U. S. Department of Commerce
John T. Connor, Secretary
National Bureau of Standards
A. V. Astin, Director

Certificate of Analysis

Standard Reference Materials 355, 356

Oxygen in Titanium-Base Materials

(By Vacuum and Inert Gas Fusion Methods)

No.	Type	Oxygen, ppm	Uncertainty, ppm[c]
355	Unalloyed[a]	3031[d]	57
356	Alloy, 6Al–4V[b]	1332[e]	77

Size: 355, rods ½ in. in diameter and 2 in. long; 356, rods 0.425 in. in diameter and 1¾ in. long.

[a] Determination on 0.15g sample.

[b] Determination on 0.10g samples.

[c] Standard deviation (1-sigma). These values include variations in the precision of the method, as well as differences which may be due to any inhomogeneity of the material.

[d] This value, obtained at NBS, is the average of 63 determinations on 18 rods. An average value of 2905 parts per million was obtained by 10 cooperating laboratories, two of which reported results by two different methods. For the sets of results, 5 were within the 2-sigma limit with an average of 2968, 2 others within the 3-sigma limit, and 5 were outside the 3-sigma limit. Of the latter, four sets were low.

[e] This value, obtained at NBS, is the average of 72 determinations of 7 rods at three positions. An average value of 1309 parts per million was obtained by 9 cooperating laboratories, one of which reported results by two different methods. For the sets of results, 5 were within the 1-sigma limit and all 10 were within the 2-sigma limit.

CAUTION: Oxygen determinations should be made on thoroughly and freshly cleaned samples that represent the cross-section of the rods, such as pie-shaped segments.

List of Analysts

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5. R. L. Rudolph, Crucible Steel Company of America, Midland Works, Midland, Pa.
6. S. Vigo and F. P. Valenti, U. S. Army Materials Research Agency, Watertown, Mass.
7. M. J. Miles, Henderson Technical Laboratories, Titanium Metals Corporation of America, Henderson, Nevada. (Results also were reported by the Toronto Laboratories of the Titanium Metals Corporation of America.)
8. M. W. Mallett, Thermal Chemistry Group, Battelle Memorial Institute, Columbus, Ohio.
9. J. M. Martin, National Research Corporation, Cambridge, Mass.

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W. Wayne Meinke, Chief
Office of Standard Reference Materials

(Over)

SUPPLEMENTARY INFORMATION

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 then handled only with clean forceps.
4. Analysis should be made as soon as possible after cleaning the sample.
5. Because some radial segregation has been observed, it is recommended first, a slice about 0.1 inch thick be taken that is representative of the full cross-section of the rod, and second, this slice then be quartered through the center using a jeweler's hacksaw. The resulting four segments of approximately the same weight should be suitable for analysis.

CONDITIONS FOR ANALYSIS AT NBS:

Method	Vacuum fusion
Furnace temperature	1950°C.
Furnace pressure	10^{-5} Torr.
Collection time	4 mins.
Bath material	Platinum (2-3 g)
Flux material	Platinum (Platinum-to-sample ratio = 10:1)
Carbon monoxide determination	Infrared absorption

The carefully cleaned specimen was wrapped in platinum wire (0.032 in. in diameter) that had been specially prepared to have a low oxygen content (approximately 4 ppm). The weight of platinum was chosen such that the ratio of platinum-to-sample was at least 10 to 1. A 2- to 3-g bath of platinum was used and a sample of the unalloyed titanium, treated exactly as a regular analysis-specimen, was added initially to condition the bath and apparatus.

A complete report on the testing and analysis of these materials will be published in the NBS Misc. Publ. 260 Series.

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