## Effect of Atmospheric Humidity and Temperature on the Relation between Moisture Content and Electrical Conductivity of Cotton \*

#### By ALBERT C. WALKER

THE data given in this paper show the effect of successive equilibrium humidity cycles on the relation between (a) relative humidity and moisture content; (b) insulation resistance and relative humidity; and (c) insulation resistance and moisture content, for raw and water-boiled cotton at constant temperature (25° C.). These data have been of considerable assistance in explaining the behavior of cotton, particularly the fact that its d.-c. insulation resistance, when measured at some definite test condition,1 is dependent, to a surprising extent, upon previous treatment, e.g. the manner in which wet cotton is dried, temperature of drying, and the atmospheric conditions to which it is exposed after drying, before being measured under the comparable test condition.

The information secured as a result of this investigation has been valuable in improving the practical methods of inspection used to control the quality of textiles for electrical insulation in telephone apparatus.

Previously it was shown 2 that the relation between the insulation resistance (I.R.) and percentage moisture content (per cent M.C.) of cotton can be expressed by the equation

$$log I.R. = -A log per cent M.C. + B.$$

It is now known that a single value of the slope A of this linear function does not suffice for all cottons, nor even for one sample of cotton. The slope may have values between 10 and 12 for the same sample depending upon the previous treatment of the cotton. Further, this equation holds only between about 3 per cent and 10 per cent

\* This is one of three papers by Walker and Quell, published in the March and April 1933 issues of *The Journal of the Textile Institute*. Abstracts of the other two papers appear in the Abstracts section of this issue of the *Bell System Technical Journal*. In the April 1929 *Bell System Technical Journal* there are two papers by R. R. Williams and E. J. Murphy, and E. B. Wood and H. H. Glenn, respectively, dealing with the problem of textile insulation.

It is the practice to compare the electrical insulating quality of different cotton samples by measuring the d.-c. insulation resistance after bringing the samples to equilibrium with 75 per cent relative humidity at 25° C., or at 85 per cent relative humidity at 37.8° C. (100° F.), equilibrium being approached from a lower humidity.

<sup>2</sup> Murphy and Walker, J. Phys. Chem., 32, 1761, 1928.

moisture content—corresponding to a range of relative humidity (hereinafter written R.H.) from 15 per cent to 85 per cent at 25° C. Nearly the whole range of moisture adsorption 3 of cotton between dryness and saturation may be characterized by three equations, as follows:

Below 3 per cent moisture content 4

$$log I.R. = -A per cent M.C. + B$$
 (I)

Between 3 per cent and 10 per cent moisture content

$$log I.R. = -A log per cent M.C. + B$$
 (II)

Between 10 per cent moisture content and saturation (about 25 per cent M.C.)

$$log I.R. = -A per cent R.H. + B$$
 (III)

Different values of A satisfy these equations, depending, as noted above, upon the previous treatment of the cotton and upon the direction of approach to equilibrium; whether this approach is from the dry state (along an absorption cycle), or from the wet state (along a desorption cycle). The experimental data include results of tests on one sample of raw cotton and two of water-boiled cotton. The following tabulation gives some idea of the limiting values of A and the conditions under which they will satisfy the equations:

|                       | Е            | quation I   | Equat                   | ion II               | Eq             | uation III     |
|-----------------------|--------------|-------------|-------------------------|----------------------|----------------|----------------|
| Absorption Desorption | 1.16<br>1.06 | No values 5 | Raw 10.5 -11 9.88-10.15 | Water-boiled 12 10.2 | 0.143<br>0.076 | 0.111<br>0.075 |

#### EXPERIMENTAL METHOD

Samples of cotton were brought to equilibrium with a flowing stream of air at 25° C., in which the partial pressure of water vapor could be adjusted to any desired value and maintained constant within 0.0115

<sup>3</sup> The word "absorption" is used to denote the taking up of a vapor, "desorption" the giving up of a vapor, and "adsorption" the general process without special indication of gain or loss. The use of these terms implies no assumptions with regard to the mechanism of the processes they denote.

<sup>4</sup> Below 2 per cent M.C., the I.R. of even raw cotton is difficult to measure, since it is above the limiting sensitivity ( $10^{13}$  ohms/mm. at 100 volts) of the insulation-resistance bridge used. Further tests are being made on this low range, using a more suitable type of cotton sample.

<sup>5</sup> Difficult to measure water-boiled cotton in the range where Equation I might apply.

mm. This is equivalent to variations of less than 15 parts per million in the water-vapor content of the air, or 0.05 per cent R.H. at 25° C. Insulation-resistance and moisture-content measurements were made at equilibrium <sup>6</sup> for a series of relative humidities in both absorption and desorption cycles on separate samples of the same cotton.

The *moisture content* was determined by mounting about 0.08 gram of cotton, wound in the form of a small skein, on a calibrated quartz-fiber balance, as described by McBain and Bakr.<sup>7</sup> The sensitivity of this spring was 0.03 gram = 1 inch deflection. The deflection caused by moisture adsorption was measured with a cathetometer, calibrated to 0.0001 inch. Measurements were reproducible to 0.0005 inch; thus the moisture adsorbed could be determined to 0.02 per cent.

The *insulation resistance* was measured by mounting 90 threads of cotton, each  $\frac{1}{2}$  inch long between metal electrodes, described in a previous communication.<sup>8</sup> This sample weighed about 0.05 gram.

The quartz spring was suspended in a long glass tube mounted within an air thermostat. A metal box with a hard-rubber top on which were mounted the electrodes was also contained in this thermostat. The flowing air streams from the same humidity apparatus were passed through the glass tube and the box in parallel.

A continuous record was obtained of the humidity of the flowing air mixture during each experiment, using an exceedingly sensitive humidity recorder, accurate to 0.05 per cent R.H. at 25° C., and sensitive to changes of but 0.02 per cent R.H. The humidity apparatus and the recorder are both described elsewhere.

Since the humidity apparatus supplied air of fixed absolute humidity, it was essential that constant temperature be maintained in the air thermostat; also that the electrode test box and quartz-spring tube be kept at the same temperature, to insure equilibrium of the samples at the same relative humidity. The air thermostat had walls  $5\frac{1}{2}$  in thick, including 3 in. of cork insulation. Copper-constantan thermocouples were mounted in each end of the electrode test-box and in the tube in close proximity to the samples. Efficient circulation of the air within the thermostat, by means of a fan driven from a motor mounted outside the thermostat, together with a sensitive mercury thermo-regulator operating a vacuum tube relay heat control, made it

<sup>&</sup>lt;sup>6</sup> Below 90 per cent R.H., equilibrium could be practically reached in but two to three hours, using this flowing stream or so-called "dynamic" method. Above 90 per cent, the time for equilibrium increases appreciably, being greater the nearer the test humidity is to saturation. Reference to the data in Table I will show the small differences between two to three hours' exposure and overnight values after 20 hours' exposure.

<sup>20</sup> hours' exposure.

<sup>7</sup> McBain and Bakr, Jour. Amer. Chem. Soc., 48, 690, 1926.

<sup>8</sup> April 1929 B.S.T.J., H. H. Glenn and E. B. Wood, Vol. VIII, p. 254.

<sup>9</sup> Walker and Ernst, Jour. Ind. and Engg. Chem. Analyt. Ed., 2, 134, 1930.

possible to maintain the thermocouples to within 0.01° C. of each other, and the temperature at any point within the thermostat remained constant to at least 0.01° C.

For several years prior to the development of the flowing air stream, or "dynamic" method of testing textiles, insulation-resistance measurements had been made on samples mounted on electrodes in a closed vessel in which 76 per cent R.H. was maintained by saturated NaCl solution. This vessel, in turn, was placed in an air thermostat nearly surrounded by a water bath maintained at 25° C.  $\pm$  0.1° C. Since the atmosphere above the salt solution is relatively stationary as compared with that in the flowing stream method, this procedure is defined as a "static" method. A statistical analysis, made by Dr. W. A. Shewhart of these laboratories, on data taken with both the static and dynamic methods, using samples from the same spool of cotton, clearly showed the superiority of the dynamic method. 10

#### EXPERIMENTAL DATA

Table I contains equilibrium data on moisture content and insulation resistance measurements of raw cotton made at a series of different relative humidities at 25° C., in both absorbing and desorbing cycles. Tables II and III contain similar data for two samples of water-

TABLE I

Moisture Content and Insulation Resistance Data on Raw Cotton in
Equilibrium with Constant Atmospheric Humidities during Repeated Absorption and Desorption Cycles at 25° C.

|  | - DOORI I   | TON CICEE  |   |  |
|--|---|--|---|--|
| Equilibrium Relative<br>Humidity at 25° C.   | Moistu  | re Content   | Insulation Re<br>½-in. Lengt<br>ply Cotton  | h of 30/2-   |
| %  | % M.C.  | log % M.C.   | megohms   | log megohms  |
| First Cycle of Increase  | ng Hum  | idity—Abs  | orption   |  |
| 8.8. 17.6. 26.3 (2 hours). 26.3 (overnight—20 hours) 45.7. 61.0. 71.5 (3 hours). 71.5 (overnight—21 hours) 82.3. 87.5. 92.7 (6 hours). 93.0 (overnight—24 hours) 99.2. Saturated air (1 hour exposure) | 2.19<br>3.10<br>3.76<br>3.83<br>5.19<br>6.49<br>7.66<br>9.39<br>11.00<br>13.95<br>14.25<br>22.30<br>24.50 | 0.340<br>0.491<br>0.575<br>0.584<br>0.72<br>0.813<br>0.882<br>0.885<br>0.973<br>1.041<br>1.145<br>1.154<br>1.349 | $\begin{array}{c} 1.76 \times 10^9 \\ 2.18 \times 10^8 \\ 2.21 \times 10^7 \\ 2.03 \times 10^7 \\ 5.81 \times 10^5 \\ 6.33 \times 10^4 \\ 8.84 \times 10^3 \\ 8.61 \times 10^3 \\ 1.05 \times 10^3 \\ 2.58 \times 10^2 \\ 41.6 \\ 38.0 \\ 5.75 \\ 4.17 \end{array}$ | 9.25<br>8.34<br>7.34<br>7.31<br>5.76<br>4.80<br>3.95<br>3.94<br>3.02<br>2.41<br>1.62<br>1.58<br>0.76<br>0.62 |

<sup>&</sup>lt;sup>10</sup> This analysis has been published by Dr. Shewhart, as an illustration of testing control in a book, "Economic Control of Quality of Manufactured Product," D. Van Nostrand, 1931. His conclusion regarding this analysis was, "We assume, therefore, upon the basis of this test, that it is not feasible for research to go much further in eliminating causes of variability." Page 21.

#### First Cycle of Decreasing Humidity—Desorption

Samples dried 20 hours with dry air at 25° C.

#### Second Cycle of Increasing Humidity—Absorption

| Saturated air (6 hours exposure)   30.00 11   1.48   2.64   0 | 26.2<br>36.2<br>56.5<br>71.5 (2 hours)<br>72.5 (overnight—18 hours)<br>Saturated air (6 hours exposure) | 4.68<br>6.47<br>8.35<br>8.45 | 0.591<br>0.670<br>0.811<br>0.922<br>0.927<br>1.48 | $\begin{array}{c} 1.11 \times 10^{7} \\ 1.39 \times 10^{6} \\ 3.87 \times 10^{4} \\ 3.34 \times 10^{3} \\ 2.79 \times 10^{3} \\ 2.64 \end{array}$ | 7.05<br>6.14<br>4.59<br>3.52<br>3.45<br>0.42 |
|---|---|------------------------------|---|---|--|
|---|---|------------------------------|---|---|--|

#### TABLE I (Continued)

| TABLE                                      | 1 (Conti | nuea)                  |   |                      |
|--|----------|------------------------|---|----------------------|
| Equilibrium Relative<br>Humidity at 25° C. | Moistu   | re Content             | Insulation Re<br>½-in. Lengt<br>ply Cotton  | h of 30/2-           |
| %  | % M.C.   | log % M.C.             | megohms   | log megohm           |
| Second Cycle of Decrea                     | sing Hur | nidity—Des             | sorption  |                      |
| 45.0 (2 hours)                             |          | 0.79<br>0.784<br>0.537 | $\begin{array}{c} 6.24 \times 10^{4} \\ 6.97 \times 10^{4} \\ 1.91 \times 10^{7} \end{array}$ | 4.80<br>4.84<br>7.28 |
| Samples dried 20 hours with dry air        | at 25° C |                        |   |                      |
| Third Cycle of Increas                     | ing Hum  | idity—Abs              | orption   |                      |
| 26.2                                       | 3.88     | 0.589                  | 9.95×10 <sup>6</sup>  | 7.00                 |
| Desorption from 26.29                      | % to 5%  | Relative H1            | umidity   |                      |
| 5.0  | 1.72     | 0.236                  | 5.67 ×108   | 8.75                 |

# Samples removed from apparatus and oven-dried at $80^{\circ}$ C. for 20 hours.

### First Cycle of Increasing Humidity—after oven-drying

| 45.7 | 5.07 | 0.705 | $4.76 \times 10^{5}$ | 5.68 |
|------|------|-------|----------------------|------|

 $<sup>^{\</sup>rm 11}$  Under the "saturated" condition in this case, moisture as dew was visible on the cotton.

boiled cotton, designated A and B respectively. These raw and water-boiled samples initially came from the same lot of raw insulating cotton.

The arrangement of the data in these tables shows the sequence in which the equilibrium values were obtained.

On Fig. 1 are plotted curves showing the relations between (a) per cent M.C. and per cent R.H., and (b) log I.R. and per cent R.H. for the raw cotton data in Table I. Fig. 2 contains a single curve showing the relation between log I.R. and log per cent M.C. for the raw cotton. Fig. 3 contains all three of these different types of curves for the two samples of water-boiled cotton. Since the data for these two water-boiled samples checked with one another so well, only one curve of each type was necessary to express the relations for both samples. Fig. 4 shows the relation between log I.R. and per cent M.C. for only the lower range of the experimental data for raw cotton, since up to about 5 per cent moisture content this relation as expressed by equation I on page 432 appears to hold better than equation II.

#### DISCUSSION OF EXPERIMENTAL DATA

#### Moisture Content-Relative Humidity Data

Exposure of raw cotton to a saturated atmosphere causes a reduction in the area of the moisture content-relative humidity hysteresis loop <sup>12</sup> (Fig. 1). Conversely, no reduction in the area of the loop on successive cycles is observed in the case of water-boiled cotton, perhaps due to this previous water treatment.

Sheppard and Newsome <sup>13</sup> found reductions in the area of this type of hysteresis loop for a treated cotton on successive cycles of exposure to high and low humidities. Our data show—(a) no change occurs in the position of the absorption curve for water-boiled cotton during two absorption cycles; (b) identical desorption curves for two different water-boiled samples; (c) identical desorption curves for raw cotton in three cycles, as well as a suggestion that the third absorption curve (only one point obtained—at 26 per cent R.H.) coincides with the second absorption curve; (d) a reduction in area in the raw cotton hysteresis loop on the second absorption cycle; (e) this reduced area for the raw cotton differs but little, both in area and location, from the hysteresis loop for the water-boiled cottons.

<sup>12</sup> This type of hysteresis loop in the moisture adsorption properties of cotton has been discussed at length by Urquhart and Williams, Jour. Text. Inst., 15, T138, 1924; also Shirley Inst. Mem., 3, 49, 1924.
13 Sheppard and Newsome, Jour. Phys. Chem., 33, 1819, 1929.

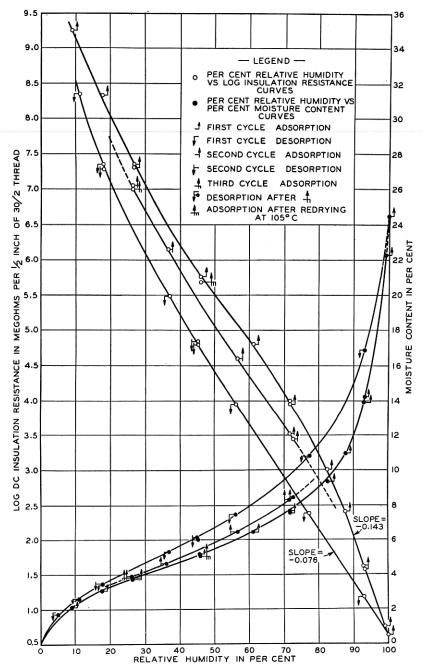


Fig. 1—Relations between relative humidity and the moisture content and log insulation resistance of raw cotton at 25° C.

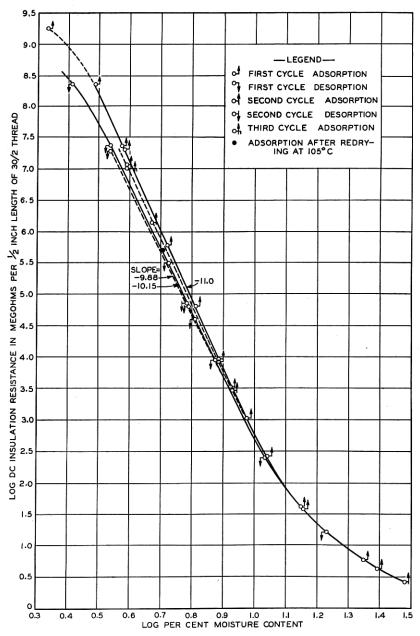


Fig. 2—Relation between log of per cent moisture content and log insulation resistance of raw cotton at  $25^{\circ}$  C.

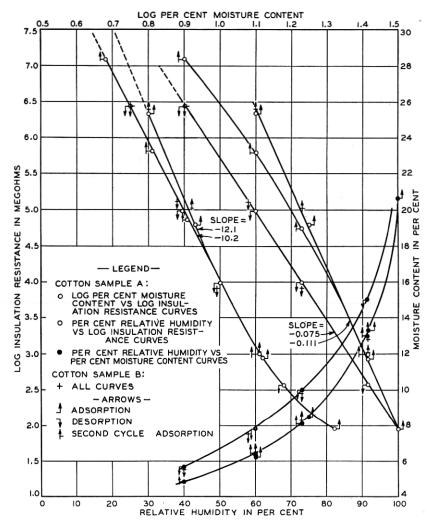


Fig. 3—Relations between relative humidity, moisture content and log insulation resistance of water-boiled cotton at 25° C.

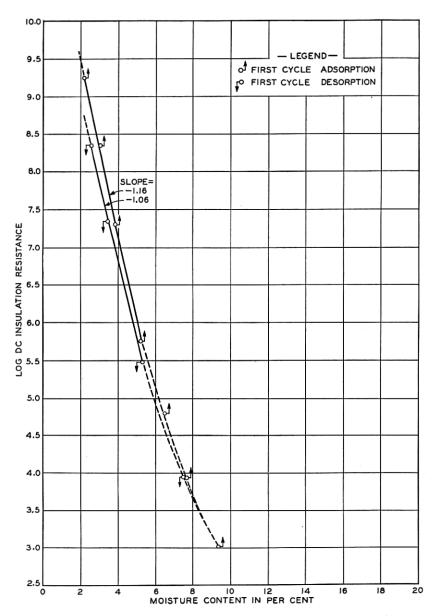


Fig. 4—Relation between per cent moisture content and log insulation resistance of raw cotton at 25° C.

This evidence is considered to indicate the close control of the testing conditions made possible with the dynamic method, and suggests that the decreases in area in the loops obtained by Sheppard and Newsome may be due to small variations in thermostat temperature about a mean value. On absorption this would have the effect of giving too high a moisture content at equilibrium, due to hysteresis; on desorption the equilibrium value would be too low.

TABLE II

Moisture Content and Insulation Resistance Data on Water-Boiled Cotton in Equilibrium with Constant Atmospheric Humidities during Absorption and Desorption Cycles at 25° C.

30/2 Cotton—Sample A

| 30/2 Cono  | n—Sam <sub>1</sub>             | ne n                          |   |  |
|--|--------------------------------|-------------------------------|---|--|
| Equilibrium Relative<br>Humidity at 25° C.             | Moistu                         | re Content                    | Insulation R ½-in. Leng ply Cotto   | esistance per<br>th of 30/2-<br>n Thread |
| %  | % M.C.                         | log % M.C.                    | megohms   | log megohms                              |
| First Cycle of Increase                                | ng Hum                         | idity—Abso                    | orption   |  |
| 60.0<br>75.0<br>91.5<br>Saturation (20 hours exposure) | 6.29<br>8.53<br>13.32<br>20.70 | 0.80<br>0.93<br>1.12<br>1.32  | $\begin{array}{c} 2.21 \times 10^{6} \\ 6.3 \times 10^{4} \\ 8.93 \times 10^{2} \\ 9.35 \times 10 \end{array}$      | 6.34<br>4.80<br>2.95<br>1.97             |
| First Cycle of Decreas                                 | ing Hum                        | idity—Desc                    | orption   |  |
| 91.5<br>73.0<br>60.0<br>40.0                           | 15.00<br>10.05<br>7.85<br>5.62 | 1.18<br>1.00<br>0.895<br>0.75 | $\begin{array}{c} 3.80 \times 10^{2} \\ 9.75 \times 10^{3} \\ 9.46 \times 10^{4} \\ 2.77 \times 10^{6} \end{array}$ | 2.58<br>3.99<br>4.98<br>6.44             |
| Samples dried 20 hours with dry air                    | at 25° C                       | •                             |   |  |
| Second Cycle of Increa                                 | sing Hun                       | nidity—Ab                     | sorption  |  |
| 40.0<br>60.0<br>73.0<br>91.5                           |                                | 0.68<br>0.81<br>0.91<br>1.11  | $\begin{array}{c} 1.25 \times 10^{7} \\ 6.45 \times 10^{5} \\ 5.95 \times 10^{4} \\ 1.00 \times 10^{3} \end{array}$ | 7.097<br>5.81<br>4.75<br>3.00            |

## Insulation Resistance-Relative Humidity Data

Figs. 1 and 3 show hysteresis loops in the log I.R.—per cent R.H. curves, for both raw and water-boiled cotton. Hysteresis loops in this relation were shown in a previous paper <sup>2</sup> but no evidence was available to show the effect on the loop area of exposure of the <sup>2</sup> loc. cit.

textile to air saturated with water vapor. From the evidence given in this paper it is seen that exposure to saturated air causes a reduction in the hysteresis loop area for both raw and water-boiled cotton. This behavior is in contrast to the moisture content-relative humidity relation in which a reduction in loop area is observed for raw, but not for water-boiled cotton.

Between 11 per cent moisture content (about 88 per cent relative humidity) and saturation, the log I.R.—per cent R.H. relation appears to be nearly linear for raw cotton, and on the desorption curve the relation is linear down to about 45 per cent R.H. For water-boiled

#### TABLE III

MOISTURE CONTENT AND INSULATION RESISTANCE DATA ON WATER-BOILED COTTON IN EQUILIBRIUM WITH CONSTANT ATMOSPHERIC HUMIDITIES DURING ABSORPTION AND DESORPTION CYCLES AT 25° C.

| 30/2 Cotton—Sam | рle | B |
|-----------------|-----|---|
|-----------------|-----|---|

| Equilibrium Relative<br>Humidity at 25° C.                   | Moistu      | re Content            | Insulation Re<br>½-in. Lengt<br>ply Cotto   | th of 30/2-          |  |
|--|-------------|-----------------------|---|----------------------|--|
| %  | % M.C.      | log % M.C.            | megohms   | log megohms          |  |
| First Cycle of Inc   | reasing Hum | idity—Abs             | orption   |                      |  |
| 73.0<br>60.0<br>91.5   | 6.33        | 0.92<br>0.80<br>1.11  | $\begin{array}{c} 1.08 \times 10^5 \\ 2.565 \times 10^6 \\ 1.05 \times 10^3 \end{array}$      | 5.03<br>6.41<br>3.02 |  |
| Exposed to air at 100% R.H. overnight—no measurements taken. |             |                       |   |                      |  |
| First Cycle of Deci  | reasing Hum | idity—Desc            | orption   |                      |  |
| 73.0   | 7.57        | 0.99<br>0.88<br>0.748 | $\begin{array}{c} 8.33 \times 10^{3} \\ 1.31 \times 10^{5} \\ 2.78 \times 10^{6} \end{array}$ | 3.92<br>5.12<br>6.44 |  |

cotton, this relation appears to be substantially linear over the full range investigated, from 60 per cent R.H. to saturation on the absorption curve, and from saturation down to about 40 per cent R.H. on the desorption cycle. Curiously, the second absorption cycles for both raw and water-boiled cotton do not exhibit such a linear relation, although in the range above 90 per cent R.H. it is possible that these second absorption curves join the initial absorption curves and become linear in the upper range.

These curves emphasize the necessity for systematic treatment of textiles in making electrical measurements under definite humidity conditions, since the hysteresis in the per cent R.H.—per cent M.C.

curves indicates that similar hysteresis in the log I.R.—R.H. curves is due to adsorption of different amounts of moisture by cotton, even when exposed to the same relative humidity. The amount of moisture adsorbed is dependent upon the direction from which equilibrium is approached.

Unfortunately, the behavior of cotton is still further complicated, so that additional precautions must be taken in measuring its electrical properties.

The difference in the effect of saturation on the area of the hysteresis loops for raw and water-boiled cotton as shown by the log I.R.—per cent R.H. and per cent R.H.—per cent M.C. curves suggests that some change in structure of cotton occurs when it absorbs much moisture, and this change in structure has a more or less permanent effect on the subsequent behavior of the material. Verification of this suggestion is found in the log I.R.—log per cent M.C. relation which will now be discussed. The study of this log relation has led to many improvements in methods now employed in the fundamental investigation of the electrical properties of cotton and in inspection methods employed in the commercial purification of cotton for electrical purposes.

#### Insulation Resistance-Moisture Content Data

The curves expressing the relation between log I.R.—log per cent M.C. are shown, in Figs. 2 and 3, to be curved, and not linear over the whole range as suggested in an earlier paper.<sup>2</sup> The data on raw cotton extends over the wider range, and the curve appears to be sigmoid in shape, exhibiting curvature above 10 per cent and below 3 per cent moisture content. Only in the middle range between these moisture content limits is the curve sufficiently linear so that equation II applies. The accuracy of the curve below about 5 per cent M.C. progressively decreases, due to difficulties in measuring the extremely high resistances, and about all that can be said of this range at present is that the log I.R.—per cent M.C. relation expressed by equation I, appears to fit the data better than the log I.R.—log per cent M.C. relation as expressed by equation II.

The definite curvature above 10 per cent M.C., not observed previously,<sup>2</sup> was found through the use of the dynamic method and the measurement of insulation resistance and moisture content values simultaneously on similar samples of cotton taken from the same supply.<sup>14</sup>

<sup>14</sup> In the vicinity of saturation, an effect similar to polarization can cause errors in the measurement of insulation resistance. The errors result in high insulation resistance values, accentuating the curvature of the curve above 10 per cent moisture

In the range where equation II is applicable the relation is seen to be a family of convergent lines with slopes (the constant A in this equation) having values between 10 and 12. These convergent lines focus at about 10 per cent M.C. (log per cent M.C. = 1).15 The actual value of the slope A in any test depends upon several factors. It is primarily dependent upon the previous treatment of the cotton. Water-boiled cotton which has been dried from the wet state at high temperature in such a manner as to secure a high I.R. for a given moisture content, in consequence, gives a line with maximum slope. Exposure to high humidities, or saturation of the cotton with water vapor causes the subsequent desorption and absorption equilibrium values to lie on a line of less slope. In the case of raw cotton, the more moisture absorbed by the cotton from a saturated atmosphere, the lower is the desorption value of A; its lower limit appears to depend to some extent upon the time of exposure and the amount of moisture absorbed. (Note the difference in the desorption slope after the first and second exposure of the raw cotton to saturated air. After the first cycle with 24.5 per cent maximum moisture content, A = 10.15; after the second with 30 per cent M.C.,  $A = 9.88^{16}$  This difference is greater than experimental error.)

Raw cotton shows a distinct difference from water-boiled cotton in one respect. On the second absorption cycle the slope A has a value

content. This effect is not readily detectable, using the slow-period H.S. type Leeds and Northrop galvanometer. When first found, it was assumed that the entire curvature of the curve above 10 per cent M.C. was due to this effect, but such was not the case. The effect is not true polarization, but is simply due to electrical heating. Above 90 per cent relative humidity for raw cotton and above 98 per cent R.H. for washed cotton, the measuring current, using 100 volts potential is sufficient to heat the cotton appreciably. This I<sup>2</sup>R loss can raise the textile temperature about 0.1° C. at 90 per cent R.H., and about 10° C. at saturation for raw cotton. These temperature rises were measured, using thermocouples of No. 40 wire braided into the threads of textile mounted on the electrodes. The heating effect causes evaporation of moisture from the cotton, thus raising the insulation resistance.

All measurements in this paper above 75 per cent R.H. for raw cotton and above 90 per cent R.H. for washed cotton were made with a special micro-ammeter having a period of but 0.8 second, as compared with the period of the H.S. type galvanometer of about 40 seconds. The temperature rise at saturation does not become evident for at least three seconds after voltage application. Until this short interval has elapsed the micro-ammeter gives a steady reading identical with the instantaneous value, and as the thermocouple records increasing temperature the meter deflection

drops.

This behavior is a hysteresis effect of a somewhat different character from that observed in the two relative humidity relations previously discussed, since in this case the effect is independent of relative humidity, and appears to be related to the distribution of moisture in the cotton and to the manner in which this moisture is

held by the cellulose. This will be discussed somewhat more fully later.

16 The value of 24.5 per cent M.C. does not necessarily indicate a true saturation value, but only a M.C. after exposure to a definite saturated atmosphere for one hour. The 30 per cent value probably represents some value above the critical saturation point at exactly 100 per cent R.H. (which would be exceedingly difficult to obtain), since actual deposits of dew were visible on the sample.

# TABLE IV

EFFECT OF HIGH RELATIVE HUMIDITY (88%) AT DIFFERENT TEMPERATURES ON THE INSULATION RESISTANCE OF COTTON AT 75% RELATIVE HUMIDITY AND 25° C.

| Sequence of Equilibrium Conditions  75 % R.H.—25 ° C 88% R.H.—22 ° C 9 Dried overnight 75% R.H.—25 ° C 2 Bried overnight Dried overnight 75% R.H.—38 ° C 88% R.H.—38 ° C 1 Dried overnight 75% R.H.—25 ° C 88% R.H.—38 ° C 1 Dried overnight 75% R.H.—25 ° C 88% R.H.—38 ° C 1 Dried overnight 75% R.H.—25 ° C | 1<br>73<br>9.0<br>9.0<br>11.5<br>4.3 | 2<br>88<br>9.8<br>50<br>2.1<br>31<br>0.84<br>4.3 | 3<br>80<br>111.3<br>57<br>2.1<br>34<br>0.78 | Washed 4 4 4 90 12.0 60 2.6 3.6 3.6 2.9 2.9 2.9 2.9 | Insulation Resistance of Cotton in Kilomegolums per 4-in. Thread Washed Cotton Samples 17         Raw Compute Samples 11         Raw Compute Samples 11         Raw Compute Samples 11         Raw Compute Samples 11         Raw Compute Samples 12         A Sam Compute Sam Compute Samples 12         A Sam Compute Sam | Resistan Samples 1  6  6  102 12.5 65 2.6 41 11.53 | 7 7 100 9.5 2.6 3.6 1.90 3.1 6.0 | 8<br>8<br>159<br>150<br>15.0<br>94<br>3.8<br>58<br>2.3<br>7.3 | Avg. 100 11.0 61 2.6 36 1.111 28 2.8 5.5 | 1 1 4.6 4.5 4.5 4.6 | R 4.8 0.48 0.48 0.09 0.09 | Raw Cotton Samples 18  3 4 (a)  4.7 4.7 4.7 4.7  0.47 4.6 4.6  1.95 4.3 4.3  1.95 4.3 4.3  0.09 — —  1.7 4.6 4.6  0.36 — — | nn Sam<br>4 4.7 4.7 4.6 | Avg. (3) 4.7 4.7 4.6 4.6 4.6 | Average (b) Exposed to 88% R.H.  0.48  2.95 0.137 1.95 0.09 |
|--|--------------------------------------|--|---|---|---|--|----------------------------------|---|--|---------------------|---------------------------|--|-------------------------|------------------------------|---|
| C  | 34                                   | 33   | 34  | 34  | 32  | 38   | 38                               | 20  | 37                                       | 4.5                 | 2.1                       | 1.8  | 4.5                     | 4.5                          | 1.95  |

Two of these raw cotton samples (1 and 4) were used as controls to check the reproducibility of the 75% humidity condition. They were not exposed to the 88% humidity conditions. Therefore the averages of 1 and 4 are given under (a). The averages of the other two (2 and 3), which were exposed to the sequence of 88% conditions, are given under (b). 17 These samples were washed at 40° C. in accordance with the procedure described in the paper, "Naturally Occurring Ash Constituents of Cotton," by Walker and Quell.

intermediate between the initial absorption and desorption slopes, thus indicating some reversibility in the properties of the cotton which determine these slopes, due to the drying effect after the initial desorption test. Water-boiled cotton does not show this effect, the slope of the second absorption curve being identical with that of the initial desorption curve, *under the conditions of drying used for these tests*. This behavior is consistent with some experiments made to determine if the initially high insulation resistance observed in some cases with water-boiled cotton could be restored by some simple means after the resistance had been adversely affected by exposure to high atmospheric humidities.

In the course of some I.R. tests made on washed cotton the control samples of raw cotton used to check each I.R. experiment to assure the same humidity and temperature conditions were found to have suddenly changed from 4.5 kilomegohms—their normal value under the test conditions—to 1.8 kilomegohms under these conditions. These controls had been exposed to atmospheric humidity conditions of 83 per cent R.H. at 32° C., while a new set of washed cotton samples were being prepared for test. Since it was particularly desirable to continue the use of the same control samples, an attempt was made to restore them to their original conditions by drying. Air at less than 0.1 per cent R.H. at 25° C., was passed over these samples for 40 hours at room temperature. When subsequently measured their resistances had increased from 1.8 to 2.9. Further drying for 48 hours at 105° C. caused a further gain of but 0.1 kilomegohm. Conversely, similar tests on washed cotton showed no improvement. A bundle of washed cotton was dried at 105° C. Instead of giving an I.R. of between 100 and 400 kilomegohms, normal for other similarly washed and dried samples, the resistance was but 23 kilomegohms. Chemical analyses of this cotton gave no indication that this low value was due to electrolytic contamination. Neither redrying of this cotton in a vacuum oven at 80° C., nor drying in an air-oven at 105° C., gave any improvement; in fact the resistance after such redrying was but 18 kilomegohms.

However, this washed cotton was greatly increased in I.R. by simply rewetting with excess water and drying rapidly at 105° C.<sup>19</sup>

From this discussion of the data it is seen that three types of linear equations may be used to express fairly accurately the relation between insulation resistance and the moisture-absorbing properties of cotton over a range of atmospheric relative humidity from saturation down

 $<sup>^{19}</sup>$  Samples A and B used to secure the data in Tables II and III were from this test. After rewetting and oven-drying at 105° C., sample A gave 108 kilomegohms and B gave 63 kilomegohms at 75 per cent R.H.—25° C.

to nearly dryness. These equations, with the respective ranges of relative humidity (and, therefore, of moisture content) over which each is significant, are given on page 432.

It is concluded that exposure of cotton to high atmospheric humidity causes a change in the gel structure due to absorption of moisture, since the insulation resistance of the material as measured at some comparable condition (75 per cent R.H. at 25° C.) is less after such high humidity exposure than before, even if the cotton is well dried before testing.

The temperature of such exposure to high atmospheric humidity also affects the subsequent electrical properties of the cotton. Data to show this temperature effect are given in Table IV.

#### Temperature Effects

Effect of Temperature at High Humidity on I.R. of Air-dried Cotton

Table IV contains the results of a series of tests on the I.R. of samples of raw and washed cotton which were exposed to several cycles of high humidity and dry air, each cycle being as follows:

- (a) Equilibrated and measured at 75% R.H.—25° C.
- (b) Equilibrated and measured at 88% R.H.—at to C.
- (c) Dried for 16 hours with a stream of dry air at 25° C.

This cycle was repeated four times, the only difference in each case being the temperature (t° C.) at which the 88 per cent R.H. equilibrium These temperatures were successively—22°, 30.2°, tests were made. 38°, and 22° C. In all, eight samples of washed cotton and four samples of raw cotton were used in the test. Two of the raw cotton samples (1 and 4) were not exposed to the 88 per cent humidity conditions, but were used as control samples to check the reproducibility of the 75 per cent humidity conditions in each cycle.20

Table V is a condensation of Table IV. The decreases in insulation

20 Five measurements each were made on these two control samples during the course of the test, giving a mean value of 4.52 kilomegohms, with a standard deviation of but 0.13 kilomegohms.

The differences in the initial values of I.R. for the eight washed samples are not due to lack of control, either in the method of washing or in the method of testing,

due to lack of control, either in the method of washing or in the method of testing, but to actual differences in the equilibrium moisture contents. For example—sample 1 gave 73 kilomegohms initially, and sample 6 gave 102 kilomegohms. Their respective moisture contents, under the test conditions, were 8.17% and 8.00%. Using Equation II, and with the constant A=10, the values of B were calculated in this equation as 13.99 and 14.05 respectively for samples 1 and 6. Assuming these samples to be of equal purity, since they were washed in an efficient manner, 21 it is reasonable to take B=14.03 for both samples. From this value of B, the IR of sample 1 was calculated at a moisture content of 8.00 per cent. giving 98 I.R. of sample 1 was calculated at a moisture content of 8.00 per cent, giving 98 kilomegohms, a satisfactory check with sample 6 at the same moisture content.

21 Walker & Quell, Jour. Text. Inst. 24, T141, 1933.

resistance of both raw and washed cotton when measured at 75 per cent relative humidity and 25° C., after exposure to the 88 per cent relative humidity conditions and dried,<sup>22</sup> are given in percentage of the *initial* 75 per cent—25° C. insulation resistances.

#### TABLE V

Percentage Reduction in the Insulation Resistance of Raw and Washed Cottons at 75 Per Cent Relative Humidity and 25° C., after Successive Exposures to 88 Per Cent Relative Humidity at t° C.

| Temperature (t° C.) of the Successive 88% R.H. Cycles | % Reduction in Insulation 25° C. after each Washed | on Resistance at 75%—<br>88% R.H. Cycle<br>Raw |
|---|--|--|
| 22° C   | 39%  | 37%<br>58.5%                                   |
| 38.0° C   |  | 64.5%<br>59.5%                                 |

Exposure of cotton to high humidity (in this case 88 per cent) alters the properties of the material in such a way that its insulation resistance when subsequently measured at 75 per cent relative humidity and 25° C., is lower than the insulation resistance measured at the 75 per cent condition before such exposure to 88 per cent humidity. This decrease in insulation resistance observed at 75 per cent humidity and 25° C., becomes progressively greater the higher the temperature of the 88 per cent humidity exposure, but on again exposing the cotton to 88 per cent humidity at the reduced temperature of 22° C., after exposure at 38° C., the insulation resistance subsequently measured at 75 per cent humidity and 25° C., is greater than after the 88 per cent—38° exposure, but less than when measured at this condition after the original exposure to 88 per cent humidity and 22° C., thus indicating that some reversal occurs in the temperature effect.

The fact that in each test the percentage reduction is of the same order of magnitude for raw and washed cotton, suggests that the effect is structural and not related to the quantity of electrolytic impurities which may be present.

An important feature of the data recorded in Table IV is that the insulation resistance of washed cotton is reduced by exposure to 88 per cent R.H. A natural question is—What would be the resistance of this cotton if exposed to 100 per cent R.H. instead of 88 per cent, or brought directly to equilibrium with 75 per cent R.H. at 25° C., from the wet state without oven-drying? Tests have been made to determine these points. Washed cotton, dried at 105° C., then con-

<sup>22</sup> The samples were dried with a stream of very dry air at 25° C. after each exposure to the 88 per cent humidity conditions to avoid the hysteresis effect, which would occur if the samples were brought back to the 75 per cent humidity condition directly from the higher humidity. Before starting the test all samples were similarly dried.

ditioned at 100 per cent R.H., gave an I.R. when tested at 75 per cent R.H. at 25° C., of 25 kilomegohms.<sup>23</sup> Its insulation resistance on being brought directly from the wet state to 75 per cent R.H. at 25° C., was but 3.7 kilomegohms, being in this case lower than the resistance of raw, unwashed cotton in Table IV. Of course, if the raw cotton could be wet with water without undergoing any change due to reduction in ash content, no doubt its resistance would be much lower than that of similarly treated water-washed cotton, since this effect appears to be structural and certainly is not dependent upon electrolytic impurities.

Effect of Temperature of Drying Wet Cotton on its Insulation Resistance

The higher the temperature at which wet, water-boiled cotton is dried, the higher is its insulation resistance. Such cotton, dried at 105° C., 120° C., and 162° C., from the wet state, gave 139, 171, and 201 kilomegohms respectively, when subsequently equilibrated at 75 per cent R.H. at 25° C.

THEORY

The most important fact to be derived from these experimental data is that cotton may have a range of insulation resistance values for any single moisture content over at least the average atmospheric humidity range, from about 15 to 85 per cent R.H. Another interesting fact is that the insulation resistance of cotton when measured at definite test conditions depends to a surprising extent upon the previous exposure of the material to prevailing atmospheric humidity and temperature conditions, prior to such tests.

This behavior suggests that the absorption of appreciable quantities of moisture causes changes in the cotton structure, which affect the mechanism of current conduction. This change in structure, no doubt a result of swelling, an effect investigated by Collins,<sup>24</sup> appears to be a difficultly reversible alteration in the colloidal gel structure of the cellulose, even after subsequent removal of the moisture by drying. These effects, rather small to be detected by ordinary methods, are revealed by the extremely sensitive electrical tests, since very small changes in moisture content cause large changes in insulation resistance.

Since the substitution of acetyl for hydroxyl groups in cellulose is accompanied by a marked reduction in the moisture adsorption,<sup>25</sup>

 $<sup>^{23}</sup>$  This oven-dried material gave 80 kilomegohms when not exposed to the 100 per cent R.H. before test.

 <sup>&</sup>lt;sup>24</sup> Collins, Jour. Text. Inst., 21, T311, 1930.
 <sup>25</sup> Wilson and Fuwa, Jour. Ind. and Engg. Chem., 14, 913, 1922. (This lower moisture adsorption of cellulose acetate has been observed in our own experiments. See also reference <sup>13</sup>.)

it appears likely that adsorption of moisture is largely a function of free hydroxyl groups. From our data it appears that when wet cotton is dried rapidly at high temperatures, the internal or micelle surface contains a minimum of hydroxyl groups. As the cotton is permitted to absorb more and more moisture, the hydroxyl groups which were oriented into the interior of the micelles by the drying process where their hygroscopic property is, in effect, neutralized by attraction of associated molecules, are attracted to the surface to hold the absorbed moisture. On drying, these hydroxyl groups do not return readily to the interior and a greater number of water molecules are held at any relative humidity, thus accounting for the normal hysteresis effect observed in the moisture content-relative humidity relation.

Practically all of the experimental data discussed in this paper were secured during 1928 and 1929, and the above theory was proposed at that time. Apparently at about the same time Urquhart questioned the explanation offered some years previously by Urquhart and Williams <sup>26</sup> to account for hysteresis in the moisture relations of cotton, depending upon a modification of the Zsigmondy pore theory. In June 1929 <sup>27</sup> Urquhart proposed a theory comprising the essential features of the orientation of hydroxyl groups as offering a better explanation than the pore theory for the moisture-adsorbing properties of cotton. The general outline just given in connection with the study of the electrical properties of cotton is much the same as the more complete theory discussed by Urquhart.

However, further consideration of our experimental data led to the conclusion that neither the pore theory nor the orientation of hydroxyl groups completely accounts for the hysteresis effect in the log I.R.—log per cent M.C. relation.

As mentioned above, rapid drying of wet cotton under proper conditions is assumed to give internal surfaces containing a minimum of hydroxyl groups. This idea can be qualified as follows: Either such drying conditions are conducive to the presence of a minimum of hydroxyl groups on the internal surfaces, or they are conducive to a less uniform distribution of these groups on these internal surfaces.

Consequently, on initially absorbing moisture from such a dried condition, the moisture associated with hydroxyls will not be uniformly distributed and the conduction of current through the cotton along these internal surfaces will be somewhat *discontinuous*. On desorption from saturation, moisture will be removed in a more regular

Urquhart and Williams, Jour. Text. Inst., 15, T433, 1924; also Shirley Inst. Mem., 3, 197, 1924.
 Urquhart, Jour. Text. Inst., 20, T125, 1929.

fashion from more uniformly distributed hydroxyls, and therefore on any descending curve the conduction of current can be considered to be along more continuous paths. This difference in continuity of moisture paths is sufficient to account for high insulation resistance values on absorption and low values on desorption curves, for each equilibrium moisture content. The actual insulation resistance in any given case depends upon the degree of continuity of such moisture paths and this in turn depends upon the previous treatment of the material.

Also it seems reasonable to consider that some of the properties of cotton under discussion may be explained to better advantage by the pore theory initially proposed by Urquhart and Williams, since it does not appear that all of the moisture which saturated cotton can absorb is necessarily associated with hydroxyl groups. In considering the pore theory, high insulation resistance values during absorption can be accounted for by a blocking effect of the pore entrances by a few water molecules. This pore blocking effect suggested by Peirce swould cause greater discontinuities in moisture paths through the cotton, and therefore higher insulation resistance for a given moisture content.

Since it is planned to discuss this theory more in detail in a separate paper when experimental data now being secured are available, only the above brief outline is given at this time.

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<sup>&</sup>lt;sup>28</sup> Peirce, Jour. Text. Inst., 20, T133, 1929.