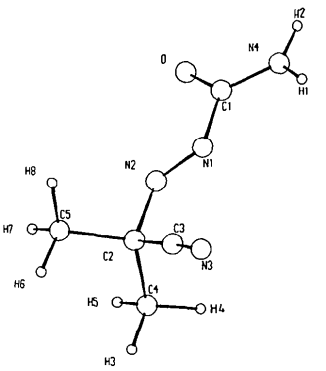


Fig. 1. Molecule of (1-cyano-1-methylethylazo)formamide with atomic labelling.

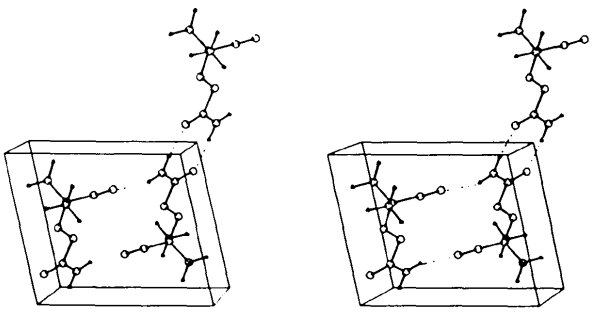

Fig. 2. Projection on (100) showing hydrogen bonding and unit cell.

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 $\Delta F$ 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 $\Delta F$ 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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*Acta Cryst.* (1990). C**46**, 1979–1980

# 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$^{-3}$, $\lambda$(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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Table 1. *Fractional atomic coordinates* ($\times 10^4$) *and* $U_{eq}$ *values* ($Å^2 \times 10^3$)

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

| | $x$ | $y$ | $z$ | $U_{eq}$ |
|---|---|---|---|---|
| C(1) | 5752 (1) | 2518 (2) | 4021 (1) | 31 (1) |
| C(2) | 7964 (1) | 6785 (2) | 3476 (1) | 37 (1) |
| C(3) | 7750 (2) | 8110 (3) | 2463 (1) | 43 (1) |
| C(4) | 8725 (2) | 4596 (3) | 3267 (2) | 63 (2) |
| C(5) | 8716 (2) | 8348 (3) | 4277 (2) | 53 (1) |
| N(1) | 6023 (1) | 4405 (2) | 3440 (1) | 37 (1) |
| N(2) | 6727 (1) | 6233 (2) | 3936 (1) | 36 (1) |
| N(3) | 7612 (2) | 9190 (3) | 1715 (1) | 67 (1) |
| N(4) | 6231 (1) | 2469 (2) | 5032 (1) | 41 (1) |
| O | 5117 (1) | 870 (2) | 3610 (1) | 40 (1) |

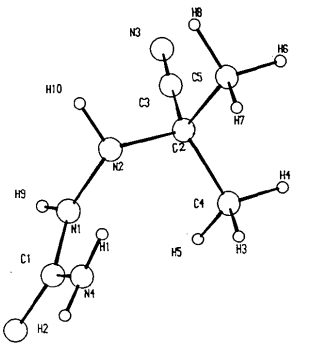


Fig. 1. The 4-cyano-4-methyl-2,3-diazapentanamide molecule with atomic labelling.

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

| | | | |
|---|---|---|---|
| C(1)—O | 1·250 (2) | O—C(1)—N(4) | 122·2 (1) |
| C(1)—N(4) | 1·347 (2) | O—C(1)—N(1) | 120·4 (1) |
| C(1)—N(1) | 1·356 (2) | N(4)—C(1)—N(1) | 117·4 (1) |
| N(1)—N(2) | 1·407 (2) | C(1)—N(1)—N(2) | 118·5 (1) |
| N(2)—C(2) | 1·480 (2) | N(1)—N(2)—C(2) | 115·0 (1) |
| C(2)—C(4) | 1·522 (2) | N(2)—C(2)—C(3) | 111·5 (1) |
| C(2)—C(5) | 1·533 (2) | N(2)—C(2)—C(4) | 111·3 (1) |
| C(2)—C(3) | 1·502 (2) | N(2)—C(2)—C(5) | 106·2 (1) |
| C(3)—N(3) | 1·138 (2) | C(3)—C(2)—C(4) | 108·7 (1) |
| N(4)—H(1) | 0·88 (2) | C(3)—C(2)—C(5) | 108·1 (1) |
| N(4)—H(2) | 0·91 (2) | C(4)—C(2)—C(5) | 111·0 (1) |
| N(1)—H(9) | 0·85 (2) | C(2)—C(3)—N(3) | 177·2 (2) |
| N(2)—H(10) | 0·94 (2) | C(1)—N(4)—H(1) | 120 (1) |
| O···N(4$^{i}$) | 2·996 (3) | C(1)—N(4)—H(2) | 116 (1) |
| O···H(2$^{i}$) | 2·10 (3) | H(1)—N(4)—H(2) | 121 (2) |
| O···N(1$^{ii}$) | 2·914 (3) | H(9)—N(1)—C(1) | 117 (1) |
| O···H(9$^{ii}$) | 2·10 (3) | H(9)—N(1)—N(2) | 123 (1) |
| N(3$^{iii}$)···N(4) | 3·152 (3) | H(10)—N(2)—C(2) | 107 (1) |
| N(3$^{iii}$)···H(1) | 2·35 (3) | H(10)—N(2)—N(1) | 110 (1) |

Symmetry code: (i) $1-x,-y,1-z$; (ii) $1-x,-\frac{1}{2}+y,\frac{1}{2}-z$; (iii) $x,1\frac{1}{2}-y,\frac{1}{2}+z$.

lized from methanol. Cell dimensions initially from Weissenberg photographs, $\lambda$(Cu $K\alpha$) = 1·542 Å, refined from setting angles of 25 reflexions on STADI-2 two-circle diffractometer (graphite-monochromatized Mo $K\alpha$ radiation). STADI-2 also used for measurement of 2047 intensities for the layers *h*0*l* to *h*7*l*, standard measured every 20 reflexions, maximum $\sin\theta/\lambda$ 0·65 $Å^{-1}$. Lp correction applied but no absorption correction. 1792 unique intensities of which 1501 with $I > 3\sigma(I)$ used in the refinement. All calculations including structure solution (*EEES*) using *SHELX*76 (Sheldrick, 1976). H-atom positions from $\Delta F$ map, least-squares refinement (based on $F$) of positions and $U_{ij}$ of all non-H atoms, of individual isotropic $U$ of all H atoms, of positions of H(1), H(2), H(9) and H(10) (methyl H riding on parent atoms with C—H = 1·08 Å). Interlayer scale factors refined at an intermediate stage, largest $\Delta/\sigma = 0{\cdot}002$, largest variation in final $\Delta F$ map ±0·19 e $Å^{-3}$. Final $R = 0{\cdot}048$, $wR = 0{\cdot}064$, $S = 2{\cdot}41$, $w = 3{\cdot}738/[\sigma^2(F) + 0{\cdot}000317F^2]$.

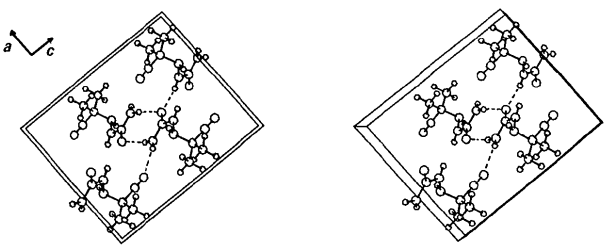


Fig. 2. Molecular packing with unit cell on (010), showing hydrogen bonding.

Atomic 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. Molecular packing is shown in Fig. 2.

**Related literature.** The title compound may be compared with urea (Swaminathan, Craven & McMullan, 1984) and is a reduced form of (1-cyano-1-methyl)ethylazoformamide (Small, 1990).

* Lists of structure factors, H-atom coordinates and anisotropic vibrational factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52973 (10 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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