Acta Cryst. (1990). C**46**, 1977–1978

Ethyl (*N*-Phenylcarbamoyl)azoformate

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(*Received* 28 *February* 1990; *accepted* 6 *April* 1990)

Abstract. $C_{10}H_{11}N_3O_3$, $M_r = 221{\cdot}24$, monoclinic, $P2_1/c$, $a = 10{\cdot}032$ (2), $b = 11{\cdot}352$ (2), $c = 9{\cdot}685$ (2) Å, $\beta = 93{\cdot}81$ (1)°, $V = 1100{\cdot}52$ Å^3, $Z = 4$, $D_x = 1{\cdot}335$, $D_m = 1{\cdot}331$ g cm^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 7{\cdot}57$ cm^{-1}, $F(000) = 464$, $T = 290$ K, photographic data measured densitometrically, $R = 0{\cdot}044$ for 1477 unique observed reflexions. The azoformamido group is planar to within 0·03 Å. There is no internal hydrogen bonding; *c*-glide related molecules are N—H···O hydrogen bonded, N···O = 2·883 (3) Å.

Table 1. *Fractional atomic coordinates* (× 10^4) *and* U_{eq} *values* (Å^2 × 10^3)

$U_{eq} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	x	*y*	*z*	U_{eq}
C(1)	3594 (3)	4208 (3)	4445 (3)	36 (2)
C(2)	2839 (4)	4509 (3)	5544 (3)	48 (3)
C(3)	1912 (4)	5396 (4)	5389 (4)	56 (4)
C(4)	1727 (4)	6009 (3)	4153 (4)	57 (3)
C(5)	2504 (4)	5720 (3)	3076 (4)	56 (4)
C(6)	3428 (4)	4835 (3)	3205 (3)	46 (3)
C(7)	4932 (3)	2618 (3)	5594 (3)	38 (3)
C(8)	7558 (3)	510 (3)	5808 (3)	46 (3)
C(9)	8286 (4)	−1343 (4)	5070 (4)	56 (4)
C(10)	8892 (5)	−1920 (5)	6351 (5)	73 (5)
N(1)	4558 (3)	3294 (2)	4511 (2)	39 (2)
N(2)	6013 (3)	1833 (3)	5185 (3)	43 (2)
N(3)	6406 (3)	1189 (3)	6146 (3)	48 (3)
O(1)	4512 (2)	2628 (2)	6735 (2)	49 (2)
O(2)	8650 (3)	892 (2)	5991 (3)	68 (2)
O(3)	7190 (2)	−552 (2)	5398 (2)	51 (2)

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

C(1)—C(2)	1·389 (4)	C(6)—C(1)—N(1)	117·0 (3)
C(2)—C(3)	1·372 (5)	N(1)—C(1)—C(2)	123·4 (3)
C(3)—C(4)	1·387 (5)	C(2)—C(1)—C(6)	119·6 (3)
C(4)—C(5)	1·383 (5)	C(1)—C(2)—C(3)	119·8 (3)
C(5)—C(6)	1·368 (5)	C(2)—C(3)—C(4)	120·9 (3)
C(6)—C(1)	1·396 (4)	C(3)—C(4)—C(5)	118·9 (4)
C(1)—N(1)	1·417 (4)	C(4)—C(5)—C(6)	121·2 (3)
N(1)—C(7)	1·334 (4)	C(5)—C(6)—C(1)	119·7 (3)
C(7)—O(1)	1·209 (3)	C(1)—N(1)—C(7)	127·8 (3)
C(7)—N(2)	1·477 (4)	N(1)—C(7)—O(1)	128·0 (3)
N(2)—N(3)	1·228 (3)	O(1)—C(7)—N(2)	123·9 (3)
N(3)—C(8)	1·446 (4)	N(2)—C(7)—N(1)	108·0 (2)
C(8)—O(2)	1·181 (4)	C(7)—N(2)—N(3)	111·1 (2)
C(8)—O(3)	1·314 (4)	N(2)—N(3)—C(8)	111·5 (3)
O(3)—C(9)	1·470 (4)	N(3)—C(8)—O(2)	121·2 (3)
C(9)—C(10)	1·496 (6)	O(2)—C(8)—O(3)	128·5 (3)
N(1)—H(1)	0·87 (4)	O(3)—C(8)—N(3)	110·2 (3)
O(1)···N(1)	2·883 (3)	O(3)—C(9)—C(10)	110·9 (3)
O(1)···H(1)	2·20 (4)		

Experimental. The material was supplied by Dr K. Dawes of the Malaysian Rubber Producers Research Association. Crystals were orange coloured laths (elongated on *c*), crystal size 0·1 × 0·3 × 0·5 mm, density by flotation.

Cell dimensions were obtained from 32 2θ values in the range 20–160°, measured on a manual four-circle diffractometer. Intensities were measured by the SERC Film Densitometry Service from equi-inclination Weissenberg photographs. Layers *h*,0,*l* to *h*,10,*l* were recorded with packs of six Agfa–Gevaert Osray M3 film (for the higher layers packs of three films sufficed). Layers *hk*0 and *hk*1 were also recorded and used for cross-scaling. 1447 unique intensities (maximum $\sin\theta/\lambda = 0{\cdot}64$ Å^{-1}, $h = -12$ to 12, $k = 0$ to 14, $l = 0$ to 12) were obtained [approximately 240 other reflexions with I(measured) ≤ 0 were considered to be unobserved], Lp corrections but no absorption corrections. An early version of *MULTAN* (Germain, Main & Woolfson, 1971) was used to solve the structure, all other calculations by *SHELX*76 (Sheldrick, 1976). H-atom positions from difference Fourier maps. Least-squares refinement (based on F) of all positions, of anisotropic U_{ij} for

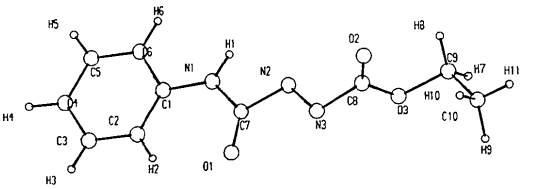

Fig. 1. The ethyl (*N*-phenylcarbamoyl)azoformate molecule with atomic labelling.

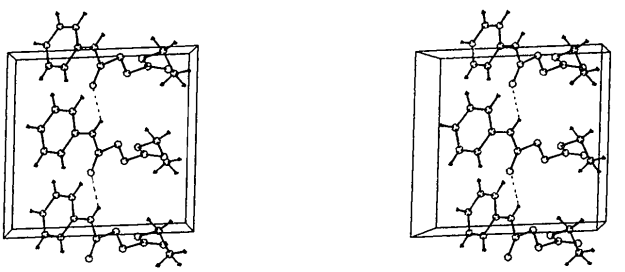

Fig. 2. Packing diagram on (010), showing hydrogen bonding and the unit cell (screw-axis-related molecules are omitted for clarity). The *a* axis is horizontal and the *c* axis vertical.

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non-H atoms and isotropic U for H atoms until $(\Delta/\sigma)_{max}$ was <0.03. Variations in the final ΔF map $+0.18$ to -0.21 e Å^{-3}. Until weights. $R = 0.044$, $wR = 0.055$, $S = 0.78$, 189 parameters, number of reflexions/number of parameters = 7.66. Scattering factors from *International Tables for X-ray Crystallography* (1974). Atomic parameters are given in Table 1,* bond distances and angles in Table 2, and the molecule, with atomic labelling, is shown in Fig. 1. Packing of the hydrogen-bonded layers is shown in Fig. 2.

* Lists of structure factors, H-atom coordinates, anisotropic vibrational factors and least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52972 (12 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Related literature. The title compound is a derivative of urea (Swaminathan, Craven & McMullan, 1984). Only one other structure containing the azoformamido group has been reported, (1-cyano-1-methyl)-ethylazoformamide (Small, 1990).

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Acta Cryst. (1990). C**46**, 1978–1979

(1-Cyano-1-methylethylazo)formamide

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(*Received* 28 *February* 1990; *accepted* 30 *April* 1990)

Abstract. $C_5H_8N_4O$, $M_r = 140.1$, triclinic, $P\bar{1}$, $a = 5.75$ (1), $b = 7.85$ (1), $c = 9.13$ (1) Å, $\alpha = 77.3$ (1), $\beta = 79.6$ (1), $\gamma = 94.2$ (1)°, $V = 392.6$ (9) Å^3, $Z = 2$, $D_x = 1.183$ g cm^{-3}, λ(Mo $K\alpha$) = 0.7107 Å, $\mu = 0.56$ cm^{-1}, $F(000) = 148$, $T = 290$ K, $R = 0.048$ for 1341 unique observed reflexions. N—H···O hydrogen bonding occurs between centrosymmetrically related pairs of molecules with N···O = 2.923 (2) Å. N—H···N hydrogen bonding occurs also between further centrosymmetrically related molecules with N···N = 3.081 (2) Å. The azoformamide group is planar to within 0.10 Å.

Experimental. Pale yellow crystals tabular on {100} were recrystallized from dichloromethane. Cell dimensions, initially from Weissenberg photographs, λ(Cu $K\alpha$) = 1.542 Å, refined from setting angles of 25 reflexions in the range $10 < 2\theta < 40°$ on STADI-2 two-circle diffractometer (graphite-monochromated Mo $K\alpha$), which was also used for measurement of 1847 intensities for the layers $0kl$ to $7kl$, standard measured every 20 reflexions, k −9 to 9, l −11 to 11 with max. $\sin\theta/\lambda$ 0.65 Å^{-1}. Variable ω scan, $2\theta'$ fixed, stationary background count. Lp correction applied but absorption correction considered unnecessary. 1665 unique intensities, 1341 of which with $I > 3\sigma(I)$ were used in the refinement. The

Table 1. *Fractional atomic coordinates and equivalent isotropic temperature factors*

$U_{eq} = (1/3)\sum_i\sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U_{eq}(Å^2)
C(1)	0.3628 (3)	0.7627 (2)	0.1408 (2)	0.055 (1)
C(2)	0.0454 (3)	0.3212 (2)	0.2896 (2)	0.051 (1)
C(3)	0.1302 (3)	0.2833 (2)	0.4369 (2)	0.053 (1)
C(4)	0.1303 (4)	0.1909 (3)	0.1936 (2)	0.068 (1)
C(5)	−0.2235 (3)	0.3131 (3)	0.3219 (3)	0.075 (1)
N(1)	0.2766 (2)	0.5858 (2)	0.2460 (1)	0.051 (1)
N(2)	0.1402 (3)	0.5017 (2)	0.1935 (2)	0.058 (1)
N(3)	0.1927 (3)	0.2486 (2)	0.5505 (2)	0.074 (1)
N(4)	0.5437 (3)	0.8364 (2)	0.1819 (2)	0.065 (1)
O	0.2731 (3)	0.8262 (2)	0.0334 (2)	0.080 (1)

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

C(1)—O	1.219 (2)	O—C(1)—N(4)	126.0 (1)
C(1)—N(4)	1.307 (2)	O—C(1)—N(1)	123.7 (1)
C(1)—N(1)	1.495 (2)	N(4)—C(1)—N(1)	110.3 (1)
N(1)—N(2)	1.217 (2)	C(1)—N(1)—N(2)	111.1 (1)
N(2)—C(2)	1.497 (2)	N(1)—N(2)—C(2)	115.3 (1)
C(2)—C(4)	1.538 (2)	N(2)—C(2)—C(3)	112.2 (1)
C(2)—C(5)	1.516 (2)	N(2)—C(2)—C(4)	106.7 (1)
C(2)—C(3)	1.490 (2)	N(2)—C(2)—C(5)	106.6 (1)
C(3)—N(3)	1.143 (2)	C(3)—C(2)—C(4)	110.3 (1)
N(4)—H(1)	0.89 (2)	C(3)—C(2)—C(5)	109.7 (1)
N(4)—H(2)	1.02 (3)	C(4)—C(2)—C(5)	111.3 (1)
O···N(4)^i	2.923 (3)	C(2)—C(3)—N(3)	177.8 (2)
O···H(2)^i	1.91 (2)		
N(3)···N(4)^{ii}	3.079 (2)		
N(3)···H(1)^{ii}	2.24 (2)		

Symmetry code: (i) $1-x, 2-y, -z$; (ii) $1-x, 1-y, 1-z$.

0108-2701/90/101978-02$03.00