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Acta Cryst. (1993). **C49**, 354–355

1,3-Dicyanomethylhexahydropyrimidine

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(Received 4 October 1991; accepted 19 May 1992)

Abstract. Hexahydropyrimidine-1,3-diacetonitrile, C₈H₁₂N₄, *M_r* = 164.21, monoclinic, *P*2₁/*c*, *a* = 8.303 (1), *b* = 8.733 (1), *c* = 12.998 (2) Å, β = 107.73 (2)°, *V* = 897.7 (4) Å³, *Z* = 4, *D_x* = 1.21 g cm⁻³, Cu *Kα* radiation, λ = 1.5418 Å, μ = 6.4 cm⁻¹, *F*(000) = 352, *T* = 293 K, final *R* = 0.035 (*wR* = 0.050) for 1005 observed reflections. The pyrimidine ring adopts the chair conformation with C(7) and C(9) in equatorial positions.

Experimental. The title compound (Fig. 1) was prepared by dropwise addition of a saturated aqueous solution of potassium cyanide (2 equivalents) to an ice-bath-cooled aqueous solution of formaldehyde (3 equivalents) containing 1,3-propanediamine dihydrochloride (1 equivalent). The aqueous solution was extracted several times with ether and the combined ether layers were dried. The solvent was then removed by distillation at reduced pressure and the residue crystallized from absolute ethanol. Data were collected on an Enraf–Nonius CAD-4 diffractometer with graphite monochromator, for a crystal of dimensions 0.25 × 0.25 × 0.30 mm. Cell parameters were measured on the diffractometer using 25 reflections in the 2θ range 20–40°. Data were collected over range of indices $-9 \leq h \leq 9$, $0 \leq k \leq 9$, $0 \leq l \leq 14$ ($\theta \leq 60^\circ$). Three standards (110, 022 and 102), measured after every 200 reflections, showed a variation of 0.1%. No absorption corrections but Lorentz and polarization corrections were applied. 1333 unique (*R_{int}* = 0.013) reflections were measured, of which 1005 were observed with $|F_o| > 3\sigma(|F_o|)$. Direct methods (*MULTAN82*; Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982) were used for structure determination. H atoms were located by difference Fourier synthesis. Anisotropic full-matrix least-squares refinement (on *F*) was performed for non-H atoms, isotropic for H atoms. In the last cycle the H atoms were fixed at idealized

Table 1. Final fractional coordinates and equivalent isotropic temperature factors (Å²) for non-H atoms with *e.s.d.*'s in parentheses

$$B_{eq} = \frac{4}{3} \sum_i \sum_j \beta_{ij} a_i \cdot a_j$$

| | <i>x</i> | <i>y</i> | <i>z</i> | <i>B_{eq}</i> |
|-------|------------|------------|------------|-----------------------|
| N(1) | 0.5997 (2) | 0.1089 (2) | 0.4213 (1) | 3.30 (3) |
| N(2) | 1.0267 (3) | 0.3770 (2) | 0.2759 (1) | 7.43 (6) |
| N(3) | 0.8891 (2) | 0.1775 (2) | 0.4632 (1) | 3.40 (3) |
| N(4) | 0.3418 (2) | 0.1822 (2) | 0.1619 (1) | 5.96 (5) |
| C(2) | 0.7417 (2) | 0.1249 (2) | 0.3781 (1) | 3.26 (3) |
| C(4) | 0.8595 (2) | 0.3275 (2) | 0.5048 (1) | 3.88 (4) |
| C(5) | 0.7066 (2) | 0.3201 (2) | 0.5449 (1) | 4.29 (4) |
| C(6) | 0.5558 (2) | 0.2569 (2) | 0.4583 (1) | 3.92 (4) |
| C(7) | 1.0385 (2) | 0.1755 (2) | 0.4280 (2) | 4.26 (4) |
| C(8) | 1.0339 (2) | 0.2874 (2) | 0.3406 (2) | 4.69 (4) |
| C(9) | 0.4559 (2) | 0.0342 (2) | 0.3453 (1) | 3.82 (4) |
| C(10) | 0.3882 (2) | 0.1173 (2) | 0.2408 (1) | 4.05 (4) |

Table 2. Bond lengths (Å), bond angles (°) and selected torsion angles (°) with *e.s.d.*'s in parentheses

| | | | |
|---------------------|-----------|---------------------|-----------|
| N(1)–C(2) | 1.459 (2) | N(3)–C(7) | 1.447 (3) |
| N(1)–C(6) | 1.464 (2) | N(4)–C(10) | 1.132 (2) |
| N(1)–C(9) | 1.452 (2) | C(4)–C(5) | 1.513 (3) |
| N(2)–C(8) | 1.137 (3) | C(5)–C(6) | 1.510 (2) |
| N(3)–C(2) | 1.452 (2) | C(7)–C(8) | 1.490 (3) |
| N(3)–C(4) | 1.467 (2) | C(9)–C(10) | 1.490 (2) |
| C(2)–N(1)–C(6) | 110.7 (1) | N(3)–C(4)–C(5) | 109.9 (1) |
| C(2)–N(1)–C(9) | 112.0 (1) | C(4)–C(5)–C(6) | 110.6 (2) |
| C(6)–N(1)–C(9) | 112.6 (1) | N(1)–C(6)–C(5) | 109.5 (1) |
| C(2)–N(3)–C(4) | 111.5 (1) | N(3)–C(7)–C(8) | 114.1 (2) |
| C(2)–N(3)–C(7) | 111.1 (1) | N(2)–C(8)–C(7) | 177.0 (2) |
| C(4)–N(3)–C(7) | 112.5 (1) | N(1)–C(9)–C(10) | 113.9 (2) |
| N(1)–C(2)–N(3) | 109.2 (1) | N(4)–C(10)–C(9) | 177.8 (2) |
| C(6)–N(1)–C(2)–N(3) | 62.4 (2) | C(2)–N(3)–C(4)–C(5) | 57.0 (2) |
| C(2)–N(1)–C(6)–C(5) | –59.3 (2) | N(1)–C(6)–C(5)–C(4) | –54.5 (2) |
| C(4)–N(3)–C(2)–N(1) | –61.2 (2) | N(3)–C(4)–C(5)–C(6) | –53.2 (2) |

positions (0.94–1.06 Å). $\sum w(|F_o| - |F_c|)^2$ was minimized, where the non-Poisson $w = 4F^2/[\sigma(I)^2 + (pF^2)^2]$; $p = 0.04$, $R = 0.035$, $wR = 0.050$, $S = 1.650$, for 158 variables. Maximum $\Delta/\sigma = 0.01$. Maximum peak height in the final difference Fourier map was 0.24 e Å⁻³. Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). Computing used the Enraf–Nonius *SDP* (Frenz, 1984). Atomic parameters are given in

Table 1;* the bond distances, bond angles and relevant torsion angles are presented in Table 2. Atomic numbering is shown in Fig. 1, and the packing diagram is presented in Fig. 2.

Related literature. The torsion angles around the hexahydropyrimidine ring alternate in sign and range from 53.2 (2) to 62.4 (2) $^{\circ}$ (Table 2) with a mean value of 57.9 (2) $^{\circ}$. This value is the same as that found for the tetrahydro-1,3-oxazine ring (Shoja & Saba, 1991). A mean value of 55.9 $^{\circ}$ was observed for cyclohexane (Clark & McKerver, 1979). The cyanomethyl groups are perpendicular to the mean plane of the heterocyclic ring with an average dihedral angle of 88.9 (1) $^{\circ}$.

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and least-squares-planes data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55459 (19 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: CR0398]

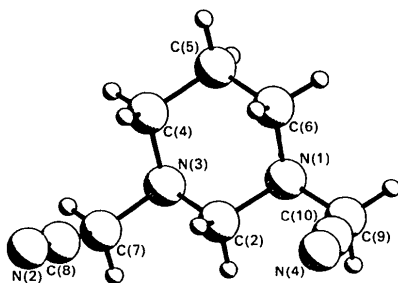


Fig. 1. Numbering of atoms and conformation of the molecule.

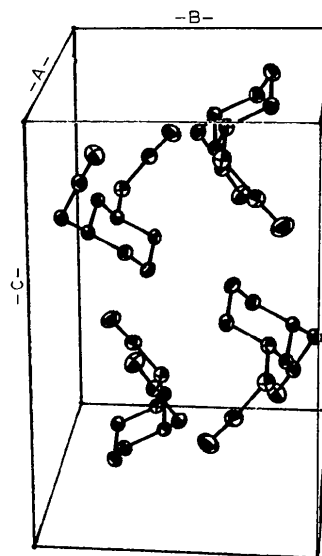


Fig. 2. Molecular packing diagram.

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Acta Cryst. (1993). C49, 355–357

Structure of Orthorhombic Triphenylphosphine Oxide: a Redetermination at Room Temperature*

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(Received 17 March 1992; accepted 8 June 1992)

Abstract. C₁₈H₁₅OP, $M_r = 278.3$, orthorhombic, *Pbca*, $a = 29.085$ (15), $b = 9.149$ (4), $c = 11.266$ (5) Å, $V = 2997.9$ Å³, $Z = 8$, $D_x = 1.233$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 1.170$ cm⁻¹, $F(000) = 1168$, $T =$

295 K, $R = 0.048$ for 1156 observed reflections. The P—O and three P—C bond lengths are 1.487 (3), and 1.795 (5), 1.799 (5) and 1.804 (5) Å, respectively. The valence angles at P fall into two categories: the three O—P—C angles, 111.8 (2), 112.0 (2) and 113.3 (2) $^{\circ}$, and the three C—P—C angles, each significantly smaller at 106.4 (2) $^{\circ}$.

* Contribution from the Crystallography Unit, Universities of Aston and Birmingham, England.