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 1090

### Oxygen in Ingot Iron

This Standard Reference Material (SRM) is intended for use in the evaluation of methods and the calibration of equipment used in the determination of oxygen in iron or ferrous materials. SRM 1090 is in the form of an ingot iron rod, 0.635 cm in diameter and 10.2 cm long (1/4 in. diameter and 4 in. long).

The oxygen content of SRM 1090 was determined by three independent methods of analysis, namely vacuum fusion, inert gas fusion, and 14 MeV neutron activation analysis. A single certified value is not reported because of small but undetermined systematic biases which exist in one or more of the methods used. Therefore, all the analytical data are presented in tabular form below.

| | Oxygen Content, μg/g | | |
|---|---|---|---|
| | Vacuum Fusion | 14 MeV Neutron Activation | Inert Gas Fusion |
| $\bar{x}$ = | 484 | 492 | 497 |
| $s^a$ = | 14 | 28 | 13 |
| n = | 216 | 6 | 12 |

$\bar{x}$ = mean oxygen value; s = standard deviation of a single determination; n = number of independent determinations

[a]The standard deviation includes error due both to the imprecision of the analytical method and to possible heterogeneity of the material analyzed.

NOTE: Oxygen determinations should be made on thoroughly and freshly cleaned samples that represent the full cross-section of the rod.

#### Preparation, Testing and Analysis

The base material for SRM 1090 is ingot iron that was obtained from the Armco Steel Corporation. It was supplied as 0.635 cm diameter centerless ground rods approximately 91 cm long (1/4 in. and 3 feet long) from a single heat preparation. It is a low alloy material with a low carbon content and relatively high oxygen content. The nominal composition of this material is as follows: C = 0.026%, Mn = 0.043%, P = 0.004%, S = 0.016%, Si = 0.004%, Cu = 0.095%.

The analyses reported in this certificate were performed by staff members of the Center for Analytical Chemistry, National Bureau of Standards.

Additional technical information on this SRM, including the names of cooperators who participated in the original analysis, is in NBS Publication 260-14, Standard Reference Materials: Determination of Oxygen in Ferrous Materials; SRM's 1090, 1091 and 1092 (1966).

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Gaithersburg, MD 20899
(Revision of Certificates
dated 3-1-66 & 4-2-69)

Stanley D. Rasberry, Chief
Office of Standard Reference Materials

(Over)

**Directions for Use:** Transfer approximately 0.40 g of this compound from the bottle to a small beaker and dry over fresh phosphorus pentoxide in a desiccator for 48 h. (Tightly close the bottle containing the remainder of the compound.) Quickly and accurately transfer 0.360 g of this dried salt to a weighed 200 mL flask. (This weight of salt is equivalent to 50 mg of nickel.) Add 3 mL of xylene and 5 mL of 2-ethylhexanoic acid and heat the flask on a hot plate, with swirling and without charring, until a clear solution forms. Add to the hot solution 80 to 90 mL of lubricating oil and gently shake the flask to mix the contents. Allow the flask to cool to room temperature and add enough lubricating oil to bring the total weight of the contents of the flask to 100 $\pm$ 0.5 g. Stopper the flask and shake gently to ensure a homogeneous solution. The concentration of nickel in this solution is 500 $\mu$g/g.

**Source of Material:** The nickel cyclohexanebutyrate was prepared by Distillation Products Industries of Rochester, NY.

## SUPPLEMENTARY INFORMATION

Although not certified, the nitrogen content of this SRM was determined by the analytical methods shown below:

| Methods of Analysis | Nitrogen (ppm by weight) | | |
|---|---|---|---|
| | $\bar{x}$ | s | n |
| Acid digestion-distillation-indophenol-photometry | 61.5 | 3.5 | 14 |
| Pressure bomb-distillation-indophenol-photometry | 59.4 | 2.0 | 4 |
| Vacuum Fusion | 60 | 1 | 15 |

The symbols $\bar{x}$, s, and n have the same meaning as given for the oxygen determinations.