

Certificate of Analysis
Standard Reference Material 2176
Propane-1,1,1-d₃
CH₃CH₂CD₃

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

Chemical purity greater than 99.9 mole percent

Isotopic purity 99.4 ± 0.2 mole percent CD₃C₂H₅

This propane-1,1,1-d₃ was prepared by photolyzing a mixture (1:8) of 3-pentanone and acetone-d₆ (99.8 ± 0.1 atom percent deuterium) at approximately 90 °C and a total pressure of 2.7 x 10⁴ N/m² (200 torr) with light essentially at 300 nm wavelength. The conversion was kept low (~ 10%) in order to prevent secondary photolysis of the reaction products. This, as well as the high ratio of acetone-d₆ to 3-pentanone, insures a high isotopic purity of propane-1,1,1-d₃. The reaction vessel consisted of 16 high-intensity lamps which surrounded the three liter reaction cell. 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 propane-1,1,1-d₃ was purified from the remaining products by trapping at -196 °C the effluent gas from a gas chromatograph equipped with a silica gel column. It was further purified by low temperature distillation into a storage vessel. The chemical purity is greater than 99.9 mole percent. The isotopic purity is 99.4 ± 0.2 mole percent CD₃C₂H₅; 0.6 ± 0.2 mole percent CD₂HC₂H₅.

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 impurities were approximately 0.03 mole percent ethane and approximately 0.01 mole percent ethylene. The isotopic purity was calculated from the purity of the starting materials. 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.

This 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
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W. Wayne Meinke, Chief
Office of Standard Reference Materials

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Mass Spectrometer Pattern for Standard Reference Material No. 2176, Propane-1,1,1-d₃

m/e	Relative Abundance	m/e	Relative Abundance
24	1.1	37	7.3
25	4.1	38	13.4
26	28.6	39	21.0
27	87.8	40	39.8
28	171.9	41	35.0
29	250.5	42	16.7
30	109.2	43	31.9
31	42.0	44	28.4
32	221.0	45	21.2
34	0.9	46	106.9
36	2.6	47	100.0