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## Structure Reports

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

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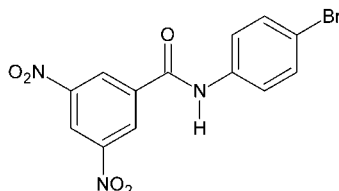
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Key indicators: single-crystal X-ray study;  $T = 296$  K; mean  $\sigma(\text{C}-\text{C}) = 0.004$  Å;  $R$  factor = 0.035;  $wR$  factor = 0.108; data-to-parameter ratio = 12.1.

The title molecule,  $\text{C}_{13}\text{H}_8\text{BrN}_3\text{O}_5$ , is slightly twisted, with the dihedral angle between the two benzene rings being  $5.9$  ( $1$ )°. In the crystal,  $\text{N}-\text{H}\cdots\text{O}$  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.* (2011a,b).



### Experimental

#### Crystal data

$\text{C}_{13}\text{H}_8\text{BrN}_3\text{O}_5$

$M_r = 366.13$

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<sup>3</sup>

$Z = 4$   
Mo  $K\alpha$  radiation  
 $\mu = 2.96$  mm<sup>-1</sup>  
 $T = 296$  K  
 $0.56 \times 0.34 \times 0.30$  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  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.060$

#### 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  
 $\Delta\rho_{\text{max}} = 0.25$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.46$  e Å<sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N1}-\text{H1N}\cdots\text{O1}^i$	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

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**supplementary materials**

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

S. Saeed, N. Rashid, R. Hussain and W.-T. Wong

### 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.

### 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.

### 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  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{Carrier})$ . The N-bound H-atoms were located from difference Fourier map and refined isotropically.

### Figures

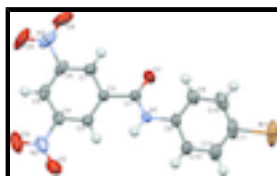


Fig. 1. The title molecule showing at the 50% probability displacement ellipsoids and the atom numbering scheme.

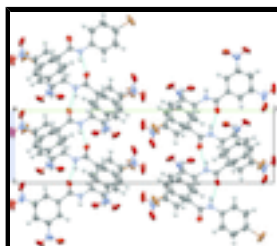


Fig. 2. The packing diagram projected down the *a* axis of the compound showing 50% probability displacement ellipsoids. The cyan dotted lines indicate N—H···O hydrogen bonding interactions.

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

### *Crystal data*

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

### *Data collection*

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

### *Refinement*

Refinement on $F^2$	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.035$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.108$	$w = 1/[\sigma^2(F_o^2) + (0.0472P)^2 + 0.9282P]$
$S = 1.10$	where $P = (F_o^2 + 2F_c^2)/3$
2476 reflections	$(\Delta/\sigma)_{\text{max}} < 0.001$
204 parameters	$\Delta\rho_{\text{max}} = 0.25 \text{ e \AA}^{-3}$
0 restraints	$\Delta\rho_{\text{min}} = -0.46 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	Extinction correction: <i>SHELXL97</i> (Sheldrick, 2008), $F_c^* = kFc[1 + 0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
	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 ( $\text{\AA}^2$ )*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
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)*

## supplementary materials

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### Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
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 ( $\text{\AA}$ , $^\circ$ )

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 ( $\text{\AA}$ ,  $^\circ$ )

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1N $\cdots$ O1 <sup>i</sup>	0.93 (3)	1.93 (3)	2.818 (3)	159 (3)

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

**Table 2**

**Table 2.  $\pi$ - $\pi$  interactions ( $\text{\AA}$ ,  $^\circ$ )**

$CgI$  and  $CgJ$  are centroids of the rings  $C2-C7$  and  $C8-C13$  respectively,  $CgI\cdots CgJ$  is the distance between ring centroids. The dihedral angle is that between the planes of the rings  $I$  and  $J$ .  $CgI\_Perp$  is the perpendicular distance of  $CgI$  from ring  $J$ .  $CgJ\_Perp$  is the perpendicular distance of  $CgJ$  from ring  $I$ .

$I$	$J$	$CgI\cdots CgJ$	Dihedral angle	$CgI\_Perp$	$CgJ\_Perp$
1	2 <sup>i</sup>	3.7391 (17)	6.24 (14)	3.5945 (11)	-3.6827 (13)
1	2 <sup>ii</sup>	3.6467 (17)	6.24 (14)	-3.5254 (12)	3.4038 (13)

symmetry operators: <sup>i</sup>:  $-1/2+x, 1/2-y, 1/2+z$  <sup>ii</sup>:  $1/2+x, 1/2-y, 1/2+z$

Fig. 1

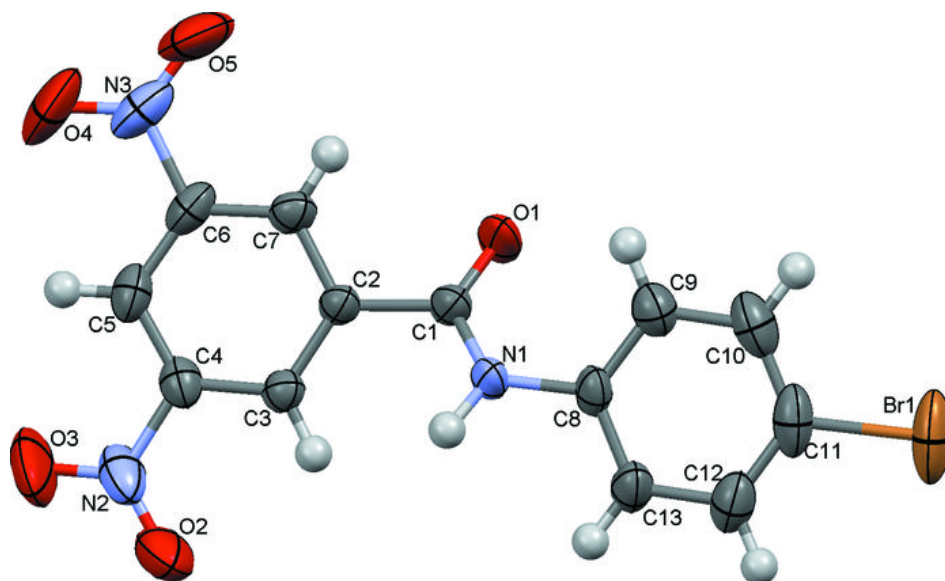




Fig. 2

