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trans-2-(Dimethoxyphosphinoyl)-1-phenyl-1-(1,1,3,3-tetramethyl-2,3-dihydro-1*H*-isoindol-2-yloxy)ethene

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.057
wR factor = 0.200
Data-to-parameter ratio = 15.2

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

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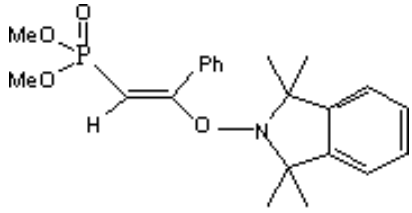
The central feature in the title molecule, $C_{22}H_{28}NO_4P$, is a tetrahedral P atom attached to two methoxy groups, a phosphate oxygen and an isoindoloxy group.

Comment

The title compound, (I), was part of an investigation using 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)

There is a possible C2—H2···N5 intramolecular hydrogen bond [2.745 (3) Å, with an angle of 107 (2)°], and an intermolecular link.

Experimental

The title compound (I) was obtained in 85.4% yield from the reaction of dimethyl phosphite (337 µl, 10 equivalents), di-*tert*-butyl peroxyoxalate (DTBP) (86 mg, 1 equivalent) and phenylacetylene (2.0 ml) (solvent) in the presence of the radical scavenger 1,1,3,3-tetramethyl-

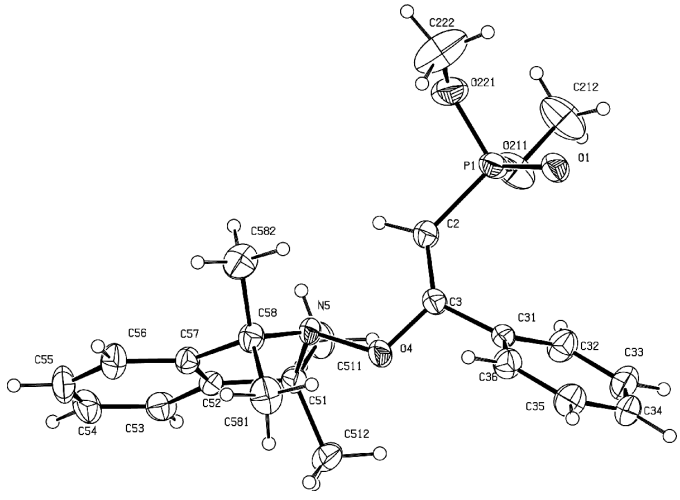


Figure 1
Molecular structure of (I), showing displacement ellipsoids at the 50% probability level.

2,3-dihydro-1*H*-isoindol-2-yloxyl (160 mg, 2.1 equivalents). Dimethyl-(1,1,3,3-tetramethyl-2,3-dihydro-1*H*-isoindol-2-yloxy) phosphate, (II) (14.6%), was also isolated from the reaction.

Reaction mixtures were degassed using repeated freezing/evacuating/thawing cycles on a high vacuum line, then sealed under vacuum in glass and heated for 10 half lives of the initiator, DTBP (68 minutes at 333 K). The reaction mixture was then separated by HPLC (Whatman Partisil 10–ODS-3 500 × 10 mm C18) using an isocratic 80:20 methanol:water, 6.0 ml min^{-1} method.

The two phosphorus-containing reaction products were eluted from the reversed-phase HPLC column in the following order.

(I) *trans*-2-(Dimethoxyphosphinoyl)-1-phenyl-1-(1,1,3,3-tetramethyl-2,3-dihydro-1*H*-isoindol-2-yloxy)-ethene. ^{1}H NMR (250 MHz, CD_3OD): δ 1.42, 1.43, 1.61, 12H, 4CH_3; 4.91, *s*, 6H, $(OCH_3)_2$; 6.03, *d*, $^2J_{P,H}$ 9.7, 1H, CHP; 7.11, *m*, 7.20, *m*, H4, H7; 7.29, *m*, 2H, H5, H6; 7.45, *m*, 3H, 7.64, *m*, 2H, Phenyl; ^{13}C NMR (62.8 MHz, CD_3OD): δ 25.8, 26.5, 30.0, 33.5, 4 × ring CH_3; 51.3, $^2J_{P,C}$ 172.0, $(OCH_3)_2$; 70.4, C1, C3; 86.0. $^1J_{P,C}$ 210.0, *h.c.p.*; 122.7, C4, C7; 123.3, *ortho* C; 127.5, C5, C6; 129.0, $^2J_{P,C}$ 3.1, NOC(Ph)CH; 129.3, C5, C6; 129.9, *para* C; 131.6, *meta* C; 134.8, *ipso* C; 146.2, C3a, C7a. ^{31}P NMR (121 MHz, CD_3OD): δ 24.03. Structural confirmation by X-ray structural analysis.

(II) Dimethyl (1,1,3,3-tetramethyl-2,3-dihydro-1*H*-isoindol-2-yloxy) phosphate. Identical to an authentic sample (Busfield *et al.*, 1994).

Crystal data

$C_{22}H_{28}NO_4P$	D_x = 1.212 Mg m^{-3}
M_r = 401.42	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 25 reflections
a = 8.490 (4) Å	θ = 10.0–12.0°
b = 13.971 (2) Å	μ = 0.15 mm^{-1}
c = 18.552 (8) Å	T = 293 (2) K
β = 90.15 (2)°	Block, colourless
V = 2200.5 (14) Å^3	0.25 × 0.12 × 0.10 mm
Z = 4	

Data collection

Enraf–Nonius CAD-4 diffractometer	R_{int} = 0.027
ω–2θ scans	θ_{max} = 25.0°
Absorption correction: ψ scan (North *et al.*, 1968)	h = 0 → 10
T_{min} = 0.974, T_{max} = 0.999	k = 0 → 16
4131 measured reflections	l = −22 → 22
3853 independent reflections	3 standard reflections
2480 reflections with $I > 2\sigma(I)$	frequency: 120 min
	intensity decay: none

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.057	$w = 1/[\sigma^2(F_o^2) + (0.1P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.200	$(\Delta/\sigma)_{max} < 0.001$
S = 1.37	$\Delta\rho_{max}$ = 0.42 e Å^{-3}
3853 reflections	$\Delta\rho_{min}$ = −0.33 e Å^{-3}
253 parameters	

Table 1
Selected geometric parameters (Å, °).

P1—O1	1.4531 (19)	C2—C3	1.329 (3)
P1—C2	1.753 (3)	C3—O4	1.372 (3)
P1—O221	1.563 (2)	O4—N5	1.457 (2)
P1—O211	1.587 (2)		
O1—P1—O221	114.49 (12)	C2—C3—O4	125.1 (2)
O1—P1—O211	115.14 (13)	C2—C3—C31	128.0 (2)
O221—P1—O211	100.15 (14)	C3—C2—P1	126.7 (2)
O1—P1—C2	118.34 (12)	C3—O4—N5	114.18 (17)
O221—P1—C2	105.00 (13)	C51—N5—C58	111.61 (18)
O211—P1—C2	101.36 (13)		

There is a great variation of U_{aniso} for some of the non-H atoms. H atoms were positioned geometrically (C—H = 0.93–0.96 Å) and refined as riding, with U_{iso}(H) = 1.2 or 1.5 times U_{eq}(C).

Data collection: *SDP* (Frenz, 1985); cell refinement: *SDP*; data reduction: *WinGX* (Farrugia, 1999); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *PLATON*98 (Spek, 1988); software used to prepare material for publication: *SHELXL*97.

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