

shows comparable values of bond distances and angles. The N-C bond lengths are indeed equivalent and this confirms Larsen & Jerslev's statement that their observed differences are not significant. Also confirmed is the fact that the N(1)-C-O(2) and N(2)-C-O(2) angles are different by 10σ . The molecular planarity is also in agreement with the previous results.

References

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Crystallographic data for derivatives of retusamine. By J. A. WUNDERLICH, *Division of Applied Chemistry, C.S.I.R.O., Chemical Research Laboratories, Melbourne, Australia*

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The unit-cell dimensions and space groups of the hydrobromide, ethobromide, chromate, fluorotitanate, and monohydrate of retusamine ($C_{19}H_{25}O_7N$) are reported.

During the search for a crystalline derivative of the alkaloid retusamine [Fig. 1(a)] suitable for a complete X-ray analysis, a number were prepared and briefly investigated. The most suitable was the monohydrated α' -bromocamphor-*trans*- π -sulphonate, the crystal and molecular structure of which has been described (Wunderlich, 1962, 1966).

The cell dimensions were obtained from oscillation and Weissenberg photographs (Cu $K\alpha$ radiation). Densities were not measured owing to the small amount of material available.

Since the alkaloid is optically active and can be regenerated unchanged from its hydro-salts, it has been assumed that the space groups of the three monoclinic species described below possess neither a mirror plane nor a center of symmetry.

Retusamine hydrobromide

$(C_{19}H_{26}O_7N)^+Br^-$

F.W. 460.33

Monoclinic, very fine needles, $a = 22.33 \pm 0.06$, $b = 6.71 \pm 0.07$, $c = 15.51 \pm 0.06$ Å and $\beta = 103.8 \pm 0.3^\circ$, $U = 2256$ Å³,

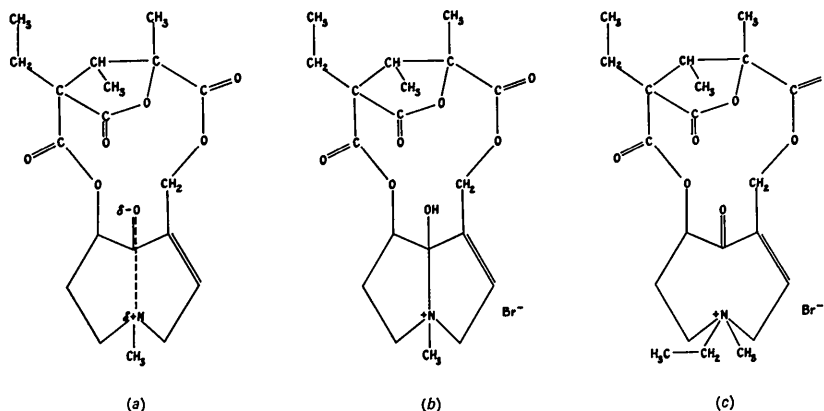


Fig. 1. (a) Retusamine. (b) Retusamine hydrobromide. (c) Retusamine ethobromide.

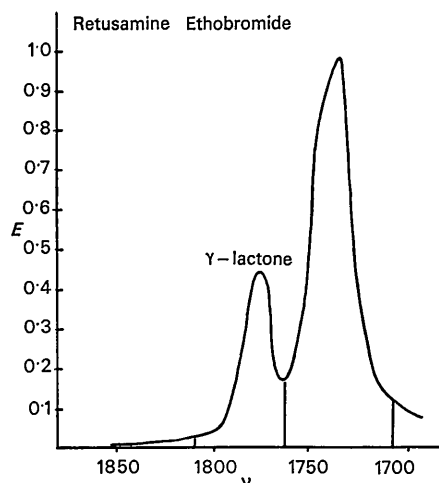


Fig. 2. Infrared absorbance (E) of retusamine ethobromide versus frequency (ν).

$Z=4$, $D_x=1.37$, $F(000)=952$. The space group is $P2_1$, and the very weak intensities of hkl reflexions with $h+k=2n+1$ indicate approximate C -centring.

The asymmetric unit consists of two independent retusamine hydrobromide molecules. The disappearance of the infrared spectral band at about 1610 cm^{-1} indicates that protonation takes place on the O atom at C(8) accompanied by transannular reaction between N(4) and C(8) [Fig. 1(b)].

Retusamine ethobromide

$(C_{21}H_{30}O_7N)^+Br^-$ F.W. 488.39
Orthorhombic, prismatic needles, $a=36.36\pm 0.04$, $b=6.61\pm 0.03$, $c=10.95\pm 0.06\text{ \AA}$, $U=2632\text{ \AA}^3$, $Z=4$, $D_x=1.23$, $F(000)=1016$.

Space group $P2_12_12_1$ from systematic absences.

When the infrared absorbance in the carbonyl region was plotted against frequency (Fig. 2), the areas under the curves at 1775 and 1735 cm^{-1} were in the ratio of 1:3. Since the former band is that due to the γ -lactone group, the latter results from the two ester carbonyl groups and that at C(8) [Fig. 1(c)] showing that ethylation has occurred on the nitrogen atom.

Retusamine chromate

$(C_{19}H_{26}O_7N)_2^+CrO_4^{2-}$ F.W. 876.84
Monoclinic, yellow needles, $a=21.62\pm 0.04$, $b=6.67\pm 0.01$, $c=15.43\pm 0.02\text{ \AA}$, $\beta=96.2\pm 0.1^\circ$, $U=2212\text{ \AA}^3$, $Z=2$, $D_x=1.32$, $F(000)=924$.
Space group $P2_1$; no systematic absences.

Retusamine fluorotitanate

$(C_{19}H_{26}O_7N)_2^+TiF_6^{2-}$ F.W. 922.74
Monoclinic, colourless needles, $a=21.20\pm 0.04$, $b=6.65\pm 0.03$, $c=15.50\pm 0.05\text{ \AA}$, $\beta=105.1\pm 0.1^\circ$, $U=2186\text{ \AA}^3$, $Z=2$, $D_x=1.40$, $F(000)=964$.
Space group $P2_1$; no systematic absences.

Retusamine monohydrate

$C_{19}H_{25}O_7N\cdot H_2O$ F.W. 397.43
Orthorhombic, efflorescent colourless needles, $a=41.7$, $b=6.7$, $c=8.8\text{ \AA}$, (estimated error of about 1% in each case), $U=2455\text{ \AA}^3$, $Z=4$, $D_x=1.08$, $F(000)=848$.
Space group $P2_12_12_1$, from systematic absences.

References

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Notes and News

Announcements and other items of crystallographic interest will be published under this heading at the discretion of the Editorial Board. The notes (in duplicate) should be sent to the General Secretary of the International Union of Crystallography (G. Boom, Department of Metallurgy, University of Oxford, Parks Road, Oxford, England. Publication of an item in a particular issue cannot be guaranteed unless the draft is received 8 weeks before the date of publication.

International Conference on Crystal Growth

July 1968

The second International Conference on Crystal Growth will be held at the University of Birmingham, England, from 15 to 19 July (inclusive), 1968. The Organizing Committee invite contributions on:

1. Basic theory of crystal growth, nucleation, kinetics and transport, and experiments related to basic theory.
2. Assessment of crystalline perfection as related to the growth process.
3. Studies of processes of fundamental importance to crystal growth of metals and non-metals including organic

materials, subdivided into the following types of growth process: (i) melt, (ii) vapour, (iii) flux, (iv) solution, (v) hydrothermal and high-pressure, (vi) recrystallization, and post crystal-growth phenomena, (vii) electrothermal deposition.

4. New techniques of crystal growth.
5. A cine film session.

Requests for further information or to be put on the mailing list should be addressed to Professor A. D. McQuilgan, Department of Physical Metallurgy and Materials Science, University of Birmingham, Birmingham 15, England. Details of the conference will be mailed to registrants in July 1967. Participants at the I.C.C.G. of 1966 in Boston are already on the mailing list.