Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Triisopropylphosphine sulfide

Richard J. Staples* and Brent M. Segal

Department of Chemistry and Chemical Biology, Harvard University, Cambridge, MA 02138, USA

Correspondence e-mail: staples@chemistry.harvard.edu

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.009 Å
R factor = 0.069
wR factor = 0.218
Data-to-parameter ratio = 20.1

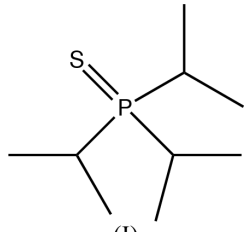
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 12 March 2001
Accepted 16 March 2001
Online 12 April 2001

The title compound, $C_9H_{21}PS$, was sublimed to give crystals suitable for single-crystal X-ray diffraction studies and shows a single $^{31}P\{^1H\}$ NMR signal in C_6D_6 and CD_3CN of 72.25 and 78.93 p.p.m., respectively, *versus* H_3PO_4.

Comment

The use of phosphines for the abstraction of sulfur from metal complexes has been used for years to promote favorable reaction chemistry (Liu *et al.*, 1998). In our research, it was necessary to synthesize and characterize triisopropylphosphine sulfide, (I), to confirm its production in the reaction *via* $^{31}P\{^1H\}$ NMR techniques (Osterloh *et al.*, 2000). As part of this characterization, the X-ray crystal structure was carried out. Related phosphine sulfides which have been characterized by X-ray diffraction include triphenylphosphine sulfide (Codding & Kerr, 1978; Foces-Foces & Llamas-Saiz, 1998) and tricyclohexylphosphine sulfide (Kerr *et al.*, 1977; Reibenspies *et al.*, 1996).



(I)

Experimental

Solid sulfur (0.05 g, 1.6 mmol) was slurried in 10 ml of tetrahydrofuran (THF) to which was added dropwise (36 µl, 1.6 mmol) triisopropylphosphine in 1 ml of THF. The sulfur dissolved rapidly yielding a pale yellow solution, which was filtered over celite. The nearly colorless solution was reduced in volume forming an oil. Sublimation at 273 K yielded 0.19 g (63%) of colorless block-shaped crystals. The compound was further characterized by $^{31}P\{^1H\}$ NMR; (C_6D_6) d 72.25 (s), (CD_3CN) d 78.93 p.p.m (s).

Crystal data

$C_9H_{21}PS$
M_r = 192.29
Orthorhombic, *Ibam*
a = 13.573 (1) Å
b = 13.8717 (9) Å
c = 12.688 (1) Å
V = 2388.9 (3) Å^3
Z = 8
D_x = 1.069 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 89 reflections
θ = 2.5–18.8°
μ = 0.36 mm^{-1}
T = 293 (2) K
Block, colorless
0.15 × 0.10 × 0.10 mm

Data collection

Bruker CCD diffractometer	$R_{int} = 0.097$
0.3° ω scans	$\theta_{max} = 25.0°$
6072 measured reflections	$h = -16 \rightarrow 14$
1108 independent reflections	$k = -16 \rightarrow 15$
525 reflections with $I > 2\sigma(I)$	$l = -15 \rightarrow 9$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0861P)^2 + 1.4788P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.069$	$(\Delta/\sigma)_{max} = 0.052$
$wR(F^2) = 0.218$	$\Delta\rho_{max} = 0.25$ e Å^{-3}
$S = 1.14$	$\Delta\rho_{min} = -0.31$ e Å^{-3}
1108 reflections	
55 parameters	
H-atom parameters constrained	

Data collection: *ASTRO* (Bruker, 1997); cell refinement: *SMART* (Bruker, 1997); data reduction: *SAINT* (Bruker, 1999); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1998); software used to prepare material for publication: *CIFTAB* in *SHELXL*97.

The CCD-based X-ray diffractometer at Harvard University was purchased through an NIH grant (1S10RR11937-01). The authors would also like to thank the laboratory of Professor Richard H. Holm for use of his facilities to complete this work and a grant from the National Institutes of Health (GM 28856).

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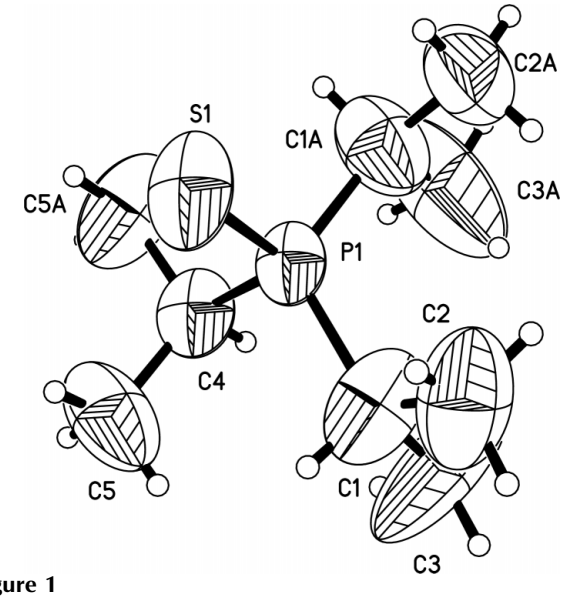

Figure 1
Displacement ellipsoid plot (50% probability) of the title compound. H atoms have been drawn as spheres with arbitrary radii.

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