the altered allanite. They may be also due to concentration of the radioelements in some of the new red-brown varieties of allanite after the breakdown of the original allanite to red brown allanite, clinozoisite and epidote. Compared with clinozoisite, green epidote granules occasionally found in the clinozoisite veinlets are slightly more radioactive.

ACKNOWLEDGMENTS

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A DIFFRACTOMETER MOUNT FOR SMALL SAMPLES*

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Buerger and Kennedy (1958) discuss the problem of scattered *x*-radiation from a diffractometer sample holder and describe improved results using a BT-cut quartz oscillator plate. They point out that when a small amount of sample is supported in the *x*-ray path, background noise from various mounting media yields random deflections that may be indistinguishable from low-intensity peaks of the diffraction pattern. Specimens large enough to be packed, pelleted, or slabbed are quasi-infinite in thickness and thus mask the background contributed by the holder or support. However, where samples are very small, any contribution from the substrate may add significantly to the background scattering.

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We have borrowed a technique from electron microscopy (Fig. 1) to solve the scattering problem in a manner that compares favorably with the use of a quartz plate. The powdered specimen is supported on, under, or between membrane substrates made from any of several plastics. These materials are transparent to x-rays when fashioned into very thin films. A flat aluminum sample holder with a rectangular opening, such as those regularly supplied with diffractometer equipment, can be used as a supporting frame for the membrane. A special holder, however, with an oversized opening gives superior results. The special holder has a narrow rim which lies almost entirely outside the primary x-ray beam, particularly at low 2θ angles.

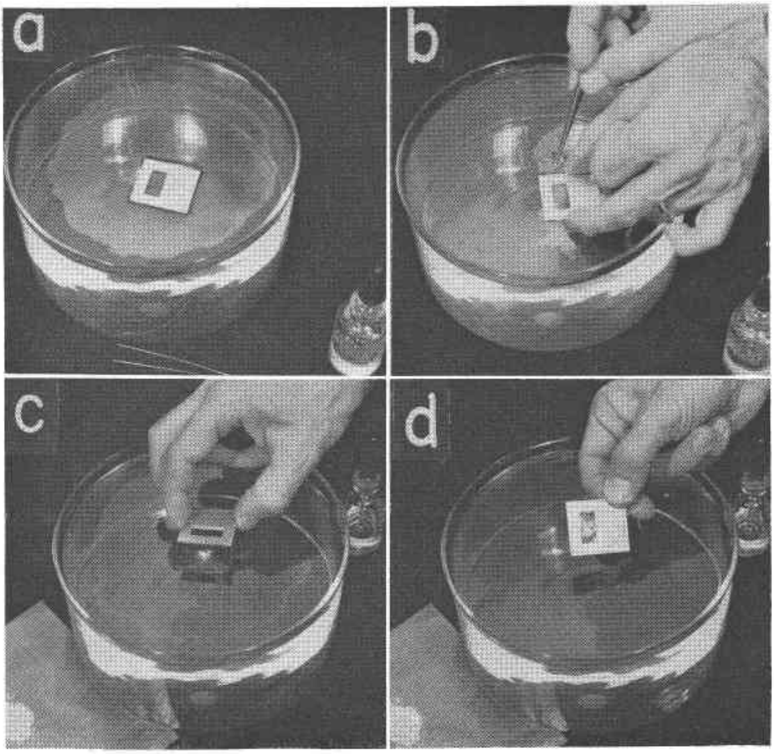

FIG. 1. Photographs of materials and method for preparing membranes used as sample substrates for diffractometer.

a. A diffractometer sample mount placed on floating membrane.
b. Removal of excess membrane.
c. Powdered sample placed on second membrane.
d. Completed mount with sample between first and second membranes.

Membranes are quickly and easily made from plastics such as collodion dissolved in a lyophobic solvent. Accordingly, water-miscible alcohols and the like are not suitable. The following list of plastics has been tried, and all have been found equally adaptable:

Plastic	*Solvent*	*Setting time (on water)*
Ethyl cellulose	Toluene	2 to 4 minutes
Ethyl cellulose	Toluene and ether (1:1)	30 to 60 seconds
Collodion	Ether	5 to 10 seconds
Collodion	Amyl acetate	1 to 2 minutes
Parlodion	Amyl acetate	1 to 2 minutes

The mixtures of plastic and solvent must be thin enough for a drop to flow freely from an ordinary dropping pipette (medicine dropper). Empirical factors which can be readily adjusted by the user govern the amounts of plastic and solvent to be mixed. Similarly, the size of drops needed for a membrane is quickly determined by a few minutes of experimentation.

One or more drops of the plastic mixture dropped in quick succession onto a water surface will spread out into a thin film. This film will "set" to form a membrane as the solvent vaporizes—the elapsed time depending on the volatility of the solvent, the temperature of the room, and temperature of the water. One can determine when the membrane has set and estimate its thickness, from the appearance of the interference colors produced by the film. Some membranes may crinkle slightly; but usually this does not affect the mount, since the membranes will stretch taut in the holder as they dry. If the wrinkles remain and are pronounced, it will be necessary to repeat the mounting procedure.

Gently place a sample holder, top surface down, against the membrane so as to cover the opening (Fig. 1-*a*). For best results this operation should be performed when the film is still slightly tacky, although the intersurface cohesion may make a good bond even when the membrane has set. Trim or tear away the surrounding floating membrane while it is still on the water (Fig. 1-*b*) so as to avoid flipping the excess onto the mount. Slip the holder out of the water sideways with a minimum of downward pressure on the stretched membrane. Shake the water off gently and dry the mount. Heat from a steam bath or from an infra-red lamp will hasten drying though excessive heating may rupture the film.

The sample may be placed on the membrane in several ways. The sample may be dusted or sprinkled on top. A water suspension may be dried on top of the membrane. The latter procedure, however, may cause the substrate to rupture. The sample may be spread on the membrane prior to attaching the membrane to the sample holders. This yields a sample suspended under the film.

A preferred technique is to sandwich the sample between two films. The thick under-membrane is prepared and mounted on a holder. Next a thin membrane is spread on the water and the sample placed on this surface (Fig. 1-*c*). The holder with the relatively thicker substrate is positioned over the sample and then the sample and second membrane are picked up as described above. The sample is thus held securely in the correct geometric position with respect to the top of the flat holder surface (Fig. 1-*d*).

From the preceding, it can be seen that no special equipment, and no more dexterity nor skill are required than one needs for the usual methods of sample preparation. Although the membrane is fragile when compared with other mounts, it has sufficient strength to support a static load of well over one gram. This is adequate for the purpose when one considers that the 10 by 20 mm. cavity in the regular holders can be packed to hold from 0.3 to 0.5 gm. of powder. Samples sprinkled or dusted on or under the substrate can be readily recovered by gentle brushing. "Sandwiched" samples can be easily salvaged, by dissolving and rinsing away the plastic with acetone.

BT-cut quartz plates were not available for testing, but AT-cut plates were obtained and tried as a substitute. Reflections from the $(10\bar{1}0)$ and $(10\bar{1}1)$ planes are present as small broad peaks in one orientation. When the plate is turned 90° in the plane of its surface, very strong reflections from these planes are produced.

In Fig. 2 are shown comparisons between diffractometer patterns recording the background scatter at constant machine settings from various substrates. The first four sets of patterns (Fig. 2*a* to *d*) are a related series of records of background scatter only. Three traces are on each diagram—the "instrumental background, *x*-rays off" is the lowest trace present in each figure. The other two traces compare the significant results of the investigation. It should be noted that the hump at low 2θ angles (Fig. 2-*b*, *c*) is contributed by white radiation scattered from the portions of the aluminum holder that are in the *x*-ray path. This is

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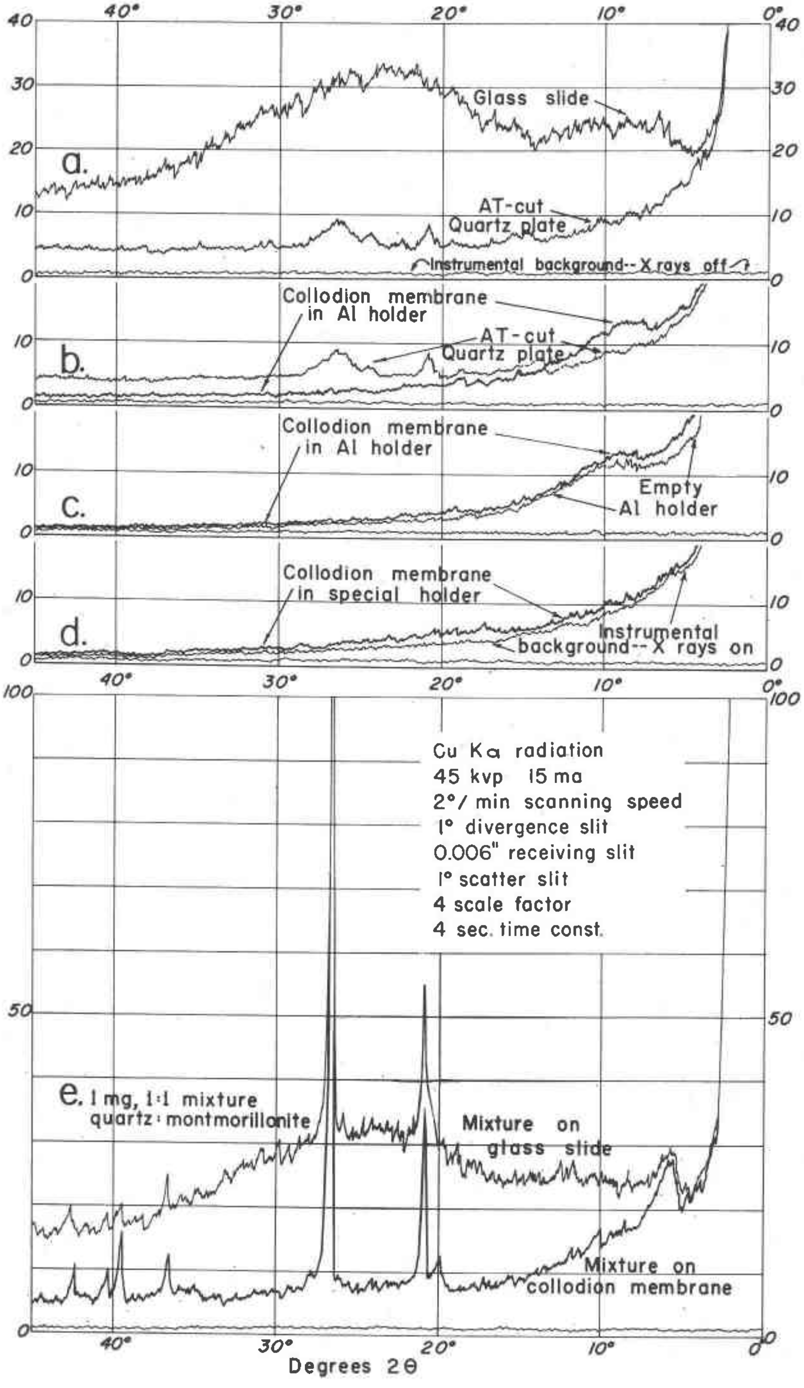
FIG. 2. Diffractometer patterns of the background scattering from various sample substrates (a, b, c, d) and the patterns of a mixed sample on a glass slide and on a membrane (e).

a. Glass slide and AT-cut quartz plate.
b. AT-cut quartz plate and membrane in stanoard Al holder.
c. Membrane in standard holder and empty holder.
d. Membrane in oversize holder and air scatter (no holder).
e. 1:1 (by vol.) quartz:montmorillonite on glass slide and on membrane in standard holder.


40°
30°
20°
10°
0°
40
30
20
10
0
a.
Glass slide
AT-cut
Quartz plate
Instrumental background-- X rays off
b.
Collodion membrane
in Al holder
AT-cut
Quartz plate
c.
Collodion membrane
in Al holder
Empty
Al holder
d.
Collodion membrane
in special holder
Instrumental
background-- X rays on
100
50
Cu Kα radiation
45 kvp 15 ma
2°/ min scanning speed
1° divergence slit
0.006" receiving slit
1° scatter slit
4 scale factor
4 sec. time const.
e. 1 mg, 1:1 mixture
quartz : montmorillonite
Mixture on
glass slide
Mixture on
collodion membrane
Degrees 2Θ

eliminated in Fig. 2-*d* by the use of a holder with a larger opening and with very little metal bathed by the primary *x*-ray beam.

The final display (Fig. 2-*e*) is a comparison of the record from a milligram of a 1:1 mixture by volume of quartz and montmorillonite. This amount of material is about half the amount of powder shown being mounted in Figure 1-*c*, *d*. The excessive "hash" in the glass-slide record masks the (02) montmorillonite band at 20° 2θ and yields many random deflections that might be erroneously interpreted as true diffraction maxima.

APPENDIX

Materials required for this method:

1. Collodion or similar plastic dissolved in a lyophobic solvent.
2. Dropper bottle.
3. Petri dish or similar container filled with water.
4. Tweezers, toothpick, or any other instrument for trimming away excess membrane.
5. Standard diffractometer sample holders.

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THE MORPHOLOGY OF ESKOLAITE, Cr_2O_3

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Kouvo & Vuorelainen* described in 1958 the new mineral eskolaite, natural Cr_2O_3 isotypic with Al_2O_3 and Fe_2O_3, forming idiomorphic crystals up to about 5 mm. in size, from Outokumpu, Finland. The authors mention that the prismatic or platy crystals show the forms: $\{11\bar{2}0\}$, $\{0001\}$, and $\{11\bar{2}3\}$, but the rhombohedrons $\{01\bar{1}2\}$ and $\{10\bar{1}4\}$ which should be dominant, were not observed.

The Institute of Mineralogy of the Technical University of Berlin got some well developed eskolaite crystals from the type locality, with a length up to 12 mm. (Fig. 1). They have, in addition to the reported forms, also the rhombohedron *r* $\{10\bar{1}1\}$ in the combinations of Fig. 2. Our indices are derived from the morphological axial ratio $c/a = 1{,}3715$ as used for Al_2O_3 and Fe_2O_3 in Dana (System of Mineralogy 1944) and in Strunz (Mineralogische Tabellen 1957).

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* Kouvo, O. and Vuorelainen, Y. (1958), Eskolaite, a new chromium mineral. *Am. Mineral.*, **43**, 1098–1106.