s8.m27.p12 **Portable simultaneous XRF/XRD device for an in-line phase monitoring of ferroelectric's production.** Ibraimov N.S.[1], Likhoushina E.V.[1], Yudina M.V.[1], Bolotokov A. A. [1], Dolgaya Zh.A.[2], Golovin V.A.[2], [1] *Institute for Roentgen Optics, Moscow, Russia,* [2] *ELMA, Zelenograd,Russia. E-mail: nar@iroptic.ru*

Keywords: Ferroelectrics; Production

Modern high-tech industry is based on application of substances featuring special physical properties. Such substances include ferrielectrics, piezoceramics like $Pb_{30}Ti_xZr_yO_3$ (lead titanate-zirconate, LTZ). Capable of transforming electric signals into acoustic (or vice versa: acoustic into electric), LTZ ceramics products are widely used in hydroacoustics, common engineering and even household equipment (sound-perceptive devices in mobile telephones). To control the structure of piezoceramics in the course of their manufacture, portable x-ray complex developed based on polycapillary optics was used. Polycapillary optics permits to render the instrument significantly smaller in size, use low-power x-ray source (-30W), improve resolution, and decrease the exposure time. Formation by Kumakhov half-lens of the primary parallel x-ray beam and filtration of diffracted radiation by a special polycapillary system enables successful express analysis of the structure in the production environment. Analysis was performed of completeness of the $Pb_{30}Ti_xZr_yO_3$ phase formation in case of low-temperature (synthesis) and high-temperature baking of the mixture of components, presence of free Pb, observance of specified ratio of zirconium content to titanium. Ferrielectrics feature maximum piezoelectric properties only within a narrow range of phase and element composition ($x\approx48\pm2$, $y\approx52\pm2$). This interval is correspondent to pseudo-cube lattice with $a=b\approx c$ crystalline cell parameters. A correlation between electrophysical and structural parameters of piezoceramic elements has been established. Presence of tetragonal and rhombohedral splittings depending on technological factors (phase composition of raw material, its dispersion, temperature and time of baking) has been shown. The suggested In-line Monitoring XRF/XRD System is not specific to manufacture of ferrielectrics - it can be adapted to other processes (such as, pharmaceutics, cement, electronics, mining, metallurgy).

s8.m27.p13 **Crystal Structure Solution from Powder Data of Selected Organic Compounds.** Wieslaw Lasocha[1], Alicja Rafalska-Lasocha[1], Krzysztof Surówka[2], [1]*Faculty of Chemistry, Jagiellonian University, Ingardena 3, 30-060 Krakow, POLAND,* [2]*Agricultural University of Cracow, Dept. of Refrigeration and Food Concentrates, ul Balicka 122, 30-149 Kraków, Poland. E-mail: lasocha@chemia.uj.edu.pl*

Keywords: Powder diffraction; Structure solution; Rietveld refinement

In the recent years, thanks the to efforts of many research groups, enormous progress in the area of structure solution from powder diffraction data was achieved. [1-4]. Nowadays the crystal structures of quite complex, flexible organic compounds are easily solved nearly in 'black box' mode. Speed of modern computers enables the crystal structure solution and Rietveld refinement to be completed in few hours. Nevertheless, similarly as in the early years of powder diffractometry, the most difficult problems one still encounters in the process of structure solution from powder data are indexing and ambiguity in the space group determination. To overcome this obstacles, the best quality powder diffraction pattern is of great importance.

As an illustration of these statements a few examples of crystal structure solution of organic compounds, consisting of a few independent parts and about 20-40 independent atoms in the asymmetric unit will be presented. Investigated compounds belong to a group of carboxylic acids and their complexes with strong organic bases such as proton sponges. We focussed our attention on DMAN: 1,8 -bis(dimethylamino)naphthalene with chemical formula $C_{14}H_{18}N_2$ which very eagerly combines with proton donors, and resulting ionic complexes are interesting subjects of investigation of biological systems, crystal engineering as well as they are challenging testing samples for ab initio X-ray crystal structure investigations by powder methods. During recent years, in the literature appeared scarce, but very novelty information about a key role of proton sponges in gene therapy.

Applied software and time required by each step, with indication where intervention of a crystallographer is still essential, will be highlighted and discussed.

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