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The absolute configuration of natural strychnine.

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In order to illustrate that the X-ray method of determining absolute configuration may be applied to compounds of arbitrary complexity we proposed the determination on strychnine hydrobromide 2 aq., the atomic coordinates of which have been given by Robertson & Beevers (1951). These authors give a comparison of observed and calculated values of about 80 structure factors. Recalculating these, we found the agreement better than the authors' comparison shows. We may thus expect the coordinates to be well within the range given.

After the experience of Peterson (1955) showing that Cu $K\alpha$ rays have a sufficiently large anomalous effect with Br atoms, we dispensed with the U $L\alpha$ rays which have previously been used for exciting Br (Trommel & Bijvoet, 1954). The values of $\Delta f'_{\mathrm{Br}}$ and $\Delta f''_{\mathrm{Br}}$ for copper rays amount to $-0{\cdot}9$ and $1{\cdot}5$ respectively (Dauben & Templeton, 1955).

The crystal used in the determination was obtained from a solution of strychnine hydrobromide in water with a slight excess of HBr. As described by Robertson & Beevers, it had the form of a lath parallel to the *a* axis with a rectangular cross-section of about $0{\cdot}03 \times 0{\cdot}12$ mm. Because of this rectangular cross-section no spurious intensity differences by absorption effects between *hkl* and $\bar{h}\bar{k}\bar{l}$ reflexions were to be feared, and consequently cylindrical grinding could be omitted.

An equi-inclination Weissenberg diagram was made of the first layer line around the *a* axis with the aid of an integrating Weissenberg camera.

Previous calculation had shown that the series of reflexions 111, 112, 113 and 114 were most suitable for our determination. The differences between reflexion and counter-reflexion were clearly observable in this series and could be measured with a photometer. The calculated and observed values of the intensities—expressed in different units—are given in Table 1.

Table 1. *Calculated and observed intensities of reflexions hkl and $\bar{h}\bar{k}\bar{l}$ from strychnine* HBr . 2H$_2$O (Cu $K\alpha$ *rays*)

	Calculated			Observed		
hkl	I_{hkl}		$I_{\bar{h}\bar{k}\bar{l}}$	I_{hkl}		$I_{\bar{h}\bar{k}\bar{l}}$
111	295	>	264	40·8	<	45·5
112	513	<	567	82·5	>	75·0
113	200	<	259	37·1	>	31·6
114	372	~	378	56·6	=	56·6

The former are based on the model corresponding to the coordinates of Robertson & Beevers in a right-handed coordinate system.

The comparison of the observed and calculated differences shows that the model used in the calculations is the mirror image of the actual molecule.

Natural strychnine, therefore, has the configuration of Fig. 1. It may be remarked that in the 111 reflexion the contribution to the structure factor coming from the normally scattering atoms is such that a shift of 3° in all atomic positions cannot change the sign of the resulting component normal to the bromine contribution, even when all changes in this component accumulate. As the coordinates are accurate within this limit the reliability of the calculated difference for the 111 and $\bar{1}\bar{1}\bar{1}$ reflexions is beyond doubt.

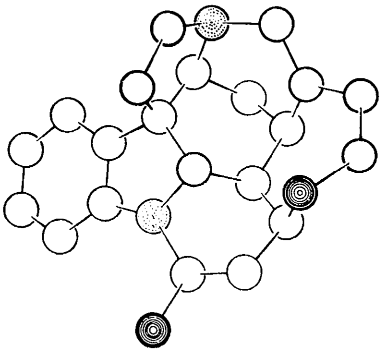

Fig. 1. Absolute configuration of the natural strychnine molecule.

References

Dauben, C. H. & Templeton, D. H. (1955). *Acta Cryst.* **8**, 841.

Peterson, S. W. (1955). *Nature, Lond.* **176**, 395.

Robertson, J. H. & Beevers, C. A. (1951). *Acta Cryst.* **4**, 270.

Trommel, J. & Bijvoet, J. M. (1954). *Acta Cryst.* **7**, 703.

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Crystallographic properties of 9-aminoacridine hemihydrate.

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The compound 9-aminoacridine is interesting as it belongs to the space group D_{2h}^{24}–*Fddd*. This assignment is based on *b*-axis and *c*-axis single-crystal rotation patterns and *b*-axis and *c*-axis zero-layer Weissenberg patterns. According to Donnay & Nowacki (1954) there is no other strictly organic compound, that is, group 7 aside from

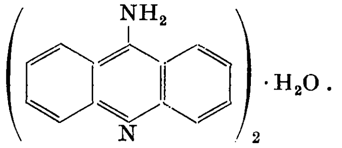
Table 1. *X-ray powder diffraction data for 9-aminoacridine hemihydrate*

d_o (Å)	I/I_1	*hkl*	d_c (Å)
8·65	0·10	131, 311	8·65, 8·65
6·56	0·50	022, 202	6·56, 6·55
6·10	0·50	151, 511, 440	6·11, 6·11, 6·10
5·48	0·05	351, 531	5·46, 5·46
		260, 620	5·46, 5·46
5·22	0·30	242, 422	5·22, 5·22
4·63	0·80	171, 711	4·62, 4·62
4·46	0·10	062, 602	4·47, 4·47
4·33	1·00	133, 313	4·34, 4·34
		262, 622	4·32, 4·32
		731, 371	4·32, 4·32
		080, 800	4·32, 4·32
4·07	0·30	660	4·07
3·86	0·30	480, 840	3·86, 3·86
3·53	0·50	004	3·54
3·39	0·05	173, 713	3·40, 3·40
3·27	0·30	044, 404	3·28, 3·27
		373, 733	3·27, 3·27
3·10	0·30	682, 862	3·11, 3·10
3·06	0·30	2,10,2, 10,2,2	3·06, 3·05
2·97	0·30	—	—
2·86	0·05	—	—
2·73	0·20	—	—
2·60	0·20	—	—
2·43	0·05	—	—
2·31	0·05	—	—
2·22	0·05	—	—
2·15	0·05	—	—
2·09	0·05	—	—
2·05	0·05	—	—
1·987	0·10	—	—
1·905	0·05	—	—
1·843	0·05	—	—
1·770	0·05	—	—

group 7*a*, in this space group. *Fddd* is one of the rarer space groups, now having a total of 10 representatives.

By examining the near equivalence of the *a* and *b* dimensions, one might be led to believe that the crystal is tetragonal. This is not so because an 001 section of the crystal is birefringent and shows a small but definite biaxial interference figure. On examining the data for the other crystals in this space group, it appears that they all have two axes nearly equal or one about one-half of another.

There is some confusion about the numbering of the acridine ring system but the compound discussed here is represented by the structure formula:



The water content of the crystals was checked by the Karl Fisher method and found to be 5·03%, compared to 4·43% theoretical.

Crystallization by slow evaporation of a water–acetone solution results in rods elongated parallel to the *c* axis and showing the macropinacoid {100}, brachypinacoid {010}, prism {110}, brachydome {011}, and the orthodome {101}. The polar interfacial angle (110):($\bar{1}$10) is 90° 04′ (calculated from X-ray data), 90° 03′ (by optical measurement). The crystals show fair 001 cleavage. The sample used for this study lost water at about 135° C. and melted 238–240° C. The crystal system is orthorhombic with space group D_{2h}^{24}–*Fddd* and 32 molecules of $(C_{13}H_{10}N_2)_2.H_2O$ per cell. The observed density is 1·277 g.cm.$^{-3}$ (flotation), while the density calculated from the X-ray data is 1·280 g.cm.$^{-3}$. The cell dimensions are

$$a = 34{\cdot}52,\quad b = 34{\cdot}56,\quad c = 14{\cdot}17\ \text{Å}\,.$$

The optical properties are:

$\alpha > 1{\cdot}70$, $\beta > 1{\cdot}70$, $\gamma > 1{\cdot}70$; negative; $2V$ about 5°. The optic plane is parallel to 010, $\alpha = c$.

The powder data (Table 1) were obtained using a camera 114·6 mm. in diameter with chromium radiation and a vanadium filter. A wavelength value of 2·2896 Å was used in the calculations.

Reference

Donnay, J. D. H. & Nowacki, W. (1954). *Crystal Data.* New York: The Geological Society of America.

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Die Kristallstruktur des wasserhaltigen Natriumhexafluorophosphats, $NaPF_6.H_2O$.

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Das von Lange & Müller (1930) zuerst dargestellte $NaPF_6.H_2O$ kristallisiert in farblosen Prismen von hexagonalem Habitus. Das Hydrat lässt sich ohne Zersetzung entwässern zum kubisch kristallisierenden $NaPF_6$; über diese Struktur wurde bereits berichtet (Bode & Teufer, 1952). Nach eigenen Beobachtungen zeigen die Kristalle von $NaPF_6.H_2O$ gerade Auslöschung und sind optisch zweiachsig negativ, gehören also einer rhombischen (pseudohexagonalen) Kristallklasse an. Aus Drehkristallaufnahmen erhält man folgende Abmessungen der Elementarzelle:

$$a = 7{,}962 \pm 0{,}005,\quad b = 10{,}594 \pm 0{,}010,\quad c = 6{,}116 \pm 0{,}005\ \text{Å}\,.$$

Die Aufstellung der Elementarzelle entspricht den *Internationale Tables* (1952). Das röntgenographisch ermittelte Achsenverhältnis beträgt $a:b:c = 0{,}7516:1:0{,}5773$. Dem pseudohexagonalen Charakter entsprechend ist die b-Achse gleich $c\sqrt{3}$. Die pyknometrische Dichte ist 2,39 g.cm.$^{-3}$; demzufolge enthält die Elementarzelle vier Molekeln $NaPF_6.H_2O$, und die röntgenographische Dichte berechnet sich zu 2,391 g.cm.$^{-3}$.

Aus Röntgengoniometeraufnahmen nach Sauter–Schie-