Acta Crystallographica Section C
Crystal Structure Communications
ISSN 0108-2701

A monoclinic polymorph of bis(*tert*-butylperthiophosphonic) dianhydride

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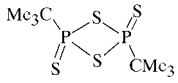
Received 4 February 2000
Accepted 9 February 2000

Data validation number: IUC0000041

In comparison with the known orthorhombic polymorph of *trans*-2,4-di-*tert*-butyl-2,4-dithioxo-1,3-dithia-2,4-diphosphetane, $C_8H_{18}P_2S_4$, (I) [Shore, Pennington, Noble & Cordes (1988). *Phosphorous Sulfur*, **39**, 153–157], the new crystallographic modification is monoclinic and the corresponding solid density is markedly higher. In both structures, the molecules have 2/*m* symmetry imposed by space-group symmetry and all corresponding bond lengths and angles are equal within the limits of errors.



(I)

Experimental

The title compound, *trans*-2,4-di-*tert*-butyl-1,3-dithia-2,4-diphosphetane-2,4-dithione, was prepared during the reaction of *tert*-butyllithium with pyridine (py) stabilized dithiometaphosphoryl chloride py→PS_2Cl. Slow dropping of one equivalent of *tert*-butyllithium into a suspension of py→PS_2Cl in benzene at 323 K, filtration, evaporation at high vacuum, and recrystallization from toluene produced crystals suitable for X-ray analysis.

Crystal data

$C_8H_{18}P_2S_4$	D_x = 1.422 Mg m^{-3}
M_r = 304.40	Mo $K\alpha$ radiation
Monoclinic, $C2/m$	Cell parameters from 62 reflections
a = 9.7313 (8) Å	θ = 13.0–17.1°
b = 9.2898 (11) Å	μ = 0.858 mm^{-1}
c = 8.1150 (10) Å	T = 180 (2) K
β = 104.252 (8)°	Square plate, colorless
V = 711.03 (13) Å^3	0.57 × 0.57 × 0.26 mm
Z = 2	

Data collection

Stoe Stadi-4 diffractometer	R_{int} = 0.038
$2\theta/\omega$ scans, ratio = 1.0, width(ω) = 1.55–1.7°	θ_{max} = 27.56°
Absorption correction: ψ scan (North *et al.*, 1968)	$h = -12 \rightarrow 12$
T_{min} = 0.640, T_{max} = 0.808	$k = -12 \rightarrow 12$
3495 measured reflections	$l = -10 \rightarrow 10$
876 independent reflections	3 standard reflections
846 reflections with $I > 2\sigma(I)$	frequency: 120 min
	intensity decay: 4.5%

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0387P)^2 + 0.2726P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.025	$(\Delta/\sigma)_{max}$ = 0.007
$wR(F^2)$ = 0.064	$\Delta\rho_{max}$ = 0.56 e Å^{-3}
S = 1.145	$\Delta\rho_{min}$ = −0.42 e Å^{-3}
876 reflections	Extinction correction: *SHELXL*97
59 parameters	Extinction coefficient: 0.038 (3)
All H-atom parameters refined	

Data collection: *STADI*4-1.06 (Stoe & Cie, 1997); cell refinement: *STADI*4-1.06; data reduction: *XRED*-1.07 (Stoe & Cie, 1997); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); software used to prepare material for publication: *SHELXL*97.

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