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rac-Diethyl [(1*S*,2*R*)-1-(4-bromophenyl)-6-hydroxy-3-oxo-2,3-dihydro-1*H*-benzo[*f*]chromen-2-yl]phosphonate

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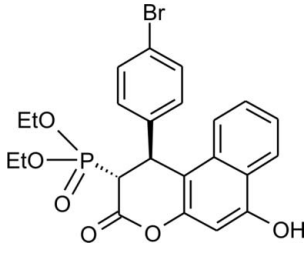
Key indicators: single-crystal X-ray study; T = 90 K; mean σ(C–C) = 0.002 Å; disorder in main residue; R factor = 0.023; wR factor = 0.064; data-to-parameter ratio = 18.3.

In the title compound, $C_{23}H_{22}BrO_6P$, the δ-valerolactone ring adopts a distorted screw-boat conformation, with the diethoxyphosphoryl substituent occupying an axial position. An unusual eclipsed conformation is found for the P—C bond. The molecules form centrosymmetric dimers connected by O—H···O hydrogen bonds. One of the methyl groups is disordered, with site occupancies of *ca* 0.8:0.2.

Related literature

For related literature, see: Bernstein *et al.* (1995); Boeyens (1978); Cremer & Pople (1975); Etter (1990); Krawczyk *et al.* (2007). For biologically active 4-aryl-3,4-dihydrocoumarins, see: Bailly *et al.* (2003; Zhang *et al.* (2006); Roelens *et al.* (2005).



Experimental

Crystal data

$C_{23}H_{22}BrO_6P$
M_r = 505.29
Monoclinic, $P2_1/n$
a = 12.8328 (5) Å
b = 11.7500 (5) Å
c = 15.8477 (7) Å
β = 93.184 (1)°
V = 2385.91 (17) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 1.83 mm^{-1}
T = 90 (2) K
0.20 × 0.20 × 0.10 mm

Data collection

Bruker SMART APEX diffractometer
Absorption correction: multi-scan (*SHELXTL*; Bruker, 2003)
T_{min} = 0.697, T_{max} = 0.839
54317 measured reflections
6239 independent reflections
5698 reflections with $I > 2\sigma(I)$
R_{int} = 0.021

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.023
$wR(F^2)$ = 0.064
S = 1.04
6239 reflections
341 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.43 e Å^{-3}
$\Delta\rho_{min}$ = −0.38 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Br—C17	1.9255 (12)	P—C2	1.8560 (11)
P—O4	1.4964 (9)	O1—C1	1.3708 (13)
P—O3	1.5762 (8)	O1—C9	1.4199 (13)
P—O5	1.5790 (9)	O2—C1	1.2085 (14)
O4—P—O3	115.66 (5)	O4—P—C2	114.94 (5)
O4—P—O5	113.99 (5)	O3—P—C2	99.90 (5)
O3—P—O5	104.07 (5)	O5—P—C2	106.75 (5)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O6—H6···O4^{i}	0.79 (2)	1.95 (2)	2.737 (1)	175 (2)

Symmetry code: (i) $-x+1, -y+1, -z+1$.

Data collection: *SMART* (Bruker, 2003); cell refinement: *SAINT-Plus* (Bruker, 2003); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXTL* (Bruker, 2003); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2512).

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supplementary materials

Acta Cryst. (2007). E**63**, o4148 [**doi:10.1107/S1600536807045576**]

rac-Diethyl [(1*S*,2*R*)-1-(4-bromophenyl)-6-hydroxy-3-oxo-2,3-dihydro-1*H*-benzo[*f*]chromen-2-yl]phosphonate

H. Krawczyk, L. Albrecht, J. Wojciechowski and W. M. Wolf

Comment

Neoflavonoids show a wide range of biological activities (Bailly *et al.*, 2003; Zhang *et al.*, 2006; Roelens *et al.*, 2005). The title compound is a key product in the novel synthesis of 4-aryl-3,4-dihydrocoumarins based on CF_3SO_3H promoted Friedel Crafts reaction of electron-rich hydroxyarenes with the acids bearing electron-withdrawing substituents on the aromatic ring (Krawczyk *et al.*, 2007). A view of (I), with atom numbering scheme is shown in Fig. 1. The δ-valerolactone and naphthalene moieties are almost coplanar with one another. The former ring adopts conformation close to a $^4S_3$ screw-boat (Boeyens, 1978), with O1, C1, C3, C4 and C9 almost coplanar (the average r.m.s. deviation from the mean plane is 0.05 Å) and C2 situated at the flap. The Cremer & Pople (1975) puckering parameters for the ring atom sequence O1/C1/C2/C3/C4/C9 are: Q = 0.48 (2) Å, θ = 116.6 (2) and φ = 315.84 (2)°. Both exocyclic substituents, namely the diethoxyphosphoryl and phenyl groups occupy axial positions in respect to the δ-valerolactone ring. The former adopts unusual eclipsed conformation when looking along the C2—P bond (Fig. 2). This particular arrangement is locked by either Coulombic attraction between phosphoryl O4 and carbonyl C1 or the C—H···π(naphthalene) interactions [H223···C5 2.89 (2) Å]. In the crystal molecules form centrosymmetric dimers connected by strong hydrogen bonds linking phosphoryl and hydroxyl groups of both monomers. In terms of Etter's graph-set terminology (Etter, 1990; Bernstein *et al.*, 1995) this system can be described as $R^2_2(20)$. The Br atom is involved in a short contact [3.183 (1) Å] with the endocyclic O1 atom of the neighbouring [0.5 − x, y − 1/2, 0.5 − z] molecule.

Experimental

The CF_3SO_3H promoted Friedel Crafts reaction of electron-rich hydroxyarenes with the acids bearing electron-withdrawing substituents on the aromatic ring. Details of the synthesis are published elsewhere (Krawczyk *et al.*, 2007). Good quality single crystals were obtained from the ethyl ether solution.

Refinement

The C23 atom of the diethoxyphosphoryl group displayed conformational disorder and was refined in a two-site disorder model. The occupancy of the major component refined to a value of 0.825 (5). H atoms were located on a difference Fourier maps calculated during the anisotropic refinement. H atoms of the diethoxyphosphoryl group were refined as riding on their parent C atoms. Positional and isotropic displacement parameters of the remaining H's were allowed to refine freely.

Figures

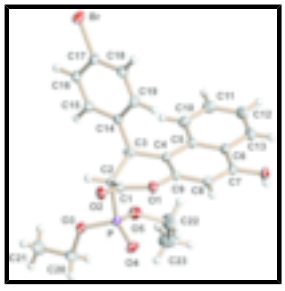

Fig. 1. View of the molecule. Displacement elipsoids are drawn at the 50% probability level. The C23 methyl group is disordered and was refined over two sites. The picture shows sites for which the occupation factor was 0.825 (5).

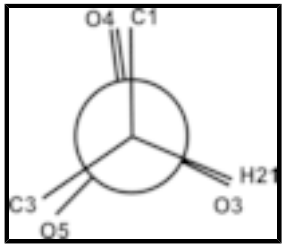

Fig. 2. Newman projection along the C2—P bond.

rac-Diethyl [(1*S*,2*R*)-1-(4-bromophenyl)-6-hydroxy-3-oxo-2,3-dihydro-1*H*- benzo[*f*]chromen-2-yl]phosphonate

Crystal data

$C_{23}H_{22}BrO_6P$	F_{000} = 1032
M_r = 505.29	D_x = 1.407 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 12.8328 (5) Å	Cell parameters from 7599 reflections
b = 11.7500 (5) Å	θ = 2.2–31.1º
c = 15.8477 (7) Å	μ = 1.83 mm^{-1}
β = 93.1840 (10)º	T = 90 (2) K
V = 2385.91 (17) Å^3	Prism, colourless
Z = 4	0.20 × 0.20 × 0.10 mm

Data collection

Bruker SMART APEX diffractometer	6239 independent reflections
Radiation source: fine-focus sealed tube	5698 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.021
T = 90(2) K	θ_{max} = 29.0º
ω scans	θ_{min} = 2.0º
Absorption correction: multi-scan (SHELXTL; Bruker, 2003)	h = −16→17
T_{min} = 0.697, T_{max} = 0.839	k = −15→15
54317 measured reflections	l = −21→20

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.023$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.064$	$w = 1/[\sigma^2(F_o^2) + (0.0365P)^2 + 0.8812P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.04$	$(\Delta/\sigma)_{max} = 0.001$
6239 reflections	$\Delta\rho_{max} = 0.43$ e Å^{-3}
341 parameters	$\Delta\rho_{min} = -0.38$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Br	0.284650 (10)	−0.133212 (11)	0.117426 (9)	0.02620 (5)	
P	0.67307 (2)	0.19775 (2)	0.525826 (18)	0.01323 (6)	
O1	0.44301 (6)	0.30270 (7)	0.45792 (5)	0.01483 (16)	
O2	0.40612 (7)	0.14418 (7)	0.52698 (6)	0.01900 (17)	
O3	0.71414 (6)	0.08202 (7)	0.56545 (5)	0.01764 (17)	
O4	0.64137 (7)	0.28582 (7)	0.58760 (6)	0.02162 (18)	
O5	0.76326 (7)	0.23729 (8)	0.46864 (6)	0.02367 (19)	
O6	0.51248 (8)	0.64805 (7)	0.31004 (6)	0.02113 (18)	
H6	0.4708 (15)	0.6687 (17)	0.3414 (13)	0.037 (5)*	
C1	0.46538 (8)	0.19325 (9)	0.48278 (7)	0.0134 (2)	
C2	0.56568 (8)	0.14374 (9)	0.45337 (7)	0.0125 (2)	
H21	0.5641 (11)	0.0631 (13)	0.4642 (9)	0.013 (3)*	
C3	0.57717 (8)	0.16968 (9)	0.35592 (7)	0.0127 (2)	
H31	0.6466 (12)	0.1471 (12)	0.3433 (9)	0.015 (3)*	
C4	0.56215 (8)	0.29631 (9)	0.34215 (7)	0.0128 (2)	
C5	0.60639 (9)	0.35682 (9)	0.27266 (7)	0.0138 (2)	
C6	0.58971 (9)	0.47646 (9)	0.26287 (7)	0.0154 (2)	

C7	0.52702 (9)	0.53432 (9)	0.32264 (7)	0.0155 (2)	
C8	0.48201 (9)	0.47477 (9)	0.38750 (7)	0.0151 (2)	
H81	0.4406 (13)	0.5087 (15)	0.4253 (11)	0.029 (4)*	
C9	0.50004 (9)	0.35658 (9)	0.39486 (7)	0.0130 (2)	
C10	0.66465 (9)	0.29916 (10)	0.21058 (8)	0.0185 (2)	
H101	0.6760 (12)	0.2190 (14)	0.2166 (10)	0.023 (4)*	
C11	0.70274 (10)	0.35731 (11)	0.14187 (8)	0.0223 (3)	
H111	0.7417 (14)	0.3187 (16)	0.1015 (11)	0.031 (4)*	
C12	0.68582 (10)	0.47584 (11)	0.13268 (8)	0.0230 (3)	
H121	0.7108 (13)	0.5141 (15)	0.0840 (11)	0.030 (4)*	
C13	0.63152 (10)	0.53456 (11)	0.19212 (8)	0.0198 (2)	
H131	0.6213 (14)	0.6112 (16)	0.1843 (11)	0.029 (4)*	
C14	0.50280 (8)	0.09639 (9)	0.29837 (7)	0.0132 (2)	
C15	0.52697 (9)	−0.01874 (10)	0.28509 (7)	0.0174 (2)	
H151	0.5900 (13)	−0.0521 (14)	0.3119 (11)	0.028 (4)*	
C16	0.46203 (10)	−0.08798 (10)	0.23236 (8)	0.0192 (2)	
H161	0.4786 (13)	−0.1668 (15)	0.2241 (11)	0.029 (4)*	
C17	0.37239 (9)	−0.04082 (10)	0.19235 (8)	0.0182 (2)	
C18	0.34500 (9)	0.07230 (10)	0.20549 (8)	0.0200 (2)	
H181	0.2817 (15)	0.1014 (16)	0.1791 (11)	0.034 (5)*	
C19	0.41108 (9)	0.14036 (10)	0.25875 (8)	0.0177 (2)	
H191	0.3946 (12)	0.2175 (14)	0.2675 (10)	0.024 (4)*	
C20	0.80761 (10)	0.07514 (11)	0.62439 (8)	0.0232 (2)	
H201	0.7904	0.0994	0.6818	0.028*	
H202	0.8638	0.1248	0.6049	0.028*	
C21	0.84199 (12)	−0.04818 (12)	0.62500 (10)	0.0307 (3)	
H211	0.7840	−0.0967	0.6406	0.046*	
H212	0.9011	−0.0582	0.6662	0.046*	
H213	0.8631	−0.0695	0.5686	0.046*	
C22	0.79348 (14)	0.35651 (15)	0.45674 (12)	0.0447 (5)	
H221	0.7280	0.3974	0.4403	0.054*	0.175 (5)
H222	0.8161	0.3849	0.5137	0.054*	0.175 (5)
H223	0.7798	0.3683	0.3966	0.054*	0.825 (5)
H224	0.7522	0.4054	0.4912	0.054*	0.825 (5)
C23A	0.8601 (7)	0.3928 (8)	0.4080 (6)	0.035 (3)	0.175 (5)
H231	0.9277	0.3568	0.4224	0.052*	0.175 (5)
H232	0.8668	0.4755	0.4142	0.052*	0.175 (5)
H233	0.8378	0.3743	0.3494	0.052*	0.175 (5)
C23B	0.91138 (16)	0.3623 (2)	0.47651 (14)	0.0459 (7)	0.825 (5)
H234	0.9462	0.3051	0.4429	0.069*	0.825 (5)
H235	0.9266	0.3473	0.5368	0.069*	0.825 (5)
H236	0.9369	0.4383	0.4625	0.069*	0.825 (5)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br	0.02545 (7)	0.01887 (7)	0.03304 (9)	−0.00433 (4)	−0.00962 (5)	−0.00515 (5)
P	0.01309 (12)	0.01055 (12)	0.01589 (14)	0.00006 (9)	−0.00051 (10)	−0.00060 (10)

O1	0.0152 (4)	0.0130 (4)	0.0168 (4)	0.0027 (3)	0.0054 (3)	0.0036 (3)
O2	0.0181 (4)	0.0180 (4)	0.0214 (4)	−0.0010 (3)	0.0058 (3)	0.0047 (3)
O3	0.0168 (4)	0.0131 (4)	0.0222 (4)	0.0020 (3)	−0.0059 (3)	−0.0002 (3)
O4	0.0215 (4)	0.0166 (4)	0.0263 (5)	0.0036 (3)	−0.0034 (3)	−0.0081 (3)
O5	0.0178 (4)	0.0275 (5)	0.0259 (5)	−0.0091 (3)	0.0025 (3)	0.0021 (4)
O6	0.0292 (5)	0.0105 (4)	0.0240 (5)	0.0015 (3)	0.0038 (4)	0.0033 (3)
C1	0.0140 (5)	0.0131 (5)	0.0131 (5)	0.0006 (4)	−0.0003 (4)	0.0002 (4)
C2	0.0125 (5)	0.0103 (5)	0.0147 (5)	0.0002 (4)	0.0001 (4)	0.0003 (4)
C3	0.0133 (5)	0.0111 (5)	0.0139 (5)	0.0009 (4)	0.0022 (4)	−0.0003 (4)
C4	0.0134 (5)	0.0109 (5)	0.0142 (5)	−0.0003 (4)	0.0011 (4)	0.0004 (4)
C5	0.0132 (5)	0.0145 (5)	0.0138 (5)	−0.0024 (4)	0.0018 (4)	−0.0003 (4)
C6	0.0165 (5)	0.0149 (5)	0.0149 (5)	−0.0033 (4)	0.0007 (4)	0.0010 (4)
C7	0.0184 (5)	0.0110 (5)	0.0168 (5)	−0.0008 (4)	−0.0014 (4)	0.0009 (4)
C8	0.0179 (5)	0.0128 (5)	0.0148 (5)	0.0027 (4)	0.0018 (4)	−0.0010 (4)
C9	0.0143 (5)	0.0125 (5)	0.0124 (5)	−0.0006 (4)	0.0014 (4)	0.0020 (4)
C10	0.0177 (5)	0.0185 (5)	0.0200 (6)	−0.0014 (4)	0.0061 (4)	−0.0021 (4)
C11	0.0206 (6)	0.0277 (6)	0.0195 (6)	−0.0056 (5)	0.0078 (5)	−0.0031 (5)
C12	0.0238 (6)	0.0273 (6)	0.0185 (6)	−0.0106 (5)	0.0053 (5)	0.0029 (5)
C13	0.0223 (6)	0.0181 (6)	0.0191 (6)	−0.0061 (4)	0.0012 (4)	0.0043 (4)
C14	0.0154 (5)	0.0119 (5)	0.0123 (5)	−0.0004 (4)	0.0014 (4)	0.0000 (4)
C15	0.0196 (5)	0.0134 (5)	0.0188 (5)	0.0030 (4)	−0.0029 (4)	−0.0007 (4)
C16	0.0240 (6)	0.0118 (5)	0.0216 (6)	0.0011 (4)	−0.0014 (5)	−0.0008 (4)
C17	0.0197 (5)	0.0152 (5)	0.0193 (6)	−0.0042 (4)	−0.0026 (4)	−0.0014 (4)
C18	0.0173 (5)	0.0180 (5)	0.0241 (6)	0.0018 (4)	−0.0044 (5)	−0.0003 (4)
C19	0.0181 (5)	0.0129 (5)	0.0218 (6)	0.0025 (4)	−0.0014 (4)	−0.0012 (4)
C20	0.0201 (5)	0.0240 (6)	0.0243 (6)	0.0013 (5)	−0.0098 (5)	−0.0001 (5)
C21	0.0297 (7)	0.0297 (7)	0.0316 (7)	0.0116 (6)	−0.0093 (6)	0.0027 (6)
C22	0.0430 (9)	0.0416 (9)	0.0475 (10)	−0.0302 (7)	−0.0147 (8)	0.0203 (7)
C23A	0.028 (4)	0.042 (5)	0.035 (5)	−0.020 (4)	0.011 (3)	0.001 (4)
C23B	0.0330 (10)	0.0670 (16)	0.0380 (12)	−0.0320 (10)	0.0053 (8)	−0.0058 (10)

Geometric parameters (Å, °)

Br—C17	1.9255 (12)	C12—H121	0.963 (17)
P—O4	1.4964 (9)	C13—H131	0.918 (18)
P—O3	1.5762 (8)	C14—C19	1.4015 (16)
P—O5	1.5790 (9)	C14—C15	1.4064 (15)
P—C2	1.8560 (11)	C15—C16	1.4062 (17)
O1—C1	1.3708 (13)	C15—H151	0.976 (17)
O1—C9	1.4199 (13)	C16—C17	1.3969 (17)
O2—C1	1.2085 (14)	C16—H161	0.961 (18)
O3—C20	1.4810 (14)	C17—C18	1.3935 (16)
O5—C22	1.4684 (17)	C18—C19	1.4112 (17)
O6—C7	1.3625 (13)	C18—H181	0.956 (19)
O6—H6	0.79 (2)	C19—H191	0.943 (16)
C1—C2	1.5096 (15)	C20—C21	1.5146 (19)
C2—C3	1.5889 (15)	C20—H201	0.9900
C2—H21	0.963 (15)	C20—H202	0.9900
C3—C4	1.5146 (14)	C21—H211	0.9800

C3—C14	1.5452 (15)	C21—H212	0.9800
C3—H31	0.961 (15)	C21—H213	0.9800
C4—C9	1.3815 (15)	C22—C23A	1.259 (8)
C4—C5	1.4526 (15)	C22—C23B	1.530 (3)
C5—C6	1.4290 (15)	C22—H221	0.9900
C5—C10	1.4379 (15)	C22—H222	0.9900
C6—C13	1.4414 (15)	C22—H223	0.9700
C6—C7	1.4462 (15)	C22—H224	0.9699
C7—C8	1.3948 (15)	C23A—H223	1.0758
C8—C9	1.4117 (15)	C23A—H231	0.9800
C8—H81	0.914 (17)	C23A—H232	0.9800
C10—C11	1.3967 (17)	C23A—H233	0.9800
C10—H101	0.957 (16)	C23B—H234	0.9800
C11—C12	1.4158 (19)	C23B—H235	0.9800
C11—H111	0.949 (18)	C23B—H236	0.9800
C12—C13	1.3863 (18)		
O4—P—O3	115.66 (5)	C14—C15—H151	120.4 (10)
O4—P—O5	113.99 (5)	C17—C16—C15	119.09 (10)
O3—P—O5	104.07 (5)	C17—C16—H161	120.3 (10)
O4—P—C2	114.94 (5)	C15—C16—H161	120.7 (10)
O3—P—C2	99.90 (5)	C18—C17—C16	121.24 (11)
O5—P—C2	106.75 (5)	C18—C17—Br	119.10 (9)
C1—O1—C9	120.84 (8)	C16—C17—Br	119.66 (9)
C20—O3—P	122.65 (8)	C17—C18—C19	118.80 (11)
C22—O5—P	124.17 (11)	C17—C18—H181	119.4 (11)
C7—O6—H6	107.5 (15)	C19—C18—H181	121.8 (11)
O2—C1—O1	119.06 (10)	C14—C19—C18	121.39 (10)
O2—C1—C2	124.74 (10)	C14—C19—H191	118.5 (10)
O1—C1—C2	116.19 (9)	C18—C19—H191	120.1 (10)
C1—C2—C3	110.67 (9)	O3—C20—C21	106.36 (10)
C1—C2—P	107.08 (7)	O3—C20—H201	110.5
C3—C2—P	115.46 (7)	C21—C20—H201	110.5
C1—C2—H21	107.3 (8)	O3—C20—H202	110.5
C3—C2—H21	111.4 (8)	C21—C20—H202	110.5
P—C2—H21	104.4 (9)	H201—C20—H202	108.6
C4—C3—C14	113.13 (9)	C20—C21—H211	109.5
C4—C3—C2	108.09 (9)	C20—C21—H212	109.5
C14—C3—C2	112.29 (9)	H211—C21—H212	109.5
C4—C3—H31	110.7 (8)	C20—C21—H213	109.5
C14—C3—H31	105.8 (9)	H211—C21—H213	109.5
C2—C3—H31	106.7 (9)	H212—C21—H213	109.5
C9—C4—C5	118.06 (10)	C23A—C22—O5	126.5 (5)
C9—C4—C3	119.30 (9)	C23A—C22—C23B	54.0 (5)
C5—C4—C3	122.56 (9)	O5—C22—C23B	106.36 (16)
C6—C5—C10	118.01 (10)	C23A—C22—H221	105.7
C6—C5—C4	120.09 (10)	O5—C22—H221	105.7
C10—C5—C4	121.87 (10)	C23B—C22—H221	147.9
C5—C6—C13	119.37 (10)	C23A—C22—H222	105.7
C5—C6—C7	118.47 (10)	O5—C22—H222	105.7

C13—C6—C7	122.10 (10)	C23B—C22—H222	64.0
O6—C7—C8	122.75 (10)	H221—C22—H222	106.1
O6—C7—C6	116.21 (10)	C23A—C22—H223	55.9
C8—C7—C6	121.00 (10)	O5—C22—H223	103.2
C7—C8—C9	118.79 (10)	C23B—C22—H223	108.5
C7—C8—H81	122.9 (11)	H221—C22—H223	64.4
C9—C8—H81	118.3 (11)	H222—C22—H223	151.1
C4—C9—C8	123.49 (10)	C23A—C22—H224	123.8
C4—C9—O1	122.29 (9)	O5—C22—H224	109.6
C8—C9—O1	114.09 (9)	C23B—C22—H224	115.1
C11—C10—C5	121.38 (11)	H221—C22—H224	51.6
C11—C10—H101	120.1 (9)	H222—C22—H224	55.2
C5—C10—H101	118.5 (9)	H223—C22—H224	113.2
C10—C11—C12	120.21 (11)	C22—C23A—H223	48.3
C10—C11—H111	120.8 (11)	C22—C23A—H231	109.5
C12—C11—H111	118.9 (11)	H223—C23A—H231	138.7
C13—C12—C11	119.98 (11)	C22—C23A—H232	109.5
C13—C12—H121	120.9 (10)	H223—C23A—H232	111.1
C11—C12—H121	119.1 (10)	C22—C23A—H233	109.5
C12—C13—C6	121.04 (11)	H223—C23A—H233	63.6
C12—C13—H131	118.1 (11)	C22—C23B—H234	109.5
C6—C13—H131	120.9 (11)	C22—C23B—H235	109.5
C19—C14—C15	118.31 (10)	H234—C23B—H235	109.5
C19—C14—C3	122.33 (10)	C22—C23B—H236	109.5
C15—C14—C3	119.36 (10)	H234—C23B—H236	109.5
C16—C15—C14	121.13 (11)	H235—C23B—H236	109.5
C16—C15—H151	118.5 (10)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O6—H6···O4^{i}	0.79 (2)	1.95 (2)	2.737 (1)	175 (2)

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*+1.

Fig. 1

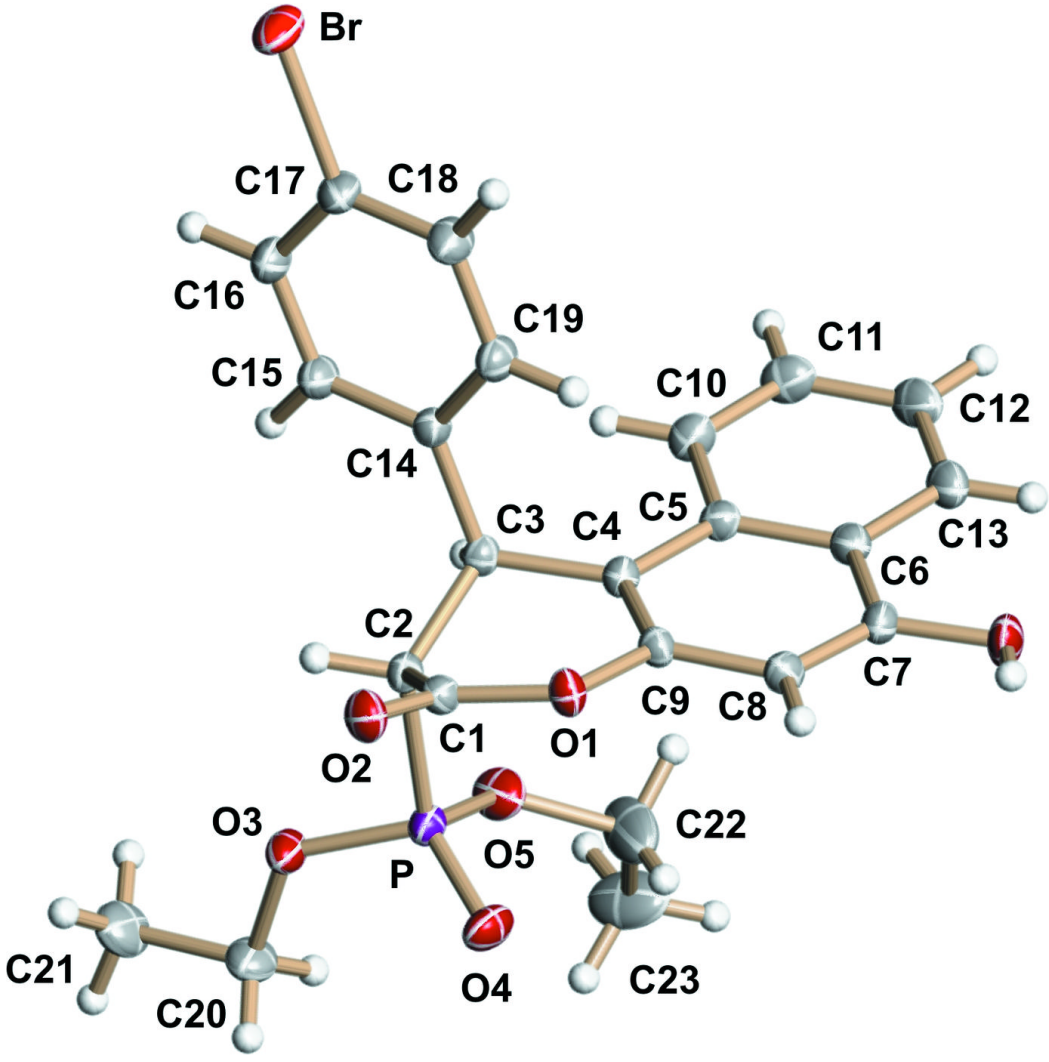
Br
C17
C18
C16
C19
C15
C11
C10
C12
C14
C3
C4
C5
C13
C6
C2
C7
C9
C8
C1
O1
O2
O3
O5
C22
P
O4
C21
C20
C23

Fig. 2