An improved water cell for the Emmons double-variation procedure

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Summary. An improved water cell for the Universal Stage is described for use in the Emmons double-variation procedure. The cell provides an increased heat flow to the mineral grains, and the technique of orientating and retaining the grains in the centre of the field of view is greatly simplified.

EMMONS' 1943 monograph on the Universal Stage included a detailed discussion of the advantages of using the double-variation procedure in refractive-index determination by the immersion method. However, while the procedure is sound in theory and workable in practice, there are a number of intricate manipulations or small irritating difficulties associated with the technique that are often sufficient to deter the beginner from using what is otherwise an extremely quick and accurate method. Many of these difficulties are associated with the design of the water cell and with the orientation of mineral grains. Emmons himself (1943, p. 18) states the position concisely ... 'Beginners frequently experience difficulty in retaining grains in place, but this difficulty is almost invariably due to insensitive fingers . . .'.

Insensitive fingers or no, the main difficulties associated with the technique can be summarized: The water cells described by Emmons, or those available commercially, substitute for the normal glass plate of the Universal Stage; in general the inlet and outlet tubes lie together on one side of the cell, thus giving rise to a complex and turbulent current in the circulating water and hence to the air bubbles that invariably accumulate in this part of the system and lie in the line of vision; clearly a through-flow system is preferable in order to remove the air-bubble irritation (cf. Vigfussen, 1940). Secondly, the recommended method for mounting mineral grains is to wedge them between two coverglasses between the water cell and the upper hemisphere; this is most unsatisfactory, since it commonly happens that the grain under observation is either crushed on tightening the upper hemisphere, or rolls out of view on tilting the stage, or is effectively smothered by other grains, which move on tilting the stage; further, the oriented grain may be disturbed whilst rotating the stage to match a second vibration direction with the liquid, or by accident; and platy or prismatic grains always lie with a strong preferred orientation using this system, thus necessitating high tilt angles with consequent risk of dislodgement. Thirdly, since the heat flow to the mineral grain takes place through two thicknesses of glass and a film of liquid, there is always some doubt as to how close the temperature of the immersion liquid approaches that of the circulating water, and hence that of the immersion liquid in the refractometer. Finally, while Emmons went to considerable pains to ensure that his range of double-variation liquids covered the refractive-index scale with a fair degree of overlap, it is often difficult, particularly with minerals of higher birefringence, to measure more than one principal refractive index using a single liquid and without going to extremes of temperature and wavelength or using a graphical technique. Mixing of adjacent liquids to overcome this difficulty is precluded owing to differential volatility, which may become marked at higher temperatures.

The present cell (fig. 1), which has been used in this department for some three years, has been designed to overcome some of these difficulties and to allow greater flexibility, accuracy, and speed in measurement. Basically it consists of a brass collar with upper and lower optical glass disks and, like other designs, replaces the glass plate on the Universal Stage. The smaller upper glass disk is inset into the collar and lies flush with its upper surface. Both disks are glued to the brass collar by Araldite epoxy resin; this gives a bond which is almost as strong as the glass itself, and adds a major improvement to the cells used two decades ago which frequently failed owing to inadequate bonding. The resin is unaffected by the normal immersion liquids or by heat.

Two identical inlet and outlet nipples are soldered on either side of the upper surface of the brass collar. Thin flexible rubber tubes carrying the circulating water project above the plane of the water cell at about 30°. These cause less obstruction during rotation or tilting of the stage than tubes carried below the stage. Brass flanges serve a dual purpose both in strengthening the nipples and in forming a groove to take the upper (rectangular) hemisphere of the stage; this renders the cell fixed and rigid when the upper hemisphere is screwed down, making it impossible to rotate it accidentally on rotation of the inner stage. Having the inlet and outlet nipples on opposite sides of the cell gives a considerably increased flow of water (over 1 litre per minute) and there is no problem of trapped bubbles.

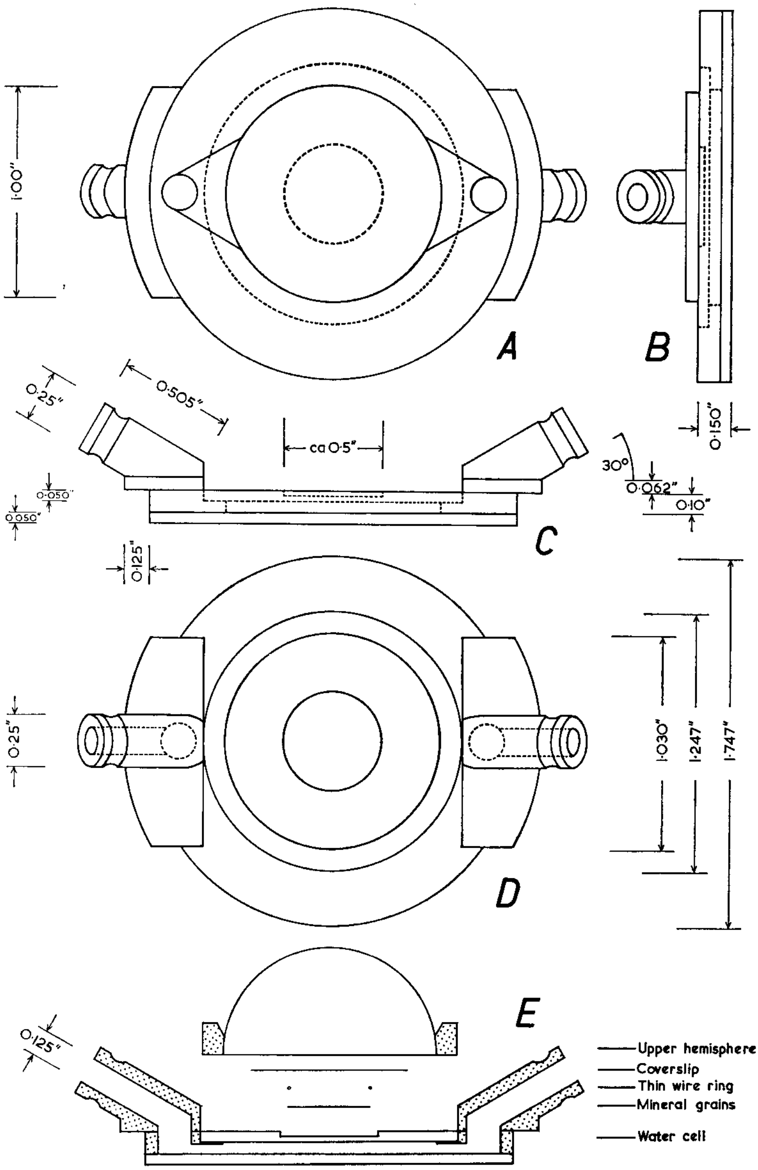

FIG. 1. Improved water cell for the Universal Stage. *a*, view of underside; *b* and *c*, side view; *d*, top view; *e*, section through water cell showing depression in upper glass disk into which fits the lower coverslip carrying the mineral grains and the circular spring clip. The depression is covered by the larger coverslip and the upper hemisphere.

A further modification to the upper glass disk greatly improves the process of mounting and orienting mineral grains in the centre of the field of view (fig. 1*e*). The disk has a circular depression *c*. 0·5 in. in diameter and 0·025 in. deep at its centre. Into this depression is placed a thin 1 cm diameter coverslip sprinkled lightly on its upper side with the mineral grains. A thin (0·01 in.) wire ring of about 1 cm diameter is placed over and around this coverslip. The depression is then filled with the immersion liquid and a wider, 0·75 in. diameter, coverslip is placed over the depression, taking care to exclude air bubbles. A further drop of immersion liquid is placed on top of the coverslip and the upper hemisphere is screwed down; this effectively seals off the depression containing the immersion liquid, and the pressure transmitted through the wire clip simultaneously makes the coverslip immobile.

The mineral grains may be fixed to the coverslip by the method described by Olcott (1960). In this procedure the upper surface of the coverslip is coated with a 10 % gelatine solution and gently warmed until hard. The surface is then lightly moistened with a water–acetone–formalin solution until tacky, the mineral grains sprinkled on top, and the gelatine again warmed until hard. This fixes each grain firmly to the coverslip by a single corner or edge.

The advantages of this modification can be summarized as follows:

The mineral grains have more or less random orientation and, when the upper hemisphere is tightened up, will not be dislodged even at high tilts; unlike earlier cells, there is no pressure on the mineral grains from the upper hemisphere.

It will be noted that the coverslip is smaller than the depression into which it fits; this allows a particular mineral grain to be centred simply by loosening the upper hemisphere slightly to reduce the pressure on the spring clip and coverslip, tilting the stage in the appropriate direction until the grain is in position, and re-tightening the hemisphere.

Since the mineral grains lie in a depression in the upper glass disk of the water cell, the heat flow to the grains is considerably increased. The temperature difference between the mineral grains in the depression and the circulating water was tested by using pure crystalline samples of ethyl carbamate (M.Pt. 46° C) and diphenylamine (M.Pt. 53° C), the melting points of these samples being tested independently using the capillary technique. A difference of only $\frac{1}{2}$° C was recorded even at the higher temperature. A secondary advantage of the system is that the time taken to reach temperature equilibrium is shortened, thus speeding up the measurements considerably.

The depression containing the immersion liquid is virtually sealed off by the upper coverslip, thus reducing volatilization of the liquid to a minimum. It is therefore possible with this modification to use mixtures of immersion liquids, provided that the film of immersion liquid in the refractometer be renewed before each measurement.

References

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