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ISSN 2414-3146

N′-[(1*E*)-4-Bromobenzylidene]-5-phenyl-1*H*-pyrazole-3-carbohydrazide

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Received 8 May 2016
Accepted 20 May 2016

Edited by W. T. A. Harrison, University of Aberdeen, Scotland

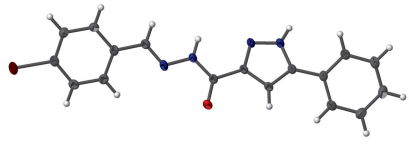
Keywords: crystal structure; pyrazole derivatives; C—H ···O hydrogen bonds.

CCDC reference: 1481143

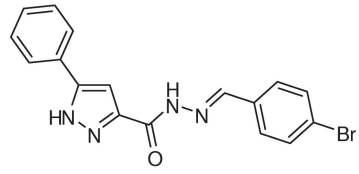
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_{17}H_{13}BrN_4O$, the dihedral angles between the pyrazole ring and the pedant phenyl and bromobenzene rings are 21.61 (11) and 28.09 (11)°, respectively. In the crystal, N—H···O hydrogen bonds link the molecules into [010] chains, which are reinforced by C—H···O interactions.

3D view



Chemical scheme



Structure description

As a continuation of our studies of pyrazole carbohydrazides (Karrouchi *et al.*, 2015), we have studied the action of 4-bromobenzaldehyde towards 5-phenyl-1*H*-pyrazole-3-carbohydrazide. This readily leads to the title compound (Fig. 1) in good yield. The molecule is distinctly twisted from end to end as indicated by the dihedral angle of 21.61 (7)° between the pendant C1–C6 phenyl ring and the pyrazole ring, and the angle of 28.10 (7)° between the latter and the C12–C17 benzene ring.

In the crystal, a N1—H1*A*···O1 hydrogen bond links the molecules into chains running parallel to [010] (Table 1 and Fig. 2) with the chains stacking along the *c* axis direction (Fig. 3). The chain linkage is reinfoced by a weak C—H···O interaction.

Synthesis and crystallization

To a solution of 5-phenyl-1*H*-pyrazole-3-carbohydrazide (1 mmol, 250 mg) in 10 ml of ethanol, was added an equimolar amount of the 4-bromobenzaldehyde in the presence of acetic acid. The mixture was maintained under reflux for 2 h, until TLC indicated the end of reaction. Then, the mixture as poured into cold water, and the precipitate formed was filtered out washed with ethanol. Single crystals of the title compound were obtained on slow evaporation of the solvent (DMF). Yield 85%; m.p. 294–296°C

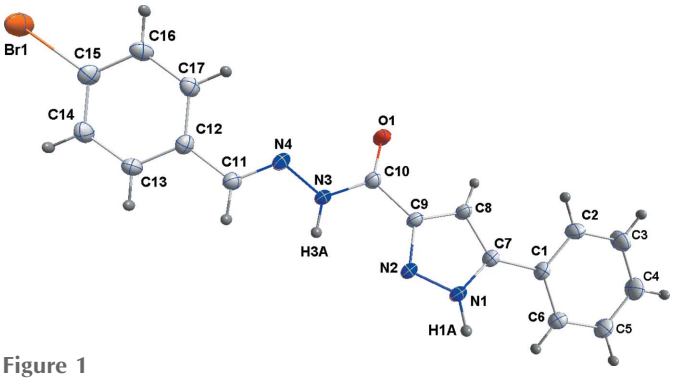


Figure 1
The title molecule with labeling scheme and 50% probability ellipsoids.

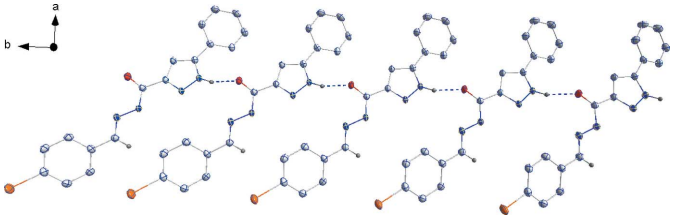


Figure 2
A portion of one chain viewed along the *c* axis with the N—H···O hydrogen bonds shown as dotted lines.

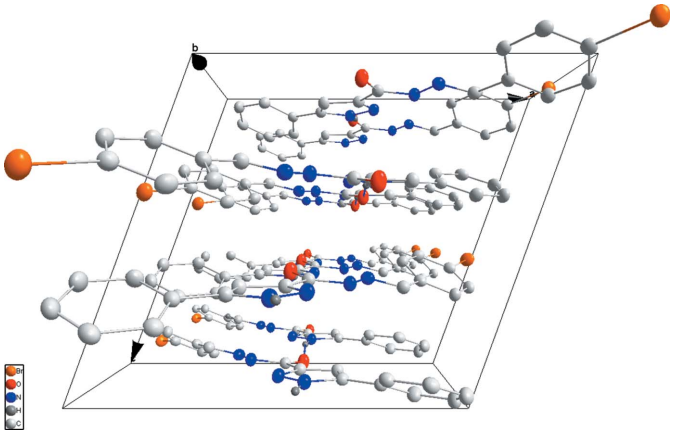


Figure 3
Packing viewed along the *b* axis showing a stack of hydrogen-bonded chains.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The methyl group is rotationally disordered over two distinct sites. The two orientations were restrained to have comparable geometries.

Acknowledgements

The support of NSF–MRI Grant No. 1228232 for the purchase of the diffractometer and Tulane University for support of the Tulane Crystallography Laboratory are gratefully acknowledged.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3*A*···N2	0.90 (3)	2.45 (3)	2.787 (2)	103 (2)
N1—H1*A*···O1^{i}	0.90 (3)	1.91 (3)	2.783 (2)	163 (3)
C6—H6···O1^{i}	0.95	2.46	3.151 (3)	130

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{17}H_{13}BrN_4O$
M_r	369.22
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	150
a, *b*, *c* (Å)	15.5151 (3), 7.1752 (1), 14.4593 (3)
β (°)	110.072 (1)
V (Å^3)	1511.90 (5)
Z	4
Radiation type	Cu $K\alpha$
μ (mm^{-1})	3.79
Crystal size (mm)	0.20 × 0.08 × 0.02
Data collection	
Diffractometer	Bruker D8 VENTURE PHOTON 100 CMOS
Absorption correction	Multi-scan (*SADABS*; Bruker, 2016)
T_{min}, T_{max}	0.74, 0.93
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	19026, 2958, 2704
R_{int}	0.040
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.617
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.030, 0.076, 1.04
No. of reflections	2958
No. of parameters	216
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.63, −0.42

Computer programs: *APEX3* and *SAINT* (Bruker, 2016), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2014* (Sheldrick, 2015*b*), *DIAMOND* (Brandenburg & Putz, 2012) and *SHELXTL* (Sheldrick, 2008).

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full crystallographic data

IUCrData (2016). **1**, x160830 [doi:10.1107/S2414314616008300]

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Crystal data

$C_{17}H_{13}BrN_4O$
M_r = 369.22
Monoclinic, $P2_1/c$
a = 15.5151 (3) Å
b = 7.1752 (1) Å
c = 14.4593 (3) Å
β = 110.072 (1)°
V = 1511.90 (5) Å^3
Z = 4
$F(000)$ = 744
D_x = 1.622 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54178 Å
Cell parameters from 9982 reflections
θ = 3.0–72.1°
μ = 3.79 mm^{-1}
T = 150 K
Plate, colourless
0.20 × 0.08 × 0.02 mm

Data collection

Bruker D8 VENTURE PHOTON 100 CMOS diffractometer
Radiation source: INCOATEC IμS micro–focus source
Mirror monochromator
Detector resolution: 10.4167 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2016)
T_{min} = 0.74, T_{max} = 0.93
19026 measured reflections
2958 independent reflections
2704 reflections with $I > 2\sigma(I)$
R_{int} = 0.040
θ_{max} = 72.1°, θ_{min} = 3.0°
h = −19→19
k = −8→8
l = −17→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.076
S = 1.04
2958 reflections
216 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0406P)^2 + 1.1336P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.63 e Å^{-3}
$\Delta\rho_{min}$ = −0.42 e Å^{-3}

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. H-atoms attached to carbon were placed in calculated positions (C—H = 0.95 - 0.98 Å) and included as riding contributions with isotropic displacement parameters 1.2 - 1.5 times those of the attached atoms. The methyl group is rotationally disordered over two distinct sites. The two orientations were restrained to have comparable geometries and included as riding contributions.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Br1	0.08242 (2)	1.44181 (3)	0.40787 (2)	0.03152 (10)
O1	0.55572 (10)	0.7240 (2)	0.39651 (12)	0.0274 (3)
N1	0.56205 (12)	0.1066 (2)	0.36792 (13)	0.0209 (3)
H1A	0.548 (2)	−0.014 (4)	0.372 (2)	0.037 (7)*
N2	0.50212 (11)	0.2396 (2)	0.37161 (12)	0.0207 (3)
N3	0.41822 (12)	0.5828 (2)	0.37237 (13)	0.0208 (3)
H3A	0.3856 (19)	0.477 (4)	0.3598 (19)	0.029 (7)*
N4	0.37729 (12)	0.7495 (2)	0.37822 (12)	0.0216 (3)
C1	0.72343 (14)	0.0664 (3)	0.36873 (14)	0.0201 (4)
C2	0.81039 (14)	0.1483 (3)	0.40431 (15)	0.0248 (4)
H2	0.8169	0.2714	0.4300	0.030*
C3	0.88713 (15)	0.0518 (3)	0.40256 (17)	0.0300 (5)
H3	0.9459	0.1090	0.4271	0.036*
C4	0.87849 (15)	−0.1286 (3)	0.36503 (16)	0.0289 (5)
H4	0.9311	−0.1948	0.3637	0.035*
C5	0.79246 (15)	−0.2107 (3)	0.32966 (15)	0.0254 (4)
H5	0.7863	−0.3337	0.3039	0.030*
C6	0.71507 (14)	−0.1153 (3)	0.33133 (15)	0.0219 (4)
H6	0.6565	−0.1733	0.3071	0.026*
C7	0.64455 (13)	0.1770 (3)	0.37096 (14)	0.0196 (4)
C8	0.63687 (14)	0.3686 (3)	0.37568 (15)	0.0208 (4)
H8	0.6821	0.4596	0.3781	0.025*
C9	0.54810 (14)	0.3990 (3)	0.37609 (15)	0.0194 (4)
C10	0.50863 (14)	0.5828 (3)	0.38258 (14)	0.0198 (4)
C11	0.28977 (14)	0.7463 (3)	0.35493 (15)	0.0218 (4)
H11	0.2566	0.6344	0.3322	0.026*
C12	0.24174 (14)	0.9165 (3)	0.36375 (14)	0.0207 (4)
C13	0.14574 (14)	0.9207 (3)	0.32523 (15)	0.0226 (4)
H13	0.1129	0.8155	0.2910	0.027*
C14	0.09828 (14)	1.0785 (3)	0.33688 (15)	0.0234 (4)
H14	0.0332	1.0824	0.3103	0.028*
C15	0.14737 (14)	1.2290 (3)	0.38761 (15)	0.0223 (4)
C16	0.24238 (14)	1.2301 (3)	0.42531 (15)	0.0234 (4)
H16	0.2746	1.3359	0.4596	0.028*
C17	0.28952 (14)	1.0742 (3)	0.41216 (15)	0.0223 (4)
H17	0.3547	1.0742	0.4361	0.027*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.03319 (15)	0.02154 (14)	0.03802 (15)	0.00873 (9)	0.00988 (10)	−0.00237 (9)
O1	0.0251 (7)	0.0134 (7)	0.0448 (9)	−0.0016 (6)	0.0134 (7)	−0.0016 (6)
N1	0.0225 (8)	0.0126 (8)	0.0281 (9)	0.0014 (6)	0.0093 (7)	0.0000 (7)
N2	0.0210 (8)	0.0128 (8)	0.0276 (9)	0.0022 (6)	0.0074 (7)	−0.0008 (6)
N3	0.0209 (8)	0.0136 (8)	0.0283 (9)	0.0006 (6)	0.0092 (7)	−0.0029 (7)
N4	0.0251 (9)	0.0160 (8)	0.0238 (8)	0.0044 (6)	0.0084 (7)	−0.0005 (6)
C1	0.0222 (10)	0.0191 (10)	0.0199 (9)	0.0035 (7)	0.0084 (8)	0.0012 (7)
C2	0.0264 (10)	0.0211 (10)	0.0264 (11)	−0.0006 (8)	0.0082 (8)	−0.0053 (8)
C3	0.0214 (10)	0.0364 (13)	0.0319 (11)	0.0007 (9)	0.0088 (9)	−0.0039 (9)
C4	0.0273 (11)	0.0324 (12)	0.0294 (11)	0.0088 (9)	0.0127 (9)	0.0007 (9)
C5	0.0320 (11)	0.0218 (10)	0.0247 (10)	0.0056 (8)	0.0128 (9)	0.0006 (8)
C6	0.0267 (10)	0.0183 (9)	0.0217 (10)	0.0008 (8)	0.0097 (8)	0.0009 (8)
C7	0.0216 (9)	0.0166 (9)	0.0208 (9)	0.0018 (7)	0.0077 (7)	0.0002 (7)
C8	0.0230 (10)	0.0136 (9)	0.0271 (10)	0.0000 (7)	0.0103 (8)	0.0011 (7)
C9	0.0227 (9)	0.0127 (9)	0.0227 (9)	0.0007 (7)	0.0076 (8)	0.0000 (7)
C10	0.0214 (9)	0.0163 (9)	0.0221 (9)	0.0012 (7)	0.0079 (8)	0.0009 (7)
C11	0.0235 (10)	0.0188 (10)	0.0234 (10)	−0.0002 (8)	0.0085 (8)	−0.0009 (8)
C12	0.0231 (10)	0.0200 (10)	0.0206 (9)	0.0032 (7)	0.0096 (8)	0.0024 (7)
C13	0.0216 (10)	0.0202 (10)	0.0264 (10)	−0.0028 (7)	0.0086 (8)	−0.0024 (8)
C14	0.0202 (9)	0.0248 (10)	0.0258 (10)	0.0020 (8)	0.0086 (8)	0.0014 (8)
C15	0.0265 (10)	0.0189 (10)	0.0224 (10)	0.0033 (8)	0.0094 (8)	0.0024 (8)
C16	0.0258 (10)	0.0197 (10)	0.0228 (10)	−0.0012 (8)	0.0059 (8)	0.0005 (8)
C17	0.0209 (9)	0.0235 (10)	0.0213 (9)	0.0012 (8)	0.0055 (8)	0.0010 (8)

Geometric parameters (Å, º)

Br1—C15	1.907 (2)	C5—C6	1.389 (3)
O1—C10	1.225 (2)	C5—H5	0.9500
N1—N2	1.346 (2)	C6—H6	0.9500
N1—C7	1.362 (3)	C7—C8	1.384 (3)
N1—H1A	0.90 (3)	C8—C9	1.397 (3)
N2—C9	1.338 (3)	C8—H8	0.9500
N3—C10	1.359 (3)	C9—C10	1.470 (3)
N3—N4	1.370 (2)	C11—C12	1.459 (3)
N3—H3A	0.90 (3)	C11—H11	0.9500
N4—C11	1.282 (3)	C12—C13	1.400 (3)
C1—C2	1.398 (3)	C12—C17	1.401 (3)
C1—C6	1.400 (3)	C13—C14	1.392 (3)
C1—C7	1.468 (3)	C13—H13	0.9500
C2—C3	1.385 (3)	C14—C15	1.379 (3)
C2—H2	0.9500	C14—H14	0.9500
C3—C4	1.392 (3)	C15—C16	1.385 (3)
C3—H3	0.9500	C16—C17	1.385 (3)
C4—C5	1.386 (3)	C16—H16	0.9500
C4—H4	0.9500	C17—H17	0.9500

N2—N1—C7	112.98 (16)	C7—C8—H8	127.5
N2—N1—H1A	119.1 (19)	C9—C8—H8	127.5
C7—N1—H1A	127.4 (19)	N2—C9—C8	112.11 (17)
C9—N2—N1	104.11 (15)	N2—C9—C10	122.89 (18)
C10—N3—N4	118.45 (16)	C8—C9—C10	125.00 (18)
C10—N3—H3A	120.8 (17)	O1—C10—N3	123.57 (18)
N4—N3—H3A	120.7 (17)	O1—C10—C9	120.95 (18)
C11—N4—N3	116.10 (17)	N3—C10—C9	115.48 (17)
C2—C1—C6	118.90 (18)	N4—C11—C12	119.27 (19)
C2—C1—C7	118.12 (18)	N4—C11—H11	120.4
C6—C1—C7	122.97 (19)	C12—C11—H11	120.4
C3—C2—C1	120.7 (2)	C13—C12—C17	119.30 (18)
C3—C2—H2	119.6	C13—C12—C11	119.27 (18)
C1—C2—H2	119.7	C17—C12—C11	121.41 (18)
C2—C3—C4	120.2 (2)	C14—C13—C12	120.29 (19)
C2—C3—H3	119.9	C14—C13—H13	119.9
C4—C3—H3	119.9	C12—C13—H13	119.9
C5—C4—C3	119.4 (2)	C15—C14—C13	118.87 (19)
C5—C4—H4	120.3	C15—C14—H14	120.6
C3—C4—H4	120.3	C13—C14—H14	120.6
C4—C5—C6	120.8 (2)	C14—C15—C16	122.17 (19)
C4—C5—H5	119.6	C14—C15—Br1	118.98 (15)
C6—C5—H5	119.6	C16—C15—Br1	118.84 (16)
C5—C6—C1	119.9 (2)	C17—C16—C15	118.86 (19)
C5—C6—H6	120.0	C17—C16—H16	120.6
C1—C6—H6	120.0	C15—C16—H16	120.6
N1—C7—C8	105.89 (17)	C16—C17—C12	120.46 (19)
N1—C7—C1	125.45 (18)	C16—C17—H17	119.8
C8—C7—C1	128.65 (19)	C12—C17—H17	119.8
C7—C8—C9	104.91 (17)		

C7—N1—N2—C9	0.6 (2)	C7—C8—C9—C10	178.65 (19)
C10—N3—N4—C11	170.82 (18)	N4—N3—C10—O1	−0.2 (3)
C6—C1—C2—C3	0.1 (3)	N4—N3—C10—C9	179.64 (17)
C7—C1—C2—C3	−178.9 (2)	N2—C9—C10—O1	172.58 (19)
C1—C2—C3—C4	0.1 (3)	C8—C9—C10—O1	−6.2 (3)
C2—C3—C4—C5	−0.1 (3)	N2—C9—C10—N3	−7.3 (3)
C3—C4—C5—C6	−0.1 (3)	C8—C9—C10—N3	173.97 (19)
C4—C5—C6—C1	0.3 (3)	N3—N4—C11—C12	177.16 (17)
C2—C1—C6—C5	−0.3 (3)	N4—C11—C12—C13	170.84 (19)
C7—C1—C6—C5	178.70 (18)	N4—C11—C12—C17	−10.8 (3)
N2—N1—C7—C8	−0.8 (2)	C17—C12—C13—C14	−1.4 (3)
N2—N1—C7—C1	179.63 (18)	C11—C12—C13—C14	176.94 (18)
C2—C1—C7—N1	−159.20 (19)	C12—C13—C14—C15	−0.6 (3)
C6—C1—C7—N1	21.8 (3)	C13—C14—C15—C16	1.6 (3)
C2—C1—C7—C8	21.3 (3)	C13—C14—C15—Br1	−177.73 (15)
C6—C1—C7—C8	−157.7 (2)	C14—C15—C16—C17	−0.5 (3)

N1—C7—C8—C9	0.6 (2)	Br1—C15—C16—C17	178.84 (15)
C1—C7—C8—C9	−179.86 (19)	C15—C16—C17—C12	−1.6 (3)
N1—N2—C9—C8	−0.2 (2)	C13—C12—C17—C16	2.6 (3)
N1—N2—C9—C10	−179.13 (18)	C11—C12—C17—C16	−175.78 (18)
C7—C8—C9—N2	−0.2 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3*A*···N2	0.90 (3)	2.45 (3)	2.787 (2)	103 (2)
N1—H1*A*···O1^{i}	0.90 (3)	1.91 (3)	2.783 (2)	163 (3)
C6—H6···O1^{i}	0.95	2.46	3.151 (3)	130

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