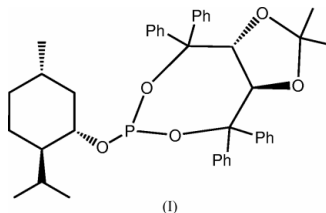


(1*R*,7*R*)-4-[(1*R*,2*S*,5*R*)-2-isopropyl-5-methylcyclohexoxy]-9,9-dimethyl-2,2,6,6-tetraphenyl-3,5,8,10-tetraoxa-4-phosphabicyclo[5.3.0]decane**Benjamin L. Rupert,*
Bryan E. F. Tiedemann and
Sarah M. Vespremi**College of Chemistry, University of California,
Berkeley, Berkeley, CA 94720, USACorrespondence e-mail:
brupert@socrates.berkeley.edu**Key indicators**Single-crystal X-ray study
T = 133 K
Mean σ (C–C) = 0.007 Å
R factor = 0.038
wR factor = 0.039
Data-to-parameter ratio = 7.4For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.The crystal structure of the title compound, C₄₁H₄₇PO₅, has been determined. The absolute configuration assignment was based on knowledge of the chirality of the starting material.Received 12 May 2003
Accepted 3 June 2003
Online 17 June 2003**Comment**Phosphorus-containing compounds are widely used as non-reactive ligands in metal catalysts. The title compound, (I), has been shown to induce enantioselectivity in Cu-mediated conjugate addition. This compound is also of interest as a ligand for Rh(I)-catalysed C–H bond activation. The bulky chiral phosphite is expected to induce enantioselectivity in the addition of C–H bonds across imine π -bonds.

The title compound contains a seven-membered heterocyclic backbone comprised of TADDOL (TADDOL = $\alpha,\alpha,\alpha',\alpha'$ -tetraphenyl-2,2'-dimethyl-1,3-dioxolane-4,5-dimethanol) and phosphorus ($\times 2$), the third substituent on phosphorus being a menthol bonded through oxygen. Bond lengths and angles are typical for organophosphites, and the structure of the TADDOL–phosphorus ring is similar to that of previously reported compounds (Keller *et al.*, 1998 and Sakaki *et al.*, 1993). In these previous examples, the third substituent on phosphorus has been relatively small and achiral (dimethylamine and phenyl, respectively). In the title compound, the larger chiral menthol provides an additional point of asymmetry, and is expected to increase the enantioselectivity of reactions carried out using metal complexes of this ligand.

Experimental

The title compound was prepared by the stepwise reaction of (+)-menthol and (–)-TADDOL with phosphorus trichloride, according to a literature procedure (Alexakis *et al.*, 2000). X-ray quality crystals were obtained by slowly cooling a concentrated solution of the title compound in an ether/pentane mixture to 238 K.

Crystal data

$C_{41}H_{47}O_5P$
 $M_r = 650.79$
 Orthorhombic, $P2_12_12_1$
 $a = 17.884$ (1) Å
 $b = 8.9233$ (6) Å
 $c = 22.395$ (2) Å
 $V = 3574.0$ (4) Å³
 $Z = 4$
 $D_x = 1.209$ Mg m⁻³

Mo $K\alpha$ radiation
 Cell parameters from 4403 reflections
 $\theta = 2.3$ – 22.4°
 $\mu = 0.12$ mm⁻¹
 $T = 133.2$ K
 Column, colorless
 $0.27 \times 0.20 \times 0.09$ mm

Data collection

Bruker SMART 1K CCD diffractometer
 ω scans
 Absorption correction: multi-scan (Blessing, 1995)
 $T_{\min} = 0.967$, $T_{\max} = 0.989$
 15797 measured reflections

5861 independent reflections
 3158 reflections with $I > 3\sigma(I)$
 $R_{\text{int}} = 0.069$
 $\theta_{\text{max}} = 24.7^\circ$
 $h = -21 \rightarrow 20$
 $k = -9 \rightarrow 10$
 $l = -22 \rightarrow 26$

Refinement

Refinement on F
 $R = 0.038$
 $wR = 0.039$
 $S = 0.96$
 3158 reflections
 424 parameters

H-atom parameters constrained
 $w = 1/\sigma^2$
 $(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 0.21$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.32$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

P1–O1	1.610 (3)	P1–O3	1.656 (3)
P1–O2	1.629 (3)		
O1–P1–O2	93.8 (1)	O2–P1–O3	99.8 (1)
O1–P1–O3	95.9 (1)		

When the absolute configuration analysis was performed in *teXsan*, very little change was observed in calculated *versus* observed intensity differences between Friedel pairs upon inversion. The correct enantiomer shows 1225 pairs of reflections with agreement between calculated and observed intensities, and 1184 in disagreement. The known chirality of the starting materials were therefore used to assign the enantiomer.

Data collection: *SMART* (Bruker, 1995–1999); cell refinement: *SAINTE* (Bruker, 2002); data reduction: *SAINTE*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993); program(s) used to refine structure: *teXsan* (Molecular Structure Corporation, 1985, 1992); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *teXsan*.

We thank Reema Thalji for providing crystals of the title compound, Dr Frederick J. Hollander and Dr Allen G. Oliver

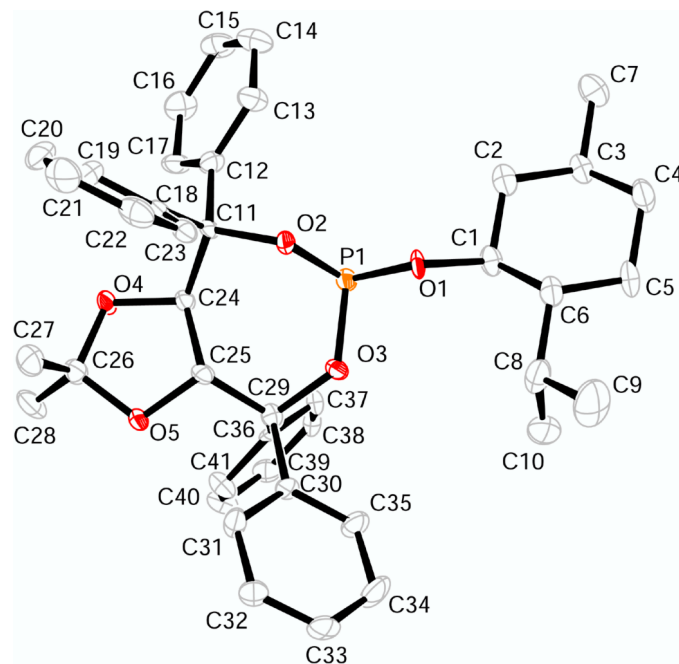


Figure 1

The title compound, with displacement ellipsoids drawn at the 50% probability level. H atoms are omitted for clarity.

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