

MS28-P4 Crystal structure and DFT calculations of 2-naphthalenecarboxamide, 3-hydroxy-N(2-methylphenyl)

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The structure of title compound 2-Naphthalenecarboxamide, 3-Hydroxy-N(2-methylphenyl) $C_{18}H_{15}NO_2$, has been synthesized. The Schiff base compound has been characterized by single-crystal X-ray diffraction technique. The compound crystallizes in the monoclinic space group P21/c with the following unit-cell dimensions $a=15.1334(13)\text{Å}$, $b=7.0994(4)\text{Å}$, $c=12.7712(12)\text{Å}$, $\alpha=90.000(0)^\circ$, $\beta=97.221(7)^\circ$, $\gamma=90.000(0)^\circ$, $V=1361.23(19)\text{Å}^3$, $Z=4$. The crystal structure contains intramolecular N-H...N hydrogen bond. X-ray measurements was carried out on a STOE IPDS II diffractometer with MoK α radiation. The molecular structure was solved by direct method using SHELXS97 and refinement by full-matrix least-squares on F^2 using SHELXL97 program. An Ortep-3 view of the molecule of title compound is shown in Fig. 1.

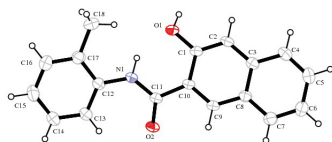


Figure 1. Ortep-3 diagram of the title compound

Keywords: Schiff base, tautomeric effect, DFT Calculations

MS28-P5 Free energy determination applied for gallic acid monohydrate polymorphs

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The contribution of both entropy and enthalpy in crystal structure relative stability determination play an important role in understanding the formation and behaviour of a specific polymorph. Investigating the effects of vibrational entropy is a key factor in achieving a better understanding the relative stability of polymorphic materials. Due to low-frequency molecular vibrations, it is rather difficult to estimate vibrational entropy accurately. Therefore, the thermodynamic contribution should be extracted by combining the calculated vibration entropy with temperature-controlled crystallographic measurements. The kinetic contribution will be as well estimated by combining the calculated vibrational energies with crystallographic measurements. Gallic acid monohydrate has been shown to crystallise into five different polymorphs and it was rather difficult to obtain meaningful crystal structure prediction.¹ Hence, we considered is a good candidate for free energy investigation. Single crystal X-ray diffraction experiments were carried out at four different temperatures (10 K, 95 K, 123 K, and 175K) at a relatively high diffraction angle. The data obtained was then used in an aspherical atom refinement implemented in XD software² in order to obtain accurate atomic and anisotropic displacement parameters (adps). In a subsequent step the aspherical atom refinement data together with periodic ab-initio calculations were used in a normal mode refinement for gaining the vibrational entropy. The entropy was calculated using the periodic ab-initio calculations for each form. The 10K data collection for form III reveal a change in the unit cell parameters with $Z'=3$ for the asymmetric unit. The volume is three times expanded and the molecules in the asymmetric unit show *pseudo* symmetry. Therefore, the structure can be defined from only one molecule with *pseudo*- $Z'=1$ as in case of the room temperature data of this form. Some of the H atoms involved in the (8) ring were found to show disorder. This is a possible explanation of the lack in H atom positions determination in the previous crystal structure prediction of gallic acid monohydrate polymorphs.¹

Reference:

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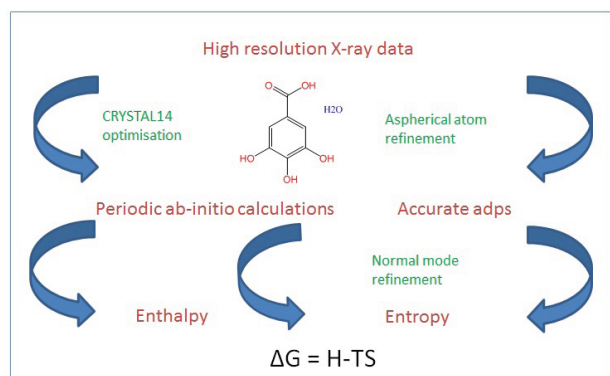


Figure 1.

Keywords: high resolution X-ray diffraction, periodic ab-initio calculations, gallic acid

MS28-P6 Experimental electron density of cytosinium chloride in crystalline state

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Comprehensive goal of my work is to analyze charge density distribution and intermolecular interactions between nucleobases in crystalline state. It is crucial to know precisely potential of this molecules to form particular type of interactions, especially in the context of RNA structure predictions. Presented work is focused on cytosinium chloride.

Single crystals of cytosinium chloride were obtained by slow evaporation of solvent from mixture of cytosine and 4-thiouracil dissolved in water with small amount of hydrochloric acid. High resolution (0.5 Å) X-ray diffraction data were collected on monocrystal diffractometer at 90 K. The data were next subject to data reduction, structure solution, independent atom model refinement and finally multipolar refinement procedures. Several strategies of data reduction and multipolar refinement were tested to obtain the best model of crystal electron density.

Cytosine chloride crystallizes in $P2_1/n$ group in monoclinic system. Unit cell consists of one protonated cytosine molecule and one chloride ion located almost in the same plane. Cytosine forms dimers through double hydrogen bond between O2 and N3 atoms (see Figure 1). Hydrogen bonds, besides cation – anion interactions, play important role in building the crystal structure and determination of crystal lattice energy.

I will present comparison of chosen data reduction and refinement strategy with others and discuss which parameters decided on its superiority. The second part will be dedicated to more profound description of cytosine chloride structure and evaluation of intermolecular interactions based on charge density.