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2-Bromo-5-methoxy-*N*′-[(*E*)-(4-nitrophenyl)methylene]benzohydrazide

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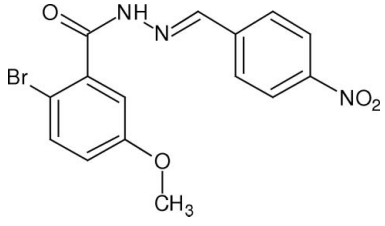
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.005 Å; R factor = 0.042; wR factor = 0.103; data-to-parameter ratio = 13.2.

The geometric parameters of the title molecule, $C_{15}H_{12}BrN_3O_4$, are in the usual ranges. The dihedral angle between the two benzene rings is 49.69 (10)°. In the crystal structure, molecules are connected by intermolecular N—H···O hydrogen bonds to form a one-dimensional chain in the *c*-axis direction.

Related literature

For related structures, see: Shan *et al.* (2004); Ali *et al.* (2004); Yathirajan *et al.* (2006). For related literature, see: Varma *et al.* (1986); Misra *et al.* (1981); Agarwal *et al.* (1983); Singh & Dash (1988); Hodnett & Dunn (1970).



Experimental

Crystal data

$C_{15}H_{12}BrN_3O_4$
M_r = 378.19
Monoclinic, $P2_1/c$
a = 7.2604 (8) Å
b = 21.8931 (19) Å
c = 9.5795 (12) Å
β = 97.882 (9)°
V = 1508.3 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 2.75 mm^{-1}
T = 173 (2) K
0.33 × 0.32 × 0.30 mm

Data collection

Stoe IPDS II two-circle diffractometer
Absorption correction: multi-scan (*MULABS*; Spek, 2003; Blessing, 1995)
T_{min} = 0.414, T_{max} = 0.436
9259 measured reflections
2818 independent reflections
2368 reflections with $I > 2\sigma(I)$
R_{int} = 0.060

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.103
S = 1.02
2818 reflections
214 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.79 e Å^{-3}
$\Delta\rho_{min}$ = −1.01 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1^{i}	0.85 (4)	2.05 (4)	2.889 (4)	171 (3)

Symmetry code: (i) $x, -y+\frac{1}{2}, z-\frac{1}{2}$.

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2458).

References

Agarwal, R., Chaudhary, K. C. & Misra, V. S. (1983). *Indian J. Chem. Sect. B*, **22**, 308–310.
Ali, H., Khamis, N. A. & Yamin, B. M. (2004). *Acta Cryst.* E**60**, o1873–o1874.
Blessing, R. H. (1995). *Acta Cryst.* A**51**, 33–38.
Hodnett, E. M. & Dunn, W. J. (1970). *J. Med. Chem.* **13**, 768–770.
Misra, V. S., Singh, S., Agarwal, R. & Chaudhary, K. C. (1981). *J. Chem. Soc. Pak.* **3**, 209–213.
Shan, S., Wang, X.-J., Hu, W.-X. & Xu, D.-J. (2004). *Acta Cryst.* E**60**, o1345–o1347.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.
Singh, W. M. & Dash, B. C. (1988). *Pesticides*, **22**, 33–37.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Stoe & Cie (2001). *X-AREA*. Stoe & Cie, Darmstadt, Germany.
Varma, R. S., Prakash, R., Khan, M. M. & Ali, A. (1986). *Indian Drugs*, **23**, 345–349.
Yathirajan, H. S., Vijaya Raj, K. K., Narayana, B., Sarojini, B. K. & Bolte, M. (2006). *Acta Cryst.* E**62**, o5013–o5014.

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supplementary materials

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2-Bromo-5-methoxy-*N*'-[(*E*)-(4-nitrophenyl)methylene]benzohydrazide

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Comment

Schiff bases are used as substrates in the preparation of number of industrial and biologically active compounds *via* ring closure, cycloaddition and replacement reactions. Some Schiff base derivatives were reported to possess antimicrobial, anti-inflammatory and central nervous system activities and moreover, Schiff bases are also known to have biological activities such as antimicrobial, antifungal, antitumor, and as herbicides (Varma *et al.*, 1986; Misra *et al.*, 1981; Agarwal *et al.*, 1983; Singh *et al.*, 1988; Hodnett *et al.*, 1970). The title compound has been synthesized and its crystal structure is reported herein.

The geometric parameters of the title molecule, $C_{15}H_{12}BrN_3O_4$, are in the usual ranges. The dihedral angle between the two benzene rings is 49.69 (10)°. In the crystal structure, molecules are connected by intermolecular N—H···O hydrogen bonds to form one-dimensional chain in the *c* axis direction.

Similar structures related to the title compound that have already been reported are 2-chloro-3,4-dimethoxybenzaldehyde (4-nitrophenyl)hydrazone (Shan *et al.*, 2004), 1-(4-fluoro-2-hydroxyphenyl)ethanone 4-nitrobenzoylhydrazone (Ali *et al.*, 2004) and 3-(2-bromo-5-methoxyphenyl)-5-methyl-1-(4-phenyl-1,3-thiazol-2-yl)- 1*H*-1,2,4-triazole (Yathirajan *et al.*, 2006).

Experimental

A mixture of 2-bromo-5-methoxybenzohydrazide (0.735 g, 0.003 mol) and 4-nitrobenzaldehyde (0.453 g, 0.003 mol) in 15 ml of absolute alcohol containing 2 drops of dilute sulfuric acid was refluxed for about 3 h. On cooling, the solid separated was filtered and recrystallized from ethyl acetate (m.p.: 462–464 K). Analysis for $C_{15}H_{12}BrN_3O_4$: Found (Calculated): C: 47.55 (47.64); H: 3.16 (3.20); N:11.06% (11.11%).

Refinement

H atoms were found in a difference map, but those bonded to C were refined using a riding model with C—H = 0.95 Å and U_{iso}(H) = 1.2U_{eq}(C) [C—H = 0.98 Å and U_{iso}(H) = 1.5U_{eq}(C) for the methyl group, which was allowed to rotate but not to tip]. The H atom bonded to N was freely refined.

Figures

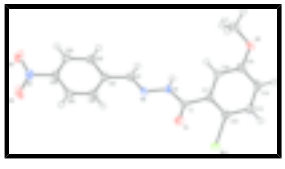

Fig. 1. The molecular structure with the atom numbering; displacement ellipsoids are at the 50% probability level.

Fig. 2. The reaction scheme

2-Bromo-5-methoxy-*N*'-[(*E*)-(4-nitrophenyl)methylene]benzohydrazide

Crystal data

$C_{15}H_{12}BrN_3O_4$	F_{000} = 760
M_r = 378.19	D_x = 1.665 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 9399 reflections
a = 7.2604 (8) Å	θ = 3.6–25.7º
b = 21.8931 (19) Å	μ = 2.75 mm^{-1}
c = 9.5795 (12) Å	T = 173 (2) K
β = 97.882 (9)º	Block, light yellow
V = 1508.3 (3) Å^3	0.33 × 0.32 × 0.30 mm
Z = 4	

Data collection

Stoe IPDS II two-circle diffractometer	2818 independent reflections
Radiation source: fine-focus sealed tube	2368 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.060
T = 173(2) K	θ_{max} = 25.7º
ω scans	θ_{min} = 3.5º
Absorption correction: multi-scan (MULABS; Spek, 2003; Blessing, 1995)	h = −8→8
T_{min} = 0.414, T_{max} = 0.436	k = −24→26
9259 measured reflections	l = −11→9

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)]$ = 0.042	$w = 1/[\sigma^2(F_o^2) + (0.0687P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.103	$(\Delta/\sigma)_{max}$ = 0.001
S = 1.02	$\Delta\rho_{max}$ = 0.79 e Å^{-3}
2818 reflections	$\Delta\rho_{min}$ = −1.01 e Å^{-3}
214 parameters	Extinction correction: SHELXL97 (Sheldrick, 1997), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.0053 (10)

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	0.32579 (4)	0.100500 (15)	0.57802 (4)	0.02733 (15)
O1	0.5025 (3)	0.22410 (11)	0.7115 (2)	0.0269 (5)
O2	1.0036 (3)	0.15890 (12)	0.3018 (3)	0.0331 (6)
O3	0.1301 (4)	0.64942 (13)	0.5891 (4)	0.0529 (8)
O4	0.0367 (4)	0.60609 (13)	0.7701 (3)	0.0408 (7)
N1	0.4881 (3)	0.28121 (12)	0.5111 (3)	0.0196 (5)
H1	0.502 (4)	0.2827 (16)	0.425 (4)	0.017 (8)*
N2	0.4182 (3)	0.33185 (12)	0.5697 (3)	0.0204 (5)
N3	0.1128 (4)	0.60476 (13)	0.6635 (3)	0.0293 (7)
C1	0.5290 (4)	0.22977 (14)	0.5886 (3)	0.0184 (6)
C2	0.3788 (4)	0.37684 (15)	0.4844 (3)	0.0219 (6)
H2	0.3959	0.3726	0.3883	0.026*
C11	0.6152 (4)	0.18073 (14)	0.5101 (3)	0.0186 (6)
C12	0.5502 (4)	0.12071 (15)	0.5056 (3)	0.0208 (6)
C13	0.6396 (4)	0.07531 (16)	0.4399 (4)	0.0251 (7)
H13	0.5944	0.0346	0.4383	0.030*
C14	0.7945 (5)	0.08914 (16)	0.3766 (4)	0.0261 (7)
H14	0.8587	0.0576	0.3350	0.031*
C15	0.8564 (4)	0.14933 (16)	0.3737 (3)	0.0232 (7)
C16	0.7690 (4)	0.19505 (15)	0.4417 (3)	0.0205 (6)
H16	0.8133	0.2359	0.4418	0.025*
C17	1.0707 (5)	0.22025 (19)	0.2950 (5)	0.0428 (10)
H17A	0.9718	0.2463	0.2471	0.064*
H17B	1.1775	0.2208	0.2426	0.064*
H17C	1.1089	0.2356	0.3907	0.064*
C21	0.3078 (4)	0.43461 (14)	0.5336 (3)	0.0197 (6)
C22	0.2838 (4)	0.48389 (15)	0.4405 (3)	0.0216 (6)
H22	0.3100	0.4789	0.3467	0.026*
C23	0.2223 (4)	0.54005 (15)	0.4830 (4)	0.0230 (7)
H23	0.2067	0.5736	0.4197	0.028*
C24	0.1842 (4)	0.54577 (14)	0.6200 (4)	0.0221 (7)
C25	0.2056 (4)	0.49770 (15)	0.7154 (3)	0.0234 (7)

H25	0.1775	0.5028	0.8087	0.028*
C26	0.2688 (4)	0.44214 (15)	0.6716 (3)	0.0221 (7)
H26	0.2858	0.4089	0.7358	0.026*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0317 (2)	0.0242 (2)	0.0281 (2)	−0.00598 (13)	0.01127 (13)	0.00084 (14)
O1	0.0430 (12)	0.0234 (12)	0.0163 (12)	0.0023 (10)	0.0114 (9)	0.0011 (9)
O2	0.0347 (12)	0.0298 (14)	0.0395 (14)	0.0040 (10)	0.0224 (10)	−0.0036 (11)
O3	0.077 (2)	0.0188 (14)	0.068 (2)	0.0059 (13)	0.0277 (17)	−0.0039 (15)
O4	0.0419 (14)	0.0404 (17)	0.0426 (16)	0.0102 (11)	0.0143 (12)	−0.0153 (13)
N1	0.0288 (12)	0.0176 (13)	0.0143 (13)	0.0045 (10)	0.0094 (10)	0.0007 (11)
N2	0.0234 (12)	0.0165 (13)	0.0224 (14)	0.0013 (10)	0.0075 (10)	−0.0029 (11)
N3	0.0263 (13)	0.0224 (16)	0.0389 (18)	0.0001 (11)	0.0031 (12)	−0.0104 (14)
C1	0.0185 (12)	0.0187 (16)	0.0184 (16)	−0.0015 (11)	0.0041 (11)	0.0015 (13)
C2	0.0221 (13)	0.0228 (17)	0.0215 (16)	0.0008 (12)	0.0058 (12)	0.0021 (13)
C11	0.0231 (13)	0.0178 (16)	0.0150 (14)	0.0038 (11)	0.0033 (11)	0.0017 (12)
C12	0.0243 (14)	0.0195 (16)	0.0187 (15)	0.0003 (12)	0.0032 (12)	0.0017 (13)
C13	0.0329 (15)	0.0158 (16)	0.0265 (18)	0.0025 (13)	0.0039 (13)	0.0013 (14)
C14	0.0320 (16)	0.0218 (17)	0.0253 (17)	0.0088 (12)	0.0065 (13)	−0.0055 (14)
C15	0.0233 (14)	0.0283 (18)	0.0186 (15)	0.0050 (12)	0.0048 (11)	0.0002 (13)
C16	0.0217 (13)	0.0180 (15)	0.0221 (16)	0.0016 (11)	0.0036 (11)	−0.0004 (13)
C17	0.0408 (19)	0.037 (2)	0.058 (3)	−0.0026 (16)	0.0311 (18)	−0.001 (2)
C21	0.0154 (12)	0.0183 (15)	0.0252 (16)	−0.0023 (11)	0.0020 (11)	−0.0018 (13)
C22	0.0254 (14)	0.0222 (17)	0.0184 (15)	0.0016 (12)	0.0069 (11)	−0.0004 (13)
C23	0.0226 (13)	0.0180 (16)	0.0280 (17)	−0.0002 (11)	0.0026 (12)	0.0016 (14)
C24	0.0171 (12)	0.0193 (16)	0.0298 (17)	−0.0011 (11)	0.0026 (12)	−0.0079 (14)
C25	0.0210 (13)	0.0267 (17)	0.0237 (17)	−0.0014 (12)	0.0073 (12)	−0.0025 (14)
C26	0.0198 (13)	0.0241 (17)	0.0225 (16)	−0.0015 (11)	0.0037 (11)	0.0013 (13)

Geometric parameters (Å, °)

Br1—C12	1.908 (3)	C13—H13	0.9500
O1—C1	1.225 (4)	C14—C15	1.394 (5)
O2—C15	1.364 (4)	C14—H14	0.9500
O2—C17	1.434 (5)	C15—C16	1.394 (4)
O3—N3	1.226 (4)	C16—H16	0.9500
O4—N3	1.227 (4)	C17—H17A	0.9800
N1—C1	1.359 (4)	C17—H17B	0.9800
N1—N2	1.371 (4)	C17—H17C	0.9800
N1—H1	0.85 (4)	C21—C22	1.395 (4)
N2—C2	1.287 (4)	C21—C26	1.400 (5)
N3—C24	1.473 (4)	C22—C23	1.388 (5)
C1—C11	1.497 (4)	C22—H22	0.9500
C2—C21	1.468 (5)	C23—C24	1.384 (5)
C2—H2	0.9500	C23—H23	0.9500
C11—C12	1.395 (4)	C24—C25	1.389 (5)
C11—C16	1.405 (4)	C25—C26	1.385 (5)

C12—C13	1.385 (5)	C25—H25	0.9500
C13—C14	1.382 (5)	C26—H26	0.9500
C15—O2—C17	117.5 (3)	C14—C15—C16	120.0 (3)
C1—N1—N2	120.8 (3)	C15—C16—C11	120.0 (3)
C1—N1—H1	121 (2)	C15—C16—H16	120.0
N2—N1—H1	118 (2)	C11—C16—H16	120.0
C2—N2—N1	114.9 (3)	O2—C17—H17A	109.5
O3—N3—O4	124.0 (3)	O2—C17—H17B	109.5
O3—N3—C24	117.8 (3)	H17A—C17—H17B	109.5
O4—N3—C24	118.2 (3)	O2—C17—H17C	109.5
O1—C1—N1	123.9 (3)	H17A—C17—H17C	109.5
O1—C1—C11	122.8 (3)	H17B—C17—H17C	109.5
N1—C1—C11	113.3 (3)	C22—C21—C26	119.3 (3)
N2—C2—C21	120.9 (3)	C22—C21—C2	118.7 (3)
N2—C2—H2	119.6	C26—C21—C2	122.0 (3)
C21—C2—H2	119.6	C23—C22—C21	121.0 (3)
C12—C11—C16	118.9 (3)	C23—C22—H22	119.5
C12—C11—C1	121.9 (3)	C21—C22—H22	119.5
C16—C11—C1	119.2 (3)	C24—C23—C22	118.2 (3)
C13—C12—C11	120.8 (3)	C24—C23—H23	120.9
C13—C12—Br1	118.4 (2)	C22—C23—H23	120.9
C11—C12—Br1	120.6 (2)	C23—C24—C25	122.5 (3)
C14—C13—C12	120.2 (3)	C23—C24—N3	118.1 (3)
C14—C13—H13	119.9	C25—C24—N3	119.4 (3)
C12—C13—H13	119.9	C26—C25—C24	118.5 (3)
C13—C14—C15	120.0 (3)	C26—C25—H25	120.7
C13—C14—H14	120.0	C24—C25—H25	120.7
C15—C14—H14	120.0	C25—C26—C21	120.5 (3)
O2—C15—C14	115.6 (3)	C25—C26—H26	119.7
O2—C15—C16	124.3 (3)	C21—C26—H26	119.7
C1—N1—N2—C2	−177.6 (3)	C14—C15—C16—C11	−1.6 (4)
N2—N1—C1—O1	2.2 (4)	C12—C11—C16—C15	−1.5 (4)
N2—N1—C1—C11	−176.2 (2)	C1—C11—C16—C15	177.0 (3)
N1—N2—C2—C21	−178.3 (2)	N2—C2—C21—C22	174.0 (3)
O1—C1—C11—C12	51.0 (4)	N2—C2—C21—C26	−4.1 (4)
N1—C1—C11—C12	−130.5 (3)	C26—C21—C22—C23	0.1 (4)
O1—C1—C11—C16	−127.4 (3)	C2—C21—C22—C23	−178.1 (3)
N1—C1—C11—C16	51.0 (3)	C21—C22—C23—C24	−0.4 (4)
C16—C11—C12—C13	2.6 (4)	C22—C23—C24—C25	0.1 (4)
C1—C11—C12—C13	−175.8 (3)	C22—C23—C24—N3	−177.9 (3)
C16—C11—C12—Br1	−172.9 (2)	O3—N3—C24—C23	−16.3 (4)
C1—C11—C12—Br1	8.6 (4)	O4—N3—C24—C23	162.5 (3)
C11—C12—C13—C14	−0.6 (5)	O3—N3—C24—C25	165.6 (3)
Br1—C12—C13—C14	175.1 (2)	O4—N3—C24—C25	−15.7 (4)
C12—C13—C14—C15	−2.6 (5)	C23—C24—C25—C26	0.5 (4)
C17—O2—C15—C14	179.4 (3)	N3—C24—C25—C26	178.5 (2)
C17—O2—C15—C16	−0.6 (5)	C24—C25—C26—C21	−0.8 (4)
C13—C14—C15—O2	−176.3 (3)	C22—C21—C26—C25	0.5 (4)

C13—C14—C15—C16	3.7 (5)	C2—C21—C26—C25	178.7 (3)
O2—C15—C16—C11	178.4 (3)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1^{i}	0.85 (4)	2.05 (4)	2.889 (4)	171 (3)

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

Fig. 1

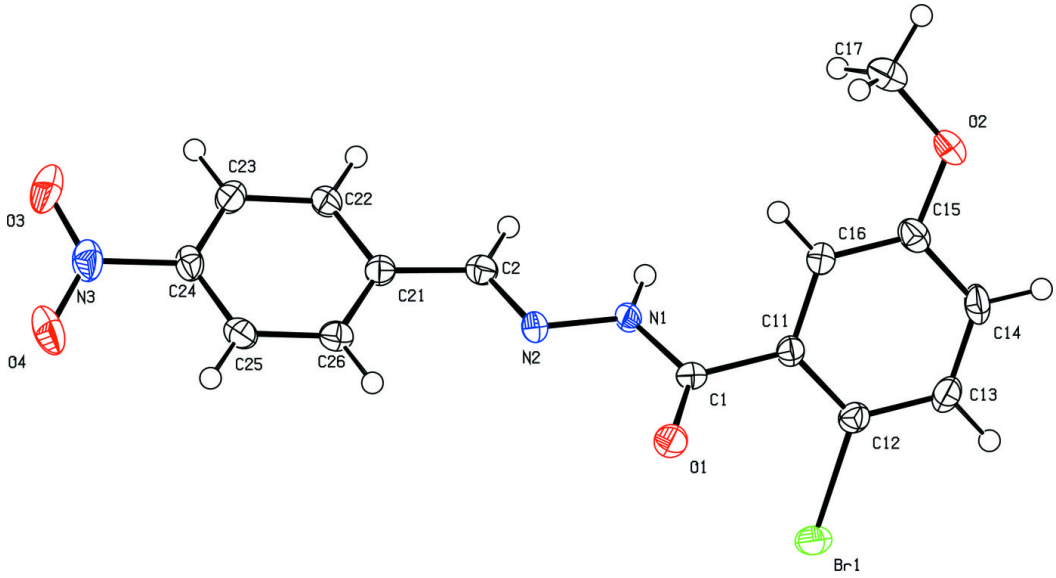
C17
O2
C15
C16
C14
C13
C12
Br1
C11
C1
O1
N1
N2
C2
C21
C22
C23
C24
C25
C26
N3
O3
O4

Fig. 2