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(Diazoalkyl)phosphoranes from [4 + 1]-Cycloadditions of Tetrahalo-orthobenzoquinones to (Diazoalkyl)phosphanes [1]

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ABSTRACT

The (diazoalkyl)phosphanes **4a–e** *undergo [4 + 1]-cycloaddition reactions with the tetrahalo-orthobenzoquinones* **5a,b** *to furnish the previously unknown (diazoalkyl)phosphoranes* **6a–h**. *The structure of product* **6h** *has been investigated by X-ray crystallography.*

INTRODUCTION

Aliphatic diazo compounds can be prepared by two synthetic methods. In the first method, the diazo groups are introduced systematically onto an appropriately functionalized hydrocarbon skeleton, whereas, in the second method, the diazo carbon atom ("electrophilic diazoalkane substitution" [2]) or a more distant position in the carbon chain is modified (for reviews, see Refs. [3]–[5]).

The present communication describes an extension of the second method starting from the (diazoalkyl)phosphanes **3** which are, in turn, easily accessible through electrophilic diazoalkane substitution reactions [2] of the lithiated diazo compounds **1** with the chlorophosphanes **2** [6]. The subsequent alterations in these cases take place at the $\lambda^3\sigma^3$-phosphorus atoms, comprising their transformation into $\lambda^5\sigma^5$-phosphorus atoms, and do not effect the diazo groups.

RESULTS AND DISCUSSION

When the (diazoalkyl)phosphanes **4a–e** are mixed with equimolar amounts of the tetrahalo-orthobenzoquinones **5a,b** in diethyl ether, rapid reactions take place even at −78°C, as can be observed by the decoloration of the hetero-1,3-diene. The (diazoalkyl)phosphoranes **6a–h** are formed by a [4 + 1]-cycloaddition of the electron-poor reaction partner to the phosphorus atom of **4**. Under identical reaction conditions, analogous reactions between silylated diazomethylphosphanes (**4**; Me_3Si in place of *t*Bu [6]) and **5a,b** were completely unselective.

[4 + 1]-Cycloaddition reactions of 1,3-dienes and heterodienes with phosphanes are well known in principle [7–9]. Orthobenzoquinones occupy a special position here, because the regeneration of the aromatization energy is most certainly the driving force behind their cycloaddition potential [10]. With the exception of **6a** (see Table 4), all of the (diazoalkyl)phosphoranes **6a–h** are yellow to orange crystalline substances and their compositions have been confirmed by elemental analysis.

The IR spectra of **6a–h** reveal that the diazo moiety of **4** does not participate in the reactions with the orthobenzoquinones **5**; intense diazo valency vibrations are still observed at 2020–2085 cm^{-1} [11]. Furthermore, the retention of the diazo car-

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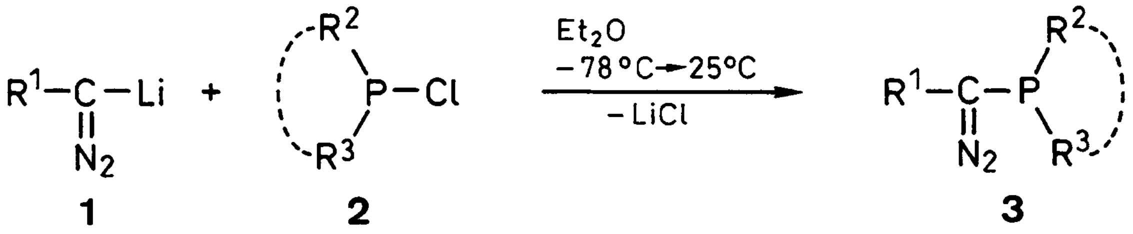
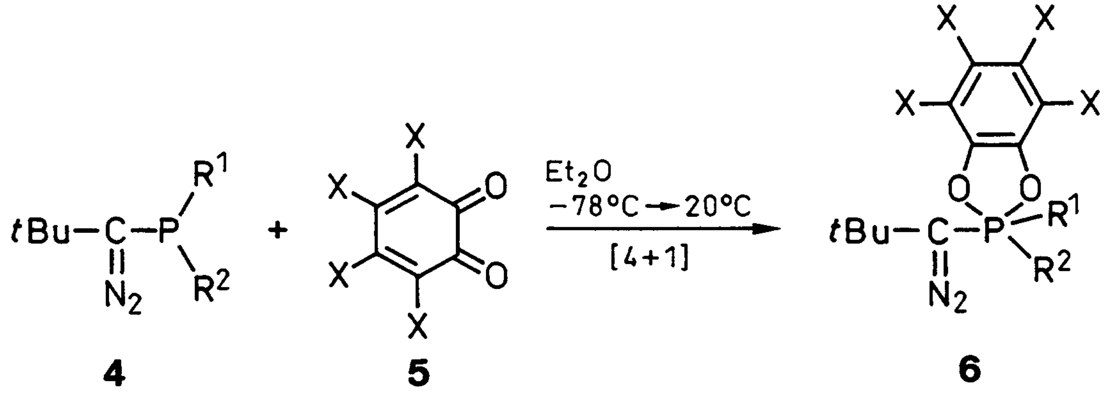


FORMULA 1



4	a	b	c	d	e
R^1	*i*Pr	*t*Bu	Me	NEt$_2$	N*i*Pr$_2$
R^2	*i*Pr	*t*Bu	NEt$_2$	NEt$_2$	N*i*Pr$_2$

5	a	b
X	Cl	Br

6	a	b	c	d	e	f	g	h
R^1	*i*Pr	*t*Bu	Me	NEt$_2$	N*i*Pr$_2$	*t*Bu	NEt$_2$	N*i*Pr$_2$
R^2	*i*Pr	*t*Bu	NEt$_2$	NEt$_2$	N*i*Pr$_2$	*t*Bu	NEt$_2$	N*i*Pr$_2$
X	Cl	Cl	Cl	Cl	Cl	Br	Br	Br

FORMULA 2

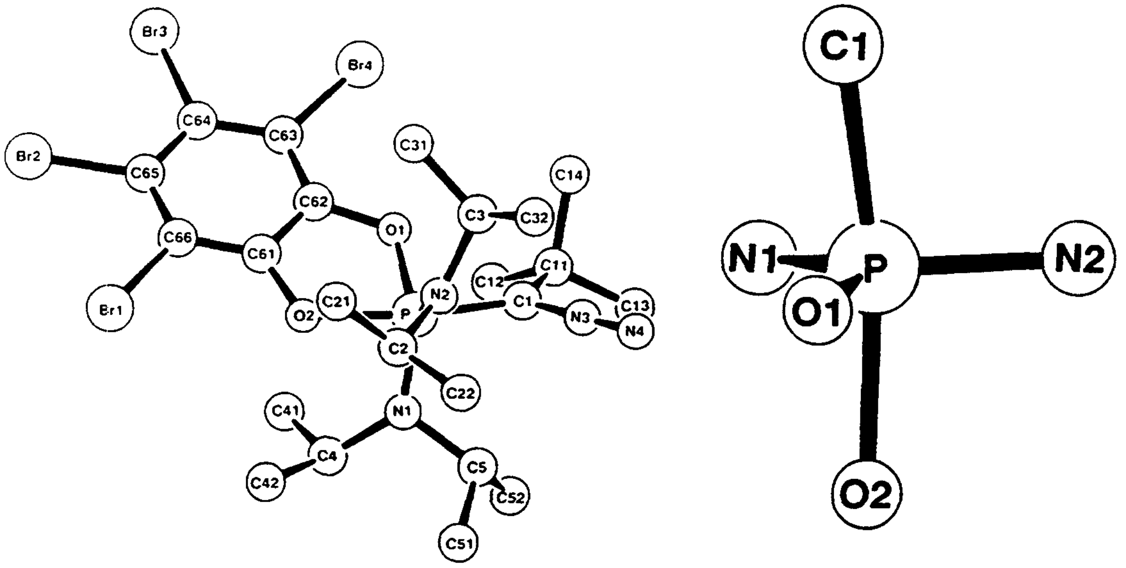


FIGURE 1 (**a**) RSA plot of **6h** (Schakal [20]. (**b**) Substituent arrangement around the central phosphorus atom of **6h**.

bon atom is unequivocally confirmed by the ^{13}C NMR signals in the region 50.8 to 57.5 ppm. These signals are split into doublets by ${}^{1}J$(C,P) couplings of 63.0–237.0 Hz [12]. In comparison to the (diazoalkyl)phosphanes **4** (δ = 49.7–52.5, ${}^{1}J$(C,P) = 31.6–76.0 Hz [6]), the pronounced increases in the carbon/phosphorus coupling constants on going from $\lambda^3\sigma^3$ to $\lambda^5\sigma^5$ phosphorus atoms are apparent; in the cases of the amino-substituted compounds **6c–e**, **g–h**, extremely large values of 177.0–237.0 Hz are observed (see also Table 1).

From the results of the X-ray crystal structure analysis of **6h** (see below), it can be predicted that the (diazoalkyl)phosphoranes should each exhibit signals for six magnetically different aromatic carbon atoms in their ^{13}C NMR spectra. However, only three sharp singlets are in fact observed and are assigned to the carbon atom pairs C1/C6, C2/C5, and C3/C4 (see Table 1); product **6h** represents an exception, because the C2/C5 signal is split by a C,P coupling (see Table 1). This equivalence of the aromatic carbon atoms may be attributed to a rapid, in terms of the NMR time scale, dynamic exchange process [13]. Attempts to prove this by low temperature measurements of the spectrum of **6h** (T = 250 K, toluene-d_8) have not yet been successful. The further NMR spectroscopic data for compounds **6a–h** are listed in Table 1 and require no further interpretation.

The crystal structure analysis of **6h** finally confirmed the cycloaddition of the orthobenzoquinones **5** to the phosphorus atoms of the (diazoalkyl)phosphanes **4**. An RSA (SCHAKAL) plot of the molecule of **6h** is shown in Figure 1a, selected bond lengths and angles are listed in Table 2, and positional parameters of the atoms of **6h** are given in Table 3.

Compound **6h** has a strongly distorted trigonal bipyramidal structure about its central phosphorus atom as a result of the bulky substituents (R^1 = R^2 = NiPr$_2$) and/or their steric interactions. This is also reflected both in the differing basal angles (113.8, 131.2, and 114.4°, see Table 2) and the deviations (13.2°) of the two axial bonds from linearity (C1-P-O2 = 166.8°). A comparison of the bond lengths P-O1 and P-O2 [1.683(4) and 1.825(4) Å, respectively] demonstrates clearly that one equatorial (O1) and one axial (O2) position are occupied by oxygen atoms originating from the orthobenzoquinone. The remaining two equatorial positions are taken by the amino groups, as confirmed by the almost identical bond lengths [1.657(5) and 1.672(5) Å]. Thus, the diazoalkyl group must be in the remaining axial position (see Figure 1b). In contrast to the values for comparable O-substituted phosphoranes [14], the axial and equatorial P–O bonds of **6h** are appreciably lengthened. The P-O2 bond length [1.825(4) Å] is very close to the sum of the covalent radii of phosphorus and oxygen (ca. 1.84 Å), and this must be taken as an indication for a rather weak bond between these two atoms. When the crystal structure data of **6h** are compared with those of **4e** [6], it is seen that the oxidative increase in the coordination number at phosphorus is accompanied by an opening of the P-C1-C11 bond angle by 8.4°. In addition, the P–C1, C1–N3, and N3–N4 bond lengths are increased while the linearity of the CN$_2$ group remains unchanged [15].

CONCLUSIONS

The preparation of the previously unknown (diazoalkyl)phosphoranes **6a–h** has opened a new

TABLE 1 IR and NMR Data of the (Diazoalkyl)phosphoranes **6a–h**

Compound	*IR (cm^{-1})* $\nu_{C=N_2}$	*^{1}H NMR ($CDCl_3$), δ, J (Hz)*	*^{13}C NMR ($CDCl_3$), δ, J (Hz)*	*^{31}P NMR ($CDCl_3$), δ*
6a	2020	1.20 (dd, 6H, ^{3}J(P,H) = 7.2, ^{3}J(H,H) = 7.0, CH-C*H*$_3$), 1.42 (dd, 6H, ^{3}J(P,H) = 6.0, ^{3}J(H,H) = 7.0, CH-C*H*$_3$), 1.27(s, 9H, *t*Bu), 2.3–2.8 (m, 2H, C*H*-CH$_3$)	17.9 (d, ^{2}J(P,C) = 4.6, CH-*C*H$_3$), 18.1 (d, ^{2}J(P,C) = 5.3, CH-*C*H$_3$), 30.7 (s, C(*C*H$_3$)$_3$, 33.3 (d, ^{2}J(P,C) = 1.6 (*C*(CH$_3$)$_3$), 37.5 (d, ^{1}J(P,C) = 82.5, *C*H-CH$_3$), 57.5 (d, ^{1}J(P,C) = 84.5, C=N$_2$), 114.2 (d, ^{3}J(P,C) = 9.0, C-2/C-5$_{arom}$), 122.1 (s, C-3/C-4$_{arom}$), 143.3 (s, C-1/C-6$_{arom}$)	22.7
6b	2020	1.38 (d, 18H, ^{3}J(P,H) = 4.2, P*t*Bu), 1.61 (s, 9H, C*t*Bu)	29.4 (s, P-C(*C*H$_3$)$_3$), 29.9 (s, C-C(*C*H$_3$)$_3$), 30.1 (d, ^{2}J(P,C) = 9.0, C(*C* (CH$_3$)$_3$), 35.6 (s, P-*C*(CH$_3$)$_3$), 50.8 (d, ^{1}J(P,C) = 74.0, C=N$_2$), 117.2 (s, C-2/C-5$_{arom}$), 121.3 (s, C-3/C-4$_{arom}$), 144.5 (s, C-1/C-6$_{arom}$)	52.0
6c	2085	1.07 (t, 6H, ^{3}J(H,H) = 7.0, CH$_2$-C*H*$_3$), 1.31 (s, 9H, *t*Bu), 2.61 (d, 3H, ^{3}J(P,H) = 14.0, PMe), 3.1–3.7 (m, 4H, C*H*$_2$-CH$_3$)	13.2 (d, ^{3}J(P,C) = 2.9, CH$_2$-*C*H$_3$), 19.7 (d, ^{1}J(P,C) = 110.6, P-CH$_3$), 29.4 (s, C(*C*H$_3$)$_3$), 33.2 (d, ^{2}J(P,C) = 8.4, *C* (CH$_3$)$_3$), 40.9 (d, ^{2}J(P,C) = 2.2, *C*H$_2$-CH$_3$), 52.4 (d, ^{1}J(P,C) = 177.0, C=N$_2$), 119.4 (s, C-2/C-5$_{arom}$), 120.2 (s, C-3/C-4$_{arom}$), 146.6 (s, C-1/C-6$_{arom}$)	28.3
6d	2065	1.02 (t, 12H, ^{3}J(H,H) = 7.0, CH$_2$-C*H*$_3$), 1.28 (s, 9H, *t*Bu), 3.0–3.6 (m, 8H, C*H*$_2$-CH$_3$)	12.5 (s, CH$_2$-*C*H$_3$), 28.4 (s, C(*C*H$_3$)$_3$), 33.0 (d, ^{2}J(P,C) = 8.0, *C*(CH$_3$)$_3$), 40.0 (s, *C*H$_2$-CH$_3$), 52.6 (d, ^{1}J(P,C) = 237.0, C=N$_2$), 118.2 (s, C-2/C-5$_{arom}$), 122.4 (s, C-3/C-4$_{arom}$), 147.9 (s, C-1/C-6$_{arom}$)	48.9
6e	2034	1.16, 1.21 (each d, each 12H, ^{3}J(H,H) = 7.0, CH-C*H*$_3$), 1.20 (s, 9H, *t*Bu), 4.10 (sept, 4H, ^{3}J(H,H) = 7.0, C*H*-CH$_3$)	23.7 (d, ^{3}J(P,C) = 3.9 CH-*C*H$_3$), 24.3 (d, ^{3}J(P,C) = 4.7, CH-*C*H$_3$), 28.8 (s, C(*C*H$_3$)$_3$), 34.2 (d, ^{2}J(P,C) = 7.0, *C*(CH$_3$)$_3$), 48.8 (d, ^{2}J(P,C) = 5.0, *C*H-CH$_3$), 53.1 (d, ^{1}J(P,C) = 219.0, C=N$_2$), 119.3 (s, C-2/C-5$_{arom}$), 119.4 (s, C-3/C-4$_{arom}$), 145.9 (s, C-1/C-6$_{arom}$)	7.3
6f	2021	1.33 (d, 18H, ^{3}J(P,H) = 2.7, P*t*Bu), 1.56 (s, 9H, C*t*Bu)	26.1 (s, C-C(*C*H$_3$)$_3$), 26.8 (d, ^{2}J(P,C) = 4.0, *C*(CH$_3$)$_3$), 29.6 (s, P-C(*C*H$_3$)$_3$), 34.9 (s, P-*C*(CH$_3$)$_3$), 51.4 (d, ^{1}J(P,C) = 63.0, C=N$_2$), 108.3 (s, C-2/C-5$_{arom}$), 117.2 (s, C-3/C-4$_{arom}$), 144.6 (s, C-1/C-6$_{arom}$)	51.1
6g	2064	1.05 (t, 12H, ^{3}J(H,H) = 7.0, CH$_2$-C*H*$_3$), 1.32 (s, 9H, *t*Bu), 3.0–3.6 (m, 8H, C*H*$_2$-CH$_3$)	12.5 (d, ^{3}J(P,C) = 3.3, CH$_2$-*C*H$_3$), 28.6 (s, C(*C*H$_3$)$_3$), 33.0 (d, ^{2}J(P,C) = 8.1, *C*(CH$_3$)$_3$), 40.3 (d, ^{2}J(P,C) = 3.7, *C*H$_2$-CH$_3$), 53.0 (d, ^{1}J(P,C) = 224.7, C=N$_2$), 113.1 (s, C-2/C-5$_{arom}$), 115.9 (s, C-3/C-4$_{arom}$), 148.7 (s, C-1/C-6$_{arom}$)	
6h	2028	1.08, 1.33 (each d, each 12H, ^{3}J(H,H) = 7.0, CH-C*H*$_3$), 1.29 (s, 9H, *t*Bu), 3.83, 3.93 (each sept, each 2H, ^{3}J(H,H) = 7.0, C*H*-CH$_3$)	24.1 (d, ^{3}J(P,C) = 4.0, CH-*C*H$_3$), 29.4 (C(*C*H$_3$)$_3$), 33.5 (d, ^{2}J(P,C) 5.0, *C*(CH$_3$)$_3$), 48.6 (s, *C*H-CH$_3$), 57.5 (d, ^{1}J(P,C) = 209.0, C=N$_2$), 108.4 (d, ^{3}J(P,C) = 10.0, C-2/C-5$_{arom}$), 115.8 (s, C-3/C-4$_{arom}$), 145.6 (s, C-1/C-6$_{arom}$)	13.4

TABLE 2 Selected Bond Lengths (Å) and Angles (°) for the (Diazoalkyl)phosphorane **6h**

P-O1	1.683(4)	P-O2	1.825(4)
P-N1	1.657(5)	P-N2	1.672(5)
N4-N3	1.148(7)	P-C1	1.874(6)
N3-C1	1.293(8)	C11-C1	1.541(9)
O(2)-P-O(1)	85.1(2)	N(1)-P-O(1)	113.8(2)
N(1)-P-O(2)	93.2(2)	N(2)-P-O(1)	131.2(3)
N(2)-P-O(2)	85.5(2)	N(2)-P-N(1)	114.4(3)
C(1)-P-O(1)	83.3(2)	C(1)-P-O(2)	166.8(3)
C(1)-P-N(1)	97.1(3)	C(1)-P-N(2)	97.6(3)
C(1)-N(3)-N(4)	179.6(5)	N(3)-C(1)-P	114.9(5)
C(11)-C(1)-P	132.6(5)	C(11)-C(1)-N(3)	112.3(6)

TABLE 3 Positional Parameters of the Atoms with Equivalent Temperature Factors ($Å^2 \times 10^3$)

Atom	*x/a*	*y/b*	*z/c*	U_{eq}
BR(1)	0.2971(1)	0.3057(1)	−0.0027(1)	54(1)
BR(2)	0.1718(1)	0.2569(1)	−0.3456(1)	49(1)
BR(3)	0.3572(1)	0.1699(1)	−0.5272(1)	55(1)
BR(4)	0.6603(1)	0.1285(1)	−0.3601(1)	58(1)
P	0.7452(2)	0.2587(1)	0.1285(2)	26(1)
O(1)	0.5888(4)	0.2800(3)	0.0799(4)	29(2)
O(2)	0.7193(4)	0.2110(3)	−0.0505(4)	31(2)
N(1)	0.7959(5)	0.1469(4)	0.1984(5)	31(3)
N(2)	0.8556(5)	0.3238(4)	0.1043(5)	30(3)
N(3)	0.8159(5)	0.2899(5)	0.4113(7)	40(3)
N(4)	0.8850(6)	0.2582(5)	0.5136(6)	56(4)
C(1)	0.7378(6)	0.3259(5)	0.2966(7)	28(3)
C(11)	0.6495(6)	0.4119(5)	0.3259(7)	36(4)
C(12)	0.5404(7)	0.3679(6)	0.3555(9)	57(5)
C(13)	0.5992(7)	0.4825(6)	0.2033(8)	47(4)
C(14)	0.7176(8)	0.4725(6)	0.4563(8)	59(5)
C(2)	0.8619(7)	0.3599(6)	−0.0331(7)	43(4)
C(21)	0.7410(7)	0.4140(6)	0.1222(8)	47(5)
C(22)	0.9191(8)	0.2851(6)	−0.1164(8)	56(5)
C(3)	0.9605(6)	0.3552(6)	0.2288(8)	43(4)
C(31)	1.0879(7)	0.3232(7)	0.2130(9)	59(5)
C(32)	0.9526(8)	0.4715(7)	−0.7415(9)	67(6)
C(4)	0.8901(6)	0.0870(5)	0.1474(7)	40(4)
C(41)	1.0109(7)	0.0635(7)	0.2672(8)	60(5)
C(42)	0.8446(8)	−0.0062(6)	0.0601(9)	59(5)
C(5)	0.7166(7)	0.0985(5)	0.2657(7)	38(4)
C(51)	0.7919(8)	0.0308(6)	0.3832(8)	59(5)
C(52)	0.6073(8)	0.0449(6)	0.1636(9)	58(5)
C(61)	0.5227(6)	0.2527(5)	−0.0540(7)	28(4)
C(62)	0.5999(6)	0.2136(5)	−0.1289(7)	26(3)
C(63)	0.5509(6)	0.1856(5)	−0.2666(7)	34(4)
C(64)	0.4246(6)	0.2014(5)	−0.3322(6)	32(4)
C(65)	0.3458(6)	0.2383(5)	−0.2586(7)	32(4)
C(66)	0.3959(6)	0.2631(5)	−0.1155(7)	31(4)

chapter in the chemistry of compounds possessing $\lambda^5\sigma^5$ phosphorus atoms. The manifold synthetic possibilities both with retention and with loss of the diazo nitrogen atoms have not yet been delineated.

EXPERIMENTAL

All reactions were performed in a glass apparatus previously baked out in an argon atmosphere. The anhydrous solvents were distilled and stored under argon prior to use. Melting points: Mettler FP 61 apparatus (heating rate 3°C/min). Microanalyses: Perkin Elmer Elemental Analyzer 240. IR spectra: Perkin Elmer 881 spectrophotometer. ^{1}H NMR spectra: Varian EM 390 and Bruker AMX 400 spectrometers (TMS as internal standard). ^{13}C NMR spectra: Bruker AMX 400 spectrometer (100.61 MHz, TMS as internal standard). ^{31}P NMR spectra: Bruker AMX 400 spectrometer (161.98 MHz, 85% orthophosphoric acid as external standard).

(Diazoalkyl)phosphoranes (**6a–h**)*; General Procedure*

A solution of the (diazoalkyl)phosphane **4a–e** [6] in diethyl ether (20 mL) (for amounts, see Table 4) was cooled to −78°C, and a solution of an equimolar amount of the tetrahalo-orthobenzoquinone **5a,b** [16] in diethyl ether (30 mL) precooled to the same temperature was added dropwise with stirring. The mixture was allowed to warm to room temperature and then filtered over "filter flakes." Evaporation at 25°C/15 mbar almost to dryness furnished analytically pure yellow to orange crystals of the respective product. For IR and NMR spectroscopic data, see Table 1; for yields, melting points, and elemental analyses, see Table 4.

Crystal Structure Analysis of the (Diazoalkyl)phosphorane **6h** *[17]*

Crystal Data. $C_{23}H_{37}Br_4N_2O_2P$, M = 752.2 g/mol, crystal dimensions 0.3 × 0.3 × 0.25 mm, $a = 11.331(4)$, $b = 13.359(6)$, $c = 10.017(3)$ Å, $\alpha = 93.06(3)$, $\beta = 108.79(2)$, $\gamma = 85.34(3)°$, space group $P\bar{1}$ (Int. Tab. No. 2), $V = 1430.15(8)$ Å^3, $Z = 2$, $d_{calc} = 1.682$ g·cm^{-3}, $\mu = 56.5$ cm^{-1}.

Data Collection. Data were collected using an automatic four-circle diffractometer (Enraf-Nonius CAD 4, Mo-Kα radiation, graphite monochromator). Exact lattice constants were determined from the least-squares refinement of the 2Θ values of 25 reflections.

Structure Solution and Refinement. Structure solution and refinement were performed by direct methods (SHELXS-86 [18] and difference Fourier syntheses (SHELX-76 [19])). Graphical presentation of the molecule was achieved with the SCHAKAL program [20]. A total of 4479 reflections were measured in the range $2.0° \leq \Theta \leq 48.0°$, of which 3523 with $I \geq 2\sigma_{(I)}$ were considered in the refinement. The number of parameters was 338, heavy atoms were refined anisotropically, and the

TABLE 4 Yields, Melting Points, and Analytical Data of the (Diazoalkyl)phosphoranes **6a–h**

Product	*Starting Material (mmol)*	*Yield (%) (Color)*	*Mp (°C)*	*Molecular Formula (Mol. Mass)*	*Analysis Calcd/Found (%)*		
					C	*H*	*N*
6a	0.9	30	—[a]	$C_{17}H_{23}Cl_4N_2O_2P$	44.37	5.04	6.09[b]
		(orange)		(460.2)	43.8	5.0	4.7
6b	2.6	16	150	$C_{19}H_{27}Cl_4N_2O_2P$	46.74	5.57	5.74
		(orange)		(488.2)	46.8	5.6	5.5
6c	1.4	42	116	$C_{16}H_{22}Cl_4N_3O_2P$	41.67	4.81	9.11
		(yellow)		(461.2)	41.4	4.8	9.1
6d	1.1	49	136	$C_{19}H_{29}Cl_4N_4O_2P$	44.03	5.64	10.81
		(yellow)		(518.3)	44.3	6.0	10.2
6e	0.9	54	166	$C_{23}H_{37}Cl_4N_4O_2P$	48.10	6.49	9.75
		(yellow)		(574.4)	48.1	6.5	9.8
6f	2.5	38 (yellow to	137	$C_{18}H_{27}Br_4N_2O_2P$	34.26	4.09	4.21
		orange)		(666.0)	34.6	4.0	4.2
6g	1.7	21	152	$C_{19}H_{29}Br_4N_4O_2P$	32.79	4.20	8.05
		(yellow)		(696.1)	32.9	4.0	8.0
6h	0.9	49	173	$C_{23}H_{37}Br_4N_4O_2P$	36.73	4.96	7.45
		(orange)		(752.2)	36.7	4.9	7.4

[a] Partly oily, partly crystalline product.
[b] In spite of correct NMR data and the absence of signals for impurities, a better elemental analysis could not be obtained.

positions of the H atoms were calculated geometrically with group-wise common temperature factors. The structure refinement converged at R = 0.0758 and R_w = 0.0842, the difference Fourier synthesis on the basis of the final structural model showed a maximum of 0.79 $e \cdot Å^{-3}$ and a minimum of −0.89 $e \cdot Å^{-3}$, and the largest shift/esd was 0.54.

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