

Acta Cryst. (1973). B29, 2971**8-[4-(4-Fluorophenyl)-3-pentenyl]-1-phenyl-1,3,8-triaza-spiro[4,5]decan-4-one (Spirilene)**

BY M. H. J. KOCH

Laboratoire de Chimie Physique et de Crystallographie, Université de Louvain, Schapenstraat 39, B-3000 Leuven, Belgium

AND G. EVRARD

Département de Chimie, Facultés Universitaires, 61 rue de Bruxelles, B-5000 Namur, Belgium

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Abstract. Monoclinic; $P2_1/n$; $a=17.703$ (5), $b=6.277$ (3), $c=20.320$ (5) Å; $\beta=114.31$ (2)°; 25°C; $C_{24}H_{28}FN_3O$; $M=393.49$; $Z=4$; $F(000)=840$.

Introduction. This compound is a neuroleptic related to spiperone. Transparent needles were obtained by slow evaporation from ethanol.

The lattice parameters were obtained by the accurate measurement of the Bragg angle of twelve reflexions on a Nonius CAD-4 computer controlled diffractometer. Weissenberg photographs showed systematic absences corresponding to space group $P2_1/n$ ($0k0$: k odd; $h0l$: $h+l$ odd). The instrumental settings used during the data collection are given in Table 1.

The structure was solved by direct methods with *MULTAN* (Germain, Main & Woolfson, 1971). The most probable map contained the whole structure.

Table 1. *Experimental conditions*

Source Cu $K\alpha$; $\lambda=1.5418$ Å, ω - 2θ scan; graphite monochromator; $\Delta 2\theta=0.7+0.3 \text{ tg } \theta$; $\theta_{\min}=2.0^\circ$; $\theta_{\max}=70.0^\circ$; aperture $=3.0+0.5 \text{ tg } \theta$. Confidence level: 2.7.
Total number of independent reflexions: 4032
Total observed: 2437

Block-diagonal least-squares refinement with the program written by Ahmed, Hall, Pippy & Huber (1966) resulted in $R=\sum||F_o|-|F_c||/\sum|F_o|=0.09$ for all observed reflexions.* The weight of each reflexion in

* A list of structure factors has been deposited with the National Lending Library, England, as Supplementary Publication No. SUP 30211 (23 pp., 1 microfiche). Copies may be obtained through the Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Table 2. *Final positional and thermal parameters* ($\times 10^4$) (with standard deviations in parentheses)
$$B = \exp [-(B_{11}h^2 + B_{22}k^2 + B_{33}l^2 + B_{12}hk + B_{13}hl + B_{23}kl)].$$

	x/a	y/b	z/c	B_{11}	B_{22}	B_{33}	B_{23}	B_{13}	B_{12}
N(1)	927 (1)	5934 (4)	10780 (1)	32	321	21	11	13	-55
C(2)	1685 (2)	6060 (5)	11427 (1)	34	348	22	1	13	-91
N(3)	2099 (1)	7875 (4)	11277 (1)	28	327	19	9	7	-61
C(4)	1734 (1)	8422 (4)	10497 (1)	25	220	19	0	14	-17
C(5)	940 (1)	7090 (4)	10242 (1)	24	256	23	-13	17	-18
C(6)	1508 (2)	10795 (5)	10362 (1)	33	230	27	-22	22	-5
C(7)	1202 (1)	11392 (4)	9562 (1)	30	223	28	9	17	8
N(8)	1807 (1)	10789 (4)	9277 (1)	32	262	24	20	18	-17
C(9)	1921 (2)	8480 (5)	9315 (1)	35	264	23	10	23	11
C(10)	2268 (1)	7711 (5)	10101 (1)	26	275	22	11	17	14
C(11)	1519 (2)	11535 (6)	8523 (2)	45	417	26	52	15	-26
C(12)	2167 (2)	11202 (6)	8207 (2)	67	429	28	-3	46	-105
C(13)	2900 (2)	12661 (6)	8569 (2)	60	331	31	32	45	-27
C(14)	3544 (2)	12811 (5)	8381 (2)	56	303	31	41	46	32
C(15)	4204 (2)	14338 (5)	8720 (1)	46	346	28	56	35	41
C(16)	5012 (2)	13948 (7)	8759 (2)	56	474	51	89	60	88
C(17)	5627 (2)	15504 (8)	9094 (2)	46	608	62	149	46	30
C(18)	5441 (2)	17349 (8)	9356 (2)	49	571	37	50	21	-84
C(19)	4691 (2)	17712 (7)	9325 (2)	49	558	39	-40	32	-93
C(20)	4069 (2)	16250 (6)	9006 (2)	45	435	35	-36	34	-7
C(21)	2928 (1)	8215 (5)	11759 (1)	26	305	18	-17	12	-19
C(22)	3403 (2)	9896 (5)	11674 (1)	29	371	26	-7	16	-54
C(23)	4210 (2)	10223 (6)	12183 (1)	28	499	27	-33	16	-57
C(24)	4554 (2)	8900 (6)	12783 (1)	29	504	27	-45	15	1
C(25)	4105 (2)	7245 (6)	12869 (1)	34	424	24	-9	9	31
C(26)	3289 (2)	6873 (5)	12363 (1)	36	341	21	-5	12	13
O(27)	392 (1)	7112 (3)	9618 (1)	28	337	24	12	3	-60
C(28)	3597 (3)	11374 (7)	7786 (2)	88	400	46	-19	75	31
F(29)	6055 (1)	18828 (5)	9659 (1)	58	806	54	13	26	-174

the refinement is given by:

$$w = 1/(6.72 + |F_o| + 0.0136|F_o|^2).$$

Table 3. *Intramolecular bond distances and angles (with standard deviations in parentheses)*

N(1)—C(2)	1.443 (4) Å
N(1)—C(5)	1.320 (3)
C(2)—N(3)	1.454 (4)
N(3)—C(4)	1.485 (3)
N(3)—C(21)	1.404 (3)
C(4)—C(5)	1.531 (4)
C(4)—C(6)	1.538 (4)
C(4)—C(10)	1.539 (4)
C(5)—O(27)	1.240 (3)
C(6)—C(7)	1.533 (4)
C(7)—N(8)	1.461 (4)
N(8)—C(9)	1.461 (4)
N(8)—C(11)	1.478 (4)
C(9)—C(10)	1.533 (3)
C(11)—C(12)	1.542 (5)
C(12)—C(13)	1.508 (5)
C(13)—C(14)	1.347 (5)
C(14)—C(15)	1.448 (5)
C(14)—C(28)	1.541 (5)
C(15)—C(16)	1.421 (6)
C(15)—C(20)	1.396 (5)
C(16)—C(17)	1.411 (6)
C(17)—C(18)	1.370 (7)
C(18)—C(19)	1.321 (6)
C(18)—F(29)	1.368 (5)
C(19)—C(20)	1.373 (6)
C(21)—C(22)	1.404 (4)
C(21)—C(26)	1.407 (4)
C(22)—C(23)	1.393 (4)
C(23)—C(24)	1.391 (5)
C(24)—C(25)	1.364 (5)
C(25)—C(26)	1.405 (4)
C(2)—N(1)—C(5)	112.5 (2)°
N(1)—C(2)—N(3)	102.2 (2)
C(2)—N(3)—C(4)	111.7 (2)
C(2)—N(3)—C(21)	116.4 (2)
C(4)—N(3)—C(21)	125.4 (2)
N(3)—C(4)—C(5)	99.8 (2)
N(3)—C(4)—C(6)	112.4 (2)
N(3)—C(4)—C(10)	113.6 (2)
C(5)—C(4)—C(6)	109.5 (2)
C(5)—C(4)—C(10)	109.9 (2)
C(6)—C(4)—C(10)	111.0 (2)
N(1)—C(5)—C(4)	110.2 (2)
N(1)—C(5)—O(27)	125.5 (3)
C(4)—C(5)—O(27)	124.3 (2)
C(4)—C(6)—C(7)	112.2 (2)
C(6)—C(7)—N(8)	111.1 (2)
C(7)—N(8)—C(9)	110.1 (2)
C(7)—N(8)—C(11)	109.5 (2)
C(9)—N(8)—C(11)	110.5 (2)
N(8)—C(9)—C(10)	110.8 (2)
C(4)—C(10)—C(9)	112.6 (2)
N(8)—C(11)—C(12)	113.0 (3)
C(11)—C(12)—C(13)	110.6 (3)
C(12)—C(13)—C(14)	124.2 (3)
C(13)—C(14)—C(15)	121.5 (3)
C(13)—C(14)—C(28)	120.9 (3)
C(15)—C(14)—C(28)	117.6 (3)
C(14)—C(15)—C(16)	120.6 (3)
C(14)—C(15)—C(20)	121.7 (3)
C(16)—C(15)—C(20)	117.7 (3)
C(15)—C(16)—C(17)	117.8 (4)
C(16)—C(17)—C(18)	120.7 (4)
C(17)—C(18)—C(19)	121.6 (4)

Table 3 (cont.)

C(17)—C(18)—F(29)	118.3 (4)
C(19)—C(18)—F(29)	120.1 (4)
C(18)—C(19)—C(20)	120.1 (4)
C(15)—C(20)—C(19)	122.0 (3)
N(3)—C(21)—C(22)	122.3 (2)
N(3)—C(21)—C(26)	119.1 (2)
C(22)—C(21)—C(26)	118.5 (3)
C(21)—C(22)—C(23)	120.2 (3)
C(22)—C(23)—C(24)	120.5 (3)
C(23)—C(24)—C(25)	120.0 (3)
C(24)—C(25)—C(26)	120.7 (3)
C(21)—C(26)—C(25)	120.0 (3)

Table 4. *Torsion angles defining the conformation of the title compound (A) and spiperone (B)*

	A	B
C(7)—N(8)—C(11)—C(12)	173°	168°
N(8)—C(11)—C(12)—C(13)	-69	-64
C(11)—C(12)—C(13)—C(14)	-176	170
C(12)—C(13)—C(14)—C(15)	176	175
C(13)—C(14)—C(15)—C(20)	-28	-13
C(2)—N(3)—C(21)—C(22)	179	-178
C(2)—N(3)—C(21)—C(26)	-3	0

The scattering factors used are those given in *International Tables for X-ray Crystallography* (1962). The final coordinates and their standard deviations are given in Table 2.

Discussion. The conformation of the molecule and the atomic numbering scheme are shown in Fig. 1.

The intramolecular bond distances and angles are given in Table 3. The conformation of the molecule is very similar to that of spiperone (Koch, 1973), as

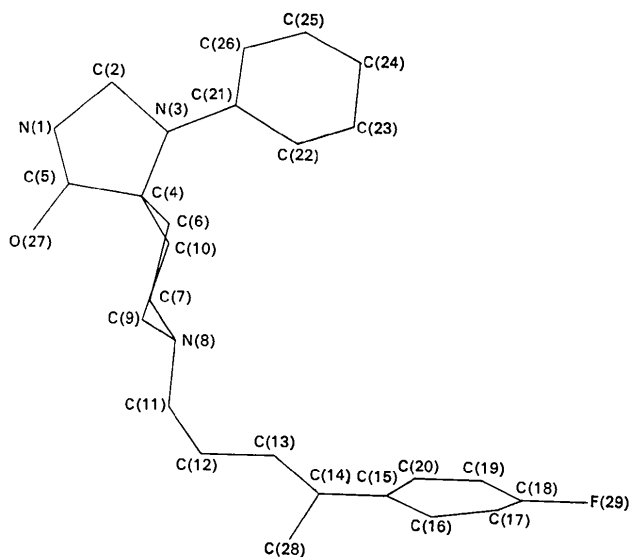


Fig. 1. Conformation and atom numbering scheme of $C_{24}H_{28}FN_3O$.

shown by comparison of the relevant torsion angles given in Table 4.

The molecules form centrosymmetric dimers similar to those found in spiperone: N(1)–H...O(27): 2·865 Å [O(27): $-x, 1-y, 2-z$].

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SHORT COMMUNICATIONS

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Acta Cryst. (1973). **B29**, 2973

Crystallographic and spectroscopic studies on ethylenediaminetetraacetic acid (EDTA). II. Comparison of two structure determinations of β -EDTA. By M. F. C. LADD and D. C. POVEY, *Department of Chemical Physics, University of Surrey, Guildford, Surrey, England*

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Two structure determinations of β -EDTA are compared and the hydrogen atom positions and bonding discussed.

Recently, we published a structure determination of β -ethylenediaminetetraacetic acid (β -EDTA) (Ladd & Povey, 1973). Subsequently, our attention was drawn to another report on the structure of the same compound (Cotrait, 1972) which pre-dates our paper.

It is interesting to compare the two structure determinations. Fig. 1 is a schematic drawing of one half of the EDTA molecule, to relate the two atom-numbering schemes. In each investigation similar amounts of reflexion data were collected from small crystals on a Siemens four-circle diffractometer, using Cu $K\alpha$ radiation, and corrected for Lorentz and polarization effects but not for absorption. The structures were solved by direct methods and refined by least-squares calculations with the programs of Ahmed (Cotrait) and the X-RAY 70 system (Ladd & Povey). A selection of general structural data is listed in Table 1. Three of the reflexions, $\bar{1}52$, 517 and $\bar{1}4,2,6$, used in each starting set were the same, although their magnitudes differed by about 15%.

In each structure, the hydrogen atoms were located on a difference map. Cotrait refined the coordinates of the hydrogen atoms with a fixed isotropic temperature factor of 2 \AA^2 for each. Ladd & Povey did not refine these coordinates and allocated isotropic temperature factors 0.5 \AA^2 higher ($1.8\text{--}3.0 \text{ \AA}^2$) than those of the carbon atoms to which they are bonded.

Because of certain differences in the two sets of bond lengths we re-calculated Cotrait's values using his published data and the *BONDLA* program of the X-RAY 70 system.

Table 1. *Structural data for β -EDTA*

	Cotrait (1972)	Ladd & Povey (1973)
a (Å)	13.286 (2)	13.273 (4)
b	5.578 (5)	5.575 (7)
c	16.120 (3)	16.110 (6)
β (°)	96.30 (5)	96.26 (3)
V_c (Å ³)	1188	1185 (2)
D_m (g cm ⁻³)	1.65 (2)	1.65 (1)
D_c	1.635	1.638 (3)
Z	4	4
Space group	$C2/c$	$C2/c$
Crystal dimensions (mm)	0.2, 0.3, 0.3	0.1, 0.4, 0.1
Number of reflexions	1203	1020
Final R	5.3%	5.6%

The results are presented in Table 2,* with his original values and our own with estimated standard deviations corrected from those reported in our 1973 paper.

The differences in the bond lengths among the heavier atoms, columns (b) and (c) in Table 2, are not significant ($< 3\sigma$). Bonds involving hydrogen show greater discrepancies. The two Cotrait sets are slightly different, especially where hydrogen atoms are involved. Our C–H and N–H bond lengths are all appreciably longer. We agree with Cotrait, and contrary to our previous paper, that there are three hydrogen bonds of similar importance involving the nitrogen atom.

* Cotrait's atom numbering has been used throughout.