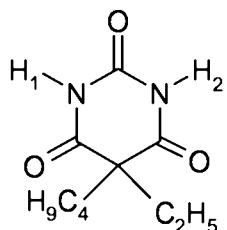


[s9.m1.p11] Solid State Investigation of the Polymorphic System of Butethal- 5-ethyl-5-N-Butyl Barbituric Acid. O. Navon¹, J. Bernstein¹, ¹*Department of Chemistry, Ben-Gurion University of the Negev, Beer Sheva 84105 Israel.*
Keywords: molecular interactions, hydrogen bond.

The polymorphic system of Butethal is composed four different forms.

The system is part of a family of the Barbiturates, which compose a class of drugs which exhibit widespread polymorphism. In an early survey it was found that of the barbiturates used medicinally about 70% were polymorphic¹. In the course of investigating a number of compounds in this family we studied on the molecule 5-ethyl-5-n-butylbarbituric acid.



We will present the solid state characterization of the different forms, utilizing a variety of techniques, including: single crystal X-ray structure analysis, X-ray powder diffraction, solid state NMR thermal analysis, graph set analysis, and lattice energy calculations, in an effort to understand and to control the structure and polymorphic form.

[s9.m1.p12] Synthesis and crystal structure of two acidic selenates: (C₆H₁₄N₂)(HSeO₄)₂ and (C₆H₁₆N₂)(HSeO₄)₂. M.A. Zakharov^{*}, S.I. Troyanov^{*}, L.A. Aslanov^{*}, E. Kemnitz^{**}. ^{*}*Moscow State University, Chemistry Department*, ^{**}*Institut für Chemie, Humboldt Universität Berlin.*

Keywords: molecular interactions, hydrogen bond.

Two new acid selenates have been synthesized: (C₆H₁₄N₂)(HSeO₄)₂ (**I**) and (C₆H₁₆N₂)(HSeO₄)₂ (**II**), where C₆H₁₄N₂ is 1,4-diazabicyclo-[2.2.2]octaneH₂ (DABCOH₂) and C₆H₁₆N₂ is N,N'-dimethylpiperazineH₂ (DMPPAH₂). Crystals of **I** and **II** were grown by slow evaporation of aqueous solutions with stoichiometric ratio of corresponding amine and selenic acid.

I and **II** were investigated by X-ray single crystal diffraction on an IPDS (Stoe) and a STADI-4 (Stoe), respectively. Unit cell parameters are a=7.447(2), b=17.755(3), c=10.269(3)Å, β=111.20(3)°, V=1266.0(6)Å³, Z=4, sp.gr. P2₁/n for **I** and a=6.78(1), b=12.20(3), c=8.230(4)Å, β=108.1(3)°, V=646.9(4)Å³, Z=2, sp.gr. P2₁/c for **II**.

Asymmetric unit of **I** contains one cation [DABCOH₂]²⁺ and two kinds of anions [HSeO₄]⁻. One of anions was found to be disordered around Se-O bond. Both kinds of tetrahedra are linked by two hydrogen O...O bonds 2.57(1) and 2.60(1)Å forming infinite chains along *a*. DABCOonium cations connect these chains along [201] by two hydrogen N...O bonds 2.652(8) and 2.705(8)Å to layers parallel to *xOz* plane. There are van-der-Waals interactions between layers.

In the structure **II** there are dimethylpiperazonium cations in chair conformation and tetrahedral HSeO₄ anions. These tetrahedra form infinite chains along *c*-axis by means of hydrogen O...O bonds 2.61(1)Å. The cations of dimethylpiperazonium join these chains by hydrogen N...O bonds 2.78(1)Å. Thus packed layers are formed parallel to *Oyz* plane. The van-der-Waals interactions are present between layers.

The infinite chains of HSeO₄ tetrahedra are the typical case of hydrogen bonding system for acid selenates. This case is compared to the cyclic hydrogen bonding systems, for instance closed dimers.

[1] Kuhnert-Brandstatter, M., "Thermomicroscopy in the Analysis of Pharmaceuticals., International Series of Monographs in Analytical Chemistry, Pergamon Press, New York, vol 45, 1972.