U. S. Department of Commerce
Philip M. Klutznick
Secretary

National Bureau of Standards
Ernest Ambler, Director

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

This Standard Reference Material is certified for chemical composition and for homogeneity 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 4.5 mm in diameter and 0.8 mm thick, as cut with a diamond blade saw. The alloy ingot, from which wafers were cut, was produced by arc fusion in an inert atmosphere of argon. Pure chromium, nickel, and iron of 99.9% minimum purity were used. The ingot was repeatedly swaged and annealed until the final diameter was reached. The ingot was finally diffusion annealed for 5 days at 1120 °C.

CHEMICAL COMPOSITION

Element	Weight Percent	Standard Deviation
Nickel	10.9	0.1
Chromium	18.1	0.1
Iron (by difference)	71.0 (by difference)	

Nickel was determined spectrophotometrically by the nickel dimethylglyoxime method and chromium was oxidized to chromate and titrated with a standard ammonium sulfate solution. The certified values are based on duplicate analyses made on four samples.

HOMOGENEITY

The three elements (iron, chromium, and nickel) were tested simultaneously for homogeneity with the NBS electron microprobe. The testing procedures included random sampling analyses and a new technique using a periodic integrator (R.B. Marinenko, K.F.J. Heinrich and F.C. Ruegg, Nat. Bur. Stand. (U.S.) Spec. Publ. 260-65 (1979)) to measure concentration fluctuations across the sample. Analyses were made with an operating voltage of 20 kV with LiF crystal spectrometers and proportional detectors. Four specimens were tested, one from an end of the ingot and the others from 1/4, 1/2, and 3/4 positions along the length of the ingot.

Periodic integrator ratemeter traces were made by moving the specimen in 5-μm steps under a 1-μm electron beam using counting periods of 10 s. Two traces, normal to each other, on each specimen did not indicate inhomogeneities within the specimens.

The preparation of the ingot and subsequent thermal and mechanical processing steps to improve chemical homogeneity were conducted by F.S. Biancaniello, P.A. Boyer, and A.W. Ruff of the Metallurgy Division.

The wet chemical analyses of nickel and chromium content were performed by B.I. Diamondstone of the Inorganic Analytical Research Division.

The determinations of homogeneity with the electron microprobe were made by R.B. Marinenko and P.C. Johnson of the Gas and Particulate Science Division.

The statistical evaluation of the data was performed by L. De Robertis of the 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.K. Kirby.

Washington, D.C. 20234
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George A. Uriano, Chief
Office of Standard Reference Materials

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A total of 36 analyses were made on each specimen in three different random sampling tests using counting periods of 20 s. The variations that were determined from these tests, expressed as standard deviations, include the differences among specimens, S_S, the variation within a specimen, S_B, and measurement imprecision, S_E.

	S_S	S_B	S_E
		Weight Percent	
Nickel	0.08	0.02	0.05
Chromium	.16	.06	.08
Iron	.47	.26	.18

With experimental conditions similar to those used in these homogeneity tests, the average of sixteen randomly chosen points on a single specimen should, with 99 percent probability, lie within the intervals:

	Weight Percent
Nickel	10.7 - 11.1
Chromium	17.6 - 18.6
Iron	69.6 - 72.4

The 99 percent confidence interval, $\overline{P} + 3S_P$, for each constituent was calculated from

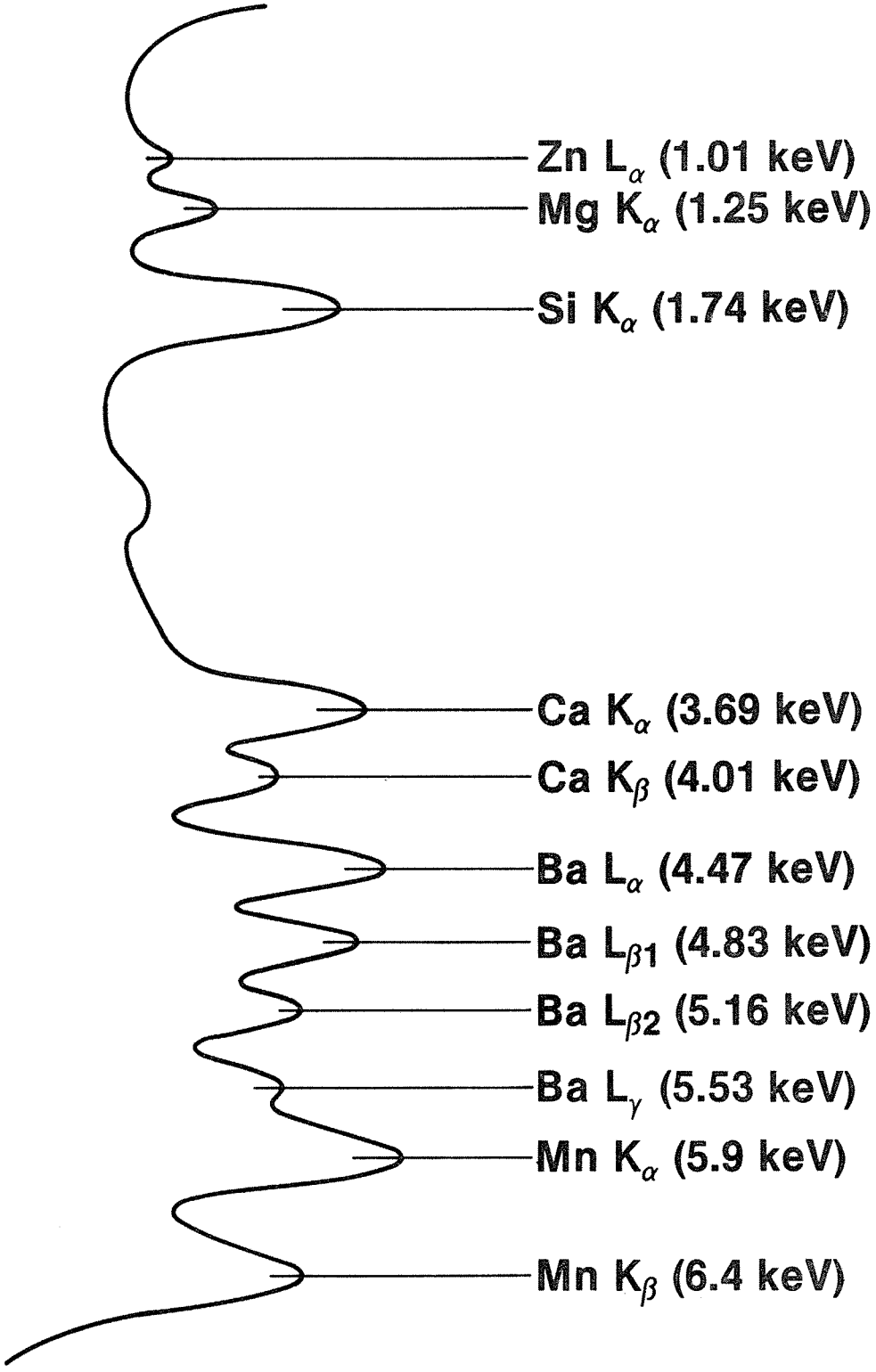
$$S_P^2 = \frac{S_S^2}{n_S} + \frac{S_B^2}{n_S n_B} + \frac{S_E^2}{n_S n_B n_E}$$

where n_E independent measurements are made at each of n_B randomly chosen points in each of n_S randomly chosen specimens. In applying these formulae users should substitute the measurement imprecision, S_E, for their apparatus.

SUPPLEMENTARY INFORMATION

The specimens used for the microprobe studies were prepared by grinding with a series of SiC papers, polishing with 6-μm and then 0.25-μm diamond powder, and finally polishing with 0.05-μm Al_2O_3 powder.

To test the utility of SRM 479a, a quantitative electron probe microanalysis was carried out on four specimens using a specimen of SRM 479 (the original lot) to calibrate the counts obtained in 40-second periods. The results confirmed (average values were within 1.3 percent) the values obtained by chemical analysis.



Characteristic Spectrum Obtained with an 8 μm Beryllium Window