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Optical Properties and Preliminary X-Ray Investigation of Retamine and its Bromide and Chloride

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The morphological, optical and X-ray properties of retamine and its bromide and chloride have been determined. These compounds crystallize in the enantiomorphic monoclinic class, space group $P21$; they are biaxial positive with optical axial angles of 66° and 77° respectively. The bromide and chloride are isomorphous.

Introduction

Retamine, an alkaloid contained in *Genista sagittalis*, was isolated and studied by Prof. Ribas Marquès and his collaborators of the Department of Organic Chemistry at the University of Santiago de Compostela (Spain) (see *e.g.* Ribas Marquès, 1961; Font-Altaba, 1957; Bosch-Figueroa, 1963). The molecules of retamine and sparteine have similar structures, which differ only in the presence of a hydroxyl group in position 7 or 9; this is indicated by hydrogenation of retamine, which produces sparteine and not α- or β-isosparteine.

Morphological and optical properties

Retamine and its bromide and chloride appear as colourless enantiomorphic crystals of tabular habit with a very simple combination of forms.

Retamine bromide presents the better crystals, with {100} as the predominant form, which confers on them a tabular habit (Fig. 1).

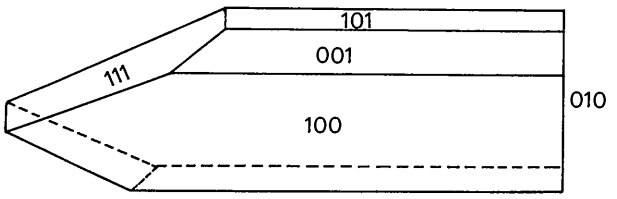

Fig. 1. Crystal of retamine bromide.

Crystals of retamine are of very varied size and thickness, very frequently with rounded edges and very rarely in quadrangular shapes. They lie frequently on the face ($\bar{1}01$).

Retamine chloride forms small crystals, and this makes its study difficult.

As retamine chloride is isomorphous with retamine bromide, we give in Table 1 the optical properties, and morphological and crystallographic data only of retamine and its bromide.

Table 1. *Morphological data of retamine and retamine bromide*

Retamine			Retamine bromide		
hkl	φ	ϱ	*hkl*	φ	ϱ
001	90°0′	15°07′	100	90°0′	90°0′
$\bar{1}01$	270 0	28 48	010	0 0	90 0
011	11 15	54 08	001	90 0	10 38
$\bar{1}00$	270 0	90 0	$\bar{1}01$	270 0	31 27
			$\bar{1}\bar{1}\bar{1}$	223 33	41 35
a:*b*:*c*:	1·7137	:1:1·3565	*a*:*b*:*c*:	0·81704	:1:0·6440
β	105°07		β	100°38	

By means of the polarizing microscope and the universal stage the orientation of the optical ellipsoid was determined. Refractive indices were measured by the immersion method. The results are shown in Table 2. Stereographic projections of retamine and retamine bromide are shown in Fig. 2.

Table 2. *The optical properties for sodium light of retamine and retamine bromide*

	Retamine No pleochroism	Retamine bromide No pleochroism
α	$1{\cdot}594_6$	1·602
β	1·600	1·614
γ	1·614	1·636
$\frac{1}{4}(\alpha+2\beta+\gamma)$	1·602	1·6165
Maximum birefringence	$0{\cdot}019_4$	0·034
Medium birefringence	$0{\cdot}012_9$	0·023
Acute bisectric	$=\gamma$, in a direction 56°12′ from the c axis in the obtuse angle	$=\gamma$, in a direction 60°30′ from the c axis in the obtuse angle
Optic axial plane	(010)	(010)
Optical axial angle	$2V=66°48'$	$2V=77°0'$
Optic sign	positive	positive

X-ray oscillation and Weissenberg photographs (using Cu $K\alpha$ Ni-filtered radiation, $(K\alpha_1)=1{\cdot}54051$ Å) were taken on the two types of crystal described above.

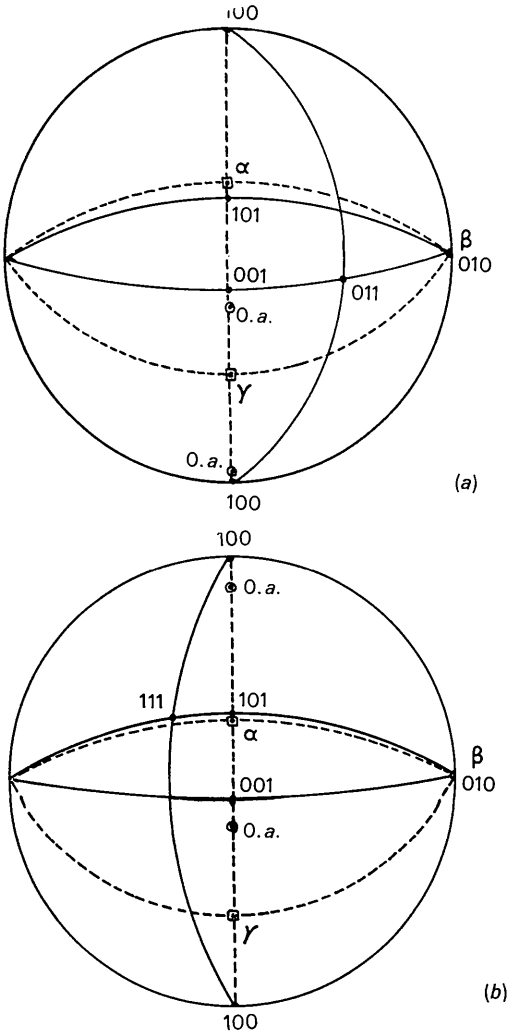

Fig. 2. Stereographic projections of crystal faces and optical indicatrix of (*a*) retamine and (*b*) retamine bromide. Full lines represent crystal zones and dashed lines the principal sections of the optical indicatrix.

In Table 3 are recorded the space group and unit-cell dimensions of both compounds.

Table 3. *Single-crystal data of retamine and retamine bromide*

	Retamine	Retamine bromide
a_0	11·873 Å	9·319 Å
b_0	6·774	11·298
c_0	9·227	7·298
β	105°08′	100°20′
Space group	$P2_1$	$P2_1$

Powder photographs of retamine and its bromide and chloride have been taken by the asymmetric focalization technique of Guinier, as modified by de Wolff, in a Nonius quadruple camera which corresponds to a camera of diameter 229·2 mm.

Tables 4, 5 and 6 give the interplanar spacing, relative peak intensities and indices of the low-order lines, the indices of the higher-order lines being omitted.

Table 4. *Powder-photograph data of retamine*

Interplanar spacing	Relative peak intensity	Indices
8·908 Å	45	001
8·028	60	$10\bar{1}$
6·281	30	010
5·813	60	110
5·731	30	200
5·378	100	011
5·192	30	$11\bar{1}$
4·607	60	111
4·550	25	$10\bar{2}$
4·460	45	002
4·331	45	210, 201
4·300	20	$21\bar{1}$
4·015	45	$20\bar{2}$
3·820	15	300, 102, $11\bar{2}$
3·749	10	012
3·623	30	211
3·451	5	$21\bar{2}$
3·387	45	020, $30\bar{2}$, $31\bar{1}$
3·325	10	112, $3\bar{1}0$
3·164	10	021, 202
3·121	45	$12\bar{1}$
2·975	45	003, $20\bar{3}$, $40\bar{1}$, 121
2·905	15	220, 311
2·872	10	400, $22\bar{1}$
2·842	5	212
2·808	10	$11\bar{3}$
2·717	30	013, 103, $30\bar{3}$, $12\bar{2}$, $21\bar{3}$, $41\bar{1}$
2·693	15	022
2·595	30	302, $22\bar{2}$
2·536	5	401, 320, 122
2·512	20	113, $31\bar{3}$
2·400	15	203, $40\bar{3}$, 312, $32\bar{2}$
2·360	5	411
2·319	45	321
etc.		

Table 5. *Powder photograph data of retamine bromide*

Interplanar spacing	Relative peak intensity	Indices
9·135 Å	30	100
7·131	45	001, 110
6·237	30	$10\bar{1}$

Table 5 (*cont.*)

Interplanar spacing	Relative peak intensity	Indices
6·069	30	011
5·684	15	020
5·443	100	11$\bar{1}$
4·830	10	120
4·740	15	111
4·584	15	200
4·459	30	021
4·200	30	20$\bar{1}$, 12$\bar{1}$
3·962	15	21$\bar{1}$
3·836	60	121
3·573	45	002, 220, 10$\bar{2}$, 201
3·490	30	130
3·425	25	211
3·412	15	012
3·343	30	031
3·321	10	131
3·154	10	102
3·121	10	20$\bar{2}$
3·059	30	300
3·039	30	022, 112, 221
2·492	5	310
2·910	15	230, 31$\bar{1}$
2·833	15	040
2·816	15	23$\bar{1}$
2·753	20	122
2·686	10	320
2·622	5	32$\bar{1}$
2·595	5	032, 202, 13$\bar{2}$, 231
2·574	10	30$\bar{2}$, 14$\bar{1}$, 311
2·536	20	212
2·485	5	141
2·403	5	23$\bar{2}$, 321
etc.		

Table 6. *Powder photograph data of retamine chloride*

Interplanar spacing	Relative peak intensity	Indices
9·136 Å	60	100
7·160	45	001
7·075	60	110
6·259	15	10$\bar{1}$
6·008	15	011
5·527	60	020
5·427	100	11$\bar{1}$
5·177	5	101

Table 6 (*cont.*)

Interplanar spacing	Relative peak intensity	Indices
4·728	10	120
4·679	10	111
4·561	5	200
4·373	60	021
4·134	45	12$\bar{1}$
3·953	10	21$\bar{1}$
3·773	60	121
3·573	10	002, 10$\bar{2}$, 201
3·511	10	220
3·406	60	012, 130, 11$\bar{2}$
3·355	10	221, 211
3·272	45	031
3·170	15	13$\bar{1}$
3·116	10	102, 20$\bar{2}$
3·034	15	300, 30$\bar{1}$
3·000	45	022, 12$\bar{2}$, 112, 131, 21$\bar{2}$
2·984	5	221
2·928	10	310
2·900	5	31$\bar{1}$
2·859	5	230
2·774	15	23$\bar{1}$
2·762	5	040
2·721	15	122, 22$\bar{2}$
2·659	5	320
2·644	15	140, 301, 32$\bar{1}$
2·581	5	041, 30$\bar{2}$, 202
2·553	15	032, 13$\bar{2}$, 231, 311
etc.		

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