A SIMPLE AND EFFICIENT PREPARATION OF 2,2,6,6- TETRA - DEUTERIOCYCLOHEXANONE.

Received on May 23, 1973

The preparation of 2,2,6,6-tetradeuteriocyclohexanone is reported [1] using a 10 % DCl-D_3PO_4 exchange solution, generated from PCl_5 and D_2O. Another procedure[2] uses a K_2CO_3-D_2O exchange solution,which results in a significant amount of base-catalyzed aldol condensation. An alternate acid-catalyzed procedure for the preparation of 2,2,6,6-tetradeuteriocyclohexanone is reported here, utilizing a p-toluenesulfonic acid-d-DCl exchange solution, generated from p -toluenesulfonyl chloride (TsCl) and D_2O. TsCl is less hygroscopic than PCl_5, and can lead to an exchange medium of greater isotopic (deuterium) purity. Thus, 1 gram of cyclohexanone is treated twice for 24 hr periods with 1 gram of TsCl and 20 ml of D_2O at reflux under an inert atmosphere. Short-path distillation of the concentrated pentane extract affords 2,2,6,6-tetradeuteriocyclohexanone, which shows 96%-d_4 and 4% -d_3 by mass spectral analysis. Since TsCl does not hydrolize very quickly in water at 25° and p-toluenesulfonic acid is very water-soluble, the TsCl can be washed before use with D_2O. This will lead to an exchange medium of even greater isotopic purity. The use of TsCl to generate acid-catalyzed exchange solutions should prove to be generally useful because of its advantages over other reagents.

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