Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

4-(5-Bromo-1,3-dioxoisoindolin-2-yl)-benzoic acid *N*,*N*-dimethylformamide solvate

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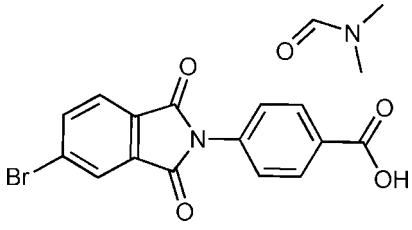
Received 19 May 2007; accepted 28 May 2007

Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.005 Å; R factor = 0.040; wR factor = 0.118; data-to-parameter ratio = 14.9.

In the crystal structure of the title compound, $C_{15}H_8BrNO_4\cdot C_3H_7NO$, the constituent species interact by C—H···O and O—H···O hydrogen bonds. The dihedral angle between the phthalimide group and the benzene ring is 51.4 (2)°.

Related literature

For a related structure, see: Liang *et al.* (2006).



Experimental

Crystal data

$C_{15}H_8BrNO_4\cdot C_3H_7NO$
M_r = 419.23
Triclinic, $P\overline{1}$
a = 5.9686 (9) Å
b = 8.3214 (12) Å
c = 19.058 (3) Å
α = 101.231 (2)°
β = 93.417 (3)°
γ = 107.273 (2)°
V = 879.6 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 2.37 mm^{-1}
T = 294 (2) K
0.26 × 0.22 × 0.12 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
T_{min} = 0.578, T_{max} = 0.764
5114 measured reflections
3561 independent reflections
2643 reflections with $I > 2\sigma(I)$
R_{int} = 0.022

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.118
S = 1.13
3561 reflections
239 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.40 e Å^{-3}
$\Delta\rho_{min}$ = −0.42 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3···O5^{i}	0.82	1.77	2.583 (3)	170
C5—H5···O1^{ii}	0.93	2.54	3.343 (4)	144
C14—H14···O1^{iii}	0.93	2.49	3.413 (4)	170
C16—H16···O4^{ii}	0.93	2.54	3.352 (4)	146
C18—H18*A*···O5^{iii}	0.96	2.53	3.262 (5)	133

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

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

This work was supported by the Doctoral Foundation of Weifang University.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2427).

References

Bruker (1997). *SADABS* (Version 2.01), *SMART* (Version 5.044), *SAINT* (Version 5.01) and *SHELXTL* (Version 5.10). Bruker AXS Inc., Madison, Wisconsin, USA.
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supplementary materials

Acta Cryst. (2007). E**63**, o3065 [**doi:10.1107/S1600536807025913**]

4-(5-Bromo-1,3-dioxoisoindolin-2-yl)benzoic acid *N*,*N*-dimethylformamide solvate

Z.-P. Liang, J. Li, Y. Hua and H.-L. Wang

Comment

Phthalimides and N-substituted phthalimides are an important class of compounds because of their interesting biological activities. Phthalimides have also served as starting materials and intermediates for the syntheses of alkaloids and pharmacophores. The asymmetric unit of (I) contains one 4-(5-bromo-1,3-dioxoisoindolin-2-yl)benzoic acid (BDA) molecule and one dimethylformamide (DMF) solvent molecule (Fig. 1).

The bond lengths and angles in (I) agree with those in the similar compound 4-phthalimidobenzoic acid *N*,*N*-dimethylformamide solvate (Liang *et al.*, 2006). The phthalimide group in (I) is essentially planar, with a mean deviation of 0.033 (3) Å. The dihedral angle between the phthalimide group and the C9—C14 benzene ring is 51.4 (2) °. The crystal structure of (I) is stabilized by an O—H···O hydrogen bond which connects the BDA and DMF molecules and by a C—H···O hydrogen bond which links the BDA molecules together (Fig. 2 and Table 1).

Experimental

A mixture of 5-bromoisobenzofuran-1,3-dione (0.01 mol) and 4-aminobenzoic acid (0.01 mol) in acetic acid (20 ml) was refluxed for 4 h. After cooling, filtration and drying, the compound 4-(5-bromo-1,3-dioxoisoindolin-2-yl)benzoic acid was obtained. 10 mg of this compound was dissolved in DMF (7 ml), and the solution was then allowed to evaporate at room temperature; colorless single crystals of (I) were formed after 12 d.

Refinement

The H atoms were positioned geometrically (O—H = 0.82 Å, C—H = 0.93–0.96 Å) and refined as riding with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(O, methyl C).

Figures

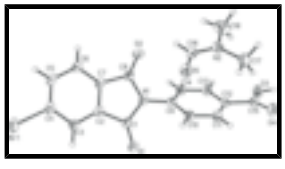

Fig. 1. The molecular structure of (I), drawn with 30% probability ellipsoids (arbitrary spheres for the H atoms).

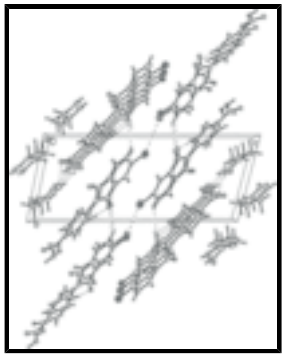

Fig. 2. The crystal packing of (I), viewed along the *a* axis. Hydrogen bonds are indicated by dashed lines.

4-(5-Bromo-1,3-dioxoisoindolin-2-yl)benzoic acid *N*,*N*-dimethylformamide solvate

Crystal data

$C_{15}H_8BrNO_4 \cdot C_3H_7NO$	$Z = 2$
$M_r = 419.23$	$F_{000} = 424$
Triclinic, $P\overline{1}$	$D_x = 1.583$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 5.9686$ (9) Å	Cell parameters from 2063 reflections
$b = 8.3214$ (12) Å	$\theta = 2.6$–26.1º
$c = 19.058$ (3) Å	$\mu = 2.37$ mm^{-1}
$\alpha = 101.231$ (2)º	$T = 294$ (2) K
$\beta = 93.417$ (3)º	Block, colorless
$\gamma = 107.273$ (2)º	0.26 × 0.22 × 0.12 mm
$V = 879.6$ (2) Å^3	

Data collection

Bruker SMART CCD diffractometer	3561 independent reflections
Radiation source: fine-focus sealed tube	2643 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.022$
$T = 294$(2) K	$\theta_{max} = 26.4$º
ω scans	$\theta_{min} = 1.1$º
Absorption correction: multi-scan (SADABS; Bruker, 1997)	$h = -7 \rightarrow 7$
$T_{min} = 0.578$, $T_{max} = 0.764$	$k = -10 \rightarrow 10$
5114 measured reflections	$l = -15 \rightarrow 23$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.040$	$w = 1/[\sigma^2(F_o^2) + (0.061P)^2 + 0.029P]$ where $P = (F_o^2 + 2F_c^2)/3$

$wR(F^2) = 0.118$	$(\Delta/\sigma)_{max} = 0.002$
$S = 1.13$	$\Delta\rho_{max} = 0.40$ e Å^{-3}
3561 reflections	$\Delta\rho_{min} = -0.42$ e Å^{-3}
239 parameters	Extinction correction: SHELXL97, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.062 (4)
Secondary atom site location: difference Fourier map	

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 $F^2 > 2\text{sigma}(F^2)$ 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}
Br1	−0.43214 (7)	0.20856 (4)	0.48540 (2)	0.0632 (2)
O1	−0.1509 (4)	0.8323 (3)	0.37081 (13)	0.0516 (6)
O2	0.3821 (4)	0.5901 (3)	0.26776 (14)	0.0597 (6)
O3	0.7047 (4)	1.3643 (3)	0.17597 (14)	0.0619 (7)
H3	0.7489	1.4260	0.1475	0.093*
O4	0.3495 (4)	1.3123 (3)	0.11588 (13)	0.0604 (6)
N1	0.1307 (4)	0.7455 (3)	0.30854 (14)	0.0405 (6)
C1	−0.0493 (5)	0.7278 (4)	0.35266 (16)	0.0390 (7)
C2	−0.0781 (5)	0.5619 (4)	0.37446 (16)	0.0369 (6)
C3	−0.2363 (5)	0.4823 (4)	0.41681 (17)	0.0415 (7)
H3A	−0.3506	0.5283	0.4351	0.050*
C4	−0.2140 (6)	0.3285 (4)	0.43040 (17)	0.0442 (7)
C5	−0.0431 (6)	0.2596 (4)	0.40437 (17)	0.0469 (8)
H5	−0.0324	0.1581	0.4156	0.056*
C6	0.1121 (6)	0.3415 (4)	0.36169 (17)	0.0443 (7)
H6	0.2279	0.2968	0.3438	0.053*
C7	0.0890 (5)	0.4929 (4)	0.34652 (16)	0.0399 (7)
C8	0.2262 (6)	0.6062 (4)	0.30239 (18)	0.0429 (7)
C9	0.2166 (5)	0.8872 (4)	0.27445 (16)	0.0374 (7)
C10	0.0590 (5)	0.9331 (4)	0.23213 (16)	0.0398 (7)
H10	−0.1025	0.8760	0.2275	0.048*
C11	0.1456 (5)	1.0653 (4)	0.19680 (16)	0.0406 (7)
H11	0.0410	1.0969	0.1684	0.049*
C12	0.3859 (5)	1.1507 (4)	0.20329 (15)	0.0359 (6)

C13	0.5403 (5)	1.1039 (4)	0.24672 (17)	0.0426 (7)
H13	0.7019	1.1609	0.2517	0.051*
C14	0.4556 (5)	0.9732 (4)	0.28260 (17)	0.0425 (7)
H14	0.5595	0.9436	0.3121	0.051*
C15	0.4749 (5)	1.2844 (4)	0.16040 (17)	0.0430 (7)
O5	−0.1165 (4)	0.5819 (3)	0.09856 (14)	0.0597 (6)
N2	0.2246 (4)	0.7231 (4)	0.06190 (15)	0.0479 (7)
C16	0.0984 (6)	0.6147 (4)	0.09704 (18)	0.0497 (8)
H16	0.1766	0.5586	0.1226	0.060*
C17	0.1156 (6)	0.8105 (5)	0.0182 (2)	0.0603 (9)
H17A	−0.0530	0.7689	0.0173	0.090*
H17B	0.1554	0.7881	−0.0300	0.090*
H17C	0.1721	0.9325	0.0384	0.090*
C18	0.4768 (7)	0.7532 (7)	0.0622 (3)	0.0818 (13)
H18A	0.5272	0.6866	0.0920	0.123*
H18B	0.5599	0.8736	0.0808	0.123*
H18C	0.5102	0.7193	0.0138	0.123*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0765 (3)	0.0469 (2)	0.0669 (3)	0.00944 (18)	0.01407 (19)	0.02855 (19)
O1	0.0564 (13)	0.0408 (12)	0.0702 (16)	0.0257 (11)	0.0207 (12)	0.0218 (11)
O2	0.0655 (15)	0.0520 (14)	0.0746 (17)	0.0320 (12)	0.0304 (13)	0.0169 (12)
O3	0.0464 (13)	0.0750 (17)	0.0739 (18)	0.0141 (12)	0.0162 (12)	0.0443 (14)
O4	0.0634 (15)	0.0670 (16)	0.0588 (15)	0.0195 (12)	0.0041 (12)	0.0350 (13)
N1	0.0437 (14)	0.0333 (13)	0.0491 (15)	0.0152 (11)	0.0108 (12)	0.0136 (11)
C1	0.0420 (16)	0.0320 (15)	0.0457 (18)	0.0150 (13)	0.0036 (14)	0.0103 (13)
C2	0.0414 (15)	0.0314 (14)	0.0398 (17)	0.0142 (12)	0.0018 (13)	0.0087 (12)
C3	0.0427 (16)	0.0351 (16)	0.0478 (18)	0.0124 (13)	0.0059 (14)	0.0114 (14)
C4	0.0550 (18)	0.0310 (15)	0.0424 (18)	0.0064 (13)	−0.0025 (14)	0.0125 (13)
C5	0.059 (2)	0.0298 (15)	0.052 (2)	0.0165 (14)	−0.0048 (16)	0.0097 (14)
C6	0.0519 (18)	0.0313 (15)	0.0508 (19)	0.0181 (13)	0.0017 (15)	0.0054 (14)
C7	0.0439 (16)	0.0299 (14)	0.0447 (18)	0.0121 (12)	−0.0010 (14)	0.0070 (13)
C8	0.0476 (17)	0.0331 (15)	0.0499 (19)	0.0161 (13)	0.0064 (15)	0.0083 (14)
C9	0.0437 (16)	0.0327 (15)	0.0399 (16)	0.0157 (12)	0.0087 (13)	0.0107 (13)
C10	0.0336 (14)	0.0418 (16)	0.0436 (17)	0.0115 (12)	0.0035 (13)	0.0093 (14)
C11	0.0419 (16)	0.0467 (17)	0.0391 (17)	0.0195 (13)	0.0039 (13)	0.0147 (14)
C12	0.0384 (15)	0.0390 (15)	0.0344 (16)	0.0163 (12)	0.0079 (12)	0.0103 (13)
C13	0.0338 (15)	0.0483 (18)	0.0462 (18)	0.0099 (13)	0.0046 (13)	0.0164 (15)
C14	0.0376 (16)	0.0460 (17)	0.0467 (18)	0.0144 (13)	−0.0010 (13)	0.0168 (15)
C15	0.0487 (18)	0.0422 (17)	0.0429 (18)	0.0165 (14)	0.0146 (14)	0.0144 (14)
O5	0.0533 (14)	0.0570 (15)	0.0764 (18)	0.0148 (11)	0.0215 (12)	0.0325 (13)
N2	0.0406 (14)	0.0561 (17)	0.0522 (17)	0.0185 (12)	0.0119 (12)	0.0176 (14)
C16	0.062 (2)	0.0484 (19)	0.049 (2)	0.0273 (16)	0.0127 (16)	0.0164 (16)
C17	0.060 (2)	0.067 (2)	0.065 (2)	0.0222 (18)	0.0136 (18)	0.034 (2)
C18	0.045 (2)	0.115 (4)	0.090 (3)	0.029 (2)	0.018 (2)	0.024 (3)

Geometric parameters (Å, °)

Br1—C4	1.895 (3)	C9—C10	1.387 (4)
O1—C1	1.210 (3)	C10—C11	1.387 (4)
O2—C8	1.197 (4)	C10—H10	0.9300
O3—C15	1.321 (4)	C11—C12	1.385 (4)
O3—H3	0.8200	C11—H11	0.9300
O4—C15	1.199 (4)	C12—C13	1.391 (4)
N1—C1	1.395 (4)	C12—C15	1.497 (4)
N1—C8	1.425 (4)	C13—C14	1.382 (4)
N1—C9	1.433 (3)	C13—H13	0.9300
C1—C2	1.484 (4)	C14—H14	0.9300
C2—C7	1.375 (4)	O5—C16	1.233 (4)
C2—C3	1.382 (4)	N2—C16	1.312 (4)
C3—C4	1.396 (4)	N2—C17	1.448 (4)
C3—H3A	0.9300	N2—C18	1.450 (4)
C4—C5	1.383 (5)	C16—H16	0.9300
C5—C6	1.384 (4)	C17—H17A	0.9600
C5—H5	0.9300	C17—H17B	0.9600
C6—C7	1.389 (4)	C17—H17C	0.9600
C6—H6	0.9300	C18—H18A	0.9600
C7—C8	1.483 (4)	C18—H18B	0.9600
C9—C14	1.377 (4)	C18—H18C	0.9600
?···?	?		
C15—O3—H3	109.5	C12—C11—C10	120.8 (3)
C1—N1—C8	111.7 (2)	C12—C11—H11	119.6
C1—N1—C9	125.2 (2)	C10—C11—H11	119.6
C8—N1—C9	123.1 (2)	C11—C12—C13	119.0 (3)
O1—C1—N1	125.7 (3)	C11—C12—C15	119.4 (3)
O1—C1—C2	128.4 (3)	C13—C12—C15	121.5 (3)
N1—C1—C2	105.8 (2)	C14—C13—C12	120.6 (3)
C7—C2—C3	122.2 (3)	C14—C13—H13	119.7
C7—C2—C1	108.7 (3)	C12—C13—H13	119.7
C3—C2—C1	129.1 (3)	C9—C14—C13	119.6 (3)
C2—C3—C4	115.9 (3)	C9—C14—H14	120.2
C2—C3—H3A	122.1	C13—C14—H14	120.2
C4—C3—H3A	122.1	O4—C15—O3	124.2 (3)
C5—C4—C3	122.8 (3)	O4—C15—C12	122.9 (3)
C5—C4—Br1	119.2 (2)	O3—C15—C12	112.8 (3)
C3—C4—Br1	118.1 (3)	C16—N2—C17	121.3 (3)
C4—C5—C6	120.1 (3)	C16—N2—C18	121.3 (3)
C4—C5—H5	119.9	C17—N2—C18	117.3 (3)
C6—C5—H5	119.9	O5—C16—N2	124.7 (3)
C5—C6—C7	117.7 (3)	O5—C16—H16	117.6
C5—C6—H6	121.1	N2—C16—H16	117.6
C7—C6—H6	121.1	N2—C17—H17A	109.5
C2—C7—C6	121.3 (3)	N2—C17—H17B	109.5
C2—C7—C8	109.0 (2)	H17A—C17—H17B	109.5

C6—C7—C8	129.7 (3)	N2—C17—H17C	109.5
O2—C8—N1	125.2 (3)	H17A—C17—H17C	109.5
O2—C8—C7	130.0 (3)	H17B—C17—H17C	109.5
N1—C8—C7	104.8 (3)	N2—C18—H18A	109.5
C14—C9—C10	120.8 (3)	N2—C18—H18B	109.5
C14—C9—N1	119.5 (2)	H18A—C18—H18B	109.5
C10—C9—N1	119.7 (3)	N2—C18—H18C	109.5
C11—C10—C9	119.1 (3)	H18A—C18—H18C	109.5
C11—C10—H10	120.4	H18B—C18—H18C	109.5
C9—C10—H10	120.4		
C8—N1—C1—O1	175.1 (3)	C2—C7—C8—O2	−177.9 (3)
C9—N1—C1—O1	−3.3 (5)	C6—C7—C8—O2	3.0 (6)
C8—N1—C1—C2	−2.1 (3)	C2—C7—C8—N1	1.4 (3)
C9—N1—C1—C2	179.4 (2)	C6—C7—C8—N1	−177.6 (3)
O1—C1—C2—C7	−174.2 (3)	C1—N1—C9—C14	128.3 (3)
N1—C1—C2—C7	3.0 (3)	C8—N1—C9—C14	−50.0 (4)
O1—C1—C2—C3	4.4 (5)	C1—N1—C9—C10	−53.4 (4)
N1—C1—C2—C3	−178.5 (3)	C8—N1—C9—C10	128.3 (3)
C7—C2—C3—C4	0.9 (4)	C14—C9—C10—C11	1.2 (4)
C1—C2—C3—C4	−177.4 (3)	N1—C9—C10—C11	−177.1 (3)
C2—C3—C4—C5	0.9 (4)	C9—C10—C11—C12	0.1 (4)
C2—C3—C4—Br1	−177.9 (2)	C10—C11—C12—C13	−0.9 (4)
C3—C4—C5—C6	−1.3 (5)	C10—C11—C12—C15	175.8 (3)
Br1—C4—C5—C6	177.4 (2)	C11—C12—C13—C14	0.4 (5)
C4—C5—C6—C7	0.0 (5)	C15—C12—C13—C14	−176.2 (3)
C3—C2—C7—C6	−2.2 (5)	C10—C9—C14—C13	−1.7 (5)
C1—C2—C7—C6	176.4 (3)	N1—C9—C14—C13	176.6 (3)
C3—C2—C7—C8	178.6 (3)	C12—C13—C14—C9	0.9 (5)
C1—C2—C7—C8	−2.7 (3)	C11—C12—C15—O4	−6.1 (5)
C5—C6—C7—C2	1.7 (4)	C13—C12—C15—O4	170.5 (3)
C5—C6—C7—C8	−179.3 (3)	C11—C12—C15—O3	175.2 (3)
C1—N1—C8—O2	180.0 (3)	C13—C12—C15—O3	−8.2 (4)
C9—N1—C8—O2	−1.5 (5)	C17—N2—C16—O5	−2.6 (5)
C1—N1—C8—C7	0.5 (3)	C18—N2—C16—O5	−179.3 (4)
C9—N1—C8—C7	179.1 (2)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3···O5^{i}	0.82	1.77	2.583 (3)	170
C5—H5···O1^{ii}	0.93	2.54	3.343 (4)	144
C14—H14···O1^{iii}	0.93	2.49	3.413 (4)	170
C16—H16···O4^{ii}	0.93	2.54	3.352 (4)	146
C18—H18A···O5^{iii}	0.96	2.53	3.262 (5)	133

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

Fig. 1

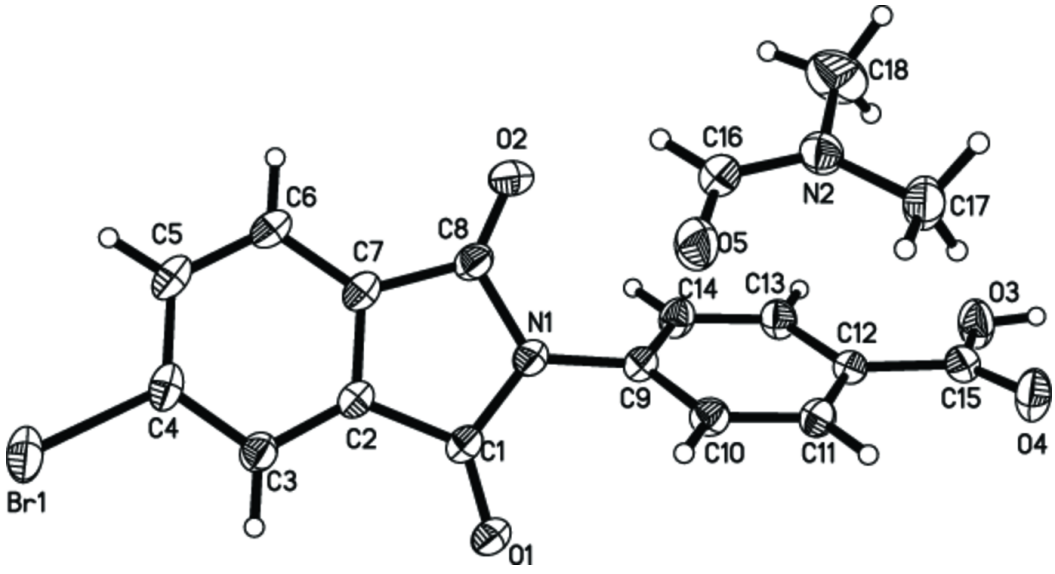
C18
C16
O2
N2
C17
C6
C8
O5
C5
C7
C14
C13
O3
N1
C12
C9
C15
C4
C2
C1
C10
C11
O4
C3
Br1
O1

Fig. 2