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NOTES

A METAL-MEMBRANE MOUNT FOR X-RAY POWDER DIFFRACTION

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Key Words—Diffraction, Metal-Membrane, Mount, Sample-Holder, X-ray

Silver membrane filters can be used as substrates for oriented clay samples; they are not affected by heat or organic solvent treatments, and can be prepared rapidly and easily using readily available laboratory equipment. The silver of the substrates gives no maxima at diffraction angles less than about 38°2θ using copper radiation. This contrasts with glass, plastic-filter, or ceramic tile substrates, which produce significant amorphous or crystalline diffraction effects between about 10° and 30°2θ. The high mass-absorption coefficient of silver to copper radiation results in a lower overall background than these materials, yet does not decrease diffraction intensities produced by the sample; the silver underlies, but is not mixed with the sample. Silver metal-membrane filters, however, present problems when used with an automated diffractometer. A new method for mounting the silver substrates has been designed which can be readily adapted to existing holders. The new device[1] described herein (Figure 1) permits easy installation and removal of silver filters, presents a flat, even sample surface in the correct position relative to the X-ray beam, and fits automatic sample changers.

Other mounting methods include glued mounts, vacuum mounts, and top-clamped mounts. Each of these techniques has significant disadvantages. The glued mount is inconvenient and often leaves the sample in a condition in which further treatments that are important in the identification of clay minerals cannot be made. If separate mounts are prepared, the differences in sample thickness retained on each silver filter result in faulty surface positions. Also, duplicate filters may show variations in preferred orientation and thus give different diffracted intensities. The vacuum mount is unacceptable because it can be adapted only with great complexity to an automatic sample changer. A mount in which the filter is held in place by clamps on the top of the mount likewise will not reproduce an even sample surface consistently; the vertical position of the sample is highly variable, and the clamps tend to produce diffraction and fluorescence effects at low goniometer angles. The device described below avoids these disadvantages.

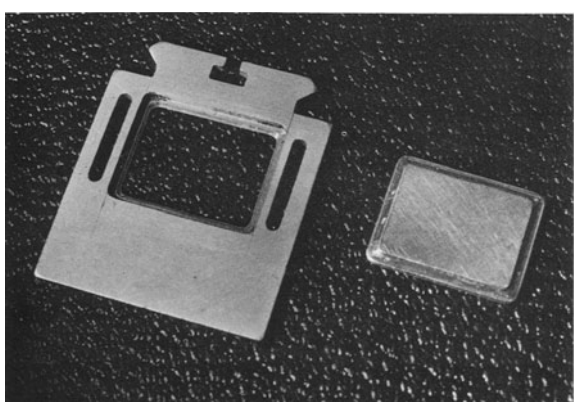


Figure 1. Left—Photograph of the back side of the metal-membrane filter mount described in this paper modified from a standard aluminum sample holder for a Philips automatic sample changer. Right—The top side of the insert used to hold the filter in place. The insert is 2.3 cm long.

The particular model of sample holder shown in Figure 1 is machined from a standard aluminum holder for a Philips[2] automatic sample changer. The insert shown is aluminum; however, plastic serves as well. The filters used in this laboratory are porous silver discs 47 mm in diameter, 0.05 to 0.06 mm in thickness, and have a pore size of 0.45 μm. The filters are obtainable from Selas Flotronics of Spring Hill, Pennsylvania. Specimens about 0.05 mm thick are collected on the filters in standard filtering equipment such as that made by Millipore Corporation (Figure 2). By using half a disc and a suitable rubber mask in the vacuum-funnel system, two separate specimens may be prepared from each 47-mm filter.

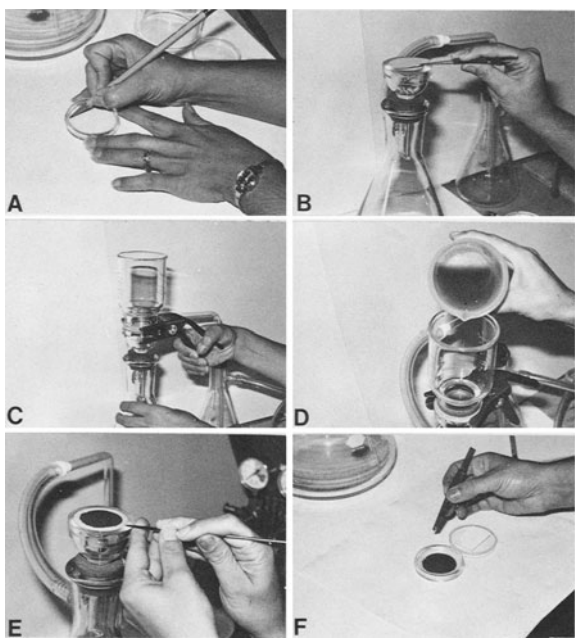


Figure 2. Procedure for mounting sample on metal-membrane filter: (A) Label filter around edge with pencil. (B) Center filter on vacuum filter apparatus. (C) Place filter funnel on top of the filter and clamp. (D) Pour a small amount of sample suspension into filter funnel (enough to cover filter). Repeat after water is pulled through the filter. (E) Remove clamp and filter funnel, and pry up edge of filter with a pointed spatula. (F) Lift filter with filter forceps and place in a plastic capsule to dry.

[1] Government patent applied for.

[2] The use of commercial names in this article is for identification purposes only and does not constitute endorsement by the U.S. Geological Survey.

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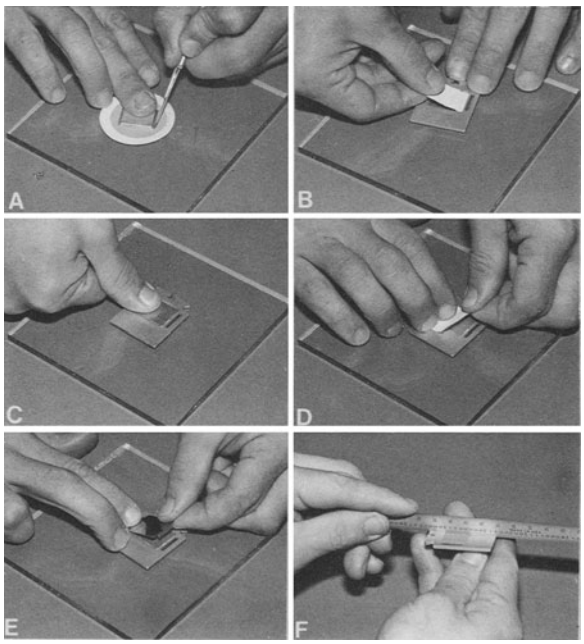


Figure 3. Procedure for mounting metal-membrane filters in an X-ray diffractometer sample holder: (A) Using the insert as a template, cut the filter with a scalpel or sharp knife. (B) Position the modified Philips sample holder face down on the clean glass plate, and place the cut-out piece of silver filter, sample side down, into the rear opening of the mount. (C) Place the mount insert on top of the silver filter and press firmly down. (D) Secure the assembly with plastic tape or equivalent. (E) If standard Philips sample changer mounts are used, the metal clips supplied may be used to secure the assembly. (F) Check the specimen surface to insure that the sample is flat and flush with the surface of the mount.

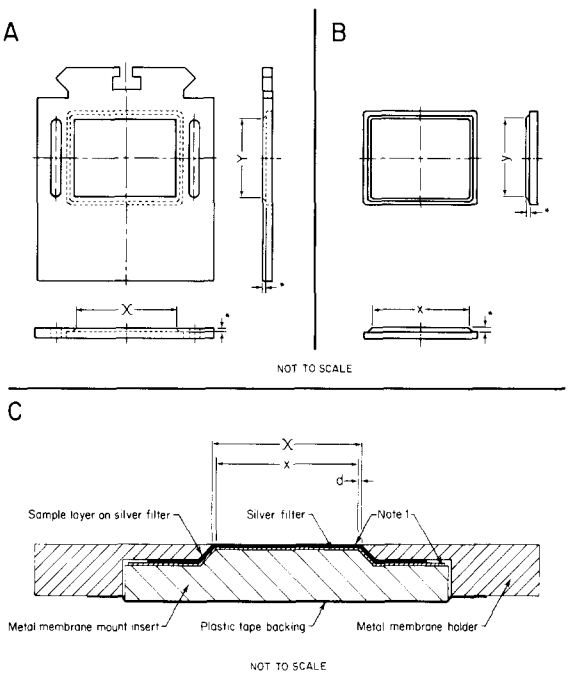


Figure 4. Plans showing the modification of a standard Philips automatic sample-changer mount. (A) Aluminum mount showing recess to be milled to accommodate insert and metal-membrane (silver) filter. (B) Aluminum or plastic insert. Dimension marked by asterisk is critical; insert and holder must match within 0.025 mm. Tolerance on other dimensions is 0.25 mm. (C) Enlarged cross section showing assembled device.

Note 1: The thickness of the filter plus sample layer on it spaces the insert so that the sample surface lies flush with the surface of the holder.

Mounting a sample-coated silver or other metal-membrane filter in the holder requires a hard, clean flat surface, such as a glass plate. The steps in mounting the sample are shown in Figure 3. Plans for the metal-membrane X-ray diffraction mount are shown in Figure 4. The top surfaces of the holder and the insert must match when the insert is placed in the holder with no filter in place. This insures that thickness of the filter plus sample will automatically space the insert so as to produce a sample surface that is flush with the surface of the holder (see Figure 4, note 1). This feature is critical to proper positioning of the sample relative to the focusing circle of the X-ray goniometer. Failure of the sample surface to lie in the surface plane of the holder will result in displacement of the X-ray diffraction maxima from their proper 2θ locations.

Note that a critical feature of the design is the beveling of the holder opening and the corresponding part of the insert at an angle considerably less than 90° to avoid shearing of the membrane. The slope of 45° depicted in Figure 4 is not critical; a lower angle may be used, but higher values are not recommended.

Dimensions x and y of Figure 4 must be less than corresponding dimensions X and Y by an amount (d) dependent on the bevel angle and the thickness of the metal membrane used, or the membrane will be sheared. The relationship is:

$$x = X - 2d \text{ and } y = Y - 2d$$

where:

$$d = t\left(\frac{1 - \cos\phi}{\sin\phi}\right) = t\left(\tan\frac{\phi}{2}\right), \text{ and}$$

t = thickness of metal membrane plus layer of sample
ϕ = angle of bevel

The silver filters in use by the authors in their laboratory have a maximum thickness (including the sample thickness) of 0.1 mm. For this thickness, given a bevel angle of 45°, dimensions x and y should be at least 0.08 mm less than dimensions X and Y. However, the machining or forming of the parts need not be this precise as long as x and y are about 0.25 mm less than X and Y. The only truly critical dimension is that marked by asterisks in Figure 4. It must be the same on insert and holder in order to satisfy the requirement, described above, that the surface of the insert be flush with the top of the mount when the two are assembled with no filter in place.

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