

# Certificate of Analysis

## Standard Reference Material 1093

### Oxygen in Valve Steel

This standard was developed primarily for application in the neutron activation method for the determination of oxygen in ferrous materials. The high manganese content of the valve steel material (9 to 10 %) makes the determination of oxygen by vacuum and inert gas fusion methods difficult because of the gettering action of the manganese.

SRM No.	Description	Oxygen, ppm	Uncertainty, ppm
1093	Valve steel	60 <sup>a</sup>	5 <sup>b</sup>

<sup>a</sup> Results determined by 14 MeV neutron activation analysis on 12 g sample weights [1 - 3].  
<sup>b</sup> Uncertainty at the 95 percent confidence level is expressed as  $ts/\sqrt{n}$ , where  $n$  = number of samples. Eight determinations per sample on seven samples were made and the uncertainty shown is given as a measure of precision only and not accuracy since only one analytical technique was used.

This standard is supplied in rods 1/4 in (0.6 mm) in diameter and 4 in (8.2 cm) long.

The material for this standard was furnished to NBS by the Allegheny Ludlum Steel Corp., Brackenridge, Pennsylvania.

Analyses were performed by S. S. Nargolwalla, G. W. Smith, and J. E. Suddueth.

The overall direction and coordination of technical measurements leading to the certification were under the chairmanship of P. D. LaFleur.

The technical and support aspects concerning 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  
March 4, 1969

W. Wayne Meinke, Chief  
Office of Standard Reference Materials

CAUTION: Oxygen determinations should be made only on thoroughly and freshly cleaned samples that represent the full cross section of the rods.

(over)

## SUPPLEMENTARY INFORMATION

**OTHER ELEMENTS:** Although not certified at this time nitrogen, at  $4807 \pm 10$  ppm, was determined by a pressure-bomb-distillation-titrimetric method. The nitrogen value obtained by vacuum fusion was  $3640 \pm 76$  ppm. Work is now underway at NBS to discover and resolve systematic biases existing in either or both of these methods.

### PREPARATION FOR THE DETERMINATION OF OXYGEN:

- (1) Samples should be cut from the original rod in such a manner as to minimize heating of the sample; *i.e.*, by a hand hacksaw.
- (2) All surfaces of the cut sample should be thoroughly cleaned with a fine file.
- (3) Samples should be washed with C. P. ether, acetone, or other suitable solvent, dried in a stream of warm clean air, and thereafter handled only with clean forceps.
- (4) Analyses should be made as soon as possible after cleaning the sample.

**CONDITIONS FOR ANALYSIS AT NBS:** The rods were cut to 2 in lengths with a hand hacksaw and cleaned in 1,1,1-trichloroethane in an ultrasonic bath for 30 minutes. The samples were dried in a dry nitrogen atmosphere and analyzed with 14 MeV neutrons using the  $^{16}\text{O}(n,p)^{16}\text{N}$  reaction.

### REFERENCES:

- [1] Nargolwalla, S. S., Crambes, M. R., and DeVoe, J. R., *Anal. Chem.* **40**, 666 (1968).
- [2] Lundgren, F. A. and Nargolwalla, S. S., *Anal. Chem.* **40**, 672 (1968).
- [3] Nargolwalla, S. S., Przybylowicz, E. P., Suddueth, J. E., and Birkhead, S. L., *Anal. Chem.* **41**, 168 (1969).