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Structure of a Substituted Isoxazoline

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Abstract. 3-Cyano-4,4,5,5-tetramethyl-4,5-dihydroisoxazole *N*-oxide, $C_8H_{12}N_2O_2$, $M_r = 168{\cdot}20$, orthorhombic, $Pna2_1$, $a = 10{\cdot}283\ (8)$, $b = 12{\cdot}389\ (4)$, $c = 7{\cdot}268\ (3)$ Å, $V = 925{\cdot}2\ (9)$ Å^3, $Z = 4$, $D_x = 1{\cdot}207$ Mg m^{-3}, $\lambda(\text{Cu } K\alpha) = 1{\cdot}54178$ Å, $\mu = 0{\cdot}691$ mm^{-1}, $F(000) = 360$, $T = 295$ K, final $R = 0{\cdot}065$, $wR = 0{\cdot}065$ for 352 independent observed reflections. In this compound the isoxazoline ring is a non-planar envelope. Intermolecular contacts are at normal van der Waals separations.

Experimental. A clear $0{\cdot}10 \times 0{\cdot}15 \times 0{\cdot}32$ mm data crystal was provided by J. Boyer of the University of Illinois at Chicago. Data crystal coated with an acrylic polymer to inhibit degradation. Automated Nicolet *R*3*m* diffractometer with incident-beam graphite monochromator, 18 centered reflections within $18 \le 2\theta \le 60°$ used for determining lattice parameters. $(\text{Sin}\theta/\lambda)_{max} = 0{\cdot}46$ Å^{-1}, range of *hkl*: $0 \le h \le 9$, $-3 \le k \le 10$, $0 \le l \le 6$. Standards 006, 080, 004, monitored every 60 reflections with linear variation of 27% over data collection, $\theta/2\theta$ mode, scan width $(2{\cdot}0 + \Delta_{\alpha 1\alpha 2})°$, 2θ scan rate 30° min^{-1}. 513 reflections measured, 421 unique, $R_{int} = 0{\cdot}047$, 352 observed $F_o > 3\sigma(F_o)$. Data corrected for Lorentz and polarization effects, but not absorption. Structure solved by direct methods. The least-squares refinement used *SHELXTL* (Sheldrick, 1980). $\sum w(|F_o| - |F_c|)^2$ minimized where $w = 1/[\sigma^2(|F_o|) + g(F_o)^2]$, $g = 0{\cdot}00030$. Secondary-extinction parameter $p = 0{\cdot}003\ (1)$ in $F_c^* = F_c/[1{\cdot}0 + 0{\cdot}002(p)F_o^2/\sin 2\theta]^{0{\cdot}25}$. 121 parameters refined: atom coordinates, anisotropic temperature factors for all non-H atoms, methyl-H atoms included using riding model, C–H = 0·96 Å, H–C–H = 109·5°, $U(\text{H}) = 1{\cdot}2U_{eq}(\text{C})$. $(\Delta/\sigma)_{max} = 0{\cdot}118$, $(\Delta/\sigma)_{mean} = 0{\cdot}021$, $R = 0{\cdot}065$, $wR = 0{\cdot}065$, $S = 1{\cdot}820$. Final difference Fourier excursions 0·22 and −0·22 e Å^{-3}. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974).† Atom numbering for Tables 1 and 2, which give atom coordinates, bond distances and bond angles, follows that shown in Fig. 1.

† Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43400 (6 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 1. *Atom coordinates* (×10^4) *and thermal parameters* (Å^2 ×10^3)

	x	*y*	*z*	U_{eq}*
O(1)	3426 (7)	2448 (5)	2886 (15)	67 (3)
N(2)	2510 (10)	2091 (10)	1619 (17)	65 (4)
C(3)	1738 (11)	2865 (10)	1112 (19)	53 (5)
C(4)	2118 (11)	3939 (9)	1957	49 (4)
C(5)	2913 (11)	3517 (9)	3583 (23)	58 (5)
O(6)	2547 (10)	1132 (7)	1144 (19)	93 (4)
C(7)	916 (12)	4587 (10)	2617 (23)	79 (5)
C(8)	2878 (11)	4592 (9)	551 (18)	76 (6)
C(9)	2189 (13)	3239 (10)	5271 (19)	80 (6)
C(10)	4175 (11)	4133 (9)	4061 (26)	76 (6)
C(11)	764 (14)	2682 (11)	−230 (25)	73 (6)
N(12)	−19 (12)	2539 (11)	−1331 (22)	95 (6)

* Equivalent isotropic *U* defined as one-third of the trace of the orthogonalized U_{ij} tensor.

Table 2. *Bond lengths* (Å) *and bond angles* (°)

O(1)–N(2)	1·389 (15)	O(1)–C(5)	1·512 (14)
N(2)–C(3)	1·298 (17)	N(2)–O(6)	1·238 (15)
C(3)–C(4)	1·516 (16)	C(3)–C(11)	1·416 (21)
C(4)–C(5)	1·528 (17)	C(4)–C(7)	1·551 (16)
C(4)–C(8)	1·519 (15)	C(5)–C(9)	1·475 (21)
C(5)–C(10)	1·545 (17)	C(11)–N(12)	1·148 (21)
N(2)–O(1)–C(5)	105·3 (9)	O(1)–N(2)–C(3)	111·6 (11)
O(1)–N(2)–O(6)	117·9 (11)	C(3)–N(2)–O(6)	130·5 (12)
N(2)–C(3)–C(4)	112·0 (11)	N(2)–C(3)–C(11)	120·6 (12)
C(4)–C(3)–C(11)	127·0 (11)	C(3)–C(4)–C(5)	98·7 (9)
C(3)–C(4)–C(7)	112·0 (9)	C(5)–C(4)–C(7)	111·4 (8)
C(3)–C(4)–C(8)	109·1 (7)	C(5)–C(4)–C(8)	115·3 (10)
C(7)–C(4)–C(8)	110·0 (9)	O(1)–C(5)–C(4)	103·1 (11)
O(1)–C(5)–C(9)	104·5 (10)	C(4)–C(5)–C(9)	116·9 (10)
O(1)–C(5)–C(10)	102·4 (9)	C(4)–C(5)–C(10)	117·0 (11)
C(9)–C(5)–C(10)	110·6 (13)	C(3)–C(11)–N(12)	179·3 (15)

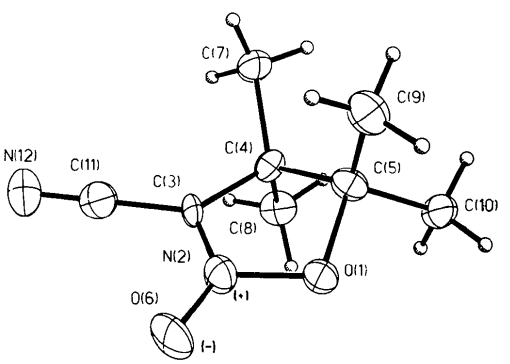

Fig. 1. Thermal-ellipsoid plot of 3-cyano-4,4,5,5-tetramethyl-4,5-dihydroisoxazole *N*-oxide with ellipsoids drawn at 20% probability level. The C(3)–N(2) bond is formally a double bond.

Related literature. Preparation of similar compounds, 4-nitroisoxazoline *N*-oxides, have been reported by Gabitov, Kremleva & Fridman (1978), and a heterocycle with the same ring connectivity has been reported by Delugeard, Baudour & Messager (1974).

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Structure of an Azapyrimidine Derivative

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Abstract. 5-Aza-6-methoxyuracil (1,2,3,4-tetrahydro-6-methoxy-1,3,5-triazine-2,4-dione), $C_4H_7N_3O_3$, M_r = 145·12, monoclinic, $P2_1/c$, a = 9·994 (5), b = 4·316 (1), c = 15·891 (8) Å, β = 115·40 (3)°, V = 619·1 (4) Å^3, Z = 4, D_x = 1·557 Mg m^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, μ = 1·11 mm^{-1}, $F(000)$ = 304, T = 295 K, final R = 0·050, wR = 0·058 for 756 independent observed reflections. The azapyrimidine ring is in a shallow boat configuration, with the methoxy axial to the N(1)–C(2)–N(5) plane of the boat. The puckering parameter, the angle between the base plane N(1)–C(2)–C(4)–N(5) and the N(1)–C(6)–N(5) and C(2)–N(3)–C(4) planes, is 30·0 (3) and 10·7 (3)° respectively. Each of the three ring N atoms participates in an intermolecular hydrogen bond; N(1) acts as a donor to O(6)′($1-x$, $y-0{\cdot}5$, $0{\cdot}5-z$), N(3) is a donor to O(2)′($1-x$, $-y$, $-z$), and N(5) is a donor to O(4)′($-x$, $y-0{\cdot}5$, $0{\cdot}5-z$), with N···O′ distances of 2·905 (4), 2·853 (4), and 2·817 (3) Å respectively.

Experimental. A clear, colorless 0·12 × 0·20 × 0·30 mm data crystal was provided by C. Coon of Lawrence Livermore Laboratory. Automated Nicolet *R*3*m* diffractometer with incident-beam graphite monochromator, 25 centered reflections within $40 \le 2\theta \le 77°$ used for determining lattice parameters. $(\mathrm{Sin}\theta/\lambda)_{max}$ = 0·55 Å^{-1}, range of *hkl*: $0 \le h \le 10$, $0 \le k \le 4$, $-16 \le l \le 15$. Standards 300, 024, 004, monitored every 60 reflections with random variation of 3·1% over data collection, $\theta/2\theta$ mode, scan width (2·0 + $\Delta_{\alpha1\alpha2}$)°, scan rate a function of count rate (2·0° min^{-1} minimum, 30° min^{-1} maximum), 1102 reflections measured, 846 unique, R_{int} = 0·008, 756 observed with $F_o > 3\sigma(F_o)$. Data corrected for Lorentz and polarization, but not absorption effects. Structure solved by direct methods. The least-squares refinement

Table 1. *Atom coordinates* (×10^4) *and temperature parameters* (Å^2 ×10^3)

	x	y	z	U_{eq}
N(1)	4386 (3)	−45 (6)	3215 (2)	40 (1)*
C(2)	4640 (3)	−1972 (7)	3937 (2)	35 (1)*
N(3)	3399 (2)	−3238 (6)	3956 (2)	37 (1)*
C(4)	1972 (3)	−2294 (7)	3415 (2)	37 (1)*
N(5)	1834 (3)	−335 (6)	2712 (2)	40 (1)*
C(6)	2969 (3)	94 (7)	2417 (2)	38 (1)*
O(2)	5891 (2)	−2519 (5)	4535 (1)	45 (1)*
O(4)	938 (2)	−3139 (6)	3581 (1)	51 (1)*
O(6)	2967 (2)	−2267 (5)	1786 (1)	42 (1)*
C(7)	1712 (3)	−2099 (9)	884 (2)	54 (1)*
H(1)	5092 (37)	724 (74)	3166 (21)	48
H(3)	3524 (35)	−4318 (75)	4418 (22)	48
H(5)	1044 (39)	220 (77)	2384 (23)	48
H(6)	2932 (31)	2601 (72)	2165 (20)	48

* Equivalent isotropic U defined as one-third of the trace of the orthogonalized U_{ij} tensor.

Table 2. *Bond lengths* (Å), *bond angles* (°) *and hydrogen-bond parameters* (Å,°)

N(1)–C(2)	1·350 (4)	N(1)–C(6)	1·441 (3)
C(2)–N(3)	1·367 (4)	C(2)–O(2)	1·226 (3)
N(3)–C(4)	1·374 (3)	C(4)–N(5)	1·349 (4)
C(4)–O(4)	1·226 (4)	N(5)–C(6)	1·424 (5)
C(6)–O(6)	1·428 (4)	O(6)–C(7)	1·446 (3)
C(2)–N(1)–C(6)	121·9 (3)	N(1)–C(2)–N(3)	115·1 (2)
N(1)–C(2)–O(2)	122·3 (3)	N(3)–C(2)–O(2)	122·6 (3)
C(2)–N(3)–C(4)	125·1 (3)	N(3)–C(4)–N(5)	114·3 (3)
N(3)–C(4)–O(4)	121·4 (3)	N(5)–C(4)–O(4)	124·2 (2)
C(4)–N(5)–C(6)	122·9 (2)	N(1)–C(6)–N(5)	108·7 (3)
N(1)–C(6)–O(6)	107·1 (2)	N(5)–C(6)–O(6)	122·6 (2)
C(6)–O(6)–C(7)	133·5 (2)		
H(1)···O(6)′	2·09 (4)	N(1)···O(6)′	2·905 (4)
H(3)···O(2)′	2·03 (4)	N(3)···O(2)′	2·853 (4)
H(5)···O(4)′	2·04 (4)	N(5)···O(4)′	2·817 (3)
N(1)–H(1)···O(6)′	168·1 (2·3)	N(3)–H(3)···O(2)′	173·2 (2·4)
N(5)–H(5)···O(4)′	174·4 (2·4)		

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