ETCH TESTS ON CALAVERITE, KRENNERITE, AND SYLVANITE

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This paper is supplementary to a comprehensive study of the gold and silver tellurides which has been carried on by Tunell and Ksanda and which is still in progress. The complex crystallography of calaverite has been thoroughly described by Goldschmidt, Palache and Peacock (1931). That of sylvanite and krennerite has long been known.

Owing to the close similarity in physical and chemical properties between calaverite, krennerite, and sylvanite, authentic specimens of any particular one of these three minerals are difficult to obtain. The resemblance between calaverite and krennerite is especially close and one cannot be certain of the identity of either of these except by measurement on the reflection goniometer or by *x*-ray study.

Authentic crystals of all three minerals were supplied by Prof. Palache and Dr. Peacock. These crystals were subsequently investigated structurally by Tunell and Ksanda by means of the Weissenberg *x*-ray goniometer (1935, 1936, 1937).

As crystallographic and *x*-ray investigations are somewhat complicated and the necessary equipment is not at the disposal of most workers, it seemed desirable to supplement these studies by a determination of etching and other tests usually made under the reflecting microscope. Accordingly, crystals of the three tellurides were mounted in bakelite in known orientations and polished in the Laboratory of Economic Geology at Harvard University under the direction of Dr. Tunell, and these specimens were lent by him to the present writer for the purpose of mineragraphic study.

The reagents commonly used in routine mineragraphic tests are 1:1 HNO_3, 1:1 HCl, 20 per cent KCN, 20 per cent $FeCl_3$, 40 per cent KOH, and 5 per cent $HgCl_2$. Of these reagents only nitric acid yields definite etch patterns on the above-mentioned tellurides. Previous etch tests made by Dr. Tunell indicated that concentrated nitric acid gives better results than the 1:1 reagent, hence the writer decided to conduct tests with nitric acid of both strengths.

The time of etching is an important factor in the results produced. The standard practice in routine mineragraphic tests is to etch one minute with each reagent. As this practice has long been established as regards 1:1 HNO_3, it was decided to choose this time for this strength of acid. On the other hand, owing to the stronger action of concentrated acid, one minute is too long, as an etch cleavage which may form with a shorter etch period, may be destroyed by etching as long as one minute.

After some experimentation, 40 seconds was the period chosen for etching with concentrated acid. The results obtained on each specimen by etching with 1:1 acid and concentrated acid respectively were totally different. This was a surprise and led to further experimentation with acid of strength midway between the two above-mentioned strengths, i.e., 3:2 HNO_3 (three parts by volume of conc. HNO_3 added to two parts of water). The time of etching with this reagent was 40 seconds. The results of these tests are shown in Figures 1 to 7.

Calaverite. Two specimens were investigated. One specimen shown in Figures 1*a*, 1*b*, and 1*c*, is sectioned in the orthodome zone. The other shown in Figures 2*a* and 2*b* is sectioned parallel to the side pinacoid. When these polished surfaces are etched with 1:1 acid no etch cleavage is apparent until the drop is removed by squirting a stream of water against it. When the water is removed and the surface dried, the surface of the specimen breaks up into irregular areas, each consisting of a flake a few microns thick. These tend to curl at the edges, giving the specimen somewhat the appearance of shingles on an old roof. In the orthodome section, the flakes seem to show no parallelism (Fig. 1*a*). In the side pinacoid section, the parallelism is pronounced (Fig. 2*a*). The surface of the specimen beneath these flakes is not rough, as when chalcocite is etched, but is apparently smooth. (The white areas between the flakes in Figures 1*a* and 2*a* show the unetched under-surface.)

Apparently the surface layer acts as a coating protecting the layer beneath it, in a manner somewhat analogous to the human epidermis. When this layer is removed by polishing, the former under-layer now becomes the surface layer ("epidermis") and reacts to etching in exactly the same way as the first "epidermis."

Small circular areas scattered irregularly over the surface have resisted the etching in part. These circular areas deserve further study in relation to the problems of the peculiar morphology of calaverite first described by Penfield and Ford and the adventive diffraction spots found by Tunell and Ksanda, since these resistant areas may indicate some kind of inhomogeneity. They are not visible on an unetched surface. These circular areas were too small to test separately. They do not appear when the specimen is etched with concentrated acid.

With 3:2 acid, a distinct parallel etch structure is developed (Fig. 1*b*). The areas between the etch partings tend to break off irregularly giving an appearance similar to boards on the side of an old house. The circular areas already mentioned have escaped corrosion. With concentrated acid a much more uniform etch cleavage is developed (Figs. 1*c* and 2*b*). From the excellent development of etch cleavage on the side pinacoidal section, it is probable that this cleavage is parallel to some face in the orthodome zone.

Krennerite. Two specimens of krennerite were available. One was sectioned parallel to the base (001). As stated by Tunell, this is analogous to the face (010) of calaverite. The results of etching this surface are shown in Figures 3*a*, 3*b*, and 3*c*. The other specimen is sectioned parallel to the plane (430) in krennerite (this plane is structurally analogous to the plane (100) of calaverite). The results of etching this specimen are shown in Figures 4*a*, 4*b*, and 4*c*. With 1:1 acid flaking is pronounced in both specimens. In the basal section two directions at right angles are distinctly discernible (Fig. 3*a*). In the other specimen the flakes are entirely irregular (Fig. 4*a*).

With 3:2 acid the two directions of etch cleavage are more pronounced in the basal section. Circular areas of apparently unetched material are scattered irregularly over the surface. Examined with a higher power, the circular areas likewise show a rectangular etch pattern, but on a much more minute scale.

When etched with concentrated acid the rectangular pattern is no longer evident on the basal section (Fig. 3*c*) but is pronounced on the other section (Fig. 4*c*). The circular areas disappear when the specimen is etched with concentrated nitric acid.

Sylvanite. Three specimens of sylvanite were studied. One sectioned parallel to (010) is shown in Figures 5*a*, 5*b*, and 5*c*. The second specimen, sectioned parallel to the *b*-axis, is shown in Figures 6*a*, 6*b*, and 6*c*. The third, sectioned in a random direction, is shown in Figures 7*a*, 7*b*, and 7*c*.

Sylvanite differs from calaverite in that when etched with 1:1 nitric acid the etching appears while the drop is still in contact with the surface (i.e., the acid drop does not have to be removed and the surface washed with water and dried before the etching appears).

With 1:1 acid the etching differs with the orientation. In Figure 5*a* the etching takes the form of short discontinuous cracks which give the surface a vermicular appearance. Most of these are parallel, but a small percentage show some divergence from parallelism. With 3:2 acid this effect is exactly reproduced on this specimen.

In Figure 6*a* two cleavages at right angles are shown. The vertical is more prominent than the horizontal. With 3:2 acid the two etch directions are more clearly brought out and seem to be of equal importance.

The effect of time on etching is brought out in Figures 5*b* and 5*c*. Both show etching of the same specimen with concentrated HNO_3; Figure 5*b* shows the result of 10 seconds etching, and Figure 5*c* shows another place on the same specimen etched 40 seconds. In Figure 5*b* only one direction is brought out. In Figure 5*c* two directions are shown. One shows prominent wide cracks relatively few in number. These probably correspond to the cleavage shown in Figure 5*b*. The other cleavage is less prominent but the cracks are closely spaced and much more numerous than the first set.

In Figure 6*c* two cleavages at right angles are shown. These seem to be about equally prominent, but the specimen seems to show some distortion, probably the result of crushing during the mounting in bakelite under pressure.

Summary. The results of etching the three tellurides are not as simple and conclusive as had been hoped for. The variations in results when different strengths of acid and different times of etching are employed call for control of both factors. On the other hand, the etch patterns are not capricious; each figure illustrates several experiments performed under the same conditions, and the conclusion is justified that, under the same conditions, a given specimen will yield the same etch patterns.

Calaverite and krennerite give similar patterns, but krennerite will give two etch-cleavages at right angles in certain sections whereas calaverite apparently will give an etch-cleavage in only one direction. Both krennerite and calaverite flake, and many specimens of both minerals develop circular areas that may represent spherical inclusions when etched with 1:1 HNO_3. Etch-cleavage in one direction indicates, but does not prove calaverite.

Sylvanite gives an etch-cleavage similar to that of krennerite but the cleavages develop while the drop is on the specimen and the surface does not flake when washed and dried.

In conclusion, etching with both 1:1 and concentrated nitric acid will usually lead to a decision whether an anisotropic gold and silver telluride is sylvanite or one of the other two minerals, calaverite and krennerite. It is difficult to distinguish calaverite from krennerite by etching with nitric acid in some cases, but if two etch-cleavages at right angles are developed the mineral is probably krennerite.

References

Goldschmidt, V., Palache, C., and Peacock, M. A. (1931): *N. Jb. Min.*, B.B. **63**A, 1–58.

Tunell, G., and Ksanda, C. J. (1935): *Jour. Washington Acad. Sci.*, **25**, 32–33.

——— ——— (1936): *Jour. Washington Acad. Sci.*, **26**, 507–409.

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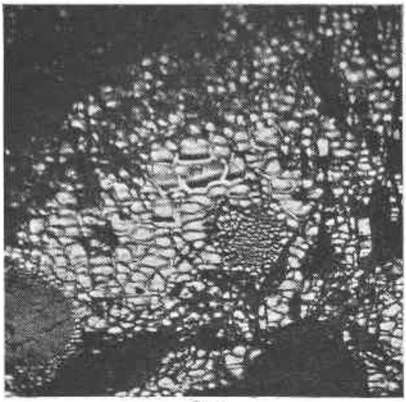
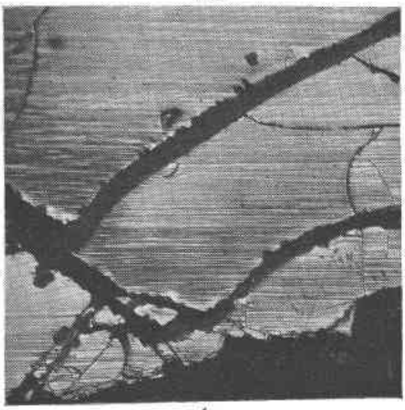
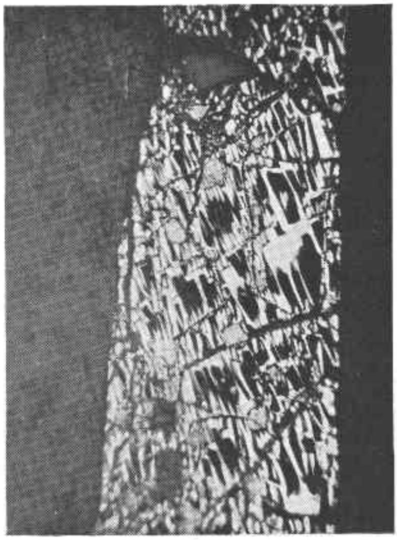


1a

1b



1c



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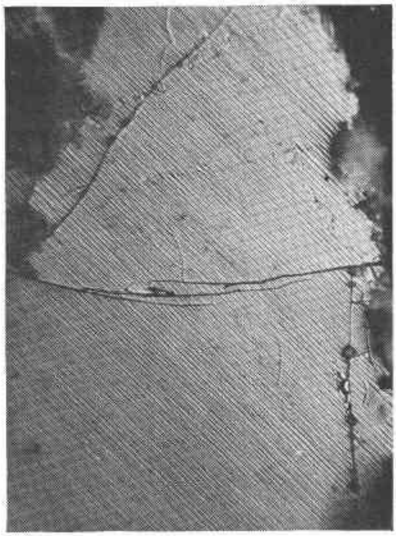


2b

PLATE I

Etch Cleavage on Calaverite

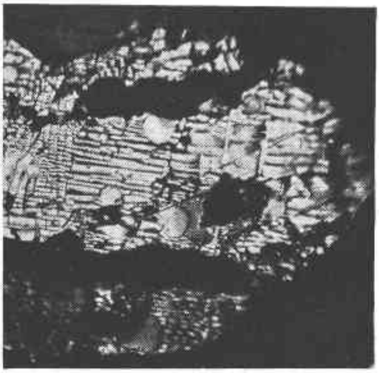
FIG. 1*a*—Calverite, Cripple Creek. Section parallel to b-axis (i.e., in orthodome zone). Section etched 60 seconds with 1:1 HNO_3.—×100

FIG. 1*b*—Same specimen as 1*a*. Section etched 40 seconds with 3:2 HNO_3.—×55

FIG. 1*c*—Same specimen as 1*a*. Section etched 40 seconds with concentrated HNO_3.—×55

FIG. 2*a*—Calaverite, Cripple Creek. Section parallel to 010. Section etched 60 seconds 1:1 HNO_3.—×52

FIG. 2*b*—Same specimen as 2*a*. Section etched 40 seconds with concentrated HNO_3.



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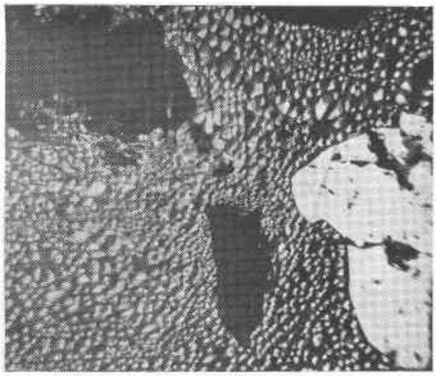
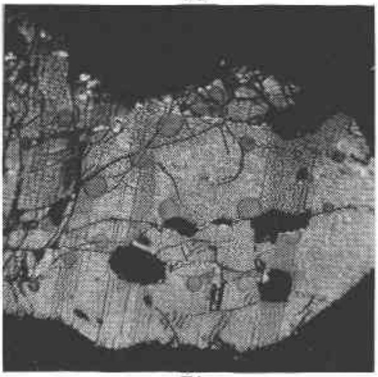


4a



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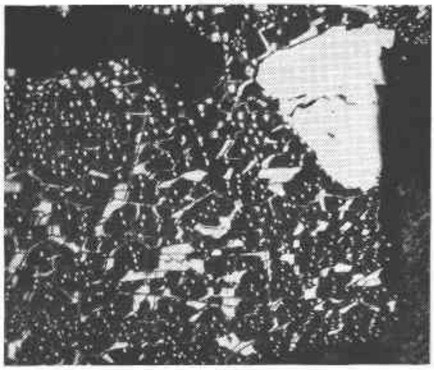

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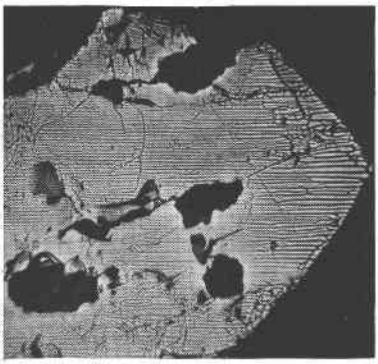

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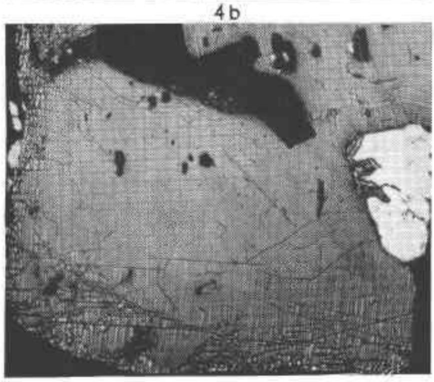


4c

PLATE II

Etch Cleavage on Krennerite

FIG. 3*a*—Krennerite, Cripple Creek. Section parallel to krennerite 001 (analogous to 010 of calaverite). Etched 60 seconds with 1:1 HNO_3.—×87

FIG. 3*b*—Same field as 3*a*. Etched 40 seconds with 3:2 HNO_3.—73

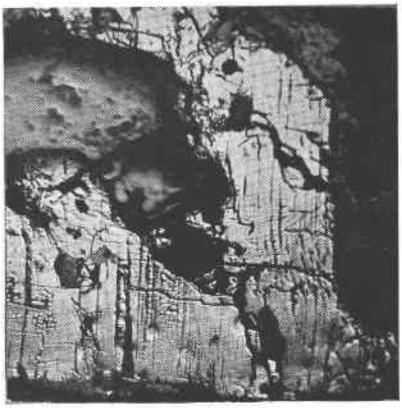
FIG. 3*c*—Same field as 3*a*. Etched 40 seconds with concentrated HNO_3.—×95

FIG. 4*a*—Krennerite, Cripple Creek. Section parallel to plane 430 in krennerite (this plane is structurally analogous to plane 100 of calaverite). Etched 60 seconds with 1:1 HNO_3. Small inclusion of tetrahedrite at center of right border of field is unetched. Tetrahedrite shows incipient alteration to covellite.—×87

FIG. 4*b*—Same field as 4*a*. Etched 40 seconds with 3:2 HNO_3. Surface breaks into irregular flakes showing no parallelism.—×73

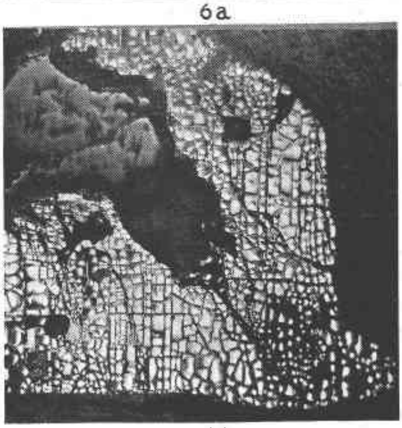
FIG. 4*c*—Same field as 4*a*. Etched 40 seconds with concentrated HNO_3.—×55

5a



6a

5b



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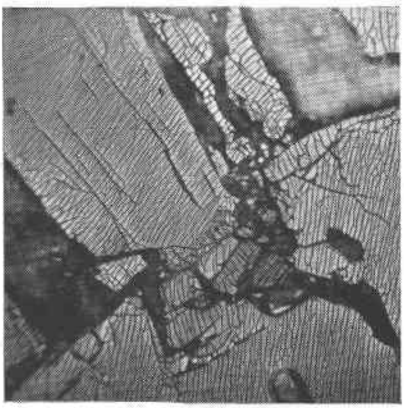

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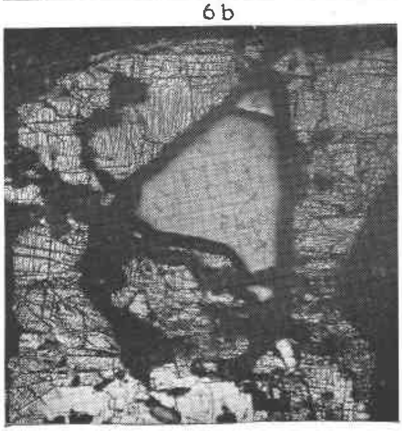


6c

PLATE III

Etch cleavage on Sylvanite

FIG. 5*a*—Sylvanite, Cripple Creek. Section parallel to 010. Etched 60 seconds with 1:1 HNO_3.—×87

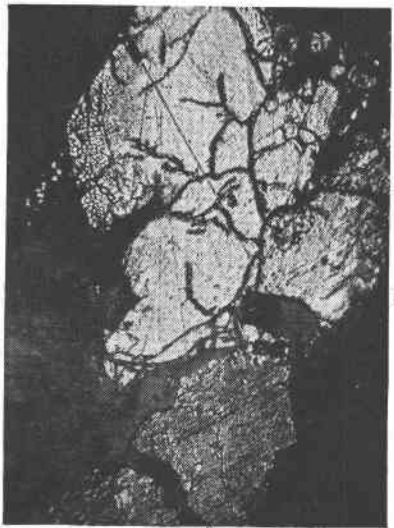
FIG. 5*b*—Same specimen as 5*a*. Etched 10 seconds with concentrated HNO_3.—×87

FIG. 5*c*—Same specimen as 5*a*. Etched 40 seconds with concentrated HNO_3.—×210

FIG. 6*a*—Sylvanite, Cripple Creek. Section parallel to *b*-axis. Etched 60 seconds with 1:1 HNO_3.—×87

FIG. 6*b*—Same field as 6*a*. Etched 40 seconds with 3:2 HNO_3.—×87

FIG. 6*c*—Same specimen as 6*a*. Etched 40 seconds with concentrated HNO_3.—×55



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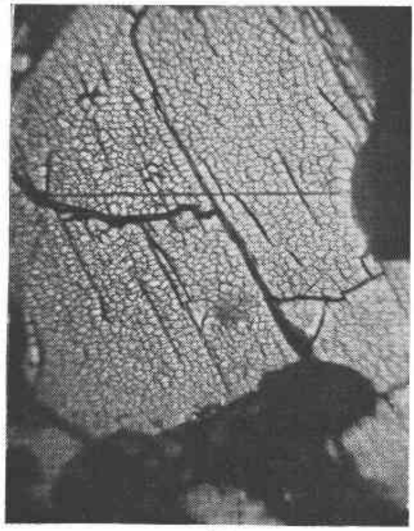
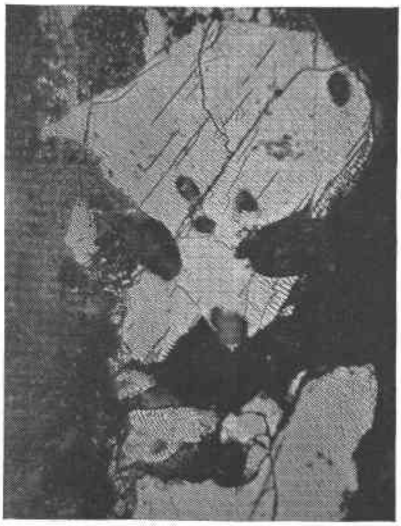


7b



7c

PLATE IV

Etch Cleavage on Sylvanite

FIG. 7*a*—Sylvanite, Săcărâmbu (Nagy-Ág), Transilvania. Random section. Etched 60 seconds with 1:1 HNO_3.—×87

FIG. 7*b*—Same specimen as 7*a*. Etched 40 seconds with 3:2 HNO_3.—×210

FIG. 7*c*—Same specimen as 7*a*. Etched 40 seconds with concentrated HNO_3.—×95