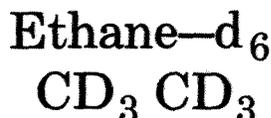


Certificate of Analysis

Standard Reference Material 2175



This Standard Reference Material is certified as a chemical standard to aid in the analysis of deuterated and partially deuterated ethane by mass spectrometric techniques.

Chemical purity greater than 99.9 mole percent

Isotopic purity 99.8 ± 0.05 atom percent deuterium

This ethane-d₆ was prepared by photolyzing acetone-d₆ (99.8 ± 0.1 atom percent deuterium) at approximately 90 °C and 2.4 x 10⁴ N/m² (180 torr) with light at essentially 300 nm wavelength. In order to insure a high isotopic purity of ethane and to prevent secondary photolysis of the reaction products, the conversion was kept low (~ 25%). The reaction vessel consisted of 16 high-intensity lamps which surrounded the three liter reaction cell. A high intensity of light favors the dimerization of methyl radicals to form ethane over the abstraction reaction to form methane. After photolysis the products were distilled from the undecomposed acetone at -130 °C and the non-condensable products were pumped away at -196 °C. The ethane-d₆ was purified from the remaining products by trapping at -196 °C the effluent gas from a gas chromatograph equipped with a silica gel column and then introduced into a storage vessel by low temperature distillation. The chemical purity is greater than 99.9 mole percent. The isotopic purity is 99.8 ± 0.5 atom percent deuterium (98.9 ± 0.1 mole percent C₂D₆; 1.1 ± 0.1 mole percent C₂D₅H).

The chemical purity was determined by use of a gas chromatograph equipped with a flame ionization detector and a 6 foot alumina column. The only detectable impurity was propane (~ 0.002 mole percent). The isotopic purity was determined by mass spectroscopy. The pattern shown on the reverse was obtained using a Consolidated Model 21-101 mass spectrometer at an ionizing current of 50 microamperes. This pattern has been corrected for C₁₃ abundance.

The Standard Reference Material was prepared, purified, and characterized by P. J. Ausloos, R. E. Rebbert, and R. M. David of the NBS Radiation Chemistry Section.

Washington, D. C. 20234
July 25, 1968

W. Wayne Meinke, Chief
Office of Standard Reference Materials

Mass Spectrometer Pattern for Standard Reference Material No. 2175, Ethane-d₆

m/e	Relative Abundance	m/e	Relative Abundance
12	6.2	26	20.0
14	14.0	27	0.7
15	0.4	28	119.6
16	29.8	29	1.0
17	0.9	30	153.4
18	30.7	31	3.6
19	.2	32	565.9
20	.3	33	0.5
24	3.6	34	83.4
25	0.2	35	1.1
		36	100.0