A METHOD FOR MEASURING THE DIELECTRIC CONSTANT
OF LIQUIDS

A Thesis submitted in partial fulfilment of the requirements for the degree of Master of Applied Science at the University of British Columbia.

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Accepted on behalf of the Department of Chemistry
Head of the Dept. of Chemistry
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I. **Object of the Research**

Work done on the isomers of decahydronaphthalene in this laboratory during the last few years has indicated that there is a singularity in some of the physical properties of the cis isomer near 50°C. The object of this research is to measure the dielectric constant of pure cis- and trans-decahydronaphthalene and to observe whether any singularity exists in the curve showing the variation of the dielectric constant of the cis-isomer with temperature.

II. **Materials Used**

A. **Cis Decahydronaphthalene**

Rectification of a mixture of the isomers at about 9 mm. absolute pressure in a Stedman Column (1) effected a separation into quite pure cis-decalin and trans-decalin. The purest fractions of each isomer had a purity

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of about 99.5 - 99.8%.

The purest fraction of cis decalin was further purified by recrystallization in a dry-ice bath, following the method used by Seyer and Walker (2). After successive recrystallizations a product was obtained whose freezing point was constant at -43.15°C, as measured by a Leeds and Northrup platinum resistance thermometer. This sample of cis decalin is considered to be as pure a sample as can be obtained.

B. Trans Decahydronaphthalene

Successive recrystallizations of the purest fraction of trans decalin yielded a product with a constant freezing point of -30.64°C.

C. Benzene

"Merck thiophene free benzene" was tested for thiophene by shaking a sample of the benzene with a few cc of C.P. conc. H₂SO₄. Since no darkening of the acid layer was observed, it was assumed that no thiophene was present. About 500 cc. of this benzene was crystallized in an ice bath until all but some 50 cc of benzene had solidified. This residue was rejected. After the fourth recrystallization, the product had a constant freezing point of 5.37°C as measured by a Leeds and Northrup platinum resistance thermometer.

(2) Seyer and Walker, *J.A.C.S.* 60 2125, (1938)
III. Equipment for and Method of Measuring Dielectric Constant

An excellent discussion of the methods of measuring dielectric constant can be found in G. P. Smyth's monograph "Dielectric Constant and Molecular Structure". The method and equipment used in this research is essentially that described by Alexander (3). A test cell is constructed, and its capacity determined by calibration with a liquid of known dielectric constant as the dielectric. Its capacity is again determined when the liquid of unknown dielectric constant is the dielectric, and from this capacity the dielectric constant of the test liquid is calculated. The test cell, in parallel with three variable condensers and an inductance, forms the tuned plate circuit of a vacuum tube oscillator; and a crystal oscillator in parallel with a resistor forms the tuned grid circuit. The vacuum tube used is one of type 6E5, an "electron ray" tube, which serves both as an oscillator and as a means of detecting when the crystal oscillator "snaps in"; that is, when the plate circuit is tuned to oscillate to the characteristic frequency of the crystal.

(3) F. C. Alexander, "Dielectric Constant Meter", *Electronics*, 18, #4, p 116 (April 1945)
A. Test Cell.

The two metal parts of the test cell were machined from stainless steel according to the specifications shown in fig. 2, and a small sapphire mounted in a little brass holder was screwed into the end of the centre electrode to orient the middle electrode in the cup of the test cell. According to Alexander (4), such a centering device increases the reproducibility of results from 1% error without the sapphire to 0.3% error with the sapphire. The point of the sapphire fits into a small hole drilled in the bottom of the cup of the test cell. The centre electrode was fixed in its porcelain holder in the following manner. The electrode was centered in the outer part of the cell and three or four asbestos-paper washers were put between the top of the electrode and the under side of the porcelain holder in order to make the

(4) loc cit
FIG. 2. PARTS OF TEST CELL.
electrode bear against its holder when centered and touching the bottom of the well in the outer part of the cell. Finally, a nut was screwed down lightly over the threaded projection - shown in fig. 3. With the cell thus assembled and held firmly together by hand in an upsidedown position, some "Chicago China Handle Cement", a quick drying cement resembling plaster of Paris, was worked into the space between the electrode and the side of the holder. After the cement had set hard, two or three asbestos washers and a brass washer were put over the top of the holder and the assembly was made secure by tightening down the nuts which screwed onto the threaded contact projecting up through the holder from the centre electrode.

Later it was found that liquids put in the cell evaporated so rapidly that even decalin would evaporate enough to cause considerable change in the capacity of the test cell after two or three days of continuous operation. A fibre collar was therefore made to fit around the thin portion of the centre electrode and to cover the annular space containing the dielectric. Since Cenco Label Varnish was found to be resistant to the action of benzene and decalin, a coat of this varnish was used as a cement to fix the fibre collar to the centre electrode. A coat of varnish also prevented the cement
FIG. 3. Assembly of Test Cell in Oven.

A. Threaded projection from centre electrode
B. Copper bus-bar
C. Red fibre insulator
D. "Shoulder" of test cell
E. Fibre cap for reducing evaporation
F. Fan
G. High frequency lead, grounded side
H. H.F. lead.
J. Auxiliary heating coil
K. Fibre insulator
in the holder from becoming soft from the action of water, benzene, or decalin, and prevented any particles of cement falling into liquid in the test cell.

B. Constant-Temperature Oven

A constant temperature oven for the test cell was made from a sheet metal box 4" x 4" x 6" high. The bottom of the box was reinforced by a 1/8" brass plate, and one side by a thinner copper plate. A small fibre block was mounted on this side to support the copper bus bar which made contact with the centre electrode of the test cell. The box was covered by one layer of asbestos paper over which 45 to 50 feet of Chromel A #22 gauge resistance wire were wound, giving a 45 ohm heating coil. The oven was insulated on the bottom and sides with 1 1/2" of rock wool insulation. In order to obtain close temperature control, a small coil of Chromel resistance wire was put inside the oven. The power input to the main heating coil was controlled by a variac; and the input to the secondary coil inside the oven could be changed by means of a variable resistance in series with it. The temperature control unit was an American Instrument Company "Metastatic" mercury thermal-regulator used with an Aminco mercury relay, which controlled the input to the secondary heating coil only.
The air in the oven was circulated by a small fan driven by a variable speed stirring motor.

Two holes were drilled and tapped in the bottom of the test cell, so that it could be screwed onto a brass plate about 2 1/2" square. Holes were then drilled and tapped in this plate and in the base plate at the bottom of the oven. This allowed the cell to be screwed firmly to the bottom of the oven, the screws making good electrical contact between oven and test cell; and the cell could be easily removed from the oven for washing, filling or repairs. In addition to serving as an oven, the sheet metal box, which was grounded, acted as an electrical shield for the test cell.

C. Remainder of Electrical Circuit

The remainder of the circuit shown in fig. 1 was assembled in a box shielded electrically by sheet metal. \( C_1 \) was a 250 \( \mu \)f condenser used for tuning the plate circuit roughly to the oscillating frequency of the crystal. \( C_2 \) was a small variable condenser which allowed the calibrated dial of condenser \( C_3 \) to be set accurately at any desired reading when the test cell contained only air as the dielectric. \( C_3 \) was a variable condenser with semicircular plates, such that its capacity varied very nearly linearly with the number of degrees through which the moveable plates were turned with respect to the fixed
plates. A dial calibrated from 0 to 100 through 180° was mounted with \( C_3 \) so that when the dial read zero, capacity \( C_3 \) was a maximum, and when the dial read 100, capacity \( C_3 \) was a minimum, the total variation in capacity being \( 23 \mu F \). When variations in the capacity of the test cell with temperature or with change of dielectric altered the frequency of oscillation of the plate circuit from that of the quartz crystal, this condenser was used to re-tune the circuit to the frequency of the crystal.

The inductance in the plate circuit consisted of 30 turns of cotton insulated copper wire wound on a 1 1/2 inch diameter form.

The quartz crystal was a Y-cut type of about 2 megacycles frequency.

As noted previously, a 6E5 type tube acted both as an oscillator and a detector to indicate when the plate circuit was tuned to the frequency of the crystal. The power for the plate circuit was supplied by two 45 volt, #385 Eveready dry cells. The filament circuit was heated by a number of 1.5 volt, #6, dry cells.

It was found that operating the filament circuit continuously greatly decreased the voltage delivered by the dry cells within a short time. Since variation in filament voltage was one factor contributing to erratic
results, this voltage was kept constant at 5 volts by connecting in parallel 2 or 3 series-sets of 4 cells and placing a small variable rheostat in the filament circuit. Still later, the filament was run from a 6 volt lead storage cell.

During some of the first observations made, it was found that variation in room temperature caused apparent variation in the capacity of the test cell even though the cell was kept at constant temperature in the oven. The main portion of the r.f. circuit was, therefore, enclosed in a 5-ply box which acted as thermal insulation, and the box was kept at the constant temperature of 30°C by a small chromel resistance wire heating coil acting in conjunction with a mercury thermal regulator and a relay. The air in this constant temperature bath was circulated by a fan operated by a variable speed stirring motor.

The leads between the test cell and the rest of the circuit were fixed in position as rigidly as possible and were electrically shielded. They were run through small pyrex tubing and shielded, one by copper tubing and the other by silver foil. The lead which was not grounded had to be more rigidly fixed than the other.

IV. Calibration of Equipment

To determine over what range of dial readings the variation in capacity $C_3$ was linear, the capacity of con-
denser $C_3$ was measured, for various dial readings, by a Boonton Radio Corporation Q-Meter, Type 160-A. The capacity dial on the Q-Meter was calibrated from 30 to $450 \mu\text{f}$ and was used with a vernier dial calibrated over a range of $6 \mu\text{f}$ in tenths of a $\mu\text{f}$. Changes in capacity could be estimated to $0.01 \mu\text{f}$. The condenser was calibrated after it had been mounted in the shielded box. Since a sharp resonant peak was not obtained in the circuit used for the calibration, several calibrations were made and a weighted average of the differences in capacity between successive dial readings was struck as shown in table I. The plot of these results in fig. 5 shows that the capacity of the tuning condenser $C_3$ varies linearly between 15 and 85 on the dial. For this reason, the dial was set at 15.0 when the test cell had minimum capacity; i.e., when it contained air as dielectric.

Before considering how the test cell was calibrated, it will not be out of place to mention the difference between dielectric constant and specific inductive capacity.

From Coulomb's law we can define electrical intensity as the force exerted by charge $q$ on unit charge

$$E \propto \frac{q}{r^2}$$

or

$$E = \frac{1}{\mu} \frac{q}{r^2}$$

where $\mu$ = constant of proportionality.
<table>
<thead>
<tr>
<th>Dial Reading</th>
<th>Capacity Increments</th>
<th>Weighted Avg.</th>
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<tr>
<td></td>
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<td>$\Delta_2$</td>
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</tr>
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<tr>
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<td>0.25</td>
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</tr>
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</table>
FIG. 5.
Calibration curve for tuning condenser $C$. 

Capacity (pF)  

DIELECTRIC CONSTANT  

Dial Reading
Electrostatics also defines electrical displacement as
\[ D = \frac{q}{\kappa^2} \]
Then intensity and displacement are related by the factor \( \mu \); thus
\[ D = \mu E \]
\( \mu \) is the inductive capacity or the permittivity of the medium. Vacuum has an inductive capacity of \( \mu_0 \).

Consider two charged plates which form a condenser. Let \( q \) be the charge on each plate. With vacuum as dielectric we have \( q = C_0 V_0 \) where \( C_0 \) is the capacity and \( V_0 \) is the potential difference between the plates when vacuum is dielectric. With some substance as dielectric
\[ q = C_1 V_1 \]
Since \( q \) does not change when we change the dielectric
\[ C_0 V_0 = C_1 V_1 \]
Experiment shows \( C_1 \propto C_0 \)

or \( C_1 = \varepsilon_1 C_0 \) where \( \varepsilon_1 \) is called the dielectric constant. Then
\[ C_0 V_0 = \varepsilon_1 C_0 V_1 \]
\[ V_1 = \frac{1}{\varepsilon_1} V_0 \]

Now the potential difference between plates A and B is
\[ V = -\int_A^B (E \cdot ds) \]
When vacuum is the dielectric
\[ V_0 = -\int_A^B (E_0 \cdot ds) = -\int_A^B \frac{q}{\mu_0 \kappa^2} ds \cos \theta \]
\[ = \frac{1}{\mu_0} \int_A^B \frac{q}{\kappa^2} ds \cos \theta \]
where \( \theta \) is the angle between \( E \) and \( ds \).
When any substance is dielectric,
\[ V_i = - \int_A^B \left( \vec{E}_i \cdot d\vec{s} \right) = -\int_A^B \frac{Q_i}{\mu_i \mu_0} ds \cos \theta \]
\[ = \frac{1}{\mu_i} \int_A^B \frac{Q_i}{\mu_0} ds \cos \theta \]

Then
\[ V_0 / \mu_0 = V_i / \mu_i \]

Also
\[ V_i = \frac{1}{\varepsilon_i} V_0 \]

\[ \therefore \varepsilon_i = \mu_i / \mu_0 \]

In the cgs system of e.s. units \( \mu_0 = 1 \) and inductive capacity and dielectric constant are the same numerically. In other systems of units, however, \( \mu_0 \neq 1 \) and \( \varepsilon \neq \mu \). For any system, however, the dielectric constant of any one substance is the same.

From the relation \( \varepsilon_i = \mu_i / \mu_0 \), it can be seen that for vacuum as dielectric; i.e. when \( \mu_i = \mu_0 \), \( \varepsilon = 1.0000 \). Now air has a dielectric constant of 1.00059 at 0°C which is indistinguishable from that of vacuum with the apparatus used. Hence to calibrate the test cell, condenser \( C_3 \) was set to read 15 (page 7) when the test cell contained only air as dielectric and the circuit was tuned with \( C_2 \). The test cell was then filled to the shoulder (fig. 3) with benzene, the circuit tuned with condenser \( C_3 \), and the new dial reading observed. Then 15.0 on the dial corresponds to a dielectric constant of 1.000, and the dial reading for benzene corresponds to the dielectric constant of benzene (2.283 at 20°C (5)).

(5) F. C. Alexander, op. cit.
If \( C_0 \) is the capacity of the test cell when air is dielectric, and if \( C_x \) is its capacity when a test liquid is dielectric, the dielectric constant of the liquid is

\[
\varepsilon = \frac{C_x}{C_0}
\]

Let \( C_x - C_0 = C \), which is the capacity change made in the tuning condenser. Substituting for \( C_x \) we get

\[
\varepsilon C_0 - C_0 = C
\]

\[
\varepsilon = \frac{C + C_0}{C_0}
\]

Thus it is shown that dielectric constant varies linearly with the change in capacity of the condenser \( C_0 \), and between 15 and 85 on the \( G_3 \) dial, dial reading and dielectric constant are linearly related.

The thermometer used to take the temperature of the constant temperature oven was calibrated from \(-10^\circ C\) to \(110^\circ C\) in tenths. The ice and steam points were checked and a correction for the inaccuracy of the original calibration was applied to all temperature readings in addition to a correction for stem exposure. The stem exposure correction is given by the formula

\[
t = 0.000165 (t - t_s)(\theta)
\]

where
- \( t \) = observed temperature
- \( t_s \) = temperature of exposed portion of stem
- \( \theta \) = no. of degrees on the scale over which the mercury is exposed.
V. Experimental Difficulties

It was considered desirable to observe the capacity of the empty cell at various temperatures before measuring the dielectric constant of decalin over a range of temperatures. The temperature of the cell was raised to 50°C and capacity readings were taken periodically over a 10 1/2 hour period, during which the temperature of the rest of the electrical circuit was kept at 30°C ± 1°C. The voltage applied across the filament of the 6E5 was observed when each reading was taken. The apparent capacity of the test cell was found to decrease with time, as shown in fig. 6. When these measurements were made, the filament of the 6E5 was supplied by 3 or 4 dry cells in series. Since the current, 0.3 amp., taken from the cells was very heavy, it was necessary to add another cell to the series from time to time; and large sudden variations in filament voltage occurred during the course of these observations. These have also been plotted against time in fig. 6 and it can be readily seen that a relation exists between the filament voltage and the measured capacity of the test cell. This relation, however, appears to be superimposed upon a continuous drift toward decreasing capacity with time. Since it was suggested that change in plate voltage might account for this drift, a capacity and resistance were put in the
FIG. 6.

Shows the drift in the capacity of the empty test cell and its relation to change in filament voltage.

1 dial division = 0.23 μμf
= 2.3% of capacity of empty test cell
plate circuit, as shown in fig. 7, to eliminate any change in the capacity of the voltage source, and the plate voltage was decreased by 22 volts.

![Plate Circuit Diagram](image)

This voltage change produced a change of capacity of $-0.000035 \mu F$ per volt, which is an insignificant error. At the same time the effect of changing filament voltage was checked, and it was found that a change of 1.5 volts on the filament had no effect on the measured capacity either immediately or within 15 minutes. Still later measurements showed a change in capacity with filament voltage similar to that shown in fig. 6. To determine whether this observed change was caused merely by change in the capacity in the filament circuit when another dry cell was added, an old cell whose potential was only 0.2 volts was put in series with the other dry cells in the filament circuit. The resulting change in the apparent capacity of the test cell was only 0.02 $\mu F$. It was therefore assumed that the voltage across the filament,
and not the capacity of the filament circuit determined the variation in dial readings.

Since on two occasions, once over a 6 1/2 hour period and once over a 4 1/2 hour period, the capacity was found to be constant when the test cell was at room temperature, it was considered possible that small quantities of grease or ammonium chloride might be distilling out from the cell at the elevated temperature. The cell was, therefore, scrubbed out with soap and water, rinsed with distilled water and baked at about 70°C for a few hours. For a few days after that, the measured capacity of the test cell remained constant with time at any one temperature. Later, the measured capacity of the cell again decreased with time at 50°C; but even after washing the cell, first with soap, then with cold water for 4 hours, then rinsing it out with twice distilled alcohol, and finally with twice distilled ether, the drift with time was not eliminated.

With the filament voltage maintained at 5.0 v, the cell was tested for drift when it contained trans decalin as dielectric. The measured capacity of the cell varied quite inconsistently; at room temperature both drift toward decreasing capacity and unreasonably large fluctuations of capacity were observed. (fig. 8).
FIG. 8.
Shows variation in measured capacity of test cell containing trans-decalin.

1 div. = 0.23 μF
= 2.3% of capacity of empty test cell.
To determine whether this undesirable behaviour was caused by the test cell or by the remainder of the circuit, the test cell was removed and dial readings were taken periodically. The circuit was kept at 30°C and for the first few hours the filament voltage was 5.0 v. As shown in fig. 9, drift continued at much the same rate as before (compare with figs. 6 and 8). Since the rated filament voltage for a 6E5 tube is 6.3 volts, it was thought possible that operating the tube at 5 volts made it sensitive to very small changes in filament voltage and that a change in the characteristics of the tube was responsible for the observed drift. For this reason the filament voltage was increased to 6.1 volts, the filament now being heated from a lead storage cell. Figure 9 shows that the capacity drift persisted after the voltage had been changed.

Although the high frequency circuit is sensitive to temperature change, the drift toward lower capacity is not accounted for by small fluctuations in the temperature of the circuit since such temperature fluctuations should produce corresponding increases and decreases in the measured capacity of the system. The relation between the temperature of the circuit and the dial reading is illustrated in figures 10, 11, 12 and 13. Although the relationship is sometimes irregular, the graphs
Figure 9.

Test cell removed from the circuit
Old crystal used
1 dial div. = 0.23 µuf
= 2.3% of capacity of empty cell

Figure 10.

Test cell removed from circuit
New crystal (3502 kc) used
Filament voltage = 6 v
indicate that after considerable time lag the dial reading increases as the temperature of the circuit increases, and that the average rate of increase is 0.5 dial division per degree, which represents an increase in the measured capacity of the cell of 0.11 μf, or an increase of 0.012 in the dielectric constant of any liquid in the test cell. It should be emphasized, however, that the drift toward lower capacity is not accounted for by even quite large fluctuations in the temperature of the circuit.

A new 3502 Kilocycle Pedersen Radio Company crystal was bought and installed in the circuit. Since its temperature coefficient was at most 2 cycles per mega-cycle per degree centigrade, it was probably AT-cut. With the test cell still out of the circuit and the filament voltage at 6 volts, the performance of the apparatus was again tested by taking dial readings two or three times each hour. The results are plotted in figures 10 and 11. A continual drift in capacity in one direction was no longer observed, but this drift was replaced by periodic changes in capacity in either direction. Some, but not all, of this change can be attributed to variation in the temperature of the circuit.

Since it might have been the tube that was causing the trouble, a new 6E5 was put in the circuit and the
FIG. 11.
Test cell not in the circuit.
New crystal used.
Filament voltage = 6.3 v.
1 dial division = 0.23 μs.
= 2.3% of capacity
of empty test cell.

FIG. 12.
Test cell not in the circuit.
New crystal used.
New tube used.
Circuit temp. 30.3° ± 0.1°C
Filament voltage = 6.4 v.
behaviour of the apparatus observed as before. During these observations the temperature was kept at $30.3 \pm 0.1^\circ C$. The results, which are plotted in fig. 12 show

(a) the change in tubes did not change the operating characteristics of the apparatus.

(b) maintaining the circuit at a temperature constant within $0.2^\circ C$ does not eliminate the periodic variations in capacity also observed previously.

Since there was a possibility that a relationship might exist between humidity and the dial reading, the humidity was observed over an 11 hour period during which dial readings were being taken. The results, plotted in fig. 12, indicate that no such relation exists.

Inspection of the data plotted in fig. 9 shows, in the light of subsequent data, that the variation in dial reading during the period when filament voltage was at 6.1 volts and the old crystal was in the circuit was not larger than variations observed after the new crystal had been put in the circuit. To test, then, whether the change of crystal or change of voltage had produced the more constant dial readings, the old crystal was returned to the circuit and the apparatus run with 6.8 volts across the filament of the 6E5 and with the cell still out of the circuit. The results, plotted in fig. 13, resemble
FIG. 13.
No cell in circuit; old crystal used.
Filament voltage = 6.8 V.
Circuit temp. = 30.3°C ± 0.1°C.

FIG. 14.
Empty cell in circuit; new crystal used.
Filament voltage = 6.4 V.
the results obtained with the new crystal. As a further check, the filament voltage was lowered to 5.0 volts and readings taken over six hours. This operating condition seems less stable than the one in which the filament is run at or above its rated voltage.

During most of the tests, the crystal was kept oscillating continuously; and the question arose as to whether the mechanical strain so produced caused fatigue in the crystal which was responsible for variation in its resonant frequency, and consequent variation in dial reading. The data plotted in figures 11 and 12 was taken with the crystal oscillating only long enough to determine each dial reading. Since variations of the same magnitude as had been found previously still occurred, it was concluded that crystal fatigue due to continuous oscillation did not account for the poor results.

In summary, then, we have the following results.

1. The temperature of the circuit should be controlled to within ±0.1°C to keep the measured capacities independent of circuit temperature.

2. The continual drift toward lower capacity, observed during the first tests, is not entirely characteristic of the apparatus without the test cell. Although figure 9 was at first thought to indicate that the drift persisted when the old Y-out crystal was in the
21.

Circuit, figure 13 shows that the drift is not as persistent as that originally observed (fig. 6). The new AT-cut crystal appears, however, to be slightly more stable than the other. Why the drift did not return when the test cell was put back in the circuit is not explained. (These data are plotted in figure 14.)

3. Around four volts, change in filament voltage has a pronounced effect on the measured capacity of the test cell. Most satisfactory operation is probably obtained by having 6.3 volts, or a little more, across the filament.

4. Although Bender (6) states that erratic operation may be expected in humid weather, no direct connection was observed between humidity and dial reading. Since the humidity is almost always high in Vancouver, it is suggested that Bender's recommendation of rinsing/tuning condenser plates in absolute alcohol be tried in any further research.

Although a fibre cap had been made to fit over the annular opening of the test cell to reduce evaporation loss from the cell, it was found that evaporation still continued at an excessive rate. At 22°C, with the fan in the oven operating, and with benzene in the cell,

evaporation losses caused a decrease in the capacity of the cell of $0.0015 \mu F$ per minute; and when the fan was not running, the rate of decrease was $0.00015 \mu F$ per minute. At higher temperatures, loss of decalin by evaporation is an error of similar magnitude; an error which makes the determination of $d\sigma /dT$ quite unfeasible.

VI. Accuracy of the Method

1. Precision of Tuning Condenser

Since the tuning capacitor was a good quality condenser with semicircular plates, one would expect change in capacity to be linear over most of the capacity range. The absolute values of capacity measured by the Q-meter, which was used to check the tuning condenser for linearity, are not important; but differences in capacity should be accurately measured. Individual values of capacity on the Q-meter dial are accurate to $\pm 1 \mu F$ or $\pm 1\%$, whichever is the greater. In checking the tuning condenser, the range between $30 \mu F$ and $60 \mu F$ on the Q-meter was used. Hence, if the Q-meter was calibrated against a standard capacity at $10 \mu F$ intervals in this range, a possible error of $20\%$ may exist in the measurement of a capacity difference. Although this error is possible, it is not considered probable.

It was found that capacity readings on the Q-meter wander as the instrument continues in operation, even
after a few hours. The rate of wandering is of the order of $0.1 \mu \text{f}$ per hour, and for this reason the Q-meter itself is unsuitable for measuring $\frac{d\varepsilon}{dT}$ when the capacity of the test cell is small. This wandering would not affect the calibration of the tuning condenser because only the difference between successive Q-meter readings, which were taken every minute or two, are important in this calibration.

2. Orientation of the Centre Electrode

Since a small change in the position of the centre electrode produces an appreciable change in the capacity of the test cell, it is important to be able to reproduce, as accurately as possible, the setting of this electrode. It was found easier to orient the electrode when the fibre cap, used to cut down evaporation, was not in the cell. Dial readings were taken for a series of different settings of the middle electrode. These are shown in Table II together with the average deviation introduced into the values of $\varepsilon$. The probable error in the dielectric constant of decalin ($\varepsilon_{\text{decalin}} \cong 2.15$) will be very close to

$$0.845 \left( \frac{\text{Avg. deviation for benzene}}{\sqrt{n}} \right)$$

where $n$ is the number of observations. That is, the probable error in $\varepsilon_{\text{decalin}}$ caused by orientation of the
Table II

(a) Fibre Cap Absent

<table>
<thead>
<tr>
<th></th>
<th>Cell empty</th>
<th>Cell filled to shoulder with Benzene</th>
<th>Cell filled to the top with Benzene</th>
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<tr>
<td>15.0</td>
<td>65.0</td>
<td>67.0</td>
<td></td>
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<tr>
<td>15.2</td>
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<td>67.3</td>
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</tr>
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<td>67.0</td>
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</tr>
<tr>
<td>15.1</td>
<td>64.7</td>
<td>67.0</td>
<td></td>
</tr>
<tr>
<td><strong>Mean 15.0</strong></td>
<td><strong>64.9</strong></td>
<td><strong>67.1</strong></td>
<td></td>
</tr>
</tbody>
</table>

Avg. deviation 0.1 division = 0.023 $\mu$F

Capacity of test cell empty 9.8 $\mu$F

Capacity of test cell with benzene 22.4 $\mu$F

Avg. deviation in $\varepsilon$ for air 0.2%

Avg. deviation in $\varepsilon$ for benzene 0.1%

(b) Fibre Cap Present

<table>
<thead>
<tr>
<th></th>
<th>Cell empty</th>
<th>Cell filled to shoulder with Benzene</th>
</tr>
</thead>
<tbody>
<tr>
<td>15.0</td>
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<td>65.5</td>
</tr>
<tr>
<td>14.5</td>
<td></td>
<td>65.3</td>
</tr>
<tr>
<td>14.0</td>
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<td>65.4</td>
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<td>14.7</td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>Mean 14.8</strong></td>
<td><strong>14.6</strong></td>
<td><strong>65.1</strong></td>
</tr>
</tbody>
</table>

Avg. deviation 0.3 div. 0.36 div.

Avg. deviation in $\varepsilon$ 0.7% 0.4%

Capacity of that part of the cell which will be or is filled with liquid.
middle electrode is not more than 0.2%, even when the fibre collar is in the cell.

3. Anomalous Dispersion(7)

When high frequencies are used in the measurement of dielectric constant, the phenomenon of anomalous dispersion may be encountered; i.e., the dielectric constant becomes smaller at high frequencies. The phenomenon is confined to polar liquids, and commences at a frequency whose reciprocal is equal to the relaxation time of the dipolar molecules. The relaxation time is given by

\[ \tau = \frac{4\pi \eta a^3}{kT} \]

where \( \eta \) = viscosity
\( a \) = molecular radius
\( k \) = Boltzmann's constant
\( T \) = absolute temperature

Although decalin has been found to have zero moment(8), it is conceivable that the unsymmetrical cis form has a small dipole moment, and hence that anomalous dispersion might be encountered in measuring \( \varepsilon \) for decalin. The critical frequency at which the phenomenon commences may be shown to be much higher than 3502 k.c., the maxi-

(7) C.P. Smyth, Dielectric Constant and Molecular Structure, Chemical Catalog Co., 1931, p.p. 34-38

(8) Estermann, Z. physik. Chem., (B) 1, 161 (1928); 2, 287 (1928).
mum frequency used. Thus, if we substitute

$$\eta = 0.0338 \text{ poise (9)}$$
$$a = 6 \times 10^{-8} \text{ cm (10)}$$
$$k = 1.38 \times 10^{-16} \text{ erg/degree}$$
$$T = 293^\circ\text{K}$$

into

$$\tau = \frac{4\pi \eta a^3}{k T}$$

we get $\tau = 2.29 \times 10^{-9} \text{ sec.}$ for cis decalin at $20^\circ\text{C}$. Hence the critical frequency is of the order of 400 megacycles.

4. **The Accuracy of $\varepsilon$**

If the dielectric constant of a liquid is desired at one temperature only, this method and apparatus provides an easy and accurate method of making the measurement. If the measurement can be made before the loss of liquid from the cell by evaporation is appreciable, then the main error is the variation in capacity with time, inherent in the apparatus as it stands at present. The maximum variation found latterly is about $0.25 \mu \mu \text{f}$. For a substance whose dielectric constant is 2, this variation represents a maximum error of 1.3% in the determined value of $\varepsilon$.


(10) Smyth, *op.cit.*, p. 38, quotes the molecular radius of nitrobenzene as approx. $3 \times 10^{-8} \text{ cm}$. An approximation for the molecular radius of decalin is, then, $6 \times 10^{-8} \text{cm}$.
5. Accuracy of $\frac{d\varepsilon}{dT}$

According to the Clausius - Mosotti equation, the molecular polarization $P$ is given by

$$P = \frac{\varepsilon - 1}{\varepsilon + 2} \frac{M}{\rho}$$

where $\varepsilon$ = dielectric constant

$M$ = molecular weight

$\rho$ = density

Now if the dipole moment of a substance is zero, $P \not= f(T)$, and hence for decalin we may write, assuming it to be non-polar,

$$\frac{d}{dT} \left( \frac{\varepsilon - 1}{\varepsilon + 2} \right) = \frac{P}{M} \frac{dP}{dT}$$

$$\frac{3}{(\varepsilon + 2)^2} \frac{d\varepsilon}{dT} = \frac{P}{M} \frac{dP}{dT}$$

$$\frac{d\varepsilon}{dT} = \frac{(\varepsilon + 2)(\varepsilon - 1)}{3\rho} \frac{d\rho}{dT}$$

$\varepsilon \approx 2.15$ for decalin

$$\frac{d\rho}{dT} = -0.000734 \text{ for cis decalin (11)}$$

$\rho = 0.8967$ at 20°C for cis (11)

Then $\frac{d\varepsilon}{dT} \approx -0.0013$ per degree C for cis decalin.

If $\varepsilon$ be measured at 10 degree intervals, and if the maximum error in the difference between two successive values of $\varepsilon$ be 40%, then the maximum allowable error in either measurement is $\pm 0.003$, or $\pm 0.14\%$. The difficulties of capacity variation with time and of evaporation make accuracy of that order quite unattainable.

(11) Seyer and Davenport, J.A.C.S., 63, 2426 (1941)
VII. Corrections for Changes in Dimensions of the Cell with Temperature.

The capacity of a condenser formed by two coaxial cylinders is given by

\[ C = \frac{0.2416 \ell}{\log_{10} \frac{r_2}{r_1}} \]

where \( \ell \) = length of cylinder
\( r_1 \) = radius of outer cylinder
\( r_2 \) = radius of inner cylinder

Using the dimensions given in figure 2, the capacity of the part of the test cell indicated by the shading in fig. 17 can be calculated as 8.07426 stat-farads at 20°C. Taking the coefficient of linear expansion of 18-8 stainless steel as \( \alpha_s = 18.0 \times 10^{-6} \) per degree C. We calculate that the capacity of the same part of the cell is 8.08874 stat-farads at 120°C. Then the increase in the capacity of the empty cell is 0.0018% per degree C.

Observations showed that the measured capacity of the test cell increased, on the average, at the rate of 0.0087 \( \mu \) \( \mu \)f\per degree C., or 0.087% per degree. Then probably 1/4 of this increase occurs in a part of the cell where a test liquid can replace air as dielectric, and the rest where the air is not replaced. When the cell is filled with decalin, a correction must be subtracted from the observed dial reading, for all readings
taken above 20°C, to compensate for the change in the
capacity of the cell which is caused by a change in the
dimensions of the cell rather than by a change in the
dielectric constant of the test liquid. Expressed in dial
divisions, the correction is
\[- (t - 20) \left[ 2.15 \left( \frac{0.038}{4} \right) + \frac{3}{4} (0.038) \right] \]
where \( t \) = temperature in degrees C.

2.15 \( \approx \) \( \epsilon \) for decalin

0.038 dial divisions per degree correspond to

0.0087 \( \mu \) \( \text{F} \) per degree.
The correction reduces to

\[- (t - 20)(0.049) \]
The increase in the annular volume indicated by the
shaded area in fig. 17 is given by

3\( \alpha_s \) \( V \) per degree C.

where \( \alpha_s \) = coeff. of linear expansion of steel

= 18.0 \( \times \) 10\(^{-6} \)

\( V \) = the annular volume

= 0.0814 cu.in. at 20°C

The increase in the volume of test liquid in the cell is
given by

\( \gamma V \) per degree C

where \( \gamma \) = coeff. of volume expansion of the test liquid

= 81.8 \( \times \) 10\(^{-5} \) for cis decalin

\( V \) = 0.0814 cu.in. at 20°C
\[ V_t = (1 + \gamma t)V_0 \]

and \[ \rho_t = \frac{\rho_0}{(1 + \gamma t)} \approx \rho_0(1 - \gamma t) \]

also \[ \rho_t = \rho_0 + t \frac{d\rho}{dt} \]

Hence \[ \gamma = -\frac{1}{\rho_0} \frac{d\rho}{dt} = +\frac{1}{0.897}(0.000734) \text{ at } 20^\circ C \]

Then the excess volume of cis decalin, occupying the space above the shoulder of the cell, is \(6.2 \times 10^{-5}\) cu. in. for each degree rise in temperature. From this one may calculate that the liquid rises through \(0.14\%\) of the height from shoulder to top of the cell with each degree rise in temperature. From Table II it is seen that when the cell contains benzene, filling to the shoulder and filling to the top involves a difference in dial reading of 2.2 divisions, or a difference in capacity of \(0.51\mu\text{f}\). Then when the cell is filled to the shoulder at \(20^\circ C\) with cis decalin, each degree rise in temperature increases the capacity of the cell by \((0.0014)(2.15)(0.51) = 0.00067\mu\text{f}\), due to the expansion of the liquid. This discussion has, of course, ignored loss of liquid by evaporation.

**VIII. Results**

Although the two major difficulties of variation of capacity with time and loss of liquid from the cell by evaporation had not been cleared up, one run was made with each of cis and trans decalin to conclude this first year of investigation. The tuning condensers were set so that the dial reading was 15.0 when the test cell was
empty. The cell was then filled with 1.40 cc of benzene at 20°C and dial readings were taken quickly before much benzene could evaporate. Two successive dial readings, a different sample of benzene being used for each of these readings, showed 65.5. Since this checks well with the difference between dial readings for air and benzene shown in Table II part (b), 65.5 was considered to correspond to \( \varepsilon = 2.282 \), the dielectric constant of benzene at 20°C.

The benzene was removed from the test cell, the cell cleaned out with ether, and then filled with 1.38 cc of trans decalin. The temperature of the oven and cell was raised from 20°C to 85°C in 12 hours so that evaporation losses would be as small as possible. Even then, about 0.3 cc was lost by evaporation. The following day the cell was cleaned out and filled with cis decalin to the shoulder. The temperature was raised from 20°C to 90°C in 7 1/2 hours. Loss by evaporation was some 0.25 cc. During all these measurements the fibre cap was on the cell to reduce evaporation. Other operating data are

- frequency 3502 kc
- filament voltage 6.4 volts
- circuit temperature 30.0°C ± 0.2°

Results are shown in Table III
A Correction for evaporation

Since the vapour of the test liquid in the cell must diffuse through the small spaces between the fibre cap and the cell in order to escape, the problem of loss of liquid from the cell may perhaps be treated by the kinetic theory of interdiffusion of gases.

Let us assume that on one side of the fibre cap the test liquid and its vapour are in equilibrium at temperature $T$, giving a vapour pressure $p$; and that on the other side the concentration of vapour in the air is negligible, since the fan in the oven is operating. The number of molecules of vapour diffusing across unit area at the upper face of the fibre disc in unit time is given by

$$\frac{dN}{dt} = D \frac{dn}{dz}$$

where $t$ = time

$D$ = diffusion constant

$dn/dz$ = distance rate of change of concentration of vapour in the direction of diffusion.

Loeb (12) gives

$$D = \frac{1}{3} \left[ (1 - F_A) C_A L_A + F_A C_B L_B \right]$$

where $F_A$ = mole fraction of gas $A$ at the cross section where $dN/dt$ is being measured.

$U$ = avg. speed

$L$ = mean free path

At point "A" we are considering that \( F_0 = 0 \), gas A being the vapour of the test liquid. Hence

\[
\frac{dN}{dt} \propto c_A L_A \frac{dn}{dz}
\]

Now

\[
\frac{dn}{dz} \propto \frac{dp}{dz} \propto p
\]

\[
c_A \propto \sqrt{\frac{T}{M_A}}
\]

where \( M_A \) is the molecular weight of A.

Also \( L = \frac{1}{\sqrt{2 \pi n \sigma^2}} \)

But \( p = \frac{1}{3} n m c^2 \)

where \( m c^2 \) is a constant

Hence \( L \propto \frac{1}{p \sigma^2} \)

and \( \frac{dN}{dt} \propto \sqrt{\frac{T}{M}} \frac{1}{p \sigma^2} p = \frac{1}{\sigma^2} \sqrt{\frac{T}{M}} \)

Let us compare the rates of loss of benzene and decalin at 20°C.

\[
\left( \frac{dN}{dt} \right)_{c_{10}H_{18}} \propto \sqrt{\frac{293}{138}} \frac{1}{\sigma_{B2}^2}
\]

\[
\left( \frac{dN}{dt} \right)_{c_{10}H_{18}} \propto \sqrt{\frac{293}{138}} \frac{1}{\sigma_{Decalin}^2}
\]

Let \( \sigma_{Decalin} = 2 \sigma_{B2} \).

Then

\[
\left( \frac{dN}{dt} \right)_{c_{10}H_{18}} = \left( \sqrt{\frac{138}{138}} \right) \left( \frac{1}{4} \right) \left( \frac{dN}{dt} \right)_{c_{10}H_{25}}
\]

Now evaporation of benzene at 20°C caused a lowering of dial reading of 0.065 divisions per minute when the fan was operating, hence the predicted lowering for decalin at 20°C would be

\[
\frac{1}{4} \left( \sqrt{\frac{138}{138}} \right) \left( 2.15 \right) \left( 0.065 \right) = 0.0115 \text{ divisions per minute.}
\]
Details of corrections for temperature change and loss of liquid are shown in Tables IV and V. The change in dielectric constant with temperature, so determined, is shown in figures 15 and 16. No claim is made for the reliability of the results. Two interesting coincidences should be pointed out, however.

(a) The value of $\frac{d\varepsilon}{dT}$ for trans beyond $55^\circ C$ is not far from the theoretically calculated value.

(b) The sharp discontinuity in the curve for cis near $51^\circ C$ occurs at a temperature close to the point at which phenomena have been observed in other physical properties of the cis isomer.

The positive values obtained for $\frac{d\varepsilon}{dT}$ below $50^\circ C$ are contrary to theory (page 26) and set all these results on a very unsteady foundation.
<table>
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<th>Temperature</th>
<th>Reading</th>
<th>Time</th>
<th>Temperature</th>
<th>Reading</th>
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<td>Time Interval</td>
<td>Temp</td>
<td>Uncorrected Reading</td>
<td>Correction for change in cell with temp.</td>
<td>Rate of change in dial reading with loss of liquid from cell</td>
<td>Total change of dial reading from loss of liquid</td>
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TABLE IV. Corrections for trans-decalin
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<th>Uncorrected Reading</th>
<th>Correction for change in cell with loss of liquid from cell</th>
<th>Rate of change in dial reading with temp.</th>
<th>Total change of dial reading from loss of liquid</th>
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**TABLE V.** Corrections for cis-decalin
Method of Calculation:

Column 4 = \((\text{Col. 1} - 20)(0.49)\) (page 28)

Column 5 = \(0.0115 \sqrt{\text{Avg. temp. during interval in}^\circ\text{K}}\)

\(\frac{293}{293}\) (page 32)

Column 6 = \((\text{Col. 5})(\text{Col. 1})\)

Column 8 = \(\text{Col. 3} - \text{Col. 4} + \text{Col. 7}\)

Column 9 is obtained from fig. 5.
FIG 16

Values obtained for the dielectric constant of cis-decalin.
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