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FINAL REPORT

AUTOGENOUS ACCELERATED CURING OF CONCRETE CYLINDERS

Part II

Development of a Moisture Measuring Method

by

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Virginia Highway Research Council
(A Cooperative Organization Sponsored Jointly by the Virginia
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PREFACE

The Research Council's studies of early determination of compressive strength of concrete stored in water baths at elevated temperatures were initiated in 1967 as a part of the State funded research program. The results of this research were presented by K. H. McGhee in his report entitled "Water Bath Accelerated Curing of Concrete".

Under the work plan by L. M. Cook entitled "An Investigation of the Moisture-Temperature Relationships -- Autogenous Accelerated Curing for Early Determination of Concrete Strength Potential", the study was extended to autogenous curing. The extended study was approved for financing under Federal Highway Planning and Research Funds on May 14, 1969. The objectives of this project were:

1. To extend knowledge of the thermal and moisture behavior of concrete subjected to high curing temperatures during autogenous curing.
2. To examine the influence that variables such as cement type, cement factor, water-cement ratio, and admixtures have on moisture and temperature.
3. To correlate the accelerated strengths of autogenously cured cylinders with those of 28 and 91 day old moist cured cylinders.

Concurrently with the Council's research project, ASTM Committee C-9 was developing standard methods of testing. Several questions raised during the ASTM efforts were closely related to the Council's work. As a result of a discussion with Federal Highway Administration personnel in October 1969, a limited study of the curing container characteristics and storage conditions was undertaken to supplement the major project effort.

The total project ultimately involved preparation of approximately 300 batches of concrete in the laboratory with all of the necessary testing. Calibration of moisture measuring instrumentation and continuous recording of temperature and moisture for the test specimens resulted in voluminous data.

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For maximum intelligibility and usefulness, the report on this project has been subdivided into five parts as follows:

Part I -- Strength Results

Part II -- Development of a Moisture Measuring Method

Part III -- Temperature Relationships

Part IV -- Moisture Relationships

Part V -- ASTM Cooperative Testing Program with Additional Emphasis on the Influence of Container and Storage Characteristics (Supplemented by Data on Water Bath Curing From an Earlier Council Project)

In Part V, it was deemed desirable to include data from the earlier study by McGhee so as to give a comprehensive picture of the Council's portion of the ASTM Cooperative Testing Program. While some of the work reported in Part V was not a part of the autogenous curing study, most of it was done as a part of the project so that its inclusion in the project report seems logical.

Each part of the report contains sufficient background information to enable it to stand alone as coverage of the aspect of the project reflected in its title. The titles, in general, reflect the project objectives. Taken together, these five reports represent the final report on the study of Autogenous Accelerated Curing of Concrete Cylinders.

SUMMARY

Through a literature study, approximately fifty methods of measuring moisture were investigated to ascertain a suitable method for use in measuring moisture changes in 6 inch by 12 inch concrete cylinders cured by the autogenous accelerated curing method.

The resistance method was selected and the Bouyoucos moisture gage was evaluated in the laboratory to determine its' limitations and capabilities. Operation and calibration procedures were developed and the moisture gages used in the autogenous accelerated curing study were grouped and calibrated in a statistically defensible manner. Correction factors for the influence of salt ions and temperature were also developed.

Results of this study led to the following conclusions:

1. Of the various methods described in the literature, the Bouyoucos moisture gage was judged to be a satisfactory method of measuring moisture in the concrete cylinders subjected to autogenous curing.
2. The influence of salt ions and temperature on the moisture gage resistance can be measured and the necessary correction factors applied to the results.

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INTRODUCTION

Under conditions of autogenous curing, the internal moisture content of concrete assumes a larger role in the strength development than for conventional curing, because external water is not present to provide uniform moist curing conditions. It is conceivable that for very low water-cement ratios the process of self-desiccation would reduce the humidity below the 80 percent stated by Powers (1) as necessary for continued hydration. The movement of moisture and its relation to temperature are of considerable importance for conditions of autogenous curing.

McGhee (2) has studied the temperature relationships of accelerated methods employing water as a curing medium for accelerated compression testing using water temperatures ranging from 95°F to 212°F. He found that efficiencies (the ratio of accelerated to 28-day strengths) ranged from 35 to 65 percent. Because of the possibility of variations in moisture during curing, the relationships between strength developed and changes in moisture and temperature accompanying autogenous curing are especially important (3) and refinement of the method would be benefited by a detailed study similar to that reported by McGhee.

OBJECTIVES

In order to accomplish the objectives of a detailed study of moisture changes during autogenous curing, Part IV of this report(4), a method of measuring moisture had to be developed. The scope of this investigation was restricted to methods applicable to 6" x 12" cylinders cured by the autogenous accelerated curing method (5) and included literature studies concerning accelerated strength development, thermal and moisture properties of freshly mixed concrete, and methods of measuring moisture movements and contents in freshly mixed and hydrated concrete. This study ultimately involved an investigation of the properties and the capabilities of the moisture gages used in this study and to group and calibrate the gages in a statistically defensible manner.

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MOISTURE MEASUREMENT

Numerous methods have been developed for determining the moisture characteristics of many different materials, including concrete. Although a great deal of knowledge regarding cement hydration has been acquired by determining the evaporable and non-evaporable water contents, (6) these determinations may destroy the specimen or may interject discontinuities into the moisture behavior. In general, the various methods that have been utilized with concrete are broken down into two classes, or groups, (7) as follows:

Group I. Direct, destructive, and/or disturbed — This group includes those methods in which the water in the material is disturbed. All the methods in this group will destroy the specimen and/or interject discontinuities in the moisture behavior.

Group II. Indirect, nondestructive, and/or undisturbed — This group comprises the methods in which the water in the material is not disturbed.

A discussion of the moisture measuring methods within each group, including factors affecting selection and advantages and disadvantages, has been presented by the author. (8)

Selection of Moisture Sensing Method

In the study of moisture changes during autogenous accelerated curing, 6" by 12" concrete cylinders cast in thin metal molds were sealed in autogenous curing containers (9) for 47 hours. The curing medium was the heat generated by the cement hydration, while the autogenous containers acted as insulators to hold in the heat of hydration. Therefore, the cylinders could not be removed from the containers during the curing period to be tested for moisture parameters. Consequently, the moisture sensing elements had to be embedded in the concrete and thus sealed in the curing containers. It was also essential that the curing containers not be disturbed — tilted, vibrated, or moved — during these 47 hours because of possible disruption of the developing concrete structure. It was estimated (5) that a temperature range from at least 70°F to 120°F would be encountered during the 47-hour curing period. The interval between moisture measurements was established as one half hour, and at least two sensing elements per cylinder were employed. Because of the large number of sensing elements involved, then, these elements had to be adaptable to automatic recording.

After investigation of approximately 20 different methods of measuring moisture, the resistance method was selected. The sensing element used was the Bouyoucos gypsum type moisture gage. The following were reasons for selecting the resistance method. The gages were:

1. Relatively inexpensive,
2. small and therefore would essentially measure moisture at a point,

3. of a size that would allow moisture movement to be determined in a standard 6" by 12" concrete cylinder,

4. easy to install and could be sealed inside the cylinder mold,

5. used with lead wires that adapted themselves very nicely to the autogenous containers so that no heat loss occurred,

6. easily read (less than six seconds),

7. adaptable to automatic recording,

8. of sufficient range for the temperatures encountered during testing,

9. capable of being calibrated to determine the quantity of water involved, as well as the moisture distribution and rate of drying,

10. capable of being corrected for effect of concrete temperature on the gage resistance through established correction curves,

11. capable of being corrected for the effect of salt ions on gage resistance, and

12. previously evaluated and used successfully by Czaban(10, 11) in an investigation of curing compounds for concrete pavements.

Bouyoucos Moisture Gage

The commercially available Bouyoucos moisture gage(12, 13) consisted of a pair of stainless steel screen electrodes firmly positioned and cast in plaster of paris. The stainless steel electrodes ran parallel to the long surface and were spaced 1/4 inch apart. These electrodes were 15/16 inch long and 4/16 inch wide.(13) Each gage measured 5/8 inch thick by 1-1/4 inches wide by 1-5/8 inches long. (See Figure 1.) The plaster of paris provided a uniform environment around the electrodes and also eliminated the fluctuation in contact resistance between the electrodes and the cement paste.

When the gage was embedded in fresh concrete it absorbed and relinquished moisture very freely, and as cement hydration proceeded any variation in moisture content produced a drastic change in the internal resistance of the gage. This change in electrical resistance was then related to moisture changes from prior calibrations of the gages.

Moisture gage resistance measurements were made with an AC ohmmeter. The ohmmeter was designed and constructed by William H. Dancy, Head of the Instrumentation Development Group of the Research Laboratories for the Engineering Sciences at the University of Virginia. The meter and its operation are discussed in reference 8. Two gages may be connected to the meter at any one time, and each gage is read manually. To accommodate the approximately 5,000 resistance readings necessary for the study of

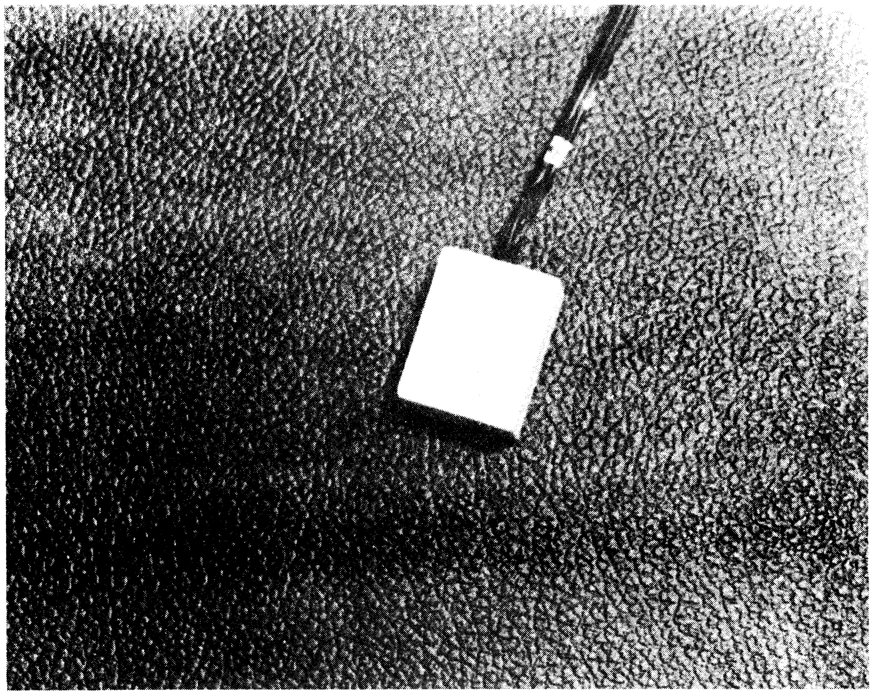

Figure 1. Bouyoucos gypsum type moisture gage.(13)

autogenous curing an AC ohmmeter identical to the manually read ohmmeter was constructed. The second ohmmeter was used in conjunction with a Minneapolis Honeywell Type 153 Universal ELECTRONIK Multipoint Recorder, having 24 points with single color printing to allow for automatic data recording. The ohmmeter and Honeywell recorder are shown in Figure 2 in the process of recording resistance data from four cylinders (eight moisture gages) undergoing autogenous curing. Resistances from 12 gages may be recorded at any one time. The Honeywell recorder was wired with appropriate microswitches so that both low-scale and high-scale resistances were recorded. Temperatures were recorded by a second 24-point Minneapolis Honeywell Type 153 Universal ELECTRONIK Multipoint Recorder and 20-gauge copper-constantan thermocouples.

Since the moisture gage resistance is affected by changes in temperature, a correction factor has to be applied to change all resistances to a common base temperature selected to be 60^{o} F.

In selecting a temperature correction chart, careful consideration was given to the parameters involved and to the development of the Bouyoucos(13) and the Coleman temperature correction charts.(14) It was decided that the most applicable chart is that shown in Figure 3, which is Coleman's.

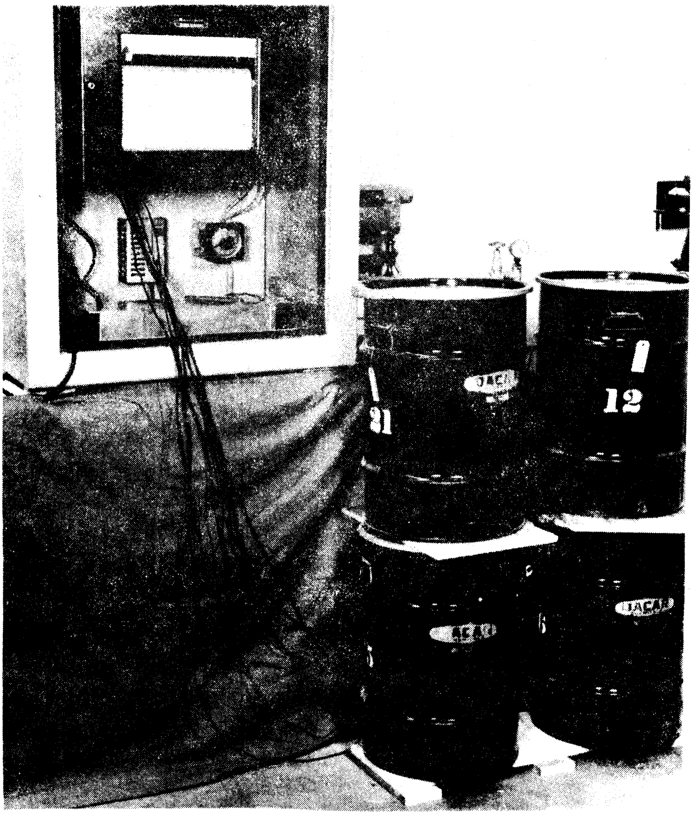

Figure 2. AC ohmmeter and Honeywell recorder with moisture gage connections from four autogenous curing containers.

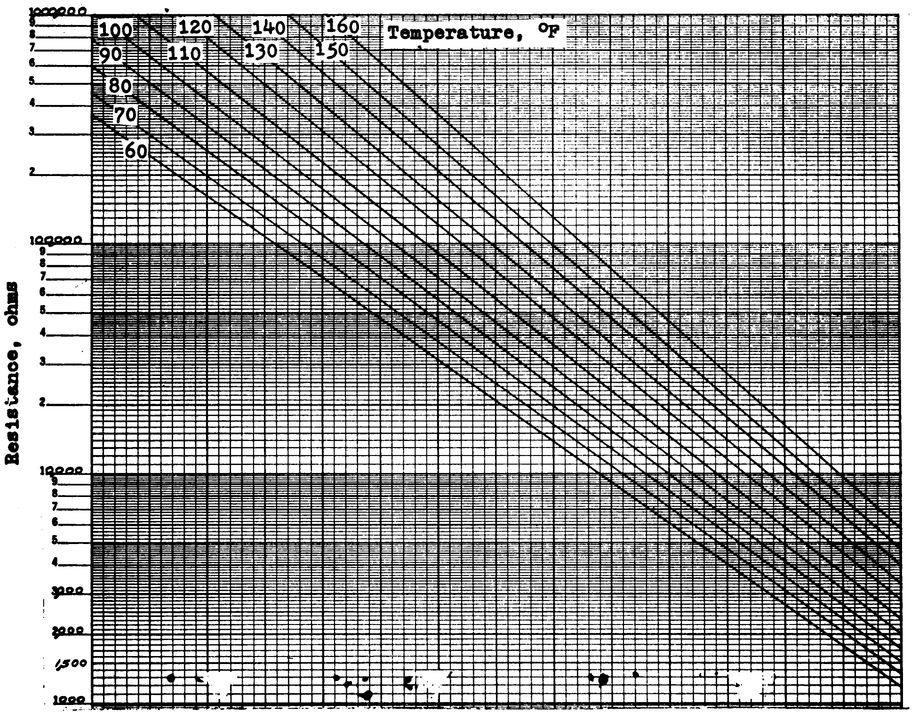

Figure 3. Moisture gage resistance temperature correction chart after Coleman.

To correct a resistance of 1,500 ohms measured at 100°F, enter Figure 3 at 1,500 ohms and move horizontally to the 60°F line. Then move vertically upward to the 100°F line and read a resistance of 2,600 ohms. Therefore, 1,500 ohms at 100°F has been corrected to 2,600 ohms at 60° F.

CALIBRATION OF MOISTURE GAGES

After a thorough search of the literature, it was concluded that no one except Czaban(10) had ever used the Bouyoucos moisture gage in concrete and that no calibration procedure for moisture determination in concrete had been developed. Consequently, a complete investigation of the gage properties and behavior was undertaken on 130 gages or on a representative sample thereof, depending upon the characteristics under study.

Calibration at Known Relative Humidities Using Salt Solutions

After the literature review, it was decided to relate gage resistance to relative humidity by using salt solutions to develop atmospheres of known relative humidities(13, 15-19) The gages were also to be calibrated for percent moisture content by gage dry weight with the use of distilled water by going through a number of wetting and subsequent drying cycles. These procedures would indicate the behavioral characteristics of the gages and would provide a relationship between evaporable water content and relative humidity(20)(ratio of actual vapor pressure to vapor pressure at saturation).

It was necessary to start from the same reference point for each gage tested, i.e., the unit dry weight of the gage. So that the detrimental effects of temperature drying at 230°F could be avoided, the gages were first weighed, as received, at atmospheric pressure and were then placed in a vacuum chamber at 28 inches of mercury for a period of 24 hours. At the end of 24 hours the gages were again weighed, and this weight was taken as the unit dry weight.

The method used for achieving the desired relative humidities was patterned after the work done by Wexler and Hasegawa(18). The six salts shown in Table I, were chosen as representing relative humidities from 12 to 97 percent.(18)

Humidity chambers were set up in accordance with work done by Martin(21) and Wylie (19). The airtight humidity chambers used were wide-mouth Mason fruit jars as shown in Figure 4.

Initially, two gages were placed in separate relative humidity chambers, each at 97.2 percent relative humidity (R.H.), one gage at 75.5 percent relative humidity, two gages at 54.9 percent relative humidity, and one gage at 12.4 percent relative humidity.

TABLE I

RELATIVE HUMIDITIES FOR VARIOUS SATURATED SALT SOLUTIONS AT 68°F (18)

Salts	Relative humidity at 68°F, percent
Lithium chloride ($LiCl \cdot H_2O$)	12.4
Magnesium chloride ($MgCl_2 \cdot 6\ H_2O$)	33.6
Magnesium nitrate ($Mg(NO_3)_2 \cdot 6\ H_2O$)	54.9
Sodium chloride (NaCl)	75.5
Potassium nitrate (KNO_3)	93.2
Potassium sulfate (K_2SO_4)	97.2

Figure 4. Calibration of moisture gages in distilled water.

The moisture gages remained suspended in the chambers above the saturated salt solutions for 60 days. At the end of 60 days no moisture registered on the ohmmeter for the gages suspended in relative humidities of 12.4, 54.9, and 75.5 percent. The two gages at 97.2 percent relative humidity did register a decrease in resistance after 45 minutes in the chamber and reached an equilibrium condition at approximately 30 days. These two gages remained in the chamber for 60 days, with no appreciable change in resistance after the first 30 days. The results were that for the same condition (97.2 percent relative humidity), one gage had a resistance of 6,800 ohms and the other gage had a resistance of 230,000 ohms.

Two basic facts resulted from these tests. First, it would be impossible to calibrate the gages at relative humidities below about 90 percent and, second, it could not be assumed that the gages had the same physical properties (i.e., that under the same moisture conditions all gages would have the same resistance). Therefore, this method of calibration was abandoned.

Statistical Calibration at 100 Percent Saturation in Distilled Water

It was then decided to subject each gage to 100 percent saturation in distilled water in order to establish its equilibrium resistance. Accordingly, the gages were grouped statistically so that the maximum coefficient of variation

$$\left(\frac{\text{standard deviation}}{\text{mean}} \quad 100 \; = \; \text{percent}\right)$$

would be less than 3.0 percent for each group of gages.

The gages were submerged in distilled water to the same depth so that the pressure head would be constant (Figure 4). Resistance readings were taken for nine days. The gages reached the equilibrium resistance in approximately 36 hours, with minor fluctuations in resistance for the next 20 hours. Figure 5 shows the distribution of equilibrium resistances of the moisture gages in distilled water. Table II indicates the five groupings selected for the gages, that had coefficients of variation of less than 2.0 percent, instead of the anticipated 3.0 percent.

Consideration was also given to gage reproducibility, the hysteresis effects of subsequent wetting/drying cycles, and the influence of mix variables on the moisture versus resistance relationship. Gages were picked at random and were retested three times at 100 percent saturation in distilled water to check equilibrium resistances. After each saturation test the gages were allowed to dry in still air at 70^{o}F and 45 percent relative humidity until the gage resistance became 1,000,000 ohms, which was the maximum capability of the AC ohmmeter. For the same drying conditions — i.e., the same moisture content — the resistances varied by not more than ± 2 percent for all gages tested. Since the gages tested were capable of reproducing the same results under identical situations and since these results were in agreement with the work of Czaban,(10) it was postulated that the remaining gages would have the same quality of reproducibility.

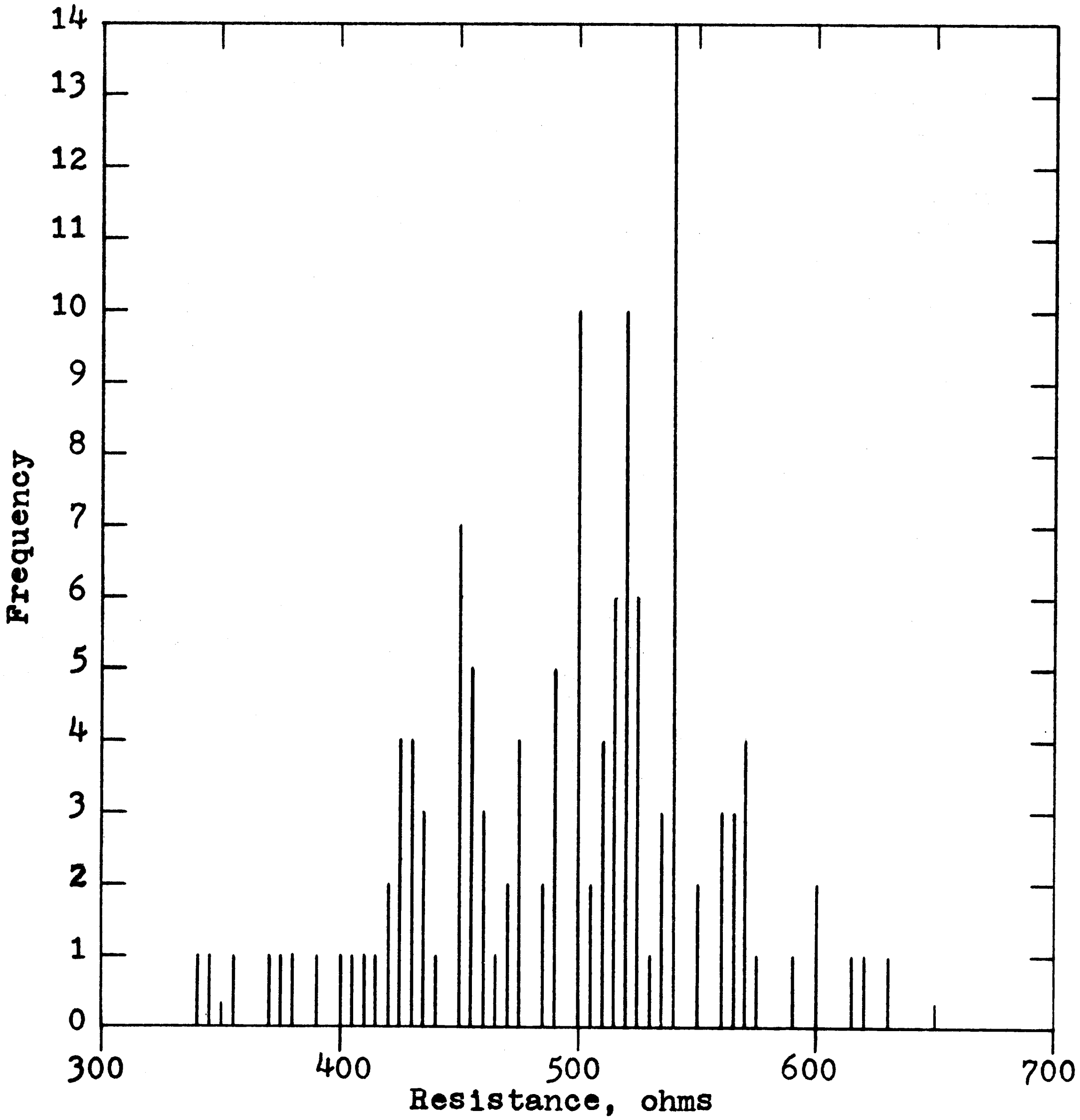

Figure 5. Histogram of resistances resulting from calibration of moisture gages in distilled water.

TABLE II

STATISTICAL EVALUATION AND GROUPING OF THE MOISTURE GAGES

Gage group number	Number of gages in group	Group range, ohms	Mean, ohms	Coefficient of variation, percent
1	12	560 - 590	568	1.41
2	36	520 - 550	532	1.80
3	29	485 - 515	502	1.90
4	22	450 - 475	460	2.05
5	16	410 - 440	427	1.80
All gages	129	340 - 630	495	11.71

During the study of autogenous curing, for which the gages were to be used, all moisture data would involve continuous decreasing humidity within the concrete; therefore, hysteresis effects would not be a factor.

Czaban found that the coarse aggregate gradation had a pronounced effect on the relationship between gage resistance and percent moisture content by dry weight for the concrete cylinders. In the autogenous experiment the coarse aggregate and fine aggregate gradations would remain constant. The effects of other factors investigated by Czaban, such as water-cement ratio, type and brand of cement, entrained-air content, on resistance-moisture relationships would not be applicable to the autogenous experiment because his tests were conducted on hardened concrete after the pore structure had been formed. In autogenous curing, the pore structure is in the process of forming during the majority of the study period.

Calibration in Concrete Cylinders

As a result of the grouping of the gages, 14 gages did not fall within any group and were discarded. A total of 111 gages was used for the moisture tests. Since each group had such a low coefficient of variation and since the gages that were calibrated in concrete could not be used during actual testing, it was decided to calibrate the gage having an equilibrium resistance closest to the mean of that particular group. If the five gages thus calibrated showed significantly different results, then additional gages would be calibrated.

The concrete mixture used in the calibration was the "average" for the anticipated study of autogenous curing. Its main characteristics were as follows:

Cement factor = 550 lb/cu yd

Water-cement ratio = 0.5

Cement type = II

Air content = 5.5 ± 0.5 percent

Initial mix temperature = $73^{o}F \pm 3^{o}F$

Admixtures = None

From a single batch of concrete, seven cylinders were made: five containing moisture gages for calibration and two with thermocouples.

The procedure which follows was used to calibrate the moisture gages during the initial concrete curing period and two subsequent cycles of resaturation and drying.

First Drying Period

1. The moisture gages were saturated in distilled water for 48 hours prior to embedding them in the fresh concrete. Resistance measurements were taken to check equilibrium.

2. The seven 6" x 12" cylinders were cast using procedures meeting the requirements of ASTM procedure C 192.(22) Special care was taken not to damage the moisture gages during casting.

3. The moisture gages were placed in the centers of the cylinders at mid-height of each cylinder, with the gage sides having the largest surface area facing the top and bottom of the cylinders. The lead wires were brought out of the top of the cylinders, as shown in Figure 6.

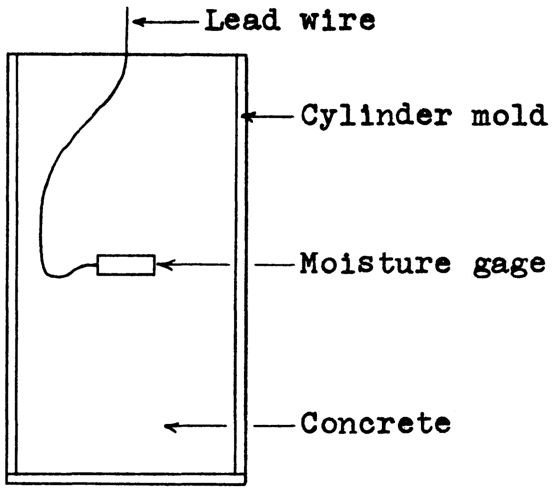

Figure 6. Position of moisture gage in concrete cylinder.

4. All the cylinders were allowed to cure for 7 hours before removal of the molds (final set as measured by ASTM — C403 occurred at 6 hours and 20 minutes). When the molds were removed, the base plate and cylinder were left intact and the cylinders were allowed to cure in still air at $73^{o}F \pm 3^{o}F$ and 50 percent R.H. ± 5 percent R.H. for the remaining curing period.

5. After removal of the molds, each cylinder/base plate unit was placed on a plywood tray to facilitate ease in handling the cylinders. Each cylinder was weighed to the nearest tenth of a pound, and at the same time a resistance and a temperature reading were recorded. The readings were

to be taken either until the gage resistance or the cylinder weight became constant or until the gage resistance surpassed the capability of the ohmmeter. These weight, resistance, and temperature readings were taken every hour for the first 24 hours, then every 2 hours for the next 24 hours. By the end of 48 hours the rate of drying had slowed considerably, and readings were taken twice a day for the next 4 days, after which time one reading a day was taken for the next 8 days. After 14 days the readings were stopped and the seven cylinder/base plate units were placed in an airtight, dust free unit.

First Resaturation and Second Drying Cycle

6. Later, the seven units were resaturated for 8 days until resistance equilibrium was established. Then the second drying period was started under the same conditions as described in step 4. The total drying period lasted 14 days, as did the first drying cycle. Weight, resistance, and temperature readings were taken every half hour for the first 48 hours, then every hour for the next 2 days, after which time two readings a day were taken for the next 10 days. The reason for taking the readings more frequently are discussed in the following section entitled "Calibration Curves".

Second Resaturation and Third Drying Cycle

7. Again the seven units were saturated for 8 days, at the end of which time the third drying period was initiated. This time drying was accomplished in still air at a temperature of $80^{O}F \pm 3^{O}F$ and a relative humidity from 25 to 30 percent. This increase in drying conditions was applied primarily to increase the rate of drying to see if the increased rate would cause a shift in the calibration curve. The frequency of measurement was the same as for the second drying period, except that the test took 10 days instead of 14 days.

8. The final step in this procedure was to place the five moisture gage cylinders with base plates into the oven at $221^{O}F$ to drive off all the evaporable water. The cylinders were in the oven for 48 hours, after which time they were weighed to determine their dry weight. The difference between the cylinder weights at the end of the third drying period and at the end of oven drying was exactly 1 pound of water for each cylinder.

Calibration Curves

There will be no attempt to differentiate between or analyze the various states of water in concrete as discussed in Part IV.(4) It will be assumed that the evaporable moisture content of a concrete cylinder at any instance can be determined, based on the oven-dry weight of the cylinder. The moisture content of the calibration cylinders was determined by using the following equation:

$$M = \frac{W - W_d}{W_d} (100) = \text{percent moisture}$$

where:

W = weight of cylinder at time of resistance reading

W_d = weight of oven-dry cylinder

Since the procedure for evaluating the raw data received from the drying periods is the same for all five gage groups, only the procedure for group 4 data will be shown.

The weight-resistance data for group 4 were corrected for temperature; i.e., resistances were corrected to 60°F by using the curves of Figure 3. The resulting curves for the three drying periods are shown in Figure 7. These curves are the actual plot of the raw data without any curve fitting. The stepped effect of the curve results from the fact that all weight readings are to the nearest tenth of a pound. For example, in the first drying period (heavy solid line) the weight did not change abruptly from 29.1 pounds to 29.0 pounds as shown; the change was a gradual one. During the second and third drying periods the weight readings were taken more frequently and were estimated to the nearest 0.05 pound, resulting in the plotting of resistance values for 29.0, 29.05, and 29.1 pounds. The vertical portions of each curve represent numerous resistance readings recorded for the same weight.

When the gages are calibrated in concrete during the initial drying, or hardening, of the concrete, the following time intervals for weight and resistance measurements should provide adequate data for establishing reliable percent moisture-resistance calibration curves:

Interval between readings	Time period
30 minutes	First day
1 hour	Second day
2 hours	Third day
4 hours	Thereafter

There are three basic conclusions which can be drawn from this set of curves. First, the slope or drying rate for the first drying period is different from that of the second and third drying periods. Second, even though the drying conditions were just slightly more severe in the third drying period than in the second period, there was a shift in the curve. That is, by increasing the rate of drying for the same resistance, the weight was decreased. Third, the weight losses and resistances for drying periods two and three did not include the range of values needed for the experiment, and it would not have been wise to have extended the curves to cover the needed range. The total quantity of water absorbed by the concrete cylinder during the second resaturation was 1.7 pounds, or 6.2 percent by dry weight. The drying conditions for the third drying

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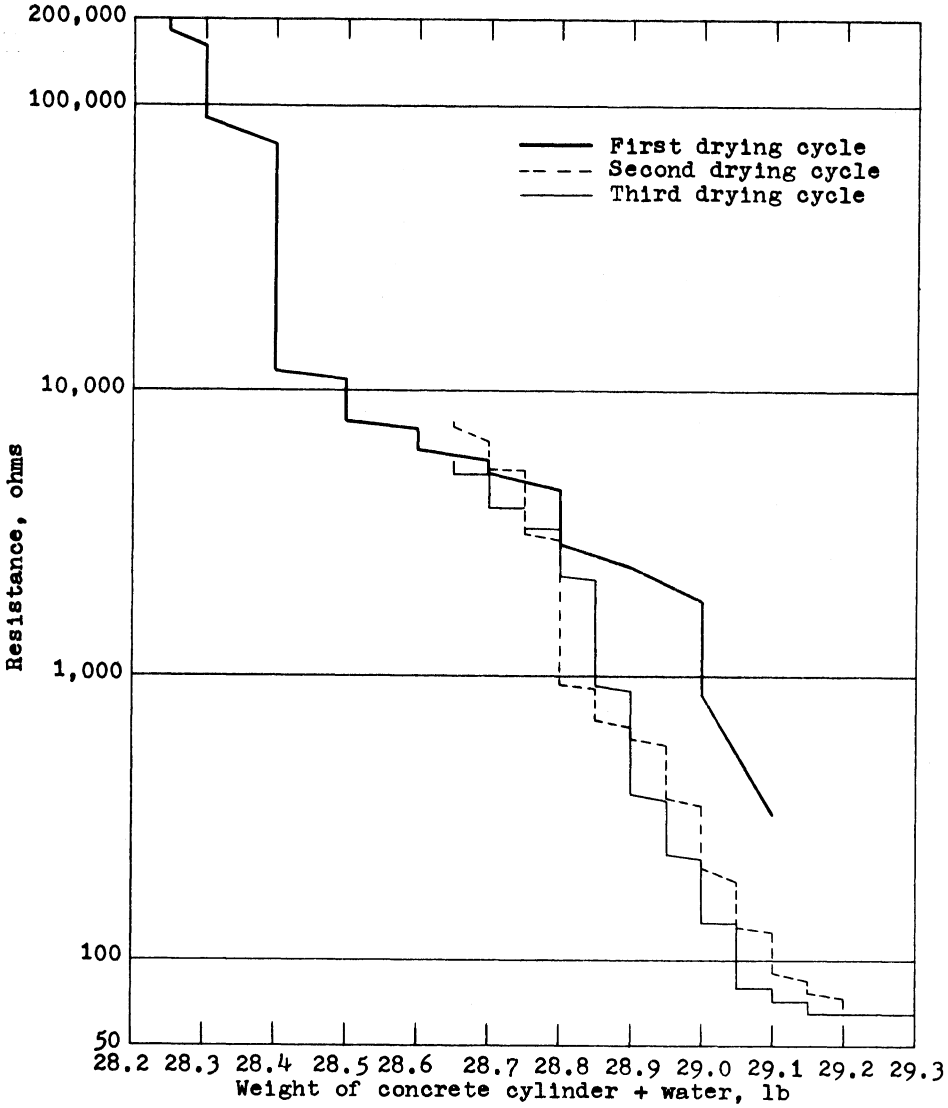

Figure 7. Weight-resistance curves for the three drying periods of Group 4 gages calibrated in concrete.

cycle, described in step 7, were not severe enough to drive out all the absorbed water; only 0.7 of a pound. The remaining pound was driven off in the oven at 221°F. At the termination of the third drying cycle the gage resistance reading was approximately 8,000 ohms, as shown in Figure 7, which is well within the capability of the moisture measuring method. Therefore it was the ambient drying conditions and not the moisture method which caused the equilibrium condition to be reached at such a low resistance after only 41 percent of the total evaporable water had been lost.

Subsequently it was decided that, for the reasons stated above plus the fact that the gages used during autogenous curing would evaluate the moisture content without drying from external influences, the curves from the first drying period of the calibration cylinders would be used.

The procedure for constructing the final calibration curve for the gages in group 4 is shown in Figure 8. In looking at the solid line between 645 ohms and 1,230 ohms, it is evident that if the readings had been taken more frequently, the 645 ohm point would have been higher and the 1,230 ohm point would have been lower. In other words, if the 1,230 ohm reading had been taken 1/2 hour earlier, the resistance might have been 1,000 ohms for the same weight. Therefore, the 645 ohm point and the 1,230 ohm point were raised and lowered, respectively, by taking the average of the two values, or 940 ohms. The dotted line now replaces the solid line. The same procedure is used between 2,220 and 2,925 ohms, resulting in the dotted line at 2,575 ohms.

Now the vertical distance between 645 and 940 ohms at 5.2 percent moisture represents the range of resistances which has a corresponding value of 5.2 percent moisture. Likewise, the vertical distance between 940 and 2,575 ohms represents the range of resistances having a corresponding value of 4.9 percent moisture. Therefore, a curve must be drawn through the average resistance of each subsequent vertical portion of the stepped curve. These points are represented by the circled points in Figure 8.

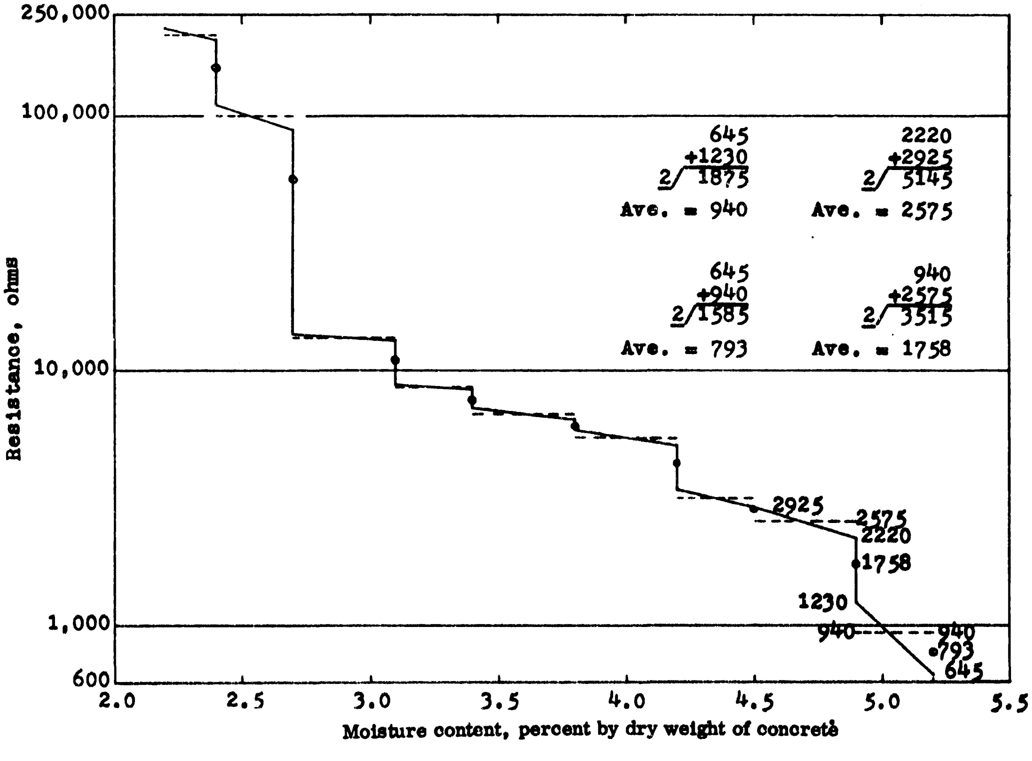

Figure 8. Curve-fitting procedure of final calibration curve for gages in group 4.

A statistical analysis was conducted on the data from the five calibrated gages. For the statistical analysis, resistances and values of moisture content were converted to logarithms to give a straight-line relationship. The results of the linear regression analysis on the converted data are presented in Table III.

TABLE III

RESULTS OF LINEAR REGRESSION ANALYSIS ON CALIBRATION CURVES FOR MOISTURE GAGES

Gage Group	Data Points	Equation*	Correlation Coefficient	Standard Error
1	9	Y = 2.514 - 0.135 X	-0.967	0.0795
2	9	Y = 2.442 - 0.121 X	-0.983	0.0510
3	8	Y = 2.380 - 0.118 X	-0.940	0.0892
4	10	Y = 2.567 - 0.140 X	-0.973	0.0739
5	8	Y = 2.424 - 0.116 X	-0.975	0.0579
Total	44	Y = 2.476 - 0.127 X	-0.958	0.0763

*Y = Log (percent water) and X = Log (ohms).

In Figure 9, which shows the curve of best fit and the corresponding 95 percent confidence interval for average values, the values have been reconverted from logarithms for easier use. The variability increases as percent moisture content decreases because of the variability in resistance readings on the high scale of the ohmmeter. The high-scale resistance curve becomes very steep at resistances between 500,000 and 1,000,000 ohms. This means that a small variation in dial reading would result in a large variation in resistance.

Even though one calibration curve could have been used, the moisture curves resulting from the autogenous curing study were based on the individual calibration curve developed for each of the five gage groups.

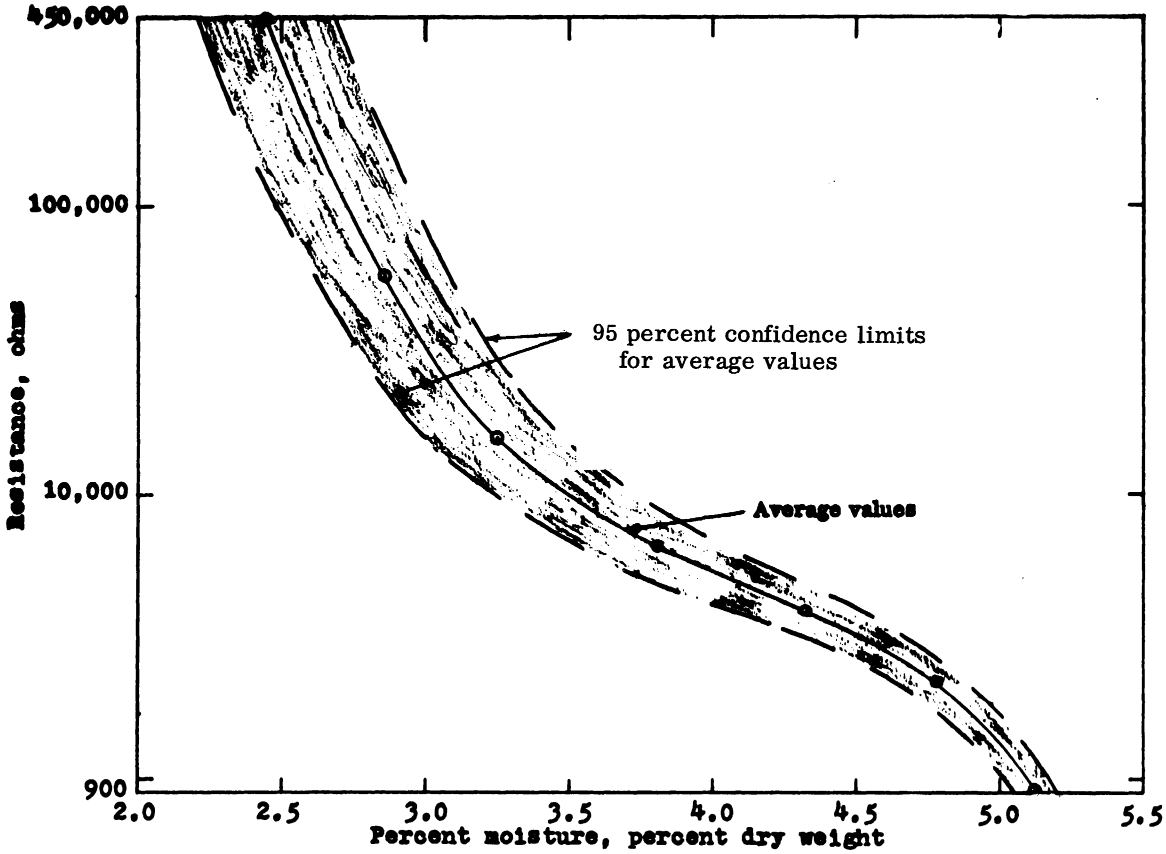

Figure 9. Curve of best fit for all five calibration curves.

Correction Factors for Calibration Curves and Moisture Curves

The final calibration curves were not plotted until after all the moisture data for the entire autogenous curing project had been gathered and plotted. The reasons they were not plotted will become evident in the succeeding paragraphs.

Salt Ions

The presence of salt ions in fresh concrete will have an effect on the resistance of the moisture gages; i.e., an increase in salt content decreases the resistance. In the autogenous experiment, resistance readings were taken at half hour intervals on 104 gages (52 cylinders) during the curing period from 1 hour to 47 hours. A typical plot of resistance versus time is shown in Figure 10. Since the gage was saturated prior to placement in the fresh concrete, the gage resistance should have steadily increased from the equilibrium value as the concrete cured. Instead, all 104 curves showed a drop in resistance of between 195 ohms and 250 ohms with the maximum drop occurring at approximately 6 hours from time zero; this drop was attributed to the content of dissolved salts.

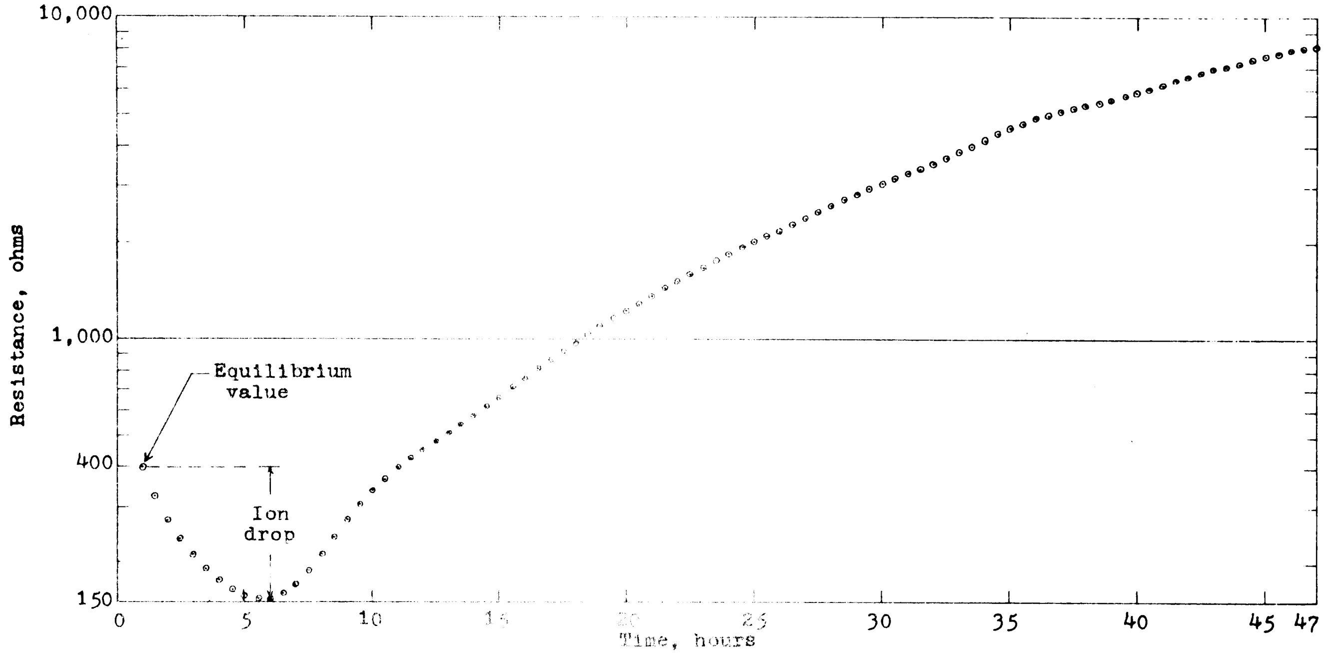

Figure 10. Typical curve of resistance versus time for a moisture gage in a concrete cylinder.

In order to substantiate this phenomenon, tests were conducted on various water-cement mixtures to determine the effect of cement type, cement content, water-cement ratio, and admixtures on the reduction in gage resistance.

The AC ohmmeter with a pair of metal probes was used to measure resistance. A glass beaker was filled with water whose resistance was 55,000 ohms for the probes used. Then, known quantities of cement were added to the water and corresponding resistance readings were recorded. The resulting curve of resistance versus cement-water ratio is shown in Figure 11. At a cement-water ratio of 0.5 (W/C = 2.0), the resistance dropped from 55,000 ohms to 215 ohms and remained steady as the cement-water ratio was increased. Since water-cement ratios for concrete are well below 1.0, the effect of salts on resistance should remain nearly constant; the resistance drops for 104 tests of autogenous curing proved this to be true. This same test was conducted for the different cements used in the autogenous experiment and showed that the three cements did not affect the resistance loss. Admixtures (retarder and accelerator) caused a resistance drop when added to water; but when added to a mixture with a cement-water ratio greater than 0.5, they did not cause a further decrease in resistance. Therefore, the cement was judged to be the controlling factor in reducing the moisture gage resistance.

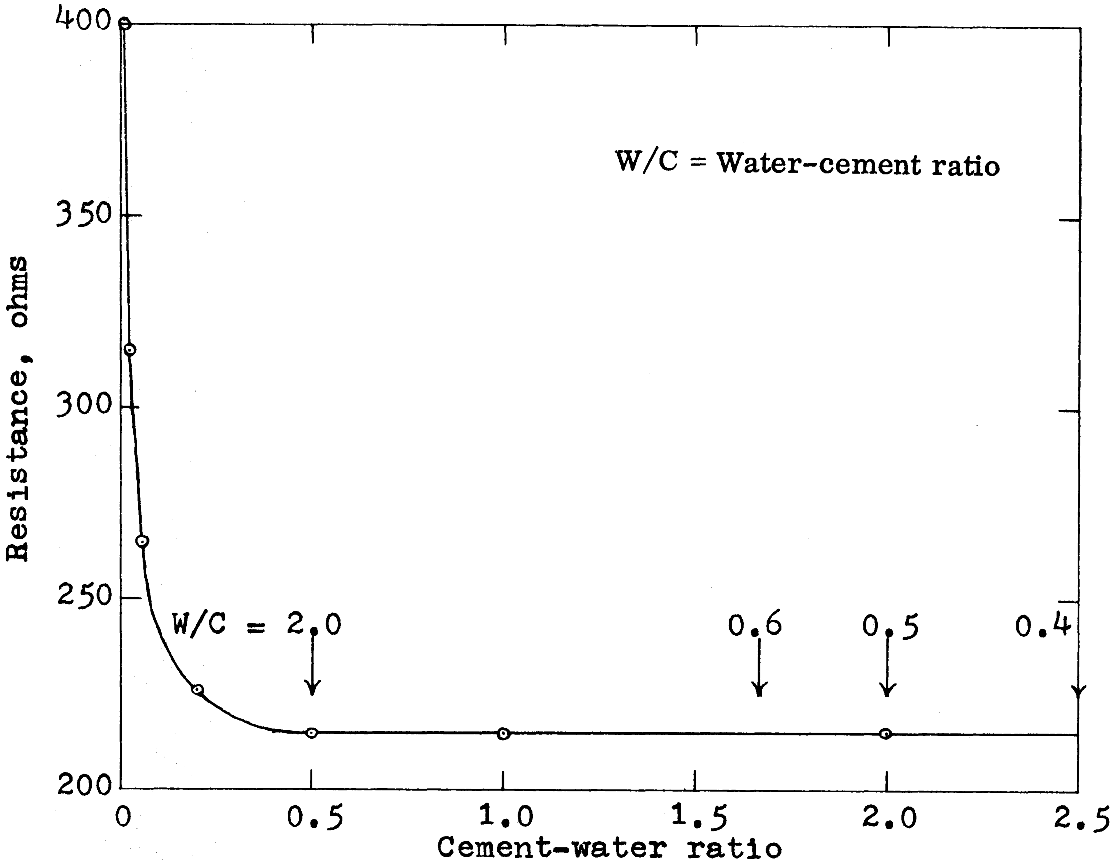

Figure 11. Effect of cement content on resistance of a cement-water solution.

To compare the shape of the resistance-time curve, the resistance drop, and the length of time to maximum resistance drop with the actual resistance-time curves of the 104 gages, one additional test was performed. A moisture gage was placed in a glass beaker filled with a water-cement mixture having a water-cement ratio of 0.5; the gage was saturated with water before being immersed in the mixture. The resulting curve, shown in Figure 12 resembles in all respects the curves obtained for moisture gages in concrete.

Based on the results of these tests, an average value of total resistance drop was determined from the 104 resistance-time curves for each of the moisture gage groups. The values so obtained are shown in Table IV. These average resistance drop values were added to the resistances of the resistance-time curves of the moisture gage calibration cylinders only. The actual resistance drop obtained for each of the 104 experimental curves was applied to its corresponding curve. The reason for determining an average value of resistance drop and applying it to the calibration data was that weight and resistance measurements were not taken on the calibration cylinders until after seven hours of curing, and the maximum resistance drop occurred at six hours. Therefore, the resistance drop effect was not recorded for the calibration cylinders. The methods used for determining percent-moisture/time curves will be discussed subsequently.

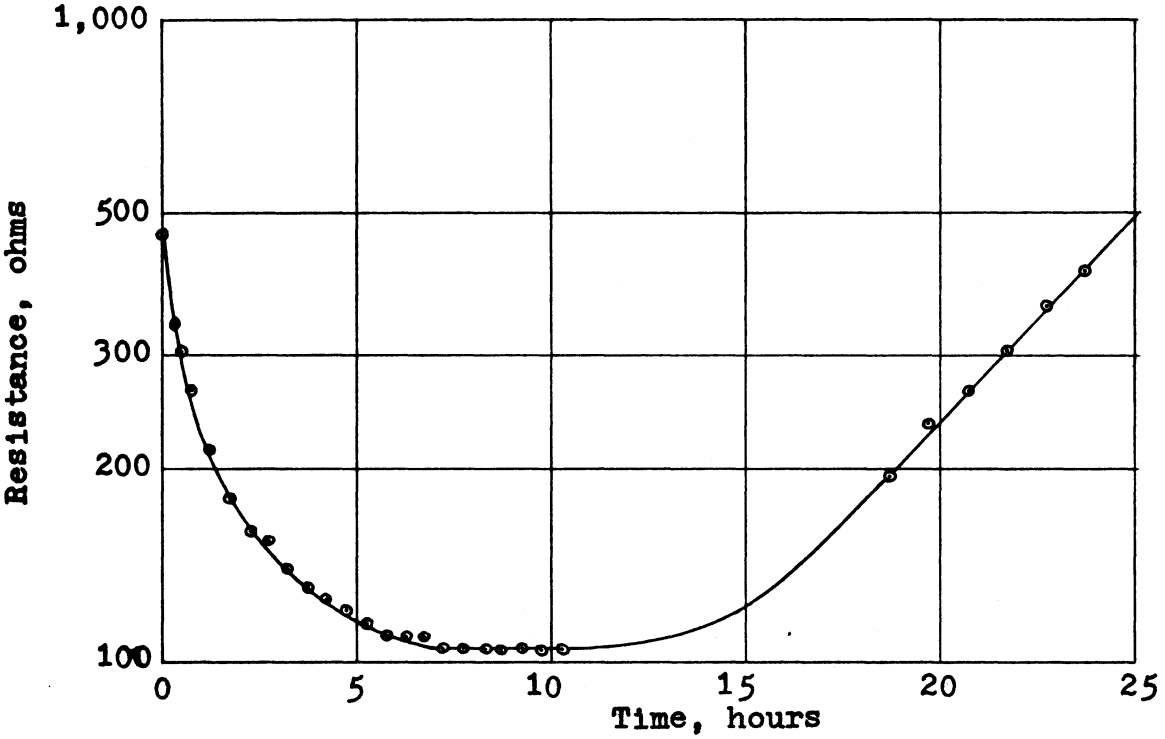

Figure 12. Resistance-time curve for moisture gage in cement-water matrix.

TABLE IV

RESISTANCE DROP VALUES, DUE TO SALT IONS, TO BE APPLIED TO CALIBRATION CURVE FOR EACH MOISTURE GAGE GROUP

Gage Group	Resistance Drop — average values, ohms
1	235
2	250
3	235
4	235
5	195

Temperature

The resistances of the calibration cylinders were corrected for temperature to a base temperature of 60°F by using the curves of Figure 3.

For the calibration curves the average resistance drops (Table IV) were added to the respective gage group resistances; the resistances were then corrected for temperature and the resulting final calibration curves were developed.

The mechanics involved in correcting an original resistance-time curve for the influence of salt ions and temperature are shown in Figure 13. Curve A is a plot of typical original data as received from the Honeywell recorder for one moisture gage (note the smoothness of the curve). The moisture gage for curve A at one hour was in a condition of 100 percent saturation in distilled water. The total ion drop for this gage was 250 ohms, which occurred between one and six hours. If curve A were corrected for the ion drop alone, then the results would be curve B, showing an increase in resistance of 167 percent at six hours and only a 3 percent increase at 47 hours. For the majority of the gages the final resistance at 47 hours ranged from 20,000 to 50,000 ohms, resulting in an ion drop effect from only one percent to 0.5 percent, respectively.

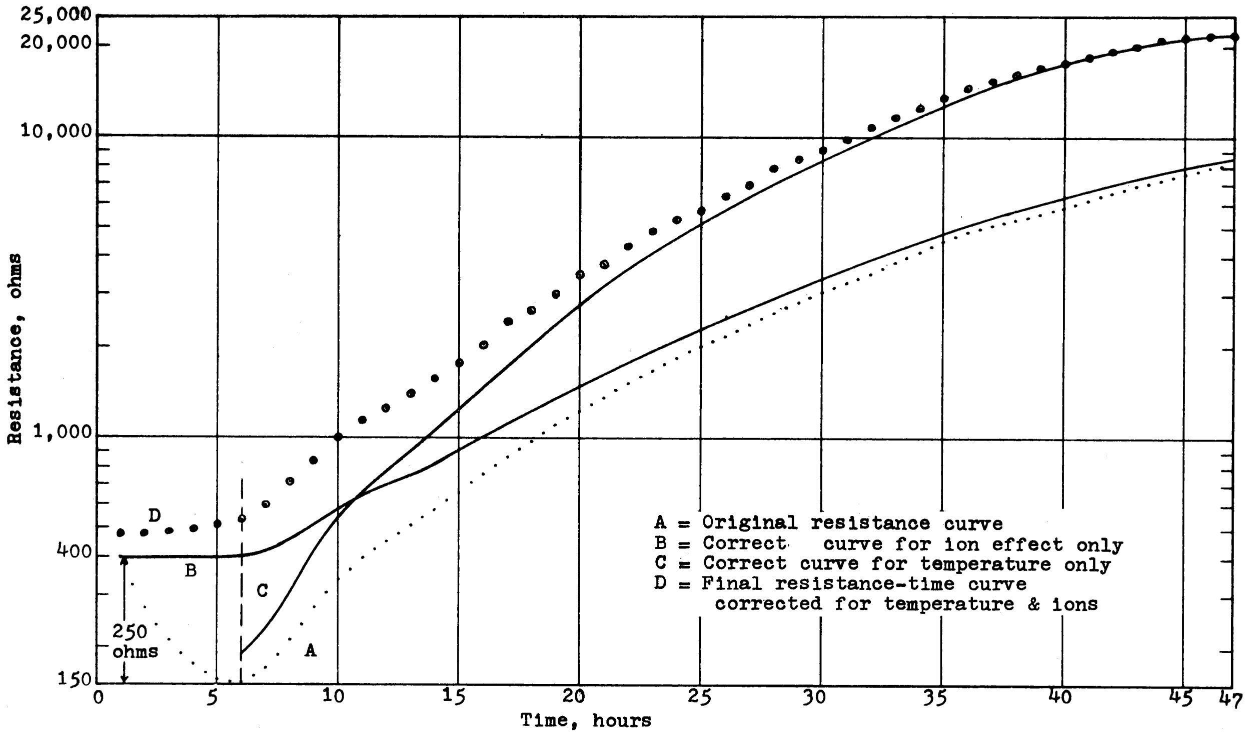

Figure 13. Mechanics involved in applying correction factors to original resistance-time moisture curves.

Correcting curve A resistances for temperature only results in curve C. All resistances are corrected to a base temperature of 60°F. Curve C shows a resistance increase of 27 percent at 6 hours and 163 percent at 47 hours. Consequently, the temperature effect is greater (from a percentage standpoint) at 47 hours, and the ion drop effect is greater at 6 hours.

Curve D results when both temperature and ion drop corrections are made. First, temperature corrections are applied to the resistance value of 400 ohms (1 hour) until the point where curve A reaches the low value of 150 ohms (6 hours); then, the ion drop resistance (250 ohms) is added to the resistance values of curve A between $6\frac{1}{2}$ hours and 47 hours, and the new values are subsequently corrected to a base temperature of 60°F. Therefore, curve D is the final resistance-time curve used in conjunction with the calibration curve for resistance and percent moisture (Figure 9) to determine the percent moisture-time curve.

MOISTURE RESULTS GENERATED BY THE DATA

A typical plot of percent moisture against time is shown in Figure 14. The following items designated in Figure 14 may be derived from all the moisture curves:

A = Total water lost, percent dry weight

B = Percent of total water lost by time of maximum autogenous temperature

C = Absolute maximum rate of water loss, percent per hour

D = Average time of absolute maximum rate of water loss, hours

E = Average maximum rate of water loss, percent per hour

F = Average time of average maximum rate of water loss, hours.

B is the water lost at the time of maximum temperature expressed as a percentage of A, the total water lost at 47 hours. E, the average maximum rate of water loss, occurs during the period of maximum rate of water loss, and was determined by taking the average rate between points ① and ② in Figure 14. These points were determined by constructing tangents to the curves and bisecting the resulting interior angles. The datum point of the moisture curve nearest to the bisector was taken as the end point. There is an error involved in estimating the tangent locations, but the average rate of water loss more nearly defines the actual drying process than does the absolute maximum rate of water loss. This is illustrated by data drawn from the autogenous curing experiment(4) and is shown in Figures 15 and 16. These figures show the relationships between C and E, and D and F, respectively. The absolute rate is approximately twice the average rate, yet the average times of both rates of water loss are approximately the same. Consequently, the average maximum rate of water loss gives a more realistic picture of what is actually taking place.

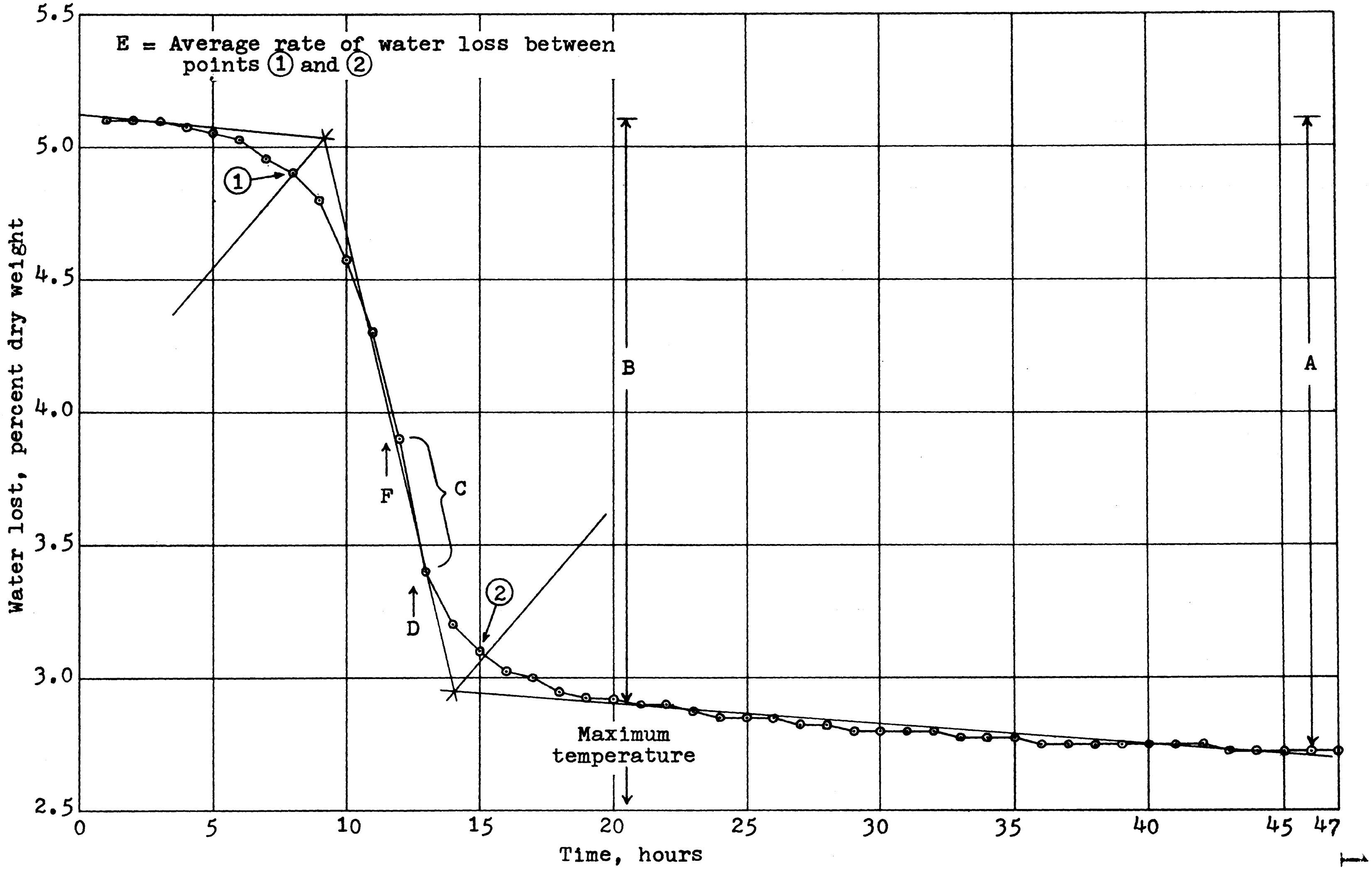

Figure 14. Typical curve of percent moisture plotted against time for autogenously cured concrete cylinder.

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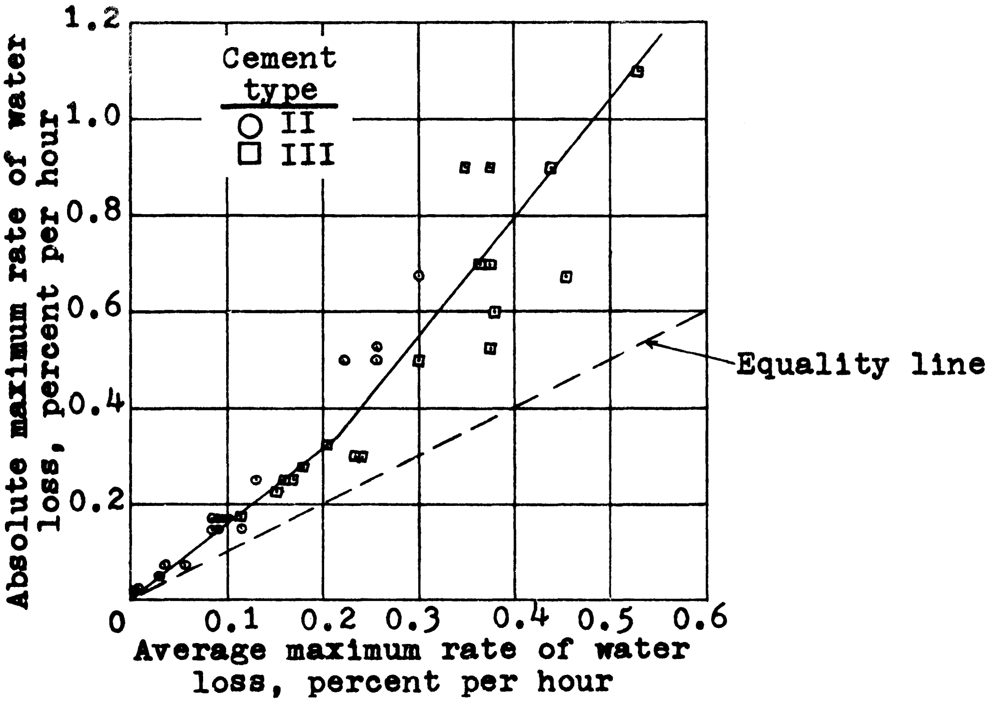

Figure 15. Relationship between absolute maximum rate of water loss and average maximum rate of water loss for several concretes with an initial mix temperature of 70°F.

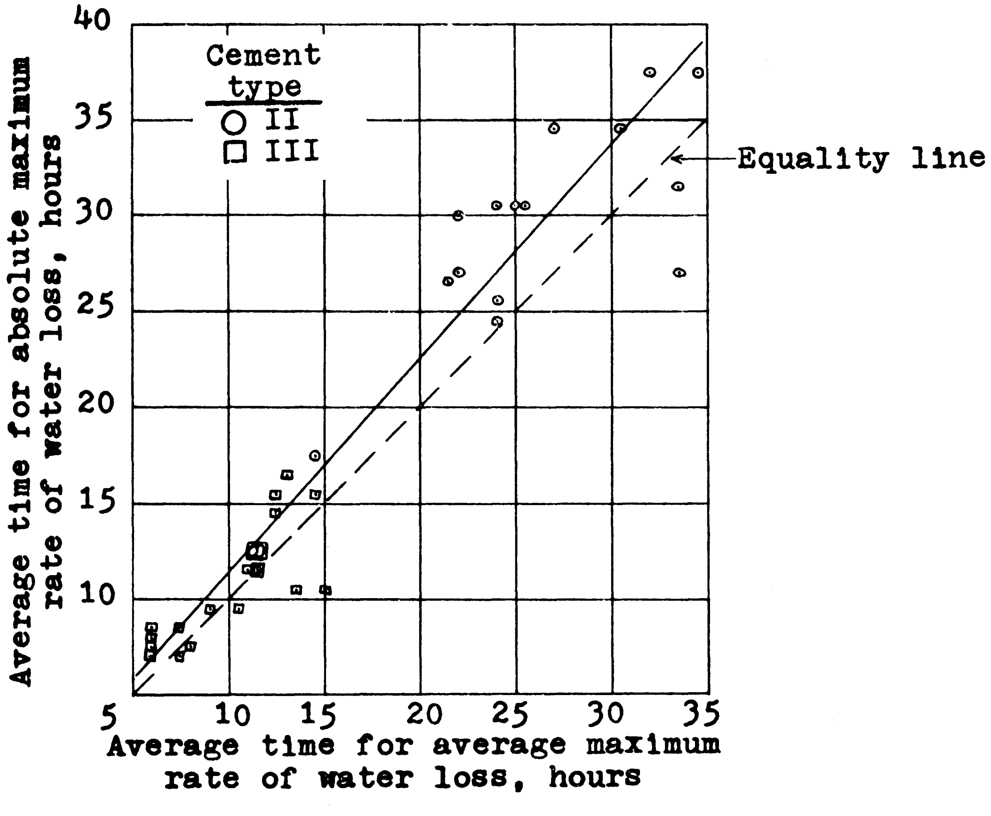

Figure 16. Relationship between average time of absolute maximum rate of water loss and average time of average maximum rate of water loss for several concretes with an initial mix temperature of 70°F.

CONCLUSIONS

1. Of the various methods described in previous literature, the Bouyoucos moisture gage was judged to be a satisfactory method of measuring moisture in the concrete cylinders subjected to autogenous curing.
2. The influence of salt ions and temperature on the moisture gage resistance can be measured and the necessary correction factors applied to the results.

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