REACTION OF TETRAPHOSPHORUS TRISULFIDE WITH THE

DIETHYLDITHIOACETAL OF BENZALDEHYDE

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We have found that P_4S_3 reacts with the diethyldithioacetal of benzaldehyde taken in 1:6 ratio in a sealed tube at 200°C over 12 h to form previously unreported 5-(1-ethylthiobenzyl)-1-ethylthiobenzylethyldithiophosphinate (I) and S_3S' -diethyl-1-ethylthiobenzyltrithiophosphonate (II) in 50.8% yield.

SC(SEt)HPh

 $\begin{array}{c} P_4S_3 + PhCH(SEt)_2 \rightarrow EtP(S)C(SEt)HPh + (EtS)_2P(S)C(SEt)HPh \\ (I) & (II) \end{array}$

In addition, triethyl tetrathiophosphate (III) (13% yield), ethyl benzyl sulfide (IV) (21.4% yield), and diethyl sulfide (V) (33% yield) were also isolated from the reaction mixture. Products (I) and (II) were isolated on a film evaporator, while (III)-(V) were obtained upon fractionation. The physical constants of (III)-(V) corresponded to reported data [1-4].

<u>S-(1-Ethylthiobenzyl)-1-ethylthiobenzylethyldithiophosphinate (I)</u> was obtained in 35.7% yield. The temperature of the evaporator spiral was $220-225^{\circ}C$ (0.002 mm), $nD^{2\circ}$ 1.6419. IR spectrum (v, cm⁻¹): 3090, 3064, 3032 (arom. =C-H), 1600, 1494 (arom. C=C), 645 (P=S), 515 (P-S). ³¹P NMR spectrum at 10.2 MHz relative to 85% H₃PO₄: 85 ppm. PMR spectrum for the diastereomer mixture at 60 MHz in CC1₄ (δ , ppm, J, Hz): 0.84 d. t (CH₃CP, ³J_{HH} = 7.5, ³J_{HP} = 25.0), 1.20 t (CH₃CS, ³J_{HH} = 7.0), 1.53-3.00 m (CH₂S), δ_1 , 4.00, δ_2 4.10 two d (CHP, ²J_{HP} = 12.0 and 11.5), δ_1 4.92, δ_2 4.95 two d (CHSP, ³J_{HP} = 15.0 and 13.0). Found, %: C 56.20, H 6.33, P 7.51, S 30.08. M⁺ 426.0746 (obtained on an MKh-1310 mass spectrometer). C₂₀H₂₇PS₄. Calculated, %: C 56.30, H 6.38, P 7.26, S 30.06. M 426.0733.

LITERATURE CITED

- V. A. Al'fonsov, I. S. Nizamov, E. S. Batyeva, and A. N. Pudovik, Zh. Obshch. Khim., 56, 709 (1986).
- G. M. Kosolapoff and L. Maier, Organic Phosphorus Compounds, Vol. 7, New York (1976), p. 791.
- R. D. Obolentsev and N. G. Marina, in: The Chemistry of Organosulfur Compounds in Petroleum and Petroleum Products [in Russian], Vol. 8, Izd. Vysshaya Shkola, Moscow (1968), p. 103.
- 4. Dictionary of Organic Compounds, Vol. 2, Oxford Univ. Press, NJ (1965).

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