used to prepare material for publication: *PARST* (Nardelli, 1983*b*).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: NA1323). Services for accessing these data are described at the back of the journal.

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4-Acetyl-4-methylheptanedinitrile at 173 K

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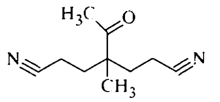
Abstract

The title compound, $C_{10}H_{14}N_2O$, serves as an precursor for ring-closure reactions to afford, for example, naphthyridines and quinolinediones. In the crystal, the molecule does not show any pre-organization favouring a ring closure; the heptanedinitrile chain is almost anti-peri-planar, with the C—C—C angles having nearly perfect tetrahedral values.

Comment

γ-Methylpimelonitrile and its derivatives can be used in cyclization reactions, with inorganic acids as catalysts, resulting in tetrahydropyridine derivates. Zondler & Pfleiderer (1972) showed that decahydro-1,8-naphthyridines can be synthesized, while Richtzenhain *et al.* (1966) synthesized substituted 2,3-dimethyl-3-(γ-aminopropyl)piperidine after reduction of the nitriles with Raney nickel and hydrogen. Koelsch & Walker (1950) found that Δ^8-octahydroquinoline-2,7-dione could be obtained by reaction with concentrated sulfuric acid at 393–413 K. We were interested in the question of whether the precursor of the above-mentioned reactions is predisposed towards ring closure. However, the crystal structure analysis revealed that all torsion angles of the heptanedinitrile chain are antiperiplanar. The molecule, (I), exhibits approximate non-crystallographic *m* symmetry. Atoms C1, C2, O2, C3 and C4 lie in a common plane (r.m.s. deviation = 0.001 Å) and the torsion angles of the remaining atoms have similar absolute values, differing only in sign.



(I)

Bond lengths and angles in (I) do not show unusual values. There were no additional crystallographic data for similar compounds in the Cambridge Structural Database (Allen & Kennard, 1993).

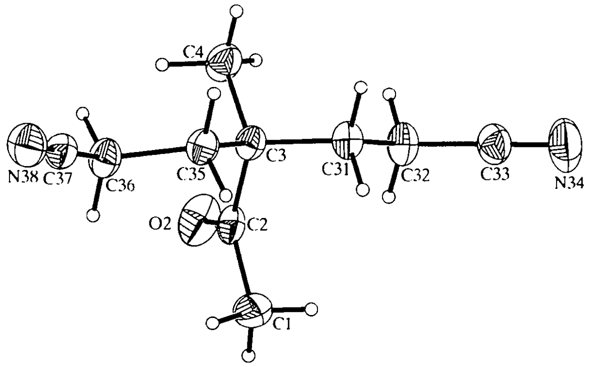


Fig. 1. Perspective view of the title compound with the atom numbering; displacement ellipsoids are at the 50% probability level.

Experimental

The title compound was obtained by dissolving 2-butanone (0.1 mol) in *tert*-butanol (20 ml) and adding this solution dropwise to 30% methanolic KOH (0.5 ml) with stirring and ice cooling. Afterwards, acrylonitrile (0.2 mol) in *tert*-butanol (20 ml) was added dropwise to the reaction mixture while keeping the temperature of the mixture under 263 K, and the resulting mixture stirred for 30 min. Petroleum ether (10 ml) was then added and the precipitate filtered off. Suitable crystals were obtained by recrystallization from petroleum ether.

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Crystal data

$C_{10}H_{14}N_2O$
$M_r = 178.23$
Monoclinic
$P2_1/n$
$a = 8.2790\,(1)$ Å
$b = 13.2031\,(1)$ Å
$c = 9.6525\,(1)$ Å
$\beta = 102.852\,(1)°$
$V = 1028.667\,(18)$ Å^3
$Z = 4$
$D_x = 1.151$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 6792 reflections
$\theta = 1–25°$
$\mu = 0.076$ mm^{-1}
$T = 173$ K
Block
$0.90 \times 0.40 \times 0.40$ mm
Colourless

Data collection

Siemens CCD three-circle diffractometer
ω scans
Absorption correction: none
7842 measured reflections
2100 independent reflections
1916 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.019$
$\theta_{\max} = 26.39°$
$h = -10 \rightarrow 10$
$k = -16 \rightarrow 16$
$l = -11 \rightarrow 12$
260 standard reflections
frequency: 1200 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.108$
$S = 1.076$
2100 reflections
119 parameters
H atoms: see below
$w = 1/[\sigma^2(F_o^2) + (0.0493P)^2 + 0.3147P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.218$ e Å^{-3}
$\Delta\rho_{\min} = -0.171$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

C2—O2	1.2143 (15)	C36—C37	1.4637 (16)
C32—C33	1.4626 (17)	C37—N38	1.1417 (16)
C33—N34	1.1371 (18)		
N34—C33—C32	178.59 (14)	N38—C37—C36	178.92 (14)
C2—C3—C31—C32	57.06 (13)	C3—C31—C32—C33	−178.38 (10)
C4—C3—C31—C32	−64.65 (13)	C31—C32—C33—N34	24 (6)
C35—C3—C31—C32	175.19 (10)	C35—C36—C37—N38	46 (7)

The data collection nominally covered over a sphere of reciprocal space, by a combination of seven sets of exposures; each set had a different φ angle for the crystal and each exposure covered 0.3° in ω. The crystal-to-detector distance was 5.98 cm. Coverage of the unique set is over 97% complete to at least 26° in θ. Crystal decay was monitored by repeating the initial frames at the end of data collection and analysing the duplicate reflections. H atoms were located by difference Fourier synthesis and refined with fixed individual displacement parameters $[U(\text{H}) = 1.5U_{\text{eq}}(\text{C}_{\text{methyl}})$ or $1.2U_{\text{eq}}(\text{C})]$ using a riding model with methyl C—H = 0.98 or secondary C—H = 0.99 Å. The C1 methyl group was allowed to rotate about its local threefold axis.

Data collection: *SMART* (Siemens, 1995). Cell refinement: *SMART*. Data reduction: *SAINT* (Siemens, 1995). Program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997). Molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 1991).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: NA1367). Services for accessing these data are described at the back of the journal.

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3,3′-(1-Silacyclohexane-1,1-diyl)bis(1-methylimidazolium) Dibromide Acetonitrile Solvate at 173 K

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Abstract

The title compound, $C_{13}H_{22}N_4Si^{2+}.2Br^-.C_2H_3N$, crystallizes from the solution of *N*-methylimidazole and 1,1-dibromosilacyclohexane in which it was prepared. Two Br^- ions of the starting material are removed from the first area of coordination of the Si atom. The resulting species is a dication. The structure displays a six-membered silaheterocycle containing a tetracoordinated Si atom. The crystal was a racemic twin.

Comment

Recently, we reported the synthesis and structure determination of tetrakis(*N*-methylimidazole)silacyclopentane dichloride (Hensen *et al.*, 1997), the first Lewis acid–base adduct consisting of a silaheterocycle and *N*-methylimidazole (NMI). However, the homologous

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