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5-Bromo-4,6-dimethylisoxazolo[3,4-*b*]pyridin-3(1*H*)-one

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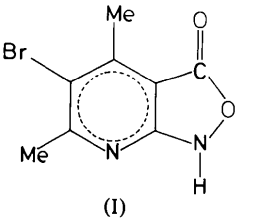
Abstract. $C_8H_7BrN_2O_2$, triclinic, $P\bar{1}$, $a = 7{\cdot}52$ (1), $b = 6{\cdot}89$ (1), $c = 9{\cdot}23$ (1) Å, $\alpha = 80{\cdot}35$ (5), $\beta = 66{\cdot}97$ (5), $\gamma = 72{\cdot}86$ (5)°, $V = 420$ Å^3, $Z = 2$, $D_m = 1{\cdot}90$, $D_c = 1{\cdot}92$ Mg m^{-3}, $F(000) = 240$. The structure was solved by direct and heavy-atom methods and refined by full-matrix least squares to a final $R = 0{\cdot}059$ for 1301 intensities. The crystal structure consists of dimers and the molecules are planar.

Introduction. The crystal structure determination of the title compound (I) was carried out in order to confirm the existence of the isoxazolone ring (Khan & Rafla, 1975) and also to establish the nature of the intermolecular interactions. The crystals were grown from methanol as brown needles elongated along the *a* axis. The crystal system was determined from precession photographs. A crystal of approximate dimensions 0·6 × 0·4 × 0·3 mm was used to collect intensities on a Hilger & Watts four-circle diffractometer. The cell dimensions were refined by a least-squares fit of 17 high-angle reflections. 1301 unique reflections up to $\theta_{max} = 24°$ were measured using the θ–2θ scan technique and Nb-filtered Mo $K\alpha$ radiation. The position of the Br atom was found from an *E* map using the *MULTAN* 78 package of programs (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978). The remaining atoms (C, N and O) were found from a weighted electron density map and H was located on a difference electron density map. The structure was refined by full-matrix least-squares methods using *SHELX* 76 (Sheldrick, 1976). Anisotropic temperature factors for atoms other than H and a single isotropic temperature factor for all H atoms were allowed. Throughout the refinement 1301 structure factors were used.‡ The function minimized was $\sum |\,|F_o| - |F_c|\,|^2$. The final *R* value was 0·059, where $R = \sum |\,|F_o| - |F_c|\,| / \sum |F_o|$. The highest peaks of residual electron density observed on the final difference map did not exceed 0·45 e Å^{-3}. The necessary calculations were performed on the University of York DEC-10 computer.



(I)

Table 1. *Positional parameters of the nonhydrogen atoms* (×10^4) *with e.s.d.'s in parentheses*

	x	*y*	*z*
Br	8556 (1)	2946 (2)	4742 (1)
N(1)	9480 (9)	7820 (9)	1596 (7)
C(2)	11195 (11)	6968 (11)	358 (8)
C(3)	12115 (10)	4934 (11)	420 (8)
C(4)	11436 (11)	3612 (11)	1703 (9)
C(5)	9656 (11)	4562 (11)	2923 (8)
C(6)	8743 (10)	6564 (11)	2847 (8)
N(7)	12021 (10)	8048 (9)	−908 (8)
C(8)	13836 (11)	4636 (11)	−1053 (8)
O(9)	15128 (8)	3188 (8)	−1637 (6)
O(10)	13738 (7)	6542 (8)	−1836 (6)
C(11)	6863 (15)	7643 (16)	4117 (11)
C(12)	12476 (15)	1426 (12)	1755 (12)

Table 2. *Positional* (×10^4) *and thermal parameters of the hydrogen atoms with e.s.d.'s in parentheses*

	x	*y*	*z*	*U* (Å^2 × 10^4)
H(70)	11497 (171)	9738 (187)	−983 (137)	1887 (431)
H(111)	6833 (150)	9330 (170)	4218 (121)	979 (323)
H(112)	6021 (165)	7419 (163)	3949 (125)	937 (383)
H(113)	7504 (182)	7523 (194)	4771 (146)	1307 (444)
H(121)	13292 (119)	1102 (117)	2368 (95)	386 (218)
H(122)	11535 (144)	398 (146)	2155 (112)	682 (280)
H(123)	12920 (202)	752 (202)	791 (163)	1605 (519)

Table 3. *Bond distances* (Å) *with e.s.d.'s in parentheses*

Br–C(5)	1·89 (0·5)	C(4)–C(5)	1·43 (1)
N(1)–C(2)	1·39 (1)	C(4)–C(12)	1·48 (1)
N(1)–C(6)	1·36 (1)	C(5)–C(6)	1·35 (1)
C(2)–C(3)	1·37 (1)	C(6)–C(11)	1·52 (1)
C(2)–N(7)	1·31 (1)	N(7)–O(10)	1·46 (1)
C(3)–C(4)	1·39 (1)	C(8)–O(9)	1·20 (1)
C(3)–C(8)	1·46 (1)	C(8)–O(10)	1·36 (1)

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‡ Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33990 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Table 4. *Bond angles* (°) *with e.s.d.'s in parentheses*

C(2)–N(1)–C(6)	118 (1)	Br–C(5)–C(4)	118 (1)
N(1)–C(2)–C(3)	121 (1)	Br–C(5)–C(6)	119 (1)
N(1)–C(2)–N(7)	123 (1)	C(4)–C(5)–C(6)	123 (1)
C(3)–C(2)–N(7)	117 (1)	N(1)–C(6)–C(11)	113 (1)
C(2)–C(3)–C(4)	123 (1)	C(5)–C(6)–C(11)	125 (1)
C(2)–C(3)–C(8)	105 (1)	C(2)–N(7)–O(10)	103 (1)
C(4)–C(3)–C(8)	132 (1)	C(3)–C(8)–O(9)	134 (1)
C(3)–C(4)–C(5)	114 (1)	C(3)–C(8)–O(10)	105 (1)
C(3)–C(4)–C(12)	122 (1)	O(9)–C(8)–O(10)	121 (1)
C(5)–C(4)–C(12)	124 (1)	N(7)–O(10)–C(8)	111 (1)

Table 5. *The least-squares plane and atomic deviations* (Å) *expressed in orthogonal ångström space*

Equation $-0{\cdot}8728X - 0{\cdot}2231Y - 0{\cdot}4342Z = 9{\cdot}856$

Br	0·010	C(5)	−0·008	O(9)	−0·026
N(1)	0·011	C(6)	−0·011	O(10)	0·009
C(2)	0·001	N(7)	0·015	C(11)	−0·014
C(3)	−0·010	C(8)	0·000	C(12)	0·017
C(4)	0·006				

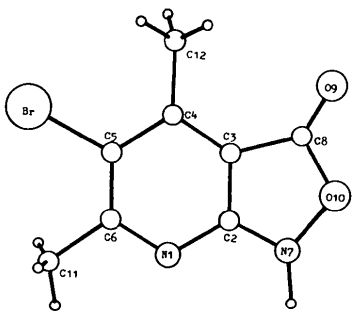

Fig. 1. A view of (I) showing the atom-numbering scheme.

Discussion. The fractional coordinates of the atoms are given in Tables 1 and 2. The bond distances and angles are given in Tables 3 and 4. The results obtained show that the molecule consists of fused pyridine and isoxazolone rings. The molecule is planar. The deviations of the atoms from the least-squares plane are listed in Table 5 and the molecule is shown in Fig. 1.

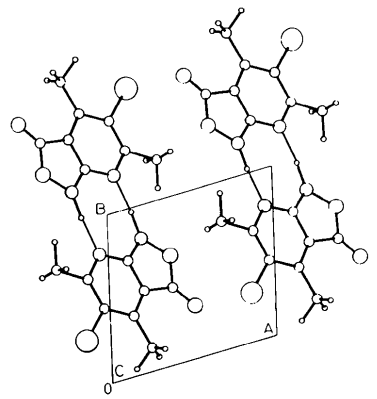

Fig. 2. A diagram showing the molecular packing.

The crystal packing is illustrated in Fig. 2. There is a strong intermolecular hydrogen bond of the type N(7)–H(70)···N(1) which binds the molecules into dimers. The distances are N(7)–H(70) = 1·11, H(70)···N(1) = 1·72 and N(7)···N(1) = 2·80 Å. Between the dimers there are no intermolecular forces other than weak van der Waals interactions.

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