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(*E*)-2-[2-(4-Bromobenzoylmethylene)-imidazolidin-1-yl]ethyl 4-bromobenzoate

Chu-Yi Yu,[a]* Xue-Ning Yuan,[b] Li-Ben Wang[a] and Zhi-Tang Huang[a]*

[a]Beijing National Laboratory for Molecular Science (BNLMS), Laboratory for Chemical Biology, Institute of Chemistry, Chinese Academy of Sciences, Beijing 100080, People's Republic of China, and [b]Graduate University of the Chinese Academy of Sciences, Beijing 100080, People's Republic of China
Correspondence e-mail: yucy@iccas.ac.cn, huangzt@public.bta.net.cn

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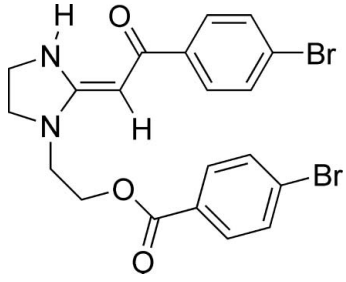
Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.008 Å; R factor = 0.050; wR factor = 0.137; data-to-parameter ratio = 13.7.

The title compound, $C_{20}H_{18}Br_2N_2O_3$, exhibits intermolecular N—H···O hydrogen bonds which form pseudo-dimers across inversion centres. Intramolecular N—H···O hydrogen bonds and weak intramolecular C—H···π hydrogen-bonding interactions are also present within the molecule.

Related literature

For related literature, see: Huang & Wang (2002); Wang & Huang (1996); Wang *et al.* (1987).



Experimental

Crystal data

$C_{20}H_{18}Br_2N_2O_3$
M_r = 494.18
Triclinic, $P\overline{1}$
a = 7.3733 (15) Å
b = 11.117 (2) Å
c = 12.954 (2) Å
α = 104.635 (10)°
β = 105.108 (11)°
γ = 96.700 (9)°
V = 972.7 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 4.19 mm^{-1}
T = 294 (2) K
0.39 × 0.37 × 0.34 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
T_{min} = 0.292, T_{max} = 0.330
(expected range = 0.213–0.241)
4924 measured reflections
3389 independent reflections
2404 reflections with $I > 2\sigma(I)$
R_{int} = 0.024

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.137
S = 1.04
3389 reflections
247 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.88 e Å^{-3}
$\Delta\rho_{min}$ = −0.78 e Å^{-3}

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

*Cg*1 is the centroid of the C6–C11 benzene ring.

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1	0.78 (5)	2.16 (5)	2.706 (5)	128 (5)
N1—H1···O1^{i}	0.78 (5)	2.48 (6)	3.020 (5)	128 (5)
C3—H3*A*···O3^{ii}	0.97	2.43	3.296 (7)	148
C8—H8···O1^{iii}	0.93	2.40	3.264 (5)	154
C19—H19···*Cg*1	0.93	3.07	3.841 (6)	141

Symmetry codes: (i) $-x+2, -y+1, -z+1$; (ii) $-x+1, -y, -z$; (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: *ORTEPIII* (Burnett & Johnson, 1996), *ORTEP-3 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

The authors thank Mr Haibin Song of Nankai University for the X-ray crystallographic determination.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DN2178).

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supplementary materials

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(*E*)-2-[2-(4-Bromobenzoylmethylene)imidazolidin-1-yl]ethyl 4-bromobenzoate

C.-Y. Yu, X.-N. Yuan, L.-B. Wang and Z.-T. Huang

Comment

Heterocyclic ketene aminals (HKAs) are bis-nucleophiles which are valuable synthons for heterocyclic synthesis (Huang & Wang, 2002). The title compound, (I), (Fig. 1) is an *N*-alkylation product of a HKA. The crystal structure of (I) was determined in order to provide information regarding its electronic conjugation properties and to examine a possible intramolecular hydrogen bond (Wang *et al.*,1987).

In the title compound, the two substituted phenyl rings make dihedral angle of 84.0 (2)°, such conformation results from the occurrence of C—H···π interaction between the C15—C20 and C6—C11 rings (Fig.1, Table 1). The H atom attached to N1 is engaged in intramolecular and intermolecular hydrogen bonds. The intermolecular hydrogen bond results in the formation of pseudo dimers which are further interconnected through C—H···O weak hydrogen bonds to form a three dimensionnal network (Fig. 2, Table 1). The bond distances and bond angles are within the expected range observed for related compounds.

Experimental

Compound (I) was prepared according to the procedure of Wang *et al.* (1996) and purified by recrystallization from acetone in 19% yield (m.p. 449–450 K). Anal. Calcd for $C_{20}H_{18}Br_2N_2O_3$: C 48.61, H 3.67, N 5.67, Br 32.34%; found: C 48.64, H 3.64, N 5.38, Br 32.47%.

Refinement

All H atoms attached to C were fixed geometrically and treated as riding with C—H = 0.93 Å (aromatic) or 0.97 Å (methylene) and with U_{iso}(H) = 1.2U_{eq}(C). H attached to N atom were freely refined with U_{iso}(H) = 1.2U_{eq}(N)

Figures

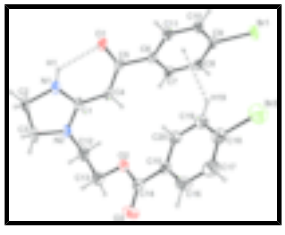

Fig. 1. The molecular structure of (I), showing the atom-numbering scheme. Ellipsoids are drawn at the 30% probability level. H atoms are represented as small spheres of arbitrary radii. The C—H···π and N—H···O intramolecular interactions are shown as dashed lines (CT1 is the centroid of the C6–C11 phenyl ring).

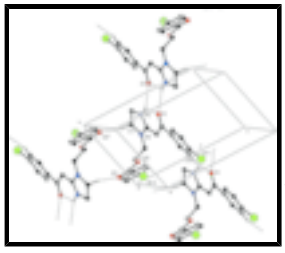

Fig. 2. Partial packing view showing the N—H···O and C—H···O hydrogen bonds network. Hydrogen bonds are shown as dashed lines. H atoms not involved in hydrogen bonding have been omitted for clarity. [Symmetry codes: (i) $2-x$, $1-y$, $1-z$; (ii) $1-x$, $-y$, $-z$; (iii) $x-1$, y, z].

(*E*)-2-[2-(4-Bromobenzoylmethylene)imidazolidin-1-yl]ethyl 4-bromobenzoate

Crystal data

$C_{20}H_{18}Br_2N_2O_3$	$Z = 2$
$M_r = 494.18$	$F_{000} = 492$
Triclinic, $P\bar{1}$	$D_x = 1.687$ Mg m^{-3}
Hall symbol: -P 1	Melting point: 449 K
$a = 7.3733$ (15) Å	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$b = 11.117$ (2) Å	Cell parameters from 1807 reflections
$c = 12.954$ (2) Å	$\theta = 2.9$–25.4°
$\alpha = 104.635$ (10)°	$\mu = 4.19$ mm^{-1}
$\beta = 105.108$ (11)°	$T = 294$ (2) K
$\gamma = 96.700$ (9)°	Prism, colourless
$V = 972.7$ (3) Å^3	0.39 × 0.37 × 0.34 mm

Data collection

Bruker SMART CCD area-detector diffractometer	3389 independent reflections
Radiation source: fine-focus sealed tube	2404 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.024$
$T = 294$(2) K	$\theta_{max} = 25.0^\circ$
φ and ω scans	$\theta_{min} = 1.7^\circ$
Absorption correction: multi-scan (SADABS; Bruker, 1997)	$h = -4 \rightarrow 8$
$T_{min} = 0.292$, $T_{max} = 0.330$	$k = -13 \rightarrow 11$
4924 measured reflections	$l = -15 \rightarrow 15$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.050$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.137$	$w = 1/[\sigma^2(F_o^2) + (0.0744P)^2 + 0.4017P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.04$	$(\Delta/\sigma)_{max} < 0.001$

3389 reflections	$\Delta\rho_{max} = 0.88$ e Å^{-3}
247 parameters	$\Delta\rho_{min} = -0.78$ 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 $F^2 > 2\text{sigma}(F^2)$ 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	0.11210 (7)	0.92243 (5)	0.38589 (6)	0.0615 (2)
Br2	0.22475 (12)	0.75878 (7)	−0.04472 (7)	0.0909 (3)
N1	0.8470 (6)	0.3228 (4)	0.3643 (4)	0.0445 (11)
H1	0.904 (8)	0.388 (5)	0.405 (5)	0.053*
N2	0.5887 (5)	0.1767 (4)	0.2816 (4)	0.0459 (10)
O1	0.8064 (4)	0.5665 (3)	0.4319 (3)	0.0473 (9)
O2	0.2955 (6)	0.2177 (3)	0.0964 (3)	0.0669 (12)
O3	0.2102 (9)	0.1289 (4)	−0.0866 (4)	0.121 (2)
C1	0.6565 (6)	0.3019 (4)	0.3296 (4)	0.0346 (10)
C2	0.9211 (7)	0.2063 (5)	0.3468 (5)	0.0569 (14)
H2A	0.9879	0.1942	0.4175	0.068*
H2B	1.0075	0.2062	0.3017	0.068*
C3	0.7435 (8)	0.1057 (5)	0.2862 (5)	0.0583 (15)
H3A	0.7447	0.0616	0.2116	0.070*
H3B	0.7313	0.0445	0.3269	0.070*
C4	0.5481 (6)	0.3957 (4)	0.3401 (4)	0.0343 (10)
H4	0.4154	0.3720	0.3129	0.041*
C5	0.6293 (6)	0.5236 (4)	0.3895 (4)	0.0312 (10)
C6	0.4998 (6)	0.6184 (4)	0.3882 (3)	0.0289 (9)
C7	0.3073 (6)	0.5882 (4)	0.3806 (3)	0.0318 (10)
H7	0.2549	0.5059	0.3755	0.038*
C8	0.1921 (6)	0.6777 (4)	0.3803 (4)	0.0355 (11)
H8	0.0641	0.6568	0.3764	0.043*
C9	0.2711 (6)	0.7988 (4)	0.3861 (4)	0.0361 (11)
C10	0.4601 (6)	0.8321 (4)	0.3943 (4)	0.0405 (12)
H10	0.5111	0.9142	0.3980	0.049*
C11	0.5735 (6)	0.7422 (4)	0.3969 (4)	0.0388 (11)
H11	0.7028	0.7652	0.4046	0.047*

C12	0.3886 (8)	0.1157 (5)	0.2361 (5)	0.0628 (16)
H12A	0.3151	0.1671	0.2748	0.075*
H12B	0.3738	0.0342	0.2505	0.075*
C13	0.3079 (9)	0.0958 (5)	0.1128 (6)	0.079 (2)
H13A	0.3906	0.0557	0.0735	0.095*
H13B	0.1820	0.0415	0.0843	0.095*
C14	0.2466 (9)	0.2216 (6)	−0.0085 (6)	0.0720 (19)
C15	0.2433 (7)	0.3529 (5)	−0.0155 (4)	0.0515 (14)
C16	0.2172 (8)	0.3767 (6)	−0.1179 (5)	0.0683 (17)
H16	0.2028	0.3100	−0.1814	0.082*
C17	0.2120 (9)	0.4968 (6)	−0.1280 (5)	0.0667 (16)
H17	0.1944	0.5119	−0.1971	0.080*
C18	0.2339 (7)	0.5940 (5)	−0.0324 (5)	0.0540 (14)
C19	0.2565 (8)	0.5734 (5)	0.0685 (5)	0.0549 (14)
H19	0.2674	0.6401	0.1313	0.066*
C20	0.2631 (8)	0.4530 (5)	0.0773 (4)	0.0559 (14)
H20	0.2812	0.4391	0.1468	0.067*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0415 (3)	0.0467 (3)	0.0999 (5)	0.0273 (2)	0.0168 (3)	0.0240 (3)
Br2	0.1142 (6)	0.0704 (5)	0.0953 (6)	0.0032 (4)	0.0313 (5)	0.0441 (4)
N1	0.032 (2)	0.049 (3)	0.056 (3)	0.0215 (19)	0.011 (2)	0.017 (2)
N2	0.036 (2)	0.037 (2)	0.064 (3)	0.0206 (18)	0.005 (2)	0.017 (2)
O1	0.0207 (17)	0.0457 (19)	0.073 (2)	0.0117 (14)	0.0064 (16)	0.0178 (17)
O2	0.069 (3)	0.035 (2)	0.067 (3)	0.0199 (17)	−0.021 (2)	0.0036 (18)
O3	0.173 (6)	0.066 (3)	0.070 (3)	0.057 (3)	−0.024 (3)	−0.023 (3)
C1	0.030 (2)	0.043 (3)	0.040 (3)	0.017 (2)	0.011 (2)	0.022 (2)
C2	0.044 (3)	0.061 (3)	0.069 (4)	0.036 (3)	0.012 (3)	0.019 (3)
C3	0.054 (3)	0.056 (3)	0.084 (4)	0.042 (3)	0.027 (3)	0.034 (3)
C4	0.024 (2)	0.044 (3)	0.039 (3)	0.0146 (19)	0.0058 (19)	0.021 (2)
C5	0.024 (2)	0.042 (3)	0.031 (2)	0.0128 (19)	0.0062 (19)	0.017 (2)
C6	0.023 (2)	0.036 (2)	0.029 (2)	0.0089 (18)	0.0058 (18)	0.0131 (19)
C7	0.024 (2)	0.037 (2)	0.035 (3)	0.0072 (18)	0.0079 (19)	0.013 (2)
C8	0.022 (2)	0.041 (3)	0.043 (3)	0.0079 (19)	0.008 (2)	0.012 (2)
C9	0.030 (2)	0.037 (2)	0.043 (3)	0.0172 (19)	0.007 (2)	0.013 (2)
C10	0.026 (2)	0.033 (2)	0.064 (3)	0.0073 (19)	0.012 (2)	0.019 (2)
C11	0.024 (2)	0.041 (3)	0.054 (3)	0.0082 (19)	0.012 (2)	0.017 (2)
C12	0.050 (3)	0.037 (3)	0.104 (5)	0.012 (2)	0.015 (3)	0.032 (3)
C13	0.060 (4)	0.032 (3)	0.110 (6)	0.008 (3)	−0.022 (4)	0.009 (3)
C14	0.063 (4)	0.056 (4)	0.063 (4)	0.028 (3)	−0.021 (3)	−0.009 (3)
C15	0.038 (3)	0.054 (3)	0.043 (3)	0.020 (2)	−0.008 (2)	−0.002 (3)
C16	0.065 (4)	0.072 (4)	0.051 (4)	0.020 (3)	0.012 (3)	−0.008 (3)
C17	0.067 (4)	0.084 (5)	0.050 (4)	0.012 (3)	0.020 (3)	0.021 (3)
C18	0.043 (3)	0.061 (3)	0.060 (4)	0.005 (3)	0.014 (3)	0.024 (3)
C19	0.062 (4)	0.050 (3)	0.044 (3)	0.015 (3)	0.007 (3)	0.007 (3)
C20	0.068 (4)	0.051 (3)	0.039 (3)	0.019 (3)	0.000 (3)	0.010 (3)

Geometric parameters (Å, °)

Br1—C9	1.908 (4)	C7—H7	0.9300
Br2—C18	1.886 (6)	C8—C9	1.378 (6)
N1—C1	1.331 (6)	C8—H8	0.9300
N1—C2	1.452 (6)	C9—C10	1.368 (6)
N1—H1	0.78 (5)	C10—C11	1.377 (6)
N2—C1	1.346 (6)	C10—H10	0.9300
N2—C12	1.447 (6)	C11—H11	0.9300
N2—C3	1.459 (6)	C12—C13	1.499 (9)
O1—C5	1.258 (5)	C12—H12A	0.9700
O2—C14	1.324 (8)	C12—H12B	0.9700
O2—C13	1.434 (6)	C13—H13A	0.9700
O3—C14	1.194 (7)	C13—H13B	0.9700
C1—C4	1.387 (6)	C14—C15	1.488 (8)
C2—C3	1.503 (8)	C15—C20	1.379 (7)
C2—H2A	0.9700	C15—C16	1.387 (8)
C2—H2B	0.9700	C16—C17	1.378 (9)
C3—H3A	0.9700	C16—H16	0.9300
C3—H3B	0.9700	C17—C18	1.381 (8)
C4—C5	1.390 (6)	C17—H17	0.9300
C4—H4	0.9300	C18—C19	1.355 (8)
C5—C6	1.503 (6)	C19—C20	1.377 (7)
C6—C11	1.385 (6)	C19—H19	0.9300
C6—C7	1.391 (5)	C20—H20	0.9300
C7—C8	1.382 (6)		
C1—N1—C2	112.6 (4)	C9—C10—C11	119.0 (4)
C1—N1—H1	119 (4)	C9—C10—H10	120.5
C2—N1—H1	125 (4)	C11—C10—H10	120.5
C1—N2—C12	125.8 (4)	C10—C11—C6	121.6 (4)
C1—N2—C3	111.5 (4)	C10—C11—H11	119.2
C12—N2—C3	122.6 (4)	C6—C11—H11	119.2
C14—O2—C13	116.7 (5)	N2—C12—C13	113.7 (5)
N1—C1—N2	109.0 (4)	N2—C12—H12A	108.8
N1—C1—C4	124.7 (4)	C13—C12—H12A	108.8
N2—C1—C4	126.3 (4)	N2—C12—H12B	108.8
N1—C2—C3	103.2 (4)	C13—C12—H12B	108.8
N1—C2—H2A	111.1	H12A—C12—H12B	107.7
C3—C2—H2A	111.1	O2—C13—C12	107.6 (4)
N1—C2—H2B	111.1	O2—C13—H13A	110.2
C3—C2—H2B	111.1	C12—C13—H13A	110.2
H2A—C2—H2B	109.1	O2—C13—H13B	110.2
N2—C3—C2	103.6 (4)	C12—C13—H13B	110.2
N2—C3—H3A	111.0	H13A—C13—H13B	108.5
C2—C3—H3A	111.0	O3—C14—O2	122.6 (6)
N2—C3—H3B	111.0	O3—C14—C15	125.3 (6)
C2—C3—H3B	111.0	O2—C14—C15	112.1 (5)
H3A—C3—H3B	109.0	C20—C15—C16	118.2 (5)

C1—C4—C5	122.9 (4)	C20—C15—C14	121.9 (5)
C1—C4—H4	118.6	C16—C15—C14	119.9 (5)
C5—C4—H4	118.6	C17—C16—C15	121.6 (5)
O1—C5—C4	124.2 (4)	C17—C16—H16	119.2
O1—C5—C6	117.2 (4)	C15—C16—H16	119.2
C4—C5—C6	118.6 (4)	C16—C17—C18	117.9 (6)
C11—C6—C7	117.7 (4)	C16—C17—H17	121.1
C11—C6—C5	119.4 (4)	C18—C17—H17	121.1
C7—C6—C5	122.9 (4)	C19—C18—C17	121.9 (5)
C8—C7—C6	121.5 (4)	C19—C18—Br2	119.6 (4)
C8—C7—H7	119.2	C17—C18—Br2	118.5 (4)
C6—C7—H7	119.2	C18—C19—C20	119.5 (5)
C9—C8—C7	118.5 (4)	C18—C19—H19	120.3
C9—C8—H8	120.7	C20—C19—H19	120.3
C7—C8—H8	120.7	C19—C20—C15	120.9 (5)
C10—C9—C8	121.6 (4)	C19—C20—H20	119.6
C10—C9—Br1	119.9 (3)	C15—C20—H20	119.6
C8—C9—Br1	118.5 (3)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1	0.78 (5)	2.16 (5)	2.706 (5)	128 (5)
N1—H1···O1^{i}	0.78 (5)	2.48 (6)	3.020 (5)	128 (5)
C3—H3A···O3^{ii}	0.97	2.43	3.296 (7)	148
C8—H8···O1^{iii}	0.93	2.40	3.264 (5)	154
C19—H19···Cg1	0.93	3.07	3.841 (6)	141

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

Fig. 1

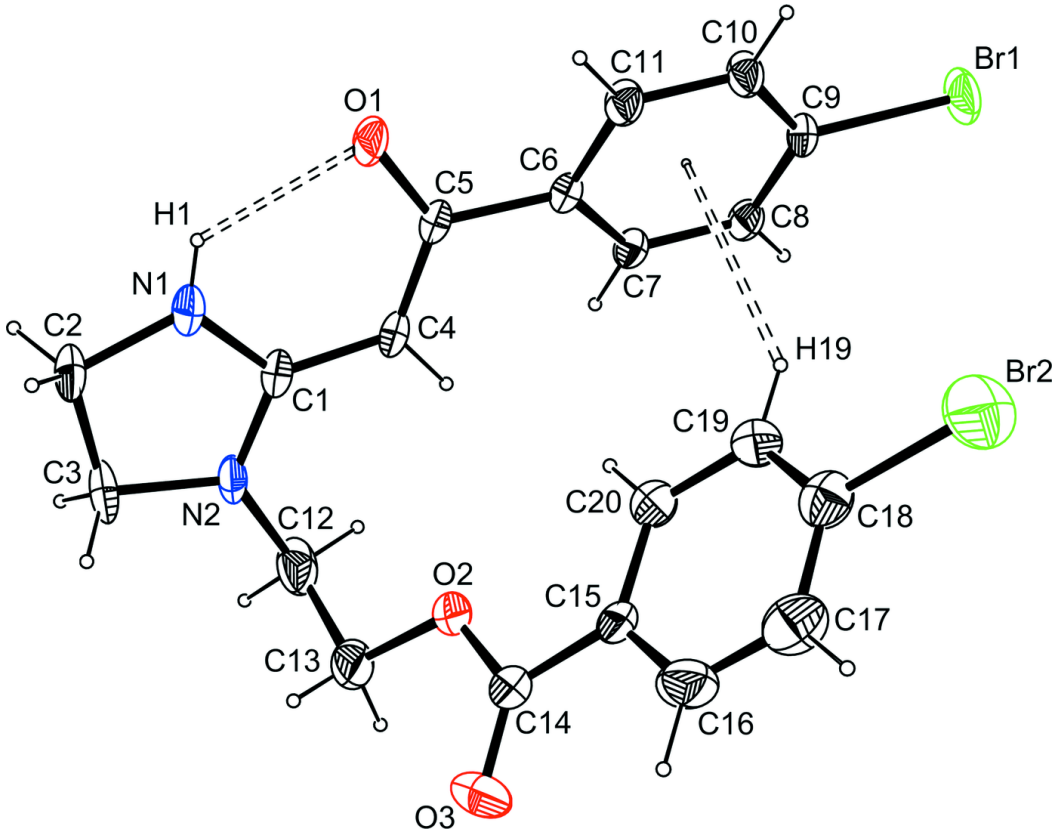
C10
C11
Br1
O1
C9
C6
C5
H1
C8
N1
C7
C2
C4
H19
Br2
C1
C19
C3
C20
C18
N2
C12
O2
C15
C17
C13
C14
C16
O3

Fig. 2