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Crystallographic data for heteratisine and some derivatives. By R. ANEJA and S. W. PELLETIER,
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Heteratisine was isolated from the roots of *Aconitum heterophyllum* by Jacobs & Craig (1942). Some of its chemistry (Aneja & Pelletier, unpublished) is markedly different from that of the other aconite alkaloids, largely owing to the presence of a lactone ring in its molecule. This structural feature has not been encountered in the aconites studied earlier.

We have prepared some derivatives of heteratisine and obtained preliminary X-ray crystallographic data for them, as also for the parent alkaloid (Table 1). The unit-cell dimensions were measured from precession photographs taken with Cu $K\alpha$ radiation ($\lambda = 1.5418 \text{ \AA}$). The space groups were deduced from systematic absences and the fact that these compounds are optically active. The densities were measured by flotation, in aqueous cesium chloride for heteratisine and in mixtures of carbon tetrachloride and benzene for the hydrohalides.

Diffraction spots of the hydrobromide (II) and the hydroiodide showed tails, evidently due to disorder in

the growth of the crystals. Hydrobromide (I) was efflorescent and gradually crumbled to a powder on loss of solvent of crystallization. Moreover, its asymmetric unit contains two molecules. The crystals were, therefore, considered unsuitable for a complete structure analysis and no further work on these derivatives is contemplated.

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References

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Table 1. *Crystal data for heteratisine and derivatives*

	Heteratisine	Heteratisine hydrobromide (I)	Heteratisine hydrobromide (II)	Heteratisine hydroiodide
Solvent of crystallization	Ethanol	Methanol	Acetonitrile	Ethanol
m.p.	267-69°	280-83°	272-74°	274-76°
$[\alpha]_D^{25}$ ($c = 1.0$, CH_3OH)	+40°	+24°	+26°	+19°
Mol. formula	$\text{C}_{22}\text{H}_{33}\text{O}_5\text{N}$	$\text{C}_{22}\text{H}_{34}\text{O}_5\text{NBr} \cdot 0.5\text{CH}_3\text{OH}$	$\text{C}_{22}\text{H}_{34}\text{O}_5\text{NBr}$	$\text{C}_{22}\text{H}_{34}\text{O}_5\text{NI}$
% C, H, N (found)	67.57, 8.60, 3.74	55.25, 7.42, 2.91	55.89, 7.26, 2.88	50.86, 6.44, 2.82
% C, H, N (calc.)	67.47, 8.50, 3.58	55.25, 7.55, 2.86	55.85, 7.25, 2.96	50.87, 6.60, 2.69
Crystal system	Monoclinic	Orthorhombic	Monoclinic	Orthorhombic
a (Å)	13.62	18.66	10.17	18.26
b (Å)	10.65	16.40	11.13	10.00
c (Å)	8.89	15.55	9.15	23.78
β	133° 18'	—	92° 23'	—
V (Å ³)	983.0	4758.6	1035.2	4342.2
Z	2	8	2	8
M	391.49	488.43	472.41	519.41
D_x (g.cm ⁻³)	1.322	1.362	1.515	1.570
D_m (g.cm ⁻³)	1.32	1.355	1.505	1.56
Space group	$P2_1$	$P2_12_12$	$P2_1$	$C222_1$

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Least-squares structure refinement assuming molecular rigidity. By G. S. PAWLEY, *Department of Chemistry, Harvard University, Cambridge, Massachusetts, U.S.A.*

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Cruikshank (1956) has shown how to treat the results of structure refinement to get the rigid-body translational and rotational components of the thermal vibration. The method neglects the effect of internal vibrations and assumes that the temperature factors for all the atoms depend solely on the rigid-body motion. Following this assumption we can write the structure factor equation

$$\mathbf{F}_h = c \sum_{i \text{ atoms}} f_i \exp(2\pi i \mathbf{h} \cdot \mathbf{x}_i) \times \exp(-\mathbf{h} \cdot \mathbf{A}^{-1} \cdot \{\mathbf{T} + \mathbf{V}_i \cdot \boldsymbol{\omega} \cdot \tilde{\mathbf{V}}_i\} \cdot \tilde{\mathbf{A}}^{-1} \cdot \tilde{\mathbf{h}}) = \sum_i \mathcal{F}_i \quad (1)$$

where

$\mathbf{h} = (h, k, l)$, the reflection indices,
 $\mathbf{x}_i = (x_i, y_i, z_i)$, the crystal coordinates for the i th atom,