National Bureau of Standards

Certificate of Analysis

Standard Reference Material 1216

Carbon Modified Silica

This Standard Reference Material (SRM) consists of three bottles, each containing approximately one g of chemically modified microparticulate silica. SRM 1216 is intended for use in the calibration of instruments used in the determination of total elemental carbon. It may also be useful in studies involving the measurement of carbon content of metals especially when gas fusion infrared detection instruments are used.

Certified Carbon Values in SRM 1216:
Three concentration levels of carbon are provided in the SRM, designated I, II, III. The certified value for carbon in each of these samples is listed below.

Bottle	Percent Carbon
I	.70 ± 0.12
II	9.06 ± 0.24
III	17.04 ± 0.43

The uncertainties listed are two standard deviations for a single determination. It is estimated that any uncertainties due to systematic error are included within these limits.

NOTICE AND WARNINGS TO USER

Expiration of Certification: This certification is valid, within the limits certified, for one year from the date of shipment. NBS will continue to monitor these materials and substantive change in their certification will be reported to the purchaser.

Use: Prior to use, samples for analysis should be dried at 85 °C for three hours, under reduced pressure(~ 100 Pa). To minimize sample "creep" during drying, one should reduce pressure gradually by equilibrating the sample at several intermediate pressures for 5-10 minutes.

Toxicity: The toxicity of this SRM has not been defined precisely; however, this material consists of finely divided siliceous particles and as such should be treated as a potential health hazard.

Analytical determinations were performed at the Center for Analytical Chemistry, Inorganic Analytical Research Division, by R.C. Gauer, T.W. Vetter, B.I. Diamondstone, and R.M. Lindstrom.

The coordination of the technical measurements leading to certification were performed under the direction of B.I. Diamondstone and L.C. Sander.

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 W.P. Reed.

November 20, 1987
Gaithersburg, MD 20899

Stanley D. Rasberry, Chief
Office of Standard Reference Materials

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Preparation and Analysis:
SRM 1216 consists of three aliquots of porous microparticulate silica (particle diameter ~20 μm, pore diameter ~30 nm). Aliquot I consists of unmodified silica, and aliquots II and III were chemically modified by reacting with octadecyltrichlorosilane. The bare and modified silica substrates were prepared by the Separations Group, Hesperia, CA. Packaging was carried out at the National Bureau of Standards. Samples from various stages of the packaging process were analyzed using gas fusion with infrared detection. No significant differences in the concentration levels of carbon were observed among samples for each aliquot. The certification measurements on these samples were made using gas fusion with infrared detection and were corroborated by prompt gamma activation analysis.

Gas Fusion:
Samples weighing approximately 100 mg were diluted with low carbon steel flux and tungsten accelerator in order to provide proper burning conditions. The carbon was extracted from the samples by gas fusion and was converted to carbon dioxide where it was measured by an infrared detector. The values provided by this technique are the result of at least 100 individual measurements at each concentration level.

Prompt Gamma Activation Analysis (PGAA):
Samples weighing 200-400 mg were weighed into aluminum foil containers and were irradiated in a neutron beam for periods of 10-17 h. Pelletized standards of sucrose, graphite, and urea were irradiated at the same time. Corrections were made for blank and pulse pileup. Uncertainty in these carbon measurements were mainly a result of counting statistics.

Reference

Diamondstone, B.I. and Gauer, R.C., Analyst, 1986, 111, p955, Determination of Total Carbon in Biological Materials.