

qui correspond à la valeur de $c_{\text{orthorhombique}} = 13,55 \text{ \AA}$.

Tracy *et al.* signalent que tous les cristaux issus de leur préparation n'étaient pas identiques. Certains avaient les mêmes paramètres que ceux du cristal étudié, mais pour d'autres, des réflexions supplémentaires sur les rangées avec h et k constants conduisaient à un paramètre c plus grand. D'ailleurs, le nombre de ces réflexions supplémentaires était variable d'un cristal à l'autre. Il est possible que Tracy *et al.* n'aient pas rencontré un seul type de cristal, mais peut être une série de polytypes dus à des irrégularités dans l'empilement des feuillettes. Mais à partir de la préparation de Guen *et al.*, nous avons toujours obtenu le même type orthorhombique que nous décrivons ici.

Conclusion

Dans les composés analogues CrF_2 , CrCl_2 , CrBr_2 , on retrouve la présence d'octaèdres CrX_6 déformés par l'effet Jahn-Teller. Ceci est dû à la présence de l'ion Cr^{2+} . Ils présentent tous 4 distances courtes (l) et 2 longues (L). La déformation de l'octaèdre qui peut être mesurée par le rapport l/L est du même ordre de grandeur dans chacun de ces composés (Tracy, Gregory & Lingafelter, 1962).

	l	L	l/L
CrF_2	1,98 Å	2,43 Å	0,82
CrCl_2	2,39	2,92	0,82
CrBr_2	2,54	3,00	0,82
CrI_2	2,74	3,24	0,85

La présence de cet octaèdre déformé explique la différence de structure entre l'iodure de chrome et les iodures MI_2 des autres éléments de transition (du titane au zinc).

Les ions divalents de ces éléments ne peuvent présenter d'effet Jahn-Teller en champ faible (excepté pour le cuivre, mais il n'existe pas d'iodure de cuivre divalent). Les atomes de ces éléments de transition se trouvent alors au centre d'octaèdres d'atomes d'iode, caractérisés par six distances M-I égales. Ils possèdent de ce fait des structures d'empilement compact d'anions, type CdI_2 ou CdCl_2 , ce qui est exclu pour l'iodure de chrome.

Références

- BUERGER, M. J. (1967). *Vector Space*, p. 204. New York; John Wiley.
- BUSING, W. R., MARTIN, K. O. & LEVY, H. A. (1962). *ORFLS*. Report ORNL-TM-305. Oak Ridge National Laboratory, Oak Ridge, Tennessee.
- CROMER, D. T. & WABER, J. L. (1965). *Acta Cryst.* **18**, 104-109.
- GUEN, L., ALLÉAUME, M., EHOUE, R. & FLAHAUT, J. (1972). *C. R. Acad. Sci. Paris, Sér. C*, **275**, 111-114.
- ÖPICK, R. T. & PRYCE, M. L. (1957). *Proc. Roy. Soc. A* **238**, 425-447.
- PAULING, L. (1960). *The Nature of the Chemical Bond*, p. 514. Ithaca: Cornell Univ. Press.
- TRACY, J. W., GREGORY, N. W. & LINGAFELTER, E. C. (1962). *Acta Cryst.* **15**, 672-674.
- TRACY, J. W., GREGORY, N. W., STEWART, S. M. & LINGAFELTER, E. C. (1962). *Acta Cryst.* **15**, 460-463.

Acta Cryst. (1973). **B29**, 1563

The Crystal Structure of Dendrocrepine Hydrobromide

BY ANNE-MARIE PILOTTI AND ANNE-CHARLOTTE WIEHAGER

Institute of Inorganic and Physical Chemistry, University of Stockholm, S-104 05 Stockholm, Sweden

(Received 14 November 1972; accepted 19 March 1973)

A three-dimensional X-ray study of the alkaloid dendrocrepine, $\text{C}_{33}\text{H}_{44}\text{N}_2\text{O}_3$, has been carried out using the hydrated hydrobromide, $\text{C}_{33}\text{H}_{44}\text{N}_2\text{O}_3 \cdot 2\text{HBr} \cdot \text{H}_2\text{O}$. The crystals are monoclinic, space group Pn , with $a = 15.720$, $b = 8.933$, $c = 11.837 \text{ \AA}$, $\beta = 96.97^\circ$, and two formula units per cell. Bromine was used as a phase-determining heavy atom and the structure was refined to an R value of 0.046. Dendrocrepine possesses a dimeric structure and is optically inactive although it contains several asymmetric carbon atoms. The compound exists as a *d,l*-structure and not as a *meso* form. The X-ray diffraction result indicates the presence of an interesting network of hydrogen bonds in the crystal.

Introduction

Dendrocrepine, $\text{C}_{33}\text{H}_{44}\text{N}_2\text{O}_3$, has been isolated from the orchid *Dendrobium crepidatum* Lindl. together with four closely related alkaloids (Elander, Leander, Rosénblom & Ruusa, 1973). Chemical and spectroscopic studies revealed some functional groups but all attempts

to determine the skeletal structures therefrom have been unsuccessful. The X-ray structure determination of one of these alkaloids, crepidine (Pilotti, 1971), elucidated the structures of all the related alkaloids. Dendrocrepine, however, possesses a dimeric structure and is optically inactive, although it contains several asymmetric carbon atoms. The X-ray analysis of this

compound was undertaken to confirm the structure and to establish whether it exists as a *d,l*-structure or as a *meso* form.

Experimental

The crystal system and approximate cell parameters were determined from oscillation and Weissenberg photographs. Systematic absences were observed in $h0l$ for $h+l$ odd. The crystal data are summarized in Table 1.

The three-dimensional intensities were collected on a Siemens four-circle automatic diffractometer up to a 2θ limit of 118° with monochromatized Cu $K\alpha$ radiation. The machine closely approximates a constant-count-per-reflexion device, except for the weakest intensities. This is accomplished by automatic selection of the measuring time, per step of 0.01° , below a maximum set by the user, and by automatic introduction of attenuators into the incident beam for very strong reflexions. The majority of reflexions are thus recorded with a nearly uniform accuracy. A crystal of irregular shape with a volume of $\sim 0.0022 \text{ mm}^3$ was mounted with the *b* axis coincident with the diffractometer φ axis. Altogether 2586 independent reflexions were collected and 2329 were considered to be observed. Three strong reflexions were chosen as a reference and measured once in every 40 reflexions. Lorentz-polarization and absorption corrections ($\mu = 36.7 \text{ cm}^{-1}$) were applied.

Table 1. *Crystal data*

Lattice constants	$a = 15.720 \pm 9 \text{ \AA}$ $b = 8.933 \pm 5$ $c = 11.837 \pm 7$ $\beta = 96.97 \pm 5^\circ$
Cell volume	$V = 1649.9 \text{ \AA}^3$
Density (X-ray)	$d = 1.400 \text{ g cm}^{-3}$
Molecules per unit cell	$Z = 2$
Space group	Pn

Structure determination and refinement

The crystal structure was solved by the heavy-atom method. The positions of the two independent bromide ions were determined from a three-dimensional Patterson map. Successive use of Fourier and difference Fourier syntheses revealed all the non-hydrogen atomic positions of the dendrocrepine molecule and the water molecule.

Refinement of the atomic parameters was accomplished by several full-matrix least-squares calculations (Gantzel, Sparks & Trueblood, 1966) with individual isotropic thermal motion of each atom. The *R* index fell to 0.104. 2329 reflexions were used in the refinement with Hughes's (1941) weighting procedure, $F_{o,\min} = 2.6$.

A difference Fourier synthesis showed no spurious features but revealed some anisotropy in the atomic shapes. The temperature factors were therefore converted to their equivalent anisotropic β_{ij} values in the equation:

$$\exp [-(h^2\beta_{11} + k^2\beta_{22} + l^2\beta_{33} + hk\beta_{12} + hl\beta_{13} + kl\beta_{23})].$$

The *R* value fell to 0.046 after seven cycles of least-squares refinement.

The positions of the hydrogen atoms were partly deduced from a difference synthesis and partly estimated from chemical considerations. However, contributions of the hydrogen atoms to the structure factors are not included since they yielded no improvement in the accuracy (σ 's) of the structure.

The atomic scattering factors used were those given by Freeman (1959) for carbon, nitrogen and oxygen and that given by Hanson, Herman, Lea & Skillman (1964) for bromine. The atomic coordinates for the non-hydrogen atoms are given in Table 2 together with their estimated standard deviations, and the thermal parameters in Table 3.* Observed and calculated structure factors are given in Table 4.*

* These tables have been deposited with the National Lending Library, England, as Supplementary Publication No. SUP 30095. Copies can be obtained through the Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CHI INZ, England.

Table 2. *Fractional atomic coordinates, with estimated standard deviations in parentheses*

	<i>x</i>	<i>y</i>	<i>z</i>
Br(1)	1.2500	0.9964 (2)	0.2500
Br(2)	1.0748 (1)	0.1644 (2)	0.5663 (2)
N(1)	1.0721 (5)	0.5681 (7)	0.3678 (6)
C(2)	0.9929 (8)	0.5531 (13)	0.4302 (8)
C(3)	1.0260 (10)	0.5874 (16)	0.5546 (9)
C(4)	1.1240 (10)	0.5977 (14)	0.5622 (9)
C(5)	1.1456 (7)	0.5247 (10)	0.4521 (8)
C(6)	1.2270 (8)	0.5747 (9)	0.4073 (8)
C(7)	1.2363 (6)	0.5078 (8)	0.2886 (9)
C(8)	1.3171 (6)	0.5581 (12)	0.2435 (12)
C(9)	1.1539 (5)	0.5391 (8)	0.2058 (7)
O(10)	1.1391 (4)	0.6961 (5)	0.1895 (5)
C(11)	1.1547 (5)	0.4602 (9)	0.0914 (7)
C(12)	1.1968 (7)	0.3258 (10)	0.0791 (9)
C(13)	1.1913 (8)	0.2556 (11)	-0.0280 (10)
C(14)	1.1409 (7)	0.3178 (10)	-0.1212 (9)
C(15)	1.0986 (7)	0.4522 (13)	-0.1096 (8)
C(16)	1.1072 (6)	0.5253 (10)	-0.0051 (8)
C(17)	1.0699 (6)	0.4911 (8)	0.2570 (7)
C(18)	1.0577 (6)	0.3220 (8)	0.2608 (7)
C(19)	0.9857 (5)	0.2719 (8)	0.1717 (7)
O(20)	0.9363 (6)	0.3595 (7)	0.1234 (8)
C(21)	0.9818 (5)	0.1066 (9)	0.1450 (8)
C(22)	0.8970 (5)	0.0651 (8)	0.0728 (7)
C(23)	0.8366 (5)	-0.0410 (9)	0.1296 (7)
C(24)	0.8181 (6)	0.0300 (11)	0.2412 (8)
C(25)	0.7474 (8)	0.1196 (13)	0.2412 (11)
C(26)	0.7306 (12)	0.1938 (21)	0.3377 (13)
C(27)	0.7795 (9)	0.1738 (19)	0.4399 (13)
C(28)	0.8482 (8)	0.0739 (15)	0.4421 (9)
C(29)	0.8654 (6)	0.0003 (11)	0.3453 (9)
O(30)	0.7642 (3)	-0.0423 (4)	0.0462 (2)
C(31)	0.8741 (5)	-0.2021 (9)	0.1414 (7)
C(32)	0.8144 (6)	-0.3127 (11)	0.1933 (9)
C(33)	0.8926 (5)	-0.2592 (8)	0.0253 (7)
C(34)	0.9507 (5)	-0.1545 (8)	-0.0289 (7)
C(35)	0.9654 (7)	-0.1835 (11)	-0.1531 (9)
C(36)	0.9937 (10)	-0.0309 (16)	-0.1935 (14)
C(37)	0.9602 (6)	0.0880 (11)	-0.1183 (10)
N(38)	0.9094 (4)	-0.0020 (6)	-0.0382 (7)
O(39)	1.0783 (1)	0.8919 (2)	0.3752 (2)

Discussion of the structure

Fig. 1 shows a perspective view of the cation and the atom numbering scheme. Bond distances and angles for the structure are listed in Tables 5 and 6. No correction for thermal vibrations has been made. The averages of the least-squares calculated estimated errors for the C-C, C-N and C-O bond lengths are

Table 5. Bond distances (Å), with estimated standard deviations in parentheses

N(1)—C(2)	1.530 (12)	C(19)—C(21)	1.508 (10)
C(2)—C(3)	1.531 (14)	C(21)—C(22)	1.540 (10)
C(3)—C(4)	1.537 (21)	C(22)—C(23)	1.551 (10)
C(4)—C(5)	1.531 (13)	C(23)—C(24)	1.524 (11)
C(5)—N(1)	1.484 (11)	C(24)—C(25)	1.370 (14)
C(5)—C(6)	1.511 (15)	C(25)—C(26)	1.371 (19)
C(6)—C(7)	1.549 (12)	C(26)—C(27)	1.364 (21)
C(7)—C(8)	1.505 (13)	C(27)—C(28)	1.399 (19)
C(7)—C(9)	1.553 (11)	C(28)—C(29)	1.375 (14)
C(9)—O(10)	1.431 (8)	C(29)—C(24)	1.386 (13)
C(9)—C(11)	1.527 (10)	O(30)—C(23)	1.413 (7)
C(11)—C(12)	1.386 (11)	C(31)—C(23)	1.555 (10)
C(12)—C(13)	1.408 (15)	C(31)—C(32)	1.541 (11)
C(13)—C(14)	1.393 (15)	C(31)—C(33)	1.526 (10)
C(14)—C(15)	1.387 (14)	C(33)—C(34)	1.504 (10)
C(15)—C(16)	1.390 (12)	C(34)—C(35)	1.537 (12)
C(16)—C(11)	1.411 (11)	C(35)—C(36)	1.528 (17)
C(17)—C(9)	1.579 (10)	C(36)—C(37)	1.521 (18)
C(17)—N(1)	1.477 (9)	C(37)—N(38)	1.537 (12)
C(17)—C(18)	1.523 (9)	N(38)—C(34)	1.506 (8)
C(18)—C(19)	1.518 (10)	N(38)—C(22)	1.478 (10)
C(19)—O(20)	1.197 (10)		

0.013, 0.011 and 0.009, respectively, and the error in the bond angles is about 0.8°.

Most bond lengths agree well with normally observed values. However, single bonds between highly substituted carbon atoms are significantly longer than those between less substituted carbon atoms, as has

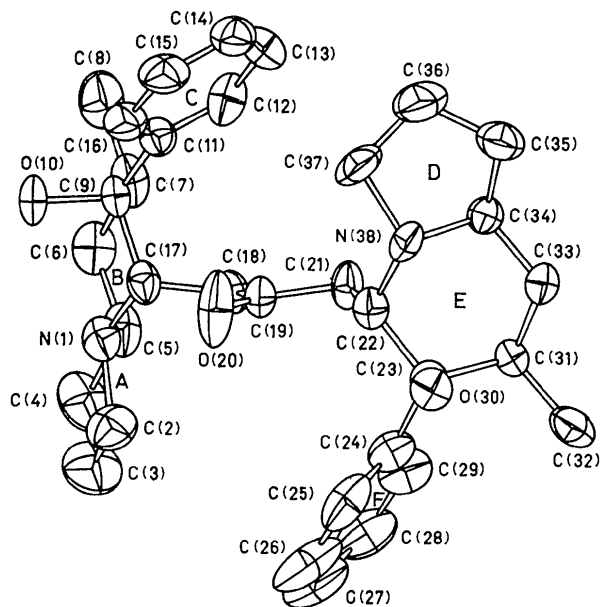


Fig. 1. A perspective view of the cation.

Table 6. Intramolecular bond angles (°), with estimated standard deviations in parentheses

C(2)—N(1)—C(5)	105.5 (0.7)	C(21)—C(19)—O(20)	122.2 (0.8)
C(2)—N(1)—C(17)	117.0 (0.7)	C(19)—C(21)—C(22)	111.2 (0.6)
C(5)—N(1)—C(17)	114.0 (0.7)	C(21)—C(22)—C(23)	116.2 (0.7)
C(3)—C(2)—N(1)	104.1 (0.9)	C(21)—C(22)—N(38)	113.0 (0.6)
C(2)—C(3)—C(4)	106.9 (1.0)	C(23)—C(22)—N(38)	107.0 (0.6)
C(3)—C(4)—C(5)	104.4 (1.0)	C(22)—C(23)—C(24)	108.2 (0.7)
C(4)—C(5)—C(6)	117.2 (0.9)	C(22)—C(23)—C(31)	111.0 (0.6)
C(4)—C(5)—N(1)	103.0 (0.9)	C(22)—C(23)—O(30)	100.7 (0.6)
C(6)—C(5)—N(1)	108.1 (0.7)	C(24)—C(23)—C(31)	114.7 (0.7)
C(5)—C(6)—C(7)	112.4 (0.8)	C(24)—C(23)—O(30)	112.7 (0.6)
C(6)—C(7)—C(8)	112.5 (0.9)	C(31)—C(23)—O(30)	108.7 (0.6)
C(6)—C(7)—C(9)	109.9 (0.8)	C(23)—C(24)—C(25)	118.9 (0.9)
C(8)—C(7)—C(9)	113.4 (0.8)	C(23)—C(24)—C(29)	123.5 (0.8)
C(7)—C(9)—O(10)	111.8 (0.6)	C(25)—C(24)—C(29)	117.4 (1.0)
C(7)—C(9)—C(11)	112.4 (0.7)	C(24)—C(25)—C(26)	121.3 (1.3)
C(7)—C(9)—C(17)	112.4 (0.7)	C(25)—C(26)—C(27)	122.0 (1.6)
C(11)—C(9)—O(10)	110.6 (0.6)	C(26)—C(27)—C(28)	116.8 (1.4)
C(11)—C(9)—C(17)	108.2 (0.6)	C(27)—C(28)—C(29)	121.3 (1.1)
C(17)—C(9)—O(10)	100.8 (0.6)	C(24)—C(29)—C(28)	120.6 (1.0)
C(9)—C(11)—C(12)	123.2 (0.8)	C(23)—C(31)—C(32)	112.6 (0.7)
C(9)—C(11)—C(16)	117.8 (0.7)	C(23)—C(31)—C(33)	109.9 (0.6)
C(12)—C(11)—C(16)	119.0 (0.8)	C(32)—C(31)—C(33)	109.6 (0.7)
C(11)—C(12)—C(13)	120.1 (0.9)	C(31)—C(33)—C(34)	111.5 (0.6)
C(12)—C(13)—C(14)	120.1 (0.9)	C(33)—C(34)—C(35)	118.2 (0.7)
C(13)—C(14)—C(15)	120.0 (0.9)	C(33)—C(34)—N(38)	108.3 (0.7)
C(14)—C(15)—C(16)	119.8 (0.9)	C(35)—C(34)—N(38)	101.2 (0.7)
C(11)—C(16)—C(15)	120.8 (0.8)	C(34)—C(35)—C(36)	103.5 (0.9)
C(9)—C(17)—C(18)	113.3 (0.6)	C(35)—C(36)—C(37)	107.7 (1.1)
C(9)—C(17)—N(1)	106.5 (0.6)	C(36)—C(37)—N(34)	103.8 (0.9)
C(18)—C(17)—N(1)	115.2 (0.6)	C(22)—N(38)—C(38)	113.8 (0.6)
C(17)—C(18)—C(19)	111.0 (0.6)	C(22)—N(38)—C(37)	118.0 (0.6)
C(18)—C(19)—C(21)	116.2 (0.6)	C(34)—N(38)—C(37)	105.5 (0.6)
C(18)—C(19)—O(20)	121.6 (0.7)		

been previously observed (Birnbaum & Ferguson, 1969). There are six C-N⁺ bonds in the molecule, each protonated nitrogen atom being attached to three C(sp³) atoms, and the mean of their lengths is 1.502 Å, normal for this kind of bond (Singh & Ahmed, 1969). In both ring systems, two of the C-N⁺ bonds are about

Table 7. Bond distances to the bromide ions shorter than 4.0 Å

Code for symmetry-related atoms:			
Superscript	Coordinates	Superscript	Coordinates
none	x, y, z	iv	$-\frac{1}{2}+x, -y, -\frac{1}{2}+z$
i	$x, -1+y, z$	v	$-\frac{1}{2}+x, 1-y, \frac{1}{2}+z$
ii	$x, y, 1+z$	vi	$-\frac{1}{2}+x, -y, \frac{1}{2}+z$
iii	$-\frac{1}{2}+x, 1-y, -\frac{1}{2}+z$	vii	$x, 1+y, 1+z$
O(10)-Br(1)	3.23 Å	C(2)-Br(2)	3.98 Å
O(39)-Br(1)	3.37	C(3)-Br(2)	3.85
C(12)-Br(1 ⁱ)	3.61	C(4)-Br(2)	3.95
O(30)-Br(1 ⁱⁱⁱ)	3.51	C(5)-Br(2)	3.71
C(35)-Br(1 ⁱⁱⁱ)	3.83	C(18)-Br(2)	3.86
C(36)-Br(1 ⁱⁱⁱ)	3.82	C(28)-Br(2)	3.78
C(37)-Br(1 ⁱⁱⁱ)	3.56	C(14 ⁱⁱ)-Br(2)	3.96
N(38)-Br(1 ⁱⁱⁱ)	3.32	C(36 ⁱⁱ)-Br(2)	3.69
		O(30)-Br(2 ^{iv})	3.21
		O(39 ⁱ)-Br(2)	3.33

Table 8. Intermolecular distances shorter than 3.8 Å between the cations

C(32)-O(20 ⁱ)	3.65 Å
C(33)-O(20 ⁱ)	3.64
C(34)-C(16 ⁱ)	3.76
C(35)-C(16 ⁱ)	3.72
C(25)-C(4 ⁱⁱⁱ)	3.69
C(26)-C(16 ^v)	3.80
C(32)-C(14 ^{vi})	3.70

equal, while the third is $\sim 4\sigma$ longer than the mean of the other two. The longer C-N⁺ bond is the one involved only in the five-membered ring. The two C(sp²)-C(sp³) single bonds agree well in their lengths with the accepted value for such bonds of 1.501 Å (Lide, 1962).

Intermolecular distances shorter than 3.8 Å between the cations are given in Table 8 and distances to the bromide ions shorter than 4.0 Å in Table 7.

An interesting feature of this crystal structure is the network of hydrogen bonds involving two bromide ions (see Fig. 2), two quaternary nitrogen atoms, two hydroxyl groups, and one molecule of water. The hydroxyl oxygen atoms O(10) and O(30) act as donors in two hydrogen bonds to the two bromide ions Br(1) and Br(2). The distances O(10)-Br(1) and O(30^{iv})-

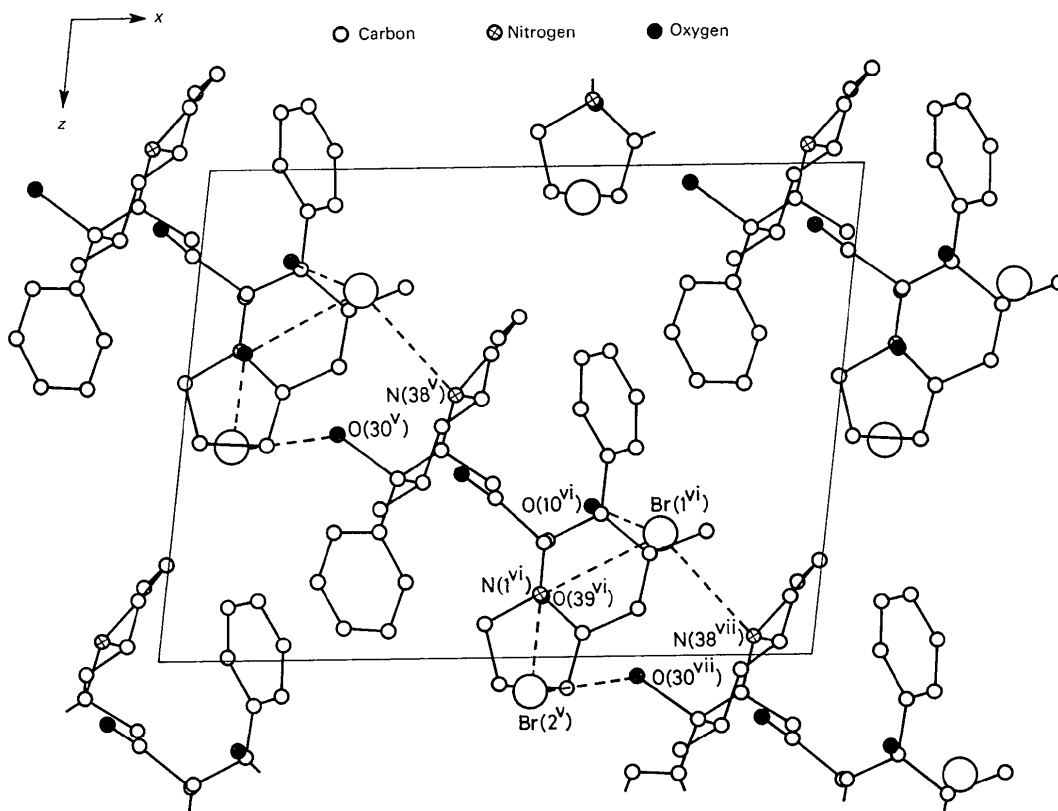


Fig. 2. The structure viewed along the *b* axis. Hydrogen bonds are marked with broken lines.
○ carbon ⊗ nitrogen ● oxygen
The large open circles denote bromide ions.

Br(2), 3.23 and 3.21 Å respectively, indicate normal hydrogen bonds (Hamilton & Ibers, 1968). The distances between the water oxygen atom O(39) and the bromide ions Br(1) and Br(2¹) are close to the weighted average of 3.34 Å obtained by Clark (1963) in a review of hydrated compounds. The bromide ion Br(1) also accepts a hydrogen atom of the quaternary nitrogen atom N(38ⁱⁱⁱ), at a distance of 3.33 Å. The distance of 2.89 Å between the quaternary nitrogen N(1) and the water oxygen O(39) also indicate a hydrogen bond (Clark, 1963).

Some calculated mean planes and their relationships to one another are given in Table 9. The five-membered

ring *D* adopts the envelope conformation, atoms C(35), C(36), C(37) and N(38) are coplanar within ± 0.013 and atom C(34) is 0.635 Å out of the plane.

Among the possible four-atom planes for the five-membered ring *A*, the set N(1), C(2), C(3) and C(4) exhibits the smallest deviation from a least-squares plane, ± 0.048 Å. C(5) has a 'normal' deviation of 0.575 Å from the plane. This departure from the ordinary envelope form of the ring is presumably an effect of intermolecular contacts and, especially, of the hydrogen bond N(1)–O(39).

The authors wish to express their sincere gratitude to Professor Peder Kierkegaard for his stimulating interest in this work. Our thanks are also due to Dr Kurt Leander for the supply of crystals. Dr Don Koenig who corrected the English of this paper is cordially thanked.

The present investigation has received financial support from the Tri-Centennial Fund of the Bank of Sweden and from the Swedish Natural Science Research Council.

References

- BIRNBAUM, K. G. & FERGUSON, G. (1969). *Acta Cryst.* **25**, 720–730.
 CLARK, J. R. (1963). *Rev. Pure Appl. Chem.* **13**, 50–90.
 ELANDER, M., LEANDER, K., ROSENBLUM, J. & RUUSA, E. (1973). *Acta Chem. Scand.* In the press.
 FREEMAN, A. J. (1959). *Acta Cryst.* **12**, 261–271.
 GANTZEL, P. K., SPARKS, R. A. & TRUEBLOOD, K. N. (1966). *IUCr World List of Crystallographic Computer Programs*, 2nd ed., program 384.
 HAMILTON, W. C. & IBERS, J. A. (1968). *Hydrogen Bonding in Solids*. New York: Benjamin.
 HANSON, H. P., HERMAN, F., LEA, J. D. & SKILLMAN, S. (1964). *Acta Cryst.* **17**, 1040–1044.
 HUGHES, E. W. (1941). *J. Amer. Chem. Soc.* **63**, 1737–1752.
 LIDE, D. R. (1962). *Tetrahedron*, **17**, 125–134.
 PILOTTI, A.-M. (1971). *Acta Cryst.* **27**, 887–892.
 SINGH, P. & AHMED, F. R. (1969). *Acta Cryst.* **25**, 1901–1910.

Table 9. *Least-squares planes and deviations*

The planes are described in terms of axes (*m, n, p*) having $m||a^*$, $n||b$ and $p||c$. The atoms indicated with asterisks were omitted from the calculations of the least-squares planes.

Plane <i>A</i>	$-0.1177m + 0.9876n - 0.1034p = 2.7691$
Plane <i>B</i>	$0.3065m + 0.9514n - 0.0281p = 6.8397$
Plane <i>C</i>	$0.8036m - 0.4982n - 0.3257p = 3.9546$
Plane <i>D</i>	$0.8281m + 0.0673n + 0.5565p = 8.5130$
Plane <i>E</i>	$0.7166m + 0.1471n + 0.6817p = 9.7799$
Plane <i>F</i>	$0.5897m + 0.7616n - 0.2688p = 7.3451$

Deviations from planes

Plane <i>A</i>		Plane <i>B</i>		Plane <i>C</i>	
N(1)	0.031 Å	N(1)*	-0.689 Å	C(11)	-0.012 Å
C(2)	-0.048	C(5)	0.008	C(12)	-0.009
C(3)	0.048	C(6)	-0.008	C(13)	0.019
C(4)	-0.031	C(7)*	0.645	C(14)	-0.009
C(5)*	-0.575	C(9)	0.008	C(15)	-0.013
		C(17)	-0.008	C(16)	0.023
Plane <i>D</i>		Plane <i>E</i>		Plane <i>F</i>	
C(34)*	0.635 Å	C(22)	0.000 Å	C(24)	0.045 Å
C(35)	0.009	C(23)	0.000	C(25)	-0.033
C(36)	-0.013	C(31)*	0.679	C(26)	0.002
C(37)	0.013	C(33)	0.000	C(27)	0.016
N(38)	-0.008	C(34)	0.000	C(28)	-0.002
		N(38)*	-0.684	C(29)	-0.028

Angles between normals to the planes

$\angle AB$	11.9°	$\angle BC$	76.3°
$\angle DE$	10.7	$\angle EF$	69.4

Acta Cryst. (1973). **B29**, 1567

Structure Cristalline de YSF (β)

PAR N. RYSANEK ET O. LOYE

Laboratoire de Physique, Faculté des Sciences Pharmaceutiques et Biologiques, Université Paris V,
4 avenue de l'Observatoire, 75270 Paris Cedex 06, France

(Reçu le 31 janvier 1973, accepté le 8 mars 1973)

The crystal structure of YSF(β) has been determined from three-dimensional X-ray diffractometer data and refined by least-squares methods to an *R* index of 5.1%. The compound crystallizes in the hexagonal space group $P6_3/mmc$ with $Z = 4$; lattice parameters are $a = 4.062$ and $c = 16.693$ Å. The structure of YSF(β) consists of compact layers (YF₂) and (YS₂) parallel to plane (001).

Introduction

L'étude cristallographique de YSF(β) fait partie d'une étude plus vaste portant sur les halogénosulfures de

terres rares. Dagrón & Thévet (1971) ont mis en évidence des composés de formule LSX où X = F, Cl, Br, I. Ces composés appartiennent à différents types cristallins dont les structures ont été étudiées au labora-