

# National Bureau of Standards

## Certificate of Analysis

### Standard Reference Material 358

#### Gasometric Standard for Unalloyed Zirconium

Analytical Program Performed at the Albany Research Center,  
U.S. Bureau of Mines, Albany, Oregon, by A. J. Mackie

This standard is in the form of wire, 1.2 mm (0.046 in) in diameter. It is intended for calibration of apparatus used in the determination of gases in metals.

CAUTION: Prior to use, the wire should be washed in acetone or trichloroethylene, and thoroughly dried.

SRM No.	358	
Designation	Unalloyed Zirconium	
	Certified Value <sup>a</sup>	Estimated Uncertainty <sup>b</sup>
<u>Element</u>	<u>Parts per Million by Weight, <math>\mu\text{g/g}</math></u>	
Hydrogen	107	3
Nitrogen	28	4

<sup>a</sup>The certified value listed is the *present best estimate* of the "true" value based on the cooperative program for certification.

<sup>b</sup>The estimated uncertainty listed is based on judgment and represents an evaluation of the combined effects of method imprecision, possible systematic errors among methods, and material variability for samples of 0.2 g (or more) for hydrogen, and samples of 1.0 g (or more) for nitrogen.

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  
June 16, 1980

George A. Uriano, Chief  
Office of Standard Reference Materials

(over)

PLANNING, PREPARATION, TESTING, ANALYSIS:

The wire material for this SRM was obtained from Teledyne Wah Chang, Albany, Oregon. At the request of NBS, selection of the base material was made to provide for a concentration difference for nitrogen between this SRM 358, and a companion unalloyed zirconium wire, SRM 357. In addition, SRM 358 was given a specialized heat treatment to produce a homogeneous stable hydride containing hydrogen at about the 100 ppm level. (The hydrogen content of SRM 357 is approximately 20 ppm.)

At the Albany Research Center, U.S. Bureau of Mines, Albany, Oregon, six wire positions were sampled representing the entire coil of wire. Hydrogen, nitrogen, and oxygen determinations were made in at least quadruplicate at each position. Prior to each determination, samples were washed in acetone and dried.

Hydrogen. Determinations were performed by vacuum hot extraction on samples weighing 0.20 to 0.23 g. (Note: The wire was bent to a compact shape for use in the apparatus.) The average of all results ( $n = 24$ ) was  $107.4 \pm 1.5$  ppm (1 sigma). Previously issued SRM 353, certified at  $98 \pm 5$  ppm (1 sigma), was also run ( $n = 5$ ) and yielded  $99.2 \pm 2.4$  (1 sigma).

Nitrogen. Determinations were performed by a semi-micro Kjeldahl method on samples weighing 1.1 to 1.4 g. The average of all results ( $n = 24$ ) was  $27.0 \pm 1.9$  ppm (1 sigma). SRM 360a, certified at 43 ppm, was also run ( $n = 5$ ) and yielded  $42.6 \pm 1.9$  ppm (1 sigma).

Oxygen. Determinations were performed by inert gas fusion with a chromatographic readout on samples weighing 0.13 to 0.17 g. Samples were wrapped in metal foil and all determinations were made in a nickel bath. The apparatus was calibrated each day with potassium acid phthalate standards containing 200, 300, and 400 micrograms of oxygen. The average of all results ( $n = 28$ ) was  $1100 \pm 62.4$  ppm (1 sigma). SRM 356, certified at  $1332 \pm 77$  ppm (1 sigma), was also run ( $n = 10$ ) and yielded  $1313 \pm 67.4$  (1 sigma).

The analytical work for homogeneity testing and certification was performed at the Albany Research Center by A. J. Mackie and D. H. Bollman.

Cooperative analyses on samples from one selected wire position were performed at the Oregon Metallurgical Corporation, Albany, Oregon, by A. D. Fryer; and at Teledyne Wah Chang, Albany, Oregon, by J. H. Schlewitz.

For hydrogen and nitrogen, one cooperating laboratory confirmed the results from the Albany Research Center.

Oxygen is *not* being certified at this time because of apparent systematic errors among methods (lack of agreement relative to the different reference materials used).

Additional studies are under way for oxygen in this SRM.

For information only, the following is given:

	<u>Approximate Value</u>
	ppm by weight
Oxygen	(1100)