

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

Dimethyl [1-(1-allyl-5-iodo-1*H*-indol-3-yl)-3-hydroxypropyl]phosphonate

Ying-Cen Guo, Xu-Fan Wang and Yu Ding*

Key Laboratory of Pesticides and Chemical Biology of Ministry of Education, College of Chemistry, Central China Normal University, Wuhan 430079, People's Republic of China

Correspondence e-mail: dingyu508@gmail.com

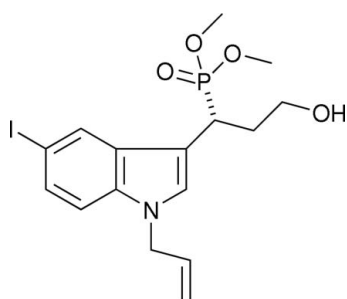
Received 29 November 2007; accepted 21 December 2007

Key indicators: single-crystal X-ray study; $T = 294$ K; mean $\sigma(\text{C}-\text{C}) = 0.007$ Å; R factor = 0.043; wR factor = 0.102; data-to-parameter ratio = 16.9.

In the title compound, $\text{C}_{16}\text{H}_{21}\text{INO}_4\text{P}$, the molecular structure is stabilized by a weak intramolecular $\text{C}-\text{H}\cdots\text{O}$ hydrogen-bond interaction. The crystal packing is stabilized by strong intermolecular $\text{O}-\text{H}\cdots\text{O}$ hydrogen-bonding interactions to form a zigzag packing arrangement.

Related literature

For asymmetric synthesis of phosphorus compounds, see: Carlone *et al.* (2007); Yang, *et al.* (2007); Ibrahim *et al.* (2007). For related structures, see: Sonar *et al.* (2006); Chen *et al.* (2007); Butcher *et al.* (2007). For related literature, see: Allen *et al.* (1989); Horiguchi & Kandatsu (1959).



Experimental

Crystal data

 $\text{C}_{16}\text{H}_{21}\text{INO}_4\text{P}$ $M_r = 449.21$ Orthorhombic, $P2_12_12_1$ $a = 7.9983$ (4) Å $b = 10.1295$ (6) Å $c = 22.3722$ (12) Å $V = 1812.57$ (17) Å³ $Z = 4$ Mo $K\alpha$ radiation $\mu = 1.87$ mm⁻¹ $T = 294$ (2) K

0.20 × 0.10 × 0.10 mm

Data collection

Bruker SMART CCD area-detector diffractometer

Absorption correction: multi-scan (SADABS; Sheldrick, 1997)

 $T_{\min} = 0.706$, $T_{\max} = 0.835$

10836 measured reflections

3564 independent reflections

3314 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.051$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.042$ $wR(F^2) = 0.102$ $S = 1.04$

3564 reflections

211 parameters

H-atom parameters constrained

 $\Delta\rho_{\text{max}} = 0.69$ e Å⁻³ $\Delta\rho_{\text{min}} = -0.30$ e Å⁻³

Absolute structure: Flack (1983),

1503 Freidel pairs

Flack parameter: 0.00 (1)

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{C12}-\text{H12}\cdots\text{O1}$	0.98	2.47	2.873 (7)	104
$\text{O1}-\text{H1}\cdots\text{O4}^i$	0.82	1.92	2.733 (6)	174

Symmetry code: (i) $-x, y - \frac{1}{2}, -z + \frac{3}{2}$.

Data collection: SMART (Bruker, 2001); cell refinement: SAINT (Bruker, 2001); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: SHELXTL (Bruker, 2001); software used to prepare material for publication: SHELXTL.

The authors acknowledge financial support from the Natural Science Foundation of Hubei Province of China (No. 2006ABA175) and the Natural Science Foundation of Central China Normal University, and are indebted to Dr Guangmin Yao of Shijiazhuang Pharmaceutical Group, Zhongqi (sjz) Pharmaceutical Technology Co. Ltd, and Professor Wenjing Xiao and Dr Xianggao Meng of Central China Normal University, for their advice and support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2520).

References

- Allen, M. C., Fuhrer, W. & Tuck, B. (1989). *J. Med. Chem.* **32**, 1652–1661.
- Bruker (2001). SMART (Version 5.628), SAINT (Version 6.45) and SHELXTL (Version 6.12). Bruker AXS Inc., Madison, Wisconsin, USA.
- Butcher, R. J., Jasinski, J. P., Yathirajan, H. S., Ashalatha, B. V. & Narayana, B. (2007). *Acta Cryst.* **E63**, o3505.
- Carlone, A., Bartoli, G. & Bosco, M. (2007). *Angew. Chem. Int. Ed.* **46**, 4504–4506.
- Chen, A., Zou, J.-W. & Zhao, W.-N. (2007). *Acta Cryst.* **E63**, o3229.
- Flack, H. D. (1983). *Acta Cryst.* **A39**, 876–881.
- Horiguchi, M. & Kandatsu, M. (1959). *Nature (London)*, **184**, 901.
- Ibrahim, I., Rios, R. & Vesely, J. (2007). *Angew. Chem. Int. Ed.* **46**, 4507–4510.
- Sheldrick, G. M. (1997). SADABS and SHELXL97. University of Göttingen, Germany.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Sonar, V. N., Parkin, S. & Crooks, P. A. (2006). *Acta Cryst.* **E62**, o3328–o3330.
- Yang, H., Hong, Y.-T. & Kim, S. (2007). *Org. Lett.* **9**, 2281–2284.

supplementary materials

Acta Cryst. (2008). E64, o384 [doi:10.1107/S1600536807068171]

Dimethyl [1-(1-allyl-5-iodo-1*H*-indol-3-yl)-3-hydroxypropyl]phosphonate

Y.-C. Guo, X.-F. Wang and Y. Ding

Comment

Phosphorus has been recognized as an essential structural constituent of many biomolecules and a crucial element in many biological transformations (Horiguchi & Kandatsu, 1959; Allen *et al.* 1989). With the advances in the development of chiral catalysts, asymmetric synthesis of some phosphorus compounds has been well documented in literatures (Carlone *et al.*, 2007; Yang, *et al.*, 2007; Ibrahim *et al.*, 2007). As part of our ongoing project on enantioselective organocatalysis, a series of α -indolyl phosphonates have been highly enantioselectively synthesized *via* Friedel-Crafts alkylation of substituted indoles with (*E*)-dialkyl 3-oxoprop-1-enylphosphonate using MacMillan's imidazolidinone catalysts. The title compound (I) was synthesized and its crystal structure is presented here.

As seen in Fig. 1, the pyrrole plane (C1/C6—C8/N1) and the phenyl ring (C1—C6) are coplanar with each other with a dihedral angle between their mean planes of 2.2 (24) $^\circ$ in the molecule of the title compound (I), and the molecular structure is stabilized by a weak C12—H12 \cdots O1 intramolecular hydrogen bonding interaction (Table 1).

The crystal packing (Fig. 2) is stabilized by strong intermolecular O—H \cdots O hydrogen bonds interaction (Table 1) to form a zigzag packing arrangement. Dipole–dipole and van der Waals interactions are also effective in the molecular packing in the crystal structure.

Experimental

The MacMillan's imidazolidinone catalysts (Fig 3) (0.02 mmol) and TFA (0.02 mmol) were stirred in 1 ml dichloromethane for 5 min at 195 K, then (*E*)-dialkyl 3-oxoprop-1-enylphosphonate 2 (0.1 mmol) was added and stirred for 15 min. And then 1-allyl-5-iodo-1*H*-indole 1 (0.11 mmol) was added. After the mixture was stirred for 48 h, the solvent (dichloromethane) was removed under reduced pressure at room temperature, and then the residue was added to a stirring solution of sodium borohydride (18.6 mg, 0.5 mmol, 5 equiv) in methanol (3.0 ml). After 15 min, the reaction was quenched with saturated aqueous NaHCO₃ and extracted with CH₂Cl₂. The combined organic layer was washed with saturated solutions of NaHCO₃ and NaCl and then dried over with Na₂SO₄. The combined organic layer was concentrated *in vacuo* and the product was purified by flash column chromatography on silica gel (ethyl acetate, R_f = 0.4), (14.3 mg, 48% yield). Compound 5 m: yellow solid; tr (minor) = 53.8 min, tr (major) = 59.5 min (Chiralcel AD—H, λ = 254 nm, 5% i-PrOH / hexanes, flow rate = 1.0 ml/min). ¹H NMR (400 MHz, CDCl₃) δ 2.10–2.18 (m, 1H), 2.28–2.35 (m, 1H), 2.59 (br, 1H), 3.49 (d, J = 10.8 Hz, 3H), 3.73 (d, J = 10.8 Hz, 3H), 3.50–3.75 (m, 3H), 4.67 (d, J = 5.2 Hz, 2H), 5.00 (d, J = 16.8 Hz, 1H), 5.19 (d, J = 10.4 Hz, 1H), 5.90–6.02 (m, 1H), 7.05–7.47 (m, 3H), 7.98 (s, 1H). ¹³C NMR (100 MHz, CDCl₃) δ 29.9, 31.3, 33.3, 48.9, 52.67, 52.75, 53.4, 53.5, 59.7, 59.8, 83.0, 107.9, 111.8, 117.5, 127.9, 128.5, 129.5, 130.2, 132.8, 135.2.; MS (EI) m/z 449 (M^+), 450 (M^+ +1), 451 (M^+ +2). Elemental Analysis calculated for C₁₆H₂₁INO₄P: C 42.78, H 4.71, N 3.12%. Found: C 42.58, H 4.94, N 3.99%. [α]_D23 = –22.62 (C=2.35, CHCl₃) (after recrystallization). Single crystals of (I) suitable for X-ray diffraction were obtained by slow evaporation of a CHCl₃ solution.

Refinement

All H atoms were positioned geometrically and treated as riding on their parent atoms, with $C-H(\text{methyl}) = 0.96 \text{ \AA}$, $C-H(\text{methylene}) = 0.97 \text{ \AA}$, $C-H(\text{methine}) = 0.98 \text{ \AA}$, $C-H(\text{aromatic}) = 0.93 \text{ \AA}$ and $O-H = 0.82 \text{ \AA}$, and with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C}_{\text{methyl}}, \text{O})$ and $1.2U_{\text{eq}}(\text{C}_{\text{aromatic}}, \text{C}_{\text{methylene}}, \text{C}_{\text{methineylene}})$.

Figures

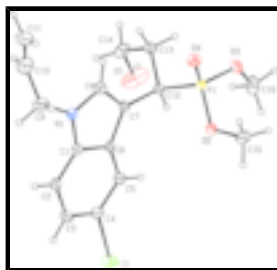


Fig. 1. The molecular structure of (I), showing the atom-labelling scheme and 50% probability displacement ellipsoids.

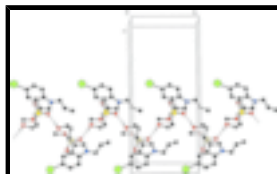


Fig. 2. The packing view of the title compound (I) along a axis, showing intermolecular hydrogen bonds as dashed lines. H atoms not involved in hydrogen bonding have been omitted for clarity..



Fig. 3. The reaction scheme.

Dimethyl [1-(1-allyl-5-iodo-1*H*-indol-3-yl)-3-hydroxypropyl]phosphonate

Crystal data

$C_{16}H_{21}INO_4P$

$M_r = 449.21$

Orthorhombic, $P2_12_12_1$

Hall symbol: P 2ac 2ab

$a = 7.9983 (4) \text{ \AA}$

$b = 10.1295 (6) \text{ \AA}$

$c = 22.3722 (12) \text{ \AA}$

$V = 1812.57 (17) \text{ \AA}^3$

$Z = 4$

$F_{000} = 896$

$D_x = 1.646 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation

$\lambda = 0.71073 \text{ \AA}$

Cell parameters from 5073 reflections

$\theta = 2.2\text{--}25.6^\circ$

$\mu = 1.87 \text{ mm}^{-1}$

$T = 294 (2) \text{ K}$

Block, colourless

$0.20 \times 0.10 \times 0.10 \text{ mm}$

Data collection

Bruker SMART CCD area-detector diffractometer

Monochromator: graphite

$T = 294(2) \text{ K}$

3314 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.051$

$\theta_{\text{max}} = 26.0^\circ$

phi and ω scans $\theta_{\min} = 1.8^\circ$
 Absorption correction: multi-scan $h = -7 \rightarrow 9$
 (SADABS; Sheldrick, 1997)
 $T_{\min} = 0.706$, $T_{\max} = 0.835$ $k = -11 \rightarrow 12$
 10836 measured reflections $l = -27 \rightarrow 27$
 3564 independent reflections

Refinement

Refinement on F^2 Hydrogen site location: inferred from neighbouring sites
 Least-squares matrix: full H-atom parameters constrained
 $R[F^2 > 2\sigma(F^2)] = 0.042$ $w = 1/[\sigma^2(F_o^2) + (0.0573P)^2 + 0.6855P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $wR(F^2) = 0.102$ $(\Delta/\sigma)_{\max} < 0.001$
 $S = 1.04$ $\Delta\rho_{\max} = 0.69 \text{ e } \text{\AA}^{-3}$
 3564 reflections $\Delta\rho_{\min} = -0.30 \text{ e } \text{\AA}^{-3}$
 211 parameters Extinction correction: none
 Primary atom site location: structure-invariant direct methods Absolute structure: Flack (1983), 1503 Freidel pairs
 Secondary atom site location: difference Fourier map Flack parameter: 0.00 (1)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
I1	-0.17244 (5)	-0.23507 (4)	1.053601 (16)	0.06002 (15)
C1	-0.1488 (6)	0.1982 (4)	0.96323 (18)	0.0340 (9)
C2	-0.2151 (6)	0.1860 (5)	1.0201 (2)	0.0408 (11)
H2	-0.2575	0.2587	1.0404	0.049*
C3	-0.2158 (7)	0.0611 (6)	1.0455 (2)	0.0460 (12)
H3	-0.2580	0.0495	1.0838	0.055*
C4	-0.1542 (7)	-0.0467 (5)	1.0142 (2)	0.0414 (11)
C5	-0.0842 (6)	-0.0348 (5)	0.95801 (19)	0.0364 (10)
H5	-0.0414	-0.1081	0.9382	0.044*
C6	-0.0794 (5)	0.0896 (4)	0.93175 (17)	0.0299 (9)
C7	-0.0233 (5)	0.1394 (4)	0.87529 (18)	0.0303 (9)

supplementary materials

C8	-0.0615 (5)	0.2707 (5)	0.87450 (19)	0.0365 (10)
H8	-0.0395	0.3272	0.8427	0.044*
C9	-0.1972 (10)	0.4394 (5)	0.9422 (2)	0.0627 (17)
H9A	-0.2818	0.4309	0.9731	0.075*
H9B	-0.1050	0.4893	0.9591	0.075*
C10	-0.2659 (14)	0.5131 (9)	0.8942 (4)	0.105 (3)
H10	-0.3574	0.4747	0.8753	0.126*
C11	-0.2201 (13)	0.6240 (7)	0.8734 (3)	0.089 (3)
H11A	-0.1294	0.6679	0.8902	0.107*
H11B	-0.2772	0.6613	0.8414	0.107*
C12	0.0602 (6)	0.0611 (5)	0.82594 (19)	0.0324 (9)
H12	0.0481	-0.0329	0.8355	0.039*
C13	-0.0169 (6)	0.0843 (5)	0.76336 (19)	0.0414 (11)
H13A	-0.0210	0.1785	0.7555	0.050*
H13B	0.0547	0.0443	0.7334	0.050*
C14	-0.1891 (8)	0.0284 (6)	0.7576 (2)	0.0541 (14)
H14A	-0.2652	0.0777	0.7829	0.065*
H14B	-0.2268	0.0374	0.7166	0.065*
C15	0.5194 (7)	0.0595 (7)	0.9005 (3)	0.0577 (15)
H15A	0.5880	0.0042	0.8757	0.087*
H15B	0.5363	0.0367	0.9417	0.087*
H15C	0.5494	0.1503	0.8943	0.087*
C16	0.3565 (9)	-0.1289 (6)	0.7724 (3)	0.0686 (18)
H16A	0.4059	-0.1626	0.8084	0.103*
H16B	0.4166	-0.1620	0.7384	0.103*
H16C	0.2419	-0.1566	0.7701	0.103*
N1	-0.1369 (5)	0.3078 (4)	0.92702 (16)	0.0379 (9)
O1	-0.1922 (9)	-0.1050 (5)	0.7740 (3)	0.112 (2)
H1	-0.2269	-0.1492	0.7459	0.168*
O2	0.3445 (4)	0.0404 (3)	0.88494 (12)	0.0386 (7)
O3	0.3641 (4)	0.0129 (4)	0.77276 (15)	0.0494 (9)
O4	0.3217 (5)	0.2376 (3)	0.81376 (16)	0.0521 (8)
P1	0.28050 (14)	0.09837 (13)	0.82350 (5)	0.0338 (3)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
II	0.0725 (3)	0.0485 (2)	0.0591 (2)	-0.00777 (18)	0.00460 (18)	0.01635 (16)
C1	0.032 (2)	0.034 (2)	0.036 (2)	0.0011 (18)	-0.0051 (17)	-0.0074 (16)
C2	0.037 (2)	0.050 (3)	0.036 (2)	0.006 (2)	0.0039 (19)	-0.006 (2)
C3	0.047 (3)	0.058 (3)	0.032 (2)	0.000 (2)	0.008 (2)	0.001 (2)
C4	0.041 (3)	0.045 (3)	0.038 (2)	-0.003 (2)	-0.003 (2)	0.0070 (19)
C5	0.032 (2)	0.037 (2)	0.040 (3)	-0.0025 (19)	0.0006 (19)	-0.0012 (19)
C6	0.024 (2)	0.038 (2)	0.028 (2)	0.0039 (18)	-0.0029 (16)	-0.0024 (17)
C7	0.023 (2)	0.038 (2)	0.030 (2)	0.0002 (18)	-0.0002 (16)	-0.0048 (17)
C8	0.037 (2)	0.038 (2)	0.035 (2)	-0.003 (2)	0.0003 (18)	-0.0033 (19)
C9	0.104 (5)	0.039 (3)	0.045 (3)	0.022 (3)	0.005 (4)	-0.002 (2)
C10	0.141 (8)	0.083 (6)	0.090 (6)	0.057 (6)	0.025 (5)	0.008 (5)

C11	0.157 (8)	0.053 (4)	0.058 (4)	0.043 (5)	0.035 (4)	0.010 (3)
C12	0.034 (2)	0.032 (2)	0.031 (2)	0.0014 (19)	0.0026 (18)	-0.0003 (18)
C13	0.042 (3)	0.054 (3)	0.028 (2)	0.002 (2)	-0.0016 (19)	-0.007 (2)
C14	0.056 (3)	0.051 (3)	0.055 (3)	0.000 (3)	-0.026 (3)	-0.006 (2)
C15	0.038 (3)	0.080 (4)	0.056 (3)	-0.006 (3)	-0.013 (2)	0.014 (3)
C16	0.060 (4)	0.062 (4)	0.083 (4)	0.011 (3)	0.004 (3)	-0.029 (3)
N1	0.042 (2)	0.035 (2)	0.0367 (19)	0.0083 (17)	0.0030 (16)	-0.0041 (15)
O1	0.153 (6)	0.054 (3)	0.129 (5)	-0.034 (4)	-0.088 (4)	0.005 (3)
O2	0.0300 (16)	0.0523 (19)	0.0334 (15)	-0.0042 (16)	-0.0025 (14)	0.0044 (13)
O3	0.035 (2)	0.071 (3)	0.0416 (18)	0.0069 (17)	0.0066 (15)	-0.0039 (17)
O4	0.0469 (19)	0.047 (2)	0.062 (2)	-0.007 (2)	-0.0022 (16)	0.0139 (16)
P1	0.0292 (6)	0.0419 (7)	0.0304 (5)	0.0007 (5)	0.0017 (4)	0.0023 (5)

Geometric parameters (Å, °)

I1—C4	2.107 (5)	C11—H11B	0.9300
C1—N1	1.378 (6)	C12—C13	1.548 (6)
C1—C2	1.383 (6)	C12—P1	1.803 (4)
C1—C6	1.419 (6)	C12—H12	0.9800
C2—C3	1.387 (7)	C13—C14	1.495 (8)
C2—H2	0.9300	C13—H13A	0.9700
C3—C4	1.387 (7)	C13—H13B	0.9700
C3—H3	0.9300	C14—O1	1.400 (7)
C4—C5	1.382 (6)	C14—H14A	0.9700
C5—C6	1.391 (7)	C14—H14B	0.9700
C5—H5	0.9300	C15—O2	1.455 (6)
C6—C7	1.432 (6)	C15—H15A	0.9600
C7—C8	1.365 (7)	C15—H15B	0.9600
C7—C12	1.514 (6)	C15—H15C	0.9600
C8—N1	1.374 (5)	C16—O3	1.438 (7)
C8—H8	0.9300	C16—H16A	0.9600
C9—C10	1.419 (10)	C16—H16B	0.9600
C9—N1	1.458 (6)	C16—H16C	0.9600
C9—H9A	0.9700	O1—H1	0.8200
C9—H9B	0.9700	O2—P1	1.580 (3)
C10—C11	1.270 (12)	O3—P1	1.576 (4)
C10—H10	0.9300	O4—P1	1.464 (4)
C11—H11A	0.9300		
N1—C1—C2	129.7 (4)	C7—C12—H12	107.8
N1—C1—C6	107.8 (4)	C13—C12—H12	107.8
C2—C1—C6	122.5 (4)	P1—C12—H12	107.8
C1—C2—C3	117.3 (4)	C14—C13—C12	112.8 (4)
C1—C2—H2	121.3	C14—C13—H13A	109.0
C3—C2—H2	121.3	C12—C13—H13A	109.0
C4—C3—C2	120.7 (4)	C14—C13—H13B	109.0
C4—C3—H3	119.7	C12—C13—H13B	109.0
C2—C3—H3	119.7	H13A—C13—H13B	107.8
C5—C4—C3	122.2 (4)	O1—C14—C13	111.1 (5)
C5—C4—H1	119.2 (4)	O1—C14—H14A	109.4

supplementary materials

C3—C4—I1	118.5 (3)	C13—C14—H14A	109.4
C4—C5—C6	118.4 (4)	O1—C14—H14B	109.4
C4—C5—H5	120.8	C13—C14—H14B	109.4
C6—C5—H5	120.8	H14A—C14—H14B	108.0
C5—C6—C1	118.8 (4)	O2—C15—H15A	109.5
C5—C6—C7	134.4 (4)	O2—C15—H15B	109.5
C1—C6—C7	106.7 (4)	H15A—C15—H15B	109.5
C8—C7—C6	106.5 (4)	O2—C15—H15C	109.5
C8—C7—C12	126.8 (4)	H15A—C15—H15C	109.5
C6—C7—C12	126.7 (4)	H15B—C15—H15C	109.5
C7—C8—N1	110.8 (4)	O3—C16—H16A	109.5
C7—C8—H8	124.6	O3—C16—H16B	109.5
N1—C8—H8	124.6	H16A—C16—H16B	109.5
C10—C9—N1	115.6 (5)	O3—C16—H16C	109.5
C10—C9—H9A	108.4	H16A—C16—H16C	109.5
N1—C9—H9A	108.4	H16B—C16—H16C	109.5
C10—C9—H9B	108.4	C8—N1—C1	108.2 (4)
N1—C9—H9B	108.4	C8—N1—C9	126.6 (4)
H9A—C9—H9B	107.4	C1—N1—C9	125.2 (4)
C11—C10—C9	129.1 (11)	C14—O1—H1	109.5
C11—C10—H10	115.5	C15—O2—P1	118.1 (3)
C9—C10—H10	115.5	C16—O3—P1	122.4 (4)
C10—C11—H11A	120.0	O4—P1—O3	109.0 (2)
C10—C11—H11B	120.0	O4—P1—O2	114.5 (2)
H11A—C11—H11B	120.0	O3—P1—O2	106.55 (19)
C7—C12—C13	113.8 (4)	O4—P1—C12	115.2 (2)
C7—C12—P1	110.1 (3)	O3—P1—C12	108.7 (2)
C13—C12—P1	109.3 (3)	O2—P1—C12	102.26 (19)
N1—C1—C2—C3	177.8 (5)	C7—C12—C13—C14	-69.1 (6)
C6—C1—C2—C3	-1.6 (7)	P1—C12—C13—C14	167.4 (4)
C1—C2—C3—C4	-0.9 (7)	C12—C13—C14—O1	-53.2 (7)
C2—C3—C4—C5	2.5 (8)	C7—C8—N1—C1	-0.1 (5)
C2—C3—C4—I1	-176.4 (4)	C7—C8—N1—C9	-179.2 (5)
C3—C4—C5—C6	-1.5 (7)	C2—C1—N1—C8	-179.9 (5)
I1—C4—C5—C6	177.4 (3)	C6—C1—N1—C8	-0.5 (5)
C4—C5—C6—C1	-1.0 (6)	C2—C1—N1—C9	-0.8 (8)
C4—C5—C6—C7	-177.9 (5)	C6—C1—N1—C9	178.7 (5)
N1—C1—C6—C5	-176.9 (4)	C10—C9—N1—C8	35.3 (10)
C2—C1—C6—C5	2.6 (6)	C10—C9—N1—C1	-143.6 (7)
N1—C1—C6—C7	0.8 (5)	C16—O3—P1—O4	175.5 (4)
C2—C1—C6—C7	-179.7 (4)	C16—O3—P1—O2	51.4 (5)
C5—C6—C7—C8	176.3 (5)	C16—O3—P1—C12	-58.1 (5)
C1—C6—C7—C8	-0.8 (5)	C15—O2—P1—O4	-51.1 (5)
C5—C6—C7—C12	-3.0 (8)	C15—O2—P1—O3	69.5 (4)
C1—C6—C7—C12	179.9 (4)	C15—O2—P1—C12	-176.4 (4)
C6—C7—C8—N1	0.6 (5)	C7—C12—P1—O4	-58.6 (4)
C12—C7—C8—N1	179.9 (4)	C13—C12—P1—O4	67.1 (4)
N1—C9—C10—C11	-120.5 (9)	C7—C12—P1—O3	178.7 (3)
C8—C7—C12—C13	-47.8 (6)	C13—C12—P1—O3	-55.6 (4)

C6—C7—C12—C13	131.4 (5)	C7—C12—P1—O2	66.3 (3)
C8—C7—C12—P1	75.3 (5)	C13—C12—P1—O2	-168.0 (3)
C6—C7—C12—P1	-105.6 (4)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
C12—H12 \cdots O1	0.98	2.47	2.873 (7)	104
O1—H1 \cdots O4 ⁱ	0.82	1.92	2.733 (6)	174

Symmetry codes: (i) $-x, y-1/2, -z+3/2$.

Fig. 1

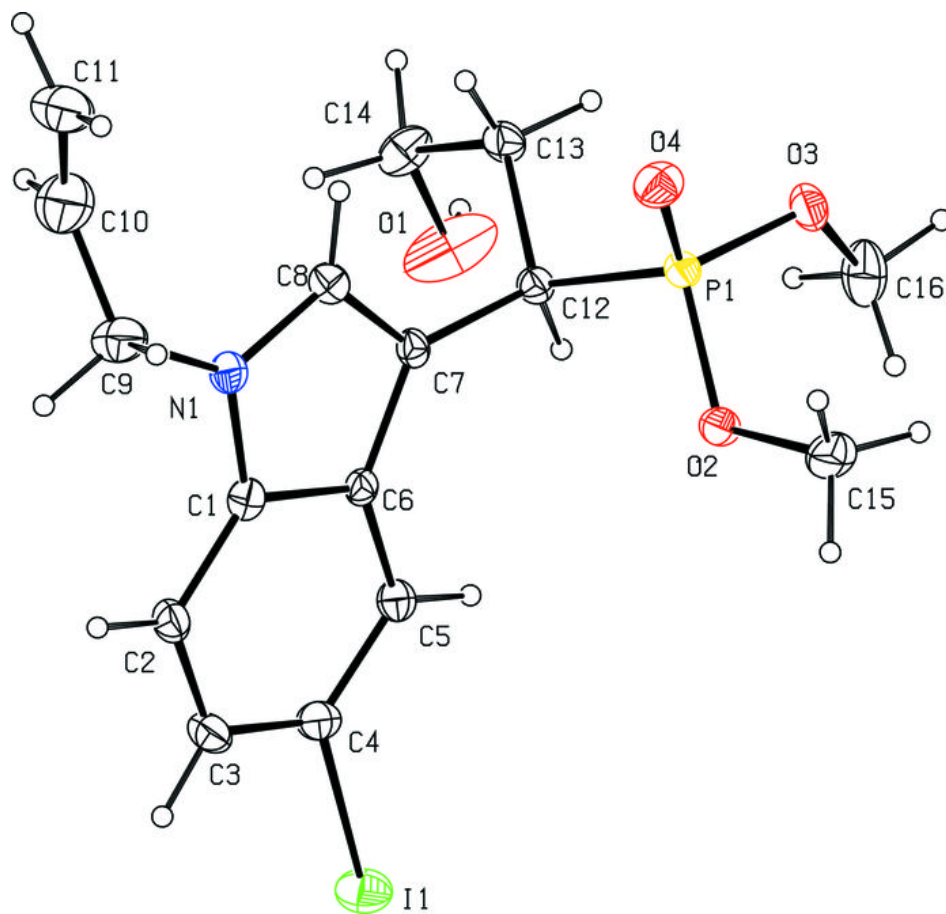


Fig. 2

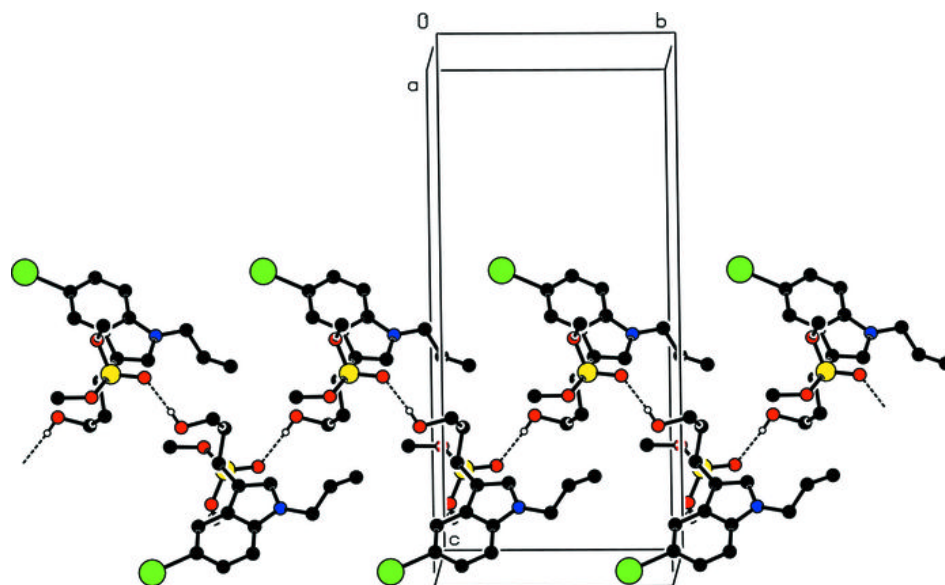


Fig. 3

