MS15-P7 Synthesis, molecular structure and spectroscopic characterization of N-(4-nitrophenyl)-2, 2-dibenzoylacetamide (NPDA): 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$_{16}$H$_{11}$N$_3$O$_5$ 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 P1$_2$/c with a = 10.023 (2) Å, b = 21.587 (5) Å, c = 9.401 (2) Å and $\beta$ = 110.29 (3)$^\circ$, 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-43d$\leftrightarrow$ P2$_1$/a $\leftrightarrow$ Ia-3d of KBSi$_2$O$_6$

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Up to now three topologically identical modifications of KBSi$_2$O$_6$ with the 3D tetrahedral framework of the ANA type [Zeolite DATABASE] are known: cubic I-43d [Ihara, Kamei 1980; Mikkol et al 1992], cubic Ia-3d [Martucci et al 2011] and monoclinic P2$_1$/a [Belokoneva et al 2010]. In present study the polycrystalline sample of cubic KBSi$_2$O$_6$ was obtained by solid-state reaction from stoichiometric mixture. The monoclinic modification of KBSi$_2$O$_6$ (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-43d$\leftrightarrow$ P2$_1$/a $\leftrightarrow$ Ia-3d. The temperature dependence looks complicated. The jumps of values of cell parameters are registered near the point of both I-43d$\leftrightarrow$ P2$_1$/a and P2$_1$/a $\leftrightarrow$ Ia-3d transformations. The volume thermal expansion coefficients are about 70, 50 and 30×10$^{-6}$°C$^{-1}$ for I-43d, P2$_1$/a and Ia-3d 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-3d polymorph. Both modifications decompose above 1000 °C with SiO$_2$ formation. In [Martucci et al 2011] the direct reversible transformation I-43d$\leftrightarrow$Ia-3d of slightly hydrated KBSi$_2$O$_6$ has been studied by Rietveld refinement from synchrotron data. Our experiment showed that the addition of Na or Rb to KBSi$_2$O$_6$ stabilized the direct transformation I-43d$\leftrightarrow$Ia-3d as well. In [Millini et al 1993] non-stoichiometrical KBSi$_2$O$_6$ 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.