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(+)-(*R*)-(*tert*-Butylvinylphosphinoyl)benzene

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Key indicators

Single-crystal X-ray study
T = 291 K
Mean σ(C–C) = 0.003 Å
R factor = 0.033
wR factor = 0.049
Data-to-parameter ratio = 16.8

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

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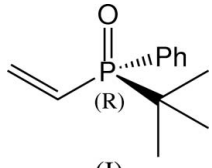
The chiral title compound, $C_{12}H_{17}OP$, with potential uses in asymmetric induction in homogeneous catalysed reactions, shows an s-*cis* conformation for the C═C—P═O unit, in common with related materials.

Comment

The title compound, (I), has been used as a substrate in a research project evaluating different routes to *P*-stereogenic *P*,*N*-ligands for homogeneous catalysis. It can also be used for Michael addition reactions (Maj, 2002), metathesis reactions (Demchuk *et al.*, 2003), and as a preligand for enantioselective transfer hydrogenation of ketones (Maj *et al.*, 1999).



(I)

In the solid state, the C22═C21—P1═O1 unit of the molecule assumes an *s-cis* conformation with a torsion angle of −8.9 (2)° (Fig. 1). This conformational preference has been found without exception in the other three vinylphosphine oxides (Pietrusiewicz, Zablocka, Wieczorek & Brandi, 1991; Pietrusiewicz, Zablocka, Kuznikowski *et al.*, 1991; Wieczorek, 1995), two vinylphosphine sulfides (Pietrusiewicz, Wieczorek et al., 1991; Pietrusiewicz *et al.*, 1992), and one vinylphosphine selenide (Pietrusiewicz, Wieczorek *et al.*, 1991) studied by X-ray diffraction, and has also been considered important in the stereoselective 1,3-dipolar cycloaddition reactions of vinylphosphine chalcogenides with nitrones (Brandi *et al.*, 1989, 1991). The crystal structure of (I) also reveals that an *R*

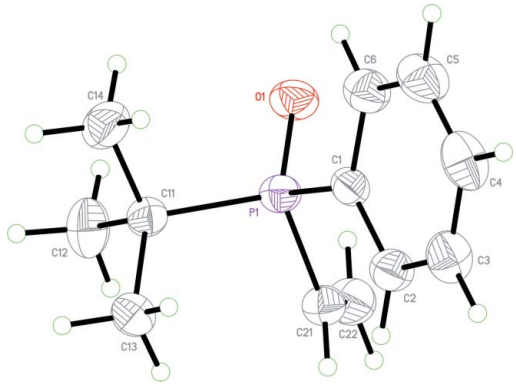


Figure 1
The molecular structure of (I), with displacement ellipsoids drawn at the 30% probability level (arbitrary spheres for the H atoms).

configuration of the P atom correlates with dextrorotatory behaviour.

Experimental

Compound (I) was prepared according to the method of Pietrusiewicz (1996). The *R* enantiomer was determined by chiral high performance liquid chromatography [chiralpak OJ, heptane–2-propanol (6:4), λ = 251 nm, c = 1 mg ml^{-1}, injection volume = 0.5 µl] to be >99% ee.

Crystal data

$C_{12}H_{17}OP$	Z = 4
M_r = 208.23	D_x = 1.159 Mg m^{-3}
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation
a = 6.0852 (4) Å	μ = 0.20 mm^{-1}
b = 7.3750 (6) Å	T = 291 (1) K
c = 26.598 (2) Å	Column, colourless
V = 1193.67 (15) Å^3	0.28 × 0.10 × 0.06 mm

Data collection

Nonius KappaCCD diffractometer	2179 independent reflections
ω scans	1367 reflections with $I > 2\sigma(I)$
Absorption correction: none	R_{int} = 0.046
9543 measured reflections	θ_{max} = 25.4°

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2)]$
$R[F^2 > 2\sigma(F^2)]$ = 0.033	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2)$ = 0.049	$\Delta\rho_{max}$ = 0.21 e Å^{-3}
S = 0.82	$\Delta\rho_{min}$ = −0.19 e Å^{-3}
2179 reflections	Absolute structure: Flack (1983), 862 Friedel pairs
130 parameters	
H-atom parameters constrained	Flack parameter: −0.10 (8)

H atoms were placed in calculated positions, with C—H = 0.93–0.97 Å, and were refined as riding, with U_{iso}(H) = 1.5U_{eq}(C) for methyl groups and 1.2U_{eq}(C) for other H atoms; the methyl groups were allowed to rotate but not to tip.

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* & *SCALEPACK*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL97*, *PARST95* (Nardelli, 1995) and *PLATON* (Spek, 2003).

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