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OF A
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FOR
FROST RESISTANCE OF CONCRETE

FINAL REPORT
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IOWA DOT PROJECT HR-272
ERI PROJECT 1775

Sponsored by the Highway Division of the
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ENGINEERING RESEARCH INSTITUTE

iowa state university

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INTRODUCTION

This report describes the research completed under the research contract entitled "Development of a Conductometric Test for Frost Resistance of Concrete" undertaken for the Iowa Highway Research Board. The objective of the project was to develop a test method which can be reasonably and rapidly performed in the laboratory and in the field to predict, with a high degree of certainty, the behavior of concrete subjected to the action of alternate freezing and thawing. The significance of the results obtained, and recommendations for use and the continued development of conductometric testing are presented in this final report.

In this project the conductometric evaluation of concrete durability was explored with three different test methods. The test methods and procedures for each type of test as well as presentation of the results obtained and their significance are included in the body of the report. The three test methods were:

1) Conductometric evaluation of the resistance of concrete to rapid freezing and thawing,
2) Conductometric evaluation of the resistance of concrete to natural freezing and thawing, and
3) Conductometric evaluation of the pore size distribution of concrete and its correlation to concrete durability.

The report also includes recommendations for the continued development of these test methods.

DESCRIPTION OF CONCRETE MIXES FOR CONDUCTOMETRIC TESTING

In the project proposal it was stated that testing would begin with concrete containing a class III aggregate and Type I portland cement and that two sets of specimens would be prepared at four different entrained air contents, the first set with constant slumps and the second with constant water-cement ratios. In review of early test data obtained in Phase II of this project it was decided to deviate from the original mix plan. This was based on the desirability to determine if conductometric analysis could be used to distinguish between durable and nondurable coarse aggregates. It was recommended that mixes be formulated using three different coarse aggregates of varying durability. This new plan required nine mixes for each aggregate with three different water-cement ratios and three different entrained air contents bringing the total number of mixes to 27.

Constituent Materials

The portland cement used for the first part of this project was Lehigh Type I. It was used with all mixes containing Alden aggregate. Concrete specimens subjected to natural freezing and thawing and those for which conductometric pore size distributions were determined also contained Lehigh cement. The remaining mixes contained blended Type I portland cement provided by the Iowa DOT.

The fine aggregate used was from the Cordova quarry. It is the same sand the IDOT uses for its durability testing. For coarse aggregate three different aggregates were tested. The first aggregate was from the Alden quarry. This Class III limestone has a 3/4 inch maximum aggregate size and IDOT durability tests have shown that the durability of concretes containing this aggregate is largely independent of air content and water-cement ratio provided the mix contained a minimal amount of entrained air. The second aggregate selected was an oolitic limestone from the Montour quarry. The maximum aggregate size for this aggregate was also 3/4 inch. Iowa DOT durability studies have shown that concretes containing this aggregate have durability factors that vary quite markedly with changes in water-cement ratio and air content. Also, the Montour oolite has a much better field service record than measured laboratory durability factors would indicate. The final aggregate tested was from the Crescent quarry. It is a nondurable Class I aggregate. In accordance with IDOT specifications this aggregate has a 3/8 inch maximum aggregate size.

Mixing water was tap water from the Town Engineering Building. Blended with the water was the air-entraining agent Protex Pro-Air. This admixture was used at concentrations of 0 ml/100 lbs. cement, 49.8 ml/100 lbs. cement and 74.7 ml/100 lbs. cement. These concentrations resulted in total air contents of approximately 2, 6, and 8 percent, respectively.

Mix Proportions

Volumetric mix proportions centered around the IDOT C3 mix. In fact, the conductometric test mix designation LM used in this report has the same volumetric proportions as the IDOT C3 mix. All mixes have the same cement content of 605 lbs. per cubic yard. For each mix, the coarse aggregate occupies 55% of the total aggregate volume and the fine aggregate the remaining 45%. All combinations of three different water-cement ratios (0.43, 0.51 and 0.60) and three target air contents (1.5%, 6% and 9%) were used for making concrete mixes with each coarse aggregate. This made a total of 27 different concrete mixes.

The actual mix volumes are shown in Table 1. Mixes were given designations consisting of a three letter code. The first letter (L, M or H) is indicative of the water-cement ratio of the mix. The letter L indicates a low water-cement ratio equal to 0.43 with the letters M and H indicating a medium (0.51) and high (0.60) water-cement ratio, respectively. Similarly, the second letter is indicative of the total air content of the mix with the letters L, M, and H representing a low (1.5%), medium (6%) and high (9%) total air content, respectively. The last letter in the designation represents the coarse aggregate used with the letters A, C, and M representing the Alden, Crescent, and Montour aggregates, respectively. Mix designations thus reflect low (L), medium (M) and high (H) water-cement ratios and air contents along with aggregate types Alden (A), Montour (M) and Crescent (C) in the following order:

(WATER-CEMENT RATIO) (AIR CONTENT) (AGGREGATE TYPE)

Mix proportions were strictly adhered to and no attempt was made to control the slump of the different mixes. As a result some of the mixes had rather high slumps and molding specimens without segregation was difficult for some of the mixes. These mixes were those with high air contents and high water-cement ratios. Since all mixes had the same cement content the amount of air-entraining admixture added did not vary with water-cement ratio. Properties of the mixes tested are listed in Tables 2, 3, 4, and 5. Tables 2, 3 and 4 list the properties of the mixes used for conductometric evaluation of freeze-thaw resistance of concrete. Properties of mixes used for conductometric evaluation of the resistance of concrete to natural freezing and thawing, and conductometric evaluation of concrete pore size distribution and correlation to concrete durability are shown in Table 5.

Table 1. Volumetric mix proportions for conductometric tests.

Materials	Mix Volumes								
	LL	LM**	LH	ML	MM	MH	HL	HM	HH
Cement	.1142	.1142	.1142	.1142	.1142	.1142	.1142	.1142	.1142
Water	.1538	.1538	.1538	.1834	.1834	.1834	.2158	.2158	.2158
Fine Agg.	.3221	.3019	.2885	.3088	.2886	.2752	.2943	.2741	.2606
Coarse Agg.	.3949	.3701	.3535	.3786	.3538	.3372	.3607	.3359	.3194
Air	.015*	.06	.09	.015*	.06	.09	.015*	.06	.09
Total	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000
W/C ratio	0.43	0.43	0.43	0.51	0.51	0.51	0.60	0.60	0.60

*No air entraining admixture added

**Volumes same as for IDOT C3 mix.

Table 2 - Properties of mixes containing the Alden aggregate from which beams were cast and subjected to rapid freezing and thawing.

Mix Designation	Water-Cement Ratio	Air Content	Slump (inches)	Unit Weight
LLA	0.43	2%	2	151.4 pcf
LMA	0.43	5.8%	4.5	142.3 pcf
LHA	0.43	6.8%	6.5	141.0 pcf
MLA	0.51	2%	6.5	152.2 pcf
MMA	0.51	5.8%	>8	141.1 pcf
MHA	0.51	7.8%	>8	138.9 pcf
HLA	0.60	2%	>8	148.7 pcf
HMA	0.60	5.9%	>8	139.9 pcf
HHA	0.60	7.9%	>8	136.1 pcf

Table 3 - Properties of mixes containing the Crescent aggregate from which beams were cast and subjected to rapid freezing and thawing.

Mix Designation	Water-Cement Ratio	Air Content	Slump (inches)	Unit Weight
LLC	0.43	1.75%	1.75	146.9 pcf
LMC	0.43	5.5%	3	145.5 pcf
LHC	0.43	9.5%	6	136.5 pcf
MLC	0.51	1.5%	6	152.1 pcf
MMC	0.51	6.4%	7	139.6 pcf
MHC	0.51	8.5%	>8	136.2 pcf
HLC	0.60	2%	>8	150.6 pcf
HMC	0.60	7%	>8	144.3 pcf
HHC	0.60	9%	>8	142.6 pcf

Table 4 - Properties of mixes containing the Montour aggregate from which beams were cast and subjected to rapid freezing and thawing.

Mix Designation	Water-Cement Ratio	Air Content	Slump (inches)	Unit Weight
LLM	0.43	1.5%	6.5	152.1 pcf
LMM	0.43	5.5%	>8	144.9 pcf
LHM	0.43	8%	>8	140.2 pcf
MLM	0.51	1.5%	>8	150.5 pcf
MMM	0.51	5%	>8	145.5 pcf
MHM	0.51	8.5%	>8	137.8 pcf
HLM	0.60	1.5%	>8	149.5 pcf
HMM	0.60	---	>8	143.2 pcf
HHM	0.60	---	>8	142.7 pcf

Table 5 - Properties of mixes from which beams were cast and subjected to natural freezing and thawing and from which cylinders were cast and conductometric pore size distributions determined.

Mix Designation	Water-Cement Ratio	Air Content	Slump (inches)	Unit Weight
LLA	0.43	1.8%	3	150.2 pcf
LMA	0.43	6.0%	7.5	141.2 pcf
LLC	0.43	2.0%	2.5	152.2 pcf
LMC	0.43	5.0%	3.5	146.6 pcf
LLM	0.43	2.0%	1.5	152.7 pcf
LLC	0.43	6.5%	6.5	139.0 pcf

CONDUCTOMETRIC TESTING OF CONCRETE

It was stated in the project proposal:

> "The ultimate goal of the proposed research is to establish specifications for concrete quality control in respect to its freeze-thaw durability. It appears to the authors of this proposal that a plausible solution (probably the only solution) to this problem could be achieved by continuously monitoring the physicochemical state of concrete during freezing and thawing cycles as a function of all the variables such as temperature, rate of cooling and warming, degree of saturation, air content, pore structure and the type of freezing-thawing medium. By physicochemical state of a sample we mean the dimensional stability and integrity of its solid and void spaces, the frozen and unfrozen fractions of its water content, and the sizes of its voids occupied by air, water (liquid) and ice. When the functional relationships between the physicochemical state and the variables are developed extrapolations can be made to various field conditions. Once these studies are completed we anticipate being able to develop a reasonably rapid test for predicting the freeze-thaw durability of concrete on the basis of changes occurring under a fixed set of ambient conditions in its physicochemical states during a limited number of cycles. Extrapolations to a different set of ambient conditions will then be possible to make using the functional relationship established as mentioned above."

The progress made during the course of this research project has allowed us to make significant steps towards the achievement of this ultimate goal. As stated earlier the three types of testing undertaken were:

1) Conductometric evaluation of the resistance of concrete to rapid freezing and thawing,

2) Conductometric evaluation of the resistance of concrete to natural freezing and thawing,

3) Conductometric evaluation of concrete pore size distributions and correlation to concrete durability.

In the first type of testing, relative dynamic moduli of elasticity, as evaluated by ASTM Method C666-84, (Standard test method for resistance of concrete to rapid freezing and thawing) are correlated to changes in the electrical conductance of concrete beams subjected to repeated freezing and thawing. In this report we will show that these changes can be used to predict the durability of concrete in much the same manner as changes in fundamental transverse frequency or pulse velocities.

In the second test (evaluation of resistance to natural freezing and thawing) the same conductometric changes were monitored in concrete beams exposed to the ambient conditions imposed by a Iowa winter. Continued development of this type of testing could allow functional relationships between the physicochemical state of the concrete and the environmental variable to be developed.

The last type of testing (conductometric pore size analysis) is the most versatile of the three. With the methodology developed for the conductometric porosimeter the physicochemical state of the concrete can be monitored under a nearly limitless variety of laboratory and field freeze-thaw conditions.

Obviously a complete investigation of all the variables contributing to the freeze-thaw durability could not be accomplished during the time duration of this research project. The purpose of this report is to present and analyze results of testing undertaken and illustrate how the experimental techniques developed during this project can be used in further investigations of concrete durability.

All mixes were tested after 28 days of moist curing. The saturation state of the concretes tested was limited to the degree of saturation that a concrete would attain at ambient temperatures if a limitless supply of water was available. Finally, two freeze-thaw conditions were imposed. For all laboratory tested samples this was the 40°F to 0°F cycle established by ASTM C666-84. For samples exposed to natural freezing and thawing the cycling was imposed by weather conditions.

Conductometric Evaluation of the Resistance of Concrete to Rapid Freezing and Thawing

Methods and Procedures

For this type of testing 1/8 inch diameter stainless steel rods were cast in one end of 3" by 3" by 16" concrete beams. To do this the 1/4" steel bottoms of the beam molds recommended by ASTM were replaced with 1" thick Plexiglas bottoms. Holes the same size as the 1/8" stainless steel electrodes were drilled in the Plexiglas bottom plates at the desired spacing. The electrode geometry for the beam samples is shown in Figure 1. The electrodes were force fitted into the holes to provide a secure geometry. The molds were then assembled and specimens were cast and allowed to cure for 24 hours in a humid curing room. The molds were then disassembled and the Plexiglas bottoms were tapped off with a rubber hammer and each electrode cut flush with the bottom of the beam.

The samples were subjected to rapid freezing and thawing in accordance with ASTM C666-84. Samples were frozen in water and thawed in water. Initially measurements were taken at 5 to 6 cycle intervals. As testing continued the number of cycles between testing was increased. The fundamental transverse frequency for each beam was measured on the half of the beam containing no electrodes. Conductance measurements were taken using each possible pair of electrodes using a Solomat model 2009 conductivity meter. Conductance measurements are particularly sensitive to changes in temperature. Thus, at the end of a cycle for which conductance readings were to be taken, samples were placed in an ice bath for at least 30 minutes to assure all testing was conducted at nearly the same concrete temperature.

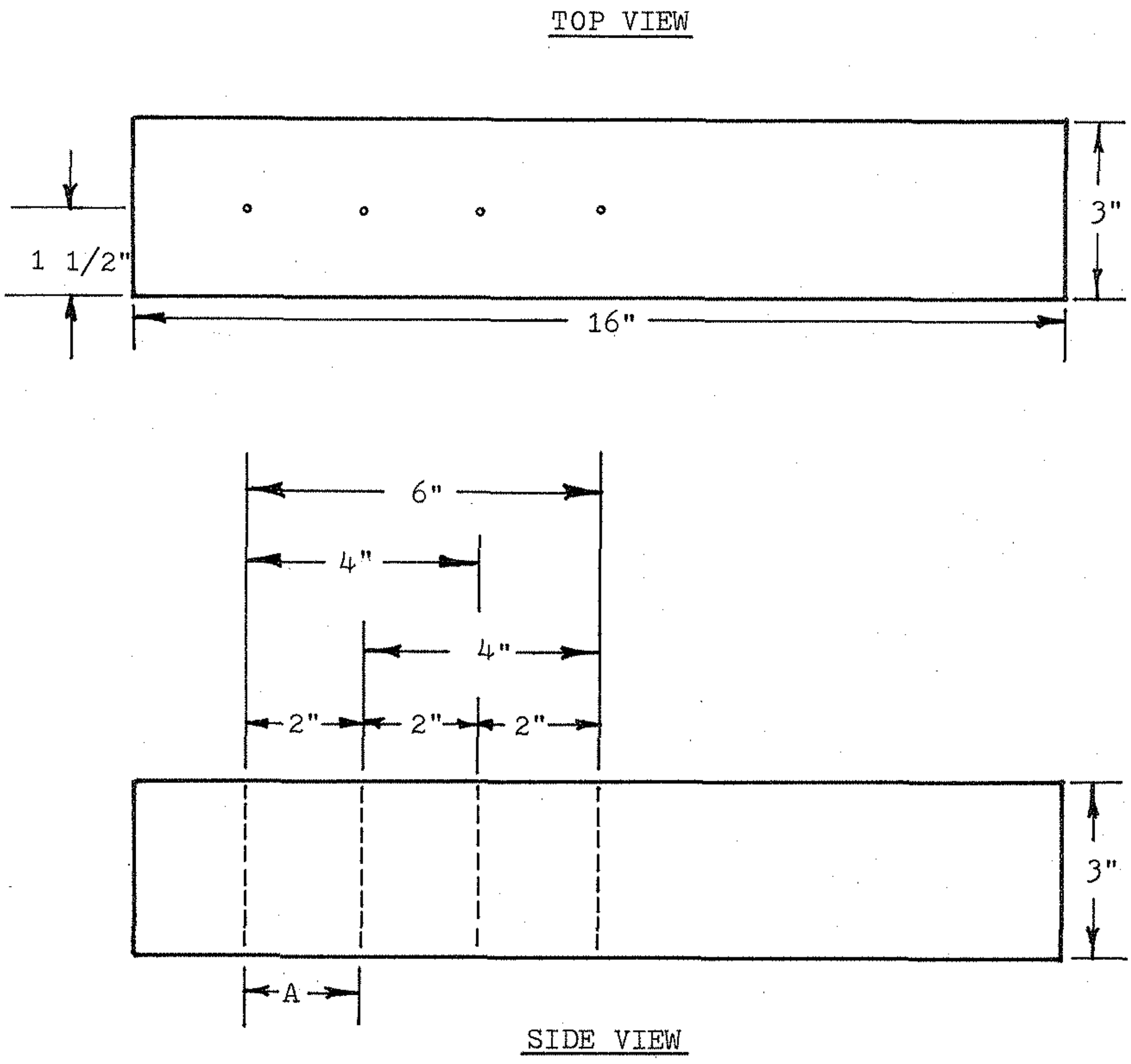


Figure 1. Electrode geometry for beam samples.

Results and Discussion

A total of 27 different mixes containing three different aggregates and having three different water-cement ratios and air contents were tested in this manner. In this report a parameter termed inverse relative conductance (IRC) is used to evaluate the data. This parameter is defined as:

$$IRC_x = C_o/C_x, \quad (1)$$

where IRC_x is the inverse relative conductance at the end of x number of freeze-thaw cycles, C_o is the conductance prior to any freeze-thaw cycling, and C_x is the conductance at the end of x number of freeze-thaw cycles. Also, test data indicated that the inverse relative conductance is independent of electrode geometry, consequently, only data for one particular electrode geometry for each mix will be presented. Thus, data presented herein are for the electrode geometry labeled A in Figure 1.

Appendix A contains graphs of inverse relative conductance versus the number of freeze-thaw cycles for all the mixes tested. Table 6 lists the number of freeze-thaw cycles, the inverse relative conductance and relative dynamic modulus of elasticity at the termination of testing for each mix. Figure 2 presents this information in a graphical format. From the examination of Figure 2 it is clear that the inverse relative conductance is directly related to relative dynamic modulus and could be used as an index of concrete durability. The solid line through the points in this graph represents a linear regression of the data. This regression results in a correlation coefficient of 0.761. There were three mixes (mix designation HMM, HHM, and HHC) tested for which the relative dynamic modulus decreased with increasing number of freeze-thaw cycles while their electrical conductances practically remained constant (see Appendix A for these mixes). In these instances the concrete mixes had a combination of

Table 6 - Inverse Relative Conductances and Relative Dynamic Moduli for concrete beam samples at the termination of freeze-thaw cycling.

Mix Designation	# of Cycles at Termination	Inverse Relative Conductance	Relative Dynamic Modulus
LLA	36	0.462	0.287
LMA	195	0.899	0.948
LHA	121	0.944	0.910
MLA	28	0.430	0.308
MMA	112	0.946	0.900
MHA	108	0.928	0.909
HLA	32	0.412	0.389
HMA	108	0.930	0.894
HHA	108	0.900	0.905
LLC	26	0.531	0.350
LMC	301	0.694	0.559
LHC	301	0.626	0.715
MLC	42	0.593	0.361
MMC	311	0.862	0.932
MHC	311	0.769	0.846
HLC	42	0.223	0.250
HMC	254	0.817	0.777
HHC	254	1.023	0.776
LLM	92	0.453	0.235
LMM	303	0.815	0.895
LHM	303	0.806	0.953
MLM	30	0.564	0.272
MMM	303	0.874	0.847
MHM	303	0.868	0.940
HLM	30	0.435	0.262
HMM	303	1.010	0.819
HHM	231	0.984	0.598

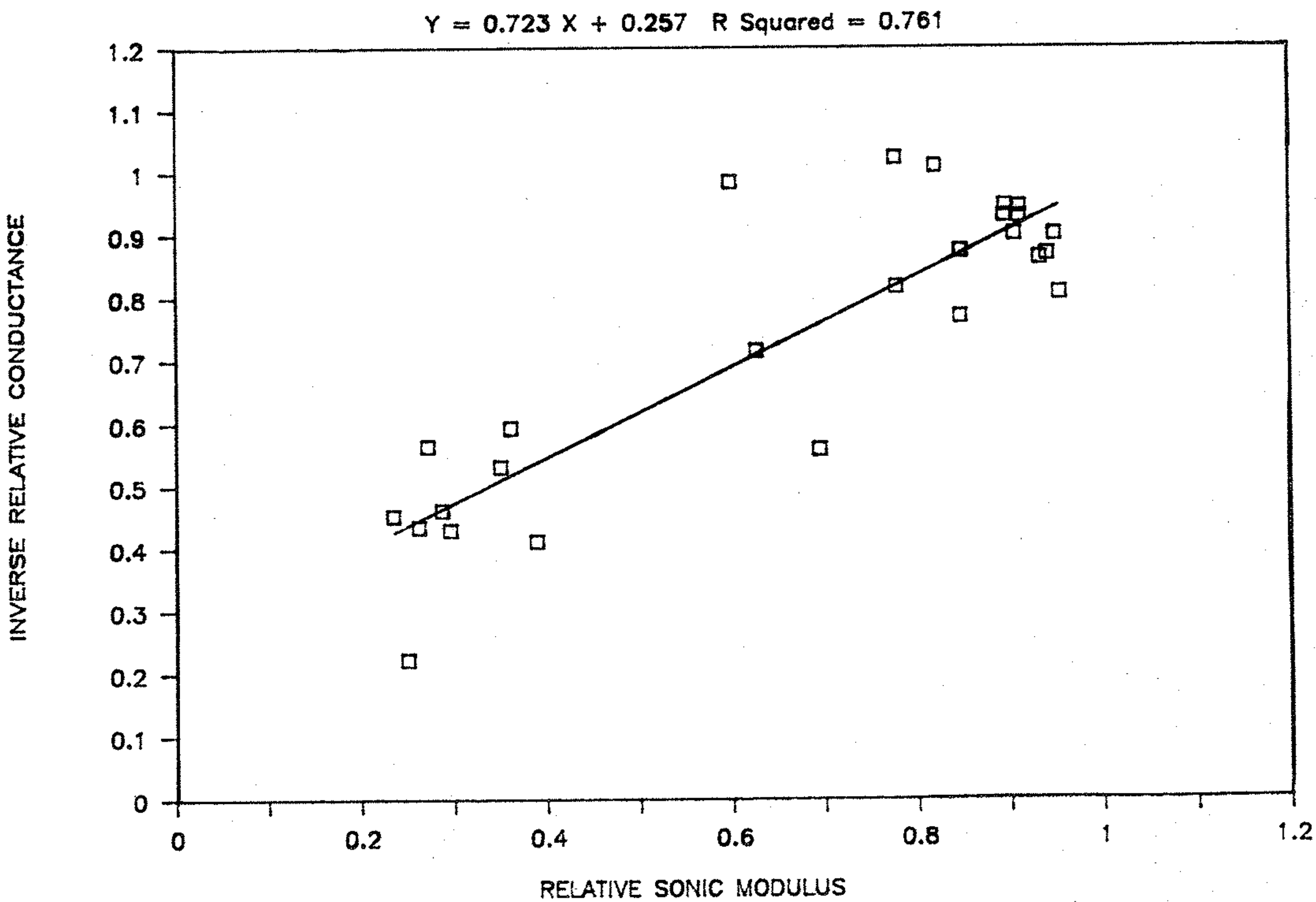


Figure 2. Correlation between inverse relative conductance and relative modulus.

high air content and high water-cement ratio which resulted in an extremely high initial conductance and extremely fluid mix with a slump which would be unacceptable for normal concrete applications. Such mixes should be expected to be nondurable due to possible segregation of the aggregate from the mortar phase on the one hand and to the high initial water content as evidenced by the high value of initial conductance on the other.

Our concrete durability research during the last six years has shown that the susceptibility of concrete to saturation (controlled by both the rate with which moisture absorption occurs and the final degree of saturation) plays a major role in the assessment of freeze-thaw durability of concrete. Recently, we have been the recipient of a National Science Foundation (NSF) grant entitled "Frost Susceptibility of Concrete in Near-Saturated States" (see Appendix D). Future activities of this new project are expected to help expand on the scope of the research conducted under the present project HR-272.

Since both freeze-thaw durability and conductance of concrete should depend on its degree of saturation and how fast this saturation occurs, conductance measurements should reflect the durability of concrete subjected to ingress of water without a recourse to freeze-thaw cycles. Although the results of the present study support this hypothesis, more work is needed to establish a functional relationship. This is one of the objectives of our NSF project. The present study concentrated on electrical conductance of concrete subjected to freeze-thaw cycles. Thus, efforts were devoted to determination of the degree of degradation caused by freeze-thaw cycles by using electrical conductance.

For concrete mixes that have acceptable values for slump, an open pore structure and aggregate segregation are not expected to occur. Therefore, for such mixes changes in electrical conductance can be used to predict their durability much in the manner as is done with dynamic modulus. Indeed, this is the case for mixes that have acceptable slumps.

In Figure 3, inverse relative conductance is plotted against relative dynamic modulus as in Figure 2 except the values for the three previously mentioned mixes (HMM, HHM and HHC) have been omitted. A linear regression of this data results in a correlation coefficient of 0.866.

Figures 4, 5, and 6 illustrate the variation of conductance with air content and water-cement ratio for mixes containing the Alden, Crescent, and Montour aggregates, respectively. Initial observations that the conductance of a concrete increases with increasing water-cement ratio seems to apply to all mixes although one deviation from this is observed in the case of the Crescent aggregate. This deviation may be due to the high absorption particles of the aggregate. As one might expect the increasing amount of excess water that is present in mixes containing absorptive aggregates and high water-cement ratios results in a concrete with a larger amount of conducting capillary pores. The variation of conductance with air content is more interesting. In the case of the durable Alden aggregate conductance increases with increasing air contents, however for the Montour and Crescent aggregates this is not the case. For both of these aggregates in the mixes with a water-cement ratio equal to 0.43 the conductance decreases with increasing air content. This suggests that there are fundamental differences in the interactions of the air void and capillary void systems of the different aggregates. In the case of mixes containing the Alden aggregate the air void system provides additional conductive paths suggesting that capillary voids and air voids are interconnected. However for mixes containing the Montour and Crescent aggregates with low water-cement ratios the presence of air voids decreases the conductance suggesting these air voids are isolated from the capillary void system. Continued investigations of these

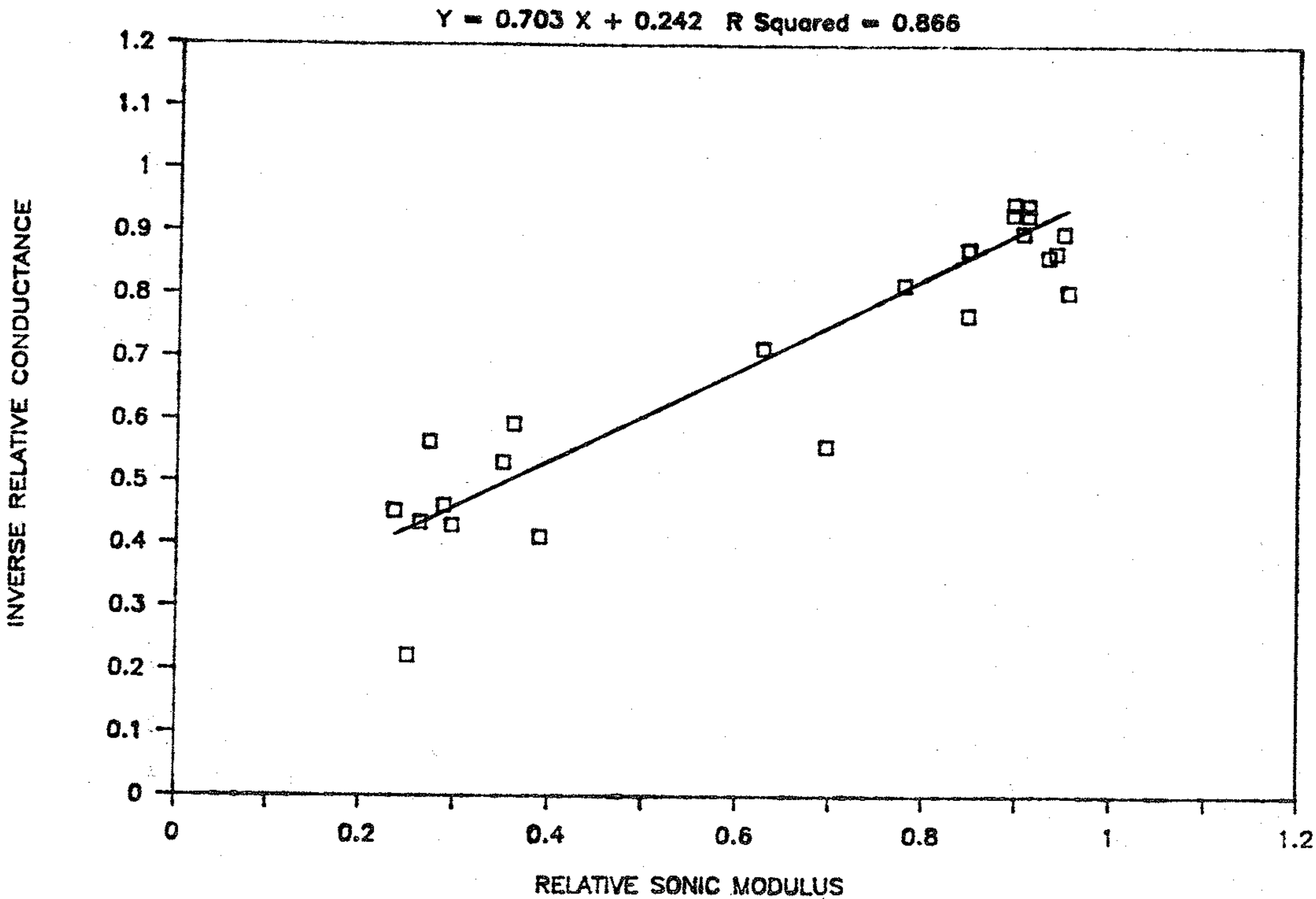


Figure 3. Correlation between inverse of relative conductance and relative modulus (same as Figure 2 except three data points are removed).

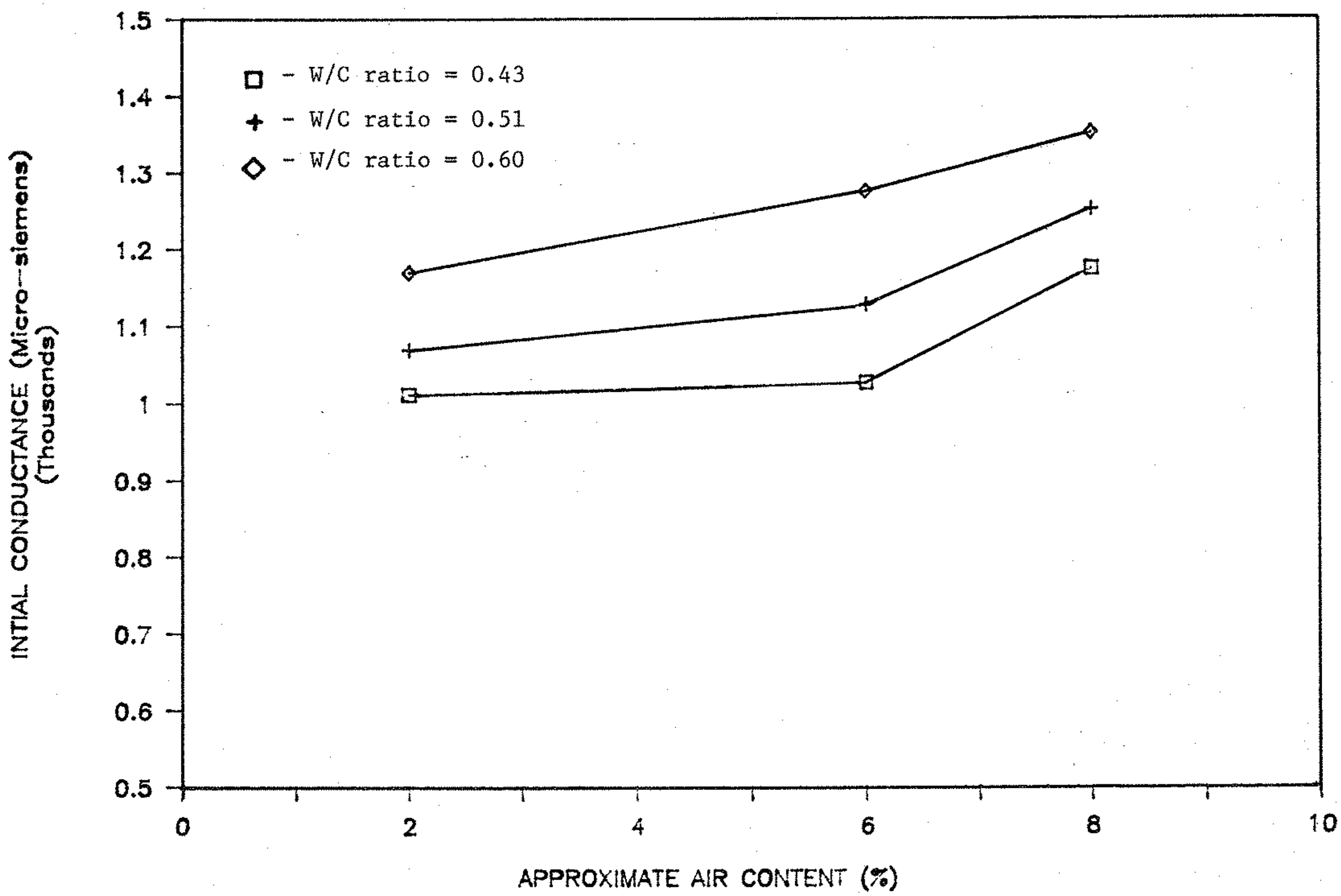


Figure 4. Variation of conductance with air content and W/C for mixes made with Alden aggregate.

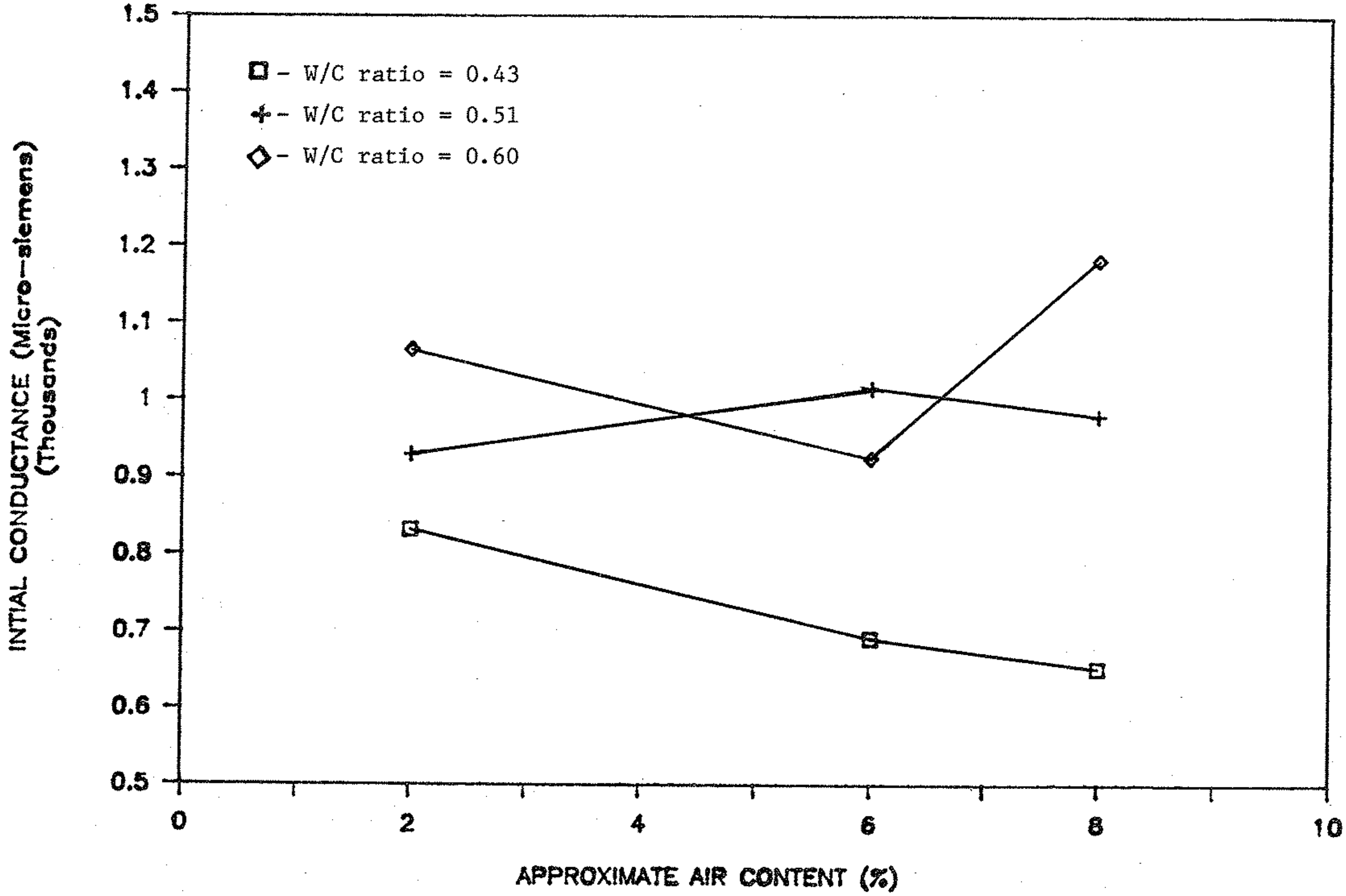


Figure 5. Variation of conductance with air content and W/C for mixes made with Crescent aggregate.

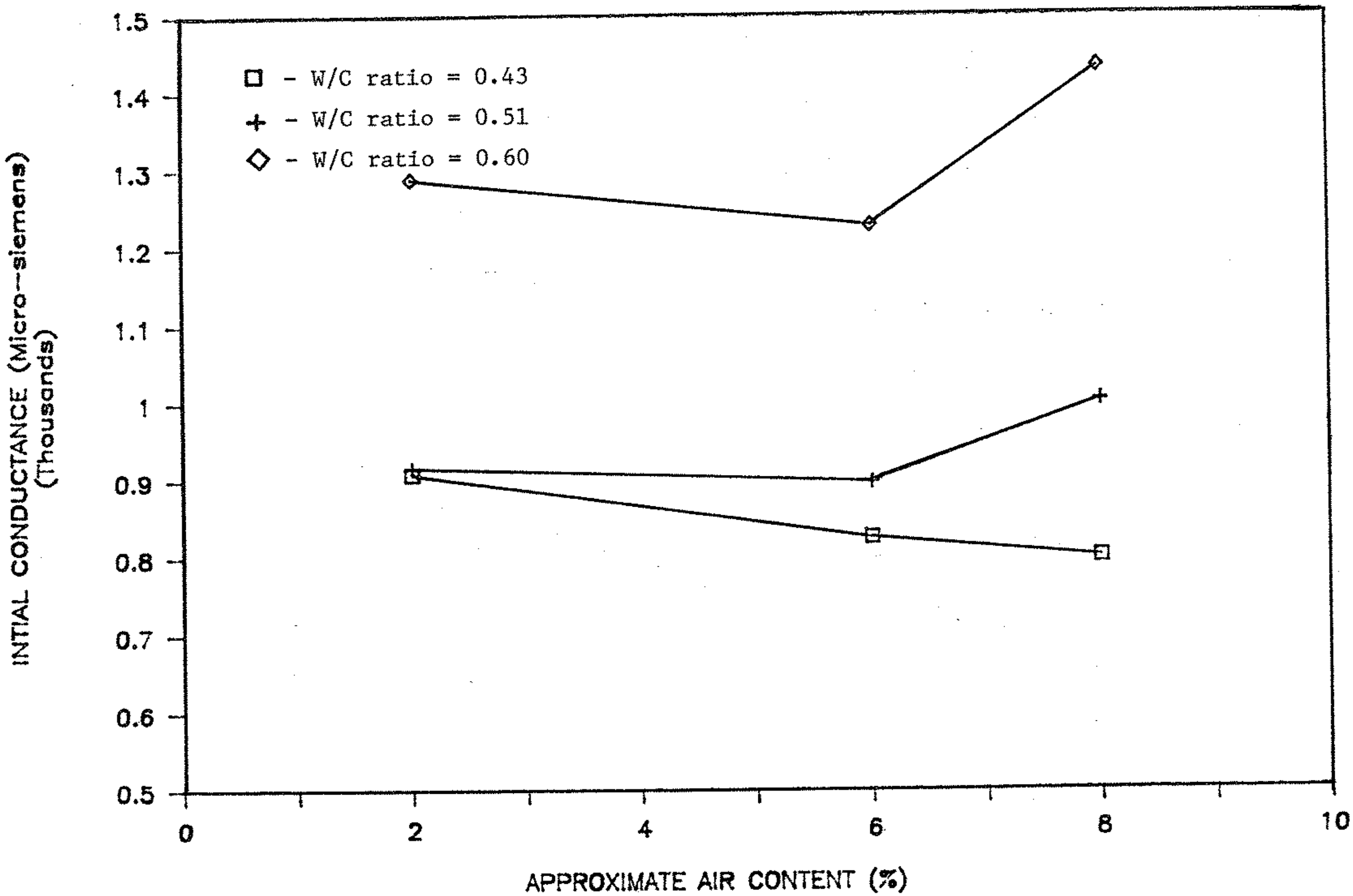


Figure 6. Variation of conductance with air content and W/C ratio for mixes made with Montour aggregate.

relationships should result in a better understanding of the relationship of the air void and capillary void system to the mechanics of freeze-thaw deterioration. Finally it appears that the overall electrical conductance of the least durable Crescent is less than that of the most durable Alden with the conductance value for the Montour being in between.

Conductometric Evaluation of the Resistance of Concrete to Natural Freezing and Thawing

Methods and Procedures

Beam specimens were prepared in the same manner as outlined previously and were placed in a bed of saturated sand in a tank and exposed to the freeze-thaw cycles of an Iowa winter. The electrical conductance of the beams was measured several times during the course of the winter. An attempt was made to take measurements when the beams were at or near 32° F. The temperature at the time of measurements varied from 28.9°F to 36.4°F. Six beams from six different concrete mixes were tested in this manner. Three of the mixes had the same proportions as the IDOT C3 paving mix. The other three had similar proportions except that they contained no air entraining admixture. Tables 1 and 5 list the mix proportions and mix properties, respectively, for these mixes.

Results and Discussion

Initially it was hoped frequent monitoring of temperatures and conductance would allow us to develop functional relationships between ambient conditions and concrete durability and allow us to modify laboratory testing procedures to these ambient conditions. It was also hoped that monitoring both the temperature and conductance would allow us to monitor the length of natural freeze-thaw cycles, the rate of cooling and warming, changes in the degree of saturation and the number of freeze-thaw cycles. It became apparent very soon after placing the samples that to follow the rapidly changing environmental conditions imposed on the samples would require nearly continuous monitoring of these measurements. This is largely due to the unanticipated variation in electrical conductance due to continued hydration and variation in the degree of saturation of the concrete. Although current technology would allow these types of continuous measurements to be taken such equipment was not available to us. Consequently, we began taking measurements over longer periods of time. We felt comparative analysis of conductance measurements taken on durable and nondurable concretes, over time, would reveal fundamental differences in the conductometric changes which occurred in these concretes. Figure 7 illustrates the results of this testing. As can be seen from this graph there is little distinguishable difference between the conductometric behavior of the nondurable non-air entrained mixes (labeled LLA, LLM and LLC) and that of the more durable air entrained counterparts (labeled LMA, LMM and LMC). Because of the short time over which these samples were monitored and especially because of the very mild winter we had in Iowa, there seems to be no discernable difference in the conductometric behavior of the different concretes. We plan to include this type of experiment in our NSF project. Copies of the reports for the NSF project will also be submitted to the IDOT.

Conductometric Evaluation of Concrete Pore Size Distributions and Correlation to Concrete Durability

Methods and Procedures

A new method for determining the pore structure of porous materials has been developed. The methodology combines relationships correlating the volume of unfrozen pore solution to electrical conductance, the pore solution freezing temperature to electrical conductance, the pore solution freezing temperature to pore radii, and the electrical conductivity of the pore solution to temperature as illustrated below:

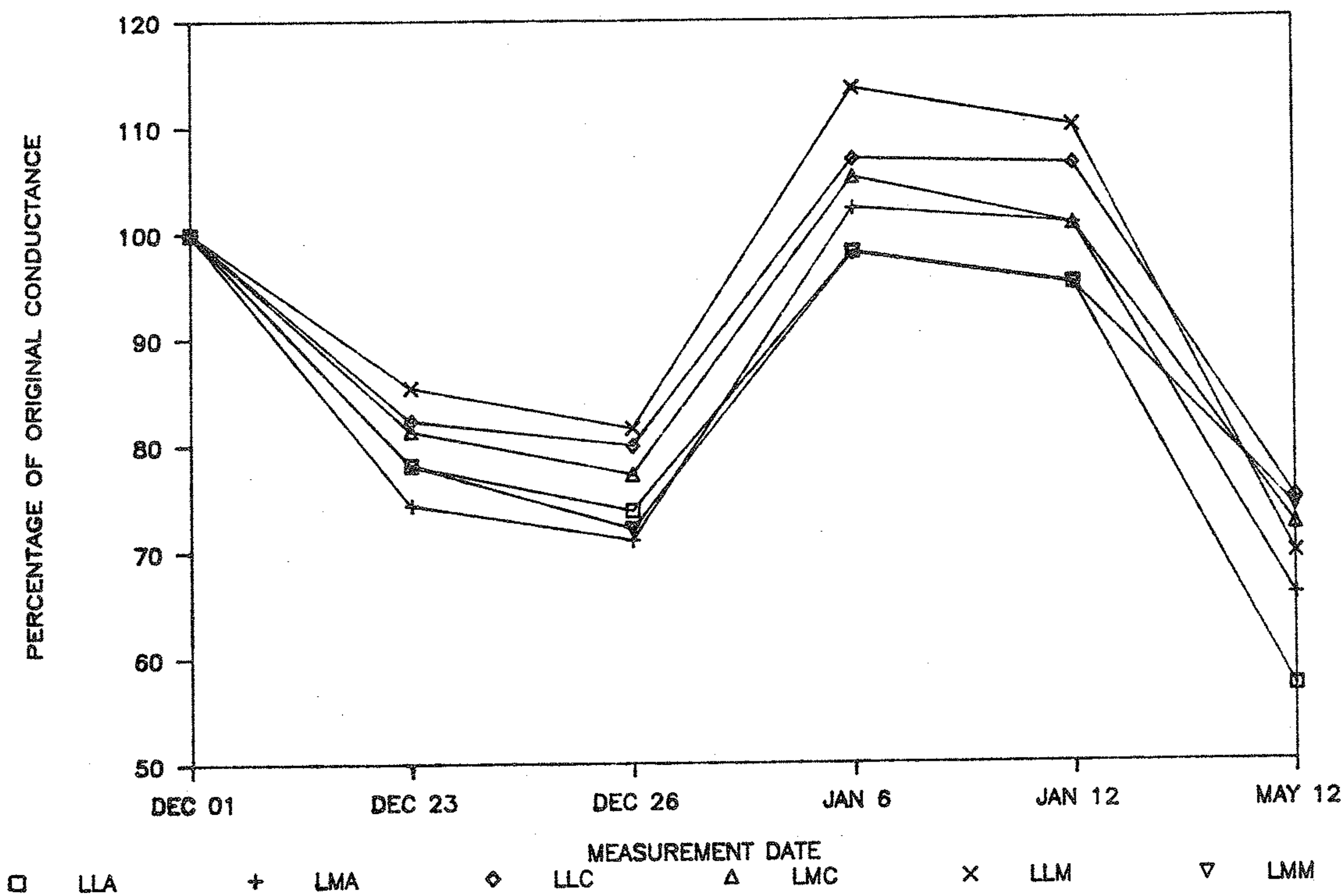
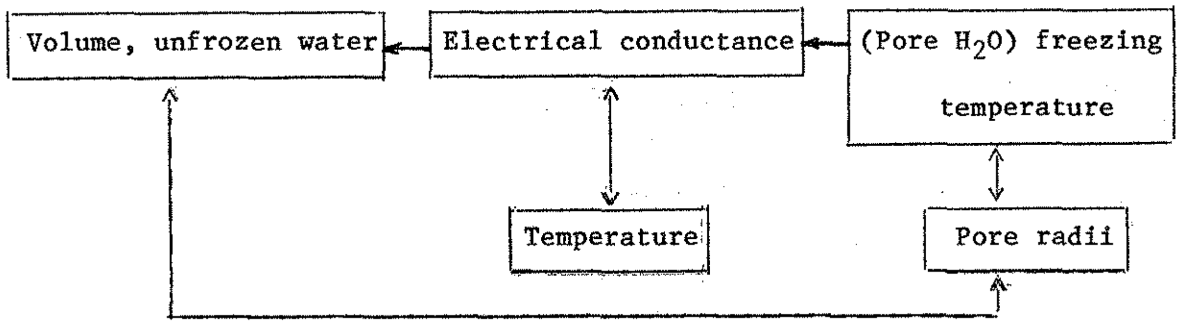


Figure 7. Relative conductance of samples subjected to natural freezing and thawing as a function of date.



These relationships are used with measurements of the electrical conductance and temperature to obtain pore size distributions of saturated porous materials subjected to a cycle of capillary freezing and melting.

Based on this methodology, a conductometric phase transition porosimeter was constructed. The pore size distribution of porous Vycor glass (a standard for pore size calibration) was measured using this porosimeter. The modal neck and body radii measured by this method are 28 and 55 angstroms, respectively. The modal neck and body radii of the same Vycor sample as determined by mercury intrusion porosimetry are 30 and 85 angstroms and by dilatometric phase transition porosimetry, 27 and 49 angstroms, respectively. Modal neck sizes determined by all three methods are comparable. Modal body sizes determined by the two phase transition methods are also comparable. The modal body size determined by mercury porosimetry is significantly larger. This difference is due to problems arising from the entrapment of mercury during the extrusion phase of mercury porosimetry testing. A complete discussion of conductometric phase transition porosimetry is included in Appendix B and for the sake of brevity will not be included in the main body of this report.

The conductometric phase transition porosimeter consists of a conductance meter, a cryostat, a thermistor, the measurement circuitry, a microcomputer, and a plotter.

Conductance measurements are made with a Solomat 2009 conductivity meter. This meter's high frequency alternating current excitation and the use of stainless steel or tungsten electrodes minimizes polarization effects. The instrument has a resolution of 0.1 micro-siemens. Digitized conductance output from the meter is relayed to the computer.

A thermistor manufactured by Thermometrics is used to measure the temperature of the sample. The thermistor is used as a variable resistor in a square wave oscillator. The oscillator's output frequency varies with corresponding changes in the resistance of the thermistor. The computer then measures the period of the square wave.

The temperature of the sample is controlled by placing the sample in a suitable container and immersing that container in a Haake cryostat filled with iso-propyl alcohol. The temperature of the cryostat is controlled by the computer through a digital to analog converter.

The computer used is an Apple IIe computer equipped wth two input/output cards from John Bell Engineering. These cards serve as an interface between the measurement circuitry and the computer. Software was developed to allow the computer to control the temperature of the cryostat, gather test data, perform necessary data processing, and output the processed data to either the computer's video monitor or a Hewlett-Packard plotter. Cycle starting and ending temperatures, cooling rates, and data collection intervals are all input by the user. In addition, the user can input the total number of cycles for a test sequence and designate different cycle parameters for each cycle within the sequence. These features allow the user to tailor the test sequence to a particular pore system. They also allow periodic determinations of pore size distribution to be made for samples subjected to repeated cycles of freezing and thawing. With this system, a wide range of ambient freezing and thawing conditions can be simulated.

Appendix C of this report contains a user's manual for the conductometric phase transition porosimeter. This manual contains instructions for the operation of the porosimeter, porosimeter circuitry schematics, computer program descriptions, and computer program listings. A patent application is being made for this methodology by Iowa State University. Earlier, a patent for the phase transition porosimeter developed under IDOT project HR-258 and utilized in the present investigation was obtained and licensed to OMICRON Company by ISU.

Two of the most significant advantages the conductometric phase transition porosimeter has over currently available commercial porosimeters, including the phase transition porosimeter, are the minimal sample preparation procedure which results in less disturbance of pore structure; and the ability to determine pore size distributions of large samples, much larger than other porosimeters can accommodate, thus minimizing sampling error.

For the evaluation of pore size distributions of portland cement concrete, 4-inch diameter by 4-inch high cylinders were molded with an imbedded electrode configuration as shown in Figure 8. The electrodes are fixed in a Plexiglas bottom plate during molding. To prevent surface conductance at the bottom of submersed specimens the bottom portions of the electrodes were embedded in small Plexiglas studs. These studs, when inserted in holes cut in the mold bottom, also held the electrodes in place during casting. Also, the upper portion of the electrodes were covered with nonconductive plastic shrink tubing to eliminate conductance between the exposed electrodes. Electrodes were cut from 1/8 inch diameter stainless steel rods. After curing, a 1 1/2 inch deep hole is drilled in the cylinder to accommodate a plastic termistor housing. The thermistor is placed in the housing to obtain the necessary temperature measurements.

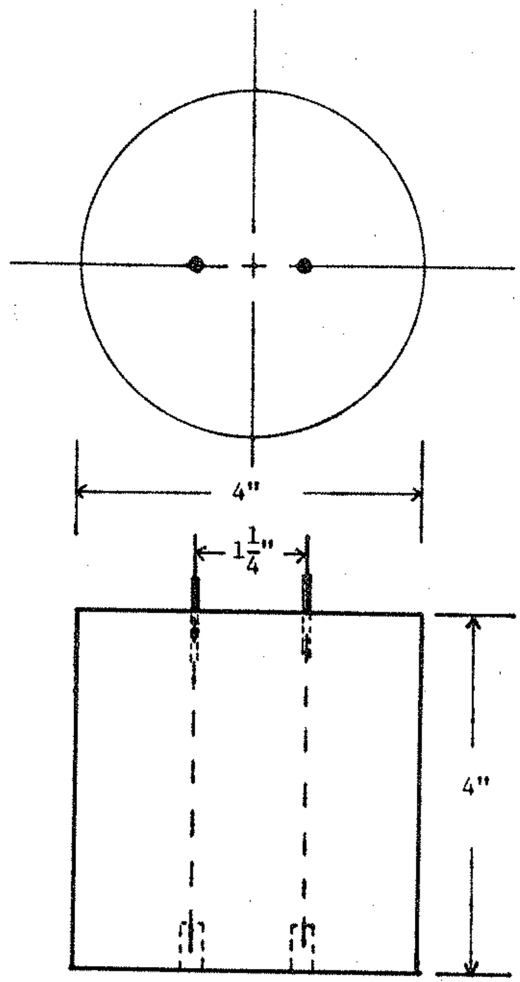


Figure 8. Electrode geometry of concrete samples for measurement of conductometric pore size distribution.

During testing, the cylinder is placed in a thin-walled plastic container, and the void between the walls of the cylinder and the container are filled with Ottawa sand. The sand is then saturated with water. Experimentation has shown that the silica crystals in the sand prevent supercooling of the bulk water surrounding the sample. This allows both warming and cooling data to be gathered.

Test sequences for the concretes tested in this manner involved alternating diagnostic cycles and rapid freeze-thaw cycles. For the diagnostic cycles the temperature of the bath was lowered from 7°C to -30°C at the rate of 3°C per hour. Conductance and temperature data collected at 0.1°C intervals composed the cooling portion of the diagnostic cycle. These data are used to calculate the pore neck size distribution for the concrete. The temperature is then raised to 7°C at the same rate. Again, conductance and temperature data were collected at 0.1°C intervals composing the warming portion of the diagnostic cycle. The latter data were used to calculate the pore body size distribution of the concrete. Diagnostic test runs were made with a 7 kilo-ohm resistor connected in series with the cylinder allowing the more sensitive range on the conductance meter to be used. Data processing routines calculated the conductance of the cylinder alone from the measured conductance. The concretes were subjected to several rapid (20°C per minute) freeze-thaw cycles between each diagnostic cycle. For these rapid freeze-thaw cycles the upper and lower limits of temperature for each cycle were 4°C and -16°C, respectively. This temperature range along with the 20°C per hour rate of temperature change resulted in cycles conforming with the specifications set by ASTM C666.

Six cylinders from six different mixes were tested in this manner. Three mixes had the same proportions as the IDOT C3 paving mix. The other three had similar proportions except they contained no air entraining admixture. Three different coarse aggregates were used (Alden, Crescent, and Montour). Tables 1 and 5 list the mix proportions and the mix properties of samples tested in this manner. For all samples tested the first cycle was a diagnostic cycle. Subsequent diagnostic cycles were the 5th, 10th, 15th, 20th and 25th cycles. After 25 cycles, testing for the non-air entrained concretes was discontinued. For the air-entrained concretes cycling was continued until 100 cycles with diagnostic cycles at 35, 45, 55, 75 and 100 cycles. At this point test cylinders were stored for a period of approximately three months. Later cycling was restarted for the three air-entrained concretes and continued until 250 cycles had been completed. During this stage of testing diagnostic cycles were run at 150, 200 and 250 cycles.

Results and Discussion

In Figures 9 and 10 the conductance of the cylindrical samples measured at 0°C is plotted versus the number of freeze-thaw cycles the sample had been subjected to at the time of the measurement. The lines drawn on these graphs represent linear regressions of the data. Figure 9 presents this information for the concrete cylinders which contained no entrained air. It can be seen from this plot that the conductance increases rapidly with the increasing number of freeze-thaw cycles for all mixes without entrained air. The behavior of the concretes containing entrained air differs, however. Figure 10 presents the same information for these mixes. From this figure it can be seen that conductance remains nearly constant for the mix containing the Alden aggregate while for the mixes containing the Montour and Crescent aggregates the conductance increases with increasing number of freeze-thaw cycles. This behavior is as expected for these mixes. IDOT durability testing has shown the Alden aggregate to have a high durability factor while the Montour and Crescent have lower durability factors. In Figures 11 and 12 the parameter inverse

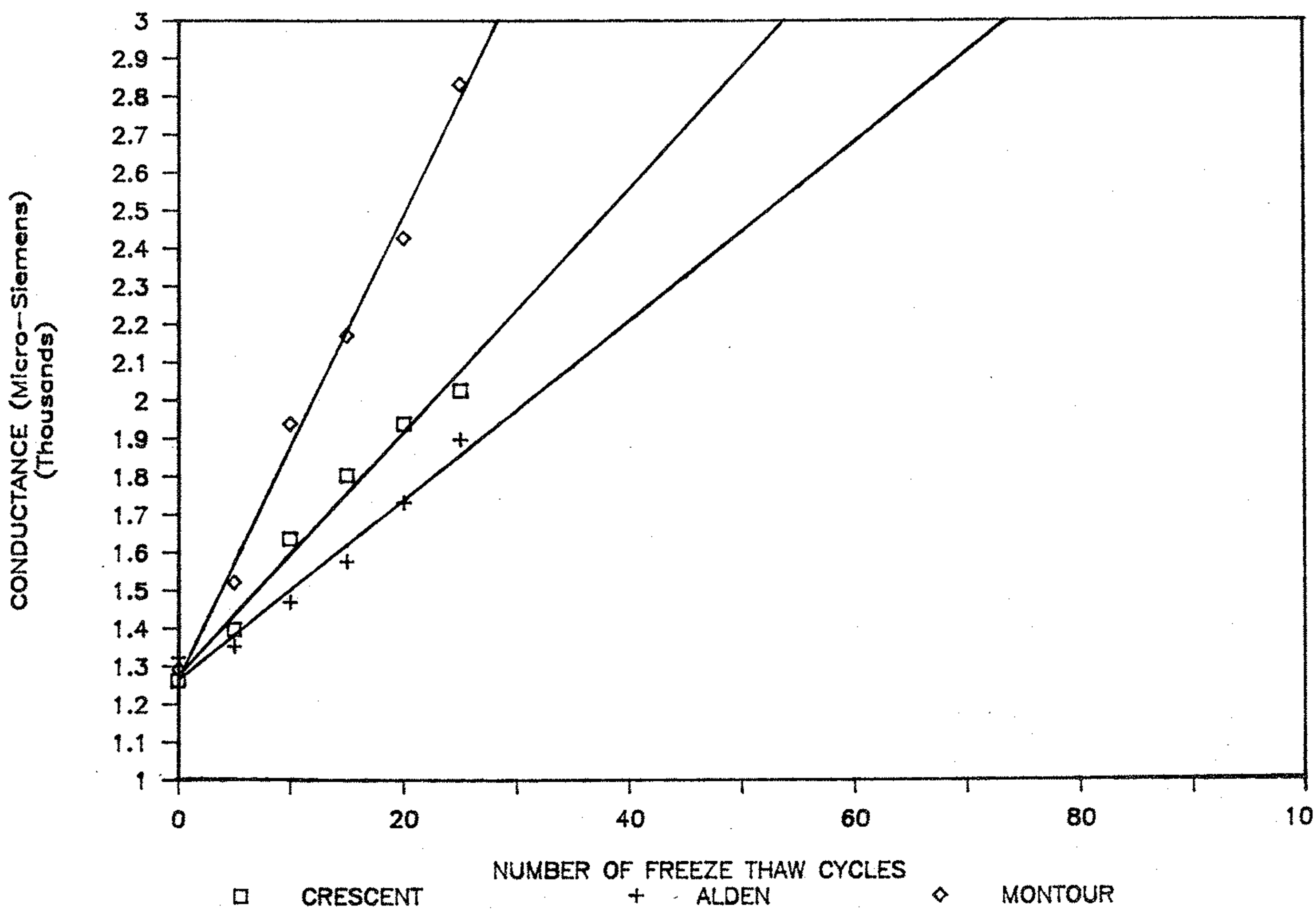


Figure 9. Variation of conductance with number of F/T cycles for mixes (IDOT C3) prepared without air entrainment.

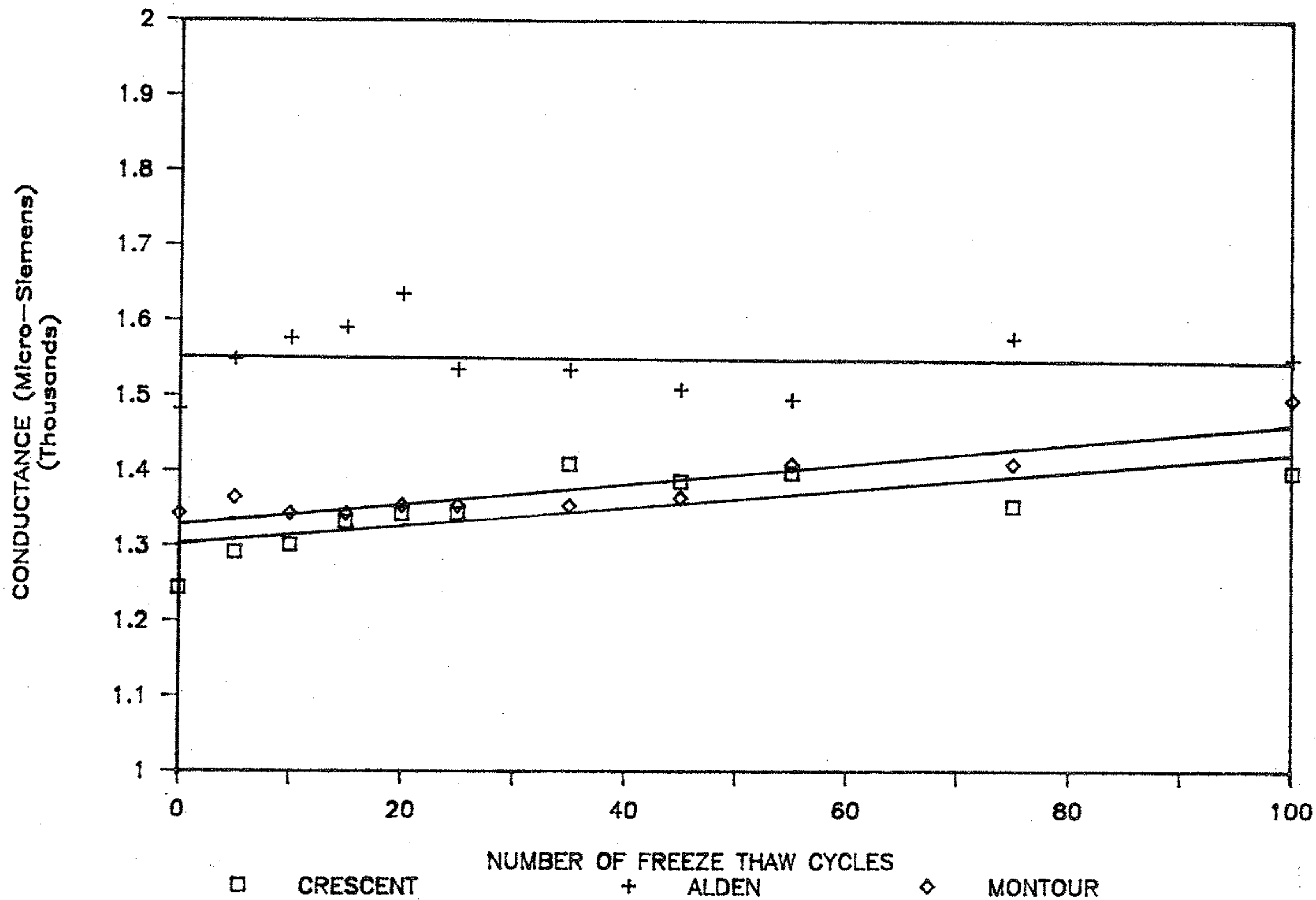


Figure 10. Variation of conductance with number of F/T cycles for air-entrained mixes (IDOT C3).

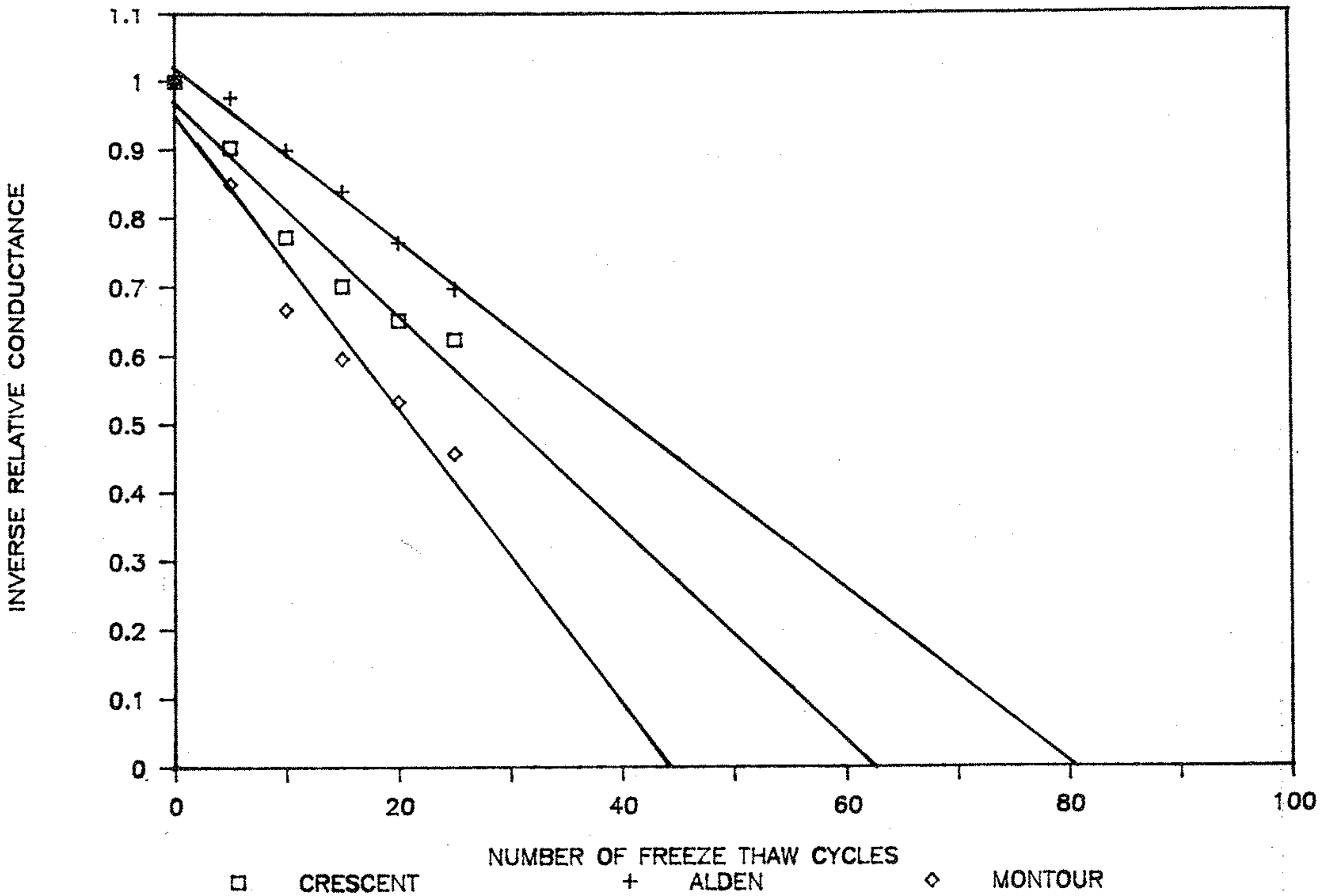


Figure 11. Inverse relative conductance versus number of F/T cycles for mixes (IDOT C3) without air entrainment.

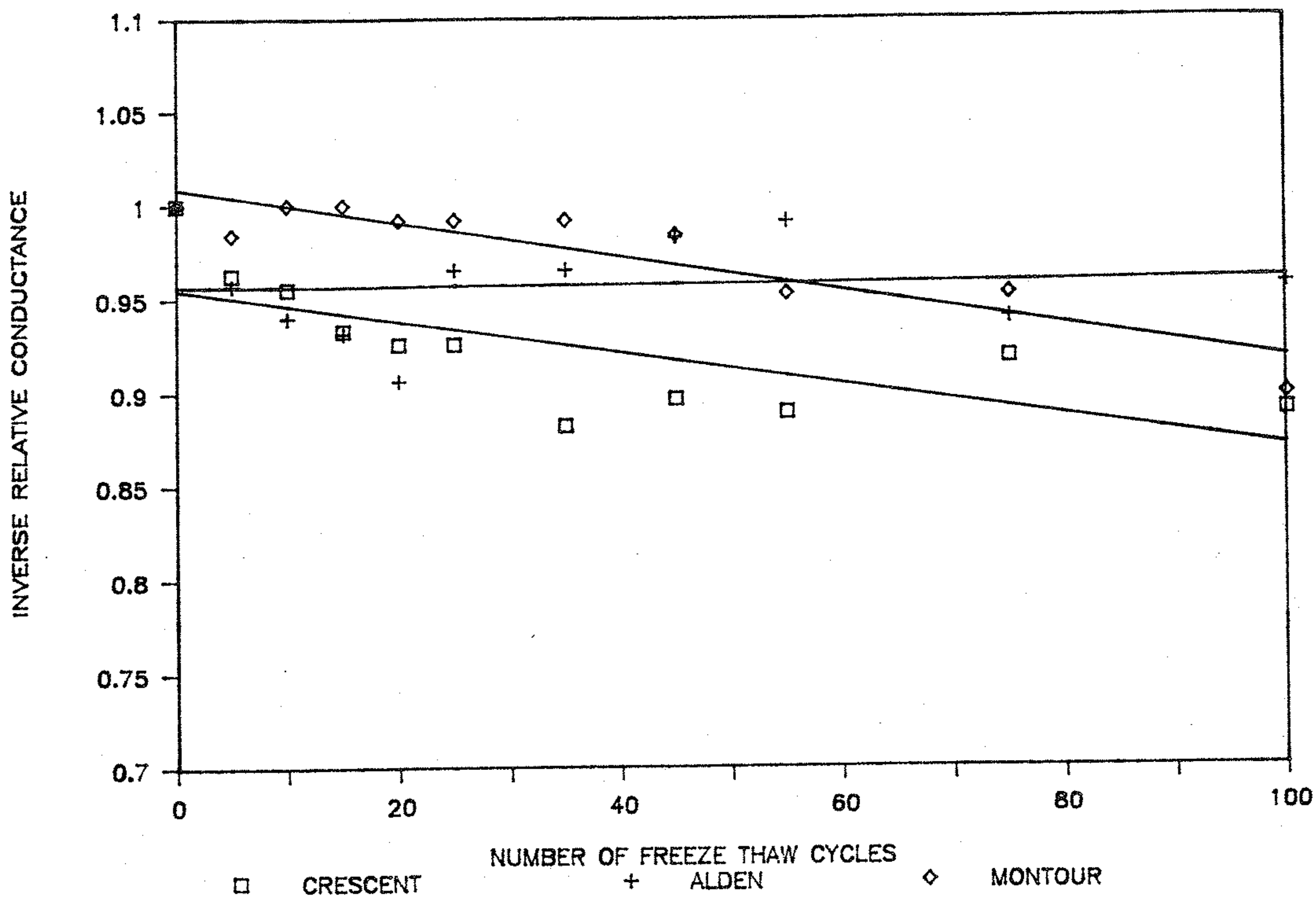


Figure 12. Inverse relative conductance versus number of F/T cycles for air-entrained mixes (IDOT C3).

relative conductance (IRC) is plotted against the number of freeze-thaw cycles for samples without and with entrained air, respectively. Again the lines in these graphs represent linear regressions of the data.

The conductometric phase transition porosimeter is capable of providing much more detailed information about the changes in the physicochemical state of a concrete subjected to repetitive freeze-thaw cycles than those provided in Figures 11 and 12. In Figure 13 the log of the conductance is plotted versus the negative inverse of the absolute temperature for the concrete cylinder containing the Montour aggregate and without entrained air. Plotted are two sets of curves. The curve represented by a solid line is that for the sample prior to any freeze-thaw cycling, while the curve represented by the dashed line is that for the sample after 25 freeze-thaw cycles. For both curves the upper branch represents data gathered during the cooling portion of the test and the lower branch represents data gathered during the warming portion of the test. Several observations can be made concerning the physicochemial changes which have occurred in this nondurable concrete. First, as previously observed the conductance at above freezing temperatures ($-1/T$ greater than -36.63×10^{-4}) has increased after being subjected to repetitive freezing and thawing. Secondly, at temperatures slightly below freezing ($-1/T$ slightly less than -36.63×10^{-4}) the curves are much steeper after the sample was subjected to repetitive freezing and thawing than they were prior to freezing and thawing. Finally, at lower temperatures ($-1/T$ much less than -36.63×10^{-4}) the conductance of the concrete after exposure to repetitive freezing and thawing is much less than prior to this exposure.

The methodology supporting conductometric phase transition porosimetry can be used to process data such as that presented in Figure 13 to give the observed conductometric changes some physical significance. The increase in total

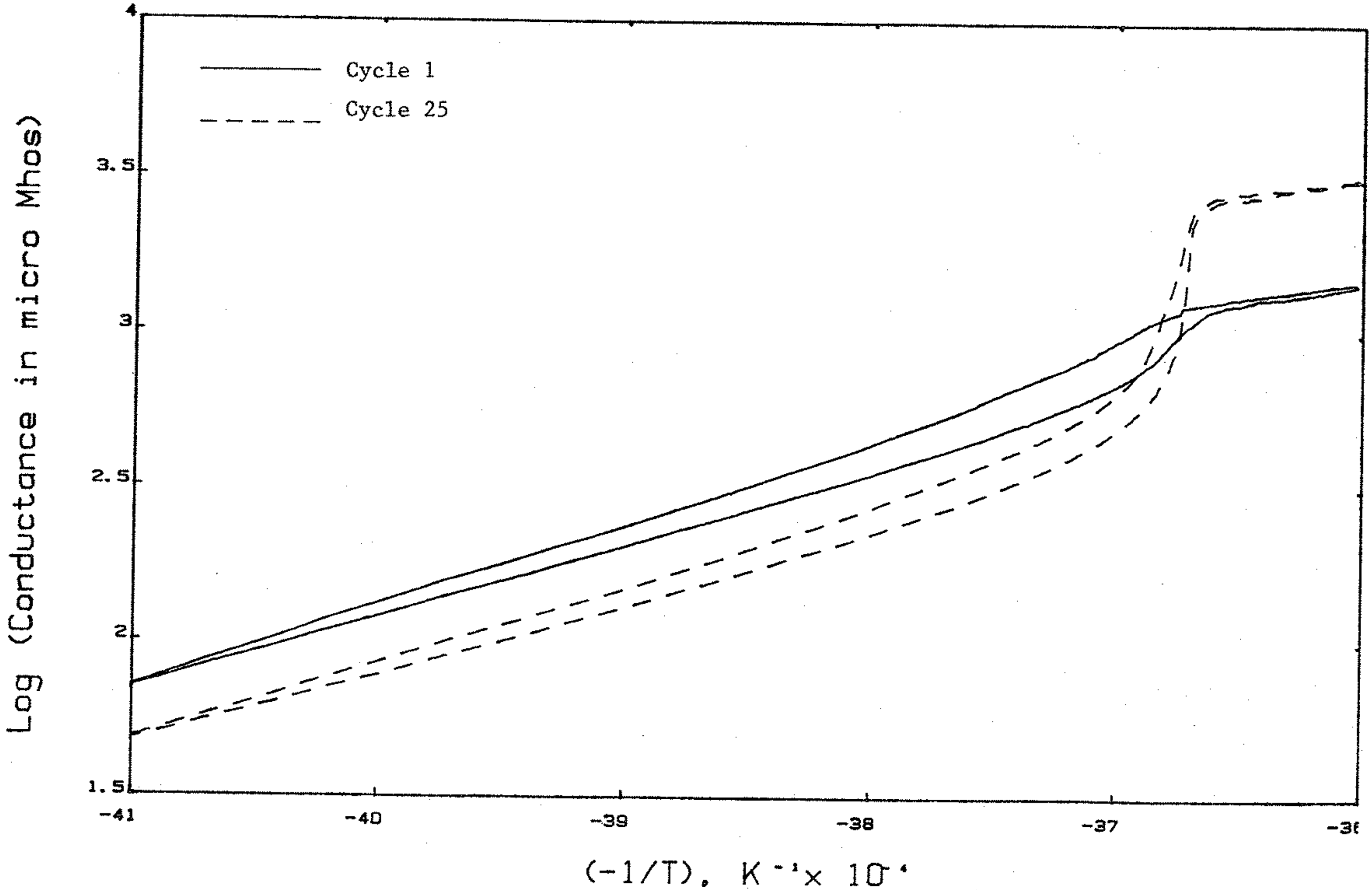


Figure 13. Variation of conductance with temperature for Montour aggregate mixes (IDOT C3) prepared without air entrainment.

conductance of the sample implies that the concrete is becoming more porous and that the degree of saturation is increasing. Or otherwise stated the conductive paths are increasing. Processing the data further allows the presentation of changes in pore size distribution illustrated in Figure 14. In this figure the cumulative relative pore volume is plotted against pore radius for the concrete at several points during the repetitive freeze-thaw exposure sequence. Points on these curves represent the fraction of the total pore volume containing pores smaller than the corresponding pore radius. From this plot the progressive nature of the failure is observable. As the concrete deteriorates the volume of small gel size (less than 100 angstrom radius) pores decreases while the volume of pores in the capillary size range (between 100 and 10,000 angstrom radius) increases. As it can be seen from Figure 14 for example, the volume of gel pores (volume of pores smaller than 100 angstroms) decreases from about 45% for cycle 1 to about 15% for cycle 25 while volume of all capillary pores (volume of pores having sizes between 100 and 10,000 angstroms) increases from 50% for cycle 1 to 80% for cycle 25. This behavior differs from the behavior of a durable concrete (mix containing the Alden aggregate and entrained air) as is illustrated in Figure 15. For this durable concrete, no significant change in the volume of gel pores is observable. The capillary size pores have increased somewhat in size but only in the range of the large capillaries without a systematic dependence on the number of F/T cycles. This would indicate that in the case of the nondurable concrete the portland cement paste has been subjected to a mechanical degradation and is no longer sound while in the durable concrete the portland cement gel structure remains intact.

Conductometric phase transition porosimetry can also be used to contrast the differences between the physicochemical changes which occur in concretes containing different aggregates. Figures 16 and 17 show the pore body size

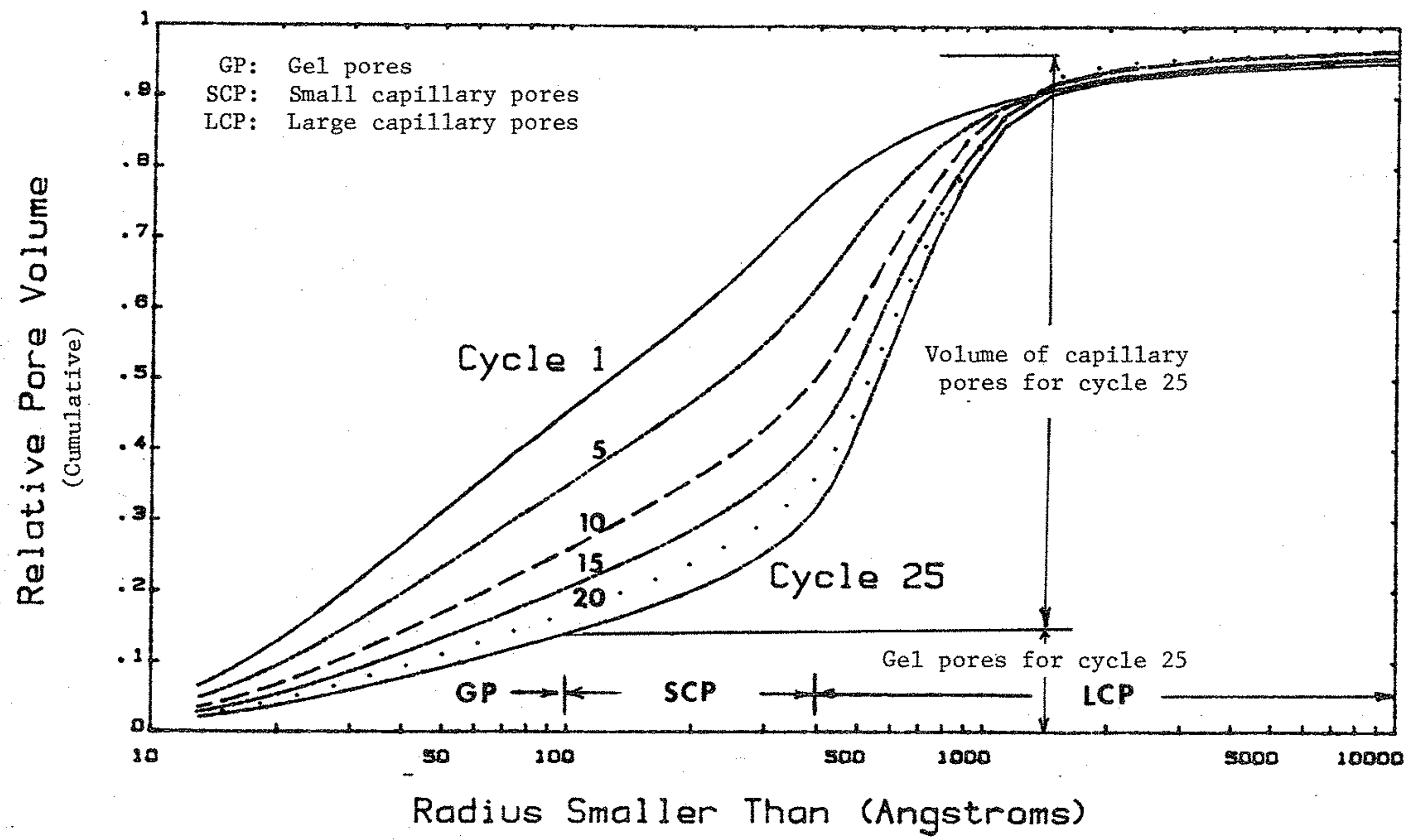


Figure 14. Effect of the number of F/T cycles on total volume distribution of sizes of body pores for the mix (IDOT C3) prepared using Montour aggregate without air entrainment.

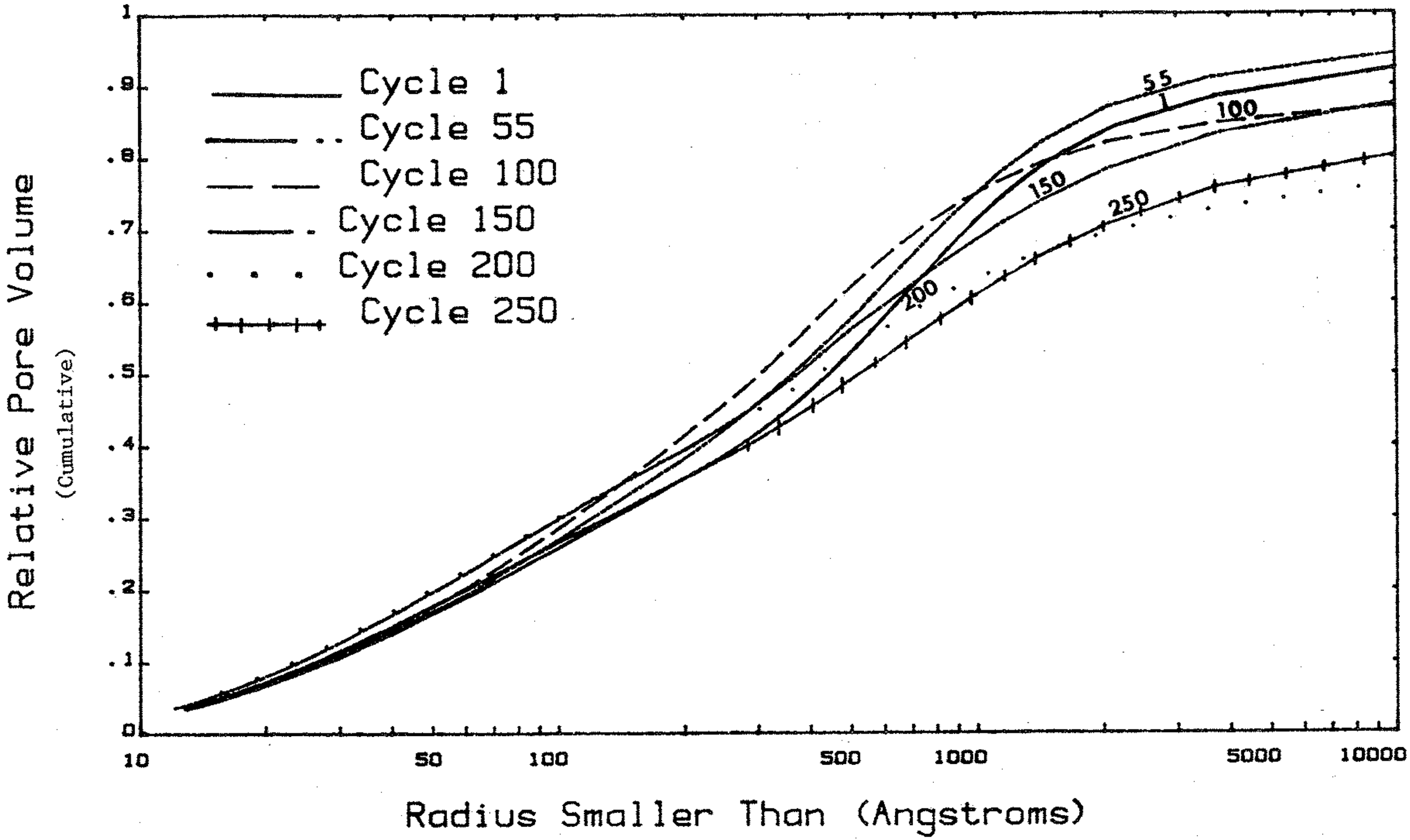


Figure 15. Effect of the number of F/T cycles on total volume distribution of sizes of body pores for the air-entrained mix prepared using Alden aggregate.

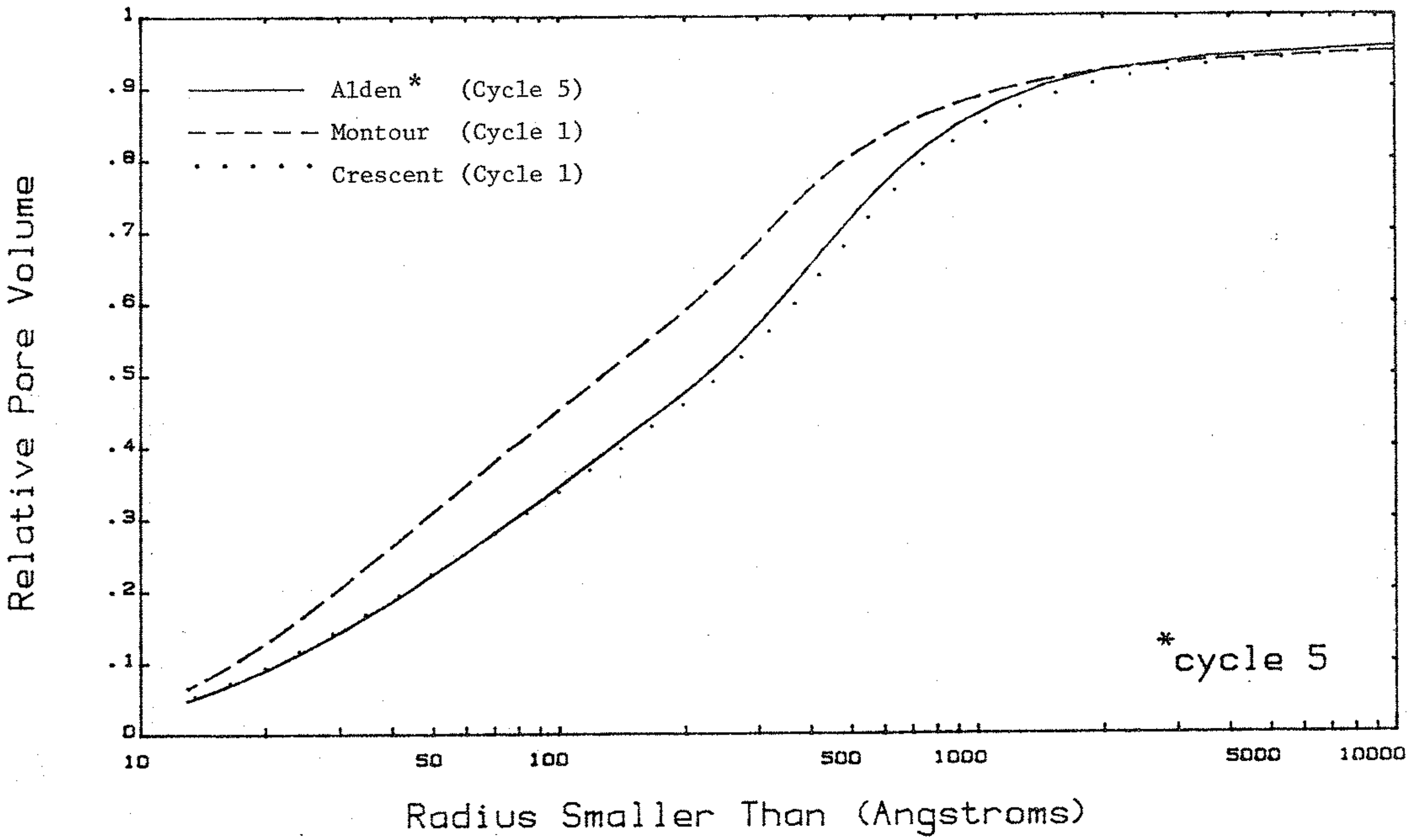


Figure 16. Total volume-size distribution of pore bodies prior to freezing and thawing for mixes prepared without air entrainment.

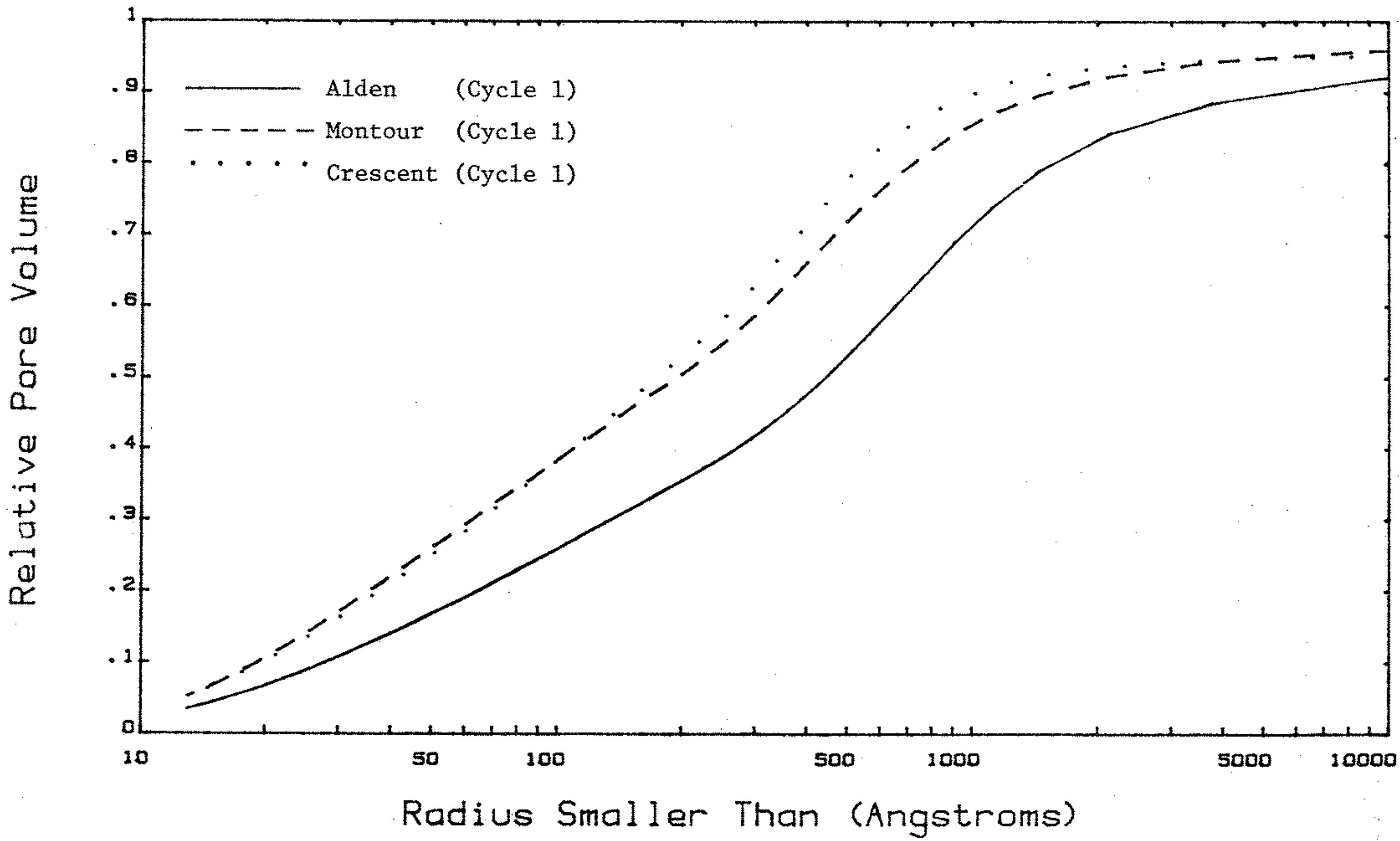


Figure 17. Total volume-size distribution of pore bodies prior to freezing and thawing for air-entrained mixes.

distributions measured prior to any freeze-thaw cycling for mixes from which concrete cylinders were cast and tested. Figure 16 contrasts distributions for mixes without any entrained air while Figure 17 contrasts distributions for mixes with entrained air. There are no significant differences between the distributions for the mixes containing the Crescent aggregate with and without air, the Alden aggregate without air and the Montour aggregate with air. The mix containing the Montour aggregate without air has a somewhat smaller initial size distribution while the mix containing the Alden aggregate with air has a somewhat larger distribution. It is not discernable from the limited data collected whether or not these differences are signficant or not. However, the changes in the physicochemical state observed for the cylinder cast from concrete containing the Alden aggregate with air were less than those observed for any of the other tested cylinders and the changes in the physicochemical state observed for the cylinder cast from concrete containing the Montour aggregate without air were greater than those observed for any of the other tested cylinders. Therefore, it is possible that continued investigations may allow researchers to imply something about differences in the durability of the concretes containing different coarse aggregates from examinations of initial pore size distributions determined in this manner. However, these differences may also be due to sampling errors resulting from inhomogeneities of concrete between the electrodes.

Since electrical conductance behavior of concrete should be largely controlled by the physicochemical state of cement matrix such differences, if not due to inhomogeneity, should reflect the effect of aggregate on the cement matrix, such as absorptivity or reactivity of the aggregate. Further studies in this area will be carried out under our recent NSF project.

It is significant to note that the pore size distribution of the cylinders with entrained air does not vary as much from those cylinders without entrained air as may be expected. This is not too surprising because the cylinders were saturated by only immersing them in water for an extended period of time. Therefore, pore size distribution determined by the conductometric method is exclusive of entrained air pores. If it were possible to saturate the entrained air voids in this manner they would not be effective in protecting the concrete from damage due to freeze-thaw action. Recalling, however, that the overall conductance of concrete containing the Alden aggregate increases with the addition of entrained air while for concretes containing the other two coarse aggregates it remains constant or decreases slightly and that the concrete containing the Alden aggregate had a somewhat larger pore size distribution than the other concretes may suggest a fundamental difference exists between how the aggregate paste and air voids interact in concretes containing different aggregates.

While the importance of differences in initial pore size distributions is somewhat speculative, interpretation of the changes which occur in pore size distributions as a concrete is subjected to repetitive freezing and thawing is more conclusive. The progressive changes which occur in the pore size distribution of a deteriorating concrete were discussed earlier and illustrative examples of the changes which occur in durable (air-entrained containing Alden aggregate) and nondurable (non air-entrained containing Montour aggregate) concretes were contrasted. These same changes, namely deterioration of the gel pore system, are observable for concrete with entrained air but containing nondurable coarse aggregates. In Figures 18 and 19 the first derivative of the relative pore volume with respect to the log of the pore radius (first derivative of Figures 14 and 15, respectively) is plotted against pore radius.

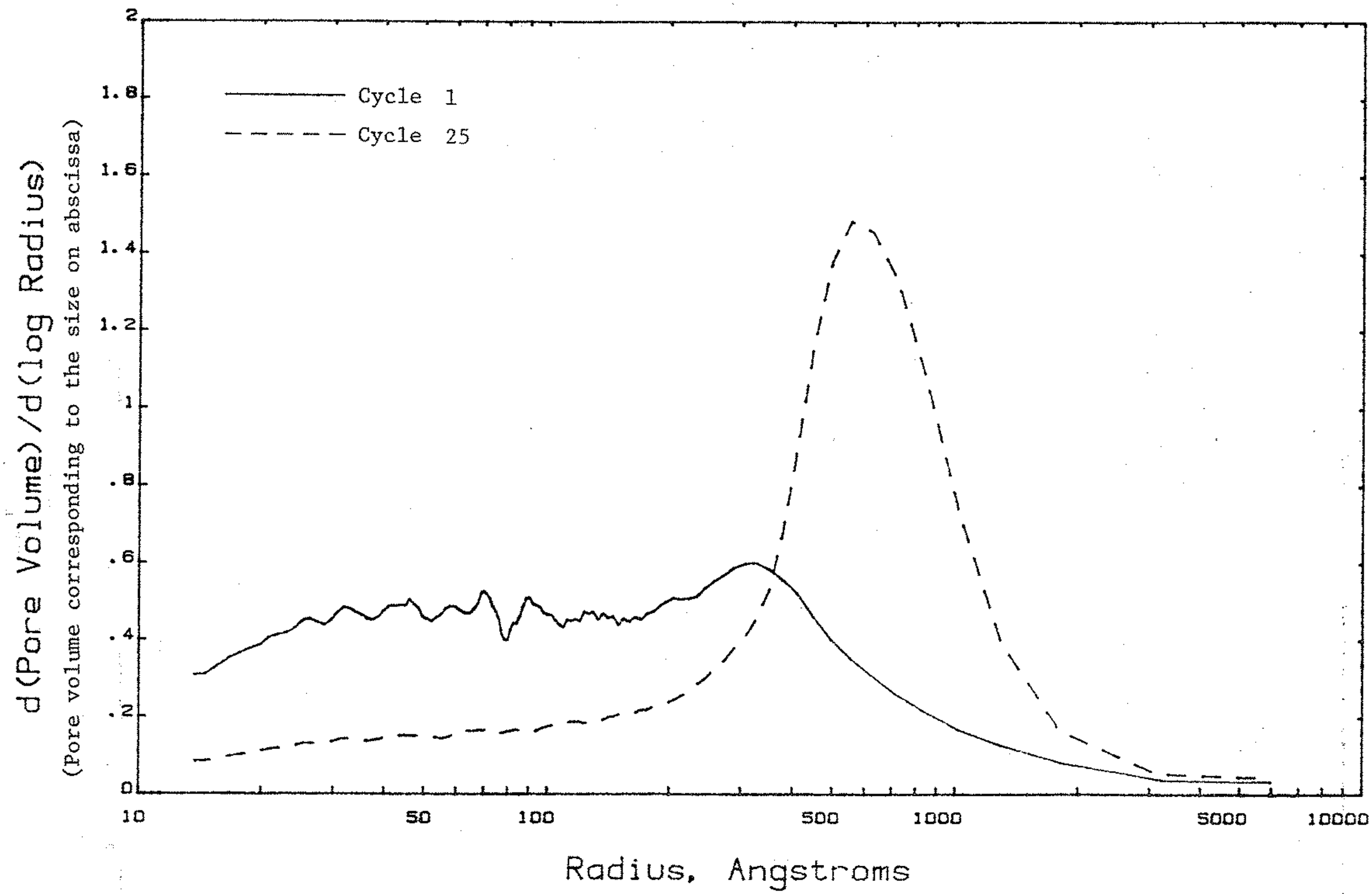


Figure 18. Variation of true pore size distribution of mix made with Montour aggregate without entrained air with F/T cycles.

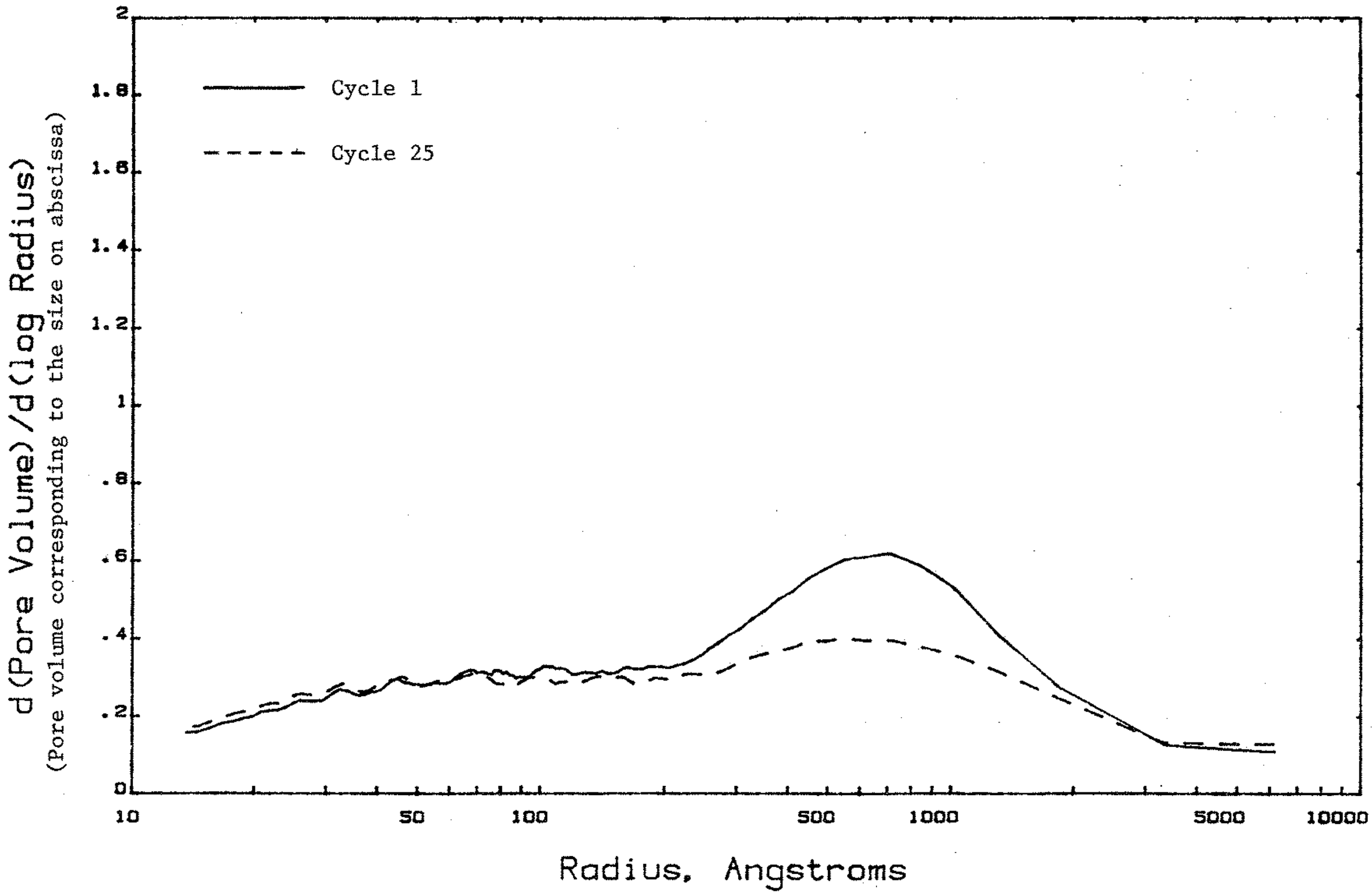


Figure 19. Variation of true pore size distribution of air-entrained mix made with Alden aggregate with F/T cycles.

Figure 18 represents behavior of a typical nondurable concrete while Figure 19 represents behavior of a durable concrete. Note that for the durable concrete the size distribution remains unchanged for pores smaller than aproximately 200 angstroms. Figures 20 and 21 give comparable information for air entrained mixes containing the Crescent and Montour aggregates. In both these plots some degradation of the small gel pores is observable. It would appear that the degradation is more extensive in the case of the Crescent aggregate. Also, the modal peak in the larger capillary pore range is less pronounced for the Montour aggregate. This would indicate that the extent of damage is less for the concrete containing the Montour aggregate. These observations provide some additional information not apparent from monitoring the conductance of the concrete at above freezing temperatures alone. The rate of change of conductance with number of freeze-thaw cycles for these two concretes was the same (see Figure 12) and the inverse relative conductance for the concrete containing the Montour aggregate was also nearly the same for both mixes.

SUMMARY AND CONCLUSIONS

During this research project several significant research advances have been made towards the development of superior test methods for evaluating the durability of portland cement concrete.

Monitoring the changes in the conductance of samples of portland cement concrete at above freezing temperatures provides an index of the concretes durability. Evaluation of concrete durability from conductance measurements can be used as a supplement to or replacement for more traditional measurements such as fundamental transverse frequency and pulse velocity. Measurements of electrical conductance could be easily incorporated into a completely automated

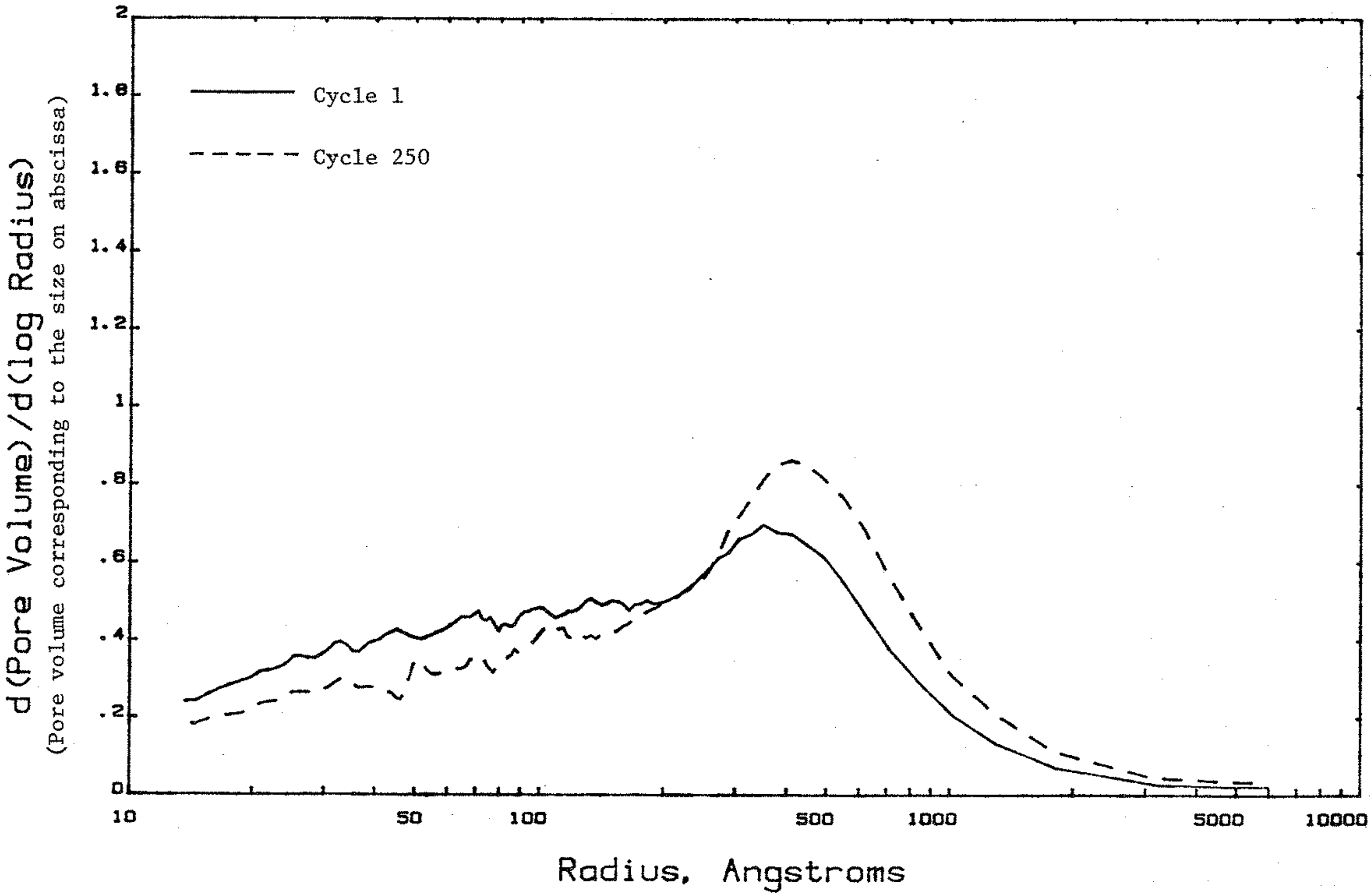


Figure 20. Variation of true pore size distribution of air-entrained mix made with Crescent aggregate with F/T cycles.

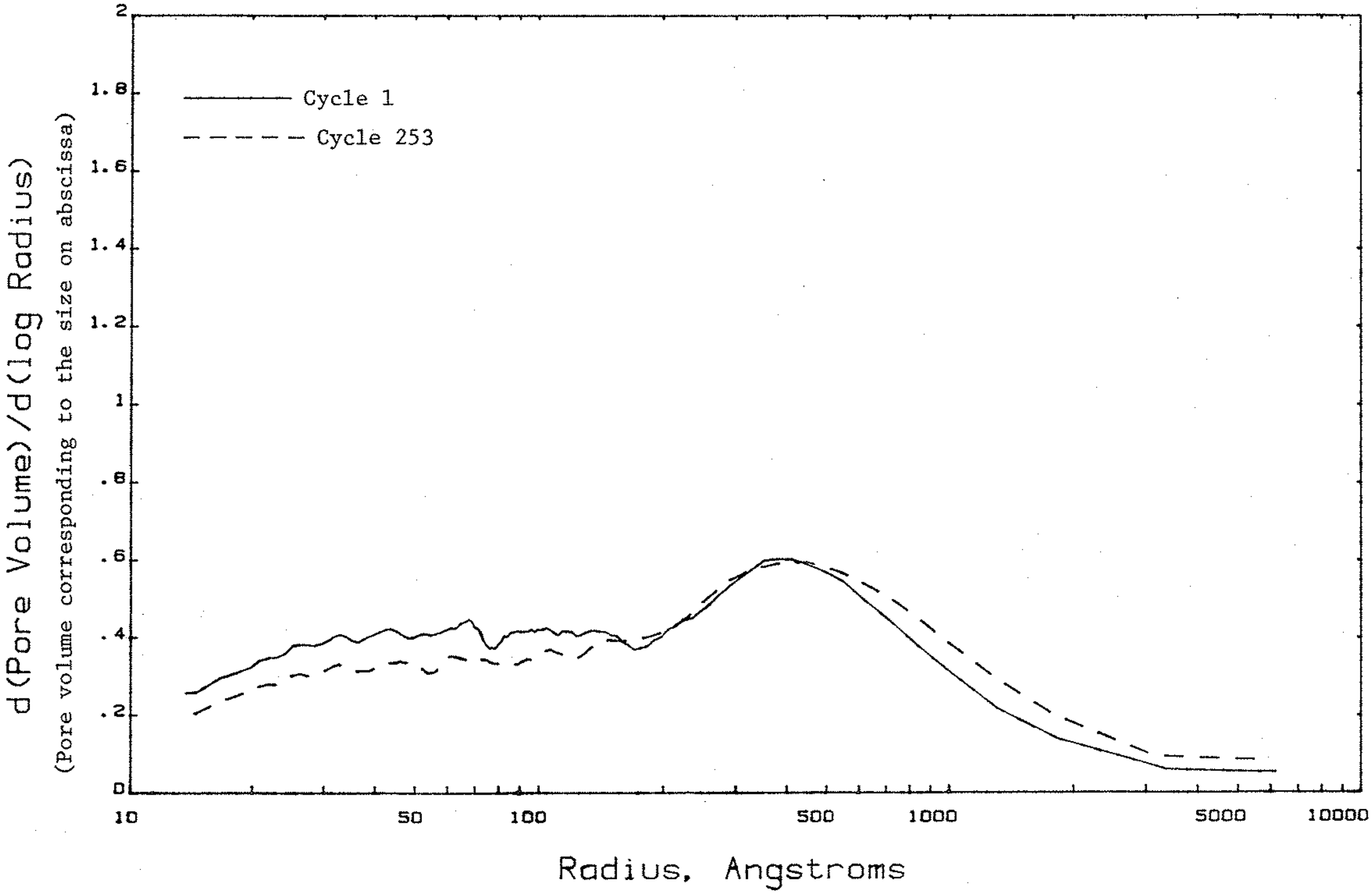


Figure 21. Variation of true pore size distribution of air-entrained mix made with Montour aggregate with F/T cycles.

repetitive freeze-thaw testing apparatus eliminating the need for removal of samples from the cabinet and manually taken measurements. These types of measurements are time intensive and increase both the cost and length of this type of test. For such an automated system measurements could easily be taken at the end of each cycle. In addition, such a system could also measure the conductance of a concrete sample at a temperature corresponding to the freezing point of pores having a radius equal to approximately 100 angstroms (approximately -5°C). The conductance of a nondurable concrete at this temperature will decrease as the concrete is exposed to repetitive freezing and thawing. This is in contrast to the conductance of the concrete at above freezing temperatures which for a nondurable concrete will increase as the concrete deteriorates.

A new method for determining pore size distributions of saturated porous materials has been developed. Based on this newly developed methodology, a conductometric phase transition porosimeter was constructed. The porosimeter is capable of determining the pore size distributions for a broad range of pore radii ranging from 12 to 5000 angstroms. The porosimeter has been used to determine the pore size distributions of portland cement concretes and monitor the changes in the distributions as the concretes are subjected to repetitive freeze-thaw cycles. This information provides a fingerprint of the changing physicochemical state of the concrete that is not available using other analytical techniques.

Conductometric porosimetry has shown that for a concrete susceptible to freeze-thaw deterioration capillary pores grow at the expense of gel pores. An early detection of this behavior during freeze-thaw cycles may help to identify nondurable concrete mixes in a much shorter time than required by the dynamic modulus method presently in use.

RECOMMENDATIONS

In order to make the transition from research to application it will be necessary to expand the data base of concretes tested by these newly developed conductometric methods. This will be necessary to establish design limits for the parameter inverse relative conductance (IRC) such as those in use for durability factors. This will be possible only as more concretes are tested in this manner. Also, it is suggested that the undertaking of such a task include concretes containing the same component materials as evaluated during this project. Mixes for these tests should be prepared at a constant slump. Researchers at Iowa State have begun to take conductance measurements along with weight and pulse velocity measurements of concretes being subjected to repetitive freezing and thawing as a part of other research activities. This will expand the current data base of information for this type of testing.

The pore size distributions of larger numbers of concrete samples need to be determined using the conductometric phase transition porosimeter. This is necessary to evaluate the amount of sampling error present due to current sample preparation techniques and to make improvements in these techniques that reduce errors if it is appropriate. These may include larger sample sizes and different electrode configurations.

Conductometric testing can also be used to determine rate and ultimate degree of saturation of concrete exposed to the ingress of water. It appears from our investigations during the last six years that there is a critical level of the free water in concrete at which a concrete mix becomes nondurable. The sooner the water content reaches this level the more susceptible a concrete is to freezing and thawing damage.

Collaboration between the Iowa State Civil Engineering Materials research team and the Iowa DOT researchers has been very productive. Continuation and furtherance of this collaboration will help Iowa further establish its leadership in transportation materials research and testing.

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Gunnink, B. W.; Schlorholtz, S. M.; Bergeson, K.; Enustun, B. V.; and Demirel, T.; Development of a Conductometric Test for Frost Resistance of Concrete, Annual Report Phase I, Iowa DOT Project HR-272, 1985.

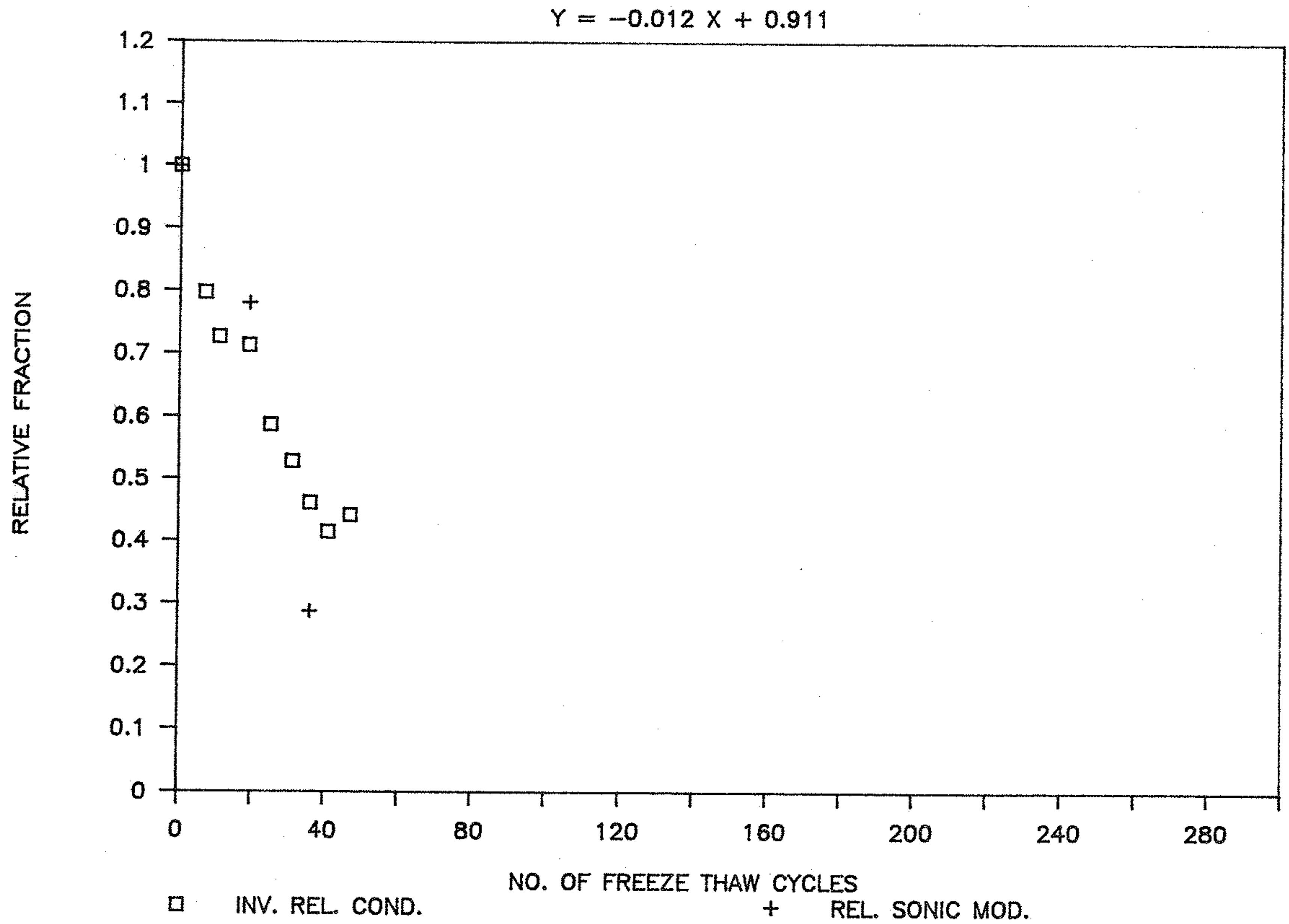
APPENDIX A

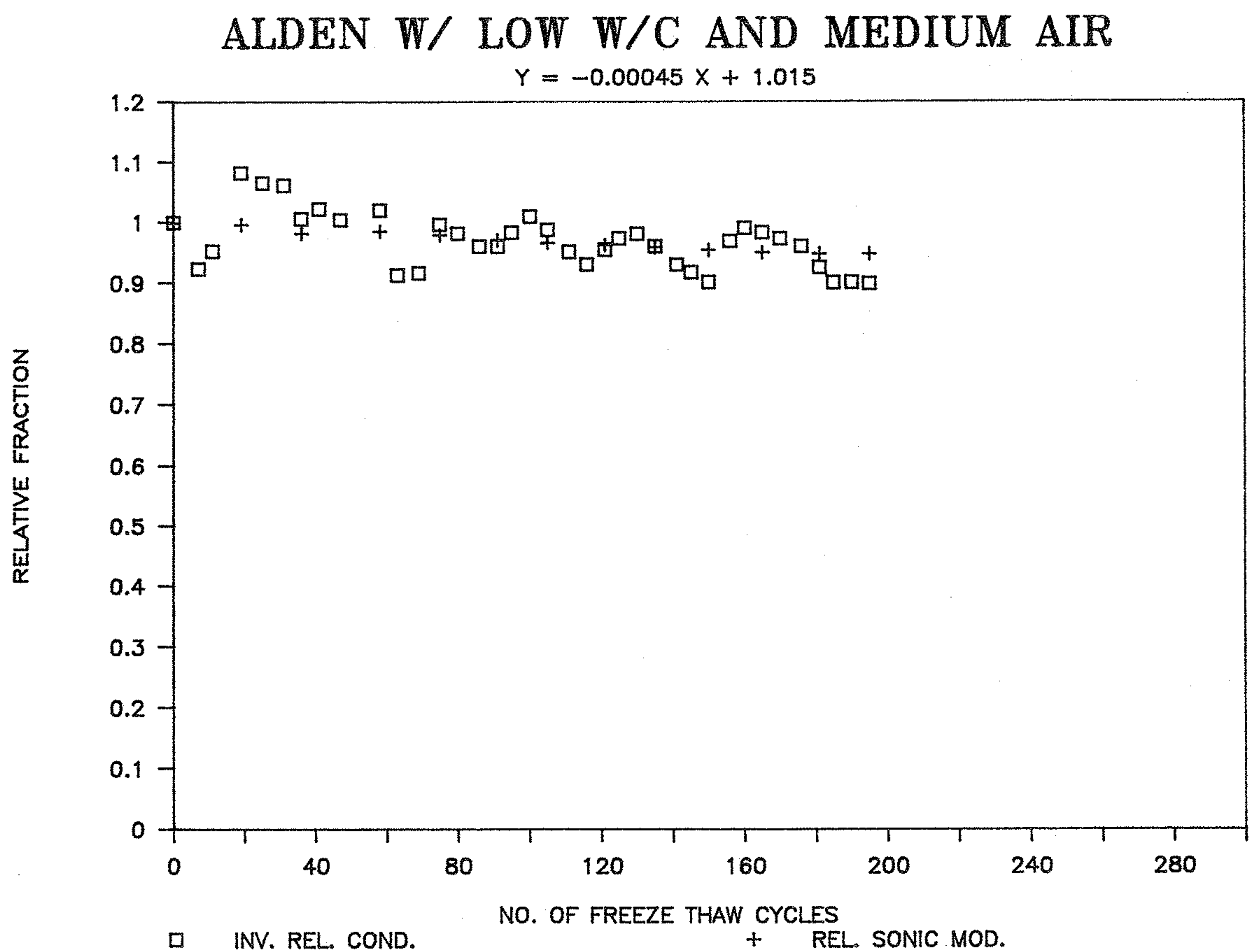
Graphs of conductance data obtained from concrete beams subjected to rapid freezing and thawing

APPENDIX A

Graphs of conductance data obtained from concrete beams subjected to rapid freezing and thawing

ALDEN W/ LOW W/C AND LOW AIR

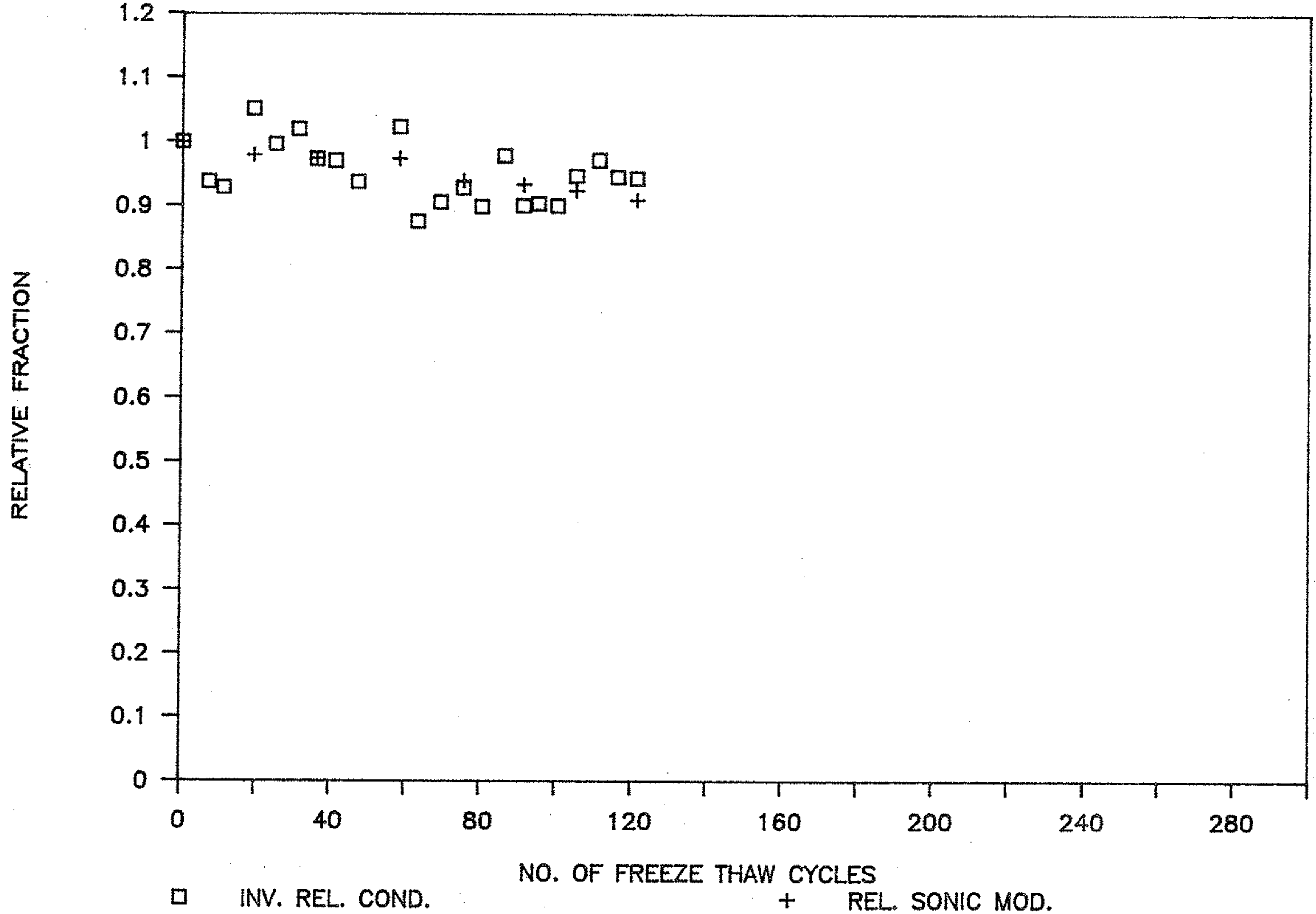


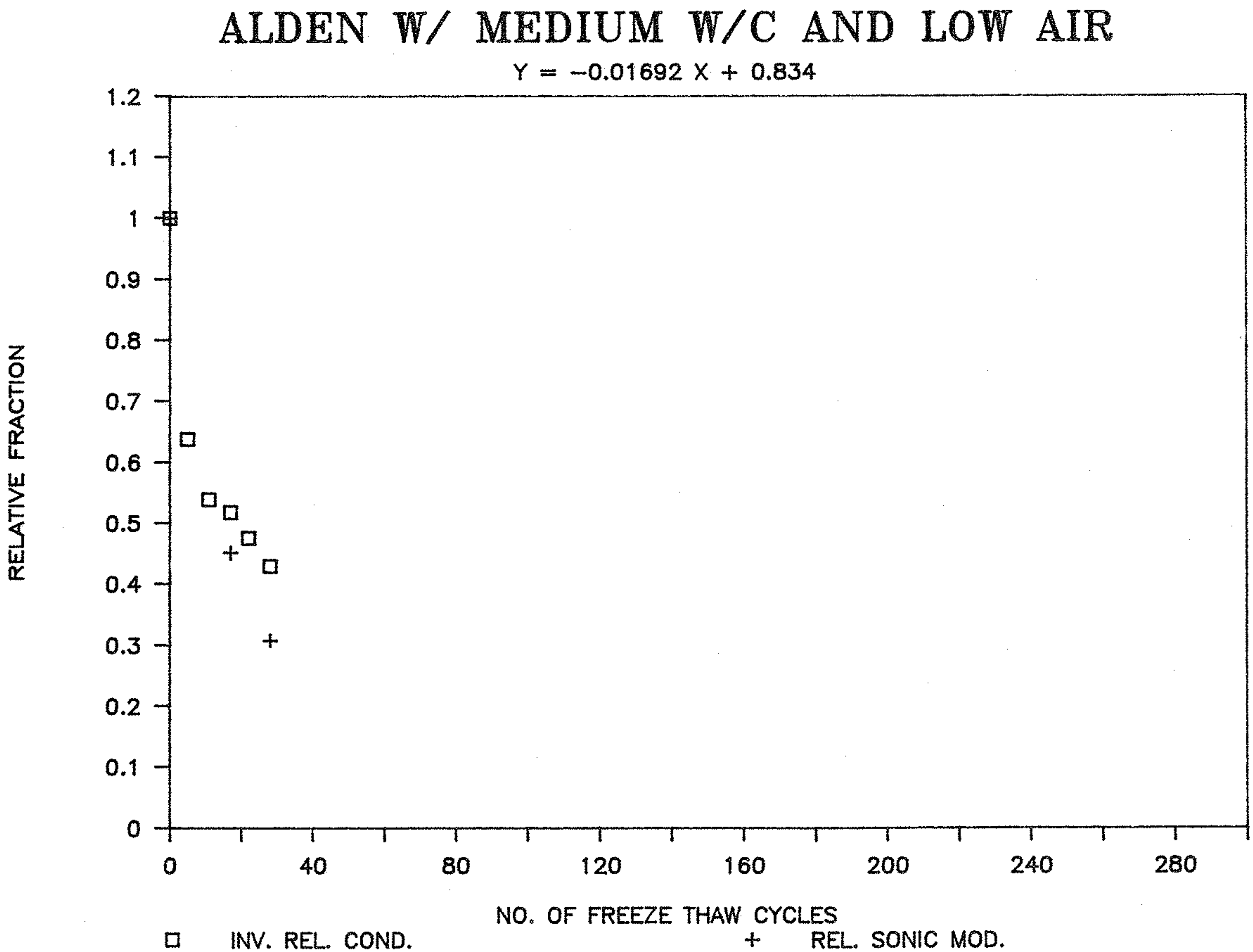

ALDEN W/ LOW W/C AND MEDIUM AIR
Y = −0.00045 X + 1.015
RELATIVE FRACTION
1.2
1.1
1
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0
0
40
80
120
160
200
240
280
NO. OF FREEZE THAW CYCLES
INV. REL. COND.
REL. SONIC MOD.

2

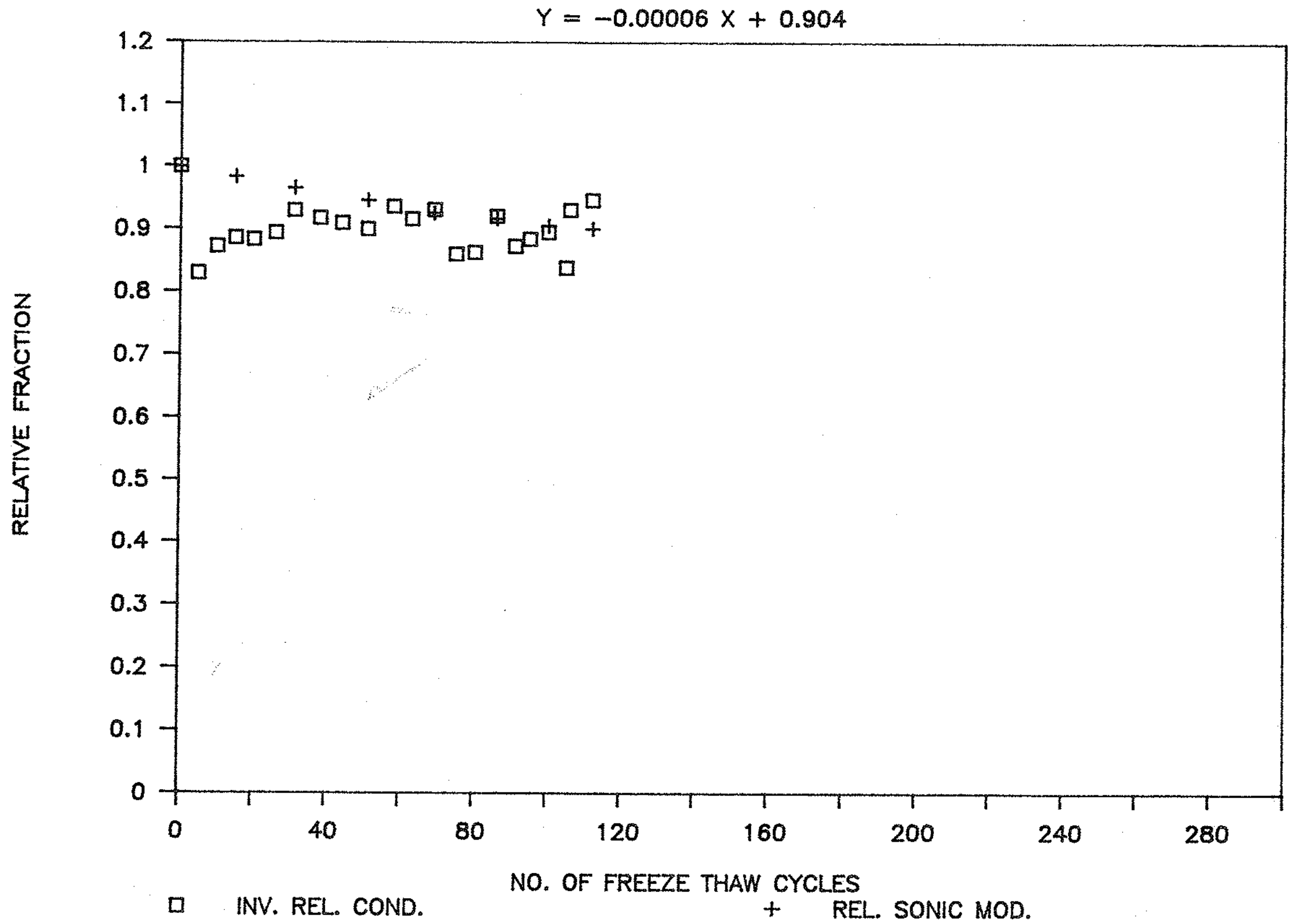
ALDEN W/ LOW W/C AND HIGH AIR

$Y = -0.00053 X + 0.986$

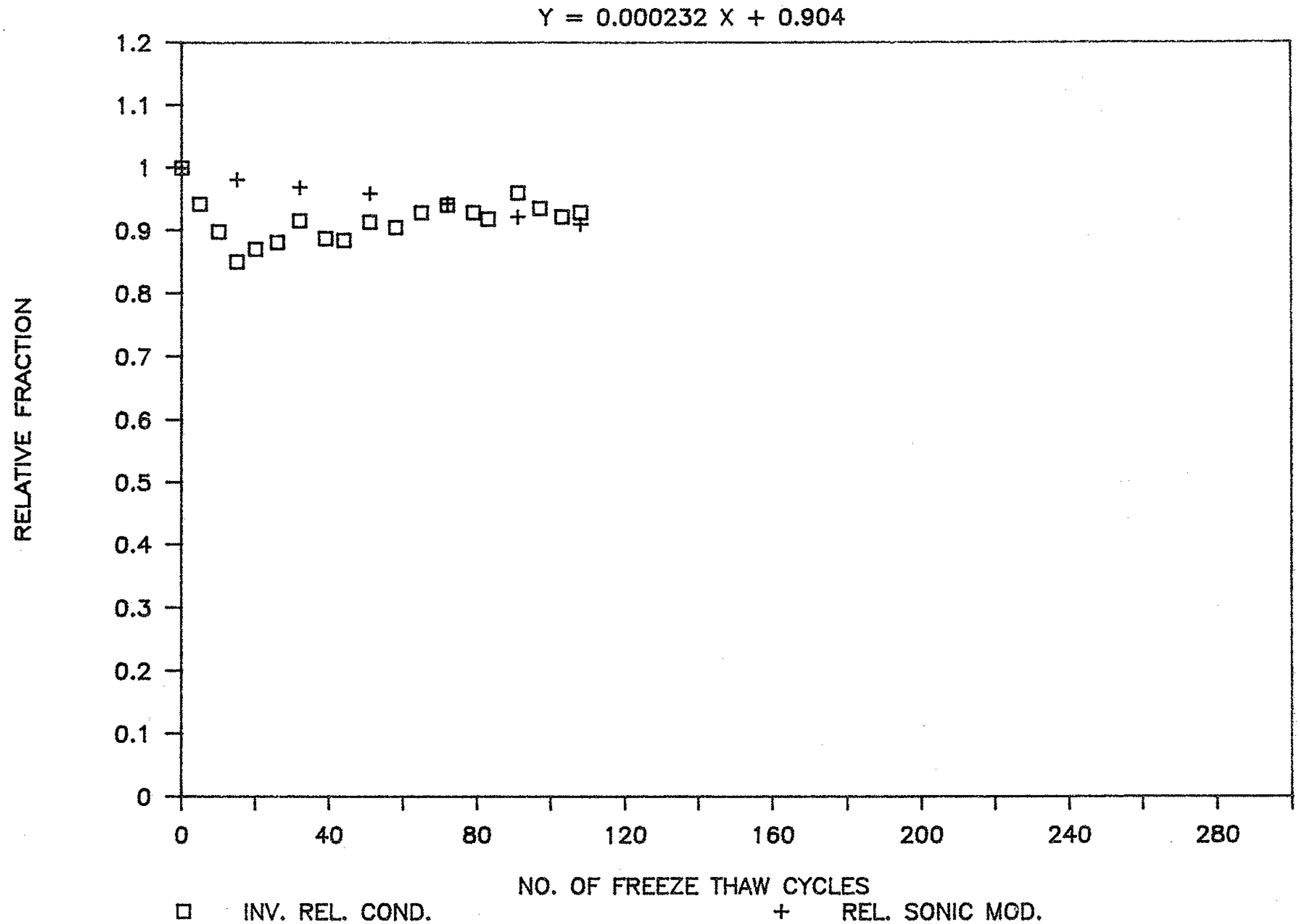


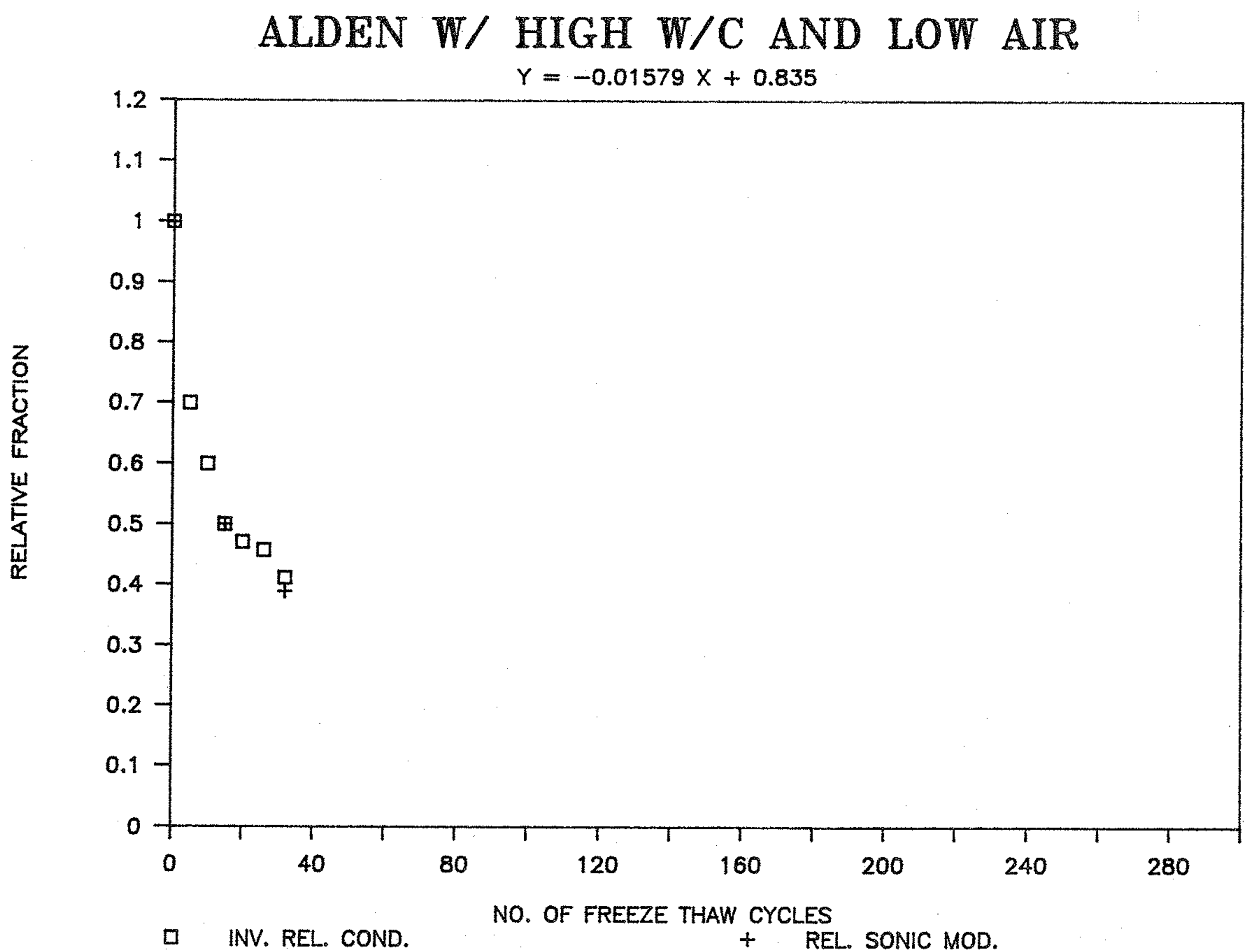

ALDEN W/ MEDIUM W/C AND LOW AIR
Y = −0.01692 X + 0.834
RELATIVE FRACTION
1.2
1.1
1
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0
0
40
80
120
160
200
240
280
NO. OF FREEZE THAW CYCLES
□ INV. REL. COND.
+ REL. SONIC MOD.

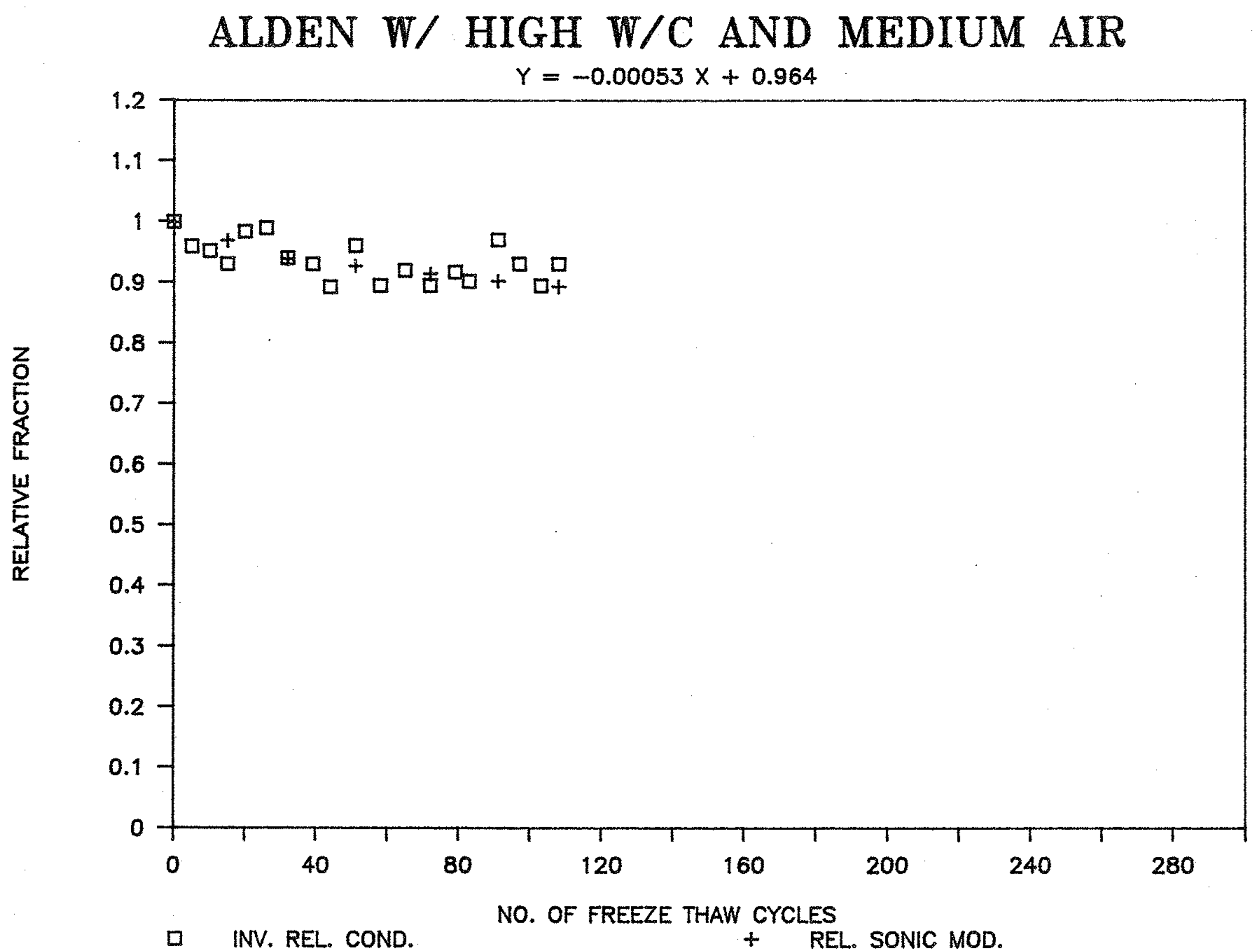
ALDEN W/ MEDIUM W/C AND MEDIUM AIR

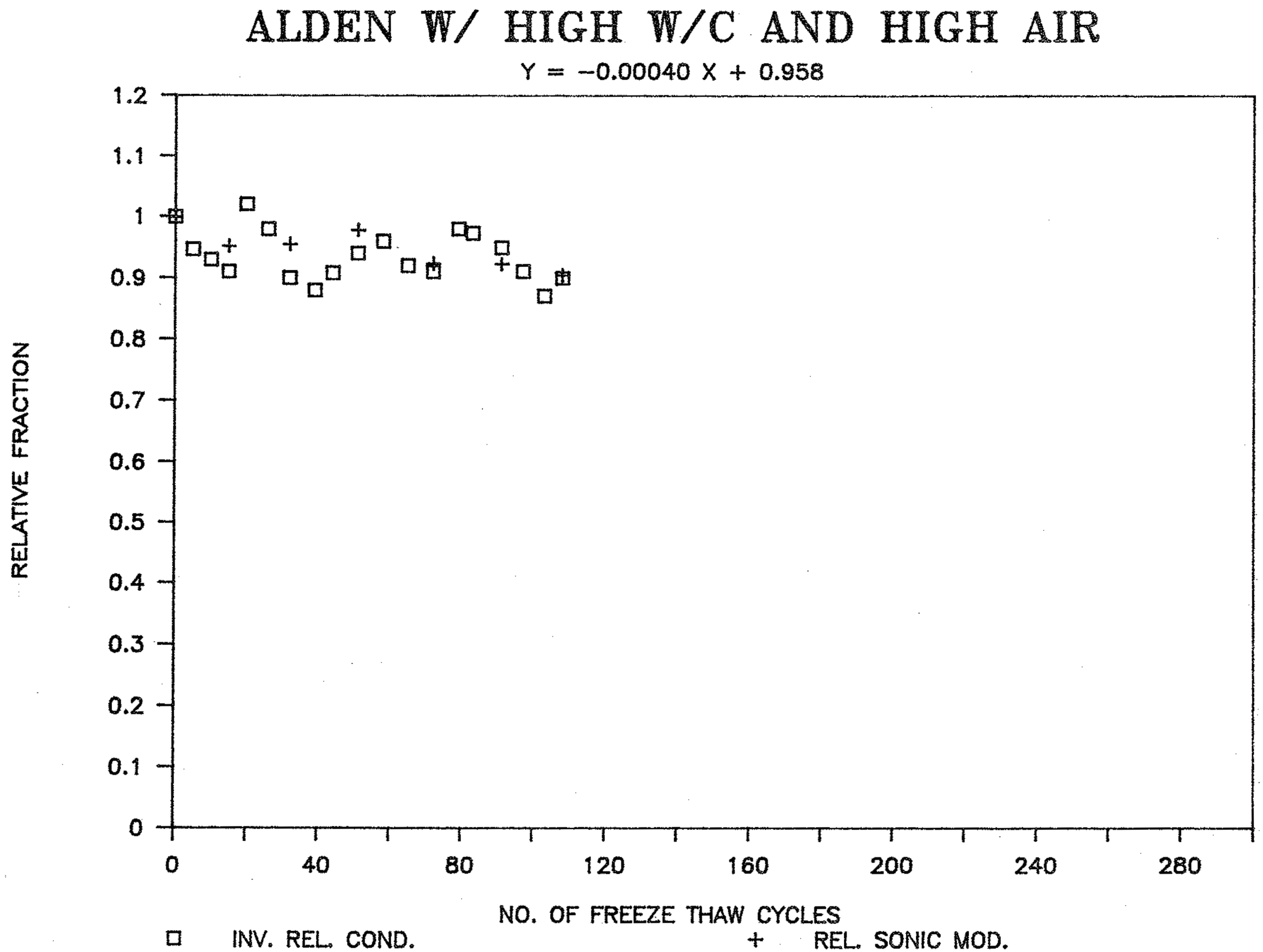


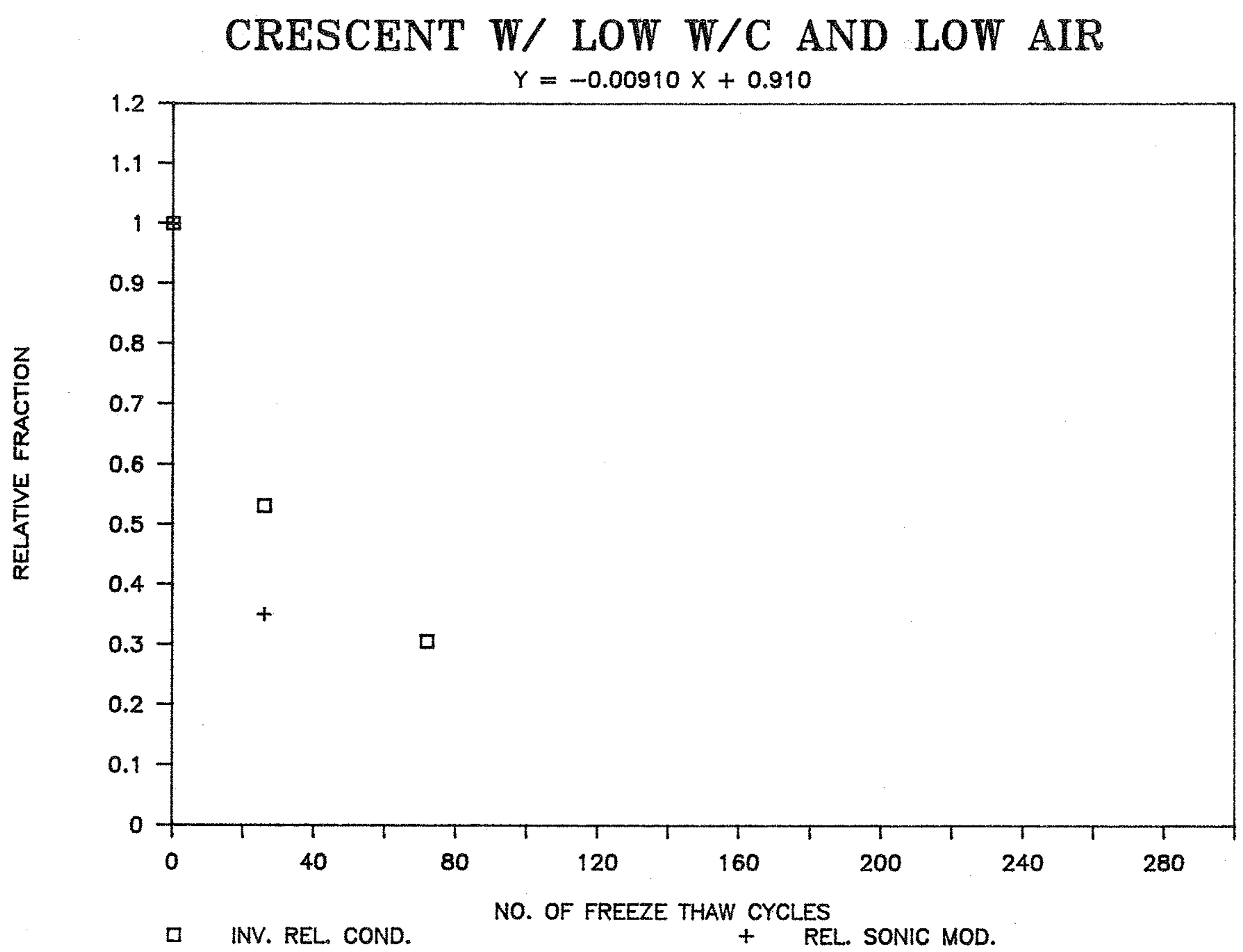
ALDEN W/ MEDIUM W/C AND HIGH AIR




ALDEN W/ HIGH W/C AND LOW AIR
Y = −0.01579 X + 0.835
RELATIVE FRACTION
1.2
1.1
1
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0
0
40
80
120
160
200
240
280
NO. OF FREEZE THAW CYCLES
INV. REL. COND.
REL. SONIC MOD.

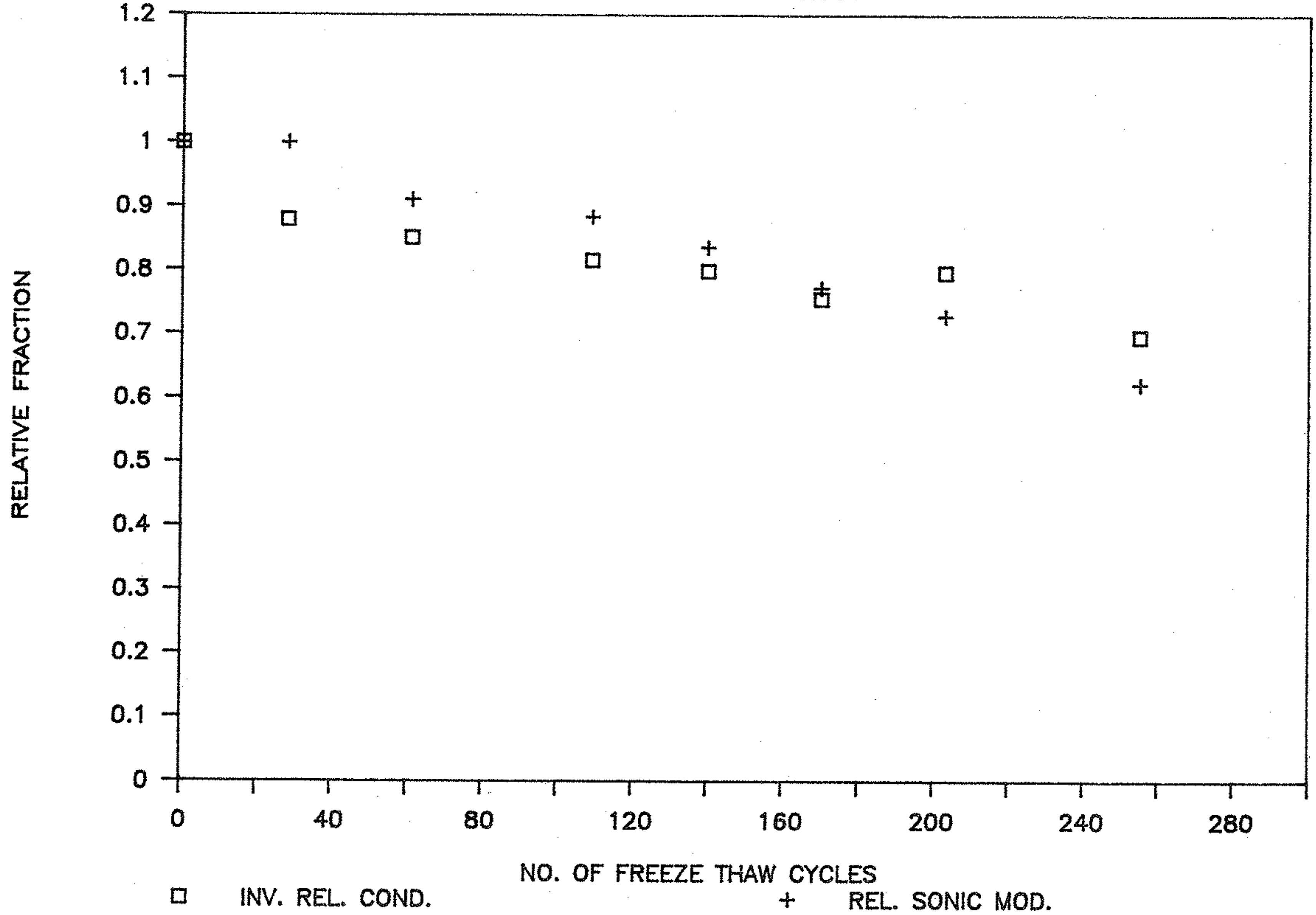

ALDEN W/ HIGH W/C AND MEDIUM AIR
Y = −0.00053 X + 0.964
RELATIVE FRACTION
1.2
1.1
1
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0
0
40
80
120
160
200
240
280
NO. OF FREEZE THAW CYCLES
INV. REL. COND.
REL. SONIC MOD.

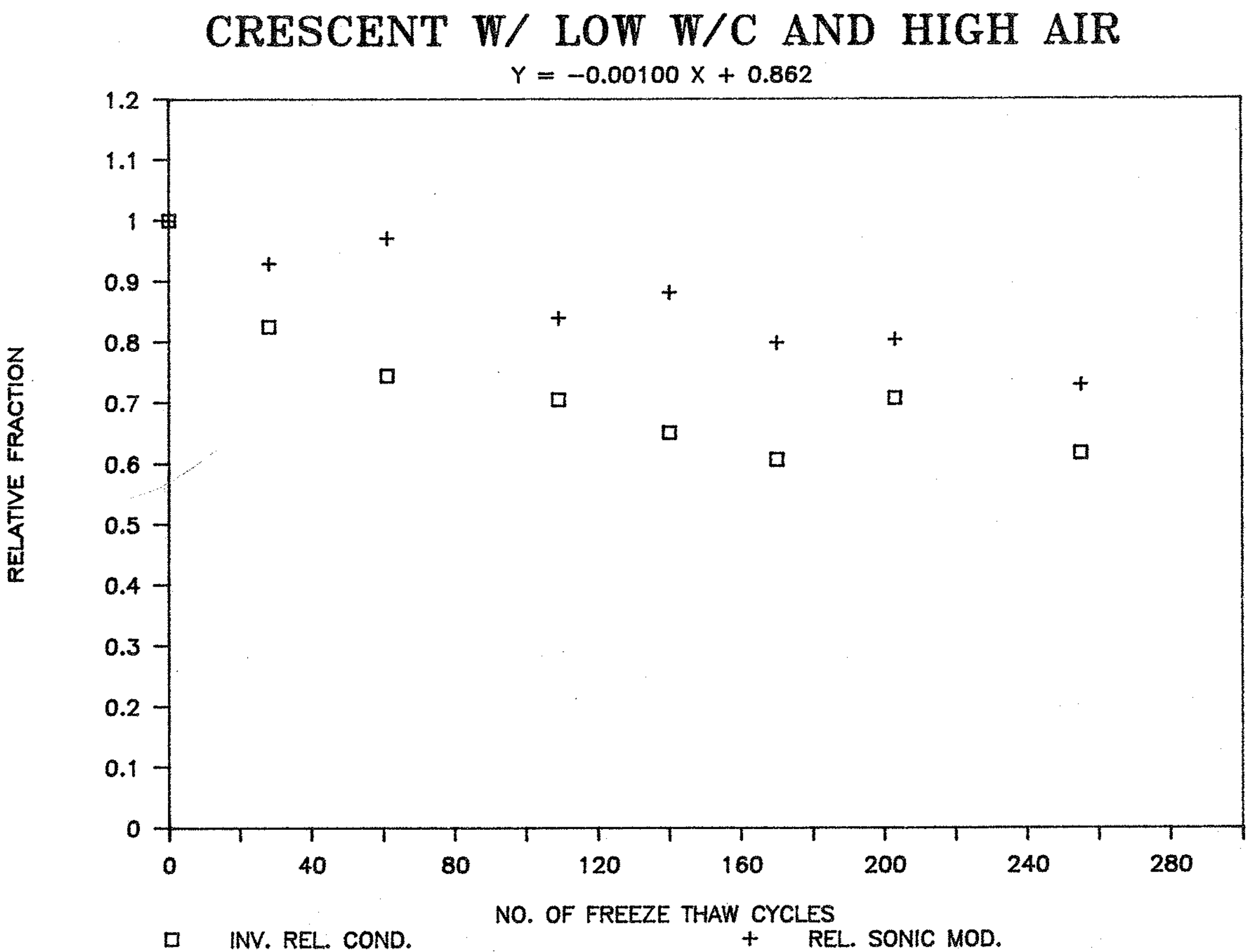

ALDEN W/ HIGH W/C AND HIGH AIR
Y = −0.00040 X + 0.958
RELATIVE FRACTION
1.2
1.1
1
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0
0
40
80
120
160
200
240
280
NO. OF FREEZE THAW CYCLES
□ INV. REL. COND.
+ REL. SONIC MOD.


CRESCENT W/ LOW W/C AND LOW AIR
Y = -0.00910 X + 0.910
RELATIVE FRACTION
1.2
1.1
1
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0
0
40
80
120
160
200
240
280
NO. OF FREEZE THAW CYCLES
INV. REL. COND.
REL. SONIC MOD.

CRESCENT W/ LOW W/C AND MEDIUM AIR

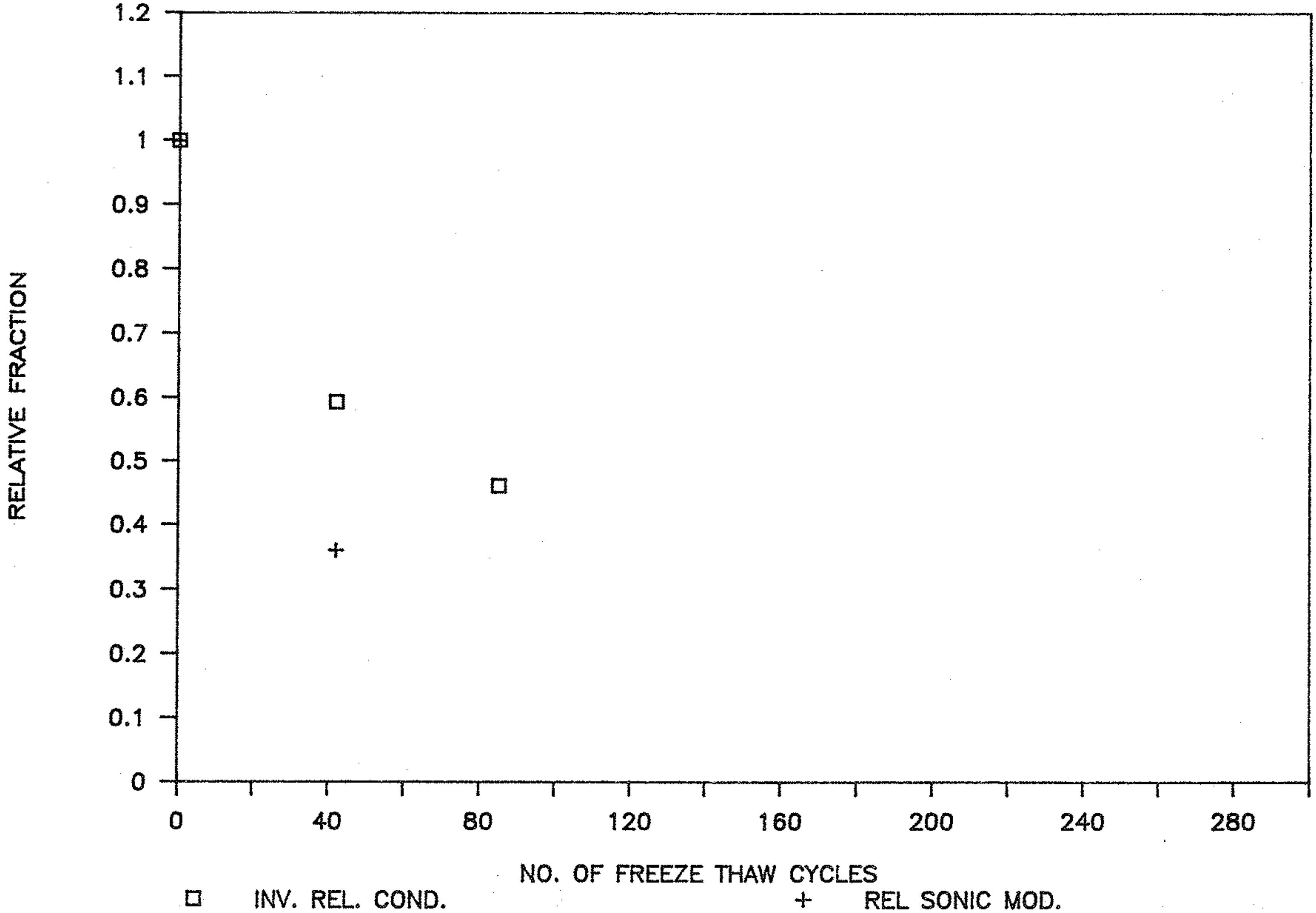
Y = −0.00086 X + 0.931



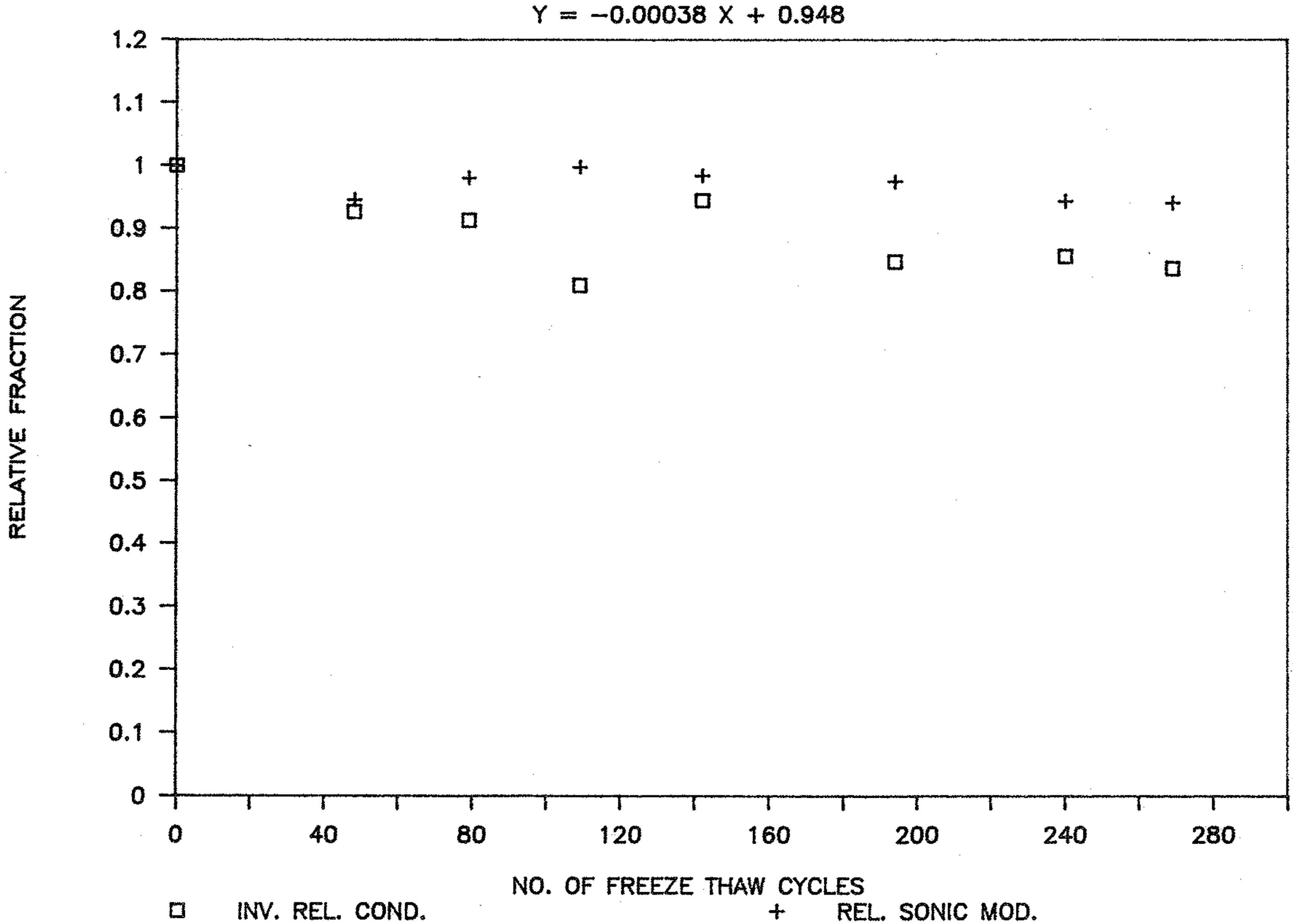

CRESCENT W/ LOW W/C AND HIGH AIR
Y = -0.00100 X + 0.862
RELATIVE FRACTION
1.2
1.1
1
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0
0
40
80
120
160
200
240
280
NO. OF FREEZE THAW CYCLES
INV. REL. COND.
REL. SONIC MOD.

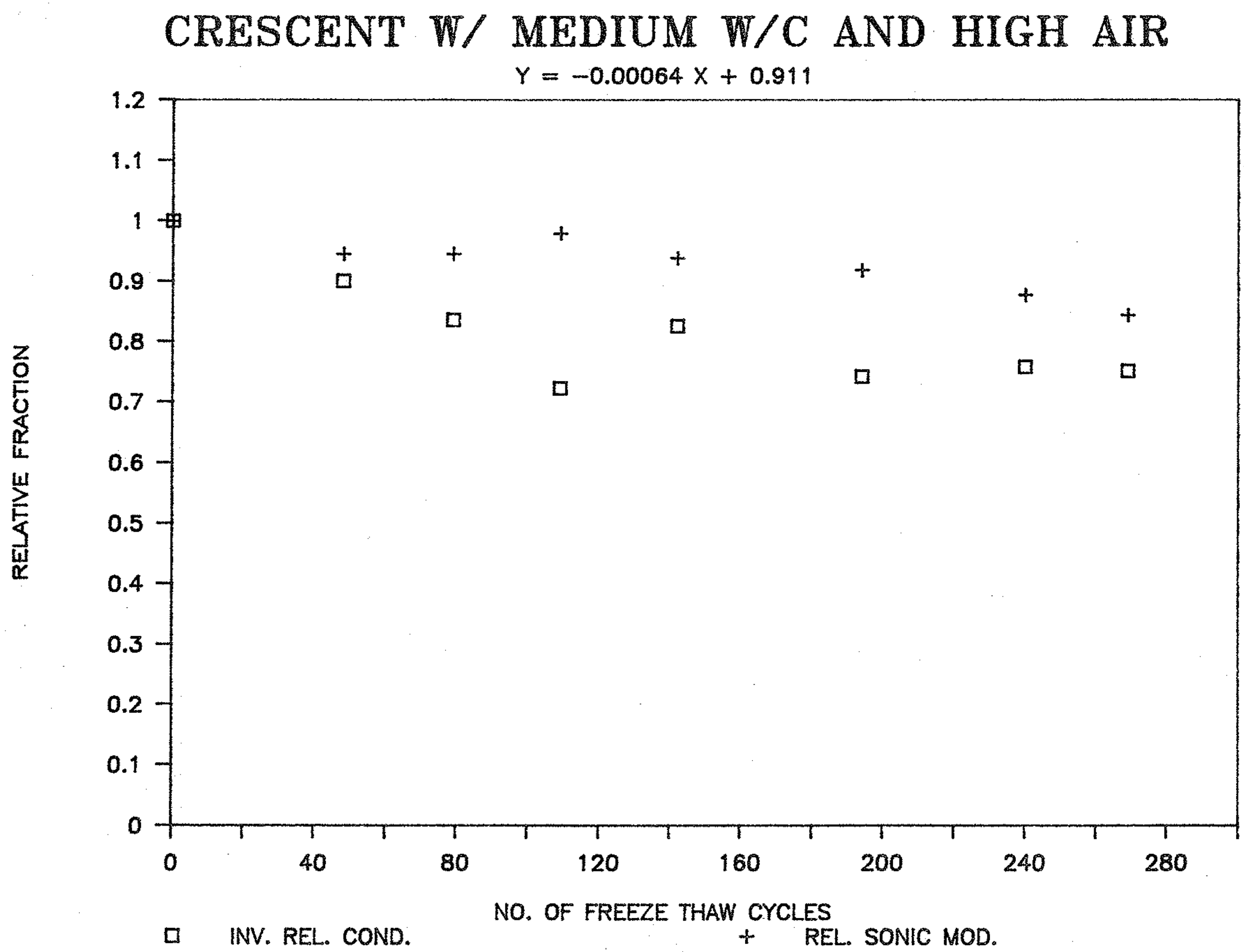
CRESCENT W/ MEDIUM W/C AND LOW AIR

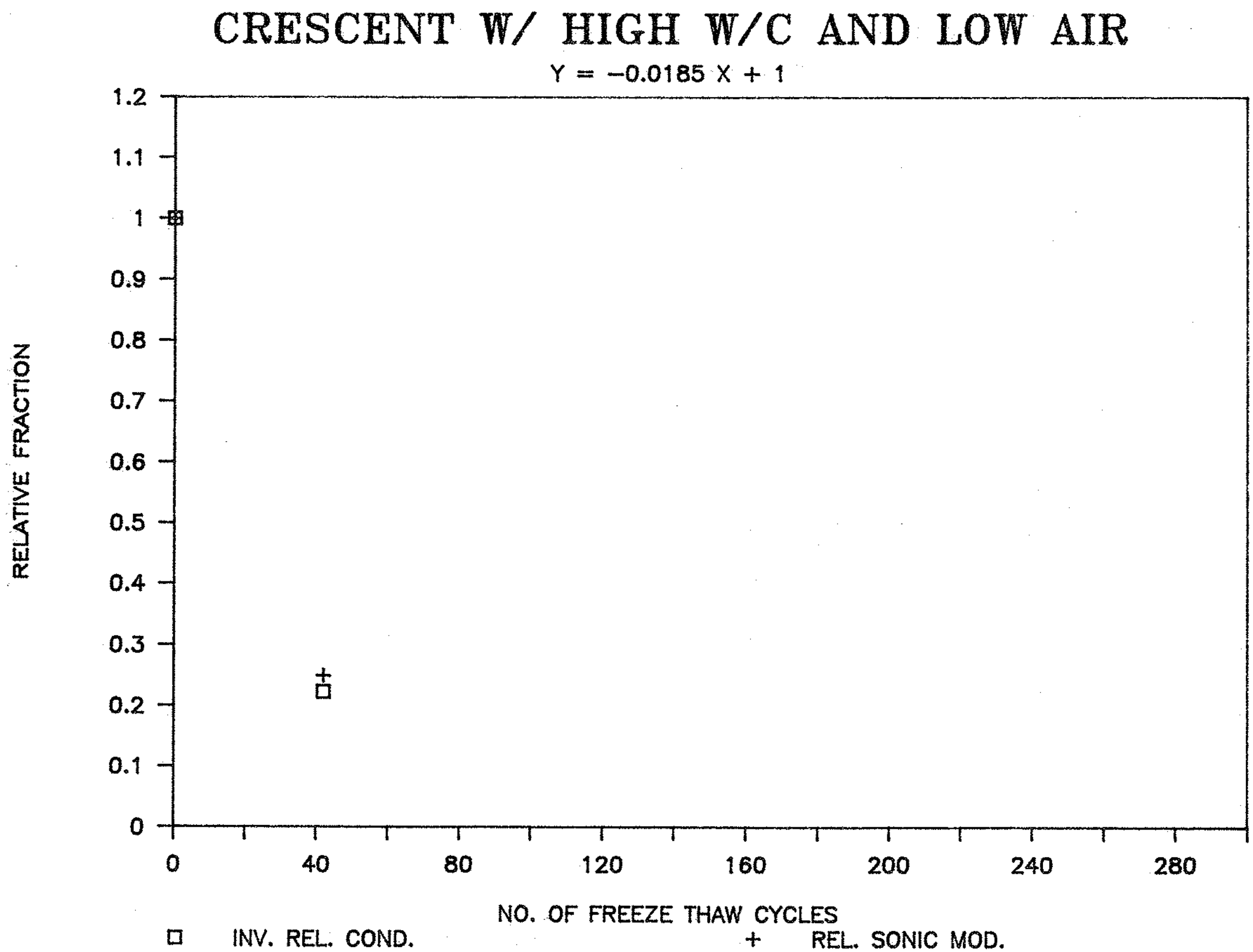
Y = −0.00631 X + 0.952



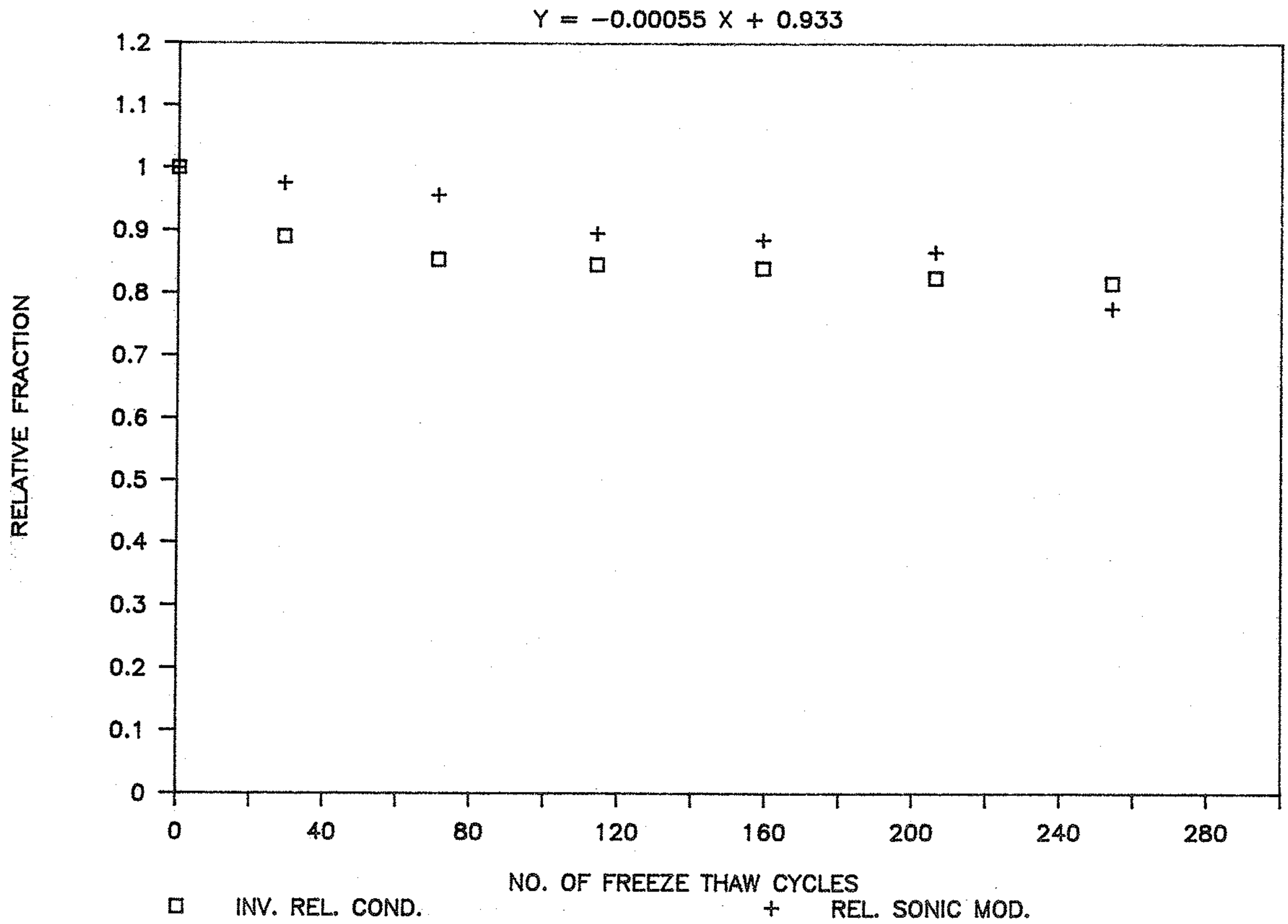
CRESCENT W/ MEDIUM W/C AND MEDIUM AIR

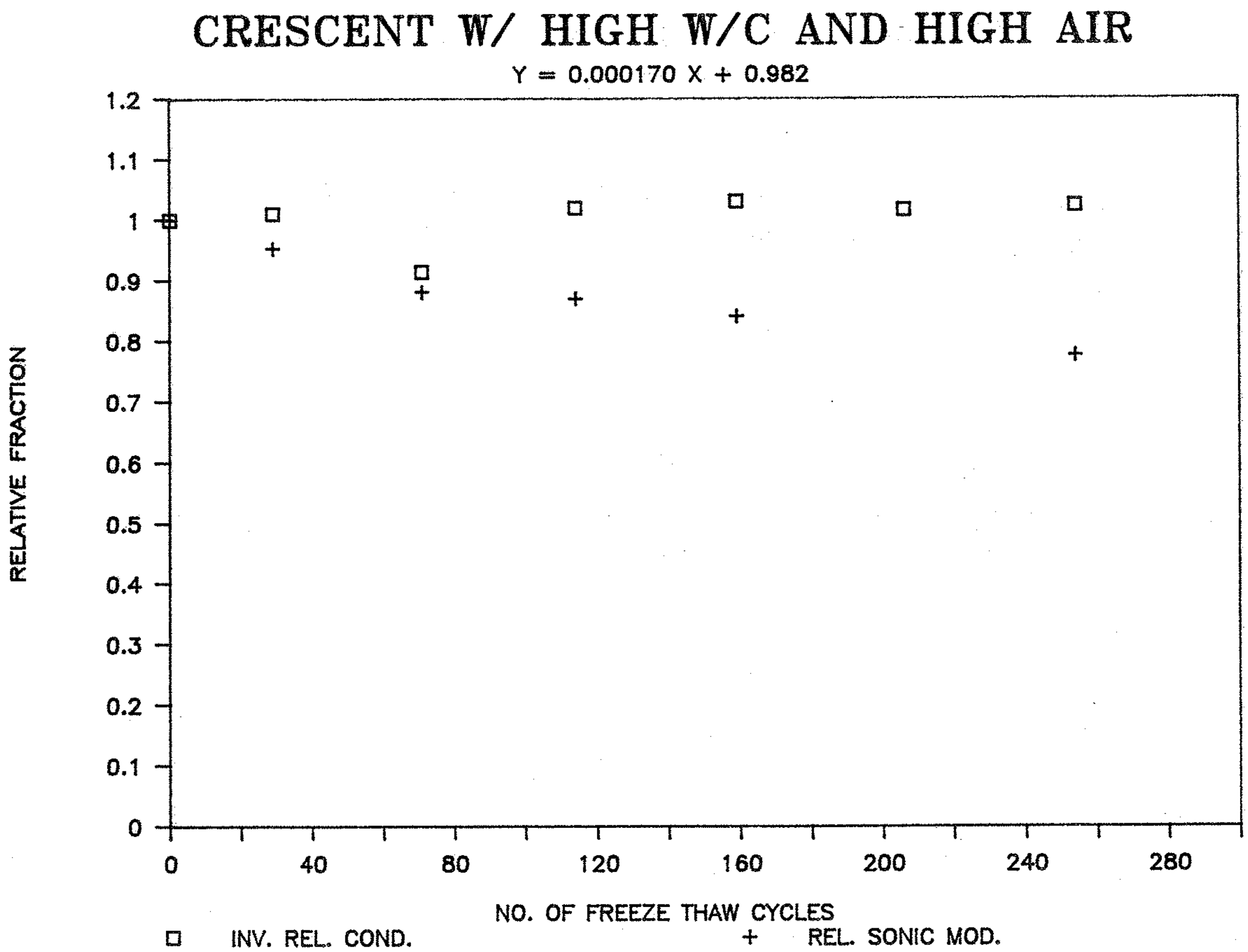


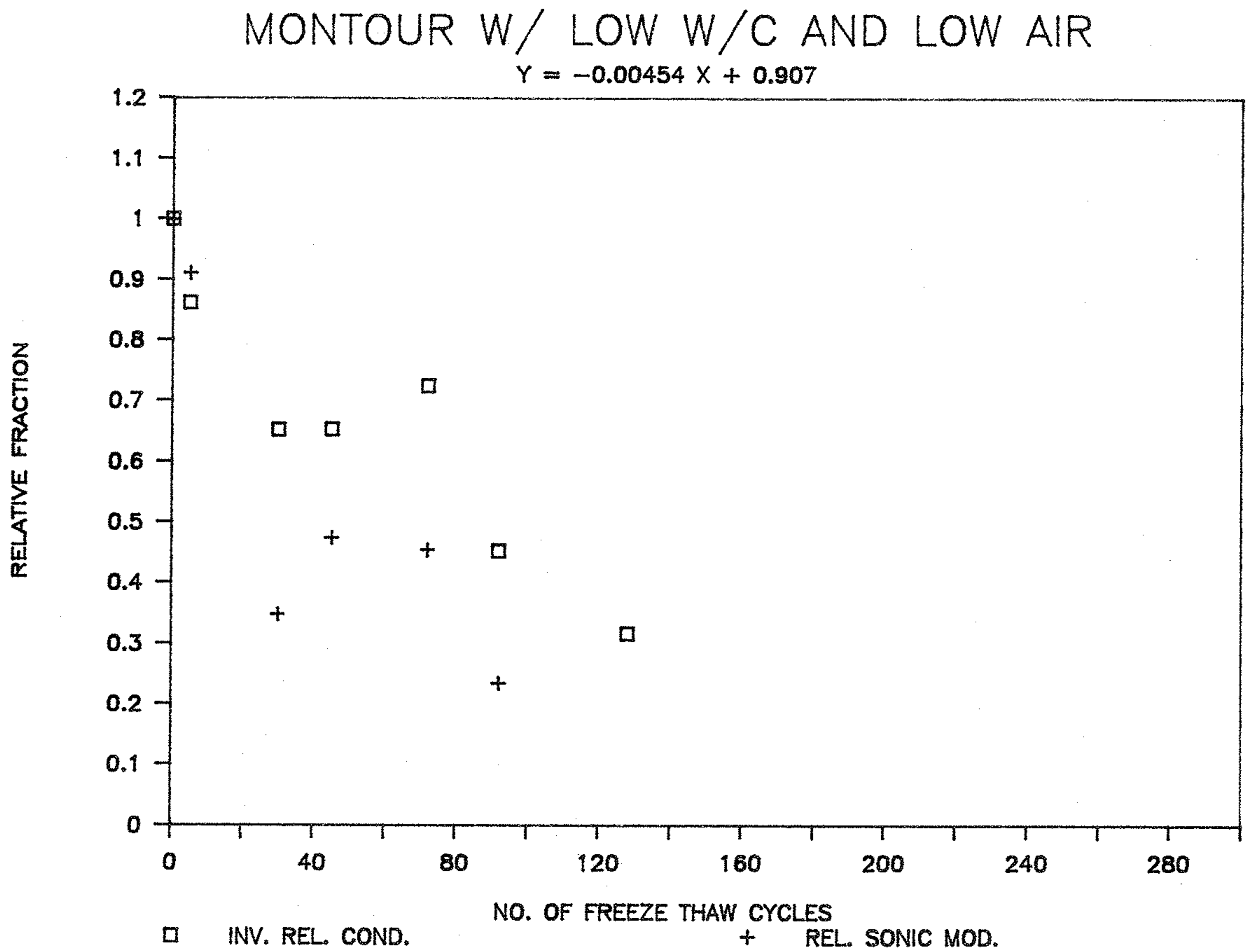

CRESCENT W/ MEDIUM W/C AND HIGH AIR
Y = −0.00064 X + 0.911
RELATIVE FRACTION
1.2
1.1
1
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0
0
40
80
120
160
200
240
280
NO. OF FREEZE THAW CYCLES
□ INV. REL. COND.
+ REL. SONIC MOD.

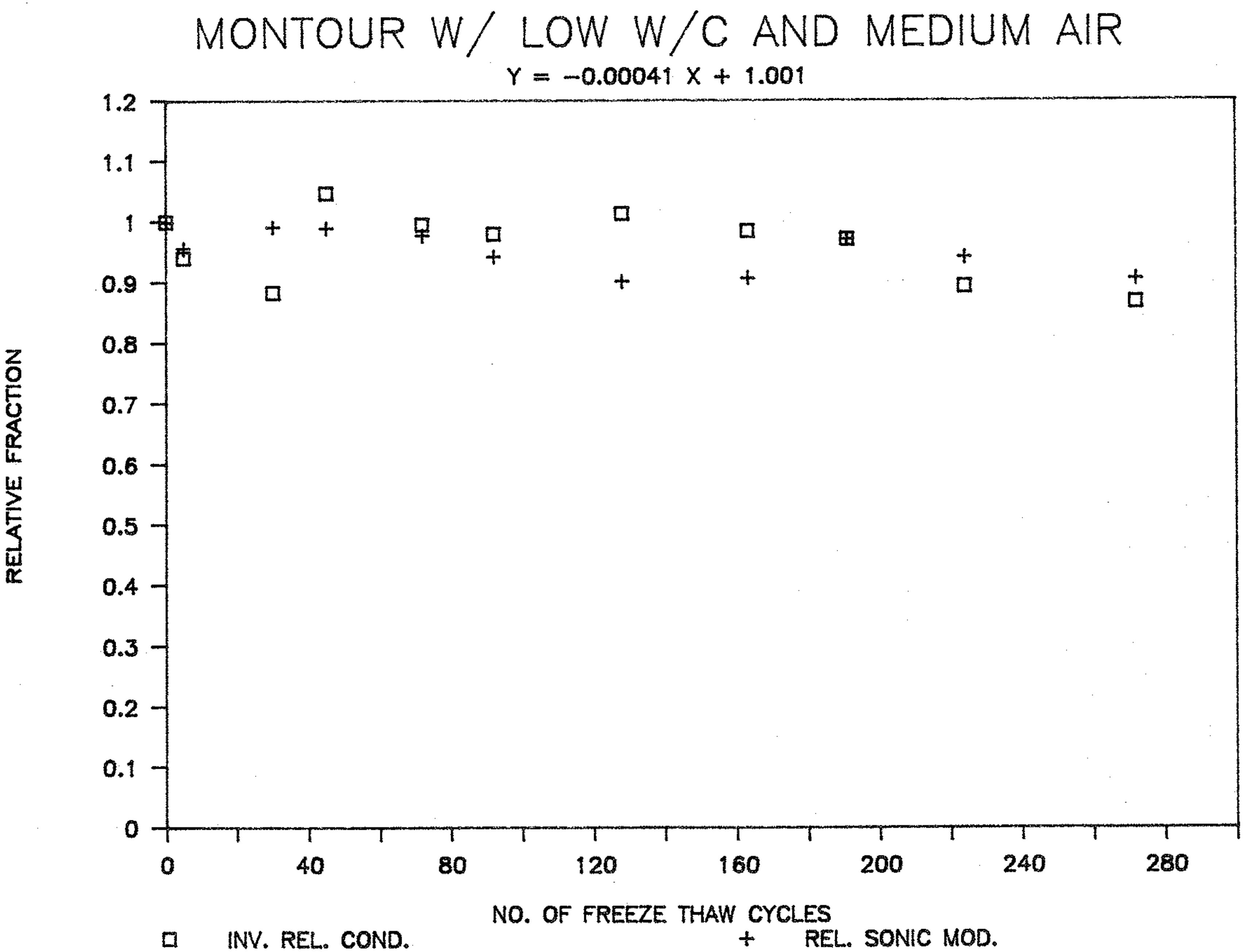

CRESCENT W/ HIGH W/C AND LOW AIR
Y = -0.0185 X + 1
RELATIVE FRACTION
1.2
1.1
1
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0
0
40
80
120
160
200
240
280
NO. OF FREEZE THAW CYCLES
INV. REL. COND.
REL. SONIC MOD.

CRESCENT W/ HIGH W/C AND MEDIUM AIR

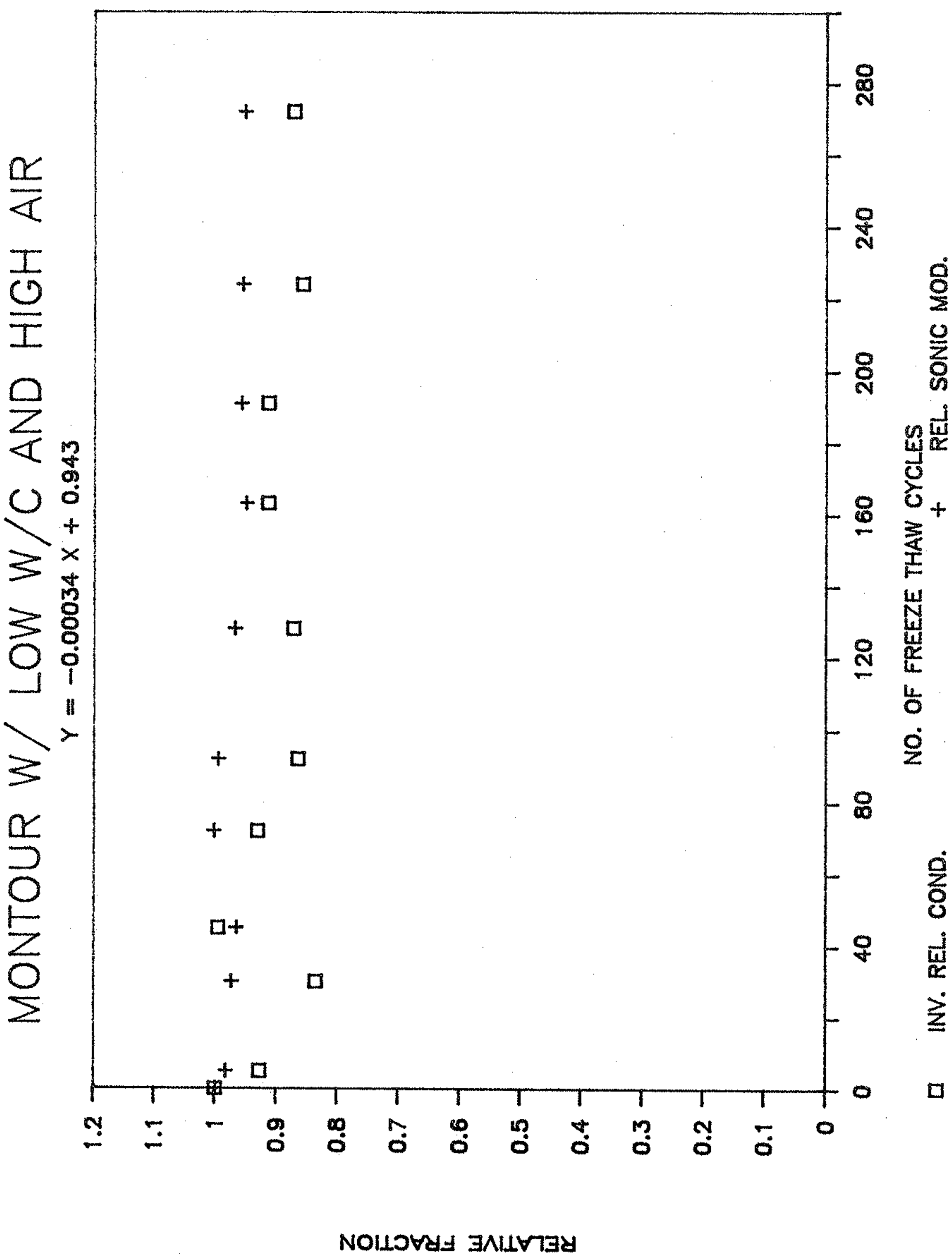


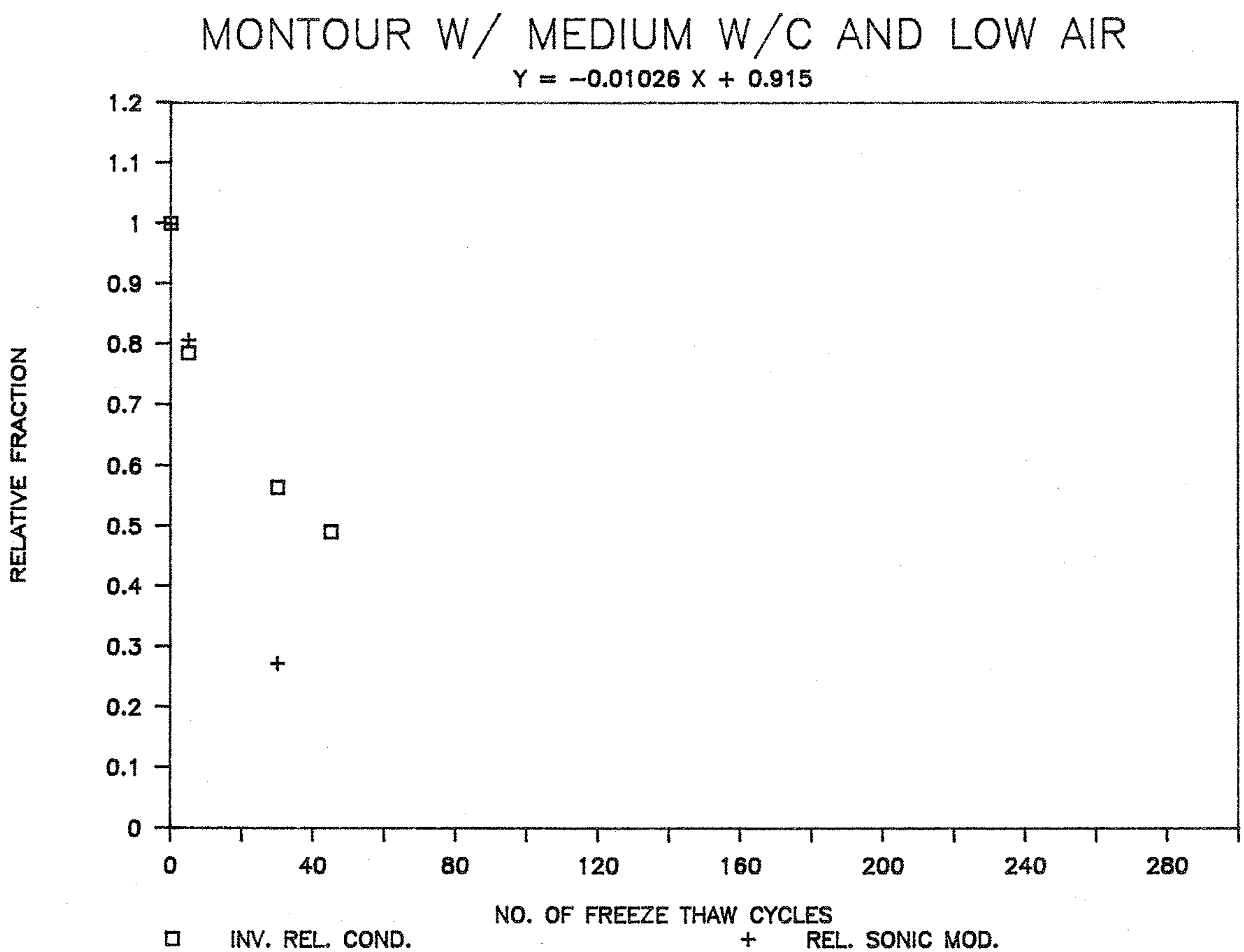

CRESCENT W/ HIGH W/C AND HIGH AIR
Y = 0.000170 X + 0.982
RELATIVE FRACTION
1.2
1.1
1
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0
0
40
80
120
160
200
240
280
NO. OF FREEZE THAW CYCLES
INV. REL. COND.
REL. SONIC MOD.

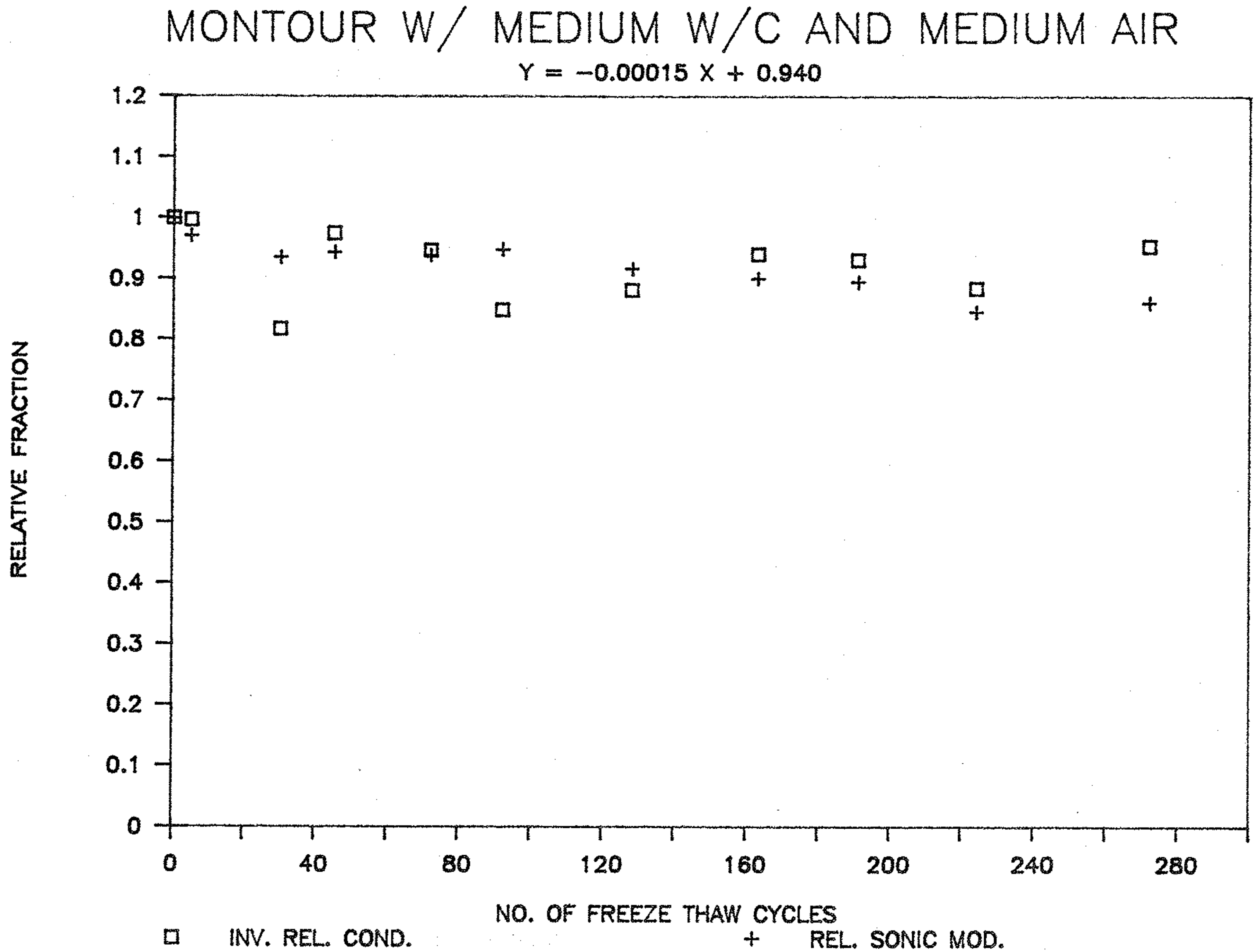

MONTOUR W/ LOW W/C AND LOW AIR
Y = −0.00454 X + 0.907
RELATIVE FRACTION
1.2
1.1
1
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0
0
40
80
120
160
200
240
280
NO. OF FREEZE THAW CYCLES
INV. REL. COND.
REL. SONIC MOD.

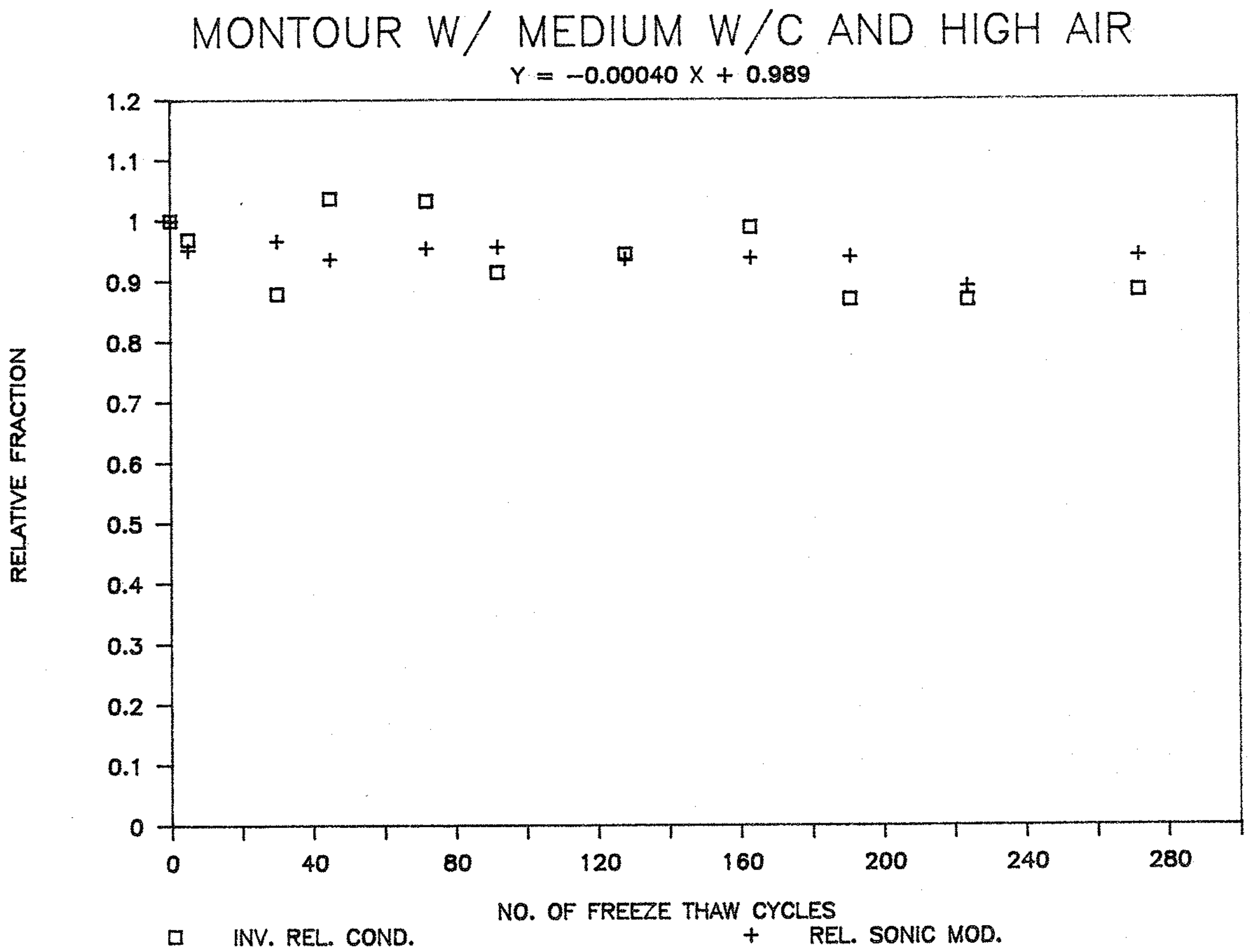


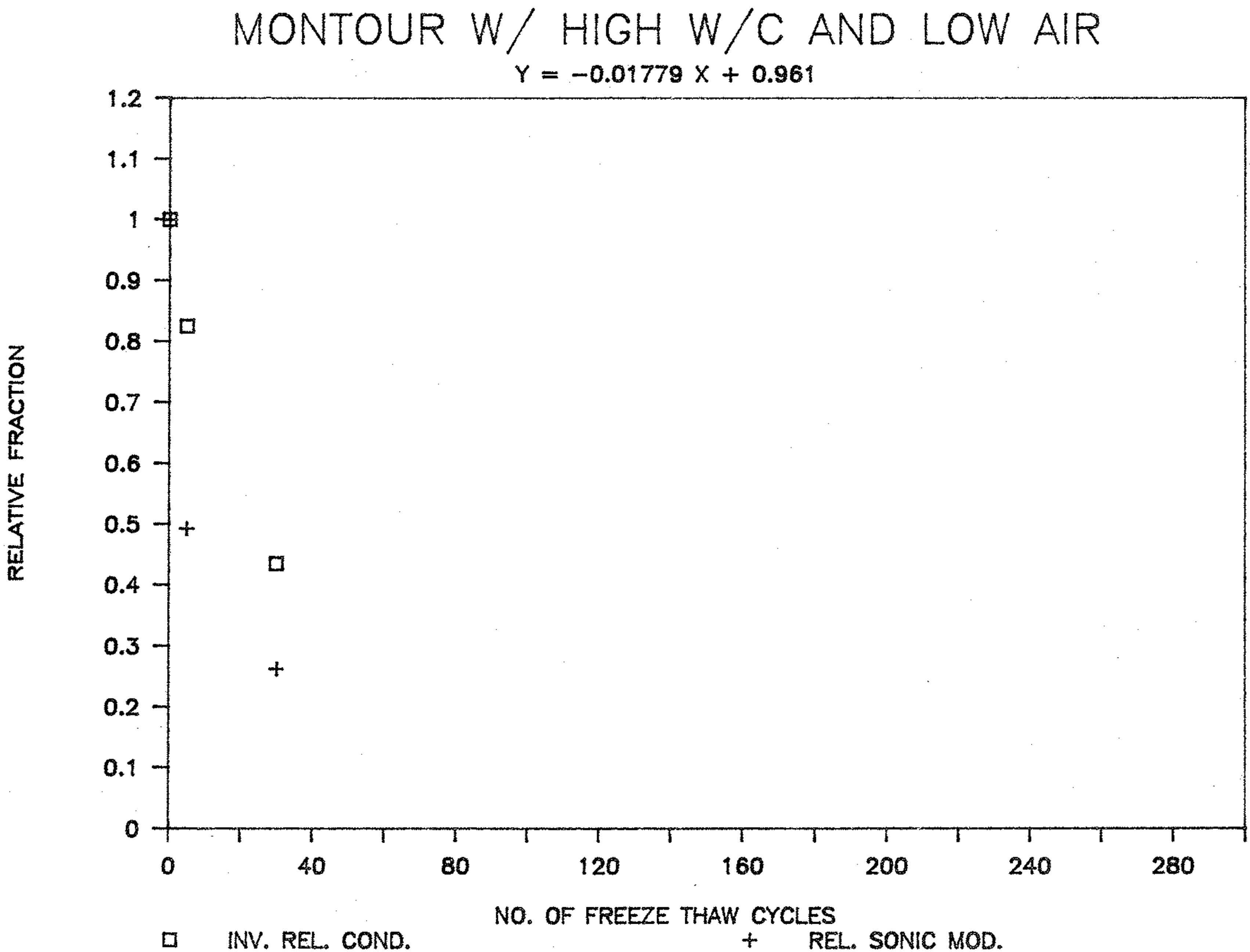
MONTOUR W/ LOW W/C AND MEDIUM AIR
Y = -0.00041 X + 1.001
RELATIVE FRACTION
1.2
1.1
1
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0
0
40
80
120
160
200
240
280
NO. OF FREEZE THAW CYCLES
□ INV. REL. COND.
+ REL. SONIC MOD.

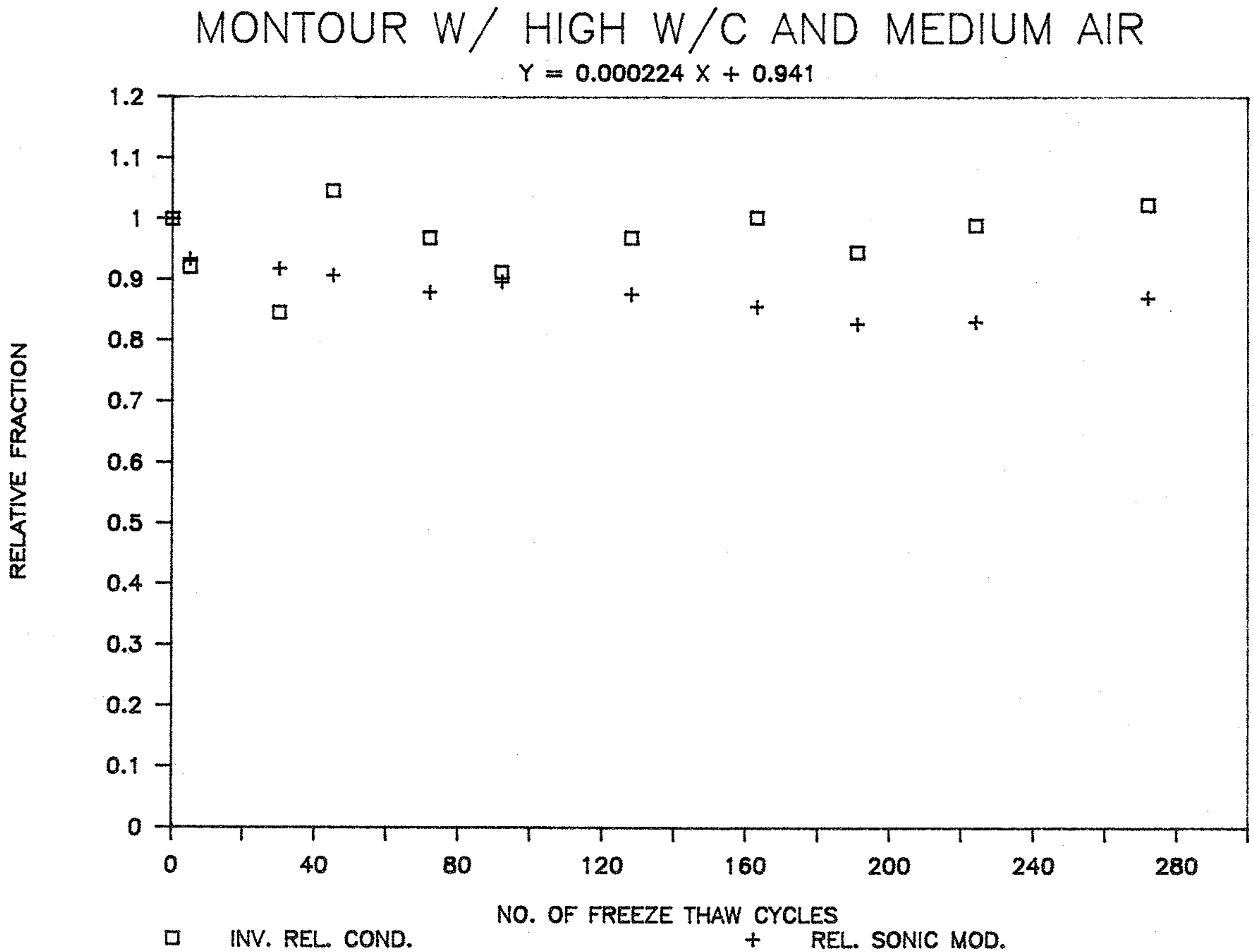

MONTOUR W/ LOW W/C AND HIGH AIR
Y = -0.00034 X + 0.943
RELATIVE FRACTION
1.2
1.1
1
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0
0
40
80
120
160
200
240
280
NO. OF FREEZE THAW CYCLES
INV. REL. COND.
REL. SONIC MOD.


MONTOUR W/ MEDIUM W/C AND LOW AIR
Y = −0.01026 X + 0.915
RELATIVE FRACTION
1.2
1.1
1
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0
0
40
80
120
160
200
240
280
NO. OF FREEZE THAW CYCLES
INV. REL. COND.
REL. SONIC MOD.


MONTOUR W/ MEDIUM W/C AND MEDIUM AIR
Y = −0.00015 X + 0.940
RELATIVE FRACTION
1.2
1.1
1
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0
0
40
80
120
160
200
240
280
NO. OF FREEZE THAW CYCLES
INV. REL. COND.
REL. SONIC MOD.


MONTOUR W/ MEDIUM W/C AND HIGH AIR
Y = −0.00040 X + 0.989
RELATIVE FRACTION
1.2
1.1
1
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0
0
40
80
120
160
200
240
280
NO. OF FREEZE THAW CYCLES
INV. REL. COND.
REL. SONIC MOD.


MONTOUR W/ HIGH W/C AND LOW AIR
Y = -0.01779 X + 0.961
RELATIVE FRACTION
1.2
1.1
1
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0
0
40
80
120
160
200
240
280
NO. OF FREEZE THAW CYCLES
□ INV. REL. COND.
+ REL. SONIC MOD.


MONTOUR W/ HIGH W/C AND MEDIUM AIR
Y = 0.000224 X + 0.941
RELATIVE FRACTION
1.2
1.1
1
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
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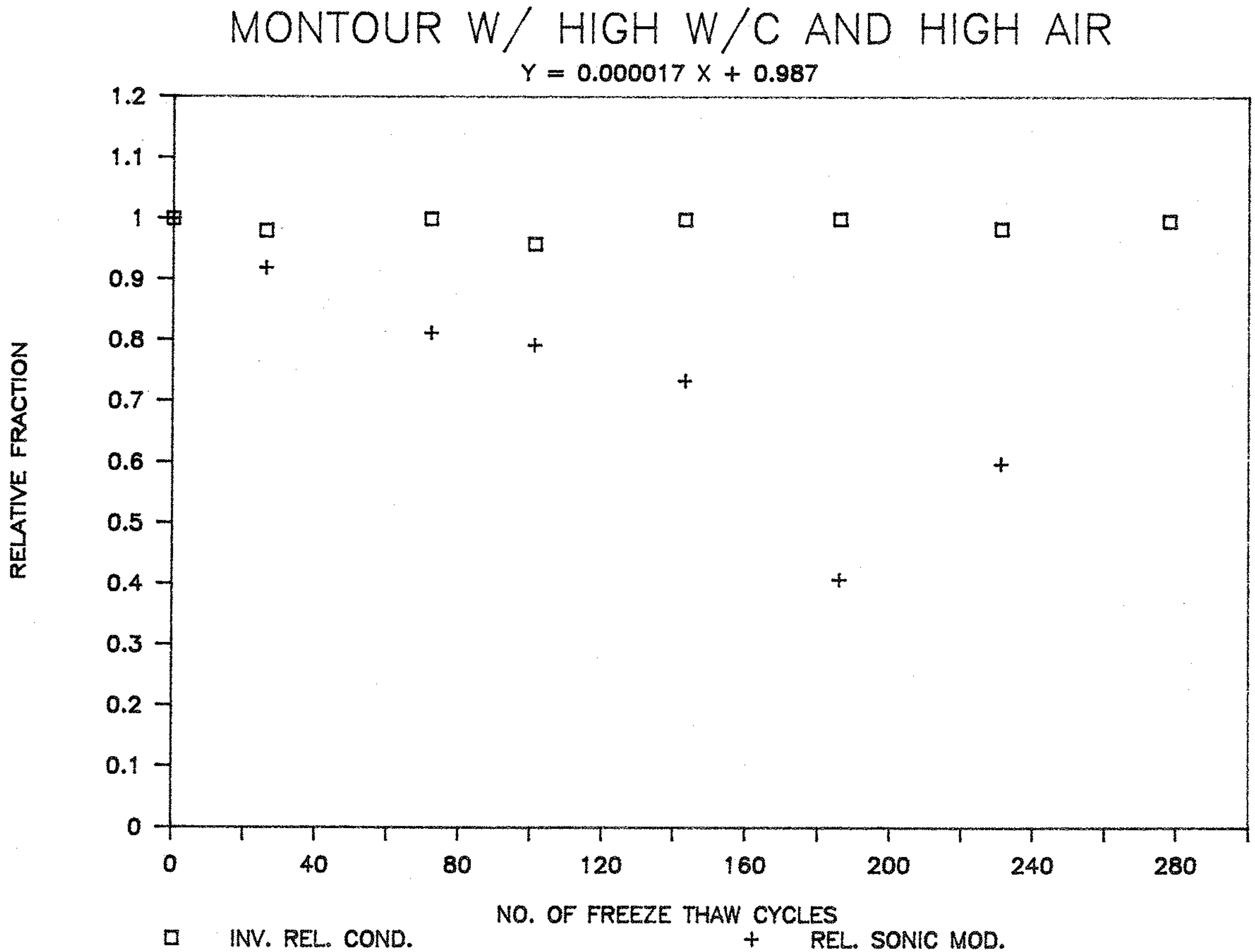
280
NO. OF FREEZE THAW CYCLES
INV. REL. COND.
REL. SONIC MOD.



MONTOUR W/ HIGH W/C AND HIGH AIR
Y = 0.000017 X + 0.987
RELATIVE FRACTION
1.2
1.1
1
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0
0
40
80
120
160
200
240
280
NO. OF FREEZE THAW CYCLES
INV. REL. COND.
REL. SONIC MOD.

APPENDIX B

Determination of the pore structure of porous materials using electrical conductance

Presented at the 18th annual meeting of the Fine Particle Society -- August 1987

(this paper presently is under review for publication)

APPENDIX B

DETERMINATION OF THE PORE STRUCTURE OF POROUS MATERIALS USING ELECTRICAL CONDUCTANCE

by

B. W. Gunnink, B. V. Enustun, and T. Demirel

ABSTRACT

A new method for determining the pore structure of porous materials has been developed. The methodology combines the plastic ice model for solid-liquid phase transitions of pore water with relationships between conductance and temperature, and conductance and pore structure. With these relationships and measurements of the electrical conductance and the corresponding temperature of a saturated porous material subjected to a cycle of capillary freezing and melting, a pore size distribution is obtainable.

Based on this methodology a conductometric phase transition porosimeter was constructed. The pore size distribution of porous Vycor glass was measured using this porosimeter. The modal neck and body radii measured by this method are 28 and 55 angstroms respectively. These results are comparable to those obtained using other techniques. The modal neck and body radii measured on the same sample by mercury porosimetry are 30 and 85 angstroms and by phase transition porosimetry, 27 and 49 angstroms, respectively.

1. INTRODUCTION

Two independent methods which have found large scale application exist for pore-size distribution analysis. The first is mercury porosimetry. Two problems associated with this method have not been solved satisfactorily: (i) the mercury/matrix contact angle, and (ii) the entrapment of mercury during extrusion. The second method, the capillary condensation method makes use of the well known Kelvin equation. It is an elaborate and time consuming method. A discussion of the limitations of these two powerful methods of porosimetry has been presented elsewhere (1,2).

Recently a method was developed based on observation of the liquid-solid phase transition point of pore water in a porous sample at the saturated state and dilatometric measurements (1,2). The method is similar to "Thermoporometry" proposed by Brun et al. (3) which is based on calorimetrically measured freezing points of water in a saturated porous material and assumes freezing is initiated by homogeneous in-situ nucleation. It is in this latter aspect phase transition porosimetry (PTP) differs from "Thermoporometry". It has been shown previously (4) that the model of plastic ice put forward by Everett (5) was applicable to freezing and melting of the pore water in a saturated porous material. In PTP the process of the phase change is followed by measuring the volume change of the sample. The principles of PTP have been discussed in detail elsewhere (1,2).

The purpose of this paper is to introduce another phase transition porosimetry method which is also based on the plastic ice model of Everett but differs from PTP in that the phase change is followed by measuring the change in electrical conductance of the sample and is refered to in this paper as conductometric phase transition porosimetry, CPTP.

2. CAPILLARY FREEZING AND MELTING

Assuming that the pore geometry of the sample can be represented by randomly intersecting spheres or cylindrical capillaries with or without constrictions, it can be shown using the plastic ice model, that the solid-liquid phase transition of water in a pore having an effective radius of r takes place at a temperature, t (^{o}C) given by (4,5):

$$t = -2\gamma T_o/(\rho\lambda r) \qquad (1)$$

where T_o is the normal melting point of ice in ^{o}K, λ is the heat of fusion of ice per unit mass, ρ is the density of water and γ is the ice/water interfacial tension at temperature t. In melting, r is the pore body radius, while in freezing it is the radius of the pore constriction.

3. PRINCIPLES OF ELECTRICAL CONDUCTANCE OF POROUS MATERIALS

Many porous materials consist of a non-conducting solid phase and a void system of randomly intersecting capillaries of various sizes. If the material is saturated the pore solution generally will exhibit electrolytic conductance, which results from the mobility of ions in the solution.

Sunberg (6), Archie (7), Winsauer et al. (8), and Perkins et al. (9) used a parameter called resistivity factor in their investigations of the resistivity of porous geomaterials. Resistivity factor is defined as the ratio of the resistivity, ρ_p, of a porous material completely saturated with an electrolyte to the resistivity, ρ_e, of the electrolyte itself.

Winsauer et al. (8) and later Perkins et al. (9) expressed a relationship between resistivity factor, F, and pore structure where

$$F = \rho_p/\rho_e = \tau/\psi \tag{2}$$

where τ is the tortuosity, defined as the ratio of the mean tortous length, L', of the pore channels in a brine-saturated rock sample traversed by an electrical current flowing between the two ends of the sample seperated by a distance, L, and ψ is the ratio of the apparent cross-sectional area, A', of the conducting electrolyte to the total cross sectional area, A, of the sample. Also these investigators related the resistivity factor to the tortuosity, τ, and porosity, η, of the sample by

$$F = \tau^2/\eta \tag{3}$$

In a commentary of the paper by Perkins et al. (9), Wyllie and de Witte (10), pointed out that this was true only if η equaled τ times ψ, or otherwise stated, the pore volume, V_p, was equal to the product of A' and L'. It can be shown that this is only true for pores with uniform cross-sectional areas.

Consider a cylindrical container of volume V having a cross sectional area, A, and length, L, made of an insulating material, but with ends of a conducting material and filled with a solution of conductivity, κ. The conductance, C, of the container is then

$$C = \kappa/(A/L) = \kappa/(V/L^2). \tag{4}$$

Let the same container be filled with a porous material saturated with the same solution. The conductance will now be considerably less since (i) the mean length of path traversed by the current is longer and (ii) the cross-sectional area which is available to current flow is smaller.

Let's define the conductance of a porous material, C', as

$$C' = \kappa\, V_p/(L^2\, \omega) \tag{5}$$

where κ and L are as defined for equation 4, V_p is the pore volume and ω is a dimensionless pore geometry factor.

An expression for the conductance of a pore system consisting of X number of parallel conducting pores can be developed from equation 5 where

$$C' = \kappa \frac{V_p}{L^2 \omega} = \sum_{j=1}^{j=x} \kappa \frac{V_{pj}}{L^2 \omega_j}, \tag{6}$$

where C', κ, V_p, L, and ω are defined earlier and V_{pj} and ω_j are the pore volume and pore geometry factor for individual conducting pore j. From this relationship it can be shown that if the pore geometry factor for each parallel conducting pore, ω_j, is the same then the pore geometry factor for the pore system, ω, is equal to ω_j.

Let us now consider an irregular single conducting pore channel of the porous material as shown in figure 1a. This pore can be represented by a pore with equivalent circular cross-sectional areas as shown in figure 1b. Such a pore would have the same volume and conductance as the original irregular pore. If this circular pore is then stretched so the curve connecting the centers of the circular cross-sections is a line, the pore can be represented by a series of right angle truncated cones as shown in figure 1c without a change in the volume or conductance. The volume of each cone, V_i, can be expressed as

$$V_i = L_i' (\pi/3)\, r_{oi}\, r_{ni} \left((r_{oi}/r_{ni}) + 1 + (r_{ni}/r_{oi})\right). \qquad (7)$$

It can be shown, using equation 4 for infinitesimally small slices, that the conductance of each cone, C_i, can be expressed as

$$C_i = \kappa\, (\pi\, r_{oi}\, r_{ni})/L_i' \qquad (8)$$

where κ is the conductivity of the pore solution, L_i' is the length of the right truncated cone, and r_{oi} and r_{ni} are the radii of the ends of the cone.

By combining equations 5, 7, and 8 it can be shown that if the pore system of a porous material when stretched consists of a single right angle truncated cone, then the dimensionless pore geometry factor, ω, can be expressed as

$$\omega = (1/3)\,(L'/L)^2 \left((r_o/r_n) + 1 + (r_n/r_o)\right). \qquad (9)$$

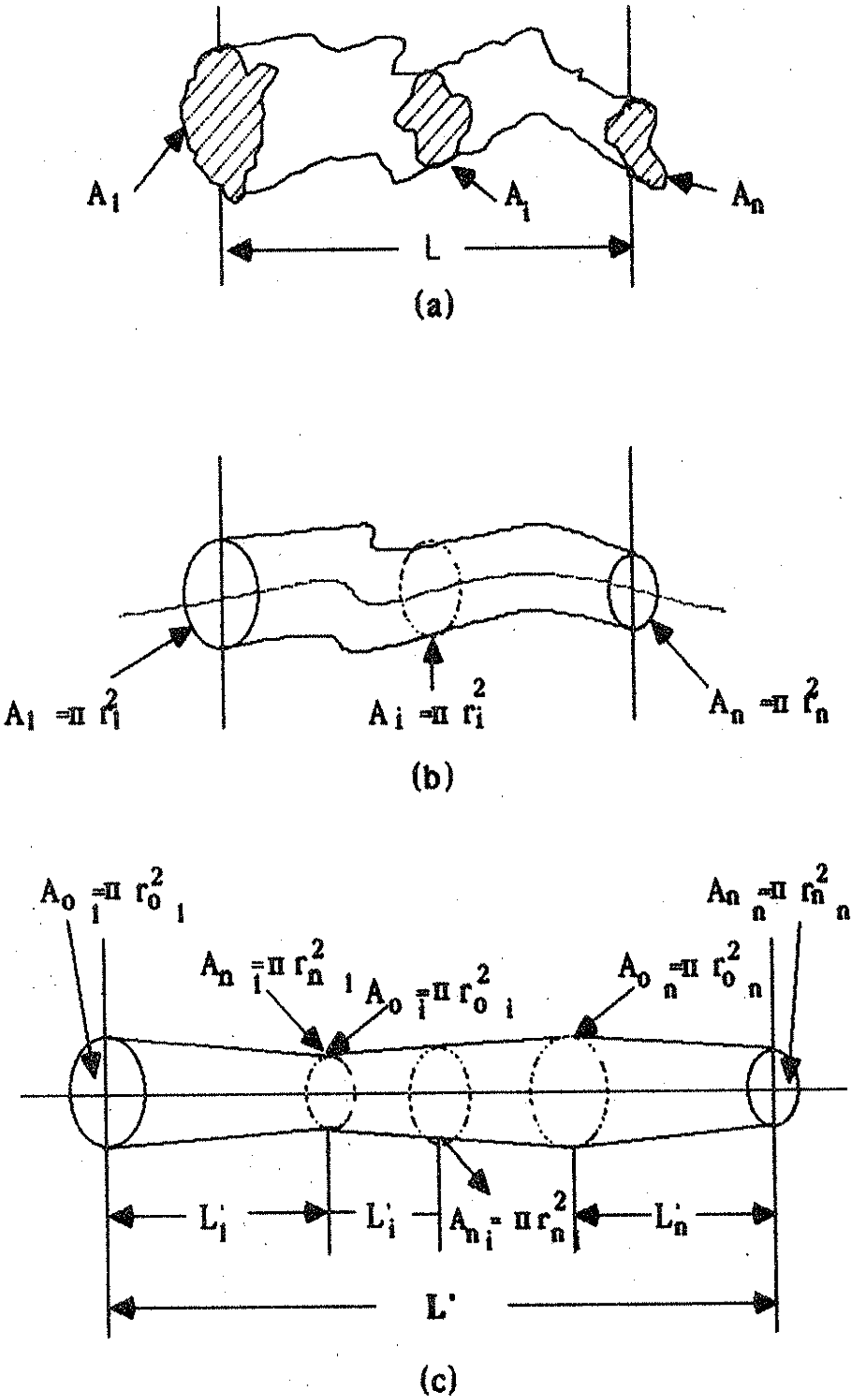


Figure 1 - Right angle truncated cone modeling of irregular pore

Using the same approach the pore geometry factor, ω, for a pore system consisting of a singular irregular pore (represented by z right angle truncated cones in a series connection, figure 1) can be expressed as

$$\omega = \frac{\sum_{i=1}^{i=z} \left(L_i' \, r_{oi} \, r_{ni} \left((r_{oi}/r_{ni}) + 1 + (r_{ni}/r_{oi})\right)\right)}{3L^2} \left(\sum_{i=1}^{i=z} L_i'/(r_{oi} \, r_{ni})\right). \qquad (10)$$

This relationship for a pore of uniform cross-sectional area is simplified to

$$\omega = (L'/L)^2, \qquad (11)$$

where L' is the sum of the lengthes of the component cylinders (right angle cones with uniform cross-sectional areas) and L is the distance between the ends of the pores as defined previously. The quantity (L'/L) has been commonly referred to as tortuosity. Also for a pore consisting of uniformly sized right angle truncated cones (r_{oi} and r_{ni} are constant) equation 10 is simplified to the relationship expressed in equation 9. Further if the connected cones lie in a straight line perpendicular to the end planes of the conducting material L' will equal L and the pore geometry factor, ω, will be a function of the ratio of the pore neck radii to the pore body radii only or a parameter heretofor referred to as necking. This illustrates two fundamental characteristics of a pore system, tortuosity and necking, exist which effect the value of the pore geometry factor, ω. Later in this paper these two parameters, tortuosity and necking, will be referred to in a qualitative sense in discussing the importance of the pore geometry factor in conductometric phase transition porosimetry.

Finally, considering any pore system to consist of x number of irregular conducting pore channels connected in parallel, an expression for the pore geometry factor, ω, for any pore system as modeled by an aglomeration of right angle truncated cones is

$$\omega = \frac{\sum_{j=1}^{j=x} \left(\sum_{i=1}^{i=z} L_i' \, r_{oi} \, r_{ni} \left((r_{oi}/r_{ni}) + 1 + (r_{ni}/r_{oi}) \right) \right)_j}{3L2} \left(\sum_{j=1}^{j=x} \left(\left(\sum_{i=1}^{i=z} L_i'/(r_{oi} \, r_{ni}) \right)^{-1} \right)_j \right)^{-1} . \quad (12)$$

Thus, equation 5 provides a relationship between electrical conductance and pore structure.

Equation 12 provides a rather complicated expression for the pore geometry factor for any pore system. If the parallel conducting pores within the system have equivalent pore geometry factors the pore geometry factor for the system could be evaluated using the relationship given for a single parallel conducting pore (equation 9). Pore systems for which this would be true would contain geometrically similar pores, or otherwise stated all pores in the system would have the same tortuosity and necking. This does not imply that all the pores in the system are identical but rather that large pores are essentially magnifications of smaller pores.

4. CONDUCTOMETRIC PHASE TRANSITION POROSIMETRY

In conductometric phase transisition porosimetry it is assumed that the electronic conductance through the solid phases of a porous material is negligible when compared to the electrolytic conductance of the pore solution. Thus, when the temperature of a mass of saturated porous material is raised from sub-freezing temperatures the frozen pore solution will melt, and an increase in electrical conductance will be observed. Capillaries will begin to melt with smaller sizes melting at lower temperatures in accordance with the plastic ice theory as expressed in equation 1.

The theory behind conductometric phase transition porosimetry is developed from a few basic relationships. First, it is assumed the relationship between the electrical conductivity of an electrolyte and the absolute temperature of the electrolyte is an Arhennius type relationship and can be expressed as

$$\ln \kappa = \frac{a}{T} + b, \qquad (13)$$

where

κ = electrical conductivity,

T = absolute temperature,

a = physical constant, and

b = physical constant.

By combing equations 5 and 13 the following equation is derived

$$y = mx + d + \log_{10}\left(V_p/(L^2\omega)\right) \qquad (14)$$

where L, V_p and ω are as defined earlier,

$x = -1/(\text{absolute temperature})$,

$y = \log_{10}(\text{conductance})$,

m = physical constant, and

d = physical constant.

A plot of the $\log_{10}$ of the conductance versus the negative inverse of the absolute temperature (y versus x) for which no phase change occurs will result in a line with a slope equal to m. Below freezing temperatures the phase change which ocur in a certain range of pores will effectively decrease the volume of conducting pores. Thus, V_p at below freezing temperatures can be considered to be the volume of conducting pores, V_{cp}.

Taking the first derivative of equation 14 yields

$$\frac{dy}{dx} = m + \frac{d\left(\log_{10}\left(V_p/(L^2\omega)\right)\right)}{dx} \qquad (15)$$

Integrating equation 15 over the definite interval, x to x_o, yields the following:

$$\log_{10}\left[\frac{\dfrac{V_{cp}(x_o)}{L^2\omega_{xo}}}{\dfrac{V_{cp}(x)}{L^2\omega_x}}\right] = \int_x^{x_o} \frac{dy}{dx}\,dx - \int_x^{x_o} m\,dx \qquad (16)$$

The right half of equation 16 can be calculated numerically from conductance test data by graphical integration as shown in figure 5 (page 19, results and discussion) and is given the variable name Z. Also, if x_o is the value of x at the pore solution melting point, then $V_{cp}(x_o)$ is simply the total conducting pore volume, V_p. Rearranging equation 16 and referring to pore volume by radius we obtain:

$$\alpha \frac{Vcp(r)}{Vp} = 10^{-Z(x,\, x_o)} , \qquad (17)$$

where $V_{cp}(r)$ is the volume of the pores with radii smaller than or equal to r containing unfrozen pore solution, V_p is the total pore volume and α is the ratio of the pore geometry factor of the total pore system, ω, to the pore geometry factor of the pores smaller than r, ω_r. If it is assumed that the shape factor, ω, is independent of pore size, then α is equal to unity and equation 17 can be reduced to

$$\frac{Vcp(r)}{Vp} = 10^{-Z(x,\, xo)} . \qquad (18)$$

Assuming α to be equal to unity implies the pore system has a certain degree of homogeneity. This does not imply that all the conducting pores are exactly the same but rather that the tortuosity and necking inherent to a given pore structure is uniform throughout the pore size distribution.

These relationships provide the theoretical background for conductometric phase transition porosimetry.

5. EXPERIMENTAL METHOD

5.1. Apparatus

The conductometric phase transition porosimeter consists of a conductance meter, a cryostat, a thermistor, the measurement circuitry, a microcomputer, and a plotter.

Conductance measurements are made with a Solomat 2009 conductivity meter. This meter's high frequency alternating current excitation and the use of stainless steel or tungsten electrodes minimize polarization effects. The instument has a resolution of 0.01 micro-siemens (μS). Digitized conductance output from the meter is relayed to the microcomputer.

A thermistor manufactured by Thermometrics is used to measure the temperature of the sample. The thermistor is used as a variable resistior in a square wave oscillator. The oscillator's output frequency varies with corresponding changes in the resistance of the thermistor. The computer then measures the period of the square wave.

The temperature of the sample is controlled by placing the sample in a suitable container and immersing that container in a Haake cryostat filled with iso-propyl alcohol. The temperature of the cryostat is controlled by the computer through a digital to analog converter.

The computer used is an Apple IIe personal computer equipped with two input/output cards from John Bell Engineering. These cards serve as an interface between the measurement circuitry and the computer. Software was developed to alow the computer to control the temperature of the cryostat, gather test data, perform necessary data processing, and output the processed data to either the computer's video monitor or a Hewlett-Packard plotter.

5.2. Experimental Procedure

The conductometric phase transition porosimeter was used to test a Vycor glass sample obtained from the Corning Glass Company. This porous glass was selected because extensive pore size infromation is available for it and thus comparative analysis with other techniques is available. A 14mm diameter and 4mm thick disk was saturated with a 0.01 molar ammonium nitrate solution. By saturating the sample with this solution measurable conductance was provided with negligible freezing point depression.

For many materials sample preparation necessary for this type of porosimetry will be minimal. It is simply a matter of embedding electrodes in a mass of the material. However, for the brittle Vycor glass a more complex electrode configuration and containment apparatus was devised (see figure 2) It consisted of a large glass test tube and two smaller sections of glass tubing. The Vycor disk was press fitted into one end of the larger tubing with a short piece of tygon tubing serving as a gasket. The smaller tubing was located at the opposite end. This tube provided the inlet for the electrode connections and thermistor. The upper end of it was filled with a dessicant and cotton to prohibit condensation inside the tubes. These two sections of tubing were then placed in the test tube. The electrical contacts with the Vycor disk were provided by mercury placed inside the tubes in contact with the top and bottom sufaces of the Vycor disk. Tungsten wires inserted through tygon tubing provided the electrical connections between the mercury contacts and the conductance meter. Finally, a thermistor was

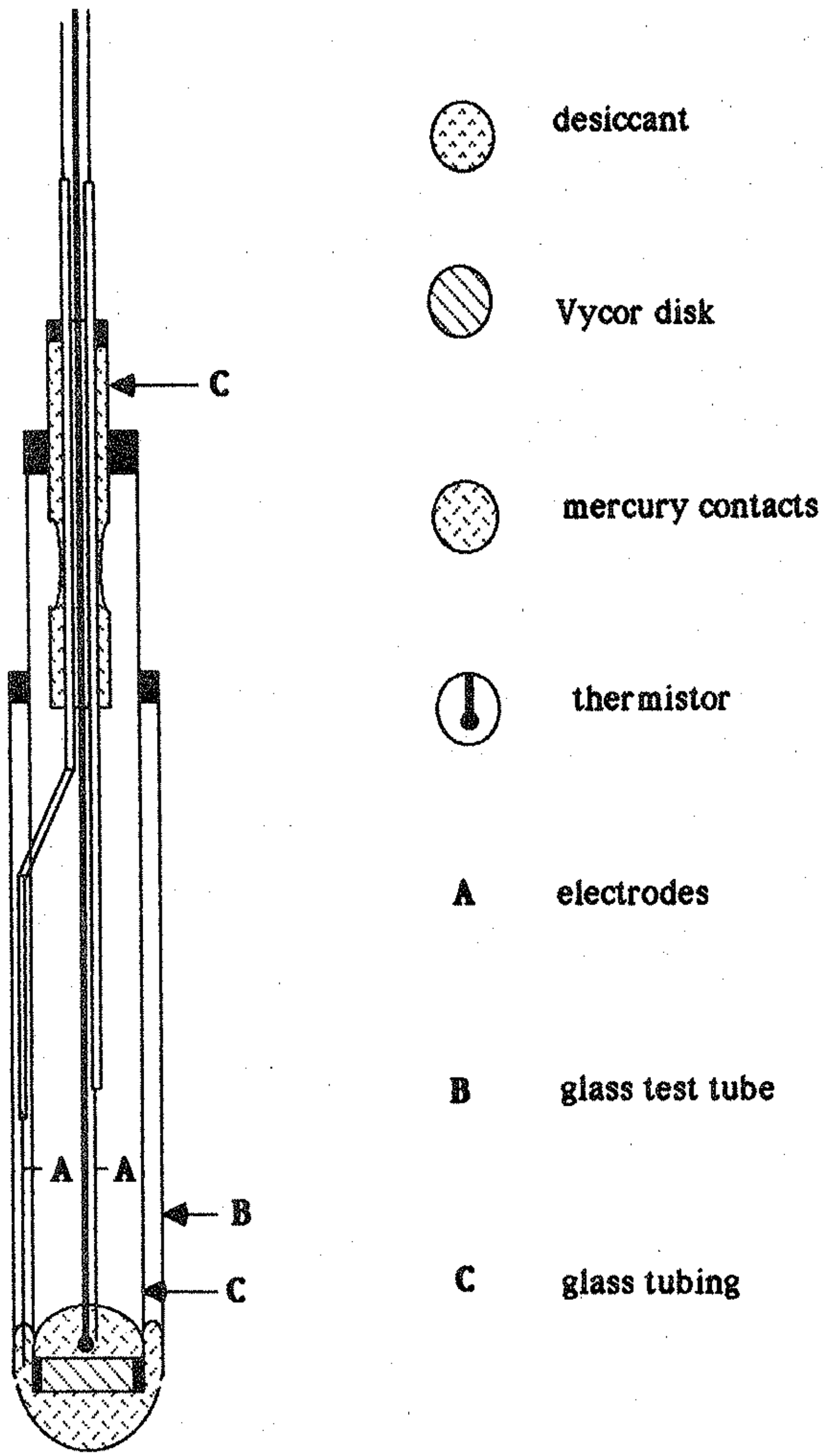


Figure 2 - Electrode configuration and containment apparatus for Vycor glass sample

placed in the mercury immediately above the Vycor sample to provide necessary temperature measurements.

The assembled apparatus was then placed in the cryostat and the testing cycle begun. The temperature of the bath was lowered from 7° C to -30° C at the rate of 3° C per hour. Conductance and temperature measurements were taken at 0.1° C intervals. These data composed the cooling portion of the test. The bath temperature was then raised from -30° C to 7° C at the same rate. Again data was gathered at 0.1° C intervals and these data composed the warming portion of the test.

After some initial experimentation a test run was made with a 5 kilo-ohm resistor connected in series with the Vycor sample. This allowed the test to be run in the more sensitive conductance range available on the conductance meter (0.01 µS resolution). Experimentally obtained values of conductance were then converted to quantitative conductance values using known relationships for conductors connected in series. The results from this testing follow.

6. RESULTS AND DISCUSSION

In figure 3 the logarithm of the conductance is plotted against the negative inverse of the absolute temperature for the Vycor sample tested. A temperature scale as measured in degrees Celsius is also included for reference purposes. The linearity of the curves prior to freezing or after melting of capillary water supports the relationship between electrical conductivity of an electrolyte and temperature as defined in equation 13. The sharp drop in conductance which occurs at approximately -8° C on the cooling curve is due to the rapid freezing of supercooled bulk water.

Figure 4 illustrates grapically the calculation of the parameter Z which is defined in equation 16. Plotted on this graph is the first derivative of the $\log_{10}$ of the conductance with respect to the negative inverse of the absolute temperature versus the negative inverse of the absolute temperature for the warming cycle of the test conducted on Vycor glass. The integration of these data over finite intervals as illustrated in figure 4 give the parameter Z as a function of the negative inverse of the alsolute temperature. Combining this realtionship with the plastic ice model relationship expressed in equation 1 gives Z as a function of pore radius. The relative conducting pore volume as a function of pore radius is then calculated using equation 18.

Figures 5 and 6 illustrate two graphical means of presenting conductometric pore size distributions.

Evaluations of these results were made by comparing them with results obtained on the same sample by phase transition porosimetry and mercury porosimetry. Figures 7 and 8 contrast the pore size distributions obtained from conductometric phase transition porosimetry, phase transition porosimetry, and mercury porosimetry for neck and body sizes respectively.

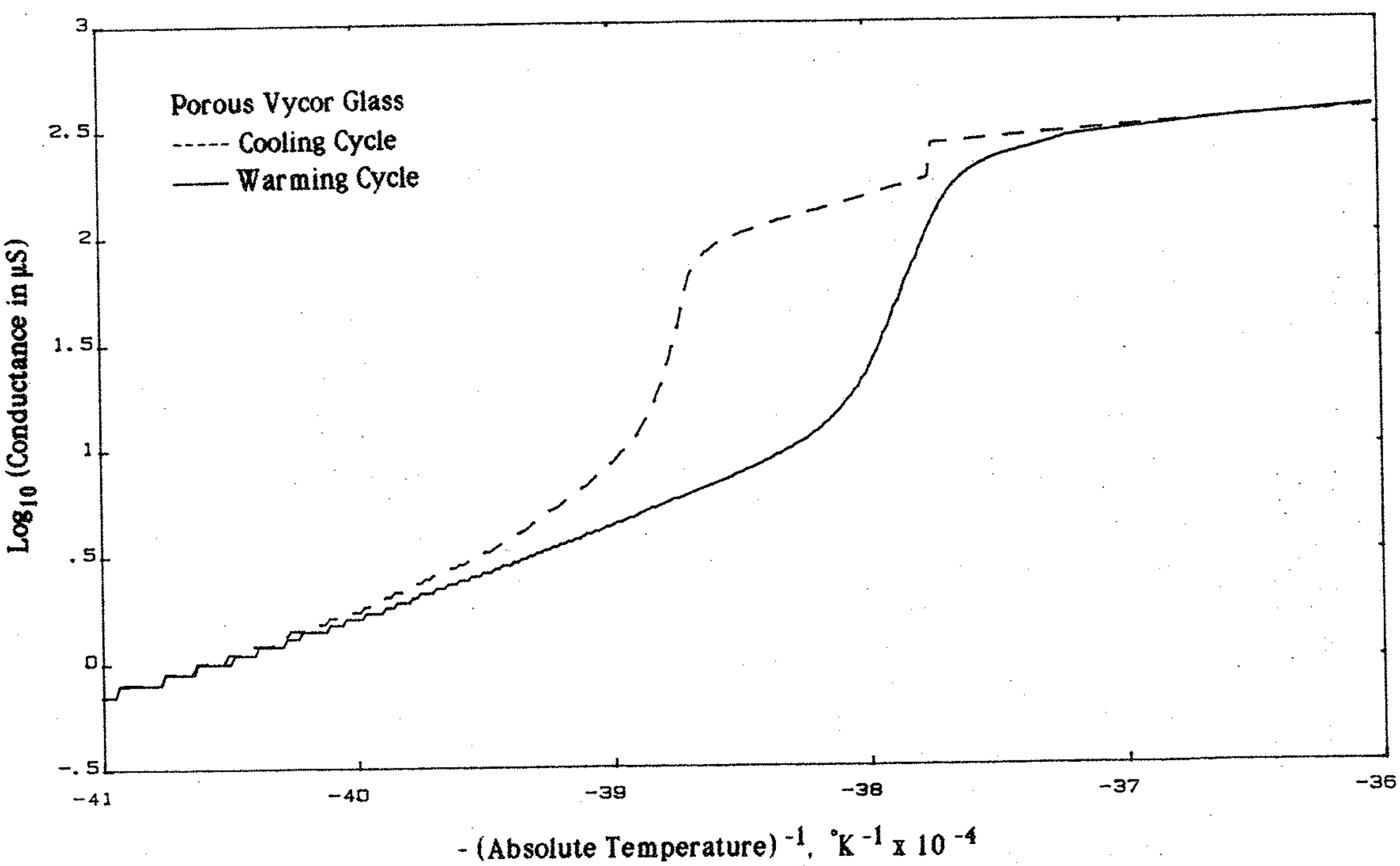


Figure 3 - Logarithm of the conductance versus negative inverse of absolute temperature (Y vs. X) for Vycor glass.

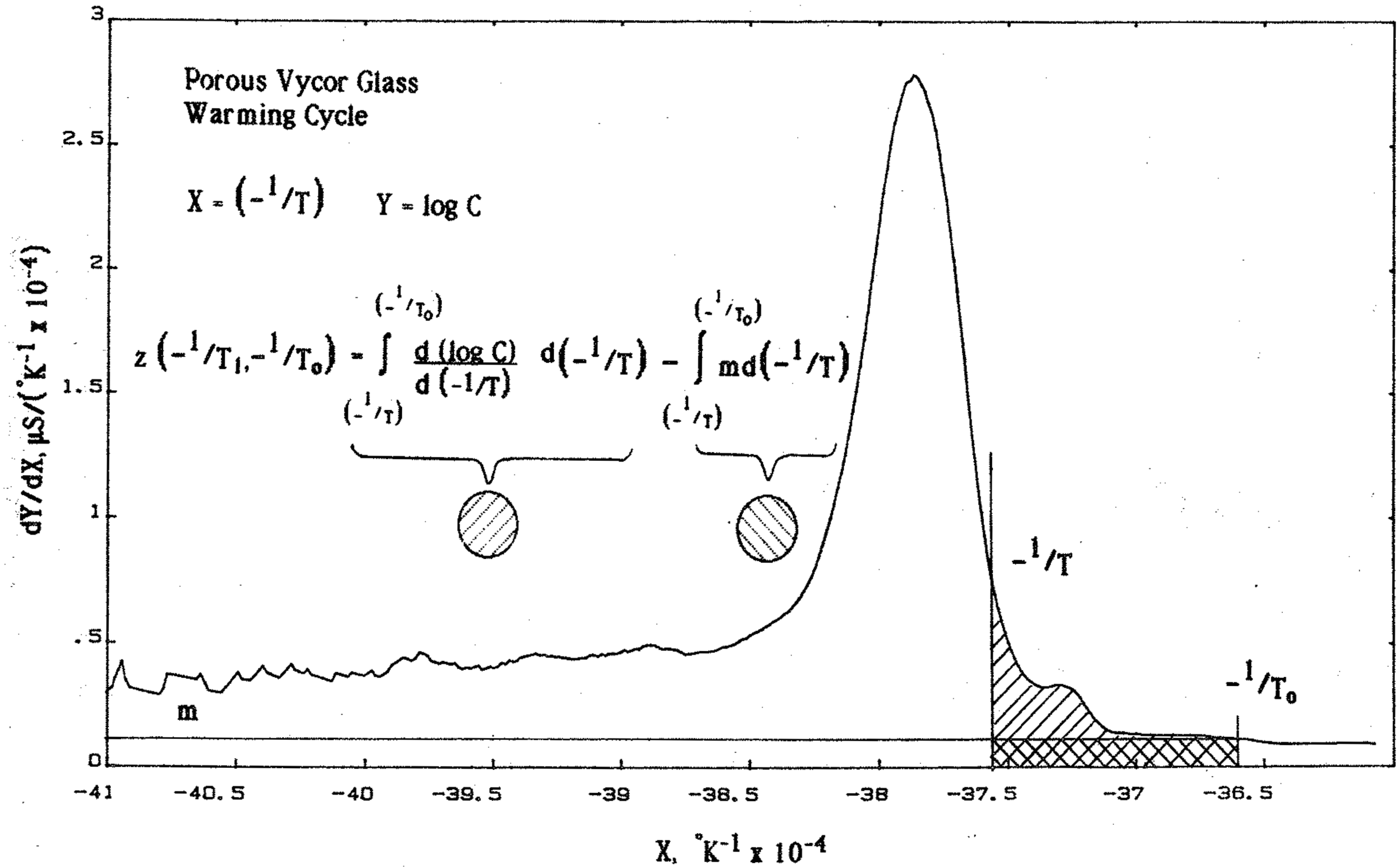


Figure 4 - Illustration of the calculation of the parameter Z in Conductance Phase Transition Porosimetry.

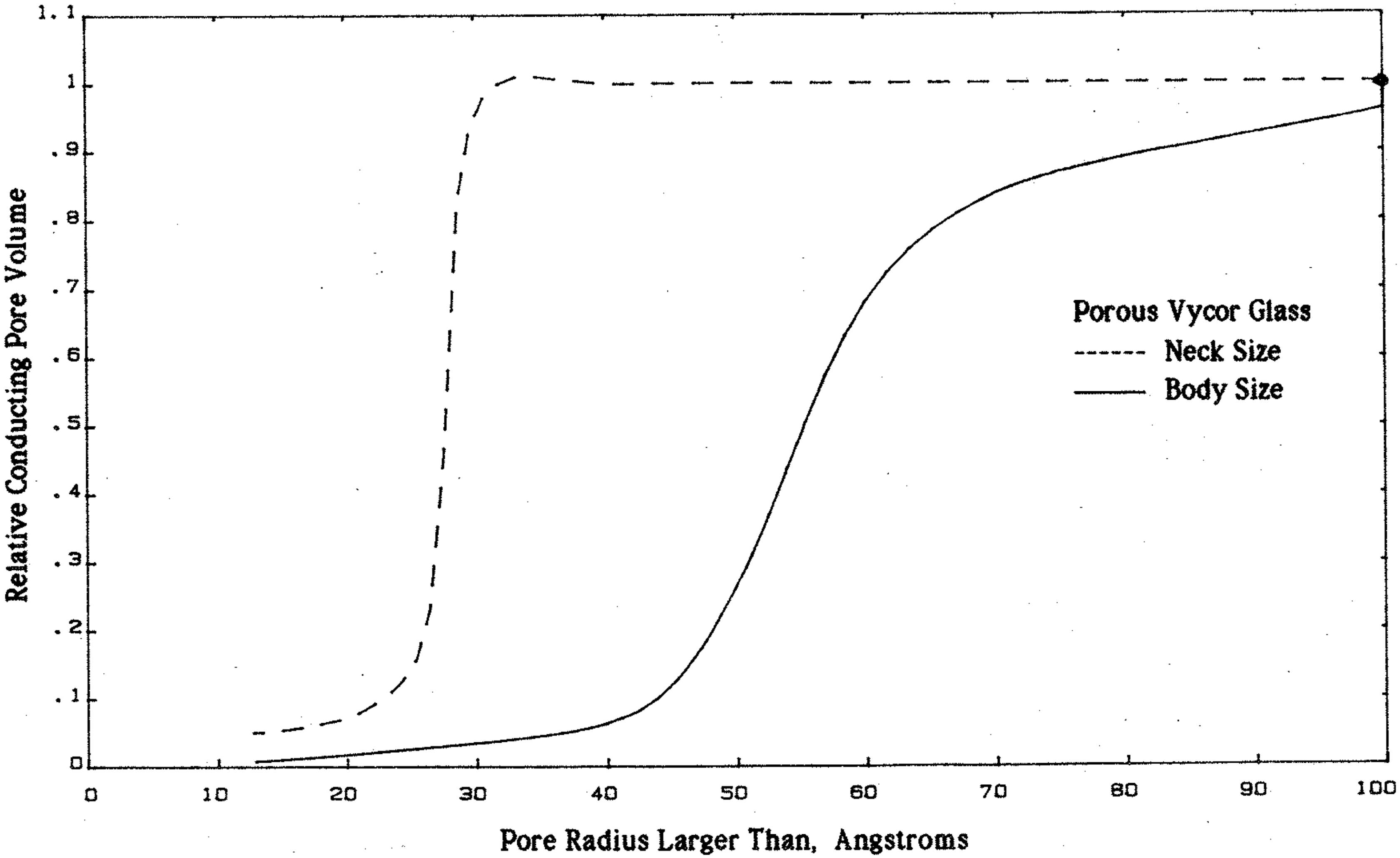


Figure 5 - Conductometric pore size distribution of Vycor glass

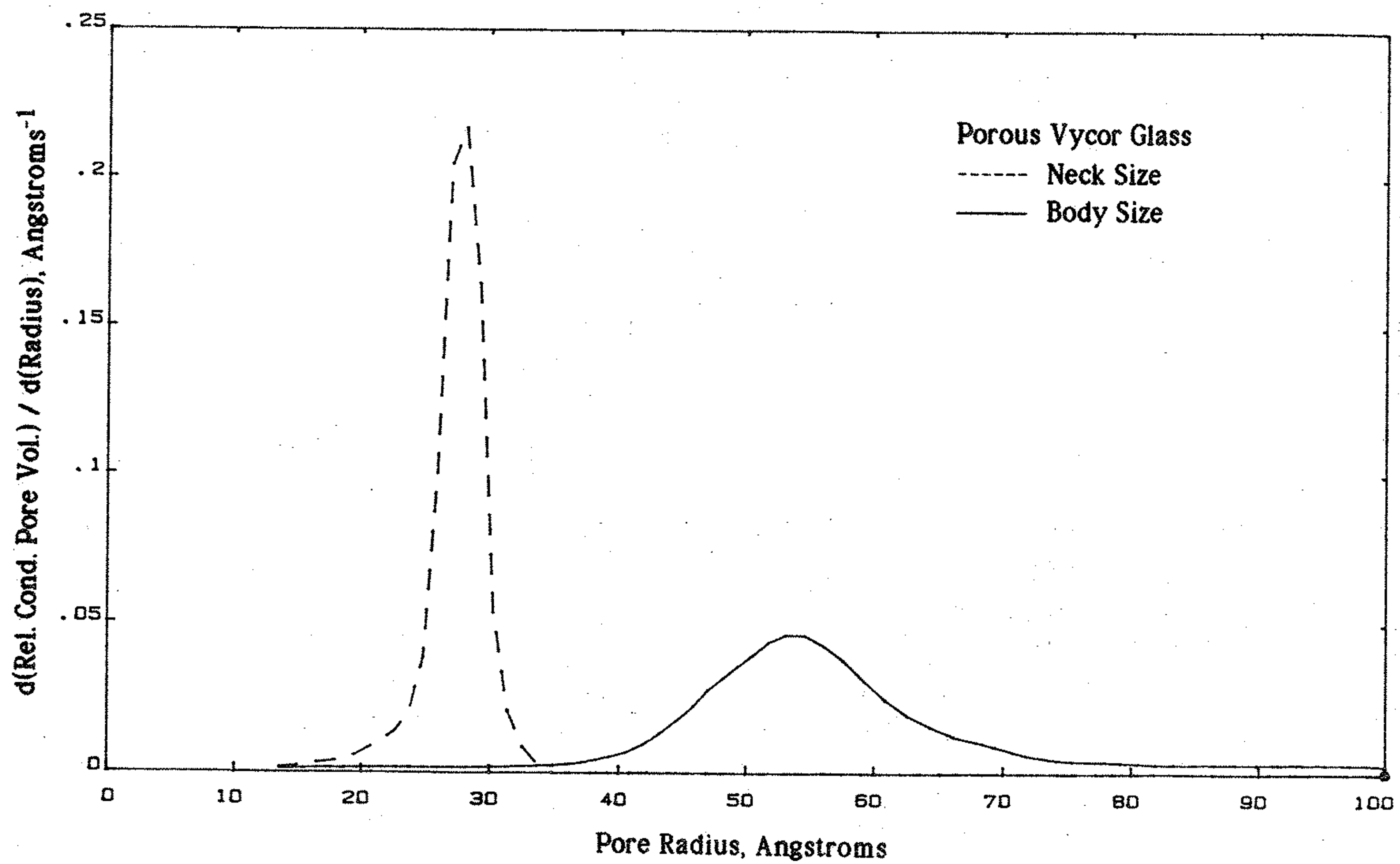


Figure 6 - Conductometric pore size distribution of Vycor glass

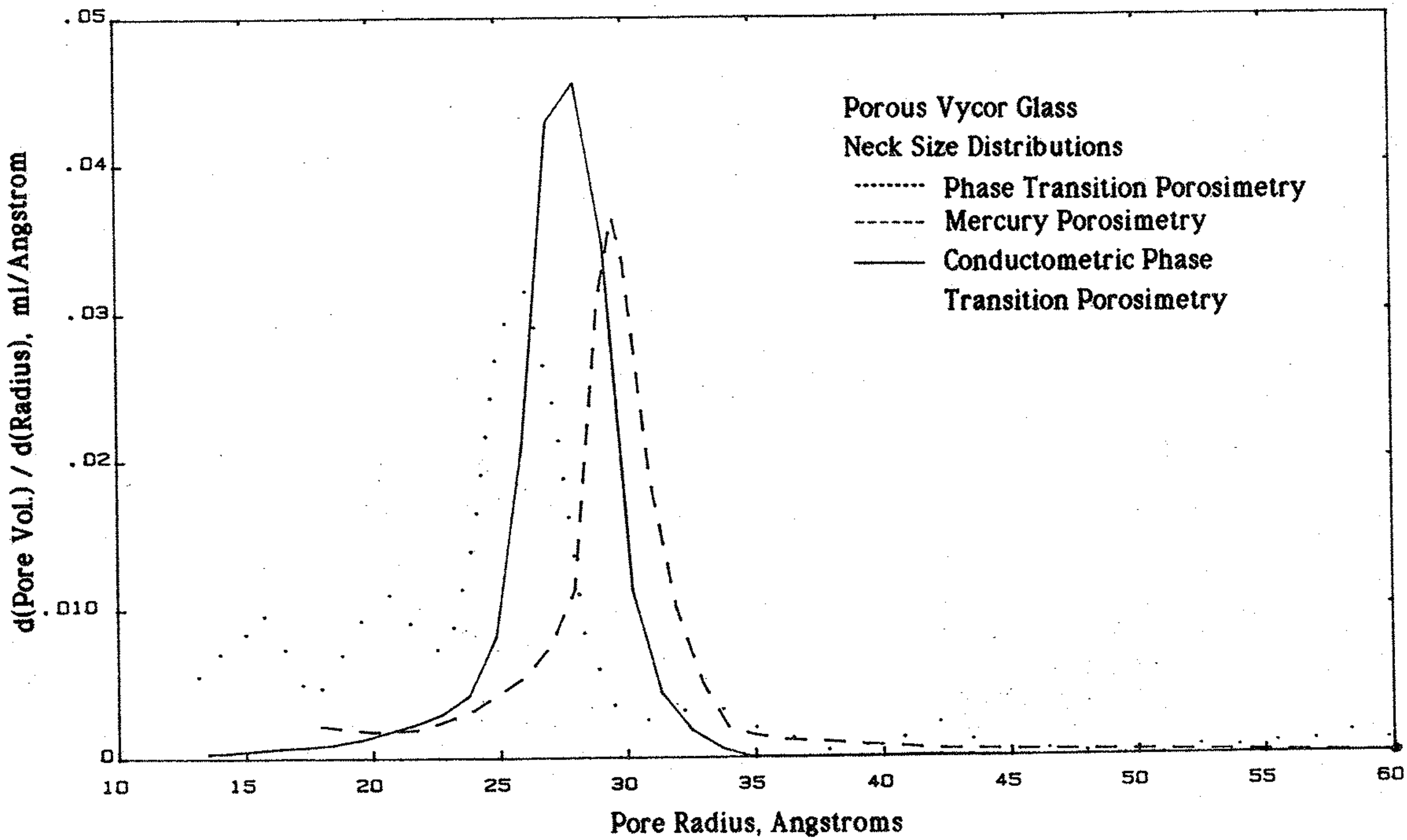


Figure 7 - Comparative neck size distributions for Vycor glass

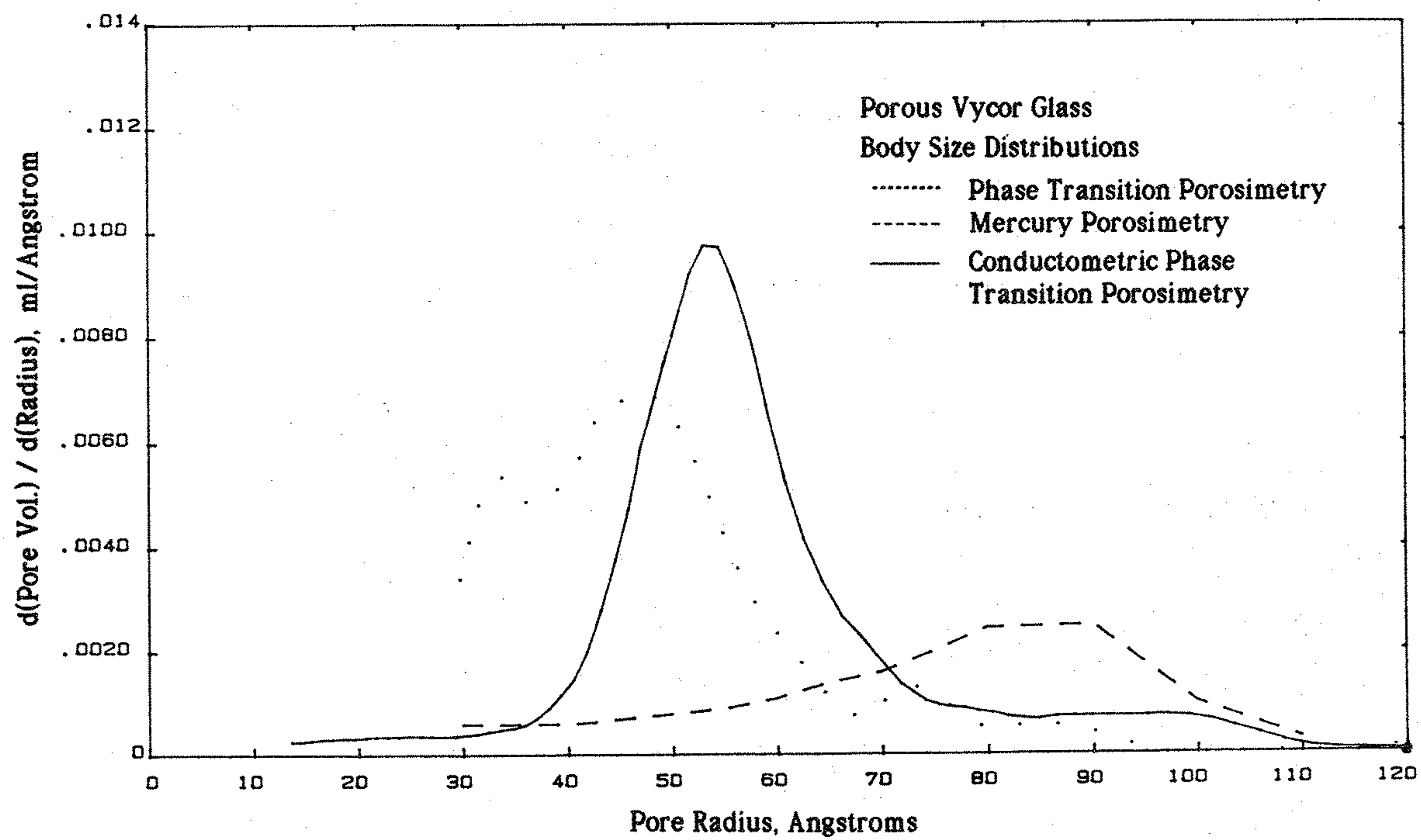


Figure 8 - Comparative body size distributions for Vycor glass

It can be seen from these figures that all three methods of porosimetry give comprable results for neck sizes. For body sizes some difference is observed. It is believed that the difference between the body size distribution as determined by phase transition techniques and that determined by mercury porosimetry is due to uncertainties involved in the entrapment of mercury during extrusion in mercury porosimetry.

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APPENDIX C

Conductometric phase transition porosimeter users manual

CONDUCTOMETRIC PHASE TRANSITION POROSIMETER

USER'S MANUAL
Volume 1
Operation Instructions

CONDUCTOMETRIC PHASE TRANSITION POROSIMETER USER'S MANUAL
Volume 1
Operation Instructions

TABLE OF CONTENTS

I. INTRODUCTION

Conductometric phase transition porosimetry is based on the assumption that the electronic conductance through the solid phases of a porous material is negligible when compared to the electrolytic conductance of the pore solution. Thus, when the temperature of a mass of saturated porous material is raised from sub-freezing temperatures the frozen pore solution will melt, and an increase in electrical conductance will be observed. As ice in capillaries melts at temperatures below the normal melting point of ice, the change in electrical conductance of the material with temperature will be a function of the pore structure of that material. The methodology combines the plastic ice model for solid-liquid phase transitions of pore water with relationships between conductance and temperature, and conductance and pore structure. The theory behind conductometric phase transition porosimetry is developed from a few basic relationships.

Assuming that the pore geometry of the sample can be represented by randomly intersecting spheres or cylindrical capillaries with or without constrictions, it can be shown using the plastic ice model, that the solid-liquid phase transition of water in a pore having an effective radius of r takes place at a temperature, t (°C) given by:

$$t = -2\gamma T_O/(\rho\lambda r) \qquad (1)$$

where T_O is the normal melting point of ice in °K, λ is the heat of fusion of ice per unit mass, ρ is the density of water and γ is the ice/water interfacial tension at t. In melting, r is the pore body radius, while in freezing it is the radius of the pore constriction.

A relationship between the ice/water interfacial tension, γ, and temperature is used where

$$\gamma = \gamma_O + kt \qquad (2)$$

where t is the temperature in °C, k is a dimensional constant with a value of 0.25 ergs cm^{-1} K^{-1}, and γ_0 is the value of γ at 0 °C. Also, for conductometric phase transition porosimetry it is helpful to develop a relationship between pore radius, r, and the negative inverse of the absolute temperature, X. With this information equation 1 can be rewritten as

$$r = \frac{-2T_0(k - \gamma_0 X + kT_0 X)}{\rho\lambda(1 + T_0 X)} \qquad (3),$$

where r, T_0, γ_0, k, ρ, and λ are as defined earlier and X = (-1/T), where T is the phase transition temperature in °K.

The numerical values of the various constants used in computations contained in this document were as follows:

$\gamma_0 = 29$ N m^{-1}

$T_0 = 273.16$ °K

$\rho_w = 1.000$ gm cm^{-3}

$\lambda = 333.3$ J gm^{-1}.

These values were obtained from physical and chemical handbooks.

The relationship between conductance of a porous material, C', and pore structure for a cylindrical sample of porous material with cross-sectional area, A, and length, L, is defined as

$$C' = \kappa\,(V_p/(L^2\,\omega)) \qquad (4)$$

where κ is the electrical conductivity of the pore solution, V_p is the pore volume and ω is a dimensionless pore geometry factor.

Also, it is assumed the relationship between the electrical conductivity of an electrolyte and the absolute temperature of the electrolyte is an Arhennius type relationship and can be expressed as

$$\ln \kappa = \frac{a}{T} + b \qquad (5)$$

where

κ = electrical conductivity,

T = absolute temperature,

a = physical constant, and

b = physical constant.

By combining equations 4 and 5 the following equation is derived

$$Y = mX + d + \log_{10}\left(V_p/(L^2\omega)\right) \qquad (6)$$

where L, V_p and ω are as defined earlier,

X = -1/(absolute temperature),

Y = $\log_{10}$ (conductance),

m = physical constant, and

d = physical constant.

A plot of the $\log_{10}$ of the conductance versus the negative inverse of the absolute temperature (Y versus X) for which no phase change occurs will result in a line with a slope equal to m. Below freezing temperatures the phase changes which ocur in a certain range of pores will effectively decrease the volume of conducting pores. Thus, V_p at below freezing temperatures can be considered to be the volume of conducting pores, V_{cp}.

Taking the first derivative of equation 6 yields

$$\frac{dY}{dX} = m + \frac{d\left(\log_{10}(V_p/(L^2\omega))\right)}{dX} \qquad (7)$$

Integrating equation 7 over the definite interval, X to X_o, yields the following:

$$\log_{10}\left[\frac{\dfrac{V_{cp}(X_o)}{L^2\omega_{X_o}}}{\dfrac{V_{cp}(X)}{L^2\omega_X}}\right] = \int_X^{X_o} \frac{dY}{dX}\,dX - \int_X^{X_o} m\,dX \qquad (8)$$

The right half of equation 8 can be calculated numerically from conductance test data and is given the variable name Z. Also, if X_o is the value of X at the pore solution melting point (273.15 °K for water), then $V_{cp}(X_o)$ is simply the total conducting pore volume, V_p. Rearranging equation 6 and referring to pore radius instead of temperature in light of equation 1 we obtain:

$$\alpha \frac{V_{cp}(r)}{V_p} = 10^{-Z(X,\, X_o)} \qquad (9)$$

where $V_{cp}(r)$ is the volume of the pores with radii smaller than or equal to r containing unfrozen pore solution, V_p is the total pore volume and α is the ratio of the pore geometry factor of the total pore system, ω, to the pore geometry factor of the pores smaller than r, ω_r. If it is assumed that the shape factor, ω, is independent of pore size, then α is equal to unity and equation 9 can be reduced to

$$\frac{V_{cp(r)}}{V_p} = 10^{-Z(X,\, X_o)} \qquad (10)$$

Assuming α to be equal to unity implies the pore system has a certain degree of homogeneity. This does not imply that all the conducting pores are exactly the same but rather that the tortuosity and necking inherent to a given pore structure is uniform throughout the pore size distribution or otherwise stated that they are geometrically similar.

These relationship provide the theoretical background for conductometric phase transition porosimetry.

The Apple IIe computerized data acquisition system has been developed. The system consists of the Apple IIe computer (with two disk drives, a monitor, a parallel interface card, and a serial interface card), a cooling bath, a temperature interface box, and a Solomat conductance meter. The computer controls the bath and records the data for a typical experiment.

II. TUTORIAL

This section will take a step by step look at a typical multicycle conductance test. For further information on any step refer to the detailed descriptions in the program or interface sections.

1. Place program disk in drive 1 and raw data disk in drive 2, then reboot the computer by pressing the CTRL, open-Apple, and RESET keys simultaneously. The computer will restart and load the Main Menu program from the program disk. The programs used in this system are menu based. By choosing a number (to change a parameter) or a letter (to choose an action) the program is controlled. While inputting a selection or data, the left and right arrows on the lower right hand corner of the keyboard may be used to correct mistakes. Press the RETURN key to enter your selection.

2. Place the sample in an appropriate container and immerse in the cooling bath. The sample should be saturated with water and surrounded by bulk water. The container should be slightly larger than the sample and able to seperate the cooling fluid from the sample. Connect the leads from the Solomat conductance meter to the probes mounted in the sample. If better resolution is needed at the lower end of the conductance scale place a resistor in series with the sample to lower the overall conductance below 160 µS and set the scale on the Solomat to 160 µS for the 1.0 cell. If a series resistor is used, the sample's conductance will be calculated by the data processing programs when the appropriate data are entered in the Main Menu before processing. If a series resistor is not used set the scale on the conductance meter to 16000 µS for the 1.0 cell. The thermistor should be placed adjacent to the sample. Turn the cooling bath on by using the green main switch first followed by the black compressor switch. Manually set the bath's temperature to the maximum temperature of the first cycle. It will take about 20 to 30 minutes for the temperature of the sample to reach that of the bath.

3. Start the controlling program by entering C for Run Conductance Test. The program will allow some notes to be entered. Then enter the number of cycle types and define each of the types by entering the maximum temperature, minimum temperature, rate of temperature change, and temperature interval between readings for each cycle type. If data collection is not needed for a paticular cycle type enter the number 0 as the

temperature interval. Next enter the total number of cycles for the test sequence and the cycle type for each cycle. Finally, enter the sample's base file name and the starting cycle number for the test sequence. The test sequence will begin. Place the temperature preset switch on the rear of the cooling bath in the ON position. The computer will control the entire test and return to the Main Menu when finished. When the test sequence is completed, turn off the cooling bath and remove the sample.

4. The next step is to process the data. The parameters shown on the Main Menu control the processing. First choose a1 for File Name. If the name of the file you wish to process is unknown, press RETURN and a catalog of the disk in drive 2 will be displayed. Enter the base file name and the cycle number. For example, Mortar.25 would be entered for the 25th cycle of the sample named mortar. If a resistor was connected in series with the sample indicate so and input the CONDUCTANCE of the resistor (μS). The derivative programs use a moving average in their algorithms to smooth the data. Enter the desired moving average for each of the derivative steps. Some of the intermediate steps generate data that may or may not be of interest. Respond with a Y to a Save File option and the data for that file will be saved to a disk. The temperature of T0 is the normal freezing point of the pore solution in degrees Kelvin (usually 273.15 K). Once the parameters are set in the desired manner, enter A and calculations will be performed.

5 About one minute into the processing the program will prompt the user to remove the raw data disk and insert a Caldat disk. The processed information is stored on a different disk than the raw data and the raw data has been loaded and processed at this point. The total process takes roughly 7-8 minutes for a cycle with 350 points per portion (warming and cooling) and the program returns to the Main Menu when finished.

6. To make plots of the data choose B for Plot Curves. The computer will load the Plotting Program and diplay a menu of data file types (from X.Y through R.DVPDLR). If selection 6 or 7 is chosen, the option of plotting the radius on a log scale is available. The plotting menu will then be displayed. Both the warming and the cooling data, may be plotted simultaneously. Enter the file name(s) of the data (base filename, cycle number, and W for warming or C for cooling, i.e., Mortar .25C for the cooling portion of the 25 cycle of a sample named mortar). Plotting parameters may be adjusted to the user's preference and the range of the data. It is advisable to make a

plot on the computer screen to ensure the parameters are set up correctly before making a plot on the plotter. The computer may be stopped while plotting to the screen at any time by pressing the ESC key. The plot is finished when the computer beeps several times. Pressing any key will return the computer to the menu.

7. When all of the necessary plots have been made, enter E to Exit Program and return to the Main Menu. When finished, return the disks to the cases and turn off the computer monitor. The computer itself is left on to maintain the stability of the amplifier circuits.

III. PROGRAMS

In this section the purpose, operation, and the structure of each program will be discussed. The structure will be given to help understand the program and solve possible problems. Each program will be listed with the following information

```
Main Menu, [Startup], (Convert TC, ...), (general sample file)
    \           \            \                 \
     \           \            \                  data files accessed
      \           \             programs it may branch to
       \            file name stored on program disk as
         program name
```

Most programs are menu based, the user options are listed on the screen by number or letter. Choosing a number allows you to edit the data or a parameter and selecting a letter chooses a particular action. While inputting a selection or data, the left and right arrows on the lower right hand corner of the keyboard may be used to correct mistakes. Press the RETURN key to enter the selection.

There are three types of disks in this system. One of the disks contains the programs to control the tests, process the data and output the results. The other two types are data disks. The raw data disk contains the temperature vs. conductance information for all cycles and the general sample file. The CALDAT disk contains the processed data for the directories listed in the introduction section. Due to disk space limitations, only about two fully processed cycles (all directories saved) or 3 partially processed cycles (only saving X.Y, R.VP, and X.DYDX) may be saved on one CALDAT disk (assuming 340 points per portion of the cycle or 680 points per cycle). More cycles may be stored on a disk if the number of points per cycle is decreased.

DIRECTORY	CONTENT
X.Y	Converted temperature and Conductance
X.DYDX	d(Y)/d(X)
X.Z	Integral of Y
X.VP	Pore Volume
R.VP	Pore Volume vs. Radius
R.DVPDR	d(Pore Volume)/d(Radius)
R.DVPDLR	d(Pore Volume)/d(Radius)

The control program will be discussed first, followed by the data processing and output programs. Program listings can be found in volume 2 of this manual.

MAIN MENU, [Startup], (Plotter, Controller, Convert XY, Derivative XY, Integrate, Convert VP, Convert R, Derivative VP}, (raw data file)

PURPOSE : Main Menu is the central program of the entire system. From Main Menu a new test sequence may be started, data plotted, or data calculations initiated.

OPERATION : Main Menu is the first program that will appear upon starting the system. From Main Menu a test may be started, data calculations initiated, or plots of data made. To start a new test press "C" and you will proceed directly to Controller. To make a plot press "B" and Plotter will be run. The format of the test may be printed on the plotter by entering "D" to run the Test Format program.

The items that appear on the screen initially are only used to control the calculation process. This data is only used for current control purposes and is not stored for each file. To load a file input a "1" for File Name and enter a file name consisting of a base file name and the cycle number. If the name or cycle number of the file is unknown, press RETURN and a catalog of the data disk will be shown. If a resistor was placed in series with the sample enter a "Y", if not a "N". If there was a resistor in series enter the conductance (not the resistance) of the resistor in uS. This data will be used to calculate the conductance of the sample. Both derivative programs use an algorithm that averages the data over a specified interval which may be adjusted. Several of the intermediate programs offer the option of saving the file. If the data from these programs is not important enter a "N" in the appropriate spaces. Not saving the intermediate data will speed the calculations and save disk space. When the temperature data is converted to radius the file may be thinned out so that there is at least one Angstrom between each of the data points. Thinning the file will cut the plotting time and also save disk space. The temperature T0 is the pore solution melting point in degrees Kelvin. A sample menu is shown on the next page.

CONDUCTANCE MENU

1.	File Name		: Mortar.25
2.	T.C to X.Y	Series Resistor	: Y
		Cond of Resistor	: 144.3
3.	X.Y to X.DYDX	Moving Average	: 3
		Save File	: N
4.	X.DYDX to X.Z	Save File	: N
5.	X.Z to X.VP	Save File	: N
6.	X.VP to R.VP	Thin File Out	: Y
		Temp of T0	: 273.15
7.	R.VP to R.DVPDR	Moving Average	: 3
8.	R.VP to R.DVPDLR	Moving Average	: 3

A. Calculate the data
B. Plot data
C. Run Conductance test
D. Print Test Format
E. Exit to system

Enter your menu choice :

CONTROLLER, [Cond2], (Main Menu, Clock, Temp Timer), (raw data, general sample file, CAL)

PURPOSE : Controller controls the cooling bath and records data during a conductance test of the sample.

OPERATION : At the beginning of the program will prompt the user to enter some information about the sample and specify the testing parameters. The computer will first prompt the user to enter some notes on the sample. Typical information would be the date and sample material. Don't put any commas into the information or the computer will ignore the rest of the line after the comma. At the end of each line press RETURN.

Now the program prompts the user to enter the number of cycle types. Any number of cycle types may be specified. For each cycle type enter the maximum temperature, minimum temperature, rate of temperature change, and the temperature interval between data readings. The computer will check the input values to insure the minimum temperature is above the maximum, a negative rate of temperature change is not entered, a rate greater than 30 C/hr is not entered, and that the temperature interval for data readings isn't greater than the difference between the maximum and minimum temperature or negative. If a temperature interval of zero is entered the program will not record any data on that cycle. This feature is useful for multiple cycle runs when only data at certain cycle intervals is needed. All of the maximum temperatures must be the same so that when the computer switches from one cycle type to another there is a smooth transition. If the current cycle has a maximum lower than the next cycle's the computer will begin taking data as soon as the current cycle is over and continue until the temperature is above the level of the next reading.

Once the data for all of the cycle types has been entered the program will prompt the user to input a test profile consisting of a sequence of the cycle types. Input the total number of cycles in the test profile and the cycle type for each cycle. The cycle types are described by number, the first one entered being number one.

Now that the test profile is completed, input the base file name for the sample and the cycle number the test is starting on. For example if a previous test of 45 cycles was run on the sample, input the same base file name and then enter the starting cycle as 46 and the cycle number of the data will now be correct.

The test will now start and a status screen will be displayed. A sample status screen is shown below.

```
COOL                                              Press F to stop

                        Temp (C)     Cond (uS)    Point
   Mortar    Sample 1   -10.22         987        367
     \                    \             \          \
      \                    \             \          Number of last point
       \                    \            Conductance at last point
        \                   Current temperature
        First 10 characters of base file name


                                     CYCLE NUMBER 11 OF 45


   Maximum Temp : 4        Minimum Temp : -30        Temp Rate : 3

               Temp Interval between readings : .1
```

The display shows the current temperature of the sample, the conductance of the last data point taken, and the number of that point. The screen also displays the cycle parameters, the current portion of the cycle (heat or cool), and the progress of the overall test (in this case on the 11th of 45 cycles).

To discontinue the experiment at any time press F and the computer will stop at the end of the cycle. The computer must stop at the end of a cycle to save the data for that cycle.

The data is saved at the end of each cycle and when the test is complete the program returns to the Main Menu.

STRUCTURE : Controller allows the user to input notes on the sample, the parameters each cycle type, and the cycle types that make up the test profile. This information is stored on the raw data disk as the CT file.

The cycle type parameters are stored in the arrays TP, BTTM, INVL,, and RA for maximum temperature, minimum temperature, temperature interval and the rate of temperature change respectively. The TD$ array is used to tell the program whether data should be taken during the cycle. If the temperature interval is set at 0 then TD$ is set to tell the computer not to take data during the cycle, otherwise TD$ is always set for taking data.

The test profile is stored in the array TC. Each element contains the cycle type corresponding to the cycle number.

The sample descriptions are used for the base file name. The starting cycle number will be one unless previous tests were run on the sample. If previous tests were run the starting cycle number is added to the current cycle number to get the cycle number the file is saved under (see the introduction for an explanation of the file names). For example if the starting cycle number is 10 the first cycle will be stored under cycle 10 and the second under 11 and so on.

When all of the data are entered the computer begins the test. There are two parts to each cycle, cooling and warming. During both portions of a cycle the same type of control cycle is performed. The temperature interval of the next reading is set and the temperature is continuously monitored. When the temperature passes the interval a conductance reading is taken and both the temperature and conductance are stored in the array SDT. The temperature of the interval is checked to see if it is past the end temperature for the current portion of the cycle. If the interval is past the end temperature then that portion of the cycle is completed. If the interval is not past the end temperature, then the next interval is set and the control cycle is repeated. While cooling a reading is taken when the temperature is lower than the temperature interval set and the cycle ends when the interval is less than the minimum temperature for the cycle. While warming, the temperature must be greater than the interval and the interval must be greater than the maximum temperature for the cycle.

The temperature of the cooling bath is controlled throughout the cycle. The bath is either cooled or warmed at the rate of temperature change specified. The temperature is controlled by sending a number between 0 and 255 to the temperature controller card. The card then converts the digital input to a voltage output that is sent to the cooling bath (for more information see the hardware section). A 0 represents 7 °C and 255 represents -35 °C. The temperature can be controlled in integer steps between these two values for a resolution of .164 °C/integer. For example 0 °C would be set by sending a 42.6 but really either a 42 or 43 would be sent for .112 or -.052 °C respectively. At the beginning of either portion of the cycle the starting and ending temperature points are set and and the time (in seconds) between integer steps is calculated. If a cycle has end points, for example, of 4 and -16 °C, the corresponding integers would be 18 and 141. If the rate of temperature change was 20 °C/hr then the time between integer steps would be

(4 - (-16)) °C / 20 °C/hr = 1 hr * 3600 sec/hr = 3600 sec

3600 sec/ (141 - 18) steps = 29 sec / step.

Each time the sample temperature is read the time is checked to see if it is time for the next temperature step. The bath temperature will continue to change until the end of the cycle even if it passes one of the end points. The bath would only stop if one of the limiting end points (7 or -35 °C) is reached.

The clock for controlling the rate of temperature change is produced by the temperature controller card. An machine language program, Clock, is loaded from the program disk converts one of the timers on the temperature controller card to a clock.

The temperature of the sample is measured by a thermistor. The variable resistance of the thermistor is converted to variable frequency by an square wave oscillator. The period of the square wave is then measured by the interface card using a machine language program, Temp Timer, loaded from the program disk. The length of the period is then read by the program. The period is converted back to a temperature by linear interpolation of a temperature table read from the CAL file on the program disk.

The conductance is measured by the Solomat conductance meter. The meter's reading is transmitted in parallel to the interface card. The conductance may then be read directly from the interface card by the program.

When the cooling portion of a test cycle is completed the data are saved to the disk, the temperature controller parameters are set for the warming portion, and the controlling loop is started. At the end of a warming portion there is one extra thing tis checked. The data is saved, but then the cycle number is checked to see if the test is completed. If there are still cycles left to complete, the next cycle type is loaded, the temperature controller parameters for the cooling portion are set, and the controlling loop is started.

At the end of a test the program returns to the Main Menu.

PLOTTER, [Plot], (Main Menu, HP Plotter), (general sample file, V, PCV, DVDP, DVR, DSR, S)

PURPOSE : Plotter allows the user to load a sample's data files from any of the directories and plot the data on the computer screen. The plotting parameters may be changed. Plotter may also pass the data to HP Plotter to make a plot of the data on a Hewlett Packard plotter.

OPERATION : Plotter first prompts the user to enter the directory to plot from. The menu below will appear.

```
MENU OF SUBDIRECTORIES

          1. X.Y
          2. X.DYDX
          3. X.Z
          4. X.VP
          5. R.VP
          6. R.DVPDR
          7. R.DVPDLR
          8. EXIT TO MAIN MENU
          ENTER NUMBER OF YOU CHOICE
```

Choose a type of data. If 6 or 7 is selected the user has the option of plotting the radius on a log scale. The plotting menu will then be displayed (a sample is shown).

```
Enter the letter of any value that you wish to change
and press RETURN.
     1.  Title ........................................ Concrete Mortar  27 March 87
     2.  Filename ..................................... Mortar.25W
     3.    Label ...................................... Warming
     4.    Linetype ................................... 7
     5.  Filename ..................................... Mortar.25C
     6.    Label ...................................... Cooling
     7.    Linetype ................................... 0
     8.   Minimum X ................................... 10
     9.   Maximum X ................................... 10000
     10.  X-Interval Size ............................. 10
     11.  Minimum Y ................................... 0
     12.  Maximum Y ................................... 1
     13.  Y-Interval Size ............................. .1
     14.  Paper Length ................................ 8.5

----------------------------------------------------------------
     A - Onscreen Plotting          D - Clear Graphics Screen   False
     B - HP722 Plotting             E - Exit Program
     C - View Graphics Screen       F - Find New Data Points

     Enter your choice and press RETURN.
```

Choices from this menu may either load data files, change plotting parameters, or choose an action. Two files may be plotted at the same time, usually the warming and cooling data from the same sample and cycle. To load a file enter 2 or 5, and enter the complete file name for the data. When a file name is entered, the computer does not load the file into an array but instead loads it during plotting. The label and the type of line associated with each file may be changed by choosing 3,and 4 or 6,and 7.

The other parameters are for adjusting the X and Y axis. The minimum and maximum value for each axis scale and also the spacing between scale markings may be adjusted. If the X *axis is radius and a log scale is chosen,* the interval for the X axis is not used so changes to the interval will have no effect on the plot. The paper size is normally set for the smaller plotters and 8.5 X 11 paper. If a larger plotter is used the paper length may be changed to 14 for 11 X14 paper.

Now that the plotting parameters and file names have been entered, the data may be plotted. It is recommended to first plot the data on the computer screen to insure the parameters are set correctly before starting a more time consuming plot on a plotter. If a "TRUE" appears after "D - Clear Graphics Screen", the graphics screen will be cleared before the new data is plotted. To plot a second set of data on top of the previous plot, make sure it says "FALSE". At any time after the axis are drawn pressing the ESC key will discontinue the plot. The computer will stop plotting and by pressing another key, the plotting menu will reappear. This escape feature only works while plotting on the computer screen.

To make the plot on paper, choose B to transfer the data to HP Plotter. HP Plotter will plot the data on the plotter and then return the data to Plotter.

To load a different sample or a different directory of files from the same sample, choose F. The program will start over by prompting the user to select a file directory.

Select E to return to the Main Menu.

STRUCTURE : The user is first prompted to select the directory of the files to plot. When the file name is entered (must include sample name, cycle number, and portion of the curve) data isn't loaded but it is ready to be loaded during plotting. Two files may be loaded and plotted at the same time. The top file is plotted first followed by the lower.

The plotting parameters set the X and Y axis. If a plot with a log scale X axis is selected the X interval is not used by the computer. The computer automatically sets up a log scale plot from the minimum X to the maximum X, but if either of these values are not a power of ten the computer rounds down to the next lowest power of ten. For example if the minimum X is set at 15 and the maximum is set at 15000 the computer will plot from 10 to 10000 with a log scale.

Line types for each of the files may be chosen from the following list.

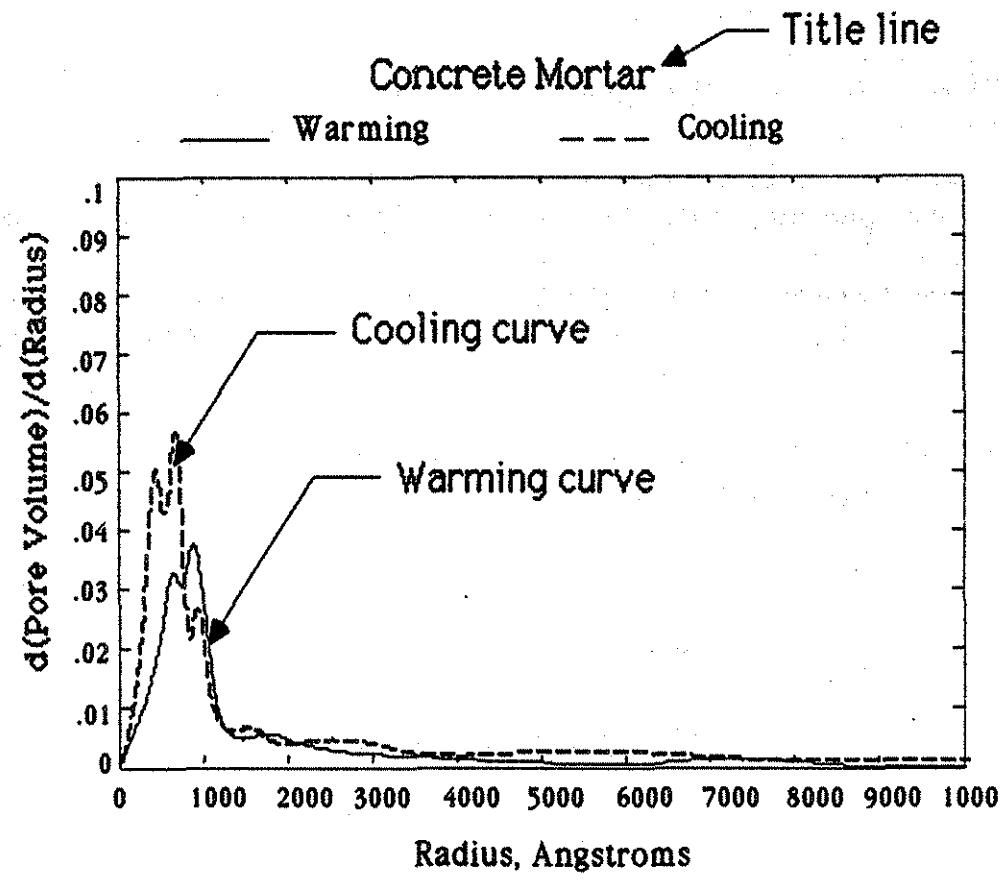
Pattern #	Line Pattern
0	only the data points are plotted
1	. . . . .
2	___ ___ ___ ___ ___
3	____ ____ ____ ____ ____
4	____ . ____ . ____ . ____ . ____ .
5	___ _ ___ _ ___ _ ___ _ ___ _
6	_ _ _ _ _ _ _ _ _ _ _ _ _ _ _ _
7	________________________________
	→| |← one pattern length

The arrays are passes to HP Plotter when a hard plot is desired.

HP PLOTTER, [Hpplot], (Plotter), (none)

PURPOSE : HP Plotter receives the parameters passed from Plotter and makes a plot on a plotter.
OPERATION : HP Plotter requires no user input.

STRUCTURE : The file names and parameters are passed to HP Plotter by Plotter. HP Plotter then initializes the Plotter and begins to draw the plot. A sample plot is shown below.



CONVERT XY, [T.C.to.X.Y], (Main Menu), (raw data file, X.Y)

PURPOSE : Convert XY reads in the raw temperature vs. conductance data from the disk and converts it to the X vs. Y format for further processing.

OPERATION : Convert XY requires no user input.

STRUCURE : Convert XY is the first in a string of data processing programs. Therefore Convert XY is responsible for loading the data from the disk and converting it into the format required for the rest of the processing programs. The Main Menu sends the file name to Convert XY. Convert XY loads the raw data from the warming and cooling files into the two-dimensional arrays A and B for the temperature and the conductance respectively. The first dimension of the array indicates whether the data are from the warming (0) or cooling (1) portion of a cycle. The second element is the numerical value of the data.

The raw data are converted to the X vs. Y format where

$$X = (-1 / T) * 10000$$

and T is the temperature in degrees Kelvin. During the conductance test you a resistor may be connected in series with the sample to narrow the conductance range of the test. This allows a lower scale on the conductance meter to be used and thus resolution is better at lower temperatures where low conductances occur. During the calculation of Y values the effect of the conductance of the series resistor is compensated for and the conductance of the sample alone is determined. The following relationships apply.

$$Y = \log (CT), \text{ and}$$

$$CT = (C * R) / (C - R),$$

where R is the conductance of the resistor in µS, and C is the conductance of the resistor and sample in µS. With the effect of the resistor is removed, CT is the conductance of the sample.

The new X vs. Y data are stored in the X.Y directory and the data are passed back to the Main Menu for further processing.

DERIVATIVE XY, [Der.X.Y], (Main Menu), (X.DYDX)

PURPOSE : Derivative XY calculates the derivative of Y with respect to X, dY/dX, for both the warming and the cooling data. The derivative algorithm also smooths the data using the moving average set in the Main Menu. The relationship between the derivative, dY/dX, and the samples pore volume is expressed in equations 7 and 8 in the Introduction.

OPERATION : Derivative XY requires no input by the user.

STRUCTURE : The data are passed from Main Menu through arrays A and B for X and Y respectively. The derivatives, dY/dX, are calculated and stored in the conductance array, therefore the conductance data in the array are lost, but the data are still in the disk file. The derivative is found with an algorithm that uses a moving average to smooth the data somewhat. The moving average (MA) is set in the Main Menu. If the user has chosen to save the data, they are stored in the X.DYDX directory. When the program is finished the data are passed back to the Main Menu for further processing.

INTEGRATE XDYDX, [Int], {Main Menu}, (X,Z)

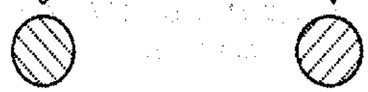
PURPOSE : Integrate XDYDX perform 2 integrations on the X, DYDX data, resulting with the calculation of the parameter Z expressed in equation 8 of the Introduction.

OPERATION : Integrate XDYDX requires no user input.

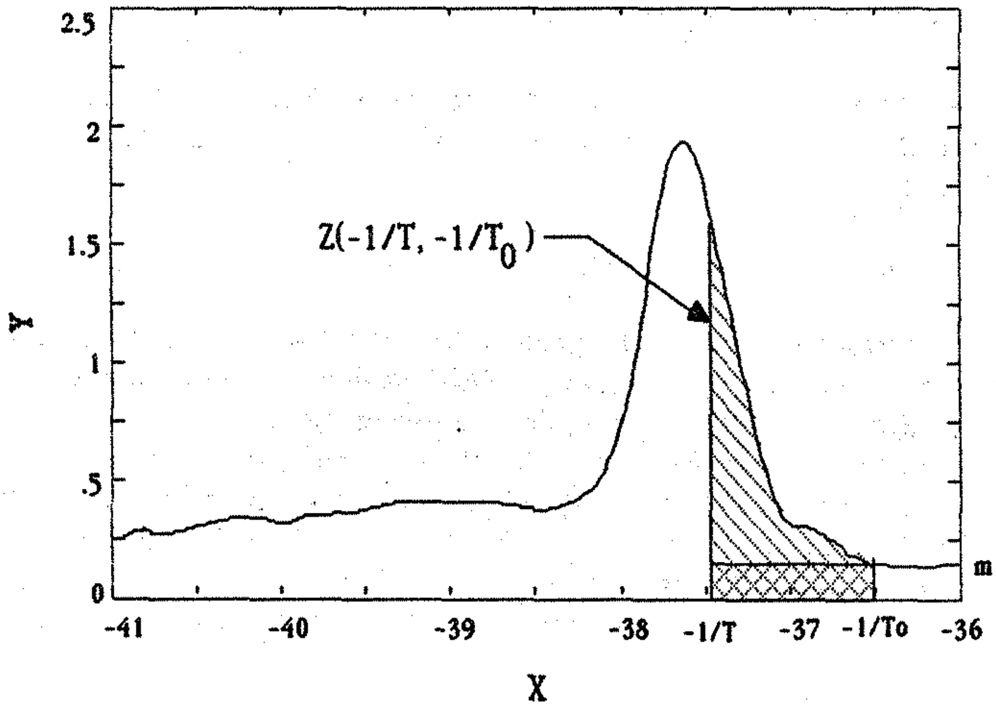
STRUCTURE : The arrays A and B are passed from the Main Menu containing the information X and dY/dXrespectively. The Z values are cumulative totals of the integral and replace the dY/dX values in the array B.

The warming file is processed first with the data pairs processed sequentially from right to left on a X vs. dY/dX plot (the larger X value pairs first). The Z value for all pairs with an X greater than -36.5 are set equal to 0. The average value of Y on the dY/DX interval from -36 to -36.5 is found and stored in Y1 as the slope m. For the rest of the file the Z value is calculated using the following formula:

$$Z\left(\frac{-1}{T},\frac{-1}{T_0}\right) = \underbrace{\int_{\frac{-1}{T}}^{\frac{-1}{T_0}} \frac{d(\log C)}{d\left(\frac{-1}{T}\right)}\, d\left(\frac{-1}{T}\right)} - \underbrace{\int_{\frac{-1}{T}}^{\frac{-1}{T_0}} m \, d\left(\frac{-1}{T}\right)}$$

where 

The calculations are graphically shown on the graph below.



The cooling portion is processed next, in a left to right manner. The same base m is used on the cooling curve. The data pairs with a dY/dX value greater than -36.5 receive the Z value of the last pair less than -36.5. When the cooling curve is complete the data are passed back to the Main Menu and stored in the directory X.Z if the user has directed the computer to save the X.Z File in the Main Menu.

CONVERT VP, [X.VP.from.X.Z], {Main Menu}, (X.VP)

PURPOSE : Convert VP calculates the total conducting pore volume from the X vs. Z data. The total conducting pore volume is the ratio of Vcp / Vp as shown in equation 10 in the Introduction.

OPERATION : Convert VP requires no user input.

STRUCTURE : The data are passed into Convert VP via the arrays A and B for X and Z respectively. The pore volume is found using the equation,

$$V_p = 1 / 10^{Z}$$

and replaces Z in the B array. If the user chooses to save the data, they are stored in the X.VP directory. The data are passed back to the Main Menu when the program has completed the calculations.

CONVERT R, [R.VP.from.X.VP], (Main Menu), (R.VP)

PURPOSE : Convert R changes the X variable to a pore radius. Relative conducting pore volume is now expressed as a function of radius, as shown on the left hand sides of equations 9 and 10 in the Introduction.

OPERATION : Convert R requires no user input.

STRUCTURE : The data are passed to Convert R via two arrays, A and B containing X and VP respectively. The X data are converted from a temperature to radius using the equation

$$R = \frac{-2 * T_0 * (.25 - (29 * X) + (.25 * T_0 * X))}{(.9998 * 3.335 \text{ X } 10^9) * (1 + T_0 * X)} * 10^8$$

where T_0 is the pore solution melting point (273.15 °K for water) which is entered in the Main Menu and X is the X variable. All data points with an original temperature greater than 0 °C are meaningless and removed from the arrays. If you have chosen to "thin" the file, the program only saves points that are spaced at least one Angstrom apart. The data are saved in the R.VP directory on the data disk and then passed back to the Main Menu for further processing.

DERIVATIVE VP, [Der.R.VP], (Main Menu), (R.DVPDR)

PURPOSE : Derivative VP calculates the derivative of Vp with respect to R, dVp/dR, for both the warming and the cooling data. The derivative algorithm also smooths the data using the moving average entered in the Main Menu.

OPERATION : Derivative VP requires no input by the user.

STRUCTURE : The data are passed from Main Menu through arrays A and B for R and Vp respectively. The derivative, dVp/dR, is calculated but not stored in the conductance array. The arrays are passed back to the Main Menu without any changes so that the derivative with respect to the log(R) can be computed. The derivative is found with an algorithm that uses a moving average to smooth the data somewhat. The moving average (MA) is set in the Main Menu. The data are stored in the R.DVPDR directory. When the program is finished the R and Vp data are passed back to the Main Menu for further processing.

DERIVATIVE LVP, [Der.LR.VP], (Main Menu), (R.DVPDLR)

PURPOSE : Derivative LVP calculates the derivative Vp with respect to log (R), dVp/dLR, for both the warming and the cooling data. The derivative algorithm also smooths the data using the moving average entered in the Main Menu.

OPERATION : Derivative LVP requires no input by the user.

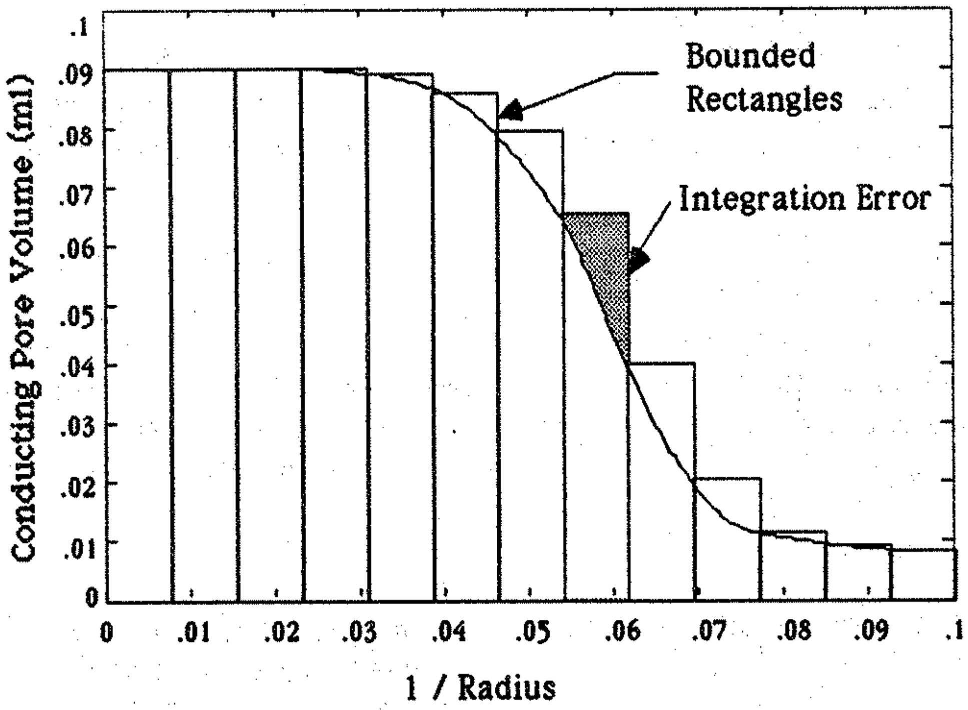
STRUCTURE : The data are passed from Main Menu through arrays A and B for R and Vp respectively. The radius is converted to the log (R) before any of the calculations and then the derivative of Vp is computed with respect to the $\log_{10}$ (R). The derivative, dVp/dLR, is calculated and stored in the conductance array. The derivative is found with an algorithm that uses a moving average to smooth the data somewhat. The moving average (MA) is set in the Main Menu. The data are stored in the R.DVPDLR directory. This is the last step in the data processing cycle and the program returns to the Main Menu when finished.

SURFACE AREA, [Int.Sa], (none), (R.VP)

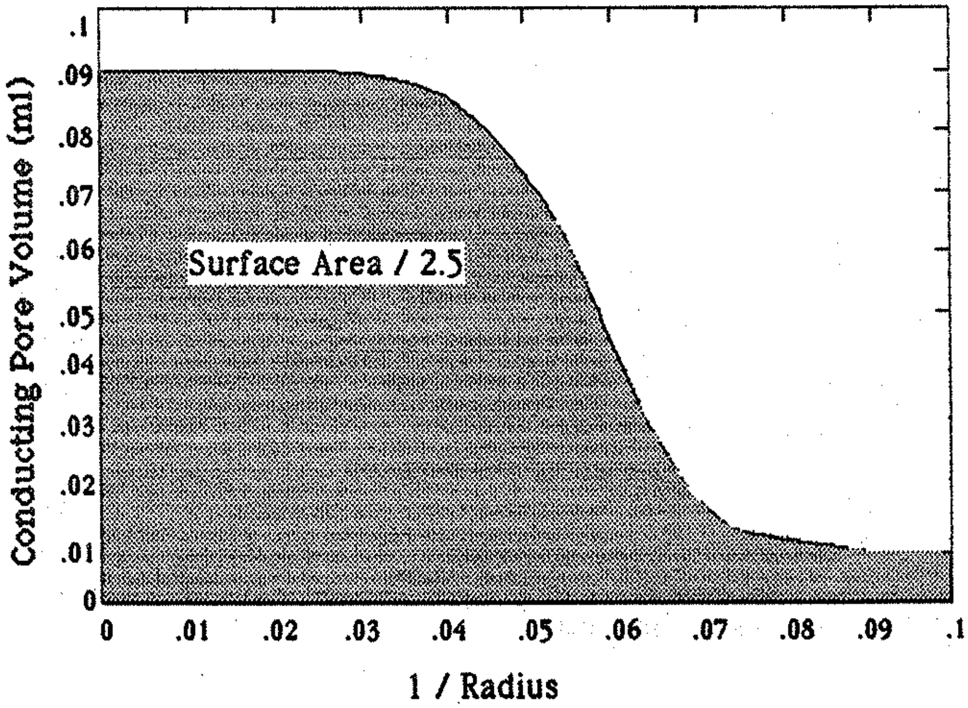
PURPOSE : Surface Area loads the radius vs. relative pore volume data and after the actual sample pore volume is entered calculates the surface area of the sample.

OPERATION : Surface Area is not offered as an option on the Main Menu, therefore to load and run the program, first exit the menu and type "RUN INT.SA,D1". Once the program is started, input a file name consisting of the base file name for the sample and the cycle number. After the R.VP file for the sample is loaded for the warming portion, enter the pore volume of the sample. The relative pore volume is multiplied by the actual total pore volume to get the pore volume at any given point. The computations will be performed and the surface area printed on the screen.

STRUCTURE : Surface area computations are only performed on the warming portion of the curve. Once the file name is entered, the pore volume data from the warming portion is loaded into an array for processing. Then a modified version of the data, changed to pore volume vs. 1/radius, is integrated from 0 to 1/12 using a method of rectangles. The change in 1/radius between points is multiplied by the height of the larger pore volume to get the area of a rectangle. The total integral is the sum of all of the rectangles. The area will be over estimated using this method but as the number of points increases and therefore the width of the rectangle decreases, the error becomes negligible. The integration method is illustrated on the next page. The width of the rectangles is exaggerated for clarity.



The surface area of the sample (excluding pores less than 12 Angstroms) is found to be the area under the curve multiplied by 2.5. The surface area is graphically represented on the next page.



The surface area is then printed to the screen and the program ended. To return to the Main Menu type "RUN STARTUP,D1"

TEST FORMAT, [Sample.Data], (Main Menu), (general sample file)

PURPOSE : Test Format prints out the test format, the cycle types and the cycles, for a test sequence.

OPERATION : To run Test Format enter a "D" in the Main Menu. Test Format will be loaded and the computer will prompt the user to insert the appropriate raw data disk. Enter the test format file name including the sample name, staring cycle number, and CT (for example Mortar.25.CT). The file will be loaded into memory and displayed on the computer screen. The user may choose to either quit and return to Main Menu or print the information on the plotter.

STRUCTURE : The data is loaded into an array and displayed on the screen. Due to limitations of screen size only 48 cycles may be displayed, the cycles over 48 will be loaded from the disk but not be shown on the screen. The program sends the data to the plotter.

CLOCK, [IO.Clock.Slot1], (Controller), (none)

PURPOSE : Clock generates a system clock that is accessed by Controller to maintain the rate of temperature change.

OPERATION : Clock requires no user input.

STRUCTURE : The operation of Clock is described in detail in Byte, March 1982 in an article by Ned Rhodes, the author of the program. The clock uses one of the 16-bit timers on the parallel interface card located in slot 1. The timer generates an interrupt every 1/60 of a second and calls Clock. Clock then displays the time on the screen and maintains a register which can be read by the program. The number in the register represents the number of interrupts since the clock was reset. Controller then divides the number in the register by 60 to get the number of seconds.

TEMP MEASUREMENT, [Cap.Res.2], {Controller}, (none)

PURPOSE : Temp Measurement determines the period of the square wave generated by the thermistor's oscillator. The period is used to determine the temperature of the sample.

OPERATION : Temp Measurement requires no user input.

STRUCTURE : Temp Measurement uses one of the 16-bit timers from the parallel interface card located in slot 1. The progam turns the timer on when it senses a rising edge on the signal. The timer then counts down until a falling edge is detected. The computer can then access the counter and subtract the initial value to get the half period of the wave.

CALIBRATE, [Calibrate], (none), (Cal)

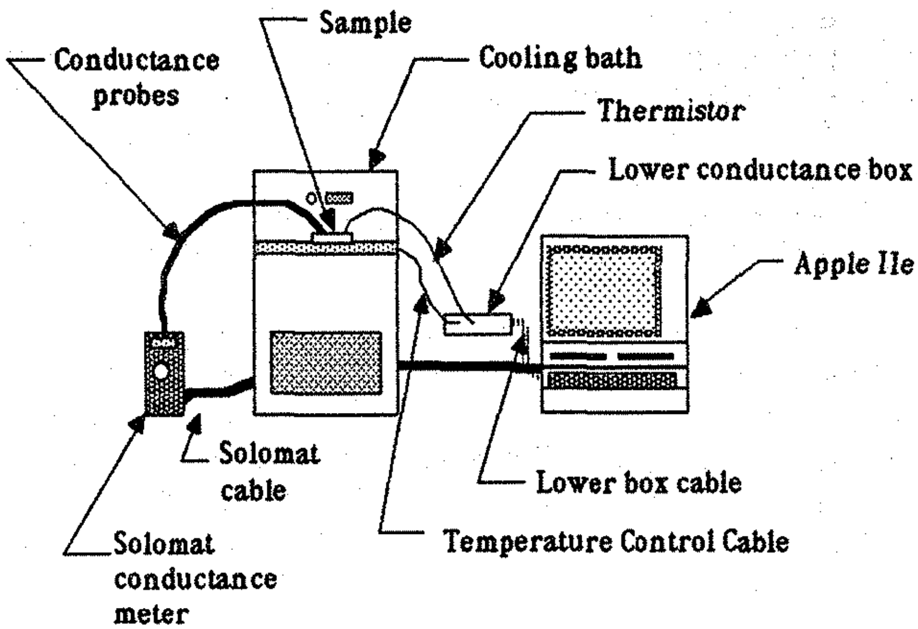
PURPOSE : Calibrate is a data editing program to allow the user to change the temperature calibration data for the thermistor.

OPERATION : Calibrate is not an option on the Main Menu. To load and run the program exit the Main Menu and type "RUN CALIBRATE,D1". The program will automatically load the Cal file and display it on the screen. The method of determining the calibration data is described in the Appendix. By using the arrows the cursor can be moved around the screen. When the cursor is at the desired location, type in the new data and press RETURN. The new data will then replace the old in the file located in the computer. When editing is completed the new version of the file in the computer may be saved to the disk, by pressing CTRL and S. If the user doesn't want the changes made to the data to be save pressing CTRL and Q will exit the program without saving the changes (NOTE : the data in the disk file is NOT changed in this case). In both cases you are returned to the Main Menu when finished.

STRUCTURE : The Cal file is automatically loaded into an array when the program is started. The user is then allowed to change the data in the array by moving the cursor on the screen to the data to be changed and entering the new data. The number of data points in the file is set at 22 and may not be changed using this program. When editing is finished the data is either written back to the file on the disk or discarded. The program returns to the Main Menu when completed.

IV. INTERFACE

The Conductance Porosimeter system consists of a Solomat conductance meter, an Apple IIe (with 2 disk drives, a monitor, and 2 parallel interface cards), an interface box, and a cooling bath. This section will mainly discuss the interface box but will also look at some of the aspects of the rest of the system. The main components are shown below.



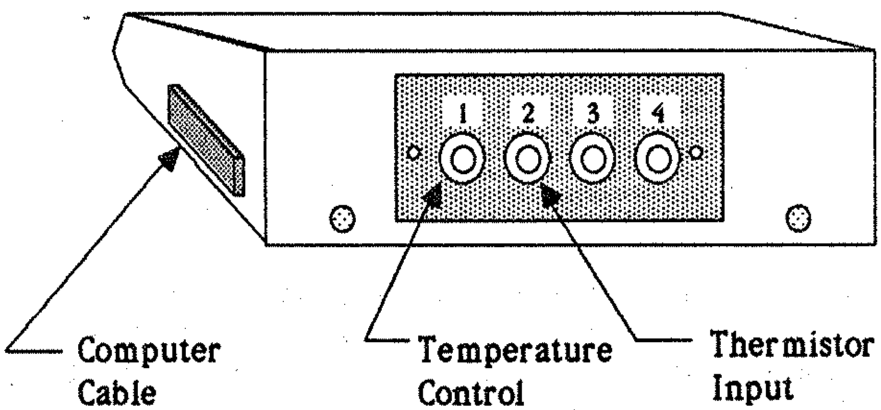
The Solomat measures the conductance of the sample and sends the digital data to the parallel interface card located in slot 4 of the Apple IIe. The temperature is converted from the variable resistance of the thermistor to a variable frequency square wave. The period of the square wave is found by a timer on the parallel interface card located in slot 1 of the Apple. Control of the cooling bath is accomplished by the parallel interface card in slot 1 sending a digital number to the temperature control circuit in the interface box. The temperature control circuit converts the digital data to a voltage level which is sent to the cooling bath. The voltage level represents the temperature level of the bath.

The following sections will take a closer look at each of these main functions of the interface.

INTERFACE BOX

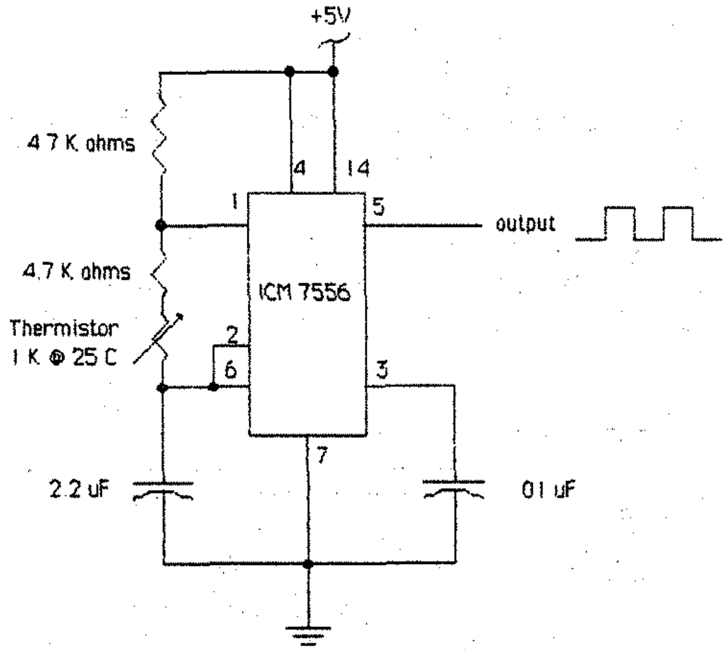
PURPOSE : The interface box provides the circuitry to control the cooling bath and record temperature readings.

OPERATION : The interface circuits are housed in a low profile box with connections for the computer cable, the thermistor, and the temperature control cable. The cables should be connected as shown.



STRUCTURE : There are two main components of the interface box, the thermistor and temperature control circuits. The thermistor circuit converts the variable resistance of the thermistor to a variable frequency square wave. The temperature control circuit converts the digital output from the computer to a voltage level that is sent to the cooling bath.

The main component of the thermistor circuit is a 556 timer chip. The chip is wired as a square wave oscillator with its frequency dependent on the capacitance and resistance on pins 6,1 and 4. Since the resistance of the thermistor is dependent on temperature, and the frequency of the oscillator is dependent on resistance, the frequency is a function of the temperature of the thermistor. The square wave is sent to the parallel interface card in slot 1. The thermistor circuit is shown on the next page.

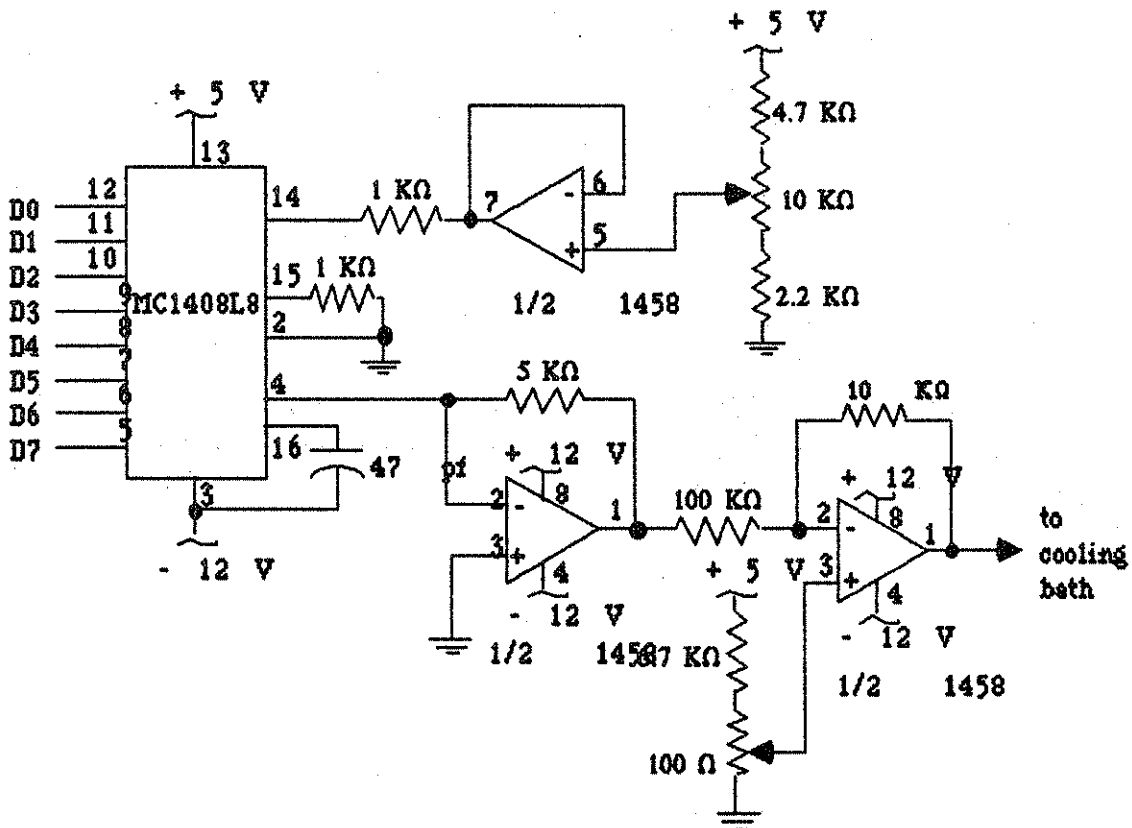


The temperature control circuit consists of a Digital-to-Analog (D/A) converter and three operational amplifiers (op amps). The temperature control output from the computer is in the form of a number from 0 to 255 corresponding to 7 and -33 °C respectively. The 8-bit number is transferred through the parallel interface card in slot 1 of the Apple. Lines PA0-7 are used to represent the 8 bits (see the parallel interface card section). The 8-bit number is converted to an analog voltage output by the D/A converter (MC1408L8).

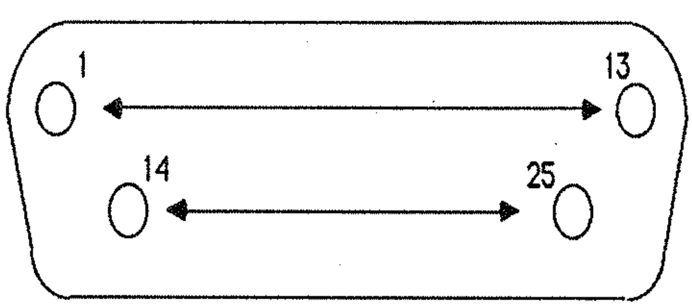
Adjusting the voltage on pin 14 of the D/A converter controls the range of voltage from the maximum to minimum inputs. The voltage on pin 14 is controlled a the voltage divider and op amp. Adjusting the 10 KΩ trim pot changes the voltage sent to the op amp. The op amp is configured as a voltage follower (just maintains the voltage input) and is connected to pin 14.

The D/A converter outputs a voltage on pin 4. The output is then amplified by the first op amp and reduced by the second one in line. The 100 Ω trim pot controls the offset voltage of the second op amp. The offset voltage is used to move the output voltage range to the proper level. The output should be .07 to -.33 V corresponding to 7 and -33 °C respectively. In practice, the 10 KΩ trim pot is used to set the low voltage and the 100 Ω for the high voltage (see the Appendix for calibrating the circuit).

The output is then connected by cable to a socket on the back of the cooling bath. The format of the socket may be found in the cooling bath's manual. The schematics of the temperature circuit are shown on the next page.



The cable connecting the interface box to the computer is a DB 25 connector configured as follows. The cable is connected to the other DB 25 connector on the back of the Apple.



Pin #	Name	Description
1	+ 5 V	Power supply
2	+ 12 V	Power
3	- 12 V	Power
4	GND	Common or Ground
5	PA0	Data line 0 for temperature control
6	PA1	" "1 " "
7	PA2	" "2 " "
8	PA3	" "3 " "
9	PA4	" "4 " "
10	PA5	" "5 " "
11	PA6	" "6 " "
12	PA7	" "7 " "
13-21	Unused	
22	CA1	Square wave output of temp oscillator
23-25	Unused	

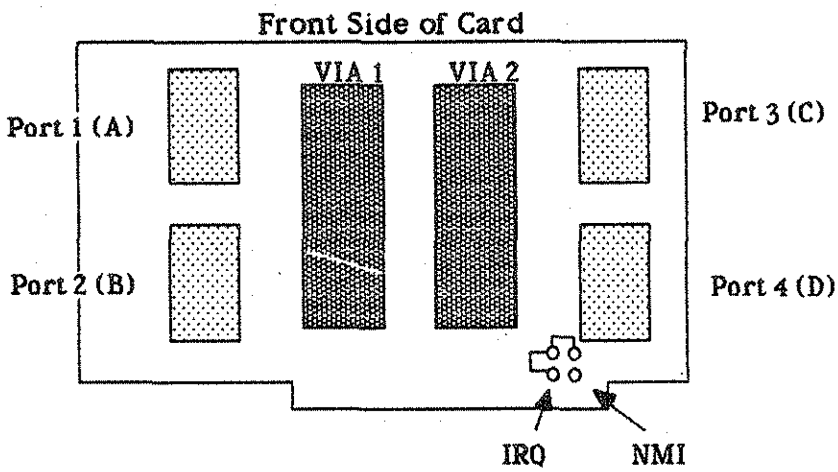
PARALLEL INTERFACE CARD

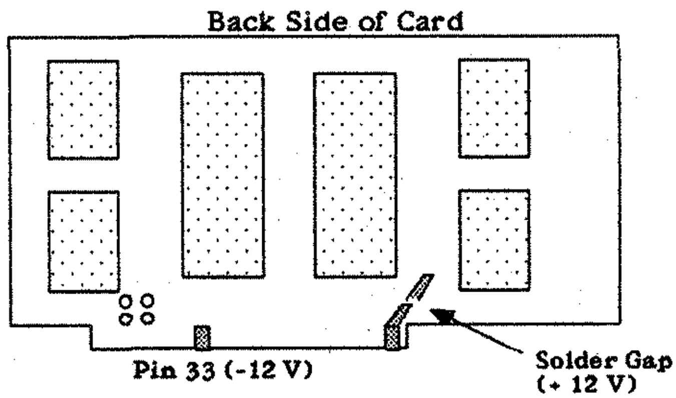
PURPOSE : The parallel interface card is used to input and output data in a parallel fashion (simultaneously on many lines rather than serially one bit at a time on one line), provide the system clock, and measure the period of the temperature signal square wave.

OPERATION : The parallel interface cards require no user intervention other than attachment of cables.

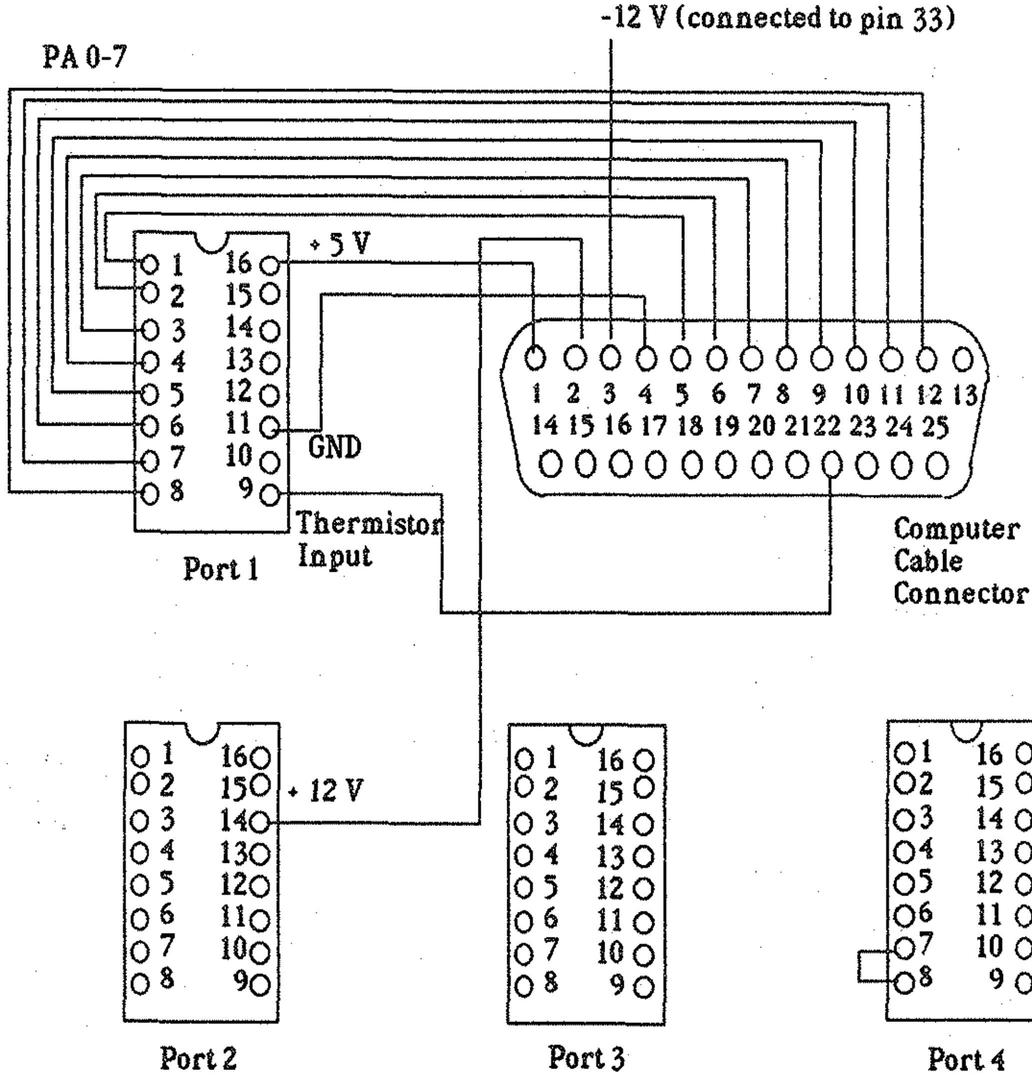
STRUCTURE : The parallel interface card used in this system is supplied by John Bell Engineering, 1014 Center St, San Carlos, CA 94070 (Tel: 415-592-8411). The card consists mainly of two 6522 Versatile Interface Adapters (VIA) with a total of four 8-bit bidirectional I/O ports and four independent 16-bit timers. More information may be found in the manuals supplied by the manufacturer.

Two parallel interface cards are used in the system. One of the cards in slot 1 of the Apple, controls the cooling bath, supplies the system clock, and measures the period of the thermistor's square wave. The other is located in slot 4 and is used to interface the Solomat conductance meter.



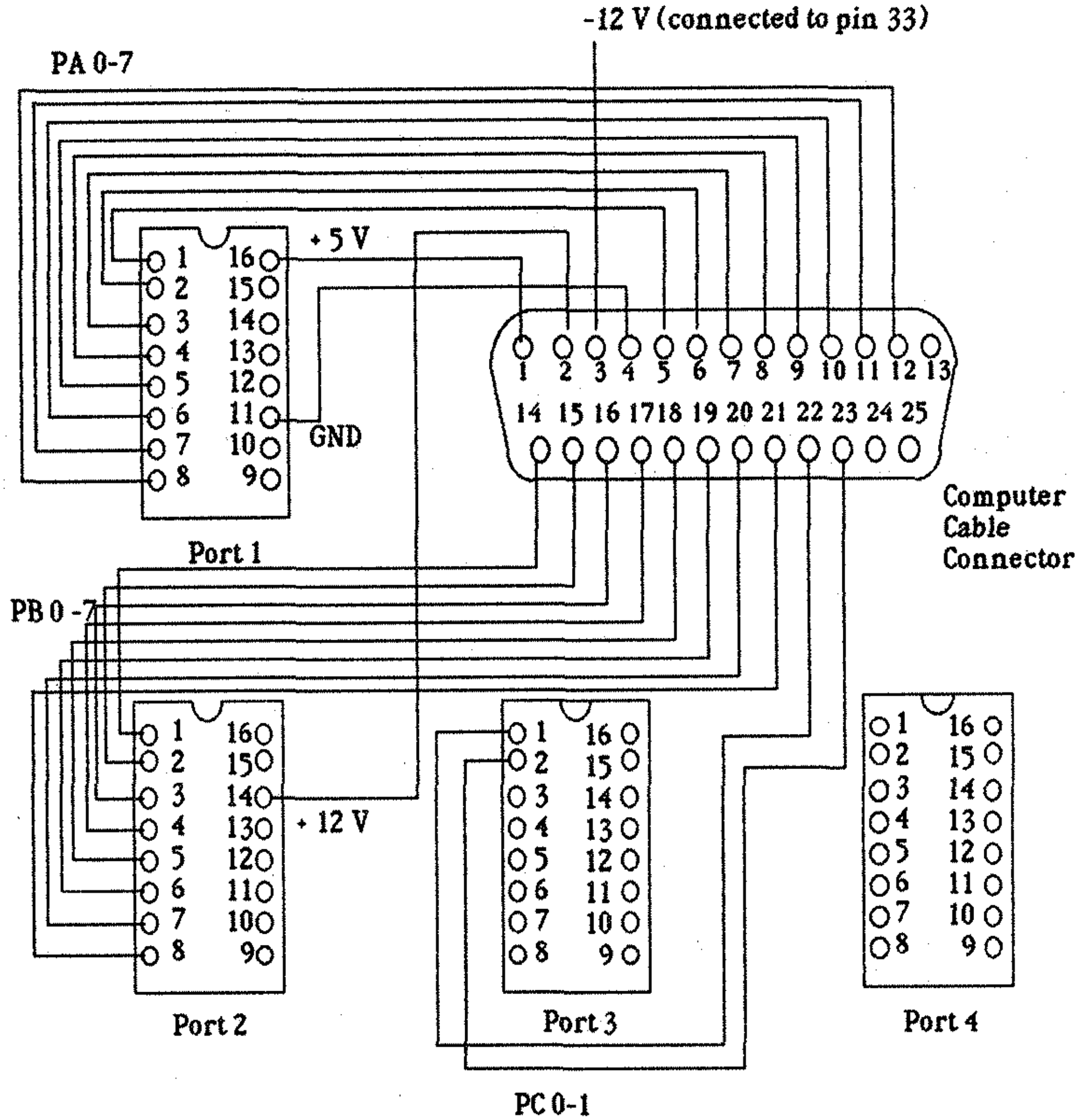


The card located in slot 1 generates the system clock through a program published in Byte, March 1982 by Ned W. Rhodes (Clock). Using the Apple's interrupts one of the 16-bit timers is triggered every 1/60 of a second. The program then reads the time from the timer. This is why the IRQ and NMI must be configured correctly. The period measurement for the thermistor is accomplished by triggering a timer on the rising edge and stopping on the falling edge. The timer counts down while operating and the program reads the timer. The temperature control data is simply passed in parallel through port 1. The connections are as shown, note the connector on port 4 and the fact that the -12 V line must be directly connected to pin 33.



Parallel Interface in Slot 1

The parallel interface card in slot 4 is used exclusively for interfacing with the Solomat. The Solomat is powered by the 5 volt line from the parallel interface card. The data is then sent back in parallel through ports 1, 2, and 3. The connections should be made as shown.



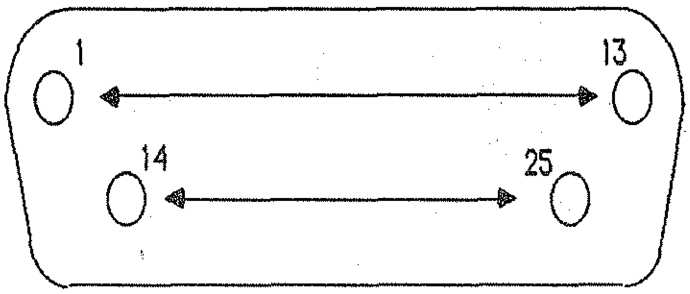
Parallel Interface Card in Slot 4

SOLOMAT CONDUCTANCE METER

PURPOSE : The Solomat conductance meter measures the conductance of the sample and sends the digital data to the Apple.

OPERATION : The conductance cables should be attached to the probes in the sample and the cable from the computer plugged into the socket on the side of the meter. The conductivity scale should be set to 16000 µS for the 1.0 cell unless a series resistor is used. A resistor is sometimes put in series with the sample to give better resolution at the lower end of the conductance cell. If a series resistor is used set the scale to 160 µS again for the 1.0 cell. The meter is powered by the computer and doesn't need to be switched on.

STRUCTURE : The Solomat is described in detail in the manufacturer's manual. The configuration of the socket is described in the section 6. All five digits are passed through the parallel interface. Power is supplied through pin 19. The configuration of the DB 25 connector on the back of the Apple is shown below. The digits are sent in Binary Coded Decimal (BCD) requiring 4 bits per digit except for digit 5 (digit 1 being the least significant) which only requires 1 bit as it can only be a one or a zero.

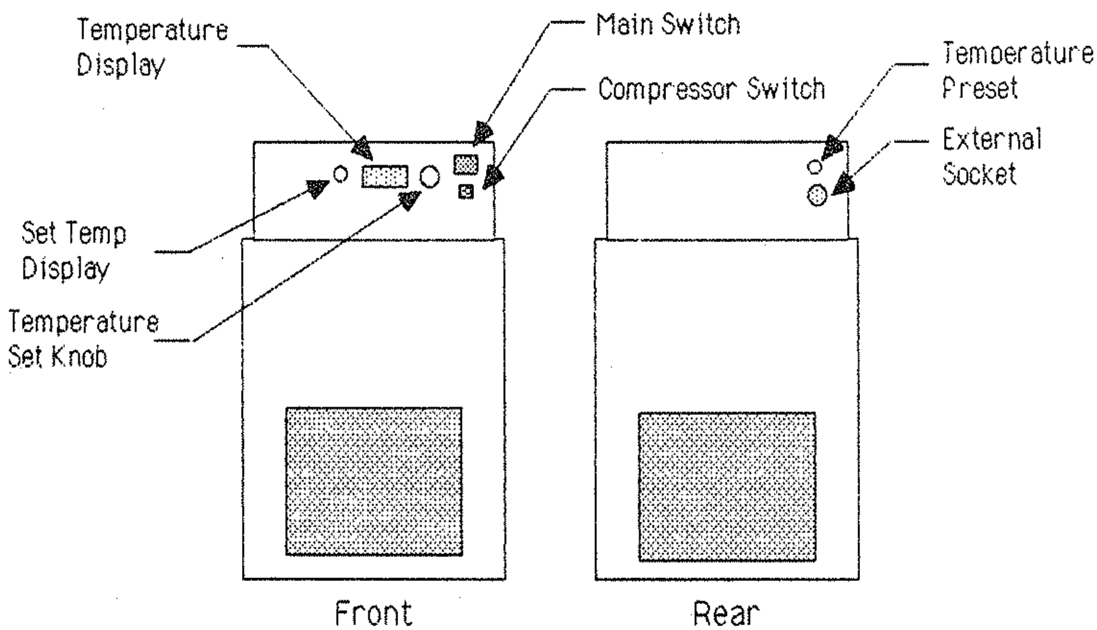


Pin #	Name	Description	
1	+ 5 V	Power supply	
2	+ 12 V	Power	
3	- 12 V	Power	
4	GND	Common or Ground	
5	PA0	Digit 1	Bit 0
6	PA1	Digit 1	Bit 1
7	PA2	Digit 1	Bit 2
8	PA3	Digit 1	Bit 3
9	PA4	Digit 2	Bit 0
10	PA5	Digit 2	Bit 1
11	PA6	Digit 2	Bit 2
12	PA7	Digit 2	Bit 3
13	GND	Common or Ground	
14	PB0	Digit 3	Bit 0
15	PB1	Digit 3	Bit 1
16	PB2	Digit 3	Bit 2
17	PB3	Digit 3	Bit 3
18	PB4	Digit 4	Bit 0
19	PB5	Digit 4	Bit 1
20	PB6	Digit 4	Bit 2
21	PB7	Digit 4	Bit 3
22	PC0	Digit 5	Bit 0
23-25	Unused		

COOLING BATH

PURPOSE : The cooling bath controls the temperature of the sample and allows the computer to cycle the sample through a freeze-thaw cycle while monitoring the conductance of the sample.

OPERATION : The controls of the cooling bath are shown below.

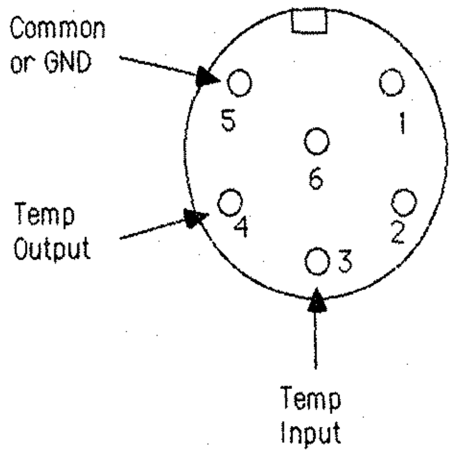


To turn on the bath, first place the main switch in the on position and then turn the compressor on. Turning them both on at the same time causes a large power surge on the power line. When turning the bath off turn off the compressor before turning the main power off.

The bath's display serves two purposes. It usually displays the current temperature of the bath, but when the set temp display button is pressed the display shows the temperature the bath has been set at. To set the bath temperature press the set temp display button and adjust the temperature set knob until the desired temperature is shown.

The external control socket is located on the rear of the bath. The cable from the temperature control circuit should be connected to the external socket. To place the bath under external control place the temperature preset switch in the ON position. The temperature of the bath will now be set by an external device, in this case the computer. A small mark will appear in the upper left hand corner of the bath's display indicating external control. The temperature the external device is setting may be shown on the bath's display by pressing the set temp display button.

STRUCTURE : The cooling bath contains a refrigerating unit and a circulator for the bath's fluid. The bath currently contains isopropyl alcohol. The bath is a Haake model A 82 supplied by Haake Inc., 244 Saddle River Road, Saddle Brook, NJ 07662. For further information of the bath see the manufacturer's manual. The format of the external socket is as shown below.



The temperature voltage is sent into the bath using pin 3. The output pin is not used but could be used to externally monitor the bath's temperature. Both the output and the input represent temperature with voltages, 0.00 volts corresponding to 0 °C and 1.00 volts for 100 °C. Therefore for our range of 7 and -33 °C, we use .07 and -.33 volts respectively.

THERMISTOR

PURPOSE : The thermistor is a temperature dependent variable resistor used to measure the temperature of the sample.

OPERATION : The thermistor requires no intervention.

STRUCTURE : The thermistor used in model SP85DA102FA1 supplied by Thermometrics, 808 U.S. Highway 1, Edison N. J. 08817. It is .085" in diameter and 1/2" long with a nominal resistance of 1 kΩ at 25 °C and stable to ± .02% per year.

V. APPENDIX

INITIALIZING DISKS

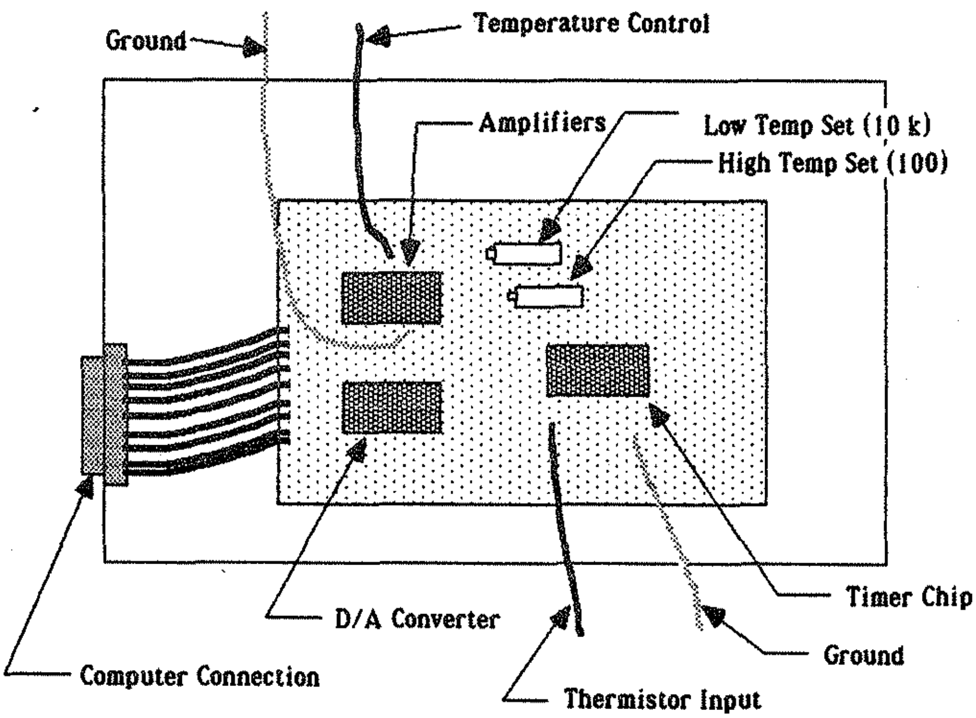
The data disks used by this system need to be initialized and directories established before data may be stored on them. To initialize a disk the user needs the ProDOS User's Disk and the /Caldat master disk. Insert the ProDOS User's Disk in drive one and press the CTRL and open-Apple, and RESET keys to reboot the system. The main menu for the ProDOS User's Disk will be displayed on the screen. Select F for ProDOS Filer (Utilities). This is a set of programs to allow the manipulation of disks. The next menu will be the ProDOS Filer menu. This time select V for Volume commands. This will further define the programs to those that manipulate entire disks rather than single files. When the Volume Commands menu is displayed select C for Copy a Volume. This program will copy the contents of an entire disk to another. This program will be used to copy the contents of the /Caldat master disk to the blank disk. The Copy a Volume screen will be displayed next. The program prompt the user to enter "THE VOLUME IN SLOT : " with a "6" being displayed. The program is asking what slot the disk drive controller card is in which in this case is the default value, slot 6. The copying will be done from drive 1 to drive 2 so answer the prompts respectively. Place the /Caldat master disk in drive 1, the blank disk in drive 2 and press RETURN. The volume name "/Caldat" will appear for the new volume name. Press RETURN to accept this as the new volume name for the blank disk. The program will format the blank disk and proceed to copy the contents from the /Caldat master disk to the blank disk. When the program is finished remove the disks and reboot the system with the porosimeter programs disk or initialize more disks. It is a wise idea to have a number of initialized data disks available.

CALIBRATING TEMPERATURE CONTROL

As described in the interface box section, the temperature of the bath is controlled by sending a number from 0 to 255 representing 7 and -33 °C respectively. The interface converts the number to a voltage level that is sent to the bath. To set the proper voltage levels the adjustment of two trim pots in the interface box will be necessary to send the appropriate maximum and minimum numbers.

The program used to calibrate the temperature control circuit isn't an option on the Main Menu. To run the program exit the menu and type "RUN I.O.CARD.TEST,D1". This program will allow the user to send a number from the parallel interface card to the interface box. The program will prompt the user to enter the port, enter 1 for port 1. Then enter the letter O for output on port 1. Only port 1 needs to be addressed as this is the port used to send the data to the interface box, so type a number 0 to begin the calibration.

The user will now need to remove the cover of the interface box to expose the trim pots that will be adjusted.



Turn the cooling bath on (the compressor doesn't need to be running) and place the external temperature preset switch (see cooling bath section) to the ON position. By pressing the temperature set button on the front of the bath (and holding it in), the temperature the control circuit is sending in will be shown on the bath's display.

To set the trim pots, alternate between sending a 0 and a 255 to the temperature control circuit and adjusting the appropriate trim pot so that the correct temperature will be displayed on the bath. Enter 1/0 into the computer for port 1 with a 0 output. Then adjust the 100 Ω trim pot (High Temp Set) until 7 °C is shown on the display. Now enter 1/255 and adjust the 10 kΩ trim pot (Low Temp Set) until -35 °C is displayed. Repeat these steps until no adjustments to the trim pots need to be made.

Replace the cover on the interface box. To return to the Main Menu type "RUN STARTUP,D1" and the calibration is completed.

CALIBRATION OF THERMISTOR

New thermistors do not have the same temperature-resistance charateristices and individual thermistors themselves change over time. Therefore the calibration file may need to be updated occasionally. The thermistor circuit converts the variable resistance of the thermistor to a variable frequency square wave. The period of the square wave is then measured by the parallel interface card so that the program may read the period. The program converts the period back to temperature using the table of values in the Cal file.

The Cal file contains 22 period - temperature pairs. The controlling program reads a period and then searches the table. If the period is within the range of the table, the program linearly interpolates between the two closest points of the table to calculate the temperature of the sample. If the period is outside of the boundaries of the table, either the maximum temperature or minimum is assumed accordingly.

To find the 22 data points needed place a thermocouple and the thermistor in a test tube and immerse the tube in the cooling bath. The thermocouple is used to determine the temperature of the thermistor. Exit the Main Menu and type "RUN TEMP.PERIOD,D1" to load a program that will display the square wave's period. Two numbers will be shown on the screen, the top number is the current reading of the thermistor and the lower a moving average of 30 readings.

Turn the bath on and set the temperature at 7 °C. When the temperature of the thermocouple stabilizes record the temperature and the period of the thermistor's square wave shown on the screen. This is the first data pair. Continue changing the bath's temperature and making readings until you have taken 22 pairs. The readings should be spread over the range of 7 and -33°C with a higher concentration near zero where the temperature is critical in the data processing. Press the ESC key to exit the program.

The program Calibrate will automatically be loaded. As described in the software section, Calibrate allows you to view and edit the Cal file. Enter the new data points into the Cal file. Make sure to keep the order straight and put the highest temperature pair at the beginning of the file (point 1). The algorithms in the controlling program assume this order. When finished save the file and return to the Main Menu. The calibration is now completed.

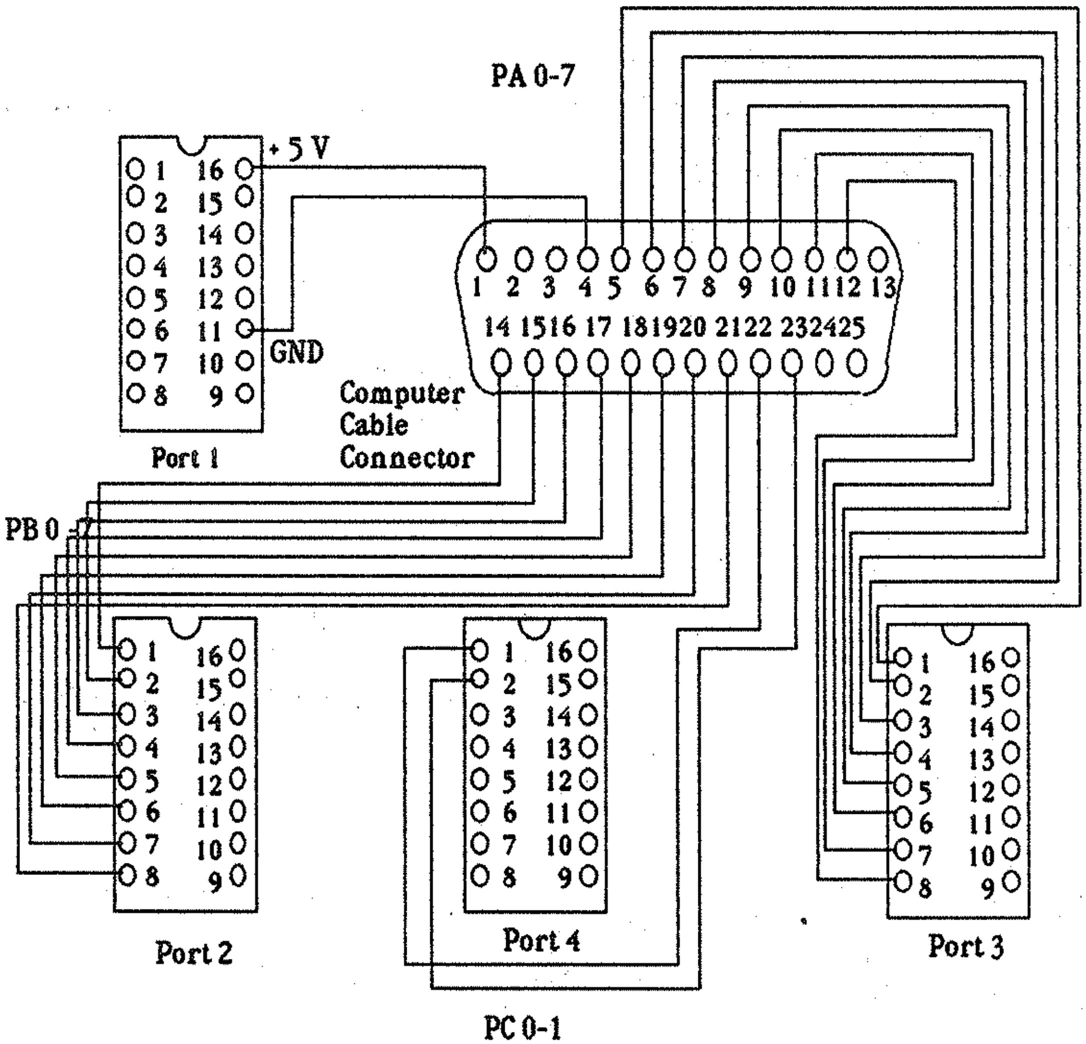
SIMPLIFICATIONS

The conductance porosimeter system uses two parallel interface cards because the original system was designed to handle both conductance PTP and phase transition porosimetry. In phase transition porosimetry the height of mercury in a capillary tube is monitored using capacitance. The variable capacitance was converted to a variable frequency square wave by a circuit similar to the one used for the thermistor. Because the amount of capacitance is so small, the noise from the temperature circuit necessitated two separate interface boxes and separate parallel interface cards. Therefore the conductance system can be set up so that only one parallel interface is used.

There are some changes to the programs and to the wiring to convert to only one interface card. The Solomat data must now be channelled through the card in slot 1 along with the thermistor input and the temperature control output. In the controlling program, Controller, change A1, B1 and C1 in lines 2040-2057 to these new values:

```
2040  A1 = -16256 + ISLOT * 256 + 1: B1 = -16384 + ISLOT * 256
2050  A3 = A1 + 2 : B3 = B1 + 2
2057  C1 = -16256 + ISLOT * 256 : C3 = C1 + 2
```

This changes the addresses the computer reads from the location in slot 4 to the card in slot 1. A1 is the location of port 1 and lines PA 0 - 7. It is used to send the digits 1 and 2 from the Solomat. Since the temperature control circuit uses port 1 of the card in slot 1 to send the control data to the temperature control circuit, A1 is also changed to port 3 and C1 (PC 0 or digit 5) is moved to port 4. The power line (+ 5V) remains with port 1. The +12 V line is not used and therefore eliminated. The computer connector for the interface box doesn't change but the new connector for the Solomat is as shown on the next page.


PA 0-7
+5 V
GND
Port 1
Computer
Cable
Connector
PB 0-7
Port 2
Port 4
Port 3
PC 0-1

VI. INDEX

CONDUCTOMETRIC PHASE TRANSITION POROSIMETER

USER'S MANUAL
Volume 2
Program Listings

CONDUCTOMETRIC PHASE TRANSITION POROSIMETER
USER'S MANUAL
Volume 2
Program Listings

LIST OF PROGRAMS

MAIN MENU

```
100  REM   *****************************************************
110  REM   *  MAIN MENU FOR COND CALC                          *
120  REM   *  DOES ALL OF THE CALCULATIONS IN ORDER            *
130  REM   *  BASED ON THE INPUT PARAMETERS SET HERE           *
140  REM   *****************************************************
150 D$ =  CHR$ (4): SPEED= 255: PRINT D$"PR#3"
160 SR$ = "Y":CR$ = "144.3":M1$ = "7":S1$ = "N"
170 S2$ = "N":S3$ = "N":K$ = "Y":M2$ = "3":C$(1) = "C":C$(0) = "W"
172 M3$ = "3"
175 TO$ = "273.15"
180  GOSUB 3000: REM  PRINT MENU OUT
190  VTAB 21: INPUT "Enter your menu choice :";MN$
200  IF MN$ = "" THEN 190
210  IF  LEN (MN$) > 1 THEN 190
220  IF  ASC (MN$) > 56 OR  ASC (MN$) < 49 THEN 250
230  ON  ASC (MN$) - 48 GOSUB 400,700,850,1000,1100,1200,1300,1362
240  GOTO 190
250  IF  ASC (MN$) < 65 OR  ASC (MN$) > 68 THEN 190
260  ON  ASC (MN$) - 64 GOTO 1400,1700,1800,270
270  END
400  REM   *************************************************
410  REM   *  ENTER THE FILENAME                           *
420  REM   *************************************************
430  VTAB 4: POKE 36,40
440  INPUT "";FIL$
450  IF FIL$ <  > "" THEN 520
460  POKE 34,4
470  HOME
480  ONERR  GOTO 600
490  PRINT D$"CATALOG /RAWDAT"
495  POKE 216,0
500  TEXT
510  GOTO 430
520  ONERR  GOTO 650
530  PRINT D$"OPEN "FIL$;"C,D2"
535  PRINT D$"READ "FIL$;"C"
537  INPUT Z
540  PRINT D$"CLOSE"
545  POKE 216,0
550  GOSUB 3000
560  RETURN
600  REM  NOT RAWDAT DISK
610  PRINT "Please insert the RAWDAT disk and press any key ...."
620  GET KB$
630  GOSUB 490
640  GOTO 190
650  REM  FILE NOT FOUND
655  PRINT D$"CLOSE"
```

MAIN MENU

```
660  VTAB 23
670  PRINT "File not found on drive 2..."
675  PRINT D$"DELETE "FIL$;"C"
680  GOSUB 430
690  GOTO 190
700  REM  ***************************************************
710  REM  *  SET X.Y PARAMS                                  *
720  REM  ***************************************************
730  VTAB 5: POKE 36,40
740  INPUT "";SR$
750  IF SR$ < > "Y" AND SR$ < > "N" THEN 730
760  IF SR$ = "N" THEN CR$ = "": GOSUB 3000: GOTO 800
770  VTAB 6: POKE 36,40
780  INPUT "";CR$
790  IF  VAL (CR$) < = 0 THEN 770
800  RETURN
850  REM  ***************************************************
860  REM  *  SET DERIVATIVE PARAMS                           *
870  REM  ***************************************************
880  VTAB 7: POKE 36,40
890  INPUT "";M1$
900  IF  VAL (M1$) < 3 THEN 880
910  VTAB 8: POKE 36,40
920  INPUT "";S1$
930  IF S1$ < > "Y" AND S1$ < > "N" THEN 910
940  RETURN
1000  REM  *************************************************
1010  REM  *  SET X.Z PARAMS                                *
1020  REM  *************************************************
1030  VTAB 9: POKE 36,40
1040  INPUT "";S2$
1050  IF S2$ < > "Y" AND S2$ < > "N" THEN 1030
1060  RETURN
1100  REM  ************************************************
1110  REM  *  SET X.VP PARAMS                              *
1120  REM  ************************************************
1130  VTAB 10: POKE 36,40
1140  INPUT "";S3$
1150  IF S3$ < > "Y" AND S3$ < > "N" THEN 1130
1160  RETURN
1200  REM  *************************************************
1210  REM  *  SET R.VP PARAMS                               *
1220  REM  *************************************************
1230  VTAB 11: POKE 36,40
1240  INPUT "";K$
1250  IF K$ < > "Y" AND K$ < > "N" THEN 1230
1260  VTAB 12: POKE 36,40
1270  INPUT "";TO$
```

MAIN MENU

```
1280  RETURN
1300  REM  ***********************************************
1310  REM  *  SET R.DVPDR  PARAMS                        *
1320  REM  ***********************************************
1330  VTAB 13: POKE 36,40
1340  INPUT "";M2$
1350  IF  VAL (M2$) < 3 THEN 1330
1360  RETURN
1362  REM  ***********************************************
1365  REM  *  SET R.DVPDLR PARAMS                        *
1370  REM  ***********************************************
1375  VTAB 14: POKE 36,40
1380  INPUT "";M3$
1385  IF  VAL (M3$) < 3 THEN 1375
1390  RETURN
1400  REM  ************************************************
1410  REM  *  RUN CALCULATIONS                             *
1420  REM  ************************************************
1422  IF FIL$ = "" THEN 190
1424  HOME
1428  PRINT "Performing the calculations..."
1430  REM  CALL T.C.TO.X.Y
1440 CR =  VAL (CR$)
1450  PRINT D$"CHAIN /CONPROG1/PROGRAMS/T.C.TO.X.Y"
1460  REM  CALL DER.X.Y
1470 MA =  VAL (M1$):SA = 1
1480  IF S1$ = "Y" THEN SA = 0
1490  PRINT D$"CHAIN /CONPROG1/PROGRAMS/DER.X.Y"
1500  REM  CALL INT
1510 SA = 1
1520  IF S2$ = "Y" THEN SA = 0
1530  PRINT D$"CHAIN /CONPROG1/PROGRAMS/INT"
1540  REM  CALL X.VP.FROM.X.Z
1550 SA = 1
1560  IF S3$ = "Y" THEN SA = 0
1570  PRINT D$"CHAIN /CONPROG1/PROGRAMS/X.VP.FROM.X.Z"
1580  REM  CALL R.VP.FROM.X.VP
1590  PRINT D$"CHAIN /CONPROG1/PROGRAMS/R.VP.FROM.X.VP"
1600  REM  CALL DER.R.VP
1610 MA =  VAL (M2$)
1620  PRINT D$"CHAIN /CONPROG1/PROGRAMS/DER.R.VP"
1625 MA =  VAL (M3$)
1630  PRINT D$"CHAIN /CONPROG1/PROGRAMS/DER.LR.VP"
1700  REM  *********************************************
1710  REM  *   CALL PLOT                               *
1720  REM  *********************************************
1730  PRINT D$"RUN /CONPROG1/PROGRAMS/P"
1800  REM  *************************************
```

MAIN MENU

```
1810  REM  *  RUN CONDUCTANCE TEST                *
1820  REM ************************************
1830  PRINT D$"RUN COND2,D1"
3000  REM   ******************************************
3010  REM   *   PRINT OUT THE MENU                       *
3020  REM   ******************************************
3030  HOME : PRINT
3040  HTAB 32: PRINT "CONDUCTANCE MENU"
3050  PRINT
3060  PRINT "1. Base Filename                           : ";FIL$
3070  PRINT "2. T.C to X.Y         Series Resistor  : ";SR$
3080  PRINT "                      Cond of Resistor : ";CR$
3090  PRINT "3. X.Y to X.DYDX      Moving Average   : ";M1$
3100  PRINT "                      Save File        : ";S1$
3110  PRINT "4. X.DYDX to X.Z      Save File        : ";S2$
3120  PRINT "5. X.Z to X.VP        Save File        : ";S3$
3130  PRINT "6. X.VP to R.VP       Thin File Out    : ";K$
3135  PRINT "                      Temp of T0       : ";T0$
3140  PRINT "7. R.VP to R.DVPDR    Moving Average   : ";M2$
3150  PRINT "8. R.VP to R.DVPDLR   Moving Average   : ";M3$
3155  PRINT : PRINT
3160  PRINT "  A.  Calculate the data"
3170  PRINT "  B.  Plot data"
3180  PRINT "  C.  Run Conductance test"
3185  PRINT "  D.  Exit to system"
3190  RETURN
```

CONTROLLER

```
10  REM   CONDUCTANCE CONTROLLER FOR ONE SAMPLE
11  REM    MODIFIED TO ONE SAMPLE ON 2 OCT 86
12  HOME
15 D$ =  CHR$ (4)
18  HIMEM: 256 *  PEEK (116) +  PEEK (115) - 1024
20  PRINT D$"BLOAD CAP.RES.2,A$340,D1"
30  PRINT D$"BLOAD IO.CLOCK.SLOT1,A$9800"
45  POKE 1535,0
50  POKE 1407,0
55  POKE 1279,0
60  CALL  - 26624
65  POKE 1919,1: REM    DISPLAYTIME
70  GOTO 2000
100  POKE 824,0: CALL 832
110  IF  PEEK (822) > 3 THEN 140
120 TV =  PEEK (823): IF TV <  > 255 THEN  POKE 823,TV + 1: GOTO 140
130  POKE 823,0
140 R = 65536 - ( PEEK (823) * 256 +  PEEK (822))
150 R = R - 25000: IF R < 0 THEN R = R + 40536
160 RR = RR + R - AR(SR):AR(SR) = R: IF MR > SR THEN SR = SR + 1: GOTO 180
170 SR = 1
180 TEMP =  INT (RR / MR + .5)
182  IF TEMP > P(NMPT) THEN TEMP = P(NMPT)
184  IF TEMP < P(1) THEN TEMP = P(1)
186  IF (TEMP < P(TI)) AND (TI > 1) THEN TI = TI - 1: GOTO 186
188  IF (TEMP > P(TI + 1)) AND (TI < NMPT - 1) THEN TI = TI + 1: GOTO 188
190 TEMP =  INT (((TEMP - P(TI)) * (T(TI + 1) - T(TI)) / (P(TI + 1) -
    P(TI)) + T(TI)) * 1E3 + .5) / 1E3
192  VTAB 6: HTAB 30
194  PRINT "           ";: HTAB 30
196  PRINT TEMP;
200 OSEC =  PEEK (1279) + 60 *  PEEK (1407) + 3600 *  PEEK (1535)
210  RETURN
300  REM  SWITCH LEADS AND READS CONDUCTANCE
310 PO = (PO -  INT (PO / 2) * 2) +  INT (PO / 4) * 4
320  POKE C1,PO
340 CX =  PEEK (C1):CY =  PEEK (B1):CZ =  PEEK (A1)
350 CX = (CX -  INT (CX / 2) * 2) * 10000
360 CY = (CY -  INT (CY / 16) * 6) * 100
370 CZ = CZ -  INT (CZ / 16) * 6
380 CND = CX + CY + CZ
390  RETURN
400  REM  CALCULATE TEMP CONTROL INFO
410 OT =  INT ((M1 * S9 + B) + .5)
420 PT =  INT ((M1 * F9 + B) + .5)
430 TS = 60 * ( ABS (F9 - S9) / (RA(TC(CCYCLE)) / 60)) /  ABS (OT - PT)
440 SD =  SGN (S9 - F9)
450 U6 = OSEC
```

EXPECTED SIGNIFICANCE OF THE PROJECT

It is expected that the proposed project will lead (1) to understanding the fundamental processes involved in the freezing of pore water in concrete, and on the basis of this, (2) to establishing criteria for assessing the frost susceptibility of concrete and thus to developing a simple and reliable testing method for predicting its performance in the field without resorting to tedious freeze-thaw cycles.

11. Philleo, R. E., "Freezing and Thawing Resistance of High-Strength Concrete," Report, NCHRP Project 20-5, Topic 16-05, (Jan. 1986).

12. Powers, T. C., "A Working Hypothesis for Further Studies of Frost Resistance to Concrete," Am. Concrete Inst. J., 41, 245-272 (1945).

13. Powers, T. C., and Helmuth, R. A., "Theory of Volume Changes in Hardened Portland Cement Paste During Freezing," Highway Res. Board Proc., 32, 285-297 (1953).

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1. A.S.T.M., Annual Standards, Part 14, 285-300 (1982).
2. Demirel, T., and Enüstün, B. V., "Ice Porosimeter," U.S. Patent 4,453,398; June 12, 1984.
3. Demirel, T., Enüstün, B. V., and Gunnink, B. W., "Development of a Conductometric Test for Frost Resistance of Concrete," Iowa Department of Transportation Project HR-272, Annual Report Phase II, Nov. 1, 1986.
4. Eckrich, J., Enüstün, B. V., and Demirel, T., "Phase Transition Porosimeter," American Laboratory, 80-92 (March, 1986).
5. Enüstün, B. V., Eckrich, J. and Demirel, T., "Phase Transition Porosimetry," Proc. of Condensed Papers, Int. Symposium-Workshop on Particulate and Multi-phase Processes and 16th Annual Meeting of the Fine Particle Soc., April 22-26, 1985, Miami Beach, Florida.
6. Enüstün, B. V., Enuysal, M., and Dösemeci, M., "Solubility of $SrSO_4$ in Capillaries," J. Colloid and Interface Sci., 57, 143-147 (1976).
7. Enüstün, B. V., Gunnink, B., Dubberke, W., and Demirel, T., "Frost Action in Rocks and Concretes," Final Report, Iowa Department of Transportation, Project HR-258, April 30, 1986.
8. Everett, D. H., "The Thermodynamics of Frost Damage to Porous Solids," Trans. Faraday Soc., 57, 1541-1551 (1961).
9. Everett, D. H., and Haynes, J. M., RILEM Bull., 27, 31 (1965).
10. Neville, A. M., Properties of Concrete, p. 463, London: Pitman, 1981.

structure on repeated freezing and thawing will be examined systematically in relation to the magnitude of the characteristic volume change on freezing.

The rate of water uptake of all samples will also be determined when they are immersed at a constant temperature. The time required to bring each sample to the corresponding characteristic (critical or mid-range) saturation will be noted. A frost susceptibility index may then be assigned to each sample, for instance, by dividing the measured characteristic volume change per unit volume of sample by the saturation time determined. Then the discriminating ability of the index will be evaluated.

The research plan also includes collaboration with the Iowa Department of Transportation to take samples from concrete pavements with good performance records, which therefore justifies the assumption that they have not suffered significant structural changes since their placement. These samples will be tested to determine their frost susceptibility index as described above. These numerical results will be compared with similar results previously obtained with the lab-made samples to make a final judgment on how well the proposed index indicates field performance.

pore water freezes in one step irrespective of the degree of saturation and will leave some liquid in these pores.

To achieve Purpose (2), we will determine the characteristic volume change per unit volume of each sample at critical saturation, if any, or else at mid-saturation range (i.e., mean of threshold saturation and 100%) in the case of the existence of a threshold saturation.

In a research project in progress [3], we have recently observed by electrical conductance measurements that a consistent and progressive shift in pore structure of concrete upon freezing and thawing at near-saturation is characteristic for its frost susceptibility. Therefore, the duplicate of each sample will be subjected to freeze-thaw cycles at critical saturation, or at mid-range saturation as the case may be. Then they will be subjected both to Phase Transition Porosimetry [2,4,5,7], developed in this laboratory to establish its pore structure and size distribution, as well as to mercury porosimetry. The former method comprises saturating the sample completely by applying vacuum or by boiling in water under reduced pressure and then measuring the volume changes by a dilatometer as a function of temperature while the sample is gradually frozen and thawed. This method is capable of providing pore-body-size and pore-neck-size distributions. If needed, scanning electron microscopy will also be used. An original triplicate sample will also be analyzed for pore-size distribution by these methods. This sample will serve as a control sample to establish structural changes caused by freeze-thaw cycles. The shifts in pore intermediate strength but not indicative of the performance of high-strength concretes as mentioned in the Introduction.

3. PROPOSED WORK

The proposed work has a dual purpose:

(1) To investigate the applicability of the two-stage model put forward above for the mechanism of freezing in concrete in near-saturated states. We would use dilatometric measurements in this investigation.

(2) To investigate the possibility of correlating the measured volume increase that accompanies bulk-ice-induced freezing in near-saturated states to frost susceptibility of concrete. Ultimately we hope to devise a laboratory testing method to predict the performance of concrete in the field more reliably than do the existing methods and to do so without resorting to tedious freeze-thaw cycles.

To achieve Purpose (1), we will prepare plain and air-entrained mortar samples with various water/cement ratios but with other compositional variables kept the same. These samples will be subjected to dilatometric measurements during freezing at a specified temperature and at various levels of saturation. If the plots of volume change versus saturation are of the type shown in Fig. 5 (signifying critical saturations), then the model is verified. However, if the plots indicate a threshold saturation rather than a critical saturation, as shown in Fig. 5 by the dotted line (a), it will mean that the freezable that nature), but would not fall strikingly below it. (The line corresponding to this is also indicated in Fig. 5 by (a). In other words, the Powers model based on one-stage freezing cannot explain the observed abruptness in frost resistance, as does the two-stage freezing model presently put forward.

The magnitude of volume change upon freezing a unit volume of the sample at a critical saturation and at a temperature compatible with the climatic conditions in the field is expected to be indicative of the frost susceptibility of a sample at this saturation. Another factor that should determine its performance in the field is the tendency of concrete to acquire this critical saturation under the prevalant saturating conditions. For instance, the rate with which an air-dried sample of a standard shape and size takes up water to reach critical saturation in an immersed state may be used as such a factor. An appropriate combination of the volume change and the rate of saturation may characterize the overall frost susceptibility in the field.

In as much as the entrained air spacing factor is important in protecting concrete from mild internal frost stresses below critical saturation, air entrainment in general is probably effective in protecting concrete from severe frost stresses at or above critical saturation. The reason is that incorporating air voids much larger than already existing capillary voids in concrete may retard its saturation and thus prevent it from reaching the critical saturation level and from being exposed to severe frost stresses. This may explain why the spacing factor is critical for the performance of concretes of

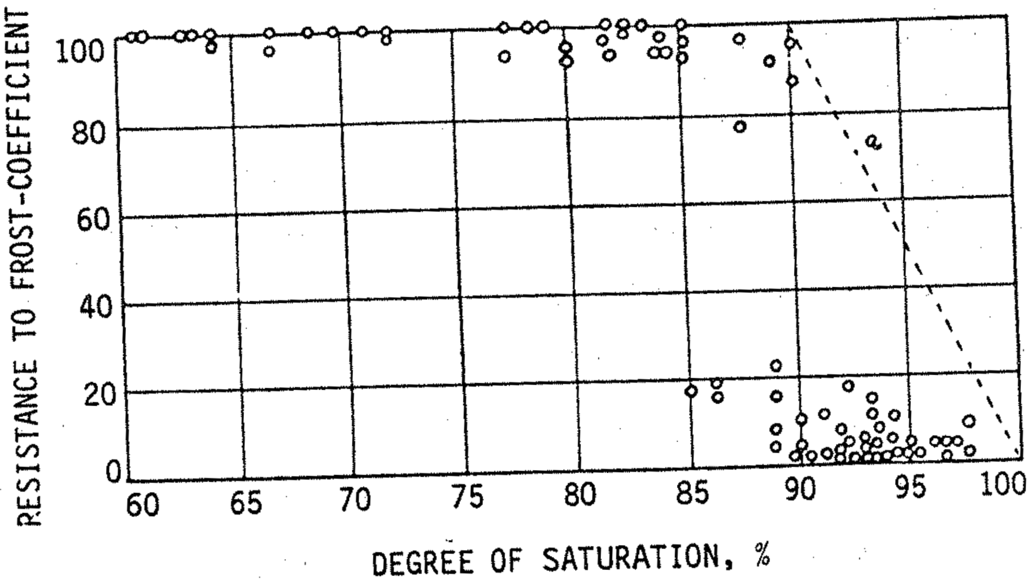

Fig. 6. Influence of saturation of concrete on its resistance to frost (Adapted from Ref. 10).

PROJECT DESCRIPTION

1. INTRODUCTION

Damage caused to concrete structures by the action of frost is one of the greatest concerns in civil and construction engineering practice in temperate climates. That frost damage to concrete is related to its pore structure is well known. During the past four decades a considerable amount of outstanding research has been done and much progress has been made on elucidating the mechanism of frost action in concrete.

Since the 1940s, air entrainment has been used to affect the pore structure of concrete and to render it nonsusceptible to frost damage. Although using air entrainment has proved effective, it has not prevented frost damage in some cases. The Powers theory [12,13] has been most successful in explaining the role of entrained air. In accordance with this theory, the use of the average spacing between air bubbles has been put forward [1] to characterize frost resistance. In general, the correlation is satisfactory for spacing factors less than 0.008 in. For larger spacing factors, however, the correlation breaks down as in the case of concretes of super low ratios of water to cement [11].

The Powers model of frost damage is valid as long as concrete is far from the state of saturation. In near-saturated states greater stresses, however, are developed within the structure than in the Powers model (see Section 2.2). Therefore, the question is whether air entrainment minimizes the frost damage by reducing the internal frost stresses as in this model or by protecting concrete from severe

PROJECT SUMMARY

The objective of the proposed work is to present a basic understanding of the freezing mechanism of pore water and on the basis of this understanding to establish criteria for assessing the freeze-thaw durability of concrete. Two mechanisms appear to control the freezing of pore water, namely, in situ nucleation and bulk-ice-initiated freezing. The first mechanism operates at low levels of saturation whereas the second controls the freezing process at high levels of saturation. The operation of the two mechanisms are reflected in the volumetric expansion of concrete upon freezing as a function of the degree of saturation. The damage to concrete depends on the internal cyclic stresses developed during freezing and thawing because of flow of water or ice.

The proposed work consists of a systematic study of the shifts in pore structure and expansion of the sample upon freezing as well as the rate of saturation of samples from the laboratory and the field to establish a correlation between these variables and the freeze-thaw durability of concrete.

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APPENDIX D

Frost susceptibility of concrete in near-saturated states

NSF project proposal

APPENDIX D

CALIBRATE

```
4050  PRINT "You must have a filename to save data.";
4060  GET KB$
4070  GOSUB 1300
4080  GOSUB 1900
4090  GOTO 530
4200  REM  ********************************************
4210  REM  *  ESCAPE SO EXIT OR NOT                    *
4220  REM  ********************************************
4230  POKE 34,23: POKE 35,24
4240  HOME
4250  PRINT "This will leave the data unchanged.  Continue (Y/N  Default =
      N).";
4260  GET KB$
4270  IF KB$ = "Y" THEN 9000
4280  GOSUB 1300
4290  GOSUB 1900
4300  GOTO 530
9000  REM  WE GONE BYE BYE
9010  PRINT D$"RUN STARTUP,D1"
```

```
3080  IF X = 2 THEN Y(N + Y - 5) =  VAL (KB$)
3090  GOSUB 1900: REM  RESET CURSOR
3100  PRINT  VAL (KB$);"     ";
3110  IF X = 1 THEN X = 2: GOTO 3140
3120  IF (Y - 5) + N <  > NMPT THEN Y = Y + 1:X = 1
3130  IF Y > 23 THEN N = N + 1: GOSUB 1700
3140  GOSUB 1900
3150  GOTO 530
3200  REM   *************************************************
3210  REM   *  LEFT ARROW                                   *
3220  REM   *************************************************
3230  IF X = 2 THEN X = 1: GOSUB 1900
3240  GOTO 530
3300  REM   *************************************************
3310  REM   *  RIGHT ARROW                                  *
3320  REM   *************************************************
3330  IF X = 1 THEN X = 2: GOSUB 1900
3340  GOTO 530
3400  REM   *************************************************
3410  REM   *  DOWN ARROW                                   *
3420  REM   *************************************************
3430  IF (Y - 5) + N <  > NMPT THEN Y = Y + 1
3440  IF Y > 23 THEN N = N + 1:Y = 23: GOSUB 1700
3450  GOSUB 1900
3460  GOTO 530
3500  REM   *************************************************
3510  REM   *  UP ARROW                                     *
3520  REM   *************************************************
3530  IF Y = 5 AND N <  > 1 THEN N = N - 1: GOSUB 1500: GOTO 3560
3540  IF Y <  > 5 THEN Y = Y - 1
3550  GOSUB 1900
3560  GOTO 530
3600  REM   *************************************************
3610  REM   * CONTROL-S  SAVE DATA                          *
3620  REM   *************************************************
3625  IF FIL$ = "" THEN 4000
3630  POKE 34,23: POKE 35,24
3640  HOME
3650  PRINT "Saving ";FIL$;" ...";
3660  GOSUB 2300
3670  POKE 34,0
3680  CLEAR
3690  GOTO 9000
4000  REM   *********************************************
4010  REM   *  TRIED TO SAVE WITHOUT A FILE NAME        *
4020  REM   *********************************************
4030  POKE 34,23: POKE 35,24
4040  HOME
```

CALIBRATE

```
1950  IF X = 2 THEN  POKE 36,45
1960  RETURN
2000  REM   *****************************************************
2010  REM   *  RETRIEVE FILE                                     *
2020  REM   *****************************************************
2030  PRINT D$"OPEN "FIL$",D1"
2040  PRINT D$"READ "FILE$
2050  INPUT NMPT
2060  DIM X(NMPT),Y(NMPT)
2065  IF CO = 1 THEN 2080
2070  INPUT XA$
2072  IF XA$ = "" THEN  INPUT XA$
2075  INPUT YA$
2077  IF YA$ = "" THEN  INPUT YA$
2080  FOR I = 1 TO NMPT
2090  INPUT X(I),Y(I)
2100  NEXT I
2110  PRINT D$"CLOSE"
2120  RETURN
2300  REM   ****************************************************
2310  REM   *  SAVE FILE                                        *
2320  REM   ****************************************************
2330  PRINT D$"OPEN "FIL$",D1"
2340  PRINT D$"WRITE"FIL$
2350  PRINT NMPT
2352  IF CO = 1 THEN 2370
2355  IF XA$ = "" THEN XA$ = "none"
2357  IF YA$ = "" THEN YA$ = "none"
2360  PRINT XA$: PRINT YA$
2370  FOR I = 1 TO NMPT
2380  PRINT X(I): PRINT Y(I)
2390  NEXT I
2400  PRINT D$"CLOSE"
2410  RETURN
3000  REM   **************************************************
3010  REM   *   ENTER THE DATA INTO ARRAY                     *
3020  REM   **************************************************
3025  PRINT KB$;
3030  GET IN$
3040  IF (IN$ >  = "0" AND IN$ <  = "9") OR IN$ = "." OR IN$ = "-" OR IN$ =
      "E" THEN KB$ = KB$ + IN$: PRINT IN$;: GOTO 3030
3042  IF  ASC (IN$) = 8 AND  LEN (KB$) = 1 THEN KB$ = "": PRINT  CHR$ (8);"
       "; CHR$ (8);
3044  IF  ASC (IN$) = 8 AND  LEN (KB$) > 1 THEN KB$ =  LEFT$ (KB$, LEN
      (KB$) -1): PRINT  CHR$ (8);" "; CHR$ (8);
3050  IF  ASC (IN$) = 13 THEN 3070
3060  GOTO 3030
3070  IF X = 1 THEN X(N + Y - 5) =  VAL (KB$)
```

CALIBRATE

```
1020  REM   ***************************************************
1030  POKE 34,0: REM  SET TOP TO LINE 0
1040  POKE 35,4: REM  SET BOTTO TO LINE 3
1050  HOME
1060  PRINT "Filename : ";: HTAB 40: PRINT "# of points : "
1070  PRINT "X-Axis Label : "
1080  PRINT "Y-Axis Label : "
1090  PRINT "Point #"; TAB( 30);"X";: POKE 36,50: PRINT "Y";
1100  POKE 34,4: POKE 35,23
1110  RETURN
1300  REM   **************************************************
1310  REM   *  PRINTS BOTTOM LINE                            *
1320  REM   **************************************************
1330  POKE 34,23: POKE 35,24
1340  HOME
1350  PRINT "Control-S to Save     Control-Q to Exit";
1360  POKE 34,4: POKE 35,23
1370  RETURN
1500  REM   **************************************************
1510  REM   *  DISPLAY A PAGE OF DATA (19 LINES)             *
1520  REM   *  N = THE DATA NUMBER TO START FROM             *
1530  REM   **************************************************
1540  HOME
1550  FOR I = N TO N + 18
1560  IF I > NMPT THEN 1590
1570  PRINT  TAB( 3);I; TAB( 25);X(I);: POKE 36,45: PRINT Y(I);
1580  IF I <  > N + 18 THEN  PRINT
1590  NEXT I
1600 X = 1:Y = 5
1610  GOSUB 1900
1620  RETURN
1700  REM   **************************************************
1710  REM   *  SCROLL UP ONE LINE                            *
1720  REM   **************************************************
1725  IF N + 18 > NMPT THEN 1800
1730  VTAB 23: POKE 36,78
1735  PRINT
1740 I = N + 18
1750  IF I > NMPT THEN 1780
1760  PRINT  TAB( 3);I; TAB( 25);X(I);: POKE 36,45: PRINT Y(I);
1780 X = 1:Y = 23
1790  GOSUB 1900
1800  RETURN
1900  REM   ***************************************************
1910  REM   *  MOVE CURSOR TO APPROPRIATE X,Y POSITION        *
1920  REM   ***************************************************
1930  VTAB Y
1940  IF X = 1 THEN  HTAB 25
```

CALIBRATE

```
100  REM   ***********************************************
110  REM   *  DATA ENTRY PROGRAM                         *
120  REM   ***********************************************
125 D$ =  CHR$ (4)
235 CO = 1
240  REM  RETRIEVE AND DISPLAY FILE
250  HOME : GOSUB 1000
260  POKE 34,0
270  VTAB 1: HTAB 12
280 FIL$ = "CAL": PRINT "CAL"
285  VTAB 1: HTAB 40: PRINT "# of points : ";
290  GOSUB 2000
295 N = 1
300  VTAB 1: POKE 36,55: PRINT NMPT
310  VTAB 2: HTAB 16: PRINT XA$
320  VTAB 3: HTAB 16: PRINT YA$
330  GOSUB 1300
340 N = 1
350  GOSUB 1500
360 X = 1:Y = 5
370  GOSUB 1900
380  GOTO 500
400  REM  INPUT NEW FILE
410  HOME : GOSUB 1000
415  POKE 34,0
420  VTAB 1: POKE 36,55
430  INPUT "";NMPT
440  DIM X(NMPT),Y(NMPT)
445  POKE 34,4
450 N = 1
460  GOSUB 1500
480  GOSUB 1300
485 X = 1:Y = 5
490  GOSUB 1900
500  REM   ****************************************************
510  REM   *  MAIN CONTROLLER FOR EDITING AND ENTRY           *
520  REM   ****************************************************
530  GET KB$
540  IF (KB$ >  = "0" AND KB$ <  = "9") OR KB$ = "." OR KB$ = "-" THEN 3000
550  IF  ASC (KB$) = 8 THEN 3200: REM  LEFT ARROW
560  IF  ASC (KB$) = 21 OR  ASC (KB$) = 32 THEN 3300: REM   RIGHT ARROW
570  IF  ASC (KB$) = 10 THEN 3400: REM  DOWN ARROW
580  IF  ASC (KB$) = 11 THEN 3500: REM  UP ARROW
590  IF  ASC (KB$) = 19 THEN 3600: REM  CONTROL S
610  IF  ASC (KB$) = 17 THEN 4200: REM   CONTROL Q
620  GOTO 530
1000  REM   **************************************************
1010  REM   *   DISPLAY HEADINGS                             *
```

TEMP MEASUREMENT

VARIABLES

C100	BASE ADDRESS FOR I/O CARD
C100	OUTPUT REGISTER B
C101	OUTPUT REGISTER A
C104	TIMER 1, LATCH LOW
C105	TIMER 1, COUNTER HIGH
C108	TIMER 2, LATCH LOW
C109	TIMER 2, COUNTER HIGH
C10B	AUXILLIARY CONTROL REGISTER
C10C	PERIPHERAL CONTROL REGISTER
C10D	INTERRUPT FLAG REGISTER

PROGRAM

```
0340   A2 70      LDX #$70
0342   8E A2 70   STX $70A2     CA1 POSITIVE ACTIVE EDGE
0345   8E 0C C1   STX $C10C     CB1 NEGATIVE ACTIVE EDGE
0348   A0 00      LDY #$00
034A   8C 0B C1   STY #C10B     T2 TIMED INTERRUPTS
034D   A9 02      LDA #$02      SWITCHING MASK
034F   AC 38 03   LDY $0338
0352   F0 02      BEQ $0356     IF EQUAL THEN DON'T SWITCH
0354   A9 10      LDA #$10      MASK
0356   A0 FF      LDY #$FF
0358   8C 0D C1   STY $C10D     CLEAR INTERRUPT FLAGS
035B   2C 0D C1   BIT $C10D     FIRST ACTIVE EDGE?
035E   F0 FB      BEQ $0358     NO, THEN CONTINUE POLLING
0360   8C 08 C1   STY $C108
0363   8C 09 C1   STY $C109     START COUNTING PERIOD
0366   8C 0D C1   STY $C10D     CLEAR INTERRUPT FLAGS
0369   2C 0D C1   BIT $C10D     SECOND ACTIVE EDGE?
036C   F0 FB      BEQ $0369     NO, THEN CONTINUE POLLING
036E   AC 08 C1   LDY $C108     GET LOW BYTE OF PERIOD
0371   AE 09 C1   LDX $C109     GET MID BYTE OF PERIOD
0374   8C 36 03   STY $0336     STORE PERIOD
0377   8E 37 03   STX $0337     STORE PERIOD
037A   60         RTS           RETURN TO PROGRAM
```

TEST FORMAT

```
1194  PRINT "LB"TC(Y + 1);C$
1198  NEXT Y
1200  PRINT "PA1000,1750;LBNotes :";C$
1210  PRINT "PA1250,1550;LB";N1$;C$
1220  PRINT "PA1250,1350;LB";N2$;C$
1230  PRINT "PA1250,1150;LB";N3$;C$
1240  PRINT "PA1250,950 ;LB";N4$;C$
1250  PRINT D$"PR#3"
1260  RETURN
```

TEST FORMAT

```
600  PRINT N1$
610  PRINT N2$
620  PRINT N3$
630  PRINT N4$
640  VTAB 23: HTAB 10
650  PRINT "  1.  Plot Out to HP"
655  VTAB 23: POKE 36,40
660  PRINT "  2.  Quit"
670  INPUT "Enter menu choice (1-2) : ";TMP$
680  IF TMP$ > "2" OR TMP$ < "1" THEN 510
700  IF TMP$ = "1" THEN  RETURN
710  PRINT "DONE"
720  END
1000  REM **************************************************
1010  REM   *  PLOT OUT TO PLOTTER                            *
1020  REM   ************************************************
1030  REM  INIT HP
1035 C$ =  CHR$ (3)
1040  PRINT D$"PR#2"
1050  PRINT  CHR$ (27)".("
1060  PRINT "IN;"
1070  PRINT  CHR$ (27)".I40;0;17:"
1075  PRINT  CHR$ (27)".N;19:"
1080  PRINT "SP1;SI.4,.6;"
1090  PRINT "DI1,0;VS20;"
1095  PRINT "PA"5150 -  LEN (F$) / 2 * 240;",6500;LB";F$;C$
1096  PRINT "SI.2,.3;"
1100  PRINT "PA1000,5650;"
1110  PRINT "LBCycle Type    Top Temp       Bottom Temp    Rate       Sample
      Interval";C$
1120  PRINT "PA1000,5450;PD;PA9280,5450;PU;"
1130  FOR X = 1 TO CT
1140  PRINT "PA1600,";5450 - 200 * X;";LB";X;C$
1150  PRINT "PA3400,";5450 - 200 * X;";LB";TP(X);C$
1160  PRINT "PA5080,";5450 - 200 * X;";LB";BTTM(X);C$
1170  PRINT "PA6520,";5450 - 200 * X;";LB";RTE(X);C$
1180  PRINT "PA8440,";5450 - 200 * X;";LB";IT(X);C$
1182  NEXT X
1183  PRINT "PA1000,4250;PD;PA9280,4250;PU;"
1184  PRINT "PA1000,4050;"
1185  PRINT "LBCycle Type  Cycle Type  Cycle Type  Cycle Type  Cycle Type
      Cycle Type";C$
1186  PRINT "PA1000,3850;PD;PA9280,3850;PU;"
1187  FOR Y = 0 TO MCYCLE - 1
1188 X = 2 +  INT (Y / 8) * 12
1190  PRINT "PA"1000 + X * 120",""3650 - 200 * (Y -  INT (Y / 8) * 8)";"
1192  PRINT "LB"Y + SCYCLE;C$;"PA"1720 + X * 120","3650 - 200 *
      (Y -  INT (Y / 8) * 8)";"
```

TEST FORMAT

```
10  REM   READ DATA ON SAMPLE AND PLOT OUT
15 D$ =  CHR$ (4)
20  HOME : VTAB 5
30  PRINT "Insert a /RAWDAT disk in drive 2 and hit a key...";
40  GET TMP$
50  PRINT D$"CATALOG,D2"
60  PRINT : INPUT "Enter new file name : ";F$
70  IF F$ = "" THEN 60
80  GOSUB 200: REM  READ IN DATA
90  GOSUB 500: REM  PRINT TO SCREEN
100  GOSUB 1000: REM  PLOT OUT
110  GOTO 90
200  PRINT D$"OPEN ";F$;",D2"
210  PRINT D$"READ ";F$
215  INPUT SCYCLE
220  INPUT CT
230  FOR X = 1 TO CT
240  INPUT TP(X),BTTM(X),RTE(X),IT(X),TD$(X)
245  IF TD$(X) = "N" THEN IT(X) = 0
250  NEXT X
251  INPUT MCYCLE
253  DIM TC(MCYCLE)
254  FOR X = 1 TO MCYCLE
256  INPUT TC(X)
258  NEXT X
260  INPUT N1$,N2$,N3$,N4$
270  PRINT D$"CLOSE ";F$
280  RETURN
500  REM  PRINT TO SCREEN
510  HOME : VTAB 2
520  HTAB  INT ( LEN (F$) / 2)
530  PRINT F$
540  VTAB 4
550  PRINT "Cycle Type";: POKE 36,15: PRINT "Top Temp";: POKE 36,30:
     PRINT "Bottom Temp";: POKE 36,45: PRINT "Rate";: POKE 36,55:
     PRINT "Sample Interval"
560  FOR X = 1 TO CT
570  PRINT  TAB( 5);X;: POKE 36,20: PRINT TP(X);: POKE 36,34:
     PRINT BTTM(X);: POKE 36,46: PRINT RTE(X);: POKE 36,62: PRINT IT(X)
572  NEXT X
574  VTAB 9: PRINT "Cycle Type  Cycle Type  Cycle Type  Cycle Type  Cycle
     Type  Cycle Type"
576  FOR Y = 0 TO MCYCLE - 1
578 X = 2 +  INT (Y / 8) * 11
580  VTAB 10 + (Y -  INT (Y / 8) * 8): POKE 36,X
582  PRINT Y + SCYCLE;: POKE 36,X + 6: PRINT TC(Y + 1)
584  NEXT Y
590  VTAB 18: PRINT "NOTES : "
```

SURFACE AREA

```
100  REM   *******************************************
110  REM   * PROGRAM TO CALCULATE SURFACE AREA       *
120  REM   *******************************************
125 D$ =  CHR$ (4)
130  PRINT "Calculating the surface area..."
135  IF DT = 0 THEN  GOSUB 2000
140 A = 0
150  FOR L = 2 TO NMPTS(0)
155  IF A(0,L) <  = 12 THEN 170
160 A = A + (1 / A(0,L - 1) - 1 / A(0,L)) * (PV * B(0,L - 1)) *  - 1E4
170  NEXT L
210 A =  INT (A * 2.5 * 1E4) / 1E4
220  PRINT "Surface Area = ";A
230  PRINT : PRINT
260  END
2000  REM   *******************************************
2010  REM   *   FILE LOADER                           *
2020  REM   *******************************************
2030  INPUT "Base Filename : ";FIL$
2040  IF FIL$ = "" THEN  PRINT D$"CATALOG /CALDAT/R.VP/": GOTO 2000
2050  PRINT D$"OPEN /CALDAT/R.VP/";FIL$;",D2"
2060  PRINT D$"READ /CALDAT/R.VP/";FIL$
2070  INPUT NMPTS(0)
2080  DIM A(1,NMPTS(0)),B(1,NMPTS(0))
2090  FOR I = 1 TO NMPTS(0)
2100  INPUT A(0,I),B(0,I)
2110  NEXT I
2120  PRINT D$"CLOSE"
2130  INPUT "Pore volume of Sample : ";PV$
2140 PV =  VAL (PV$)
2150  RETURN
2160  INPUT DIL$,CO$,DT$
2165  INPUT MA$,N1$,N2$
2170  PRINT D$"CLOSE"
2180  RETURN
```

DERIVATIVE LVP

```
820 OT = TS:OV = VS
830  NEXT I
835 NMPTS(K) = NMPTS(K) - 1
850  PRINT D$"OPEN /CALDAT/R.DVPDLR/"FIL$;C$(K)
860  PRINT D$"WRITE /CALDAT/R.DVPDLR/"FIL$;C$(K)
870  PRINT NMPTS(K)
880  FOR L = 1 TO NMPTS(K)
885 A(K,L) = 10 © A(K,L)
890  PRINT A(K,L): PRINT B(K,L)
900  NEXT L
910  PRINT D$"CLOSE"
920  NEXT K
925  FOR T = 1 TO 5: PRINT  CHR$ (7): NEXT T
930  PRINT D$"CHAIN STARTUP,@180 ,D1"
1000  REM   *************************
1010  REM   * TO LOG R              *
1020  REM   *************************
1025 LT =  LOG (10)
1030  FOR K = 0 TO 1
1040  FOR L = 1 TO NMPTS(K)
1050 A(K,L) =  LOG (A(K,L)) / LT
1060  NEXT L
1070  NEXT K
1080  RETURN
```

DERIVATIVE LVP

```
100  REM  *********************************************
110  REM  * THIS PROGRAM CALCULATES THE DERIVATIVE OF   *
120  REM  * THE PORE VOLUME W/RESPECT THE LOG OF THE    *
130  REM  * PORE RADIUS                                 *
140  REM  *********************************************
150  GOSUB 1000
160  PRINT "Converting R.VP to R.DVPDLR"
440  FOR K = 0 TO 1
445 FL = 0
446  FOR S = 1 TO MA:T1(S) = 0:V1(S) = 0: NEXT S
450  FOR S = 1 TO MA - 1:T1(S) = A(K,S):V1(S) = B(K,S)
460  NEXT S
465 PO = 0:PI = MA - 1
470  FOR I =  INT (MA / 2 + 1) TO MA - 1:T = 0:V = 0
480  FOR J = 1 TO I:T = T + T1(J):V = V + V1(J): NEXT J
490 TS = T / I
500 VS = V / I
510  IF  NOT FL THEN FL = 1: GOTO 570
520 DVDT =  INT ((VS - OV) / (TS - OT) * 1E5 + .5) / 1E5
530 WT =  INT ((TS + OT) / 2 * 1E3 + .5) / 1E3
540 FL = FL + 1
550 PO = PO + 1
560 A(K,PO) = WT:B(K,PO) = DVDT: IF FL = 2 THEN PO = PO + 1:A(K,PO) =
    WT:B(K,PO) = DVDT
570 OT = TS:OV = VS
580  NEXT I
590  FOR I = 1 TO NMPTS(K) - MA + 1
600 PI = PI + 1:TE = A(K,PI):VO = B(K,PI)
610  IF I > NMPTS(K) - 2 * MA + 2 THEN Z = I - NMPTS(K) + 2 * MA - 1:T2(Z)
     = TE:V2(Z) = VO
620 T = T + TE - T1(S):T1(S) = TE
630 V = V + VO - V1(S):V1(S) = VO
640 S = S + 1: IF MA < S THEN S = 1
650 TS = T / MA
660 VS = V / MA
680  IF TS = OT THEN DVDT = OO: GOTO 700
690 DVDT =  INT ((VS - OV) / (TS - OT) * 1E5 + .5) / 1E5:OO = DVDT
700 WT =  INT ((TS + OT) / 2 * 1E3 + .5) / 1E3
710 PO = PO + 1:A(K,PO) = WT:B(K,PO) = DVDT
720 OT = TS:OV = VS
730  NEXT I
740  FOR I = 2 TO  INT (MA / 2 + 1):T = 0:V = 0
750  FOR J = I TO MA:T = T + T2(J):V = V + V2(J): NEXT J
760 TS = T / (J - I)
770 VS = V / (J - I)
790 DVDT =  INT ((VS - OV) / (TS - OT) * 1E5 + .5) / 1E5
800 WT =  INT ((TS + OT) / 2 * 1E3 + .5) / 1E3
810 PO = PO + 1:A(K,PO) = WT:B(K,PO) = DVDT
```

DERIVATIVE VP

```
810  PRINT WT: PRINT DVDT
820 OT = TS:OV = VS
830  NEXT I
910  PRINT D$"CLOSE"
920  NEXT K
930  PRINT D$"CHAIN STARTUP,@1630,D1"
```

DERIVATIVE VP

```
100  REM  ************************************************
110  REM  * THIS PROGRAM CALCULATES THE DERIVATIVE OF    *
120  REM  * OF THE PORE VOLUME WITH RESPECT TO           *
130  REM  * PORE RADIUS                                  *
140  REM  ************************************************
160  PRINT "Converting R.VP to R.DVPDR"
360  DIM T1(MA),V1(MA),T2(MA),V2(MA)
440  FOR K = 0 TO 1
442  PRINT D$"OPEN /CALDAT/R.DVPDR/"FIL$;C$(K)
443  PRINT D$"WRITE /CALDAT/R.DVPDR/"FIL$;C$(K)
444  PRINT NMPTS(K) - 1
445 FL = 0
446  FOR S = 1 TO MA:T1(S) = 0:V1(S) = 0: NEXT S
450  FOR S = 1 TO MA - 1:T1(S) = A(K,S):V1(S) = B(K,S)
460  NEXT S
465 PI = MA - 1
470  FOR I =  INT (MA / 2 + 1) TO MA - 1:T = 0:V = 0
480  FOR J = 1 TO I:T = T + T1(J):V = V + V1(J): NEXT J
490 TS = T / I
500 VS = V / I
510  IF  NOT FL THEN FL = 1: GOTO 570
520 DVDT =  INT ((VS - OV) / (TS - OT) * 1E5 + .5) / 1E5
530 WT =  INT ((TS + OT) / 2 * 1E3 + .5) / 1E3
540 FL = FL + 1
560  PRINT WT: PRINT DVDT: IF FL = 2 THEN  PRINT WT: PRINT DVDT
570 OT = TS:OV = VS
580  NEXT I
590  FOR I = 1 TO NMPTS(K) - MA + 1
600 PI = PI + 1:TE = A(K,PI):VO = B(K,PI)
610  IF I > NMPTS(K) - 2 * MA + 2 THEN Z = I - NMPTS(K) + 2 * MA - 1:
     T2(Z) = TE:V2(Z) = VO
620 T = T + TE - T1(S):T1(S) = TE
630 V = V + VO - V1(S):V1(S) = VO
640 S = S + 1: IF MA < S THEN S = 1
650 TS = T / MA
660 VS = V / MA
680  IF TS = OT THEN DVDT = OO: GOTO 700
690 DVDT =  INT ((VS - OV) / (TS - OT) * 1E5 + .5) / 1E5:OO = DVDT
700 WT =  INT ((TS + OT) / 2 * 1E3 + .5) / 1E3
710  PRINT WT: PRINT DVDT
720 OT = TS:OV = VS
730  NEXT I
740  FOR I = 2 TO  INT (MA / 2 + 1):T = 0:V = 0
750  FOR J = I TO MA:T = T + T2(J):V = V + V2(J): NEXT J
760 TS = T / (J - I)
770 VS = V / (J - I)
790 DVDT =  INT ((VS - OV) / (TS - OT) * 1E5 + .5) / 1E5
800 WT =  INT ((TS + OT) / 2 * 1E3 + .5) / 1E3
```

CONVERT R

```
100  REM ***********************************************
110  REM *THIS PROGRAM CONVERTS THE X VS PORE VOLUME FILE *
120  REM * TO RADIUS VS PORE VOLUME.                    *
130  REM ***********************************************
135 T0 =  VAL (T0$)
140  PRINT "Converting X.VP to R.VP"
320  FOR K = 0 TO 1
325 P0 = 0:R1 = 0
330  FOR L = 1 TO NMPTS(K)
422 X0 = A(K,L) * 1E - 4
425 R = (( - 2 * T0 * (.25 - (29 * X0) + (.25 * T0 * X0))) /
    ((.9998 * 3.335E9) * (1 + T0 * X0))) * 1E8
427  IF R < 0 THEN 490
428  IF ( ABS (R - R1) < 1) AND (K$ = "Y") THEN 490
450 P0 = P0 + 1:R1 = R
460 A(K,P0) = R:B(K,P0) = B(K,L)
490  NEXT L
500 NMPTS(K) = P0
510  NEXT K
520  FOR K = 0 TO 1
530  PRINT D$"OPEN /CALDAT/R.VP/"FIL$;C$(K)
540  PRINT D$"WRITE /CALDAT/R.VP/"FIL$;C$(K)
550  PRINT NMPTS(K)
560  FOR L = 1 TO NMPTS(K)
570  PRINT A(K,L): PRINT B(K,L)
580  NEXT L
590  PRINT D$"CLOSE"
600  NEXT K
610  PRINT D$"CHAIN STARTUP,@1600,D1"
```

CONVERT VP

```
100  REM    *****************************************
110  REM    *  CONVERTS THE X.Z FILE TO X.VP        *
120  REM    *  BASES THE LOW POINT ON THE AVERAGE OF *
130  REM    *  COOLING DATA FROM -36 TO -36.5       *
140  REM    *****************************************
150  PRINT "Converting X.Z to X.VP"
330  FOR K = 0 TO 1
340  FOR L = 1 TO NMPTS(K)
360 VP = 1 / 10 © B(K,L)
370 B(K,L) = VP
380  NEXT L
390  NEXT K
400  IF (SA) THEN 500
410  FOR K = 0 TO 1
420  PRINT D$"OPEN /CALDAT/X.VP/"FIL$;C$(K)
430  PRINT D$"WRITE /CALDAT/X.VP/"FIL$;C$(K)
440  PRINT NMPTS(K)
450  FOR L = 1 TO NMPTS(K)
460  PRINT A(K,L): PRINT B(K,L)
470  NEXT L
490  NEXT K
500  PRINT D$"CHAIN STARTUP,@1580,D1"
```

INTEGRATE XDYDX

```
100  REM ****************************************************
110  REM * THIS PROGRAM INTEGRATES X, DY/DX DATA            *
120  REM * TO CALCULATE THE PARAMETER Z                     *
130  REM ****************************************************
150 D$ =  CHR$ (4): SPEED= 255: PRINT D$"PR#3"
160  PRINT "Converting X.DYDX to X.Z"
260  REM  PROCESS DATA
305 I = 1:Z = 0:TS = 0:TN = 0
330  IF A(1,I) <  =  - 36 THEN 370
340 B(1,I) = 0
350 I = I + 1
360  GOTO 330
370  IF A(1,I) <  =  - 36.5 THEN 420
380 TS = TS + B(1,I)
390 TN = TN + 1
400 I = I + 1
405 B(1,I) = 0
410  GOTO 370
420 Y1 = TS / TN:X1 = A(1,I - 1)
440  FOR K = I TO NMPTS(1)
450 Z = Z + (X1 - A(1,K)) * (B(1,K) - Y1)
460 B(1,K) = Z
475 X1 = A(1,K)
490  NEXT K
520 Z = 0:X1 = A(0,1)
540  FOR K = 1 TO NMPTS(0)
550  IF A(0,K) >  - 36.5 THEN 570
560 Z = Z + (X1 - A(0,K)) * (B(0,K) - Y1)
570 B(0,K) = Z
580 X1 = A(0,K)
590  NEXT K
600  IF (SA) THEN 700
610  FOR K = 0 TO 1
620  PRINT D$"OPEN /CALDAT/X.Z/"FIL$;C$(K)
630  PRINT D$"WRITE /CALDAT/X.Z/"FIL$;C$(K)
640  PRINT NMPTS(K)
650  FOR L = 1 TO NMPTS(K)
660  PRINT A(K,L): PRINT B(K,L)
670  NEXT L
680  PRINT D$"CLOSE"
690  NEXT K
700  PRINT D$"CHAIN STARTUP, @1540,D1"
```

DERIVATIVE XY

```
830  NEXT I
835 NMPTS(K) = NMPTS(K) - 1
840  IF (SA) THEN 920
850  PRINT D$"OPEN /CALDAT/X.DYDX/"FIL$;C$(K)
860  PRINT D$"WRITE /CALDAT/X.DYDX/"FIL$;C$(K)
870  PRINT NMPTS(K)
880  FOR L = 1 TO NMPTS(K)
890  PRINT A(K,L): PRINT B(K,L)
900  NEXT L
910  PRINT D$"CLOSE"
920  NEXT K
930  PRINT D$"CHAIN STARTUP,@1500,D1"
```

DERIVATIVE XY

```
100  REM  ************************************************
110  REM  * THIS PROGRAM FINDS THE DERIVATIVE            *
120  REM  * OF X W/RESPECT TO Y, DY/DX                   *
140  REM  ************************************************
160  PRINT "Converting X.Y to X.DYDX"
360  DIM TE(MA),VO(MA),TZ(MA),VZ(MA)
440  FOR K = 0 TO 1
445 FL = 0
446  FOR S = 1 TO MA:TE(S) = 0:VO(S) = 0: NEXT S
450  FOR S = 1 TO MA - 1:TE(S) = A(K,S):VO(S) = B(K,S)
460  NEXT S
465 PO = 0:PI = MA - 1
470  FOR I =  INT (MA / 2 + 1) TO MA - 1:T = 0:V = 0
480  FOR J = 1 TO I:T = T + TE(J):V = V + VO(J): NEXT J
490 TS = T / I
500 VS = V / I
510  IF  NOT FL THEN FL = 1: GOTO 570
520 DVDT =  INT ((VS - OV) / (TS - OT) * 1E5 + .5) / 1E5
530 WT =  INT ((TS + OT) / 2 * 1E3 + .5) / 1E3
540 FL = FL + 1
550 PO = PO + 1
560 A(K,PO) = WT:B(K,PO) = DVDT: IF FL = 2 THEN PO = PO + 1:
    A(K,PO) = WT:B(K,PO) = DVDT
570 OT = TS:OV = VS
580  NEXT I
590  FOR I = 1 TO NMPTS(K) - MA + 1
600 PI = PI + 1:TE = A(K,PI):VO = B(K,PI)
610  IF I > NMPTS(K) - 2 * MA + 2 THEN Z = I - NMPTS(K) + 2 * MA - 1:
     TZ(Z) = TE:VZ(Z) = VO
620 T = T + TE - TE(S):TE(S) = TE
630 V = V + VO - VO(S):VO(S) = VO
640 S = S + 1: IF MA < S THEN S = 1
650 TS = T / MA
660 VS = V / MA
680  IF TS = OT THEN DVDT = OO: GOTO 700
690 DVDT =  INT ((VS - OV) / (TS - OT) * 1E5 + .5) / 1E5:OO = DVDT
700 WT =  INT ((TS + OT) / 2 * 1E3 + .5) / 1E3
710 PO = PO + 1:A(K,PO) = WT:B(K,PO) = DVDT
720 OT = TS:OV = VS
730  NEXT I
740  FOR I = 2 TO  INT (MA / 2 + 1):T = 0:V = 0
750  FOR J = I TO MA:T = T + TZ(J):V = V + VZ(J): NEXT J
760 TS = T / (J - I)
770 VS = V / (J - I)
790 DVDT =  INT ((VS - OV) / (TS - OT) * 1E5 + .5) / 1E5
800 WT =  INT ((TS + OT) / 2 * 1E3 + .5) / 1E3
810 PO = PO + 1:A(K,PO) = WT:B(K,PO) = DVDT
820 OT = TS:OV = VS
```

CONVERT XY

```
100  REM ***************************************************
110  REM * THIS PROGRAM CONVERTS RAW DATA OF TEMP AND     *
120  REM * CONDUCTANCE INTO X,Y DATA PAIRS                *
130  REM ***************************************************
140 SU = 1
142  IF SR$ = "Y" THEN SU = 10
145  DEF  FN CN(X) =  LOG (X / SU) /  LOG (10)
170  PRINT "Converting Raw Data to X.Y"
180  DEF  FN CN(X) =  LOG ((X * CR / SU) / (CR - X / SU)) /  LOG (10)
190  IF SR$ = "N" THEN 240
200  PRINT D$"OPEN  "FIL$;C$(0)",D2"
210  PRINT D$"OPEN  "FIL$;C$(1)
220  PRINT D$"READ "FIL$;C$(0)
230  INPUT NMPTS(0)
240  PRINT D$"READ "FIL$;C$(1)
250  INPUT NMPTS(1)
260 N = NMPTS(0)
270  IF NMPTS(1) > NMPTS(0) THEN N = NMPTS(1)
280  DIM A(1,N),B(1,N)
285  PRINT D$"READ "FIL$;C$(0)
287  FOR K = NMPTS(0) TO 1 STEP  - 1
290  INPUT A(0,K),B(0,K)
295  NEXT K
300  PRINT D$"READ "FIL$;C$(1)
305  FOR K = 1 TO NMPTS(1)
307  INPUT A(1,K),B(1,K)
310  NEXT K
320  PRINT D$"CLOSE"
325  INPUT "Please insert the CALDAT disk in drive 2 and press
     RETURN...";KB$
327  IF KB$ <  > "" THEN 325
328  ONERR  GOTO 1000
329  FOR L = 0 TO 1
330  PRINT D$"OPEN/CALDAT/X.Y/"FIL$;C$(L)
340  PRINT D$"WRITE/CALDAT/X.Y/"FIL$;C$(L)
344  PRINT NMPTS(L)
350  FOR K = 1 TO NMPTS(L)
360 A(L,K) =  - 1 / (A(L,K) + 273.6) * 1E4
370 B(L,K) =  FN CN(B(L,K))
380  PRINT A(L,K): PRINT B(L,K)
390  NEXT K
400  PRINT D$"CLOSE /CALDAT/X.Y/"FIL$;C$(L)
410  NEXT L
495  POKE 216,0
500  PRINT D$"CHAIN STARTUP,@1460,D1"
1000  PRINT "Check disk in drive 2"
1010  GOTO 325
```

HP PLOTTER

```
5120  NEXT K
5130  PRINT "SPO;"
5200  PRINT D$"PR#3": PRINT D$"CHAIN  /CONPROG1/PROGRAMS/P,@390,D1"
```

HP PLOTTER

```
4095  IF YP > YB THEN YP = YB
4100  IF (YP < YA) THEN YP = YA
4120  PRINT "PA"XP","YP";PD;PA"
4130  FOR I = 2 TO NMPTS
4135  INPUT X,Y
4140 XP =  FN XL(X)
4150 YP =  FN YL(Y)
4160  IF XP > XB THEN XP = XB
4170  IF (XP < XA) THEN XP = XA
4180  IF YP > YB THEN 4210
4190  IF (YP < YA) THEN 4210
4200  PRINT XP","YP","
4210  NEXT I
4215  PRINT D$"CLOSE"
4220  PRINT XP","YP";PU;"
4225  NEXT K
4230  RETURN
4500  REM   ******************************************
4510  REM   *   PLOT AXIS LABELS                     *
4520  REM   ******************************************
4530  PRINT "SP1;SI.24,.38;"
4540 XP = (XB + XA) / 2 -  LEN (BT$) / 2 * 150
4560 YP = YA - 600
4570  PRINT "PA"XP","YP";LB"BT$;C$
4575  IF A > 4 THEN 4580
4577  PRINT "SI.1,.12;PA"(XB + XA) / 2 - 50 +  LEN (BT$) / 2 * 150",
      "YA - 500";LB - 1"C$
4578  PRINT "SI.24,.38;"
4580 YP = (YB + YA) / 2 -  LEN (EL$) / 2 * 150
4590  PRINT "DI0,1;PA"XA - 700","YP";LB"EL$;C$
4595  PRINT "DI1,0;"
4600  RETURN
5000  REM   *******************************************
5010  REM   *  TITLES                                 *
5020  REM   *******************************************
5025  PRINT "SI.30,.50;"
5030 XP = (XB + XA) / 2 -  LEN (TI$) / 2 * 160
5040 YP = YB + 700
5050  PRINT "PA"XP","YP";LB"TI$;C$
5055  FOR K = 1 TO 2
5057  IF F$(K) = "" THEN 5120
5060 LIN = L(K)
5070  IF LIN = 0 THEN LIN = 2
5080  PRINT "SI.22,.30;LT"LIN",2;"
5085  IF LIN = 7 THEN  PRINT "LT;"
5090  PRINT "PA"XA + 600","YB + 170";PD;PA"XA + 1275","YB + 170";PU;"
5100  PRINT "PA"XA + 1475","YB + 170";LB"L$(K);C$
5110 XA = (XA + XB) / 2
```

```
2590  FOR YS = Y1 TO Y2 STEP YIN
2600 YN =  FN YL(YS)
2610  PRINT "PA"XB","YN";PD;PA"XB - 25","YN";PU;"
2620  NEXT YS
2630  RETURN
3000  REM   *******************************************
3010  REM   *    X LABELS                             *
3020  REM   *******************************************
3030 RF = 0
3040  IF XIN <  > 0 THEN RF =  -  INT ( LOG ( ABS (XIN)) / LT)
3050 XS =  INT (XS * 10 © RF + .5) / 10 © RF
3060  IF XS = 0 OR  ABS (XS) >  = .01 THEN XS$ =  LEFT$
      (BL$,7 -  LEN ( STR$ (XS))) +  STR$ (XS): GOTO 3100
3065 XS$ =  STR$ ( INT ( ABS (XS) * 10 © RF + .5))
3070  IF XS < 0 THEN XS$ =  MID$ (SN$,1 + RF,7 -  LEN (XS$)) + XS$: GOTO
3100
3080 XS$ =  MID$ (SP$,1 + RF,7 -  LEN (XS$)) + XS$
3090 XS$ =  RIGHT$ (XS$, LEN (XS$) - 1)
3100  RETURN
3200  REM   *******************************************
3210  REM    *    Y LABELS                             *
3220  REM   *******************************************
3230 RF = 0
3240  IF YIN <  > 0 THEN RF =  -  INT ( LOG (YIN) / LT)
3250 YS =  INT (YS * 10 © RF + .5) / 10 © RF
3260  IF YS = 0 OR  ABS (YS) >  = .01 THEN YS$ =  LEFT$ (BL$,7 -  LEN
      ( STR$ (YS))) +  STR$ (YS): GOTO 3300
3265 YS$ =  STR$ ( INT ( ABS (YS) * 10 © RF + .5))
3270  IF YS < 0 THEN YS$ =  MID$ (SN$,1 + RF,7 -  LEN (YS$)) + YS$:
      GOTO 3300
3280 YS$ =  MID$ (SP$,1 + RF,7 -  LEN (YS$)) + YS$
3290 YS$ =  RIGHT$ (YS$, LEN (YS$) - 1)
3300  RETURN
4000  REM   *****************************************
4010  REM   *   PLOT FUNCTION                       *
4020  REM   *****************************************
4040  FOR K = 1 TO 2
4041  IF F$(K) = "" THEN 4225
4042  PRINT "SP1;LT"L(K)",2;"
4045  PRINT D$"OPEN /"DR$"/"FIL$"/"F$(K)
4047  PRINT D$"READ /"DR$"/"FIL$"/"F$(K)
4048  INPUT NMPTS
4050 I = 1
4055  INPUT X,Y
4060 XP =  FN XL(X)
4070 YP =  FN YL(Y)
4080  IF XP > XB THEN XP = XB
4090  IF (XP < XA) THEN XP = XA
```

```
2060  GOSUB 2500: REM   LINEAR Y
2070  RETURN
2100  REM   ********************************************
2110  REM   *   LINEAR X PLOT                          *
2120  REM   ********************************************
2130  FOR XS = X1 TO X2 STEP XIN
2140 XN =  FN XL(XS)
2150  GOSUB 3000: REM  X LABEL
2160  PRINT "PA"XN - 550","YA - 200";LB"XS$;C$
2170  PRINT "PA"XN","YA";PD; PA"XN","YA + 50";PU;"
2180  NEXT XS
2190  FOR XS = X1 TO X2 STEP XIN
2200 XN =  FN XL(XS)
2210  PRINT "PA"XN","YB";PD;PA"XN","YB - 25";PU;"
2220  NEXT XS
2230  RETURN
2300  REM   *******************************************
2310  REM   *  LOG X PLOT                             *
2320  REM   *******************************************
2330  FOR K =  LOG ( ABS (X1)) / LT TO  LOG ( ABS (X2)) / LT - 1
2340  FOR K1 = 1 TO 10
2345 XS = K1 * 10 © K
2350 XN = ( LOG (XS) / LT - XT) / HARSCAL + XA
2360  PRINT "PA"XN","YA";PD;PA"XN","YA + 50";PU;"
2370  IF (K1 <  > 1) AND (K1 <  > 5) AND ((K1 <  > 10) OR
      (K <  > ( LOG ( ABS (X2)) / LT - 1))) THEN 2400
2375 XIN = 10 © K
2380  GOSUB 3000: REM  X LABEL
2390  PRINT "PA"XN - 550","YA - 200";LB"XS$;C$
2400  NEXT K1
2410  NEXT K
2420  FOR K =  LOG ( ABS (X1)) / LT TO  LOG ( ABS (X2)) / LT - 1
2430  FOR K1 = 1 TO 10
2435 XS = K1 * 10 © K
2440 XN =  FN XL(XS)
2450  PRINT "PA"XN","YB";PD;PA"XN","YB - 25";PU;"
2460  NEXT K1
2470  NEXT K
2480  RETURN
2500  REM   ******************************************
2510  REM   *  LINEAR Y PLOT                         *
2520  REM   ******************************************
2530  FOR YS = Y1 TO Y2 STEP YIN
2540 YN =  FN YL(YS)
2550  GOSUB 3200: REM  Y LABEL
2560  PRINT "PA"XA - 640","YN";LB"YS$;C$
2570  PRINT "PA"XA","YN";PD;PA"XA + 50","YN";PU;"
2580  NEXT YS
```

HP PLOTTER

```
100  REM   ******************************************
110  REM   *  HARD PLOTS TO THE HP FROM PLOT PROGRAM *
120  REM   ******************************************
130 D$ =  CHR$ (4):LT =  LOG (10):C$ =  CHR$ (3)
135 XNUM = .15:YNUM = .185
140 SP$ = "       .00000":SN$ = "      -.00000":BL$ = "           "
150  GOSUB 1000: REM  INITIALIZE HP
160  GOSUB 1200: REM  DEFINE FUNCTIONS
170  PRINT "PA"XA","YA";"
180  PRINT "PD;PA"XA","YA","XB","YA","XB","YB","XA","YB","XA","YA",
     "XB","YA","XB","YB","XA","YB","XA","YA";PU;"
190  GOSUB 2000: REM  PLOTS SCALES
200  GOSUB 4500: REM  PLOTS LABELS
210  GOSUB 4000: REM  PLOTS POINTS
220  GOTO 5000: REM  GRAPH TITLES
1000  REM   ***************************************
1010  REM   *  INIT FOR THE HP                     *
1020  REM   ***************************************
1030  PRINT D$"PR#2"
1040  PRINT  CHR$ (27)".("
1050  PRINT " IN;"
1060  PRINT  CHR$ (27)".I40;0;17:"
1070  PRINT "SP1;SI"XNUM","YNUM";"
1080  PRINT "DI1,0;VS20;"
1090  IF PL = 14 THEN XA = 900:XB = 9470:YA = 900:YB = 6400: GOTO 1110
1100 XA = 900:XB = 9470:YA = 900:YB = 6400
1110  RETURN
1200  REM   ****************************************
1210  REM   * DEFINE FUNCTIONS                      *
1220  REM   ****************************************
1230  IF A <  > 5 THEN 1290
1240  REM  LOG FUNCTION
1250 HARSCAL = ( LOG ( ABS (X2 / X1)) / LT) / (XB - XA)
1260 XT =  LOG ( ABS (X1)) / LT
1270  DEF  FN XL(R) = ( LOG ( ABS (R)) / LT - XT) / HARSCAL + XA
1280  GOTO 1380
1290  REM  LINEAR FUNCTION
1300 HARSCAL = (X2 - X1) / (XB - XA)
1310  DEF  FN XL(R) = (R - X1) / HARSCAL + XA
1380  REM  LINEAR FUNCTION
1390 VERSCAL = (Y2 - Y1) / (YB - YA)
1400  DEF  FN YL(T) = (T - Y1) / VERSCAL + YA
1410  RETURN
2000  REM   ********************************************
2010  REM   *  PLOTS SCALES                             *
2020  REM   ********************************************
2030  IF A <  > 5 THEN  GOSUB 2100: GOTO 2060
2040  GOSUB 2300: REM  LOG X
```

PLOTTER

```
3700  REM  Y INT
3710  VTAB 15: HTAB 40
3720  INPUT "";YIN
3730  GOTO 520
3750  REM  PAPER LENGTH
3760  VTAB 16: HTAB 40
3770  INPUT "";PL
3780  GOTO 520
```

PLOTTER

```
3130  GOTO 3060
3150  REM  LABEL
3160  VTAB 5: HTAB 40
3170  INPUT "";L$(1)
3180  GOTO 520
3200  REM  LINETYPE
3210  VTAB 6: HTAB 40
3220  INPUT "";L(1)
3230  GOTO 520
3250  REM  FILENAME 2
3260  VTAB 7: HTAB 40
3270  INPUT "";F$(2)
3280  IF F$(2) <  > "" THEN 390
3290  POKE 34,9
3300  HOME
3310  PRINT D$"CATALOG /"DR$"/"FIL$
3320  TEXT
3330  GOTO 3260
3350  REM  LABEL
3360  VTAB 8: HTAB 40
3370  INPUT "";L$(2)
3380  GOTO 520
3400  REM  LINETYPE
3410  VTAB 9: HTAB 40
3420  INPUT "";L(2)
3430  GOTO 520
3450  REM  MIN X
3460  VTAB 10: HTAB 40
3470  INPUT "";X1
3475  IF X1 = 0 AND KB$ = "Y" THEN X1 = 1
3477  VTAB 10: HTAB 40: PRINT X1
3480  GOTO 520
3500  REM  MAX X
3510  VTAB 11: HTAB 40
3520  INPUT "";X2
3530  GOTO 520
3550  REM  X INT
3560  VTAB 12: HTAB 40
3570  INPUT "";XIN
3580  GOTO 520
3600  REM  MIN Y
3610  VTAB 13: HTAB 40
3620  INPUT "";Y1
3630  GOTO 520
3650  REM  MAX Y
3660  VTAB 14: HTAB 40
3670  INPUT "";Y2
3680  GOTO 520
```

PLOTTER

```
1480  REM  * CHECKING THE DISK IN DRIVE 2 FOR*
1490  REM  * THE PREFIX OF /CALDAT.          *
1500  REM  **********************************
1510  PRINT "THE DISK IN DRIVE 2 IS NOT CALDAT.  PLEASE REPLACE THE DISK IN
      DRIVE 2 WITH CALDAT."
1520  INPUT "AND PRESS RETURN TO CONTINUE.";X$
1530  POKE 216,0: GOTO 100
2000  PRINT  CHR$ (17): POKE  - 16304,0: POKE  - 16302,0: POKE  - 16299,0:
      POKE  - 16297,0
2001  GET A$
2010  POKE  - 16303,0: POKE  - 16300,0
2020  PRINT D$"PR#3": PRINT : GOTO 390
2100  REM  SETS UP X AXIS ON A LOG SCALE
2105  IF X1 = 0 THEN X1 = 1
2110  FOR K =  LOG (X1) /  LOG (10) TO  LOG (X2) /  LOG (10) - 1
2120  FOR K1 = 1 TO 10
2130 XP =  FN CN(K1 * 10 © K)
2135  IF XP = 280 THEN 2150
2140  HPLOT XP,159 TO XP,154
2150  NEXT K1
2160  NEXT K
2180  GOTO 862
2300  REM  ROUNDS DOWN TO THE NEXT CYCLE
2310  VTAB (L): HTAB (MM)
2320  INPUT "";X1
2330 X1 = 10 © ( INT ( LOG (X1) /  LOG (10)))
2340  VTAB (L): HTAB (MM)
2350  PRINT X1;"  "
2360  GOTO 520
2400  REM  ROUNDS TO NEAREST CYCLE
2410  VTAB (L + 1): HTAB (MM)
2420  INPUT "";X2
2430 X2 = 10 © ( INT ( LOG (X2) /  LOG (10) + .5))
2440  VTAB (L + 1): HTAB (MM)
2450  PRINT X2;"  "
2460  GOTO 520
3000  REM  TITLE
3010  VTAB 3: HTAB 40
3020  INPUT "";TI$
3030  GOTO 520
3050  REM  FILENAME
3060  VTAB 4: HTAB 40
3070  INPUT "";F$(1)
3080  IF F$(1) <  > "" THEN 390
3090  POKE 34,9
3100  HOME
3110  PRINT D$"CATALOG /"DR$"/"FIL$
3120  TEXT
```

PLOTTER

```
1260  RETURN
1270 LAB$ = "":PL = 8.5:LIN = 7:PEN = 1:X1 =  - 41:X2 =  - 36:Y1 = 0:
     Y2 = 1:YIN = .1:XNUM = .15:YNUM = .185:XINC = 1
1280 EL$ = "Relative Conducting Pore Volume":BT$ = "(-1/T), K  x 10":
     FIL$ = "X.VP"
1290  DEF  FN MOH(T) = 159 - (T) * YA / (Y2 - Y1)
1300  DEF  FN CN(R) = ((R - X1) * XB / (X2 - X1)) + 1
1310  RETURN
1320 LAB$ = "":PL = 8.5:X1 = 10:X2 = 10000:Y1 = 0:Y2 = 1.1:YIN = .1:
     XNUM = .15:YNUM = .185:XINC = 10:BT$ = "Radius, Angstroms":
     EL$ = "Relative Conducting Pore Volume":FIL$ = "R.VP"
1321  DEF  FN MOH(T) = 159 - (T - Y1) * (YA - YB) / (Y2 - Y1)
1322  DEF  FN CN(R) = ( LOG (R) /  LOG (10) -  LOG (X1) /  LOG (10)) /
      ( LOG (X2) /  LOG (10) -  LOG (X1) /  LOG (10)) * XB + 1
1324  IF KB$ = "N" THEN  DEF  FN CN(R) = ((R - X1) * XB / (X2 - X1)) + 1:
      A = 6:X1 = 0:XINC = 1000
1325 PEN = 1:LIN = 7
1326  RETURN
1330 LAB$ = "":PL = 8.5:YIN = 1:XIN = 1:X1 =  - 41:X2 =  - 36:Y1 = 0:
     Y2 = 5:PEN = 1:LIN = 2:XNUM = .15:YNUM = .185:BT$ = "(-1/T) K  x 10":
     EL$ = "      Z       "
1333 PEN = 1:LIN = 7:FIL$ = "X.Z"
1340  DEF  FN MOH(T) = 159 - (T - Y1) * (YA - YB) / (Y2 - Y1)
1350  DEF  FN CN(R) = ((R - X1) * XB / (X2 - X1)) + 1
1360  RETURN
1361 LAB$ = "":PL = 8.5:X1 = 10:X2 = 10000:Y1 = 0:Y2 = 2:YIN = .2:
     XNUM = .15:YNUM = .185:XINC = 10:BT$ = "Radius, Angstroms "
1362 EL$ = "d(Pore Volume)/d(log Radius) ":PEN = 1:LIN = 7:
     FIL$ = "R.DVPDLR"
1363  IF A = 6 THEN EL$ = "d(Pore Volume)/d(Radius)":FIL$ = "R.DVPDR":
      Y2 = .1:YIN = .01
1364  IF KB$ = "N" THEN X1 = 0:XINC = 1000
1365  DEF  FN MOH(T) = 159 - (T - Y1) * (YA - YB) / (Y2 - Y1)
1366  IF KB$ = "Y" THEN  DEF  FN CN(R) = ( LOG (R) /  LOG (10) -  LOG (X1)
      / LOG (10)) / (LOG (X2) / LOG (10) -  LOG (X1) / LOG (10)) * XB + 1:
      A = 5: GOTO 1368
1367  DEF  FN CN(R) = ((R - X1) * XB / (X2 - X1)) + 1
1368  RETURN
1370  HOME
1380  PRINT D$"CATALOG /"DR$"/"FIL$
1390  INPUT "ENTER NEW FILE NAME     ";SUB$
1400  IF SUB$ = "" THEN  GOTO 1370
1410  RETURN
1420  ONERR  GOTO 1510
1430  PRINT D$"OPEN /CALDAT, TDIR"
1440  PRINT D$"CLOSE /CALDAT"
1450  POKE 216,0: GOTO 240
1470  REM  **********************************
```

PLOTTER

```
865  IF F$(I) = "" THEN 1021
870  PRINT  CHR$ (4)"OPEN /"DR$"/"FIL$"/"F$(I)
880  ONERR  GOTO 1090
890  PRINT  CHR$ (4)"READ/"DR$"/"FIL$"/"F$(I)
900  INPUT NMPTS
920  POKE 216,0
930  FOR T = 1 TO NMPTS
935  IF  PEEK ( - 16384) = 27 THEN T = NMPTS: GOTO 1010
936  POKE  - 16368,0
940  INPUT X,Y
950  IF (X <  = X2) AND (X >  = X1) THEN J =  FN CN(X)
960  IF (Y <  = Y2) AND (Y >  = Y1) THEN K =  FN MOH(Y)
970  IF (X > X2) OR (X < X1) THEN J = 0
980  IF (Y > Y2) OR (Y < Y1) THEN K = 1
990  IF (J = 0) OR (K = 1) THEN J = 0:K = 1
1000  HPLOT J,K
1010  NEXT T
1020  PRINT  CHR$ (4)"CLOSE/"DR$"/"FIL$"/"F$(I)
1021  NEXT I
1022  PRINT  CHR$ (7); CHR$ (7); CHR$ (7)
1030  GET A$: PRINT  CHR$ (18): TEXT : PRINT D$"PR#3":K = 10: HOME :
      GOTO 390
1040  PRINT  CHR$ (4)"RUN STARTUP,D1"
1050  REM  ****************************************************
1060  REM  *THIS IS AN ERROR HANDLING ROUTINE WRITTEN TO CLEAR*
1070  REM  *ERRORS IN INPUTING FILE NAMES.                    *
1080  REM  ****************************************************
1090  TEXT : PRINT  CHR$ (4)"CLOSE/"DR$"/"FIL$"/"SUB$
1100  PRINT  CHR$ (4)"DELETE /"DR$"/"FIL$"/"SUB$: POKE 216,0
1110  GET A$: PRINT D$"PR#3": GOTO 150
1130  END
1140  REM  ******************************************************
1150  REM  *THIS IS A LOOKUP TABLE FOR VARIABLE DEFAULTS AND    *
1160  REM  *EQUATIONS.                                          *
1170  REM  ******************************************************
1180 LAB$ = "Degrees Celcius":PL = 8.5:X1 =  - 41:X2 =  - 36:Y1 =  - 1:
     Y2 = 4:XNUM = .15:YNUM = .185:EL$ = "Log (Conductance in micro Mho)":
     BT$ = "(-1/T), K  x 10"
1190 SL = 2:XIN = 1:YIN = .5:LIN = 7:PEN = 1:FIL$ = "X.Y"
1200  DEF  FN MOH(T) = 159 - (T - Y1) * (YA - YB) / (Y2 - Y1)
1210  DEF  FN CN(R) = ((R - X1) * XB / (X2 - X1)) + 1
1220  RETURN
1230 LAB$ = "":PL = 8.5:YINC = .5:XIN = 1:PEN = 1:X1 =  - 41:X2 =  - 36:
     Y1 = 0:LIN = 7:Y2 = 3:XNUM = .15:YNUM = .185:BT$ = "(-1/T), K  x 10":
     EL$ = "d(log(Conductance))/d(-1/T)"
1231 FIL$ = "X.DYDX"
1240  DEF  FN MOH(T) = 159 - (T - Y1) * (YA - YB) / (Y2 - Y1)
1250  DEF  FN CN(R) = ((R - X1) * XB / (X2 - X1)) + 1
```

PLOTTER

```
407  HTAB (K): PRINT "7.   LINETYPE ....................  "L(2)
410  HTAB (K): PRINT "8.  MINIMUM X .....................  "X1
420  HTAB (K): PRINT "9.  MAXIMUM X .....................  "X2
430  HTAB (K): PRINT "10. X-INTERVAL SIZE ...............  "XINC
440  HTAB (K): PRINT "11. MINIMUM Y .....................  "Y1
450  HTAB (K): PRINT "12. MAXIMUM Y .....................  "Y2
460  HTAB (K): PRINT "13. Y-INTERVAL SIZE ...............  "YINC
480  HTAB (K): PRINT "14. PAPER LENGTH ..................  "PL
495  PRINT "-------------------------------------------"
500  PRINT "A-ONSCREEN PLOTTING";: HTAB 30: PRINT "D-CLEAR GRAPHICS
     SCREEN";CS$(CS)
502  PRINT "B-HP722 PLOTTING ";: HTAB 30: PRINT "E-EXIT PROGRAM"
504  PRINT "C-VIEW GRAPHICS SCREEN";: HTAB 30: PRINT "F-FIND NEW DATA
     POINTS"
510  PRINT
520  VTAB (23): INPUT "ENTER YOUR CHOICE AND PRESS RETURN.";SE$
525  IF SE$ = "" THEN 520
530  IF ( VAL (SE$) < 1 OR  VAL (SE$) > 13) AND (SE$ < "A" OR SE$ > "F")
     THEN 520
540 M =  VAL (SE$)
550  ON M GOTO 3000,3050,3150,3200,3250,3350,3400,3450,3500,3550,3600,3650,
     3700,3750
560  IF  ASC ( LEFT$ (SE$,1)) = 65 THEN 720: REM  A
570  IF  ASC ( LEFT$ (SE$,1)) = 66 THEN  PRINT D$"CHAIN
     /CONPROG1/PROGRAMS/PLOT": REM  B
580  IF  ASC ( LEFT$ (SE$,1)) = 67 THEN  GOTO 1200: REM  C
590  IF  ASC ( LEFT$ (SE$,1)) = 68 THEN CS =  NOT CS: GOTO 390: REM  D
600  IF  ASC ( LEFT$ (SE$,1)) = 69 THEN  PRINT D$"RUN STARTUP,D1": REM  E
610  IF  ASC ( LEFT$ (SE$,1)) = 70 THEN 160: REM  F
720  PRINT  CHR$ (17)
730  IF  NOT CS THEN  POKE  - 16304,0: POKE  - 16302,0: POKE  - 16299,0:
     POKE  - 16297,0: GOTO 755
740  HGR2
750  POKE  - 16368,0
755 YA = 159:YB = 0:XA = 1:XB = 279
760  HCOLOR= 3: IF  NOT CS THEN  GOTO 862
770  HPLOT 0,0 TO 279,0 TO 279,159 TO 1,159 TO 1,0
790 VARSCAL = (YA - YB) * YINC / (Y2 - Y1)
800  FOR K = 1 + VARSCAL TO YA STEP VARSCAL
810  HPLOT 1,K TO 5,K: HPLOT 279,K TO 274,K: NEXT K
815  IF A = 5 THEN 2100: REM  PLOT X ON LOG SCALE
820 HARSCAL = (XB - XA) * XINC / (X2 - X1)
825  IF HARSCAL < 1 THEN HARSCAL = 1
830  FOR K = 1 TO XB - HARSCAL STEP HARSCAL
840  HPLOT K,159 TO K,154
850 K =  INT (K)
860  NEXT K
862  FOR I = 1 TO 2
```

PLOTTER

```
100  REM ************************************************
110  REM * THIS PROGRAM PLOTS ONSCREEN PLOTS OR SAVES THE*
120  REM * VARIABLES IN A VARIABLE FILE AND PLOTS ON THE *
130  REM * HP7220 PLOTTER.                               *
140  REM ************************************************
150  TEXT :CS$(1) = "TRUE":CS$(0) = "FALSE"
155  DIM AS%(30)
160  HOME :D$ =  CHR$ (4)
170 DR$ = "CALDAT"
180 L(1) = 7:L(2) = 2
200 CS = 1
235  REM ********************************************
236  REM * CHECKING TO SEE IF DISK IN DRIVE TWO IS   *
237  REM * CALDAT OR NOT.                            *
238  REM ********************************************
240 K = 10: PRINT "MENU OF SUBDIRECTORIES"
250  PRINT : PRINT
260  HTAB (K): PRINT "1.  X.Y"
270  HTAB (K): PRINT "2.  X.DYDX"
275  HTAB (K): PRINT "3.  X.Z"
280  HTAB (K): PRINT "4.  X.VP"
290  HTAB (K): PRINT "5.  R.VP"
300  HTAB (K): PRINT "6.  R.DVPDR"
302  HTAB (K): PRINT "7.  R.DVPDLR"
305  HTAB (K): PRINT "8.  EXIT TO MAIN MENU"
310  INPUT "          ENTER NUMBER OF YOUR CHOICE  ";A$
320  IF (A$ < "1") AND (A$ > "8") THEN  GOTO 240
330 A =  VAL (A$)
332  IF A$ = "8" THEN 1040
333  IF A >  = 2 AND A <  = 4 THEN  PRINT : INPUT "DO YOU WISH PORE RADIUS
     MARKS ON TOP (Y/N  DEFAULT = N)  ";PZ$
334 K1$ = "Y": IF A$ = "6" THEN K1$ = "N"
335  IF A$ <  > "6" AND A$ <  > "7" AND A$ <  > "5" THEN 338
336  PRINT : PRINT "DO YOU WISH TO PLOT THE RADIUS ON A LOG SCALE (Y/N
     DEFAULT = ";K1$;")  ";: INPUT TEMP$
337 KB$ = TEMP$: IF TEMP$ = "" THEN KB$ = K1$
338  IF A$ < "5" THEN L$(1) = "WARMING":L$(2) = "COOLING": GOTO 340
339 L$(2) = "NECK SIZES":L$(1) = "BODY SIZES"
340  ON A GOSUB 1180,1230,1330,1270,1320,1361,1361
390  HOME : PRINT "ENTER THE LETTER OF ANY VALUE THAT YOU WISH TO CHANGE."
391 L = 3:K = 2
400  PRINT "AND PRESS RETURN"
401  HTAB (K): PRINT "1.  TITLE ........................  "TI$
402  HTAB (K): PRINT "2.  FILENAME .....................  "F$(1)
403  HTAB (K): PRINT "3.    LABEL ......................  "L$(1)
404  HTAB (K): PRINT "4.    LINETYPE ...................  "L(1)
405  HTAB (K): PRINT "5.  FILENAME .....................  "F$(2)
406  HTAB (K): PRINT "6.    LABEL ......................  "L$(2)
```

CONTROLLER

```
6340  RETURN
6500  REM  DISPLAY ITEMS
6510  VTAB 18: HTAB 4
6520  PRINT "Maximum Temp : ";TP(TC(CCYCLE));"   ";
6530  HTAB 30
6540  PRINT "Minimum Temp : ";BTTM(TC(CCYCLE));"   ";
6550  POKE 36,58
6560  PRINT "Temp Rate : ";RA(TC(CCYCLE));"   ";
6570  VTAB 20: HTAB 10
6580  PRINT "Temp Interval between readings : ";
6590  IF TD$(TC(CCYCLE)) = "N" THEN  PRINT "0": GOTO 6610
6600  PRINT INVL(TC(CCYCLE))
6610  RETURN
7000  REM    SAVE OFF CYCLE TYPE DESCRIPTIONS ON DISK
7010 D$ =  CHR$ (4)
7030  PRINT D$;"OPEN  ";DIS$;".";SCYCLE;".CT,D2"
7040  PRINT D$;"WRITE ";DIS$;".";SCYCLE;".CT"
7045  PRINT SCYCLE
7050  PRINT CT
7060  FOR N = 1 TO CT
7070  PRINT TP(N)
7072  PRINT BTTM(N)
7074  PRINT RA(N)
7076  PRINT INVL(N)
7078  PRINT TD$(N)
7080  NEXT N
7081  PRINT MCYCLE
7082  FOR N = 1 TO MCYCLE
7083  PRINT TC(N)
7084  NEXT N
7085  PRINT N1$: PRINT N2$: PRINT N3$: PRINT N4$
7090  PRINT D$;"CLOSE ";DIS$;".";SCYCLE;".CT"
7110  RETURN
9999  POKE  - 15986,64: HIMEM: 38400: END
```

CONTROLLER

```
3500  REM  NOTES ON SAMPLE
3510  HOME : VTAB 3: HTAB 29
3520  PRINT "Conductance Controller"
3530  VTAB 8: PRINT "Notes on Sample (press 'Return' at end of line) :"
3540  INPUT ">";N1$
3550  INPUT ">";N2$
3560  INPUT ">";N3$
3570  INPUT ">";N4$
3580  RETURN
4000  PRINT D$"OPEN CAL,D1"
4005  PRINT D$"READ CAL"
4010  INPUT NMPT
4020  DIM P(NMPT),T(NMPT)
4025  FOR TI = 1 TO NMPT
4030  INPUT P(TI),T(TI)
4040  NEXT TI
4042 TI = 1
4045  PRINT D$"CLOSE"
4050  RETURN
6000  REM  NOW SPECIFY EACH CYCLE
6010  HOME : VTAB 16: HTAB 10
6020  PRINT "Number of Cycles : ";
6030  GOSUB 1900
6040  IF TMP =  > 1 THEN 6070
6050  HTAB 29: PRINT "     ";: HTAB 29
6060  GOTO 6030
6070 MCYCLE =  INT (TMP)
6075  DIM TC(MCYCLE)
6080  FOR CCYCLE = 1 TO MCYCLE
6090  HOME : VTAB 16: HTAB 10
6100  PRINT "Cycle Number : ";CCYCLE;
6110  POKE 36,40
6120  PRINT "Cycle Type : ";
6130  GOSUB 1900
6140  IF TMP =  > 1 AND TMP <  = CT THEN 6160
6150  POKE 36,53: PRINT "     ";: POKE 36,53
6155  GOTO 6130
6160 TC(CCYCLE) =  INT (TMP)
6170  GOSUB 6500: REM  PRINT OUT PARAMETERS
6180  VTAB 24: HTAB 5
6190  PRINT "Is this the correct cycle type ?";
6200  GET TMP$
6210  IF TMP$ = "Y" OR TMP$ = "y" THEN 6250
6220  IF TMP$ = "N" OR TMP$ = "n" THEN 6090
6230  GOTO 6200
6250  VTAB 24: HTAB 1: CALL  - 868
6255  NEXT CCYCLE
6330  VTAB 16: HTAB 1: CALL  - 868
```

CONTROLLER

```
2635  CALL  - 868: INVERSE : PRINT " Press 'C' to not halt ": NORMAL
2636  GOTO 2639
2637 FSTP = 0: VTAB 3: POKE 36,50
2638  PRINT "Press F to stop at cycle"
2639  POKE  - 16368,0
2640  IF COOL AND TEMP <  = RTEMP OR  NOT COOL AND TEMP >  = RTEMP THEN
      GOTO 2655
2650  GOTO 2630: REM   NEXT TEMP
2655  IF TD$(TC(CCYCLE)) = "N" THEN 2730
2660  GOSUB 300: REM  TAKE CND READING
2670 SDT(PNT,0) = TEMP
2680 SDT(PNT,1) = CND
2705  HTAB 40: PRINT "        ";
2710  HTAB 40: PRINT CND;
2715  POKE 36,51: PRINT "       ";
2720  POKE 36,51: PRINT PNT
2730  IF RTEMP <  = BTTM(TC(CCYCLE)) AND COOL OR RTEMP =  > TP(TC(CCYCLE))
      AND  NOT COOL THEN 3000: REM  AT END OF CYCLE FOR SAMPLE
2740 RTEMP =  INT ((RTEMP + INVL(TC(CCYCLE))) * 100) / 100
2750 PNT = PNT + 1
2760  GOTO 2630
3000  REM  SAVE OFF DATA
3010  GOSUB 1000: REM  SAVE TO TEMPORARY DISK
3020  IF  NOT FSTP OR COOL AND FSTP THEN 3130
3030  GOSUB 500: REM  SHUT OFF HEATER
3040  VTAB 15: HTAB 5
3050  PRINT "Program halted, do you wish to continue?";
3060  GET TMP$
3070  IF TMP$ = "Y" OR TMP$ = "y" THEN 3100
3080  IF TMP$ = "N" OR TMP$ = "n" THEN  CALL  - 868: GOTO 3140
3090  GOTO 3060
3100  HTAB 5: CALL  - 868: REM  ERASE LINE
3110  VTAB 3: POKE 36,50: PRINT "Press F to stop at cycle"
3120 FSTP = 0
3130  IF (CCYCLE - LCYCLE = NCYCLE OR CCYCLE = MCYCLE) AND  NOT COOL THEN
      3140
3135  GOTO 3180
3140  REM   NO NEED TO TRANSFER DATA TO PERMANENT DISKS
3165 LCYCLE = CCYCLE
3170  IF (CCYCLE = MCYCLE) OR FSTP THEN 3210
3180  IF COOL THEN  GOSUB 600: GOTO 3200: REM  TURN ON HEATER
3190  GOSUB 550: REM  TURN ON COMPRESSOR
3200  GOTO 2610: REM  START NEXT CYCLE
3210  GOSUB 500: REM  TURN OFF HEATER AND QUIT
3220  VTAB 24: HTAB 5
3230  PRINT "Program finished    "
3240  PRINT : PRINT
3250  PRINT D$"RUN STARTUP,D1"
```

CONTROLLER

```
2174  POKE 36,70: PRINT "    ";: POKE 36,70
2175  GOTO 2172
2178 RA(X) = TMP
2250  VTAB 20: HTAB 10
2260  PRINT "Temp Interval Between Readings : ";
2270  GOSUB 1900
2280  IF TMP <  = (TP(X) - BTTM(X)) AND TMP >  = 0 THEN 2302
2290  POKE 36,42: PRINT "     ";: POKE 36,42
2300  GOTO 2270
2302  IF TMP = 0 THEN TD$(X) = "N":INVL(X) = .1: GOTO 2440
2304 TD$(X) = "Y"
2305 INVL(X) = TMP
2440  VTAB 24: HTAB 5
2450  PRINT "Is all information correct?";
2460  GET TMP$
2470  IF TMP$ = "Y" OR TMP$ = "y" THEN  HTAB 5: CALL  - 868: GOTO 2492
2480  IF TMP$ = "N" OR TMP$ = "n" THEN 2080
2490  GOTO 2460
2492  NEXT X
2493  GOSUB 6000: HOME
2496 CCYCLE = 0
2500  POKE 35,15: HOME : TEXT : VTAB 5: HTAB 30
2510  PRINT "Sample Description     Starting Cycle"
2515  VTAB 6: HTAB 18: CALL  - 868: PRINT "Sample  1";: INPUT DIS$
2516  VTAB 6: POKE 36,55: GOSUB 1900:SCYCLE = TMP
2520  VTAB 15: HTAB 5: PRINT "Is This name correct? ";: GET TMP$
2522  IF TMP$ = "Y" OR TMP$ = "y" THEN 2528
2525  IF TMP$ = "N" OR TMP$ = "n" THEN 2500
2527  GOTO 2520
2528  POKE 35,15: HOME : TEXT : GOSUB 7000
2530  VTAB 5: HTAB 30
2532  PRINT "Temp (C)  Cond (uS)  Point"
2534  VTAB 6: HTAB 5: PRINT  LEFT$ (DIS$,1)
2535  VTAB 6: HTAB 18: CALL  - 868: PRINT "Sample  1"
2545  VTAB 3: POKE 36,50: PRINT "Press F to stop at cycle"
2550  DIM SDT(400,1)
2562 MR = 30
2565  DIM AR(MR)
2580 RTEMP = TP(TC(1))
2600  GOSUB 550: REM  START COMPRESSOR
2610  GOSUB 6500
2620  GOSUB 700: REM  INITIALIZE
2630  GOSUB 100: REM  READ TEMP
2631  IF  ABS (OSEC - U6) >  = TS THEN U6 = OSEC:OT = OT + SD: IF OT >  = 0
      AND OT <  = 255 THEN  POKE D1,OT
2632  IF  PEEK ( - 16384) = 195 THEN 2637
2633  IF  PEEK ( - 16384) <  > 198 THEN 2639
2634 FSTP = 1: VTAB 3: POKE 36,50
```

```
1950  IF  ASC (K$) = 8 AND  LEN (TMP$) = 1 THEN TMP$ = "": PRINT  CHR$ (8);
      " "; CHR$ (8);
1952  IF  ASC (K$) = 8 AND  LEN (TMP$) > 1 THEN TMP$ =  LEFT$ (TMP$, LEN
      (TMP$) - 1): PRINT  CHR$ (8);" "; CHR$ (8);
1955  GOTO 1920
1960 TMP$ = TMP$ + K$
1965  PRINT K$;
1970  GOTO 1920
1980 TMP =  VAL (TMP$)
1990  RETURN
2000  REM  PRE MAIN PROGRAM
2005  GOSUB 4000
2020 PSLOT = 4:ISLOT = 1:COOL = 0:LCYCLE = 0:D$ =  CHR$ (4):INVL = 0
2030 FSTP = 0:CCYCLE = 0
2035 M1 = 255 / ( - 35 - 7):B = 7 *  - M1:D1 =  - 16384 + ISLOT * 256 + 1:
OKE D1 + 2,255
2040 A1 =  - 16384 + PSLOT * 256 + 1:B1 =  - 16384 + PSLOT * 256
2050 A3 = A1 + 2:B3 = B1 + 2
2057 C1 =  - 16256 + PSLOT * 256 + 1:C3 = C1 + 2
2058  POKE A3,0: POKE B3,0: POKE C3,254
2059  GOSUB 3500: REM  GET NOTES
2060  HOME
2062  VTAB 16: HTAB 2: PRINT "# of Cycle Types : ";
2064  GOSUB 1900
2066  IF TMP =  > 1 THEN 2072
2068  HTAB 21: PRINT "     ";: HTAB 21
2070  GOTO 2064
2072 CT =  INT (TMP)
2074  DIM TP(CT),BTTM(CT),INVL(CT),RA(CT),TD$(CT)
2076  FOR X = 1 TO CT
2080  HOME : VTAB 16: HTAB 2: PRINT "Cycle Type : ";X
2085  VTAB 18: HTAB 4
2087  PRINT "Maximum Temp : ";
2090  GOSUB 1900
2092  IF TMP <  = 7 THEN 2100
2094  POKE 36,18: PRINT "    ";: POKE 36,18
2096  GOTO 2090
2100 TP(X) = TMP
2110  HTAB 30
2120  PRINT "Minimum Temp : ";
2130  GOSUB 1900
2140  IF TMP < TP(X) THEN 2170
2150  POKE 36,44: PRINT "       ";: POKE 36,44
2160  GOTO 2130
2170 BTTM(X) = TMP
2171  VTAB 18: POKE 36,58: PRINT "Temp Rate : ";
2172  GOSUB 1900
2173  IF TMP > 0 AND TMP <  = 30 THEN 2178
```