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

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The title compound, $C_{24}H_{26}NOP$, 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.

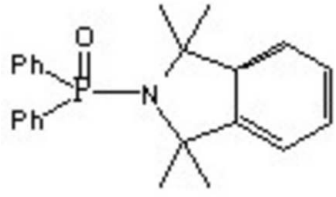
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–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.

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-yloxyl to study the reaction of phosphorus-centred radicals with alkenes and alkynes (Busfield *et al.*, 1994; Bottle *et al.*, 1994).



(I)

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

$C_{24}H_{26}NOP$
M_r = 375.43
Monoclinic, $P2_1$
a = 14.48 (2) Å
b = 8.323 (1) Å
c = 18.455 (5) Å
β = 112.280 (12)°
V = 2058 (3) Å^3
Z = 4
D_x = 1.212 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.15 mm^{-1}
T = 293 (2) K
Prism, colourless
0.35 × 0.22 × 0.13 mm

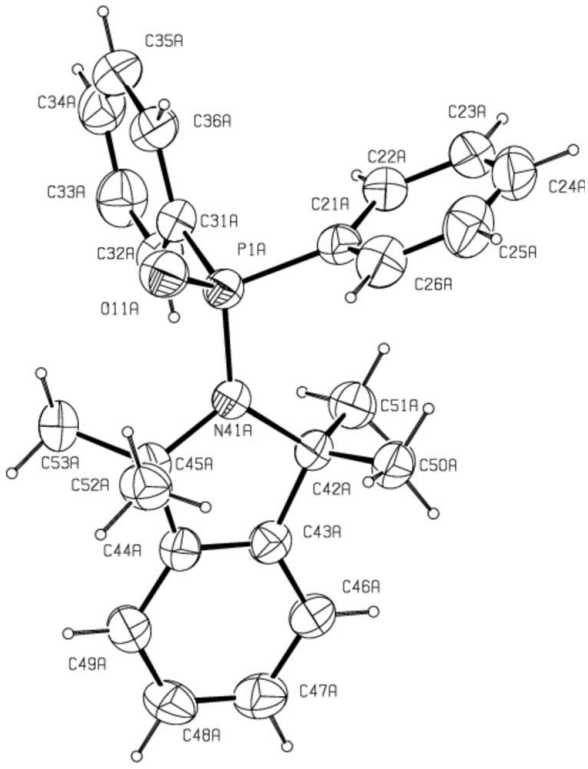


Figure 1
The molecular structure of molecule *A*, showing displacement ellipsoids drawn at the 50% probability level. H atoms are shown as small spheres

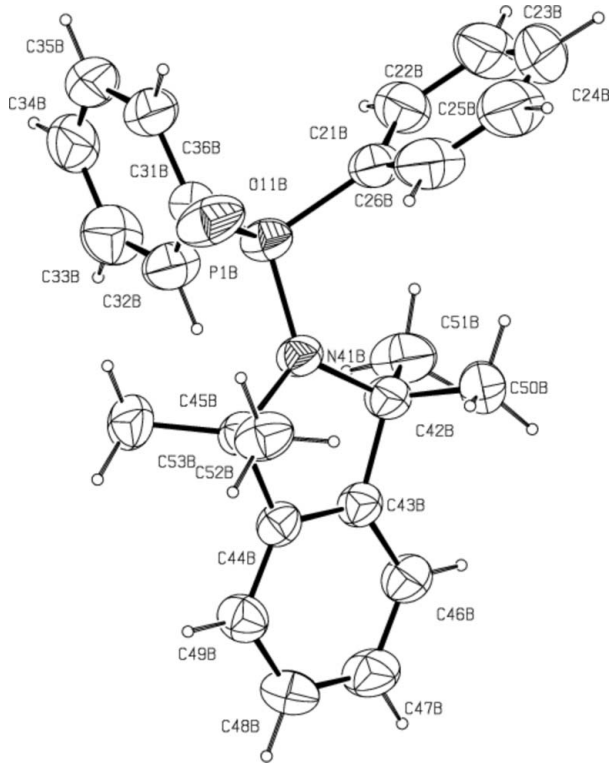


Figure 2
The molecular structure of molecule *B*, showing displacement ellipsoids drawn at the 50% probability level. H atoms are shown as small spheres

Data collection

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

Refinement

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

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

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