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REPORT ON PHOTOGRAPHIC METHODS OF URANIUM DETERMINATION RELEASED

A report describing several methods of photographic determination of the presence of uranium-bearing minerals in rocks and ores has been placed in open file today, Secretary of the Interior Oscar L. Chapman announced today. This work was completed as a part of the program on radioactive materials undertaken by the Geological Survey on behalf of the Atomic Energy Commission.

The photographic methods used by Survey Geologists to examine radioactive materials are described in detail. Paths traversed by charged particles (alpha particles) that have been emitted by uranium and its daughter products are recorded as black tracks in special photographic emulsions and are studied under a microscope.

This type of analysis gives information on the location and concentration of radioactive elements which can be obtained in no other way. The report specifically describes some new techniques in preparing different types of photographic emulsions for use in the study of radioactive rocks, minerals, and ores.

Entitled "The preparation of nuclear-track plates and stripping films for the study of radioactive minerals," and prepared by L. R. Stieff and T. W. Stern, the report has been placed in open file for public inspection in the Geological Survey Library, Room 1033, General Services Administration Building, Washington, D. C.

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GEOLOGICAL SURVEY

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THE PREPARATION OF NUCLEAR-TRACK PLATES AND STRIPPING FILMS
FOR THE STUDY OF RADIOACTIVE MINERALS

by

L. R. Stieff and T. W. Stern

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THE PREPARATION OF NUCLEAR-TRACK PLATES AND STRIPPING FILMS
FOR THE STUDY OF RADIOACTIVE MINERALS

by

L. R. Stieff and T. W. Stern

Abstract

Some modifications of current techniques for preparing nuclear plates for mineralogical studies are described. A method of mounting nuclear-track stripping film on thin sections is given. Illustrations are presented of the application of these techniques to the study of the radioactivity of powdered rocks, minerals, or thin sections.

Introduction

Since Becquerel's discovery of radioactivity in 1896 by the exposure of an unactivated uranyl sulfate phosphor to a photographic emulsion, the photographic method has become a valuable tool in the study of radioactive minerals. Photographic emulsions may be used in three ways to recognize radioactive minerals and to study the distribution of radioactivity in rocks. In addition photographic emulsions have been used in many specialized mineralogic studies, not discussed in this report, such as the semiquantitative determination of both uranium and thorium in the same rocks or minerals (Coppens, 1950, p. 21; Picciotto, 1949, p. 80; Yagoda, 1949, p. 160), the departure of uranium minerals from radioactive equilibrium (Yagoda,

1949, pp. 101, 182), the artificial autoradiography of minerals (Goodman and Thompson, 1943, p. 456), and the natural fission rates and neutron fluxes in uranium ores (Yagoda and Kaplan, 1949, p. 702).

Of the three ways of using photographic emulsions for the study of radioactivity in rocks the autoradiographic method is the oldest and simplest. A polished rock or mineral section is placed in contact with the emulsion of a photographic plate sensitive to either light or X-rays. After proper exposure and development, the radioactive minerals are recognized by comparing the pattern on the photographic plate with the polished specimen. This method, however, is severely limited by the difficulties encountered in the precise registration of the developed autoradiograph and the radioactive minerals in the specimen. Because of this shortcoming and because this simple method has been fully described by Yagoda (1946a, p. 87) and others, no further elaboration of this technique is given herein.

The dispersed-grain method of Tyler and Marais (1941, p. 146) and of Yagoda (1949, p. 176) has solved the problem of registering the developed alpha or beta tracks with the radioactive grain, which emitted the energized particle, by entrapping powdered rock or mineral fragments in a water-softened emulsion. This method uses special photographic plates covered with thick, fine-grained emulsions (nuclear emulsions) capable of recording the trajectories of alpha and beta particles emitted by both natural and artificial radioactive elements. Although this technique provides a means for recognizing radioactive grains, obviously it can give little information on the spatial distribution of the radioactive minerals in the rock. In spite of this draw-

back, this technique has many important uses, and the modifications of the method developed in this laboratory are described below.

The stripping-film method has been used in tracer studies of histological thin sections by Pelc (1947, p. 749), Doniach and Pelc (1950, p. 184), Boyd and Williams (1948, p. 225), MacDonald, Cobb, Solomon, and Steinberg (1949, p. 117), and Heller (1950, p. 8), but, to our knowledge, stripping film has not been used previously for geological studies. The use of stripping film permits positive recognition of radioactive grains, and the permanent registration of the emulsion and the thin section also provides a means of studying the spatial distribution of the radioactivity in the rock. The method of mounting stripping films on thin sections of rock as developed in this laboratory is described below.

This work was done as part of the program the U. S. Geological Survey is conducting on behalf of the U. S. Atomic Energy Commission.

Acknowledgments.—We wish to acknowledge the assistance of Dr. Herman Yagoda, National Institute of Health, U. S. Public Health Service, who reviewed the manuscript and made valuable suggestions throughout the work. We are indebted to the following members of the Geological Survey: F. S. Reed and John Mergner for preparing thin sections; Irving May for assisting us in selecting the best bonding material; Theodore Botinelly, J. H. Eric, M. N. Girhard, John C. Rabbitt, and Jane Titcomb for reviewing the manuscript.

Dispersed-grain Method

Type of plate.---The alpha-sensitive nuclear-track plates (NTA) used in the dispersed-grain method may be obtained from the Eastman Kodak Co., and the Heinicke Instrument Corp., of Rochester, N. Y. (distributors of both Eastman and Ilford nuclear emulsions), and from Ilford Ltd., and Kodak Ltd., in England. These plates are available in various sizes from $1\frac{1}{4}$ by 10 inches to 8 by 10 inches. Emulsions are available in thicknesses of 25, 50, 100, and 200 microns. An emulsion thickness of 50 microns is recommended for mineralogical work because the maximum range in the emulsion of the most energetic alpha particles does not exceed 50 microns. From 30 to 45 days should be allowed for the delivery of these plates.

For this study NTA plates $1\frac{1}{4}$ by 10 inches with an emulsion thickness of 50 microns were used. These plates are sensitive to light and should be handled under a $7\frac{1}{2}$ -watt safelight with a Wratten series OA filter. The plates, emulsion side down, are cut into $1\frac{3}{4}$ -inch lengths by means of a small plate-cutting jig shown in figure 1. A Goodell-Pratt wheel-type glass cutter is used to score the glass side of the plate. After the plate is scored, it is advanced and broken over the end of the jig. An identifying number is then inscribed on the glass side of the plate with a diamond-pointed pencil.

When studying rocks containing trace amounts of uranium and thorium, it may be desirable first to remove the tracks that have accumulated since the film was manufactured. These tracks are due to traces of

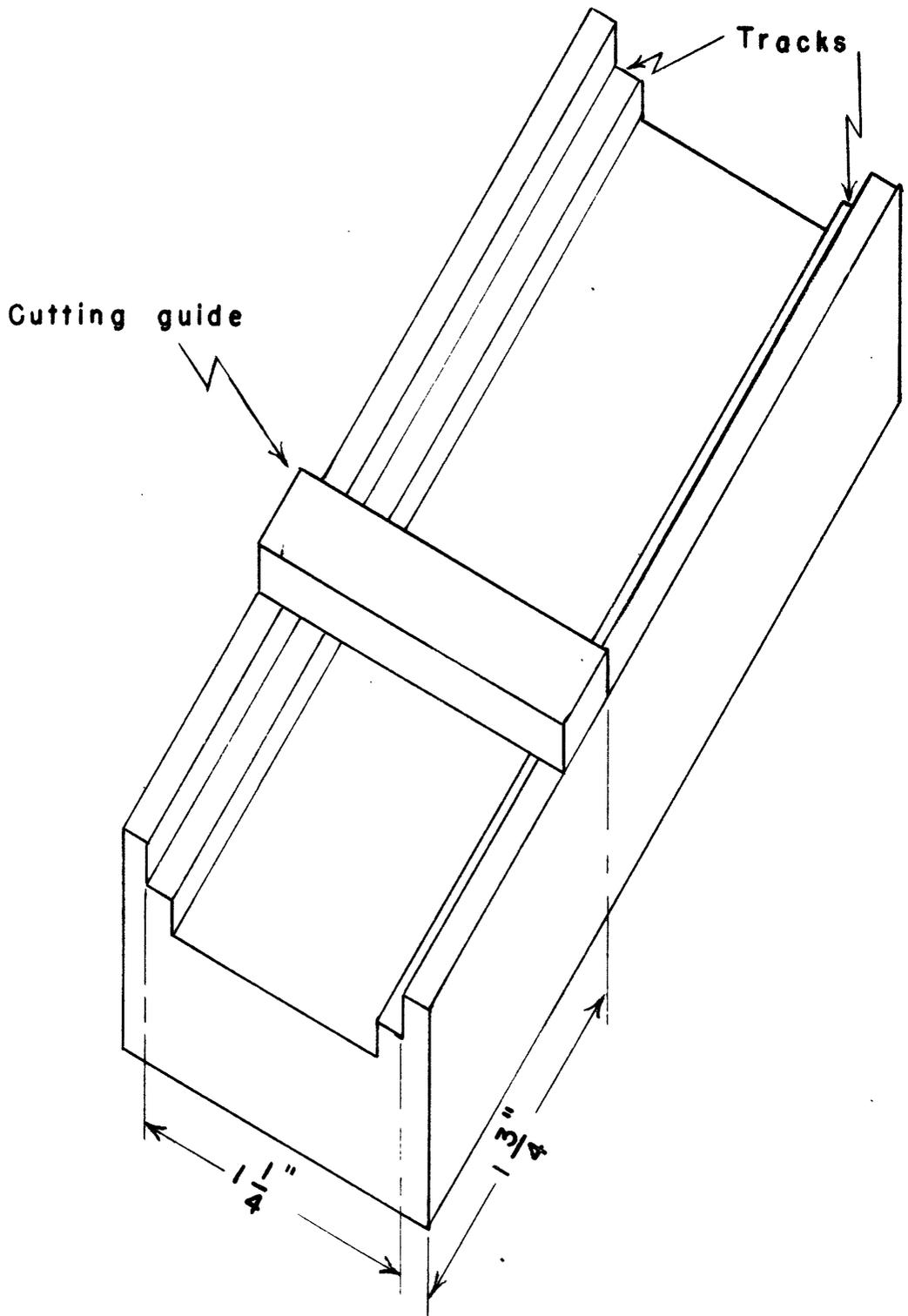


Figure 1.— Alpha-plate cutting jig

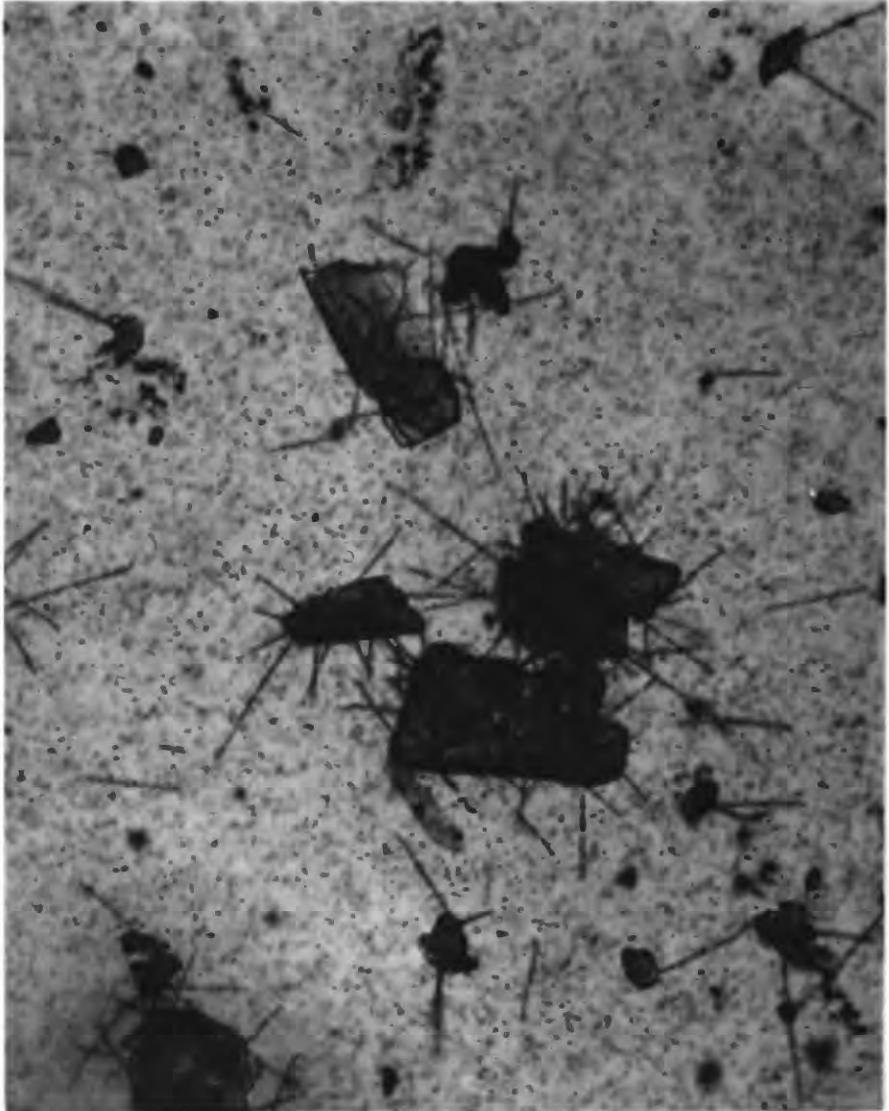
uranium and thorium in the emulsion. Yagoda (1946a, p. 94; Wiener and Yagoda, 1950, p. 39) has found that the background tracks may be eradicated by placing the plates in a lighttight container over a dilute solution of hydrogen peroxide.

Sample preparation.--Usually specimens to be studied by this method should be ground and sized. Sieve sizes from minus 80 to minus 200 mesh have been used with success (pl. 1). If necessary, the sample may be further separated by magnetic or heavy-liquid methods and the resulting separates placed on the nuclear-track plate. Also chemical separation of the sized material before dispersal is possible. After the sample is prepared, the sized fractions should be stored in a closed container for approximately one month to allow the radium and its daughter products in the sample to return to radioactive equilibrium. Approximately 20 mg of the sized material is dispersed in 10 ml of distilled water in a capped 50-ml bottle.

Plate preparation.--A 6-cm petri dish is filled with 10 ml of distilled water at room temperature. The 1 3/4-inch alpha plate is placed emulsion side up in the petri dish and allowed to soak for five minutes. The emulsion has then softened sufficiently to trap the dispersed material which now is added near the edge of the petri dish. Care must be taken not to pour the dispersed material directly over the plate as this loads it unevenly. The plate is now covered with approximately 8 mm of water. The time required for the sample to settle out depends on the distribution of particle sizes in the

Plate 1. A photomicrograph of crystalline "carnotite" dispersed on an NTA plate. The tracks in the emulsion diverging from the carnotite grains are the paths taken by the alpha particles emitted by uranium or its radioactive daughter products. Alpha plate exposure, 5 days. Magnification, approximately 600 X.

PLATE 1



sample and can be calculated from Stokes' law. Five minutes settling time usually is sufficient for all but the finest-sized samples. The plate, now being loaded with approximately 0.007 mg of sample per sq mm, is carefully removed from the petri dish with tweezers, and placed to dry, emulsion side up, on a flat surface. When the plate has dried, it should be stored in a lighttight box. If it is necessary to control the loss of daughter products by emanation from hydrous minerals, the lighttight box may be placed in a closed container. If prolonged exposure (1,000 hours or more) is necessary, the background fog may be minimized by placing the box containing the plate in a refrigerator (Picciotto, 1949, p. 77).

Exposure.--The length of exposure is related to the following: the uranium or thorium content of the sample, the degree to which the uranium or thorium is in radioactive equilibrium with its daughter products, the size and shape of the grains, and the manner in which the radioactivity is distributed in the sample. Generally, a minimum of two diverging alpha tracks is necessary to establish the radioactivity of a grain. Small pitchblende grains can be recognized easily after a 72-hour exposure, but the very fine grained radioactive segregations in phosphate rocks require an exposure of at least 1,000 hours. If the length of exposure required for satisfactory results with a mineral grain is unknown, several slides should be prepared at the same time and developed at different intervals. Yagoda (1949, p. 177) has calculated that "a fragment measuring 10 microns in average diameter, containing about 50 per cent uranium, will record about 20 tracks

per day of exposure. The isolated grain contains less than 10^{-8} g of uranium"

Development.--After exposure the plate is developed from 2 to 4 minutes at 68° F. in full strength Eastman Kodak D-19 developer (or Ilford, ID-19). This type of development shows the alpha particle energy-loss rate by changes in the grain spacing of the track. However, if only track counts are to be made, the plate should be developed for 2 minutes at 68° F. in Eastman Kodak D-8 developer diluted two to one (Webb, 1948, p. 519). Alternate methods of development are given by Yagoda (1949, p. 60). The plate is then rinsed for 10 minutes with two changes of water. The plate is fixed in Eastman Kodak F-5 fixer or 30 percent hypo for twice the time required to clear the emulsion and rinsed for 15 minutes with three changes of water. Finally, the plate is allowed to drain and dry thoroughly in a dust-free atmosphere.

Plate covering.--The dispersed-grain methods of Tyler and Marais and others do not include covering the alpha plates. We have found, however, that the addition of a cover glass serves two purposes. First, the alpha plate becomes a permanent mount and may be used repeatedly without danger of dislodging the grains. Second, as most microscope objectives are designed to be used with covered specimens and as alpha plates generally are studied under high-power magnification, the use of a cover glass results in a marked improvement in the resolution of the tracks and mineral grains.

A clean glass cover slip, 22 mm square, is mounted on the alpha plate with two drops of canada balsam in xylol. The plate is now kept in an oven in a horizontal position for 48 hours at a temperature of

approximately 140° F. After removal from the oven, the plate is allowed to cool before it is examined or stored.

Microscopic examination.--The alpha tracks emitted by the radioactive grains are studied by focusing the microscope down into the emulsion (pl. 1). Although some alpha tracks may be seen at magnifications of approximately 100 diameters, quantitative track counts cannot be made successfully at magnifications of less than 500 diameters. Detailed studies of track length and grain spacing should be made at magnifications in excess of 1,000 diameters.

For rapid surveys of the alpha plates a binocular metallographic microscope is used. These surveys are made with transmitted light, a 40 X objective, and 15 X planar oculars. A 95 X oil-immersion objective (N.A. = 1.25) is used with the same oculars for detailed track and mineral studies. A larger field can be obtained by using a Bausch and Lomb 40 X fluorite oil-immersion objective (N.A. = 1.00). For identification of the radioactive minerals a petrographic microscope is employed.

If track-count studies are to be made on previously identified mineral grains, eye strain can be reduced by the use of a green monochromatic filter or by dark-field illumination.

Advantages and limitations.--The dispersed-grain method may be used to recognize the radioactive minerals of a powdered rock sample and may aid in their identification. If alpha plates are made under controlled conditions, departures from radioactive equilibrium and the approximate uranium and thorium content may be estimated. Radio-

colloids, the localized concentrations of short-lived daughter products (Yagoda, 1946b, pp. 462-470), may be studied easily. Alpha plates also may aid in the selection of samples for lead-uranium-ratio age determinations.

This method is limited to minerals that are not water soluble and that are not attacked by photographic solutions. Also, this method can give little information on the spatial distribution of the radioactive minerals in the rock.

Stripping-film Method

Type of film.--Nuclear track stripping film consists of a 150-micron supporting film to which has been bonded a 10-micron cellulose ester film covered with a 10- or 25-micron nuclear emulsion sensitive to alpha and beta particles (NTB). (See fig. 2A.) These NTB stripping films may be obtained from the Eastman Kodak Co. and the Heinicke Instrument Corp. of Rochester, N. Y. Stripping film is at present available only in boxes containing ten 4-by-5-inch sheets. Approximately 60 days should be allowed for delivery of this film.

Film preparation.--NTB emulsions are sensitive to light and should be handled before development under a 25-watt safelight equipped with a Wratten series 2 filter. A sheet of stripping film, measuring 4 by 5 inches, is cut with a print trimmer into eight pieces, each 1 by $2\frac{1}{2}$ inches.

Two cuts, parallel to the length and one-eighth of an inch from each edge, are made through the emulsion and cellulose ester film with a razor blade and straight edge. (See fig. 2B.) As the film is pressure

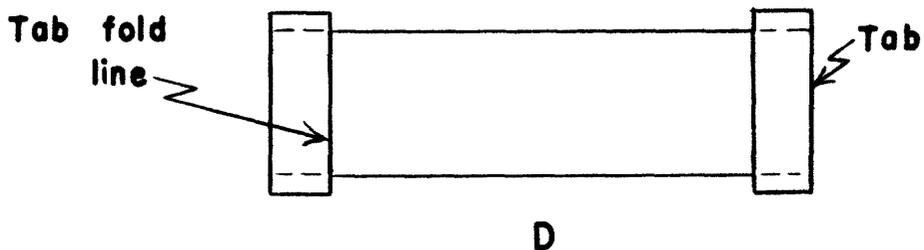
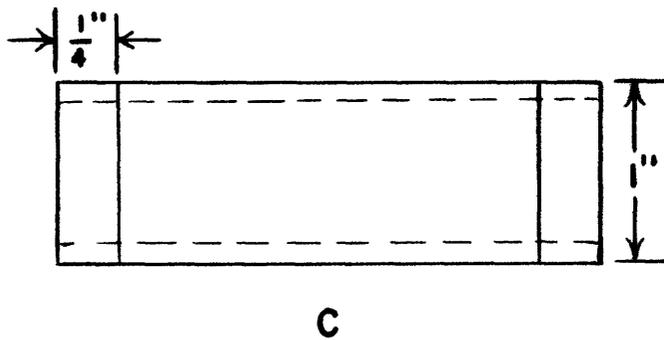
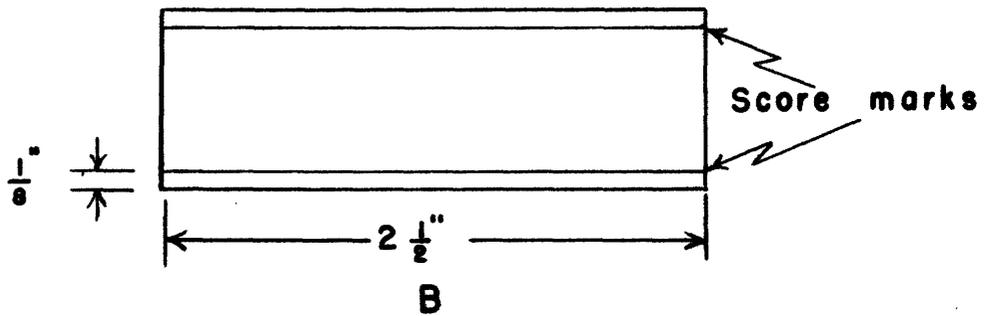
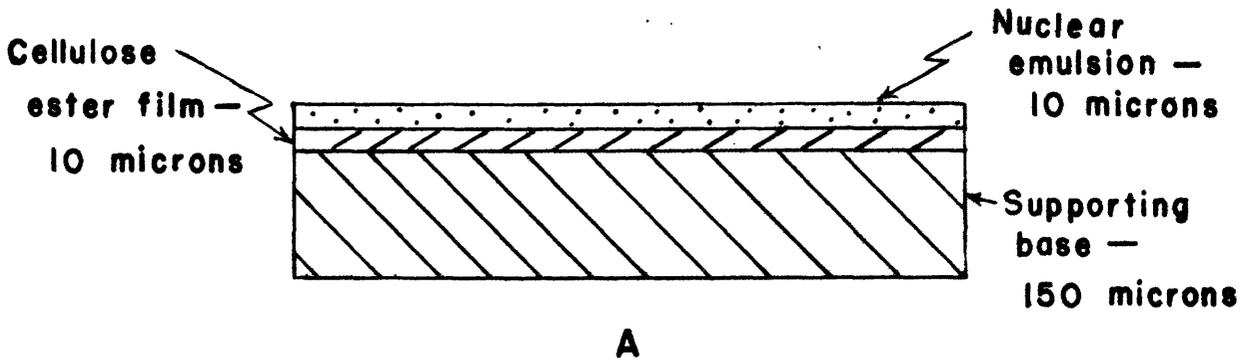


Figure 2.— Nuclear-track stripping film cross section and preparation. (A) Cross section. (B) Film, emulsion side up. (C) Film, supporting base up. (D) Unsupported emulsion.

sensitive, care must be taken to avoid touching the part of the emulsion between the score marks. The razor should not cut through the 150-micron supporting base. We have found that the tendency of the film to tear during the stripping process is minimized if the film has been scored as described above.

Approximately half an inch of one end of the film then is placed, emulsion side down, on a small wooden block and a cut 1 inch long and approximately a quarter of an inch from the end of the piece of film is made with a razor blade and straight edge (fig. 2C). This cut is deeper than the $2\frac{1}{2}$ -inch score marks and should go about halfway through the supporting base. A similar cut is made on the other end of the film.

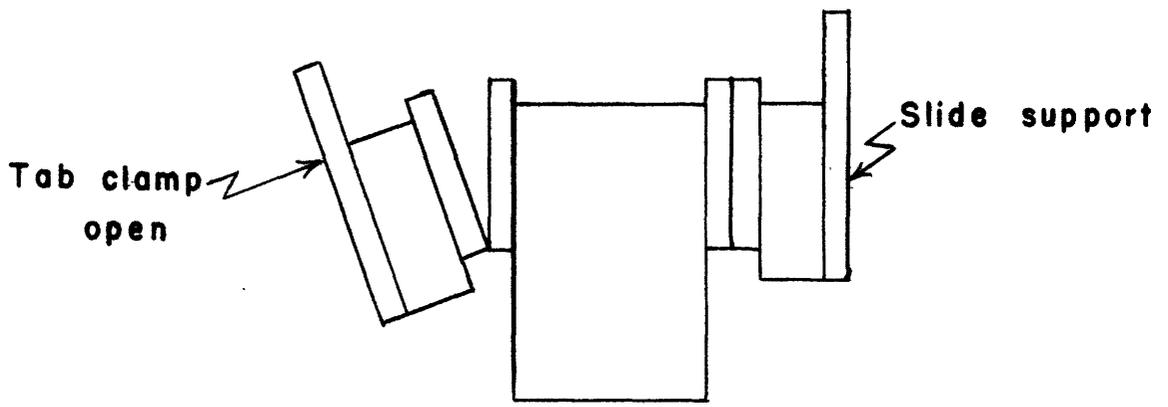
The film is held by the edges, emulsion side down, and the supporting base is broken carefully along the one-inch cut, bending the tab towards the emulsion. This operation is repeated for the other end of the film. With the film held in the left hand and the tab in the right hand, gently start to strip the emulsion and the cellulose ester film from the supporting base. The emulsion and cellulose ester film should be freed from the supporting base all along the one-inch cut before repeating this operation for the other end of the film. The film is now ready for stripping. Holding the film with the left hand, emulsion side toward the operator, slowly pull the tab downward, freeing the emulsion and cellulose ester film from the supporting base. If the film begins to tear, the tear usually will not cross the lengthwise score marks. If the film is stripped without difficulty, the 1/8-inch ribbons can be removed with tweezers. (See fig. 2D.) It is essential

that the film be stripped slowly to prevent fogging by static electricity. It has been found that the film can be stripped more easily on days of high relative humidity. When the relative humidity is low the film should be placed in a box with a piece of moistened blotting paper for about 15 minutes before the above operation is started.

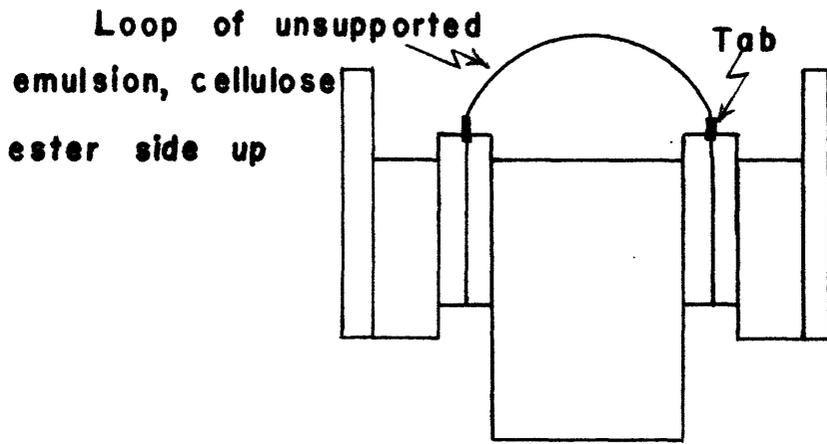
Stripping-film jig.--The jig used to hold the stripping film during mounting is shown in figure 3A. This jig may be made of metal, wood, or glass slides, and its dimensions depend on the size of the specimen to be covered. A rubber band, not shown in figure 3, holds the two tab clamps firmly closed. Tweezers are used to place the tabs in the tab clamps of the jig. The loop of film is mounted in the jig with the cellulose ester side up. The loop is centered in the jig and, if necessary, adjusted by means of the tabs until the reflection of the safelight across the top of the loop is parallel to the ends of film jig slide supports. In this position the stripping film is ready to be mounted on the thin section. (See fig. 3B.)

Preparation of thin sections.--Thin sections may be prepared in the usual manner but with the cover glass omitted; however, special precautions against contamination must be taken with samples containing small amounts of uranium. The use of disposable "metallographic" emery papers to minimize contamination has been described by Yagoda (1949, p. 39). The rock slice may be mounted on the glass slide with either canada balsam or Lakeside 70.

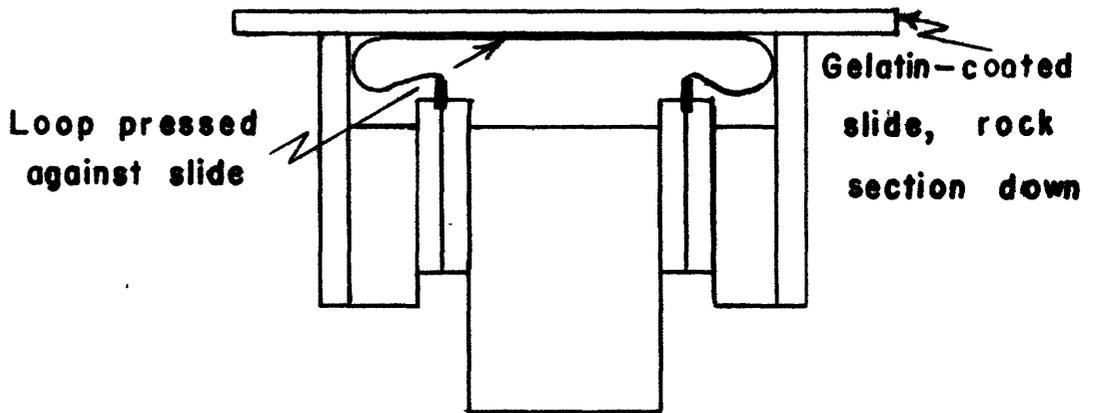
In order to allow the thin section of the sample to return to its original radioactive-equilibrium condition and to minimize the loss of



A



B



C

Figure 3.— Jig for mounting nuclear-track stripping film. (A) Film jig. (B) Film mounted in jig. (C) Slide resting on slide supports.

daughter products by emanation from hydrous minerals, the thin section should be stored for approximately one month in a desiccator.

Covering thin section.--The uncovered thin section is first dipped in a solution of gelatin at approximately 150° F. This gelatin solution is prepared by placing 12 g of gelatin (Eastman Kodak flake gelatin, item no. P1099) in 150 ml of distilled water, allowing the gelatin to hydrate for 30 minutes, adding 2.5 ml of 2 percent chrome alum, and adding 5 ml of 0.2 percent aqueous wetting agent (Yagoda, 1949, p. 95) such as aerosol. The solution then should be gently heated. The gelatin is kept in a tightly stoppered wide-mouth bottle on a hot plate except when the slide is being immersed. When not in use, the gelatin solution is stored in a refrigerator to inhibit bacterial growth. To minimize contamination, very low grade samples should not be dipped in the same gelatin solution that is used for high-grade samples.

After dipping, the slide is allowed to drain for approximately one minute to remove excess gelatin. It is ready to be covered when clear finger-print impressions can be made in the gelatin film on the back of the slide. The slide, rock section down, is centered over the loop of film and gently lowered until the slide rests on the film jig slide supports. (See fig. 3C.)

The slide is chilled by placing a small ice-filled bottle or chilled metal bar on the slide. This rapidly sets the gelatin and prevents the film from pulling away from the thin section during the next operation. The film is cut with a scalpel along the tab fold lines, freeing the film loop from the tabs. After the ice-filled bottle is removed, the

slide is taken from the jig. It is placed, thin section up, on the table, and the excess film is removed with a razor and straight edge. If for any reason the slide has been covered imperfectly, the stripping film may be removed by soaking the slide in warm water. A solution of Eastman Kodak rapid mounting cement and ethyl acetate diluted one to three is used to paint down and secure the edges of the film to the slide. The slide is then stored in a lighttight box for exposure.

If the operator is not familiar with stripping film, it is suggested that several practice mounts be made in daylight before attempting this method under darkroom conditions.

Exposure.--The same general factors governing exposure discussed in the dispersed-grain method apply to the stripping-film method. It should be mentioned, however, that because of the relatively small amounts of material represented by a thin section of a radioactive grain, the exposure time may be increased by a factor of three or four over the exposure used for the dispersed-grain method. The optimum exposure time of any particular type of material can be determined only by the exposure of several slides of the same material. If for any reason the exposure was incorrect and another thin section cannot be obtained, the stripping film may be removed from the thin section. This is accomplished by first gently rubbing the cemented edges with a cotton swab dipped in ethyl acetate and then placing the slide in warm (110° F.) water for about 10 minutes.

Development.--The development recommended above for nuclear-track plates may also be used for NTB stripping films. Movement of the strip-

ping film due to shrinkage of the emulsion during development and fixation has not been noticed.

Microscopic examination.---In contrast with the dispersed-grain method, the alpha tracks emitted by radioactive grains are studied by focusing up into the emulsion. (See plates 2 and 3.) The combination of objectives and oculars used on alpha plates may be used with equal success on stripping film. For oil-immersion work a drop of immersion oil is placed directly on the developed emulsion. The oil is removed by wiping the slide gently with a soft absorbent cloth or tissue. To remove the last traces of the oil, the emulsion is cleaned with a drop of xylol on an absorbent tissue. Because the cellulose ester is isotropic, the determination of the optical properties of the mineral grains in the thin section is made without difficulty.

The use of a vertical illuminator with a metallographic microscope has aided the study of alpha tracks that have been emitted directly over opaque grains. The peripheral tracks emitted by opaque grains can be studied easily by transmitted light.

Advantages and limitations.---The stripping-film method represents a marked improvement over the mechanical methods of registering autoradiographic plates and thin sections (Poole and Bremner, 1949, p. 130), the method of correlating track patterns with grain shape (Coppens, 1950, p. 21), and the method of simultaneously exposing to light both the alpha plate and thin section (Picciotto, 1949, p. 79). The use of stripping film assures a positive registration between the developed film and the radioactive grains. Studies have been made that suggest that the

Plate 2. A photomicrograph of a euhedral grain of "uranothorite" in fluorite covered with NTB stripping film. The "uranothorite" grain is surrounded by a composite halo consisting of a deep-purple zone and an outer bleached zone. The alpha tracks are most apparent where they cross the bleached zone. The tracks directly over the "uranothorite" grain are so numerous that the mineral seems to be almost opaque. The tracks crossing the purple zone cannot be seen by transmitted light. Stripping film exposure, 13 days. Magnification, approximately 600 X.

PLATE 2

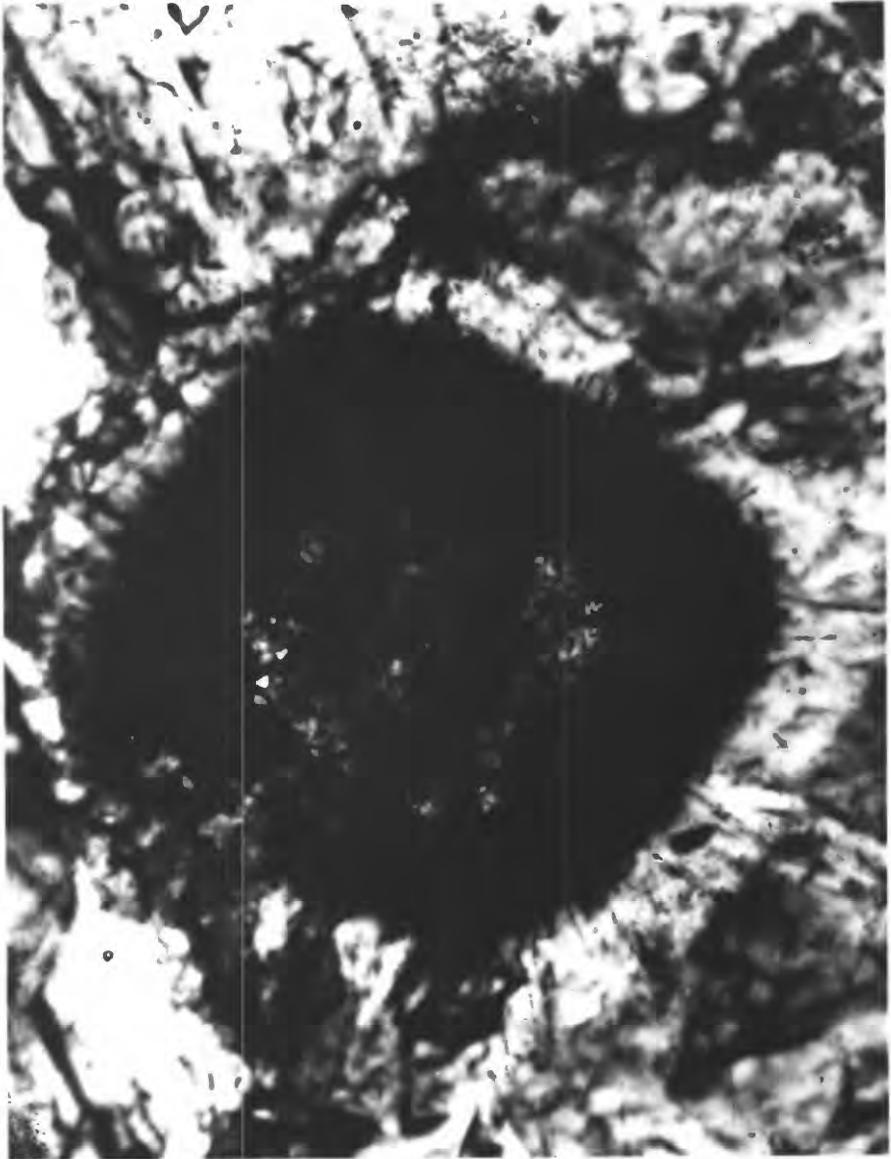
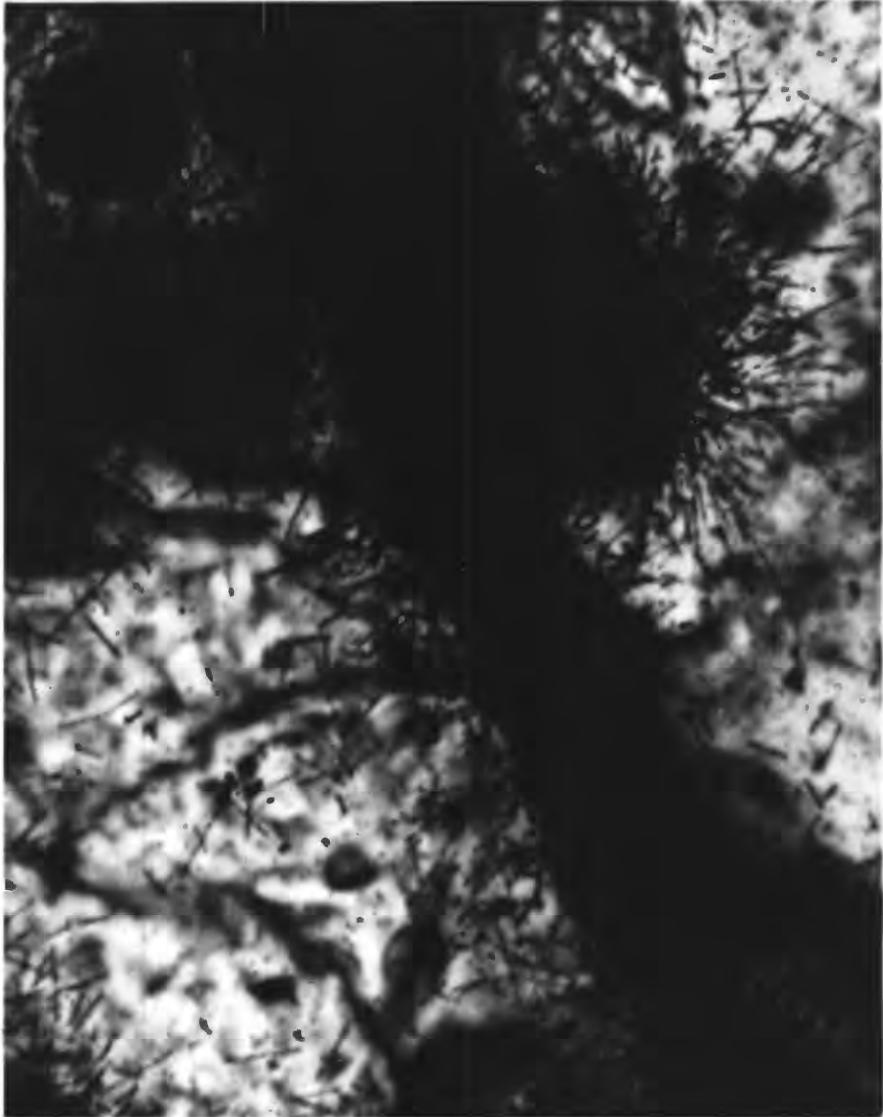


Plate 3. A photomicrograph of carnotite-bearing sandstone covered with NTB stripping film. The dark band diagonally crossing the field is interstitial microcrystalline carnotite. The light areas on either side of the dark band are quartz grains. The burst of alpha tracks on the upper right is probably a radiocolloid, that is, a local concentration of radium and its daughter products. Stripping film exposure, 5 days. Magnification, approximately 600 X.

PLATE 3



cellulose ester film protects the thin section from chemical attack by either the developer or hypo and minimizes the loss of daughter products by emanation. It should be mentioned that great dexterity is not required to cover thin sections with nuclear-track stripping film. After several trials in daylight the average operator can make satisfactory mounts under darkroom conditions.

The inherent limitations of the stripping-film method are a consequence of the methods of preparing the thin sections. Radioactive minerals may be lost easily in grinding and, if precautions are not taken, the slide may be contaminated by the re-use of grinding powders and rouges. A thorough study of the distribution of radioactivity in a rock can be made only by examining many thin sections. At least one week's exposure is required before any results can be obtained.

We believe that the stripping-film method, which has been described above, may be used without modification for histological studies and with some modifications for polished-section and metallographic studies.

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