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(*S*)-Methyl 2-(1,3-dioxoisoindolin-2-yl)-3-(4-iodophenyl)propionate

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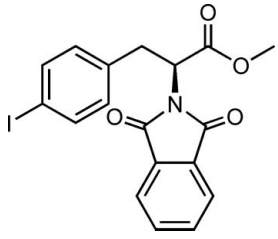
Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.009 Å; disorder in main residue; R factor = 0.041; wR factor = 0.108; data-to-parameter ratio = 13.5.

In the chiral title compound, $C_{18}H_{14}INO_4$, the dihedral angle between the aromatic ring planes is 54.0 (3)°. The crystal packing is stabilized by C—H···O interactions. The methoxy group is disordered essentially equally over two positions.

Related literature

For related literature, see: Paquette *et al.* (1997); Hutton (1997).



Experimental

Crystal data

$C_{18}H_{14}INO_4$
M_r = 435.20
Hexagonal, $P6_1$
a = 10.1246 (6) Å
c = 29.528 (3) Å
V = 2621.3 (3) Å^3
Z = 6
Mo $K\alpha$ radiation
μ = 1.85 mm^{-1}
T = 294 (2) K
0.26 × 0.20 × 0.18 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
T_{min} = 0.634, T_{max} = 0.732
15228 measured reflections
3215 independent reflections
2893 reflections with $I > 2\sigma(I)$
R_{int} = 0.041

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.108
S = 1.07
3215 reflections
238 parameters
29 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.46 e Å^{-3}
Absolute structure: Flack (1983), 1383 Friedel pairs
Flack parameter: 0.02 (3)

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

D—H···A	D—H	H···A	D···A	D—H···A
C12—H12···O2^{i}	0.93	2.43	3.236 (8)	145
C8—H8···O1^{ii}	0.93	2.46	3.379 (8)	170

Symmetry codes: (i) $y, -x+y+1, z-\frac{1}{6}$; (ii) $x-y+1, x, z+\frac{1}{6}$.

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*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2624).

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supplementary materials

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(*S*)-Methyl 2-(1,3-dioxoisoindolin-2-yl)-3-(4-iodophenyl)propionate

Z. Wu, P. Shen and S. Jiang

Comment

The title compound, (I), is an intermediate in the synthesis of β-hydroxyphenylalanine (Hutton, 1997), a compound which occurs in a wide range of important biologically active peptides. A view of the molecular structure of (I) is shown in Fig. 1. A l l bonds lengths and angles are normal.

The dihedral angle between the aromatic ring planes is 54.0 (3)°. The dihedral angle between the mean planes of C9/C17/O3/O4 and the benzene ring is 73.06 (69) Å. That between C9/C17/O3/O4 and the 3a,7a-dihydro-isoindole-1,3-dione ring is 71.85 (34) Å.

Neighboring molecules in the crystal are linked together *via* weak C—H···O hydrogen bonds (Table 1, Fig. 2).

Experimental

To a suspension of 4-iodo-*L*-phenylalanine methyl ester hydrochloride (0.49 g, 1.4 mmol) and phthalic anhydride (0.38 g, 2.6 mmol) in toluene (20 ml) was added triethylamine (0.5 ml, 3.6 mmol) and the resulting mixture was heated to reflux for three hours with stirring in an oil-bath at 413–418 K. After cooling to room temperature, the solvent was evaporated and the residue was washed with water, then extracted with ethyl acetate (2 × 20 ml). The combined organic layers were dried (Na_2SO_4) and evaporated to give a crude product which was purified by column chromatography on silica gel with petroleum ether-ethyl acetate (2:1 *v*/*v*) as eluent to give the product (0.60 g, 97%) (Paquette *et al.*, 1997). Crystals of (I) were grown by slow evaporation of solution of ethyl acetate and petroleum ether (1:4 *v*/*v*) at room temperature over a period of 3 days.

Refinement

The H atoms were positioned geometrically (C—H = 0.93–0.97 Å) and refined as riding with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C).

Figures

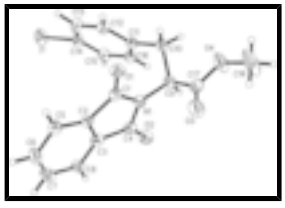

Fig. 1. A view of the molecular structure of (I). Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as spheres of arbitrary radius.

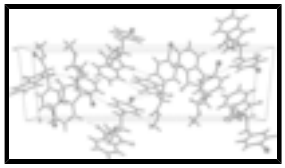

Fig. 2. The packing diagram for (I).

Methyl 2-(1,3-dioxoisoindolin-2-yl)-3-(4-iodophenyl)propionate

Crystal data

$C_{18}H_{14}INO_4$	$Z = 6$
$M_r = 435.20$	$F_{000} = 1284$
Hexagonal, $P6_1$	$D_x = 1.654$ Mg m^{-3}
Hall symbol: P 61	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 10.1246$ (6) Å	Cell parameters from 6976 reflections
$b = 10.1246$ (6) Å	$\theta = 2.3$–26.0º
$c = 29.528$ (3) Å	$\mu = 1.85$ mm^{-1}
$\alpha = 90$º	$T = 294$ (2) K
$\beta = 90$º	Block, colorless
$\gamma = 120$º	0.26 × 0.20 × 0.18 mm
$V = 2621.3$ (3) Å^3	

Data collection

Bruker SMART CCD diffractometer	3215 independent reflections
Radiation source: fine-focus sealed tube	2893 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.041$
$T = 294$(2) K	$\theta_{max} = 26.5$º
ω scans	$\theta_{min} = 2.3$º
Absorption correction: multi-scan (SADABS; Bruker, 1997)	$h = -12 \rightarrow 12$
$T_{min} = 0.634$, $T_{max} = 0.732$	$k = -10 \rightarrow 12$
15228 measured reflections	$l = -23 \rightarrow 37$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.041$	$w = 1/[\sigma^2(F_o^2) + (0.0545P)^2 + 3.4009P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.108$	$(\Delta/\sigma)_{max} = 0.002$
$S = 1.07$	$\Delta\rho_{max} = 1.24$ e Å^{-3}
3215 reflections	$\Delta\rho_{min} = -0.46$ e Å^{-3}
238 parameters	Extinction correction: none

29 restraints

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Absolute structure: Flack (1983), 1383 Friedel pairs

Flack parameter: 0.02 (3)

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}	Occ. (<1)
I1	0.77592 (5)	1.07946 (5)	0.10858 (3)	0.06792 (18)	
N1	0.4928 (5)	0.3527 (5)	0.05051 (16)	0.0360 (10)	
O1	0.4372 (5)	0.3960 (5)	−0.02138 (15)	0.0465 (10)	
O2	0.6200 (5)	0.3471 (6)	0.11397 (17)	0.0600 (12)	
O3	0.2869 (6)	0.0642 (6)	0.0247 (2)	0.0713 (16)	
C1	0.5277 (6)	0.4077 (6)	0.00639 (19)	0.0341 (11)	
C2	0.6966 (6)	0.4763 (6)	0.0025 (2)	0.0359 (12)	
C3	0.7501 (6)	0.4599 (6)	0.0432 (2)	0.0396 (12)	
C4	0.6223 (7)	0.3823 (7)	0.0750 (2)	0.0411 (13)	
C5	0.7908 (7)	0.5437 (7)	−0.0339 (2)	0.0445 (13)	
H5	0.7540	0.5577	−0.0613	0.053*	
C6	0.9429 (7)	0.5898 (7)	−0.0280 (3)	0.0563 (17)	
H6	1.0097	0.6341	−0.0522	0.068*	
C7	0.9983 (7)	0.5724 (7)	0.0123 (3)	0.0562 (18)	
H7	1.1014	0.6038	0.0149	0.067*	
C8	0.9028 (7)	0.5082 (7)	0.0499 (3)	0.0499 (15)	
H8	0.9399	0.4986	0.0778	0.060*	
C9	0.3423 (6)	0.2826 (6)	0.0706 (2)	0.0377 (11)	
H9	0.3529	0.2611	0.1023	0.045*	
C10	0.2728 (6)	0.3867 (6)	0.0705 (2)	0.0420 (13)	
H10A	0.2273	0.3810	0.0411	0.050*	
H10B	0.1925	0.3510	0.0930	0.050*	
C11	0.3893 (6)	0.5489 (7)	0.0803 (2)	0.0380 (12)	
C12	0.4320 (7)	0.6593 (7)	0.0470 (2)	0.0442 (13)	
H12	0.3870	0.6317	0.0186	0.053*	
C13	0.5407 (8)	0.8105 (7)	0.0554 (2)	0.0519 (15)	
H13	0.5649	0.8837	0.0330	0.062*	
C14	0.6121 (7)	0.8516 (6)	0.0965 (2)	0.0449 (14)	
C15	0.5726 (7)	0.7436 (7)	0.1301 (2)	0.0455 (13)	

H15	0.6206	0.7719	0.1581	0.055*	
C16	0.4634 (7)	0.5953 (7)	0.1225 (2)	0.0430 (13)	
H16	0.4377	0.5240	0.1455	0.052*	
C17	0.2436 (7)	0.1291 (7)	0.0471 (2)	0.0461 (14)	
O4	0.1059 (13)	0.0680 (14)	0.0656 (8)	0.053 (4)	0.49 (4)
C18	−0.0093 (14)	−0.0829 (17)	0.0483 (10)	0.071 (6)	0.49 (4)
H18A	0.0404	−0.1362	0.0374	0.107*	0.49 (4)
H18B	−0.0651	−0.0702	0.0240	0.107*	0.49 (4)
H18C	−0.0783	−0.1406	0.0723	0.107*	0.49 (4)
O4'	0.0968 (8)	0.0875 (16)	0.0475 (8)	0.051 (4)	0.51 (4)
C18'	−0.0090 (15)	−0.0543 (18)	0.0238 (10)	0.067 (6)	0.51 (4)
H18D	0.0251	−0.0502	−0.0067	0.100*	0.51 (4)
H18E	−0.1092	−0.0660	0.0236	0.100*	0.51 (4)
H18F	−0.0123	−0.1394	0.0392	0.100*	0.51 (4)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
I1	0.0592 (3)	0.0433 (2)	0.0838 (3)	0.0125 (2)	0.0126 (3)	−0.0134 (3)
N1	0.038 (2)	0.040 (2)	0.030 (2)	0.020 (2)	0.0019 (19)	0.0005 (19)
O1	0.047 (2)	0.058 (3)	0.039 (2)	0.030 (2)	−0.0060 (19)	0.0018 (19)
O2	0.062 (3)	0.082 (3)	0.042 (3)	0.040 (2)	−0.004 (2)	0.002 (2)
O3	0.059 (3)	0.054 (3)	0.098 (4)	0.026 (2)	0.020 (3)	−0.017 (3)
C1	0.042 (3)	0.031 (2)	0.031 (3)	0.019 (2)	−0.002 (2)	−0.004 (2)
C2	0.036 (3)	0.028 (2)	0.045 (3)	0.018 (2)	−0.001 (2)	−0.003 (2)
C3	0.045 (3)	0.038 (3)	0.039 (3)	0.023 (2)	−0.009 (2)	−0.007 (2)
C4	0.046 (3)	0.043 (3)	0.039 (3)	0.026 (3)	−0.008 (2)	−0.005 (2)
C5	0.046 (3)	0.041 (3)	0.044 (3)	0.019 (3)	0.003 (3)	0.001 (3)
C6	0.044 (3)	0.043 (3)	0.067 (5)	0.010 (3)	0.016 (3)	0.002 (3)
C7	0.032 (3)	0.050 (4)	0.082 (6)	0.017 (3)	−0.005 (3)	−0.015 (3)
C8	0.036 (3)	0.050 (3)	0.064 (4)	0.022 (3)	−0.012 (3)	−0.009 (3)
C9	0.036 (3)	0.041 (3)	0.037 (3)	0.020 (2)	0.005 (2)	0.001 (2)
C10	0.040 (3)	0.044 (3)	0.045 (4)	0.023 (3)	0.003 (2)	−0.005 (2)
C11	0.040 (3)	0.050 (3)	0.029 (3)	0.027 (3)	0.004 (2)	0.004 (2)
C12	0.050 (3)	0.044 (3)	0.036 (3)	0.023 (3)	−0.005 (3)	−0.004 (2)
C13	0.059 (4)	0.042 (3)	0.046 (4)	0.018 (3)	0.003 (3)	0.005 (3)
C14	0.042 (3)	0.033 (3)	0.051 (4)	0.012 (2)	0.005 (3)	−0.006 (2)
C15	0.047 (3)	0.049 (3)	0.040 (3)	0.024 (3)	−0.007 (3)	−0.014 (3)
C16	0.042 (3)	0.047 (3)	0.040 (3)	0.022 (3)	0.005 (2)	0.005 (2)
C17	0.046 (3)	0.036 (3)	0.057 (4)	0.021 (3)	0.001 (3)	−0.005 (3)
O4	0.044 (5)	0.039 (5)	0.067 (8)	0.015 (4)	0.013 (5)	−0.002 (5)
C18	0.064 (8)	0.064 (8)	0.076 (10)	0.024 (6)	0.007 (7)	−0.023 (7)
O4'	0.040 (5)	0.046 (6)	0.061 (8)	0.017 (4)	0.002 (4)	−0.014 (5)
C18'	0.054 (8)	0.044 (7)	0.082 (10)	0.008 (5)	0.008 (6)	−0.013 (7)

Geometric parameters (Å, °)

I1—C14	2.091 (6)	C10—H10A	0.9700
N1—C1	1.391 (7)	C10—H10B	0.9700

N1—C4	1.393 (7)	C11—C12	1.386 (9)
N1—C9	1.448 (7)	C11—C16	1.407 (8)
O1—C1	1.191 (7)	C12—C13	1.389 (9)
O2—C4	1.201 (8)	C12—H12	0.9300
O3—C17	1.162 (7)	C13—C14	1.368 (10)
C1—C2	1.494 (8)	C13—H13	0.9300
C2—C3	1.363 (8)	C14—C15	1.377 (9)
C2—C5	1.371 (9)	C15—C16	1.367 (9)
C3—C8	1.383 (8)	C15—H15	0.9300
C3—C4	1.469 (9)	C16—H16	0.9300
C5—C6	1.379 (9)	C17—O4	1.327 (9)
C5—H5	0.9300	C17—O4'	1.327 (9)
C6—C7	1.364 (11)	O4—C18	1.474 (9)
C6—H6	0.9300	C18—H18A	0.9600
C7—C8	1.402 (11)	C18—H18B	0.9600
C7—H7	0.9300	C18—H18C	0.9600
C8—H8	0.9300	O4'—C18'	1.469 (9)
C9—C17	1.530 (8)	C18'—H18D	0.9600
C9—C10	1.531 (8)	C18'—H18E	0.9600
C9—H9	0.9800	C18'—H18F	0.9600
C10—C11	1.495 (8)		
C1—N1—C4	112.1 (5)	C11—C10—H10B	109.2
C1—N1—C9	125.1 (5)	C9—C10—H10B	109.2
C4—N1—C9	122.6 (5)	H10A—C10—H10B	107.9
O1—C1—N1	125.2 (5)	C12—C11—C16	117.3 (6)
O1—C1—C2	129.6 (5)	C12—C11—C10	120.3 (5)
N1—C1—C2	105.2 (5)	C16—C11—C10	122.4 (5)
C3—C2—C5	122.2 (5)	C11—C12—C13	121.2 (6)
C3—C2—C1	107.8 (5)	C11—C12—H12	119.4
C5—C2—C1	130.0 (6)	C13—C12—H12	119.4
C2—C3—C8	121.7 (6)	C14—C13—C12	120.0 (6)
C2—C3—C4	109.4 (5)	C14—C13—H13	120.0
C8—C3—C4	128.8 (6)	C12—C13—H13	120.0
O2—C4—N1	123.9 (6)	C13—C14—C15	119.8 (6)
O2—C4—C3	130.8 (6)	C13—C14—I1	119.6 (5)
N1—C4—C3	105.4 (5)	C15—C14—I1	120.6 (5)
C2—C5—C6	116.7 (6)	C16—C15—C14	120.6 (6)
C2—C5—H5	121.6	C16—C15—H15	119.7
C6—C5—H5	121.6	C14—C15—H15	119.7
C7—C6—C5	122.0 (6)	C15—C16—C11	121.1 (6)
C7—C6—H6	119.0	C15—C16—H16	119.5
C5—C6—H6	119.0	C11—C16—H16	119.5
C6—C7—C8	121.2 (6)	O3—C17—O4	125.1 (8)
C6—C7—H7	119.4	O3—C17—O4'	120.3 (8)
C8—C7—H7	119.4	O4—C17—O4'	25.8 (7)
C3—C8—C7	116.1 (6)	O3—C17—C9	126.4 (6)
C3—C8—H8	121.9	O4—C17—C9	106.7 (7)
C7—C8—H8	121.9	O4'—C17—C9	112.2 (7)
N1—C9—C17	107.7 (4)	C17—O4—C18	116.4 (9)

N1—C9—C10	113.3 (5)	C17—O4'—C18'	117.4 (9)
C17—C9—C10	114.0 (5)	O4'—C18'—H18D	109.5
N1—C9—H9	107.2	O4'—C18'—H18E	109.5
C17—C9—H9	107.2	H18D—C18'—H18E	109.5
C10—C9—H9	107.2	O4'—C18'—H18F	109.5
C11—C10—C9	111.9 (5)	H18D—C18'—H18F	109.5
C11—C10—H10A	109.2	H18E—C18'—H18F	109.5
C9—C10—H10A	109.2		
C4—N1—C1—O1	179.5 (5)	C1—N1—C9—C10	−58.6 (7)
C9—N1—C1—O1	−4.8 (8)	C4—N1—C9—C10	116.7 (6)
C4—N1—C1—C2	1.2 (6)	N1—C9—C10—C11	−38.7 (7)
C9—N1—C1—C2	176.9 (5)	C17—C9—C10—C11	−162.4 (5)
O1—C1—C2—C3	−178.8 (6)	C9—C10—C11—C12	112.3 (6)
N1—C1—C2—C3	−0.6 (6)	C9—C10—C11—C16	−65.6 (7)
O1—C1—C2—C5	0.2 (10)	C16—C11—C12—C13	−1.6 (9)
N1—C1—C2—C5	178.4 (5)	C10—C11—C12—C13	−179.6 (6)
C5—C2—C3—C8	−1.1 (9)	C11—C12—C13—C14	2.6 (10)
C1—C2—C3—C8	177.9 (5)	C12—C13—C14—C15	−2.0 (10)
C5—C2—C3—C4	−179.3 (5)	C12—C13—C14—I1	179.8 (5)
C1—C2—C3—C4	−0.2 (6)	C13—C14—C15—C16	0.5 (9)
C1—N1—C4—O2	179.0 (6)	I1—C14—C15—C16	178.7 (4)
C9—N1—C4—O2	3.3 (9)	C14—C15—C16—C11	0.4 (9)
C1—N1—C4—C3	−1.4 (6)	C12—C11—C16—C15	0.2 (8)
C9—N1—C4—C3	−177.1 (5)	C10—C11—C16—C15	178.1 (5)
C2—C3—C4—O2	−179.5 (6)	N1—C9—C17—O3	15.4 (9)
C8—C3—C4—O2	2.5 (11)	C10—C9—C17—O3	142.0 (7)
C2—C3—C4—N1	0.9 (6)	N1—C9—C17—O4	−179.1 (12)
C8—C3—C4—N1	−177.0 (6)	C10—C9—C17—O4	−52.6 (13)
C3—C2—C5—C6	2.3 (9)	N1—C9—C17—O4'	−152.3 (13)
C1—C2—C5—C6	−176.6 (6)	C10—C9—C17—O4'	−25.7 (14)
C2—C5—C6—C7	−1.3 (9)	O3—C17—O4—C18	−14 (2)
C5—C6—C7—C8	−0.8 (10)	O4'—C17—O4—C18	74 (2)
C2—C3—C8—C7	−1.0 (9)	C9—C17—O4—C18	−179.4 (13)
C4—C3—C8—C7	176.8 (6)	O3—C17—O4'—C18'	8(2)
C6—C7—C8—C3	2.0 (10)	O4—C17—O4'—C18'	−101 (3)
C1—N1—C9—C17	68.4 (6)	C9—C17—O4'—C18'	176.9 (12)
C4—N1—C9—C17	−116.4 (6)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C12—H12···O2^{i}	0.93	2.43	3.236 (8)	145
C8—H8···O1^{ii}	0.93	2.46	3.379 (8)	170

Symmetry codes: (i) *y*, −*x*+*y*+1, *z*−1/6; (ii) *x*−*y*+1, *x*, *z*+1/6.

Fig. 1

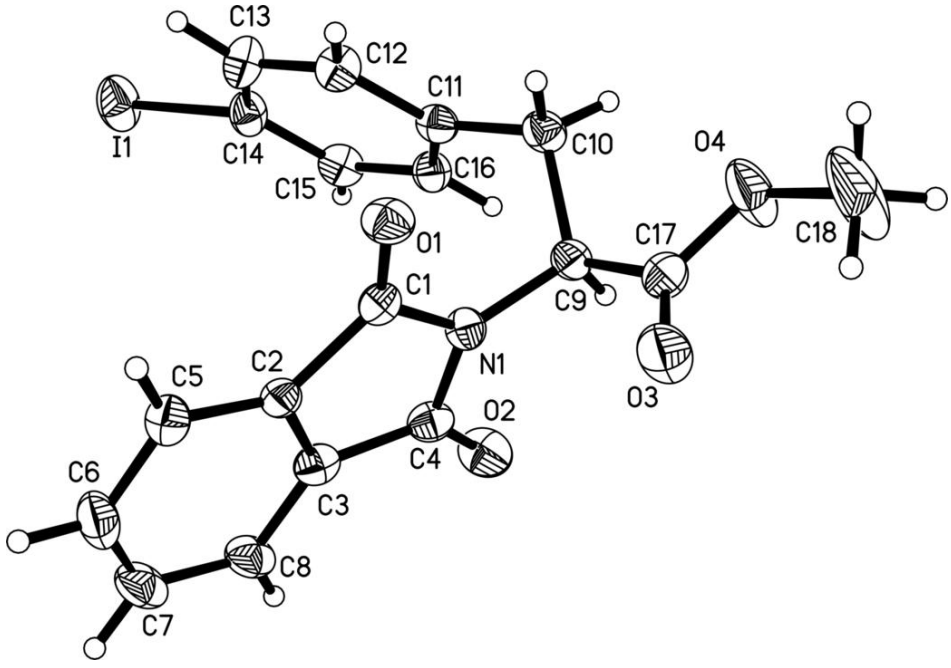
C13
C12
C11
I1
C14
C10
O4
C16
C15
O1
C17
C18
C1
C9
N1
C5
C2
O3
O2
C6
C4
C3
C8
C7

Fig. 2