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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71822 (15 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: SH1085]

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X-ray Study of Static Disorder in *N*-Methylacetamide

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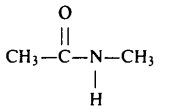
Abstract

N-Methylacetamide (NMA), C_3H_7NO, is the smallest molecule to contain a peptide bond and is considered to be the basic structural unit of peptide chains in proteins. An X-ray electron density study at 110 K showed the presence of static disorder. The present work describes this disorder and the configuration of this biologically important molecule.

Comment

NMA is a small planar molecule of fundamental importance which has been extensively investigated by IR and Raman spectroscopy (Mizushima & Shimanouchi, 1950). A preliminary report of the structure of NMA at 238 K has been published (Katz & Post, 1960) in which the authors describe the structure as ordered (space group *Pnma* with *R* = 0.13). The results of an accurate electronic density study of the molecule would be an important contribution to complete the work cited above in order to gain a better understanding of the peptide bond.



$CH_3-\overset{\overset{O}{\|}}{C}-\underset{\underset{H}{|}}{N}-CH_3$

NMA

The refinement was initially carried out using the least-squares program *LINEX* (Coppens, 1974). An anisotropic refinement in which the coordinates of the H atoms were adjusted to result in tetrahedral bond angles and C—H bond lengths of 1.09 Å gave $R = 0.09$. A difference Fourier synthesis revealed a second position for the molecule (Fig. 1). An occupation factor of 0.9 was assigned to the first molecule (molecule *A*) and a factor of 0.1 was assigned to the second one (molecule *B*). The positions of the atoms of the second molecule were corrected to adjust its geometry to that of molecule *A*.

Further refinement was carried out using the least-squares program *ORION* (André, Fourme & Renaud, 1971), in which atoms may be constrained in groups. The thermal motion of the molecules was analysed initially in terms of rigid-body motion, but this resulted in a singularity in the least-squares normal matrix, giving the errors 'the atoms lie on a quadratic curve' (Schomaker & Trueblood, 1968). Therefore, the five non-H atoms of molecule *A* were allowed to refine freely with anisotropic displacement factors, while molecule *B* was treated as a rigid body with isotropic displacement factors *B* of 2.03 $Å^2$ for the non-H atoms; for H atoms an isotropic Debye–Waller factor refined to 1.81 $Å^2$. This second refinement procedure resulted in a decreased *R* value of 0.066. The structure of molecule *A* is shown in Fig. 2.

In order to determine the nature of this disorder a second X-ray study was carried out at 210 K. Using the same refinement procedure described above, a

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smaller value of R was obtained for the same occupation factors found at 110 K. From this it was concluded that the disorder is static.

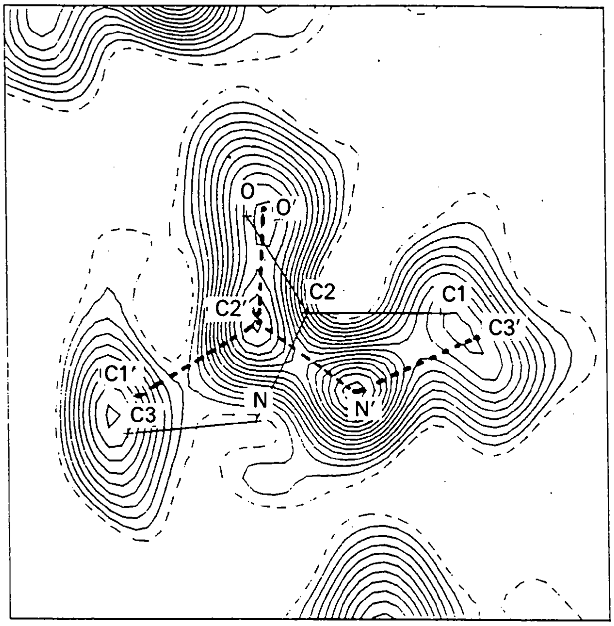

Fig. 1. A difference electron density map in the plane of molecule A, showing the position of molecule B. Molecule A: C1, C2, C3, O, N. Molecule B: C1', C2', C3', O', N'. H atoms are not shown. Contours are drawn at intervals of 0.04 e Å^{-3} from ρ = 0.02 e Å^{-3}.

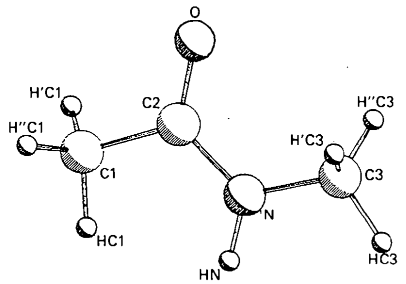

Fig. 2. Perspective view of molecule A.

Experimental

N-Methylacetamide is very hygroscopic. Samples were filtered through a molecular sieve to remove traces of water and were sealed in a thin-walled glass capillary, 0.5 mm in diameter. The experimental procedures used to grow and maintain single crystals at low temperatures have been described (Post, Schwartz & Fankuchen, 1951).

Crystal data

C_3H_7NO
M_r = 73.094
Orthorhombic
$Pn2_1a$
a = 9.65 (10) Å
b = 6.33 (15) Å
c = 7.17 (14) Å
V = 437.5 Å^3
Z = 4
D_x = 1.11 Mg m^{-3}
D_m = 1.02 Mg m^{-3}
D_m by flotation in CCl_4/toluene

Mo $K\alpha$ radiation
λ = 0.71069 Å
Cell parameters from 25 reflections
θ = 11-25°
μ = 0.078 mm^{-1}
T = 110 (3) K
Cylinder, 0.5 mm (diameter), 0.7 mm (length)
Colourless

Data collection

Nonius CAD-4 diffractometer
$\omega/2\theta$ step scans
Absorption correction: none
5690 measured reflections
3578 independent reflections
1264 observed reflections $[I \geq 3\sigma(I)]$

R_{int} = 0.039
θ_{max} = 65°
$h = 0 \rightarrow 19$
$k = 0 \rightarrow 12$
$l = 0 \rightarrow 13$
5 standard reflections frequency: 120 min intensity variation: none

Refinement

Refinement on F
R = 0.066
wR = 0.066
S = 1.12
1264 reflections
51 parameters
H atoms: constrained refinement
Unit weights applied
$(\Delta/\sigma)_{max}$ = 0.7

$\Delta\rho_{max}$ = 0.49 e Å^{-3}
$\Delta\rho_{min}$ = −0.05 e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV) for non-H and Stewart, Davidson & Simpson (1965) for H atoms

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$B_{eq} = (4/3)\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	B_{eq}
C1	0.1268 (2)	0.2617 (22)	0.0295 (3)	2.1766
C2	0.1642 (2)	0.2577 (17)	0.2347 (3)	1.7218
C3	0.0823 (2)	0.2572 (20)	0.5543 (3)	2.1348
N	0.0592 (2)	0.2629 (15)	0.3539 (2)	1.8700
O	0.2869 (1)	0.2602 (1)	0.2888 (3)	2.2679
C1'	0.1245 (2)	0.2923 (22)	0.5260 (3)	2.0300
C2'	0.1636 (2)	0.2418 (17)	0.3279 (3)	2.0300
C3'	0.0874 (2)	0.2159 (20)	0.0080 (3)	2.0300
N'	0.0623 (2)	0.2602 (15)	0.2022 (2)	2.0300
O'	0.2825 (1)	0.1859 (1)	0.2812 (3)	2.0300

Table 2. *Selected geometric parameters* (Å, °)

C1—C2	1.515 (3)	C1'—C2'	1.50
C2—N	1.325 (3)	C2'—N'	1.33
C2—O	1.246 (2)	C2'—O'	1.24
C3—N	1.454 (3)	C3'—N'	1.44
C1—C2—N	116.3 (6)	C1'—C2'—N'	116
C1—C2—O	121.9 (6)	C1'—C2'—O'	123
N—C2—O	121.7 (6)	N'—C2'—O'	121
C2—N—C3	121.3 (6)	C2'—N'—C3'	121

Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71709 (9 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: DU1056]

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1*H*-Benzimidazolium Hydrogen L-Tartrate Dihydrate

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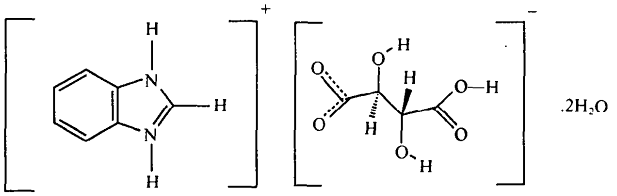
Abstract

The structure of the title compound, $C_7H_7N_2^+.C_4H_5O_6^-.2H_2O$, is dominated by infinite chains of hydrogen L-tartrate anions linked in a head-to-tail manner by a short hydrogen bond, O···O = 2.576 (4) Å. The cation and the water molecules provide several crosslinks between adjacent anionic chains, creating a complex three-dimensional hydrogen-bond network.

Comment

The structures and properties of salts of tartaric acid have captivated scientists from a remarkably diverse range of disciplines, including solid-state physics (Fousek, 1991), wine-making technology (Clark, Fuglesang & Gump, 1988) and chiral synthesis (Marshall & Luke, 1991). This family of compounds is also beginning to attract significant attention because of its ability to exhibit nonlinear optical effects (Aakeröy, Hitchcock & Seddon, 1992; Zyss, Pecaut, Levy & Masse, 1993). Furthermore, the possibilities of using infinite sheets of hydrogen tartrate anions as building blocks ('scaffolding') for crystal engineering have also been explored recently (Aakeröy & Hitchcock, 1993).

We are currently conducting an extensive structural investigation of hydrogen tartrate salts in order to determine the role played by the cation in inducing, or discriminating between, different anionic aggregate types. In addition, the physico-chemical characteristics of the cations within hydrated *versus* anhydrous hydrogen tartrate salts are being evaluated with a view to predicting and controlling the structural features of novel, unknown crystalline materials (Aakeröy, Rzepa & Seddon, 1994). The synthesis and crystallographic study of the title compound (I) forms part of this investigation. The title compound was prepared by mixing equimolar ethanolic solutions of 1*H*-benzimidazole and L-tartaric acid. After evaporation of the solvent, the product was collected by filtration and recrystallized from water.



(I)

The cation and anion do not display any unusual structural features (Fig. 1). The cation exhibits the expected planar geometry and the C—C—C—C torsion angle of the anion is close to 180°.

The anions are arranged in infinite chains, parallel to **b**, *via* a short O—H···O hydrogen bond, with an O—H···O angle of 160 (3)°, which links adjacent anions in a head-to-tail fashion. However, there are no direct hydrogen bonds between adjacent chains, which prevents the formation of the commonly

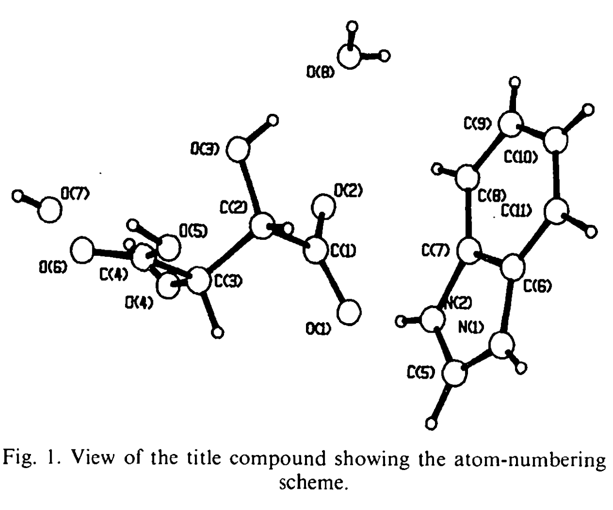


Fig. 1. View of the title compound showing the atom-numbering scheme.

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