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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 [$N\equiv C_{sp}\cdots C_{sp}\equiv N$ 4.4 (5)°], and to the *a* axis. The angle between the C32— and C72— C_{sp^3} — $C_{sp}\equiv N$ planes is 2.6 (6)°. The principal dimensions include: $N\equiv C_{sp}$ 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.

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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 $N\equiv C_{sp}$ 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 [$N\equiv C_{sp}\cdots C_{sp}\equiv N$ 4.4 (5)°] and the intramolecular $N\cdots N$ separation is 4.70 (1) Å.

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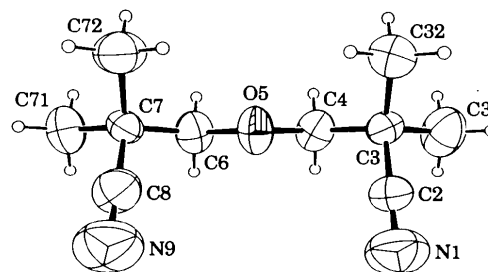


Fig. 1. An ORTEP view of molecule (I), with the non-H atoms shown with their thermal ellipsoids drawn at the 50% probability level. For clarity, the H atoms are drawn as spheres of arbitrary size.

Experimental

Crystal data

$C_{10}H_{16}N_2O$
 $M_r = 180.25$
Monoclinic
 $P2_1/c$
 $a = 6.6495$ (4) Å
 $b = 9.8627$ (7) Å
 $c = 17.6172$ (12) Å
 $\beta = 91.286$ (5)°

$D_x = 1.04$ Mg m $^{-3}$
Mo $K\alpha$ radiation
 $\lambda = 0.71073$ Å
Cell parameters from 25 reflections
 $\theta = 7.5$ – 15.6 °
 $\mu = 0.064$ mm $^{-1}$
 $T = 293$ K
Block

$V = 1155.1 (1) \text{ \AA}^3$	$0.30 \times 0.15 \times 0.15 \text{ mm}$
$Z = 4$	Colourless
Data collection	
Enraf-Nonius CAD-4 diffractometer	$R_{\text{int}} = 0.01$
$\omega/2\theta$ scans	$\theta_{\text{max}} = 25.0^\circ$
Absorption correction: none	$h = -7 \rightarrow 7$
2196 measured reflections	$k = 0 \rightarrow 11$
2021 independent reflections	$l = 0 \rightarrow 20$
588 observed reflections	3 standard reflections
$[I_{\text{net}} > 2.0\sigma(I_{\text{net}})]$	frequency: 60 min
	intensity variation: 33%
	decay during data collection

Refinement

Refinement on F	$\Delta\rho_{\text{max}} = 0.15 \text{ e \AA}^{-3}$
$R = 0.050$	$\Delta\rho_{\text{min}} = -0.17 \text{ e \AA}^{-3}$
$wR = 0.042$	Extinction correction: Larson (1970)
$S = 1.30$	Extinction coefficient: 9798 (1598)
588 reflections	Atomic scattering factors from <i>International Tables for X-ray Crystallography</i> (1974, Vol. IV, Table 2.2B)
119 parameters	
H atoms refined as riding, C—H 0.95 \AA	
$w = 1/[\sigma^2(F) + 0.0004F^2]$	
$(\Delta/\sigma)_{\text{max}} < 0.001$	

Table 1. Fractional atomic coordinates and equivalent isotropic thermal parameters (\AA^2)
$$U_{\text{eq}} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	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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Lists of structure factors, anisotropic thermal parameters, H-atom coordinates, torsion angles and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71475 (8 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: MU1064]

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Structures of 4-Hydroxyimino-4-phenylbutanoic Acid, C₁₀H₁₁NO₃ (I), and 5-Hydroxyimino-5-phenylpentanoic Acid, C₁₁H₁₃NO₃ (II), at 223 K

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Abstract

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