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Structure of 2,2,6,6-Tetramethyl-4-oxaheptanedinitrile

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Abstract

The title molecule, $C_{10}H_{16}N_2O$ (I), has approximate mirror symmetry with the main chain fully extended. The main torsion angles defining the molecular conformation are $O—C_{sp^3}—C_{sp^3}—C_{sp}$ 58.9 (4) and −60.5 (4)°. The mean principal dimensions include $N≡C_{sp}$ 1.13 (1), $C_{sp}—C_{sp^3}$ 1.46 (1) and $C_{sp^3}—O$ 1.410 (6) Å. The two nitrile groups are oriented *cis* to one another [$N≡C_{sp}···C_{sp}≡N$ 4.4 (5)°] and the intramolecular N···N separation is 4.70 (1) Å.

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Comment

The structural determination of 2,2,6,6-tetramethyl-4-oxaheptanedinitrile (I) was undertaken as part of a research program directed towards the development of new macrocyclic ligands for use in the synthesis of transition-metal complexes (Ferguson, McAlees, McCrindle, Restivo & Roberts, 1977; McCrindle, Ferguson, McAlees, Parvez, Ruhl, Stephenson & Wieckowski, 1986; McCrindle, Ferguson, McAlees, Parvez & Stephenson, 1982, 1983; McCrindle, Ferguson, McAlees & Roberts, 1981; McCrindle, Ferguson & Parvez, 1984; McAlees & McCrindle, 1981).

The crystal structure determination establishes the overall conformation and the orientation of the two nitrile moieties in the solid state, Fig. 1. (I) has pseudo-mirror symmetry with the mirror plane passing through the central ether O atom; the two nitrile groups are oriented *cis* and are almost parallel to one another [$N≡C_{sp}···C_{sp}≡N$ 4.4 (5)°], and to the *a* axis. The angle between the C32— and C72—C_{sp^3}—$C_{sp}≡N$ planes is 2.6 (6)°. The principal dimensions include: $N≡C_{sp}$ 1.127 (10) and 1.129 (10) Å; $C_{sp}—C_{sp^3}$ 1.464 (10) and 1.467 (10) Å; $C_{sp^3}—C_{sp^3}$ in the range 1.506 (8)–1.532 (8) Å with a mean value of 1.512 (7) Å; $C_{sp^3}—O$ 1.409 (6) and 1.412 (6) Å. The main torsion angles defining the molecular conformation are $O—C_{sp^3}—C_{sp^3}—C_{sp}$ 58.9 (4) and −60.5 (4)°. The distance between N1 and N9 is 4.70 (1) Å, and this makes the molecule a potentially useful bridging ligand in metal-cluster chemistry. Full details of other molecular dimensions are available as supplementary material.
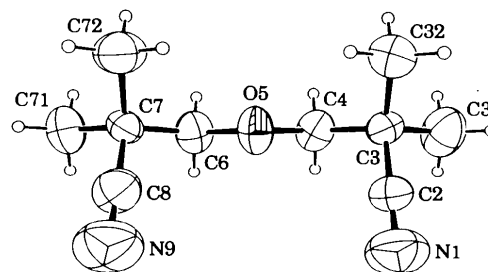

Fig. 1. An *ORTEP*II view of molecule (I), with the non-H atoms shown with their thermal ellipsoids drawn at the 50% probability level. For clarity, the H atoms are drawn as spheres of arbitrary size.

Experimental

Crystal data

$C_{10}H_{16}N_2O$	D_x = 1.04 Mg m^{-3}
M_r = 180.25	Mo $K\alpha$ radiation
Monoclinic	λ = 0.71073 Å
$P2_1/c$	Cell parameters from 25 reflections
a = 6.6495 (4) Å	θ = 7.5–15.6°
b = 9.8627 (7) Å	μ = 0.064 mm^{-1}
c = 17.6172 (12) Å	T = 293 K
β = 91.286 (5)°	Block

$V = 1155.1$ (1) $\mathring{A}^3$

$Z = 4$

0.30 × 0.15 × 0.15 mm

Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer

$\omega/2\theta$ scans

Absorption correction: none

2196 measured reflections

2021 independent reflections

588 observed reflections [$I_{net} > 2.0\sigma(I_{net})$]

$R_{int} = 0.01$

$\theta_{max} = 25.0°$

$h = -7 \rightarrow 7$

$k = 0 \rightarrow 11$

$l = 0 \rightarrow 20$

3 standard reflections frequency: 60 min intensity variation: 33% decay during data collection

Refinement

Refinement on F

$R = 0.050$

$wR = 0.042$

$S = 1.30$

588 reflections

119 parameters

H atoms refined as riding, C—H 0.95 $\mathring{A}$

$w = 1/[\sigma^2(F) + 0.0004F^2]$

$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.15$ e $\mathring{A}^{-3}$

$\Delta\rho_{min} = -0.17$ e $\mathring{A}^{-3}$

Extinction correction: Larson (1970)

Extinction coefficient: 9798 (1598)

Atomic scattering factors from International Tables for X-ray Crystallography (1974, Vol. IV, Table 2.2B)

Table 1. Fractional atomic coordinates and equivalent isotropic thermal parameters ($\mathring{A}^2$)

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

	x	y	z	U_{eq}
N1	0.1872 (8)	0.5964 (6)	0.1899 (3)	0.094 (4)
N9	0.1592 (9)	0.2151 (7)	0.0299 (4)	0.111 (5)
C2	0.0185 (12)	0.5934 (7)	0.1958 (3)	0.061 (5)
C3	−0.2004 (9)	0.5938 (5)	0.2030 (3)	0.051 (4)
C31	−0.2694 (9)	0.7393 (5)	0.2181 (3)	0.087 (4)
C32	−0.2564 (9)	0.5017 (5)	0.2673 (3)	0.078 (4)
C4	−0.2957 (8)	0.5463 (5)	0.1283 (3)	0.059 (4)
O5	−0.2236 (5)	0.4149 (3)	0.1124 (2)	0.058 (2)
C6	−0.3034 (9)	0.3607 (5)	0.0441 (3)	0.060 (4)
C7	−0.2303 (9)	0.2171 (5)	0.0347 (3)	0.047 (4)
C71	−0.3152 (9)	0.1600 (5)	−0.03982 (24)	0.077 (4)
C72	−0.2950 (9)	0.1295 (5)	0.1009 (3)	0.077 (4)
C8	−0.0100 (12)	0.2177 (6)	0.0320 (4)	0.066 (5)

The crystal decomposed by 33% during data collection and this was allowed for in the data reduction by using a five-point smoothing correction in the scaling of the data. The H atoms, visible in the difference maps at an intermediate stage of refinement, were positioned geometrically and included as riding atoms in the structure-factor calculations. Data collection: *CAD-4 Software* (Enraf-Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: *NRCVAX DATRD2* (Gabe, Le Page, Charland, Lee & White, 1989). Program(s) used to solve structure: *NRCVAX SOLVER*. Program(s) used to refine structure: *NRCVAX LSTSQ*. Molecular graphics: *NRCVAX; ORTEPII* (Johnson, 1976). Software used to prepare material for publication: *NRCVAX TABLES*.

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Structures of 4-Hydroxyimino-4-phenylbutanoic Acid, $C_{10}H_{11}NO_3$ (I), and 5-Hydroxyimino-5-phenylpentanoic Acid, $C_{11}H_{13}NO_3$ (II), at 223 K

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Abstract

Infinite polar chains of molecules with strong intermolecular hydrogen bonds (O—H···N and O—H···O) are observed between carboxyl and oxime