Reactivity of Fused 1,3-Benzothiaphospholes: Formation of Pentacoordinated Derivatives

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ABSTRACT

Fused 1,3-benzothiaphospholes 2 (cis-2,6,10-trimethyl-[1,3]benzothiaphospholo[2,3-b][1,3]benzothiaphosphole) reacts with H_2O_2 and S_8, giving the corresponding oxide and sulfide, respectively. The reactions of 2 with diethyl azodicarboxylate (DEAD)/catechol or DEAD/o-aminophenol, o-azidophenol, and tetrachloro-o-benzoquinone (TOB) give the first examples of spiro pentacoordinated phosphorus derivatives of this heterocyclic system. The X-ray structural analysis of spiro compound 3 showed a trigonal bipyramidal configuration at phosphorus in which the three rings assume axial-equatorial positions.

INTRODUCTION

Recently, we devised [1] the facile and highly stereoselective synthesis of cis-2,10-dimethyl[1,2,3]-benzothiadiphospholo[2, 3-b][1, 2, 3]benzothiadiphosphole 1, a new fused heterocyclic system containing an unusual P-P(S₂) unit, by treating p-

methylthioanisole with PCl₃ and AlCl₃. This new system was highly unstable to the conditions under which phosphines normally react, and, from the oxidation with H₂O₂, the reaction with diethyl azodicarboxylate (DEAD)/catechol, or the reaction with o-azidophenol, unidentifiable products were obtained. The instability of 1 might be due to the high reactivity of the phosphorus atom in the 6-position. This hypothesis was confirmed by a Friedel Craft acylation (CH₃COCl/AlCl₃) to replace the P-6 atom by the carbonyl carbon atom of acetyl chloride in a highly stereoselective fashion, obtaining a new heterocycle cis-2 in good yield [2].

2,6,10-Trimethyl-[1,3]benzothiaphospholo[2,3-b][1,3]benzothiaphosphole 2 shows a cis relationship between the P-12 lone pair and the 6-methyl substituent, as evidenced by X-ray diffraction analysis [2]. We have begun to study the reactivity of this heterocycle with regard to the reactions previously carried out on the system 1.

A spiro pentacovalent phosphorus derivative (3) has now been characterized by an X-ray crystal structure determination in order to elucidate the geometry at the phosphorus atom.

RESULTS AND DISCUSSION

Treatment of 2 with H₂O₂ and, in a separate experiment, elemental sulfur afforded the phosphine

oxide **4** [2] and sulfide **5**, respectively, in good yields, and the products were fully characterized by spectroscopic and analytical data.

The reaction of *cis*-**2** with typical reagents for the synthesis of phosphoranes [3] are shown in Scheme 1; from the reaction of **2** with *ortho*-azidophenol, the spiro compound **6** was obtained in good yield. The ^{31}P NMR signal of **6** ($\delta = -30.2$) is characteristic of a pentacoordinated phosphorus compound.

The spiro phosphorane **7** was readily obtained by the oxidative addition of 1 equiv of tetrachloro-*o*-benzoquinone to *cis*-**2**. The reaction could be followed by disappearance of the red color of the quinone until complete consumption of the starting material had been achieved. The $\delta^{31}P = 6.4$ for compound **7**, downfield relative to the usual pentacoordinate region, indicates the presence of a predominant betaine form in fast exchange with **7**.

Compound **6** was also prepared by treating **2** with DEAD/*ortho*-aminophenol, the reaction occurring in benzene. The reaction was also successful if catechol was employed in place of *o*-aminophenol, and, in this case, phosphorane **3** was formed. The phosphine oxide **4** was recovered as a by-product of the reaction, presumably derived by hydrolysis of a 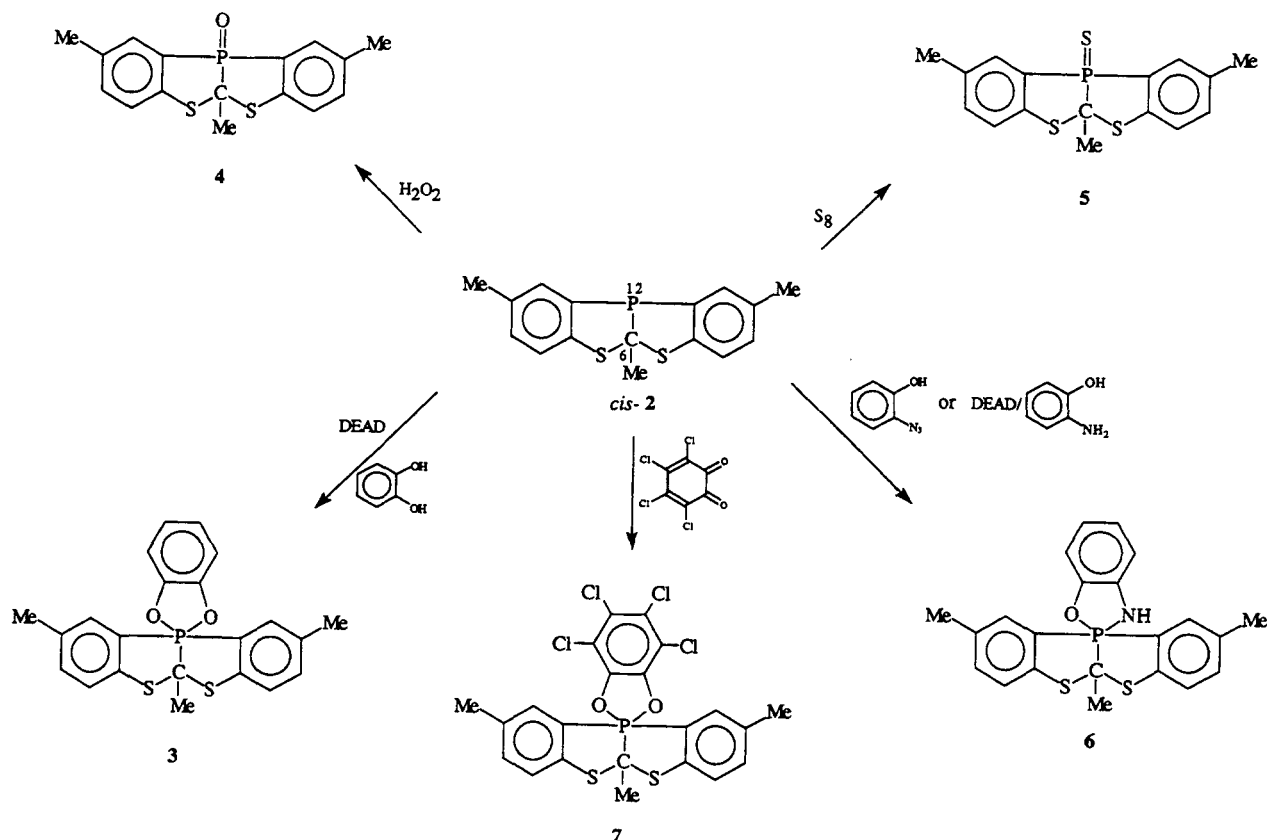compound **2**-DEAD adduct. The compounds **3**, **6**, and **7** are stable to both air and moisture; thermal decomposition commences only at a temperature in excess of 150 °C. The structural proof for these spiro compounds **3**, **6**, and **7** rests on analytical and spectroscopic data as well as, in the case of **3** on an X-ray diffraction study.

MOLECULAR GEOMETRY OF 3

The arbitrary numbering scheme used in the crystal structure analysis of **3** is shown in Figure 1.

Bond distances, angles, and torsion angles listed in Table 1 indicate that the conformational geometry of the product derived from the two original molecules that formed the asymmetric unit is quite similar and in good agreement with similar compounds of pentacovalent phosphorus [4].

It is known [5] that compounds containing pentacoordinated P in small rings arrange themselves, in order to minimize the ring strain, in either a square pyramid or a trigonal bipyramid. In compound **3**, the pentacoordinated P participates in a five-membered ring associated with two condensed five-membered rings and has a conformation close to an undistorted trigonal bipyramid with axial annulation [6]. Bond distances of the same type assume values significantly different; thus,
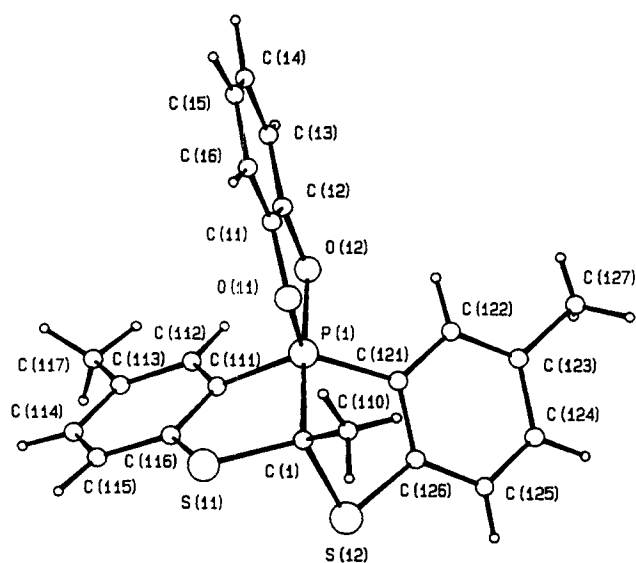

SCHEME 1

FIGURE 1   Perspective view of compound 3.

TABLE 1   Selected Bond Distances (Å), Bond Angles (°), and Torsion Angles (°) with Esds in Parentheses

	Molecule 1 X = 1	Molecule 2 X = 2
S(X1)–C(X)	1.815(3)	1.806(3)
S(X1)–C(X16)	1.760(3)	1.760(4)
S(X2)–C(X)	1.834(3)	1.844(3)
S(X2)–C(X26)	1.757(3)	1.763(3)
P(X)–O(X1)	1.659(3)	1.660(3)
P(X)–O(X2)	1.766(3)	1.748(3)
P(X)–C(X)	1.921(3)	1.920(3)
P(X)–C(X11)	1.806(3)	1.816(3)
P(X)–C(X21)	1.815(2)	1.819(3)
O(X1)–C(X1)	1.397(3)	1.387(3)
O(X2)–C(X2)	1.354(4)	1.357(3)
C(X)–S(X1)–C(X16)	94.9(2)	94.6(2)
C(X)–S(X2)–C(X26)	94.0(2)	92.9(1)
O(X1)–P(X)–O(X2)	88.7(1)	89.3(2)
O(X1)–P(X)–C(X)	87.7(2)	89.1(2)
O(X1)–P(X)–C(X11)	114.5(1)	116.2(2)
O(X1)–P(X)–C(X21)	126.0(2)	120.6(1)
O(X2)–P(X)–C(X)	175.5(2)	177.9(2)
O(X2)–P(X)–C(X11)	92.4(2)	91.6(2)
O(X2)–P(X)–C(21)	88.9(2)	89.5(1)
C(X)–P(X)–C(X11)	91.5(1)	90.4(2)
C(X)–P(X)–C(X21)	91.2(2)	90.1(1)
C(X11)–P(X)–C(X21)	119.6(2)	123.2(2)
P(X)–O(X1)–C(X1)	114.5(2)	114.2(2)
P(X)–O(X2)–C(X2)	111.9(2)	112.0(2)
S(X1)–C(X)–S(X2)	107.5(2)	108.3(2)
S(X1)–C(X)–P(X)	107.5(2)	108.2(2)
S(X1)–C(X)–C(X10)	110.4(2)	110.1(3)
S(X2)–C(X)–P(X)	104.9(2)	103.7(2)
S(X2)–C(X)–C(X10)	109.6(2)	109.4(2)
P(X)–C(X)–C(X10)	116.6(2)	116.7(3)
C(X)–S(X1)–S(X16)–C(X11)	−18.1(3)	−16.4(3)
C(X)–S(X2)–C(X26)–C(X21)	−25.3(3)	−27.2(3)
O(X1)–P(X)–C(X)–S(X1)	81.4(2)	82.2(2)
O(X1)–P(X)–C(X)–SS(X2)	−164.5(2)	−162.9(2)
O(X1)–P(X)–C(X)–C(X10)	−43.0(3)	−42.6(3)
O(X2)–P(X)–C(X)–S(X1)	116.8(22)	123.3(48)
O(X2)–P(X)–C(X)–S(X2)	−129.0(22)	−121.8(48)
O(X2)–P(X)–C(X)–C(X10)	−7.6(24)	−1.5(49)
C(X11)–P(X)–C(X)–S(X1)	−33.1(2)	−34.1(2)
C(X21)–P(X)–C(X)–S(X2)	−38.5(2)	−42.3(2)

axial P(X)-C(X) distances [1.1921(3) and 1.1920(3) Å in molecules 1 and 2, respectively] are longer than the equatorial P(X)-C(11) and P(X)-C(21) distances [1.806(3), 1.815(2) and 1.816(3), 1.819(3) Å in molecules 1 and 2, respectively], and also the axial P(X)-O(X2) distance [1.766(3) and 1.748(3)] are longer than typical equatorial P(X)-O(X1) distances [1.659(3) and 1.660(3) Å].

As shown in Table 2, the five-membered rings are not planar; the conformational analysis [7] indicates that the dioxaphosphole ring of molecule 2 adopts a conformation intermediate between twist and envelope with C_2 symmetry and with a pseudo twofold axis through C(21) [8], while all the other five-membered rings adopt an envelope conformation with C_s symmetry. All the benzene rings are planar within the experimental errors.

EXPERIMENTAL

Methods and Materials

^{1}H and ^{31}P NMR spectra were recorded at 300 and 120.75 MHz, respectively, with use of a Varian Gemini 300 instrument. ^{1}H NMR chemical shifts are given in parts per million from Me_4Si, and ^{31}P NMR chemical shifts are given in parts per million from H_3PO_4 85% (external standard) in $CDCl_3$ solutions. Mass spectra (by electronic impact) were recorded with a VG 7070 spectrometer or with an HP 59970 workstation formed by an HP-5890 gas chromatograph equipped with a methyl silicone capillary column and by an HP-5970 mass detector. Melting points are uncorrected and were determined with a Buchi apparatus. o-Azidophenol was synthesized according to literature methods [9]. Commercial catechol, o-aminophenol, tetra-

chloro-o-benzoquinone (TOB), and DEAD were used without purification. Benzene was purified by distillation over sodium. For purification of crude reaction mixtures, flash chromatography using silica gel 70–230 mesh was used in most cases. Analytical thin layer chromatography was performed by using precoated silica gel F-254 plates.

Reaction of 2 with Sulfur

Sulfur (60 mg, 1.9 mmol) was added to a solution of 576 mg of **2** (1.9 mmol) in benzene (10 mL), and the mixture was refluxed for 1 hour. Compound **5**

TABLE 2 Analysis of the Planarity
(a) Distances (Å × 10³) of relevant atoms from the mean plane with esds in parentheses

	Molecule 1	Molecule 2
Plane *A*: S(X1),P(X),C(X11),C(X16)		
S(X1)	10(7)	−15(5)
P(X)	−10(7)	14(5)
C(X11)	25(7)	−40(6)
C(X16)	−25(7)	41(6)
C(X)[a]	663(7)	−666(6)
Plane *B*: S(X2),P(X),C(X21),C(X26)		
S(X2)	−1(2)	3(6)
P(X)	1(2)	−3(6)
C(X21)	−4(4)	7(7)
C(X26)	4(4)	−8(7)
C(X)[a]	−795(5)	871(7)
Plane *C*: O(X1),O(X2),C(X1),C(X2)		
O(X1)	4(6)	9(5)
O(X2)	−4(6)	−9(5)
C(X1)	−6(6)	17(6)
C(X2)	7(6)	−18(6)
P(X)[a]	240(6)	216(5)
(b) Angles (°) between planes		
A-B	73.2(3)	71.8(2)
A-C	78.4(3)	76.4(3)
B-C	52.6(2)	59.8(3)

was purified by flash chromatography over silica gel using *n*-hexane/diethyl ether 4:1 as an eluant and was obtained in 87% yield.

Compound **5.** White solid; R_F 0.60 [light petroleum (40–60°C)/diethyl ether 1:1]. Mp 194–196°C. ^{1}H NMR δ: 2.06 (d, 3H, $^3J_{HP}$ = 14.7 Hz, CH$_3$–C), 2.36 (s, 6H, CH$_3$–C$_{ar}$), 7.10 (dd, 2H $^3J_{HH}$ = 8.0 Hz, $^4J_{HH}$ = 3.8 Hz, H–C$_{ar}$), 7.22 (bd, 2H, $^3J_{HH}$ = 8.0 Hz, H–C$_{ar}$), 7.55 (bd, 2H, $^3J_{HP}$ = 12.0 Hz, H–C–C–P); ^{31}P NMR δ: 87.4. Mass spectra *m/z* 334.0085 (100%) M$^+$ (calcd for C$_{16}$H$_{15}$PS$_3$: 334.0073), 301, 275, 243, 211, 181, 153, 63, 59. Anal. calcd for C$_{16}$H$_{15}$PS$_3$%: C, 57.48; H, 4.53, P, 9.27; S, 28.72. Found: C, 57.53; H, 4.57; P, 9.22; S, 28.55.

Reaction of 2 with o-Azidophenol

o-Azidophenol (350 mg, 2.5 mmol) dissolved in dry benzene (10 mL) was added dropwise to a solution of **2** (800 mg, 2.5 mmol) in 50 mL of benzene at room temperature under a nitrogen atmosphere, and the mixture was stirred for 1 hour. Gc-ms analysis showed the presence of the spiro compound **6** as the main product and the oxide **4** as a by-product of the reaction; compound **6** was isolated in 75% yield by flash chromatography over silica gel using light petroleum (40–60°C)/diethyl ether 4:1 as eluant.

Compound **6.** White solid; R_F 0.52 [light petroleum (40–60°C)/diethyl ether 1:1]. Mp 161–164°C. ^{1}H NMR δ: 1.95 (d, 3H, $^3J_{HP}$ = 6.6 Hz, CH$_3$–C), 2.31 (s, 6H, CH$_3$–C$_{ar}$), 4.98 (d, 1H, $^2J_{HP}$ = 17.4, HN–P), 6.65–7.26 (m, 8H, H–C$_{ar}$), 7.63 (d, 2H, $^3J_{HP}$ = 11.2 Hz, H–C–C–P); ^{31}P NMR δ; −30.2. Mass spectra *m/z* 409.0743 M$^-$ (calcd for C$_{22}$H$_{20}$NOPS$_2$: 409.0724), 267, 239 (100%), 153, 63, 52. Anal. calcd for C$_{22}$H$_{20}$NOPS$_2$%: C, 64.54; H, 4.93; P, 7.57; S, 15.63; N, 3.42. Found: C, 64.55; H, 4.92; P, 7.59; S, 15.64; N, 3.40.

Reaction of 2 with DEAD/o-Aminophenol

DEAD (0.20 mL, 1.32 mmol) and *o*-aminophenol (144 mg, 1.32 mmol) were added successively to a stirred solution of **2** (400 mg, 1.32 mmol) in benzene (10 mL) at room temperature. The reaction was immediate. Gc-ms analysis showed the presence of the spiro compound **6** as the main product and the oxide **4** as a by-product (ca. 10%) of the reaction; compound **6** was isolated in 64% yield by flash chromatography over silica gel using light petroleum (40–60°C)/diethyl ether 4:1 as eluant.

Reaction of 2 with DEAD/Catechol

DEAD (0.20 mL, 1.32 mmol) and catechol (145 mg, 1.32 mmol) were successively added to a stirred solution of **2** (400 mg, 1.32 mmol) in benzene (10 mL) at room temperature. The reaction was immediate. Gc-ms analysis showed the presence of the spiro compound **3** as the main product and the oxide **4** as a by-product (ca. 10%) of the reaction; compound **3** was isolated in 70% yield by flash chromatography over silica gel using cyclohexane:benzene 30:1 as eluant. Product **3** was crystallized from diethyl ether.

Compound **3.** White solid; R_F 0.60 [light petroleum (40–60°C)/diethyl ether 1:1]; mp 165–167°C. ^{1}H NMR δ: 2.06 (d, 3H, $^3J_{HP}$ = 7.7 Hz, CH$_3$–C), 2.32 (s, 6H, CH$_3$–C$_{ar}$), 6.68–6.78 (m, 1H, H–C$_{ar}$), 6.84–7.02 (m, 3H, H–C$_{ar}$), 7.11–7.26 (m, 4H, H–C$_{ar}$), 7.93 (bd, 2H, $^3J_{HP}$ = 10.5 Hz, H–C–C–P); ^{31}P NMR δ: −2.2. Mass spectra *m/z* 410.0559 M$^+$ (calcd for C$_{22}$H$_{19}$O$_2$PS$_2$: 410.0564), 267, 239 (100%), 181, 153, 63, 52. Anal. calcd for C$_{22}$H$_{19}$O$_2$PS$_2$%: C, 64.38; H, 4.67; P, 7.55; S, 15.59. Found: C, 64.39; H, 4.70; P, 7.53; S, 15.59.

Reaction of 2 with Tetrachloro-o-benzoquinone

Tetrachloro-o-benzoquinone (72 mg, 0.28 mmol) in benzene (5 mL) was added dropwise to a solution of **2** (85 mg, 0.8 mmol) in benzene (5 mL) at room temperature. The reaction was immediate and quantitative. Product **7** was crystallized from diethyl ether.

Compound **7**. White solid; R$_F$ 0.60 [light petroleum (40–60°C)/diethyl ether 1:1]; mp 209°C with decomposition. ^{1}H NMR δ: 2.11 (d, 3H, $^3J_{HP}$ = 8.2 Hz, CH$_3$–C), 2.35 (s, 6H, CH$_3$–C$_{ar}$), 7.15 (dd, 2H $^3J_{HH}$ = 7.8 Hz, $^4J_{HH}$ = 6.3 Hz, H–C$_{ar}$) 7.27 (bd, 2H, $^3J_{HH}$ = 7.8 Hz, H–C$_{ar}$), 7.93 (bd, 2H, $^3J_{HP}$ = 10.2 Hz, H–C–C–P); ^{31}P NMR δ: 6.4. Mass spectra m/z 545.9030 M$^-$ (calcd for C$_{22}$H$_{15}$O$_2$PS$_2$Cl$_4$: 545.9009), 267, 239 (100%), 181, 153, 52. Anal. calcd for C$_{22}$H$_{15}$O$_2$PS$_2$Cl$_4$%: C, 48.36; H, 2.77; P, 5.67; S, 11.71; Cl, 25.62. Found: C, 48.39; H, 2.79; P, 5.65; S, 11.70; Cl, 25.60.

X-Ray Crystal Structure Determination of 3

Crystals were colorless flattened prisms. Lattice parameters were determined using a program which repeatedly rectifies on the diffractometer the values of (θ, χ, φ) angles of 30 reflections to obtain the maximum of the peak when the angles are not moving more than 0.01°.

Crystal Data. C$_{22}$H$_{19}$O$_2$PS$_2$, M = 410.5. Triclinic a = 21.555(4), b = 10.409(2), c = 9.486(2) Å, α = 97.4(1), β = 77.4(1), γ = 104.0(1)°, U = 2008.9(13) Å^3, Z = 4, D_c = 1.36 g cm^{-3}; Cu K_α radiation λ = 1.5418 Å, μ = 32.7 cm^{-1}. Space group $P\bar{1}$ (C$_i^1$ No. 2) from structure determination.

X-ray measurements were performed at T = 295 K on a Siemens AED single crystal diffractometer in the range 3 $\leq$ θ $\leq$ 70° using Ni-filtered Cu K_α radiation. The angles for every reflection were determined on the basis of the orientation matrix, and the outline of the diffraction peak was collected in the θ-2θ step scanning mode using a scan width from (θ – 0.60)° to (θ + 0.60 + $\Delta\lambda/\lambda$ tgθ)°. The intensities I_{hkl} were determined by analyzing the reflection profiles [10]. 7640 independent reflections (–25 $\leq$ h $\leq$ 25, –12 $\leq$ k $\leq$ 12, 0 $\leq$ l $\leq$ 11) were measured of which 5701 having I_{hkl} > 2σ(I_{hkl}) [σ(I) based on statistic counting] were used in the refinement. One standard reflection, measured every 50 collected reflections to monitor crystal decomposition and instrumental linearity, showed no significant variation. Intensities I_{hkl} were corrected for Lorentz and polarization effects. The dimensions of the crystal were 0.17, 0.14, and 0.43 mm. No absorption corrections were applied.

Structure Analysis and Refinement. The structure was solved by direct methods by use of the SHELXS86 program [11] and refined by SHELX76 [12] with cycles of full-matrix anisotropic least squares (hydrogen atoms isotropically) up to R = 0.037 and R_w = 0.044; the weighting function was of the form w = 0.2434/[σ^2(F_0) + 0.0073F_0^2]. Positional parameters for nonhydrogen atoms are given in Table 3.

All the hydrogen atoms were located in the difference-Fourier map. Atomic scattering factors were

TABLE 3 Fractional Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Thermal Parameters ($\times 10^4$ Å^2) for non-H Atoms with Esds in Parentheses

$$U_{eq} = 1/3\Sigma_i\Sigma_j U_{ij}a_i^* a_j^*(a_i \cdot a_j)$$

	x	y	z	U$_{eq}$
Molecule 1				
S(11)	4132(0)	7178(1)	3794(1)	477(2)
S(12)	2782(0)	7189(1)	3662(1)	488(3)
P(1)	3220(0)	4639(1)	2973(1)	344(2)
O(11)	3530(1)	4015(2)	4116(2)	409(2)
O(12)	3137(1)	3087(2)	1947(2)	418(6)
C(1)	3294(1)	6252(2)	4223(3)	394(9)
C(11)	3684(1)	2784(2)	3627(3)	386(8)
C(12)	3459(1)	2282(2)	2368(3)	361(8)
C(13)	3564(1)	1073(3)	1681(3)	450(10)
C(14)	3899(2)	391(3)	2329(4)	558(12)
C(15)	4123(2)	918(3)	3595(4)	602(13)
C(16)	4018(2)	2146(3)	4272(3)	513(12)
C(110)	3088(2)	6103(3)	5842(3)	521(12)
C(111)	3818(1)	5459(3)	1530(3)	394(9)
C(112)	3906(1)	4974(3)	61(3)	441(9)
C(113)	4413(1)	5606(3)	–968(3)	501(10)
C(114)	4845(1)	6719(3)	–499(4)	560(13)
C(115)	4773(1)	7223(3)	922(4)	529(12)
C(116)	4251(1)	6599(3)	1950(3)	437(9)
C(117)	4497(2)	5090(5)	–2546(4)	716(18)
C(121)	2361(1)	4552(2)	3063(3)	359(7)
C(122)	1892(1)	3400(3)	2815(3)	413(9)
C(123)	1253(1)	3444(3)	2845(3)	461(10)
C(124)	1078(1)	4669(3)	3140(3)	500(11)
C(125)	1525(1)	5823(3)	3433(3)	478(10)
C(126)	2171(1)	5774(3)	3381(3)	403(9)
C(127)	760(2)	2188(4)	2513(6)	695(16)
Molecule 2				
S(21)	835(0)	6963(1)	7400(1)	543(3)
S(22)	269(0)	7301(1)	10,523(1)	559(2)
P(2)	1705(0)	7836(1)	9606(1)	374(2)
O(21)	2137(1)	8936(2)	8444(2)	411(6)
O(22)	2425(1)	7744(2)	10,112(2)	415(7)
C(2)	926(1)	8008(3)	9049(3)	459(10)
C(21)	2806(1)	9084(3)	8261(3)	400(8)
C(22)	2962(1)	8354(2)	9200(3)	367(8)
C(23)	3597(1)	8310(3)	9157(3)	466(10)
C(24)	4075(1)	9054(4)	8181(4)	584(12)
C(25)	3920(2)	9810(4)	7257(4)	646(13)
C(26)	3271(1)	9825(3)	7282(3)	521(10)
C(210)	874(2)	9413(3)	8871(4)	531(11)
C(211)	1569(1)	6106(3)	8900(3)	410(9)
C(212)	1876(1)	5159(3)	9231(3)	465(9)
C(213)	1825(2)	3909(3)	8483(3)	525(10)
C(214)	1460(2)	3616(3)	7385(4)	592(12)
C(215)	1144(2)	4512(3)	7050(3)	567(11)
C(216)	1203(1)	5760(3)	7815(3)	468(10)
C(217)	2168(2)	2918(4)	8816(6)	789(18)
C(221)	1389(1)	8375(3)	11,470(3)	410(9)
C(222)	1776(1)	9066(3)	12,450(3)	440(9)
C(223)	1492(2)	9452(3)	13,853(3)	490(11)
C(224)	821(2)	9108(3)	14,261(3)	568(11)
C(225)	423(2)	8450(3)	13,301(3)	569(12)
C(226)	709(1)	8093(3)	11,876(3)	471(9)
C(227)	1922(2)	10,180(4)	14,901(4)	689(15)

taken from the *International Tables for X-ray Crystallography* [13].

All the calculations were carried out on the GOULD 6040 POWERNODE computer at the Centro di Studio per la Strutturistica Diffrattometrica del C.N.R. of Parma, and the Cambridge Structural Database Files were used for bibliographic searches through the Servizio Italiano di Diffusione Dati Cristallografici di Parma.

SUPPLEMENTARY MATERIAL AVAILABLE

Tables of H-atoms fractional coordinates and a list of anisotropic thermal parameters and structure factors are available. Ordering information is given on any current masthead page.

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