

## 06-Crystallography of Organic Compounds

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(Maurin, Winnicka-Maurin, Paul, Curtin, 1992).

The two title compounds (Figure 1 and 2, respectively), unlike any other mentioned before, form cyclic tetrameric units in crystalline state. The molecules are bonded together by pairs of hydrogen bonds between oxime and carboxyl groups. Seemingly similar

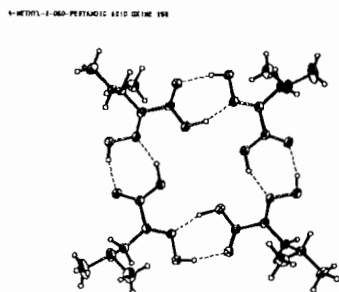


Figure 1

LOWA-1111 ACID OXIME

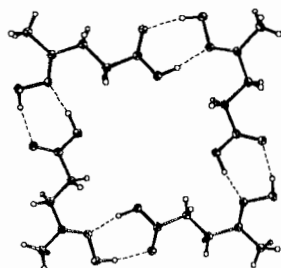


Figure 2

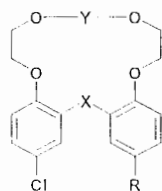
structures differ significantly both in molecular geometries and hydrogen bond dimensions. The dramatic changes in C=N and N-O bond lengths as well as in O...O and O...N hydrogen bond lengths in 4-methyl-2-oxopentanoic acid oxime comparing to levulinic acid oxime and other compounds could be interpreted as the result of resonance interaction between oxime and carboxyl group in the first compound. This reasoning is strongly supported by quantum mechanic calculations done for both title compounds.

## References.

- Maurin, J.K., Paul, I.C. & Curtin, D.Y. (1992a). *Acta Cryst.*, **C48**, 0000-0000.  
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 Padmanabhan, K., Paul, I.C. & Curtin, D.Y. (1989). *Acta Cryst.*, **B45**, 411-416.

**PS-06.03.05 X-RAY STUDY OF DIPHENYLAMINE CONTAINING CROWNS.** By Zs. Böcskei<sup>1</sup>, V. Németh<sup>2</sup>, B. Ágai<sup>2</sup>, K. Simon<sup>1</sup>, <sup>1</sup>Chinoin Pharmaceutical and Chemical Works Ltd, Budapest, POB 110, H-1325, Hungary, <sup>2</sup>Department of Organic Chemical Technology, Technical University Budapest, H-1521, Hungary.

Three crowns, **1** (R=H, X=NH, Y=CH<sub>2</sub>-CH<sub>2</sub>), **2** (R=H, X=NH, Y=CH<sub>2</sub>-CH<sub>2</sub>-O-CH<sub>2</sub>-CH<sub>2</sub>), **3** (R=Cl, X=S, Y=CH<sub>2</sub>-CH<sub>2</sub>-O-CH<sub>2</sub>-CH<sub>2</sub>) have been analyzed by X-ray crystallography. A water molecule is situated in the middle of crown **2** H-bonded to the N-H group and two oxygen atoms. The conformation of the three macrocycles will be compared.



Compound	1	2	3
Formula	C <sub>18</sub> H <sub>20</sub> ClNO <sub>4</sub>	C <sub>20</sub> H <sub>24</sub> ClNO <sub>5</sub> ·H <sub>2</sub> O	C <sub>20</sub> H <sub>22</sub> Cl <sub>2</sub> O <sub>5</sub>
a, Å	11.147	12.463	12.741
b, Å	12.356	12.619	13.516
c, Å	13.743	13.422	12.992
β, °	107.81	102.86	98.10
V, Å <sup>3</sup>	1802	2058	2215
Z	4	4	4
S.G.	P2 <sub>1</sub> /c	P2 <sub>1</sub> /c	P2 <sub>1</sub>
N <sub>refl</sub>	2753	1668	1736
R	0.08	0.10	in progress

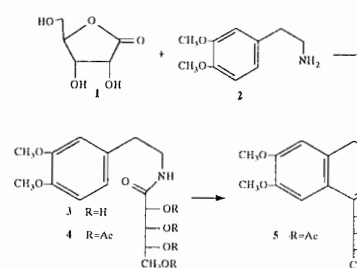
**PS-06.03.06 ENANTIOSELECTIVE SYNTHESIS OF ISOQUINOLINE ALKALOIDS FROM SIMPLE SUGAR.** by Z. Czarnocki<sup>1</sup>, *Department of Chemistry, Warsaw University, Pasteura 1, 02-093 Warsaw, Poland*, J.K. Maurin and K. Wieteska, *Institute of Atomic Energy, Solid State Physics Department, 05-400 Otwock-Świerk, Poland*.

In the course of our study on enantioselective synthesis of isoquinoline alkaloids from various naturally occurring chiral substrates, we launched a project of using a simple sugar - D-ribose as a chiral building block. The synthetic sequence started with a condensation of **1** with 3,4-(dimethoxyphenyl)ethylamine **2** to give an amide **3**, which after acetylation to **4** and Bischler-Napieralski cyclization gave a very unstable imine **5** (Czarnocki 1992).

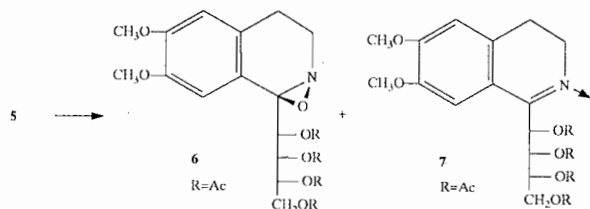
When **5** was subjected to m-chloroperbenzoic acid (MCPBA) oxidation, compounds **6** and **7** were formed in a relative ratio depending on the temperature (Czarnocki, 1992).

The synthetic utility of nitron **7** was further elucidated by its transformation, in several steps, into two isoquinoline alkaloids

Scheme 1



Scheme 2



(R)-calycotomine, and (S)-xylopinine. Both compounds, **6** and **7**, were characterized by spectral means (<sup>1</sup>H and <sup>13</sup>C-NMR) as well as by X-ray structural analysis.