

time since the gate pulse. This allows the number of counts within a designated energy window to be binned as a function of time since the trigger. Time resolution to the  $\mu\text{s}$  level is possible this way.

The module can also count the number of times the sync input toggles following each gate pulse and tag x-rays with this information as well. This allows the x-rays to be binned by this information as well, allowing spectra to be collected synchronously with a modulating sample state (phase locked mode) at rates up to a few 100 kHz. We present an example of EXAFS from a high-T superconductor cycling between its normal and SC states.

**PS01.11.11 A CONTAINER FOR THE TRANSPORT OF MOUNTED CRYSTALS.** Marc Whitlow, Berlex Biosciences, 15049 San Pablo Avenue, Richmond, CA 94804

An airtight crystal shipping tube has been produced by modifying a 15 ml pressure reaction tube to hold a mounted crystal. A crystal mounted on a specimen pin is held in place in the crystal shipping tube by a set screw. The crystal shipping tubes are then inserted in holes prepared for them in two foam rubber blocks. The foam rubber blocks fit into a Styrofoam box, and the whole package is shipped by overnight courier. Both the foam rubber packing and the fact that the specimen pin is firmly held in place minimize the mechanical stresses that occur during transport. Furthermore, the airtight container eliminates any pressure changes that may occur. In addition, the mounted crystals are visible through the glass shipping tube. A number of macromolecular data sets have been successfully collected from crystals shipped in this way.

**PS01.11.12 ELECTRON DIFFRACTOMETRY AND CRYSTAL STRUCTURE OF BRUCITE.** A. P. Zhukhlistov<sup>1</sup>, A. S. Avilov<sup>2</sup>, G. Ferraris<sup>3</sup>, B. B. Zvyagin<sup>1</sup>, V. P. Plotnikov<sup>1</sup>, <sup>1</sup>Inst. Ore Mineralogy RAS, <sup>2</sup>Inst. Crystallography RAS, Moscow, Russia, <sup>3</sup>University of Torino, Italy

The principles of direct measurements of the electron diffraction (ED) intensities developed in the State Optical Institute (St. Petersburg) and Institute of Crystallography RAS (Moscow) and a commercial EMR-102 ED-camera have been adapted for analysis of two-dimensional intensity distributions, in particular those of the most most informative oblique-texture patterns. With stationary detector, the ED-pattern is moved within electromagnetic fields with variable steps and along chosen directions. Special instrumental and software improvements provide corrections of different effects (e.g., incident beam instability) and increase precision, resolution, linearity and dynamic range of the measurements.

A set of intensities collected on brucite,  $\text{Mg}(\text{OH})_2$ , showed that the described system of electron diffractometry is the most reliable ever produced for this purpose and opens new prospects in the field of structural crystallography by electron diffraction. In fact, it provides reliable Volt-values of the crystalline electrostatic potential field, whereas the structural features of brucite have been revealed with a precision comparable with that of X-ray diffraction for O and Mg atoms, and of neutron diffraction for the H atom.

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**PS01.11.13 X-RAY DIFFRACTION AT NON-AMBIENT TEMPERATURE CONDITIONS.** B. Koppelhuber-Bitschnau, F. A. Mautner, Institute of Physical and Theoretical Chemistry, Technical University Graz, 8010 Graz, Austria, and P. Doppler, Anton Paar GmbH, Graz, Austria

Several Cameras for X-ray Diffraction at low and high Temperature range were developed, most features and benefits of the following four Temperature Attachments are presented.

With the HTK 16 High-Temperature Camera investigations in the temperature range from room temperature to 1850 K can be carried out either under vacuum, air or inert gas. The HTK 16 can easily be fit to most available goniometers. Integrated alignment slit allows precise positioning even at high temperatures, the heating filament is optimized for minimum temperature gradient, the linear compensation of the heating filament elongation guarantees for sample position stabilization.

The TTK 450 Low-Temperature Camera can be operated with most of the available goniometers, both horizontal and vertical ones. It permits temperature studies by X-Ray methods at temperatures from 80 to 700 K either under vacuum, air or inert gas.

The XRK X-Ray Reactor Chamber mounted on a goniometer permits studies of solid state and solid state-gas reactions at temperatures from room temperature to approx. 1250 K. The experiments may be carried out either in reduced, inert or oxidizing atmospheres at pressure from approx 1 mbar to 10 bar. No temperature gradients within surface and whole bulk of samples, no condensation of reaction gases because housing thermostatable up to 400 K.

The He-TTK Low Temperature Attachment equipped with a closed cycle Helium cryostat permits temperature studies to be made by X-ray methods at temperatures from approx. 12 to 700 K. The He-TTK can be operated with most of the vertical goniometers available, and is suitable for powders, sheets, single crystals and thin films.

**PS01.11.14 THE ANALYSIS OF MACROMOLECULES UNDER AQUEOUS AND NON-AQUEOUS CONDITIONS DETERMINING THE PARTICLE SIZE DISTRIBUTION AND MOLECULAR WEIGHT DISTRIBUTION USING LOW AND RIGHT ANGLE LASER LIGHT SCATTERING AND PHOTON CORRELATION SPECTROSCOPY (PCS) DETECTION WITH SIZE EXCLUSION CHROMATOGRAPHY.** Trevor Havard and Peter Wallace. Precision Detectors Inc., 160 Old Farm Road, Amherst, MA 01002 Tel 413 256 0516.

The objective of this paper is the separation and analysis of the particle size and molecular weight of macromolecules like Poly-Carbohydrates and Proteins, carried out under aqueous conditions, and macromolecules like polystyrene, star branched polystyrene and dendrimers. The paper will describe a **new instrument** that enables the molecular weight and particle size distribution to be obtained simultaneously. The paper will also describe how an instrument of this type can be used for the first time in flow mode to determine the molecular weight distribution as well as the hydrodynamic radius of the macromolecule eluting from an SEC system. This system is especially useful in determining the amount of aggregation that occurs while trying to dissolve certain macromolecular systems like carbohydrates and proteins into solution under different conditions where pH, temperature may be varied. The results show the effective use of Photon Correlation Spectroscopy (PCS) to determine aggregation, branching, and size, independent of the usual chromatographic integration and baseline user selection. The use of Photon Correlation Spectroscopy as a technique for characterizing particles is well documented. However, until now there has never been an attempt to make these important measurements in a flowing SEC system.