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SAMPLE CUPS AND A TECHNIQUE FOR SIDEWARD PACKING OF *X*-RAY DIFFRACTOMETER SPECIMENS

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Sideward filling of specimen cups has been employed by a number of workers (*e.g.*, Engelhardt, 1955; Gordon and Harris, 1956; Niskanen, 1964) to reduce the preferred orientation of mineral powders, especially of clays. However, there are some inherent difficulties in achieving the reproducible charging of a sideward-packed specimen cup, and recommendations given by earlier writers such as how many times the cup should be tapped to assist the settling of the mineral powder in the cup, or the number of times the cup should fall a certain distance, do not seem to eliminate the element of operator dependence. Sideward packing, nevertheless, appears to be superior to other methods for the reduction of preferred orientation, and has been adopted for the present work. To overcome the personal factor in sample preparation, a special vibrator is used.

Rotation of the specimen is often recommended in order to expose a statistical powder sample to the *x*-ray beam, especially when accurate intensity measurements are desired. However, with the Philips assembly PW 1064, the holder used for rotation of packed powder specimens cannot be used for specimens prepared on glass slides, *e.g.* oriented specimens of clays, and by running such specimens alternately with packed powder specimens a change of holders is necessary. This means that the alignment has to be checked and adjusted on each change over. To overcome this we have constructed a simple stationary cup for packed specimens to be used with the stationary holder, and in the course of routine work on

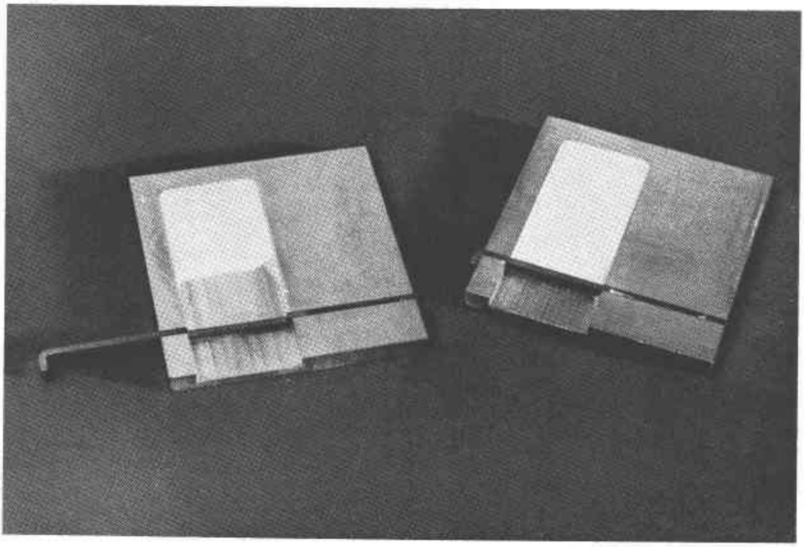

FIG. 1. Stationary cup of aluminum, with brass rod to close the cavity. The block is 42×38×3 mm, with an excavation 30×15×1 mm, which has a recessed extension 2 mm deep to the edge of the block serving to hold the dispensing funnel. To close the cavity, a brass rod, 45 mm long and with a cross section 2×1 mm, runs in a groove. The rod is bent at one end to make pushing easier and it has a 1 mm deep recess, that matches the open side of the cavity. The bottom corners of the cavity are rounded to allow the cup to be properly filled without entrapping air. Left, the cup half filled with rod in open position; right, ready for exposure.

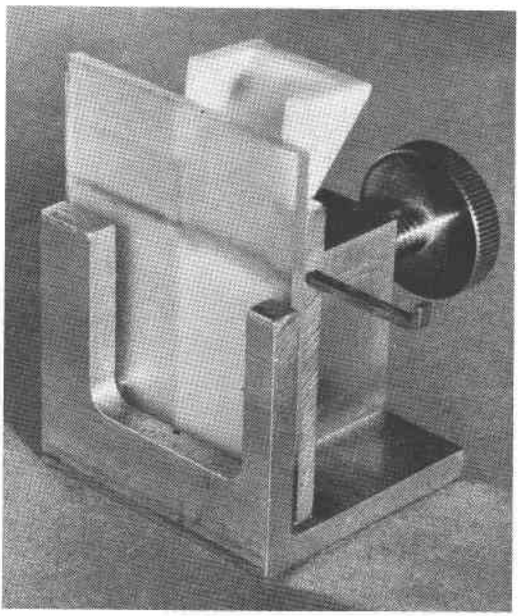

Fig. 2. The stationary cup when it is being charged. Note the dispensing funnel (in this case of teflon) and the ground glass slide. The brass rod is in the open position.

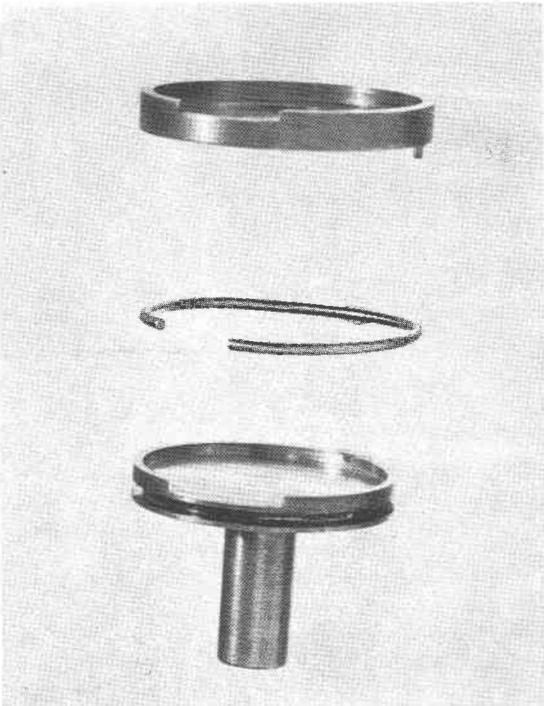

FIG. 3. A sideward-packing sample cup for use with the Philips rotation assembly, PW 1064. In the figure, the cup is dismantled. *Lower:* Cup body with outer cup diam. 21.5 mm, with a 1 mm deep groove around the edge and a 10 mm opening in the rim. The stem is 6 mm wide, height of stem + cup 16 mm, height of rim 1 mm. *Middle:* Steel wire diam. 0.8 mm, 60 mm long, coiled to fit in the cup groove. *Upper:* Ring to fit outside the cup body, 3 mm high, outer diam. 23 mm. The ring has a 10 mm opening corresponding to the one in the rim of the cup, and a groove (less deep than in the cup) running around the inside of the ring. The ring and cup are held together by the tension of the steel wire. The tag seen to the right of the ring allows the ring to be rotated around the cup body. The top of the ring lies very slightly below the plane of the rim of the cup, to facilitate its rotation when the cup is in the packing stand (Fig. 4).

geological material, principally clays, the use of the stationary cup has been found to be both convenient and time-saving. In comparison with the rotating cup, no noticeable loss of accuracy has been entailed; on the contrary, the 2θ values are more easily reproduced, as the stationary-cup surface lies in a fixed plane, whereas the surface of the rotating cup has to be adjusted each time.

In Figs. 1 and 2, the stationary cup is illustrated and details of it are found in the legend. The cup is made of aluminum. The dispensing funnel shown in Fig. 2 is made of teflon, which, however, is too soft and a funnel

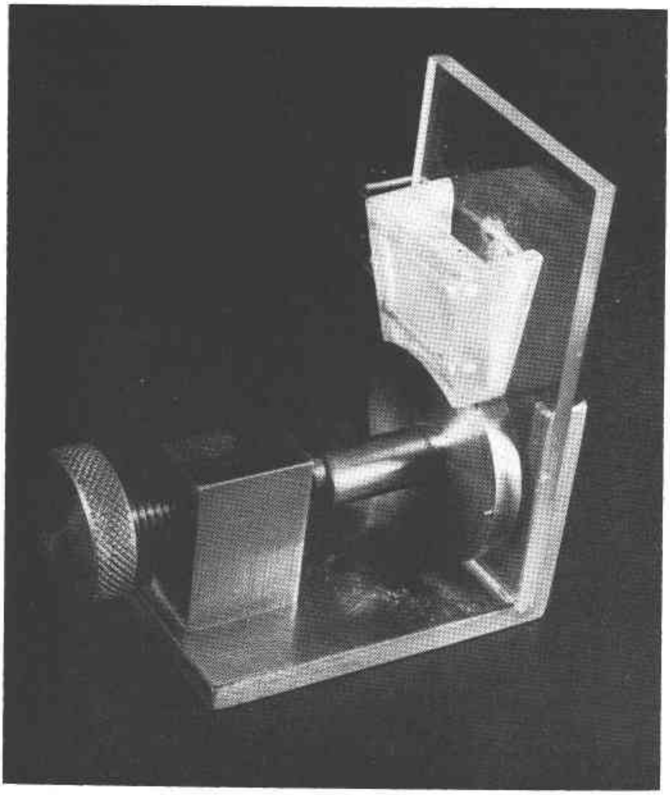

FIG. 4. The rotatable cup in packing position. The stand is similar to the one in Fig. 2, with a U-shaped opening in the front, so that the cup surface may be seen during charging. The cup is pressed against the glass slide by firmly turning the screw. A perspex funnel is machined to fit the rim and held by a spring wire. When the cup is being charged the ring is rotated to the open position and when filled the ring is rotated to close the opening while the cup is still in the stand. The slide and cup are then removed together and when in a horizontal position, the slide is carefully lifted to avoid electrostatic disturbances. The cup is placed in the rotatable holder.

of aluminum is to be preferred. Two sizes of stationary cups have been made, one which is shown here, the other with a smaller cavity for use when little material is available. The glass slide, as shown in Fig. 2, has a coarsely ground surface to prevent platy minerals from gliding along its surface (Norrish and Taylor, 1962). The rotating cup is illustrated in Figs. 3 and 4 and the constituent parts of its closing mechanism can be seen in Fig. 3. In Fig. 4, the rotating cup is shown in the packing stand.

The cups are filled with the aid of a Sortimat vibrator, type Sm I.[1]

[1] Sortimat Creuz and Co., Winnenden/Württ., West Germany.

This vibrator was originally designed with a transport spiral to feed out screws and similar items; for the present purposes the transport spiral is removed and the stand holding the cup (Figs. 2, 4) is placed directly onto the vibrator. The important feature of this vibrator is the circular motion given in the horizontal plane which accompanies the overall vibration effect. This circular vibration effect is produced by a trigonal arrangement of three steel bands and an electromagnet working on them. The circular motion helps the mineral powder to float into the specimen cup and diminishes the tendency of fine powders to entrap air. The cup is filled by fixing it in the stand and placing the stand onto the vibrator; when charging the powder, the stand is lightly tapped to get the powder firmly packed. A smooth and planar surface is achieved, and similarly packed cups are produced by different workers, showing that operator dependence is eliminated at this point.

Mr. H. Johansson carried out the machine working of the cups and his contribution to the development of the present models is gratefully acknowledged. Mrs. Ulla Bokén gave technical assistance which was supported by a grant (1959–61) from the Swedish Natural Science Research Council. The English of this manuscript was kindly revised by Dr. M. J. McNamara.

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THE PREPARATION OF THIN SECTIONS OF FRAGMENTAL MATERIALS USING EPOXY RESIN

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Introduction

Techniques of preparing thin sections of fragmental materials, such as sand grains, drill cuttings and mill products, either have required lengthy