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3-Bromo-6-phenylhydrazono-β-lapachone

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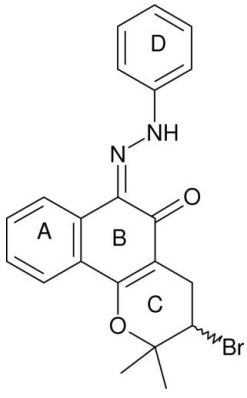
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.035; wR factor = 0.079; data-to-parameter ratio = 17.8.

The title compound, $C_{21}H_{19}BrN_2O_2$, exists in the hydrazo tautomeric form in the crystal structure, being stablilized by an intramolecular N—H···O hydrogen bond. Aromatic π–π stacking helps to establish the packing [centroid-to-centroid separation = 3.6804 (14) Å].

Related literature

For background, see: De Simone *et al.* (2002); Carvalho *et al.* (2002). For reference structural data, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_{21}H_{19}BrN_2O_2$
M_r = 411.29
Monoclinic, $P2_1/c$
a = 6.3799 (3) Å
b = 20.3789 (11) Å
c = 14.4125 (8) Å
β = 101.269 (1)°
V = 1837.72 (17) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 2.25 mm^{-1}
T = 293 (2) K
0.39 × 0.34 × 0.11 mm

Data collection

Bruker SMART1000 CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1999)
T_{min} = 0.473, T_{max} = 0.790
13653 measured reflections
4225 independent reflections
2488 reflections with $I > 2\sigma(I)$
R_{int} = 0.036

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.079
S = 0.85
4225 reflections
237 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.38 e Å^{-3}
$\Delta\rho_{min}$ = −0.46 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O2	0.86	1.88	2.562 (2)	136

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK2208).

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supplementary materials

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3-Bromo-6-phenylhydrazono-β-lapachone

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Comment

The title compound, (I), is a derivative of 3-bromo-β-lapachone (De Simone *et al.*, 2002). It was prepared as part of our on-going studies of azo dyes with possible medical applications (Carvalho *et al.*, 2002).

Compound (I) exists in its hydrazo tautomeric form in the solid-state (Fig. 1), in which the mobile H atom is attached to the N2 atom. NMR spectroscopic measurements indicate that the azo tautomer [with the H atom attached to O2 and the B (C3/C4/C8/C13–C15) ring aromatic; see Scheme] also occurs in solution. The hydrazo conformation in (I) is stabilized by an intramolecular N—H···O hydrogen bond (Table 1).

In the solid-state structure of (I), the carbon-carbon bond lengths in the B ring (see Scheme) indicate its weak aromatic character, with four of the six C—C bonds longer than 1.44 Å and one shorter than 1.36 Å. Even so, it is almost flat (r.m.s. deviation = 0.008 Å). The A ring and B ring planes are slightly twisted [dihedral angle = 3.71 (13)°]. The D ring plane makes an angle of 4.01 (13)° with the B ring mean plane. The heterocyclic C ring adopts a half-chair conformation with C2/C3/C4/O1 approximately co-planar (r.m.s. deviation = 0.012 Å) and C1 and C5 deviating from the plane by −0.341 (4) Å and 0.449 (4) Å, respectively. Such a conformation was also seen in 3-bromo-β-lapachone (De Simone *et al.*, 2002). The title molecule is chiral: in the arbitrarily chosen asymmetric molecule C1 has *R* configuration, but crystal symmetry generates a racemic mixture. Otherwise, the geometric parameters of (I) may be regarded as normal (Allen *et al.*, 1987).

In the crystal, aromatic π–π stacking involving the B and D rings helps to consolidate the packing [B···D^{i} centroid-centroid separation = 3.6804 (14) Å, i = x − 1, y, z].

Experimental

The title compound was prepared by reacting 3-bromo-β-lapachone (De Simone *et al.*, 2002) with phenylhydrazone in refluxing methanol for 24 h. Recrystallization from an ethanol solution of (I) afforded red blocks.

Refinement

The N-bound H atom was located in a difference map, relocated to an idealized position (N—H = 0.86 Å) and refined as riding with U_{iso}(H) = 1.2U_{eq}(N). The remaining hydrogen atoms were geometrically placed (C—H = 0.93–0.98 Å) and refined as riding with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C). The methyl groups were allowed to rotate, but not to tip, to best fit the electron density.

Figures

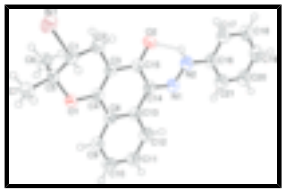


Fig. 1. View of the molecular structure of (I) showing 50% displacement ellipsoids (arbitrary spheres for the H atoms). The hydrogen bond is indicated by a double-dashed line.

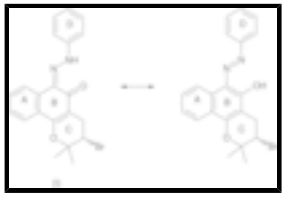


Fig. 2. The tautomeric forms of the title compound.

3-Bromo-6-phenylhydrazono-β-lapachone

Crystal data

$C_{21}H_{19}BrN_2O_2$	F_{000} = 840
M_r = 411.29	D_x = 1.487 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 8040 reflections
a = 6.3799 (3) Å	θ = 2.0–27.2º
b = 20.3789 (11) Å	μ = 2.25 mm^{-1}
c = 14.4125 (8) Å	T = 293 (2) K
β = 101.269 (1)º	Block, red
V = 1837.72 (17) Å^3	0.39 × 0.34 × 0.11 mm
Z = 4	

Data collection

Bruker SMART1000 CCD diffractometer	4225 independent reflections
Radiation source: fine-focus sealed tube	2488 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.036
T = 293(2) K	θ_{max} = 27.5º
ω scans	θ_{min} = 2.0º
Absorption correction: multi-scan (SADABS; Bruker, 1999)	h = −8→7
T_{min} = 0.473, T_{max} = 0.790	k = −26→26
13653 measured reflections	l = −18→18

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.035$

$wR(F^2) = 0.079$

$S = 0.85$

4225 reflections

237 parameters

Primary atom site location: structure-invariant direct methods

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0398P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.38$ e Å^{-3}

$\Delta\rho_{min} = -0.46$ e Å^{-3}

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 $F^2 > \sigma(F^2)$ 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}
C1	0.2158 (3)	0.41562 (10)	0.36365 (17)	0.0432 (6)
H1	0.1327	0.3875	0.3982	0.052*
C2	0.4456 (3)	0.39307 (10)	0.38700 (18)	0.0460 (6)
H2A	0.5372	0.4260	0.3670	0.055*
H2B	0.4894	0.3872	0.4548	0.055*
C3	0.4676 (3)	0.32921 (10)	0.33710 (16)	0.0390 (5)
C4	0.3083 (3)	0.30539 (10)	0.26907 (16)	0.0384 (5)
C5	0.1201 (3)	0.40928 (10)	0.25768 (17)	0.0434 (6)
C6	0.2454 (4)	0.44491 (12)	0.19404 (18)	0.0604 (7)
H6A	0.3913	0.4303	0.2076	0.091*
H6B	0.2407	0.4913	0.2051	0.091*
H6C	0.1838	0.4358	0.1291	0.091*
C7	−0.1144 (4)	0.42674 (12)	0.2355 (2)	0.0606 (7)
H7A	−0.1885	0.4021	0.2759	0.091*
H7B	−0.1727	0.4164	0.1706	0.091*
H7C	−0.1312	0.4728	0.2460	0.091*
C8	0.3172 (3)	0.24149 (10)	0.22508 (16)	0.0391 (5)
C9	0.1427 (4)	0.21550 (11)	0.16214 (17)	0.0455 (6)
H9	0.0165	0.2394	0.1474	0.055*
C10	0.1553 (4)	0.15525 (12)	0.12204 (18)	0.0544 (7)
H10	0.0373	0.1382	0.0808	0.065*
C11	0.3429 (4)	0.11943 (12)	0.14250 (18)	0.0545 (7)

H11	0.3510	0.0786	0.1146	0.065*
C12	0.5170 (4)	0.14396 (11)	0.20380 (18)	0.0506 (6)
H12	0.6431	0.1198	0.2161	0.061*
C13	0.5081 (3)	0.20479 (10)	0.24819 (16)	0.0396 (5)
C14	0.6863 (3)	0.23012 (10)	0.31862 (16)	0.0397 (5)
C15	0.6641 (3)	0.29275 (10)	0.36498 (17)	0.0411 (6)
C16	1.2036 (3)	0.17164 (11)	0.42902 (17)	0.0440 (6)
C17	1.3754 (4)	0.19699 (11)	0.49211 (18)	0.0496 (6)
H17	1.3666	0.2384	0.5182	0.060*
C18	1.5606 (4)	0.16053 (13)	0.51634 (19)	0.0584 (7)
H18	1.6767	0.1774	0.5589	0.070*
C19	1.5736 (4)	0.09970 (14)	0.4779 (2)	0.0638 (7)
H19	1.6989	0.0754	0.4939	0.077*
C20	1.4013 (4)	0.07436 (12)	0.4156 (2)	0.0646 (8)
H20	1.4106	0.0328	0.3900	0.078*
C21	1.2151 (4)	0.11001 (12)	0.39096 (19)	0.0565 (7)
H21	1.0986	0.0927	0.3492	0.068*
N1	0.8577 (3)	0.19224 (9)	0.33927 (14)	0.0430 (5)
N2	1.0202 (3)	0.21120 (9)	0.40449 (14)	0.0469 (5)
H2	1.0145	0.2482	0.4325	0.056*
O1	0.1235 (2)	0.33873 (7)	0.23736 (11)	0.0465 (4)
O2	0.8114 (2)	0.31524 (7)	0.42808 (12)	0.0537 (4)
Br1	0.19534 (4)	0.505529 (12)	0.40785 (2)	0.06343 (12)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0428 (13)	0.0301 (12)	0.0565 (16)	0.0019 (9)	0.0091 (12)	0.0015 (11)
C2	0.0436 (13)	0.0377 (13)	0.0539 (16)	0.0045 (10)	0.0025 (12)	−0.0059 (11)
C3	0.0375 (13)	0.0320 (12)	0.0459 (15)	0.0015 (9)	0.0038 (11)	−0.0001 (10)
C4	0.0351 (12)	0.0364 (12)	0.0425 (14)	0.0014 (10)	0.0044 (11)	0.0027 (11)
C5	0.0409 (13)	0.0334 (12)	0.0537 (16)	0.0054 (9)	0.0043 (12)	0.0034 (11)
C6	0.0657 (17)	0.0534 (16)	0.0636 (19)	0.0021 (12)	0.0163 (15)	0.0107 (13)
C7	0.0440 (15)	0.0535 (16)	0.078 (2)	0.0097 (12)	−0.0025 (14)	0.0031 (14)
C8	0.0421 (13)	0.0349 (12)	0.0390 (13)	−0.0025 (10)	0.0050 (11)	0.0021 (10)
C9	0.0441 (14)	0.0423 (14)	0.0457 (15)	−0.0026 (10)	−0.0015 (12)	0.0017 (11)
C10	0.0638 (17)	0.0459 (15)	0.0474 (16)	−0.0141 (12)	−0.0038 (13)	−0.0026 (12)
C11	0.0672 (17)	0.0395 (14)	0.0526 (17)	−0.0024 (12)	0.0014 (14)	−0.0086 (12)
C12	0.0541 (15)	0.0419 (14)	0.0542 (17)	0.0061 (11)	0.0069 (13)	−0.0052 (12)
C13	0.0427 (13)	0.0365 (13)	0.0388 (14)	−0.0009 (10)	0.0059 (11)	0.0011 (10)
C14	0.0385 (13)	0.0352 (12)	0.0448 (15)	0.0042 (10)	0.0067 (11)	0.0024 (11)
C15	0.0399 (13)	0.0351 (12)	0.0457 (15)	−0.0005 (10)	0.0017 (12)	0.0027 (11)
C16	0.0410 (13)	0.0420 (13)	0.0489 (16)	0.0087 (10)	0.0084 (12)	0.0078 (11)
C17	0.0468 (15)	0.0475 (15)	0.0526 (17)	0.0045 (11)	0.0044 (13)	0.0020 (12)
C18	0.0486 (16)	0.0621 (18)	0.0590 (19)	0.0054 (12)	−0.0027 (13)	0.0083 (14)
C19	0.0527 (16)	0.0649 (19)	0.071 (2)	0.0235 (14)	0.0045 (15)	0.0107 (15)
C20	0.0654 (18)	0.0479 (16)	0.075 (2)	0.0192 (13)	0.0014 (16)	0.0000 (14)
C21	0.0549 (16)	0.0465 (15)	0.0637 (18)	0.0063 (12)	0.0004 (13)	−0.0021 (13)

N1	0.0405 (11)	0.0409 (11)	0.0461 (13)	0.0033 (9)	0.0050 (10)	0.0014 (9)
N2	0.0427 (11)	0.0420 (11)	0.0534 (14)	0.0056 (9)	0.0032 (10)	−0.0027 (10)
O1	0.0378 (9)	0.0378 (9)	0.0585 (11)	0.0037 (7)	−0.0042 (8)	−0.0031 (8)
O2	0.0422 (9)	0.0459 (10)	0.0640 (12)	0.0045 (7)	−0.0121 (9)	−0.0130 (9)
Br1	0.06445 (19)	0.04274 (16)	0.0824 (2)	0.00948 (12)	0.01259 (15)	−0.01133 (14)

Geometric parameters (Å, °)

C1—C2	1.511 (3)	C10—C11	1.384 (3)
C1—C5	1.535 (3)	C10—H10	0.9300
C1—Br1	1.952 (2)	C11—C12	1.371 (3)
C1—H1	0.9800	C11—H11	0.9300
C2—C3	1.507 (3)	C12—C13	1.401 (3)
C2—H2A	0.9700	C12—H12	0.9300
C2—H2B	0.9700	C13—C14	1.462 (3)
C3—C4	1.357 (3)	C14—N1	1.324 (3)
C3—C15	1.445 (3)	C14—C15	1.460 (3)
C4—O1	1.360 (2)	C15—O2	1.260 (2)
C4—C8	1.454 (3)	C16—C21	1.378 (3)
C5—O1	1.468 (2)	C16—C17	1.380 (3)
C5—C7	1.510 (3)	C16—N2	1.408 (3)
C5—C6	1.515 (3)	C17—C18	1.382 (3)
C6—H6A	0.9600	C17—H17	0.9300
C6—H6B	0.9600	C18—C19	1.367 (3)
C6—H6C	0.9600	C18—H18	0.9300
C7—H7A	0.9600	C19—C20	1.377 (4)
C7—H7B	0.9600	C19—H19	0.9300
C7—H7C	0.9600	C20—C21	1.379 (3)
C8—C9	1.396 (3)	C20—H20	0.9300
C8—C13	1.412 (3)	C21—H21	0.9300
C9—C10	1.366 (3)	N1—N2	1.314 (2)
C9—H9	0.9300	N2—H2	0.8600
C2—C1—C5	112.08 (19)	C9—C10—C11	120.3 (2)
C2—C1—Br1	109.67 (14)	C9—C10—H10	119.9
C5—C1—Br1	111.39 (14)	C11—C10—H10	119.9
C2—C1—H1	107.8	C12—C11—C10	120.2 (2)
C5—C1—H1	107.8	C12—C11—H11	119.9
Br1—C1—H1	107.8	C10—C11—H11	119.9
C3—C2—C1	109.48 (18)	C11—C12—C13	121.1 (2)
C3—C2—H2A	109.8	C11—C12—H12	119.5
C1—C2—H2A	109.8	C13—C12—H12	119.5
C3—C2—H2B	109.8	C12—C13—C8	118.1 (2)
C1—C2—H2B	109.8	C12—C13—C14	122.3 (2)
H2A—C2—H2B	108.2	C8—C13—C14	119.62 (19)
C4—C3—C15	120.2 (2)	N1—C14—C15	123.8 (2)
C4—C3—C2	121.83 (19)	N1—C14—C13	116.5 (2)
C15—C3—C2	117.92 (19)	C15—C14—C13	119.64 (18)
C3—C4—O1	122.82 (19)	O2—C15—C3	119.9 (2)
C3—C4—C8	123.22 (19)	O2—C15—C14	121.54 (19)

O1—C4—C8	113.95 (18)	C3—C15—C14	118.6 (2)
O1—C5—C7	103.95 (17)	C21—C16—C17	120.4 (2)
O1—C5—C6	108.58 (19)	C21—C16—N2	121.9 (2)
C7—C5—C6	112.2 (2)	C17—C16—N2	117.7 (2)
O1—C5—C1	105.21 (16)	C16—C17—C18	119.7 (2)
C7—C5—C1	112.05 (19)	C16—C17—H17	120.2
C6—C5—C1	114.06 (19)	C18—C17—H17	120.2
C5—C6—H6A	109.5	C19—C18—C17	120.1 (2)
C5—C6—H6B	109.5	C19—C18—H18	119.9
H6A—C6—H6B	109.5	C17—C18—H18	119.9
C5—C6—H6C	109.5	C18—C19—C20	120.0 (2)
H6A—C6—H6C	109.5	C18—C19—H19	120.0
H6B—C6—H6C	109.5	C20—C19—H19	120.0
C5—C7—H7A	109.5	C19—C20—C21	120.6 (3)
C5—C7—H7B	109.5	C19—C20—H20	119.7
H7A—C7—H7B	109.5	C21—C20—H20	119.7
C5—C7—H7C	109.5	C16—C21—C20	119.2 (2)
H7A—C7—H7C	109.5	C16—C21—H21	120.4
H7B—C7—H7C	109.5	C20—C21—H21	120.4
C9—C8—C13	119.6 (2)	N2—N1—C14	119.41 (19)
C9—C8—C4	121.9 (2)	N1—N2—C16	120.81 (19)
C13—C8—C4	118.47 (19)	N1—N2—H2	119.6
C10—C9—C8	120.7 (2)	C16—N2—H2	119.6
C10—C9—H9	119.7	C4—O1—C5	117.88 (16)
C8—C9—H9	119.7		
C5—C1—C2—C3	−45.1 (2)	C8—C13—C14—N1	177.8 (2)
Br1—C1—C2—C3	−169.34 (16)	C12—C13—C14—C15	−177.7 (2)
C1—C2—C3—C4	11.2 (3)	C8—C13—C14—C15	0.6 (3)
C1—C2—C3—C15	−167.68 (19)	C4—C3—C15—O2	179.9 (2)
C15—C3—C4—O1	−177.3 (2)	C2—C3—C15—O2	−1.2 (3)
C2—C3—C4—O1	3.8 (3)	C4—C3—C15—C14	−0.2 (3)
C15—C3—C4—C8	4.1 (3)	C2—C3—C15—C14	178.8 (2)
C2—C3—C4—C8	−174.8 (2)	N1—C14—C15—O2	0.8 (4)
C2—C1—C5—O1	63.2 (2)	C13—C14—C15—O2	177.8 (2)
Br1—C1—C5—O1	−173.54 (13)	N1—C14—C15—C3	−179.1 (2)
C2—C1—C5—C7	175.48 (18)	C13—C14—C15—C3	−2.2 (3)
Br1—C1—C5—C7	−61.2 (2)	C21—C16—C17—C18	0.7 (4)
C2—C1—C5—C6	−55.7 (2)	N2—C16—C17—C18	−178.4 (2)
Br1—C1—C5—C6	67.6 (2)	C16—C17—C18—C19	0.1 (4)
C3—C4—C8—C9	173.5 (2)	C17—C18—C19—C20	−0.6 (4)
O1—C4—C8—C9	−5.1 (3)	C18—C19—C20—C21	0.4 (4)
C3—C4—C8—C13	−5.7 (3)	C17—C16—C21—C20	−0.9 (4)
O1—C4—C8—C13	175.69 (19)	N2—C16—C21—C20	178.1 (2)
C13—C8—C9—C10	−0.6 (3)	C19—C20—C21—C16	0.4 (4)
C4—C8—C9—C10	−179.8 (2)	C15—C14—N1—N2	−0.5 (3)
C8—C9—C10—C11	−0.8 (4)	C13—C14—N1—N2	−177.58 (19)
C9—C10—C11—C12	0.5 (4)	C14—N1—N2—C16	179.1 (2)
C10—C11—C12—C13	1.2 (4)	C21—C16—N2—N1	−4.5 (3)
C11—C12—C13—C8	−2.6 (4)	C17—C16—N2—N1	174.6 (2)

C11—C12—C13—C14	175.8 (2)	C3—C4—O1—C5	17.2 (3)
C9—C8—C13—C12	2.3 (3)	C8—C4—O1—C5	−164.19 (18)
C4—C8—C13—C12	−178.5 (2)	C7—C5—O1—C4	−166.46 (19)
C9—C8—C13—C14	−176.1 (2)	C6—C5—O1—C4	74.0 (2)
C4—C8—C13—C14	3.1 (3)	C1—C5—O1—C4	−48.5 (2)
C12—C13—C14—N1	−0.5 (3)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O2	0.86	1.88	2.562 (2)	136

Fig. 1

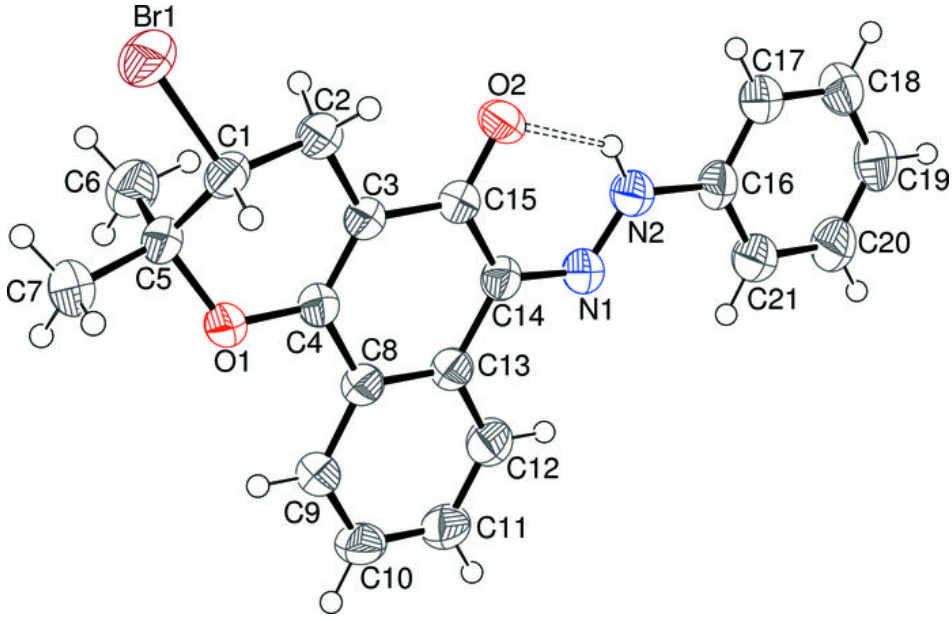
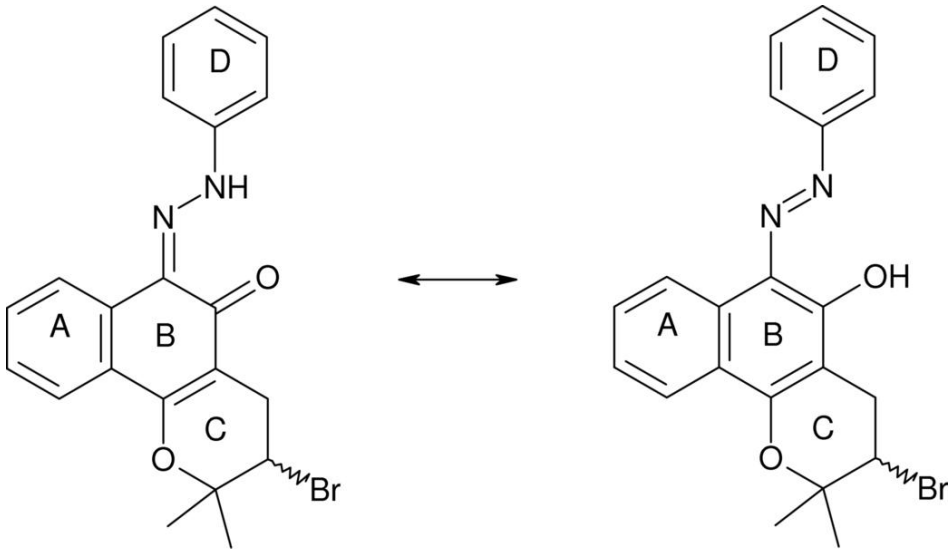

Br1
C1
C2
C3
C4
C5
C6
C7
C8
C9
C10
C11
C12
C13
C14
C15
C16
C17
C18
C19
C20
C21
O1
O2
N1
N2

Fig. 2



(I)