Acta Cryst. (1973). B**29**, 2969

8-(2-Phenoxyethyl)-1-phenyl-1,3,8-triazaspiro[4,5]decan-4-one

BY M.H.J. KOCH

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

AND O. DIDEBERG

Laboratoire de Cristallographie, Institut de Physique, Université de Liège, 4000 *Liège, Belgium*

(*Received* 27 *August* 1973; *accepted* 10 *September* 1973)

Abstract. Monoclinic; $P2_1/c$; $a=15{\cdot}053$ (3), $b=6{\cdot}403$ (2), $c=20{\cdot}909$ (6) Å; $\beta=113{\cdot}76$ (6)°; 25°C; $C_{21}H_{25}N_3O_2$; $M=351{\cdot}43$; $Z=4$; $F(000)=752$.

Introduction. This compound is related to spiperone, a very potent neuroleptic. Transparent needles were obtained by slow evaporation from a mixture of ethanol and dichloromethane.

The lattice parameters were obtained by least-squares refinement of the setting angles of twelve reflexions using Cu $K\bar{\alpha}$ radiation. The space group was determined from Weissenberg photographs.

Intensities were collected on a Hilger & Watts computer-controlled diffractometer. The experimental conditions are given in Table 1. The structure was solved by direct methods with *MULTAN* (Germain, Main & Woolfson, 1971).

Table 1. *Experimental data*

Crystal dimensions 0·25 × 0·25 × 0·15 mm
Source Cu $K\bar{\alpha}$; $\lambda=1{\cdot}5418$ Å; ω–2θ step scan
$\theta_{min}=2°$; $\theta_{max}=70°$
Confidence level: 2·5
Total number of independent reflexions: 2839
Total observed: 2074

Block-diagonal least-squares refinement with the program written by Ahmed, Hall, Pippy & Huber (1966) resulted in $R=\sum||F_o|-|F_c||/\sum|F_o|=0{\cdot}13$ for all observed reflexions.* The weight assigned to each re-

* A list of structure factors has been deposited with the National Lending Library, England, as Supplementary Publication No. SUP 30210 (17 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)	4878 (2)	4186 (4)	9112 (1)	87	411	32	−42	45	−99
C(2)	4788 (2)	4134 (5)	8405 (1)	99	354	33	−45	50	−116
N(3)	4081 (2)	2456 (4)	8105 (1)	76	411	28	−12	40	−75
C(4)	3658 (2)	1729 (4)	8588 (1)	70	300	28	0	36	−14
C(5)	4248 (2)	2969 (4)	9246 (1)	72	311	32	−11	38	−5
C(6)	2559 (2)	2222 (5)	8340 (1)	56	415	33	8	29	24
C(7)	2111 (2)	1168 (6)	8809 (1)	79	605	35	−25	57	−31
N(8)	2326 (2)	−1042 (5)	8880 (1)	93	501	36	−36	60	−189
C(9)	3371 (2)	−1457 (5)	9217 (1)	79	340	43	15	58	−1
C(10)	3854 (2)	−631 (4)	8761 (1)	83	288	40	−1	49	28
C(11)	1846 (3)	−2090 (8)	9294 (2)	124	942	42	−40	65	−412
C(12)	948 (3)	−3316 (7)	8786 (2)	107	655	51	54	97	−37
O(13)	630 (2)	−4462 (5)	9220 (1)	131	642	47	−37	79	−198
C(14)	−13 (2)	−6039 (6)	8905 (2)	69	462	59	−81	50	−86
C(15)	−523 (3)	−6258 (6)	8168 (2)	77	512	52	10	56	−18
C(16)	−1172 (3)	−7944 (6)	7938 (2)	86	491	45	−47	40	−30
C(17)	−1307 (3)	−9274 (6)	8390 (2)	117	478	52	−16	60	−47
C(18)	−829 (3)	−8986 (6)	9101 (2)	122	445	59	33	76	33
C(19)	−184 (3)	−7348 (6)	9363 (2)	109	485	41	27	54	−1
C(20)	3679 (2)	2250 (4)	7378 (1)	73	373	28	−11	38	25
C(21)	3962 (2)	3611 (5)	6970 (1)	74	399	37	18	55	26
C(22)	3561 (2)	3434 (6)	6238 (1)	81	545	38	32	56	18
C(23)	2901 (2)	1874 (6)	5905 (1)	83	681	31	8	47	8
C(24)	2641 (2)	479 (6)	6313 (1)	82	577	35	−82	42	−74
C(25)	3018 (2)	619 (5)	7038 (1)	79	456	33	−41	44	−43
O(26)	4138 (2)	2916 (3)	9794 (1)	104	413	28	−39	49	−134

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

N(1)—C(2)	1·429 (4) Å
N(1)—C(5)	1·340 (4)
C(2)—N(3)	1·464 (4)
N(3)—C(4)	1·470 (4)
N(3)—C(20)	1·396 (3)
C(4)—C(5)	1·526 (4)
C(4)—C(6)	1·554 (4)
C(4)—C(10)	1·554 (4)
C(5)—O(26)	1·225 (3)
C(6)—C(7)	1·551 (5)
C(7)—N(8)	1·446 (5)
N(8)—C(9)	1·467 (5)
N(8)—C(11)	1·492 (6)
C(9)—C(10)	1·507 (5)
C(11)–C(12)	1·554 (6)
C(12)–O(13)	1·392 (5)
O(13)–C(14)	1·370 (5)
C(14)–C(15)	1·424 (5)
C(14)–C(19)	1·372 (5)
C(15)–C(16)	1·404 (6)
C(16)–C(17)	1·346 (5)
C(17)–C(18)	1·378 (5)
C(18)–C(19)	1·383 (6)
C(20)–C(21)	1·400 (4)
C(20)–C(25)	1·420 (4)
C(21)–C(22)	1·407 (4)
C(22)–C(23)	1·382 (5)
C(23)–C(24)	1·396 (5)
C(24)–C(25)	1·390 (4)
C(2)—N(1)—C(5)	115·0 (3)°
N(1)—C(2)—N(3)	102·1 (2)
C(2)—N(3)—C(4)	112·2 (2)
C(2)—N(3)—C(20)	117·2 (2)
C(4)—N(3)—C(20)	126·9 (2)
N(3)—C(4)—C(5)	101·4 (2)
N(3)—C(4)—C(6)	113·8 (2)
N(3)—C(4)—C(10)	112·0 (2)
C(5)—C(4)—C(6)	110·5 (2)
C(5)—C(4)—C(10)	108·0 (2)
C(6)—C(4)—C(10)	110·6 (2)
N(1)—C(5)—C(4)	108·6 (2)
N(1)—C(5)—O(26)	125·9 (3)
C(4)—C(5)—O(26)	125·5 (3)
C(4)—C(6)—C(7)	112·1 (2)
C(6)—C(7)—N(8)	110·8 (3)
C(7)—N(8)—C(9)	112·2 (3)
C(7)—N(8)—C(11)	111·1 (3)
C(9)—N(8)—C(11)	108·5 (3)
N(8)—C(9)—C(10)	109·3 (3)
C(4)—C(10)–C(9)	113·2 (2)
N(8)—C(11)–C(12)	108·7 (3)
C(11)–C(12)–O(13)	104·7 (3)
C(12)–O(13)–C(14)	116·0 (3)
O(13)–C(14)–C(15)	124·1 (3)
O(13)–C(14)–C(19)	114·2 (3)
C(15)–C(14)–C(19)	121·5 (4)
C(14)–C(15)–C(16)	116·4 (3)
C(15)–C(16)–C(17)	121·7 (4)
C(16)–C(17)–C(18)	120·8 (4)
C(17)–C(18)–C(19)	120·4 (4)
C(14)–C(19)–C(18)	119·1 (4)
N(3)—C(20)–C(21)	120·0 (3)
N(3)—C(20)–C(25)	121·6 (3)
C(21)–C(20)–C(25)	118·4 (3)
C(20)–C(21)–C(22)	120·7 (3)
C(21)–C(22)–C(23)	120·8 (3)
C(22)–C(23)–C(24)	118·5 (3)
C(23)–C(24)–C(25)	122·2 (3)
C(20)–C(25)–C(24)	119·3 (3)

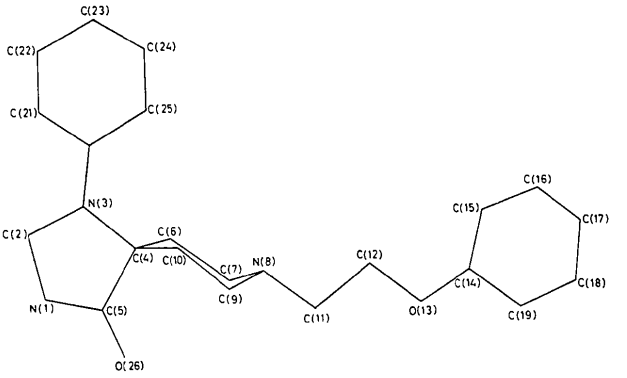

Fig. 1. Conformation and atomic numbering scheme of $C_{21}H_{25}N_3O_2$.

flexion during the refinement is given by:

$$w = 1/(20 + |F_o| + 0{\cdot}01|F_o|^2) .$$

The atomic 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 atomic numbering scheme and the conformation of the molecule are shown in Fig. 1. The intramolecular bond distances and angles are given in Table 3.

The conformation of the side chain is defined by the torsion angles given in Table 4. The molecules form centrosymmetric dimers similar to those found in spiperone (Koch, 1973). The dimerization is due to the amide groups which are hydrogen bonded: O(26)–N(1), 2·852 Å [N(1): $1-x$, $1-y$, $2-z$].

Table 4. *Torsion angles defining the conformation of the side chain*

C(7)—N(8)—C(11)–C(12)	104°
C(9)—N(8)—C(11)–C(12)	−132
N(8)—C(11)–C(12)–O(13)	172
C(11)–C(12)–O(13)–C(14)	−165
C(12)–O(13)–C(14)–C(15)	−15
C(12)–O(13)–C(14)–C(19)	170

We thank Dr P. A. Janssen (Janssen Pharmaceutica–Beerse–Belgium) for providing the sample. M. K. thanks the Fonds National de la Recherche Scientifique for a fellowship.

References

AHMED, F. R., HALL, S. R., PIPPY, M. E. & HUBER, C. P. (1966). *World list of Crystallographic Computer Programs*, 2nd ed. Appendix, p. 52. Utrecht: Oosthoek.

GERMAIN, G., MAIN, P. & WOOLFSON, M. M. (1971). *Acta Cryst.* A**27**, 368–376.

International Tables for X-ray Crystallography (1962). Vol. III. Birmingham: Kynoch Press.

KOCH, M. H. J. (1973). *Acta Cryst.* B**29**, 379–382.