Technical Paper 3

A new suction apparatus for mounting clay specimens on small-size porous plates for X-ray diffraction

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A new suction device for mounting clay specimens on small porous plates for X-ray diffraction

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In X-ray diffraction analysis of clay samples, suction-on-porous plate facilitates a convenient and rapid mounting of oriented specimens. Procedures were described by Kinter & Diamond (1956), Dümmler & Schroeder (1965), Rich (1969) and Shaw (1972). Gibbs (1965) found that specimens made by this procedure compared well with specimens made by some other techniques. However they used only large plates (about 50 mm x 30 mm x 5 mm). A new suction device was designed as the technique is eminently suitable for routine analysis (no centrifugal rinse is needed) and as some of the recently introduced automatic diffractometer sample changers can only hold much smaller specimens. The device described here is for porous plates of size 18 mm x 14 mm x 1.9 mm as used in the Philips PW 1170 sample changer but can just as well be made to accept plates of other seizes.

Device and specimen preparation

A cross-section of the device is shown in Figure 1. It consists of two parts made on a lathe from commercially available PVC rod (diam. 50 mm). Figure 2 shows the different stages of assembly in the laboratory.

When suction is applied, the water-soaked porous plate is pulled onto the lower washer (Fig. 1-6), ensuring a water-tight seal (so this washer need not be glued in the trough), After fitting the top section, a suspension containing about 10 mg clay is poured into the device. Although the amount is not critical, experience has shown that more clay does not significantly increase peak intensities and may induce curling of the clay layer upon drying and especially upon heating. If the concentration of the suspension is not known, a useful aid in routine work is to dilute a suspension in a test tube (diam. 15 mm) until a fluorescent lamp of the laboratory lighting becomes just visible through it as a narrow bright line; 5 ml of this suspension usually yields a satisfactory specimen. If only the fine clay is used, an even more translucent suspension can be used. The turn-away bolts facilitate a quick check of the specimen (a clip or spring connexion may also be devised).

Usually, the sample is prepared as a sodium clay. Since in X-ray diffraction analysis, samples are normally saturated with magnesium and potassium, the specimen can conveniently be saturated with Mg^{2+} or K^{+} by sucking through 5 ml of solution of substance concentration of $\frac{1}{2}MgCl_2$ or KCl 1mol/litre and then washing twice with 5 ml deionized water. After drying, the specimen can be analysed. The dry sample is still suitable for ion exchange and thus facilitates comparative studies on the same specimen. The specimen can be treated with glycerol by spraying with a glycerol/ethanol mixture (volume ratio 1 to 1) using a vaporizer and allowing it to dry on filter paper. The potassium-saturated sample is more suitable for heating

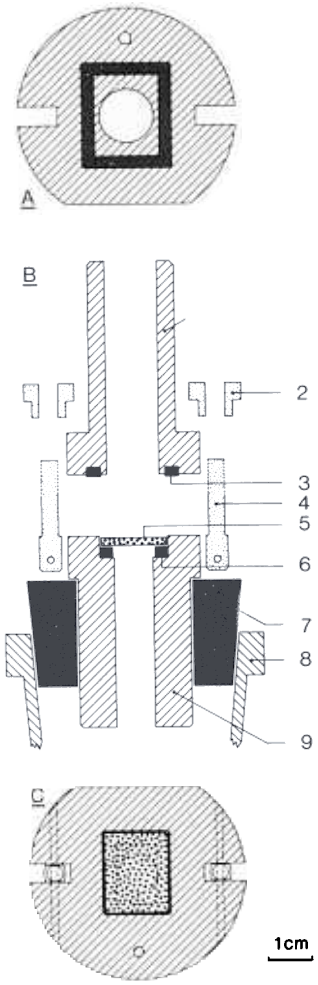

Fig. 1. Cross-section of suction device. A. Bottom view of top section. B. Cross-section of unassembled device. C. Top view of bottom section. 1. Detachable top section. 2. Nut. 3. Neoprene or rubber washer (glued in groove). 4. Turn-away bolt for rapid assembly. 5. Porous plate. 6. Neoprene or rubber washer (need not be glued). 7. Perforated rubber stopper. 8. Rim of vacuum flask. 9. Bottom section. Note the small hole in the flange of the top section (A) and the fitting pin in the bottom section (C) for easy assembly.

tests than the magnesium-saturated one, which is hygroscopic.

If the method of Rich (1969) be preferred, in which a more concentrated suspension is pipetted directly onto the porous plate, then the device can be used without the top section.

A diffractrogram of a clean porous plate is useful as control if small peaks due to the plate material show up when the clay mount happens to be too thin.

Preparation of porous plates

For the manufacture of porous plates, two materials have proved suitable so far: the white unglazed non-vitreous ceramic wall tile obtainable from almost any tile

Fig. 2. Different stages of assembly.

factory, and 'Diapor M8G' (Schumacher Fabrik, Bietigheim/Württemberg, West Germany), available in plates of ca. 500 mm x 300 mm x 5 mm consisting mainly of mullite. These materials differ in some respects. Unglazed tile is much harder and, therefore, has a longer life in repeated use. It has a more pitted surface, however, and is somewhat less permeable than 'Diapor'. Both have excellent heating properties.

The small plates can be prepared like thin sections of rock specimens or impregnated soil samples. If prepared by hand, tiles must be cut to a small working size (e.g sufficient for 6 small plates), they are then ground to the desired thickness, scored with a sharp object, and the small plates snapped apart.

With a flat-grinding machine, preparation is much quicker since larger units can be handled (e.g. sufficient for 40 small plates): one man can then prepare over 500 small plates in a day.

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