

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

N'-(*E*)-4-Bromobenzylidene]pyrazine-2-carbohydrazone

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Received 13 June 2013; accepted 18 June 2013

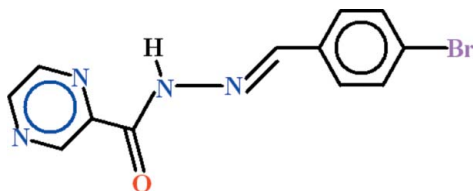
 Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.005$ Å;

 R factor = 0.039; wR factor = 0.084; data-to-parameter ratio = 15.5.

In the title compound, $\text{C}_{12}\text{H}_9\text{BrN}_4\text{O}$, the *N'*-methylidene-pyrazine-2-carbohydrazone and 4-bromobenzene groups are oriented at a dihedral angle of 10.57 (7)°. The hydrazone N—H group is involved in intramolecular N—H···N interaction, which generates an *S*(5) motif. A short C—H···O interaction is formed between the methylidene H atom and the carbonyl O atom. It connects molecules into chains extending along [100]. In addition, molecules are arranged into stacks extending along [010] via π – π interactions between pyrazine and benzene rings, with centroid–centroid distances of 3.837 (2) and 3.860 (2) Å.

Related literature

For a related crystal structure and related studies, see: Hearn & Cynamon (2004); Jin *et al.* (2006); Yuan *et al.* (2006). For graph-set notation, see: Bernstein *et al.* (1995).



Experimental

Crystal data

 $\text{C}_{12}\text{H}_9\text{BrN}_4\text{O}$
 $M_r = 305.14$

 Triclinic, $P\bar{1}$
 $a = 5.8947$ (9) Å

 $b = 7.6941$ (12) Å

 $c = 14.029$ (2) Å

 $\alpha = 83.273$ (7)°

 $\beta = 80.086$ (7)°

 $\gamma = 72.440$ (6)°

 $V = 596.11$ (16) Å³
 $Z = 2$

 Mo $K\alpha$ radiation

 $\mu = 3.44$ mm^{−1}
 $T = 296$ K

 $0.26 \times 0.22 \times 0.20$ mm

Data collection

Bruker Kappa APEXII CCD

diffractometer

Absorption correction: multi-scan

(SADABS; Bruker, 2005)

 $T_{\min} = 0.425$, $T_{\max} = 0.503$

7382 measured reflections

2529 independent reflections

 1403 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.074$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.039$
 $wR(F^2) = 0.084$
 $S = 0.94$

2529 reflections

163 parameters

H-atom parameters constrained

 $\Delta\rho_{\text{max}} = 0.28$ e Å^{−3}
 $\Delta\rho_{\text{min}} = -0.30$ e Å^{−3}
Table 1

Hydrogen-bond geometry (Å, °).

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
C6—H6···O1 ¹	0.93	2.24	3.132 (4)	161
N3—H3A···N1	0.86	2.27	2.671 (3)	108

 Symmetry code: (i) $x + 1, y, z$.

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012) and *PLATON* (Spek, 2009); software used to prepare material for publication: *WinGX* (Farrugia, 2012) and *PLATON*.

The authors acknowledge the provision of funds for the purchase of a diffractometer and encouragement by Dr Muhammad Akram Chaudhary, Vice Chancellor, University of Sargodha, Pakistan. The authors are also thankful to the Higher Education Commission (HEC) of Pakistan for financial support. MA is also thankful to the Pakistan Council of Scientific and Industrial Research (PCSIR) Laboratories of Pakistan for financial support throughout his study leave.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GK2580).

References

- Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
- Bruker (2005). *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Bruker (2007). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.
- Hearn, M. J. & Cynamon, M. H. (2004). *J. Antimicrob. Chemother.* **53**, 185–191.
- Jin, L., Chen, J., Song, B., Chen, Z., Yang, S., Li, Q., Hu, D. & Xu, R. (2006). *Bioorg. Med. Chem. Lett.* **16**, 5036–5040.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Spek, A. L. (2009). *Acta Cryst.* **D65**, 148–155.
- Yuan, C.-C., Wu, L., Xing, Z.-Y. & Shi, X.-F. (2006). *Acta Cryst.* **E62**, o3274–o3275.

supporting information

Acta Cryst. (2013). E69, o1141 [https://doi.org/10.1107/S1600536813016917]

***N'*-[*(E)*-4-Bromobenzylidene]pyrazine-2-carbohydrazide**

Shahid Hameed, Mushtaq Ahmad, M. Nawaz Tahir, Muhammad Abdullah Shah and Hazoor Ahmad Shad

S1. Comment

The title compound (Fig. 1) was prepared to study biological activities of hydrazone compounds (Hearn & Cynamon, 2004; Jin *et al.*, 2006).

Crystals of the earlier reported 4-chlorobenzaldehyde(pyrazine-2-carbonyl) hydrazone (Yuan *et al.*, 2006) are practically isostructural with the title compound.

In the title compound the *N'*-methylidenepyrazine-2-carbohydrazide (A) (C1–C6/N1–N4/O1) and 4-bromophenyl (B) (C7–C12/Br1) moieties are almost planar with r. m. s. deviations of 0.061 Å and 0.009 Å, respectively. The dihedral angle between A/B is 10.57 (7)°. There exists intramolecular N—H···N hydrogen bond (Table 1, Fig. 2) forming *S*(5) motif (Bernstein *et al.*, 1995). The intermolecular hydrogen bonds of C—H···O type (Table 1, Fig. 2) generate *C*(6) chains (Bernstein *et al.*, 1995) along the crystallographic *a*-axis. There exist π – π interactions with a distance of 3.838 (2) Å [*Cg*1—*Cg*2^{*i*} & *Cg*2—*Cg*1^{*i*}: *i* = -*x*, 2 - *y*, -*z*] and 3.860 (2) Å [*Cg*1—*Cg*2^{*ii*} & *Cg*2—*Cg*1^{*ii*}: *ii* = -*x*, 1 - *y*, -*z*], between the centroids of pyrazine (*Cg*1) and benzene (*Cg*2) rings.

S2. Experimental

The title compound was prepared by the condensation of equimolar ratio of pyrazine-2-carbohydrazide (0.50 g, 3.6 mmol) and 4-bromobenzaldehyde (0.67 g, 3.6 mmol) in methanol by the reflux of 5 h. The resulting reaction mixture was allowed to cool over night. The precipitated solid was filtered, washed with petroleum ether and recrystallized from chloroform in petroleum ether and dried under reduced pressure over CaCl₂ to give white prisms. Rf: 0.40 (30% acetone in petroleum ether): Yield: 83%, soluble in chloroform; m.p. 546–547 K.

S3. Refinement

The H atoms were positioned geometrically (N–H = 0.86 Å, C–H = 0.93 Å) and refined as riding on their carriers with $U_{\text{iso}}(\text{H}) = xU_{\text{eq}}(\text{C}, \text{N})$, where $x = 1.2$ for all H-atoms.

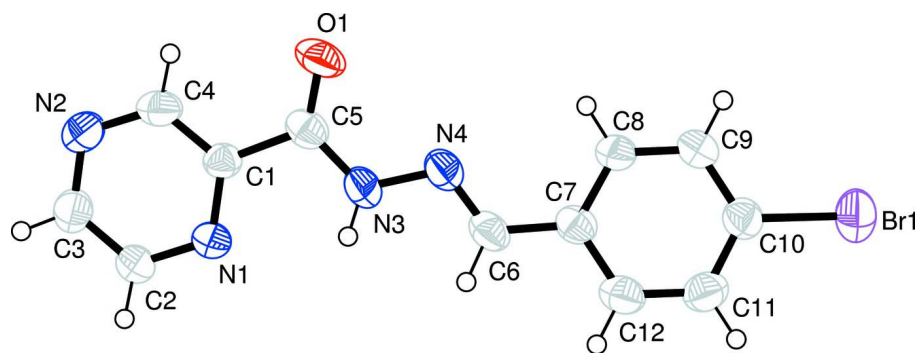


Figure 1

View of the title compound with the displacement ellipsoids drawn at the 50% probability level. H atoms are shown by small circles of arbitrary radii.

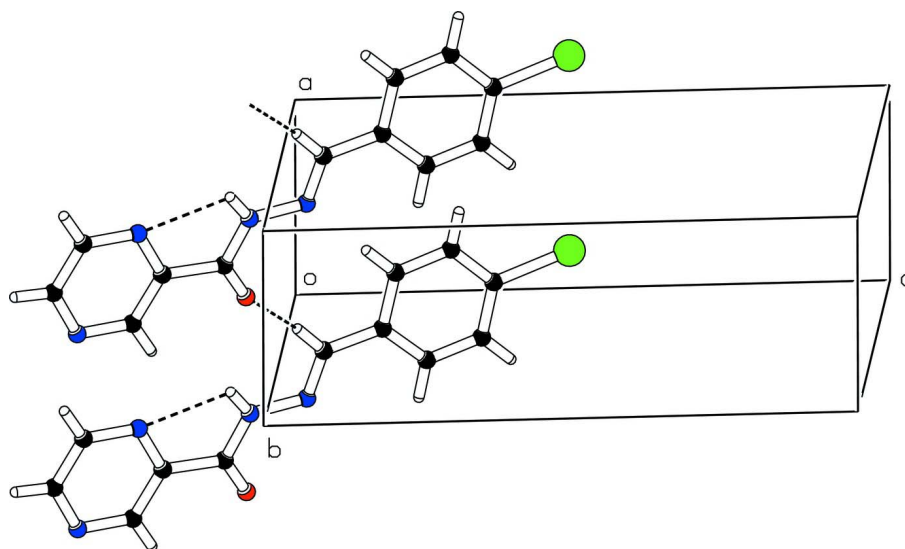


Figure 2

Packing diagram of the title compound showing $S(5)$ motif and $C(6)$ chains along $[1\ 0\ 0]$.

N'-[(*E*)-4-Bromobenzylidene]pyrazine-2-carbohydrazide

Crystal data

$C_{12}H_9BrN_4O$

$M_r = 305.14$

Triclinic, $P\bar{1}$

Hall symbol: $-P\ 1$

$a = 5.8947\ (9)\ \text{\AA}$

$b = 7.6941\ (12)\ \text{\AA}$

$c = 14.029\ (2)\ \text{\AA}$

$\alpha = 83.273\ (7)^\circ$

$\beta = 80.086\ (7)^\circ$

$\gamma = 72.440\ (6)^\circ$

$V = 596.11\ (16)\ \text{\AA}^3$

$Z = 2$

$F(000) = 304$

$D_x = 1.700\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 1403 reflections

$\theta = 2.1\text{--}25.5^\circ$

$\mu = 3.44\ \text{mm}^{-1}$

$T = 296\ \text{K}$

Prism, white

$0.26 \times 0.22 \times 0.20\ \text{mm}$

Data collection

Bruker Kappa APEXII CCD
diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 8.10 pixels mm⁻¹
 ω scans
Absorption correction: multi-scan
(*SADABS*; Bruker, 2005)
 $T_{\min} = 0.425$, $T_{\max} = 0.503$

7382 measured reflections
2529 independent reflections
1403 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.074$
 $\theta_{\max} = 27.1^\circ$, $\theta_{\min} = 1.5^\circ$
 $h = -7 \rightarrow 6$
 $k = -9 \rightarrow 9$
 $l = -17 \rightarrow 17$

Refinement

Refinement on F^2
Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.039$
 $wR(F^2) = 0.084$
 $S = 0.94$
2529 reflections
163 parameters
0 restraints
Primary atom site location: structure-invariant
direct methods

Secondary atom site location: difference Fourier
map
Hydrogen site location: inferred from
neighbouring sites
H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0355P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.28 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.30 \text{ e } \text{\AA}^{-3}$

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 $F^2 > \sigma(F^2)$ 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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Br1	0.36405 (7)	0.25644 (5)	0.47485 (3)	0.07332 (19)
O1	-0.4594 (4)	0.8146 (3)	-0.03918 (16)	0.0633 (7)
N1	-0.0274 (4)	0.9547 (3)	-0.21291 (18)	0.0486 (7)
N2	-0.4307 (5)	1.1093 (4)	-0.3071 (2)	0.0599 (8)
N3	-0.0701 (5)	0.7949 (3)	-0.03380 (18)	0.0523 (7)
H3A	0.0565	0.8198	-0.0649	0.063*
N4	-0.0613 (5)	0.7038 (3)	0.05682 (19)	0.0508 (7)
C1	-0.2458 (5)	0.9456 (4)	-0.1727 (2)	0.0401 (8)
C2	-0.0148 (6)	1.0405 (4)	-0.3008 (2)	0.0538 (9)
H2	0.1337	1.0501	-0.3321	0.065*
C3	-0.2126 (6)	1.1151 (4)	-0.3467 (2)	0.0549 (9)
H3	-0.1927	1.1724	-0.4084	0.066*
C4	-0.4426 (6)	1.0230 (4)	-0.2191 (3)	0.0540 (9)
H4	-0.5918	1.0148	-0.1877	0.065*
C5	-0.2732 (6)	0.8455 (4)	-0.0745 (2)	0.0476 (8)
C6	0.1460 (6)	0.6511 (4)	0.0827 (2)	0.0496 (9)

H6	0.2748	0.6734	0.0398	0.060*
C7	0.1898 (6)	0.5575 (4)	0.1764 (2)	0.0449 (8)
C8	0.0083 (6)	0.5360 (4)	0.2494 (2)	0.0496 (8)
H8	-0.1509	0.5833	0.2386	0.060*
C9	0.0571 (6)	0.4466 (4)	0.3372 (2)	0.0524 (9)
H9	-0.0679	0.4339	0.3853	0.063*
C10	0.2919 (6)	0.3757 (4)	0.3537 (2)	0.0500 (8)
C11	0.4775 (6)	0.3941 (4)	0.2827 (2)	0.0555 (9)
H11	0.6361	0.3455	0.2939	0.067*
C12	0.4264 (6)	0.4854 (4)	0.1945 (2)	0.0543 (9)
H12	0.5516	0.4989	0.1467	0.065*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0815 (3)	0.0777 (3)	0.0601 (3)	-0.0224 (2)	-0.0225 (2)	0.01461 (19)
O1	0.0580 (15)	0.0762 (17)	0.0622 (15)	-0.0398 (13)	0.0071 (13)	0.0006 (12)
N1	0.0420 (16)	0.0576 (17)	0.0482 (17)	-0.0216 (13)	-0.0063 (13)	0.0069 (13)
N2	0.0479 (18)	0.070 (2)	0.064 (2)	-0.0208 (15)	-0.0186 (16)	0.0107 (15)
N3	0.0564 (19)	0.0604 (18)	0.0429 (16)	-0.0271 (14)	-0.0057 (15)	0.0106 (13)
N4	0.0555 (19)	0.0552 (17)	0.0433 (17)	-0.0238 (14)	-0.0035 (14)	0.0059 (13)
C1	0.0373 (19)	0.0392 (18)	0.0455 (19)	-0.0143 (15)	-0.0058 (16)	-0.0009 (14)
C2	0.048 (2)	0.066 (2)	0.050 (2)	-0.0259 (17)	-0.0076 (18)	0.0125 (17)
C3	0.057 (2)	0.061 (2)	0.051 (2)	-0.0225 (18)	-0.0159 (19)	0.0060 (17)
C4	0.041 (2)	0.062 (2)	0.062 (2)	-0.0221 (17)	-0.0034 (18)	-0.0014 (18)
C5	0.052 (2)	0.047 (2)	0.048 (2)	-0.0234 (17)	-0.0001 (18)	-0.0048 (16)
C6	0.055 (2)	0.053 (2)	0.045 (2)	-0.0293 (17)	-0.0008 (18)	0.0027 (16)
C7	0.048 (2)	0.0429 (18)	0.048 (2)	-0.0225 (15)	-0.0039 (17)	-0.0031 (15)
C8	0.0401 (19)	0.053 (2)	0.057 (2)	-0.0192 (16)	-0.0062 (17)	0.0068 (17)
C9	0.048 (2)	0.059 (2)	0.051 (2)	-0.0221 (17)	-0.0024 (18)	0.0051 (17)
C10	0.052 (2)	0.049 (2)	0.051 (2)	-0.0199 (16)	-0.0092 (18)	0.0019 (15)
C11	0.043 (2)	0.060 (2)	0.065 (2)	-0.0157 (17)	-0.0119 (19)	-0.0025 (18)
C12	0.048 (2)	0.064 (2)	0.054 (2)	-0.0247 (17)	0.0008 (18)	-0.0048 (18)

Geometric parameters (Å, °)

Br1—C10	1.886 (3)	C3—H3	0.9300
O1—C5	1.204 (3)	C4—H4	0.9300
N1—C2	1.329 (4)	C6—C7	1.447 (4)
N1—C1	1.332 (3)	C6—H6	0.9300
N2—C3	1.322 (4)	C7—C8	1.383 (4)
N2—C4	1.332 (4)	C7—C12	1.393 (4)
N3—C5	1.346 (4)	C8—C9	1.370 (4)
N3—N4	1.379 (3)	C8—H8	0.9300
N3—H3A	0.8600	C9—C10	1.376 (4)
N4—C6	1.268 (4)	C9—H9	0.9300
C1—C4	1.370 (4)	C10—C11	1.377 (4)
C1—C5	1.506 (4)	C11—C12	1.382 (4)

C2—C3	1.368 (4)	C11—H11	0.9300
C2—H2	0.9300	C12—H12	0.9300
C2—N1—C1	115.4 (3)	N4—C6—C7	122.5 (3)
C3—N2—C4	114.6 (3)	N4—C6—H6	118.7
C5—N3—N4	121.3 (3)	C7—C6—H6	118.7
C5—N3—H3A	119.4	C8—C7—C12	117.9 (3)
N4—N3—H3A	119.4	C8—C7—C6	123.3 (3)
C6—N4—N3	114.5 (3)	C12—C7—C6	118.7 (3)
N1—C1—C4	121.5 (3)	C9—C8—C7	121.6 (3)
N1—C1—C5	118.5 (3)	C9—C8—H8	119.2
C4—C1—C5	119.9 (3)	C7—C8—H8	119.2
N1—C2—C3	122.4 (3)	C8—C9—C10	119.7 (3)
N1—C2—H2	118.8	C8—C9—H9	120.2
C3—C2—H2	118.8	C10—C9—H9	120.2
N2—C3—C2	122.8 (3)	C9—C10—C11	120.4 (3)
N2—C3—H3	118.6	C9—C10—Br1	120.6 (3)
C2—C3—H3	118.6	C11—C10—Br1	119.0 (3)
N2—C4—C1	123.2 (3)	C10—C11—C12	119.5 (3)
N2—C4—H4	118.4	C10—C11—H11	120.2
C1—C4—H4	118.4	C12—C11—H11	120.2
O1—C5—N3	125.4 (3)	C11—C12—C7	120.9 (3)
O1—C5—C1	121.7 (3)	C11—C12—H12	119.6
N3—C5—C1	112.9 (3)	C7—C12—H12	119.6
C5—N3—N4—C6	174.4 (3)	C4—C1—C5—N3	-173.2 (3)
C2—N1—C1—C4	-1.1 (4)	N3—N4—C6—C7	178.3 (3)
C2—N1—C1—C5	178.2 (3)	N4—C6—C7—C8	-7.6 (5)
C1—N1—C2—C3	0.3 (5)	N4—C6—C7—C12	172.5 (3)
C4—N2—C3—C2	-0.7 (5)	C12—C7—C8—C9	-0.1 (5)
N1—C2—C3—N2	0.7 (5)	C6—C7—C8—C9	-180.0 (3)
C3—N2—C4—C1	-0.1 (5)	C7—C8—C9—C10	-0.1 (5)
N1—C1—C4—N2	1.0 (5)	C8—C9—C10—C11	0.0 (5)
C5—C1—C4—N2	-178.2 (3)	C8—C9—C10—Br1	178.7 (2)
N4—N3—C5—O1	-1.9 (5)	C9—C10—C11—C12	0.4 (5)
N4—N3—C5—C1	179.1 (2)	Br1—C10—C11—C12	-178.3 (2)
N1—C1—C5—O1	-171.5 (3)	C10—C11—C12—C7	-0.6 (5)
C4—C1—C5—O1	7.8 (5)	C8—C7—C12—C11	0.5 (5)
N1—C1—C5—N3	7.6 (4)	C6—C7—C12—C11	-179.7 (3)

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C6—H6 \cdots O1 ⁱ	0.93	2.24	3.132 (4)	161
N3—H3A \cdots N1	0.86	2.27	2.671 (3)	108

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