has 12 nearest neighbours. One of them belongs to the same molecule (chain-linked). The remaining 11 distances between the centroids of the nearest non-linked cages are near to $b/2$ (= 7·05 Å) and range from
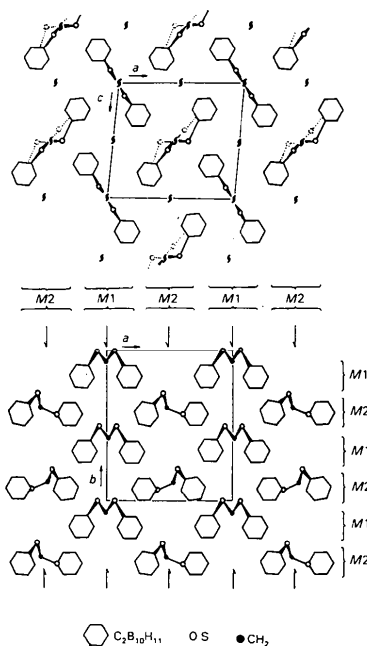

Fig. 2. Schematic view (H atoms omitted, icosahedra represented by hexagons) of the packing showing the positions of the molecules with respect to the screw axes and the layers of molecules 1 and molecules 2. In the projection along **b** (upper part of the figure) the molecules 1 eclipse one another and the molecules 2 are partially eclipsed (dotted lines). The projection along **c** (lower part of the figure) shows the nearly hexagonal close packing of the icosahedra.

6·913 (5) to 7·473 (5) Å (see Table 3). Owing to this the arrangement of the icosahedra in the crystal simulates hexagonal close packing with the principal axis parallel to [001]. This suggests that the packing of the icosahedra plays a more important role than the bonding properties of the linking —S—CH₂—S— chain in determining the crystal structure.

The shortest intermolecular distances range from 2·50 for H(21)—H(102) to 3·22 Å.

References

AHMED, F. R., HALL, S. R., PIPPY, M. E. & HUBER, C. P. (1966). NRC Crystallographic Programs for the IBM/360 System. *World List of Crystallographic Computer Programs*, 2nd ed., Appendix, p. 52. Utrecht: Oosthoek.
BUERGER, M. J. (1959). *Vector Space and its Application in Crystal-Structure Investigation*. New York: John Wiley.
HAMILTON, W. C. (1959). *Acta Cryst.* **12**, 609–610.
International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press.
MAIN, P., WOOLFSON, M. M., LESSINGER, L., GERMAIN, G. & DECLERCQ, J.-P. (1974). *MULTAN74. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain-la-Neuve, Belgium.
SHOEMAKER, D. P. (1970). *J. Appl. Cryst.* **3**, 179–180.
STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.
ŠUBRTOVÁ, V., LÍNEK, A. & HAŠEK, J. (1980). *Acta Cryst.* **B36**, 858–861.

Structure of N-(3-*tert*-Butyl-2-fluoro-1-indanyl)acetamide, C₁₅H₂₀FNO

By ELIANE LAURENT, HÉLÈNE LEFRANC AND ROBERT TARDIVEL

Laboratoire de Chimie Organique III, ERA CNRS 611, Université Lyon I, 43 Boulevard du 11 Novembre 1918, 69622 Villeurbanne, France

AND RENÉ FAURE AND HENRI LOISELEUR

Laboratoire de Chimie Analytique II, Université Lyon I, 43 Boulevard du 11 Novembre 1918, 69622 Villeurbanne, France

Abstract. $M_r = 249\cdot3$, orthorhombic, *Pbca*, $a = 9\cdot740$ (2), $b = 27\cdot692$ (3), $c = 21\cdot460$ (2) Å, $V = 5788$ (2) Å³, $Z = 16$, $D_x = 1\cdot14$ Mg m⁻³, λ(Cu $K\bar{\alpha}$) = 1·5424 Å, $\mu = 0\cdot66$ mm⁻¹, $F(000) = 2144$, $T = 298$ K. Final $R = 0\cdot031$ for 689 observed reflections. The molecules show a *cis* configuration concerning the F atom and the acetamide group, both of them being in a *trans* position with respect to the *tert*-butyl group.

Introduction. In acetonitrile solution containing triethylamine trihydrofluoride, indene is electrochemically oxidized in a regiospecific and stereoselective way to cis-N-(2-fluoro-1-indanyl)acetamide (Bensadat, Laurent & Tardivel, 1981). The study of the stereoselectivity was carried out with 1-tert-butylindene as a reactant. Among the four possible stereoisomers only two are formed ($A/B = 87/13$). ^{1}H and ^{19}F NMR spectra of the major isomer A elucidated the relative configuration of the F atom and the acetamide group but not that of the tert-butyl group. Thus its X-ray structure determination was carried out.

Experimental. Colourless, very thin plates from petroleum ether–benzene 10:1 (v/v), $0.025 \times 0.40 \times 0.50$ mm. CAD-4 Nonius diffractometer, graphite monochromator, ω–$\frac{4}{3}\theta$ scan, $2 < 2\theta < 146°$. 25 reflections used for lattice-parameter refinement. 4786 reflections ($0 \leq h \leq 12$, $0 \leq k \leq 26$, $0 \leq l \leq 22$), only 689 observed according to $I > 1.2\sigma(I)$ and $I > 1.3I_{background}$. Lp correction, absorption ignored. Systematic absences: $0kl$, k odd; $h0l$, l odd; $hk0$, h odd. Standard reflection $0\bar{6}6$ varied within 4% range. Two molecules in asymmetric unit. Direct-methods program

MULTAN (Germain, Main & Woolfson, 1970) involving 250 first $|E| > 1.60$. Anisotropic refinement on F; H from ΔF, isotropic, each refined with B equal to B_{eq} of bound atom. Last refinement involved β_{ij} and x,y,z of all atoms; final $R = 0.031$, $wR = 0.039$, $S = 0.64$; $w = (a + b|F_o|)^{-2}$. $(\Delta/\sigma)_{max} = 0.3$ for non-H atoms, $\Delta\rho \leq 0.08$ e Å^{-3}. f_i's from International Tables for X-ray Crystallography (1974).

Discussion. Atomic parameters are given in Table 1, bond distances and angles in Table 2.* Only one molecule of A (molecule 1 in Table 1) is depicted in Fig. 1 (drawn with ORTEP, Johnson, 1965). Molecule (2) is isostructural.

---

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and least-squares-planes' data have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38662 (28 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Refined relative coordinates and equivalent isotropic thermal parameters* (Å^2) *for the non-H atoms of the two isostructural molecules*

$$B_{eq} = \frac{4}{3}\sum_i \sum_j \beta_{ij}\,\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}
Molecule (1)				
F(1)	0.0909 (5)	0.4869 (2)	0.3582 (2)	6.4 (4)
O(1)	0.4225 (5)	0.4166 (2)	0.2655 (2)	5.8 (5)
N(1)	0.1975 (6)	0.4171 (2)	0.2852 (3)	4.4 (5)
C(1)	0.1921 (9)	0.4592 (3)	0.3885 (4)	4.9 (9)
C(2)	0.2133 (7)	0.4129 (3)	0.3537 (3)	4.5 (7)
C(3)	0.1102 (7)	0.3786 (3)	0.3819 (3)	3.8 (6)
C(4)	0.0606 (8)	0.3347 (3)	0.3591 (4)	5.0 (6)
C(5)	−0.0377 (8)	0.3104 (4)	0.3949 (4)	5.8 (7)
C(6)	−0.0807 (10)	0.3281 (4)	0.4500 (4)	7.4 (8)
C(7)	−0.0321 (9)	0.3724 (4)	0.4720 (4)	5.7 (8)
C(8)	0.0689 (7)	0.3987 (3)	0.4383 (3)	3.9 (6)
C(9)	0.1337 (9)	0.4465 (3)	0.4538 (4)	4.4 (7)
C(10)	0.2384 (8)	0.4467 (3)	0.5078 (3)	4.8 (6)
C(11)	0.3568 (10)	0.4126 (5)	0.4914 (6)	8.6 (9)
C(12)	0.1676 (12)	0.4281 (5)	0.5660 (4)	8.1 (8)
C(13)	0.2882 (12)	0.4979 (4)	0.5176 (6)	7.5 (9)
C(14)	0.3048 (9)	0.4201 (3)	0.2459 (4)	5.4 (7)
C(15)	0.2699 (10)	0.4275 (4)	0.1781 (4)	6.3 (7)
Molecule (2)				
F(2)	0.3954 (5)	0.1976 (2)	0.2214 (2)	7.7 (5)
O(2)	0.6725 (6)	0.0800 (2)	0.2698 (2)	7.0 (6)
N(2)	0.4594 (7)	0.1098 (3)	0.2603 (4)	5.6 (5)
C(16)	0.4855 (8)	0.1992 (3)	0.2748 (4)	4.9 (8)
C(17)	0.4755 (8)	0.1492 (3)	0.3036 (4)	4.2 (6)
C(18)	0.3563 (8)	0.1558 (3)	0.3476 (4)	4.5 (6)
C(19)	0.2893 (10)	0.1167 (4)	0.3742 (4)	6.3 (8)
C(20)	0.1747 (12)	0.1276 (4)	0.4120 (5)	8.1 (9)
C(21)	0.1407 (10)	0.1753 (5)	0.4239 (5)	7.8 (9)
C(22)	0.2117 (10)	0.2119 (4)	0.3955 (4)	7.1 (7)
C(23)	0.3278 (9)	0.2016 (3)	0.3581 (4)	5.2 (8)
C(24)	0.4267 (9)	0.2361 (3)	0.3223 (4)	5.6 (7)
C(25)	0.5369 (8)	0.2637 (3)	0.3607 (4)	6.6 (7)
C(26)	0.6305 (12)	0.2278 (4)	0.3933 (5)	6.6 (9)
C(27)	0.4608 (13)	0.2929 (4)	0.4099 (5)	8.0 (9)
C(28)	0.6226 (13)	0.2938 (4)	0.3144 (5)	8.4 (8)
C(29)	0.5534 (10)	0.0754 (3)	0.2465 (4)	5.2 (7)
C(30)	0.5164 (11)	0.0346 (4)	0.2069 (5)	5.9 (7)

Table 2. *Main intramolecular bond lengths* (Å) *and angles* (°)

	Molecule (1)		Molecule (2)
C(1)–F(1)	1.407 (8)	C(16)–F(2)	1.443 (7)
C(2)–N(1)	1.481 (8)	C(17)–N(2)	1.442 (9)
C(2)–C(1)	1.498 (10)	C(17)–C(16)	1.519 (10)
C(3)–C(2)	1.509 (9)	C(18)–C(17)	1.506 (9)
C(4)–C(3)	1.396 (9)	C(19)–C(18)	1.387 (10)
C(5)–C(4)	1.401 (10)	C(20)–C(19)	1.412 (12)
C(6)–C(5)	1.346 (10)	C(21)–C(20)	1.387 (12)
C(7)–C(6)	1.398 (11)	C(22)–C(21)	1.369 (12)
C(8)–C(3)	1.391 (9)	C(23)–C(18)	1.319 (9)
C(8)–C(7)	1.422 (10)	C(23)–C(22)	1.415 (10)
C(9)–C(1)	1.554 (11)	C(24)–C(16)	1.554 (9)
C(9)–C(8)	1.504 (10)	C(24)–C(23)	1.558 (10)
C(10)–C(9)	1.543 (9)	C(25)–C(24)	1.555 (11)
C(11)–C(10)	1.531 (11)	C(26)–C(25)	1.520 (12)
C(12)–C(10)	1.517 (10)	C(27)–C(25)	1.522 (13)
C(13)–C(10)	1.512 (10)	C(28)–C(25)	1.542 (13)
C(14)–O(1)	1.224 (8)	C(29)–O(2)	1.270 (9)
C(14)–N(1)	1.346 (8)	C(29)–N(2)	1.353 (9)
C(15)–C(14)	1.509 (11)	C(30)–C(29)	1.459 (11)
C(2)–N(1)–C(14)	123.0 (6)	C(17)–N(2)–C(29)	126.9 (7)
F(1)–C(1)–C(2)	109.4 (6)	F(2)–C(16)–C(17)	104.9 (6)
F(1)–C(1)–C(9)	106.5 (6)	F(2)–C(16)–C(24)	108.5 (6)
C(2)–C(1)–C(9)	107.9 (7)	C(17)–C(16)–C(24)	107.9 (7)
C(1)–C(2)–N(1)	114.5 (7)	C(16)–C(17)–N(2)	115.7 (7)
C(1)–C(2)–C(3)	104.3 (6)	C(16)–C(17)–C(18)	101.2 (7)
N(1)–C(2)–C(3)	112.1 (6)	N(2)–C(17)–C(18)	114.3 (7)
C(2)–C(3)–C(4)	129.6 (7)	C(17)–C(18)–C(19)	121.7 (8)
C(2)–C(3)–C(8)	106.8 (7)	C(17)–C(18)–C(23)	112.7 (8)
C(4)–C(3)–C(8)	123.6 (8)	C(19)–C(18)–C(23)	125.5 (9)
C(3)–C(4)–C(5)	117.6 (8)	C(18)–C(19)–C(20)	116.2 (10)
C(4)–C(5)–C(6)	121.3 (10)	C(19)–C(20)–C(21)	119.9 (10)
C(5)–C(6)–C(7)	120.7 (10)	C(20)–C(21)–C(22)	120.1 (11)
C(6)–C(7)–C(8)	120.8 (8)	C(21)–C(22)–C(23)	120.5 (11)
C(3)–C(8)–C(7)	116.0 (8)	C(18)–C(23)–C(22)	117.3 (9)
C(3)–C(8)–C(9)	115.0 (7)	C(18)–C(23)–C(24)	112.0 (8)
C(7)–C(8)–C(9)	128.9 (7)	C(22)–C(23)–C(24)	130.6 (9)
C(1)–C(9)–C(8)	98.8 (6)	C(16)–C(24)–C(23)	98.5 (6)
C(1)–C(9)–C(10)	115.7 (6)	C(16)–C(24)–C(25)	114.7 (7)
C(8)–C(9)–C(10)	116.6 (6)	C(23)–C(24)–C(25)	117.8 (7)
C(9)–C(10)–C(11)	108.9 (6)	C(24)–C(25)–C(26)	109.6 (8)
C(9)–C(10)–C(12)	108.4 (7)	C(24)–C(25)–C(27)	107.1 (8)
C(9)–C(10)–C(13)	108.7 (7)	C(24)–C(25)–C(28)	107.3 (8)
C(11)–C(10)–C(12)	108.8 (9)	C(26)–C(25)–C(27)	108.7 (9)
C(11)–C(10)–C(13)	111.6 (8)	C(26)–C(25)–C(28)	109.0 (9)
C(12)–C(10)–C(13)	110.4 (8)	C(27)–C(25)–C(28)	115.1 (9)
O(1)–C(14)–N(1)	120.5 (8)	O(2)–C(29)–N(2)	117.5 (7)
O(1)–C(14)–C(15)	123.6 (7)	O(2)–C(29)–C(30)	122.1 (9)
N(1)–C(14)–C(15)	115.9 (8)	N(2)–C(29)–C(30)	120.4 (9)

The F atom and the acetamide group are in *cis* position and the *tert*-butyl group is *trans* to the F atom.
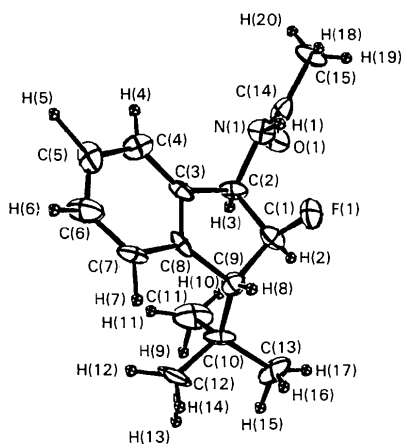

Fig. 1. 25% probability thermal ellipsoids for molecule (1) (H atoms are arbitrarily scaled).

Thus, this isomer is the major product of the electro-fluoroacetamidation process. Comparison of the ^{1}H and ^{19}F NMR spectra of A with those of the major isomer obtained in the same way from non-substituted indene has confirmed that in this latter case the major isomer is also *cis*.

Finally, as a structural feature we may note that the least-squares mean-plane calculations (deposited) involving the six C atoms of the phenyl ring show that C(2) and C(9) [C(17) and C(24) for molecule 2] lie in this plane while C(1) [C(16)] deviates by more than 0·4 Å.

References

BENSADAT, A., LAURENT, E. & TARDIVEL, R. (1981). *Nouv. J. Chim.* **5**, 397–411.
GERMAIN, G., MAIN, P. & WOOLFSON, M. M. (1970). *Acta Cryst.* **B26**, 274–285.
International Tables for X-ray Crystallography (1974). Vol. IV, p. 99. Birmingham: Kynoch Press.
JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee.

A Hydrated 1:1 Complex between Niflumic Acid and Ethanolamine,*
$C_{13}H_8F_3N_2O_2^-.C_2H_8NO^+.H_2O$

BY V. DHANARAJ AND M. VIJAYAN

Molecular Biophysics Unit, Indian Institute of Science, Bangalore-560012, India

Abstract. $M_r = 361\cdot3$, triclinic, $P\bar{1}$, $a = 6\cdot239$ (2), $b = 11\cdot280$ (2), $c = 12\cdot451$ (2) Å, $\alpha = 101\cdot2$ (1), $\beta = 92\cdot3$ (1), $\gamma = 99\cdot9$ (1)°, $V = 844\cdot123$ Å^3, $Z = 2$, $D_x = 1\cdot42$, $D_m = 1\cdot42$ (1) Mg m^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 1\cdot102$ mm^{-1}, $F(000) = 376$, $T = 293$ K. Final $R = 0\cdot064$ for 2150 observed reflections. The niflumic acid anions consist essentially of three planar groupings, namely, two six-membered rings and a carboxylate group attached to one of them. The invariant common structural features observed in the crystal structures of fenamates, namely, the coplanarity of the carboxyl group and the six-membered ring bearing it, and the internal hydrogen bond between the carboxyl group and the imino N atom that bridges the two six-membered rings, are retained in the complex. The amino N atom is *gauche* with respect to the terminal hydroxyl group in the ethanolamine cation. The complexation between the two molecules is achieved through ionic and hydrogen-bonded interactions involving the carboxylate group in niflumic acid.

Introduction. Non-steroidal anti-inflammatory analgesics like salicylates, pyrazole derivatives and fenamates are believed to act through the inhibition of prostaglandin biosynthesis (Flower, 1974). We have been carrying out X-ray studies on analgetic pyrazole derivatives and fenamates, and their complexes in an attempt to understand their molecular geometry, the nature of the non-covalent interactions they are likely to be involved in, and the geometric and electronic consequences of such interactions (Singh & Vijayan, 1974, 1977; Krishna Murthy & Vijayan, 1981a). The crystal structures analysed include niflumic acid (Krishna Murthy & Vijayan, 1979), meclofenamic acid (Krishna Murthy & Vijayan, 1981b) and flufenamic acid (Krishna Murthy, Bhat & Vijayan, 1982). The crystal structures of mefenamic acid and a different form of flufenamic acid have been reported earlier (McConnell, 1973, 1976). Here we report the crystal structure of a 1:1 complex of niflumic acid (2-{[3-(tri-fluoromethyl)phenyl]amino}-3-pyridinecarboxylic acid) (I) with ethanolamine (2-aminoethanol) (II). This represents the first structure determination of a complex involving an analgetic fenamate.