

National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 3120

Spectrometric Standard Solution

Germanium

Batch Code 692401

This Standard Reference Material (SRM) is intended for use in atomic absorption spectrometry, inductively coupled plasma spectrometry, spectrophotometry, or any other analytical technique that requires aqueous standard solutions for calibrating instruments. SRM 3120 is a single element solution prepared gravimetrically to contain a nominal 10 mg/mL of germanium with an approximate oxalic acid volume fraction of 10 %. The certified value (Y) is based on the mass of pure metal dissolved and diluted to known volume. 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 at 22 °C is 1.016 g/mL.

Metal	Concentration (Y) (mg/mL)	Source Purity, %	Acid Volume Fraction, Approximate
Gemanium	10.00 ± 0.03	Ge metal, (99.999)*	Oxalic Acid, 10 %

^{*}This high-purity material was analyzed by optical emission spectromety and atomic absorption spectrometry and found to contain no detectable impurities.

The uncertainty in the certified value is calculated as

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

where u_c is the "combined uncertainty" calculated according to the ISO and NIST Guides [1,2]. The value 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 germanium metal. The additional 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 combined uncertainty consists of a Type A component associated with replicate weighings of the starting material and Type B components due to uncertainty in the material purity, material handling, and dilution.

SRM 3120 was prepared and analyzed by T.A. Butler; atomic emission and atomic absorption spectrometric analyses were made by J.A. Norris and T.A. Butler of the NIST Analytical Chemistry Division.

The support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the Standard Reference Materials Program by B.S. MacDonald.

Gaithersburg, MD 20899 February 20, 1996 Thomas E. Gills, Chief Standard Reference Materials Program

Procedures for Use

Stability: 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 °C \pm 1 °C before use and all glass or plastic surfaces coming into contact with the standard must have been previously cleaned. A working standard solution can be prepared from the SRM solution by serial dilution. Dilutions should be made with certified volumetric class A flasks and 5 mL or 10 mL class A pipets. All volumetric transfers of solutions should be performed using a proven analytical technique. Each dilution should be acidified with an appropriate high-purity acid and diluted to calibrated volume using high-purity water. The stability of the working standard solution will depend on the final acid concentration; therefore, care should be exercised to ensure that the final acid concentration of the dilution closely approximates that of the SRM. The analyst should prepare daily working solutions from 100 μ g/mL dilutions of the originial SRM solution.

NOTICE AND WARNING TO USERS

For some instrumental techniques, small differences in acid type and concentration between the SRM and sample may lead to erroneous results. Therefore, the same acid mixture as is listed on this SRM certificate should be used in making appropriate dilutions and working standards.

REFERENCES

- [1] Guide to the Expression of Uncertainty in Measurement, ISBN 92-67-10188-9, 1st Ed. ISO, Geneva, Switzerland, (1993).
- [2] 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, D.C., (1994).