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5-Bromoanthranil

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Key indicators

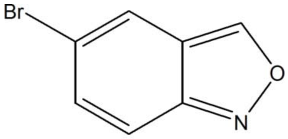
Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.011 Å
R factor = 0.027
wR factor = 0.077
Data-to-parameter ratio = 14.9

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

Crystals of the title compound, C_7H_4BrNO, are twinned by a twofold rotation about [001]. The crystal structure is stabilized by π-stacking of the virtually planar fused ring system along the crystallographic a axis, and through weak intermolecular C—H···O, C—H···N and C—H···Br interactions.

Comment

As part of our continuing studies into potentially bioactive indoles (Macleod *et al.*, 2003), the title compound 5-bromoanthranil, (I), was isolated as an unexpected major product in a key synthetic step. In order to identify the compound, a single-crystal structure determination was essential. Compound (I) was first reported by Bamberger & Lublin (1909), and a more recent synthesis given by Wünsch & Boulton (1967). The fused ring system is virtually planar, with a maximum deviation of 0.023 (4) Å from the mean plane defined by the nine ring atoms for C10 (r.m.s. deviation for the nine fitted atoms is 0.012 Å). In the unit cell, the molecule stacks in chains roughly parallel to the a axis through π-stacking. Molecules are related by a translation along a, with an intermolecular distance of 3.6 Å. Adjacent chains are also connected by weak C—H···O, C—H··· N and C—H···Br interactions.



(I)

Experimental

The title compound was prepared by refluxing a stirred solution of 2-azido-5-bromobenzaldehyde (0.512 g, 2.2 mmol) and p-toluenesulfonic acid monohydrate (4 mg, 0.02 mmol) in toluene (6 ml) for 9 h in a Dean–Stark apparatus under argon. The solution was allowed to cool to room temperature with stirring. The reaction mixture was then added to aqueous sodium bicarbonate (5 ml), the organic phase separated and washed with water (2 × 10 ml). Removal of the solvent *in vacuo*, followed by recrystallization from hexane, gave 5-bromoanthranil as colourless needles (yield 0.451 g, 92%).

Crystal data

C_7H_4BrNO
$M_r = 198.02$
Monoclinic, $P2_1$
$a = 3.8265 (2)$ Å
$b = 9.6148 (6)$ Å
$c = 8.9964 (5)$ Å
$\beta = 97.948 (4)^\circ$

$V = 327.81 (3)$ Å^3
$Z = 2$
Mo $K\alpha$ radiation
$\mu = 6.18$ mm^{-1}
$T = 100 (2)$ K
0.5 × 0.05 × 0.05 mm

doi:10.1107/S1600536807017278

Data collection

Enraf–Nonius KappaCCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2004; Blessing, 1995)
$T_{\min} = 0.335$, $T_{\max} = 0.734$

4797 measured reflections
1428 independent reflections
1341 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.033$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.027$
$wR(F^2) = 0.077$
$S = 1.18$
1428 reflections
96 parameters
1 restraint

H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\max} = 0.72$ e Å^{-3}
$\Delta\rho_{\min} = -0.54$ e Å^{-3}
Absolute structure: Flack (1983), 674 Friedel pairs
Flack parameter: 0.43 (3)

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

D—H···A	D—H	H···A	D···A	D—H···A
C11—H11···Br1$^{\rm i}$	0.95	3.17	3.919 (8)	138
C12—H12···Br1$^{\rm ii}$	0.95	3.37	4.004 (7)	126
C12—H12···O1$^{\rm iii}$	0.95	2.59	3.412 (8)	145
C15—H15···Br1$^{\rm iv}$	0.95	3.10	3.938 (8)	148
C16—H16···N1$^{\rm v}$	0.98 (5)	2.85 (5)	3.618 (8)	136 (3)
C16—H16···N1$^{\rm vi}$	0.98 (5)	2.71 (5)	3.402 (8)	128 (4)

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

All the crystals examined were non-merohedral twins. Application of the twin law $\bar{1}00\ 0\bar{1}0\ 0.65,0,1$, which corresponds to a twofold rotation about [001], gave a refinement with the best residuals. The HKLF5 format reflection file as written by *PLATON* (Spek, 2003) was used for refinement. The number of reflections affected by the twin contribution was 496. The twinning leads to a pseudo-orthorhombic lattice with a tripled c axis, which was the lattice automatically chosen by the indexing software. The proportions of the main and secondary twin domains were 0.882 (3):0.118 (3), as determined from the least-squares refinement. The value of the Flack parameter indicates that inversion twinning is also present, though due to the twinning, the absolute configuration could not be unambiguously determined. The absolute configuration reported here gives a marginally lower R value. The positional and thermal parameters for the atom H16 were refined freely; this is a non-standard situation regarding the H-atom placement routines in *SHELXL97* (Sheldrick, 1997). The remaining H atoms were refined with a riding model from calculated positions with C—H = 0.95 Å and with and with $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$.

Data collection: *COLLECT* (Nonius, 2004); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *SCALEPACK* and *DENZO* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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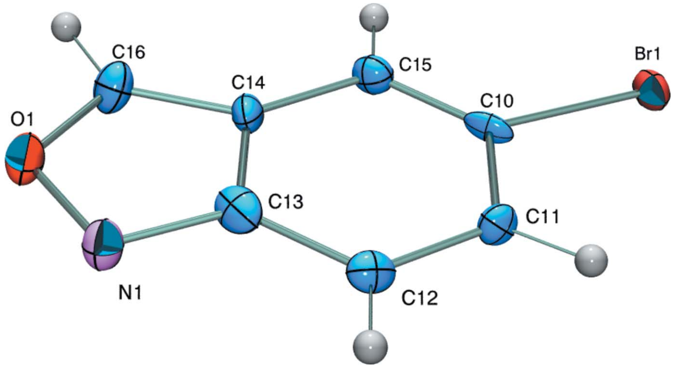

Figure 1
The molecular structure of (I), shown with 50% probability displacement ellipsoids.

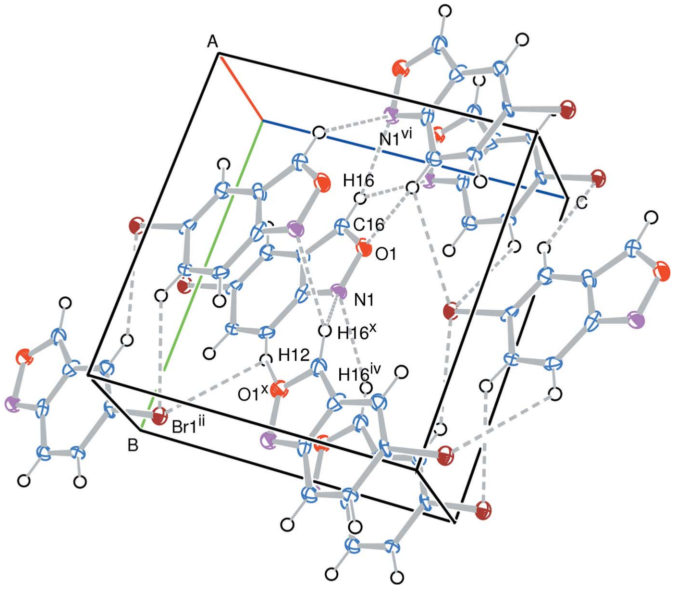

Figure 2
Packing diagram of (I); dashed lines indicate hydrogen-bond interactions. [Symmetry codes: (ii) $-x, \frac{1}{2}+y, 1-z$; (iv) $-x, -\frac{1}{2}+y, 1-z$; (vi) $1+x, 1+y, 1+z$; (x) $1-x, -\frac{1}{2}+y, 1-z$.]

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