parameters refined. Atomic scattering factors were obtained from Cromer & Mann (1968).

The final positional parameters are given in Table 1,* bond lengths and angles in Table 2. Inter- and intramolecular hydrogen bonds are listed in Table 3. Fig. 1 shows a stereoview of the molecule. The intermolecular hydrogen bonds are shown in Fig. 2.

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and bond distances and angles involving H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55468 (37 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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3-Cyano-3-dimethylamino-4-ethoxy-1-phenyl-2-azetidinone

BY J. FENEAU-DUPONT AND J.-P. DECLERCQ

Laboratoire de chimie physique et de cristallographie, Université Catholique de Louvain, 1 place Louis Pasteur, 1348 Louvain la Neuve, Belgium
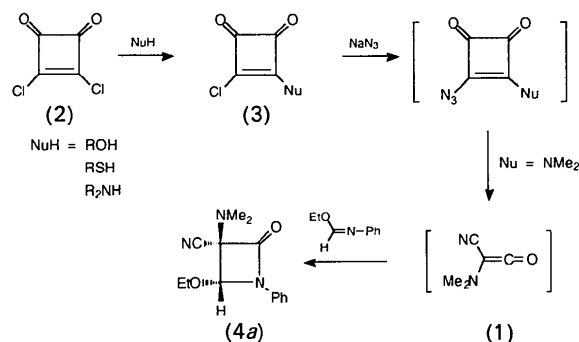
AND M. C. LABILLE, Z. JANOUSEK AND H. G. VIEHE

Laboratoire de chimie organique, Université Catholique de Louvain, 1 place Louis Pasteur, 1348 Louvain la Neuve, Belgium

Abstract. 3-Dimethylamino-4-ethoxy-2-oxo-1-phenyl-3-azetidinecarbonitrile, $C_{14}H_{17}N_3O_2$, $M_r = 259.3$, monoclinic, $P2_1/n$, $a = 6.105$ (2), $b = 21.184$ (5), $c = 11.356$ (7) Å, $\beta = 91.14$ (3)°, $V = 1468.4$ (11) Å^3, $Z = 4$, $D_x = 1.17$ g cm^{-3}, λ(Cu $K\alpha$) = 1.54178 Å, $\mu = 6.63$ cm^{-1}, $F(000) = 552$, $T = 291$ K, $R = 0.061$ for 1864 observed reflections. The azetidinone ring is nearly planar with endocyclic angles of 2, -2, 2 and $-2°$ (maximum deviation from the plane: 0.015 Å for the N atom). The relative configurations at the chiral centres are R,R (or S,S).

Experimental. Captodative cyanoketenes (1) have become accessible from dichlorocyclobutenedione (2) (squaric acid dichloride) by two successive nucleophilic substitutions (Labille, Janousek & Viehe, 1991). Monochlorocyclobutenediones (3) treated with sodium azide decompose immediately with a loss of carbon monoxide and nitrogen to produce (1). Ketene intermediates (1) can be trapped with imidates to give novel β-lactam derivatives.

The title compound (4a) was obtained in 42% yield and has been characterized by ^{1}H, ^{13}C NMR, IR and MS. Its stereochemistry is now elucidated by X-ray analysis.

A parallelepiped crystal with dimensions 0.25 × 0.37 × 0.40 mm was used for data collection. Lattice parameters were refined using 17 reflections in the range $16 \le 2\theta \le 64°$. A Huber four-circle diffractom-

Table 1. *Atomic coordinates* $(\times 10^4)$ *and equivalent isotropic temperature factors* $(\text{Å}^2 \times 10^3)$

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a^*_i a^*_j \mathbf{a}_i.\mathbf{a}_j$$

	x	y	z	U_{eq}
N1	−825 (3)	9413 (1)	7122 (2)	68 (1)
C2	−2367 (4)	8959 (1)	7023 (2)	73 (1)
C3	−897 (4)	8474 (1)	7676 (2)	71 (1)
C4	743 (4)	9034 (1)	7790 (2)	68 (1)
C5	−1782 (5)	8262 (1)	8818 (2)	90 (1)
N6	−2488 (5)	8075 (2)	9657 (3)	130 (1)
N7	−175 (4)	7961 (1)	6941 (2)	80 (1)
C8	−1966 (6)	7548 (2)	6548 (3)	115 (1)
C9	1641 (6)	7614 (1)	7483 (3)	106 (1)
O10	1046 (3)	9283 (1)	8910 (1)	83 (1)
C11	2983 (6)	9063 (2)	9490 (3)	104 (1)
C12	2999 (7)	9319 (2)	10721 (3)	129 (2)
O13	−4180 (3)	8945 (1)	6579 (2)	98 (1)
C14	−628 (4)	10037 (1)	6689 (2)	69 (1)
C15	−2339 (5)	10313 (1)	6071 (2)	83 (1)
C16	−2035 (6)	10935 (2)	5657 (3)	100 (1)
C17	−138 (7)	11248 (2)	5869 (3)	106 (1)
C18	1561 (6)	10966 (1)	6473 (3)	105 (1)
C19	1318 (5)	10356 (1)	6883 (3)	89 (1)

Table 2. *Bond distances* (Å) *and angles* (°)

C2—N1	1.349 (3)	C4—N1	1.452 (3)
C14—N1	1.416 (3)	C3—C2	1.543 (3)
O13—C2	1.208 (3)	C4—C3	1.556 (3)
C5—C3	1.484 (3)	N7—C3	1.445 (3)
O10—C4	1.385 (3)	N6—C5	1.126 (3)
C8—N7	1.464 (4)	C9—N7	1.457 (3)
C11—O10	1.422 (3)	C12—C11	1.499 (4)
C15—C14	1.377 (3)	C19—C14	1.381 (4)
C16—C15	1.412 (4)	C17—C16	1.352 (5)
C18—C17	1.370 (5)	C19—C18	1.382 (4)
C4—N1—C2	95.8 (2)	C14—N1—C2	134.5 (2)
C14—N1—C4	129.6 (2)	C3—C2—N1	92.1 (2)
O13—C2—N1	133.1 (2)	O13—C2—C3	134.8 (2)
C4—C3—C2	84.3 (2)	C5—C3—C2	113.8 (2)
C5—C3—C4	113.8 (2)	N7—C3—C2	113.8 (2)
N7—C3—C4	114.7 (2)	N7—C3—C5	113.4 (2)
C3—C4—N1	87.8 (2)	O10—C4—N1	110.1 (2)
O10—C4—C3	116.2 (2)	N6—C5—C3	176.6 (4)
C8—N7—C3	113.1 (2)	C9—N7—C3	112.0 (2)
C9—N7—C8	112.5 (2)	C11—O10—C4	113.2 (2)
C12—C11—O10	107.7 (3)	C15—C14—N1	120.3 (2)
C19—C14—N1	118.7 (2)	C19—C14—C15	121.0 (2)
C16—C15—C14	117.5 (3)	C17—C16—C15	121.2 (3)
C18—C17—C16	120.7 (3)	C19—C18—C17	119.5 (3)
C18—C19—C14	120.1 (3)		

Fig. 1. Stereoscopic view of the molecule and atom numbering.

eter and Rigaku rotating anode, with graphite-monochromated Cu $K\alpha$ radiation and ω–θ scan mode, were used to measure 2657 independent reflections with $\sin\theta/\lambda \le 0.60 \text{ Å}^{-1}$ and $-7 \le h \le 7$, $0 \le k \le 25$, $0 \le l \le 13$, 1864 of which had $I \ge 2.5\sigma(I)$. A standard reflection ($2\bar{2}\bar{2}$), checked every 50 reflections, showed no significant deviation. Lp correction. No correction for absorption. The structure was solved by direct methods using *SHELXS*86 (Sheldrick, 1985). H atoms were placed in computed positions, except that of C4 which was found from a difference Fourier synthesis. Anisotropic least-squares refinement (*SHELX*76; Sheldrick, 1976), based on F, included 176 parameters; H atoms were treated isotropically with a common refined temperature factor ($U = 0.14 \text{ Å}^2$). $R = 0.061$, $wR = 0.077$, $w = 1/(\sigma^2 + 0.0097F^2)$, $S = 1.0$ for 1864 observed reflections.

Final maximum shift to e.s.d. ratio was 0.04. Maximum and minimum heights in the final difference Fourier synthesis were 0.21 and −0.28 e Å^{-3}, respectively. Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV).

The atomic parameters are given in Table 1.* Fig. 1 is a stereoscopic view of the molecule, showing the numbering of the atoms (*PLUTO*; Motherwell & Clegg, 1978). Bond distances and angles are given in Table 2.

Related literature. Related compounds include 1-aza-2-cyclobutanone (Yang, Seiler & Dunitz, 1987), 3-chloro-3-cyano-1-cyclohexyl-4-(phenylthio)-2-aze-tidinone (Chambers & Doedens, 1980), 4,4-di-chloro-1-methyl-3,3-diphenyl-2-azetidinone (Van Meerssche, Germain, Declercq, Le Clef & Viehe, 1983), 3-chloro-1,3,4-triphenyl-2-azetidinone (Colens, Declercq, Germain, Putzeys & Van Meerssche, 1974), 4-triphenylmethylthio-2-azetidinone (Bando, Takano, Miyahara, Tanaka, Nakatsuka & Ishiguro, 1989) and L-1-benzyl-4-hydroxymethyl-2-azetidinone (Lee, Cho, Kim, Shin, Ruble & Craven, 1990).

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