16. APPARATUS AND TECHNIQUES

16.6-01 CHARACTERIZATION AND QUANTITATIVE DETERMINATION BY X-RAY DIFFRACTION OF A QUARTZ, CALCITE AND THE CLAY MINERALS ILLITE, KAOLINITE AND CHLORITE IN SOME LAKE, PLUVIAL AND SEA SEDIMENTS. By C.J. Toussaint, Joint Research Centre, Ispra Establishment, Italy and R. Boniforti, C.N.R., Marine Environmental Laboratory, Flasscherino, Italy.

Sediments are deposited in a variety of geological environments such as lakes, rivers and oceans. They are very important in the geochemical cycles of many elements because they act as "traps" for material introduced into the environment by both natural and anthropogenic processes. In the framework of a project where the distribution of stable elements with different physicochemical characteristics will be studied in several sites along the Italian coasts, in particular to existing and future nuclear sites, also a mineralogical characterization of the sediments has been found to be useful. Sediment samples were collected from the Gulf of La Spezia and surrounding zones. (Vera and Magra river, Massaciuccoli lake) and from the Sardegna coast. For the characterization of the various clay minerals oriented aggregates have been used, obtained by sedimenting a dilute dispersion. Auxiliary techniques, using organic swelling agents, treatment with dilute hydrochloric acid and high temperature treatments have been utilized. Quantitative determinations were carried out using an X-ray diffractometer with graphite monochromator, utilizing internal standard and active dilution techniques.

16.6-02 A COMPARISON BETWEEN THE CONVENTIONAL BRAGG-BRENTANO TECHNIQUE AND THE TRANS- MISSION TECHNIQUE IN POWDER DIFFRACTION. By E. Wolfsel, SPD Application Laboratory, Hilpertstr. 10, 6100 Darmstadt, West Germany.

The STOE/SSX automatic diffractometer system will be described, which consists of two powder diffractometers, whereby the horizontal X-ray tube housing is mounted on the Bragg-Brentano diffractometer, whereas the focusing transmission diffractometer of Guinier type can be easily adjusted towards the opposite port of the X-ray tube. The diffractometers are controlled by a DEC LSI 11 computer.

With this system reflection and transmission techniques can be optionally applied. Comparison of results in various fields of applications will show the advantages and disadvantages of Bragg-Brentano, transmission and Debye-Scherrer capillary techniques. The following points will be discussed:

1) Diffractometer alignment
2) Specimen preparation
3) Data collection
4) Precision lattice constants measurement
5) Preferred orientation: The combined reflection-transmission scan
6) Intensity measurement: Line profile analyses
7) Quantitative analyses of multiphase specimens
8) The use of linear position sensitive detectors in powder diffractometry.

16.6-03 THE ACCURACY OF A FAST SCANNING-PSD GUINIER-DIFFRACTOMETER. By H. Gobel, Siemens Forschungslaboratorium, München, Germany.

The jet age of X-ray powder diffraction started with the installation of a linear position-sensitive detector (PSD) on a diffractometer (H. Gobel, Adv. in X-ray Anal. 22 (1979) 255), allowing data-collection speeds of several hundred degrees per minute. This is about ten times faster than the fastest scans reported with conventional systems (G.L. Ayers et al., J. Appl. Cryst. 11 (1978) 229). The method takes advantage of the capability of a PSD to collect all X-rays over a range of several degrees of 2Theta in parallel and accumulates the entire pattern by scanning the PSD continuously along the arc, adding quanta of equal diffraction angles into equal channels of a multichannel.

While the use and accuracy of this technique was well studied for the case of a Bragg-Brentano diffractometer (H. Gobel, Adv. in X-ray Anal. 26 (1981) in press), its application to a correctly focusing Guinier-diffractometer was demonstrated more qualitatively (H. Gobel, ACA Spring Meeting 1979, Honolulu, Hawaii). It was shown that well-plottable diagrams with low background and pure Kα lines could be obtained at scanning speeds of up to 100 degrees per minute. This goniometer had been developed for fast analyses of small amounts of sample material and accurate positioning of diffraction lines at low angles. It was now studied systematically under these aspects.

For the numerical evaluations, parts of the minicomputer software system "DIFFRAC 11" were used, mainly a peak-search and line-correction program (written by B. Jobst), a full-featured PGPS-Search-program (written by R. Snyder and C. Mallory) and a version of the Appleman-program (overlaid by C.C. Johnson and G. Power) for lattice-parameter refinements and indexing.


A low-temperature attachment for Weissenberg cameras operating in the range 10-300 K is described. It consists of an Oxford Instruments continuous-flow helium cryostat and a laboratory-built microprocessor (Intersil 6100) based temperature controller-programmer. This latter unit has a memory of 16K words and is connected to a visual-display unit (V.D.U.) and a printer. The user may input information via the V.D.U. using a high-level-language (POCAL). The cryostat has mylar windows, allowing examination of a 240° range of 2θ (symmetrically-disposed about 2θ = 90°) and an angular range of greater than 1.2° from the equatorial plane. Inclined-beam photographs up to 2θ = 30° may also be taken. The temperature stability and temperature accuracy are both to better than ± 0.1 K over the entire range of operation. The helium consumption is 0.2 1/hr at 200 K, 0.13 1/hr at 100 K, 0.65 1/hr at 30 K and 1.3 1/hr at 10 K, giving continuous operation for up to 100 hrs when connected to a 50 l helium Dewar. In addition to the usual modes of operation, slight modifications to the Weissenberg camera enable it to run in a "continuous-recording" mode, in which a series of oscillation photographs of the crystal may be taken at different temperatures. The very sophisticated temperature programming system allows this operation to be carried out completely automatically. The operation of the device is primarily intended for the study of low-temperature structural phase transitions, is illustrated by results obtained for the ferroelectric material pyrochlore, Ag₃Ih₄O₆H₆.