Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

Tris(1-naphthyl) phosphite

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Received 13 March 2007
Accepted 19 April 2007

The title compound, $C_{30}H_{21}O_3P$, has a distorted trigonal–pyramidal geometry around the P atom. The P—O bond lengths are between 1.609 (1) and 1.643 (1) Å, and O—P—O bond angles vary between 94.73 (5) and 101.91 (6)°. The crystal structure is stablized by weak π—π and C—H$\cdots\pi$ interactions.

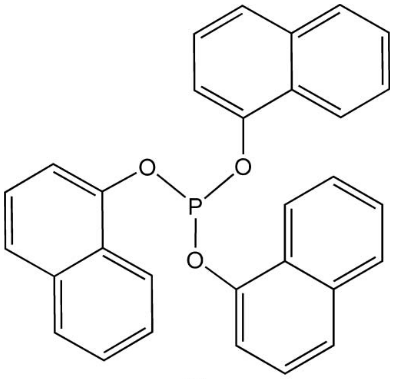
Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.002 Å
R factor = 0.040
wR factor = 0.106
Data-to-parameter ratio = 17.6

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

Comment

Phosphites are well known ligands in homogeneous catalysis and were first used in rhodium-catalysed hydroformylation in the late 1960s (Pruett & Smith, 1969). Up to now the structures of only a few monodentate phosphite ligands have been characterized (Baker *et al.*, 1992; Crous *et al.*, 2005; Golovanov *et al.*, 2005; Senker & Lüdecke, 2001). The title compound, (I), is presented as the first monodentate phosphite ligand containing a naphthyl ring system as the aryl substituent.



(I)

Compound (I) crystallizes with a distorted trigonal-pyramidal (pseudo-tetrahedral) geometry about the P atom (Fig. 1). One of the naphthyl ring systems is oriented so that the O—C bond is directed away from the electron lone pair of the P atom, as indicated by the O—P—O—C and P—O—C—C torsion angles (Table 1 and Fig. 1). Calculation of the Tolman solid-state cone angle (Tolman, 1977) results in a value of 163°, which gives an indication of the steric demand of the phosphite ligand. This value correlates well with that obtained for a Rh^I Vaska-type complex containing a (2-naphthyloxy)diphenylphosphine ligand (Kirsten *et al.*, 2007) of 162°. The main contributor to the steric bulk in these structures is the naphthyl ring system. It is interesting to note that the bulky phosphite ligand tris(2,4-di-tert-butylphenyl) phosphite, $(PObtbp)_3$ (Crous *et al.*, 2005), crystallizes on a pseudo-threefold rotation axis in the pure form and also when co-crystallized with a Rh^I complex, and that tris(2-methoxy-

doi:10.1107/S1600536807019629

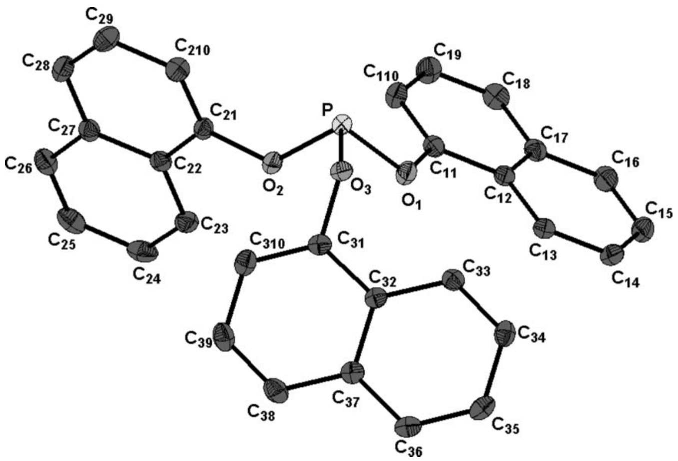


Figure 1
The molecular structure of (I), shown with 50% probability displacement ellipsoids. H atoms have been omitted for clarity.

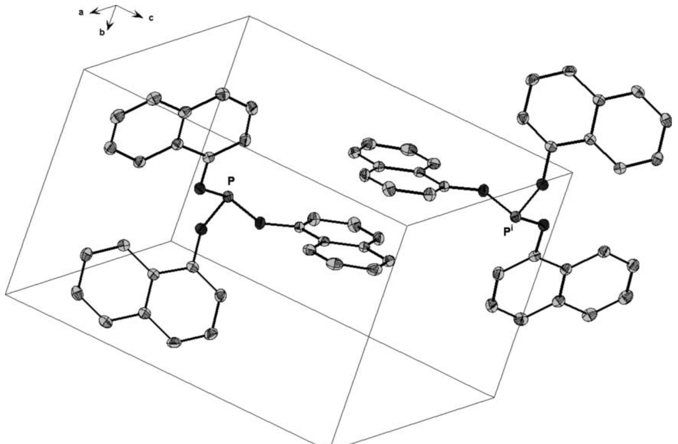


Figure 2
The unit cell contents, showing the weak π–π interaction in the structure [symmetry code: (i) $-x, 1-y, 1-z$].

phenyl) phosphite (Baker *et al.*, 1992) crystallizes on a C_3 axis, whereas there is no crystallographic C_3 symmetry present in compound (I).

A weak π–π intermolecular interaction is present in the crystal packing of (I) with an interplanar distance of 3.391 (2) Å [Fig. 2; C21$\cdots$C210 and C21$\cdots$C210($-x$, $1-y$, $1-z$)] and with ring slippage illustrated by the larger centroid-centroid distance of 3.6704 (9) Å for the C22–C27 ring.

The angle between the P—O—C plane and the least-squares plane of the corresponding benzene ring (bonded to the O atom) gives an indication of the orientation of each naphthyl group in the phosphite. These angles are given in Table 2 along with the angles for similar phosphites in the literature. In (I), two of these interplanar angles are quite similar, while the largest angle correlates with the orientation as described alternatively in the previous paragraph.

Weak C—H$\cdots\pi$ interactions are observed in (I). The most significant interactions are C25—H25$\cdots$C13($1-x$, $1-y$, $1-z$) and C38—H38$\cdots$centroid(C21–C210)($1-x$, $2-y$, $1-z$) with $D\cdots A$ distances of 3.016 (2) and 3.362 Å and D—H$\cdots A$ angles of 173 and 171°, respectively.

Experimental

The title compound was synthesized by the dropwise addition of BuLi (44 ml, 70 mmol, 1.6 *M* solution in hexanes) to a cooled (273 K) solution of 1-naphthol (10 g, 69 mmol) in diethyl ether (100 ml) over *ca*. 30 min. A solution of PCl_3 (2.0 ml, 22 mmol) in diethyl ether (50 ml) was then added dropwise over *ca* 30 min and the reaction mixture was allowed to reach room temperature and stirred for 18 h. Water (300 ml) was added to the yellow suspension and the organic layer separated and dried over anhydrous magnesium sulfate (10 g), filtered, and concentrated to give a yellow oil. Recrystallization from hexane resulted in single crystals suitable for X-ray diffraction (yield 5.5 g, 54%).

Crystal data

$C_{30}H_{21}O_3P$
$M_r = 460.44$
Triclinic, $P\overline{1}$
$a = 7.5057$ (2) Å
$b = 10.8272$ (4) Å
$c = 14.6167$ (5) Å
$\alpha = 106.782$ (2)°
$\beta = 97.780$ (2)°
$\gamma = 92.890$ (1)°
$V = 1121.75$ (6) Å^3
$Z = 2$
Mo $K\alpha$ radiation
$\mu = 0.15$ mm^{-1}
$T = 100$ (2) K
0.24 × 0.12 × 0.10 mm

Data collection

Bruker X8 APEXII diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1998)
$T_{\min} = 0.964$, $T_{\max} = 0.985$
19931 measured reflections
5403 independent reflections
4452 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.037$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.106$
$S = 1.04$
5403 reflections
307 parameters
H-atom parameters constrained
$\Delta\rho_{\max} = 0.38$ e Å^{-3}
$\Delta\rho_{\min} = -0.38$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

P—O1	1.6425 (11)	O1—C11	1.3906 (18)
P—O2	1.6376 (11)	O2—C21	1.3889 (17)
P—O3	1.6086 (11)	O3—C31	1.4026 (17)
O2—P—O1	94.73 (5)	O3—P—O2	101.91 (6)
O3—P—O1	98.02 (5)		
O2—P—O1—C11	86.13 (10)	P—O1—C11—C12	160.36 (10)
O3—P—O2—C21	107.89 (11)	P—O2—C21—C22	161.44 (10)
O1—P—O3—C31	−65.99 (12)	P—O3—C31—C32	109.66 (13)

Table 2
Angles between P—O—C planes and the least-squares plane of the corresponding attached benzene rings of P(O*R*)$_3$ ligands (°).

R	Angle 1	Angle 2	Angle 3	Space Group	Ref.
Ph	87.1	34.1	21.7	$R\overline{3}$	*a*
Ph	82.9	57.4	26.1	$P2_1/n$	*b*
2-MeOPh	57.6	57.6	57.6	$P2_13$	*c*
2,4-di^{t}BuPh	40.9	40.4	37.5	$P\overline{1}$	*d*
2,4-di^{t}BuPh	45.2	40.3	30.9	$P\overline{1}$	*d*
1-Naphthyl	72.6 (2)	19.4 (1)	19.0 (1)	$P\overline{1}$	*e*

Notes: (*a*) Senker et al., 2001, (*b*) Golovanov *et al.*, 2005, (*c*) Baker *et al.*, 1992, (*d*) Crous *et al.*, 2005, (*e*) the title compound, (I).

All H atoms were positioned geometrically and allowed to ride on their parent atoms, with C—H = 0.95 Å and $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(parent).

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT-Plus* (Bruker, 2004); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *DIAMOND* (Brandenburg & Putz, 2006); software used to prepare material for publication: *SHELXL97*.

Financial assistance from the University of the Free State is gratefully acknowledged. Part of this material is based on work supported by the South African National Research Foundation (NRF) under grant number GUN 2068915. Opinions, findings, conclusions or recommendations expressed in this material are those of the authors and do not necessarily reflect the views of the NRF.

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