

There are no unusually close intermolecular contacts; the packing is illustrated in Fig. 2.

A more detailed discussion will be given when structural data of the pyridine analogue (2) are available.

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Structure of Tefludazine, 2-{4-[3-(4-Fluorophenyl)-6-(trifluoromethyl)-1-indanyl]piperazinyl}ethanol, C₂₂H₂₄F₄N₂O

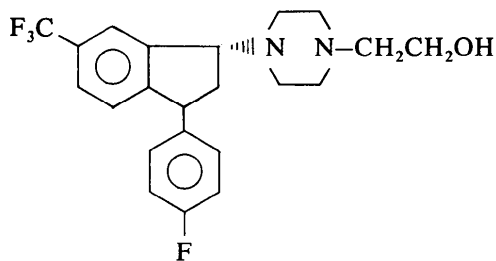
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Abstract. $M_r = 408.44$, monoclinic, $P2_1/c$, $a = 13.20$ (1), $b = 13.378$ (6), $c = 12.55$ (1) Å, $\beta = 112.60$ (8)°, $V = 2046$ (3) Å³, $Z = 4$, $D_m = 1.328$ Mg m⁻³, $D_x = 1.326$ (2) Mg m⁻³, Mo $K\alpha$, $\lambda = 0.71069$ Å, $\mu = 0.101$ mm⁻¹, $F(000) = 856$, room temperature, $R = 0.055$ for 2191 diffractometer-collected reflections. The two substituents of the indan ring system are situated *trans* to each other.

Introduction. The potent and long-acting neuroleptic compound tefludazine was developed by systematic variation of structural components (Bøgesø, 1983). The structure determination was undertaken in order to confirm the relative configuration proposed on the basis of the NMR spectra.



Experimental. Crystals grown by Dr K. P. Bøgesø, H. Lundbeck & Co A/S, from a mixture of diisopropyl ether and hexane, unit-cell parameters from least-

squares refinement of the θ angles ($|\theta| \leq 15^\circ$) for 24 reflections, Nonius CAD-3 diffractometer, D_m by flotation, irregularly shaped crystal, of approximate size 0.30 × 0.34 × 0.36 mm, mounted in silicon grease in a glass capillary, ω -scan technique, 3974 reflections measured in one quadrant with $(\sin \theta)/\lambda \leq 0.599$ Å⁻¹, $-13 \leq h \leq 15$, $-15 \leq k \leq 0$, $-14 \leq l \leq 0$, 2192 with $I_{net} \geq 3.0\sigma(I)$, where σ is the standard deviation from counting statistics; intensity of the three standard reflections did not vary significantly, no absorption correction; structure solved with *MULTAN77* (Main, Lessinger, Woolfson, Germain & Declercq, 1977), trial structure refined with *XRAY72* (Stewart, Kruger, Ammon, Dickinson & Hall, 1972); final cycles of refinement, based on $|F|$, included one scale factor and positional and anisotropic thermal parameters for all non-H atoms; H atoms located in difference electron density map included in refinement as a fixed contribution in calculated positions (C–H = 1.0 Å), with a common thermal parameter ($B = 4.0$ Å²); position of the hydroxyl hydrogen atom taken directly from the difference electron density map; unit weights [weight analysis showed them to be satisfactory] were used, final $R = R_w = 0.055$, $S = 1.53$, ratios of maximum and average shift to error in the last refinement cycle were 0.32 and 0.12, respectively, minimum and maximum heights in final difference Fourier synthesis were -0.2 and 0.3 e Å⁻³, respectively, X-ray scattering factors for H from Stewart Davidson & Simpson

(1965), for all other atoms from *International Tables for X-ray Crystallography* (1974).*

Discussion. The final coordinates and equivalent isotropic thermal parameters of all non-H atoms are given in Table 1, and in Tables 2 and 3 are listed bond distances and angles, selected torsion angles, and interplanar angles. A stereoview of the molecule is shown in Fig. 1. High values of the thermal parameters were found for the fluorine atoms and Fig. 1 may therefore leave the erroneous impression that these atoms take up much more space than the hydrogen atoms. The five-membered ring has a rather flat

* Lists of structure factors, positional parameters for H atoms and anisotropic thermal parameters for non-H atoms have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38480 (18 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Positional parameters ($\times 10^4$) and equivalent isotropic thermal parameters ($\text{\AA}^2 \times 10^2$)

	x	y	z	U_{eq}^*
C(1)	2443 (3)	5026 (3)	6898 (3)	4.0 (3)
C(2)	2064 (3)	5126 (3)	5569 (4)	4.6 (3)
C(3)	2353 (3)	4120 (3)	5115 (4)	4.3 (3)
C(4)	4011 (4)	2953 (3)	6234 (4)	5.2 (3)
C(5)	4821 (4)	2716 (4)	7299 (4)	5.3 (3)
C(6)	4895 (3)	3248 (3)	8277 (4)	4.5 (3)
C(7)	4161 (3)	4002 (3)	8231 (4)	4.1 (3)
C(8)	3341 (3)	4228 (3)	7177 (4)	4.1 (3)
C(9)	3282 (3)	3713 (3)	6187 (4)	4.0 (3)
C(61)	5820 (4)	3000 (4)	9400 (5)	6.2 (4)
F(62)	5812 (3)	3542 (3)	10296 (3)	8.2 (3)
F(63)	6796 (2)	3155 (3)	9363 (3)	8.9 (4)
F(64)	5789 (3)	2058 (3)	9716 (3)	10.2 (4)
N(11)	352 (3)	3618 (3)	8365 (3)	4.5 (2)
C(12)	857 (4)	4583 (3)	8828 (4)	4.9 (3)
C(13)	1840 (4)	4759 (3)	8506 (4)	4.6 (3)
N(14)	1504 (3)	4790 (3)	7243 (3)	3.7 (2)
C(15)	935 (3)	3847 (3)	6744 (4)	4.2 (3)
C(16)	-28 (3)	3666 (3)	7103 (4)	4.7 (3)
C(17)	-542 (4)	3390 (4)	8759 (4)	5.8 (3)
C(18)	-1084 (3)	2383 (4)	8345 (4)	5.6 (3)
O(19)	-301 (2)	1605 (2)	8667 (3)	5.6 (2)
C(21)	2651 (3)	4225 (3)	4066 (3)	4.1 (3)
C(22)	3518 (4)	4834 (4)	4074 (4)	5.2 (3)
C(23)	3798 (4)	4924 (4)	3122 (4)	5.7 (3)
C(24)	3202 (4)	4389 (4)	2155 (4)	5.2 (3)
C(25)	2349 (4)	3772 (4)	2107 (4)	5.1 (3)
C(26)	2075 (4)	3691 (3)	3063 (4)	4.7 (3)
F(27)	3477 (3)	4464 (3)	1212 (3)	7.3 (2)
H(19)	-700	930	8350	

$$* U_{eq} = (U_1 U_2 U_3)^{1/3}.$$

Table 2. Bond lengths (\AA)

C(1)—C(2)	1.552 (6)	C(13)—C(12)	1.518 (8)
C(2)—C(3)	1.565 (6)	C(12)—N(11)	1.467 (6)
C(3)—C(9)	1.530 (6)	N(11)—C(16)	1.468 (6)
C(9)—C(8)	1.396 (7)	C(16)—C(15)	1.522 (7)
C(8)—C(1)	1.532 (6)	C(15)—N(14)	1.479 (6)
C(9)—C(4)	1.386 (6)	N(11)—C(17)	1.476 (8)
C(4)—C(5)	1.390 (6)	C(17)—C(18)	1.520 (7)
C(5)—C(6)	1.389 (7)	C(18)—O(19)	1.412 (5)
C(6)—C(7)	1.384 (6)	C(3)—C(21)	1.518 (7)
C(7)—C(8)	1.383 (6)	C(21)—C(22)	1.402 (7)
C(6)—C(61)	1.505 (6)	C(22)—C(23)	1.385 (8)
C(61)—F(62)	1.341 (8)	C(23)—C(24)	1.368 (7)
C(61)—F(63)	1.323 (7)	C(24)—C(25)	1.379 (8)
C(61)—F(64)	1.327 (7)	C(25)—C(26)	1.384 (8)
C(1)—N(14)	1.495 (6)	C(26)—C(21)	1.392 (6)
N(14)—C(13)	1.473 (6)	C(24)—F(27)	1.368 (7)

Table 3. Interbond angles, selected torsion angles, and angles between planes ($^\circ$)

E.s.d.'s on interplanar angles are $< 1^\circ$.			
C(1)—C(2)—C(3)	107.1 (3)	C(1)—N(14)—C(15)	113.6 (4)
C(2)—C(3)—C(9)	102.2 (3)	N(11)—C(12)—C(13)	109.6 (4)
C(3)—C(9)—C(8)	111.0 (4)	C(12)—C(13)—N(14)	110.9 (4)
C(9)—C(8)—C(1)	111.5 (3)	C(13)—N(14)—C(15)	109.0 (4)
C(8)—C(1)—C(2)	102.0 (4)	N(14)—C(15)—C(16)	110.9 (4)
C(3)—C(9)—C(4)	127.4 (4)	C(15)—C(16)—N(11)	110.4 (3)
C(8)—C(9)—C(4)	121.6 (4)	C(16)—N(11)—C(12)	107.7 (4)
C(9)—C(4)—C(5)	118.3 (5)	C(12)—N(11)—C(17)	110.4 (4)
C(4)—C(5)—C(6)	120.0 (5)	C(16)—N(11)—C(17)	112.6 (3)
C(5)—C(6)—C(7)	121.7 (4)	N(11)—C(17)—C(18)	113.6 (4)
C(6)—C(7)—C(8)	118.5 (4)	C(17)—C(18)—O(19)	111.0 (3)
C(7)—C(8)—C(9)	119.9 (4)	C(2)—C(3)—C(21)	114.6 (4)
C(7)—C(8)—C(1)	128.6 (4)	C(9)—C(3)—C(21)	114.1 (4)
C(5)—C(6)—C(61)	118.1 (4)	C(3)—C(21)—C(22)	122.0 (3)
C(7)—C(6)—C(61)	120.2 (4)	C(3)—C(21)—C(26)	120.2 (4)
C(6)—C(61)—F(62)	113.6 (4)	C(21)—C(22)—C(23)	122.2 (4)
C(6)—C(61)—F(63)	112.5 (5)	C(22)—C(23)—C(24)	117.8 (5)
C(6)—C(61)—F(64)	112.5 (4)	C(23)—C(24)—C(25)	122.3 (5)
F(62)—C(61)—F(63)	105.4 (4)	C(24)—C(25)—C(26)	119.3 (4)
F(62)—C(61)—F(64)	104.6 (5)	C(25)—C(26)—C(21)	120.7 (5)
F(63)—C(61)—F(64)	107.6 (4)	C(26)—C(21)—C(22)	117.7 (4)
C(8)—C(1)—N(14)	116.2 (3)	C(23)—C(24)—F(27)	118.4 (5)
C(2)—C(1)—N(14)	111.6 (3)	C(25)—C(24)—F(27)	119.3 (4)
C(1)—N(14)—C(13)	112.2 (3)		

	Experiment	MM2
C(1)—C(8)—C(9)—C(3)	1.4 (5)	0.4
C(8)—C(9)—C(3)—C(2)	13.9 (5)	13.3
C(9)—C(8)—C(1)—C(2)	-16.1 (5)	-13.8
C(9)—C(3)—C(2)—C(1)	-23.6 (4)	-21.4
C(8)—C(1)—C(2)—C(3)	24.3 (4)	21.4

\angle Plane I, plane II 80.5
 \angle Plane III, plane IV 129.0
 \angle Plane IV, plane V 124.4

Plane I C(4), C(5), C(6), C(7), C(8), C(9)
 Plane II C(21), C(22), C(23), C(24), C(25), C(26)
 Plane III C(13), N(14), C(15)
 Plane IV C(12), C(13), C(15), C(16)
 Plane V C(12), N(11), C(16)

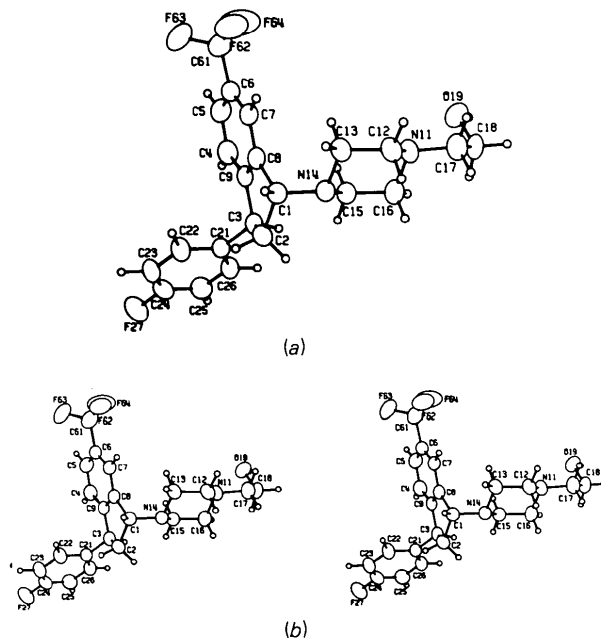


Fig. 1. (a) Atom numbering. (b) Stereoview of molecular structure. Non-hydrogen atoms are represented by their thermal ellipsoids drawn at the 50% probability level and hydrogen atoms as spheres of an arbitrary radius. The drawing was produced by ORTEP (Johnson, 1971).

envelope conformation with C(2) on the flap, 0.41 Å from plane I (*cf.* Table 3). Force-field calculations performed for a model [*trans*-3-(dimethylamino)-5-(1-methylethenyl)cyclopentene] of the central part of the title compound with the program *MM2* (Allinger, 1977) indicate that only very little energy (<5 kJ mol⁻¹) is needed to change the torsion angle C(3)–C(2)–C(1)–C(8) from the observed ~24° through zero to –20°, and no significant barrier has to be surmounted (*cf.* Fig. 2). This indicates that the molecule is rather flexible and that conformations different from the one observed in the crystal are accessible. The two benzene rings are roughly perpendicular to each other.

The piperazine ring has the normal chair conformation with both exocyclic N–C bonds equatorial. The torsion angle N(11)–C(17)–C(18)–O(19) is 55.3 (5)°. The hydroxyl H atom has no contact with N(11) but is engaged in *intermolecular* H bonding with N(14). The distances from O(19) and H(19) to N(14) are 2.886 (5) and 1.84 Å, respectively, and the angle O(19)–H(19)···N(14) is 176°. H bonds of the type described interlink the molecules in chains (*cf.* Fig. 3). All other *intermolecular* contacts are van der Waals contacts.

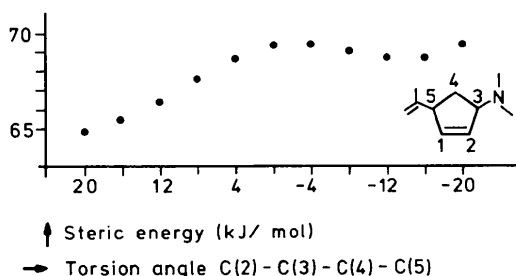


Fig. 2. Results of *MM2* calculations on the flexibility of a model [*trans*-3-(dimethylamino)-5-(1-methylethenyl)cyclopentene] of the central part of the tefudazine molecule. The torsion angle C(2)–C(3)–C(4)–C(5) of the cyclopentene ring has been varied in steps of 4°. In each step the geometry of the model was fully optimized.

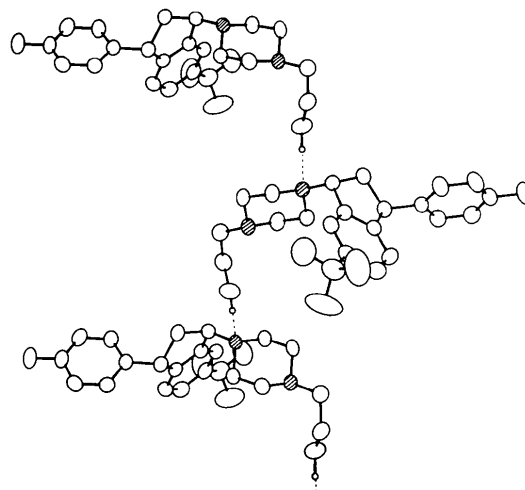


Fig. 3. Hydrogen bonds OH···N interlink the molecules in chains. Nitrogen atoms are shaded.

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trans-2-Isopropyl-*N,N*-dimethyl-1,3-dioxane-5-carbothioamide, C₁₀H₁₉NO₂S

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Abstract. $M_r = 217.33$, orthorhombic, $P2_12_12_1$, $a = 5.169$ (1), $b = 11.339$ (2), $c = 20.244$ (4) Å, $V = 1186.5$ (2) Å³, $Z = 4$, $F(000) = 472$, $D_x = 1.22$, $D_m =$

1.25 Mg m⁻³ (flotation at 298 K), $\lambda(\text{Cu } K\alpha) = 1.5418$ Å, $\mu(\text{Cu } K\alpha) = 2.22$ mm⁻¹; final $R = 0.065$ for 1255 observed reflections. The dihedral angle between the least-squares planes of the dioxane ring, which is in a distorted chair conformation, and the dimethylcarbothioamide group is 98.7 (2)°.

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