



National Institute of Standards & Technology

# Certificate of Analysis

Standard Reference Material<sup>®</sup> 3120

Standard Solution

Germanium

Lot No. 791102

This Standard Reference Material (SRM) is intended primarily for use in calibrating instruments used in atomic spectrometry, including atomic absorption spectrometry, inductively coupled plasma optical spectrometry, and inductively coupled plasma mass spectrometry. It can also be used in conjunction with any other analytical technique or procedure where an aqueous standard solution is required. One unit of SRM 3120 consists of 50 mL of a single element solution prepared gravimetrically to contain a known amount of germanium in an approximate oxalic acid volume fraction of 10 %.

Certified Value ( $Y$ ) of Germanium: 9.49 mg/mL  $\pm$  0.05 mg/mL at 22 °C

The certified value ( $Y$ ) is based 1) on the mass of high purity germanium metal, dissolved and diluted to known volume and, 2) on x-ray fluorescence spectrometry of the resulting solution. Metallic elemental impurities in the starting material were determined by inductively coupled plasma mass spectrometry, oxygen and nitrogen by inert gas fusion and hydrogen by vacuum extraction. The material was found to contain less than 130 mg/kg total metallic impurities and less than 25 mg/kg dissolved gases. The value has been adjusted upward by 0.1 % relative, based on estimated transpiration losses of solvent through the container walls of 0.2 % relative per year. The density of the solution is 1.015 g/mL  $\pm$  0.002 g/mL at 22 °C.

The uncertainty in the certified value is calculated as

$$U = (2u_c + 0.001Y + B) \text{ mg/mL}$$

where  $u_c$  is the "combined standard uncertainty" calculated according to the ISO Guide [1]. The value of  $u_c$  is intended to represent, at the level of one standard deviation, the combined effect of uncertainty components associated with volumetric and gravimetric factors, as well as the purity of the starting material. The quantity, 0.001 $Y$ , is an allowance for transpiration of the solution through the container walls, which is estimated to be  $\pm$  0.1 % of the certified value during the one-year period of validity of the certification. The quantity,  $B$ , is an allowance for between method differences.

The combined standard uncertainty consists of a Type A component associated with replicate weighings of the starting material and Type B components due to uncertainties in the material purity, material handling, and dilution.

This SRM was prepared gravimetrically by T.A. Butler and analyzed using x-ray fluorescence by A.F. Marlow of the NIST Analytical Chemistry Division. Inductively coupled plasma mass spectrometric analysis of the starting material was performed at Elemental Research, Inc., North Vancouver, BC, Canada. Gas analyses were performed at Luvak, Inc., Boylston, MA.

The support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the Standard Reference Materials Program by N.M. Trahey.

Gaithersburg, MD 20899  
Certificate Issue Date: March 25, 1997

Thomas E. Gills, Chief  
Standard Reference Materials Program

## Procedures for Use

**Expiration of Certification:** This certification is valid for one year from the shipping date, provided the solution is kept tightly capped and stored under normal laboratory conditions. NIST will monitor the stability of representative solutions from this SRM lot, and if any changes occur that invalidate this certification, NIST will notify purchasers.

**Preparation of Working Standard Solutions:** All solutions should be brought to  $22\text{ }^{\circ}\text{C} \pm 1\text{ }^{\circ}\text{C}$  before use and all glass or plastic surfaces coming in contact with the standard must have been previously cleaned. A working standard solution can be prepared from the SRM solution by serial dilution. Each dilution should be acidified with the appropriate high purity acid in high purity water. The analyst should prepare daily working solutions from 100  $\mu\text{g/mL}$  dilutions of the original SRM solution.

## REFERENCE

- [1] *Guide to the Expression of Uncertainty in Measurement*, ISBN 92-67-10188-9, 1st Ed. ISO, Geneva, Switzerland, (1993); see also Taylor, B.N. and Kuyatt, C.E., "Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results," NIST Technical Note 1297, U.S. Government Printing Office, Washington DC, (1994).