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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(1-Cyano-1-methylethylazo)formamide

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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) $1-x, 1-y, 1-z$.

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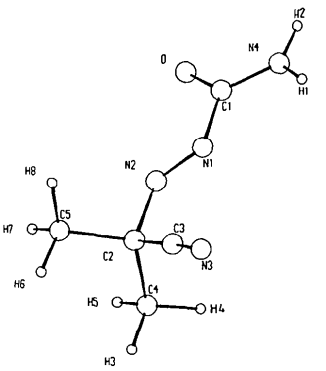

Fig. 1. Molecule of (1-cyano-1-methylethylazo)formamide with atomic labelling.

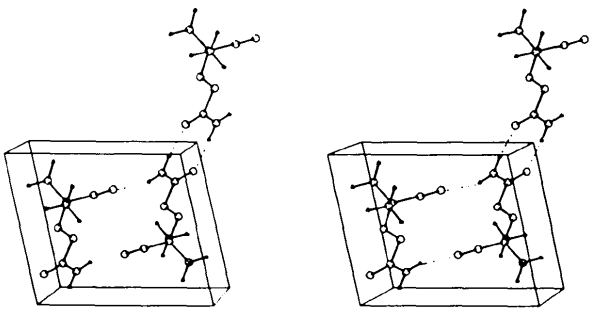

Fig. 2. Projection on (100) showing hydrogen bonding and unit cell.

structure was solved using the *EEES* direct-methods program of *SHELX*76 (Sheldrick, 1976) which was used for all other calculations. H-atom positions from ΔF map, least-squares refinement, based on F, of positions and U_{ij} of non-H atoms, of isotropic U for all H atoms and positions of H(1) and H(2) (methyl H atoms riding on parent atoms with C—H = 1·08 Å). Interlayer scale factors refined at an intermediate stage, final $\Delta/\sigma < 0{\cdot}07$. Variations in final ΔF map +0·17 to −0·17 e Å^{-3}. Final $R = 0{\cdot}048$, $wR = 0{\cdot}077$, $S = 2{\cdot}17$, 112 parameters refined, $Q = 11{\cdot}97$, $w = 2{\cdot}613/[\sigma^2(F) + 0{\cdot}00087F^2]$. Scattering factors were from *International Tables for X-ray Crystallography* (1974). Atomic parameters are given in Table 1, bond distances and angles in Table 2; the molecule with atomic labelling is shown in Fig. 1, packing of the molecules, with hydrogen bonds and unit cell in Fig. 2.*

Related literature. The title compound may be regarded as a derivative of urea (Swaminathan, Craven & McMullan, 1984). One other compound containing the azoformamide group has been reported, ethyl-*N*-phenylcarbamoylazoformate (Small, 1990).

* Lists of structure factors, H-atom coordinates, anisotropic vibrational factors and least-squares-planes data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53034 (12 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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4-Cyano-4-methyl-2,3-diazapentanamide

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Abstract. $C_5H_{10}N_4O$, $M_r = 142{\cdot}06$, monoclinic, $P2_1/c$, $a = 10{\cdot}37$ (1), $b = 5{\cdot}77$ (1), $c = 12{\cdot}72$ (1) Å, $\beta = 93{\cdot}9$ (1)°, $V = 759{\cdot}34$ Å^3, $Z = 4$, $D_x = 1{\cdot}242$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·7107 Å, $\mu = 0{\cdot}58$ mm^{-1}, $F(000) = 304$, $T = 290$ K, $R = 0{\cdot}048$ for 1501 unique observed reflexions. Hydrogen bonding occurs through centres of symmetry (N—H···O, with N···O = 2·996 Å), screw axes (N—H···O, with N···O = 2·914 Å) and glide planes (N—H···N≡C, with N···N = 3·152 Å) to form layers of molecules parallel to (100).

Experimental. Colourless crystals, 0·05 × 0·16 × 0·25 mm, which cleave parallel to (100), recrystal-

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