conformation (Berkovitch-Yellin & Leiserowitz, 1984).

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Structure of N-Methylnicotinamide

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Abstract. $C_7H_8N_2O$, $M_r = 136.2$, monoclinic, $P2_1/a$, $a = 7.055$ (1), $b = 9.849$ (6), $c = 10.066$ (4) Å, $\beta = 100.47$ (2)°, $V = 687.5$ (5) Å³, $Z = 4$, $D_m = 1.32$, $D_x = 1.315$ g cm⁻³, Cu $K\alpha$, $\lambda = 1.5418$ Å, $\mu = 7.09$ cm⁻¹, $F(000) = 288$, $T = 294$ K, $R = 0.048$ for 1134 reflections $[I > 3\sigma(I)]$. The N-methylcarboxamide group is extended with the keto O(7) transoid to C(2) [C(2)—C(3)—C(7)—O(7) ±158.9 (3), C(3)—C(7)—N(7)—C(8) ±177.1 (3), C(2)—C(3)—C(7)—N(7) ±23.2 (3) and C(4)—C(3)—C(7)—N(7) ±158.2 (3)°]. The dihedral angle between the planes of the pyridine ring and the carboxamide plane is 22°. The molecules are linked together by N—H···O hydrogen bonds involving the amino N(7) and the carbonyl O(7) atoms.

Introduction. Nicotinamide is a component of two related coenzymes, nicotinamide adenine dinucleotide (NAD) and nicotinamide adenine dinucleotide diphosphate (NADP). The crystal structure of NAD has been determined in both the free acid form (Parthasarathy & Fridey, 1984) and in the low pH

lithium salt (Reddy, Saenger, Muhlegger & Weimann, 1981). Although they had different conformations for the NAD molecules, both the structures revealed intermolecular stacking of the bases; however, no intramolecular stacking was observed as predicted by NMR studies (Sarma, Ross & Kaplan, 1968). We have undertaken a systematic analysis of several model compounds involving the bases and nicotinamide groups, with a view to studying the stacking properties in these compounds and comparing them with those found in NAD. The crystal structure of adenosine 5'-mononicotinate (Ramasubbu & Parthasarathy, 1988) revealed intra- as well as intermolecular base stacking, a feature not observed in some other model compounds studied earlier (Sakaki, Inoue, Senda & Tomita, 1978; Voet, 1973; Johnson, Frank & Paul, 1973). Recently, we determined the crystal structures of thionicotinamide and N-methylnicotinamide with a view to obtaining accurate molecular dimensions of neutral nicotinamide as well as studying the effect of sulfur substitution on its hydrogen-bonding details. We report here the crystal structure of N-methylnicotinamide.

Table 1. *Positional parameters and their isotropic thermal parameters*

The estimated standard deviations for last digits are given in parentheses.

	x	y	z	B (Å^2)
N(1)	0·1731 (3)	0·5345 (2)	0·3472 (2)	4·55 (3)
C(2)	0·1885 (3)	0·4889 (2)	0·2244 (2)	3·63 (3)
C(3)	0·2681 (2)	0·3644 (2)	0·2013 (2)	2·88 (3)
C(4)	0·3317 (3)	0·2815 (2)	0·3117 (2)	3·66 (3)
C(5)	0·3185 (3)	0·3267 (2)	0·4396 (2)	4·28 (4)
C(6)	0·2392 (3)	0·4531 (2)	0·4517 (2)	4·58 (4)
C(7)	0·2842 (2)	0·3147 (1)	0·0633 (2)	2·95 (3)
O(7)	0·3024 (2)	0·1921 (1)	0·0425 (1)	4·21 (3)
N(7)	0·2844 (2)	0·4073 (1)	−0·0326 (1)	3·34 (3)
C(8)	0·3082 (3)	0·3715 (2)	−0·1682 (2)	4·19 (4)
H(C2)	0·133 (3)	0·550 (2)	0·148 (2)	2·6 (5)*
H(C4)	0·386 (3)	0·195 (2)	0·299 (2)	1·9 (4)*
H(C5)	0·364 (3)	0·269 (2)	0·517 (2)	3·1 (5)*
H(C6)	0·230 (4)	0·496 (3)	0·538 (3)	3·9 (6)*
H(N7)	0·276 (4)	0·500 (3)	−0·020 (2)	2·8 (5)*
H1(C8)	0·416 (4)	0·314 (2)	−0·166 (3)	5·1 (7)*
H2(C8)	0·200 (4)	0·334 (2)	−0·222 (2)	3·1 (5)*
H3(C8)	0·347 (5)	0·454 (3)	−0·218 (3)	5·1 (7)*

Starred atoms were refined isotropically. Anisotropically refined atoms are given in the form of the isotropic equivalent displacement parameters defined as: $(4/3)[a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + ab(\cos\gamma)B(1,2) + ac(\cos\beta)B(1,3) + bc(\cos\alpha)B(2,3)]$.

Experimental. The compound was purchased from Sigma Chemicals; needle-shaped crystal obtained from aqueous methanol solution; D_m by flotation (bromoform and benzene); unit-cell parameters on a CAD-4 diffractometer using 25 reflections with $20 < \theta < 51°$; crystal of dimensions $0·95 \times 0·45 \times 0·1$ mm; three-dimensional data ($2\theta < 154°$ for Cu $K\alpha$ radiation), $\omega/2\theta$ scan; scan widths calculated using the expression $(0·6 + 0·14\tan\theta)°$ and aperture widths using $(3·0 + 1·2\tan\theta)$ mm; maximum time spent on any reflection measurement 100 s; faster scan used for strong reflections; intensities of three reflections measured after every hour of exposure; variation of intensities in these three reflections <2% during the entire data collection; crystal orientation checked after every 100 reflections; 1455 reflections measured out of which 1134 were significant $[I > 3\sigma(I)]$; h −5 to 5; k 0 to 13; l 0 to 19; Lorentz and polarization corrections applied; intensities of three reflections at $\chi \simeq 90°$ were measured for different values of φ from 0 to 360° in steps of 10°, and the resultant curve of transmission as a function of φ was used to calculate the absorption for all reflections; maximum and minimum transmission 0·99 and 0·77, with an average of 0·89. Structure solved using *MULTAN* (Germain, Main & Woolfson, 1971); 209 E values ($|E| > 1·46$) used as input to *MULTAN*; the correct set, with the figure of merit of 1·14 and a residual of 0·149, gave all atoms in the molecule; refinement initially with isotropic thermal parameters and later with anisotropic thermal parameters led to $R = 0·084$; difference electron density maps revealed positions of all H atoms; final cycles of refinement with

anisotropic thermal parameters for non-H atoms, isotropic for H atoms and extinction-parameter refinement ($g = 5·08 \times 10^{-6}$) led to $R = 0·048$ for 1134 reflections; $wR = 0·075$; $S = 2·09$, $\sum w[|F_o|^2 - (1/k)|F_c|^2]$ minimized; $w = 4|F_o|^2/\sigma^2(|F_o|^2)$; $\sigma^2(|F_o|^2) = [\sigma^2(I) + p^2I^2]/Lp$ where $p = 0·05$, $\sigma(I)$ is standard deviation of intensity I based on counting statistics; k is the scale factor; maximum and average $\Delta/\sigma = 0·2$ and 0·02; final $|\Delta\rho| = 0·2$ e Å^{-3}. Program and atomic scattering factors as in Enraf–Nonius (1979) *SDP*; torsion-angle programs by Dr S. T. Rao and *ORTEP* by Johnson (1965).
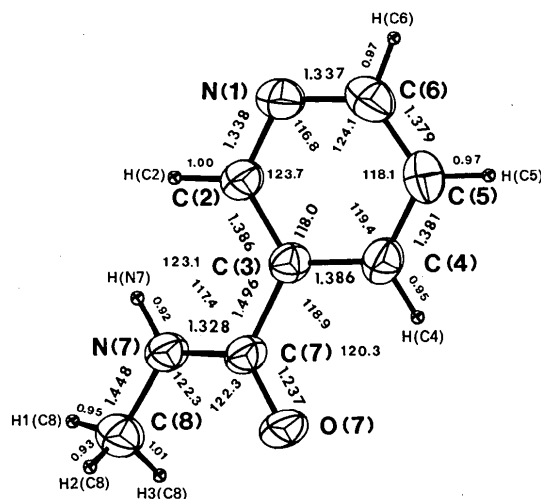

Fig. 1. An *ORTEP* drawing of N-methylnicotinamide giving the atom numbering in the molecule and the final bond distances (Å) and angles (°). The average standard deviation in bond lengths is 0·002 Å and in bond angles 0·2°.
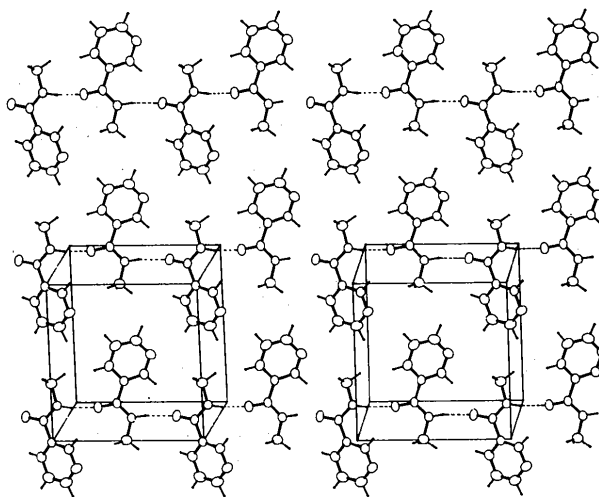

Fig. 2. A stereoscopic view of the packing of the molecules in the unit cell as viewed down the a axis. Molecules related by screw axes are linked by an N—H···O hydrogen bond.

Discussion. The final atomic coordinates are given in Table 1.* Fig. 1 gives the atom numbering as well as the final bond distances and angles in the structure. The pyridine ring is essentially planar and the maximum deviation of the ring atoms from the least-squares plane is 0·009 Å. The C(2)—N(1)—C(6) bond angle of 116·8 (2)° agrees with the values found in nicotinamide (Wright & King, 1954), isonicotinic acid (Takusagawa & Shimada, 1976), nicotinic acid (Wright & King, 1953; Gupta & Kumar, 1975; Kutoglu & Scheringer, 1983) and other non-protonated pyridine compounds (~117°) (Singh, 1965). The dihedral angle between the planes of the pyridine ring and of the methylcarboxamide groups is 22·0°.

Fig. 2 gives a stereoview of the packing of the molecules in the unit cell. Molecules related by screw axes are connected by an N(7)—H(N7)···O(7) hydrogen bond involving the amino N(7) and the carbonyl O(7) atoms [N(7)···O(7) 2·869 (1), H(N7)···O(7) 1·98 (2) Å and N(7)—H(N7)···O(7) 163 (2)°]. There is no intermolecular stacking observed in the crystal structure.

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52949 (19 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of Xanthone

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Abstract. 9*H*-Xanthen-9-one, $C_{13}H_8O_2$, $M_r = 196·21$, orthorhombic, $P2_12_12_1$, $a = 14·072 (10)$, $b = 13·627 (8)$, $c = 4·878 (3)$ Å, $V = 935·4 (10)$ Å³, $Z = 4$, $D_x = 1·393$, $D_m = 1·391 (6)$ g cm⁻³, Mo *K*α, $\lambda = 0·70926$ Å, $\mu = 0·875$ cm⁻¹, $F(000) = 408$, room temperature, $R = 0·077$ for 648 independent reflections. The molecule is planar except for the carbonyl O atom (0·13 Å out of the plane). The benzene rings are not regular hexagons but the distortion is symmetric and the molecule has approximate C_{2v} symmetry. There are no abnormally short distances between molecules.

Introduction. The molecular structure of xanthone has been studied in the gas and the solid phases. In the gas phase xanthone is planar and has C_{2v} symmetry. The benzene rings were assumed to be regular hexagons in the structure analysis, but the symmetry of the ring appeared to be lower (Iijima, Misu, Ohnishi & Onuma, 1989). On the other hand, the

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