Refinement of the crystal structure of bis(*O,O'*-diisopropylphosphorothionyl)disulfide at 173 K, [(C₃H₇O)₂PS₂]₂

E. R. T. Tiekink*

The University of Adelaide, Department of Chemistry, Australia 5005

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Abstract

C₁₂H₂₈O₄P₂S₄, triclinic, $P\bar{1}$ (No. 2), $a = 8.364(4)$ Å, $b = 8.481(4)$ Å, $c = 8.134(3)$ Å, $\alpha = 111.06(3)^\circ$, $\beta = 94.83(4)^\circ$, $\gamma = 97.78(4)^\circ$, $V = 528.0$ Å³, $Z = 1$, $R_{\text{gt}}(F) = 0.025$, $wR_{\text{ref}}(F^2) = 0.093$, $T = 173$ K.

Source of material

The title compound was isolated as an oxidation product formed during the attempted recrystallisation of (C₆F₅)₃Sb(S₂P(OiPr)₂)₂ [1].

Discussion

The low-temperature structure for centrosymmetric [(O*i*Pr)₂P(=S)₂]₂ is presented [2, 3]. Key geometric parameters: $d(\text{S1}—\text{S1}') = 2.112(1)$ Å, $d(\text{P1}—\text{S1}) = 2.080(1)$ Å, $d(\text{P1}—\text{S2}) = 1.921(1)$ Å, $\angle \text{S1}—\text{P1}—\text{S2} = 104.76(4)^\circ$, $\angle \text{O1}—\text{P1}—\text{O2} = 96.90(6)^\circ$. Chains are formed in the lattice, running parallel to the c direction, mediated by pairs of O2...H'—C3' interactions formed between centrosymmetrically related molecules such that $d(\text{O2}—\text{H5}') = 2.58$ Å, $d(\text{O2}—\text{C3}') = 3.561(2)$ Å and the angle at H5' is 160°; symmetry operation: $-x, 1-y, 2-z$.

Table 1. Data collection and handling.

Crystal:	pale-yellow block, size 0.19 × 0.23 × 0.45 mm
Wavelength:	Mo K α radiation (0.7107 Å)
μ :	6.12 cm ⁻¹
Diffractometer, scan mode:	Rigaku AFC7R, $\omega/2\theta$
$2\theta_{\text{max}}$:	59.98°
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$:	5320, 3069
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2450
$N(\text{param})_{\text{refined}}$:	157
Programs:	teXsan [4], DIRDIF92 [5], SHELXL-97 [6]

Table 2. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U_{iso}
H(1)	2i	-0.047(2)	0.135(2)	0.622(2)	0.023(4)
H(2)	2i	-0.031(3)	0.202(3)	0.994(3)	0.066(7)
H(3)	2i	-0.088(3)	0.019(3)	0.836(3)	0.065(7)
H(4)	2i	0.096(3)	0.098(3)	0.854(3)	0.055(6)
H(5)	2i	-0.238(2)	0.333(3)	0.876(3)	0.046(5)
H(6)	2i	-0.299(3)	0.157(3)	0.721(3)	0.050(5)
H(7)	2i	-0.250(3)	0.313(3)	0.677(3)	0.057(6)
H(8)	2i	0.424(2)	0.608(2)	0.623(2)	0.032(4)
H(9)	2i	0.583(3)	0.563(3)	0.846(3)	0.054(6)
H(10)	2i	0.557(3)	0.744(3)	0.993(3)	0.052(6)
H(11)	2i	0.652(3)	0.734(3)	0.834(3)	0.062(6)
H(12)	2i	0.358(2)	0.908(3)	0.896(3)	0.051(6)
H(13)	2i	0.461(3)	0.903(3)	0.737(3)	0.055(6)
H(14)	2i	0.278(2)	0.843(3)	0.693(3)	0.047(5)

* Correspondence author (e-mail: edward.tiekink@adelaide.edu.au)

Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
S(1)	2i	0.06740(4)	0.40251(4)	0.44192(4)	0.0346(2)	0.0310(2)	0.0193(2)	0.0153(1)	0.0038(1)	0.0082(1)
S(2)	2i	0.31975(4)	0.21702(4)	0.58352(5)	0.0289(2)	0.0279(2)	0.0361(2)	0.0124(1)	0.0054(1)	0.0103(1)
P(1)	2i	0.18873(3)	0.39073(3)	0.66998(4)	0.0210(2)	0.0199(2)	0.0200(2)	0.0040(1)	0.0025(1)	0.0071(1)
O(1)	2i	0.0594(1)	0.3686(1)	0.7922(1)	0.0269(4)	0.0236(4)	0.0232(4)	0.0005(3)	0.0061(3)	0.0078(3)
O(2)	2i	0.2741(1)	0.5776(1)	0.7927(1)	0.0259(4)	0.0231(4)	0.0241(4)	−0.0003(3)	0.0054(3)	0.0048(3)
C(1)	2i	−0.0591(1)	0.2074(2)	0.7402(2)	0.0254(5)	0.0242(5)	0.0278(5)	0.0018(4)	0.0052(4)	0.0106(4)
C(2)	2i	−0.0160(2)	0.1221(2)	0.8671(3)	0.0430(8)	0.0441(8)	0.059(1)	0.0038(7)	0.0016(7)	0.0359(8)
C(3)	2i	−0.2257(2)	0.2562(2)	0.7491(2)	0.0260(6)	0.0380(7)	0.0383(7)	0.0051(5)	0.0071(5)	0.0148(6)
C(4)	2i	0.4143(2)	0.6678(2)	0.7439(2)	0.0286(6)	0.0244(5)	0.0309(6)	−0.0007(4)	0.0084(5)	0.0058(5)
C(5)	2i	0.5644(2)	0.6738(2)	0.8629(3)	0.0257(6)	0.0431(8)	0.060(1)	0.0034(6)	0.0020(6)	0.0145(7)
C(6)	2i	0.3758(2)	0.8424(2)	0.7666(2)	0.0410(8)	0.0282(6)	0.0474(8)	0.0005(5)	0.0061(6)	0.0153(6)

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References

1. Mahalakshmi, H.; Jain, V. K.; Tiekink, E. R. T.: Some chemistry of pentafluorophenylantimony(V) complexes. *J. Organomet. Chem.*, in preparation.
2. Lawton, S. L.: The crystal and molecular structure of bis(*O,O'*-diisopropylphosphorothionyl)-disulphide, [(*i*-C₃H₇O)₂PS₂]₂. *Inorg. Chem.* **9** (1970) 2269–2274.
3. Tkachev, V. V.; Atovmyan, L. O.; Shchepinov, S. A.: Crystal and molecular structure of bis(*O,O'*-di-isopropylthiophosphoryl)-disulfide. *Z. Strukt. Khim.* **17** (1976) 945–947.
4. teXsan: Single Crystal Structure Analysis Software. Version 1.04. Molecular Structure Corporation. The Woodlands, TX, USA 1997.
5. Beurskens, P. T.; Admiraal, G.; Beurskens, G.; Bosman, W. P.; Garcia-Granda, S.; Gould, R. O.; Smits, J. M. M.; Smykalla, C.: The DIRDIF program system, Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands 1992.
6. Sheldrick, G. M.: SHELXL-97. Program for crystal structure refinement. University of Göttingen, Germany 1997.