

**Keywords: anilinium, hydrogensulfate and hydrogen-bond**

#### FA4-MS26-P10

**16 independent molecules for an *ortho*-palladated primary amine** Beatrice Braun (Calmuschi-Cula)<sup>a,b</sup>, Ulli Englert<sup>a</sup>, <sup>a</sup>*Institute of Inorganic Chemistry, RWTH Aachen University*, <sup>b</sup>*Institute of Chemistry, Humboldt University Berlin*

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During our efforts to build well-ordered binary crystals based on *ortho*-palladated primary amines [1-2], we encountered a rare and interesting example of  $Z' = 16$ . The complex derived from 4-methoxy-phenylethylamine crystallizes in the non-centrosymmetric space group *Pc*; the other compounds in this class are unspectacular with respect to the number of independent molecules. Only four other structures with  $Z'$  larger or equal 16 exist up to date in the CSD Database [3-7]. According to Kitaigorodskii's close packing principle [8], molecules arrange in the solid state in such a way to maximize density and minimize free volume. Void space in crystals is always unfavourable [9]. Structures with large  $Z'$  values are associated with packing conflicts [10].

In the present case, the elevated number of molecules in the asymmetric unit can be attributed to the -OMe substituent which corresponds to a soft conformational degree of freedom.

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**Keywords: chemistry organometallic, crystal packing, conformational flexibility**

#### FA4-MS26-P11

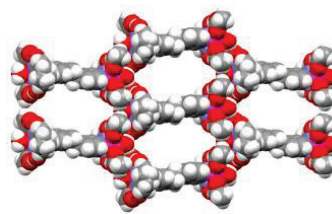
**Multi-Component Crystals of DihydroErgocornine.**

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Polymorphism and solvatomorphism are phenomena frequently observed in pharmaceutically active substances. The knowledge of potential crystalline forms is important particularly from the point of stability, process and final dosage form development.

Systematic research in the field of ergot alkaloids has shown, that structurally related compounds sometimes form nearly isostructural types of packing. Same structure type is shared with one or more solvent molecules, but also a mix of solvents



occupying the identical structure cavities. Number of members which fit to one structure type continually increases. In most cases a structure is quite unstable due to the solvent molecules

leaking out of its sites. According to thermal analysis, the solvent molecules tend to abandon structure in separated steps depending on time or temperature. A question arises, to what extent of partial desolvation the original structure is preserved. Is it possible to eliminate one of two molecules in the cavity? Will a single crystal survive partial desolvation? Is it possible to localize the remaining molecule(s)?

This work deals with solvatomorphism of semisynthetic ergot alkaloid dihydroergocornine mesylate, which forms interesting series of solvatomorphs. Single crystal X-ray structure data of dihydroergocornine mesylate solvated forms will be presented, sorted into structure types showing relationship among them. The desolvation process of selected phases will be described using combined TG-DTA-MS method. The new desolvated phases will be monitored with X-ray powder diffraction analysis. Hopefully a mixed solvent phase will be presented as well.

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**Keywords: thermal methods, X-ray structure of ergot alkaloid, solvatomorph**

#### FA4-MS26-P12

**Crystal Structure of New Binuclear Molecule**

**[Cd<sub>2</sub>(NioxH)<sub>2</sub>(bpetha)(CH<sub>3</sub>COO)<sub>4</sub>(H<sub>2</sub>O)<sub>2</sub>]** Lilia Croitor,<sup>a</sup> Eduard B. Coropceanu,<sup>b</sup> Mark M.

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We have further developed the strategy of 'metal dioxime building block' for construction of new generation of mixed-ligand coordination compounds with useful properties [1]. The ability of the oxime donors to form intramolecular hydrogen bonds with coordinated anion can be useful for stabilization of reactive intermediates or for enhancement of reactivity. Interaction in the system Cd(CH<sub>3</sub>COO)<sub>2</sub>+NioxH<sub>2</sub>+bpetha in the molar ratio 1:2:2 resulted in binuclear molecule of the composition [Cd<sub>2</sub>(NioxH)<sub>2</sub>(bpetha)(CH<sub>3</sub>COO)<sub>4</sub>(H<sub>2</sub>O)<sub>2</sub>] (1), and the dioxime-free 1D coordination polymer with the monomeric unit [Cd(bpetha)(CH<sub>3</sub>COO)<sub>2</sub>(H<sub>2</sub>O)] (2), where NioxH<sub>2</sub>=1,2-cyclohexanedionedioxime, bpetha=1,2-bis(4-pyridyl)ethane. Compound 1 crystallizes in monoclinic space group *P2<sub>1</sub>/c* with  $a = 8.773(2)$ ,  $b = 11.156(2)$ ,  $c = 21.878(4)$  Å,  $\beta = 105.41(3)^\circ$  and  $Z = 2$ .