MS15-P7 Synthesis, molecular structure and spectroscopic characterization of N-(4-nitrophenyl)-2, 2-dibenzoylacetamide (NPDBA): with experimental (X-Ray, FT-IR, ^{1}H and ^{13}C-NMR and UV-*Vis*) techniques and theoretical calculations

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The title compound, $C_{22}H_{16}N_2O_5$, was synthesized and characterized by experimental techniques (FT-IR, ^{1}H-NMR, ^{13}C-NMR, UV-*Vis* and X-Ray single crystal determination) and theoretical calculations. According to X-Ray diffraction results, the title compound crystallizes in the monoclinic space group $P12_1/c1$ with a = 10.023 (2) Å, b = 21.587 (5) Å, c = 9.401 (2) Å and β = 110.29 (3)°, and Z = 4 in the unit cell. The molecular geometry, vibrational frequencies, molecular electrostatic potential (MEP), thermodynamic properties, the dipole moments, HOMO-LUMO energy has been calculated by using the Density Functional Theory (DFT) method with 6-311G(d,p) and 6-311++G(d,p) basis sets. ^{1}H and ^{13}C-NMR chemical shifts show good agreement with experimental values.

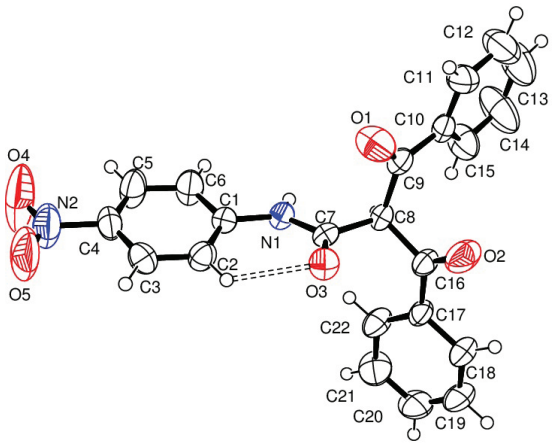

Figure 1. The molecular structure of the title compound.

Keywords: X-ray diffraction; Density functional theory; Quantum chemical calculations; Carboxamide; Characterization.

MS15-P8 Unusual thermal polymorphic transformation *I*-43*d*↔ $P2_1/a$ ↔ *Ia*-3*d* of $KBSi_2O_6$

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Up to now three topologically identical modifications of $KBSi_2O_6$ with the 3D tetrahedral framework of the ANA type [Zeolite DATABASE] are known: cubic *I*-43*d* [Ihara, Kamei 1980; Miklos et al 1992], cubic *Ia*-3*d* [Martucci et al 2011] and monoclinic $P2_1/a$ [Belokoneva et al 2010]. In present study the polycrystalline sample of cubic $KBSi_2O_6$ was obtained by solid-state reaction from stoichiometric mixture. The monoclinic modification of $KBSi_2O_6$ ($P2_1/a$) was prepared by hydrothermal synthesis at 600 °C and 5 kBar. The thermal behavior of both modifications upon heating in air was studied by high-temperature X-ray powder diffractometry (HTXRD) and differential scanning calorimetry (DSC) in the temperature range 25–1100 °C. In accord to both HTXRD and DSC results the cubic modification undergoes reversible thermal transformations: *I*-43*d*↔ $P2_1/a$ ↔ *Ia*-3*d*. The temperature dependence looks complicated. The jumps of values of cell parameters are registered near the point of both *I*-43*d* ↔ $P2_1/a$ and $P2_1/a$ ↔ *Ia*-3*d* transformations. The volume thermal expansion coefficients are about 70, 50 and 30×10^{-6} °C^{-1} for *I*-43*d*, $P2_1/a$ and *Ia*-3*d* phases, respectively. The HTXRD data on the transition temperatures are in a good agreement with DSC data both on heating and cooling. Taking into account well known tendency of substances to increase their symmetry on heating, polymorphic transformation cubic-monoclinic-cubic looks unusual. $P2_1/a$ hydrothermal phase transforms reversibly into *Ia*-3*d* polymorph. Both modifications decompose above 1000 °C with SiO_2 formation. In [Martucci et al 2011] the direct reversible transformation *I*-43*d* □ *Ia*-3*d* of slightly hydrated $KBSi_2O_6$ has been studied by Rietveld refinement from synchrotron data. Our experiment showed that the addition of Na or Rb to $KBSi_2O_6$ stabilized the direct transformation I-43d ↔ *Ia*-3*d* as well. In [Millini et al 1993] non-stoichiometrical $KBSi_2O_6$ enriched in SiO_2 was obtained by hydrothermal synthesis with *Ia-3d* symmetry. It seems that even insignificant variations in composition could lead to stabilization of different modifications of boroleucite structure.

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Keywords: borosilicate, leucite, high-temperature transformation