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A PRECISION METHOD FOR MEASURING TEMPERATURES OF REFRACTIVE INDEX LIQUIDS ON A CRYSTAL REFRACTOMETER AND ON A MICROSCOPE SLIDE[1]

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In connection with comprehensive investigations on portland cement, it has been necessary to develop methods of high precision for the identification and study of the several compounds and solid solutions which are encountered. This report presents such a method for the measurement of temperature in the determination of refractive indices. It is believed that the method will be generally useful to the microscopist engaged in any work requiring the accurate temperature measurement of single drops of liquid.

In checking refractive index values on liquids at intervals of a day or more, using an Abbe crystal refractometer, variations in index have been indicated which could not be ascribed to changes in the liquid itself. The values occasionally changed by as much as .0015 to .0017 on consecutive days and the variations were sometimes positive and sometimes negative in character. A careful study of the whole procedure indicated that the values used for the temperature of the liquids were subject to significant errors.

The change in refractive index for each degree of temperature change for liquids of $n=1.700$ and higher is .0006 to .0007. Therefore, if the value of the refractive index of such liquids is to be determined to the third decimal place, the temperature of the liquid must be known to about 1°, since other errors, though perhaps smaller, must be allowed for.

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The writers have found that by using a differential thermocouple, the temperature of the liquid on the refractometer can be measured with the desired accuracy. This procedure may be employed, also, to measure the temperature of the liquid while in use on a slide on a microscope thus eliminating another probable source of error in the determinations of refractive indices of solids by microscopic methods. Seidentopf,[2] in 1906, mentioned the use of a thermocouple for measuring the temperature of a liquid on a slide. The suggestion, however, appears not to have been adopted by microscopists in this country.

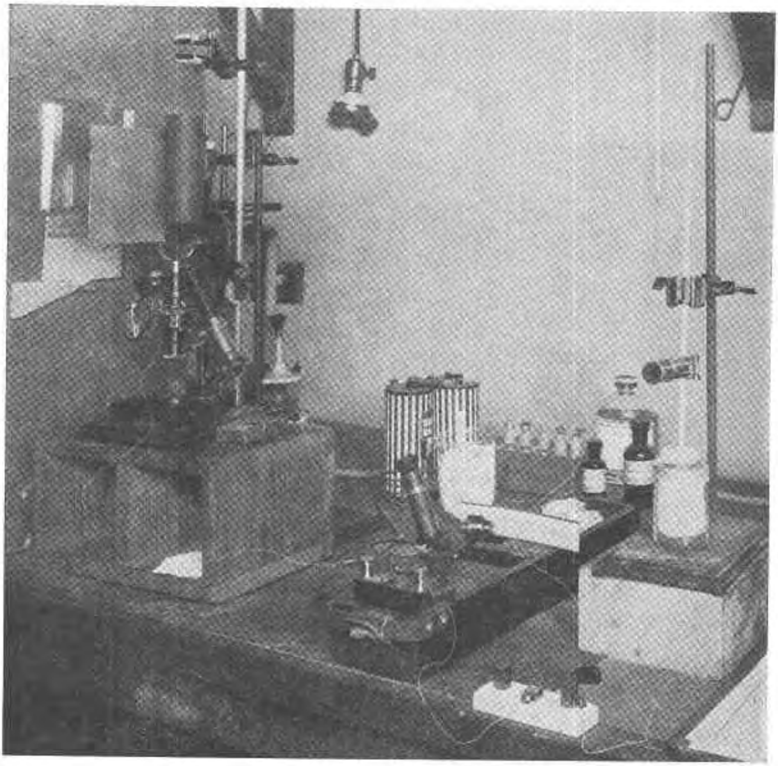

Fig. 1. Suggested arrangement showing (1) differential thermocouple with flattened junction in place on refractometer, (2) the method of protecting the thermometer bulb and its thermocouple junction, and (3) the portable galvanometer used in the work.

[2] *Z. Elektrochem.*, 12 (1906), 596.

SUGGESTED PROCEDURE

The method described below differs from those commonly used in two essential points: (1), the location and protection of the thermometer; and (2) the use of a differential thermocouple to measure temperature differences. A photograph showing the arrangement of the apparatus is reproduced in Fig. 1. The Abbe crystal refractometer (hemisphere of $N_D = 1.7972 \pm .0001$) is shielded from the Bunsen burner (the source of monochromatic light) by a wooden shield with a movable ground glass window and by a second cardboard shield[3] close to the hemisphere. The small lamp—a 6 to 8 volt Mazda bulb operated from dry cells—is a convenient light for reading the scales and gives a negligible amount of heat.

The mercurial reference thermometer used to measure the temperature of one junction is of the precision type, calibrated at several points near room temperatures. It has a temperature range of 0°–40° over a scale length of about 30 cm. and is graduated in tenths of a degree. By the aid of the small reading glass shown in the Figure, it can be read easily to 0.05°. In the arrangement of apparatus suggested, the bulb and part of the stem of the thermometer are placed in a glass jar, packed with loose cotton. This insulation is necessary to insure a reasonably close agreement in the temperatures of the thermometer bulb and the attached thermocouple junction. It also insulates the system to a considerable degree from minor fluctuations in air temperature, and makes the temperature "lag" of the thermometer more equal to the lags of the refractometer and the microscope.

The galvanometer shown in Fig. 1 is a very compact, portable instrument of the d'Arsonval type, such as is used for insulation testing. It should have a sensitivity of about 3 micro-volts per scale division.

The thermocouple[4] is conveniently made of two pieces of No.

[3] The cylindrical shield hung above the hemisphere is used only when the face of the operator is close to the liquid for some time, as then the heat from the breath causes a slight rise in the temperature of the liquid.

[4] The construction and connections of the thermocouple may be seen from Fig. 2. A and B, cotton-covered copper wire, No. 36; C, cotton-covered constantan wire, No. 36; G, galvanometer; S, switch; 1, junction to be in contact with the thermometer bulb; 2, flattened junction to be placed in liquid.

When junctions 1 and 2 are at different temperatures, completing the circuit by closing the switch (S) causes deflections in the galvanometer, which for small

36 cotton-covered copper wire silver-soldered to the ends of a similar wire of constantan, with one of the junctions rolled to a thin ribbon.

The galvanometer scale and thermocouple must be calibrated[5] for small known temperature differences (0°–5°), and then, when arranged as shown in Fig. 1, can be used in conjunction with the thermometer to obtain the correct temperature of the liquid.[6]

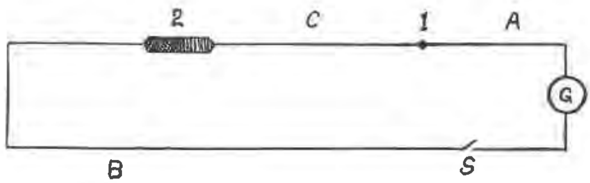

Fig. 2. Wiring diagram for the thermocouple and galvanometer.

The accuracy with which changes in temperature of the liquid on a refractometer can be followed by this method, is shown by the data in Table I, which are taken from one of the trial runs. The liquid used was a sample of pure monobrom-naphthalene.

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values near the center of the scale, are practically proportional to the temperature differences. If then, the value of the scale deflections, in terms of temperature, is known and the temperature of one junction can be read by means of a thermometer, there is provided a means of determining the temperature of the other junction very accurately.

[5] The thermocouple and galvanometer scale may be calibrated by noting the deflections resulting from known small (0°–5°) differences in temperature. The two junctions may be inserted in two separate thermos bottles containing water and calibrated thermometers. The temperature difference and the corresponding galvanometer deflection are then obtained. Several such measurements for other temperature differences should be made, and the value of one scale division in terms of temperature calculated from the mean.

[6] The following procedure is suggested: The apparatus is arranged as shown in Fig. 1, with the flattened junction of the thermocouple on the hemisphere and just to the right of the center. (It may be held in place by fastening the lead-wires to the refractometer with modeling clay). The liquid is placed on the hemisphere and the junction and liquid covered with a piece of foil, which keeps the liquid in place on the junction. The switch is closed and the refractometer set at the critical angle. The galvanometer and thermometer scales are read and the switch opened. The refractometer scale is next read and then the null point of the galvanometer is obtained. The temperature of the liquid is then calculated as the algebraic sum of the corrected thermometer reading and the temperature value of the observed galvanometer deflections.

TABLE I

Time in minutes after first reading	Corrected Thermometer Reading	Critical Angle[a]	Calculated Temperature of Liquid	Refractive Index at 21°. Calculated from liquid temperature[b]
0	25.3	67° 13.5′	24.9	1.6589
2	25.4	67° 13.5′	25.1	1.6590
4	25.5	67° 13.5′	25.2	1.6591
7	25.5	67° 12.5′	25.4	1.6590
9	25.5	67° 12.0′	25.4	1.6589
11	25.5	67° 12.0′	25.5	1.6589
13	25.5	67° 12.0′	25.5	1.6589
15	25.6	67° 11.5′	25.6	1.6589
17	25.7	67° 11.0′	25.8	1.6590
19	25.8	67° 11.0′	25.9	1.6589
21	25.8	67° 11.0′	25.9	1.6589

[a] Angular readings are the means of three settings.

[b] For calculating n at 21°, the value $dn/dt = .00048$ was used. See Larson, Bulletin **679**, *U. S. Geol. Survey*.

Attention is called to two points shown in the above data.

The refractive index calculated to 21° is practically constant, indicating a very close agreement between observed changes in critical angle and changes in temperature as measured by this method.

The temperatures of the thermometer and of the liquid rose steadily (without measurable fluctuations) but at slightly different rates. The slow and regular character of the changes makes for greater accuracy in the calculated values of the liquid temperature, and does not obtain if the thermometer is used unprotected.

Other series of determinations were made on the same liquid on other days when the temperature of the room was different. Three of such series are summarized in Table II.

TABLE II

Trial	Number of Readings	n at 21° Calculated from Temp. of Liquid	Variation
1	11	1.6590	1.6589–1.6591
2	5	1.6592	1.6591–1.6592
3	6	1.6591	1.6590–1.6591

The consistency of the results obtained in these trials indicates that the measurements of the temperature were satisfactory.

USE OF THERMOCOUPLE AT THE MICROSCOPE

When determining the refractive indices of solids by the immersion methods on a microscope, the temperature of the liquid surrounding the grains must be known. The thermocouple described above can be used to determine this temperature to a high degree of precision and with but very little inconvenience. The same arrangement of thermocouple, galvanometer and protected thermometer is employed. The flattened junction is placed under one end of the cover glass and held in place by attaching the lead-wires to the slide with modeling clay. The method of procedure is similar to that used with the refractometer and the method of calculating the temperature is the same.

USE OF MERCURIAL THERMOMETERS IN REFRACTOMETRY

The differential thermocouple and thermometer described above were used to measure the differences between the temperature of a liquid on the refractometer and that of a second unprotected thermometer, when hanging at different places near the instrument, corresponding to those commonly used.

Depending on the location of the thermometer, the differences between the temperatures of the liquid and of the uninsulated thermometer varied in amount, being as high as 5° in some cases. For any given location of the thermometer, the temperature difference was often quite variable, even within relatively short time intervals, and was sometimes positive and sometimes negative in character. These variations may be explained, in part at least, by the observation that the thermocouple responds more rapidly to variations in temperature than does the attached mercurial thermometer. This also indicates the need for suitable insulation of the thermometer bulb and its attached junction.

The best agreement between the temperatures of the liquid and uninsulated thermometer was obtained when the latter was hung about 2 mm. above the liquid and protected by the cylindrical shield as shown in Fig. 1. With this arrangement, the temperature difference generally increased rapidly for a few minutes after the flame was turned on, the thermometer being at the higher temperature. Later the difference decreased, sometimes passing through zero and becoming negative. The final temperature differences though at times larger were generally within $\pm 1°$, indicating that such an arrangement may be satisfactory for routine work, although not reliable when precise data are desired.

Measurements similar to those just described were made, also, at the microscope. Unprotected thermometers, hung at several places near the instrument, were read at the same time the temperature of the liquid on the microscope slide was determined by the thermocouple and the protected thermometer.

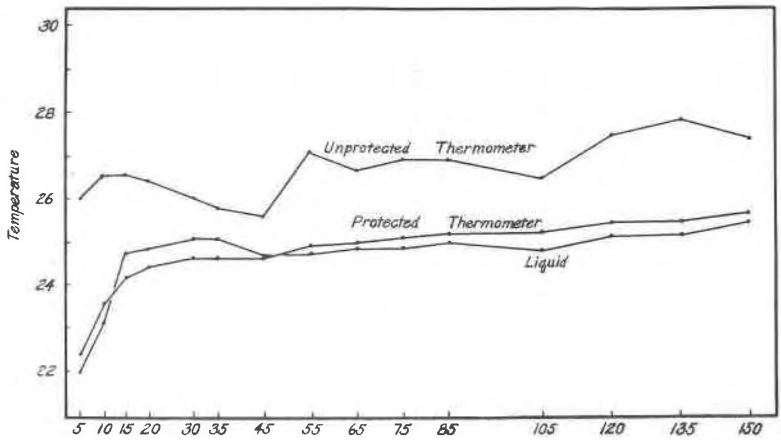

Fig. 3. Curve showing changes in temperature with time following the turning on of light at the microscope.

The data shown in Fig. 3 are typical of those obtained. In this trial all the apparatus and the liquid had been on the same table over night and the liquid had been in place on the microscope for some time before the lamp was turned on. However, five minutes after turning on the lamp, the unprotected thermometer registered a temperature about 4° higher than that of the liquid. During the entire trial, the unprotected thermometer hanging about 30 cm. from the microscope showed a temperature which was higher and more variable than that of the liquid. The significance of the temperature differences obtaining with such arrangements for temperature measurements may be clearer if stated in terms of refractive index. In the trial run reported in Fig. 3, the refractive index of a grain matching a liquid with $n=1.700$ (dn/dt=.0006) would have been too low by amounts varying from .0004 to .0023.

Examination of Fig. 3 shows a close agreement between the temperatures of the liquid and those of the protected thermometer used with the thermocouple. The differences in temperature measured by this arrangement of apparatus are small and therefore give rise to only small deflections near the center of the galvanometer scale. Were the reference junction immersed in a liquid in a thermos bottle, its temperature would remain sensibly constant and the galvanometer deflections would therefore be larger and more variable—a condition making for less accurate results.

The close agreement noted suggests the possibility of using a good thermometer similarly protected, but without the thermocouple, for routine work where temperature measurements to $\pm 2°$ are satisfactory. A few trials should indicate the proper amount of packing and the proper location of the thermometer to insure reasonably close agreement between the temperature indicated by the thermometer and that of the liquid on the slide.

Summary

The measurement of the temperature of a liquid on a crystal refractometer or on a microscope slide by means of a thermometer hung in the vicinity of the instruments was found to be unreliable.

A method is described by which these temperatures can be measured with the accuracy required when refractive indices are to be determined to 0.001. The method is simple in operation and requires apparatus which is relatively inexpensive.