National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material 276b

Tungsten Carbide

This Standard Reference Material (SRM) is intended as a calibrant for the determination of total carbon by combustion-thermal conductivity, combustion-infrared and combustion-gravimetric methods of analysis, and is in the form of a fine powder, which passes through a 74 μm sieve (#200). The SRM is issued in units of 75 g.

	Certified Value Percent by Weight
Total Carbon	6.10 ± 0.04

The certified value is on an as-received basis and was established through replicate determinations on 300 mg subsamples using induction-furnace combustion with infrared detection. This value was confirmed by the ensemble of data from cooperating laboratory analyses.

The uncertainty in the certified value is calculated as

$$U = 2u_c \text{ wt. \%}$$

where u_c is the "combined standard uncertainty" calculated according to the CIPM approach [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 replicate instrumental measurements, instrument calibration, and method uncertainty. Additional detail on the components of uncertainty is given in Table 1.

Notice to Users: The SRM is hygroscopic and should be kept tightly closed except when in use. Storage in a desiccator over a desiccant is recommended. While the material must be protected against absorption of moisture, drying is not recommended, since it has the potential of oxidizing free carbon. Certification is valid for five years from the date of shipment from NIST.

The overall coordination of the technical measurements leading to certification was performed under the direction of C.M. Beck II of the NIST Inorganic Analytical Research Division.

The statistical evaluation of the data was performed by K.R. Eberhardt of the NIST Statistical Engineering Division.

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

Gaithersburg, MD 20899
June 2, 1994

Thomas E. Gills, Chief
Standard Reference Materials Program

(over)

PLANNING, PREPARATION, AND ANALYSIS:

The material for this SRM was prepared and provided by Osram Sylvania, Towanda, PA, through the courtesy of J.L. Schoonover.

At NIST the material was blended in a small cone blender for one h and samples representative of the lot were taken from the bulk and bottled in an argon atmosphere for homogeneity testing. Subsequently, the remainder of the lot was bottled in an argon atmosphere and each unit was sealed under argon in an aluminized pouch to protect the sample from adsorption of moisture.

Homogeneity testing and certification analyses were performed at NIST by C.M. Beck II and at all cooperating laboratories. Each laboratory measured six samples from different portions of the lot in duplicate, except for NIST, where duplicate measurements were made on twelve portions. The testing showed no significant material variability for total carbon among the several portions measured in each laboratory.

The material was analyzed by NIST and four cooperating laboratories using combustion-thermal conductivity and combustion-infrared methods of analysis for total carbon.

Constituents other than total carbon have been determined as indicated below. These are *not certified*, but are given as additional information on the composition of the material.

Element	Percent by Weight	Method
"Free" Carbon	(.04)	Acid pretreatment, combustion - thermal-conductivity or infrared detection
Oxygen	(.08)	Inert gas fusion, infrared detection
Nitrogen	(.01)	Inert gas fusion, thermal conductivity detection

Analyses for SRM 276b were performed in the following laboratories:

National Institute of Standard and Technology, Inorganic Analytical Research Division, Gaithersburg, MD, C.M. Beck II.

Osram Sylvania Inc., Towanda, PA, R. Schoonover.

Luvak Inc., Boylston, MA, J. Flanagan.

Leco Corp., St. Joseph, MI, D. Lawrenz.

Rogers Tool Works, Rogers, AR, M.R. Goff.

Components of uncertainty are of two types. Type A which can be estimated by statistical means, and Type B which are estimated by non statistical approaches. Table 1 lists the components of uncertainty which were combined to calculate the uncertainty in certified value for total carbon in SRM 276b.

Table 1. Components of Uncertainty

Source	Standard Uncertainty (%C)	Degrees of Freedom	Type A or B
Replicate Measurements of 29 Samples of Material	0.0035	28	A
Measurement of Calibration Standard	0.0043	7	A
Uncertainty in Certified Value of Calibration Standard	0.0021	9	A
Method Uncertainty	0.02	∞	B
Combined Standard Uncertainty, u_c	0.021	>30	

REFERENCE

[1] *Guide to the Expression of Uncertainty in Measurement,* ISBN 92-67-10188-9, 1st Ed. ISO, Switzerland, 1993.