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5-(2-Bromo-5-methoxyphenyl)-1,3,4-oxadiazol-2-amine

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Key indicators

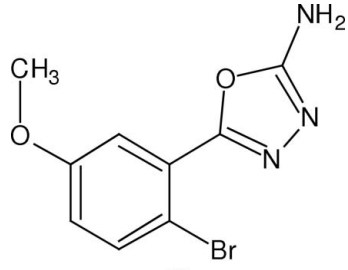
Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.010 Å
R factor = 0.073
wR factor = 0.220
Data-to-parameter ratio = 13.3

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title compound, $C_9H_8BrN_3O_2$, crystallizes with two almost identical molecules in the asymmetric unit. The crystal packing is stabilized by N—H···N hydrogen bonds and π–π stacking interactions.

Comment

2,5-Disubstituted 1,3,4-oxadiazole derivatives possess a wide range of biological activities, such as antibacterial, antifungal, antimalarial, anticonvulsant, central nervous system depressant, anti-inflammatory and anticancer properties (Jones *et al.*, 1965; Adelstein *et al.*, 1976; Zarghi *et al.*, 2005). The anti-inflammatory and analgesic activities of 1,3,4-oxadiazole derivatives have been extensively studied in recent years (Andreani *et al.*, 1994; Misra *et al.*, 1996; Amir & Shikha, 2004). We have recently reported the anti-inflammatory activity of 2,5-disubstituted 1,3,4-oxadiazole derivatives (Narayana, Ashalatha *et al.*, 2005, or Narayana, Vijaya Raj *et al.*, 2005?). Because of the important biological applications of 2,5-disubstituted-1,3,4-oxadiazole derivatives, the title compound, (I), has been synthesized and its crystal structure is reported here.



(I)

A perspective view of the asymmetric unit (two molecules) of compound (I) is shown in Fig. 1. Bond lengths and angles can be regarded as normal (Cambridge Structural Database, Version 5.27, November 2005, updated August 2006; *MOGUL*, Version 1.1; Allen, 2002). Both molecules are essentially planar (r.m.s. deviations for all non-H atoms 0.115 and 0.043 Å for the two different molecules in the asymmetric unit). The two molecules are almost identical (r.m.s. deviation for all non-H atoms 0.144 Å).

The crystal packing of (I) is stabilized by N—H···N hydrogen bonds (Table 1) and by π–π stacking interactions with the following distances: centroid(ring C11–C16)···centroid(ring C1/O1/N1/N2/C2)^{i} = 3.601 Å and centroid(ring C11*A*–C16*A*)···centroid(ring C1*A*/O1*A*/N1*A*/N2*A*/C2*A*)^{ii} = 3.441 Å [symmetry codes: (i) $1-x, 1-y, 1-z$; (ii) $2-x, 1-y, 2-z$].

Experimental

2-Bromo-5-methoxybenzoic acid hydrazide (4.9 g, 0.02 mol) and cyanogen bromide (2.3 g, 0.022 mol) in methanol (30 ml) were refluxed over a water bath for 4 h. The reaction mixture was then cooled and made alkaline using 5% sodium bicarbonate solution. The solid which separated was filtered off and purified by recrystallization from acetone. The title compound was obtained as white crystals from acetone (yield 93%; m.p. 453 K).

Crystal data

$C_9H_8BrN_3O_2$
M_r = 270.09
Monoclinic, $P2_1/c$
a = 18.1501 (19) Å
b = 10.3513 (7) Å
c = 11.0955 (10) Å
β = 95.298 (8)°
V = 2075.7 (3) Å^3
Z = 8
D_x = 1.729 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 3.94 mm^{-1}
T = 173 (2) K
Block, colourless
0.47 × 0.36 × 0.21 mm

Data collection

Stoe IPDS II two-circle diffractometer
ω scans
Absorption correction: multi-scan (*MULABS*; Spek, 2003; Blessing, 1995)
T_{min} = 0.215, T_{max} = 0.441
10488 measured reflections
3644 independent reflections
2876 reflections with $I > 2\sigma(I)$
R_{int} = 0.079
θ_{max} = 25.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.073
$wR(F^2)$ = 0.220
S = 1.05
3644 reflections
274 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1458P)^2 + 1.2938P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 1.77 e Å^{-3}
$\Delta\rho_{min}$ = −1.42 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 1997)
Extinction coefficient: 0.021 (2)

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

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3A···N2A^{i}	0.88	2.28	3.043 (9)	145
N3—H3B···N1^{ii}	0.88	2.24	3.067 (8)	158
N3A—H3A1···N2^{iii}	0.88	2.27	3.015 (9)	142
N3A—H3A2···N1A^{iv}	0.88	2.19	3.043 (8)	162

Symmetry codes: (i) $-x+2, y+\frac{1}{2}, -z+\frac{3}{2}$; (ii) $x, -y+\frac{3}{2}, z+\frac{1}{2}$; (iii) $-x+2, y-\frac{1}{2}, -z+\frac{3}{2}$; (iv) $x, -y+\frac{1}{2}, z+\frac{1}{2}$.

H atoms were found in a difference map but were subsequently positioned geometrically and allowed to ride on their parent atoms at distances of 0.88, 0.95 and 0.98 Å for N-bound H, aromatic H and methyl H, respectively. The methyl groups were allowed to rotate but not to tip. U_{iso}(H) was set to 1.2U_{eq}(C,N) or 1.5U_{eq}(C_{methyl}). The highest peak is 0.99 Å from Br1A and the deepest hole is 1.14 Å from Br1A.

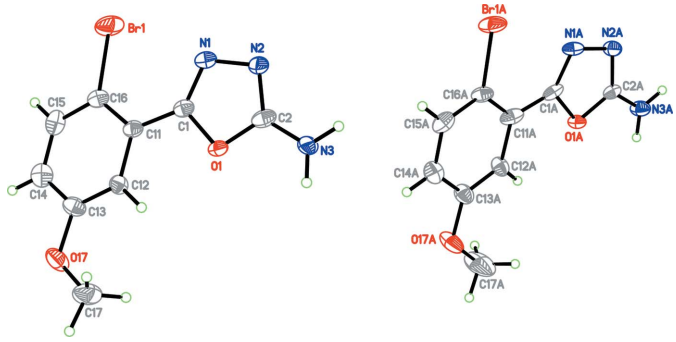


Figure 1
A perspective view of the two molecules in the asymmtric unit of (I), with the atom numbering. Displacement ellipsoids are drawn at the 50% probability level.

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL97*.

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