Crystallographic evidence of hexacoordination at phosphorus via intramolecular coordination of donor groups on phosphane and phosphane sulphide

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Received 10 July 1995

Abstract

The X-ray structure analysis of bis(8-dimethylamino-1-naphthyl)phenylphosphane (3) and of the corresponding sulphide 4 has revealed hexacoordination at phosphorus in both cases, the N ··· P separations being less than the sum of the van der Waal radii. Furthermore, in both cases the overall geometry corresponds to a distorted bicapped tetrahedron. The optimum geometry calculated for 4 via the HYPERCHEM program developed by Autodesk (MM + method) suggests that the structure of the molecule is a function not only of steric requirements but also of electronic effects.

Keywords: Phosphane; X-ray structure analysis; Phosphane sulfide; 6-coordination

1. Introduction

Numerous tertiary phosphanes containing aromatic rings ortho-substituted by dimethylamino or by dimethylaminomethyl groups [1–5] are known. Interest in this class of phosphanes derives from the fact that they can be useful in transition metal chemistry, acting as new bidentate ligands via both phosphorus and nitrogen [6–11]. The possible existence of N → P interactions in these compounds has rarely been considered [12]. However, we have recently reported [13] the X-ray analysis of phosphanes 1 and 2 (see Scheme 1) which show three weak N → P interactions. In both compounds the P atom is pseudo-seven-coordinate via three carbon atoms, three nitrogen atoms and its lone pair, which together form a tricapped tetrahedron. Furthermore we have shown that, as the N ··· P distances are rather long in these phosphanes, they have a reactivity which is d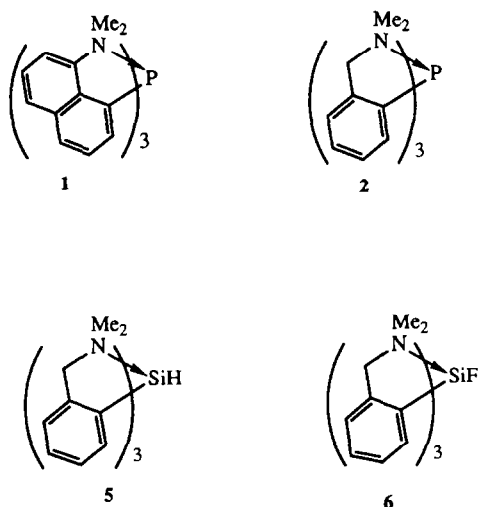ifferent from that of classical phosphanes [13,14]. In order to understand better the correlation between molecular structure and reactivity of such phosphanes, we investigated the X-ray structures of bis[(8-dimethylamino)-1-naphthyl]phenylphosphane (3) and of the corresponding sulphide 4.

2. Results and discussion

Treatment of PhPCl$_2$ with an excess of lithium (8-dimethylamino-1-naphthyl) in THF at −80°C gave a 60% yield of phosphane 3 as a yellow air-stable powder which has been fully characterized. Clear yellow crystals suitable for X-ray crystallography were grown from CH$_2$Cl$_2$/Et$_2$O. A PLUTO representation of the structure of 3 is shown in Fig. 1. Table 1 lists the most important bond lengths and bond angles, and torsion angles and other crystallographic details are given in Tables 2, 6 and 7. Some structural features follow.

The N1Me$_2$ group is trans to the P–C25 bond and the N2Me$_2$ group is opposite to the P–Cl bond, the N1 ··· P ··· N2 angle being 102.4(1)°.

Scheme 1.

Fig. 1. PLUTO drawing of the molecular structure of 3 showing the atomic numbering scheme. Hydrogen atoms are omitted for clarity.

The two $N \cdots P$ distances (2.792 Å and 2.780 Å) are significantly shorter than the sum of the N and P van der Waals radii (3.4 Å) [15], indicating weak interactions between these atoms.

The three P–C bond lengths (Table 1) differ significantly from one another and in all cases are longer than those observed in triphenylphosphane [16] (1.822 Å, 1.831 Å and 1.831 Å).

The naphthyl group connected to the longest P–C bond (P–C1, 1.872 Å) is only slightly distorted, the perpendiculars to the rings A and B forming an angle of
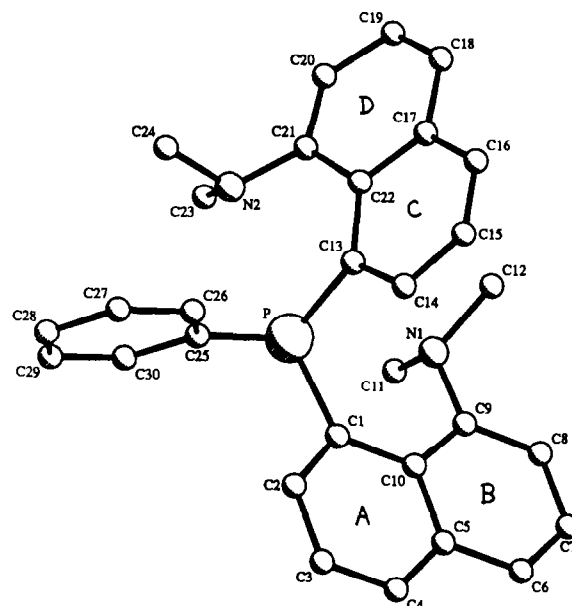
3°, while the other naphthyl group connected to the P–C13 is no longer planar, the perpendiculars to the rings C and D forming an angle of 7°. Another measure of this out-of-plane deformation is the dihedral angle $C13-P \cdots N2-C21$ of 27(1)°.

The pyramidal configuration about the phosphorus

Table 1
Selected bond lengths (Å) and bonds angles (°) of compound 3

Bond lengths			
P–C1	1.872(03)	$P \cdots N2$	2.780(02)
P–C13	1.852(02)	N1–C9	1.424(03)
P–C25	1.845(02)	N2–C21	1.432(03)
$P \cdots N1$	2.792(02)		
Bond angles			
C13–P–C1	101.4(1)	$C25-P \cdots N1$	175.1(1)
C25–P–C1	100.4(1)	$C1-P \cdots N2$	175.5(1)
C25–P–C13	101.7(1)	$C25-P \cdots N2$	82.2(1)
$N1 \cdots P \cdots N2$	102.4(1)	$C1-P \cdots N1$	74.9(1)
N1–C9–C8	120.8(2)	$C13-P \cdots N1$	78.1(1)
N1–C9–C10	118.6(2)	$C13-P \cdots N2$	74.4(1)
N2–C21–C20	122.4(3)		
N2–C21–C22	117.2(2)		

Table 2
Selected torsion angles (°) of compound 3

P–C1–C2–C3	176	C8–C9–C10–C1	175
P–C1–C10–C5	−178	N1–C9–C10–C1	−8
P–C1–C10–C9	2	P–C13–C22–C17	−161
C6–C5–C10–C1	−176	P–C13–C22–C21	17
C7–C8–C9–N1	−175	C20–C21–C22–C13	−168

Table 3
Selected bond lengths (Å) and bonds angles (°) of compound **4**

Bond lengths			
P–S	1.957(02)	P · · · N1	3.011(04)
P–C1	1.828(06)	P · · · N2	3.009(04)
P–C7	1.852(05)	N1–C13	1.428(07)
P–C17	1.835(05)	N2–C23	1.410(07)
Bond angles			
C1–P–S	111.5(2)	C1–P · · · N1	170.6(2)
C7–P–S	106.5(2)	C7–P · · · N2	176.1(2)
C7–P–C1	104.3(2)	C17–P · · · N1	72.4(2)
C17–P–S	123.5(2)	S–P · · · N2	75.6(2)
C17–P–C1	102.4(2)	C1–P · · · N2	77.7(2)
C17–P–C7	107.1(2)	S–P · · · N1	77.8(1)
N1–P · · · N2	106.9(2)	C7–P · · · N1	70.5(2)
		C17–P · · · N2	69.0(2)

atom is retained, as indicated by the average C–P–C angle of 101.1°, whereas it is 103.4° for triphenylphosphane [16]. Comparing the angles in **3** (Table 1) to the theorical angles for a bicapped tetrahedron (109.4°, 180° and 70.5°) suggests that the overall geometry of **3** corresponds to a bicapped distorted tetrahedron (taking into account the lone pair) rather than to a distorted octahedron.

Both ^{1}H and ^{13}C NMR spectra of **3** in solution exhibit four broad signals for the methyl groups at room temperature. This indicates that at room temperature (which is close to the coalescence temperature) there is an interaction of the two NMe_2 groups with the phosphorus without an intramolecular isomerization process occurring around the phosphorus atom. Further details will be described in another publication.

Table 4
Selected torsion angles (°) of compound **4**

Angles	From X-ray structural analysis	From optimum geometry calculations
P–C7–C12–C11	−155	−151.54
P–C7–C12–C13	24	31.44
C16–C11–C12–C7	171	173.45
C7–C12–C13–C14	−168	−174.07
C7–C12–C13–N1	18	16.64
C14–C15–C16–C11	7	3.48
C18–C17–C22–C23	−164	−162.18
C20–C21–C22–C23	164	166.56
C26–C21–C22–C17	166	172.9
C17–C22–C23–C24	−164	−173.86

Table 5
Selected calculated bond lengths (Å) and bonds angles (°) of compound **4**

Bond lengths			
P–S	2.0621	P · · · N1	3.266
P–C1	1.767	P · · · N2	3.253
P–C7	1.753		
P–C17	1.764		
Bond angles			
C1–P–S	107.4	S–P · · · N1	71.85
C7–P–S	105.1	C1–P · · · N1	175.86
C7–P–C1	108.2	C7–P · · · N1	68.33
C17–P–S	113.9	S–P · · · N2	68.3
C17–P–C1	109.96		

Table 6
Crystal data and intensity collection parameters

Compound	3	4
Molecular formula	$C_{30}H_{29}N_2P$	$C_{30}H_{29}N_2SP$
Molecular weight	448.5	480.6
Crystal size (mm)	0.33 × 0.35 × 0.37	0.38 × 0.25 × 0.17
Crystal system	Monoclinic	Monoclinic
Space group	$P2_1/n$	$P2_1/n$
a (Å)	12.455(1)	10.807(4)
b (Å)	9.370(2)	19.201(21)
c (Å)	21.362(3)	12.568(4)
β (°)	102.67(1)	104.15(3)
V (Å^3)	2147.8(7)	2529(3)
Z	4	4
$F(000)$	952	1016
$D_{calc.}$ (g cm^{-3})	1.39	1.26
Radiation	Mo K α	Mo K α
μ (λ = 0.71069 Å) (cm^{-1})	1.6	2.2
No. of unique data	4784 ($h = -15-u$; $k = 0-11$; $l = 0-26$)	4979 ($h = -12-12$; $k = 0-23$; $l = 0-15$)
No. of data used [$I \geqslant 2.5\sigma(I)$]	3171	2236
R	0.047	0.056
R_w	0.052 [($w = 1/\sigma^2(F) + 0.0011F^2$)]	0.064 [$w = 1/(\sigma^2(F) + 0.004F^2)$]
S (goodness of fit)	1.25	1.01
Final $\Delta\rho$ (e Å^{-3})	−0.20/+0.28	−0.28/+0.22

Table 7
Fractional atomic coordinates ($\times 10^4$) and equivalent isotropic thermal parameters (Å^2) for compound 3

| Atom	x	y	z	B_{eq}
P	1548(1)	1348(1)	4437(1)	2.6
C1	789(2)	−185(3)	3981(1)	2.9
C2	934(2)	−1534(3)	4243(2)	4.3
C3	352(3)	−2730(3)	3960(2)	5.3
C4	−381(2)	−2612(3)	3389(2)	4.3
C5	−578(2)	−1270(3)	3088(1)	3.3
C6	−1321(2)	−1162(3)	2484(1)	4.1
C7	−1519(3)	105(4)	2182(1)	4.6
C8	−1023(2)	1346(3)	2471(1)	3.9
C9	−304(2)	1302(3)	3066(1)	2.8
C10	−19(2)	−26(3)	3386(1)	2.7
N1	128(2)	2583(2)	3383(1)	2.9
C11	−609(2)	3163(3)	3763(1)	4.0
C12	452(3)	3666(3)	2965(1)	4.2
C13	2508(2)	1836(3)	3921(1)	2.7
C14	2669(2)	925(3)	3445(1)	3.7
C15	3212(2)	1379(4)	2961(1)	4.8
C16	3532(2)	2767(4)	2935(1)	4.7
C17	3397(2)	3736(3)	3412(1)	3.8
C18	3661(2)	5202(4)	3369(2)	5.1
C19	3506(3)	6144(4)	3823(2)	5.7
C20	3165(3)	5665(3)	4366(2)	4.7
C21	2920(2)	4252(3)	4438(1)	3.2
C22	2946(2)	3258(3)	3936(1)	2.9
N2	2720(2)	3692(2)	5026(1)	3.4
C23	1953(3)	4503(4)	5309(2)	5.1
C24	3759(3)	3442(4)	5490(1)	4.8
C25	2467(2)	385(3)	5097(1)	3.0
C26	3449(2)	−299(3)	5060(1)	3.7
C27	4099(2)	−954(4)	5596(2)	4.7
C28	3779(3)	−943(4)	6176(2)	5.2
C29	2808(3)	−274(4)	6218(1)	5.0
C30	2165(2)	384(3)	5687(1)	3.9
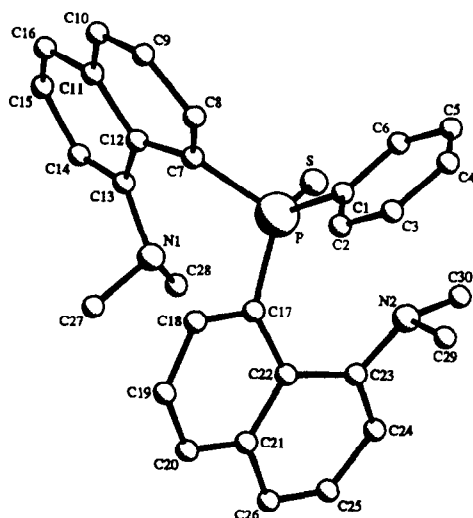
Fig. 2. PLUTO drawing of the molecular structure of 4 showing the atomic numbering scheme. Hydrogen atoms are omitted for clarity.

Structural analysis of the corresponding sulphide 4 was undertaken to investigate the effect of the sulphur atom on both the bond lengths, especially on the N → P

dative bonds and angles. The molecular geometry is presented in Fig. 2. Selected bond lengths and angles are listed in Table 3 and other crystallographic details in Tables 6 and 8. The average C–P–C angle is 104.6° compared with 103.4° for 3 and 105.7° for triphenylphosphane sulphide [17], and the average S–P–C angle is 113.8° compared with 113.1(6)° for Ph₃P=S. This indicates that the geometry at phosphorus is formally tetrahedral. However, the angle C17–P–S is 123.5(2)°, which is much larger than the ideal value. Both N atoms are directed towards the phosphorus atom with P · · · N distances (3.011 Å and 3.009 Å) longer than in 3 but still smaller that the sum of the N and P van der Waals radii (3.4 Å), suggesting weak interactions. The N1Me₂ group is trans to the P–C1 bond while the N2Me₂ group is opposite to the P–C7 bond, the N1 · · · P · · · N2 angle being 106.9(2)°. This suggests that the overall geometry of 4 can be considered as a bicapped distorted tetrahedron. The P=S bond is a little longer (1.957 Å) than the P=S bond of most triarylphosphanes sulphides [18–20] but notably shorter than that of the bulky tris(2,6-dimethoxyphenyl)phos-

Table 8
Fractional atomic coordinates (× 10⁴) and equivalent isotropic thermal parameters (Å²) for compound 4

Atom	x	y	z	B_{eq}
P	8172(1)	2367(1)	1417(1)	4.0
S	7172(2)	2536(1)	−83(1)	5.4
C1	9284(5)	1643(2)	1473(4)	4.2
C2	9861(5)	1303(3)	2443(4)	5.1
C3	10655(5)	734(4)	2470(6)	6.1
C4	10899(6)	501(4)	1500(7)	7.0
C5	10364(7)	833(4)	532(6)	6.7
C6	9553(6)	1408(3)	509(4)	5.5
C7	9213(5)	3138(3)	1815(3)	4.3
C8	10508(6)	3015(3)	2118(4)	5.3
C9	11392(6)	3571(4)	2142(4)	6.4
C10	10970(8)	4207(4)	1778(5)	6.8
C11	9644(8)	4365(3)	1459(4)	6.0
C12	8739(6)	3842(3)	1550(4)	4.7
C13	7438(7)	4052(3)	1347(4)	5.2
C14	7071(7)	4701(3)	906(4)	6.6
C15	7976(10)	5176(4)	734(5)	8.0
C16	9226(9)	5038(4)	1047(5)	7.3
C17	7455(5)	2174(2)	2565(4)	3.7
C18	8090(5)	2419(3)	3578(3)	4.6
C19	7514(7)	2396(3)	4480(4)	6.0
C20	6273(8)	2199(3)	4313(5)	6.3
C21	5583(6)	1948(3)	3290(5)	5.4
C22	6227(5)	1851(2)	2440(4)	4.2
C23	5621(6)	1432(3)	1530(5)	4.9
C24	4343(7)	1287(4)	1333(7)	7.5
C25	3680(7)	1493(5)	2104(10)	9.3
C26	4257(8)	1775(4)	3086(8)	7.8
N1	6570(5)	3603(2)	1708(3)	5.1
C27	6529(6)	3803(3)	2826(4)	5.9
C28	5267(6)	3578(3)	1014(5)	7.6
N2	6404(4)	1136(2)	902(3)	4.5
C29	7051(5)	516(3)	1436(4)	4.9
C30	5792(6)	989(3)	−247(4)	6.7

phane sulphide (1.969 Å) [21]. The lengthening of the P · · · N distances in **4** compared to those in **3** and including a more obvious deformation of naphthyl groups (Table 4) should be effected by the steric hindrance of the sulphur atom. Structural investigation of the silicon compounds **5** and **6** [22] indicated that the longer Si · · · N distances exist in **6** despite the electronegativity of the fluoride atom, which should favour the N · · · Si contact.

The important distortion of the naphthyl groups can be determined from the dihedral angles C7–P · · · N1–C13 and C17–P · · · N2–C23 which are 32°. This led us to calculate the optimum geometry of **4** (Fig. 3) using the HYPERCHEM program developped by Autodesk (MM + method). Selected bonds and angles are listed in Table 5. The calculated values tend towards an ideal tetrahedron, with two P–N bonds slightly longer than those determined by X-ray analysis; the naphthyl groups are also less deformed. This suggests that the geometry of the molecule is a result of both electronic and steric effects. The ^{1}H NMR spectrum of **4** in solution clearly shows the presence of two isomers, each exhibiting the resonances of the NMe$_2$ groups as four sharp singlets. Thus, in each isomer the NMe$_2$ groups are coordinated to the phosphorus atom in solution without intramolecular isomerization. The ^{31}P NMR spectrum in solution confirms the presence of two isomers ($\delta = 54$ ppm and 64.45 ppm). In contrast, only one signal appears in the ^{31}P CP MAS NMR spectrum ($\delta = 50.89$ ppm) which is consistent with the X-ray structure analysis of **4** which reveals only one isomer. In Scheme 2 we have represented the four isomers of **4**. Isomer **4a** is observed in the solid state, and **4b** and **4c** are also likely isomers as deduced from the X-ray structural analysis of various silicon compounds containing 8-dimethylaminonaphthyl [23–25]. However, we have no way to discount either. So far an isomer of type **4d** has never been observed.

The structural data indicate hexacoordination at the phosphorus atom in both cases. A study of the reactivity of the phosphane **3** and its comparison with the reactivity of the phosphane **1**, in which the phosphorus atom is
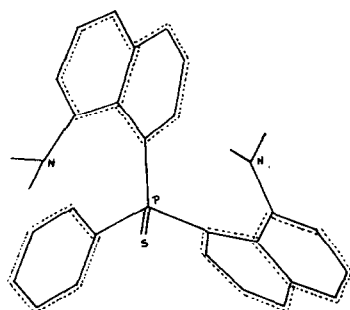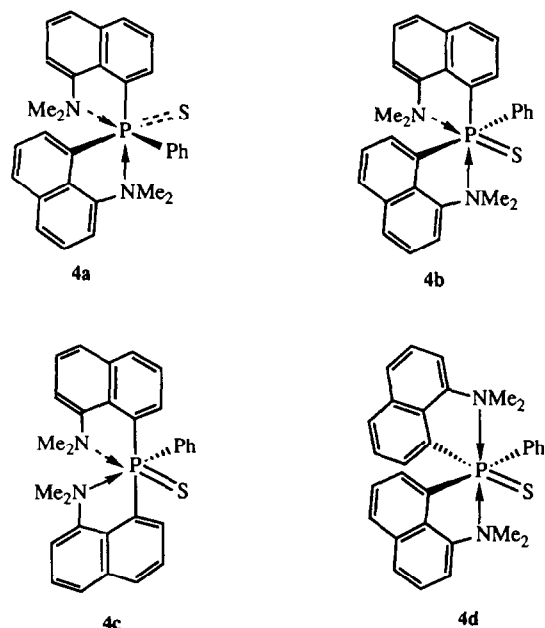

Fig. 3. Optimum geometry of **4** calculated via the HYPERCHEM program developed by Autodesk (MM + method). Hydrogen atoms are omitted for clarity.

Scheme 2.

pseudo-heptacoordinate [13], and with that of the (8-dimethylamino-1-naphthyl)diphenylphosphane is in progress. These should allow a better understanding of the relation between molecular structure and reactivity.

3. Experimental details

^{1}H, ^{13}C and ^{31}P NMR spectra were obtained using a Bruker 250 AC spectrometer. Solid-state NMR spectra were recorded on a Bruker AM-30 spectrometer. ^{1}H and ^{13}C chemical shifts are reported relative to Me$_4$Si and ^{31}P chemical shifts relative to H$_3$PO$_4$. Elementary analyses were performed by the Centre de Microanalyse du CNRS.

3.1. Bis[(8-dimethylamino)-1-naphthyl]phenylphosphane (3)

Dichlorophenylphosphane (1.84 ml, 13.6 mmol) was added with a syringe at −80°C to a solution of lithium (8-dimethylamino-1-naphthyl) (4.7 g, 2.72 mol) in THF (45 ml). The mixture was stirred overnight at room temperature, heated under reflux for about 4 h, and then cooled and hydrolyzed with 10% NaOH. The solvent was removed and the yellow solid washed with ethanol and ether, filtered and dried under vacuum. The solid was recrystallized from a dichloromethane/ether mixture to give yellow crystals of **3** (3.65 g, 60%); m.p. 203.1–204.1°C. ^{1}H NMR (C$_6$D$_6$) δ: 1.55 (s, 3H); 1.72 (s, 3H); 2.52 (s, 3H); 2.75 (s, 3H); 6.63–8.05 (m, 17H) ppm. ^{31}P NMR (CDCl$_3$) δ: 4.40 (s) ppm. ^{31}P CP MAS NMR δ: −2.707 ppm. Anal. Calc. for C$_{30}$H$_{29}$N$_2$P: C,

80.33; H, 6.05; N, 6.46%. Found: C, 80.24; H, 5.87; N, 6.43%.

3.1.1. X-Ray diffraction studies

Reflections were measured at room temperature with graphite monochromated Mo K α radiation on a Huber diffractometer. Parameters were refined using 19 reflections $(10° < 2\theta < 25°)$. No absorption correction was employed. The standard reflection (5,0,7) was checked every 50 reflections, but no significant deviation was observed. The structure was solved by direct methods using MULTAN-80 [26] with anisotropic least-squares refinement on F with SHELX 76 [27] assuming H atoms to be in the theoretical position. The structure is shown in Fig. 1 [28].

3.2. Bis[(8-dimethylamino)-1-naphthyl]phenylphosphane sulphide (4)

Phosphane 1 (600 mg, 1.3 mmol) and 0.358 g (1.4 mmol) of sulphur were mixed with butanol (40 ml). The heterogeneous mixture was heated under reflux for 6 h to form a homogeneous solution. The solution was then filtered to eliminate insoluble sulphur. After complete removal of the solvent in vacuum, the solid residue was recrystallized from a dichloromethane/ethanol mixture to give yellow crystals of 4 (0.405 g, 65%); m.p. 191–192°C. ^{1}H NMR (C_6D_6) δ: two isomers in a 4.5 : 1 ratio; (major) 1.13 (s, 3H); 1.18 (s, 3H); 2.45 (s, 3H); 2.85 (s, 3H); 6.6–8.2 (m, 17H) ppm; (minor) 1.05 (s, 3H); 1.15 (s, 3H); 2 (s, 3H); 2.75 (s, 3H); 6.6–8.2 (m, 17H) ppm. ^{31}P NMR (C_6D_6) δ: 54 (s, major), 64.4 (s) ppm. ^{31}P CP MAS NMR δ: 50.89 ppm. Anal. Calc. for $C_{30}H_{29}N_2PS$: C, 74.28; H, 6.28; N, 5.64%. Found: C, 74.23; H, 6.14; N, 5.63%.

3.2.1. X-Ray diffraction studies

Reflections were measured at room temperature with graphite monochromated Mo K α radiation on a Huber diffractometer. Parameters were refined using 18 reflections $(10° < 2\theta < 25°)$. No absorption correction was employed. The standard reflection (2,3,4) was checked every 50 reflections, but no significant deviation was observed. Part of the structure of the molecule was solved using MULTAN-80 [26] with complete resolution of structure by the use of the DIRDIF [29] program. Anisotropic least-squares refinement on F were undertaken with SHELX 76 [27] assuming H atoms to be in the theoretical position. The molecular structure is shown in Fig. 2 [28].

4. Supplementary materials available

Tables of fractional atomic coordinates of all atoms, thermal parameters, complete sets of bond distances and

bonds angles for 3 and 4 are available from the authors and have been deposited at the Cambridge Crystallographic Data Centre.

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