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1,4-Butylenebis(diphenylphosphine selenide)

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Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.003 Å
R factor = 0.026
wR factor = 0.075
Data-to-parameter ratio = 21.2

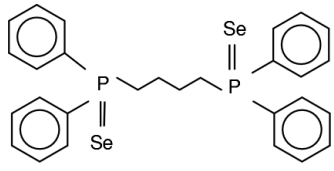
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title compound, $C_{28}H_{28}P_2Se_2$, is located on an inversion centre. The Se—P bond length is 2.1055 (5) Å.

Comment

Tertiary phosphine chalcogenides have been reported to be versatile coordinating agents and have shown extraction and catalytic behaviour (Lobana, 1989). There have been few structural accounts of tertiary phosphine selenides or their complexes; Ph_3PSe (Codding & Kerr, 1979), $HgCl_2(Ph_3PSe)$ as a chloro-bridged dimer (Glasser *et al.*, 1969), $AuCl(Ph_3PSe)$ (Hussain, 1986), $CuI(Ph_3PSe)(CH_3CN)$ as an iodo-bridged dimer (Lobana *et al.*, 1999), $ZnI_2(dppmSe_2)$ [where $dppmSe_2$ is 1,1-methylenebis(diphenylphosphine selenide); Lobana *et al.*, 2001] and $CuCl(dppeSe_2)$ as a chloro- and $dppeSe_2$-bridged dimer [where $dppeSe_2$ is 1,2-ethylenebis(diphenylphosphine selenide); Lobana *et al.*, 2001].

As part of an ongoing study of the structural chemistry of phosphine selenide ligands and their complexes, we have prepared and obtained single crystals of the title compound, (I) (Fig. 1).



(I)

Experimental

To a solution (0.500 g, 1.17 mmol) of 1,4-butylenebis(diphenylphosphine) or dppb in 20 ml dry benzene was added powdered selenium metal (0.185 g, 2.34 mmol) and the resulting solution refluxed for 4 h. The solid Se was consumed during the reflux. The solution was filtered and the volume reduced by half. The product formed on the addition of 1 ml of dry ethanol; yield 0.685 g, 95%, m.p. 461–463 K. Single crystals were obtained from a solution of 100 mg of $dppbSe_2$ in a 1:1 benzene–ethanol mixture. The preparation is based on that of Ph_3PSe (Nicpon & Meek, 1966), and analytical data and IR spectroscopy have been previously reported (Sandhu & Singh, 1976).

Crystal data

$C_{28}H_{28}P_2Se_2$
M_r = 584.36
Monoclinic, $P2_1/n$
a = 6.8206 (7) Å
b = 23.154 (3) Å
c = 8.7018 (9) Å
β = 105.380 (2)°
V = 1325.0 (2) Å^3
Z = 2
D_x = 1.465 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 918 reflections
θ = 2.6–27.7°
μ = 2.93 mm^{-1}
T = 294 (2) K
Prism, colourless
0.60 × 0.25 × 0.19 mm

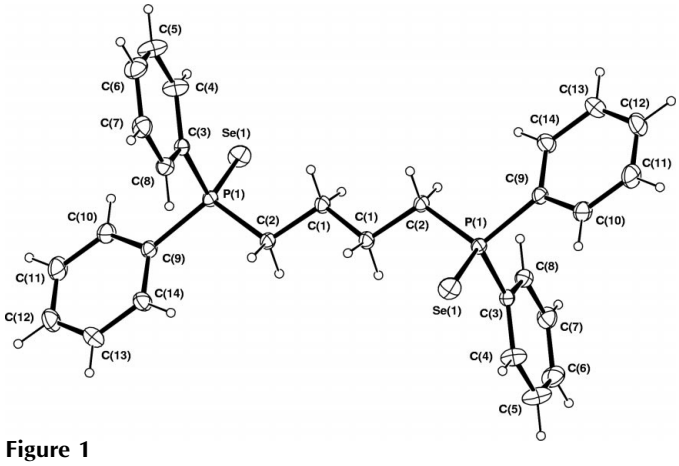


Figure 1
*ORTEP*II (Johnson, 1976; Hall *et al.*, 1999) projection of (I) with displacement ellipsoids shown at the 20% probability level.

Data collection

Bruker SMART 1000 CCD diffractometer
ω scans
Absorption correction: empirical (*SADABS*; Sheldrick, 1996; Blessing, 1995)
T_{min} = 0.382, T_{max} = 0.570
11 599 measured reflections
3080 independent reflections
2741 reflections with $I > 2\sigma(I)$
R_{int} = 0.017
θ_{max} = 28.3°
$h = -9 \rightarrow 8$
$k = -30 \rightarrow 30$
$l = -11 \rightarrow 11$
182 standard reflections
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.026
$wR(F^2)$ = 0.075
S = 1.02
3080 reflections
145 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0425P)^2 + 0.4738P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.47 e Å^{-3}
$\Delta\rho_{min}$ = −0.41 e Å^{-3}

Crystal decay was assessed with a recollection the first 182 reflections at the end of the experiment.

Data collection: *SMART* (Siemens, 1995); cell refinement: *SAINT* (Siemens, 1995); data reduction: *SAINT* and *XPREP* (Siemens, 1995); program(s) used to solve structure: *SIR*97 (Altomare *et al.*, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *TEXSAN* (Molecular Structure Corporation, 1997).

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