

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

C(6)–N(1)–C(2)	111 (1)	C(11)–C(6)–C(5)	123 (1)
C(3)–C(2)–N(1)	129 (1)	O(10)–N(7)–C(2)	106 (1)
N(7)–C(2)–N(1)	122 (1)	C(13)–N(7)–C(2)	136 (1)
N(7)–C(2)–C(3)	109 (1)	C(13)–O(7)–O(10)	117 (1)
C(4)–C(3)–C(2)	120 (1)	O(9)–C(8)–C(3)	135 (1)
C(8)–C(3)–C(4)	132 (1)	O(10)–C(8)–C(3)	104 (1)
C(5)–C(4)–C(3)	113 (1)	O(10)–C(8)–O(9)	121 (1)
C(12)–C(4)–C(3)	122 (1)	C(8)–O(10)–N(7)	111 (1)
C(12)–C(4)–C(5)	125 (1)	O(14)–C(13)–N(7)	118 (1)
C(6)–C(5)–C(4)	123 (1)	C(15)–C(13)–N(7)	117 (1)
C(5)–C(6)–N(1)	125 (1)	C(15)–C(13)–O(14)	125 (1)
C(11)–C(6)–N(1)	113 (1)		

$\sum ||F_o| - |F_c||^2$. The final *R* index was 0.108, where $R = \sum ||F_o| - |F_c|| / \sum |F_o|$. Peaks on the difference map of the final electron density did not exceed 0.50 e Å⁻³. All the calculations were performed on the DEC-10 computer of the University of York.

Discussion. The atom coordinates are presented in Tables 1 and 2. Bond lengths and angles are given in Tables 3 and 4. The findings show that the acyl residue is attached to N(7) in the isoxazolone ring. Weak van der Waals interactions are the main force responsible for molecular packing in the crystal. The molecule is shown in Fig. 1.

scan technique were used to collect 931 unique measurable reflections up to $\theta_{\max} = 50^\circ$. The structure was determined by direct methods using *MULTAN* 78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978). The refinement of the coordinates with anisotropic temperature factors for atoms other than H and a single isotropic temperature factor for H atoms was carried out by full-matrix least-squares methods using *SHELX* 76 (Sheldrick, 1976). During refinement the H atoms were allowed to 'ride' on the heavy atoms. The refinement was carried out on 931 structure factors.* The usual Lorentz and polarization corrections were applied. The function minimized was

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33991 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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4,6-Dimethylisoxazolo[3,4-*b*]pyridin-3(7*H*)-one Monohydrate

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Abstract. C₈H₈N₂O₂·H₂O, triclinic, *P*1̄, *a* = 8.43 (1), *b* = 8.67 (1), *c* = 6.80 (1) Å, $\alpha = 77.92$ (5), $\beta = 77.06$ (5), $\gamma = 64.52$ (5)°, *V* = 434 Å³, *Z* = 2, *D_m* = 1.37, *D_c* = 1.39 Mg m⁻³, *F*(000) = 192. The structure was solved by direct methods and refined by full-matrix least squares to a final *R* = 0.0669 for 882 structure

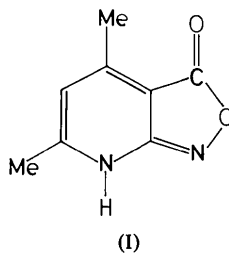
amplitudes. The movable H atom is attached to the N atom of the pyridine ring and not to that of the isoxazolone ring. Both H atoms of the water molecule are involved in a three-dimensional hydrogen-bonded network.

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Introduction. The X-ray investigation of the title compound (I) (Khan & Rafla, 1975) was undertaken to demonstrate the existence of a water molecule in the crystal lattice and to elucidate the role it plays. The © 1979 International Union of Crystallography

crystals were grown from methanol as yellow needles elongated along the *c* axis. The crystal system was found from precession photographs to be triclinic. The intensities were collected on a Hilger & Watts Y290 automatic diffractometer using a crystal of approximate dimensions 0.2 × 0.2 × 0.3 mm. The cell parameters were refined by a least-squares fit of 17 high-angle reflections. 882 unique reflections up to $\theta_{\max} = 50^\circ$ were measured with the θ - 2θ scan technique and Ni-filtered Cu $K\alpha$ radiation. The structure was solved by direct methods using the *MULTAN* 78 package of programs (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978). The positions of the atoms of the water molecule and the non-methyl H atoms were found from a difference electron density map. The methyl-group H atoms were fitted geometrically. The refinement of the structure was performed by full-matrix least-squares methods using *SHELX* 76 (Sheldrick, 1976). Refinement of the coordinates with anisotropic temperature factors for atoms other than H and a single isotropic temperature factor for all H atoms was carried out. During refinement the H atoms were allowed to 'ride' on the heavy atoms. The 882 structure factors were used throughout the refinement.* The usual Lorentz and polarization corrections were made to convert intensities into structure factors. The function minimized was $\sum |F_o| - |F_c|^2$. The final *R* value was 0.0679, where $R = \frac{\sum |F_o| - |F_c|}{\sum |F_o|}$. The residual electron density on the final difference map did not exceed 0.45 e Å⁻³. The calculations were performed on the DEC-10 computer of the University of York.



Discussion. The positional parameters of the atoms are listed in Tables 1 and 2. Bond distances and angles are presented in Tables 3 and 4. The results obtained show that in the crystalline state the molecules adopt an electronic structure different from that reported earlier (Khan & Rafta, 1975). H(10) is located at N(1) in the pyridine ring (Fig. 1), and not at N(7) in the isoxazolone ring (I). The requirements of molecular packing and maximum development in the hydrogen-bonded network are likely to be responsible for this

proton translocation. The molecules are planar. The presence of one water molecule was established and both of its H atoms are found to take part in a very strong three-dimensional hydrogen-bonded system. Fig. 2 shows clearly the details of this hydrogen-bonded network and Table 5 summarizes the distances involved.

At this stage a few additional comments and remarks should be made on the structures of 4,6-dimethylisoxazolo[3,4-*b*]pyridin-3(7*H*)-one monohydrate, 5-bromo-4,6-dimethylisoxazolo[3,4-*b*]pyridin-3(1*H*)-one (Hamid, Hempel & Hull, 1979) and 1-acetyl-4,6-dimethylisoxazolo[3,4-*b*]pyridin-3(1*H*)-one (Hamid & Hempel, 1979) in order to make the results obtained more comprehensive and plausible.

Considerable shortening of the C—N bond of the isoxazolone ring for the monohydrate and bromo compounds (1.30 and 1.31 Å) relative to that in the N-

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

	<i>x</i>	<i>y</i>	<i>z</i>
N(1)	8399 (5)	2350 (4)	2167 (5)
C(2)	10179 (6)	1325 (5)	2073 (6)
C(3)	10738 (5)	-464 (5)	2493 (6)
C(4)	9576 (6)	-1240 (5)	2972 (6)
C(5)	7759 (6)	-132 (6)	3050 (7)
C(6)	7217 (6)	1619 (6)	2654 (6)
N(7)	11413 (5)	1911 (5)	1623 (6)
C(8)	12633 (6)	-1091 (6)	2261 (7)
O(9)	13810 (4)	-2549 (4)	2462 (5)
O(10)	13044 (4)	313 (4)	1740 (5)
C(11)	5298 (6)	2845 (7)	2772 (8)
C(12)	10159 (8)	-3152 (6)	3440 (8)
O(13)	7384 (5)	5808 (4)	1359 (5)

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

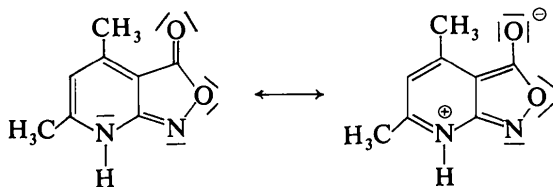
	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> (Å ² × 10 ⁴)
H(10)	7672 (5)	3822 (4)	1868 (5)	969 (196)
H(50)	6907 (6)	-520 (6)	3090 (7)	337 (117)
H(111)	4383 (6)	2243 (7)	2919 (8)	1881 (304)
H(112)	5246 (6)	3686 (7)	1349 (8)	1302 (277)
H(113)	4931 (6)	3587 (7)	4018 (8)	1558 (305)
H(121)	9032 (8)	-3478 (6)	3592 (8)	1813 (329)
H(122)	10626 (8)	-3479 (6)	4889 (8)	2998 (457)
H(123)	11211 (8)	-3863 (6)	2325 (8)	2562 (382)
H(131)	7759 (5)	6635 (4)	152 (5)	1277 (213)
H(132)	6395 (5)	6360 (4)	1671 (5)	871 (222)

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

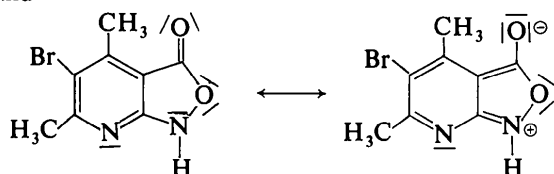
N(1)—C(2)	1.37 (1)	C(5)—C(6)	1.37 (1)
N(1)—C(6)	1.35 (1)	C(6)—C(11)	1.50 (1)
C(2)—C(3)	1.40 (1)	N(7)—O(10)	1.48 (1)
C(2)—N(7)	1.30 (1)	C(8)—O(10)	1.36 (1)
C(3)—C(4)	1.36 (1)	C(8)—O(9)	1.23 (1)
C(3)—C(8)	1.43 (1)	O(13)—H(131)	1.06 (1)
C(4)—C(5)	1.41 (1)	O(13)—H(132)	0.77 (1)
C(4)—C(12)	1.50 (1)		

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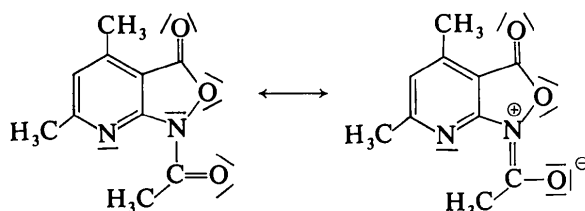
acetyl compound (1.44 Å) may be attributable to the mesomeric effects of charge separation:



and



The major electronic structure with charge separated in the N-acetyl compound is



In addition, the electronegative Br atom slightly reduces the basic character of the pyridine-ring N atom because of induction phenomena. This may be another reason for the H atom to be on the isoxazolone ring N atom. In compounds containing six-membered heterocyclic rings the valence angles of the N atoms with an extra-annular H atom are usually noticeably larger than those of N atoms without any attachment (Singh, 1965). This observation agrees well with our results, the characteristic angles being 120° for the monohydrate and 118° for the bromo compounds. The difference of 2° may not be a spectacular one but the tendency is apparent.

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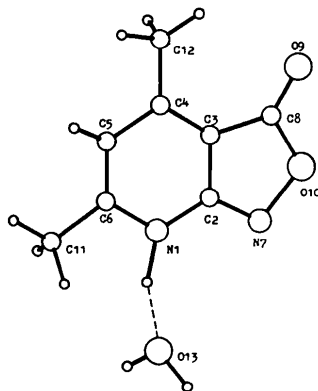


Fig. 1. A view of the molecule showing the atom-numbering scheme.

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

C(2)—N(1)—C(6)	120 (1)	C(4)—C(5)—C(6)	121 (1)
N(1)—C(2)—C(3)	120 (1)	N(1)—C(6)—C(5)	121 (1)
N(1)—C(2)—N(7)	124 (1)	N(1)—C(6)—C(11)	116 (1)
C(3)—C(2)—N(7)	117 (1)	C(5)—C(6)—C(11)	123 (1)
C(2)—C(3)—C(4)	122 (1)	C(2)—N(7)—O(10)	102 (1)
C(2)—C(3)—C(8)	104 (1)	C(3)—C(8)—O(10)	107 (1)
C(4)—C(3)—C(8)	134 (1)	C(3)—C(8)—O(9)	133 (1)
C(3)—C(4)—C(5)	116 (1)	O(9)—C(8)—O(10)	121 (1)
C(3)—C(4)—C(12)	123 (1)	N(7)—O(10)—C(8)	111 (1)
C(5)—C(4)—C(12)	121 (1)	H(132)—O(13)—H(131)	102 (1)

Table 5. Intermolecular hydrogen-bond geometry (Å)

N(1) ^I ...O(13) ^I	2.70	O(9) ^{III} ...O(13) ^I	2.72
O(13) ^I ...H(10) ^I	1.60	O(9) ^I ...H(132) ^I	1.96
N(1)—H(10)	1.15	O(13)—H(132)	0.77

N(7) ^{II} ...O(13) ^I	2.86	Symmetry code	
N(7) ^I ...H(131) ^I	1.81	(I) x, y, z	
O(13)—H(131)	1.06	(II) $2-x, 1-y, -z$	
		(III) $x-1, y+1, z$	

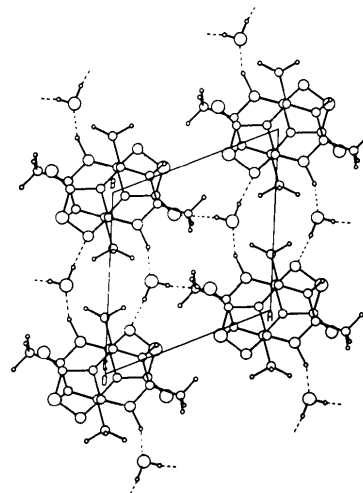


Fig. 2. The crystal packing and hydrogen-bond scheme.

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