16.5-3 TIME-RESOLVED POWDER DIFFRACTION OF REACTIONS USING LABORATORY, SYNCHROTRON, AND NEUTRON SOURCE. By P. Barnes, S.M. Clark, S.E. Tarling, E. Polak, G. Mamott and J. Anwar, Industrial Materials Group, Department of Crystallography, Birkbeck College, London, U.K.

This paper considers a wide range of powder diffraction techniques that can be used to monitor industrial reactions and transformations occurring at elevated temperatures and various environments.

For slow industrial furnace cycles (>24 hours) laboratory high temperature powder cameras can be modified and used invariably with some chemical modification to the sample and positive environmental (gas) control when powder surface decomposition is excessive compared to the real large scale industrial furnace: these points will be illustrated using the furnace-production of pigment-Ultramarine as an example.

For reactions taking place over 2-10 hours, a laboratory diffractometer with specially-designed environmental cells and position-sensitive detector has been used to collect diffraction data over 5-10 minute intervals, using a range of temperatures and gas pressures up to 10 bars. This combination is illustrated using the calcination of zirconium hydroxide to zirconia up to 700°C and the hydration of portland cements under ambient and steam (autoclave) curing conditions. Even faster data collection rates are required for studying polymorphic transformations in pharmaceutical products.

Most of these dynamic studies have also been performed using neutron and synchrotron X-ray sources. In the case of neutron diffraction, the quality of data is sufficient to enable the non-crystalline water fraction to be monitored, as well as growth of crystalline products, during the hydration of cements. The synchrotron method has been developed using hydration cells on an energy-dispersive powder diffraction system built by the authors, and these studies complement the neutron results.

This study demonstrates that quite complex reaction cycles can now be studied using a variety of time-resolved powder diffraction techniques.

16.5-4 APPLICATION OF THE CRYSTALLOGRAPHIC INVESTIGATION OF URINE IN CLINICAL PRACTICE. By V.G. Maidannik, V.D. Chebotareva and V.G. Burlai, Kiev Medical Institute, Kiev, USSR.

A procedure for the crystallographic analysis of urine is described. In a study involving 112 patients with renal disease and 20 normal subjects, the diffraction patterns of urine (solids) were recorded. There were significant differences between the diffraction patterns recorded, respectively, from the urine of patients with glomerulonephritis, patients with pyelonephritis, and the controls. The simplicity of the method suggests that it may have wide clinical applications.

16.6-1 GONIOMETRIC DEVICE AND LIQUID HELIUM CYROSTAT FOR SINGLE CRYSTALS STUDIES WITH X-RAY 4 CIRCLES DIFFRACTOMETER, DOWN TO 7 K. By J. Muller, R. Argoud, J.J. Capponi, M. Marezio, Laboratoire de Cristallographie, associé à l'U.S.T.M.G., C.N.R.S., 166 X, 38042 Grenoble Cedex (France).

For all low temperatures devices used for collecting single crystal X-ray intensity data below 77 K, it is necessary to enclose the crystal, mounted on a goniometer head, inside a liquid helium cryostat. In the apparatus we describe here, the crystal is mounted on a small goniometric device (36 mm in diameter) which is magnetically coupled to the diffractometer. This coupling is achieved by mounting on the diffractometer a master magnet in the place of the classic goniometer head. The master magnet drives a small slave-magnet fixed on the goniometric device enclosed in a flow liquid helium cryostat which remains stationary during the ϕ, χ and θ movements. The crystal is oriented by the master magnet.
The goniometric device, as the cryostat, can be easely adapted to all diffractometer types, without major mechanical modification.
By using this goniometric device (mounted on a Philips PW1100 diffractometer) we have been able :
- to refine, at room temperature, a garnet structure (YIG) with an R-factor of 1.8 % ;
- to characterize the well-known tetragonal → orthorhombic transition of $DyVO_4$ occuring at 14 K ;
- to collect two sets of data (the first at 7 K and the second at 15 K) for $LiTi_2O_4$ (which becomes superconductor at 10.8 K).

These two data collections took 6 days without any interruption. The data are now been analyzed.
The device can also be used to carry out studies under vacuum, controlled atmospheres, and under small pressures.