STRUCTURES OF ORGANIC, ORGANOMETALLIC AND COORDINATION COMPOUNDS

\[
\begin{align*}
\beta &= 108.31(4)^{\circ}, \quad V = 2401(2) \text{ Å}^3, \quad \text{space group P2}_1/n, \\
D_0 &= 1.20 \text{ g cm}^{-3}, \quad \rho (Z = 4) = 1.21 \text{ g cm}^{-3}.
\end{align*}
\]

p = 108.31(4)°, u = 3

These thiazetidine rings are planar and more symmetrical than other published thiazetidine rings because both carbon atoms of the ring have external double bonds (C-S: 1.81-1.88 Å and C-N: 1.37-1.40 Å).

MONOPOTASSIUM SALTS OF 4-CYANO AND 4-NITRO-3-HYDOXY-1,2,6-THIADIAZINE 1,1-DIOXIDE MONOHYDRATE.

By C. Esteban-Calderón, M. Martínez-Ripoll and S. García-Blanco, X-Ray Department, Instituto "Rocasolano", Serrano 119, Madrid-6, Spain.

Both compounds have been synthesized by Goya and Stud (J. Heterocyclic Chem. 1978, 15, 253-256), as a part of a research project aimed at obtaining substances with biological activity.

Crystal data are:

**4CN-K(C_4 H_2 N_3 O_3 sK.H_2 O):**
- a = 9.881 (1)
- b = 7.296 (1)
- c = 5.858 (1) Å
- \(\alpha = 103.06(1)^{\circ}\)
- \(\beta = 94.56(2)^{\circ}\)
- \(\gamma = 100.51(2)^{\circ}\)
- Z = 2; P2_1/n; n° reflex. in refinement 2835.

**4N02-K(C_3 H_2 N_3 O_5 sK.H_2 0):**
- a = 6.0744 (4)
- b = 18.583 (2) Å
- c = 7.497 (1) Å
- \(\alpha = 91.68(1)^{\circ}\)
- \(\beta = 100.51(2)^{\circ}\)
- \(\gamma = 100.51(2)^{\circ}\)
- Z = 4; P2_1/n; n° reflex. in refinement 2006.

Both structures have been solved by using single crystal data and MoKa radiation, and refined by least-squares analysis to the values of 0.042 (R_w=0.049) and 0.039 (R_w=0.050), respectively.

Major electron distributions are proposed (Fig.3 and 4), according to Linnett's model (Linnett, J.W. (1966). The Electron Structure of Molecules. A New Approach. London, Methuen).

CONFORMATIONAL AND STRUCTURAL RELATIONSHIPS BETWEEN TEN DITERPENOIDS FROM TEUCRUM SPECIES.

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The chemistry of diterpenoids occurring in the genus Teucrium, is described in a recent general review (Piozzl, Heterocycles. 1981 in press). The structures of ten of these compounds have been determined by X-Ray diffraction.

The purpose of this study is to classify these compounds according to the conformation of their rings. Special attention has been devoted to the decaline group. Parameters definition: for this study Cremer's parameters have been used (Cremer and Pople. J.Am.Chem.Soc. (1975) 97(6), 1354-58), \(\theta\) (\(^{\circ}\)), \(\phi\) (\(^{\circ}\)) and \(\psi\) (\(^{\circ}\)). Chairs occur for \(\theta=0,180\), and boat or twist for \(\theta=90\). \(\phi\) describes the boat form \(\phi=0,60,120\ldots\) and twist form \(\phi=30,90,150\ldots\), and \(\psi\) the puckering. The table shows the experimental X-Ray values for the decaline moieties. \(\psi\) is the internal torsion C1-C10-C5-C4 and \(\theta\) is C6-C5-C10-C9. It is observed that \(\psi\) is nearly constant: 108° for sp^3-sp^3 bonds and 93° for sp^2-sp^3 bonds. The 108° value is diminished by the C5-C9 axial interaction and the oxo substituted C6 in compounds I, IV and VI. For saturated cyclohexane rings \(\psi\) increases with \(\psi\) to reach a maximum at \(\psi=90\) and decreases again towards \(\phi=180\).
although for chairs \( Q_1 \) remains almost constant.

\[
\begin{align*}
\text{Table} & \quad A & \quad A & \quad A & \quad A & \quad A & \quad A & \quad A \\
1 & 222 & 0.60 & -57.8 & 102.9 & 45.1 & 0.50 & 233 & 6 \\
2 & 7 & 350 & 0.57 & -59.7 & 107.2 & 32.9 & 0.80 & 32 & 50 \\
3 & 9 & 251 & 0.61 & -62.5 & 105.2 & 16.9 & 0.76 & 71 & 78 \\
4 & 10 & 291 & 0.62 & -65.9 & 103.3 & 57.3 & 0.74 & 72 & 106 \\
5 & 18 & 309 & 0.59 & -64.2 & 114.2 & 50.2 & 0.51 & 1 & 4 \\
6 & 18 & 259 & 0.62 & -68.8 & 102.2 & 32.5 & 0.57 & 293 & 110 \\
7 & 31 & 280 & 0.57 & -71.5 & 107.4 & 25.5 & 0.54 & 38 & 94 \\
8 & 51 & 304 & 0.65 & -102.0 & 97.8 & 0.81 & 32 & 92 \\
9 & 125 & 229 & 0.51 & 1.1 & 49.3 & -48.2 & 0.57 & 118 & 186 \\
10 & 123 & 225 & 0.50 & -0.6 & 50.4 & -49.5 & 0.57 & 128 & 169 \\
\end{align*}
\]

- Compounds with ring A being cyclohexane. Ring A takes a chair conformation only disturbed by the ring B. Ring B is in a chair conformation when no substituent is present in C7 and boat otherwise. In 11, ring B is in a twist conformation due to the influence of the twist gynanoand ring. In VI, due to the lactonic bridge, ring B is a chair slightly distorted to boat.

- Compounds with ring A being cyclohexane. Rings A are envelopes with the flap at the atoms as predicted by Bucourt and Hainaut. Bull. Soc. Chim. Fr. (1975) 2, 1366-78 for cyclohexenes. In VII, ring B is forced to boat by the \( 0.8 \)-unsaturated \( Y \)-lactone ring; there is some deformation to twist induced by the \( C4 \)-alcohol substituent. The envelope A has the flap at C7. A chair conformation is completed in VIII; ring B is by the interaction between \( C4 \)-CS substituents, there is also an \( \alpha \)-H-bond \( 03\text{H} \cdot \cdot \cdot 03\text{O} \); the \( \tau \) angle induced for \( \tau \) produces envelope A with the flap at C7. The compounds IX and X with \( C4 \) H10 have rings B with no 1,3 axial interaction, being in a chair conformation; the high value of \( \tau \) induces \( \tau \) to be zero defining the envelope A with the flap at C2.

- Five-membered rings. \( Y \)-lactone rings, are envelopes with \( \tau \) 0 in the lactonic group, except for VII. The \( 0.8 \)-unsaturated \( Y \)-lactone rings are almost planar.

**9.3-01** STRUCTURAL INVESTIGATIONS OF LIQUID CLATHRATED PARENT COMPLEXES: THE CRYSTAL AND MOLECULAR STRUCTURES OF \( K_3[Al_3Me_2(SiO_3)] \), \( K_3[Al_3Me_2(SO_3)] \) - 0.5 \( \text{Me}_2\text{C}_6\text{H}_5 \), and \( K_3[Al_6O_6\text{C}_6\text{H}_5] \) \( \text{C}_6\text{H}_6 \). R. D. Rogers, R. C. H. Hainaut, and J. L. Atwood, Department of Chemistry, University of Alabama, University, Alabama, USA 35486.

The interaction of tri-alkyl aluminum with metal halides and pseudo halides to form complexes of the type \( M(\text{Al}X)_n \) has been of interest for quite some time. Our primary goal has been to find those complexes which trap aromatic molecules in either the liquid or solid state. Expansion of the metal salts capable of forming liquid clathrates has led away from compounds restricted to the general formulation above. We have recently completed two structural studies on \( K_3[Al_3Me_3(SO_3)] \). One was crystallized from benzene and contained no solvent molecules, while the compound taken from para-xylene resulted in 0.6 mole of \( \pi \)-xylene of crystallization. \( K_3[Al_3Me_3(SO_3)] \) crystallizes in the monoclinic space group \( P2_1/c \) with \( a = 10.23(4), b = 20.235(5), c = 14.039(4), \beta = 112.20(2)^\circ \), \( D_0 = 1.14 \text{ g cm}^{-2} \). This compound was refined to a final R value of 0.095 for 1576 independent observed reflections. \( K_3[Al_3Me_3(SO_3)] \) - 0.5 \( \text{Me}_2\text{C}_6\text{H}_5 \) was also monoclinic, \( P2_1/c \) with \( a = 9.773(5), b = 15.497(4), c = 20.442(4), \beta = 92.31(2)^\circ \), \( D_0 = 1.11 \text{ g cm}^{-2} \). Refined to a final R value of 0.085 for 2600 reflections.

The third compound, \( K_3[Al_6O_6\text{C}_6\text{H}_5] \) was prepared by the reaction of \( K_3[Al_3Me_3SO_3] \) with trimethylaluminum, and crystallizes in the triclinic space group \( P1 \) with \( a = 12.095(4), b = 10.322(4), c = 13.095(4), \alpha = 120.46(3), \beta = 94.52(2), \gamma = 81.99(2), D_0 = 1.08 \text{ g cm}^{-2} \). Refined to a final R value of 0.042 for 2509 observed reflections. Comparisons between structural characteristics and liquid clathrate behavior will be discussed.