

largest value of a d_{ijk} amounts to 0.34(4) pm/V. However, in spite of the relatively small coefficients d_{ijk} there are some interesting aspects for application of $\text{Li}_2\text{SO}_4\cdot\text{H}_2\text{O}$ as a SHG material: availability of large high-quality crystals, transparency in the UV region and possibility of phase-matching in the range from near UV to near IR.

[1] Ziegler G.E., *Z. Kristallogr.*, 1934, **89**, 456. [2] Hobden M.V., *J. Appl. Phys.*, 1967, **38**, 4365. [3] Becker P., et al., *Crys. Res. Technol.*, 2003, **38**, 881.

Keywords: nonlinear optics, nonlinear optical materials, optical materials

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Neutron Diffraction Study of 4-nitroaniline at 100K

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The planarity of the amino group in nitroanilines is still a matter of controversy. It has been assumed in several experimental and theoretical studies of 4-nitroaniline (pNA) [1]. Single crystal X-ray diffraction and experimental electron density analysis of pNA have been previously reported [2,3]. However, no accurate experimental coordinates and atomic displacement parameters (adps) for the hydrogen atoms are available yet. As shown by Bürgi et al. [4], accurate adps for hydrogen atoms are essential for obtaining reliable results on physical properties. With this in mind, we have carried out a single crystal neutron diffraction study of pNA at 100K. It shows a relationship between the planarity of the amino moiety and the degree of intramolecular charge transfer, as well as a hydrogen bond scheme different from that observed in other primary anilines [1].

[1] Goeta A. E., et al., *Chem Mater.*, 2000, **12**, 3342, and references therein. [2] Tonogaki M., et al., *Acta Cryst. B*, 1993, **49**, 1031. [3] Coppens P., Volkov A., *Acta Cryst. A*, 2004, **60**, 357. [4] Bürgi H. B., et al., *Chem.Eur.J.*, 2002, **8**, 3512.

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Cation Distribution in Eu^{2+} - and/or Eu^{3+} -Containing Inorganic Compounds

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Divalent Eu^{2+} ($4f^7 5s^2 5p^6 6s^2$) or trivalent Eu^{3+} ($4f^6 5s^2 5p^6 6s^2$) ions exhibit extremely interesting electrical and optical properties in various inorganic compounds. The coordination of such ions in the crystal structure, for example, would be in control of the photoluminescence of materials used for cathode-ray tube, plasma display panels and imaging plate.

The site occupation of Eu ions has been mainly examined in this study. X-ray diffraction and absorption techniques were applied to such typical compounds as YNbO_4 , $\text{Y}_2\text{O}_3\text{S}$, $\text{BaMgAl}_{10}\text{O}_{17}$ and Eu_3S_4 , which contain Eu^{2+} and/or Eu^{3+} . Fine powder crystals of $(\text{Y},\text{Eu}^{3+})\text{NbO}_4$ were successfully synthesized by the polymerizable complex method and used for structural analyses with the Rietveld method. Single crystals of Eu_3S_4 were synthesized from the powder sample with 0.06 g NH_4I flux by the vapor growth. The crystal structure of a mixed-valence compound, Eu_3S_4 has been examined by the valence-difference contrast method of anomalous scattering at the Eu L_{II} absorption edge. The hopping character of 4f electrons between adjacent Eu sites is partially frozen between Eu^{2+} and Eu^{3+} ions below $T = 210$ K. A charge-ordered tetragonal structure was determined below $T_c = 188.5$ K, where a half of Eu^{3+} ions occupy the whole 4a sites, while the remaining half of Eu^{3+} ions mix with all Eu^{2+} in the 8d

sites.

Keywords: cation distribution, structural inorganic chemistry, mixed-valence compounds

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Synthesis and Characterization of Nanocrystalline $\text{Y}_3\text{Al}_5\text{O}_{12}$ derived by a Sol-Gel Method

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Nanocrystalline pure and rare earth doped yttrium aluminum garnet (YAG) powders are promising materials for photonics

Nano-sized YAG powders were synthesized through nitrate-citrate sol-gel processing [1]. The powders were obtained by drying the YAG gel followed by calcinations at 600°C. The amorphous YAG precursor was then heat-treated at different temperatures from 900 to 1100°C. The evolution of YAG phases was investigated by X-ray powder diffraction (XRD) and Fourier Transform Infrared Spectroscopy (FTIR). The XRD pattern of powder heat-treated 3h at temperature as low as 900°C showed the formation of cubic $\text{Y}_3\text{Al}_5\text{O}_{12}$ well-crystallized phase. No intermediate phases were observed. Integral breadth of diffraction line profile was used in order to approach the changes in crystallite size with heat-treatment. The increase of YAG crystallite size from 20 to 50nm with annealing temperature increasing from 900 to 1100°C was observed.

The FTIR measurements were found to be consistent with the YAG phase crystallization process observed by XRD.

[1] Chung B.-J., Park J.-Y., Sim S.-M., *J. Ceram. Proc. Res.*, 2003, **4**, 145.

Keywords: electronic photonic materials, X-ray powder diffraction, FTIR

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Structural Study of Sn-doped In_2O_3

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Tin doped In_2O_3 (ITO) is widely used in phototronic, optoelectronic and microelectronic devices. There are numerous studies on ITO, but the understanding of its defect structure is rather incomplete. In_2O_3 possesses a cubic structure, the space group $\text{Ia}\bar{3}$ [1]. The structure contains two different six-fold-coordinated cation sites referred as sites B and D, respectively. This paper focuses on X-ray diffraction and ^{119}Sn Moessbauer studies of the polycrystalline ITO samples containing 2-14 at% Sn. Nanocrystalline ITO powders were prepared by a sol-gel technique using InCl_3 and SnCl_4 reagent grade chemicals and annealed at 300°C for 5 h. Lattice parameter a of doped In_2O_3 increased with Sn-doping level up to 8 at% and decreased above. Such behavior of lattice parameter suggests that tin substitution for In^{3+} on sites B and D is non-uniform and depends on tin content. ^{119}Sn Moessbauer spectra indicated that only the Sn^{4+} state is present in ITO samples. The least square fitting of spectra was performed by assuming presence of two doublets. In accordance with [2], doublets with isomer shifts (IS) in the range 0.09–0.17 and 0.36–0.464 mms^{-1} , respectively, correspond to the D and B sites in the cation sublattice of the In_2O_3 structure. The area ratio of the two doublets depended on tin content.

[1] Marezio M., *Acta Cryst.*, 1966, **20**, 723. [2] Binczycka H., et al., *Phys. Stat. Sol. (B)*, 2005, in press.

Keywords: Sn-doped In_2O_3 , X-ray diffraction, Moessbauer spectroscopy