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

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Abstract. $C_{10}H_{10}N_2O_3$, monoclinic, $P2_1/c$, $a = 4 \cdot 04$ (1), $b = 17 \cdot 07$ (1), $c = 14 \cdot 33$ (1) Å, $\beta = 96 \cdot 36$ (5)°, $V = 982$ Å^3, $Z = 4$, $F(000) = 432$, $D_m = 1 \cdot 38$, $D_c = 1 \cdot 39$ Mg m^{-3}. The structure was solved by direct methods and refined to $R = 0 \cdot 108$ for 931 intensities. The molecules are planar and held together by van der Waals forces. The acyl residue was found at the N atom of the isoxazolone ring.

Introduction. The crystal structure determination of the title compound (I) was undertaken to confirm that the N-acylation occurred at the N atom of the isoxazolone ring (Khan & Rafla, 1975). The crystals were grown from methanol as colourless needles elongated along the *a* axis. The space group was determined from
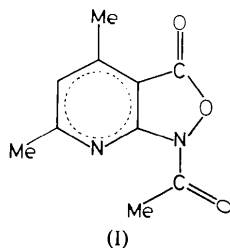
(I)

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precession photographs to be $P2_1/c$. A crystal of approximate dimensions $0 \cdot 4 \times 0 \cdot 2 \times 0 \cdot 2$ mm was used for data collection on a Hilger & Watts Y290 automatic diffractometer. It proved to be difficult to grow good quality crystals. The unit-cell parameters were refined by a least-squares fit of 17 high-angle reflections. Ni-filtered Cu $K\alpha$ radiation and the θ–2θ

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

	x	y	z	U (Å$^2 \times 10^4$)
H(50)	−2620 (19)	2270 (5)	10727 (5)	1214 (260)
H(111)	−3726 (25)	3494 (5)	11042 (6)	1650 (298)
H(112)	−4460 (25)	4257 (5)	10215 (6)	1275 (270)
H(113)	−565 (25)	4181 (5)	10898 (6)	1135 (263)
H(121)	−2050 (23)	1044 (4)	10303 (6)	1297 (262)
H(122)	1704 (23)	794 (4)	9855 (6)	1418 (294)
H(123)	−2134 (23)	651 (4)	9162 (6)	750 (199)
H(151)	1023 (26)	4466 (4)	8273 (6)	2326 (343)
H(152)	1385 (26)	4926 (4)	7186 (6)	2154 (335)
H(153)	4898 (26)	4843 (4)	8028 (6)	1591 (335)

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

N(1)–C(2)	1·35 (1)	C(6)–C(5)	1·36 (1)
N(1)–C(6)	1·37 (1)	C(11)–C(6)	1·51 (1)
C(3)–C(2)	1·36 (1)	O(10)–N(7)	1·40 (1)
N(7)–C(2)	1·44 (1)	C(13)–N(7)	1·37 (1)
C(4)–C(3)	1·41 (1)	O(9)–C(8)	1·16 (1)
C(8)–C(3)	1·49 (1)	O(10)–C(8)	1·40 (1)
C(5)–C(4)	1·39 (1)	O(14)–C(13)	1·22 (1)
C(12)–C(4)	1·48 (1)	C(15)–C(13)	1·46 (1)
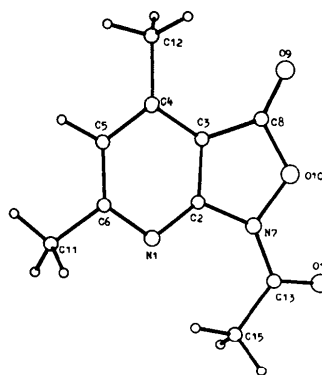
Fig. 1. A view of the molecule showing the atom-numbering scheme.

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

	x	y	z
N(1)	−192 (16)	3543 (3)	9133 (4)
C(2)	796 (18)	2971 (4)	8577 (5)
C(3)	677 (19)	2184 (4)	8700 (5)
C(4)	−642 (19)	1874 (4)	9496 (5)
C(5)	−1658 (19)	2451 (5)	10087 (5)
C(6)	−1448 (19)	3233 (5)	9903 (5)
N(7)	2319 (16)	3140 (3)	7741 (4)
C(8)	2157 (18)	1794 (4)	7914 (5)
O(9)	2645 (15)	1146 (3)	7718 (4)
O(10)	3074 (13)	2414 (3)	7358 (3)
C(11)	−2618 (25)	3855 (5)	10541 (6)
C(12)	−776 (23)	1024 (4)	9679 (6)
C(13)	3367 (20)	3797 (4)	7314 (5)
O(14)	4998 (16)	3713 (3)	6656 (4)
C(15)	2635 (26)	4559 (4)	7730 (6)

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)		

scan technique were used to collect 931 unique measurable reflections up to $\theta_{max} = 50°$. 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.

$\sum ||F_o| - |F_c||^2$. 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.

References

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

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Abstract. $C_8H_8N_2O_2.H_2O$, triclinic, $P\bar{1}$, $a = 8·43$ (1), $b = 8·67$ (1), $c = 6·80$ (1) Å, $\alpha = 77·92$ (5), $\beta = 77·06$ (5), $\gamma = 64·52$ (5)°, $V = 434$ Å^3, $Z = 2$, $D_m = 1·37$, $D_c = 1·39$ 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