conformation. It should be pointed out that for the triacetate of the KDO methyl ester not only in the crystal, but also in solution, another side-chain conformation exists. In this structure the atoms corresponding to the heptose O(6) and C(7) atoms exhibit *tg* and *gt* conformations, respectively. These deviations are comprehensible, since in KDO the C(5) substituent is axial, whereas the corresponding position of the heptose molecule shows an equatorial configuration.

However, the crystal structures of four derivatives of neuraminic acid are known (Biedl, 1971; O'Connell, 1973; Flippen, 1973; Wawra, 1974). All have the $^1C_4$ conformation, for which the stereochemical situation at the equatorially substituted C(6) atom is similar to the present structure: *gt* for the C atom and *gg* for the O atom of the side chain in question.

In the crystal structure of the title compound no close contacts of interest were observed.

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Structure of 2,2-Bis(methylthio)-1,1-ethylenedicarbonitrile

BY HANS-ULRICH HUMMEL AND HERIBERT PROCHER

Institut für Anorganische Chemie der Universität, Egerlandstraße 1, *D*-8520 *Erlangen, Federal Republic of Germany*

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Dedicated to Professor Dr K. Brodersen on the occasion of his 60*th birthday*

Abstract. $C_6H_6N_2S_2$, $M_r = 170{\cdot}3$, monoclinic, $P2_1/c$, $a = 4{\cdot}112\,(1)$, $b = 13{\cdot}406\,(1)$, $c = 15{\cdot}433\,(2)$ Å, $\beta = 111{\cdot}47\,(1)°$, $V = 791{\cdot}7$ Å^3, $Z = 4$, $D_m = 1{\cdot}40\,(3)$, $D_x = 1{\cdot}43\,(1)$ Mg m^{-3}, $\lambda(\text{Ag }K\alpha) = 0{\cdot}5591$ Å, $\mu = 0{\cdot}255$ mm^{-1}, $F(000) = 352$, $T = 298$ K, final $R = 0{\cdot}047$ for 861 observed independent reflections. The molecules are arranged nearly parallel to the *bc* plane and form polar chains along the **b** direction. The molecule has a *syn–anti* conformation with respect to the $(MeS)_2C$ group as a consequence of reduced intramolecular steric interaction. The average S—C bond length is 1·762 Å. The central C—C is 1·368 Å.

Introduction. During our investigations on the existence and molecular geometry of the free acid $(HS)_2C{=}C(CN)_2$ we decided to study the more stable $(CH_3S)_2C{=}C(CN)_2$ (Hummel & Förner, 1986). The structure analysis of the title compound is also interesting from a more theoretical point of view. It exhibits unexpected redox behaviour in our cyclic voltammetric studies (Hummel & Procher, 1985) and shows a decreased charge density at C(1) compared with the dianionic species $(S_2C_4N_2)^{2-}$ (Henriksen & Eggert, 1978). It is expected that the geometry of the molecule can give some hint of the charge distribution and the repulsive electrostatic forces in the species.

Experimental. Stoichiometric amounts of $Na_2(S_2C_4N_2).3H_2O$ and CH_3I in methanol (Söderbäck, 1963). Crystal size $0{\cdot}06 \times 0{\cdot}12 \times 0{\cdot}30$ mm. D_m determined pycnometrically. Precession photographs. Monoclinic, Laue class $2/m$, systematic extinctions *h*0*l*,

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$h = 2n+1$, $0k0$, $k = 2n+1$, space group $P2_1/a$. PW 1100 diffractometer, ω scan, scan width $(0{\cdot}6 + 0{\cdot}2\tan\theta)^\circ$. Cell dimensions based on 25 high-indexed diffractometer-measured intensities ($12 \le \theta \le 15^\circ$). 3420 symmetry-dependent reflections ($-15 \le h \le 15$, $-14 \le k \le 14$, $-4 \le l \le 4$, $2 \le \theta \le 18^\circ$) averaged to give 962 symmetry-independent reflections, $R_{\text{int}} = 0{\cdot}035$. Three standard reflections, no significant deviations from their initial intensities. Corrections for Lorentz, polarization and absorption effects (Sheldrick, 1976); transmission factors are 0·88–0·93. Structure solved by direct methods (*MULTAN*, Germain, Main & Woolfson, 1971). 861 observed reflections $[I > 4\sigma(I)]$ in blocked-matrix least-squares refinement ($\sin\theta_{\max}/\lambda = 0{\cdot}519$ Å^{-1}); 110 parameters; H atoms from difference map; with common isotropic temperature factor; $\sum w\|F_o| - |F_c\|^2$ minimized; $w = 4{\cdot}3767/[\sigma^2(F_o) + 1{\cdot}5 \times 10^{-4}F_o^2]$; *SHELX*76 system (Sheldrick, 1976); $(\Delta/\sigma)_{\max} = 0{\cdot}0003$, $(\Delta/\sigma)_{\min} = 0{\cdot}000$, $(\Delta/\sigma)_{\text{ave}} = 0{\cdot}0001$; $\Delta\rho = -0{\cdot}23$ to $0{\cdot}26$ e Å^{-3}. Final refinement converged to $R = 0{\cdot}047$, $wR = 0{\cdot}047$. Scattering factors, f' and f'' from *International Tables for X-ray Crystallography* (1974).

Discussion. Final atomic parameters are given in Table 1,* and selected distances and angles in Fig. 1. The structure is depicted in Fig. 2. It consists of discrete molecules forming polar chains parallel to the crystallographic *b* axis. Within one chain the hydrophobic $(MeS)_2C$ groups are oriented along the same direction while the 2_1 screw axis causes a space-filling arrangement of that *syn–anti* bis(methylthio) function. Chains of different polarity alternate along the *a* axis produced by the centre of symmetry. There are no important interactions other than van der Waals contacts within or between neighbouring molecules. This is in accordance with the IR and NMR spectra and a low melting point of the compound (m.p. 351 K). A similar *syn–anti* conformation is also predicted for the acid $(HS)_2C{=}C(CN)_2$ using *ab initio* HF-MO methods (Hummel & Förner, 1986). Compared to the anion $(S_2C_4N_2)^{2-}$ in the potassium salt (Hummel, 1985) the methyl derivative exhibits severe deviations from planarity. The maximum deviations from a least-squares plane for the whole molecule are 0·24 [C(6)] and 0·14 Å [C(1)]. This could be caused by intramolecular interactions as well as packing effects.

The S(1)—C(1) and S(2)—C(1) bond lengths are significantly different and correspond well to $S{-}C_{sp^2}$ single bonds (Gasparri, Nardelli & Villa, 1967). The $S{-}CH_3$ bonds are elongated but shorter than 'normal' C—S single bonds (1·82 Å) (Dräger, 1977).

Compared to the anion $(S_2C_4N_2)^{2-}$ (Hummel, 1985) the central bond length C(1)—C(2) is drastically decreased and corresponds well to a C=C double bond (Churchill & Cooke, 1970) but the bond angles around C(1) and C(2) differ greatly from the expected value of 120°. The angles S(2)—C(1)—C(2) (129·3°) and

Table 1. *Final atomic coordinates and equivalent isotropic temperature factors of the non-hydrogen atoms*

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

	x	y	z	U_{eq}(Å^2)
S(1)	0·9003 (1)	0·0518 (1)	0·6594 (3)	0·0535 (5)
S(2)	0·7133 (1)	0·1368 (1)	0·3537 (3)	0·0618 (5)
C(1)	0·2150 (2)	0·9628 (2)	0·6254 (6)	0·0499 (8)
C(2)	0·2350 (2)	0·0523 (2)	0·7965 (6)	0·0555 (8)
C(3)	0·1638 (2)	0·1274 (2)	0·7239 (7)	0·0548 (8)
C(4)	0·3208 (2)	0·0795 (2)	0·0747 (7)	0·0601 (8)
C(5)	0·0936 (2)	0·8276 (3)	0·1478 (7)	0·0603 (8)
C(6)	0·4048 (2)	0·9001 (3)	0·8888 (7)	0·0676 (9)
N(3)	0·1077 (2)	0·1867 (2)	0·6644 (7)	0·0818 (8)
N(4)	0·3857 (2)	0·1064 (2)	0·2971 (6)	0·0658 (8)

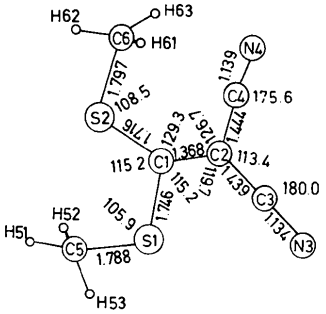

Fig. 1. The numbering and geometry of the $(MeS)_2C{=}C(CN)_2$ molecule. (Distances are in Å, angles in degrees, average e.s.d. for bond lengths 0·003 Å and for angles 0·2°.)

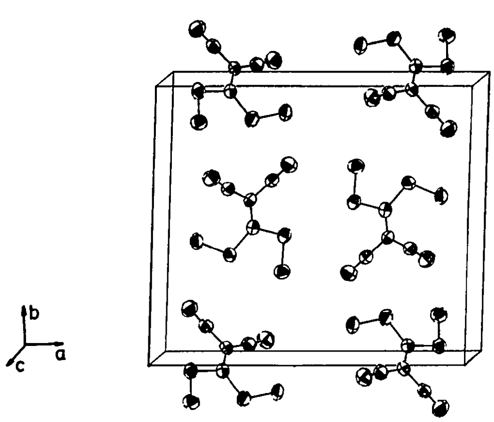

Fig. 2. The structure of $(MeS)_2C{=}C(CN)_2$.

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, intermolecular distances and mean-plane data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43146 (28 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

C(1)—C(2)—C(4) (126·7°) clearly reflect strong steric interaction between the *syn* methyl group of C(6) and the C(2)—C(4)—N(4) group of the molecule. This steric interaction is also reduced by the deviation of the C(2)—C(4)—N(4) moiety from linearity (175·6°). Nevertheless C(2)—C(3) and C(2)—C(4) are typical C_{sp^2}—C_{sp} bonds with bond order of $\frac{4}{3}$ (Bent, 1961).

The C—N triple-bond lengths are of the same order as the value in the corresponding $(S_2C_4N_2)^{2-}$ and demonstrate the low contribution of the cyanide groups to the π system of the whole molecule (Witt & Britton, 1971).

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Structure of 1-[3-(Imidazol-1-yl)propyl]thymine

BY MARIA GDANIEC AND GRAŻYNA WENSKA

Faculty of Chemistry, A. Mickiewicz University, 60–780 *Poznań, Poland*

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Abstract. $C_{11}H_{14}N_4O_2$, $M_r = 234{\cdot}3$, monoclinic, $P2_1/n$, $a = 8{\cdot}707\ (2)$, $b = 12{\cdot}786\ (4)$, $c = 10{\cdot}643\ (3)$ Å, $\beta = 102{\cdot}89\ (2)°$, $V = 1155{\cdot}0\ (5)$ Å^3, $Z = 4$, $D_x = 1{\cdot}35$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 0{\cdot}59$ cm^{-1}, $F(000) = 496$, room temperature, $R = 0{\cdot}034$ for 1268 unique reflections. The extended conformation of the molecule prevents any intramolecular photocycloaddition of thymine and imidazole C=C double bonds in the crystal [the shortest distance between the midpoints of C=C and C=N double bonds is 3·549 (3) Å].

Introduction. The photochemistry of purine–pyrimidine dinucleotides and their analogues in aqueous solution has grown in interest in the last few years (Bose, Davies, Sethi & McCloskey, 1983; Bose, Kumar, Davies, Sethi & McCloskey, 1984; Bose & Davies, 1984; Wenska, Paszyc & Skalski, 1983; Wenska, 1985). However, the available information concerning the possibility of photocycloaddition of purine to pyrimidine and the stability of the resulting photodimers is still limited and ambiguous. Thus further structural data are needed to shed more light on these problems.

Irradiation with UV light of an aqueous solution of the title compound leads to a cyclobutane product resulting from cycloaddition of imidazole and thymine C=C double bonds (Wenska, 1985). The product of this photoreaction has a *cis–syn* configuration (Gdaniec, Wenska & Kozioł, 1985). This X-ray study of the title compound was undertaken to determine the conformation of this compound in the crystal and investigate whether or not a solid-state photoreaction might lead to a similar product of intramolecular photocycloaddition.

Experimental. Colourless crystal 0·5 × 0·5 × 0·4 mm from methanol; D_m not determined; Syntex $P2_1$ diffractometer, graphite monochromator; lattice parameters from 15 reflections; profiles measured for 1724 unique reflections with $2\theta \leq 48°$ (h 0→9, k 0→14, l −12→12), profile analysis according to Lehmann & Larsen (1974), no significant intensity variation for two standard reflections; absorption ignored; 1268 reflections with $I \geq 1{\cdot}96\sigma(I)$; structure solved by direct methods with *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980); full-matrix least-squares refinement on F with *SHELX*76 (Sheldrick, 1976), $w = 1/[\sigma^2(F) + 0{\cdot}00013F^2]$; final refinement: anisotropic non-H atoms, isotropic H atoms (from ΔF map) and empirical isotropic extinction parameters x used to correct F_c according to $F_c' = F_c(1 - xF_c^2/\sin\theta)$, x converged at 0·0116 (9); $R = 0{\cdot}034$ and $wR = 0{\cdot}040$; $S = 1{\cdot}95$; in final refinement cycle $\Delta/\sigma \leq 0{\cdot}02$, largest peak in final ΔF map 0·11, largest hole −0·13 e Å^{-3}; atomic

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