Polyèdres de coordination

Les atomes de manganèse sont aux centres d'octaèdres légèrement aplatis; l'un d'eux est mis en évidence sur la Fig. 7; on en retrouve la projection sur la Fig. 9 puisque le manganèse est situé sur l'axe 3.

Les atomes de plomb que nous avons appelés $\mathrm{Pb_{II}}$, au nombre de 12, ont trois voisins d'oxygène formant un triangle équilatéral. Les autres atomes d'oxygène les plus proches sont à une distance PbO supérieure à 2,8 Å.

Les atomes de plomb en position générale $\mathrm{Pb_{I}}$ ont des polyèdres de coordination beaucoup plus irréguliers; leurs proches voisins oxygène sont aussi au nombre de trois; les trois liaisons $\mathrm{Pb_{I}}$–O ont même longueur mais le plomb se projette assez loin du centre de gravité du triangle.

Les distances Pb–O étant pour $\mathrm{Pb_{I}}$ et $\mathrm{Pb_{II}}$ de l'ordre de 2,2–2,3 Å, il est certain que les liaisons Pb–O sont des liaisons de covalence.

Enchaînement de la structure

Les groupements $\mathrm{Si_2O_7}$ qui forment la charpente du cristal sont règulièrement disposés autour des axes ternaires parallèles à **c** passant par les points 000, $\frac{1}{3}\frac{2}{3}0$, $\frac{2}{3}\frac{1}{3}0$; ils forment, en quelque sorte, des 'cheminées cylindriques' parallèles à **c**. Nous avons représenté sur la Fig. 9 la projection sur le plan 001 de ces groupements échelonnés sur une hauteur $c/3$. Dans les canaux axés sur les axes ternaires, on trouve les atomes de manganèse et de $\mathrm{plomb_{II}}$. Ces deux cations ne forment que des liaisons 'horizontales', c'est-à-dire entre groupements situés à la même cote. La cohésion 'verticale' ainsi que la cohésion entre les différentes 'cheminées' est réalisée par les atomes de plomb en position générale.

Les variances sur les paramètres d'oxygène sont trop grandes pour que nous puissions comparer valablement les différentes longueurs de liaison Si–O.

Je remercie Monsieur le Professeur Guillemin du Bureau de Recherches Géologiques et Minières qui m'a aimablement fourni les cristaux naturels de barysilite, Monsieur Bassi du Centre d'Études Nucléaires de Grenoble qui a effectué les calculs à la machine IBM 7090 et Monsieur Bertaut, Directeur Scientifique au C.N.R.S. pour l'intérêt qu'il a porté à ce travail.

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The Conformation of Non-Aromatic Ring Compounds. XVIII*. The Crystal Structure of 4-Bromo-9β,10α-pregna-4,6-diene-3,20-dione

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Crystals of 4-bromo-9β,10α-pregna-4,6-diene-3,20-dione are orthorhombic with space group $P2_12_12_1$ and $Z=4$. The unit-cell dimensions at room temperature are $a=11{\cdot}66$, $b=7{\cdot}90$, $c=20{\cdot}18$ Å. The structure was solved by construction of a minimum function M_4 and was refined by the least-squares method.

The atom C(19) is below and C(18) above the ring system of the molecule, which itself has a bent shape. Ring *C* has the conformation of a distorted chair form. The atoms C(2), C(3), C(4), C(5), C(6), C(10), O(1) and Br are approximately coplanar. The molecule is considerably flatter than inspection of Dreiding models might suggest.

Introduction

This paper records the third of a series of investigations (Geise, Romers & Rutten, 1966; Geise, Romers, 1966) of the crystal and molecular structures of steroids. The steroid under investigation, 4-bromo-9β,10α-pregna-4,6-diene-3,20-dione (also named 4-bromo-duphaston), is obtained by bromination of 9β,10α-pregna-4,6-diene-3,20-dione (Westerhof & Hartog, 1965). The latter compound has been prepared from lumisterol as the starting material (Westerhof & Reerink, 1960).

For chemists not familiar with steroid chemistry it is, perhaps, necessary to make a few explanatory remarks upon the nomenclature of the steroid in question

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* Part XVII. Geise & Romers (1966).

(IUPAC 1957 rules). Inspection of Fig. 1 shows that the molecule contains six asymmetric carbon atoms: C(8), C(9), C(10), C(13), C(14) and C(17) whose substituents can have configurations above (β) or below (α) the general plane of the molecule. In pregna-4,6-diene-3,20-dione the configuration of the substituents is 8β, 9α, 10β, 13β, 14α, 17β, implying that rings *B* and *C* as well as the rings *C* and *D* are *trans* connected. Inversion of the configuration at the carbon atoms C(9) and C(10) gives the molecule duphaston of the 9β, 10α series, in which now rings *B* and *C* are *cis* connected. The generic name pregna is derived from the molecule pregnane, which contains an ethyl group on C(17) instead of the acetyl group.

The steroids of the $9\beta,10\alpha$ series exhibit interesting physiological and biological properties (Schöler & de Wachter, 1960). Halkes & Havinga (1964, 1965) carried out a study of structural and conformational properties of 9,10-isomeric steroids, particularly by means of infrared and nuclear magnetic resonance spectra. The wish to obtain as much detailed information as possible of the geometry of this type of steroid molecule was the immediate stimulus to investigate the crystal structure of 4-bromo-duphaston. A preliminary paper has appeared elsewhere (Romers, van Heijkoop, Hesper & Geise, 1965). The numbering of the atoms given in Fig. 1 is in agreement with the usual numbering of atoms in steroids.

Experimental

Samples of 4-bromo-duphaston (m.p. 121°–122 °C) were crystallized from an acetone–methanol mixture and samples of 4-chloro-duphaston (m.p. 185°–188 °C) from methanol. The crystals are small, irregular blocks elongated along [010]. Zero layer Weissenberg photographs taken for the compounds about the *a*, *b* and *c* axes showed that the reflexions $h00$, $0k0$ and $00l$ are absent for $h=2n+1$, $k=2n+1$ and $l=2n+1$, respectively. From the observed densities (flotation method) and the measurement of the unit-cell dimensions (Table 1) it is concluded that four molecules of 4-chloro- and 4-bromo-duphaston have to be located in their isomorphous unit cells with space group $P2_12_12_1$.

A series of equi-inclination Weissenberg photographs of the 4-bromo derivative (filtered copper radiation, multiple film technique) were taken at room temperature about [100] with levels $h=0, 1, \ldots, 10$, about [010] with levels $k=0, 1, \ldots, 7$ and about [001] with zero level only. The intensities were estimated visually and corrected for Lorentz-, polarization, spot shape and absorption (cylindrical approximation) effects. The data of both zones were correlated and put on a common scale according to a least-squares procedure of Rollett & Sparks (1960).

Structure determination and refinement

Nearly equal positions were derived for the chloro and bromo atom from the respective Patterson projections $P(u, w)$ and $P(v, w)$. It was hoped to solve the structure by means of the isomorphous replacement technique in the centrosymmetric projections along [010] and [100]. Since the molecules are oriented roughly perpendicular to the long *c* axis both these projections suffer severely from overlap. Although in the projection along the *b* axis the molecule is free from overlap by neighbouring molecules (see also Fig. 4), only the atoms C(19) and O(2) could be distinguished clearly. It was not possible to obtain a better agreement than an $R(h0l)$ of 37%. Evidently, a too heavy degree of overlap brought about the failure of this otherwise powerful method.

Finally, the structure was solved by the application of a minimum function M_4 (Buerger, 1959), which was constructed from the three-dimensional Patterson function by placing the origin successively on the three Harker peaks of the bromine atom. All atoms except C(12) and C(21) were traced in the resulting map. Structure factor calculations based upon the located atoms gave an $R(hkl)$ of 46%. The two missing atoms were found in a subsequent electron density function $\varrho(x, y, z)$, from which improved parameters were obtained for the remaining atoms. This set of positional coordinates together with one overall temperature parameter ($B=4{\cdot}5$ Å^2) for all atoms was used as a starting point for the following refinement, which was performed by the method of least squares. Since the refinement (see Table 2) proceeded in the same way as described in the two preceding papers (Geise, Romers & Rutten, 1966; Geise & Romers, 1966) we will confine ourselves

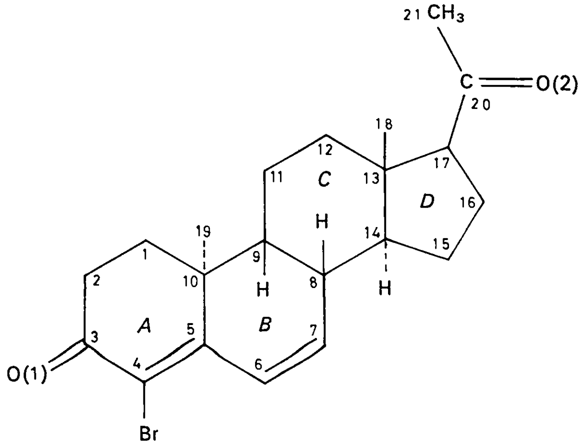

Fig. 1. The numbering of the atoms of 4-bromo-$9\beta,10\alpha$-pregna-4,6-diene-3,20-dione (4-bromo-duphaston). The rings *A*, *B*, *C* and *D* are indicated.

Table 1. *Cell dimensions* (Å), *densities* (g.cm^{-3}) *and space groups of* 4-*chloro- and* 4-*bromo-duphaston*

Compound	*a*	*b*	*c*	*d*(exp)	*d*(calc)	*Z*	Space group
4-chloro-	11·56	7·85	20·17	1·18	1·26	4	$P2_12_12_1$
4-bromo-	11·66	7·90	20·18	1·41	1·40	4	$P2_12_12_1$

to the ultimate results. The refinement stopped at an *R* value of 10·2% for observed reflexions only. Photographic copies of the observed and calculated structure factors for the 1881 reflexions are available from the authors on request.

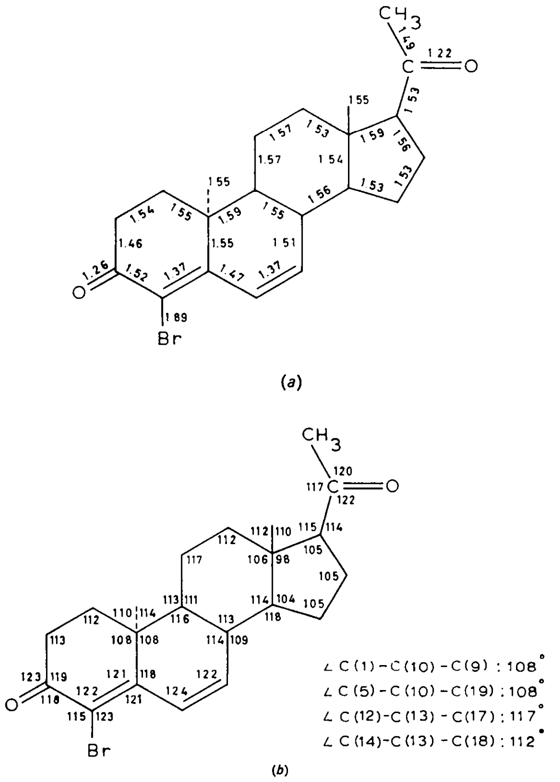

Fig. 2. (*a*) Bond lengths and (*b*) valency angles of 4-bromo-duphaston.

Results and accuracy

The final positional coordinates of 4-bromo-duphaston in fractions of the cell edges together with the standard deviations in Å are listed in Table 3. The

Table 2. *Outline of the refinement of* 4-*bromo-duphaston*

Cycle	Procedure	*R* (%)
1	Structure factors without C(12) and C(21)	46
1a	*ϱ*(*xyz*); C(12) and C(21) located	
2	Structure factors least squares*; one overall *B*=4·5 Å^2	34
3	SFLS; one overall *B*=4·5 Å^2	28·8
4	SFLS, isotropic individual *B*'s	24·2
5	SF-output; some indexing errors corrected	22·1
6	SFLS, isotropic individual *B*'s	21·2
7	As 6	19·6
7a	Erroneous spot shape correction discovered; reduction repeated; rescaling of reciprocal layers	
8	As 4	17·5
8a	Difference Fourier synthesis, indicating strong anisotropic thermal motion of the bromine atom and to a less extent of the carbon and oxygen atoms	
8b	Introduction of hydrogen atoms at calculated positions; these positions were not altered during the further refinement	
9	SFLS with Br anisotropic, C and O with isotropic individual temperature parameters; H with an overall *B*	12·8
10	SFLS anisotropic for all atoms except H	11·8
11	As 10	11·0
12	As 10	10·8
12a	SF output; some errors corrected	
13	As 10	10·2
13a	Difference Fourier synthesis; no indications of disordering [*e.g.* for atoms C(20), C(21) and O(2)].	

* Abbreviated as SFLS.

Table 3. *Positional coordinates in fractions of the cell edges and standard deviations* (Å) *of* 4-*bromo-duphaston*

	x	*y*	*z*	*σ*(*x*)	*σ*(*y*)	*σ*(*z*)
Br	+0·8229	+0·1208	+0·8178	0·001	0·001	0·001
O(1)	+0·9003	+0·4480	+0·7585	0·009	0·010	0·010
O(2)	−0·0678	+0·5485	+0·9396	0·010	0·012	0·009
C(1)	+0·6578	+0·6644	+0·8368	0·013	0·010	0·011
C(2)	+0·7450	+0·6385	+0·7801	0·012	0·011	0·012
C(3)	+0·8018	+0·4734	+0·7826	0·012	0·012	0·010
C(4)	+0·7424	+0·3281	+0·8180	0·012	0·010	0·010
C(5)	+0·6365	+0·3477	+0·8469	0·011	0·009	0·009
C(6)	+0·5785	+0·2041	+0·8790	0·011	0·010	0·010
C(7)	+0·4807	+0·2189	+0·9166	0·012	0·012	0·011
C(8)	+0·4250	+0·3882	+0·9301	0·010	0·010	0·009
C(9)	+0·4973	+0·5425	+0·9074	0·009	0·008	0·008
C(10)	+0·5697	+0·5173	+0·8415	0·010	0·009	0·009
C(11)	+0·4242	+0·7093	+0·9077	0·012	0·010	0·011
C(12)	+0·2966	+0·6959	+0·8832	0·011	0·009	0·011
C(13)	+0·2321	+0·5510	+0·9168	0·010	0·009	0·009
C(14)	+0·3004	+0·3880	+0·9016	0·008	0·009	0·009
C(15)	+0·2192	+0·2448	+0·9222	0·012	0·011	0·014
C(16)	+0·0995	+0·3048	+0·9011	0·012	0·012	0·013
C(17)	+0·1130	+0·4968	+0·8848	0·009	0·013	0·010
C(18)	+0·2176	+0·5817	+0·9924	0·012	0·014	0·010
C(19)	+0·4956	+0·5098	+0·7779	0·011	0·011	0·009
C(20)	+0·0105	+0·6044	+0·9058	0·011	0·015	0·010
C(21)	+0·0111	+0·7855	+0·8858	0·015	0·019	0·018

anisotropic thermal parameters defined by $\exp\{-2\pi^2 . (h^2a^{*2}U_{11}+k^2b^{*2}U_{22}+l^2c^{*2}U_{33}+2hka^*b^*U_{12}+2hla^*c^*U_{13}+2klb^*c^*U_{23})\}$ are summarized in Table 4. The atoms C(21) and O(2) of the side chain have conspicuously high thermal parameters. It was anticipated that these atoms might interchange their positions freely or that some kind of hindered rotation about the bond C(17)–C(20) should exist. The difference Fourier synthesis, however, calculated in the region of these atoms is very flat and does not support this hypothesis.

The averaged standard deviation in the coordinates is 0·012 Å. It follows that the standard deviation in the bond lengths is about 0·017 Å and in the valency angles about 1°. These values are probably too small and the values (0·024 Å and 1·5°) resulting from an approximative formula of Cruickshank (1960) seem to be more reliable. The errors inherent in the uncertainties in the cell edges are considerably smaller and were therefore neglected. No attempts were made to correct the distances for libration effects.

Geometry of the molecule

The atoms C(19) and C(18) are found below and above the ring system of the steroid molecule. The junction between the rings *B* and *C* is *cis*, which is consistent with the chemical notation (9*β*,10*α*). The junction between *C* and *D* is *trans*. Bond lengths and valency angles are summarized in Fig. 2. The C–C single bonds do not deviate from the accepted value for aliphatic carbon–carbon bonds. The bonds C(4)–C(5) and C(6)–C(7) are 1·37 Å, in accordance with their chemically established double bond character. The other bond lengths, C–O and C–Br, are also near the normally accepted values. The valency angles in the 3-oxo-Δ(4,6) system and in the acetyl group attached to C(17) are on the average 120°. Only the angle C(3)–C(4)–Br is too small. The angles in ring *D* are, of course, considerably smaller than the tetrahedral values, whereas those in the saturated ring *C* are larger. The same phenomena were observed in the cholestane skeleton (Geise, Romers & Rutten, 1966; Geise & Romers, 1966). The pinching of the bonds C(13)–C(17) and C(14)–C(15) consequently enlarges the angles C(12)–C(13)–C(17) and C(8)–C(14)–C(15) to about 117°.

Examination of Dreiding models reveals that the steroid skeleton is nearly planar when ring *C* has a boat form and L-shaped when ring *C* has a chair conformation. The form actually found is a distorted chair. As can be seen from the perspective drawing of the molecule (Fig. 3), rings *A* and *B* are bent away from ring *C*. This can be achieved by a torsion about the bonds C(11)–C(9), C(8)–C(9) and C(7)–C(8). The distorted molecule becomes flatter and the intramolecular distances from C(19) to C(8), C(11), C(12) and C(14), that are too small in the Dreiding models, are enlarged with an amount of 0·7–1·1 Å to reach the

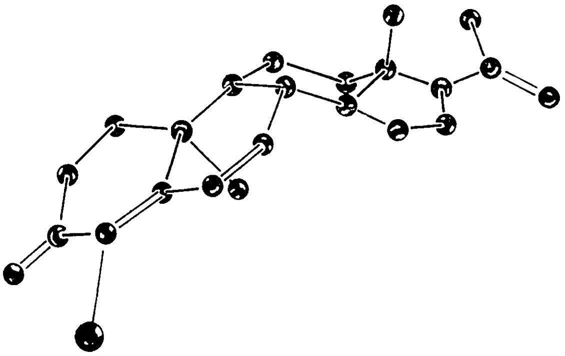

Fig. 3. A perspective drawing of the 4-bromo-duphaston molecule.

Table 4. *Anisotropic temperature parameters U_{11}, U_{22} etc. of* 4-*bromo-duphaston*

	U_{11}	U_{22}	U_{33}	$2U_{12}$	$2U_{23}$	$2U_{31}$
Br	+0·056	+0·046	+0·084	+0·031	−0·002	−0·005
O(1)	+0·052	+0·062	+0·091	+0·008	+0·016	+0·051
O(2)	+0·059	+0·090	+0·066	+0·020	+0·017	+0·028
C(1)	+0·061	+0·033	+0·053	−0·006	−0·011	+0·008
C(2)	+0·052	+0·040	+0·068	−0·011	−0·003	+0·016
C(3)	+0·054	+0·054	+0·046	−0·016	−0·002	+0·024
C(4)	+0·063	+0·037	+0·041	−0·001	−0·007	−0·011
C(5)	+0·045	+0·032	+0·039	−0·003	+0·000	−0·008
C(6)	+0·054	+0·030	+0·052	+0·010	+0·028	−0·005
C(7)	+0·051	+0·049	+0·053	+0·020	+0·021	+0·010
C(8)	+0·045	+0·038	+0·037	+0·005	+0·017	−0·006
C(9)	+0·043	+0·027	+0·031	+0·001	−0·011	+0·001
C(10)	+0·048	+0·026	+0·037	−0·002	−0·014	−0·007
C(11)	+0·055	+0·031	+0·060	+0·002	−0·022	+0·026
C(12)	+0·050	+0·028	+0·057	+0·004	+0·003	+0·001
C(13)	+0·048	+0·030	+0·037	+0·004	+0·002	+0·010
C(14)	+0·034	+0·028	+0·043	−0·011	+0·008	+0·004
C(15)	+0·056	+0·029	+0·087	−0·021	+0·008	+0·011
C(16)	+0·045	+0·046	+0·081	−0·016	−0·022	+0·008
C(17)	+0·030	−0·065	+0·049	+0·006	−0·003	+0·006
C(18)	+0·057	+0·068	+0·041	+0·003	−0·006	+0·001
C(19)	+0·047	+0·046	+0·036	−0·008	+0·001	−0·019
C(20)	+0·043	+0·086	+0·047	+0·026	+0·004	+0·019
C(21)	+0·057	+0·100	+0·106	+0·069	+0·070	+0·043

permissible range of 3·2–3·6 Å. The cyclopentane ring *D* has the conformation of a half chair.

It is interesting to note that the atoms Br, O(1), C(2), C(3), C(4), C(5), C(10) and C(6) are coplanar. The atoms C(7) and C(8), also forming part of the double bond system, are significantly outside this plane. This can be understood as the torsion around C(8)–C(9) and the subsequent distortion of ring *C* lift the atoms above the plane. Although the distortion in the neighbourhood of the bond system C(7)–C(8)–C(9)–C(11)

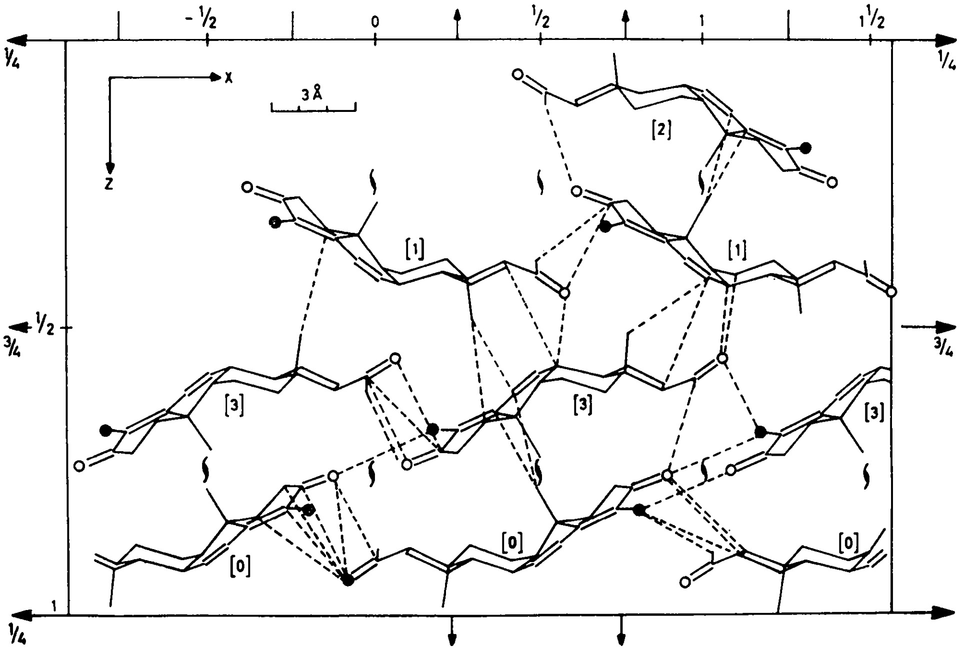

Fig. 4. Projection of the molecules along the *b* axis. The bromine atoms are given as black circles, the oxygen atoms as open circles.

Table 5. *Intermolecular distances* ≤4·00 Å *in* 4-*bromo-duphaston*

The following notation has been used:

molecule							
0a at	x,	$y+1$,	z	or	x,	$y-1$,	z
0b	$x+1$,	y,	z	or	$x-1$,	y,	z
0c	$x+1$,	$y-1$,	z	or	$x-1$,	$y+1$,	z
1	$-x$,	$-y$,	$z-\frac{1}{2}$				
2a	$x-\frac{1}{2}$,	$-y+1\frac{1}{2}$,	$-z+2$	or	$x+\frac{1}{2}$,	$-y+1\frac{1}{2}$,	$-z+2$
2b	$x-\frac{1}{2}$,	$-y+\frac{1}{2}$,	$-z+2$	or	$x+\frac{1}{2}$,	$-y+\frac{1}{2}$,	$-z+2$
3a	$-x+1$,	$y-\frac{1}{2}$,	$-z+1\frac{1}{2}$	or	$-x+1$,	$y+\frac{1}{2}$,	$-z+1\frac{1}{2}$
3b	$-x+2$,	$y-\frac{1}{2}$,	$-z+1\frac{1}{2}$	or	$-x+2$,	$y+\frac{1}{2}$,	$-z+1\frac{1}{2}$

Br · · · C(2)	0a	4·00 Å
Br · · · C(16)	0b	3·92
Br · · · C(21)	0c	3·71
Br · · · O(1)	3b	3·83
C(1) · · · O(2)	0b	3·91
C(2) · · · C(21)	0b	3·94
C(2) · · · O(2)	0b	3·96
C(3) · · · C(20)	0b	2·63
C(3) · · · O(2)	0b	3·56
C(4) · · · O(2)	0b	3·73
C(5) · · · C(19)	3a	3·98
C(6) · · · C(18)	2b	3·80
C(6) · · · C(19)	3a	3·62
C(7) · · · C(16)	2b	3·93
C(7) · · · O(2)	2b	3·64
C(11) · · · O(2)	2a	3·63
C(21) · · · O(1)	3a	3·35
O(1) · · · C(16)	0b	3·88
O(1) · · · C(17)	0b	3·58
O(1) · · · C(20)	0b	3·47
O(1) · · · O(2)	0b	3·76

is considerable, it is interesting that this strain hardly affects the conformation of the other parts (*e.g.* ring *A* or *D*) of the steroid molecule.

Conformational analyses of this and other steroids will be published in a forthcoming paper (Geise, Altona & Romers, 1966).

Molecular environment

Intermolecular distances up to 4 Å have been calculated. A projection of the structure along the *b* axis is given in Fig. 4 and a summary of the shortest intermolecular distances in Table 5. The packing of the hydrogen atoms has not been taken into account. Most close contacts exist between the original molecule and molecules related to this by a shift along the *a* axis, but also contacts are found with symmetry-related molecules. No particular short interactions are found.

We wish to express our gratitude to Prof. Dr E. Havinga for his continuous interest in this investigation. We are indebted to Dr O. A. de Bruin of Philips Duphar Research Laboratories (Weesp, The Netherlands) who provided the samples of 4-chloro- and 4-bromo-duphaston and to the direction of the above mentioned Laboratory for financial support.

All calculations were performed on the X-1 computer of the University of Leiden, with programs devised by Mrs E. W. M. Rutten, Mr W. M. H. Rutten and two of us (B. H. and H. J. G.). Finally, we thank the members of the computing staff of the Centraal Reken-Instituut of the University of Leiden for their help during the calculations.

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Structure Cristalline du Subéramide

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The crystal structure of suberamide has been determined by three-dimensional X-ray crystallographic methods. The crystals are monoclinic with the unit-cell dimensions:

$$a=14\cdot44,\ b=5\cdot13,\ c=14\cdot17\ \text{Å};\ \beta=117°\ 30'.$$

The space group is $C2/c$ with four molecules in the unit-cell. Bond lengths and angles are consistent with the currently accepted value for aliphatic compounds.

Introduction

La détermination de la structure cristalline du subéramide $CONH_2$–$[CH_2]_6$–$CONH_2$ entre dans le cadre des recherches entreprises au Laboratoire sur les diamides aliphatiques. Parmi les composés de cette série seules les structures de l'oxamide (Ayerst & Duke, 1954) et du succinamide (Davies & Pasternak, 1955) ont été publiées. Nous envisageons donc de poursuivre l'étude cristallographique des autres diamides aliphatiques.

Partie expérimentale

Cristallisation

Le subéramide cristallise par lente évaporation d'une solution alcoolique (mélange d'alcools amylique, propylique et éthylique) saturée. Les plaquettes monocliniques* obtenues se clivent facilement suivant la direction [010]. Les clichés ont été faits avec un cristal de section 0,2 × 0,3 mm.

Maille

Les paramètres sont mesurés par oscillation d'un cristal dans une chambre de Bragg.

$a = 14{,}44 \pm 0{,}02$ Å

$b = 5{,}13 \pm 0{,}01$ $\beta = 117° 30' \pm 20'$

$c = 14{,}17 \pm 0{,}02$ $V = 932$ Å^3

Densité calculée: 1,225 avec 4 molécules par maille.

Densité mesurée par flottation: 1,230.

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* La direction d'aplatissement est $(20\bar{1})$.