Technical Bulletin
AL-181

Deuterium Oxide,

100.0 atom % D, low in paramagnetic impurities, minimum isotopic purity 99.96 atom % D

revised 6/99
1 page

Description

Molecular formula	D_2O
Formula weight	20.03
bp	101.4 °C
n^{20}_D	1.3280
density	1.107

Deuterium oxide is a distilled-in-glass grade of heavy water of high isotopic purity, intended for use in relaxation-time studies and other applications where the presence of trace amounts of metal ions is undesirable. The high isotopic purity also enables the user to exchange labile protons to the extent that water-saturation techniques are not necessary in most cases.

Production and Packaging

Deuterium oxide enriched to high isotopic purity is fractionated in a glass apparatus under argon. It is packaged in USP Type 1 borosilicate glass serum bottles which have been leached for an extended period with ethylenediaminetetraacetic acid (EDTA), disodium salt. The septum closure is of silicone rubber with a bonded inner face of PTFE.

Analysis

Isotopic Purity

The isotopic purity is determined utilizing ^1H NMR. This value remains constant with prolonged storage.

Paramagnetic Impurities

Since the list of possible paramagnetic impurities is long, direct analyses for elements are not performed. Instead, relaxation times are measured before and after degassing the sample. At present, the effect of long-term storage on relaxation time is not known with certainty.

The lot-specific analytical data appears on each bottle of product. These data vary from lot to lot; a typical lot analysis is:

99.99 atom % D
T_1 24.3 ± 0.4 sec
T_1 (after degassing) 46.9 ± 1.1 sec

Handling Procedures

To Maintain Isotopic Purity

To obtain the maximum benefit of the high deuterium enrichment of this product, rigorous handling procedures are necessary. All transfers should be made in a glove box, and the water adsorbed on the syringe and NMR tube should be exchanged with deuterium oxide. This is accomplished by repeated equilibration with D_2O, allowing 24 hours for each equilibration. It is suggested that three equilibrations be performed, especially if long FT runs are anticipated.

To Eliminate Paramagnetic Impurities

Any contact of the deuterium oxide (or the material to be studied) with paramagnetic materials must be strictly avoided. Perhaps the most convenient method of sample withdrawal is with an all-glass syringe using a Teflon® needle with a Kel-F® hub. Both needle and syringe are available from Aldrich.

Glassware should be treated to remove metals. NMR tubes and syringes, in particular, usually contain substantial amounts of iron on the surface, since they are commonly manufactured by vacuum shrinking upon iron mandrels. The glassware can be leached with 0.1*M* EDTA, disodium salt for a long period, followed by thorough rinsing with deuterium oxide to remove metals.

Related Products and Equipment

19,234-1	**Deuterium oxide,** 100.0 atom % D, low in paramagnetic impurities, minimum isotopic purity 99.96 atom % D
10,631-3	**Ethylenediaminetetraacetic acid, disodium salt dihydrate,** 99+%
Z10,190-7	**Syringe,** interchangeable barrels and plungers, glass-tipped, with Luer fitting, 2 mL
Z11,731-5	**Syringe needle,** Teflon®, 20-gauge, with Kel-F® Luer hub, 12 inches long

Teflon is a registered trademark of E.I. du Pont de Nemours & Co.
Kel-F is a registered trademark of 3M Company.

ALDRICH®

Organics and Inorganics for Chemical Synthesis

1001 West Saint Paul Ave., Milwaukee, WI 53233
Telephone 414-273-3850 • 800-231-8327
Fax 414-273-4979 • 800-962-9591
Web http://www.sigma-aldrich.com
E-mail aldrich@sial.com

© 1999 Sigma-Aldrich Co. Printed in the USA. Aldrich is a member of the Sigma-Aldrich Family. Sigma-Aldrich warrants that its products conform to the information contained in this and other Sigma-Aldrich publications. Purchaser must determine the suitability of the product for its particular use. See reverse side of invoice or packing slip for additional terms and conditions of sale.