Molecules 2000, 5, M131

Methane-diphosphonic Acid Tetraethyl Ester

Didier Villemin and Frédéric Thibault

ISMRA, 6 boulevard Maréchal Juin, F-14050 Caen, France; a) UMR 6507 CNRS, E-mail: Didier.Villemin@ismra.fr b) UMR 6506 CNRS

Received: 16 December 1999 / Accepted: 17 January 2000 / Published: 24 January 2000

Keywords: phosphonate, bioactive compound, material, alkene precursor.

Methanediphosphonic acid tetraethyl ester is a useful intermediate for the synthesis of phosphonoalkenes [1] or diphosphonoalkenes [2]. Methanediphosphonic acid tetraethyl ester is also a precursor of bioactive compounds [3] and hybrid organic-inorganic materials [4]. Many synthetic methods are available in the literature [5], but none of them is very convenient and commercial methanediphosphonic acid tetraethyl ester [6] remains expensive. We have improved the method described by Horni [7]. Although our method is long (a 60 day reaction at ambient temperature), the yield is excellent (89%) and the method is simple and convenient in comparison with other methods of literature.

A solution of sodium ethoxide was prepared by adding sodium metal (3 mol, 69 g) in portions to absolute ethanol (900 ml). Diethyl phosphite (3 mol, 386 ml) was added with stirring, and the mixture stirred for 1 h at ambient temperature. The excess of alcohol was evaporated using a rotary evaporator. Dichloromethane (1000 ml) was added, and the mixture was agitated for 2 months in a closed bottle. The mixture was washed with water (500 ml) and the organic phase was dried over magnesium sulfate. The solvent was evaporated, and the product was obtained as a colourless liquid (89% yield) by distillation [Bp : 128 °C(0.02)].

RN : 1660-94-2

$^1$H NMR (CCl$_4$): 1.35 (t, J = 7 Hz, 12 H, P-O-C-CH$_3$) 2.45 (t, J = 21 Hz, 2 H, P-CH$_2$-P) 4.15 (dq, J$^1$ = J$^2$ = 7 Hz, 8 H, P-O-CH$_2$).

$^{13}$C NMR (CDCl$_3$): 16.4 (P-O-C-CH$_3$), 25.7 (t, J = 137 Hz, P-C-P) 62.55 (P-O-C).

$^{31}$P NMR (CDCl$_3$): 19.05.

IR (film) : 1250 (P=O), 1020 (P-O-C), 960, 820.

References


*Sample Availability*: Available from the authors and from MDPI.

©2000 MDPI. All rights reserved. *Molecules* website [www.mdpi.org/molecules/](http://www.mdpi.org/molecules/).