U. S. Department of Commerce
Peter G. Peterson
Secretary

National Bureau of Standards
Certificate of Analysis
Standard Reference Material 1135
High-Silicon Steel

This standard is in the form of annealed disks 31.8 mm (1 1/4 in) in diameter and 19.1 mm (3/4 in) thick, primarily for application in optical emission and x-ray spectrometric methods of analysis.

	C	Mn	P	S	Si	Cu	Ni	Cr	V	Mo	Al	Sn
ANALYST		Peroxydisulfate-Arsenite	Photometric	Combustion Iodate titration	Perchloric acid dehydration	Photometric	Weighed as Nickel dimethylglyoxime	$FeSO_4$-$KMnO_4$ titration		Photometric		
1	0.025[a]	0.096[b]	0.005	0.026[c]	3.18[d]	0.054[b]	0.051[e]	0.024[f]	<0.01[g]	0.013	0.0027[h] .0032[i]	---
2	.028[a]	.095	.008[j]	.028	3.18[d]	.056[k]	.049	.023[l]	---	.014	---	0.005[m]
3	.028	.093[n]	.006[j]	.026	3.21	.057[o]	.047[e]	.022[p]	---	.015	.0030[q]	.004[m]
4	.026[a]	.092[n]	.006[r]	.026	3.18	.056[s,t]	.052[e]	.020[p]	<.01[b]	.012	.0027[u]	.002
5	---	---	---	---	---	---	---	---	---	---	.0026[v]	---
*Recommended Value	0.027	0.094	0.006	0.026	3.19	0.056	0.050	0.022	<0.01	0.014	0.0028	0.004

*The value listed is not expected to deviate from the ''true'' value by more than ± 1 in the last significant figure reported.

[a] Combustion-chromatographic

[b] Atomic absorption method

[c] 1-g sample burned in oxygen at 1450 °C and sulfur dioxide absorbed in starch-iodide solution. Iodine liberated from iodide by titration, during the combustion, with standard KIO_3 solution.

[d] Double dehydration

[e] Spectrophotometric method

(over)

[f]Chromium separated from the bulk of the iron in a 10-g sample by $NaHCO_3$ hydrolysis, oxidized with peroxydisulfate, and titrated potentiometrically with ferrous ammonium sulfate.

[g]Vanadium separated as in (f), oxidized with HNO_3 and titrated potentiometrically with ferrous ammonium sulfate.

[h]Flame emission method

[i]Polarographic method

[j]Alkalimetric method

[k]KI-$Na_2S_2O_3$ titration

[l]Iron removed by ether separation

[m]Tin precipitated as the sulfide and titrated with standard iodate solution.

[n]Periodate spectrophotometric method

[o]Copper-ammonia complex spectrophotometric method.

[p]Diphenylcarbazide spectrophotometric method

[q]$AlPO_4$ gravimetric method

[r]Phosphorus complex extracted with isobutyl alcohol

[s]Diethyldithiocarbamate spectrophotometric method

[t]Same value obtained by atomic absorption method.

[u]Ether-mercury cathode-cupferron-Chrome Azurol S spectrophotometr method.

[v]Mercury cathode-cupferron-NaOH separation 8-quinolinol spectrophotometric method.

PLANNING, PREPARATION, TESTING, ANALYSIS: For many metal SRM's it is desirable to have the material in two forms: chips, primarily for chemical analysis, and solids, primarily for optical emission and x-ray spectrometric methods of analysis. Before SRM 1135 (solid form) was prepared, plans were made also to provide the same material in chip form also as SRM 179.

The material for this standard was prepared by the Armco Steel Corporation. A single ingot was pressed to a slab with one dimension of the cross section four times that of the other dimension. After cropping top and bottom, the slab was cut lengthwise and the center section, corresponding to about one-fourth of the original ingot, was discarded. About half of the slab sections was rolled into 5-in diameter rounds. to be chipped at NBS for SRM 179. The remaining material was hot rolled to oversized rods, annealed, and centerless ground to final rod size for SRM 1135.

Extensive homogeneity testing was performed at NBS and included metallographic studies by R. F. Brady, optical emission spectrometric analysis by D. M. Bouchette and J. L. Weber, Jr., x-ray spectrometric analysis by S. D. Rasberry

and J. McKay, and chemical analysis by J. R. Baldwin and S. A. Wicks. The testing revealed the material to be of high homogeneity.

Chemical analyses were made on a composite sample of millings cut from the full cross section of the SRM 1135 material. The laboratories and analysts cooperating in the analytical program for certification are as follows:

1. J. R. Baldwin, R. K. Bell, E. R. Deardorff, E. J. Maienthal, T. C. Rains, T. A. Rush, and S. A. Wicks, Analytical Chemistry Division, Institute for Materials Research, National Bureau of Standards.

2. E. H. Shipley, Gary Steel Works, United States Steel Corporation, Gary, Indiana.

3. G. K. Stewart, Geneva Works, United States Steel Corporation, Geneva, Utah.

4. M. Dannis and R. L. LeRoy, Armco Steel Corporation Research and Technology, Middletown, Ohio.

5. W. R. Bandi and J. L. Lutz, United States Steel Applied Research Laboratory, Monroeville, Pennsylvania.

The overall direction and coordination of the technical measurements leading to certification were performed under the chairmanship of O. Menis and J. I. Shultz.

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 and C. L. Stanley.

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J. Paul Cali, Chief
Office of Standard Reference Materials