(E)-N′-(2-Chloro-5-nitrobenzylidene)-4-methoxybenzohydrazide

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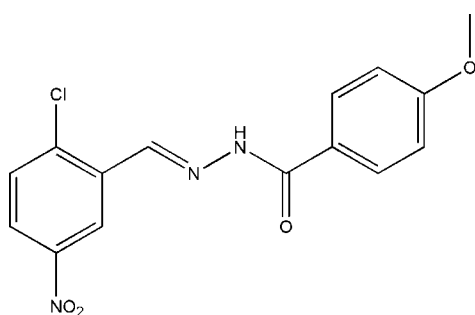Correspondence e-mail: hongyan_ban@163.com

In the title compound, $C_{15}H_{12}ClN_3O_4$, the benzohydrazide group is not planar and the molecule exists in a *trans* configuration with respect to the methylidene unit. The dihedral angle between the two substituted benzene rings is 0.4 (3)°. In the crystal structure, molecules are linked by intermolecular N—H⋯O hydrogen bonds, forming chains parallel to the *c* axis.

Related literature

For the biological activity of hydrazones, see: Zhong *et al.* (2007); Raj *et al.* (2007); Jimenez-Pulido *et al.* (2008). For related structures, see: Yehye *et al.* (2008); Fun, Patil, Jebas *et al.* (2008); Yang *et al.* (2008); Ejsmont *et al.* (2008); Fun, Patil, Rao *et al.* (2008). For reference bond lengths, see: Allen *et al.* (1987).

Experimental

Crystal data

$C_{15}H_{12}ClN_3O_4$	V = 1478.1 (5) Å^3
M_r = 333.73	Z = 4
Monoclinic, Cc	Mo $K\alpha$ radiation
a = 11.724 (2) Å	μ = 0.28 mm^{-1}
b = 13.482 (3) Å	T = 298 (2) K
c = 9.4259 (19) Å	0.20 × 0.20 × 0.17 mm
β = 97.199 (3)°	

Data collection

Bruker SMART CCD area-detector diffractometer	4284 measured reflections
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	2735 independent reflections
T_{min} = 0.946, T_{max} = 0.953	2320 reflections with $I > 2\sigma(I)$
	R_{int} = 0.015

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.035	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.089	
S = 1.04	$\Delta\rho_{max}$ = 0.14 e Å^{-3}
2735 reflections	$\Delta\rho_{min}$ = −0.19 e Å^{-3}
212 parameters	Absolute structure: Flack (1983), 1079 Friedel pairs
3 restraints	Flack parameter: −0.01 (7)

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
N2—H2⋯O1^i	0.897 (10)	2.150 (15)	2.994 (3)	156 (3)

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

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

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supplementary materials

(*E*)-*N'*-(2-Chloro-5-nitrobenzylidene)-4-methoxybenzohydrazide

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Comment

Hydrazones derived from the condensation of aldehydes with hydrazides have been demonstrated to possess excellent biological activities (Zhong *et al.*, 2007; Raj *et al.*, 2007; Jimenez-Pulido *et al.*, 2008). Due to the easy synthesis of such compounds, a great deal of hydrazones have been synthesized and structurally characterized (Yehye *et al.*, 2008; Fun, Patil, Jebas *et al.*, 2008; Yang *et al.*, 2008; Ejsmont *et al.*, 2008). We report herein the crytal structure of the title new hydrazone compound.

In the structure of the title compound (Fig. 1), the molecule exists in a *trans* configuration with respect to the methylidene unit. The dihedral angle between the two substituted benzene rings is 0.4 (3)°. In the 2-chloro-5-nitrophenyl unit, the nitro group is slightly twisted from the mean plane of the C1–C6 ring with a dihedral angle of 7.4 (3)°. The same pattern can ben observed in a similar hydrazone compound (Fun, Patil, Rao *et al.*, 2008). In the 4-methoxyphenyl unit, the methoxy group is nearly coplanar with the mean plane of the C9–C14 ring, with atom C15 deviating from the C9–C14 ring by 0.098 (2) Å. The C7-N1-N2-C8 torsion angle is 7.3 (3)°. The bond distances and angles are in normal ranges (Allen *et al.*, 1987).

In the crystal structure, molecules are linked by intermolecular N—H···O hydrogen bonds (Table 1), to form chains parallel to the *c* axis (Fig. 2).

Experimental

The compound was prepared by refluxing 2-chloro-5-nitrobenzaldehyde (1.0 mol) with 4-methoxybenzohydrazide (1.0 mol) in methanol (100 ml). Excess methanol was removed from the mixture by distillation. The colourless solid product was filtered, and washed three times with methanol. Colourless block crystals of the title compound were obtained from a methanol solution by slow evaporation in air.

Refinement

Atom H2 was located in a difference Fourier map and refined isotropically, with the N–H distance restrained to 0.90 (1) Å. Other H atoms were placed in calculated positions (C—H = 0.93-0.96 Å) and refined as riding with $U_{iso}(H) = 1.2\ U_{eq}(C)$ or $1.5U_{eq}(C)$ for methyl H atoms. A rotating group model was used for the methyl group.
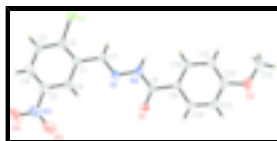
Figures

Fig. 1. The molecular structure of the title compound, showing 30% probability displacement ellipsoids for the non-hydrogen atoms.
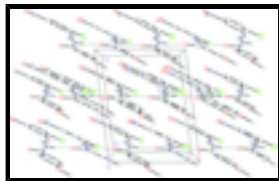
Fig. 2. Packing diagram of the title compound, viewed along the *b* axis. Hydrogen bonds are shown as dashed lines. Hydrogen atoms not involved in hydrogen bonds are omitted for clarity.

(E)-*N*'-(2-Chloro-5-nitrobenzylidene)-4-methoxybenzohydrazide

Crystal data

$C_{15}H_{12}ClN_3O_4$	$F_{000} = 688$
$M_r = 333.73$	$D_x = 1.500$ Mg m^{-3}
Monoclinic, *Cc*	Mo *Kα* radiation $\lambda = 0.71073$ Å
Hall symbol: C -2yc	Cell parameters from 1899 reflections
$a = 11.724$ (2) Å	$\theta = 2.7–26.0°$
$b = 13.482$ (3) Å	$\mu = 0.28$ mm^{-1}
$c = 9.4259$ (19) Å	$T = 298$ (2) K
$\beta = 97.199$ (3)°	Block, colourless
$V = 1478.1$ (5) Å^3	$0.20 \times 0.20 \times 0.17$ mm
$Z = 4$	

Data collection

Bruker SMART CCD area-detector diffractometer	2735 independent reflections
Radiation source: fine-focus sealed tube	2320 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.015$
$T = 298(2)$ K	$\theta_{max} = 27.5°$
ω scans	$\theta_{min} = 2.3°$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -12 \rightarrow 15$
$T_{min} = 0.946$, $T_{max} = 0.953$	$k = -17 \rightarrow 14$
4284 measured reflections	$l = -12 \rightarrow 11$

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
$R[F^2 > 2\sigma(F^2)] = 0.035$	$w = 1/[\sigma^2(F_o^2) + (0.0432P)^2 + 0.2836P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.090$	$(\Delta/\sigma)_{max} < 0.001$
$S = 1.04$	$\Delta\rho_{max} = 0.14$ e Å^{-3}
2735 reflections	$\Delta\rho_{min} = -0.19$ e Å^{-3}
212 parameters	Extinction correction: none

3 restraints

Absolute structure: Flack (1983), 1079 Friedel pairs

Primary atom site location: structure-invariant direct methods

Flack parameter: −0.01 (7)

Secondary atom site location: difference Fourier map

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 ($\mathring{A}^2$)

	x	y	z	U_{iso}*/U_{eq}
Cl1	0.12621 (9)	0.12406 (5)	0.37773 (10)	0.0861 (3)
N1	0.13488 (17)	0.41377 (14)	0.18463 (19)	0.0474 (5)
N2	0.09570 (18)	0.49328 (13)	0.2558 (2)	0.0477 (4)
N3	0.3330 (2)	0.2029 (2)	−0.1503 (3)	0.0806 (8)
O1	0.07475 (18)	0.58146 (13)	0.05053 (18)	0.0603 (5)
O2	−0.0721 (2)	0.92744 (16)	0.4294 (3)	0.0838 (7)
O3	0.3489 (3)	0.2876 (2)	−0.1823 (3)	0.1312 (12)
O4	0.3571 (2)	0.1320 (2)	−0.2230 (3)	0.1006 (8)
C1	0.1933 (2)	0.24746 (17)	0.1773 (2)	0.0466 (5)
C2	0.1870 (2)	0.15042 (18)	0.2246 (3)	0.0521 (6)
C3	0.2269 (2)	0.07102 (18)	0.1509 (3)	0.0625 (7)
H3	0.2211	0.0067	0.1848	0.075*
C4	0.2750 (2)	0.0880 (2)	0.0280 (3)	0.0626 (7)
H4	0.3019	0.0356	−0.0227	0.075*
C5	0.2829 (2)	0.1836 (2)	−0.0190 (3)	0.0574 (7)
C6	0.2439 (2)	0.26332 (17)	0.0522 (3)	0.0506 (6)
H6	0.2511	0.3273	0.0176	0.061*
C7	0.1489 (2)	0.33260 (16)	0.2508 (2)	0.0478 (5)
H7	0.1315	0.3267	0.3440	0.057*
C8	0.06846 (19)	0.57703 (16)	0.1789 (2)	0.0437 (5)
C9	0.0312 (2)	0.66442 (16)	0.2574 (2)	0.0432 (5)
C10	−0.0184 (2)	0.66089 (17)	0.3825 (2)	0.0466 (5)
H10	−0.0277	0.6001	0.4262	0.056*
C11	−0.0548 (2)	0.7478 (2)	0.4443 (3)	0.0536 (6)
H11	−0.0890	0.7449	0.5280	0.064*
C12	−0.0394 (2)	0.83779 (18)	0.3803 (3)	0.0594 (7)
C13	0.0120 (3)	0.8419 (2)	0.2577 (3)	0.0664 (7)
H13	0.0243	0.9031	0.2166	0.080*
C14	0.0454 (2)	0.75687 (18)	0.1949 (3)	0.0576 (7)

H14	0.0778	0.7607	0.1099	0.069*
C15	−0.1293 (3)	0.9299 (3)	0.5519 (5)	0.0924 (11)
H15A	−0.1978	0.8904	0.5359	0.139*
H15B	−0.1493	0.9972	0.5714	0.139*
H15C	−0.0796	0.9039	0.6320	0.139*
H2	0.085 (2)	0.490 (2)	0.3482 (13)	0.080*

Atomic displacement parameters ($Å^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.1402 (7)	0.0565 (4)	0.0680 (4)	0.0130 (5)	0.0377 (4)	0.0108 (4)
N1	0.0625 (12)	0.0416 (10)	0.0391 (10)	0.0010 (8)	0.0102 (9)	−0.0058 (8)
N2	0.0704 (12)	0.0389 (9)	0.0353 (9)	0.0033 (8)	0.0128 (9)	−0.0020 (7)
N3	0.0738 (16)	0.094 (2)	0.0798 (18)	−0.0181 (14)	0.0314 (13)	−0.0263 (16)
O1	0.0928 (13)	0.0558 (9)	0.0343 (9)	0.0018 (9)	0.0157 (8)	0.0020 (7)
O2	0.0947 (15)	0.0526 (12)	0.1063 (18)	0.0148 (10)	0.0216 (13)	−0.0121 (11)
O3	0.187 (3)	0.098 (2)	0.131 (2)	−0.042 (2)	0.109 (2)	−0.0274 (17)
O4	0.1071 (17)	0.1107 (19)	0.0928 (16)	−0.0112 (14)	0.0478 (14)	−0.0467 (14)
C1	0.0483 (12)	0.0468 (12)	0.0440 (13)	−0.0013 (10)	0.0028 (10)	−0.0074 (10)
C2	0.0607 (15)	0.0487 (12)	0.0455 (13)	0.0046 (11)	0.0015 (11)	−0.0052 (10)
C3	0.0749 (17)	0.0441 (13)	0.0663 (18)	0.0083 (12)	0.0008 (14)	−0.0078 (12)
C4	0.0590 (15)	0.0581 (15)	0.0696 (18)	0.0076 (12)	0.0029 (13)	−0.0235 (13)
C5	0.0464 (14)	0.0688 (17)	0.0574 (15)	−0.0061 (11)	0.0080 (12)	−0.0217 (13)
C6	0.0500 (13)	0.0504 (13)	0.0517 (14)	−0.0037 (12)	0.0071 (11)	−0.0082 (11)
C7	0.0631 (14)	0.0425 (12)	0.0379 (12)	−0.0009 (11)	0.0067 (10)	−0.0031 (9)
C8	0.0553 (13)	0.0426 (11)	0.0339 (11)	−0.0066 (10)	0.0085 (9)	0.0000 (9)
C9	0.0510 (13)	0.0428 (11)	0.0352 (11)	0.0004 (10)	0.0028 (10)	−0.0001 (9)
C10	0.0563 (14)	0.0431 (12)	0.0399 (12)	−0.0016 (10)	0.0046 (11)	0.0018 (10)
C11	0.0512 (13)	0.0615 (16)	0.0481 (15)	0.0030 (12)	0.0064 (12)	−0.0111 (11)
C12	0.0589 (15)	0.0406 (13)	0.0752 (19)	0.0073 (11)	−0.0051 (14)	−0.0043 (12)
C13	0.0841 (19)	0.0430 (13)	0.0721 (18)	0.0036 (13)	0.0099 (15)	0.0094 (13)
C14	0.0753 (17)	0.0485 (14)	0.0507 (14)	−0.0021 (12)	0.0144 (13)	0.0100 (11)
C15	0.077 (2)	0.081 (2)	0.121 (3)	0.0114 (16)	0.017 (2)	−0.038 (2)

Geometric parameters (Å, °)

Cl1—C2	1.725 (3)	C4—H4	0.9300
N1—C7	1.260 (3)	C5—C6	1.376 (3)
N1—N2	1.374 (2)	C6—H6	0.9300
N2—C8	1.358 (3)	C7—H7	0.9300
N2—H2	0.897 (10)	C8—C9	1.485 (3)
N3—O3	1.202 (4)	C9—C10	1.379 (3)
N3—O4	1.229 (3)	C9—C14	1.397 (3)
N3—C5	1.458 (4)	C10—C11	1.399 (3)
O1—C8	1.223 (3)	C10—H10	0.9300
O2—C12	1.366 (3)	C11—C12	1.376 (4)
O2—C15	1.406 (4)	C11—H11	0.9300
C1—C2	1.387 (3)	C12—C13	1.370 (4)
C1—C6	1.402 (3)	C13—C14	1.370 (4)

C1—C7	1.469 (3)	C13—H13	0.9300
C2—C3	1.389 (3)	C14—H14	0.9300
C3—C4	1.370 (4)	C15—H15A	0.9600
C3—H3	0.9300	C15—H15B	0.9600
C4—C5	1.369 (4)	C15—H15C	0.9600
C7—N1—N2	117.83 (18)	C1—C7—H7	120.8
C8—N2—N1	117.34 (17)	O1—C8—N2	121.9 (2)
C8—N2—H2	120.9 (19)	O1—C8—C9	120.8 (2)
N1—N2—H2	121.7 (19)	N2—C8—C9	117.29 (18)
O3—N3—O4	123.0 (3)	C10—C9—C14	118.4 (2)
O3—N3—C5	118.3 (3)	C10—C9—C8	125.4 (2)
O4—N3—C5	118.7 (3)	C14—C9—C8	116.1 (2)
C12—O2—C15	118.8 (3)	C9—C10—C11	120.7 (2)
C2—C1—C6	117.3 (2)	C9—C10—H10	119.7
C2—C1—C7	123.4 (2)	C11—C10—H10	119.7
C6—C1—C7	119.3 (2)	C12—C11—C10	119.5 (2)
C1—C2—C3	122.1 (3)	C12—C11—H11	120.3
C1—C2—Cl1	120.45 (19)	C10—C11—H11	120.3
C3—C2—Cl1	117.4 (2)	O2—C12—C13	114.9 (3)
C4—C3—C2	119.6 (3)	O2—C12—C11	125.0 (3)
C4—C3—H3	120.2	C13—C12—C11	120.1 (2)
C2—C3—H3	120.2	C14—C13—C12	120.7 (2)
C5—C4—C3	118.9 (2)	C14—C13—H13	119.7
C5—C4—H4	120.6	C12—C13—H13	119.7
C3—C4—H4	120.6	C13—C14—C9	120.6 (2)
C4—C5—C6	122.5 (3)	C13—C14—H14	119.7
C4—C5—N3	119.5 (2)	C9—C14—H14	119.7
C6—C5—N3	118.0 (3)	O2—C15—H15A	109.5
C5—C6—C1	119.6 (2)	O2—C15—H15B	109.5
C5—C6—H6	120.2	H15A—C15—H15B	109.5
C1—C6—H6	120.2	O2—C15—H15C	109.5
N1—C7—C1	118.5 (2)	H15A—C15—H15C	109.5
N1—C7—H7	120.8	H15B—C15—H15C	109.5

Hydrogen-bond geometry (Å, °)

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O1[i]	0.897 (10)	2.150 (15)	2.994 (3)	156 (3)

Symmetry codes: (i) x, $-y+1$, $z+1/2$.

Fig. 1

Fig. 2