MS28-P2 **Integrated XRD/SONICC/TPE-UVF for identifying and centering of protein crystals.** Christopher Dettmar,[a] Scott Toth,[a] Michael Becker,[b] Robert Fischetti,[b] Garth Simpson,[a] [a]*Department of Chemistry, Purdue University, West Lafayette, IN 47907,* [b]*GM/CA@APS, X-ray Science Division, Argonne National Laboratory, Argonne, IL 60439*
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An instrument is under development for integrating second order nonlinear imaging of chiral crystals (SONICC)[1] and two-photon excited ultraviolet fluorescence imaging (TPE-UVF),[2] previously developed in the Simpson Lab, with a synchrotron X-ray diffraction (XRD) beam line, targeting rapid screening for identification and centering of protein crystals. In SONICC, two photons of infrared light combine to form one photon of visible light at twice the frequency. The symmetry requirements of this process make it sensitive to non-centrosymmetric crystalline order, including the large majority (~90%) of protein crystals and excluding most common salts, cryo-protectants, solvents, and aggregated protein.[3] In TPE-UVF, the simultaneous absorption of two photons of visible light, the energy equivalent of one photon of UV light, results in emission of a photon in the near UV to blue range. The wavelengths used are sensitive to aromatic amino acid residues, particularly tryptophan, making ~80% of proteins detectable through a mechanism unique and complementary to SONICC. These imaging methods are sensitive to micron-sized crystals, including those grown in the lipidic mesophase that are otherwise difficult to visualize.[4] Positioning by nonlinear optical imaging may reduce or eliminate the need for large area X-ray raster scanning thereby reducing X-ray induced radiation damage and increasing throughput of synchrotron x-ray diffraction. Integration with the synchrotron beam line allows a sample to be mounted on the goniometer, imaged, crystal centered and ready for diffraction in just a few minutes.

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MS28-P3 **The Nd-Gd chromium borates solid solutions: structures and phase relations.** Elena Dobretsova[a], Kirill Boldyrev[b], Elena Borovikova[a], [a]*Lomonosov's Moscow State University, Russia,* [b]*Institute of Spectroscopy RAS, Russia*
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The solid solutions of rare-earth chromium borates $Nd_xGd_{1-x}Cr_3(BO_3)_4$ have been synthesized by the flux method. Such borates crystallize in space groups *R*32 and *C*2/*c* and have the polytypic nature. The non-centrosymmetrical rhombohedral modification is the most perspective as the functional material with the nonlinear optic properties. In this study the solid solutions $GdCr_3(BO_3)_4$ (*R*32) - $NdCr_3(BO_3)_4$ (*C*2/*c*) were investigated by the Rietveld method and by methods of infrared and optical spectroscopy.

Pure $GdCr_3(BO_3)_4$, $NdCr_3(BO_3)_4$ and three samples of intermediate compositions were studied by the Rietveld method. Even the extreme compositions contain a significant portion of the opposite phase. A rhombohedral phase in pure gadolinium chromium borate is about 85%. A monoclinic phase in pure neodymium chromium borate is about 62%. The phase ratio varies slightly (R32 from 56 to 47%) at x between 0.5 and 0.9.

Infrared (IR) spectra of solid solutions $Nd_xGd_{1-x}Cr_3(BO_3)_4$, where x = 0 – 0.6, mainly keep the characteristic features of rhombohedral phase spectra and vary slightly. There are four bands of stretching vibrations in the region of 1260 - 1100 cm^{-1} in the spectrum of pure $GdCr_3(BO_3)_4$ (*R*32). The spectra change significantly at x = 0.7 – 0.8. Typical for monoclinic borate spectral features, such as the band at ~ 1280 cm^{-1}, the splitting of the band at ~ 1200 cm^{-1}, and the shoulder at ~ 1240 cm^{-1}, are observed. The IR spectrum of $Nd_{0.9}Gd_{0.1}Cr_3(BO_3)_4$ is characteristic for monoclinic borate. Eight bands of asymmetric stretching vibrations of BO_3^{3-} ions appear in the region of 1360 - 1100 cm^{-1}, and 4 bands of symmetric stretching vibrations of BO_3^{3-} ions in the region of 1040 - 914 cm^{-1} are present.

The presence of two phases (monoclinic and rhombohedral) is also confirmed by optical spectroscopy. The low-temperature optical absorption spectra corresponding to the transition $^4I_{9/2} \rightarrow {}^4F_{3/2}$ in the Nd^{3+} ion are studied. The low frequency spectral line of two predicted ones shifts to lower frequencies when changing from gadolinium to neodymium chromium borate. In the optical spectra of $Nd_xGd_{1-x}Cr_3(BO_3)_4$, where x vary from 0.1 to 0.6, the widening of this line was observed. The second component of this spectral line appears as an independent line at x = 0.6. This splitting indicates the presence of a significant part of second crystalline phase in the samples with x> 0.6. An integral intensity of the splitted spectral line shows the ratio of the phases in the studied crystals.

This study shows the ratio of monoclinic (*C*2/*c*) and rhombohedral (*R*32) polytypic phases, estimated by all used methods. We have found that the rhombohedral phase prevail up to the x = 0.7 in $Nd_xGd_{1-x}Cr_3(BO_3)_4$ solid solutions. The pure Nd chromium borate contains about 38% of rhombohedral (*R*32) modification.

Keywords: Polytypic modifications; rare-earth chromium borates; Rietveld method