Acta Crystallographica Section E
**Structure Reports Online**
ISSN 1600-5368

# *N*-(4-Bromophenyl)-3,5-dinitrobenzamide

**Sohail Saeed,[a]* Naghmana Rashid,[a] Rizwan Hussain[b] and Wing-Tak Wong[c]**

[a]Chemistry Department, Research Complex, Allama Iqbal Open University, Islamabad 44000, Pakistan, [b]National Engineering & Scientific Commission, PO Box 2801, Islamabad, Pakistan, and [c]Department of Chemistry, The University of Hong Kong, Pokfulam Road, Pokfulam, Hong Kong SAR, People's Republic of China
Correspondence e-mail: sohail262001@yahoo.com

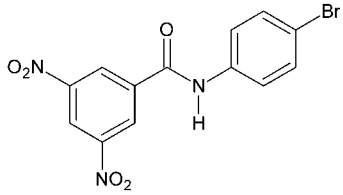
Received 28 November 2011; accepted 29 November 2011

Key indicators: single-crystal X-ray study; $T$ = 296 K; mean $\sigma$(C–C) = 0.004 Å; $R$ factor = 0.035; $wR$ factor = 0.108; data-to-parameter ratio = 12.1.

The title molecule, $C_{13}H_8BrN_3O_5$, is slightly twisted, with the dihedral angle between the two benzene rings being 5.9 (1)°. In the crystal, N—H···O hydrogen bonds link the molecules into one-dimensional chains running along [101]. Further stabilization of the crystal structure is provided by $\pi$–$\pi$ interactions [shortest centroid–centroid distance = 3.6467 (17) Å].

## Related literature

For background to the biological activity of *N*-substituted benzamides, their use in synthesis and for related structures, see: Saeed *et al.* (2011*a*,*b*).




## Experimental

### Crystal data

$C_{13}H_8BrN_3O_5$ $M_r$ = 366.13
Monoclinic, $P2_1/n$
$a$ = 7.1273 (2) Å
$b$ = 26.6676 (7) Å
$c$ = 7.5428 (2) Å
$\beta$ = 101.652 (2)°
$V$ = 1404.10 (7) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 2.96 mm$^{-1}$
$T$ = 296 K
0.56 × 0.34 × 0.30 mm

### Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min}$ = 0.288, $T_{max}$ = 0.471
18250 measured reflections
2476 independent reflections
2007 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.060

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.108
$S$ = 1.10
2476 reflections
204 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.25 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···$A$ |
|---|---|---|---|---|
| N1—H1*N*···O1$^{i}$ | 0.93 (3) | 1.93 (3) | 2.818 (3) | 159 (3) |

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

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: *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *SHELXL97*.

Dr Wesley T.-K. Chan, Professor Z.-Y. Zhou and the Hong Kong Polytechnic University are sincerely thanked for helping to collect the X-ray data.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK5029).

## References

Bruker (2007). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Macrae, C. F., Bruno, I. J., Chisholm, J. A., Edgington, P. R., McCabe, P., Pidcock, E., Rodriguez-Monge, L., Taylor, R., van de Streek, J. & Wood, P. A. (2008). *J. Appl. Cryst.* **41**, 466–470.
Saeed, S., Jasinski, J. P. & Butcher, R. J. (2011*a*). *Acta Cryst.* E**67**, o279.
Saeed, S., Rashid, N., Ng, S. W. & Tiekink, E. R. T. (2011*b*). *Acta Cryst.* E**67**, o1194.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

# supporting information

*Acta Cryst.* (2012). E**68**, o26 [doi:10.1107/S1600536811051464]

## *N*-(4-Bromophenyl)-3,5-dinitrobenzamide

**Sohail Saeed, Naghmana Rashid, Rizwan Hussain and Wing-Tak Wong**

### S1. Comment

In connection with on-going studies into *N*-substituted benzamides (Saeed *et al.*, 2011*a*,*b*), we recently determined the crystal structure of 3,5-dinitro-*N*-(1,3-thiazol-2-yl)-benzamide monohydrate (Saeed *et al.*, 2011*a*). In this paper we present the crystal structure of the title compound (I), Fig. 1.

Intermolecular N1—H1N···O1 hydrogen bonds link the molecules into 1-D chains running along [101], Table 1 and Fig. 2. The dihedral angle between the two phenyl ring planes is 5.9 (1)°. Both nitro groups are slightly twisted, 3.3 (2)° and 4.6 (2)°, respectively, from the benzene ring plane, C2—C7. There are also weak $\pi$–$\pi$ interactions between neighbouring molecules, Table 2.

### S2. Experimental

To a 250 ml round flask fitted with a condenser was added ethyl 4-bromoaniline (0.1 mol), dichloromethane (15 ml) and triethylamine(0.5 ml) with magnetic stirring. 3,5-Dinitrobenzoyl chloride (0.1 mol) was added gradually. The reaction mixture was stirred at room temperature for 1 h and then refluxed for 2 h. The product precipitated as a colourless powder, which was washed three times with water and dichloromethane. Recrystallization from ethyl acetate produced the crystals of the title compound.

### S3. Refinement

All of the C-bound H atoms are observable in a difference Fourier map but were placed at geometrical positions with C—H = 0.93 Å, and with $U_{iso}$(H) = 1.2$U_{eq}$(Carrier). The N-bound H-atoms were located from difference Fourier map and refined isotropically.

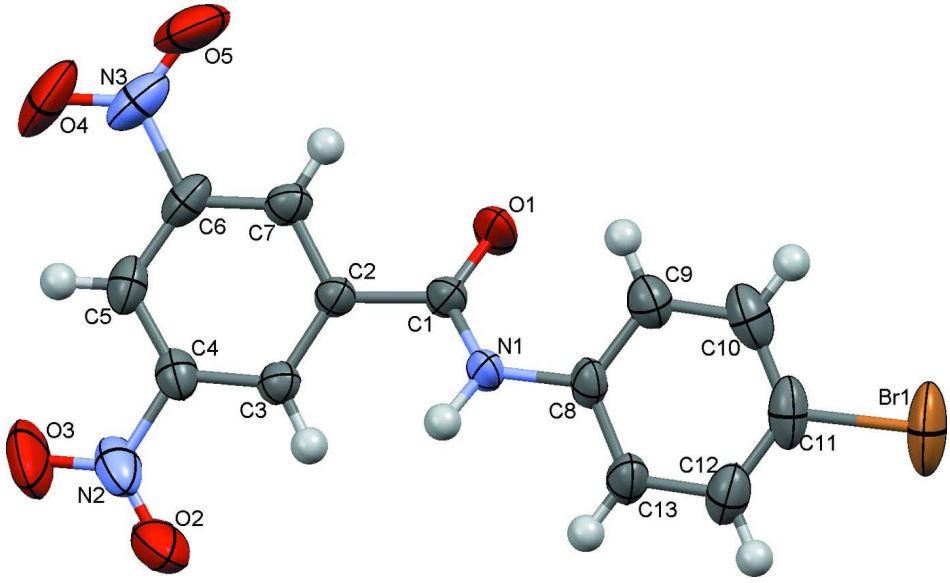



**Figure 1**

The title molecule showing at the 50% probability displacement ellipsoids and the atom numbering scheme.

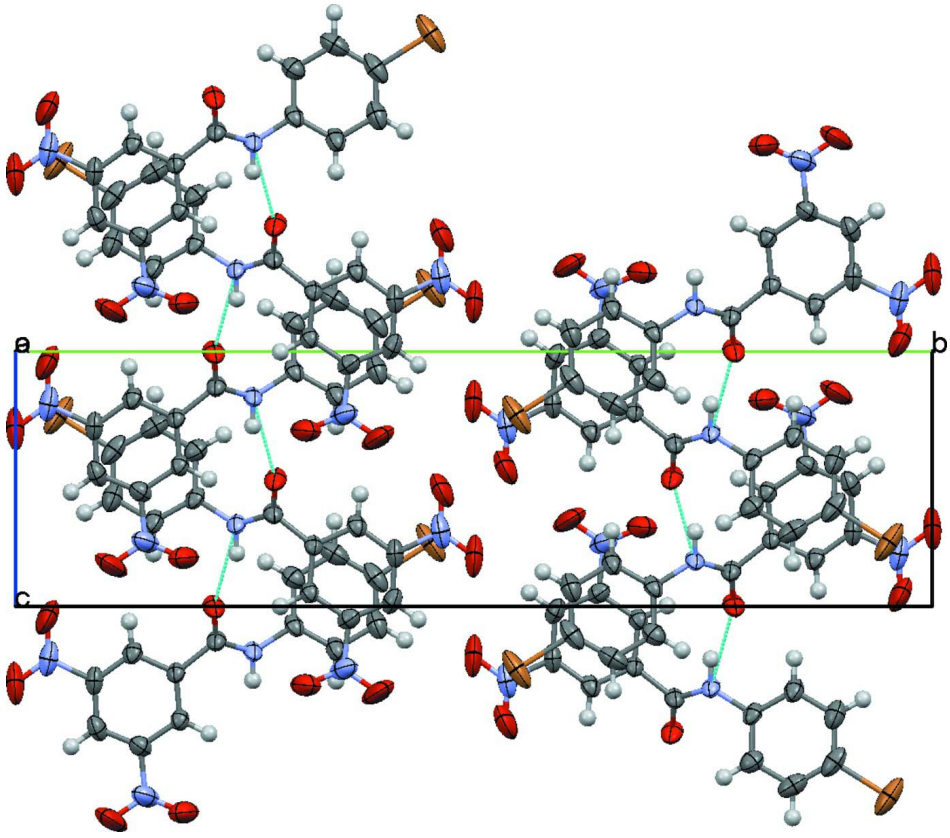



**Figure 2**

The packing diagram projected down the *a* axis of the compound showing 50% probability displacement ellipsoids. The cyan dotted lines indicate N—H···O hydrogen bonding interactions.

## *N*-(4-Bromophenyl)-3,5-dinitrobenzamide

*Crystal data*

$C_{13}H_8BrN_3O_5$
$M_r = 366.13$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 7.1273$ (2) Å
$b = 26.6676$ (7) Å
$c = 7.5428$ (2) Å
$\beta = 101.652$ (2)°
$V = 1404.10$ (7) Å$^3$
$Z = 4$
$F(000) = 728$
$D_x = 1.732$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 18250 reflections
$\theta = 2.9$–$25.0°$
$\mu = 2.96$ mm$^{-1}$
$T = 296$ K
Block, colourless
0.56 × 0.34 × 0.30 mm

*Data collection*

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega$ and $\varphi$ scan
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.288$, $T_{max} = 0.471$
18250 measured reflections
2476 independent reflections
2007 reflections with $I > 2\sigma(I)$
$R_{int} = 0.060$
$\theta_{max} = 25.0°$, $\theta_{min} = 2.9°$
$h = -8 \rightarrow 8$
$k = -31 \rightarrow 31$
$l = -8 \rightarrow 8$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.035$
$wR(F^2) = 0.108$
$S = 1.10$
2476 reflections
204 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 atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0472P)^2 + 0.9282P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.25$ e Å$^{-3}$
$\Delta\rho_{min} = -0.46$ e Å$^{-3}$
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0074 (12)

*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* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| Br1 | 0.80856 (6) | 0.046874 (16) | 0.25109 (7) | 0.0884 (2) |
| O1 | 0.6333 (3) | 0.28369 (8) | 0.5032 (3) | 0.0569 (6) |
| O2 | 0.9867 (5) | 0.31665 (12) | 1.3107 (3) | 0.0879 (9) |

| | | | | |
|---|---|---|---|---|
| O3 | 0.9761 (6) | 0.39563 (13) | 1.3387 (4) | 0.1162 (14) |
| O4 | 0.8205 (5) | 0.50043 (9) | 0.8040 (5) | 0.0986 (10) |
| O5 | 0.7279 (6) | 0.46522 (10) | 0.5447 (5) | 0.0967 (11) |
| N1 | 0.8777 (3) | 0.24105 (8) | 0.6787 (3) | 0.0395 (6) |
| N2 | 0.9563 (5) | 0.35817 (13) | 1.2475 (4) | 0.0651 (9) |
| N3 | 0.7853 (5) | 0.46389 (11) | 0.7070 (6) | 0.0655 (9) |
| C1 | 0.7638 (4) | 0.28096 (10) | 0.6368 (4) | 0.0393 (6) |
| C2 | 0.8027 (3) | 0.32503 (10) | 0.7622 (4) | 0.0341 (6) |
| C3 | 0.8632 (4) | 0.32045 (10) | 0.9473 (4) | 0.0362 (6) |
| H3 | 0.8834 | 0.2890 | 1.0013 | 0.043* |
| C4 | 0.8932 (4) | 0.36369 (11) | 1.0508 (4) | 0.0430 (7) |
| C5 | 0.8704 (4) | 0.41094 (11) | 0.9800 (4) | 0.0487 (8) |
| H5 | 0.8934 | 0.4394 | 1.0524 | 0.058* |
| C6 | 0.8110 (4) | 0.41394 (10) | 0.7935 (5) | 0.0456 (7) |
| C7 | 0.7736 (4) | 0.37237 (10) | 0.6849 (4) | 0.0405 (6) |
| H7 | 0.7293 | 0.3759 | 0.5608 | 0.049* |
| C8 | 0.8647 (4) | 0.19583 (10) | 0.5760 (4) | 0.0392 (6) |
| C9 | 0.8261 (4) | 0.19669 (12) | 0.3885 (4) | 0.0488 (7) |
| H9 | 0.8107 | 0.2271 | 0.3268 | 0.059* |
| C10 | 0.8108 (5) | 0.15204 (15) | 0.2943 (5) | 0.0580 (9) |
| H10 | 0.7828 | 0.1522 | 0.1685 | 0.070* |
| C11 | 0.8370 (4) | 0.10718 (13) | 0.3874 (5) | 0.0576 (9) |
| C12 | 0.8826 (5) | 0.10586 (12) | 0.5729 (5) | 0.0561 (8) |
| H12 | 0.9045 | 0.0754 | 0.6338 | 0.067* |
| C13 | 0.8955 (4) | 0.15065 (11) | 0.6683 (4) | 0.0481 (8) |
| H13 | 0.9247 | 0.1504 | 0.7942 | 0.058* |
| H1N | 0.958 (4) | 0.2412 (10) | 0.792 (4) | 0.035 (7)* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Br1 | 0.0662 (3) | 0.0737 (3) | 0.1252 (5) | −0.01117 (19) | 0.0187 (3) | −0.0669 (3) |
| O1 | 0.0609 (13) | 0.0435 (11) | 0.0499 (13) | 0.0067 (10) | −0.0278 (11) | −0.0061 (10) |
| O2 | 0.134 (3) | 0.084 (2) | 0.0409 (14) | −0.0258 (18) | 0.0057 (15) | 0.0037 (14) |
| O3 | 0.205 (4) | 0.095 (2) | 0.0539 (16) | −0.049 (2) | 0.038 (2) | −0.0396 (17) |
| O4 | 0.105 (2) | 0.0269 (13) | 0.161 (3) | −0.0038 (13) | 0.020 (2) | −0.0076 (16) |
| O5 | 0.133 (3) | 0.0518 (16) | 0.104 (3) | 0.0173 (17) | 0.022 (2) | 0.0374 (16) |
| N1 | 0.0429 (13) | 0.0309 (12) | 0.0361 (13) | 0.0010 (10) | −0.0123 (11) | −0.0037 (10) |
| N2 | 0.089 (2) | 0.070 (2) | 0.0407 (16) | −0.0319 (17) | 0.0224 (15) | −0.0123 (15) |
| N3 | 0.0619 (19) | 0.0326 (15) | 0.104 (3) | 0.0066 (13) | 0.0229 (18) | 0.0138 (16) |
| C1 | 0.0383 (15) | 0.0335 (14) | 0.0394 (15) | −0.0015 (11) | −0.0079 (12) | 0.0023 (12) |
| C2 | 0.0309 (14) | 0.0294 (13) | 0.0381 (15) | 0.0005 (10) | −0.0024 (11) | 0.0013 (11) |
| C3 | 0.0344 (14) | 0.0323 (13) | 0.0394 (15) | −0.0027 (11) | 0.0016 (11) | 0.0020 (11) |
| C4 | 0.0448 (16) | 0.0465 (16) | 0.0389 (16) | −0.0090 (13) | 0.0116 (13) | −0.0087 (13) |
| C5 | 0.0530 (18) | 0.0356 (15) | 0.060 (2) | −0.0102 (13) | 0.0159 (15) | −0.0130 (14) |
| C6 | 0.0410 (16) | 0.0261 (14) | 0.071 (2) | 0.0006 (11) | 0.0144 (15) | 0.0024 (13) |
| C7 | 0.0370 (15) | 0.0359 (15) | 0.0447 (16) | 0.0038 (12) | −0.0012 (12) | 0.0058 (13) |
| C8 | 0.0331 (14) | 0.0350 (14) | 0.0440 (16) | −0.0016 (11) | −0.0051 (11) | −0.0108 (12) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C9 | 0.0471 (17) | 0.0522 (18) | 0.0446 (18) | −0.0024 (14) | 0.0035 (13) | −0.0088 (14) |
| C10 | 0.0495 (19) | 0.075 (2) | 0.0485 (19) | −0.0023 (16) | 0.0085 (15) | −0.0248 (18) |
| C11 | 0.0373 (17) | 0.057 (2) | 0.077 (2) | −0.0065 (14) | 0.0087 (16) | −0.0346 (18) |
| C12 | 0.0495 (19) | 0.0402 (16) | 0.075 (2) | −0.0007 (14) | 0.0032 (16) | −0.0147 (16) |
| C13 | 0.0492 (17) | 0.0358 (15) | 0.0517 (18) | 0.0011 (13) | −0.0077 (14) | −0.0076 (13) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| Br1—C11 | 1.898 (3) | C4—C5 | 1.366 (4) |
| O1—C1 | 1.228 (3) | C5—C6 | 1.387 (5) |
| O2—N2 | 1.208 (4) | C5—H5 | 0.9300 |
| O3—N2 | 1.205 (4) | C6—C7 | 1.372 (4) |
| O4—N3 | 1.214 (4) | C7—H7 | 0.9300 |
| O5—N3 | 1.211 (5) | C8—C9 | 1.385 (4) |
| N1—C1 | 1.337 (4) | C8—C13 | 1.387 (4) |
| N1—C8 | 1.426 (3) | C9—C10 | 1.380 (5) |
| N1—H1N | 0.93 (3) | C9—H9 | 0.9300 |
| N2—C4 | 1.468 (4) | C10—C11 | 1.381 (5) |
| N3—C6 | 1.478 (4) | C10—H10 | 0.9300 |
| C1—C2 | 1.499 (4) | C11—C12 | 1.371 (5) |
| C2—C3 | 1.381 (4) | C12—C13 | 1.388 (4) |
| C2—C7 | 1.388 (4) | C12—H12 | 0.9300 |
| C3—C4 | 1.385 (4) | C13—H13 | 0.9300 |
| C3—H3 | 0.9300 | | |
| | | | |
| C1—N1—C8 | 125.1 (2) | C7—C6—C5 | 122.8 (3) |
| C1—N1—H1N | 116.5 (17) | C7—C6—N3 | 118.2 (3) |
| C8—N1—H1N | 117.7 (17) | C5—C6—N3 | 119.0 (3) |
| O3—N2—O2 | 123.0 (3) | C6—C7—C2 | 119.3 (3) |
| O3—N2—C4 | 118.1 (3) | C6—C7—H7 | 120.3 |
| O2—N2—C4 | 119.0 (3) | C2—C7—H7 | 120.3 |
| O5—N3—O4 | 124.9 (3) | C9—C8—C13 | 120.4 (3) |
| O5—N3—C6 | 117.3 (3) | C9—C8—N1 | 121.2 (3) |
| O4—N3—C6 | 117.7 (4) | C13—C8—N1 | 118.4 (3) |
| O1—C1—N1 | 124.4 (3) | C10—C9—C8 | 119.4 (3) |
| O1—C1—C2 | 118.9 (2) | C10—C9—H9 | 120.3 |
| N1—C1—C2 | 116.7 (2) | C8—C9—H9 | 120.3 |
| C3—C2—C7 | 119.6 (2) | C9—C10—C11 | 119.8 (3) |
| C3—C2—C1 | 123.3 (2) | C9—C10—H10 | 120.1 |
| C7—C2—C1 | 117.1 (2) | C11—C10—H10 | 120.1 |
| C2—C3—C4 | 118.5 (2) | C12—C11—C10 | 121.4 (3) |
| C2—C3—H3 | 120.7 | C12—C11—Br1 | 120.6 (3) |
| C4—C3—H3 | 120.7 | C10—C11—Br1 | 118.0 (3) |
| C5—C4—C3 | 123.7 (3) | C11—C12—C13 | 118.9 (3) |
| C5—C4—N2 | 118.4 (3) | C11—C12—H12 | 120.5 |
| C3—C4—N2 | 117.8 (3) | C13—C12—H12 | 120.5 |
| C4—C5—C6 | 116.0 (3) | C8—C13—C12 | 120.0 (3) |
| C4—C5—H5 | 122.0 | C8—C13—H13 | 120.0 |

| | | | |
|---|---|---|---|
| C6—C5—H5 | 122.0 | C12—C13—H13 | 120.0 |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H1*N*···O1$^{i}$ | 0.93 (3) | 1.93 (3) | 2.818 (3) | 159 (3) |

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