and 1·5° respectively. The four H atoms of the methano groups and the two C atoms to which they are bonded are coplanar within experimental uncertainty [maximum deviation 0·03 (2) Å]. The contact between the two inner bridge H atoms [H(15′)···H(16″) distance = 1·78 Å] is partly relieved by slight rotation and deformation of the $-CH_2-$ groups: the bisectors of the $H\hat{C}H$ angles deviate by about 5° from the bisectors of the related bridgehead $C\hat{C}C$ angles, and the $H\hat{C}H$ angles are both less than the tetrahedral angle, notwithstanding the small values of the $C\hat{C}C$ bridgehead angles (104°). The dihedral angle between the two bridges is increased from the 0·6° value found in 1,6:8,13-propane-1,3-diylidene[14]annulene (Gavezzotti *et al.*, 1972) to 26·6° (to be compared with 26·0° in the butane analogue BUT).

The *mm*2-averaged C—C bond distance involving the bridge C atoms is 1·498 Å in SBM, only slightly less than the corresponding length in BUT (1·505 Å); the $C\hat{C}C$ angles at the bridge C atoms are 103·9° (average value) in SBM and 102·9° in BUT; and the distances between these two C atoms are 2·921 and 2·914 Å respectively. It can therefore be stated that the two internal H atoms affect the position of the bridge C atoms, and hence the conformation of the annulene perimeter, in much the same way as the $-CH_2-CH_2-$ group does in BUT.

We wish to thank Professor E. Vogel for the sample of the crystals.

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4-(*p*-Chlorophenyl)-4-hydroxy-*N*,*N*-dimethyl-α,α-diphenylpiperidine-1-butyramide (Loperamide) Hydrate

BY G. GERMAIN, J. P. DECLERCQ* AND M. VAN MEERSSCHE

Laboratoire de Chimie Physique et de Cristallographie, Université de Louvain, 1 place Louis Pasteur, B-1348 Louvain-la-Neuve, Belgium

AND M. H. J. KOCH

Research Laboratories, Janssen Pharmaceutica, B-2340 Beerse, Belgium

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Abstract. $C_{29}H_{33}N_2O_2Cl.H_2O$, F.W. 495·0; orthorhombic, *Pbca*; a = 15·160 (3), b = 20·715 (5), c = 16·803 (3) Å; t = 25°C; Z = 8. The molecules are connected by direct hydrogen bonds and through the water molecules.

Introduction. Loperamide is a specific, long-acting antidiarrhoeal drug. Slow evaporation of a solution in ethanol yielded transparent colourless crystals. The space group was determined from Weissenberg photographs; final cell dimensions and intensities were measured on a Picker four-circle diffractometer. The experimental conditions are given in Table 1. The structure was solved with *MULTAN* (Germain, Main & Woolfson, 1971) and refined by block-diagonal least squares (Ahmed, Hall, Pippy & Huber, 1966). The final $R = \Sigma||F_o| - |F_c||/\Sigma|F_o|$ is 0·09 for all observed reflexions. The scattering factors used are those given in

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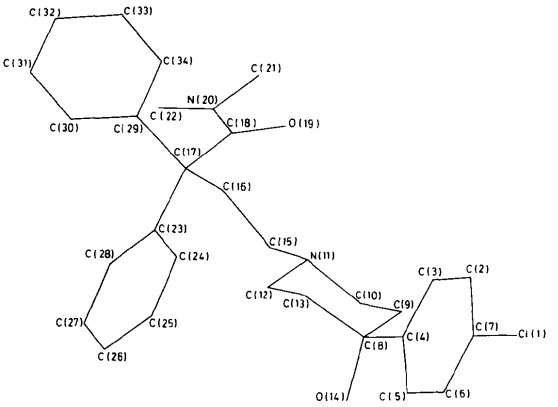

Fig. 1. Conformation and numbering scheme of loperamide.

Table 1. *Experimental conditions*

Source Cu $K\bar{\alpha}$: $\lambda = 1{\cdot}5418$ Å
ω–2θ scan: $\theta_{max} = 50°$
Confidence level: 2·5
Total number of independent reflexions: 2692
Total observed: 2231

Table 2. *Final positional parameters* ($\times 10^4$) *with standard deviations in parentheses*

	x	y	z
Cl(1)	4198 (1)	7693 (1)	1764 (1)
C(2)	4949 (3)	8214 (2)	461 (2)
C(3)	4891 (2)	8583 (2)	−254 (2)
C(4)	4118 (2)	8869 (1)	−506 (2)
C(5)	3362 (2)	8807 (2)	−33 (2)
C(6)	3388 (3)	8439 (2)	674 (2)
C(7)	4164 (2)	8160 (2)	911 (2)
C(8)	4095 (2)	9264 (2)	−1268 (2)
C(9)	4454 (2)	8883 (2)	−1975 (2)
C(10)	4538 (2)	9311 (2)	−2717 (2)
N(11)	5119 (2)	9859 (1)	−2560 (1)
C(12)	4719 (2)	10259 (2)	−1932 (2)
C(13)	4629 (2)	9879 (2)	−1152 (2)
O(14)	3207 (2)	9412 (1)	−1496 (1)
C(15)	5254 (2)	10238 (2)	−3291 (2)
C(16)	6010 (2)	10733 (2)	−3171 (2)
C(17)	6283 (2)	11119 (1)	−3927 (2)
C(18)	6791 (2)	10667 (1)	−4492 (2)
O(19)	6933 (2)	10094 (1)	−4288 (1)
N(20)	7098 (2)	10866 (1)	−5206 (2)
C(21)	7600 (3)	10414 (2)	−5698 (2)
C(22)	7087 (3)	11541 (2)	−5502 (2)
C(23)	5438 (2)	11394 (1)	−4291 (2)
C(24)	5083 (2)	11168 (2)	−5002 (2)
C(25)	4280 (2)	11399 (2)	−5284 (2)
C(26)	3810 (2)	11855 (2)	−4858 (2)
C(27)	4138 (2)	12070 (2)	−4130 (2)
C(28)	4954 (2)	11839 (2)	−3855 (2)
C(29)	6955 (2)	11619 (1)	−3643 (2)
C(30)	6875 (2)	12286 (2)	−3745 (2)
C(31)	7494 (2)	12703 (2)	−3419 (2)
C(32)	8239 (2)	12478 (2)	−3018 (2)
C(33)	8334 (2)	11813 (2)	−2938 (2)
C(34)	7720 (2)	11394 (2)	−3250 (2)
O(35)	6688 (2)	9058 (1)	7518 (2)

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

Cl(1)–C(7)	1·731 (4)	C(3)–C(2)–C(7)	116·1 (3)
C(2)–C(3)	1·428 (5)	C(2)–C(3)–C(4)	122·8 (3)
C(2)–C(7)	1·414 (6)	C(3)–C(4)–C(5)	118·8 (3)
C(3)–C(4)	1·379 (5)	C(3)–C(4)–C(8)	120·6 (3)
C(4)–C(5)	1·400 (5)	C(5)–C(4)–C(8)	120·6 (3)
C(4)–C(8)	1·520 (4)	C(4)–C(5)–C(6)	120·3 (3)
C(5)–C(6)	1·412 (5)	C(5)–C(6)–C(7)	119·7 (3)
C(6)–C(7)	1·370 (5)	Cl(1)–C(7)–C(2)	117·4 (3)
C(8)–C(9)	1·526 (4)	Cl(1)–C(7)–C(6)	120·1 (3)
C(8)–C(13)	1·522 (4)	C(2)–C(7)–C(6)	122·4 (3)
C(8)–O(14)	1·433 (4)	C(4)–C(8)–C(9)	111·7 (3)
C(9)–C(10)	1·534 (4)	C(4)–C(8)–C(13)	109·3 (3)
C(10)–N(11)	1·460 (4)	C(4)–C(8)–O(14)	111·2 (2)
N(11)–C(12)	1·472 (4)	C(9)–C(8)–C(13)	110·1 (3)
N(11)–C(15)	1·473 (4)	C(9)–C(8)–O(14)	103·8 (2)
C(12)–C(13)	1·535 (5)	C(13)–C(8)–O(14)	110·7 (3)
C(15)–C(16)	1·551 (5)	C(8)–C(9)–C(10)	111·3 (3)
C(16)–C(17)	1·558 (4)	C(9)–C(10)–N(11)	110·6 (3)
C(17)–C(18)	1·540 (4)	C(10)–N(11)–C(12)	108·6 (2)
C(17)–C(23)	1·528 (4)	C(10)–N(11)–C(15)	110·4 (2)
C(17)–C(29)	1·530 (4)	C(12)–N(11)–C(15)	110·8 (2)
C(18)–O(19)	1·255 (4)	N(11)–C(12)–C(13)	111·1 (3)
C(18)–N(20)	1·351 (4)	C(8)–C(13)–C(12)	111·5 (3)
N(20)–C(21)	1·463 (5)	N(11)–C(15)–C(16)	110·3 (2)
N(20)–C(22)	1·483 (5)	C(15)–C(16)–C(17)	115·4 (3)
C(23)–C(24)	1·391 (5)	C(16)–C(17)–C(18)	108·9 (2)
C(23)–C(28)	1·388 (4)	C(16)–C(17)–C(23)	107·2 (2)
C(24)–C(25)	1·391 (5)	C(16)–C(17)–C(29)	105·6 (2)
C(25)–C(26)	1·382 (5)	C(18)–C(17)–C(23)	113·5 (2)
C(26)–C(27)	1·393 (5)	C(18)–C(17)–C(29)	105·7 (2)
C(27)–C(28)	1·404 (5)	C(23)–C(17)–C(29)	115·5 (2)
C(29)–C(30)	1·398 (4)	C(17)–C(18)–O(19)	119·5 (3)
C(29)–C(34)	1·413 (5)	C(17)–C(18)–N(20)	122·3 (3)
C(30)–C(31)	1·388 (5)	O(19)–C(18)–N(20)	118·2 (3)
C(31)–C(32)	1·396 (5)	C(18)–N(20)–C(21)	119·0 (3)
C(32)–C(33)	1·392 (5)	C(18)–N(20)–C(22)	125·6 (3)
C(33)–C(34)	1·375 (5)	C(21)–N(20)–C(22)	114·8 (3)
		C(17)–C(23)–C(24)	122·9 (3)
		C(17)–C(23)–C(28)	118·7 (3)
		C(24)–C(23)–C(28)	118·1 (3)
		C(23)–C(24)–C(25)	121·1 (3)
		C(24)–C(25)–C(26)	120·6 (3)
		C(25)–C(26)–C(27)	119·2 (3)
		C(26)–C(27)–C(28)	119·6 (3)
		C(23)–C(28)–C(27)	121·2 (3)
		C(17)–C(29)–C(30)	124·9 (3)
		C(17)–C(29)–C(34)	118·0 (3)
		C(30)–C(29)–C(34)	117·1 (3)
		C(29)–C(30)–C(31)	120·5 (3)
		C(30)–C(31)–C(32)	122·0 (3)
		C(31)–C(32)–C(33)	117·4 (3)
		C(32)–C(33)–C(34)	121·2 (3)
		C(29)–C(34)–C(33)	121·7 (3)

Table 4. *Torsion angles defining the conformation of loperamide*

C(8)–C(13)–C(12)–N(11)	−57°
C(12)–N(11)–C(15)–C(16)	71
N(11)–C(15)–C(16)–C(17)	174
C(15)–C(16)–C(17)–C(18)	−73
C(15)–C(16)–C(17)–C(23)	50
C(15)–C(16)–C(17)–C(29)	174
C(16)–C(17)–C(18)–N(20)	179
C(16)–C(17)–C(23)–C(24)	−109
C(16)–C(17)–C(29)–C(30)	−123

Table 5. *Hydrogen bonds* (Å) *in crystals of loperamide*

N(11)–O(35)	2·903	O(35)	$x, y, z-1$
O(14)–O(19)	2·732	O(19)	$-0{\cdot}5+x, y, -0{\cdot}5-z$
O(14)–O(35)	2·965	O(35)	$-0{\cdot}5+x, y, 0{\cdot}5-z$
O(19)–O(14)	2·732	O(14)	$0{\cdot}5+x, y, -0{\cdot}5-z$

International Tables for X-ray Crystallography (1962). The final coordinates are given in Table 2.*

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 32326 (18 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Discussion. The conformation of the molecule and the numbering scheme are shown in Fig. 1 and the bond distances and angles in Table 3. The torsion angles defining the conformation are given in Table 4.

The molecules are connected as indicated in the *Abstract* and in Table 5.

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trans-(Oxoaqua)trichlorothiourearhenium(V), $ReO(H_2O)Cl_3tu$

BY T. LIS

Institute of Chemistry, University of Wrocław, 50-383 Wrocław, ul. Joliot-Curie 14, Poland

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Abstract. $CH_6N_2O_2SCl_3Re$, orthorhombic, $Pca2_1$ (C_{2v}^5), $a = 11{\cdot}032$ (8), $b = 7{\cdot}083$ (5), $c = 11{\cdot}166$ (8) Å, $M_r = 402{\cdot}7$, $V = 872{\cdot}5$ Å^3, $Z = 4$, $D_m = 3{\cdot}04$, $D_x = 3{\cdot}06$ g cm^{-3}, μ(Cu $K\alpha$, $\lambda = 1{\cdot}5418$ Å) $= 366{\cdot}0$ cm^{-1}. The molecule is somewhat distorted from true octahedral symmetry and the oxo O atom and water molecule are *trans*. The bond lengths are Re–O 1·71 (2), Re–H_2O 2·29 (2), Re–S 2·311 (7) and Re–Cl 2·347–2·425 Å. The structure was refined to an R of 0·051 for 901 diffractometer data.

Introduction. This investigation is a part of the study of coloured oxo-complexes of Re^{V} which can be obtained in the system Re^{VII}–HCl–thiourea. In the previous paper (Lis, 1976) the crystal structure of blue $[ReO(H_2O)Cl_2tu_2]Cl$ was reported. The X-ray analysis presented here has shown that the green compound has the formula $ReO(H_2O)Cl_3tu$. The crystal structure of the orange compound of the formula $ReOCl_3tu_2$ will be reported later.

The title compound was obtained by dissolving 0·1 g NH_4ReO_4 (or $KReO_4$) and 0·1 g $CS(NH_2)_2$ in 10–20 ml of concentrated HCl. On standing, well formed green crystals were obtained. Weissenberg photographs showed that the crystals are orthorhombic; the systematic absences were: $h0l$ for $h = 2n+1$, $0kl$ for $l = 2n+1$; space group $Pca2_1$ (C_{2v}^5) or $Pcam$ (D_{2h}^{11}). An almost spherical crystal, of approximate radius 0·05 mm, was used for the data collection. A Syntex $P2_1$ diffractometer and Cu $K\alpha$ radiation with a graphite monochromator were used for lattice-parameter and intensity measurements. The intensities of reflexions were measured by the 2θ–ω scan technique. The data were corrected for Lorentz and polarization effects and for absorption as for spherical crystals ($\mu r = 1{\cdot}8$). Of 941 accessible reflexions below $2\theta = 146°$, 901 with $I > 1{\cdot}96\sigma(I)$ were used for the structure determination. All calculations were performed on the NOVA 1200 computer with programs supplied by Syntex (Syntex XTL structure determination system). Neutral-atom scattering factors used were those tabulated by Cromer & Waber as given in *International Tables for X-ray Crystallography* (1974).

A three-dimensional Patterson map was computed, from which no clear basis for choice between the two possible space groups was available. The non-centrosymmetric one, $Pca2_1$, was tentatively assumed, in which all atoms occupy the fourfold general set of positions. The approximate coordinates of the Re atoms were easily determined from the Patterson map. With phases