Notes

Crystal Structure of 2,4-Bis(phenylthio)-1,3-dithia-2λ^5,4λ^5-diphosphetane 2,4-Disulphide†

Masataka Yokoyama *
Department of Chemistry, Faculty of Science, Chiba University, Yayoi-cho, Chiba City 260, Japan
Koreharu Ogata
Analysis Center, Chiba University, Yayoi-cho, Chiba City 260, Japan

The molecular structure of the oxygen–sulphur exchange reagent [PhSP(S)S]$_2$ has been determined by *X*-ray diffraction. It is monoclinic, space group $P2_1/n$, with a = 6.410(1), b = 11.701(3), c = 11.409(2) Å, β = 101.92(2)°, Z = 2, and R = 0.044 for 1 473 unique observed reflections.

Due to the physiological activity of thiopeptides [1] and the utility of dithio esters in organic synthesis,[2] effective oxygen–sulphur exchange reagents are of current interest. Hydrogen sulphide,[3] boron trisulphide,[4] thiophosphoryl bromide,[5] phosphorus pentasulphide,[6] Lawesson's reagent,[7] Davy's reagent,[8] and 2,4-bis(4-phenoxyphenyl)-1,3-dithia-2λ^5,4λ^5-diphosphetane 2,4-disulphide[9] have been used for this purpose.

We have presented 2,4-bis(arylthio)-1,3-dithia-2λ^5,4λ^5-diphosphetane 2,4-disulphide as convenient and efficient exchange reagents, superior to other related reagents in terms of their simple preparation, ease of handling, and good stability on storage.[10] Herein we report *X*-ray data for 2,4-bis(phenylthio)-1,3-dithia-2λ^5,4λ^5-diphosphetane 2,4-disulphide.

Experimental

The compound [PhSP(S)S]$_2$ was prepared by the reported procedure.[10] A mixture of tetraphosphorus decasulphide (2.8 g, 6.3 mmol), thiophenol (3.9 g, 36 mmol), and 1,2,4-trichlorobenzene (7 cm^3) was refluxed with stirring for 30 min. The resulting mixture was cooled to room temperature to precipitate yellow crystals (3 g) which were collected, washed with chloroform and diethyl ether, and recrystallized from toluene to afford the required compound as yellow needles in 58% yield (1.33 g), m.p. 167—168 °C (Found: C, 35.25; H, 2.45; S, 47.20%. M^+ 408. $C_{12}H_{10}P_2S_6$ requires C, 35.30; H, 2.45; S, 47.10%, M^+ 408.5).

Crystallography.—Crystal data. $C_{12}H_{10}P_2S_6$, M = 408.51, monoclinic, space group $P2_1/n$, a = 6.410(1), b = 11.701(3), c = 11.409(2) Å, β = 101.92(2)°, Z = 2, U = 837.4 Å^3, D_c = 1.620 g cm^{-3}, $F(000)$ = 208, λ(Cu-K_α) = 1.541 79 Å.

Lattice constants and intensity data were measured using graphite-monochromated Cu-K_α radiation on a Rigaku AFC-5 diffractometer. A total of 1 473 unique reflections with $F_o > 3\sigma(F_o)$ were obtained using the ω—2θ scanning method with a 2θ scan speed of 4° min^{-1} ($3 \leqslant 2\theta \leqslant 130°$). The structure was solved by the program MULTAN 80[11] based on the direct method, and refined to a final R value of 0.044 (w = 1), using isotropic thermal parameters for hydrogen atoms and anisotropic thermal parameters for the other atoms.

Additional material available from the Cambridge Crystallographic Data Centre comprises thermal parameters and remaining bond lengths and angles.

Table 1. Atom co-ordinates (× 10^4) with estimated standard deviations (e.s.d.s) in parentheses for [PhSP(S)S]$_2$

Atom	x	y	z
S(1)	6 839(2)	178(1)	11 087(1)
P(2)	6 463(2)	141(1)	9 205(1)
S(3)	8 120(3)	−927(1)	8 502(2)
S(4)	6 906(3)	1 776(1)	8 584(1)
C(5)	5 297(10)	2 649(4)	9 322(5)
C(6)	3 187(10)	2 878(5)	8 763(5)
C(7)	1 972(11)	3 581(5)	9 341(6)
C(8)	2 788(11)	4 021(5)	10 452(6)
C(9)	4 898(13)	3 801(5)	10 986(6)
C(10)	6 172(10)	3 113(5)	10 426(5)
H(C6)	2 483(88)	2 581(48)	7 872(49)
H(C7)	603(103)	3 673(58)	9 838(61)
H(C8)	1 725(98)	4 515(55)	10 810(57)
H(C9)	5 625(92)	4 122(53)	11 906(54)
H(C10)	7 709(87)	2 927(49)	10 737(52)

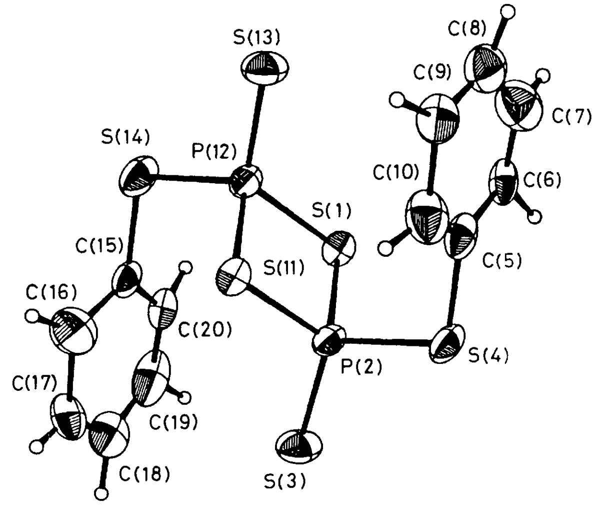

Figure. ORTEP drawing of [PhSP(S)S]$_2$. All non-hydrogen atoms are represented by 50% thermal ellipsoids; hydrogen atoms are represented by arbitrary small spheres

Results and Discussion

The ORTEP drawing, atom co-ordinates, bond lengths and angles are shown in the Figure, Table 1, and Table 2

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† *Supplementary data available:* see Instructions for Authors, *J. Chem. Soc., Dalton Trans.*, 1988, Issue 1, pp. xvii—xx.

Table 2. Bond lengths (Å) and angles (°), with e.s.d.s

S(1)–P(2)	2.112(2)	C(6)–C(7)	1.390(10)
P(2)–S(11)	2.107(2)	C(7)–C(8)	1.368(9)
P(2)–S(3)	1.920(2)	C(8)–C(9)	1.388(10)
P(2)–S(4)	2.080(2)	C(9)–C(10)	1.393(10)
S(4)–C(5)	1.782(6)	C(10)–C(5)	1.380(8)
C(5)–C(6)	1.397(8)	S(1)–P(12)	2.107(2)
P(2)–S(1)–P(12)	86.3(1)	C(5)–C(6)–C(7)	118.8(6)
P(2)–S(1)–P(12)	86.3(1)	C(6)–C(7)–C(8)	121.2(7)
S(1)–P(2)–S(4)	109.5(1)	C(7)–C(8)–C(9)	119.3(7)
S(4)–P(2)–S(3)	109.3(1)	C(8)–C(9)–C(10)	121.1(7)
S(3)–P(2)–S(11)	116.3(1)	C(9)–C(10)–C(5)	118.7(6)
S(1)–P(2)–S(3)	118.8(1)	S(1)–P(2)–S(11)	93.7(1)
S(11)–P(2)–S(4)	108.2(1)	S(1)–P(12)–S(11)	93.7(1)
P(2)–S(4)–C(5)	103.5(2)	S(1)–P(12)–S(13)	116.3(1)
C(6)–C(5)–C(10)	121.0(6)	S(1)–P(12)–S(14)	108.2(1)

respectively. In the Figure, the whole molecule is drawn on the basis of the values estimated from 15 atom co-ordinates [S(1), S(3), P(2), S(4), C(5)—C(10), and C(6)H—C(10)H]. Its structure has a centre of symmetry, which obliges the exocyclic sulphur atoms to be *trans*. The four-membered ring containing two sulphur and two phosphorus atoms exists in a diamond form [P(2)–S(1)–P(12) 86.3(1) and S(1)–P(2)–S(11) 93.7(1)°] and the P–S bond lengths are almost equal [2.112(2) and 2.107(2) Å].

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