrically by the nickel dimethylglyoxime method; chromium was oxidized to chromate and titrated with a standard ferrous ammonium sulfate solution. Analyst - R. A. Paulson

[b] Pure chromium, nickel, and iron were used in the preparation of this alloy (99.9% minimum purity for each element). (For information only, carbon was determined to be 0.012%.)

CERTIFIED HOMOGENEITY CHARACTERIZATION

Homogeneity testing for iron, chromium, and nickel was carried out by means of the NBS electron microprobe with an operating voltage of 20kV and spectrometers equipped with LiF crystals and sealed proportional detectors. Approximately 160 analyses were made on each of five samples taken from the original alloy rod ends, and the 1/4, 1/2, and 3/4 positions, respectively. The observed coefficient of variation was not greater than 1.5% for any of the three elements. For sixteen determinations made for each element, the observed 99% confidence intervals about the means were (Wt.%):

Iron	± 0.80
Chromium	± 0.20
Nickel	± 0.12

Analyst - H. Yakowitz

The preparation of the original alloy ingot and subsequent thermal and mechanical processing steps to improve chemical homogeneity were conducted by D. P. Fickle and A. W. Ruff.

The technical and support requirements associated with the issuance of the Standard Reference Material were coordinated through the Office of Standard Reference Materials by R. E. Michaelis.

Technical details concerning the preparation and characterization of this SRM are given in NBS Special Publication 260-43, Preparation and Homogeneity Characterization of an Austenitic Iron-Chromium-Nickel Alloy.

Washington, D.C. 20234
October 16, 1972

J. Paul Cali, Chief
Office of Standard Reference Materials

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SUPPLEMENTARY INFORMATION

The original alloy ingot was produced by the arc fusion method in an inert gas atmosphere of argon, gettered by titanium. The ingot was repeatedly swaged, and annealed in a sealed quartz tube until the final diameter of 4.6 mm was reached. A diffusion annealing step was included for 5 days at 1120 °C.

The rod was cut into wafers approximately 1 mm thick by electro-discharge machining in an organic solvent. The specimens selected for homogeneity characterization were ground on a series of SiC papers, followed by 6μm diamond and then 1/4μm diamond particle polishing. Final polishing was done using 0.05μm Al_2O_3 particles on Gamal cloth.

Electron probe microanalysis was performed on the 1/4, 1/2, 3/4 specimens using pure element standards (M. M. Darr, H. Yakowitz, C. E. Fiori, and R. L. Myklebust). Results are as follows:

Element	Voltage (kV)	Concentration (Wt. %)
Fe	15	70.8
Fe	20	70.1
Fe	25	71.7
Fe	30	71.1
Cr	15	18.4
Cr	20	19.2
Cr	25	19.2
Cr	30	19.3
Ni	15	10.9
Ni	20	10.7
Ni	25	10.7
Ni	30	10.7

Prior to the production of SRM 479, a portion of the original ingot was used to produce foil specimens for subsequent determination of the stacking fault energy of the alloy over the temperature range 25 to 325 °C using transmission electron microscope methods (R. M. Latanision and A. W. Ruff, Met. Trans. 2, 505 (1971)). Those results indicated that satisfactory solute homogeneity should be obtained by a suitable thermal and mechanical processing program.