Diphenyl(1,1,3,3-tetramethyl-2,3-dihydro-1*H*-isoindol-2-yl)phosphine oxide

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Key indicators

Single-crystal X-ray study

$T = 293$ K

Mean $\sigma(\text{C}—\text{C}) = 0.005$ Å

R factor = 0.029

wR factor = 0.086

Data-to-parameter ratio = 8.0

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

The title compound, $\text{C}_{24}\text{H}_{26}\text{NOP}$, crystallizes with two independent non-chiral molecules in the asymmetric unit. The molecular structure consists of a tetrahedral P atom bonded by two phenyl groups, a phosphate O atom and an isoindole group. The geometry of the two molecules is similar but not identical.

Comment

The title compound, (I), was obtained as part of an investigation of the use of the radical scavenger 1,1,3,3-tetramethyl-2,3-dihydro-1*H*-isoindol-2-ylxyl to study the reaction of phosphorus-centred radicals with alkenes and alkynes (Busfield *et al.*, 1994; Bottle *et al.*, 1994).

Experimental

The title compound was obtained in 6.3% yield from the reaction of diphenylphosphine (100 mg), di-*tert*-butyl peroxyoxalate (DTBP) (58 mg) and benzene (2.5 ml) (solvent) in the presence of the radical scavenger (103 mg). Reaction mixtures were degassed using repeated freezing/evacuating/thawing cycles on a high vacuum line, then sealed under vacuum in glass and heated for ten half-lives of the initiator, DTBP (68 min at 330 K). Interestingly, in the identical reaction to that above but with no initiator (DTBP) present, the title compound was still obtained (21%), along with the same additional five phosphorus-containing compounds. A small quantity of the title compound was taken up in methanol (HPLC grade) to produce a concentrated sample. One small drop of water (HPLC grade) was then added to the solution. The solution was allowed to stand in the freezer at 253 K (length of time unrecorded) to produce X-ray quality crystals.

Crystal data

$\text{C}_{24}\text{H}_{26}\text{NOP}$

$M_r = 375.43$

Monoclinic, $P2_1$

$a = 14.48$ (2) Å

$b = 8.323$ (1) Å

$c = 18.455$ (5) Å

$\beta = 112.280$ (12)°

$V = 2058$ (3) Å³

$Z = 4$

$D_x = 1.212$ Mg m^{−3}

Mo $K\alpha$ radiation

$\mu = 0.15$ mm^{−1}

$T = 293$ (2) K

Prism, colourless

$0.35 \times 0.22 \times 0.13$ mm

Figure 1
The molecular structure of molecule *A*, showing displacement ellipsoids drawn at the 50% probability level. H atoms are shown as small spheres

Data collection

Enraf–Nonius CAD-4 diffractometer	3878 independent reflections
ω – 2θ scans	3276 reflections with $I > 2\sigma(I)$
Absorption correction: ψ scan (North <i>et al.</i> , 1968)	$R_{\text{int}} = 0.017$
$T_{\text{min}} = 0.950$, $T_{\text{max}} = 0.981$	$\theta_{\text{max}} = 25.0^\circ$
4422 measured reflections	3 standard reflections
	frequency: 120 min
	intensity decay: 2%

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0591P)^2 + 0.1682P]$
$R[F^2 > 2\sigma(F^2)] = 0.029$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.086$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 1.03$	$\Delta\rho_{\text{max}} = 0.18 \text{ e } \text{\AA}^{-3}$
3878 reflections	$\Delta\rho_{\text{min}} = -0.18 \text{ e } \text{\AA}^{-3}$
487 parameters	Absolute structure: Flack (1983), 0
H-atom parameters constrained	Friedel pairs
	Flack parameter: 0.03 (8)

H atoms were placed in calculated positions, with C–H = 0.93 (aromatic) or 0.96 Å (methyl), and included in the refinement in riding-model approximation, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$, or $1.5U_{\text{eq}}(\text{C})$ for methyl H atoms.

Data collection: *SDP* (Frenz, 1985); cell refinement: *SDP*; data reduction: *WinGX* (Farrugia, 1999); program(s) used to solve struc-

Figure 2
The molecular structure of molecule *B*, showing displacement ellipsoids drawn at the 50% probability level. H atoms are shown as small spheres

ture: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97*; molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

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