I. Darren Grice,^a Ian D. Jenkins,^b
W. Ken Busfield,^c Karl A. Byriel^d
and Colin H. L. Kennard^{e*}

^aInstitute for Glycomics, Griffith University,
Gold Coast, Queensland, 4215, Australia,

^bNatural Product Discovery, Griffith University,
Nathan, Brisbane, Queensland, 4111, Australia,

^cSchool of Science, Griffith University, Nathan,
Brisbane, Queensland, 4111, Australia, ^dCentre
for Drug Design and Development, The
University of Queensland, Brisbane,
Queensland, 4072, Australia, and ^eChemistry
Department, School of Molecular and Microbial
Sciences, The University of Queensland,
Brisbane, Queensland, 4072, Australia

Correspondence e-mail: C.Kennard@uq.edu.au

Key indicators

Single-crystal X-ray study

$T = 293\text{ K}$

Mean $\sigma(\text{C}-\text{C}) = 0.004\text{ \AA}$

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>.

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

The central feature in the title molecule, $\text{C}_{22}\text{H}_{28}\text{NO}_4\text{P}$, is a tetrahedral P atom attached to two methoxy groups, a phosphate oxygen and an isoindoloxyl group.

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

There is a possible $\text{C2}-\text{H2}\cdots\text{N5}$ intramolecular hydrogen bond [$2.745(3)\text{ \AA}$, with an angle of $107(2)^\circ$], 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-yl-oxyl (160 mg, 2.1 equivalents). Dimethyl-(1,1,3,3-tetramethyl-2,3-dihydro-1*H*-isoindol-2-yl-oxo) 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-yl-oxo)-ethene. ¹H NMR (250 MHz, CD₃OD): δ 1.42, 1.43, 1.61, 12H, 4CH₃; 4.91, *s*, 6H, (OCH₃)₂; 6.03, *d*, ²J_{PH} 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; ¹³C NMR (62.8 MHz, CD₃OD): δ 25.8, 26.5, 30.0, 33.5, 4 × ring CH₃; 51.3, ²J_{PC} 172.0, (OCH₃)₂; 70.4, C1, C3; 86.0, ¹J_{PC} 210.0, *h.c.p.*; 122.7, C4, C7; 123.3, *ortho* C; 127.5, C5, C6; 129.0, ²J_{PC} 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. ³¹P NMR (121 MHz, CD₃OD): δ 24.03. Structural confirmation by X-ray structural analysis.

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

Crystal data

C₂₂H₂₈NO₄P
M_r = 401.42
Monoclinic, *P*₂₁/*c*
a = 8.490 (4) Å
b = 13.971 (2) Å
c = 18.552 (8) Å
β = 90.15 (2)°
V = 2200.5 (14) Å³
Z = 4

*D*_x = 1.212 Mg m⁻³
Mo *K*α radiation
Cell parameters from 25 reflections
θ = 10.0–12.0°
μ = 0.15 mm⁻¹
T = 293 (2) K
Block, colourless
0.25 × 0.12 × 0.10 mm

Data collection

Enraf-Nonius CAD-4
diffractometer
ω–2θ scans
Absorption correction: ψ scan
(North *et al.*, 1968)
*T*_{min} = 0.974, *T*_{max} = 0.999
4131 measured reflections
3853 independent reflections
2480 reflections with *I* > 2σ(*I*)

*R*_{int} = 0.027
θ_{max} = 25.0°
h = 0 → 10
k = 0 → 16
l = –22 → 22
3 standard reflections
frequency: 120 min
intensity decay: none

Refinement

Refinement on *F*²
R [*F*² > 2σ(*F*²)] = 0.057
wR(*F*²) = 0.200
S = 1.37
3853 reflections
253 parameters

H-atom parameters constrained
w = 1/[σ²(*F*_o²) + (0.1*P*)²]
where *P* = (*F*_o² + 2*F*_c²)/3
(Δ/σ)_{max} < 0.001
Δρ_{max} = 0.42 e Å⁻³
Δρ_{min} = –0.33 e Å⁻³

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: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON98* (Spek, 1988); software used to prepare material for publication: *SHELXL97*.

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