

LUPININE ALKYL METHYLPHOSPHONATES
AND LUPININE DIETHYL PHOSPHOROTHIOATE

A. A. Abduvakhabov, Kh. A. Aslanov,
A. S. Sadykov, and K. Inoyatova

UDC 1 947

In order to determine their anticholinesterase activity, we have prepared phosphoric acid esters of lupinine. The lupinine was isolated by the sulfuric acid method from technical "anabasin sulfate" [1]; the alkyl methylphosphonochloridates and potassium diethyl phosphorothioate were obtained by published methods [2-4].

Ethyl Lupinine Methylphosphonate. Lupinine (0.02 mole) was added to a dispersion made by Brühl's method [5] of 0.02 g-atom of sodium in 70 ml of absolute benzene. The mixture was boiled until the sodium had dissolved completely, and then 0.02 mole of ethyl methylphosphonochloridate was added over 5 min and the mixture was boiled again for 4 h. Then it was decomposed with water and extracted with benzene. After drying and the distillation of the solvent, the ethyl lupinine methylphosphonate $C_{13}H_{26}NO_3P$ was distilled in vacuum. Yield 30%, bp $115^\circ C$ at 4×10^{-2} mm, d_4^{20} 1.0856, n_D^{20} 1.4835.

n-Butyl lupinine methylphosphonate $C_{15}H_{30}NO_3P$ was obtained similarly. Yield 35%, bp $132-133^\circ C$ at 1 mm, d_4^{20} 1.0596, n_D^{20} 1.4800.

O,O-Diethyl S-Lupinine Phosphorothioate. With stirring, an ethanolic solution of 4 g (0.0174 mole) of bromolupinane [6] in 10 ml of ethanol was added to 3.34 g (0.0174 mole) of potassium diethyl phosphorothioate in 15 ml of absolute ethanol, and the reaction mixture was boiled for 5 h. After 12 h the product was worked up in the usual way and distilled in vacuum. Yield 62%, bp $113-114^\circ C$ at 10^{-2} mm, d_4^{20} 1.1217, n_D^{20} 1.5052, $C_{14}H_{28}NO_3PS$.

The methiodides of O,O-diethyl S-lupinine phosphorothioate $C_{10}H_{18}NS(O)P(OC_2H_5)_2 \cdot CH_3I$ (yield 90%, mp $126^\circ C$) and of n-butyl lupinine methylphosphonate $C_{10}H_{18}NO(O)P(OC_4H_9)CH_3 \cdot CH_3J$ (yield 87%, mp $150^\circ C$) were obtained by the action of methyl iodide.

LITERATURE CITED

1. A. S. Sadykov, The Chemistry of the Alkaloids of *Anabasis aphylla* [in Russian], Tashkent, 1956.
2. Methoden der Organischen Chemie, Phosphorverbindungen, Bd. XII/2, Berlin, 1963.
3. E. N. Tsvetkov and M. I. Kabachnik, Reaktsii i Methody Issledovaniya Organicheskikh Soedinenii, 13, 269, 1964.
4. G. Pesin and A. M. Khaletskii, ZhOKh, 31, 2511, 1961.
5. I. W. Brühl, Ber., 35, 3627, 1902.
6. G. R. Clemo, W. Morgan, and R. Raper, J. Chem. Soc., 1937, 965.

Lenin Tashkent State University. Translated from Khimiya Prirodykh Soedinenii, No. 6, pp. 771-772, November-December, 1970. Original article submitted July 15, 1970.

© 1973 Consultants Bureau, a division of Plenum Publishing Corporation, 227 West 17th Street, New York, N. Y. 10011. All rights reserved. This article cannot be reproduced for any purpose whatsoever without permission of the publisher. A copy of this article is available from the publisher for \$15.00.