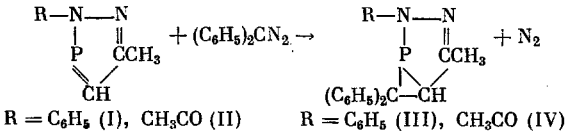
CYCLOADDITION REACTIONS TO 4-METHYL-2-PHENYL- AND 2-ACETYL-4-METHYLDIAZAPHOSPHOLES WITH THE PARTICIPATION OF DIPHENYLDIAZOMETHANE

B. A. Arbuzov and É. N. Dianova,

UDC 542.91:547.1'118

Using as examples 4-methyl-2-phenyl- and 2-acetyl-4-methyldiazaphospholes ([1] and [2], respectively) and diphenyldiazomethane we have shown for the first time the possibility of cycloaddition at a P=C bond of a doubly-coordinated phosphorus atom



R = C_6H_5 (I), CH_3CO (II) R = C_6H_5 (III), CH_3CO (IV)

The structures of (III) and (IV) were shown by elementary analyses, ^1H and ^{31}P NMR spectra, and IR spectra.

(III), mp 135-136°C (from C_2H_5OH), R_f 0.35 (C_6H_6), δ_{31P} (CH_2Cl_2) 80 ppm. PMR spectrum (CCl_4, δ, ppm): 2.181 (3 H, CH_3), 3.84 (1H, $^2J_{PH}$ = 20 Hz), 7.00 (15 H, 3 C_6H_5). IR spectrum (ν, cm^{-1}): 3065, 3030 w, 1595, 1500 s, 770 m, 755 s, 721 m, 705 s (C_6H_5); 1575 m (C=N); band at 1300 v.s. absent (pulsation vibrations of a diazaphosphole ring). Found: C 77.30; H 5.71; N 8.10; P 9.01%. $C_{22}H_{19}N_2P$. Calculated: C 77.16; H 5.59; N 8.18; P 9.05%.

(IV), mp 140-146°C, R_f 0.45 (acetone), δ_{31P} (CH_2Cl_2) 93 ppm. PMR spectrum (CCl_4, δ, ppm): 1.865 (3 H, CH_3CO), 2.115 (3H, CH_3); 3.59 (1 H, $^2J_{PH}$=20 Hz), 7.126 (10 H, 2 C_6H_5). IR spectrum (ν, cm^{-1}): 3060, 3025 w, 1605, 1490, 770, 725 m, 710 s (C_6H_5); 1665, 1650 s (C=O); 1575 w (C=N); no band at 1280 v.s. Found: C 70.06; H 5.56; N 8.69; P 9.85%. $C_{18}H_{17}N_2PO$. Calculated: C 70.11; H 5.55; N 9.08; P 10.05%.

LITERATURE CITED

1. N. I. Shvetsov-Shilovskii, N. P. Ignatova, and N. N. Mel'nikov, Zh. Obshch. Khim., 40, 1501 (1970).
2. N. P. Ignatova, N. N. Mel'nikov, and N. I. Shvetsov-Shilovskii, Khim. Geterosikl. Soedin., 753 (1967).

A. M. Butlerov Chemical Institute. V. I. Ul'yanova-Lenin Kazan State University. Translated from Izvestiya Akademii Nauk SSSR, Seriya Khimicheskaya, No. 6, pp. 1453-1454, June, 1977. Original article submitted March 1, 1977.