

Structure of Iminodiacetonitrile

BY SATOSHI KAIDA

Materials Characterization Department, Matsushita Technoresearch Inc., 3–15 Yagumo-Nakamachi, Moriguchi, Osaka 570, Japan

HISASHI MINEMOTO, TOKIHIKO SHIMIZU AND NOBUO SONODA

Central Research Laboratories, Matsushita Electric Industrial Co. Ltd, 3–15 Yagumo-Nakamachi, Moriguchi, Osaka 570, Japan

AND KUNIO MIKI* AND NOBUTAMI KASAI

Department of Applied Chemistry, Faculty of Engineering, Osaka University, Yamadaoka, Suita, Osaka 565, Japan

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Abstract. $C_4H_5N_3$, $M_r = 95.10$, monoclinic, $P2_1$, $a = 6.494$ (2), $b = 7.821$ (2), $c = 5.345$ (2) Å, $\beta = 114.31$ (2)°, $U = 247.4$ (1) Å³, $Z = 2$, $D_x = 1.277$ g cm⁻³, $\lambda(\text{Cu } K\alpha) = 1.5418$ Å, $\mu = 7.24$ cm⁻¹, $F(000) = 100$, $T = 295$ K, $R = 0.051$ for 390 observed reflections. The molecule has approximate C_s symmetry, the imino N(1) atom being located in the mirror plane. The bond distances and angles related by this mirror symmetry are in good agreement with each other.

Experimental. Crystals were grown as colorless thin plates by stepped recrystallization from acetone solution. A well-shaped crystal with dimensions of 0.50 × 0.40 × 0.10 mm was mounted on a Rigaku automated four-circle diffractometer. Unit-cell parameters were determined by a least-squares fit of 2θ values of 25 reflections in the range 44.9–69.3°. Intensities were measured by the ω - 2θ scan technique using Ni-filtered Cu $K\alpha$ radiation. The scan rate was 4° min⁻¹ in 2θ and the scan width was $\Delta(2\theta) = (2.2 + 0.30 \tan \theta)^\circ$. Background intensities were measured for 5 s at the end of each scan. Four standard reflections (001, 340, 040, 400) were remeasured every 100 reflections, no significant loss of intensity being observed. 816 reflections were collected with 2θ up to 120° ($\sin \theta / \lambda = 0.562$ Å⁻¹) and index range $h = -7$ to 7, $k = -8$ to 8, $l = 0$ to 6. By averaging Friedel-pair reflections, 399 independent reflections were obtained ($R_{\text{int}} = 0.017$). Corrections for Lorentz and polarization effects were applied, but neither absorption nor extinction corrections were carried out.

The structure was solved by direct methods using *SHELXS86* (Sheldrick, 1986) and refined by the block-diagonal least-squares procedure using the *HBLVS* program (Ashida, 1979) using 390 observed reflections [$|F_o| \geq 3\sigma(|F_o|)$]. On the difference Fourier maps all the H atoms were found at the expected positions, and were included in further refinement. The function minimized was $\sum w(|F_o| - |F_c|)^2$, where $w = [\sigma(F_o)^2 + 0.0010|F_o|^2]^{-1}$. The

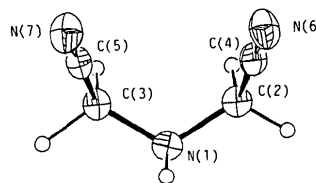


Fig. 1. ORTEP (Johnson, 1976) drawing of the molecular structure together with atomic numbering system. Non-H atoms are represented by thermal ellipsoids with 30% probability levels, whereas H atoms are drawn as spheres with $B = 1.0$ Å².

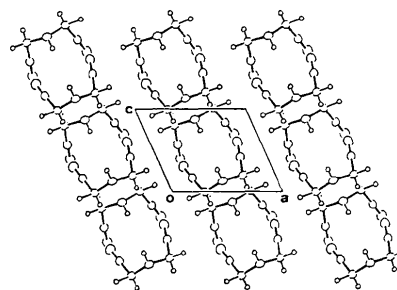


Fig. 2. ORTEP (Johnson, 1976) drawing of the crystal packing as viewed along the b axis. Atoms are represented as in Fig. 1. No abnormally short intermolecular contacts are observed.

* To whom correspondence should be addressed.

Table 1. Fractional atomic coordinates and equivalent isotropic thermal parameters with e.s.d.'s in parentheses

	x	y	z	B_{eq}^*/B^{\dagger} (\AA^2)
N(1)	0.5001 (3)	0.2500 (4)	0.1276 (4)	3.5
C(2)	0.7074 (4)	0.3449 (3)	0.1989 (5)	3.5
C(3)	0.2925 (4)	0.3442 (4)	-0.0071 (5)	3.5
C(4)	0.7580 (4)	0.4721 (4)	0.4226 (6)	3.8
C(5)	0.2420 (4)	0.4714 (4)	0.1648 (5)	3.7
N(6)	0.7926 (4)	0.5646 (4)	0.5998 (5)	5.0
N(7)	0.2054 (4)	0.5642 (4)	0.3058 (6)	5.2
H(1)	0.496 (4)	0.201 (5)	0.264 (8)	3.4†
H(2A)	0.855 (5)	0.275 (4)	0.261 (7)	3.5†
H(2B)	0.693 (6)	0.406 (4)	0.050 (8)	3.5†
H(3A)	0.129 (5)	0.256 (4)	-0.078 (7)	3.5†
H(3B)	0.294 (6)	0.405 (4)	-0.149 (7)	3.5†

* As defined by Hamilton (1959).

Table 2. Bond distances (\AA) and bond angles ($^{\circ}$) with e.s.d.'s in parentheses

N(1)—C(2)	1.444 (4)	N(1)—C(3)	1.442 (4)
C(2)—C(4)	1.485 (4)	C(3)—C(5)	1.480 (4)
C(4)—N(6)	1.139 (4)	C(5)—N(7)	1.139 (5)
C(2)—N(1)—C(3)	116.6 (3)	N(1)—C(2)—C(4)	115.5 (3)
N(1)—C(3)—C(5)	115.7 (3)	C(2)—C(4)—N(6)	176.9 (4)
C(3)—C(5)—N(7)	177.0 (4)		

ing diagram are depicted in Figs. 1 and 2, respectively. Bond distances and angles are presented in Table 2.

All computations were performed on an ACOS 930 computer at the Research Center for Protein Engineering, Institute for Protein Research, Osaka University.

Related literature. A modified and improved preparation of the title compound has recently been reported (Micovic & Ivanovic, 1986).

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number of observations per refined parameter is $390/85 = 4.59$ and $S = 2.17$. The final R and wR values are 0.051 and 0.066, respectively. $(\Delta/\sigma)_{\max}$ of non-H atoms in the final refinement cycle is 0.029. The peaks in the final $\Delta\rho$ map were between 0.35 and -0.17 e \AA^{-3} . The atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). The final atomic parameters are listed in Table 1.* The molecular structure with atomic numbering system and the crystal pack-

* Lists of anisotropic temperature factors for non-H atoms and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53031 (5 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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N-Methyl-N-(2-phenyl-4-oxocyclobutyl)-p-toluenesulfonamide

BY B. TINANT AND J.-P. DECLERCQ

Laboratoire de Chimie Physique et de Cristallographie, Université Catholique de Louvain,
1 place Louis Pasteur, 1348 Louvain la Neuve, Belgium

AND B. GOBEAUX

Laboratoire de Chimie Organique de Synthèse, Université Catholique de Louvain, 1 place Louis Pasteur,
1348 Louvain la Neuve, Belgium

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Abstract. $\text{C}_{18}\text{H}_{19}\text{NO}_3\text{S}$, $M_r = 329.42$, monoclinic, $P2_1/n$, $a = 10.573$ (5), $b = 12.437$ (6), $c = 13.045$ (6) \AA , $\beta = 106.84$ (4) $^{\circ}$, $V = 1642$ (1) \AA^3 , $Z = 4$, $D_x = 1.33 \text{ g cm}^{-3}$, $\lambda(\text{Cu } K\alpha) = 1.5418 \text{ \AA}$, $\mu = 18.24 \text{ cm}^{-1}$, $F(000) = 696$, $T = 291 \text{ K}$, $R = 0.051$ for

2154 observed reflections. The puckering (α) of the cyclobutanone ring, *i.e.* the dihedral angle between the planes defined by C1, C2, C3 and C1, C2, C4, is 14.8 (8) $^{\circ}$. The substituents are *trans* to each other in a pseudoequatorial arrangement. An *endo* deviation

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