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2-(4-Dimethylamino-2-hydroxybenzoyl)-benzoic acid methanol solvate

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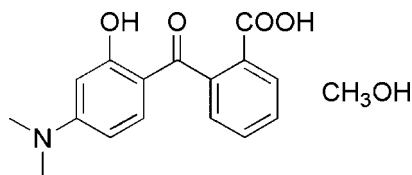
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Key indicators: single-crystal X-ray study; $T = 113$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.036; wR factor = 0.109; data-to-parameter ratio = 16.0.

In the title compound, $\text{C}_{16}\text{H}_{15}\text{NO}_4 \cdot \text{CH}_4\text{O}$, the dihedral angle between the benzene rings is $75.21(5)^\circ$. The structure is stabilized by an intramolecular $\text{O}-\text{H} \cdots \text{O}$ interaction [$\text{O} \cdots \text{O} = 2.589(2)$ Å]. The solvent molecule links symmetry-related molecules of the complex via hydrogen bonds with $\text{O} \cdots \text{O}$ separations of $2.631(2)$ and $2.815(2)$ Å. $\text{C}-\text{H} \cdots \text{O}$ hydrogen bonds are also present.

Related literature

For a related structure, see: Yan *et al.* (2006). For synthetic applications, see: Hellmut & Lamm (1977); Minru *et al.* (1977); Yojiro *et al.* (1992); Lee *et al.* (1998); Luo *et al.* (1994).



Experimental

Crystal data

$\text{C}_{16}\text{H}_{15}\text{NO}_4 \cdot \text{CH}_4\text{O}$
 $M_r = 317.33$
 Triclinic, $P\bar{1}$

$a = 7.1438(14)$ Å
 $b = 7.3021(15)$ Å
 $c = 16.613(3)$ Å

$\alpha = 83.92(3)^\circ$
 $\beta = 80.21(3)^\circ$
 $\gamma = 64.94(3)^\circ$
 $V = 773.0(3)$ Å³
 $Z = 2$

Mo $K\alpha$ radiation
 $\mu = 0.10$ mm⁻¹
 $T = 113(2)$ K
 $0.20 \times 0.18 \times 0.12$ mm

Data collection

Rigaku Saturn CCD area-detector diffractometer
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.980$, $T_{\max} = 0.988$

12548 measured reflections
 3528 independent reflections
 2679 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.034$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.036$
 $wR(F^2) = 0.109$
 $S = 1.06$
 3528 reflections
 220 parameters

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\max} = 0.31$ e Å⁻³
 $\Delta\rho_{\min} = -0.25$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H} \cdots A$	$D-\text{H}$	$\text{H} \cdots A$	$D \cdots A$	$D-\text{H} \cdots A$
$\text{O1}-\text{H1} \cdots \text{O2}$	0.92 (2)	1.75 (2)	2.589 (2)	151 (1)
$\text{O4}-\text{H4} \cdots \text{O5}^{\text{i}}$	0.94 (2)	1.70 (2)	2.631 (2)	168 (2)
$\text{O5}-\text{H5} \cdots \text{O2}$	0.87 (2)	1.95 (2)	2.815 (2)	178 (2)
$\text{C7}-\text{H7B} \cdots \text{O3}^{\text{ii}}$	0.98	2.54	3.463 (2)	156
$\text{C13}-\text{H13} \cdots \text{O5}^{\text{iii}}$	0.95	2.53	3.331 (2)	142

Symmetry codes: (i) $x + 1, y, z$; (ii) $-x + 2, -y, -z$; (iii) $x, y + 1, z$.

Data collection: *CrystalClear* (Rigaku/MSK, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalStructure* (Rigaku/MSK, 2005); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PV2121).

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supplementary materials

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2-(4-Dimethylamino-2-hydroxybenzoyl)benzoic acid methanol solvate

J.-C. Guo, Q.-H. Liu, J.-Y. Guo, M.-Z. Chen and R.-W. Zhang

Comment

The title compound, (I), is an intermediate in the synthesis of rhodamine derivatives (Hellmut & Lamm, 1977; Minru *et al.*, 1977; Yojiro *et al.*, 1992; Lee *et al.*, 1998). 2-Carboxyl-4'-diethylamino-2'-hydroxybenzophenone was synthesized (Luo *et al.*, 1994) from 3-diethylaminophenol and phthalic anhydride in toluene with the same reaction mechanism as the title compound. In the present paper, the title compound has been synthesized and the crystal structure of (I) is reported.

The bond distances and bond angles in (I) (Fig. 1) are similar to the corresponding dimensions reported for a closely related structure, 2-[4-(diethylamino)-2-hydroxybenzoyl]-3,4,5,6-tetrafluorobenzoic acid (Yan *et al.*, 2006). The 2-hydroxy-4-dimethylaminobenzoyl and *o*-benzoic acid moieties in (I) are each essentially planar and the angle between two planes is 75.21 (5)°. There are intermolecular O—H···O hydrogen bonds involving the solvent and the complex molecules stabilizing the structure (Table 1 and Fig. 2). The structure also contains an intramolecular interaction of the type O—H···O (O···O = 2.589 (2) Å) and non-classical hydrogen bonds of the type C—H···O.

Experimental

A solution of 3-dimethylamino phenol (4.11 g, 30.0 mmol) and phthalic anhydride (4.66 g, 31.5 mmol) in toluene (30 ml) was refluxed for 3 h. The solution was cooled to room temperature and the precipitate was collected to afford the title compound (yield = 75.6%). The crude product was purified by silica-gel chromatography (methanol–dichloromethane, 1:50). Crystals suitable for X-ray diffraction were obtained by slow evaporation of a solution in methanol and dichloromethane (5:1).

Refinement

The O-bound H atoms were located in a difference map and their coordinates were refined with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{O})$. The methyl and aryl H atoms were constrained to ideal geometry with C—H distances of 0.95 and 0.98 Å, and $U_{\text{iso}}(\text{H}) = 1.5$ and $1.2U_{\text{eq}}(\text{C})$, respectively, and each methyl group was allowed to rotate freely about its C—C bond.

Figures

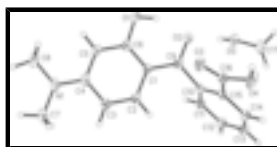


Fig. 1. A view of the title compound showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level (arbitrary spheres for the H atoms).

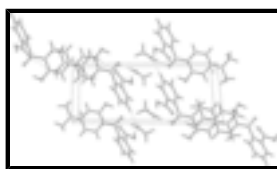


Fig. 2. The unit cell packing of (I); dashed lines indicate hydrogen-bond interactions.

2-(4-Dimethylamino-2-hydroxybenzoyl)benzoic acid methanol solvate

Crystal data

$C_{16}H_{14}NO_4 \cdot C_1H_4O_1$	$Z = 2$
$M_r = 317.33$	$F_{000} = 336$
Triclinic, $P\bar{1}$	$D_x = 1.363 \text{ Mg m}^{-3}$
Hall symbol: -P 1	Mo $K\alpha$ radiation
$a = 7.1438 (14) \text{ \AA}$	$\lambda = 0.71073 \text{ \AA}$
$b = 7.3021 (15) \text{ \AA}$	Cell parameters from 2451 reflections
$c = 16.613 (3) \text{ \AA}$	$\theta = 2.5\text{--}27.5^\circ$
$\alpha = 83.92 (3)^\circ$	$\mu = 0.10 \text{ mm}^{-1}$
$\beta = 80.21 (3)^\circ$	$T = 113 (2) \text{ K}$
$\gamma = 64.94 (3)^\circ$	Block, yellow
$V = 773.0 (3) \text{ \AA}^3$	$0.20 \times 0.18 \times 0.12 \text{ mm}$

Data collection

Rigaku Saturn CCD area-detector diffractometer	3528 independent reflections
Radiation source: rotating anode	2679 reflections with $I > 2\sigma(I)$
Monochromator: confocal	$R_{\text{int}} = 0.034$
Detector resolution: $7.31 \text{ pixels mm}^{-1}$	$\theta_{\text{max}} = 27.5^\circ$
$T = 113(2) \text{ K}$	$\theta_{\text{min}} = 2.5^\circ$
ω and φ scans	$h = -9 \rightarrow 9$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$k = -9 \rightarrow 9$
$T_{\text{min}} = 0.980$, $T_{\text{max}} = 0.988$	$l = -21 \rightarrow 21$
12548 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.036$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.109$	$w = 1/[\sigma^2(F_o^2) + (0.0647P)^2 + 0.0542P]$
$S = 1.06$	where $P = (F_o^2 + 2F_c^2)/3$
3528 reflections	$(\Delta/\sigma)_{\text{max}} = 0.002$
220 parameters	$\Delta\rho_{\text{max}} = 0.31 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\text{min}} = -0.25 \text{ e \AA}^{-3}$
	Extinction correction: none

Special details

Experimental. ^1H NMR ($\text{CD}_3\text{OD}:\text{CDCl}_3$, 5:1, δ , p.p.m.): 8.07 (d, 1H), 7.68 (t, 1H), 7.59 (t, 1H), 7.36 (d, 1H), 6.88 (d, 1H), 6.18 (d, 1H), 6.13 (s, 1H), 3.04 (s, 6H).

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 $F^2 > \sigma(F^2)$ 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)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.70462 (14)	-0.28138 (12)	0.19780 (5)	0.0243 (2)
H1	0.651 (2)	-0.228 (2)	0.2486 (10)	0.036*
O2	0.54875 (13)	-0.02370 (12)	0.31294 (5)	0.0228 (2)
O3	0.88960 (13)	0.13445 (13)	0.30456 (5)	0.0267 (2)
O4	0.83287 (14)	0.32743 (14)	0.40966 (6)	0.0345 (2)
H4	0.973 (3)	0.242 (3)	0.4146 (10)	0.052*
O5	0.21609 (13)	0.11128 (13)	0.44162 (5)	0.0245 (2)
H5	0.322 (3)	0.069 (2)	0.4029 (10)	0.037*
N1	0.81331 (15)	-0.03715 (16)	-0.07467 (6)	0.0245 (2)
C1	0.61529 (16)	0.07774 (16)	0.17518 (7)	0.0172 (2)
C2	0.60924 (17)	0.23363 (17)	0.11636 (7)	0.0192 (2)
H2	0.5611	0.3674	0.1342	0.023*
C3	0.66995 (18)	0.19999 (18)	0.03453 (7)	0.0211 (2)
H3	0.6602	0.3099	-0.0032	0.025*
C4	0.74787 (17)	-0.00015 (18)	0.00592 (7)	0.0203 (2)
C5	0.75624 (17)	-0.15813 (17)	0.06392 (7)	0.0206 (2)
H5A	0.8070	-0.2923	0.0461	0.025*
C6	0.69205 (17)	-0.12142 (16)	0.14605 (7)	0.0184 (2)
C7	0.7924 (2)	0.1273 (2)	-0.13552 (7)	0.0286 (3)
H7A	0.6454	0.2228	-0.1325	0.043*
H7B	0.8422	0.0720	-0.1902	0.043*
H7C	0.8754	0.1975	-0.1247	0.043*
C8	0.9050 (2)	-0.2431 (2)	-0.10245 (8)	0.0283 (3)
H8A	1.0167	-0.3284	-0.0704	0.042*
H8B	0.9627	-0.2441	-0.1604	0.042*
H8C	0.7975	-0.2956	-0.0953	0.042*
C9	0.54677 (17)	0.11448 (17)	0.26072 (7)	0.0177 (2)
C10	0.45044 (17)	0.32664 (16)	0.29110 (6)	0.0165 (2)
C11	0.24430 (18)	0.44749 (17)	0.28008 (7)	0.0216 (2)
H11	0.1761	0.4021	0.2476	0.026*

supplementary materials

C12	0.13725 (18)	0.63378 (17)	0.31616 (7)	0.0228 (3)
H12	-0.0030	0.7156	0.3079	0.027*
C13	0.23498 (18)	0.70024 (17)	0.36419 (7)	0.0226 (3)
H13	0.1615	0.8270	0.3893	0.027*
C14	0.44038 (18)	0.58132 (17)	0.37548 (7)	0.0204 (2)
H14	0.5068	0.6272	0.4086	0.024*
C15	0.55075 (17)	0.39511 (16)	0.33878 (6)	0.0168 (2)
C16	0.77429 (17)	0.27111 (16)	0.34857 (7)	0.0185 (2)
C17	0.2575 (2)	0.2154 (2)	0.49935 (7)	0.0287 (3)
H17A	0.1589	0.3581	0.4986	0.043*
H17B	0.4004	0.2052	0.4851	0.043*
H17C	0.2418	0.1545	0.5541	0.043*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0321 (5)	0.0178 (4)	0.0208 (4)	-0.0080 (4)	-0.0025 (4)	-0.0044 (3)
O2	0.0310 (5)	0.0189 (4)	0.0175 (4)	-0.0093 (4)	-0.0025 (3)	-0.0021 (3)
O3	0.0226 (4)	0.0258 (4)	0.0254 (4)	-0.0014 (4)	-0.0052 (3)	-0.0090 (4)
O4	0.0214 (5)	0.0383 (5)	0.0411 (6)	-0.0025 (4)	-0.0116 (4)	-0.0211 (4)
O5	0.0207 (4)	0.0280 (5)	0.0251 (4)	-0.0089 (4)	-0.0022 (3)	-0.0083 (4)
N1	0.0215 (5)	0.0316 (6)	0.0169 (5)	-0.0071 (4)	-0.0011 (4)	-0.0064 (4)
C1	0.0155 (5)	0.0189 (6)	0.0173 (5)	-0.0062 (4)	-0.0030 (4)	-0.0042 (4)
C2	0.0170 (5)	0.0191 (5)	0.0203 (5)	-0.0059 (4)	-0.0017 (4)	-0.0045 (4)
C3	0.0186 (6)	0.0234 (6)	0.0191 (5)	-0.0068 (5)	-0.0020 (4)	-0.0008 (4)
C4	0.0127 (5)	0.0288 (6)	0.0175 (5)	-0.0054 (5)	-0.0023 (4)	-0.0063 (5)
C5	0.0179 (5)	0.0211 (6)	0.0220 (6)	-0.0054 (5)	-0.0028 (4)	-0.0083 (4)
C6	0.0161 (5)	0.0191 (6)	0.0205 (5)	-0.0063 (4)	-0.0047 (4)	-0.0032 (4)
C7	0.0255 (6)	0.0406 (8)	0.0170 (5)	-0.0111 (6)	-0.0017 (5)	-0.0027 (5)
C8	0.0265 (6)	0.0397 (7)	0.0223 (6)	-0.0161 (6)	0.0031 (5)	-0.0158 (5)
C9	0.0157 (5)	0.0188 (5)	0.0190 (5)	-0.0061 (4)	-0.0045 (4)	-0.0032 (4)
C10	0.0193 (5)	0.0161 (5)	0.0132 (5)	-0.0064 (4)	-0.0011 (4)	-0.0021 (4)
C11	0.0219 (6)	0.0232 (6)	0.0192 (5)	-0.0071 (5)	-0.0057 (4)	-0.0044 (4)
C12	0.0190 (6)	0.0215 (6)	0.0228 (6)	-0.0026 (5)	-0.0045 (4)	-0.0023 (5)
C13	0.0238 (6)	0.0170 (5)	0.0234 (6)	-0.0052 (5)	-0.0007 (5)	-0.0041 (4)
C14	0.0224 (6)	0.0184 (6)	0.0220 (5)	-0.0095 (5)	-0.0029 (4)	-0.0038 (4)
C15	0.0182 (5)	0.0169 (5)	0.0154 (5)	-0.0077 (4)	-0.0012 (4)	-0.0005 (4)
C16	0.0201 (5)	0.0181 (5)	0.0186 (5)	-0.0088 (5)	-0.0025 (4)	-0.0022 (4)
C17	0.0283 (6)	0.0366 (7)	0.0220 (6)	-0.0129 (6)	-0.0028 (5)	-0.0079 (5)

Geometric parameters (\AA , $^\circ$)

O1—C6	1.3550 (14)	C7—H7A	0.9800
O1—H1	0.92 (2)	C7—H7B	0.9800
O2—C9	1.2561 (14)	C7—H7C	0.9800
O3—C16	1.2092 (14)	C8—H8A	0.9800
O4—C16	1.3240 (14)	C8—H8B	0.9800
O4—H4	0.94 (2)	C8—H8C	0.9800
O5—C17	1.4237 (15)	C9—C10	1.5075 (16)

O5—H5	0.87 (2)	C10—C11	1.3919 (16)
N1—C4	1.3569 (15)	C10—C15	1.4041 (15)
N1—C8	1.4537 (17)	C11—C12	1.3892 (17)
N1—C7	1.4587 (17)	C11—H11	0.9500
C1—C2	1.4105 (16)	C12—C13	1.3850 (17)
C1—C6	1.4258 (16)	C12—H12	0.9500
C1—C9	1.4367 (15)	C13—C14	1.3862 (17)
C2—C3	1.3699 (16)	C13—H13	0.9500
C2—H2	0.9500	C14—C15	1.3950 (16)
C3—C4	1.4281 (17)	C14—H14	0.9500
C3—H3	0.9500	C15—C16	1.4928 (16)
C4—C5	1.4101 (17)	C17—H17A	0.9800
C5—C6	1.3800 (16)	C17—H17B	0.9800
C5—H5A	0.9500	C17—H17C	0.9800
C6—O1—H1	105.2 (9)	N1—C8—H8C	109.5
C16—O4—H4	109.9 (10)	H8A—C8—H8C	109.5
C17—O5—H5	109.8 (10)	H8B—C8—H8C	109.5
C4—N1—C8	120.41 (11)	O2—C9—C1	122.52 (10)
C4—N1—C7	121.24 (10)	O2—C9—C10	116.45 (9)
C8—N1—C7	118.35 (10)	C1—C9—C10	120.82 (10)
C2—C1—C6	116.93 (10)	C11—C10—C15	119.40 (10)
C2—C1—C9	122.49 (10)	C11—C10—C9	117.74 (10)
C6—C1—C9	120.58 (10)	C15—C10—C9	122.31 (10)
C3—C2—C1	122.67 (10)	C12—C11—C10	120.65 (11)
C3—C2—H2	118.7	C12—C11—H11	119.7
C1—C2—H2	118.7	C10—C11—H11	119.7
C2—C3—C4	120.04 (11)	C13—C12—C11	120.03 (11)
C2—C3—H3	120.0	C13—C12—H12	120.0
C4—C3—H3	120.0	C11—C12—H12	120.0
N1—C4—C5	121.05 (11)	C12—C13—C14	119.83 (11)
N1—C4—C3	120.88 (11)	C12—C13—H13	120.1
C5—C4—C3	118.07 (10)	C14—C13—H13	120.1
C6—C5—C4	121.21 (11)	C13—C14—C15	120.80 (11)
C6—C5—H5A	119.4	C13—C14—H14	119.6
C4—C5—H5A	119.4	C15—C14—H14	119.6
O1—C6—C5	117.56 (10)	C14—C15—C10	119.28 (10)
O1—C6—C1	121.36 (10)	C14—C15—C16	120.61 (10)
C5—C6—C1	121.08 (11)	C10—C15—C16	120.10 (10)
N1—C7—H7A	109.5	O3—C16—O4	123.78 (11)
N1—C7—H7B	109.5	O3—C16—C15	123.12 (10)
H7A—C7—H7B	109.5	O4—C16—C15	113.10 (10)
N1—C7—H7C	109.5	O5—C17—H17A	109.5
H7A—C7—H7C	109.5	O5—C17—H17B	109.5
H7B—C7—H7C	109.5	H17A—C17—H17B	109.5
N1—C8—H8A	109.5	O5—C17—H17C	109.5
N1—C8—H8B	109.5	H17A—C17—H17C	109.5
H8A—C8—H8B	109.5	H17B—C17—H17C	109.5
C6—C1—C2—C3	1.11 (16)	C6—C1—C9—C10	-175.35 (9)

supplementary materials

C9—C1—C2—C3	-178.47 (10)	O2—C9—C10—C11	-97.87 (12)
C1—C2—C3—C4	-1.53 (17)	C1—C9—C10—C11	76.97 (14)
C8—N1—C4—C5	-3.56 (16)	O2—C9—C10—C15	73.53 (14)
C7—N1—C4—C5	175.95 (10)	C1—C9—C10—C15	-111.62 (12)
C8—N1—C4—C3	175.95 (10)	C15—C10—C11—C12	-0.42 (16)
C7—N1—C4—C3	-4.54 (16)	C9—C10—C11—C12	171.25 (10)
C2—C3—C4—N1	-178.45 (10)	C10—C11—C12—C13	-0.52 (17)
C2—C3—C4—C5	1.08 (16)	C11—C12—C13—C14	0.63 (18)
N1—C4—C5—C6	179.25 (10)	C12—C13—C14—C15	0.20 (17)
C3—C4—C5—C6	-0.27 (16)	C13—C14—C15—C10	-1.13 (16)
C4—C5—C6—O1	179.85 (10)	C13—C14—C15—C16	177.71 (10)
C4—C5—C6—C1	-0.12 (17)	C11—C10—C15—C14	1.23 (16)
C2—C1—C6—O1	179.77 (9)	C9—C10—C15—C14	-170.04 (10)
C9—C1—C6—O1	-0.65 (16)	C11—C10—C15—C16	-177.62 (10)
C2—C1—C6—C5	-0.27 (16)	C9—C10—C15—C16	11.12 (15)
C9—C1—C6—C5	179.32 (10)	C14—C15—C16—O3	-162.76 (11)
C2—C1—C9—O2	178.74 (10)	C10—C15—C16—O3	16.06 (16)
C6—C1—C9—O2	-0.82 (17)	C14—C15—C16—O4	16.69 (15)
C2—C1—C9—C10	4.21 (16)	C10—C15—C16—O4	-164.48 (10)

Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
O1—H1...O2	0.92 (2)	1.75 (2)	2.589 (2)	151 (1)
O4—H4...O5 ⁱ	0.94 (2)	1.70 (2)	2.631 (2)	168 (2)
O5—H5...O2	0.87 (2)	1.95 (2)	2.815 (2)	178 (2)
C7—H7B...O3 ⁱⁱ	0.98	2.54	3.463 (2)	156
C13—H13...O5 ⁱⁱⁱ	0.95	2.53	3.331 (2)	142

Symmetry codes: (i) $x+1, y, z$; (ii) $-x+2, -y, -z$; (iii) $x, y+1, z$.

Fig. 1

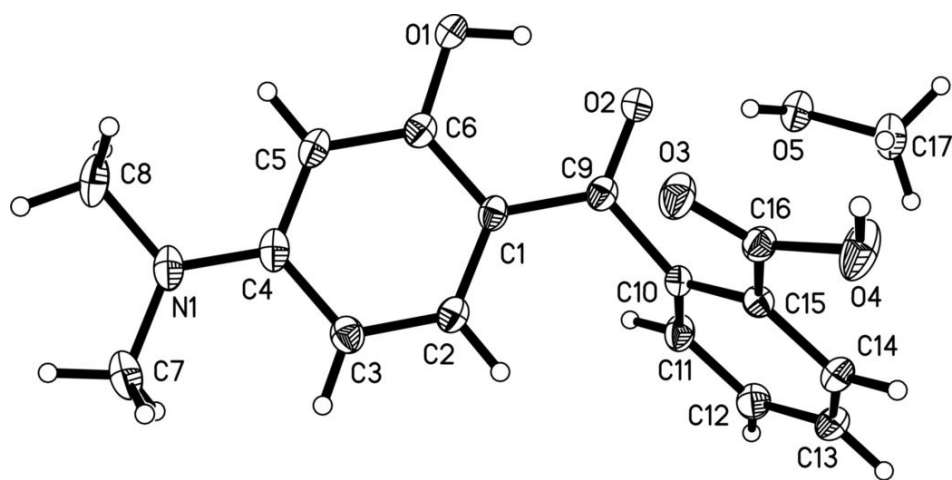


Fig. 2

