APPARATUS FOR TOTAL WATER DETERMINATION BY THE PENFIELD METHOD[1]

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The Penfield method (Hillebrand *et al.*, 1953; Kolthoff & Sandell, 1952; Penfield, 1894; Washington, 1910) for the determination of total water in rocks and minerals is a simple and reliable method well-known to rock analysts. Although more detailed and rigorous methods have been described (Groves, 1951; Hillebrand *et al.*, 1953), this method stands out in its simplicity and ease of operation.

As used in the rock and mineral analysis laboratories of the Geological Survey the method is a modified version of the original Penfield method. The sample, mixed with lead oxide (litharge) and placed in a bulb at the end of a narrow glass tube, is heated first by an ordinary bunsen burner and later by a hand torch using a fuel mixture of propane and oxygen. The water expelled from the bulb, together with other volatiles, is condensed in a cooled portion of the tube. The fused end of the tube is removed and the tube is cooled; a solid glass rod is inserted to displace gases, such as CO_2, which may be present. The tube and contents are then weighed, dried and reweighed and the difference is the weight of the total water present in the sample. Additional precautions must be taken if certain volatiles, such as fluorine, are known to be present.

It has been found convenient to use an apparatus designed to support and cool the tube during the fusion period, one that combines simplicity of construction with ease of operation. This apparatus is pictured in Figure 1; an exploded diagram is given in Figure 2. The tray holder and sides are of $\frac{1}{2}''$ plywood, while the base is made of two $\frac{3}{4}''$ plywood pieces glued and nailed together. Plywood was chosen as it provides the necessary weight required for the stability of the stand. The object of the $3\frac{1}{2}''$ wide space at the bottom half of the base is to provide room for the base of the burner during the initial stages of ignition. The heat shield is made of two $\frac{1}{4}''$ pieces of asbestos board, screwed to the front vertical support; a single sheet of $\frac{1}{2}''$ asbestos board would also suffice.

The overflow tray now in use is made of plastic in order to minimize "sweating" of the dish due to the overflow into it of ice cold water. The cooling tray which holds the mixture of water and ice is made of alumi-

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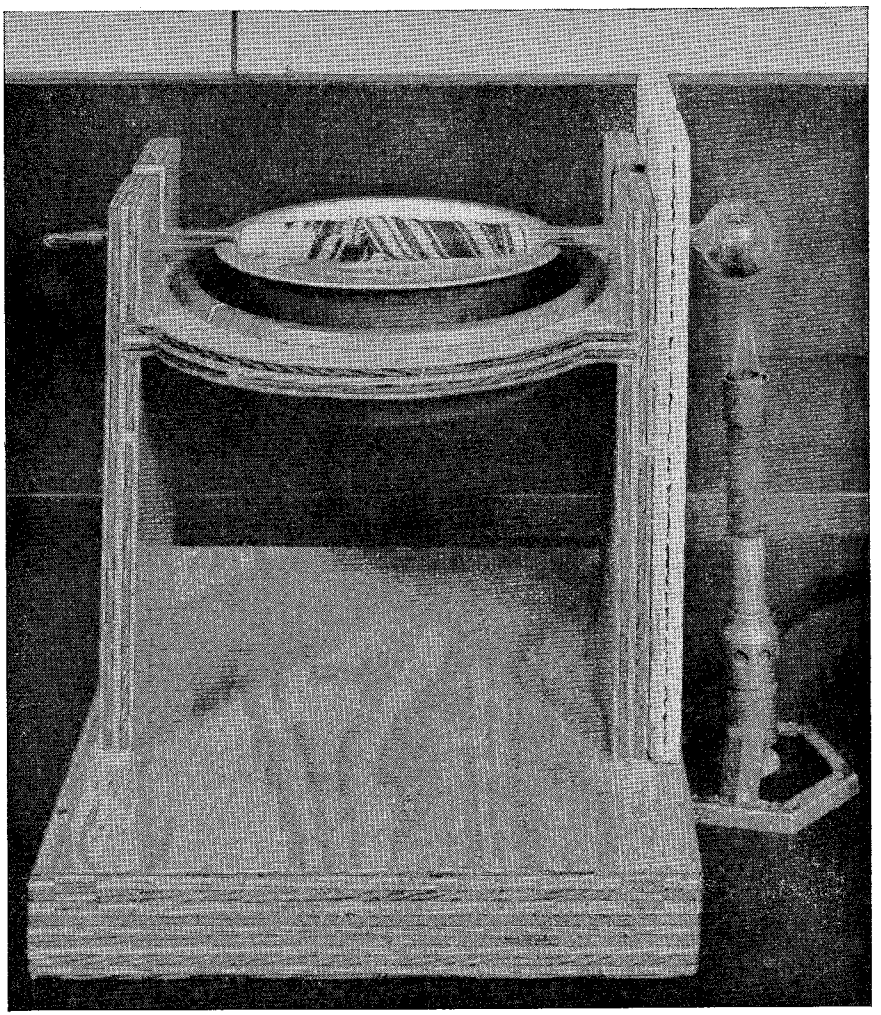

FIG. 1. Apparatus for the determination of total water by the Penfield method.

num, but could be of any other rust-resistant material. The rubber supports are made from rubber stoppers cemented to the two trays with rubber cement. The height of these supports should be such that, with the overflow tray in position, the notches in the cooling tray are level with the bottoms of the two slots in the vertical supports of the tray holder. A slight downward inclination toward the open end of the tube is recommended to prevent any condensed water from flowing back into

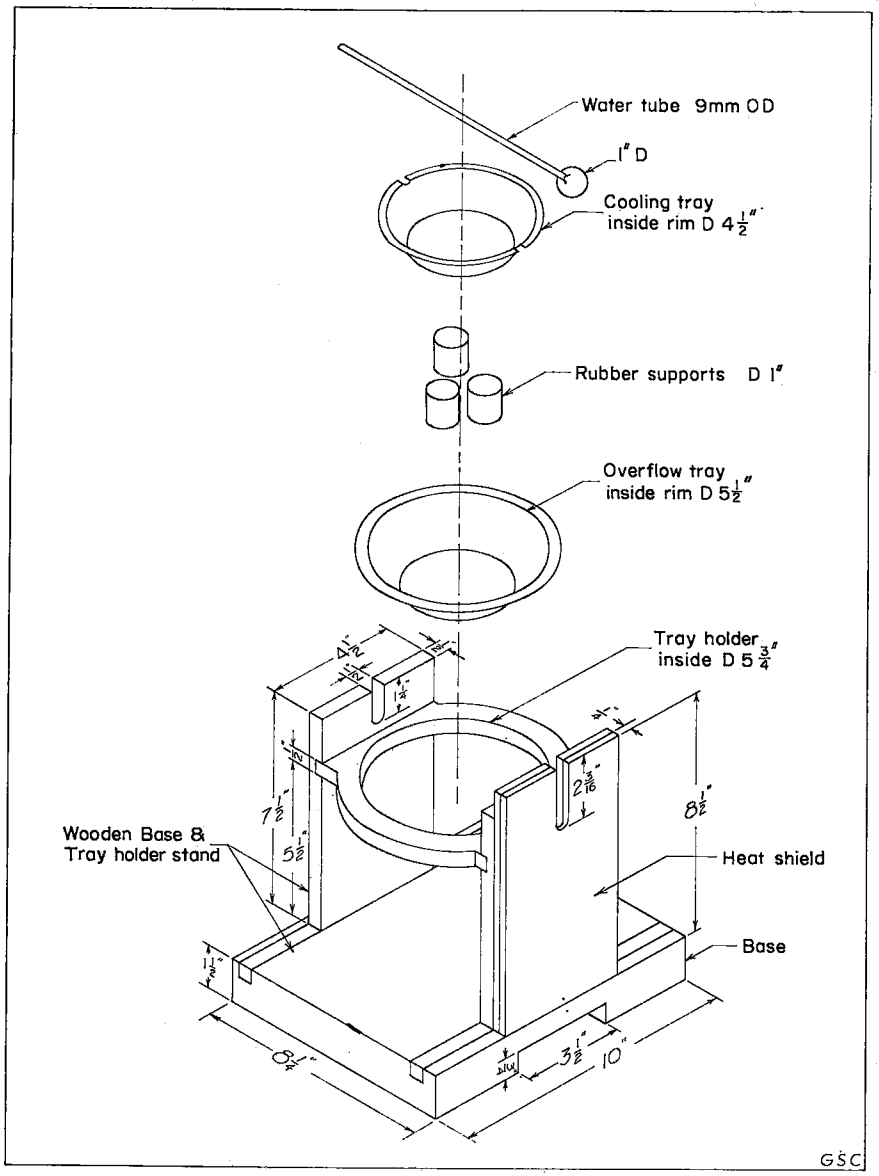

FIG. 2. Exploded diagram of apparatus.

the fusion area of the tube. During operation the centre portion of the tube is wrapped in a strip of cloth, the tube is supported in the cooling tray and slowly rotated during the heating. This serves to insure cooling

of the tube as well as mixing of the sample and flux in the bulb during ignition.

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A DIAMOND DRILL FOR CORING MINERAL SAMPLES IN THE LABORATORY

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During a current study of apatite mineralogy a large crystal was grid sampled on a basal section. Small core samples were obtained at over 100 points on a surface 11 × 14 cms. with a small coring diamond drill bit, made to specifications by J. K. Smit and Sons, 81 Tycos Drive, Toronto. The ease with which core sections were made suggests the technique will be of general value in cases where a small quantity of a rock or mineral is required for mineralogical examination or chemical analysis.

The drill bit used was 5.2 mm. in outside diameter and 3.4 mm. inside diameter, and will give a core length of approximately 1.5 cms. in one operation. Core lengths used in the present study were generally less than this, being of the order of 0.5 to 1.0 cm. (Fig. 1). The diamonds are set in a tungsten alloy matrix, and bit wear is negligible with a relatively soft mineral such as apatite.

Initially the drill bit was set in the chuck of a drill press, and water was poured over the bit to act as a coolant and wash away cuttings. This was found to be unsatisfactory since the cuttings block the inside of the bit. J. K. Smit and Sons then supplied a water swivel which fits between the