U. S. Department of Commerce
Malcolm Baldrige
Secretary

National Bureau of Standards
Ernest Ambler, Director

National Bureau of Standards

Certificate of Analysis

Standard Reference Material 1254

Calcium in Low-Alloy (Silicon) Steel

This Standard Reference Material (SRM) is in the form of disks, 32 mm (1 1/4 in) in diameter and 19 mm (3/4 in) thick, and is intended for use in calibrating with optical emission and x-ray fluorescence spectrometric methods of analysis. (A similar chemical composition material also is available in chip form, SRM 125b, primarily for application in chemical methods of analysis.

Element	Certified Value,[1] Percent by Weight	Estimated Uncertainty[2]
Calcium	0.0053	0.0003

[1]The certified value for calcium, 0.0053 percent, is the *present best estimate* of the "true" value based on analyses at NBS. The calcium was determined by Isotope Dilution-Spark Source Mass Spectrometry (ID-SSMS) J.R. Moody and P.J. Paulsen; and by Flame Emission Spectrometry (FES), T.A. Rush and T.C. Rains.

[2]The estimated uncertainty for calcium, ± 0.0003 percent, is based on judgment and represents an evaluation of the combined effects of method imprecision, possible systematic errors among methods, and material variability based on the results of flame emission spectrometry on millings cut from the full cross section of the rods at selected locations representative of the entire lot of material, (1-g samples).

PLANNING, PREPARATION, TESTING, ANALYSIS:

The material for SRM 1254 is a critically tested and evaluated portion of the material orginally used in the development of SRM's 125b and 1134, High-Silicon Steels. The material was furnished to NBS by the Armco Steel Corporation, Middletown, Ohio, courtesy of J.F. Woodruff. (For additional details on the preparation, see NBS Spec. Publ. 408, Standard Reference Materials and Meaningful Measurements, 1975, Panel on Metals, III Chemical Analysis of Metals, J.F. Woodruff, pp 528-534.)

Extensive homogeneity testing of candidate material for SRM 1254 was performed at NBS by optical emission spectrometry (OES), J.A. Norris. Of 15 rods (~8 ft long) tested, representing the candidate material, only 5 were selected for further testing. Based on the results of flame emission spectrometry (FES), T.C. Rains, on selected samples of millings (1-g samples) cut from the full cross section of the rods, four rods were finally selected for SRM 1254.

During the course of homogeneity studies of proposed SRM 1254, additional characterization of the calcium-bearing inclusions was performed by qualitative and quantitative metallography, C.H. Brady, and G.A. Hicho; and by electron microprobe analysis, D.E. Newberry.

Preliminary analyses by OES relative to other reference materials gave the following approximate results for calcium:

(0.0050%), W.R. Kennedy, American Cast Iron Pipe Company, Birmingham, Alabama, and

(0.0052%), J.A. Norris, National Bureau of Standards, Washington, D.C.

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.

Washington, D.C. 20234
April 1, 1982

George A. Uriano, Chief
Office of Standard Reference Materials

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RECOMMENDATIONS/PRECAUTIONS FOR USE OF SRM 1254

Optical Emission Analysis

The within and between disk variation for calcium (micro-inhomogeneity) is of such a magnitude that averaging 16 exposures is recommended for instrument calibration. (A lesser number of exposures may suffice in individual laboratories and will depend on the excitation source parameters, length of exposure, and the specific procedures followed.)

Sample surface preparation is critical because the intensity ratio obtained can be seriously biased by calcium present in the surfacing material. (A test of the normal surfacing procedure compared to a mechanically prepared surface is recommended.)

X-Ray Fluorescence Analysis

Care must be taken to ensure that the surface being irradiated is not one exhibiting "layered" segregation of calcium. (A simple test would be to run both surfaces of the sample. If agreement is observed, it is most probable that the two surfaces are yielding the correct calcium content.)

The surface preparation for XRF analysis is critical because the calcium secondary radiation being emitted from the sample emanates from the immediate surface. Any contamination will seriously bias the results. (A metallographic surfacing procedure using 1/4 μm diamond dust has been satisfactory for most XRF analyses. For calcium, however, some laboratories report that calcium-free belt sanding (dry) with about 80 grit is preferred.)

Elements other than calcium are present in this material as indicated below. These are *not certified,* but are given as additional information on the composition.

Element	Non-Certified Value, Percent by Weight
C	(0.03)
Mn	(.28)
P	(.03)
S	(.008)
Si	(2.9)
Cu	(0.07)
Ni	(.04)
Cr	(.02)
Mo	(.008)
Sn	(.003)
Al	(.33)