Table 3. *Selected bond lengths* (Å), *angles* (°), *torsion angles* (°) *and hydrogen-bond lengths* (Å)

	(I)	(II)
N(1)—N(2)	1·314 (3)	1·314 (6)
N(2)—N(3)	1·321 (4)	1·325 (8)
N(3)—C(4)	1·345 (4)	1·344 (7)
C(4)—C(5)	1·383 (4)	1·390 (7)
C(5)—C(6)	1·384 (4)	1·392 (7)
C(6)—N(1)	1·350 (3)	1·347 (6)
C(4)—C(7)	1·490 (6)	1·497 (9)
C(5)—C(8)	1·513 (4)	1·506 (7)
C(8)—O(9)	1·227 (3)	1·242 (7)
C(8)—N(10)	1·319 (4)	1·323 (7)
C(6)—C(11)	1·475 (4)	1·473 (7)
C(6)—N(1)—N(2)	119·8 (2)	121·3 (4)
N(1)—N(2)—N(3)	122·6 (2)	121·4 (4)
N(2)—N(3)—C(4)	119·5 (2)	119·8 (4)
N(3)—C(4)—C(5)	120·5 (2)	121·1 (4)
C(4)—C(5)—C(6)	177·2 (2)	116·4 (4)
C(5)—C(6)—N(1)	120·0 (2)	119·7 (4)
N(3)—C(4)—C(7)	114·1 (2)	115·4 (5)
C(5)—C(4)—C(7)	125·3 (2)	123·4 (5)
C(4)—C(5)—C(8)	119·0 (2)	120·0 (4)
C(6)—C(5)—C(8)	123·7 (2)	123·3 (4)
C(5)—C(8)—O(9)	120·6 (2)	117·3 (4)
C(5)—C(8)—N(10)	114·8 (2)	119·1 (4)
O(9)—C(8)—N(10)	124·4 (2)	123·5 (4)
C(5)—C(6)—C(11)	126·0 (2)	126·3 (4)
N(1)—C(6)—C(11)	113·8 (2)	113·9 (4)
C(4)—C(5)—C(8)—O(9)	−75·2 (2)	−68·3 (4)
C(6)—C(5)—C(8)—N(10)	−75·8 (2)	−74·2 (5)
C(5)—C(6)—C(11)—C(12)	−37·8 (2)	−37·6 (5)
N(10)···O(9)	2·862 (3)	
N(10)···N(2)	2·990 (3)	
N(10)···N(1)		3·059 (7)
N(10)···N(3)		3·087 (6)

parameter of each bonded atom. Major computations performed on a PANAFACOM computer with the *RCRYSTAN* (Rigaku Corporation, 1985) X-ray analysis program system. The atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). Final atomic parameters are listed in Table 2.* Selected bond lengths, angles, torsion angles and hydrogen-bond lengths are listed in Table 3. Fig. 1 shows an *ORTEP* drawing (Johnson, 1965) of form (I) of the molecule with the atom labels. Fig. 2 gives the crystal structures.

Related literature. By comparison with the similar compound 4-methyl-6-phenyl-1,2,3-triazine (Yamaguchi, Ohsawa & Itoh, 1990), the bond lengths and angles are almost consistent in the triazine rings (differences are less than 0·01 Å and 0·5°, respectively).

* Tables of H-atom coordinates, anisotropic thermal parameters and structure-factor amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54235 (11 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.

MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1980). *MULTAN80. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.

MINISCI, F., FONTANA, T. & VISMARA, E. (1990). *J. Heterocycl. Chem.* **27**, 79–96.

Rigaku Corporation (1985). *RCRYSTAN. X-ray Analysis Program System*. Rigaku Corporation, Tokyo, Japan.

YAMAGUCHI, K., OHSAWA, A. & ITOH, T. (1990). *Acta Cryst.* C**46**, 2177–2181.

YAO, J.-X., ZHENG, C.-D., QIAN, J.-Z., HAN, F.-S., GU, Y.-X. & FAN, H.-F. (1985). *SAPI85. A Computer Program for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Institute of Physics, Chinese Academy of Sciences, Beijing, People's Republic of China.

Acta Cryst. (1991). C**47**, 2258–2262

Structures of 3-(Substituted benzamido)-5,6,7,8-tetrahydro-5,8-methanoisoquinolines

BY HIROSHI NAKAI, MAMORU TAKASUKA, TADASHI IRIE AND HIROSHI TANIDA

Shionogi Research Laboratories, Shionogi & Co. Ltd, Fukushima-ku, Osaka 553, *Japan*

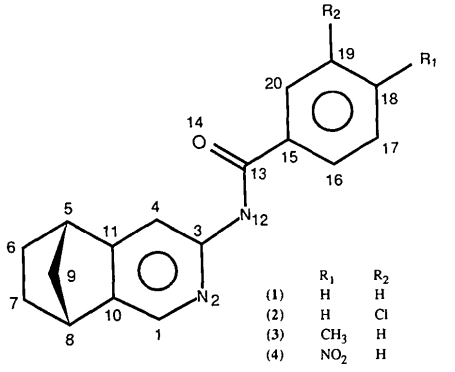
(*Received* 11 *March* 1991; *accepted* 22 *May* 1991)

Abstract. 3-Benzamido-5,6,7,8-tetrahydro-5,8-methanoisoquinoline (1): $C_{17}H_{16}N_2O$, $M_r = 264{\cdot}33$, monoclinic, $P2_1/c$, $a = 11{\cdot}168$ (4), $b = 13{\cdot}158$ (4), $c = 9{\cdot}678$ (8) Å, $\beta = 105{\cdot}68$ (3)°, $V = 1369$ (1) Å^3, $Z = 4$, $D_x = 1{\cdot}282$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, $\mu = 0{\cdot}65$ mm^{-1}, $F(000) = 560$, $T = 295$ K, $R = 0{\cdot}076$ for 2005 observed reflections. 3-(*m*-Chlorobenzamido)-5,6,7,8-tetrahydro-5,8-methanoisoquinoline (2): $C_{17}H_{15}ClN_2O$, $M_r = 298{\cdot}77$, monoclinic, $P2_1/c$, $a = 11{\cdot}515$ (3), $b = 14{\cdot}241$ (2), $c = 9{\cdot}576$ (2) Å, $\beta = 108{\cdot}03$ (2)°, $V = 1493{\cdot}1$ (6) Å^3, $Z = 4$, $D_x = 1{\cdot}329$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, $\mu =$

0108-2701/91/102258-05$03.00

2·26 mm^{-1}, $F(000) = 624$, $T = 295$ K, $R = 0·051$ for 2234 observed reflections. 3-(*p*-Methylbenzamido)-5,6,7,8-tetrahydro-5,8-methanoisoquinoline (3): $C_{18}H_{18}N_2O$, $M_r = 278·35$, triclinic, $P\bar{1}$, $a = 10·549$ (1), $b = 11·430$ (2), $c = 6·820$ (1) Å, $\alpha = 101·00$ (1), $\beta = 96·63$ (1), $\gamma = 108·67$ (1)°, $V = 750·7$ (2) Å^3, $Z = 2$, $D_x = 1·231$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, $\mu = 0·62$ mm^{-1}, $F(000) = 296$, $T = 295$ K, $R = 0·049$ for 2466 observed reflections. 3-(*p*-Nitrobenzamido)-5,6,7,8-tetrahydro-5,8-methanoisoquinoline (4): $C_{17}H_{15}N_3O_3$, $M_r = 309·32$, monoclinic, $P2_1/c$, $a = 15·080$ (3), $b = 6·034$ (2), $c = 16·818$ (3) Å, $\beta = 104·12$ (1)°, $V = 1484·0$ (5) Å^3, $Z = 4$, $D_x = 1·384$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, $\mu = 0·81$ mm^{-1}, $F(000) = 648$, $T = 295$ K, $R = 0·050$ for 2161 observed reflections. In crystals (1)–(4) the bicyclic rings are all disordered with two conformations. In (1) and (2) the molecules are linked to each other by hydrogen bonds between the NH and O atoms in the amido groups so as to form infinite chains. Molecules (3) and (4) adopt a dimer structure *via* a pair of NH···N hydrogen bonds between the amido and pyridine groups and are located around a center of symmetry.

Experimental. Rigaku AFC-5 diffractometer, graphite-monochromatized Cu $K\alpha$ radiation, no absorption corrections. Three standard reflections monitored every 100 measurements showed no change.



For each of (1)–(4), an interpretable structure was obtained by *MULTAN*84 (Main, Germain & Woolfson, 1984) but least-squares refinements of the structure failed to reduce the R value to less than 7–10%. Moreover, anisotropies of temperature factors in the bicyclic-ring atoms were very large, bond lengths and angles in the bicyclic rings were unreasonable and a large peak appeared in each difference density map. Subsequently, low-temperature diffraction data of (2) measured at 175 K were used for refinement of the structure but the situation could not be improved. Therefore, it is not likely that thermal vibrations are responsible for the anomalies. However, the anomalies could be all explained by a disorder model in which two orientations of the bicyclic ring are superimposed. This model was refined by the least-squares method allowing rigid groups with fixed isotropic B values and using the program *CRYLSQ* in *XTAL*2.6 (Hazekamp, 1989). The occupancy factors of the disordered portion were estimated from the thermal parameters. By this method the R value could be reduced to about 5–8%. In (3), six peaks corresponding to the H atoms of the methyl group were revealed in a difference density map, showing disordering of the methyl group.

H atoms in the disordered portions were located at their ideal positions. These positions were included in stucture-factor calculations but not refined. Other H atoms were located in difference density maps. Anisotropic thermal parameters for non-H atoms and isotropic for H atoms were refined by block-diagonal least squares. Atomic scattering factors were calculated by $\sum[a_i\exp(-b_i\lambda^{-2}\sin^2\theta)] + c$ ($i = 1,\cdots,4$) (*International Tables for X-ray Crystallography*, 1974, Vol. IV). Calculations were performed on a VAX station 3100 computer at Shionogi Research Laboratories.

(1): Colorless needles obtained from *n*-hexane. Crystal of dimensions 0·1 × 0·1 × 0·5 mm. Cell dimensions determined from 2θ angles for eight reflections in the range $30 < 2\theta < 44°$. Intensities measured up to $\theta = 70°$ in h −13/13, k 0/16 and l −11/0, ω-2θ scans, ω-scan width (1·2 + 0·2tanθ)°. 2528 unique reflections measured, 2005 intensities observed [$F_o > 3\sigma(F_o)$]. $\sum(w|\Delta F|^2)$ minimized, $w = 1/[\sigma^2(F_o) + 0·00217|F_o|^2]$, $w = 0$ for 193 reflections with $w^{1/2}|\Delta F| \geq 3$. Final $R = 0·076$, $wR = 0·074$, $S = 1·28$. The large R value is a consequence of the poor quality of the crystal. The highest and lowest peaks in the final difference map are 0·3 and −0·3 e Å^{-3}. Max. Δ/σ in the final cycle 0·3.

(2): Colorless prisms obtained from *n*-hexane. Crystal of dimensions 0·3 × 0·3 × 0·3 mm. Cell dimensions determined from 2θ angles for 25 reflections in the range $29 < 2\theta < 60°$. Intensities measured up to $\theta = 70°$ in h −13/13, k −17/0 and l 0/11, ω–2θ scans, ω-scan width (1·5 + 0·2tanθ)°. 2689 unique reflections measured, 2234 intensities observed [$F_o > 3\sigma(F_o)$]. $\sum(w|\Delta F|^2)$ minimized, $w = 1/[\sigma^2(F_o) + 0·00166|F_o|^2]$, $w = 0$ for 166 reflections with $w^{1/2}|\Delta F| \geq 3$. Final $R = 0·051$, $wR = 0·061$, $S = 1·27$. The highest and lowest peaks in the final difference map are 0·5 and −0·6 e Å^{-3}. Max. Δ/σ in the final cycle 0·2.

(3): Colorless prisms obtained from *n*-hexane. Crystal of dimensions 0·2 × 0·4 × 0·7 mm. Cell dimensions determined from 2θ angles for 25 reflections in the range $30 < 2\theta < 45°$. Intensities measured up to $\theta = 70°$ in h −12/0, k −13/13 and l −8/8,

Table 1. *Atomic coordinates and equivalent isotropic temperature factors* ($Å^2$)

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

	x	y	z	B_{eq}
(1) *A* (Occupancy factor 0·734)				
C(1)	1·0078 (3)	0·4394 (2)	0·3253 (3)	5·09 (10)
N(2)	0·8967 (3)	0·3954 (2)	0·3233 (3)	4·51 (7)
C(3)	0·8651 (3)	0·3110 (2)	0·2457 (3)	3·91 (7)
C(4)	0·9371 (3)	0·2643 (2)	0·1675 (3)	4·25 (8)
C(5)	1·1516 (3)	0·2872 (2)	0·1000 (4)	5·08 (9)
C(6)	1·1409 (4)	0·3693 (3)	−0·0167 (4)	5·92 (11)
C(7)	1·1832 (3)	0·4656 (3)	0·0739 (4)	5·24 (10)
C(8)	1·2105 (3)	0·4304 (2)	0·2314 (3)	4·78 (9)
C(9)	1·2656 (3)	0·3251 (3)	0·2214 (5)	6·06 (11)
C(10)	1·0845 (3)	0·3991 (2)	0·2498 (3)	4·38 (8)
C(11)	1·0498 (3)	0·3108 (2)	0·1716 (3)	4·24 (8)
(1) *B* (Occupancy factor 0·266)				
C(1)	1·0091 (10)	0·4286 (7)	0·3383 (10)	5·6 (3)
N(2)	0·9123 (5)	0·3768 (4)	0·3492 (7)	3·7 (2)
C(3)	0·8722 (6)	0·3048 (5)	0·2551 (7)	3·2 (2)
C(4)	0·9281 (9)	0·2793 (7)	0·1471 (9)	5·0 (3)
C(5)	1·1179 (8)	0·3270 (7)	0·0459 (11)	5·3 (3)
C(6)	1·2328 (12)	0·2790 (10)	0·1424 (15)	7·8 (4)
C(7)	1·2858 (9)	0·3625 (11)	0·2406 (13)	7·5 (4)
C(8)	1·1892 (10)	0·4514 (7)	0·2048 (11)	5·4 (3)
C(9)	1·1540 (10)	0·4417 (11)	0·0369 (11)	7·1 (4)
C(10)	1·0729 (11)	0·4106 (6)	0·2425 (9)	5·5 (3)
C(11)	1·0272 (9)	0·3333 (7)	0·1423 (9)	5·1 (3)
(1)				
N(12)	0·7552 (2)	0·2644 (1)	0·2631 (2)	4·23 (5)
C(13)	0·6835 (2)	0·1958 (2)	0·1729 (2)	3·78 (5)
O(14)	0·7048 (1)	0·1686 (1)	0·0611 (2)	4·97 (5)
C(15)	0·5745 (2)	0·1528 (1)	0·2159 (2)	3·75 (5)
C(16)	0·5646 (2)	0·1534 (2)	0·3543 (2)	5·10 (7)
C(17)	0·4653 (3)	0·1054 (2)	0·3875 (3)	5·98 (9)
C(18)	0·3773 (2)	0·0563 (2)	0·2842 (3)	5·82 (8)
C(19)	0·3855 (3)	0·0560 (2)	0·1460 (3)	6·40 (9)
C(20)	0·4835 (2)	0·1041 (2)	0·1110 (3)	5·50 (7)
(2) *A* (Occupancy factor 0·5)				
C(1)	0·8250 (5)	0·4530 (3)	0·3852 (4)	7·2 (1)
N(2)	0·7887 (3)	0·5424 (2)	0·3877 (3)	5·1 (1)
C(3)	0·7805 (4)	0·5925 (2)	0·2671 (4)	5·7 (1)
C(4)	0·8090 (3)	0·5606 (2)	0·1446 (3)	5·2 (1)
C(5)	0·8771 (6)	0·4053 (3)	0·0378 (6)	8·4 (2)
C(6)	1·0146 (6)	0·3858 (3)	0·1154 (9)	10·2 (3)
C(7)	1·0071 (6)	0·3228 (5)	0·2316 (7)	9·3 (2)
C(8)	0·8789 (5)	0·3089 (3)	0·2271 (5)	7·4 (1)
C(9)	0·8220 (5)	0·3158 (3)	0·0630 (6)	8·3 (2)
C(10)	0·8447 (4)	0·4107 (3)	0·2647 (4)	6·3 (1)
C(11)	0·8449 (4)	0·4697 (3)	0·1475 (4)	6·4 (1)
(2) *B* (Occupancy factor 0·5)				
C(1)	0·8856 (4)	0·4752 (3)	0·4138 (4)	6·0 (1)
N(2)	0·8296 (3)	0·5578 (2)	0·4015 (3)	5·6 (1)
C(3)	0·7758 (3)	0·5958 (2)	0·2680 (3)	4·2 (1)
C(4)	0·7825 (3)	0·5537 (2)	0·1366 (3)	4·2 (1)
C(5)	0·8706 (5)	0·4034 (3)	0·0413 (4)	6·7 (1)
C(6)	0·7887 (4)	0·3146 (3)	0·0409 (4)	6·4 (1)
C(7)	0·8565 (6)	0·2718 (4)	0·1930 (6)	9·2 (2)
C(8)	0·9590 (4)	0·3414 (3)	0·2727 (5)	6·6 (1)
C(9)	0·9971 (4)	0·3645 (3)	0·1312 (6)	7·3 (2)
C(10)	0·9052 (3)	0·4325 (2)	0·2961 (4)	5·5 (1)
C(11)	0·8452 (3)	0·4698 (3)	0·1523 (4)	5·6 (1)
(2)				
N(12)	0·7233 (1)	0·6817 (1)	0·2747 (1)	4·97 (4)
C(13)	0·6988 (2)	0·7515 (1)	0·1732 (2)	5·00 (4)
O(14)	0·7286 (2)	0·7480 (1)	0·0615 (1)	6·86 (5)
C(15)	0·6343 (2)	0·8352 (1)	0·2076 (2)	5·15 (4)
C(16)	0·5488 (2)	0·8295 (2)	0·2819 (3)	6·73 (6)
C(17)	0·4910 (2)	0·9097 (2)	0·3073 (3)	8·63 (9)
C(18)	0·5194 (2)	0·9961 (2)	0·2638 (3)	7·99 (8)
C(19)	0·6030 (2)	1·0007 (1)	0·1888 (2)	6·04 (5)
C(20)	0·6603 (2)	0·9224 (1)	0·1587 (2)	5·32 (5)
Cl(21)	0·63825 (6)	1·10998 (3)	0·13052 (7)	7·83 (2)
(3) *A* (Occupancy factor 0·67)				
C(1)	0·9873 (2)	0·7599 (1)	0·1765 (2)	4·37 (4)
N(2)	0·9564 (1)	0·8339 (1)	0·3277 (2)	4·50 (4)
C(3)	0·8254 (1)	0·8086 (1)	0·3425 (2)	3·37 (3)
C(4)	0·7179 (1)	0·7102 (2)	0·2068 (3)	4·79 (4)

Table 1 (*cont.*)

	x	y	z	B_{eq}
C(5)	0·6702 (2)	0·5237 (2)	−0·1214 (3)	6·37 (6)
C(6)	0·6805 (2)	0·5768 (3)	−0·3171 (3)	7·59 (8)
C(7)	0·8318 (2)	0·6031 (2)	−0·3365 (2)	5·63 (6)
C(8)	0·8881 (2)	0·5622 (1)	−0·1484 (2)	4·63 (5)
C(9)	0·7658 (2)	0·4497 (2)	−0·1373 (3)	5·84 (6)
C(10)	0·8881 (1)	0·6597 (1)	0·0374 (2)	3·74 (4)
C(11)	0·7529 (2)	0·6352 (1)	0·0544 (2)	4·61 (4)
(3) *B* (Occupancy factor 0·33)				
C(1)	0·9756 (3)	0·7696 (3)	0·1598 (4)	3·83 (7)
N(2)	0·9481 (2)	0·8458 (2)	0·3146 (3)	3·07 (5)
C(3)	0·8177 (3)	0·8289 (2)	0·3205 (4)	3·44 (7)
C(4)	0·7080 (3)	0·7377 (3)	0·1804 (4)	4·21 (8)
C(5)	0·6503 (4)	0·5597 (4)	−0·1584 (6)	5·93 (12)
C(6)	0·6650 (5)	0·4423 (5)	−0·1201 (10)	7·93 (17)
C(7)	0·8113 (5)	0·4548 (4)	−0·1324 (8)	6·97 (15)
C(8)	0·8724 (4)	0·5843 (4)	−0·1774 (7)	6·13 (12)
C(9)	0·7439 (7)	0·5840 (4)	−0·3207 (6)	7·60 (18)
C(10)	0·8718 (4)	0·6778 (4)	0·0150 (5)	5·30 (11)
C(11)	0·7371 (3)	0·6635 (3)	0·0227 (5)	4·82 (9)
(3)				
N(12)	0·8054 (1)	0·8950 (1)	0·5059 (1)	4·30 (2)
C(13)	0·6868 (1)	0·8932 (1)	0·5690 (2)	4·31 (3)
O(14)	0·5739 (1)	0·8270 (1)	0·4710 (1)	6·43 (3)
C(15)	0·7055 (1)	0·9800 (1)	0·7723 (1)	4·03 (3)
C(16)	0·8014 (1)	0·9872 (1)	0·9375 (2)	4·60 (3)
C(17)	0·8137 (1)	1·0674 (1)	1·1242 (2)	4·94 (3)
C(18)	0·7334 (1)	1·1421 (1)	1·1511 (2)	4·88 (3)
C(19)	0·6385 (1)	1·1336 (1)	0·9851 (2)	5·09 (3)
C(20)	0·6235 (1)	1·0531 (1)	0·7986 (2)	4·63 (3)
C(21)	0·7484 (2)	1·2302 (2)	1·3543 (2)	6·94 (5)
(4) *A* (Occupancy factor 0·71)				
C(5)	0·6045 (2)	0·0550 (6)	0·8431 (2)	4·94 (9)
C(6)	0·5606 (2)	0·0187 (6)	0·9174 (2)	5·52 (10)
C(7)	0·5879 (2)	0·2323 (6)	0·9698 (2)	5·18 (9)
C(8)	0·6456 (2)	0·3641 (5)	0·9203 (2)	4·35 (8)
C(9)	0·5948 (2)	0·3060 (7)	0·8323 (2)	5·59 (10)
C(10)	0·7319 (2)	0·2290 (4)	0·9267 (2)	3·58 (6)
C(11)	0·7072 (2)	0·0403 (5)	0·8793 (2)	3·94 (7)
(4) *B* (Occupancy factor 0·29)				
C(5)	0·5991 (5)	0·0032 (12)	0·8687 (5)	4·9 (2)
C(6)	0·5693 (5)	0·1909 (13)	0·8067 (5)	5·2 (2)
C(7)	0·5947 (6)	0·3993 (13)	0·8572 (7)	5·9 (3)
C(8)	0·6367 (5)	0·3115 (14)	0·9434 (5)	5·6 (2)
C(9)	0·5737 (5)	0·1057 (21)	0·9422 (6)	7·5 (3)
C(10)	0·7257 (4)	0·1980 (12)	0·9376 (4)	4·2 (2)
C(11)	0·7012 (5)	0·0074 (10)	0·8905 (5)	4·2 (2)
(4)				
C(1)	0·8201 (1)	0·2521 (3)	0·9682 (1)	3·67 (5)
N(2)	0·8856 (1)	0·1058 (2)	0·9615 (1)	3·27 (4)
C(3)	0·8606 (1)	−0·0708 (3)	0·9138 (1)	3·02 (4)
C(4)	0·7706 (1)	−0·1222 (3)	0·8734 (1)	3·90 (5)
N(12)	0·9360 (1)	−0·2040 (2)	0·9069 (1)	3·32 (4)
C(13)	0·9367 (1)	−0·3721 (3)	0·8535 (1)	3·21 (4)
O(14)	0·8692 (1)	−0·4526 (3)	0·8083 (1)	5·12 (4)
C(15)	1·0309 (1)	−0·4524 (3)	0·8527 (1)	3·07 (4)
C(16)	1·0891 (1)	−0·3188 (3)	0·8217 (1)	3·77 (5)
C(17)	1·1754 (1)	−0·3932 (4)	0·8193 (1)	4·17 (5)
C(18)	1·2017 (1)	−0·6015 (3)	0·8504 (1)	3·82 (5)
C(19)	1·1452 (1)	−0·7378 (3)	0·8812 (1)	3·97 (5)
C(20)	1·0587 (1)	−0·6643 (3)	0·8818 (1)	3·71 (5)
N(21)	1·2935 (1)	−0·6805 (4)	0·8494 (1)	5·48 (6)
O(22)	1·3476 (1)	−0·5503 (4)	0·8337 (2)	8·55 (8)
O(23)	1·3113 (1)	−0·8751 (4)	0·8648 (2)	9·12 (8)

ω–2θ scans, ω-scan width $(1{\cdot}5 + 0{\cdot}2\tan\theta)°$. 2763 unique reflections measured, 2466 intensities observed $[F_o > 3\sigma(F_o)]$. $\sum(w|\Delta F|^2)$ minimized, $w = 1/[\sigma^2(F_o) + 0{\cdot}00177|F_o|^2]$, $w = 0$ for 209 reflections with $w^{1/2}|\Delta F| \geq 3$. Final $R = 0{\cdot}049$, $wR = 0{\cdot}057$, $S = 1{\cdot}24$. The highest and lowest peaks in the final difference map are 0·6 and −0·4 e $Å^{-3}$. Max. Δ/σ in the final cycle 0·3.

(4): Colorless plates obtained from *n*-hexane. Crystal of dimensions 0·2 × 0·2 × 0·5 mm. Cell dimensions determined from 2θ angles for 15 reflections in the range $35 < 2\theta < 40°$. Intensities measured up to $\theta = 70°$ in h −18/17, k −7/0 and l 0/20, ω–2θ scans, ω-scan width $(1{\cdot}5 + 0{\cdot}2\tan\theta)°$. 2706 unique reflections measured, 2161 intensities observed [$F_o > 3\sigma(F_o)$ and one very strong reflection rejected]. $\sum(w|\Delta F|^2)$ minimized, $w = 1/[\sigma^2(F_o) + 0{\cdot}00132|F_o|^2]$, $w = 0$ for 81 reflections with $w^{1/2}|\Delta F| \geq 3$. The final $R = 0{\cdot}050$, $wR = 0{\cdot}061$, $S = 1{\cdot}23$. The highest and lowest peaks in the final difference map are 0·4 and −0·4 e Å^{-3}. Max. Δ/σ in the final cycle 0·2.

The occupancy factors and the final atomic parameters of (1)–(4) are given in Table 1.* Perspective views of the disordered structures are presented in Fig. 1, stereoviews are shown in Fig. 2. The two conformations, *A* and *B*, are the mirror image of each other with respect to the plane of the pyridine ring.

Related literature. Several types of acylamino molecules with an aminopyridine structure have been reported as new or potentially promising anti-ulcer agents (Bolhofer, Deana, Habecker, Hoffman, Gould, Pietruszkiewicz, Prugh, Torchiana, Cragoe & Hirschmann, 1983; Bouhayat, Piessard, Baut, Sparfel, Petit, Piriou & Welin, 1985; Moffett, Robert & Skaletzky, 1971). Compounds (1)–(4) were synthesized by Tanida, Irie & Wakisaka (1986). These results will be compared with those of IR and ^{1}H NMR spectra and those of conformational analyses using AM1 calculations in a separate paper.

* Lists of H-atom coordinates, anisotropic temperature factors of the non-H atoms, bond lengths, bond angles and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54281 (81 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

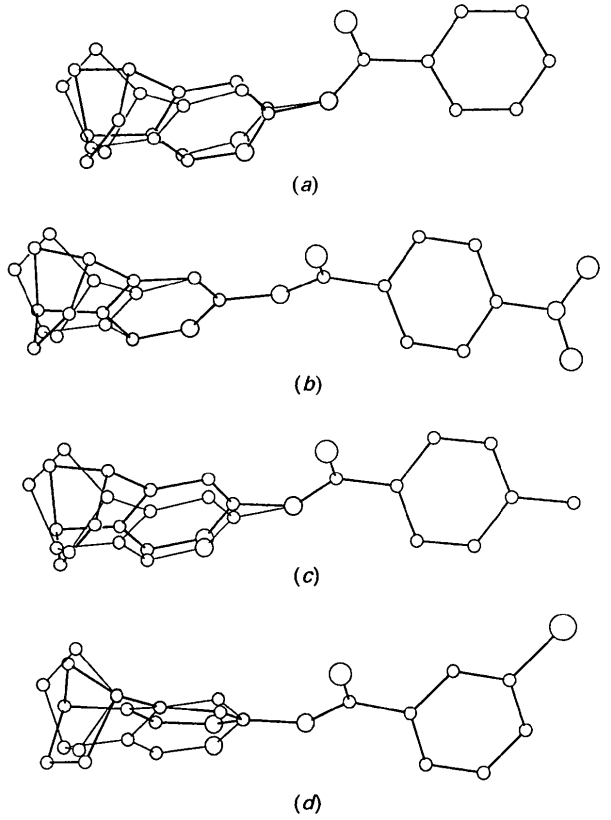


Fig. 1. Perspective views of the molecular structures (*a*) (1), (*b*) (2), (*c*) (3) and (*d*) (4), drawn by the program *PLUTO* (Motherwell & Clegg, 1978). Thin and thick lines show molecules *A* and *B*, respectively. The H atoms are omitted for clarity.

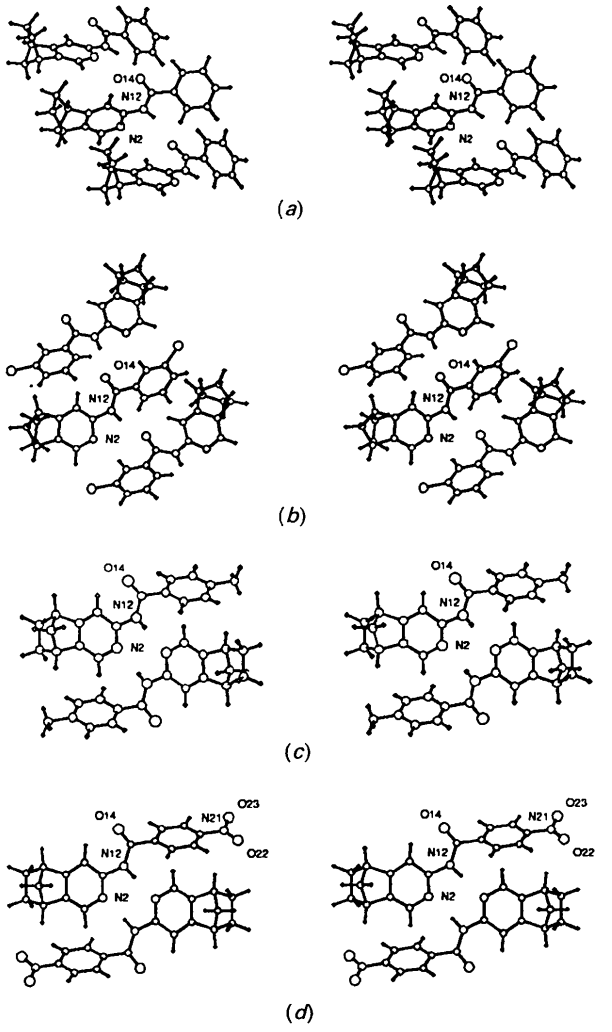


Fig. 2. Stereoviews of the intermolecular hydrogen-bonding systems (*a*) (1), (*b*) (2), (*c*) (3) and (*d*) (4) drawn by *PLUTO*. N and O atoms labeled on the molecule refer to the symmetry code (x, y, z).

The authors wish to thank Dr Hatsue Tamura of Osaka University for measurement of the low-temperature diffraction data.

References

BOLHOFER, W. A., DEANA, A. A., HABECKER, C. N., HOFFMAN, J. M., GOULD, N. P., PIETRUSZKIEWICZ, A. M., PRUGH, J. D., TORCHIANA, M. L., CRAGOE, E. J. JR & HIRSCHMANN, R. (1983). *J. Med. Chem.* **26**, 538–544.

BOUHAYAT, S., PIESSARD, S., BAUT, G. L., SPARFEL, L., PETIT, J. Y., PIRIOU, F. & WELIN, L. (1985). *J. Med. Chem.* **28**, 555–559.

HAZEKAMP, R. O. (1989). *CRYLSQ*, *XTAL2.6 User's Manual*, edited by S. R. HALL & J. M. STEWART. Univs. of Western Australia, Australia, and Maryland, USA.

MAIN, P., GERMAIN, G. & WOOLFSON, M. M. (1984). *MULTAN*84. *A Computer Program for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.

MOFFETT, R. B., ROBERT, A. & SKALETZKY, L. L. (1971). *J. Med. Chem.* **14**, 963–968.

MOTHERWELL, W. D. S. & CLEGG, W. (1978). *PLUTO*. Program for plotting molecular and crystal structures. Univ. of Cambridge, England.

TANIDA, H., IRIE, T. & WAKISAKA, Y. (1986). *J. Heterocycl. Chem.* **23**, 177–181.