

mixture contains, or is suspected of containing, feldspars (silicates) or other minerals readily confused with quartz, it is necessary to purify it further. To accomplish this, about 10 ml. of hydrofluosilicic or fluoboric acid is poured on the sample in a small platinum crucible and it is set aside for 3 to 6 days, being stirred at least twice each day with a platinum loop. At the end of the 3 to 6 days, depending upon the gross appearance of the residue, the remaining dust is filtered off, washed, dried, ignited, and weighed. It is again inspected under the petrographic microscope in order to determine the nature and purity of the residue which should now be quartz or possibly other forms of free silica, such as chalcedony, tripoli, cristobalite, and tridymite, which dissolve in the above acids slowly or one of a few other minerals such as silicon carbide, which is not removed by these combined acid treatments but is readily distinguishable. The petrographic identification of minerals is far beyond the scope of this chapter but a simple procedure for the identification of the quartz content of dusts, utilizing petrographic technique, may be stated as follows.

Place a minute amount (few particles) of the dust residue on a microscope slide, add drop of immersion oil of refractive index 1.545, and cover with a cover slip. Observe with a petrographic microscope using crossed Nicols under about 450x magnification. If quartz is present the small particles will appear as milky white irregular shapes. To determine whether an individual particle is quartz, use the crossed Nicols, rotate the stage, and observe that quartz particles (except when oriented with optic axis vertical) disappear four times per complete revolution of the stage. This characteristic establishes that quartz particles are anisotropic as distinguished from isotropic crystals, which are invisible when viewed with crossed Nicols. Bring the stage to rest with a particle extinguished, then slide the analyzer Nicol prism out of the system, focus on the edges of the crystal, and raise the microscope tube from focus. A light halo surrounding the particle will move into or away from the crystal depending upon its orientation, because this halo (Becke line) always moves toward the medium of higher refractive index when the microscope tube is raised from focus. Now turn the stage exactly 90 degrees in either direction and repeat this procedure. If the particle is quartz the halo of light will move in the opposite direction. This is true because quartz has two indices of refraction, 1.544 and 1.553, both close to, and one on each side of, 1.545, the refractive index of the immersion oil. The refractive index of a submerged crystal may be judged to be very near that of the immersion oil if there is a lack of clear distinction of boundary lines; this is true when the difference in refractive indices is less than 0.01. When the difference is less than 0.001 a submerged crystal can be seen only with difficulty. If the particle under examination has these characteristics, that is, irregular shaped, milky white between crossed Nicols, two refractive indices one just above and one barely below 1.545, it may be suspected of being quartz. It is good practice to check a sample now with oil of n_D^{20} 1.57 to make certain that the crystals have both indices less than 1.57.

The identity of the crystals may be verified by further scrutiny. Center a particle exactly under the cross hairs and align the microscope system so that the particle does not move from position during a complete revolution of the stage. Now, by means of a Wright's diaphragm, or less satisfactorily with the Bertrand lens, if the microscope is so equipped, and no Wright's diaphragm is available—isolate this crystal and observe its inference figure.²² This test cannot be applied successfully to particles appreciably smaller than 10 μ in diameter. It is done with crossed Nicols and upper lens condenser (converger) swung into place. Upon rotation of the

²² E. E. Wahlstrom, *Optical Crystallography*. Wiley, New York, 1943.

stage, as seen across on a light field, or portions of the arms of a cross, can be seen (interference figure of uniaxial crystal). If only portions of the arms making up the cross are visible, the analyst must imagine the complete figure in order to choose the section he wishes to investigate (see Figure 10).

A crystal is rarely found oriented so that the cross is in the center of the field, as in 1 (see Figure 10); infrequently, with the axis of the cross visible at or near the circumference of the field, as in 2; but usually, as in 3, where only the arms of the cross, appearing as straight or very slightly curved bars extending perpendicular to the observer, move straight across the field from left to right, or vice versa, or extending horizontal, move from top to bottom, or vice versa. The crystal may now be further classified as positive or negative by the use of a

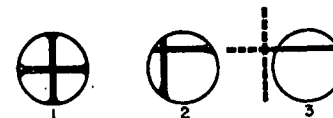


Figure 10. Interference figures of uniaxial crystals (quartz).

quartz wedge mica plate, or selenite plate. First choose the lower right quadrant, if only the arms (bars) are visible, by rotating the stage until a horizontal bar has passed across the field from the observer and a perpendicular bar is just out of view to the left. Now, as a quartz wedge is inserted into its slot as a bisector of this field, color curves will move outward if the particle is quartz (positive). With a mica plate so inserted, a black dot will appear in this and the upper left quadrant, while with a selenite plate these same areas (3 to 6, and 9 to 12 o'clock, respectively) will have a yellow tinge and the other 2 quadrants will appear blue. With mica crystals opposite results are obtained. The particle that has met these requirements may now be definitely identified as quartz because it has been found to be anisotropic, uniaxial, and with indices of refraction of barely less than 1.545 (1.544) and between 1.545 and 1.57 (1.553). No other crystalline material fits all of these characteristics.

If the sample on first observation is found to contain a large percentage of particles other than quartz (feldspars and other silicates), it is then necessary to treat with hydrofluosilicic acid. After treatment the residue is again examined and if found to be quartz, a correction of 0.5 percent per day should be added to the quartz residue to compensate for its slight solubility in hydrofluosilicic acid.

Other forms of free silica as well as all other minerals may be similarly identified by the petrographic microscope. Anyone interested in this method of identification should obtain samples of each mineral he wishes to be able to identify; pulverize some of each in a diamond mortar and practice with known pure materials until all of these characteristics are readily recognized. With the aid of samples and a table for the determination of minerals,²³ along with the good texts, the field may be broadened to include many minerals.

Sampling samples ordinarily do not have a sufficient number of particles 10 μ in diameter or larger upon which an interference figure can be obtained, and

²³ Ward's Natural Science Establishment Inc., Rochester, N. Y.

²⁴ B. S. Hart and H. Berman, *U. S. Geol. Survey Bull.* No. 848 (1934).

²⁵ W. N. Short, *U. S. Geol. Survey Bull.* No. 914, 1940. A. N. Winchell, *Elements of Mineralogy*, Wiley, New York, 1931. A. N. Winchell, *Artificial Inorganic Solid Substances and Artificial Minerals*, Wiley, New York, 1931. E. S. Dana, *A Textbook of Mineralogy*, Wiley, New York, 1932. E. M. Chamot and C. W. Mason, *Handbook of Chemical Microscopy*, Wiley, New York, 1938.