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A STUDY OF SOUTH DAKOTA DEICER NUMBER 2:

FINAL REPORT

By: T. Ashworth

Prepared for:

South Dakota Department of Transportation
700 Broadway Avenue East
Pierre, South Dakota 57501-2586

Contract Number 3213

Department of Physics
South Dakota School of Mines & Technology
Rapid City, South Dakota 57701

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FOREWORD

This final report was prepared by the South Dakota School of Mines and Technology (SDSM&T) for the South Dakota Department of Transportation under Project No. 3213. It covers the work performed and findings made during the period of the contract 1 June, 1987 to 30 September, 1988.

The report was prepared by Dr. T. Ashworth, Principal Investigator, Professor and Head of Physics, SDSM&T. Team members who have assisted in the conduct of the research include Dr. J. A. Weyland, Professor of Physics, Dr. R. W. Looyenga, Associate Professor of Chemistry, Dr. W. A. Klemm, Professor of Chemical Engineering, Messrs. Anthony P. Ewing, Louie L. Lu, and R. David Wheeler, Graduate Research Assistants and M.S. candidates in Physics, and Messrs. Stephen F. Ashworth, William P. Donley and Nancy Neuharth, Undergraduate Assistants.

T. Ashworth, Ph.D.
Principal Investigator

"A Study of South Dakota Deicer Number 2: Identification of Components and/or Synergies Responsible for Freezing Point Depression and Prevention of Ice Adhesion."

ABSTRACT

This report documents the work performed under Contract No. 3213 to the South Dakota Department of Transportation. The deicing materials developed by S.D. DOT have been evaluated together with their component materials. It has been found that the deicer designated SD2 is effective in greatly reducing the bond strength of ice to Portland Cement Concrete which has been treated with the material. Also, it is suggested that the effectiveness of SD2 is a result of a synergism between its components. An apparatus has been developed which can monitor the cooling curve of solutions through their freezing points. Experiments in this device have shown that SD2 freezes into a disordered solid, as does one of its components, sodium glycolate. It is concluded that there could be very significant benefits from a continuation of this work, not only to determine whether the properties of SD2 can be attributed to a synergistic mechanism, but also to seek a fundamental understanding which, in time, may allow the development of equally efective, but perhaps less expensive, deicers.

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I. INTRODUCTION AND OBJECTIVES

This report documents the work performed in the Department of Physics, South Dakota School of Mines and Technology under Contract Number 3213 with the South Dakota Department of Transportation. The work has been carried out during the period 1 July, 1987 to 30 September, 1988.

I-A. Objectives

Our specific objectives were to:

1) Evaluate the effectiveness of the "South Dakota Deicers", particularly South Dakota Deicer Number 2 (SD2), in reducing the interfacial strength of ice bonded to Portland Cement Concrete as determined by Shear Test Measurements. In these tests we were to study both the compound materials and the component materials; the deicing materials were used as a pre-treatment on the concrete substrates.

2) Determine some of the freezing characteristics of SD2 and its component materials. For this we chose to develop a true cooling curve apparatus rather than simply cool solutions to determine the freezing point depression.

3) Prepare a proposal suitable for submission to the SHRP IDEAS program for work whose objective is to continue efforts to understand the mechanisms which lead to the properties of SD2.

I-B. Background

A staggering economic and environmental burden is being imposed upon the United States by the continued widespread use of sodium chloride on the nation's highways - now over 10 million tons per year [1]; the extent and seriousness of the damage to highways, bridges, automobiles, water resources, and the ecology has now begun to be realized [2,3,4]. In recent years the search for effective, inexpensive, and environmentally benign deicers has begun in earnest. A number of deicing materials (alcohols, CMA, NaCl with corrosion inhibitors, etc) have been developed and tested. There is, however, still a tremendous need for understanding at the fundamental level of the physical and chemical mechanisms involved in the adhesion of ice to all types of surfaces. It is this fundamental knowledge which is necessary as the basis of advancement of all forms of deicing techniques.

It is clear that the bonding of ice to a solid surface is complex. This was apparent from the work carried out in the late 1950's, especially the important work of Jellinek [5] and of Raraty and Tabor [6] on adhesion to metals and plastics. Work described in the latter reference brings out more fully the interaction between the breaking of an ice/substrate bond and the properties of the bulk ice as well as the properties of the interface itself. Jellinek [7] and Glen [8] earlier had demonstrated the considerable complexity of the mechanical properties of ice. Work has continued over the years; several studies have been reported recently, notably those conducted at USACRREL. Currier, Schulson and St. Lawrence's work [9], for example, included measurements on carefully grown and characterized specimens, and they monitored simultaneously the acoustic emissions from the ice to obtain information on microfracture during deformation.

When ice is formed on Portland Cement Concrete or asphalt, even more factors are involved in the adhesion and abhesion processes. Still we must consider the mechanical properties of the ice and the cohesive forces between these materials, but they are now further complicated by the non-homogeneity of the pavement materials and the variety of bonding available. And the ability to develop adhesive bonding between the ice and the substrate by the mechanical interlocking of the frozen liquid into and around irregularities (especially pores and open structure) must also be incorporated into the overall adhesion picture. These considerations are reflected in the conceptual model we have described earlier. It is clear from this model that there are several ways in which the overall bond strength can be reduced.

The serious search for materials or treatments which could prevent the formation of a strong interfacial bond began in the late 1950's, for example see references [10] and [11]. With the increased recognition of the corrosion and environmental problems associated with the vast use of sodium chloride, the search for alternatives intensified in the 1970's. Several investigations and analyses resulted. The first international symposium on Snow Removal and Ice Control Research was organized at CRREL by L. D. Minsk; of the many relevant papers presented, two of the most important are Jellinek's survey of ice adhesion work [12], and Ackley and Itagaki's report on ice adhesion studies [13]. The Environmental Protection Agency issued a report on a search for new technology in 1972 [14], the State of California reported on an evaluation of deicing chemicals in 1974 [15], the National Cooperative Highway Research Program sponsored an evaluation of alternative techniques [16], and several reports appeared on attempts to find or develop hydrophobic and icephobic surfaces (for example references [17] and [18]). Under the sponsorship of the U.S. Department of Transportation, basic studies of the forces of adhesion were initiated at the South Dakota School of Mines and Technology in 1977. Results of this work and continuation work are presented in two reports and a thesis [19,20,21]; a report on additional work is in preparation. Several relevant papers were presented at the 1978 Symposium on Snow Removal and Ice Control Research. Cook reported on icephobic coating for highway surfaces, Dunn and Schenk discussed alternative deicing chemicals, and Minsk and Kehl summarized objectives for the future [22].

Work in the 1980's has concentrated on the development of the CMA deicer and the prevention of ice adhesion on metal surfaces (especially waterway structures, electrical systems, and water-eroded helicopter blades), and the use of brine deicing techniques [23,24]. Emphasis has also been placed on the minimization of deicer application rates [25]. Results of the several projects on CMA have been summarized by Chollar [26]. Considerable progress has been made using surface treatments of silicone compounds and block copolymers [27,28]. Overall it is clear that coatings and treatments can be used to reduce the adhesion of ice to surfaces. Suggestions have been made that monolayers of polymeric and other types of materials [21] may be effective. Results from the Italian brine pretreatment program are encouraging; work is needed to increase treatment retention on the pavement. Independent studies on incorporated, encapsulated calcium chloride (Verglimit) are also showing promise [29]. However, it still remains to identify treatments or coatings for highway pavement materials which are effective in preventing the formation or strong bonding of ice, are long-lasting or can easily be renewed, which do not significantly reduce the effective coefficient of friction, and which rival the traditional use of sodium chloride in economy. In this regard it would be helpful if the true cost of sodium chloride were determined, rather than having new materials or techniques compared simply to the cost of the material and its application alone.

I-C. Development of the "South Dakota" Deicers

In March of 1980, Bjorksten Research Laboratories, Inc., of Madison, Wisconsin, published their research which was directed towards the development of a non-corrosive alternative to NaCl [30]. Their report indicated two chemicals of choice, namely, methanol and calcium magnesium acetate (CMA). The evidence presented indicated that CMA has several desirable qualities which are not completely applicable to other deicing options; in particular, CMA shows no significant corrosion of steel, zinc or aluminum, it exhibits corrosion inhibition towards A-36 steel and A-3560 cast aluminum, neither the calcium nor magnesium ions present any more toxicity hazard than the sodium ion, CMA contains no nitrogen or phosphorus and therefore does not increase lake eutrophication, and it is essentially non-toxic and non-flammable.

Since the Bjorkstein report, quantities of CMA adequate for field trials have been produced. Several reports are now available on these tests. As yet, there is not complete agreement on the effectiveness of the material, nor on it's claimed non-corrosive properties; some test results were clearly favorable, while others were somewhat ambivalent. For a summary of the most recent and ongoing work under the auspices of FHWA, see FCP ANNUAL PROGRESS REPORT, YEAR ENDING SEPTEMBER 30, 1985, PROJECT 3C.

Interest in non-corrosive deicers by the South Dakota Department of Transportation led to an investigation by the Chemical Engineering Department at SDSM&T of the feasibility of producing CMA from locally available sources of dolomitic lime and cellulosic waste. Atmospheric fusion did not result in conversion to acetic acid. However, it was discovered that cellulose degradation under basic conditions at high temperatures and pressures could be used to produce a calcium lactate-acetate-glycolate compound; this was designated as "South Dakota Deicer No. 1", or SD1. Further work on similar sodium-based compounds lead to the development of SD2; in May, 1987 a patent (United States Patent Number 4,664,832) was granted to the State of South Dakota for this material. In the patent, the material is described as having a composition of:

Sodium glycolate	33.1%
Sodium formate	31.8%
Sodium acetate	26.3%
Sodium maleate	6.4%
Sodium fumarate	1.4%

and small quantities of sodium lactate, sodium malate, sodium malonate, and sodium tartrate.

Initial testing was performed by personnel in the Chemical Engineering Department and SDDOT. The results were promising. In aqueous solution, SD2 has a low eutectic point, it has high solubility in water, is non-corrosive and non-toxic.

Preliminary testing of the adhesive strength of ice to concrete treated with SD2 was performed in the Physics Department during the period December, 1986 through May, 1987. Our results were very encouraging, and led to the work described in this report. Although this work is aimed primarily at determining the properties of the materials, it also begins to address questions about how they work. For example, what is the active element in SD2? Is it one of the major components acting individually, or is it some of the components acting in combination? Rough measurements of the freezing point together with other indirect evidence suggest not, but this needed to be shown systematically.

I-D. Evaluation Program

Evaluation of the materials has been performed by determining the interfacial strength of ice formed on Portland Cement Concrete substrates which have been treated with aqueous solutions of the various salts. A carefully defined test procedure has been developed, as described below. The measurement sequences performed include one series at a fixed application rate (200#/lm equivalent) as a function of test temperature, and another series at fixed test temperatures ($-5^{o}C$ and $-15^{o}C$) as a function of application rate.

In addition to the effect of the salts on the ice-concrete interface, an attempt was made to determine some of the properties of the salts in aqueous solution. Some measurements of solubility were to be performed; this was hampered by difficulty in obtaining adequate quantities of some of the salts. The main thrust of this portion of the work was to develop an apparatus capable of monitoring the cooling and freezing process in a manner which allowed the freezing point depression and eutectic temperature to be determined. Also, the apparatus was to be capable of monitoring the cooling curve and allowing rough estimates of parameters such as the specific heat of the materials in solution and solid forms, and the latent heat of phase transformations.

Both sections of this investigation have produced remarkable results.

II. INTERFACIAL SHEAR STRENGTH TEST PROCEDURE.

II-A. General Description

The basic system for interfacial shear testing was developed as part of a previous research project "Investigation of the Basic Forces Involved in the Adhesion of Ice to Highway Surfaces" sponsored by the Federal Highway Administration. It consists of a Cal-Tester Model TH-5 5000lb tester mounted outside a freezer with the loading members passing through the side of the freezer into an insulated, temperature controlled box as shown in figure 1. Plastic bushings through the freezer wall allowed for smooth operation of the tester without significant moisture infiltration. The sample holder was mounted on the frame of the testing machine. Temperature control is achieved with a thermistor temperature sensor, an on/off regulator controlling the supply of power to an electric light bulb, and an air circulation fan. When the box remains closed, the temperature can be regulated to within one tenth of a degree Celsius. Further details of the system can be found in references [19] and [20] and the M.S. Theses of Louie L. Lu [31] and Anthony P. Ewing [32].

II-B. Substrate Specification

The concrete substrates were prepared in one batch according to ASTM specification C-192-76. Data for the concrete is as follows: Air content 6%, slump 2.25 inches, W/C ratio 0.49, 36% fine aggregate consisting of natural river sand, 64% coarse aggregate consisting of Minnekahta crushed limestone of maximum size 1", a 28 day strength of 5260 psi, unit weight 2403 kg/m^3, and date of mix 16 March, 1979.

II-C. Specimen Preparation

Test specimens are produced by freezing water in Teflon rings which are placed on the substrate being studied, as shown in figure 2. To ensure that the substrates are prepared in a standard manner, the procedure was developed as shown in the flow diagram of figure 3 and defined as follows:

1) Soak the substrate in tap water for 15-18 hours (usually overnight following the test of the previous day).
2) Use brush and running tap water to clean substrate.
3) Rinse and brush substrate in distilled water.
4) If changing to a different test series (change in deicer chemical) then let soak in distilled water for 30 minutes. Test electrical conductivity of soak-water and if less than 3 X 10^{-6} mhos go to next step, otherwise, repeat cleaning. If not changing chemicals then also go to next step.

(NOTE: at each change of deicer chemical, a reference test of interfacial shear strength on the untreated substrates must be made to ensure that the substrates are clean. If the reference test is below the original value of shear strength on the untreated substrates (within 0.5 kg/cm^2), cleaning and untreated shear tests are repeated until it is.)

5) Let substrates air dry.
6) Measure amount of chemical to be tested based on application rate X (in previously defined units of #/lm).

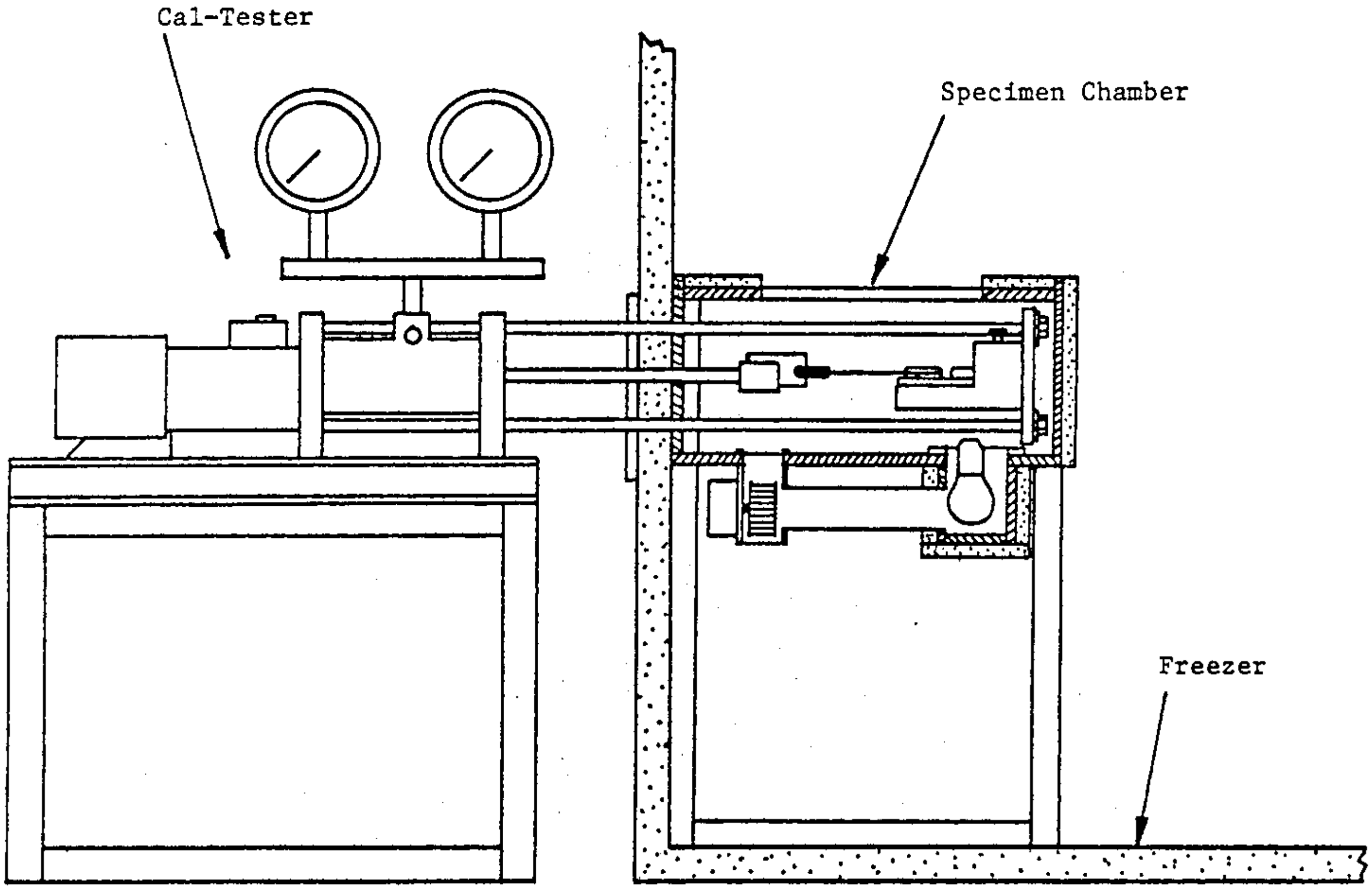

Figure 1. Shear Testing System

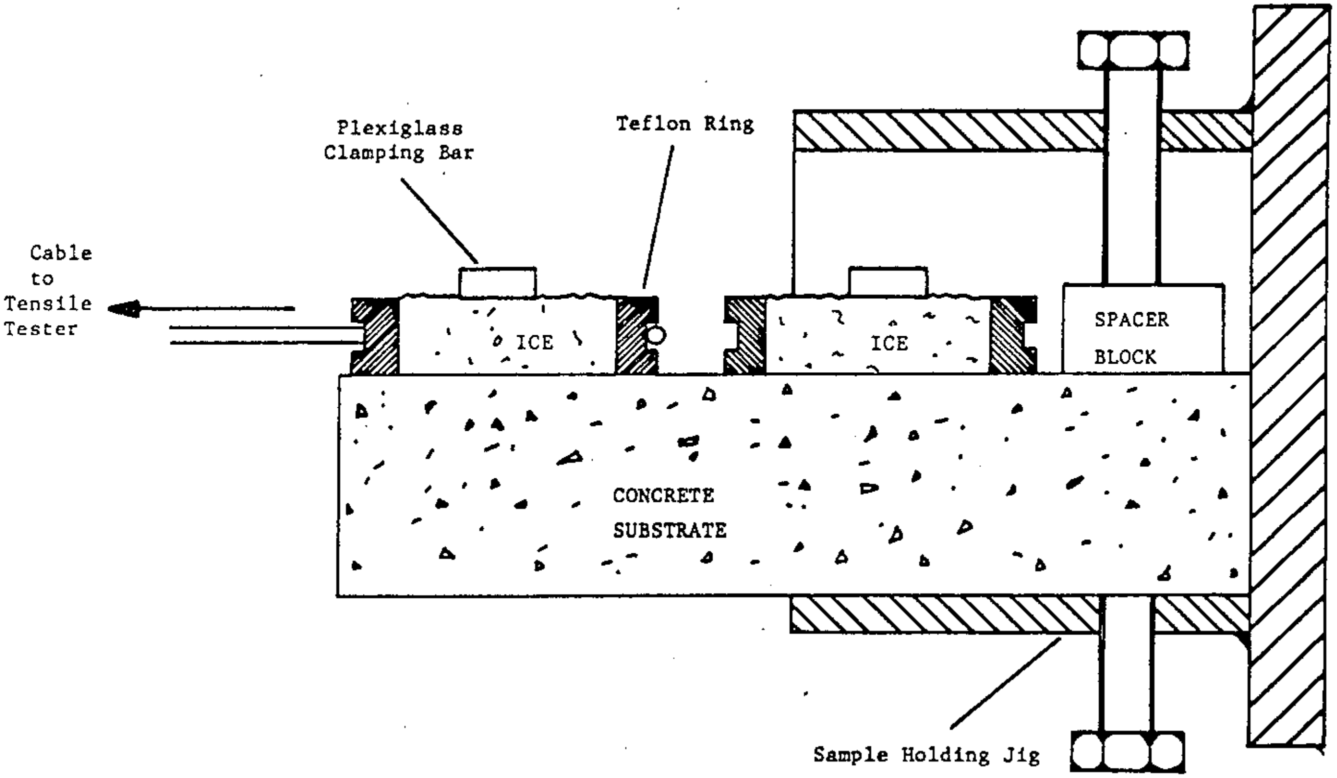

Figure 2. Test Specimen Arrangement

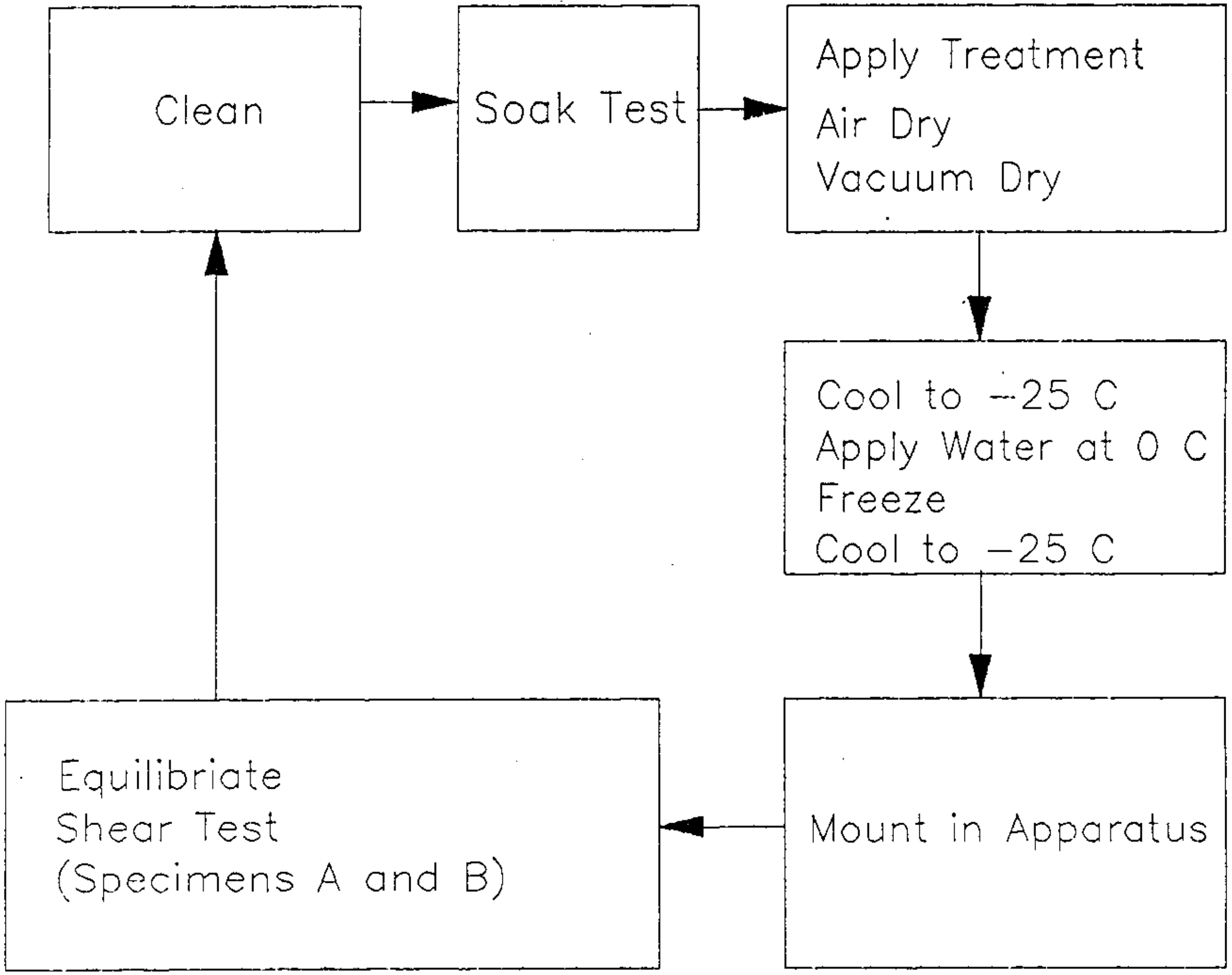

Figure 3. Flow Diagram for Shear Strength Tests

7) Add 6 ml of distilled water to the measured out chemical (1.5 ml X 4 substrates = 6 ml).
8) Pour 1.5 ml (which allows complete wetting of surface) onto each substrate and use a small brush to distribute evenly.
9) Allow prepared substrates to air dry completely (minimum 4-5 hours).
10) Using rubber bands, attach 2 teflon rings over the designated areas on each of the dried, prepared substrates.
11) Place prepared substrates in freezer overnight (-25°C).
12) Pour distilled water (at 0°C) into each of the 8 teflon rings.
13) After 1 hour, transfer substrates to temperature controlled environment containing the shearing apparatus.

II-D. Details of Testing Sequence

The temperature in the testing chamber can be controlled to within 0.05°C. However, measurements indicate that the substrate-ice interfacial temperature changes as much as 0.5°C in the two minutes it takes to change samples. In order to reduce the amount by which the freezer temperature rises while the substrate is being changed, a large sheet of aluminum-faced insulation has been inserted near the top of the freezer so that when the freezer is opened only the sample chamber is subjected to room temperature. This tends to speed up the cool-down recovery time of the test chamber. The temperature control system typically takes about two hours to reach a given temperature within the range -5°C to -15°C within the specimen test chamber. However, a much longer time is needed to get down to a temperature of -20°C, requiring it to be left overnight.

After one hour of having set the chamber temperature desired for testing, the four substrates used for a test (four substrates each with two test specimens provide eight replications for each test) are transferred from the specimen preparation freezer to the testing chamber. The first substrate is mounted in the test apparatus. Two cables are used, one for each teflon ring; this makes it possible to test both samples on a substrate without having to open the sample chamber. The arrangement is shown in figure 4.

Four hours are allowed for the substrates to come to thermal equilibrium at the test temperature. Measurements on a test specimen with a thermocouple embedded at the interface have shown this to be more than adequate. Two tests are conducted on the first substrate, then the test chamber is opened to remove the first substrate and mount the second substrate. One hour is then allowed for re-establishment of thermal equilibrium before the second substrate is tested. The same procedure is followed for testing the third and fourth substrates.

The shear rate used in all these tests is approximately 4000 lbs/minute - the fastest rate available from the Cal-Tester. Ice breaks off the substrate typically at 50-300 lbs. The force exerted at failure is recorded by the auxiliary pointer on the pressure gauge.

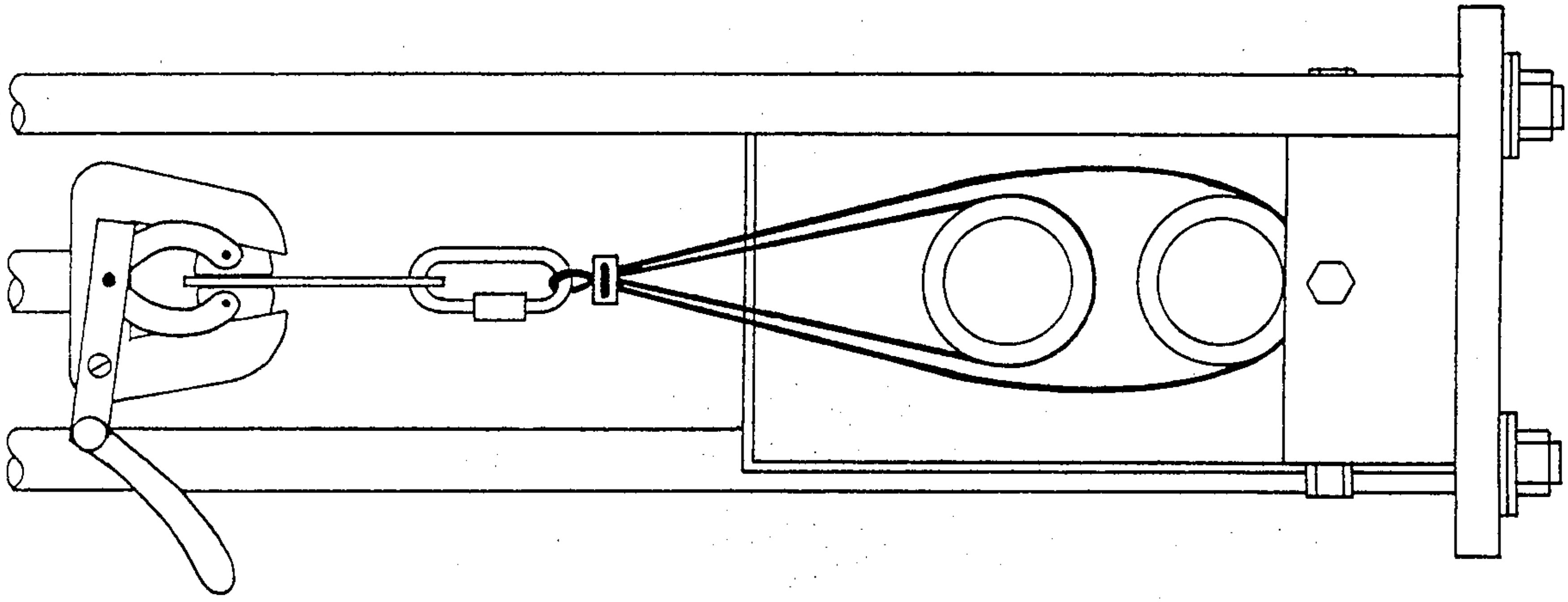

Figure 4. Specimen/Cable Arrangement

III. INTERFACIAL SHEAR STRENGTH TEST RESULTS

It has been found in previous studies that there is a significant amount of variation of the adhesive strength of ice on concrete substrates. This is also true for different sites on a given substrate, presumably due to the random distribution of aggregate and mortar in a surface. It was also found that the variation in the values of interfacial strength as measured on a particular site was significantly smaller than that for several different sites. As a result, the position of the Teflon rings on each substrate has been kept the same for each test. Also, comparison of deicing materials has only been made between tests conducted on the same set of substrates. Although there is an overall average standard deviation of 10% in the interfacial strength on untreated P.C.C., values obtained from a particular site of a given substrate usually have a standard deviation of about 6%. For tests on treated substrates, the standard deviation is usually higher than for untreated substrates. This is partly due to the fact that tests were repeated several times on untreated substrates as the test procedure was being developed, and the process of treatment adds further opportunity for variability to enter the test. Besides the possibility of variation of the distribution of treatment material due to uneven application or adsorption into the concrete, there is also a possibility of variation in the amount of treatment material which is redissolved into the water as it is poured onto the test surface. In general, the absolute scatter in measured shear strength values is proportionately smaller when the shear strength for a given treatment is smaller. Thus for all the data given, the actual standard deviation between results is approximately 20%, but the relative certainty between data points may be somewhat better than this.

All the data obtained are given in tabular form in the appendix to this report.

III-A. SD2 Compared to Sodium Chloride and Calcium Chloride

These materials were compared in measurement series A1, A2, and B1, the results of which are shown in figures 5a, 5b, and 5c. Series A1 and A2 determined the shear strength of ice on treated P.C.C. substrates as a function of application rate (at -5^{o}C and -15^{o}C respectively), and series B1 explored the dependence upon temperature for an application rate of 200 #/lm. These data show that SD2 is significantly more effective than NaCl or $CaCl_2$ in all cases where the application rate is 100 #/lm or greater. Also shown in figure 5a are data for SD1. In figure 5c, data for sodium formate and sodium glycolate are given; measurements using sodium acetate treatment are in progress.

III-B. SD2 Compared to its Component Materials

Figures 6a, 6b, and 6c allow one to compare SD2 with its component materials. Roughly speaking, SD2 is composed of equal parts of sodium glycolate, sodium formate, and sodium acetate. Because of this, for comparison of results shown on figure 6, it is well to keep in mind that an application rate of 300 #/lm of SD2 is approximately comparable to 100 #/lm of these three major individual constituents. It should also be kept in mind that the most abundant minor components, sodium maleate and sodium fumarate, constitute only 6% and 1% of SD2, respectively.

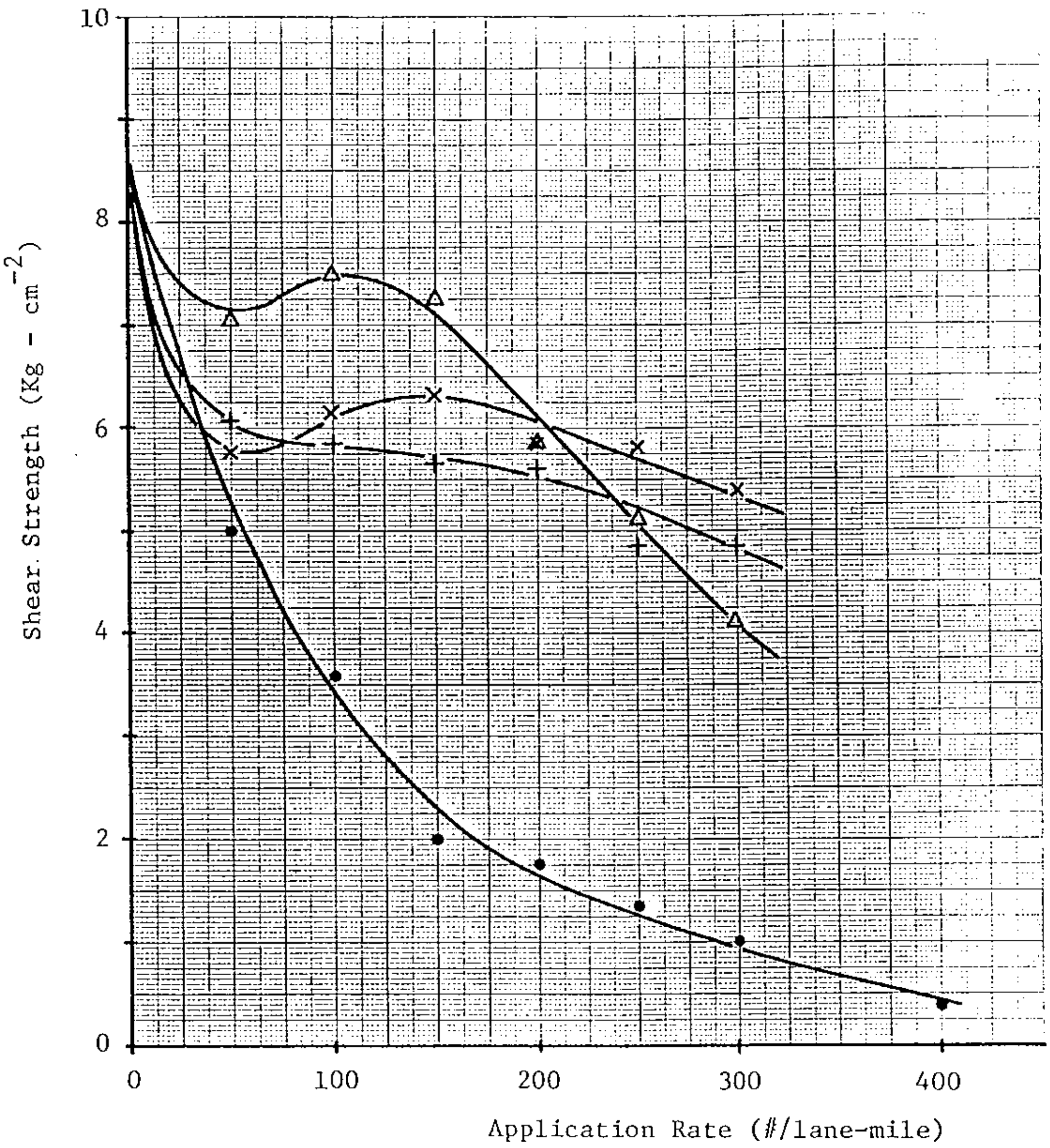

Figure 5a. Interfacial Shear Strength v. Application Rate

Measurement Series A1: P.C.C. Substrate, -5°C

● - SD2-I △ - SDI + - NaCℓ × - $CaCℓ_2$

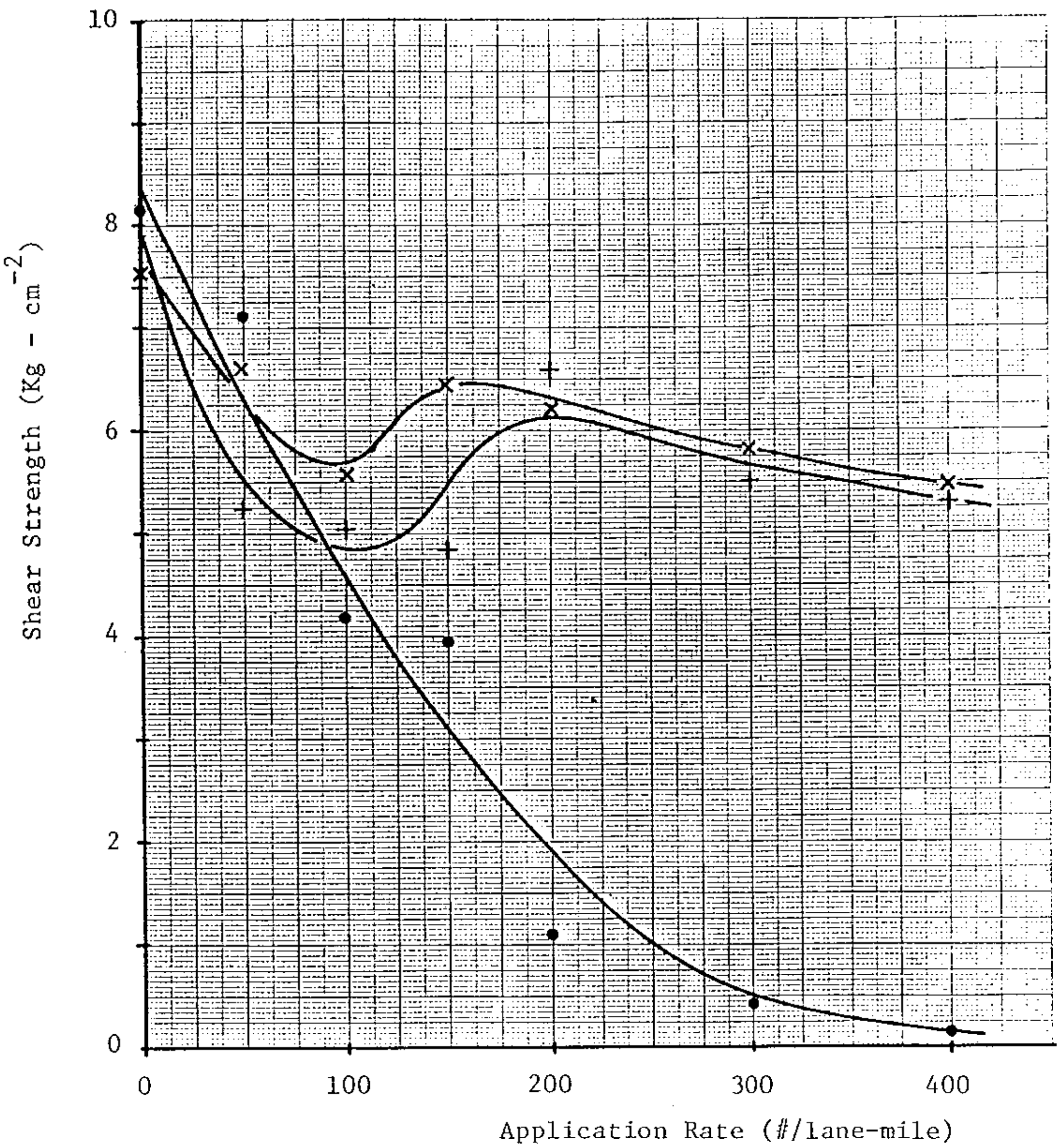

Figure 5b. Interfacial Shear Strength v. Application Rate

Measurement Series A2: P.C.C. Substrate, -15°C

● - SD2-I △ - SDI + - NaCℓ × - CaCℓ$_2$

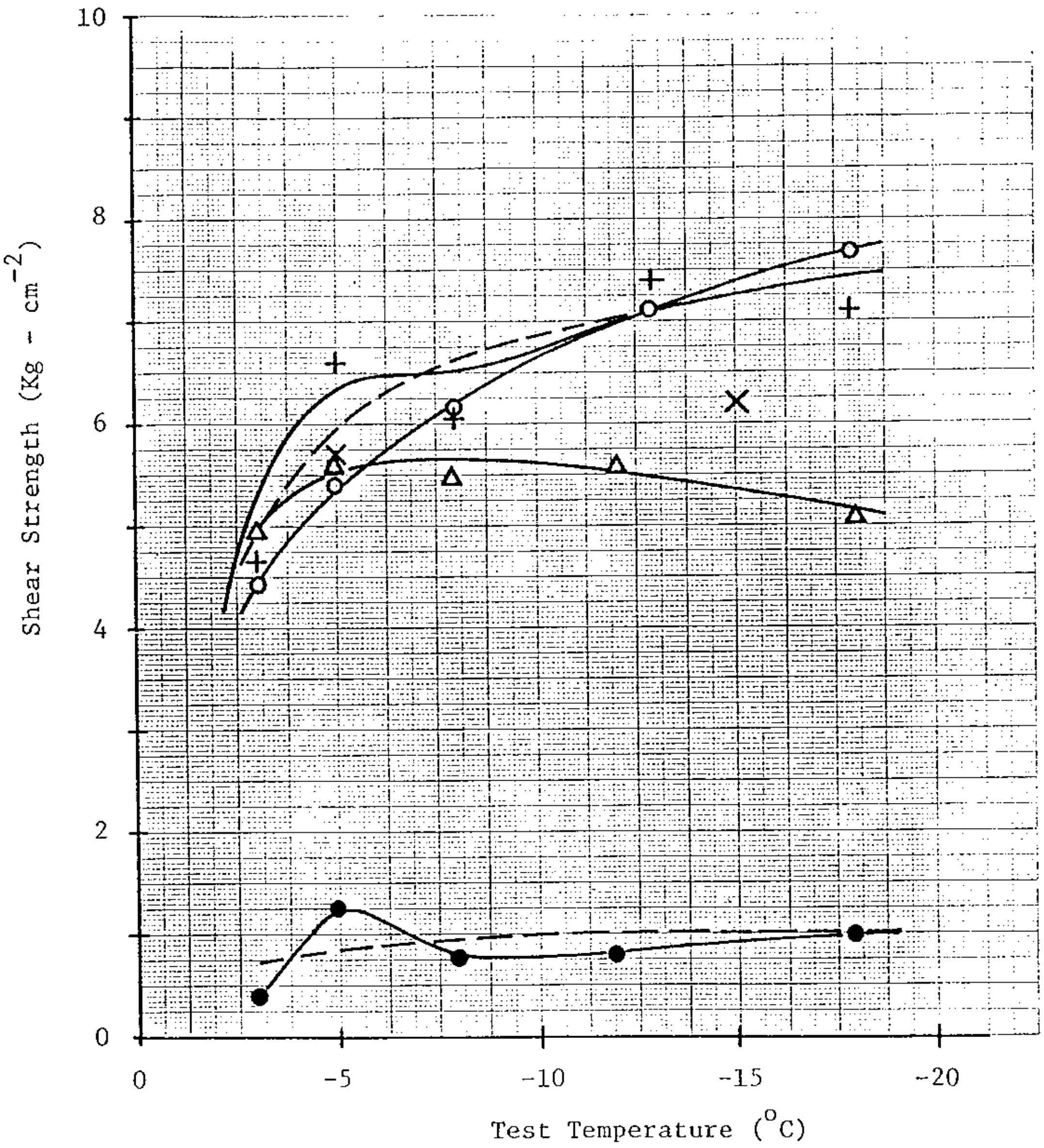

Figure 5c. Interfacial Shear Strength v. Test Temperature

Measurement Series B1: P.C.C. Substrate, 200#/lane-mile

● - SD2-I + - NaCℓ × - CaCℓ$_2$ ○ - Na Formate

△ - Na Glycolate

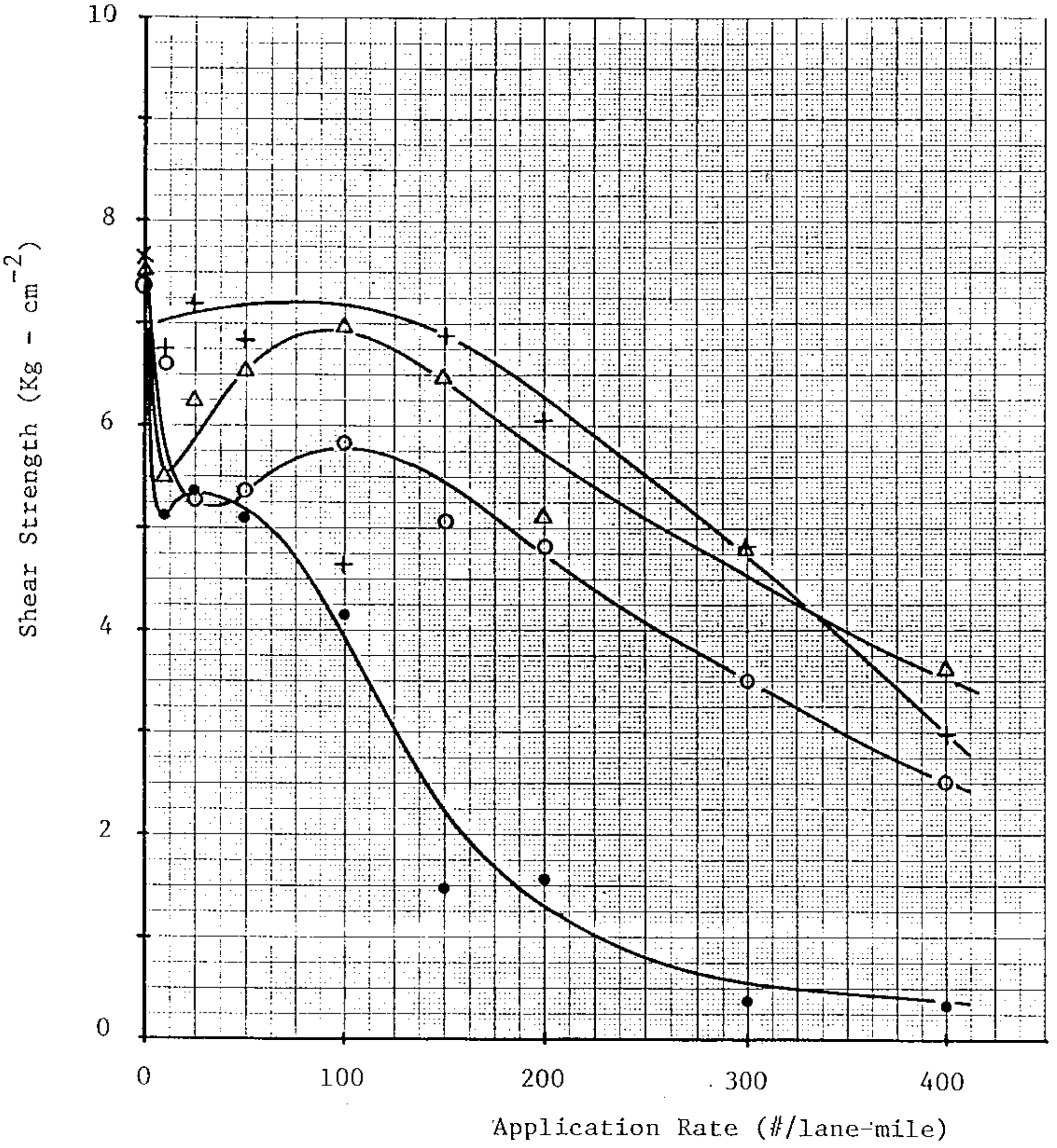

Figure 6a. Interfacial Shear Strength v. Application Rate

Measurement Series C1: P.C.C. Substrate, -5°C

● - SD2-I

○ – Na Formate   △ - Na Glycolate   + - Na Acetate

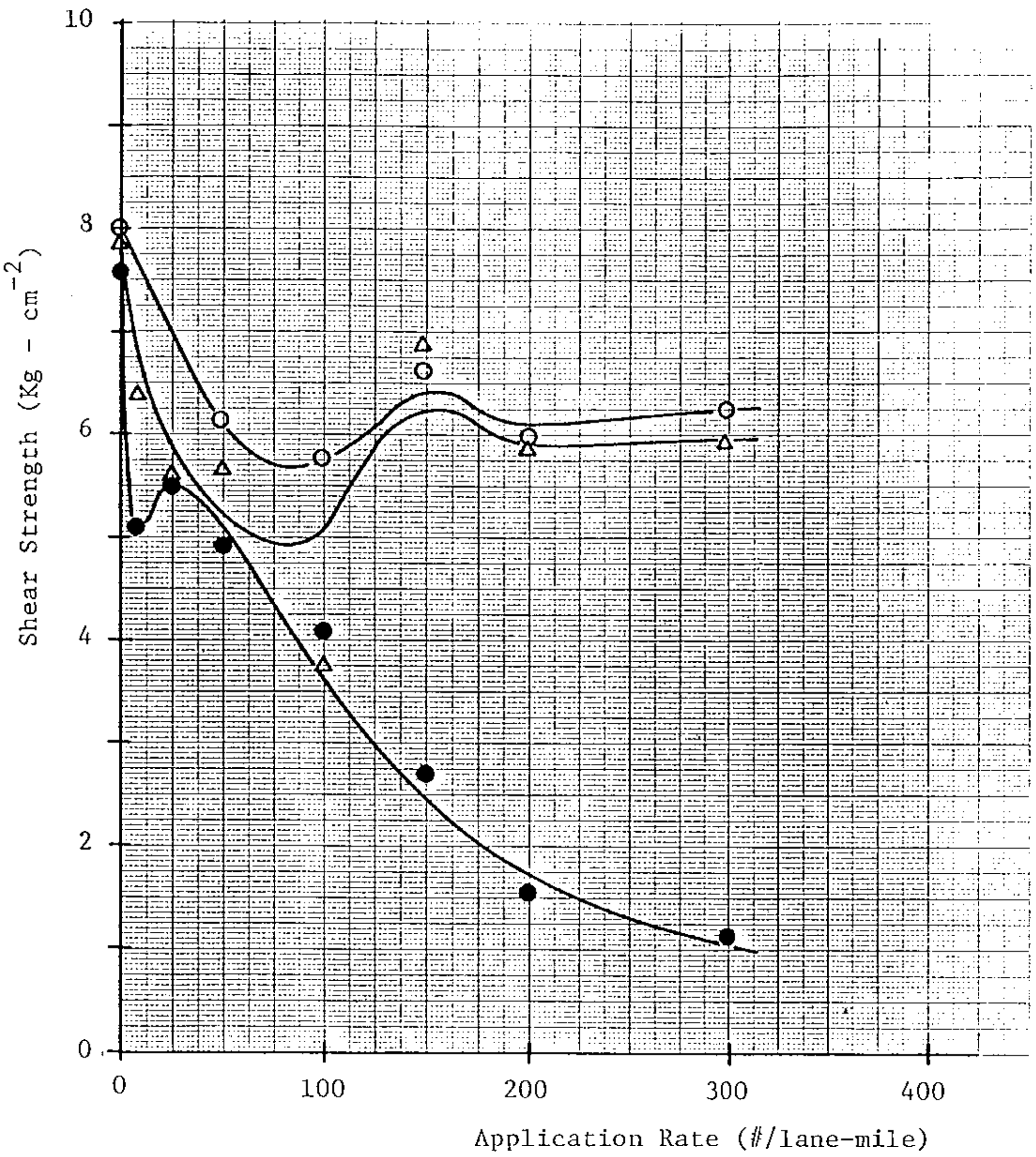

Figure 6b. Interfacial Shear Strength v. Application Rate

Measurement Series C2: P.C.C. Substrate, -15°C

● - SD2-I

○ - Na Formate △ - Na Glycolate + - Na Acetate

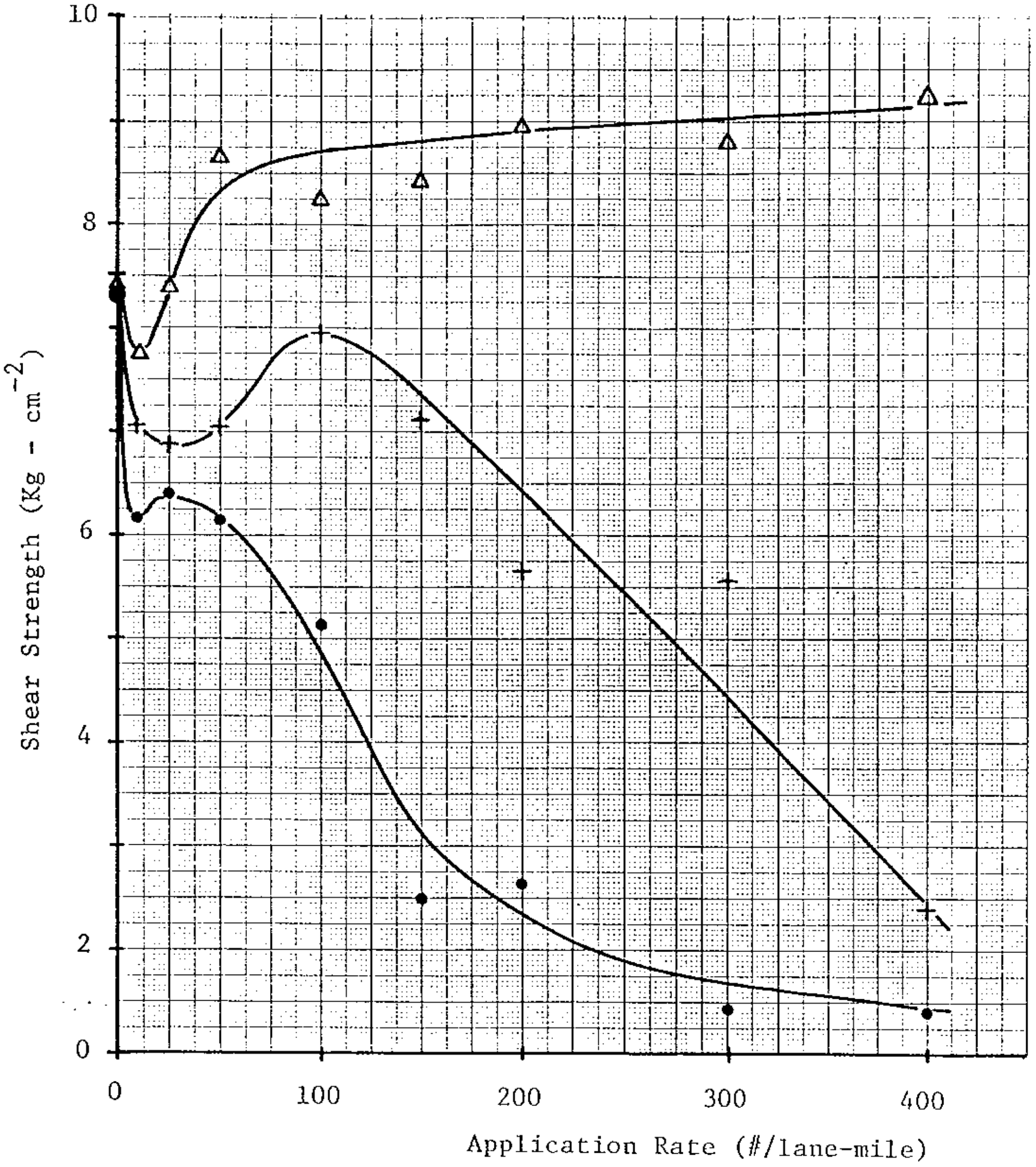

Figure 6c. Interfacial Shear Strength v. Application Rate

Measurement Series C1: P.C.C. Substrate, -5°C

● - SD2-I

+ - Na Maleate △ - Na Fumarate

III-C. Comparison of Two Batches of SD2

A new substrate series (Series DS) was prepared for the comparison of two batches of SD2. The first batch, designated as SD2-I was the original sample provided to us and used for all the tests described above. The second batch, designated as SD2-II, was provided by Mr. Dan Johnston on 16 March, 1988. Test of these materials at -15°C have been completed; the data is shown in figure 7. Clearly both materials are effective interfacial strength reducers, with the new batch being slightly more effective at application rates of 100 #/lm, but less effective in the 150 to 250 #/lm range. Both materials appear to be equally effective at higher application rates. The cause of the increase of interfacial strength for the intermediate range of treatment is not understood.

III-D. Discussion

As our program of measurements has progressed, the need for a well-defined and carefully followed experimental procedure has become very evident. Besides the care taken in the specimen cleaning and treatment procedure, the need for consistent rechecking also became very apparent. In our Quarterly Report for the period 1 January, 1988 through 31 March, 1988 it was indicated that we were having a problem getting the normal interfacial adhesive strength of ice to allegedly clean P.C.C. substrates. Several possibilities had to be investigated including possible effects due to thermal stress at the interface, possible effects of inadvertent variations in load application rate, and any other possible inadvertent variations from the standard test procedure. It was found that there is a small dependence of measured interfacial strength on the rate of application of the load, but that this effect certainly was not responsible for the large decreases observed. We were not able to determine any significant effects due to thermal stress in the interface. Eventually we concluded that although our standard procedure for determining surface cleanliness was not indicating the presence of any surface contamination, there must actually be some. Since the cleanliness soak test was not showing any contamination, it must be water insoluble. Consequently, the substrates were scrubbed with a mild detergent and scouring compound (Ajax). Following a very thorough washing and rinsing the interfacial strength tests indicated that the problem had been solved. Our conclusion was that oil had somehow go onto the substrates, and that it must have been airborne. Enquiries revealed that there had recently been a great deal of oil in the compressed air supply which is used throughout the building, and that a student had disconnected the discharge of a small rotary pump from the vent line; since this pump was frequently displacing significant volumes of air, it was putting out an oil aerosol. We now believe that this latter source was the cause of the problem. As with our previous experience with traces of diesel oil contamination, this experience indicates the high sensitivity of the adhesive strength of ice on concrete to even traces of oil. The interfacial strengths found on the oil-contaminated substrates was only about one third of the value on uncontaminated substrates. Had the tests on untreated substrates not been included in the testing sequence, some very puzzling and erroneous conclusions would have been suggested. We are confident that such problems have been eliminated in the data presented.

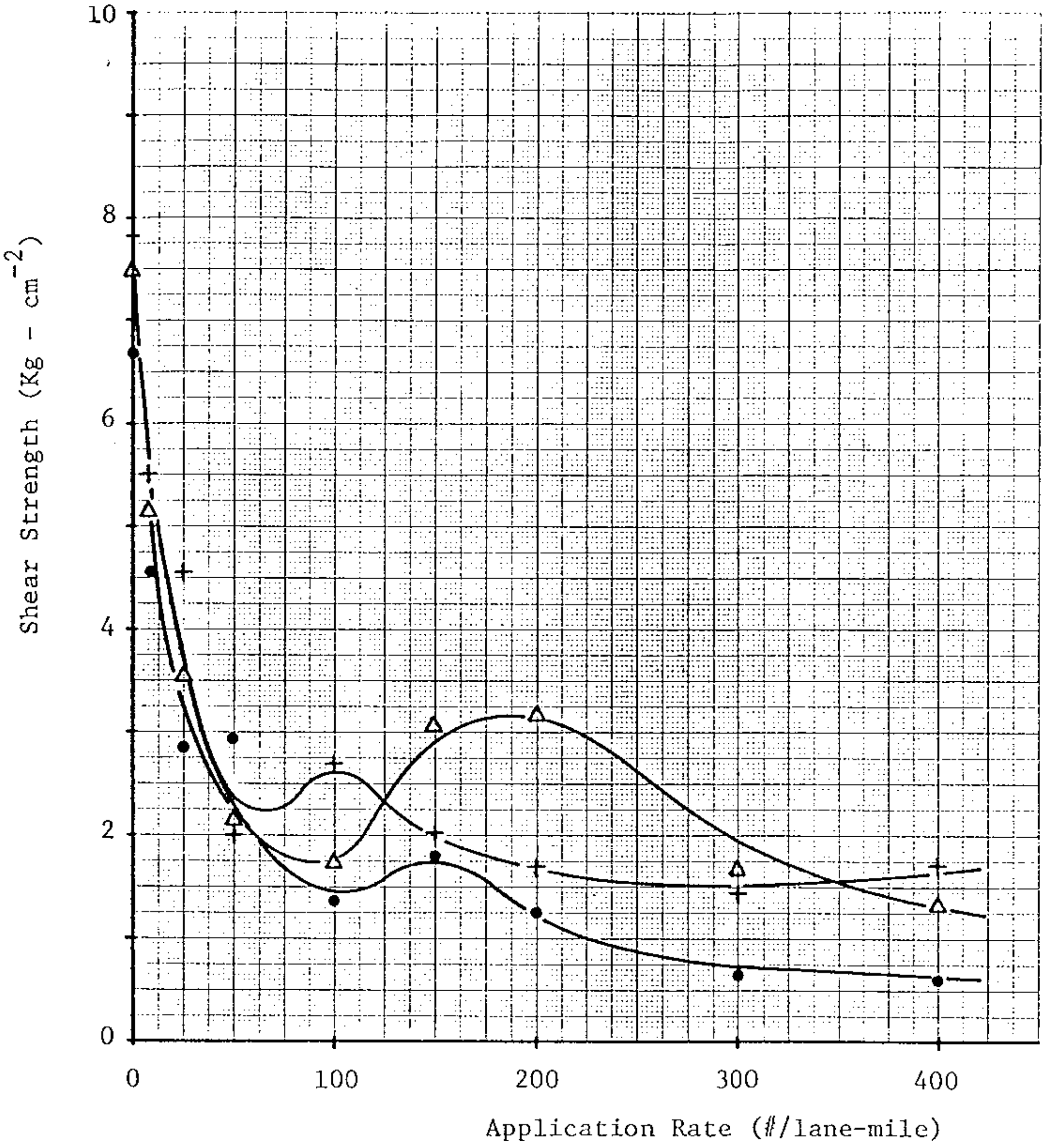

Figure 7. Interfacial Shear Strength v. Application Rate

Measurement Series DS: P.C.C. Substrate, -5°C, -15°C

● - SD2-II (-5°C) △ - SD2-II (-15°C) + - SD2-I (-15°C)

Graphs 5, 6, and 7 illustrate the quite remarkable nature of our results. In figure 5c it is seen that SD2 is more effective than the commonly used deicing materials. Whilst it is true that the difference between the results for SD2 and other materials is largest at application rates of about 200 #/lm, the extent of the relative reduction of interfacial strength by SD2 over the entire temperature range covered is striking.

Data presented in figures 6a, 6b, and 6c lead to the second important conclusion regarding SD2. That is, the bond-strength of ice to substrates treated with SD2 is significantly lower than the bond-strength to substrates treated with any of its three major components or two of its minor components. For example, at -5^{o}C, the shear strength with SD2 is approximately 0.4 kg/cm^2, while, at the equivalent application rate with its major constituents at 100 #/lm, the shear strength is over an order of magnitude higher (see figure 6a). It is interesting that the shear strength for major component treated concrete still appears to be a function of application rate at 400 #/lm. When treated at this level, the dried substrates appear to be completely covered with the salt.

Comparison of the effectiveness of SD2 with that of its two most abundant minor components indicate that they also are not individually responsible for the remarkably low shear strengths found. At -5^{o}C, 300 #/lm mile of SD2 corresponds to 18 #/lm of sodium maleate, and 4 #/lm of sodium fumarate. Again, we find an order-of-magnitude difference between shear strengths of ice to SD2 treated concrete and that of sodium maleate and sodium fumarate treated concrete. Sodium maleate, although significantly less effective, shows some of the characteristics of SD2. Sodium fumarate treatment, on the other hand, enhances the interfacial strength for application rates of 50 #/lm and greater. The interfacial strength seems still to be increasing at 400 #/lm despite the fact that the dried treatment material is clearly visible as a powder on the substrate surface.

The shear strength versus application rate of SD2 and its major components at -15^{o}C is summarized in figure 5c. One notes several changes in the shear strength behavior of the major components at this lower temperature from the comparable results at -5^{o}C. However, here again the most significant conclusion is that at this lower temperature it still is not possible to predict the shear strength behavior of SD2 from those of its major components.

These observations raise several interesting possibilities - that the deicing properties of SD2 are due to a synergistic action, or, equally fascinating, that some unknown material, making up less than 1% of SD2 or some material which has been regarded as inert, is responsible for the remarkably low shear strengths. In either case, the results require further investigation. Clearly, it would be highly desirable to have some deicing material on which a full chemical analysis has been performed.

In our earlier measurement series (series A1, and A2), measurements were performed for treatment rates of 50, 100, 150, 200, 250, 300, and 400 #/lm. For later series, values were taken also at 10 and 25 #/lm. The addition of these two points to the measurement sequence has lead to the observation of detail in the curves at low application rates. In particular, it is seen that, for every specimen for which a shear strength measurement was made for a treatment rate of 10 #/lm, the adhesive strength is significantly reduced from its value on the untreated substrate. Further, for all materials tested, there is a maximum in the interfacial shear strength for some treatment rate greater than 10 #/lm. For SD2, the maximum is near 25 #/lm; for other materials it is as high as 150 #/lm. We are lead to speculate that there may be two mechanisms at work in the ice adhesion process. At very low treatment rates the applied material forms a thin layer on the substrate. This layer disrupts the binding which water molecules normally are able to accomplish directly to the concrete - presumably by presenting to the adsorbate a surface which does not have good order. As the thickness of the layer increases, the salt is able to produce a surface which possesses a structure resembling its bulk structure. Now the mechanism of interfacial weakening may result from the availability of the salt for incorporation into the first layers of attached water molecules. This may result in a large number of impurities and structural defects in the interfacial layer of ice, thereby reducing its strength. Factors such as the adsorption of treatment material into the concrete could also be involved.

III-E. Conclusions

The following conclusions have been made as a result of our experiences while performing the interfacial shear strength test and from the data obtained.

o - In performing interfacial shear strength tests, the need for carefully developed procedures for substrate preparation, specimen fabrication, and test sequence was evident.

o - Minute traces of hydrocarbon material contamination on substrates cause very significant reduction of the adhesive strength of ice to concrete.

o - There are significant differences between different batches of SD2.

o - When used as a pretreatment material, SD2 is very effective in reducing the adhesive strength of ice to concrete.

o - The ice-substrate interfacial strength reducing characteristics of the component materials of SD2 are different to those of SD2 itself; it is therefore concluded that there must be some type of synergistic action between certain component materials.

o - From the measurements performed to date, neither the identity of the components which interact, nor the mechanism of the synergism, can yet be determined. Further measurements (which are planned for the next phase of the work) are needed in which tests are performed using treatments of combinations of the component materials at first in pairs, and then in trios if necessary. Results from the freezing curve studies, which are described in the next section of this report, will give valuable information to aid in the selection of combinations.

IV. SOLUBILITY TESTS.

IV-A. Objectives

The objectives of this segment of the research (task C1) was to determine the solubility of the SD2 deicer and its main component materials. These tests were performed by undergraduate assistants in the Department of Chemical Engineering, working under the supervision of Dr. Klemm.

IV-B. Solubility Determination and Results

By means of evaporating solutions and weighing the residues (gravimetric method) the solubilities of several of the separate salts in South Dakota Deicer Number 2 (SD2) down to various temperatures below the freezing point of pure water have been determined. Duplicate analyses were made in some cases by taking aliquots. Atomic absorption techniques, measuring the sodium ion, were also made for comparison. The salts studied were sodium oxalate, sodium formate, and sodium acetate. These tests require approximately 50 grams of specimen. For a considerable time, we were unable to identify a supplier for some of the component salts. Presently, we have managed to obtain sodium lactate and sodium fumarate, and have sodium maleate, sodium glycolate, and sodium malonate on order. The results are given in Table I. Literature values, where available, are also listed [33].

An attempt was made to determine the degree of hydration of the solid salt forms in equilibrium with the saturated solutions at various low temperatures. These are given in Table II.

IV-C. Conclusions

From the data given in Tables I and II overleaf, the following conclusions can be drawn.

- o - There is very reasonable agreement between values determined by the different methods, and our data is in good agreement with values from the literature.
- o - The solubility of sodium acetate and sodium formate is very similar to the solubility of sodium chloride, especially at -10^{o}C.
- o - Also, it is clear that additional measurements are needed on the other salts before any interpretive conclusions can be made. It is our intention to complete these measurements when all the samples presently on order are received.

Table I Solubility of Salts at Various Low Temperatures

Salt	Temp(^{o}C)	Solubility Wt % Gravimetric	Atomic Abs.	Literature [33]
NaCl	25.0	26.394		26.40
	4.0	26.3105		26.32
	-5.0	25.5968		25.6
	-10.0	25.0239		25.0
NaOxalate	4.0	2.6829		
	4.0	2.7750		
	-3.9	No determination frozen solid		
NaCarbonate	4.0	Frozen solid; likely decahydrate		8.1
NaAcetate	4.0	27.1338	27.30	27.5
	4.0	27.4986		
	-3.9	25.8963	26.00	26.0
	-3.9	26.0018		
	-12.9	23.4235	23.50	24.3
	-12.9	23.6220		
NaFormate	6.0	35.4687	34.50	35.0
	6.0	34.9251		
	-11.9	25.8583	25.90	24.5
	-11.9	25.7770		
	-16.9	24.2949	24.10	23.2
	-16.9	24.2237		

Table II Waters of Hydration of Solid Salts in Equilibrium with Saturated Solutions

Salt	Temp(^{o}C)	Waters of Hydration Experimental	Literature [33]
NaAcetate	4.0	0.437	Zero or 3*
NaOxalate	4.0	0.013	Zero
NaFormate	6.0	2.97	3.0
	-11.9	2.99	3.0
	-16.9	2.92	3.0

* Two forms shown; the zero hydrate is listed as unstable.

V. COOLING CURVES AND COOLING CURVE APPARATUS.

V-A. Objectives

The objectives of this segment of the research was to determine the freezing characteristic of the SD2 deicer and its main component materials. Specifically, tasks C2, C3, and C4 of the contract called for the building of a freezing point apparatus and investigation of the freezing points of SD2 and its major components. In order to obtain more information about the process of freezing point depression and freezing, we chose to develop an apparatus which was capable of determining the amount of energy associated with the various phases of cooling. This part of the work was performed by David Wheeler and Dr. Ashworth, with assistance from an undergraduate student in the Department of Physics.

V-B. Cooling Curves

A cooling curve is a graph of temperature versus time as heat is extracted from a test solution. At some point the liquid begins to freeze and a change in phase usually occurs. The temperature at which this occurs and the slope of the resulting curve are all dependent on the solution's components, their properties and concentration. Discussions of cooling curves can be found in references cited [34-36].

Pure Substances.

A pure substance like water changes phase at a well-defined temperature, the material system transforming from water at $0^{o}C$ to ice at $0^{o}C$. This is a first order phase transition, and will produce a cooling curve of the form illustrated in figure 8b. The heat removed during the phase-change (isothermal) portion of the curve is known as the latent heat of fusion. Once all the sample has turned to ice its temperature starts to lower; energy extracted as heat during this process is known as sensible heat.

The phase assumed by any material system for a given set of thermodynamic parameters is determined by the Gibb's Free Energy; the equilibrium phase for the system is the one with the lowest free energy. As the thermodynamic parameters are changed, a condition may be reached in which another phase has a lower free energy than the one the system is in. However, the change of phase will not occur instantaneously. Usually, the change will require the removal of large amounts of energy (latent heat), and there is often the need for a "nucleation event" to occur. Until nucleation does occur, the system will continue to follow the free energy curve for its current phase by moving into a supercooled or superheated metastable state.

In a real situation, when a very pure substance is cooled, it will often remain in a liquid state to a temperature well below its freezing point. Supercooling down to temperatures as low as $-40^{o}C$ has been reported for water [37]. This is the metastable thermodynamic state referred to in the previous paragraph. As soon as phase conversion begins, all the additional energy removed as sensible heat is then converted almost instantaneously into latent heat. This process appears in the cooling curve as a period of supercooling

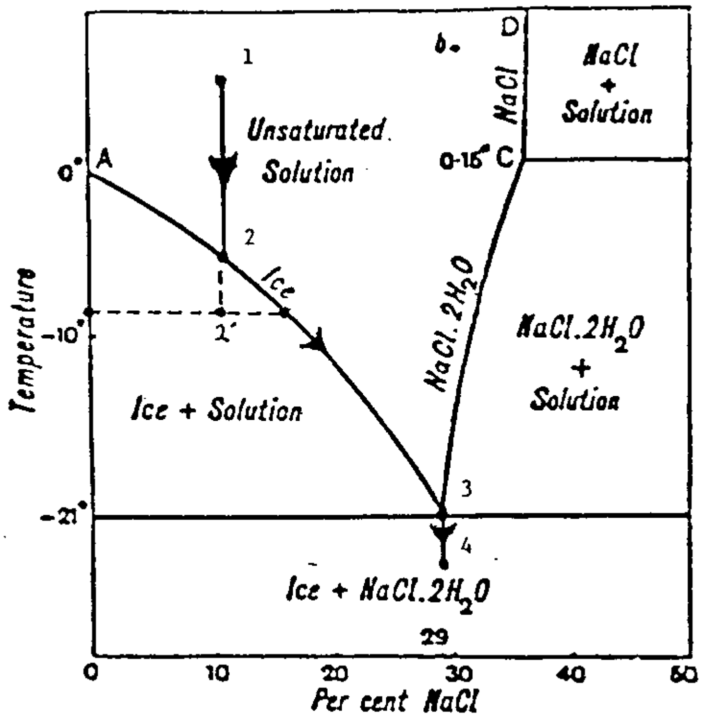

Figure 8a. Phase Diagram for NaCl Solution.

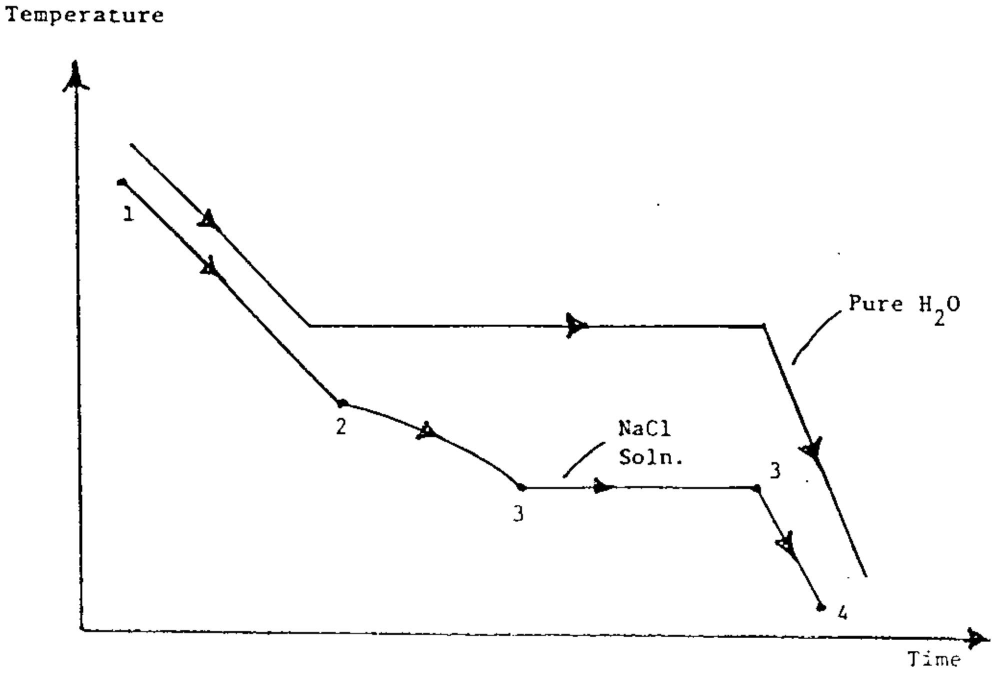

Figure 8b. Idealized Cooling Curves for Pure Water and NaCl Solution.

prior to the onset of isothermal freezing. Towards the end of the phase-change, energy has to be removed by conduction through some of the already-formed ice. This causes temperature gradients to be developed, and, as a result, the temperature measured within the specimen usually begins to decrease before all the specimen is in the solid phase. The smaller the "rounding" that this causes at the end of the freezing portion of the cooling curve, the better the equilibrium achieved within the sample cell. These effects can be seen in the data presented later in this section.

Aqueous Solutions.

In samples where there exists a mixture of water and a soluble material such as NaCl, the cooling curve is somewhat different as illustrated in figure 8b. To begin with, energy is removed as sensible heat and the temperature of the solution decreases. During this portion of the process the composition (ratio of NaCl to H_2O) of the solution does not change - segment 1-2 in the phase diagram depicted in figure 8a. Subsequently, the cooling curve is controlled by the characteristics of the phase diagram. When more energy is removed, the thermodynamic state of the whole system is then depicted as point 2' on the phase diagram. This indicates a mixture of pure ice in equilibrium with a solution of increased NaCl:H_2O ratio, which simply means that ice has precipitated out leaving a more concentrated solution. Although latent heat is being removed as the ice is precipitating, the equilibrium properties of the solution requires that the temperature of the system decrease at the same time. This produces a portion of the cooling curve like segment 2-3 in figure 8b. Ultimately, the thermodynamic state of the remaining solution reaches point 3, the eutectic temperature of the system. This is the point of lowest temperature at which the mixture can exist in liquid form. Now the solution crystallizes into a crystal form containing both NaCl and H_2O (chemical formula NaCl:$2H_2O$). As this phase change occurs, latent heat of fusion must be removed; this results in the isothermal portion 3-3 of figure 8b. It is interesting to note that, in our apparatus, the stirring rod sticks as soon as the phase transition begins for salt solutions, and not at the end of phase-change portion of the curve as it does with water. Once all of the solution is transformed, the temperature of the solid crystal decreases in a normal manner as depicted by segment 3-4 in figure 8b.

Disordered Solutions

A third type of cooling curve is observed for solution systems from which ordered crystals are not produced [35]. Here, the cooling curve has the same form to begin with, but lacks the isothermal segment when the eutectic temperature is reached. Solutions of this nature have been termed disordered substances. Instead of large ordered crystals forming, small crystals having only short range order are produced.

V-C. Design of the Cooling Curve Apparatus

A system was required to determine freezing point depression and cooling curves of various deicers and their components. A test cell was needed which would extract energy from the solution at as nearly a constant rate as possible for cooling from room temperature down to about -80°C. In order to maintain thermal equilibrium in the solution, some type of mechanical stirring was needed. This presented significant difficulties since some of the materials to be tested changed directly from a liquid of relatively low viscosity to a crystalline solid, whereas others changed gradually from a low viscosity solution into a fluid with very high viscosity. The stirring system was required to accommodate these widely varying properties; especially, it must continue to stir as long as possible, but should not self-destruct when a specimen solidifies. The sample cell was to be surrounded by insulation. Energy removal was to occur and be regulated by a thermal link, or thermal resistance, from the cell (a stainless-steel rod or tube) which was in thermal contact with a copper plate, which in turn was maintained at approximately liquid nitrogen temperature (-170°C) by a copper rod immersed in LN_2. Because a large temperature difference was present across the thermal link, the heat flow along it would reduce only slowly as the temperature of the specimen cell decreased. Figure 9 illustrates the scheme of the apparatus, which has been dubbed the "Ice Cream Machine", or I.C.M.

Stirring

The stirring system we constructed sits above the test cell and has the ability to stir up to four cells at once. Preliminary designs used a small DC motor operating through a gearbox to provide the motion. The amount of torque needed to stir even one cell required a large gear reduction. The complexity of this gearbox, along with alignment problems of the gears, proved too cumbersome to work with. A stepping motor was substituted which provided high torque and low turning speeds. The motor rotating at about 60 rpm indirectly provides the stirring motion. Control of the motor is via a TEMP-II microprocessor.

The motor turns an eccentric cam which activates a teflon capped push-rod. Details are shown in figure 10. The push-rod attaches to a "horn" which transfers the motion to the stirring rod. The cam drives the upward motion for the stirring, and the return motion is activated by a spring. To accommodate the problems associated with the phase change, spring loading was incorporated into the push-rods. Whilst the solution is in its liquid phase, the springs are not compressed significantly, but when the phase transition occurs the eccentric drive simply compresses the springs while the stirrer remains fixed.

The stirring rod itself comprises a long thin-walled stainless steel tube and a copper stirrer at the bottom, as illustrated in figure 11. The temperature of the solution was registered by a thermocouple inserted through the tube and glued into place inside the copper stirrer. The stirrer is two-tiered so that the range of motion required for complete stirring could be limited, and its presence in the cell enhances thermal equilibrium.

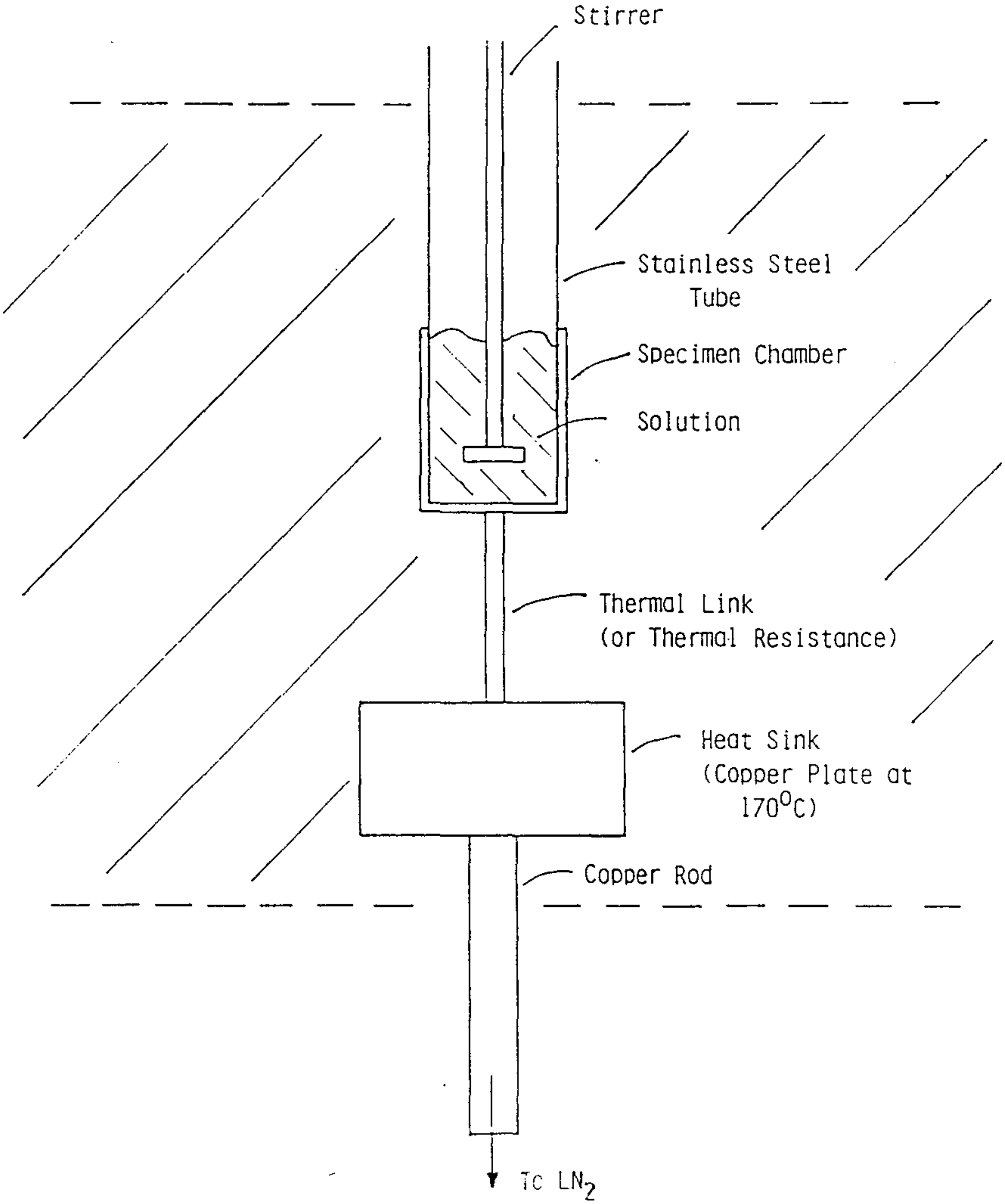

Figure 9. Scheme of Cooling Curve Apparatus

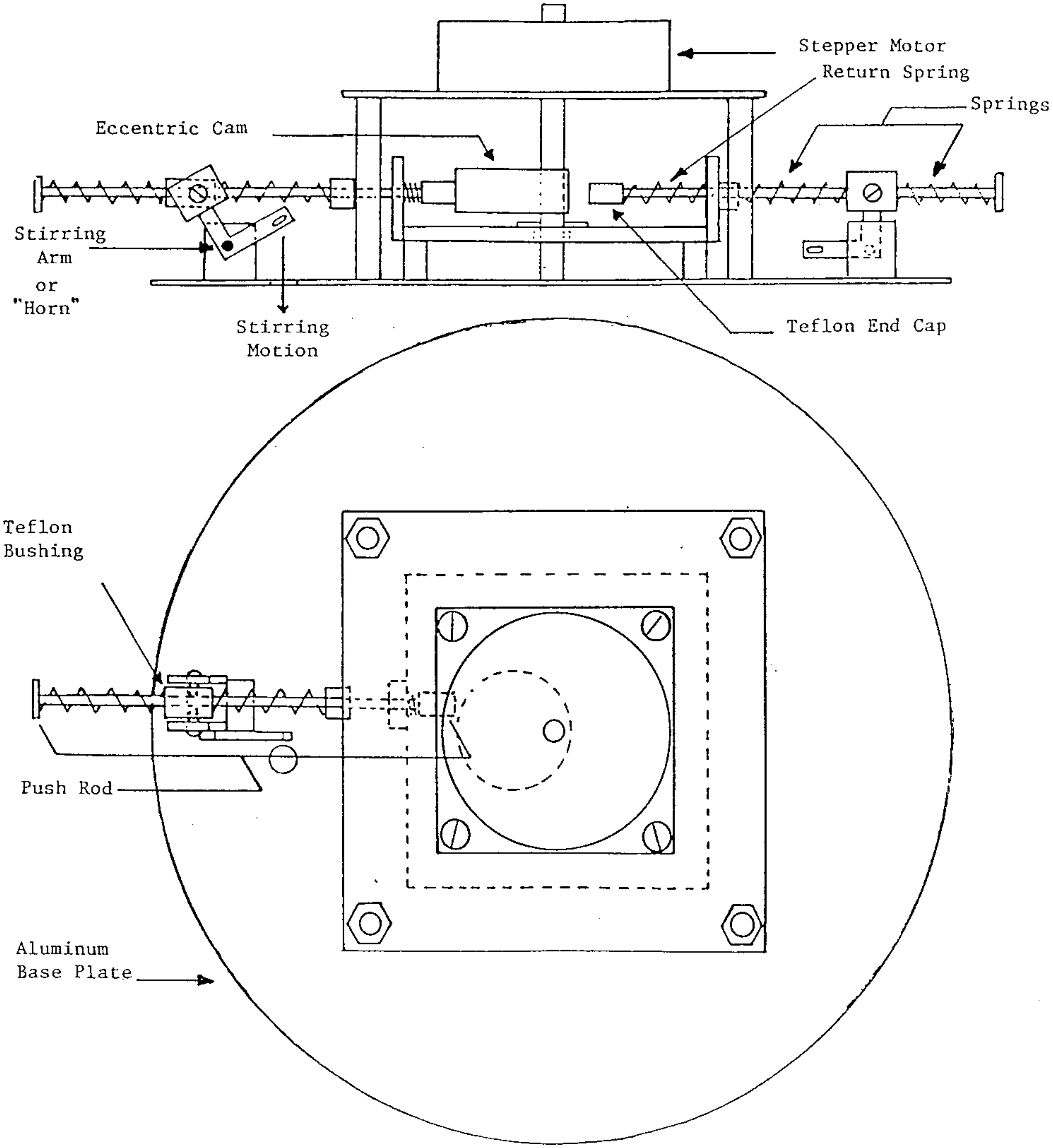

Figure 10. Stirring Mechanism.

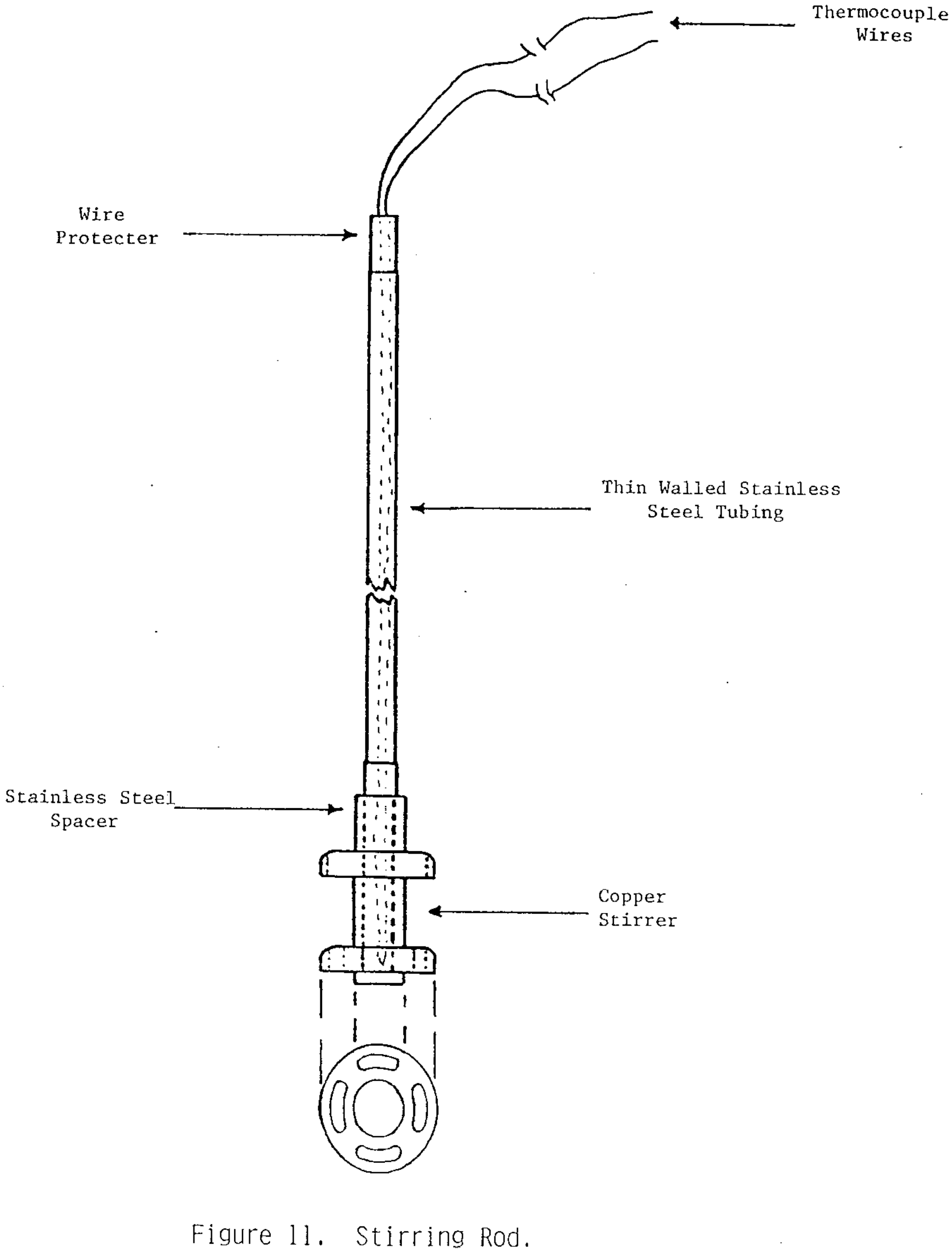

Figure 11. Stirring Rod.

V-D. Test Cell #1

The initial test cell design consisted of a thin walled stainless steel tube surrounded on one end by a copper cap -- this was the specimen chamber. At the top of the chamber a wider copper cap was connected by soft soldering to a copper coupling and another copper cap at the bottom. This bottom cap was then connected to another thin walled stainless steel tube which acted as the thermal link. Heat was removed from the top of the specimen chamber to take advantage of stirring by natural convection down to 4^{o}C. The design is illustrated in figure 12.

Initial data indicated that the cell was not performing in accordance with design. It did produce cooling curves which correctly identified the freezing point of water and the freezing point depression of salt solutions, but cooling rates were nonlinear and latent heat portion of the cooling curve was shorter than expected from heat transfer calculations. A cooling curve obtained for pure water is shown in figure 13. This data indicated that, based on the calculated heat capacities of the system, the time required for the removal of the latent heat of the water specimen was an order of magnitude smaller than expected. Also, the ratio of heat extraction before and during freezing were not correct. Several modifications and tests were performed, and eventually it was determined that the main problem was due to the heat conduction and heat capacity of the surrounding pourable insulation.

V-E. Test Cell #2

The solution to the problems caused by the insulation was to eliminate two of the major mechanisms of heat transfer (conduction and convection) and to eliminate the thermal contact between the cell and insulation by surrounding the test cell with a vacuum. Figure 14 illustrates the arrangement. This new cell design also incorporated an increase in capacity to ten milliliters of solution, a decrease in mass of copper in the specimen cell to an equivalent of less than one milliliter of water, and the use of a specially machined support tube fabricated in nylon which reduced the heat influx to the cell along this component by a factor of ten. The scheme of the apparatus is shown in figure 15, and figure 16 illustrates the overall schematic of the apparatus and the ancillary systems including the data acquisition system. With this new cell the experimental results we obtained began to agree more closely with the values calculated for cooling rates and freezing times. Rather than the estimated and measured quantities differing by more than a factor of ten, they were now in agreement to within a factor of less than two! But we were still not quite satisfied. The final source of extraneous heat transfer was found to be radiation from the cell wall to the inside wall of the vacuum jacket. Here a simple solution was to add to the jacket a heater powered by a temperature regulator activated by a differential thermocouple whose junctions were attached to the two surfaces. Now the jacket temperature was made to follow, within one tenth of a degree, that of the specimen cell and thereby reduce the radiative loss to a negligible amount. The heater was bifilarly wound in order to prevent feedback by magnetic induction.

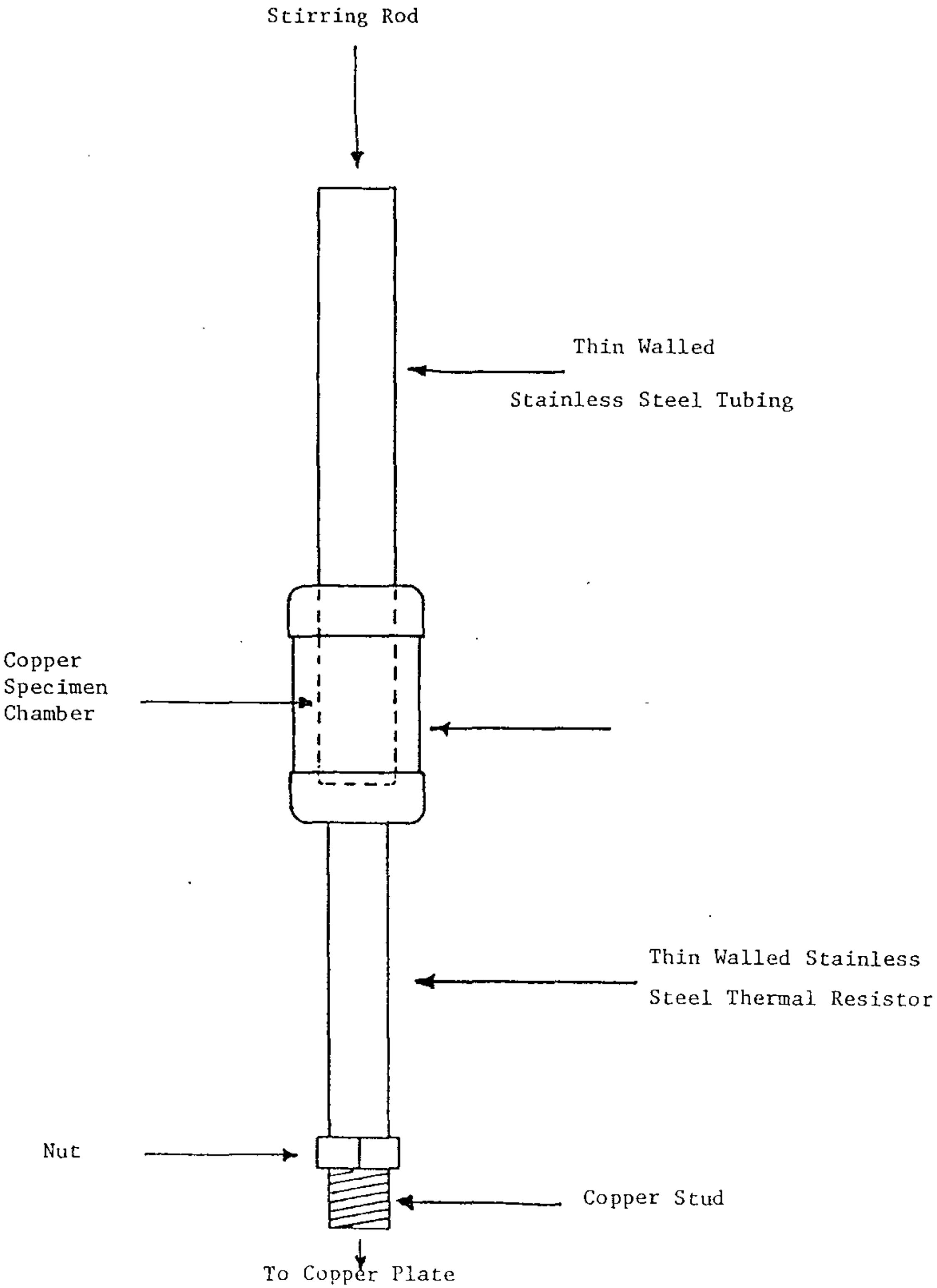

Figure 12. Test Cell #1.

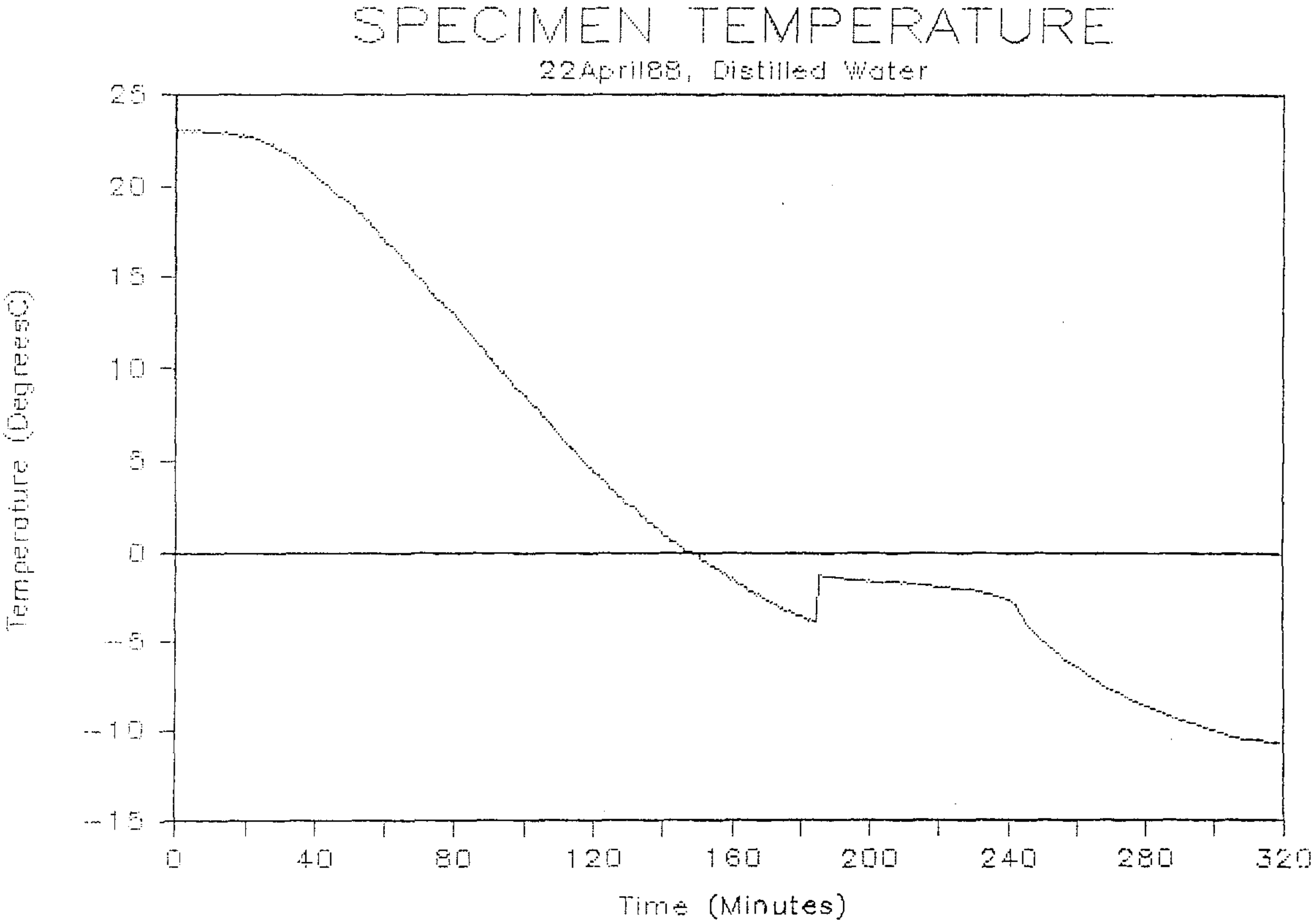

Figure 13. Cooling Curve for Distilled Water Using Cell #1.

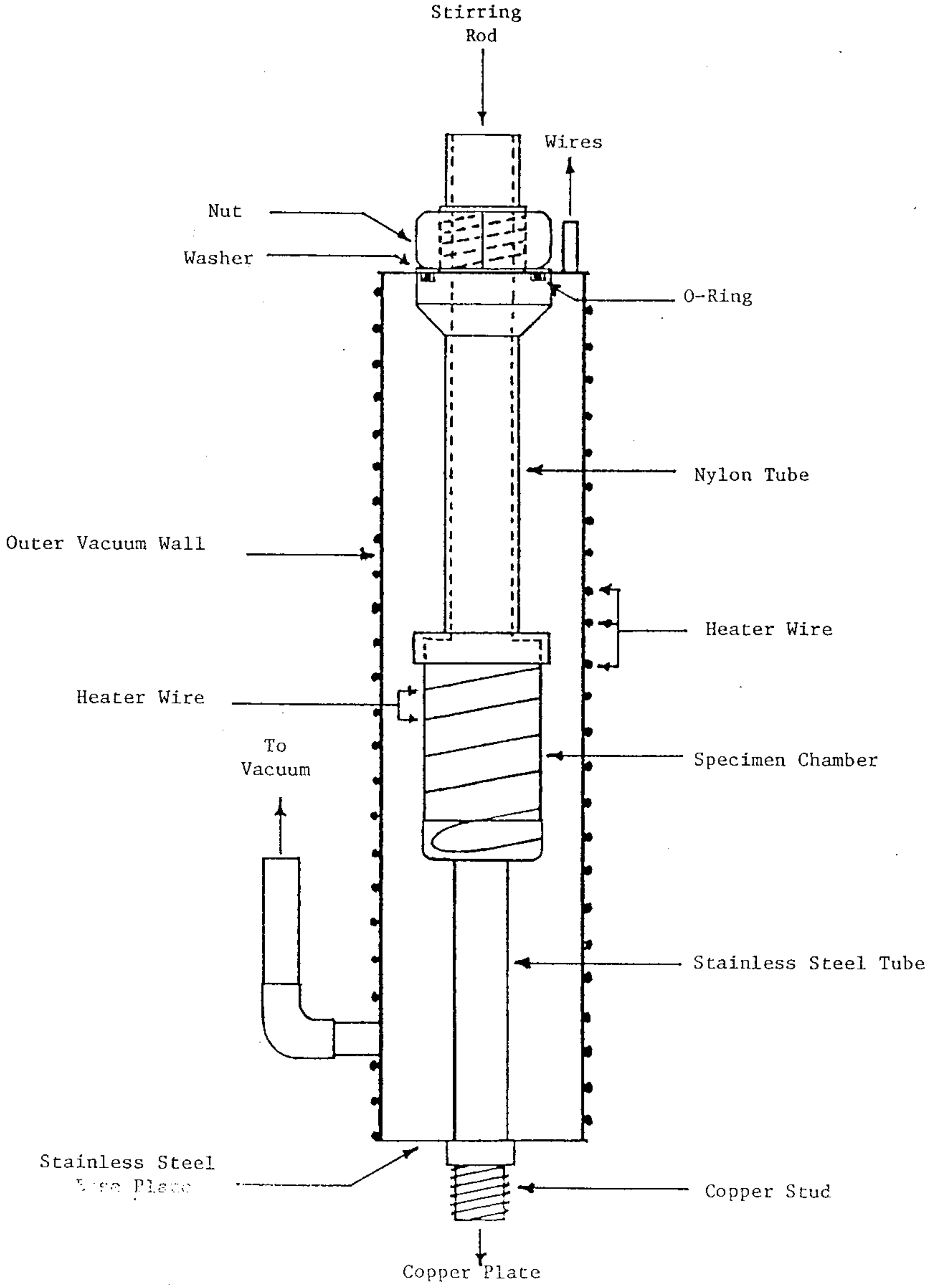

Figure 14. Test Cell #2.

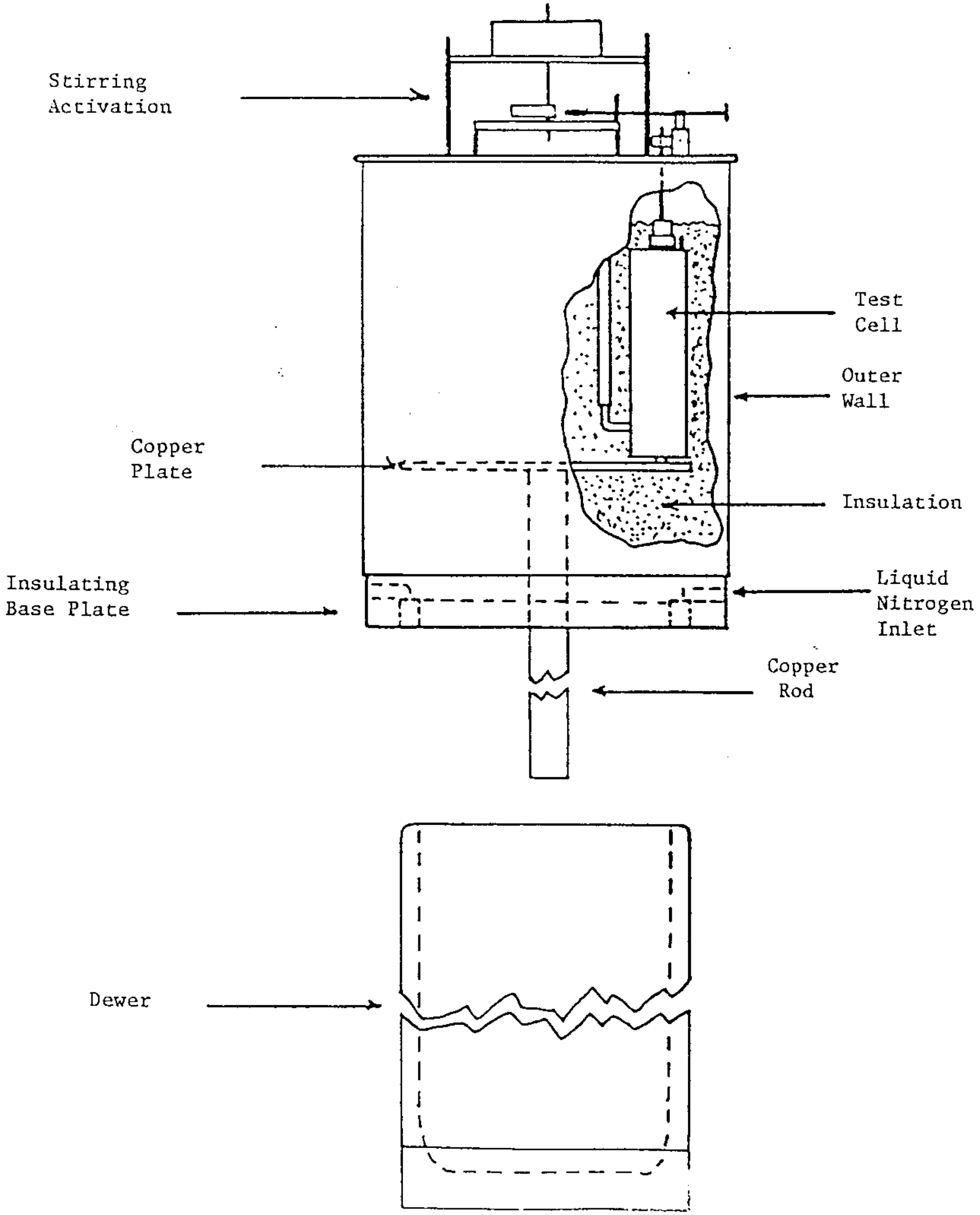

Figure 15. Cooling Curve Apparatus.

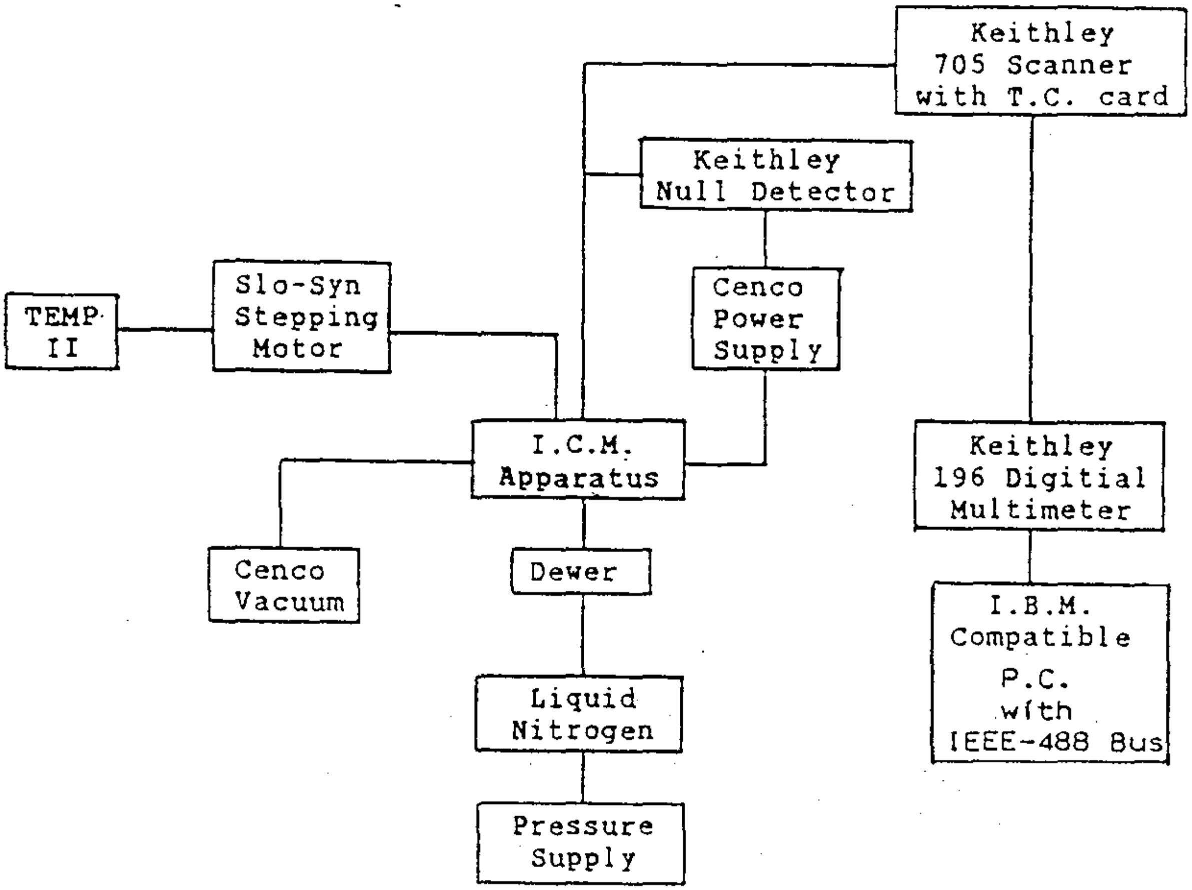

Figure 16. Flowchart of Cooling Curve Apparatus.

V-F. Final Testing and Calibration

Estimated performance and measured performance were now in close agreement. During the various tests carried out for the evaluation of thermal performance, we were also able to observe other details such as the behavior of the stirrer in different types of solution. Several small modifications were made to obtain the best stirring action without scraping all the solidifying material up into the support tube. The system was now ready for its final trials. We performed cooling curve experiments on triply-distilled (XXX) water and on a sodium chloride solution. Since the freezing behavior of both of these materials is well known, we were thus able to verify the performance.

Figures 17 and 18 show the cooling curves for the XXX water and 50% NaCl solution respectively. Several features are noted.

In the curve for water, the cooling above and below the freezing temperature is very close to a linear function of time. The rate below 0°C is approximately double that above 0°C, as expected since the specific heat of ice is approximately one half of the specific heat of water. The specimen supercools by approximately 4°C and then, when freezing nucleation occurs, the cell temperature returns to 0°C. It can be seen that the temperature measuring system recorded a temperature during the freezing section of the curve which is very slightly below 0°C; the difference is approximately 0.10°C and is due to thermals in the thermocouple leads. This simply causes a similar displacement of all measured temperatures, and therefore does not affect the accuracy of temperature differences. It is also seen that the temperature remains very close to 0°C until all the water has solidified, even after the stirrer has been frozen in place. This indicates that the cell/stirrer design is working extremely well in maintaining thermal equilibrium.

Similar observations are true for the initial cooling of the NaCl solution shown in figure 18. Now the amount of supercooling is significantly smaller, as would be expected in a binary system. The graph shows that there is a freezing point depression of -10.5°C, and the material then cools more slowly as ice precipitates from the solution and the concentration increases. At the eutectic point, there is again a small amount of supercooling before the first-order phase change to hydrous sodium chloride begins. Our apparatus recorded a temperature of -22.5°C during the removal of latent heat. At present, we do not know why this temperature is below the accepted value of -21.13°C by more than the 0.10°C mentioned in the previous paragraph. Although we are scrutinizing all aspects of measurement equipment and conversion software, we are not going to presume that the measurement is in error for the present since our technique is in many respects superior than the normal method of using a thermometer or thermistor in a test-tube which is placed in a cold bath. In our system we achieve superior thermal tempering of the sensing thermocouple leads than is possible in other designs.

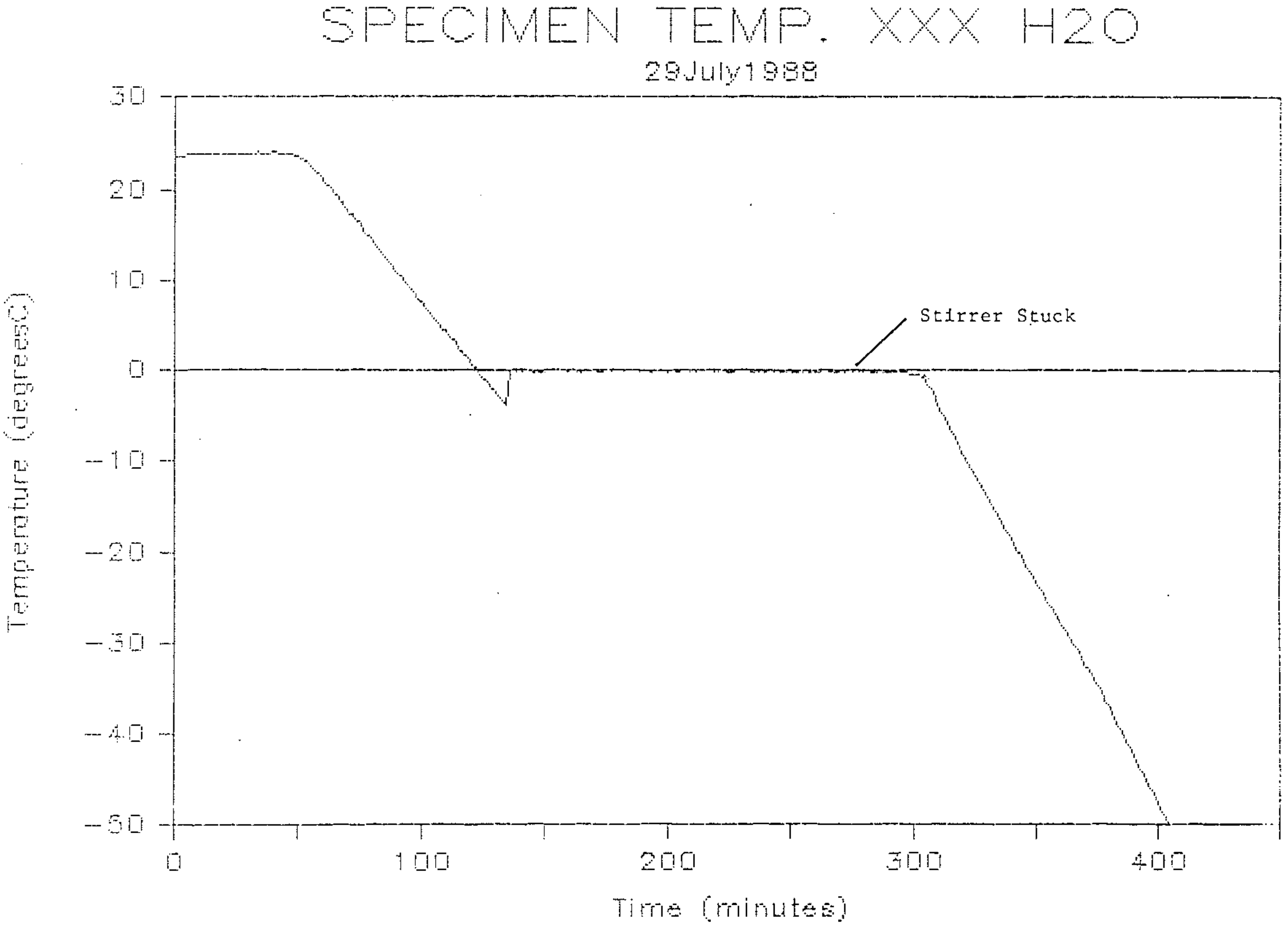

Figure 17. Cooling Curve for Triply Distilled Water.

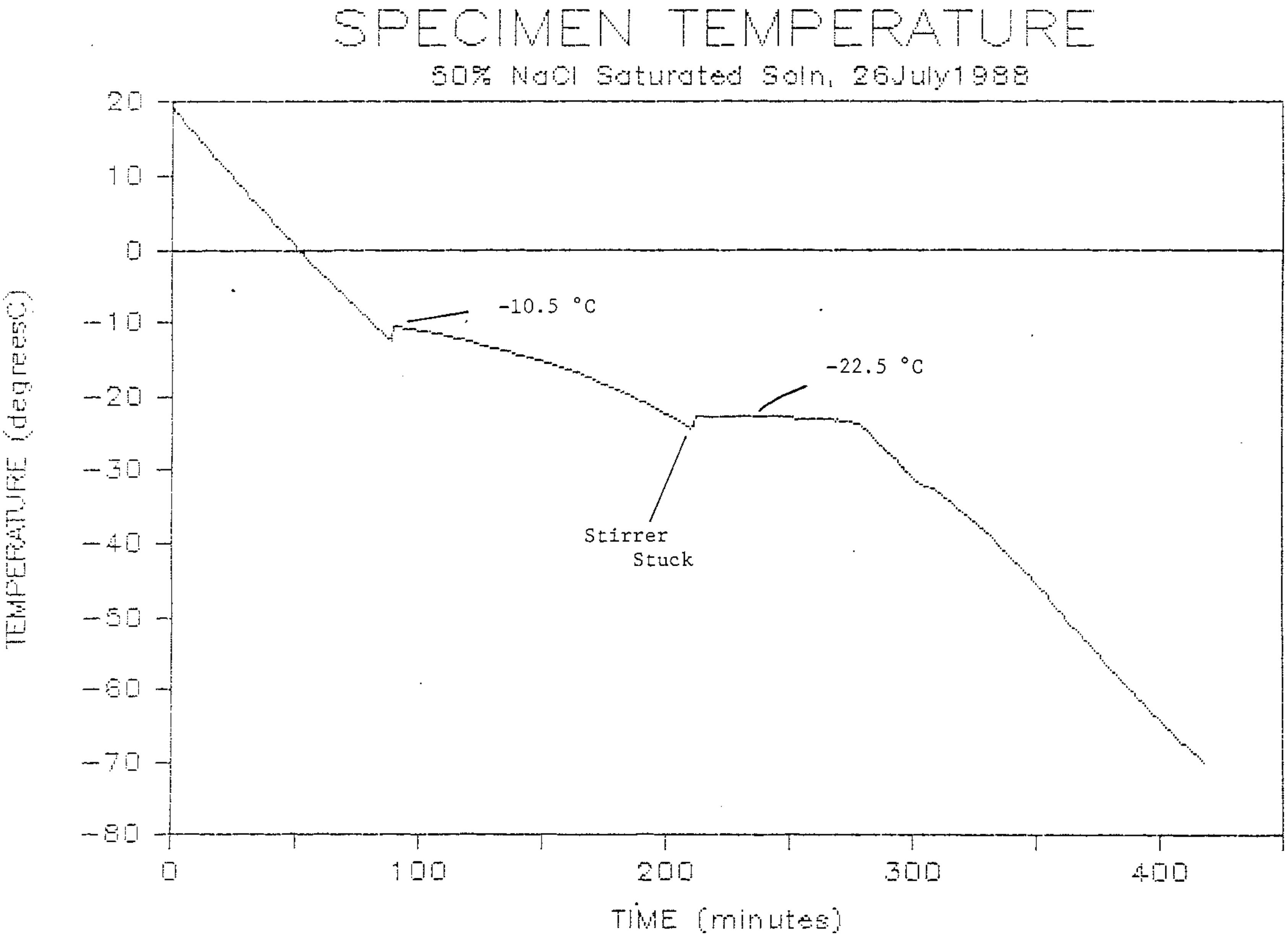

Figure 18. Cooling Curve for NaCl.

V-G. Cooling Curves for SD2 and its Component Materials

Figures 19, 20, 21, 22, and 23 show the cooling curves for SD2-II, its three major components (sodium acetate, sodium formate, and sodium glycolate), and a mixture of the three sodium salts, respectively. In each case a 50% saturated solution was used.

Chronologically, the data on SD2-II was taken before the run was made on the sodium chloride. The form of the curve for the SD2-II was so surprising that we required confirmation that the apparatus was indeed functioning correctly. The absence of a flat portion of the curve corresponding to removal of latent heat at the eutectic point was of greatest concern. It was expected that a ordered, solid phase would be produced which would have a latent heat associated with its formation! Data for sodium chloride and the other sodium salts confirmed that the SD2-II curve was indeed correct. It is better to leave further discussion of this cooling curve until the cooling curves for the component materials have been presented.

Sodium acetate generated a cooling curve (figure 20) which is most like a "normal" aqueous solution curve. A freezing point depression down to -10.14^{o}C was measured. A region of isothermal energy removal was found at -20.5^{o}C. However, it is noticed that there is some detail in the curve between the supercooling and the isothermal region. Rather than immediately rising to the eutectic temperature, the temperature has a tiny peak and then rises gradually.

With sodium formate the freezing point depression was down to -18.06^{o}C. This salt exhibits a significantly greater latent heat than the sodium acetate, and the temperature curve does not contain the detail noted for the acetate. However, we now see that there is a small amount of temperature fluctuation at the beginning of the essential isothermal region. The stirrer does not stick immediately, and the temperature fluctuations continue until about this occurrence. The remarkable feature of this cooling curve is that the eutectic point is at a higher temperature than the freezing point depression!

Sodium glycolate produced a beautiful example of a cooling curve which does not contain any trace of a first order transition. Freezing point depression is down to -7.61^{o}C, and thereafter the cooling curve indicates a smoothly increasing rate of cooling down to about -40^{o}C where the cooling rate becomes essentially constant. As discussed in section V-B, this type of cooling curve is generated when the solution solidifies into a non-ordered solid.

The features of the SD2-II cooling curve can now be understood in terms of those of its component materials. Although the freezing point depression is only down to -7.88^{o}C, we now believe that this sample contains a large amount of inert material. Clearly, the sodium glycolate is dominant in determining the freezing properties of SD2. It appears that the very small detail in the curve following the onset of ice precipitation is most likely due to the sodium formate. The cooling rate becomes constant at about -40^{o}C

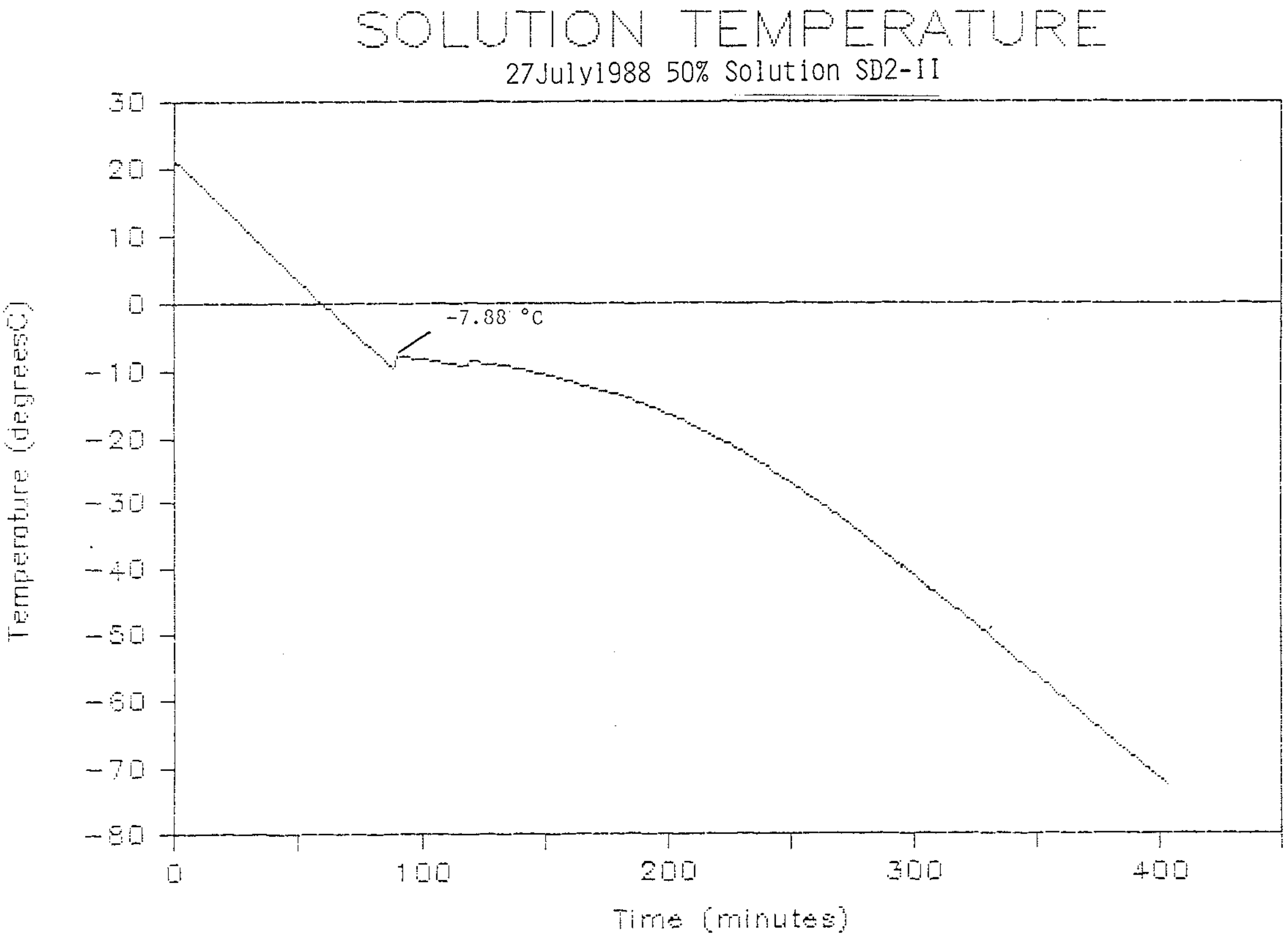

Figure 19. Cooling Curve for SD2-II.

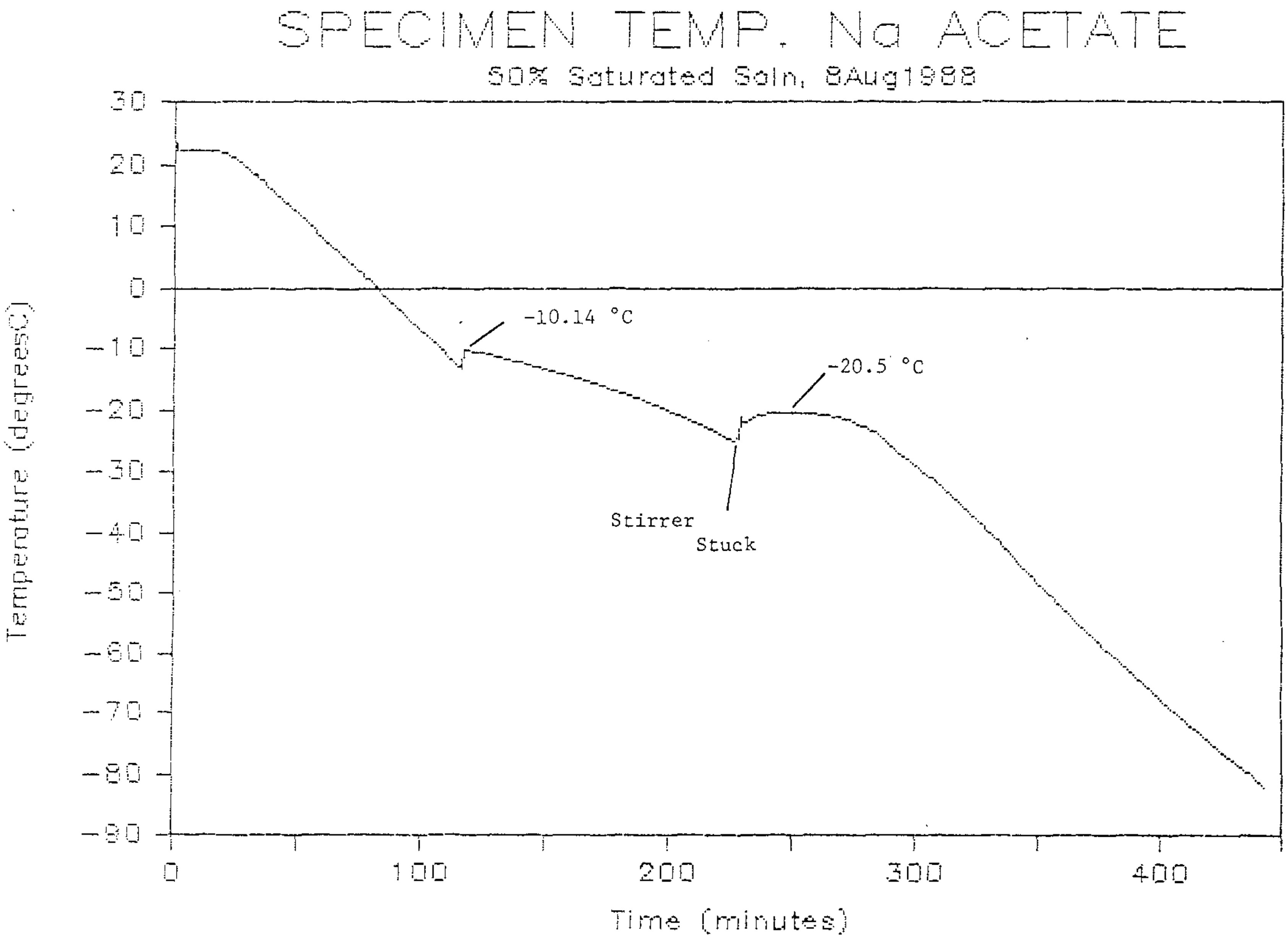

Figure 20. Cooling Curve for Sodium Acetate.

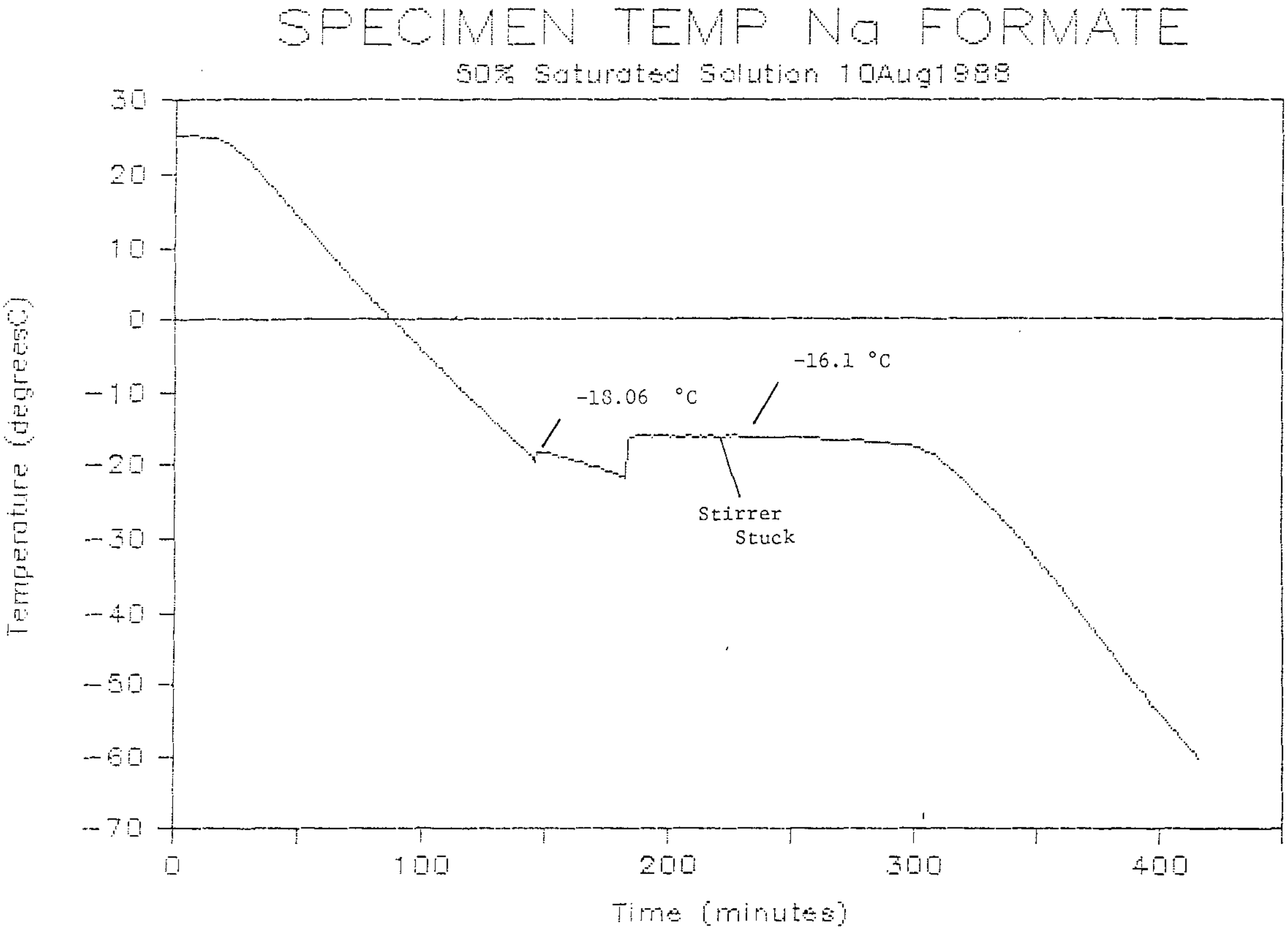

Figure 21. Cooling Curve for Sodium Formate.

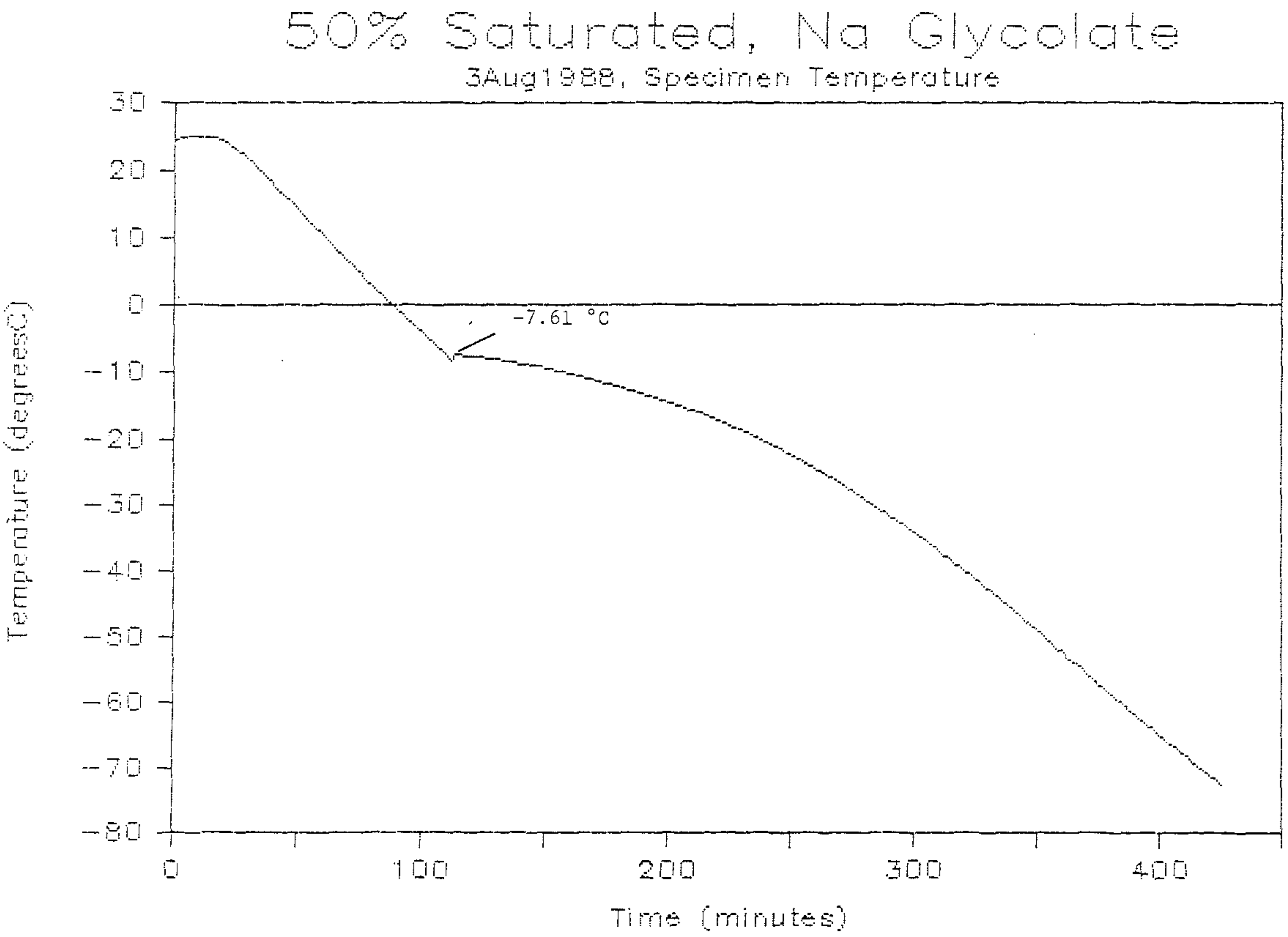

Figure 22. Cooling Curve for Sodium Glycolate.

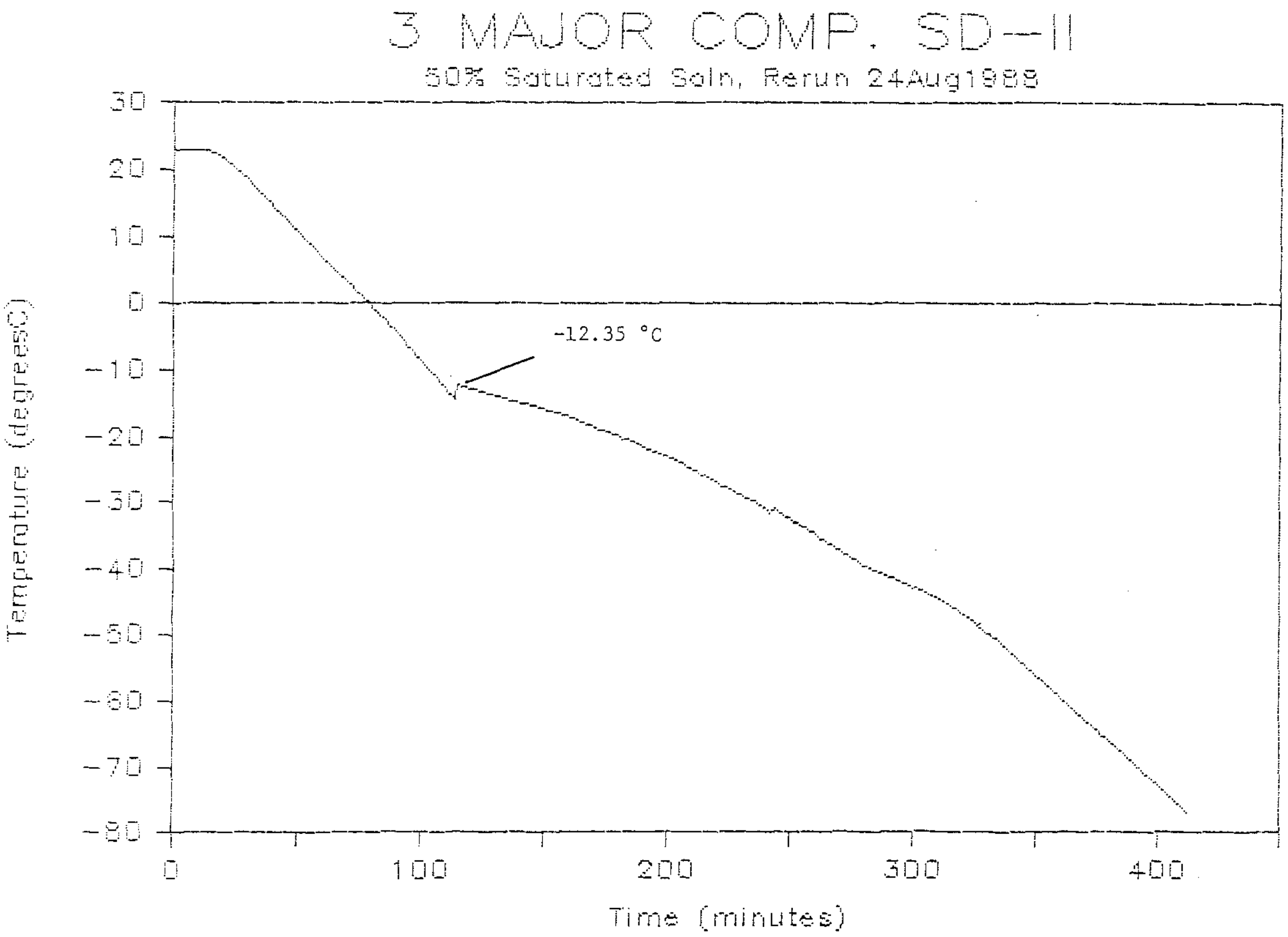

Figure 23. Cooling Curve for the Three Major Component of SD-II.

The final curve in this series (figure 23) shows the cooling curve for a 50% solution made up with equal parts by weight of the three major components of SD2. At a first glance this curve appears to be similar to that of SD2-II or sodium glycolate. However, closer inspection reveals a very small detail at about -31°C, the rate of cooling decreases significantly at about -40°C, and a constant rate of cooling is not achieved until the temperature is down to about -50°C. At the present time we cannot offer any explanation of these details.

V-H. Conclusions

- An apparatus has been developed which is capable of monitoring the cooling curves of solutions of various types. Although there is concern that there is still a small systematic error in the temperature measurement, differential temperatures are monitored with great fidelity making the system capable of determining details in the cooling process. As it stands, data obtained can be interpreted to give rough quantitative values of the energy extracted from the specimen; this has not been performed as yet.

- Two of the major components of SD2-II (sodium formate and sodium acetate) have cooling curves which resemble that of an aqueous salt solution from which a crystalline solid is formed. SD2-II, sodium glycolate, and the mixture of the three major components exhibit the disordered solution traits.

- The cooling curve of the mixture of the three salts is extremely interesting, and rather exciting. Although it shows similar behavior to SD2 in general terms, the details seen in the curve suggests that there are additional details to the process. This needs to be investigated further.

- The basic properties of the materials tested are summarized in the table below:

Material	Type	Freezing Point Depression (°C)	Eutectic Temperature (°C)
H_2O	Pure	0.0	0.0
NaCl	Aqueous	-10.5	-21.3
Na Acetate	Aqueous	-10.1	-20.5
Na Formate	Aqueous	-18.1	-16.1
Na Glycolate	Disordered	-7.6	~ -40
South Dakota II	Disordered	-7.9	~ -40
Salt Mixture	Disordered	-12.3	~ -50

VI. DISCUSSION AND CONCLUSIONS.

Detailed discussion and conclusions have been given at the end of each section. The main points are as follows:

- o - The test procedure developed for interfacial shear strength testing needs to be carefully specified to yield valid results. When this is done, the method produces a useful comparison of materials used as a pretreatment on concrete.

- o - South Dakota Deicer Number 2 has remarkable properties. In the interfacial shear strength test it is far more effective in reducing the adhesive strength of ice to concrete than the commonly used sodium and calcium chloride salts.

- o - Individual component materials of SD2 are much less effective in reducing the strength of an ice-concrete interface, and the characteristics of their actions are not the same as those of SD2. It is concluded from these interfacial strength tests that the effectiveness of SD2 arises from a synergism between two or more of the component materials. As yet, our interfacial testing program has not been carried far enough to identify which components are involved.

- o - Recently, analyses carried out on several batches of SD2 have indicated that there is a significant fraction of the material which is unidentified. Whether this or these materials play any role in the deicing action cannot be determined at the present time.

- o - The cooling curve experiments have revealed that sodium glycolate solution freezes into a solid which does not have long-range order. SD2 has a similar freezing behavior. This is highly suggestive that the glycolate plays a major role in determining the properties of SD2. However, as stated above, sodium glycolate alone is much less effective as a deicing pretreatment. Thus, the whole story is much more complicated.

- o - The mixture of the three major components of SD2 produces the greatest freezing point depression observed. Further, the cooling curve for this salt mixture contained features not seen in the curve of the individual materials.

- o - It is our speculation that an understanding of the mechanisms which produce the properties of SD2 will lead to a fundamental breakthrough in the development of deicing materials. If we can understand the details of one synergistic interaction, this may point the way to engineering similar interactions in other material systems. Thus, while the work performed has identified that an important advance has been made, it may have even further reaching implications.

VII. PRESENTATIONS, PAPERS, PROPOSALS AND THESES.

In addition to producing the interesting results presented in the foregoing sections, the project has produced other tangible output.

VII-A. Presentations and Papers

1) A presentation based on the work reported here was given at the 1988 Annual TRB Meeting:
   T. Ashworth, J. A. Weyland, L. L. Lu, and A. P. Ewing, "The effect of SD2 and CMA treatment on the adhesive strength of ice on Portland Cement Concrete".

2) A presentation of the results of the work reported here was given to the South Dakota Department of Transportation Research Review Board on August 19, 1988.

3) A paper entitled " Evaluation of South Dakota Deicer No. 2 and CMA by Shear Testing" has been submitted for presentation at the 1989 Annual Transportation Research Board Meeting in Washington, D.C. and for subsequent publication in the proceedings of the meeting.

It is expected that additional papers will result from the work on the cooling curves when we have completed a full set of data for a batch of SD2 whose composition has been determined.

VII-B. Theses

To date, there have been two Theses produced in partial fulfillment of the requirements of the Degree Master of Science in Physics. They are:

Lu, L. L., "The Effect of Surface Treatments on Ice Adhesion to Concrete," M.S. Thesis, South Dakota School of Mines and Technology, 1987.

Ewing, A. P., "Further Investigations of the Interfacial Layer Between Ice and Concrete," M.S. Thesis, South Dakota School of Mines and Technology, 1987.

It is expected that the student working on the freezing characteristics of the solutions will complete his thesis on this work before the end of the present semester.

VII-C. Proposals

One of the deliverable items specified in the contract was a proposal which included continuation work on the properties and mechanisms of SD2. It was intended that this proposal should be submitted to the I.D.E.A.S. program in the Strategic Highway Research Program (SHRP). Our intention was complicated by our successful proposal submission with Midwest Research Institute for the SHRP Project H203, "ICE-PAVEMENT BOND DISBONDING -- FUNDAMENTAL STUDY". An attempt was made to have us include the proposed work in the effort of the contract awarded. This, clearly, was unsatisfactory. Discussions with SHRP personnel have recently cleared the way for the submission of our proposal. It is expected that it can now be submitted within the next two weeks.

The thrust of the work to be submitted is a complete evaluation of the effects of SD2 and its components on the interface between ice and substrates of limestone and P.C. mortar, the further development of the cooling curve apparatus, and a full series of cooling curves for the materials including analysis of the energies associated with the various changes in state.

The preparation of the proposal for SHRP has also been affected by the State's involvement in the NSF EPSCoRE Program. A proposal was submitted for consideration in this program; it was evaluated very highly, and selected for inclusion in the main proposal. Naturally, this whole process was carried out in crisis mode, but I am pleased to report that our proposal entitled "A STUDY OF SYNERGISTIC MECHANISMS IN DEICING MATERIALS WHICH LEAD TO THE PREVENTION OF ICE ADHESION" was submitted to NSF on September 30, 1988. The work proposed includes adsorption isotherm studies on powdered limestone and mortar which have been treated with SD2 and its component salts. From these measurements we will be able to determine whether the materials demonstrate a synergism in disrupting the adsorption sites for the water molecule. This is a much more fundamental investigation than the work which is to be submitted to SHRP, and the prospect of having it funded is extremely exciting. Besides funds for the personnel, the proposal includes funds for a micro-calorimeter, an FTIR spectrometer, and a STEM.

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APPENDIX A

INTERFACIAL SHEAR STRENGTH OF ICE ON TREATED CONCRETE SUBSTRATES

TABLE A-1. Shear Strength Series A1

Data File Name: LL10D87
Test Series: A1 20FEB87-06MARCH87
Treatment: SODIUM CHLORIDE
Test Constant: TEMP = -5 °C
Calibration: based on 04JAN88 data and sample area of 12.07 cm^2
$0.038701 * GAUGE - 0.15029 = TRUE\ Kg/cm^2$.

APPL RATE (#/lm)	SHEAR STRENGTH (Kg/cm^2)	STAND DEV (Kg/cm^2)	CONVERTED DATA 1	2	3	4	5	6	7	8
0	9.63	1.64	9.29	8.75	9.09	7.58	12.1	11.4	7.58	11.0
50	7.40	0.92	6.23	8.28	6.42	6.81	7.31	8.24	6.93	8.94
100	6.24	2.68	5.03	7.62	8.24	5.50	-0.1	7.82	7.35	8.44
150	6.95	1.07	4.72	7.39	6.77	8.44	6.19	7.00	7.00	8.05
200	6.87	1.02	4.84	5.77	7.82	7.97	6.85	6.81	7.12	7.78
250	6.01	1.14	5.15	6.00	6.23	6.19	6.04	3.83	6.46	8.17
300	6.00	0.97	4.64	5.65	7.70	6.97	5.07	5.80	5.42	6.69

Data File Name: LL08D87
Test Series: A1 4FEB87-16FEB87
Treatment: CALCIUM CHLORIDE
Test Constant: TEMP = -5 °C
Calibration: based on 04JAN88 data and sample area of 12.07 cm^2
$0.038701 * GAUGE - 0.15029 = TRUE\ Kg/cm^2$.

APPL RATE (#/lm)	SHEAR STRENGTH (Kg/cm^2)	STAND DEV (Kg/cm^2)	CONVERTED DATA 1	2	3	4	5	6	7	8
50	7.11	2.87	3.33	4.37	9.56	5.22	11.4	9.52	4.49	8.90
100	7.52	2.23	3.87	4.10	8.71	6.85	8.44	8.36	9.71	10.0
150	7.71	1.58	5.73	7.86	8.40	6.42	6.27	7.12	8.94	10.8
200	7.16	1.55	5.19	5.77	6.23	5.57	7.82	8.28	9.37	9.02
250	7.12	0.98	5.61	8.24	7.78	6.54	6.04	7.04	7.08	8.59
300	6.54	1.48	3.87	5.42	7.24	5.53	6.31	8.32	6.97	8.59

TABLE A-1. Shear Strength Series A1 (continued)

Data File Name: LL14D87
Test Series: A1 3APRIL87-16APRIL87
Treatment: SD1
Test Constant: TEMP = -5^{o}C
Calibration: based on 04JAN88 data and sample area of 12.07 cm^2
0.038701 * GAUGE - 0.15029 = TRUE Kg/cm^2.

APPL RATE (#/lm)	SHEAR STRENGTH (Kg/cm^2)	STAND DEV (Kg/cm^2)	CONVERTED DATA							
			1	2	3	4	5	6	7	8
0	10.74	1.20	8.75	10.6	12.7	9.21	11.0	10.8	10.9	11.6
50	8.49	1.31	10.5	7.70	6.42	8.59	7.62	7.58	9.56	9.87
100	9.01	2.11	4.64	9.33	8.05	9.17	7.58	10.9	11.6	10.6
150	9.23	1.82	5.65	8.94	8.71	8.36	9.13	10.3	12.4	10.2
200	7.66	1.70	3.52	8.51	7.00	9.17	7.97	8.55	8.90	7.58
250	6.27	0.73	7.39	5.65	6.23	6.89	6.04	4.88	6.23	6.81
300	4.46	3.56	0.85	4.95	-0.1	6.42	1.24	4.18	11.3	6.81

Data File Name: LL06D87
Test Series: A1 17APR87-30APR87
Treatment: SD2-I
Test Constant: TEMP = -5^{o}C
Calibration: based on 04JAN88 data and sample area of 12.07 cm^2
0.038701 * GAUGE - 0.15029 = TRUE Kg/cm^2.

APPL RATE (#/lm)	SHEAR STRENGTH (Kg/cm^2)	STAND DEV (Kg/cm^2)	CONVERTED DATA							
			1	2	3	4	5	6	7	8
0	9.88	1.75	8.59	8.13	12.1	9.37	12.9	8.63		9.37
50	6.18	1.21	3.87	6.62	4.68	7.04	6.54	6.04	7.78	6.81
100	4.54	2.16	1.47	4.57	2.59	6.42	2.36	4.64	8.20	6.04
150	2.72	1.42	1.24	2.28	2.36	5.22	0.89	2.94		4.10
200	2.44	2.50	0.58	0.66	1.01	6.50	0.50	1.59		6.19
250	1.99	1.99	0.46	0.58	0.66	2.67	0.46	0.66	5.65	4.76
300	1.64	1.58	0.70	0.50	0.77	1.78	0.46	0.70	5.26	2.94
400	0.86	0.56	0.62	0.58	0.46	0.46	0.50	0.62	1.59	2.01

TABLE A-2. Shear Strength Series A2

Data File Name: LL21D87
Test Series: A2 24JUNE87-16JULY87
Treatment: SODIUM CHLORIDE
Test Constant: TEMP = -15°C
Calibration: based on 04JAN88 data and sample area of 12.07 cm^2
0.038701 * GAUGE - 0.15029 = TRUE Kg/cm^2.

APPL RATE (#/lm)	SHEAR STRENGTH (Kg/cm^2)	STAND DEV (Kg/cm^2)	CONVERTED DATA							
			1	2	3	4	5	6	7	8
0	8.34	0.41	8.05	8.36	8.51	7.97	8.75	9.13	8.13	7.82
50	6.14	1.08	4.26	7.16	7.04	6.89	6.73	4.49	6.66	5.88
100	6.21	0.83	6.04	5.80	7.82	7.35	6.04	5.57	5.73	5.30
150	5.99	0.66	4.64	6.46	6.81	6.54	5.69	6.11	5.42	6.23
200	7.20	1.84	8.59	6.31	4.22	7.78	9.52	4.53	8.44	8.20
300	6.74	0.78	6.23	6.04	7.74	7.78	6.42	6.27	7.62	5.77
400	6.49	1.62	9.13	3.91	8.01	7.78	5.65	5.46	6.69	5.26

Data File Name: LL23D87
Test Series: A2 8JULY87-22JULY87
Treatment: CALCIUM CHLORIDE
Test Constant: TEMP = -15°C
Calibration: based on 04JAN88 data and sample area of 12.07 cm^2
0.038701 * GAUGE - 0.15029 = TRUE Kg/cm^2.

APPL RATE (#/lm)	SHEAR STRENGTH (Kg/cm^2)	STAND DEV (Kg/cm^2)	CONVERTED DATA							
			1	2	3	4	5	6	7	8
0	8.87	0.73	9.13	7.39	8.78	8.71	9.95	8.94	9.64	8.36
50	8.03	0.81	7.97	7.28	8.40	7.20	8.44	8.17	9.67	7.04
100	6.80	1.00	5.42	5.46	8.09	8.20	6.46	6.39	7.00	7.35
150	7.35	1.14	5.80	5.96	8.13	8.44	8.36	6.23	8.75	7.08
200	7.53	0.60	6.27	8.05	7.89	8.17	7.97	7.47	7.12	7.24
300	7.08	1.20	4.68	7.20	7.58	5.57	8.36	7.24	8.17	7.78
400	6.68	1.43	3.91	6.85	7.58	4.68	7.66	7.58	8.05	7.04

TABLE A-2. Shear Strength Series A2 (continued)

Data File Name: LL36D87
Test Series: A2 13MAY87-27MAY87
Treatment: SD2-I
Test Constant: TEMP = -15^{o}C
Calibration: based on 04JAN88 data and sample area of 12.07 cm^2
$0.038701 * GAUGE - 0.15029 = TRUE\ Kg/cm^2$.

APPL RATE (#/lm)	SHEAR STRENGTH (Kg/cm^2)	STAND DEV (Kg/cm^2)	CONVERTED DATA							
			1	2	3	4	5	6	7	8
0	9.88	1.75	8.59	8.13	12.1	9.37	12.9	8.63		9.37
50	8.55	1.13	7.20	10.1	7.66	8.55	8.13	7.20	10.1	9.33
100	5.94	2.23	2.13	7.04	3.95	8.44	4.88	4.88	9.13	7.04
150	2.20	1.15	1.59	2.21	2.59	4.76	0.89	1.01	1.90	2.63
200	1.64	0.73	0.62	1.97	1.97	2.94	0.85	1.55	1.01	2.17
300	0.88	0.39	0.46	0.62	1.86	0.85	0.77	0.77	0.77	0.85

TABLE A-3. Shear Strength Series B1

Data File Name: LL07D87
Test Series: B1 5FEB87-10FEB87
Treatment: SODIUM CHLORIDE
Test Constant: RATE = 200 #/lm
Calibration: based on 04JAN88 data and sample area of 12.07 cm^2
0.038701 * GAUGE - 0.15029 = TRUE Kg/cm^2.

TEST TEMP (°C)	SHEAR STRENGTH (Kg/cm^2)	STAND DEV (Kg/cm^2)	CONVERTED DATA							
			1	2	3	4	5	6	7	8
-13	8.91	1.50	8.86	5.84	9.95	10.0	8.67	9.02	7.74	11.1
-8	7.81	1.01	7.51	5.65	8.51	9.02	8.28	7.12	7.70	8.67
-5	8.01	1.87	8.94	3.52	9.40	8.67	7.43	7.35	8.98	9.71
CLEAN -5	10.64	2.28	9.37	5.42	11.3	10.9	11.9	10.5	13.5	12.0

Data File Name: LL17D87
Test Series: B1 4MAY87-11MAY87
Treatment: SD2-I
Test Constant: RATE = 200 #/lm
Calibration: based on 04JAN88 data and sample area of 12.07 cm^2
0.038701 * GAUGE - 0.15029 = TRUE Kg/cm^2.

TEST TEMP (°C)	SHEAR STRENGTH (Kg/cm^2)	STAND DEV (Kg/cm^2)	CONVERTED DATA							
			1	2	3	4	5	6	7	8
-18	1.54	0.77	1.04	0.93	1.39	3.13	1.20	1.59	0.66	2.36
-12	2.13	1.69	0.81	1.01	1.01	3.48	0.62	1.20	5.65	3.25
-8	1.91	1.17	1.01	0.70	0.77	2.17	1.20	1.97	4.10	3.33
-3	0.93	0.32	0.54	0.85	0.81	1.01	0.89	0.89	0.70	1.70

TABLE A-3. Shear Strength Series B1 (continued)

Data File Name: LL09D87
Test Series: B1 12FEB87-19FEB87
Treatment: SODIUM FORMATE
Test Constant: RATE = 200 #/lm
Calibration: based on 04JAN88 data and sample area of 12.07 cm^2
0.038701 * GAUGE - 0.15029 = TRUE Kg/cm^2.

TEST TEMP (^{o}C)	SHEAR STRENGTH (Kg/cm^2)	STAND DEV (Kg/cm^2)	CONVERTED DATA							
			1	2	3	4	5	6	7	8
-13	8.62	1.23	8.40	8.94	7.74	9.56	7.35	6.62	10.3	9.95
-8	7.44	0.74	8.13	6.85	8.24	7.93	7.20	7.58	5.84	7.66
-5	6.64	0.83	7.74	5.69	6.50	6.58	6.73	5.42	6.46	7.97
CLEAN -5	9.33	1.06	11.0	8.51	10.0	9.37	7.39	8.86	9.13	10.2

Data File Name: LL22D87
Test Series: B1 29JUNE87-9JULY87
Treatment: SODIUM GLYCOLATE
Test Constant: RATE = 200 #/lm
Calibration: based on 04JAN88 data and sample area of 12.07 cm^2
0.038701 * GAUGE - 0.15029 = TRUE Kg/cm^2.

TEST TEMP (^{o}C)	SHEAR STRENGTH (Kg/cm^2)	STAND DEV (Kg/cm^2)	CONVERTED DATA							
			1	2	3	4	5	6	7	8
-18	5.08	1.18	4.64	5.07	5.46	5.26	7.04	6.27	3.48	3.37
-12	6.18	1.79	6.19	8.63	7.55	7.31	5.69	2.75	4.22	7.04
-8	6.73	1.43	8.13	7.97	8.17	7.62	5.34	7.08	5.03	4.49
-5	6.86	0.62	7.78	6.66	6.69	7.24	6.81	5.50	7.24	6.97
-3	6.14	0.52	6.46	6.42	6.62	7.00	5.65	5.80	5.69	5.46
CLEAN -5	8.38	0.62	7.97	7.43	8.75	8.36	9.13	7.78	9.37	8.24

TABLE A-4. Shear Strength Series C1

Data File Name: DW01D87
Test Series: C1 22OCT87-7NOV87
Treatment: SD2-I
Test Constant: TEMP = -5°C
Calibration: based on 04JAN88 data and sample area of 12.07 cm^2
0.038701 * GAUGE - 0.15029 = TRUE Kg/cm^2.

APPL RATE (#/lm)	SHEAR STRENGTH (Kg/cm^2)	STAND DEV (Kg/cm^2)	CONVERTED DATA 1	2	3	4	5	6	7	8
0										
10	5.15	1.81	5.69	4.06	2.71	2.28	6.69	5.46	7.43	6.85
25	5.36	1.43	4.91	4.10	6.35	6.73	7.28	5.88	5.03	2.59
50	5.10	1.48	6.85	5.11	5.80	6.19	6.66	4.26	2.52	3.41
100	4.11	2.06	7.16	4.68	4.14	4.14	7.00	2.55	1.01	2.13
150	1.46	0.38	2.01	1.20	1.39	1.66	1.97	1.01	0.97	1.43
200	1.57	1.37	4.76	2.55	1.55	1.01	1.01	0.43	0.43	0.81
300	0.40	0.10	0.27	0.19	0.43	0.43	0.46	0.46	0.43	0.46
400	0.35	0.09	0.27	0.31	0.23	0.43	0.46	0.23	0.39	0.43

Data File Name: DW02D87
Test Series: C1 9NOV87-23NOV87
Treatment: SODIUM ACETATE
Test Constant: TEMP = -5°C
Calibration: based on 04JAN88 data and sample area of 12.07 cm^2
0.038701 * GAUGE - 0.15029 = TRUE Kg/cm^2.

APPL RATE (#/lm)	SHEAR STRENGTH (Kg/cm^2)	STAND DEV (Kg/cm^2)	CONVERTED DATA 1	2	3	4	5	6	7	8
0										
10	6.78	1.01	6.89	5.53	6.81	7.58	6.97	4.88	7.39	8.17
25	7.18	0.97	6.42	6.42	6.81	8.05	8.55	5.61	7.35	8.20
50	6.81	1.19	6.62	5.50	6.42	6.04	9.13	5.46	8.01	7.28
100	4.64	2.28	5.65	5.80	6.66	3.91	1.24	4.68	7.97	1.16
150	6.91	0.89	7.62	6.23	7.20	6.69	7.97	5.30	6.23	7.97
200	6.05	1.56	6.00	2.63	6.66	7.20	7.66	4.57	7.00	6.66
300	4.84	1.64	5.65	1.78	6.69	5.65	5.46	3.52	6.58	3.33
400	2.97	1.76	0.62	0.89	5.46	3.17	4.10	1.86	5.34	2.24

TABLE A-4. Shear Strength Series C1 (continued)

Data File Name: DW03D87
Test Series: C1 25NOV87-12DEC87
Treatment: SODIUM FORMATE
Test Constant: TEMP = -5°C
Calibration: based on 04JAN88 data and sample area of 12.07 cm^2
0.038701 * GAUGE - 0.15029 = TRUE Kg/cm^2.

APPL RATE (#/lm)	SHEAR STRENGTH (Kg/cm^2)	STAND DEV (Kg/cm^2)	CONVERTED DATA							
			1	2	3	4	5	6	7	8
0										
10	6.62	0.81	5.65	6.35	5.53	6.81	7.35	5.96	7.58	7.70
25	5.22	1.25	6.08	5.65	6.11	5.65	3.17	2.98	5.84	6.23
50	5.36	1.05	4.84	5.88	6.19	4.26	5.07	3.52	6.46	6.66
100	5.82	0.97	4.80	4.68	6.42	5.53	7.39	4.88	6.97	5.88
150	5.04	1.43	5.26	3.29	7.00	5.92	5.84	3.91	2.75	6.27
200	4.81	1.53	5.26	4.88	6.04	5.38	5.84	2.79	1.82	6.42
300	3.52	1.39	2.05	2.21	5.88	4.14	3.68	2.01	5.22	2.94
400	2.52	1.43	0.43	1.01	3.95	2.55	2.82	1.78	5.19	2.44

Data File Name: DW04D87
Test Series: C1 16DEC87-6JAN88
Treatment: SODIUM GLYCOLATE
TEST CONSTANT: TEMP = -5°C
Calibration: based on 04JAN88 data and sample area of 12.07 cm^2
0.038701 * GAUGE - 0.15029 = TRUE Kg/cm^2.

APPL RATE (#/lm)	SHEAR STRENGTH (Kg/cm^2)	STAND DEV (Kg/cm^2)	CONVERTED DATA							
			1	2	3	4	5	6	7	8
0	7.63	0.71	7.00	7.24	8.36	7.74	7.97	7.58	6.39	8.75
10	5.49	1.69	5.50	4.18	5.69	8.17	2.28	4.84	7.20	6.04
25	6.28	1.06	5.73	5.50	6.08	8.05	6.81	4.33	7.04	6.69
50	6.55	1.15	6.04	5.22	6.85	6.66	7.78	4.68	8.40	6.73
100	7.00	0.74	6.39	6.04	8.17	7.58	7.66	6.08	6.81	7.24
150	6.45	0.98	7.74	5.03	7.04	5.88	7.43	5.11	7.20	6.15
200	5.16	1.84	2.21	5.42	7.74	6.73	3.33	4.68	7.20	3.95
300	4.78	1.16	3.91	2.86	6.23	5.73	5.11	3.91	6.23	4.26
400	3.61	1.48	2.24	0.66	3.99	4.72	5.34	3.10	5.11	3.71

Table A-4. Shear Strength Series C1 (continued)

Data File Name: DW05D87
Test Series: C1 14JAN87-30JAN88
Treatment: SODIUM MALEATE
Test Constant: TEMP = -5^{o}C
Calibration: based on 04JAN88 data and sample area of 12.07 cm^2
0.038701 * GAUGE - 0.15029 = TRUE Kg/cm^2.

APPL RATE (#/lm)	SHEAR STRENGTH (Kg/cm^2)	STAND DEV (Kg/cm^2)	CONVERTED DATA 1	2	3	4	5	6	7	8
0	7.43	0.93	5.84	6.97	9.13	7.78	6.97	7.00	8.32	7.39
10	6.02	0.56	6.62	5.65	6.81	6.23	6.39	5.11	5.92	5.42
25	5.85	1.04	6.04	4.99	4.53	6.66	7.55	4.37	6.23	6.42
50	6.01	0.79	5.42	5.11	7.28	6.23	6.89	4.84	6.11	6.19
100	6.90	0.77	6.42	5.92	7.93	7.28	7.58	5.61	7.39	7.00
150	6.07	0.77	6.66	5.26	7.16	6.50	6.81	5.19	5.07	5.88
200	4.61	1.06	5.07	3.56	5.73	4.30	4.10	2.75	6.04	5.34
300	4.64	0.94	5.88	2.94	5.26	4.68	5.65	3.71	4.06	4.91
400	1.37	0.90	1.20	0.50	2.48	0.97	0.58	0.73	3.17	1.32

Data File Name: DW06D87
Test Series: C1 18FEB88-4MAR88
Treatment: SODIUM FUMARATE
Test Constant: TEMP = -5^{o}C
Calibration: based on 04JAN88 data and sample area of 12.07 cm^2
0.038701 * GAUGE - 0.15029 = TRUE Kg/cm^2.

APPL RATE (#/lm)	SHEAR STRENGTH (Kg/cm^2)	STAND DEV (Kg/cm^2)	CONVERTED DATA 1	2	3	4	5	6	7	8
0	7.39	0.54	7.16	7.66	6.42	7.86	7.97	7.20	6.81	7.97
10	6.68	1.70	6.04	5.46	3.17	8.32	7.28	7.93	8.75	6.42
25	7.34	1.43	3.91	7.78	8.98	7.58	7.24	8.32	6.93	7.97
50	8.63	0.57	8.75	8.90	7.58	8.51	8.01	9.13	8.63	9.52
100	8.18	1.01	8.01	9.75	7.04	8.13	8.36	6.35	8.98	8.75
150	8.37	0.75	7.78	8.82	7.20	8.94	8.51	8.36	7.62	9.67
200	8.89	0.70	7.97	8.17	9.52	9.13	7.97	8.90	9.71	9.67
300	8.80	0.63	9.25	8.55	7.58	8.40	9.33	8.98	8.55	9.75
400	9.22	0.91	10.2	10.9	8.75	8.98	9.29	8.94	7.78	8.75

TABLE A-4. Shear Strength Series C1 (continued)

Data File Name: DW07D87
Test Series: C1 29MAR88-20APR88
Treatemnt: SODIUM CARBONATE
Test Constant: TEMP = -5°C
Calibration: based on 04JAN88 data and sample area of 12.07 cm^2
$0.038701 * GAUGE - 0.15029 = TRUE\ Kg/cm^2$.

APPL RATE (#/lm)	SHEAR STRENGTH (Kg/cm^2)	STAND DEV (Kg/cm^2)	CONVERTED DATA 1	2	3	4	5	6	7	8
0	6.72	0.90	7.00	5.19	6.23	7.39	5.84	7.16	6.66	8.24
10	7.06	1.78	4.88	3.71	6.66	9.02	7.20	8.59	7.78	8.63
25	7.79	0.98	6.85	6.15	7.82	7.82	8.13	7.58	9.75	8.17
50	5.37	1.84	4.49	3.52	7.82	8.01	6.50	5.61	2.63	4.37
100	5.58	2.03	3.37	1.82	5.84	7.20	7.04	4.30	7.78	7.24
150	6.09	2.04	3.06	3.71	6.08	7.97	7.04	9.67	5.07	6.08
200	7.25	1.71	6.27	5.84	8.36	7.43	4.33	7.00	8.40	10.2
300	6.44	2.33	4.84	2.13	6.54	6.77	4.91	10.2	8.01	7.97
400	6.40	2.92	2.13	1.16	5.73	7.43	8.71	9.29	8.32	8.36

Data File Name: DW08D87
Test Series: C1 6MAY88-24MAY88
Treatment: SODIUM OXALATE
Test Constant: TEMP = -5°C
Calibration: based on 04JAN88 data and sample area of 12.07 cm^2
$0.038701 * GAUGE - 0.15029 = TRUE\ Kg/cm^2$.

APPL RATE (#/lm)	SHEAR STRENGTH (Kg/cm^2)	STAND DEV (Kg/cm^2)	CONVERTED DATA 1	2	3	4	5	6	7	8
0	6.72	0.90	7.00	5.19	6.23	7.39	5.84	7.16	6.66	8.24
10	8.09	0.55	7.00	8.13	8.36	8.17	8.17	9.09	8.05	7.74
25	6.59	0.97	6.27	5.80	7.00	5.26	7.97	7.97	5.61	6.81
50	7.89	0.64	8.09	7.62	8.32	8.75	8.59	7.78	7.20	6.77
100	8.20	0.80	8.59	8.78	8.90	6.35	8.17	8.94	8.09	7.78
150	7.74	0.44	6.85	7.55	7.55	7.97	8.01	7.51	8.17	8.32
200	7.84	0.96	8.55	6.42	8.28	6.81	8.51	6.62	8.90	8.55
300	5.55	1.88	5.30	2.55	7.97	4.45	8.01	7.24	5.03	3.83
400	4.45	1.33	3.83	3.99	5.96	3.33	6.31	5.65	4.26	2.21

TABLE A-5. Shear Strength Series C2

Data File Name: LL33D87
Test Series: C2 11AUG87-27AUG87
Treatment: SD2-I
Test Constant: TEMP = -15^{o}C
Calibration: based on 04JAN88 data and sample area of 12.07 cm^2
0.038701 * GAUGE - 0.15029 = TRUE Kg/cm^2.

APPL RATE (#/1m)	SHEAR STRENGTH (Kg/cm^2)	STAND DEV (Kg/cm^2)	CONVERTED DATA 1	2	3	4	5	6	7	8
0	9.13	0.45	9.09	8.55	9.21	9.44	10.0	8.51	8.98	9.17
10	6.48	1.46	7.82	6.62	3.79	4.68	8.55	6.50	6.58	7.24
25	6.54	1.50	8.55	6.19	3.71	7.39	8.36	5.80	5.50	6.81
50	6.07	1.25	5.84	6.42	4.49	4.22	8.36	6.73	5.65	6.81
100	5.14	0.77	5.11	6.58	3.95	4.49	5.84	4.88	5.50	4.72
150	3.53	0.87	3.60	3.91	2.48	3.71	4.49	4.33	1.78	3.91
200	2.21	0.92	3.83	2.36	1.47	2.44	2.79	1.20	0.81	2.75
300	1.72	0.64	3.13	1.82	1.20	1.86	2.01	1.59	0.89	1.24
400	3.73	1.95	7.39	5.19	4.88	4.26	3.02	2.01	1.62	1.43

Data File Name: LL19D87
Test Serries C2 29MAY87-18JUNE87
Treatment: SODIUM ACETATE
Test Constant: TEMP = -15^{o}C
Calibration: based on 04JAN88 data and sample area of 12.07 cm^2
0.038701 * GAUGE - 0.15029 = TRUE Kg/cm^2.

APPL RATE (#/1m)	SHEAR STRENGTH (Kg/cm^2)	STAND DEV (Kg/cm^2)	CONVERTED DATA 1	2	3	4	5	6	7	8
0	9.60	1.05	8.59	10.0	9.52	8.94	10.8	7.97	11.3	9.52
10	9.28	0.97	9.52	8.36	8.75	11.0	9.37	10.2	9.21	7.74
25	9.08	0.82	9.29	8.17	9.37	8.36	10.2	7.82	9.95	9.33
50	8.74	1.07	8.40	7.58	8.94	8.86	10.8	9.13	9.13	7.00
100	8.13	1.42	8.59	7.20	7.58	7.24	10.2	6.11	10.3	7.62
150	7.64	1.07	8.20	6.81	5.73	7.20	8.90	8.36	8.94	6.97
200	7.05	1.17	7.78	6.97	5.22	7.47	9.17	5.50	7.24	7.00
300	5.73	1.30	7.78	6.23	5.65	5.07	3.95	3.87	7.58	5.65

TABLE A-5. Shear Strength Series C2 (continued)

Data File Name: LL32D87
Test Series: C2 29JULY87-8AUG87
Treatment: SODIUM FORMATE
Test Constant: TEMP = -15°C
Calibration: based on 04JAN88 data and sample area of 12.07 cm^2
0.038701 * GAUGE - 0.15029 = TRUE Kg/cm^2.

APPL RATE (#/lm)	SHEAR STRENGTH (Kg/cm^2)	STAND DEV (Kg/cm^2)	CONVERTED DATA							
			1	2	3	4	5	6	7	8
0	9.59	0.30	9.37	9.02	9.95	9.52	9.71	9.60	10.0	9.48
50	7.54	1.08	8.44	7.24	6.50	6.27	9.25	6.58	8.86	7.16
100	7.04	1.35	6.50	6.23	5.30	8.44	6.19	6.42	7.47	9.75
150	8.01	1.35	8.75	6.08	6.58	8.13	7.58	7.16	9.52	10.2
200	7.17	0.80	6.81	6.81	6.81	6.89	7.97	5.84	8.59	7.58
300	7.60	0.61	7.62	7.28	7.00	8.90	7.97	7.78	6.81	7.39

Data File Name: LL20D87
Test Series: C2 26JUNE87-8JULY87
Treatment: SODIUM GLYCOLATE
Test Constant: TEMP = -15°C
Calibration: based on 04JAN88 data and sample area of 12.07 cm^2
0.038701 * GAUGE - 0.15029 = TRUE Kg/cm^2.

APPL RATE (#/lm)	SHEAR STRENGTH (Kg/cm^2)	STAND DEV (Kg/cm^2)	CONVERTED DATA							
			1	2	3	4	5	6	7	8
0	8.84	0.82	9.95	8.75	8.05	7.58	8.36	8.75	9.13	10.1
10	7.79	1.22	7.82	6.93	6.58	6.85	7.62	7.00	10.2	9.17
25	6.93	2.00	7.39	6.27	7.08	1.93	8.17	7.78	8.55	8.20
50	6.96	0.71	8.20	6.62	6.85	6.93	7.97	6.46	6.54	6.04
100	4.69	2.26	3.33	2.55	7.00	1.55	7.78	5.46	2.79	7.00
150	8.45	0.90	9.33	6.77	8.13	8.78	9.29	7.43	9.40	8.40
200	7.07	1.03	7.97	7.08	6.97	8.55	6.08	6.42	8.09	5.34
300	7.29	0.90	8.55	6.04	6.46	7.35	6.50	6.97	8.59	7.78

TABLE A-5. Shear Strength Series C2 (continued)

Data File Name: LL24D87
Test Series: C2 10JULY87-27JULY87
Treatment: SODIUM MALEATE
Test Constant: TEMP = -15^{o}C
Calibration: based on 04JAN88 data and sample area of 12.07 cm^2
0.038701 * GAUGE - 0.15029 = TRUE Kg/cm^2.

APPL RATE (#/1m)	SHEAR STRENGTH (Kg/cm^2)	STAND DEV (Kg/cm^2)	CONVERTED DATA 1	2	3	4	5	6	7	8
0	9.87	1.19	9.21	9.13	8.59	8.94	9.17	12.0	10.4	11.4
10	8.53	0.66	8.40	7.58	8.20	8.32	8.13	9.71	8.44	9.44
25	7.70	0.86	7.62	6.66	6.89	7.70	7.00	7.58	9.13	8.98
50	7.30	1.10	7.78	5.80	6.58	9.09	5.69	7.70	7.78	7.97
100	8.12	1.03	8.59	6.81	7.86	8.55	9.09	6.50	9.75	7.78
150	6.32	2.53	7.62	1.97	3.48	4.49	9.17	6.85	9.37	7.58
200	6.78	2.30	8.36	1.01	6.35	7.20	8.59	6.81	7.78	8.13
300	6.59	1.44	8.01	7.28	4.68	4.10	8.36	5.88	7.16	7.24

Data File Name: LL34D87
Test Series: C2 22SEP87-9OCT87
Treatment: SODIUM FUMARATE
Test Constant: TEMP = -15^{o}C
Calibration: based on 04JAN88 data and sample area of 12.07 cm^2
0.038701 * GAUGE - 0.15029 = TRUE Kg/cm^2.

APPL RATE (#/1m)	SHEAR STRENGTH (Kg/cm^2)	STAND DEV (Kg/cm^2)	CONVERTED DATA 1	2	3	4	5	6	7	8
0	3.47	0.69	3.75	4.49	3.91	3.95	3.52	3.17	2.71	2.21
10	8.62	0.59	7.93	7.78	8.55	8.20	9.17	9.06	8.59	9.60
25	8.22	0.81	7.74	8.13	9.56	9.29	7.20	7.20	8.28	8.32
50	7.59	1.45	7.74	3.83	7.70	8.13	8.71	7.93	8.24	8.40
100	6.98	2.00	7.31	6.46	9.13	5.92	2.44	7.39	8.55	8.55
150	8.47	0.61	9.13	8.94	8.55	8.75	7.78	8.94	7.24	8.36
200	8.46	1.94	7.82	5.92	12.2	9.52	8.51	5.84	9.52	8.28
300	8.55	0.54	7.82	7.70	8.59	8.78	8.20	9.13	9.21	8.90

TABLE A-6. Shear Strength Series DS1 and DS2

Data File Name: DW09D88
Test Series: DS1 25APR88-17MAY88
Treatment: SD2-II
Test Constant: TEMP = -5°C
Calibration: based on 04JAN88 data and sample area of 12.07 cm^2
0.038701 * GAUGE - 0.15029 = TRUE Kg/cm^2.

APPL RATE (#/lm)	SHEAR STRENGTH (Kg/cm^2)	STAND DEV (Kg/cm^2)	CONVERTED DATA							
			1	2	3	4	5	6	7	8
0	6.69	1.72	4.99	5.22	10.2	7.55	5.53	8.17	6.04	5.65
10	4.58	0.92	3.95	4.68	4.06	5.22	3.10	4.49	4.68	6.42
25	2.85	0.53	1.97	2.67	2.55	3.79	2.94	3.10	2.40	3.33
50	2.96	0.24	2.90	2.90	2.59	2.94	2.75	3.10	2.94	3.48
100	1.39	0.47	0.89	1.39	1.04	2.17	0.85	1.78	1.04	1.90
150	1.82	0.52	0.97	1.04	1.66	2.36	1.86	2.28	2.01	2.32
200	1.30	0.34	1.01	1.28	1.01	1.59	1.59	1.62	0.66	1.59
300	0.68	0.21	0.70	0.31	0.43	0.89	0.66	1.01	0.62	0.77
400	0.63	0.15	0.39	0.43	0.70	0.85	0.62	0.62	0.77	0.62

Data File Name: DW10D88
Test Series: DS2 30MAY88-19JUN88
Treatment: SD2-II
Test Constant: TEMP = -15°C
Calibration: based on 04JAN88 data and sample area of 12.07 cm^2
0.038701 * GAUGE - 0.15029 = TRUE Kg/cm^2.

APPL RATE (#/lm)	SHEAR STRENGTH (Kg/cm^2)	STAND DEV (Kg/cm^2)	CONVERTED DATA							
			1	2	3	4	5	6	7	8
0	7.51	1.33	5.84	6.77	9.13	6.31	6.97	9.75	6.77	8.48
10	5.18	1.38	3.87	4.53	6.04	6.19	7.97	3.87	3.79	5.11
25	3.54	1.03	3.33	2.98	3.21	2.90	6.23	3.33	3.33	2.98
50	2.13	0.25	1.97	2.17	1.78	2.21	2.63	2.21	2.21	1.82
100	1.75	0.19	1.86	1.35	1.82	1.82	1.55	1.74	2.01	1.82
150	3.07	0.92	3.13	1.82	3.48	3.25	5.03	2.09	2.75	2.94
200	3.21	0.73	3.17	2.55	1.90	3.91	4.30	3.79	2.86	3.13
300	1.67	0.19	1.74	1.59	1.43	1.47	1.90	2.01	1.59	1.59
400	1.34	0.21	1.78	1.24	1.04	1.24	1.51	1.43	1.24	1.24

TABLE A-6. Shear Strength Series DS1 (continued)

Data File Name: DW11D88
Test Series: DS2 27JUNE88-3AUG88
Treatment: SD2-I
Test Constant: TEMP = -15°C
Calibration: based on 04JAN88 data and sample area of 12.07 cm^2
0.038701 * GAUGE - 0.15029 = TRUE Kg/cm^2.

APPL RATE (#/lm)	SHEAR STRENGTH (Kg/cm^2)	STAND DEV (Kg/cm^2)	CONVERTED DATA							
			1	2	3	4	5	6	7	8
0	7.83	1.19	6.42	5.65	9.44	8.55	7.82	8.17	8.94	7.58
10	5.50	0.58	5.80	4.88	5.03	5.88	6.62	5.69	4.76	5.30
25	4.60	1.07	3.10	3.29	3.52	5.42	6.08	4.88	4.84	5.65
50	2.00	0.73	1.82	1.93	1.35	2.94	0.62	2.90	2.48	1.93
100	2.71	0.64	2.01	2.32	2.28	3.33	3.48	3.71	2.28	2.21
150	2.06	0.50	1.93	1.78	1.78	2.32	2.90	2.71	1.55	1.43
200	1.71	0.33	1.78	1.78	1.55	1.97	1.90	2.17	1.51	1.01
300	1.46	0.32	1.43	1.39	1.20	1.55	2.01	1.66	0.85	1.55
400	1.66	0.20	1.51	1.55	1.62	1.93	1.35	1.74	1.55	1.97

APPENDIX B

RESEARCH PROPOSAL TO THE NATIONAL SCIENCE FOUNDATION

"A STUDY OF SYNERGISTIC MECHANISMS IN DEICING MATERIALS WHICH LEAD TO THE PREVENTION OF ICE ADHESION"

Proposal No. E&S 88-XX

Research Proposal to the National Science Foundation

by

South Dakota School of Mines and Technology
Rapid City, South Dakota 57701-3995
Federal Identification Number: 46-0256905

A STUDY OF SYNERGISTIC MECHANISMS IN DEICING MATERIALS WHICH LEAD TO THE PREVENTION OF ICE ADHESION

New Request

APPROVALS

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Proposal No. E&S 88-XX

PROPOSAL SUMMARY

A STUDY OF SYNERGISTIC MECHANISMS IN DEICING MATERIALS WHICH LEAD TO THE PREVENTION OF ICE ADHESION

Principal Investigator: T. Ashworth
Department of Physics
South Dakota School of Mines and Technology
501 East St. Joseph St.
Rapid City, South Dakota 57701-3995
Telephone (605) 394-2361

Amount Requested: $57,961 for the first year
(major equipment items not included)

Duration of Proposed Work: 36 months

Proposed Starting Date: 1 January, 1989

ABSTRACT

Researchers in South Dakota have developed an extremely promising deicer known as South Dakota Deicer Number 2 (SD2). Tests with ice formed on Portland Cement Concrete indicate that the material is very effective when used as a pretreatment in reducing the interfacial strength. However, similar behavior is not seen in tests which use the main material components separately.

We propose to make measurements of the surface area, heat of immersional wetting, and the adsorption isotherms of selected substrate materials (limestone, portland cement mortar, and amorphous silica); these measurements will be made on pure powdered material and on material which has been treated with the deicer and its components. From the data obtained, the density and energy distribution of adsorption sites will be deduced. In the first year of the project existing equipment (with some improvements) will be used. As additional instrumentation becomes available, the project will progress to more sophisticated calorimetry, bond strength determination by FTIR spectroscopy, and morphologic and elemental studies using SEM and possibly STEM techniques.

From this information we hope to be able to determine which of the component materials are involved in the synergistic action, and the mechanism by which the synergism occurs. These tests will produce a foundation of scientific results which will both aid our understanding of this particular material and increase our level of competence in the general area of interfacial science.

1. INTRODUCTION.

General Problem Area

The general class of problems in which molecules of different types interact to form an interfacial layer has become more and more important. Whenever there are different chemical species present or even different phases of one molecular type an interfacial region exists, and the nature of the interatomic interactions and the dynamics of this region determine a wide range of properties. Examples are as diverse as the electrical properties of ultra-thin and now micron-sized layers of metals and oxides on doped semiconductors in VLSI devices, the generation of heat resulting from the adsorption of water molecules and the absorption of oxygen molecules on the surface of coal, and the adhesion forces between fiber and matrix components which are an important factor in the mechanical properties of advanced composite materials. Besides the examples which are related to recent developments in high vacuum surface science and in "high tech" areas, there are a number of classical interfacial problems which are still in great need of work. One particularly important example is the adhesion of ice to highway pavements and structures (marine, power lines, etc.). Although many millions of dollars have been spent trying to prevent the effects of icing, there has been very little funding or research directed at understanding the interfacial properties which determine the efficacy of methods of preventing and destroying the adhesion of ice to surfaces onto which it forms.

Ice Adhesion Problem

The importance of highway icing lies in the need for year-round mobility of trucks, buses and passenger cars on the highway network in order to maintain local and national economic well-being and livelihood. Thus there is the overall objective of providing safe, serviceable highways during winter conditions, and this must be done with a minimum of cost. Mainly, the present approach is to use chemical deicers and physical displacement systems (snowplows). Unfortunately the mechanical systems are slow, cause significant pavement damage, are energy and labor intensive, and are relatively ineffective on hard-packed snow and ice. As a result there has been a great increase in the use of salt, usually sodium chloride, which is readily available and cheap. Application of salt or sand/salt mixtures quickly produces the serviceable highway conditions demanded by the public. For temperatures not too low, salt is effective for ice and light snow, and greatly enhances the effectiveness of plowing under conditions of heavy snow.

Because NaCl works so well there has been little incentive for research to look for alternative chemical or physical methods. Concerns about the deleterious effects on the environment of both calcium and sodium chloride have been present for many years; the full extent of the problems associated with their use has now become obvious. In the 1950's national usage was around 1 million tons annually; since about 1970 it has been around 10 million tons annually! The cost of this material is about $350MM (MM = million). However, the effective cost of its use is many times higher. A joint study by the National Bureau of Standards and Battelle Laboratories [1] concluded that the automobile corrosion costs attributable to salt use were about $12,000MM per year. Estimates made by the EPA of the cost of salt damage to bridges are even much larger than the cost of auto corrosion [2]. Thus an apparently inexpensive highway deicing methodology is in reality extremely expensive.

possibly more than one-hundred fold the cost of the material itself. In addition, the extent and cost of environmental pollution are now manifesting themselves. Although there has not yet been a nation-wide estimate of the costs arising from environmental pollution (particularly the salinaton of water supplies), the number and cost of localized problems are beginning to paint an alarming picture. At a recent NRC/TRB meeting [3], presentations included reports of additional construction costs of $2.5MM for a 1/4 mile segment of interstate from which direct salt run-off had to be avoided, $1.5MM to install city water to a small community whose water supply had been contaminated by sodium, and $4MM to develop protection wells and drainage systems to safeguard another community's water wells. These are just a very few of the many environmental problems which are arising from the use of salts. They illustrate the need for the development of alternative, environmentally safe methods. To accomplish this there is an acute need for basic knowledge of the interaction between ice and a range of materials and the nature of the interfacial layer.

Previous work on ice adhesion indicates that the attachment of ice to various surfaces is complex and involves at least two mechanisms [4,5,6,7]. Interfacial failure may be a combination of adhesive failure (failure of the chemical bond between ice and the substrate) and cohesive failure (fracture of physically interlocked ice). It is also probable that highly disordered, mobile interfacial layers add further complexity [8,9]. Mechanical removal of ice from a surface is also complicated by the interplay between the properties of the bulk material and those of the interface; fracture is strain rate dependent, and can be either brittle or ductile [10]. Unfortunately most of the previous work has been performed either using highly complex substrates (such as concrete) or without detailed substrate classification. In particular, there have been no studies on "controlled", "adjustable" substrates. Although attempting to determine detailed microscopic information about the interface between ice and a substrate, most of the investigations have used macroscopic experimental techniques. Our own studies began in this traditional macroscopic manner by determining bulk and interfacial tensile and shear strengths. This was performed under the sponsorship of the Federal Highways Administration (1978-1982). Results indicated a need for data directly related to the strength of the individual bonds. Our program was expanded to include measurements of surface area, immersional heats of wetting, and adsorption isotherms for Portland Cement mortar, sand, and limestone. Attempts to correlate heats of wetting to adhesive strength have been reported in the literature. By also performing surface and isotherm measurements on one material system (sand) for pure material and for material contaminated with diesel oil we obtained evidence that long-chain molecules may only form a single point of attachment to the substrate [11]. Previous work which contributed significantly towards the development of the concepts of ice adhesion, including the important work of Jellinek, the Bowden-Raraty-Tabor group, the US Naval Research group, the CRREL group, and many others, is summarized in our DOT reports [6,7] and the thesis of M. J. Westphal [11]. Recent important contributions have been made by Andrew's group at Queen Mary College, London, and by the CRREL group on the general problem of ice adhesion to materials.

2. RESEARCH PLAN AND EXPERIMENTAL METHODS

Current Research

Presently we have two research contracts. Both of these projects are of a distinctly applied nature and are concerned with the bonding between ice and Portland Cement Concrete treated with various materials. A small one-year contract funded by South Dakota State Department of Transportation involves evaluation of a deicing material (SD2) developed by the State DoT. The work proposed here has developed out of this investigation. We have found that the shear strength of an ice-concrete interface is greatly reduced by pretreatment of the substrate with SD2; however similar behavior is not found when the component materials are used individually as a pretreatment. It is the nature and mechanism of this synergistic effect which we wish to investigate. We have also found a similar, but not as pronounced, synergism between the components of calcium magnesium acetate (CMA). Our data indicates very rapid reduction of interfacial strength for very dilute treatments, and, in almost all cases, there is a subsequent increase and maximum in the strength as the treatment rate is increased; this is especially pronounced when the Ca++ ion is involved.

Our other research contract is a larger two year subcontract for Midwest Research Institute who have the prime contract funded by the Strategic Highway Research Program (SHRP); this latter contract may develop into a 5 year program. SRI International and Surface Research Applications of Kansas City are also subcontractors who make up the remainder of the research team. This project is looking at the disbonding of ice, and some of the work being performed by MRI involves the basic nature of ice growth on surfaces with various interfacial energies with the water system. Our component of the work is to further refine the interfacial strength testing procedures and conduct a series of definitive tests on highly characterized model substrates and substrates representative of highway surfaces using a range of carefully controlled conditions of ice growth. Later in the project we will evaluate the efficacy of disbonding materials on similar surfaces.

Research Plan

In order to carry this work to a more fundamental level, measurement of adsorption isotherms, heats of wetting, heats of solution, and other forms of interfacial energy are required. Some measurements have been made on simple equipment; much more data of a higher quality is needed for the work proposed. Measurements made with the two types of instruments described in the equipment section will provide the level of detail and sophistication required to begin evaluation in terms of physical models. From measurements in the calorimeters we will be able to determine bonding site density and area, the energy associated with the bonding sites, and some information regarding the range of influence of the substrate on the structure of the bonded material. These data will be analyzed in conjunction with information from our ongoing studies of shear strength measurement on soluble material treated substrates and the freezing and solubility curves for the aqueous solutions (phase diagrams). For the ice adhesion problem the microcalorimeter and DSC/TGA equipment will allow studies of ice growth from the vapor phase, which will give valuable information on the kinematics of the adsorption processes, nucleation site type and distribution, and the dynamics of molecular interaction in the 2D quasi-liquid of a growing crystal surface.

Thus the specific objectives for proposed research are:

(i) To perform a comprehensive evaluation of the literature incorporating material from several disciplines. As part of the SHRP contract each group has performed searches in defined areas including winter highway maintenance, ice adhesion to all types of surfaces, ice growth, atmospheric ice, surface physics, and surface chemistry. In addition, as part of a sister project (H201) funded by SHRP, searches have been performed by a group at Brooklyn Polytechnic over the areas of interfacial science and general adhesion problems. All of this data base has been prepared using ProCite citation data base software, and it is available to us. We will perform a critical evaluation of all these references together with material obtained by manual searching of current (past two years) literature.

(ii) Investigate mechanism of synergistic action between the component materials of SD2 by performing surface area, heat of immersional wetting, and adsorption isotherms for limestone and amorphous silica in their pure states and when treated with the deicer materials SD2 and CMA and their individual component materials.

(iii) To interpret the results obtained in terms of the number and characteristics of the adsorption sites and the thesis that certain combinations of treatment material disrupt the surface; the manner in which they do this is to be determined.

(iv) To continue the investigation by adding components such as optical and electron (SEM) microscope evaluation of the morphology of the substrate materials in both normal and powered form; FTIR studies of the bonding between substrate, treatment, and water molecules adsorbed on the surface and present as a very thin layer of ice; and STEM evaluation of remnant materials on mechanically disbonded surfaces. Equipment acquisition under the overall program will determine the timetable by which these investigations can be incorporated into this project.

Experimental Methods

In the first year measurements will be performed using existing equipment. Specimens are prepared for heat of wetting and adsorption isotherm determination by drying and rolling to a fine powder in a ball mill. When the material can pass through a 400-mesh screen, its surface area is measured in a Strohlein Area Meter II. The heat of wetting specimens are carefully dried and then sealed in a thin walled glass sphere. Modifications will be made to the calorimeter used for this measurement to improve the thermal isolation of the sample vessel (which will decrease the stray heat influxes and hence simplify data analysis and improve accuracy) and incorporate a data acquisition system to record the temperature-time profiles. Data reduction can then be performed directly without time-consuming data entry. Our apparatus is similar to those described by Boyd and Harkins [12] and Zettlemoyer [13]. It consists of identical flasks with stirrers which are used in the differential mode. The flask used for the specimen contains a bulb crusher; after the system has attained equilibrium the bulb is broken and the attendant temperature increase is measured with a multi-element differential thermocouple between the flasks. Adsorption isotherms are determined by the gravimetric method [14]. A Cahn

Model 2000 electrobalance with a sensitivity of 10^{-7} gram is used to monitor the changes in the mass of a small specimen of powder as the partial pressure of water vapor over its surface is increased in increments. Several precautions are taken to reduce vibration and to prevent effects due to spurious adsorption. Data obtained for similar powders have always been well defined type II isotherms which can be analyzed using the BET theory [15].

During the first year we wish to obtain, install, and learn to operate the SETARAM Model C80 microcalorimeter described in the equipment section below. By using our existing calorimeter initially, we will be able to obtain useful data which will allow us to design the experimental program for the microcalorimeter in which, during the second year, measurements will be made of the interfacial energy of materials subject to a wide range of treatment as water vapor is allowed to adsorb onto the surface. These measurements, together with the data from the adsorption isotherms will provide the base for analysis of adsorption site density and energy distribution. An existing SEM in the Department of Metallurgy will also be used in the second year to attempt to develop methods for observing ice on substrates. To date there appears to have been only one successful attempt to obtain electron micrographs of ice [16]. The morphologic structure of the interfacial layer of ice on different substrates would provide valuable complementary information.

Work proposed for the third year is highly dependent upon equipment acquisition. Several measurement series of heats of solution, simultaneous measurement of heats of adsorption and adsorption isotherms, etc. could be carried out in a DSC/TGA. Bond-strength determination for adsorbed molecules and ice films will be possible when an FTIR spectrometer is obtained. An attempt is also being made to obtain funds to purchase an STEM with full elemental analysis capability. Should such an instrument become available.there are several experiments which we would like to perform. Of most immediate interest would be the determination of the composition of the exposed surfaces when ice is broken away from a surface to which it was adhered. When treatment materials are used, knowledge of the broken surface structure and chemical composition will indicate the extent of diffusion of the materials into the ice, and the location at which mechanical failure occurred. Additional calorimetry and adsorption measurements will be included in the experimental design as appropriate.

3. IMPROVEMENT PLAN

Our improvement plan has four components;

o - to gain a wider base knowledge of interfacial science,

o - to take current expertise in the area of an applied problem and to supplement it by developing expertise in the relevant basic physical science,

o -to make available quality instrumentation for the measurement of thermophysical properties, and

o - to develop our current research into a definitive program which is nationally and internationally recognized.

The area chosen in which to generate an improved knowledge base and instrumentation facilities will complement the knowledge, interests, and equipment needs of several other faculty members, thereby facilitating interdisciplinary projects of competitive quality in the future. Instrumentation sought by other faculty members under this and other equipment proposals are complementary to the calorimeters sought in this proposal - all the equipment we seek to obtain would provide those facilities necessary for a broad program of materials science with a strong component of interfacial science.

Although we have been successful in obtaining research contracts for research of an applied nature, there is a need to significantly strengthen our knowledge base in the basic physical and chemical science relevant to surface and interfacial problems. This can only be achieved by providing faculty with the opportunity to study appropriate material. The comprehensive literature evaluation included in the first year of the study is intended to be a significant component of this. It will also be possible to maintain and enhance the current interaction with surface scientists at MRI, SRA, and SRI International through the SHRP sponsored research. Presently we have a significant opportunity, and support of the proposed work will allow us to continue to build upon it.

On our campus there exists an urgent need to obtain modern instrumentation and to significantly increase the range of equipment available. The interests and areas of expertise of the SDSM&T faculty focus on material science and interfacial science. Our Ph.D. programs and active research projects are in Geology and Materials Engineering and Science. There is a recognized need and a strong commitment to establish well equipped laboratories for Electron Microscopy, Molecular Spectroscopy, Thermophysical Properties, Heat Treatment, and Applied Interfacial Phenomena to support these programs. As indicated earlier, the addition of the requested microcalorimeter and DSC/TGA system will complement existing equipment and is an important component of the overall facility that is needed.

The specific project proposed is ideal for the SDSM&T campus because it builds on one of the strongest existing areas, and it will enhance a wide range of opportunities. Several related projects which would benefit can be identified.

Ice Adhesion to Metal Surfaces

Work on adhesion to metals will bring us more into the realm of the general adhesion problem, and into the specific ice adhesion-to-metal problem (aircraft, equipment in cold climates, shipping, etc.).

As for adhesion to geologic materials, optical and electron (SEM) microscopy will be needed to determine substrate morphology. Adsorption and calorimetric measurements will allow additional substrate characterization as well as the energetics of the subsequent bonding and ice growth. STEM can be used for investigation of fractured interfaces and FTIR measurements can be used to investigate the bonding of very thin layers of ice on a substrate.

Studies of Self-Heating and Combustion Mechanisms in Coals

For a number of years a group at SDSM&T (Rich, Munro and Ashworth) have been attempting to develop research in the area of self-heating and combustion mechanisms. Dr. Munro was successful in obtaining a small research grant, and, with the help of a small amount of internal money, we have designed and built a macro-calorimeter for this work. Acquisition of significant external funding has been prevented largely by the lack of facilities for DSC/TGA and micro-thermal analysis.

The problem to be studied is closely related to the ice adhesion problem, since in both cases one is dealing with the interaction of water, nitrogen and oxygen molecules with a complex substrate. When the interaction takes place, the interfacial energy leads to heating and eventually to chemical reaction. Isotherm, interfacial energy, SEM, STEM and spectroscopic studies similar to those of the adhesion project would now be correlated to the macro-calorimetric measurements designed to monitor actual self-heating and combustion events.

Humidity Sensing and Crack Propagation in Glass

Studies were carried out for a number of years on the properties of ice and atmospheric water vapor. This research project was sponsored by the Office of Naval Research. A large component of the work was the evaluation of humidity sensors and determination of the manner in which the water molecules interacted with anodized aluminum surfaces which have deep pores. It was determined that the interaction was extremely complex; it involved adsorption which was of a true physisorption nature, a diffusional process within the pores, and deep-seated adsorption within pores or cavities which had a very long time constant. As a result, the devices evaluated were not satisfactory for stratospheric sensing, and there were many unanswered questions regarding the relationship between water molecules and aluminum oxide molecules [17]. In order to take this work further, exactly the same type of calorimetric - adsorption isotherm - FTIR spectroscopy - SEM/STEM type of investigation is needed. This is another example of an area of existing expertise on our campus which has not been able to be developed because of the lack of facilities.

Very similar interactions are now being reported between water molecules and strained silica [18, 19, 20]. Although water molecules initially are physically adsorbed, the presence of strain apparently leads to the incorporation in a strongly bonded site at the tip of a crack. There is then a chemical binding which leads to surface hydroxyl groups, and a localized reduction of binding energy of the silica molecules. This results in the propagation of the crack by one atomic distance, and this process can be repeated. It has been found that the special relationship between the size and binding of water and silica molecules is important here; other types of molecule are much less effective. With the facilities proposed, this interesting and potentially important problem could be effectively tackled.

Projects in Metallurgy

Knowledge of bonding obtained for the purpose of destroying or preventing bonding are useful for problems for which the strength of an interface is to be enhanced. Thus there is an immediate application to problems of advanced bonded materials and advanced composites which use carbon or boron compound filaments. The work is also relevant to problems such as corrosion, metallurgical processing, and cathodic protection. In all cases the calorimetry would provide fundamental information which can be combined with microscopy and macroscopic studies to generate a systematic and comprehensive research effort.

These are just a few of a very large number of research projects of interest to and within the capability of SDSM&T personnel. This area of research does not come into direct competition with well established surface physics laboratories, but rather compliments the type of work performed there.

4. TIMETABLE OF RESEARCH

FIRST YEAR

TASK	MONTH 1	2	3	4	5	6	7	8	9	10	11	12
A. Critical Literature Survey & Experiment Design.												
1. Study SHRP H201 & H203 data bases	XXXX	XXXX	XXXX	XXXX	XXXX	XXXX	XXXX	XXXX	XXX			
2. Search of current literature									XXXX	XXXX	XXXX	XXXX
3. Evaluate design of Second Year Experiment and revise as appropriate												XXXX
B. Surface Area Measurements.												
1. Prepare powders of substrate materials	XXXX	XXXX	XXXX									
2. Determine surface areas of materials			XXXX	XXXX	XXXX							
3. Determine surface areas of treated materials						XXXX	XXXX	XXXX	XXXX	XXXX	XXXX	XXX
C. Immersional Heats of Wetting.												
1. Improve calorimeter	XXXX	XXXX	XX									
2. Freshly ground materials			XXXX	XXXX	XXXX	XXXX						
3. Material stored under various conditions					XXXX	XXXX	XXXX				XXXX	
4. Material treated with SO2								XXXX	XX			
5. Material treated with CMA									XXXX	XX		
6. Material treated with selected components of SO2 (4, 5 & 6 involve one level of treatment only)										XXXX	XXXX	XXXX
D. Adsorption Isotherms.												
1. Freshly ground materials		XX	XXXX	XXXX	XXXX							
2. Material stored under various conditions					XXXX	XXXX	XXXX				XXXX	
3. Material treated with SO2								XXXX	XX			
4. Material treated with CMA									XXXX	XX		
5. Material treated with selected components of SO2 (3, 4 & 5 involve one level of treatment only)										XXXX	XXXX	XXXX
E. Data Interpretation.												
1. Evaluate properties of substrate materials						XXXX						XXXX
2. Evaluate effect of treating materials												XXXX
F. Reports												
1. Quarterly progress report			X			X			X			
2. Prepare final report												XXXX

SECOND and THIRD YEARS

The research timetable for the second year will be decided during the last month of the first year of the project. It will be determined by the results of the first year and the time frame of the acquisition of equipment in the overall program. Likewise, the third year timetable will be determined at the end of the second year.

5. EQUIPMENT

As indicated in the Experimental Methods section, the work will be started using existing equipment. Additional instruments are necessary to improve the resolution of the calorimetry data in the second and third years of the project.

In this segment of the proposal describes the devices selected for these measurements. They have also been selected because they are capable of measuring the thermophysical properties of a very wide range of materials. The specific instruments which are needed are:

1) Microcalorimeter.
Specifications - SETARAM Model C80
Extensive capability for surface and solution studies
Wide temperature range -200°C to 200°C (or high temperature model 25°C to 1000°C)
Mixing capability and appropriate sample size
Cost - approx $60K for fully configured system.

Justification - this instrument is highly versatile and is capable of high precision; it is made by the manufacturer who has been highly recommended for the DSC system. Also, this calorimeter is based on the Calvet design, which is the best in this field.

2) D.S.C., T.G.A., + any other thermal property measurement systems
Specifications - SETARAM Model TG-DSC 111
Simultaneous DSC and TGA capability
Temperature range -123°C to 827°C
IBM P.C. or clone for data system
Wide range of measurement capabilities
Cost - approx $100K for fully configured system.

Justification - we have contacted a number of experts in the field and have discovered that there are significant problems with both the Perkin-Elmer and duPont instruments which were thought to be the appropriate choices. The thermal analysis specialists at N.B.S. Boulder have strongly recommended the instrument selected in terms of its quality, measurement capability, and suitability for the work which we wish to perform.

The selections have been made based on a comprehensive survey of equipment now available and a careful consideration of the capabilities of these systems. For the work presently envisioned the ability of the SETARAM system to perform simultaneous DSC and TGA was the primary factor for our choice.

6. REFERENCES.

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8. BUDGET

Proposal No. E&S 88-XX

Ice Adhesion and Related Studies.

FIRST YEAR

A. SENIOR PERSONNEL	CAL| ACAD |SMR			
1. T. Ashworth (P.I.)	2.25 1.00	(25% AY)		$15,933
2.	0.00 0.00			$0
3.	0.00 0.00			$0
4.	0.00 0.00			$0
5.	0.00 0.00			$0
6. (1)TOTAL SENIOR PERSONNEL				$15,933
B. OTHER PERSONNEL				
1. ( ) POST DOCTORAL ASSOCIATES				$0
2. ( ) OTHER PROFESSIONALS				$0
3. (1) GRADUATE STUDENTS	4.25 3.00	(1st yr PhD		$11,336
4. (2) UNDERGRADUATE STUDENTS	400 hr @ $5.00	(Jr/Sr)		$2,000
5. (1) SECRETARIAL-CLERICAL	100 hr @ $7.96			$796
6. ( ) OTHER				$0
7. (5) TOTAL OTHER PERSONNEL				$14,132
TOTAL SALARIES AND WAGES(A+B)				$30,065
C. FRINGE BENEFITS				
1. SENIOR PERSONNEL (STAFF)	16% of Salrs		$2,677	
2. OTHER PERSONNEL (STUDENTS)	8% of B3+B4		$1,067	
3. TOTAL FRINGE BENEFITS				$3,743
TOTAL SALARIES, WAGES AND FRINGE BENEFITS				$33,808
D. PERMANENT EQUIPMENT				
DATA SYSTEM - P.C. WITH ADDITIONS			$2,711	
- PRINTER			$600	
- DMM/SCANNER			$1,390	
TOTAL PERMANENT EQUIPMENT				$4,701
E. TRAVEL 1. DOMESTIC				$2,000
2. FOREIGN				
F. PARTICIPANT SUPPORT COSTS				
1. STIPENDS				
2. TRAVEL				
3. SUBSISTENCE				
4. OTHER				
TOTAL PARTICIPANT COSTS				$0
G. OTHER DIRECT COSTS				
1. MATERIALS AND SUPPLIES	(See Breakdown)			$2,230
2. PUBLICATION COSTS/PAGE CHARGES				$500
3. CONSULTANT SERVICES				
4. COMPUTER (ADPE) SERVICES				
5. MACHINING COSTS	(20hr@$25/hr)			$500
6. OTHER (Tuition Remission)	(1@2x12CH/Sem)			$2,196
TOTAL OTHER DIRECT COSTS				$5,426
H. TOTAL DIRECT COSTS (A THROUGH G)				$45,935
I. INDIRECT COSTS (SPECIFY)				
40.0% of SALARIES AND WAGES				
TOTAL INDIRECT COSTS				$12,026
J. TOTAL DIRECT AND INDIRECT COSTS				$57,961
K. RESIDUAL FUNDS				
L. AMOUNT OF THIS REQUEST				$57,961

BUDGET CALCULATIONS

EFFORT (Months) FALL	SPRING	SUMMR	TOTAL HRS	HRLY RATE	TOTAL MONTHS	MONTHLY SALARY	TOTAL SALARY
1.12	1.13	1.00			3.25	$4,902	$15,933
					0.00		$0
					0.00		$0
					0.00		$0
					0.00		$0
					0.00		
					0.00		
2.00	2.25	3.00	1256.7	$9.02	7.25	$4	$11,336 /GRA
			400.0	$5.00	0.00		$2,000
			100.0	$7.96	0.00		$796
					0.00		

PERMANENT EQUIPMENT BREAKDOWN

Control and Data Acquisition System		
VSS XT20C PC	$1,256	
Monitor (Color)	$425	
Modem GVC 1200-internal	$150	
2nd Floppy Drive	$150	
8087 Co-processor	$180	
GPIB Interface-internal	$550	$2,711
Printer		
Panasonic KX-P1524	$600	$600
Measuring Equipment		
Keithley 199 DMM/Scanner	$1,390	$1,390
TOTAL		$4,701

SUPPLIES AND MATERIALS BREAKDOWN

Software	$850	
IEEE-488 Cables	$180	
Miscellaneous Supplies	$1,200	$2,230
TOTAL		$2,230

TUITION REMISSION	Ttl CH	Rate/CH	
	24	$91:51	$2,196

VITAE
PRINCIPAL INVESTIGATOR

PERSONAL

Name: Dr. T. Ashworth S.S. Number: 503-66-6353
Rank: Professor of Physics
Institution: South Dakota School of Mines and Technology
Address: 501 East St. Joseph St.
Rapid City, South Dakota 57701-3995
Telephone (605) 394-2361

EDUCATION

Ph.D. Physics 1967 University of Manchester, England
B.Sc. (1st Class Honours) Physics 1961
University of Manchester, England

PROFESSIONAL EXPERIENCE:

1977-date	Professor of Physics, South Dakota School of Mines and Technology.
1985-1986	Visiting Scientist and Visiting Professor of Physics, Cornell University (Sabbatical Leave from SDSM&T).
1984-1985	Acting Head of Department of Physics, South Dakota School of Mines and Technology.
1980-date	Co-President, Ashworth and Associates, a research and consulting company. During the past three years we have obtained and completed 6 research contracts in the area of heat transfer and thermophysical properties.
1976-1979	Coordinator, Energy Research, Information and Conservation Center, South Dakota School of Mines and Technology.
1974-1975	Senior Research Fellow, National Center for Atmospheric Research, Boulder, Colorado. (Sabbatical Leave from SDSM&T).
1971-1977	Associate Professor of Physics, South Dakota School of Mines and Technology.
1968-1971	Assistant Professor of Physics, South Dakota School of Mines and Technology.
1966-1968	Turner and Newall Research Fellow, University of Manchester, Faculty of Technology.
1961-1966	Teaching Assistant, University of Manchester, Faculty of Technology.

AWARDS/HONORS

Elected Fellow, International Thermal Conductivity Conferences, 1985.
S.D.S.M.&T. Presidential Award, Outstanding Professor, 1984
Senior Research Fellowship, National Center for Atmospheric Research from August 1974 to August 1975
Turner and Newall Research Fellowship from 1966-68
S.R.C. Research Studentship, 1961-66
State Scholar, 1958-61

THESES AND DISSERTATIONS DIRECTED

* Joint supervisorship with faculty member in another department.

Engle, Michael W., Ph.D in Materials Engineering and Science.
"Heat Tranfer from Thin Films to LN2," (In Progress).

Westphal, Michael J., Ph.D in Materials Engineering and Science.
"Investigation of the Properties of Interfacial Regions between Bonded Solids", (In Progress).

Wood, Jeffery W., Ph.D in Materials Engineering and Science.
"Investigaton of Interfacial Structure and Thermophysical Properties by Thermo-Acoustic and SEM Techniques," (In Progress).
Supervised jointly with R. D. Redin.

Gu, Gong-En, M.S. in Physics,
"Evaluation of Methods for the Measurement of the Thermal Properties of Thin Films," (In Progress - expected completion May, 1989).

Hu, Yanping, M.S. in Physics,
"Evaluation of Transient Heating Methods for Thermal Conductivity Measurement," (In Progress - expected completion May, 1989).

Satter, Martin R., M.S. in Physics,
"Boundary Resistance Effects in Thermal Conductivity Measurements." (In Progress - expected competion December, 1988).

Wheeler, R. David, M.S. in Physics,
"Investigation of Interfacial Effects between Substrates and Solids Formed from Aqueous Solutions," (In Progress - expected completion December, 1988)

Ewing, Anthony P., M.S. in Physics,
"Some Aspects of Ice Adhesion," (1988).

Engle, Michael W., M.S.in Physics,
"Heat Tranfer from Thin Films to LN2," (1987).

Lu, Louie Leng, M.S. in Physics,
"Evaluation of Deicing Materials by Shear Testing," (1987).

Dixon, David J., M.S. in Chemical Engineering*,
"Effect of Moisture on Self-Heating of Coal," (1986).

Sun, You-Tai, M.S. in Mining Engineering*,
"Thermal Conductivity of Anisotropic Rocks under Uniaxial Compressive Load," (1985).

Lai, Lei-Ping, M.S. in Physics,
"The Thermal Conductivity of Tetrahydrofuran Hydrate and Ice IH," (1984).

Westphal, Michael J., M.S. in Physics,
"Effect of Diesel Oil Contamination on Powdered Sand Surface Phenomena," (1983).

THESES AND DISSERTATIONS DIRECTED (Continued)

Mahajan, J. K., M.S. in Mining Engineering*,
"Development of Re-usable Probes for Rock Temperature Measurements," (1982).

Dorland, Louis J., M.S. in Physics,
"Development of Methods for Thermal Conductivity Measurements," (1982).

Alexander, Thomas M., M.S. in Mining Engineering*,
"Investigation of Thermal Conductivity in Natural Materials," (1981).

Chancellor, R. A., M.S. in Mining Engineering*,
"Temperature Profile Studies in a Deep Hot Mine," (1981).

Murdock, Ronald A., M.S. in Mining Engineering*,
"Thermal Conductivity Systems for Measurements on Rocks Under Applied Stress," (1979).

Howe, Mark A., M.S. in Physics,
"An Investigation of the Moisture Adsorption Properties of Various Natural Materials," (1979).

Marin, J. Scott, M.S. in Electrical Engineering,
"An Investigation of Silicon Monoxide Thin Films as Relative Humidity Detectors," (1978).

Tollefsen, Bjorn, M.S. in Civil Engineering*,
"Mechanical Aspects of Ice Adhesion to Concrete." (1978).

Hsiung, Chia Yi, Ph.D. in Electrical Engineering,
"Transport Phenomena in Semiconductors," (1976)
Supervised jointly with R. D. Redin.

DeVries, J. L., M.S. in Physics,
"Models of Moisture Adsorption on Aluminum Oxide," (1975).

Moen, Richard S., M.S. in Physics,
"A Calorimetric Investigation of Surface Free Energy of Ice," (1975).

Hess, J., M.S. in Mathematics,
"Least Squares Curve Fitting With Constraints," (1974).

Loomer, J. E., M.S. in Physics,
"Transport Phenomena in Polymers," (1972).

Reiners, T. H., M.S. in Physics,
"Growth of Ice from the Vapor Phase in Electric Fields," (1972).

Pendo, M. C., M.S. in Electrical Engineering*,
"Solution of Heat Flow Problems and Optimization of Radial Flow Measurement Techniques," (1972).

Kopp, F. J., M.S. in Physics, M.S. in Physics,
"The Specific Heat of Ice," (1971).

THESES AND DISSERTATIONS DIRECTED (Continued)

Johnson, L. R., M.S. in Physics,
"Investigation of the Thermal Conductivity of Ice," (1970).

Smith, M. G., M.S. in Physics,
"Thermal Conductivity of Insulating Foams by a Radial Flow Method," (1969).

PROFESSIONAL ACTIVITIES

RESEARCH PROPOSALS SUBMITTED

FUNDED RESEARCH PROJECTS (1980 - present)

Ice-Pavement Disbonding Study
Midwest Research Institute, Strategic Highway Research Program
SHRP Prime Contract No. SHRP-87-H203, Subcontract No. 219-8977-1
Amount - $83,612 Peroid - 8 Oct. 87 - 30 Sept. 88

A Study of South Dakota Deicer #2
(S.D. State DOT)
1 July 87 - 30 June 88 $ 30,005

Developmental Work for Reliability Studies for a Computer Cryogenic System
(ETA Systems Inc.) 1 Mar 84 - 30 June 85 $ 40,000

Cosmic Ray Telescope
(University of Pennsylvania)
1 Jan. 82 - 31 May 83 $ 6,998

Guarded Hot-Plate System for Thermal Properties Lab
(N.S.F.)
1 May 82 - 30 April 83 $ 35,000

Adhesion of Ice to Highway Surfaces
(D.O.T.)
18 July 77 - 17 July 80 $180,996

SELECTED PUBLICATIONS

Steeple, H., and T. Ashworth, (1965), "The Minimum-Residual Refinement of b-Uranium from Polycrystalline Data," Acta Cryst., 21, 955-958.

Rechowicz, M., T. Ashworth, and H. Steeple, (1967), "Heat Transfer Across Pressed Contacts at Low Temperatures," Cryogenics, 7, 369-370.

Ashworth, T., and H. Steeple, (1968), "The Continuous-Heating Method of Calorimetry and its Application to the Study of Low-Temperature Specific-Heat Anomalies in Methyl-Ammonium Alum.," Cryogenics, 8, 225-234.

Ashworth, T., and M. Rechowicz, (1968), "Properties of Materials--I Application of Adhesives," Cryogenics, 8, 361-363.

Bunting, J. G., T. Ashworth, and H. Steeple, (1969), "The Specific Heat of Apiezon N Grease," Cryogenics, 9, 385-386.

Ashworth, T., L. R. Johnson, R. D. Redin, and R. D. Thomas, (1969), "Improvements of the Linear Heat-Flow Method," Proc. Ninth Conference on Thermal Conductivity, USAEC Conference Report No. 691002, pp. 497-504.

Bunting, J. G., T. Ashworth, and H. Steeple, (1970), "Specific Heat of Methyl Ammonium Chromium Alum," Phys. Lett., 33A, 37-38.

Kreitman, M. M., T. Ashworth, and M. Rechowicz, (1972), "A Correlation between Thermal Conductance and Specific Heat Anomalies and the Glass Temperature of Apiezon N and T Greases," Cryogenics. 12, 32-34.

Ashworth, T., and L. R. Johnson, (1971), "Thermal Conductivity of Nylon from 4 to 350 K," Proc. Eleventh International Conference on Thermal Conductivity.

Kopp, F. J., and T. Ashworth, (1972), "Carbon Resistors as Low Temperature Thermometers," Rev. Sci. Instr., 43, 327-332.

Ashworth, T., L. R. Johnson, C. Y. Hsuing, and M. M. Kreitman, (1973), "Use of the Linear Heat-Flow Method for Poor Conductors and its Application to the Thermal Conductivity of Nylon," Cryogenics, 13. 34-40.

Ashworth, T., (1972), "Measurement of Absolute, Instantaneous Conductivities of Poor Conductors," Proc. Fourth International Cryogenic Engineering Conference, Eindhoven,The Netherlands, May 1972, 274-278.

Ashworth, T., (1972), "Measurement of the Thermal Properties of Ice," Proc. Fourth International Cryogenic Engineering Conference, Eindhoven, The Netherlands, May 1972, 377-379.

Kreitman, M. M., and T. Ashworth, (1972), "Thermal Conductivity of Nylon," Proc. Twelfth International Conference on Thermal Conductivity, Birmingham, Alabama.

Ashworth, T., J. E. Loomer, and M. M. Kreitman, (1973), "Thermal Conductivity of Nylons and Apiezon Greases," Adv. Cryo. Eng., 18. 271-279.

SELECTED PUBLICATIONS (continued)

Frels, W., D. R. Smith, and T. Ashworth, (1974), "Vapor Pressure of Nitrogen Below the Triple Point," Cryogenics, 14, 3-7.

Ashworth, T., (1975), "On the Precision Continuous Method of Calorimetry and Its Application to the Determination of Surface Energy," in Thermal Analysis (ed. Buzas), Vol. 3, 799-805.

Ashworth, T., D. R. Smith, and J. L. DeVries, (1977), "A Convectively Mixed Humidity Chamber," Rev. Sci. Instrum., 48, 1595-1600.

Redin, R. D., C. Y. Hsiung, and T. Ashworth, (1978), "Thermal Conductivity of Heavily Doped p-type Indium Antimonide," Thermal Conductivity, (Plenum Publishing Co., N.Y.), 15, 57-62.

Ashworth, T., W. G. Lacey, and E. Ashworth, (1978), "Drift Measurement Technique Applied to Poor Conductors," ASTM S.T.P. 660. pp. 426-436.

Ashworth, T., and C. A. Knight, (1978), "Cylindrical Ice Accretions as Simulations of Hail Growth: I. Effects of Rotation and of Mixed Clouds," J. Atmos. Sci., 35, 1987-1996.

Knight, C. A., T. Ashworth, and N. C. Knight, (1978), "Cylindrical Ice Accretions as Simulations of Hail Growth: II. The Structure of Fresh and Annealed Accretions," Ibid, 1997-2009.

Ashworth, E., T. Ashworth, and C. A. Knight, (1980), "Cylindrical Ice Accretions as Simulations of Hail Growth: III. Analyses Techniques and Application to Trajectory Determination," J. Atmos. Sci., 37, 846-854.

Ashworth, T., B. Tollefsen, and J. A. Weyland, (1979). "Adhesion of Ice to Concrete Surfaces - Preliminary Findings," National Academy of Sciences, Special Report 185, Snow Removal and Ice Control Research, pp. 23-29.

Ashworth, T., and J. A. Weyland, (1979), "Investigation of the Basic Forces Involved in the Adhesion of Ice to Highway Surfaces: Interim Report," Report DOT/RSPA/DPB-50/79/28 prepared for the Office of University Research, Research and Special Programs Administration, U.S. Department of Transportation.

Ashworth, T., and J. A. Weyland, (1982), "Investigation of the Basic Forces Involved in the Adhesion of Ice to Highway Surfaces: Second Interim Report," Report DOT/RSPP/DPB-50/82/5 prepared for the Office of University Research, Research and Special Programs Administration, U.S. Department of Transportation.

Ashworth, T., R. C. Murdock, and E. Ashworth, (1983), "Thermal Conductivity Systems for Measurements on Rocks Under Applied Stress," Thermal Conductivity 16 (ed. David C. Larson), pp. 91-99, Plenum Press.

Ashworth, T., T. M. Alexander, and E. Ashworth, (1983), "Thermal Conductivity of Carbonate Gneiss under Applied Uniaxial Pressure," Thermal Conductivity 17 (ed. J. G. Hust), pp. 737-744, Plenum Press.

SELECTED PUBLICATIONS (continued)

Ashworth, E., J. E. Mahajan, and T. Ashworth, (1983), "Study of Temperature Profiles in Rock using a New Thermistor Probe." 112th AIME Annual Meeting, Atlanta, Georgia.

Ashworth, E., and T. Ashworth, (1984), "The Application of Finite Element Analysis for Comparison of In Situ and Laboratory Measurements of Rock Thermal Properties," Proceedings of 3rd International Mine Ventilation Congress (eds. M. J. Howes and J. J. Jones), pp. 343-347, Institution of Mining and Metallurgy.

Maher, S., and T. Ashworth, (1985), "A Data Acquisition System Capable of Monitoring Small Signals," in Thermal Conductivity 18 (eds. T. Ashworth and David R. Smith), pp. 153-160, Plenum Press.

Satter, M. R., and T. Ashworth, (1985), "Thermal Resistances at Interfaces," ibid, pp. 641-650.

Ashworth, E., and T. Ashworth, (1985), "Finite Element Analysis of Unguarded Thermal Conductivity Apparatii," ibid. pp. 599-609.

Ashworth, T., and D. R. Smith, (1985), Thermal Conductivity 18, (Editors), Plenum Press.

Beck, A. E., and T. Ashworth, (1985), "Thermophysical Properties of Geological Materials," High Temperatures-High Pressures, 17, 357-358.

Ashworth, T., L. R. Johnson, and L. P. Lai, (1985), "Thermal Conductivity of Pure Ice and THF Clathrate Hydrate," High Temperatures-High Pressures; 17, 413-419.

Ashworth, T., D. R. Smith, and E. Ashworth, (1985), "Measurement Methods for Thermal Transport Properties of Rocks," Research and Engineering Applications in Rock Masses (ed. E. Ashworth), pp. 797-805, A. A. Balkema, Rotterdam (1985).

Ashworth, T., and D. R. Smith, "Thermal Conductivity of Sintered Cordierite by Guarded and Unguarded Methods," Thermal Conductivity 19, (ed. D. W. Yarbrough), in Press.

Ashworth, T., and M. W. Engle, "Heat Transfer Coefficients of Metal Wires and Films Immersed in Liquid Nitrogen," in preparation.

10. CURRENT AND PENDING RESEARCH SUPPORT

Current Support

Ice-Pavement Disbonding Study
Midwest Research Institute, Strategic Highway Research Program
SHRP Prime Contract No. SHRP-87-H203, Subcontract No. 219-8977-1.
Amount - $83,612 Period - 8 Oct. 87 - 30 Sept. 88

A Study of South Dakota Deicer #2
South Dakota State Department of Transportation, Contract No. 3213.
Amount - $30,005 Period - 1 July 87 - 30 June 88.

Pending Proposals

Ice-Pavement Disbonding Study
Midwest Research Institute, Strategic Highway Research Program
SHRP Prime Contract No. SHRP-87-H203, Subcontract No. 219-8977-1.
Amount - $70,390 Period - 1 Oct. 88 - 30 Sept. 89

An Integrated Laboratory Program to Enhance Knowledge of Materials Properties and to Develop Computer-Based Instrumentation Skills
Submitted to the National Science Foundation, November, 1987.
Instrumentation and Laboratory Improvement Program, 1 year duration.
Amount requested - $19,381 (+ $19,381 matching)

9. RESULTS FROM PRIOR NSF SUPPORT

Under an NSF funded Senior Research Fellowship at the National Center for Atmmospheric Research (August, 1974 to August, 1975), and continuation work under the contract:

Analysis of the Crystalline Nature of Artificial Hail: A Corroboration and Refinement of the Techniques for Trajectory Determination.
National Center for Atmospheric Research,
NCAR Prime Contract No. NSF-C760, Subcontract No. NCAR S6018.
Amount - $7,483 Period - 21 June, 76 - 31 Dec. 77

The following journal and conference proceeding articles resulted from the work performed:

Ashworth, E., T. Ashworth, and C. A. Knight, "Cylindrical Ice Accretions as Simulations of Hail Growth: III. Analyses Techniques and Application to Trajectory Determination," J. Atmos. Sci., 37, 846-854, 1980.

Knight, C. A., T. Ashworth, and N. C. Knight, "Cylindrical Ice Accretions as Simulations of Hail Growth: II. The Structure of Fresh and Annealed Accretions," 35, 1997-2009, 1978.

Ashworth, T., and C. A. Knight, "Cylindrical Ice Accretions as Simulations of Hail Growth: I. Effects of Rotation and of Mixed Clouds," J. Atmos. Sci., 35, 1987-1996, 1978.

Knight, C. A., T. Ashworth, and N. C. Knight, "Experiments on the Factors Involved in Interpreting Natural Hail Crystal Textures," Ibid, pp. 186-193, 1978.

Ashworth, E., T. Ashworth, and C. A. Knight, "Analysis Parameters for Accreted Ice," Preprints-Conference on Cloud Physics and Atmospheric Electricity, Issaquah, Washington, July 1978, pp. 182-185, 1978.

Ashworth, T., and E. Ashworth, "Single Doppler Study of the Airflow in an evolving Cell with a Weak Echo Region," Proc. 17th Conference on Radar Meteor., 126-131, 1976.

Under NSF Instumentaion Grant:

Acquisition of Guarded Plate Apparatus for Study of Heat Transfer Mechanisms
National Science Foundation, Grant No. PRM-8205602.
Amount - $35,000 Period - 1 May 82 - 30 April 83
(includes $10,000 matching)

The apparatus was acquired. Subsequently it was used for numerous measurements on geologic and potential standard reference materials. One rounnd-robin measurement series which we participated in was reported in Dynatech Report No. 2323 to Rockwell International (Rockwell Report SD-BWI-TS-011) and has been presented by dynatech staff at several national and international meetings. Data on an international co-operative study on Cordierite has been presented and is currently in press. Dr. David R. Smith, who was co-investigator on the project, is now at the National Bureau of Standards, Boulder, Colorado; he is working specifically on further developing the guarded-plate apparatus. He has recently submitted several papers on work performed on the apparatus and continued at NBS. Several other projects for which the apparatus was an essential component have also resulted in publications. Co-operative work on the thermal conductivity of rock for several new mines with collegues at the University of Nevada, Reno, and a mine ventilation consulting company is currently in progress.

Ashworth, T., and D. R. Smith. "Thermal Conductivity of Sintered Cordierite by Guarded and Unguarded Methods," Thermal Conductivity 19, (ed. D. W. Yarbrough), in Press.

Ashworth, T., D. R. Smith, and E. Ashworth, "Measurement Methods for Thermal Transport Properties of Rocks," Research and Engineering Applications in Rock Masses (ed. E. Ashworth), pp. 797-805, A. A. Balkema, Rotterdam 1985.

Beck, A. E., and T. Ashworth, "Thermophysical Properties of Geological Materials," High Temperatures-High Pressures, 17, 357-358, 1985.

Ashworth, E., and T. Ashworth, "The Application of Finite Element Analysis for Comparison of In Situ and Laboratory Measurements of Rock Thermal Properties," Proceedings of 3rd International Mine Ventilation Congress (eds. M. J. Howes and J. J. Jones), pp. 343-347, Institution of Mining and Metallurgy, 1984.

10. CURRENT AND PENDING RESEARCH SUPPORT

Current Support

Ice-Pavement Disbonding Study
Midwest Research Institute, Strategic Highway Research Program
SHRP Prime Contract No. SHRP-87-H203, Subcontract No. 219-3977-1.
Amount - $83,612 Period - 8 Oct. 87 - 30 Sept. 88

A Study of South Dakota Deicer #2
South Dakota State Department of Transportation, Contract No. 3213.
Amount - $30,005 Period - 1 July 87 - 30 June 88.

Pending Proposals

Ice-Pavement Disbonding Study
Midwest Research Institute, Strategic Highway Research Program
SHRP Prime Contract No. SHRP-87-H203, Subcontract No. 219-3977-1.
Amount - $70,390 Period - 1 Oct. 88 - 30 Sept. 89

An Integrated Laboratory Program to Enhance Knowledge of Materials Properties and to Develop Computer-Based Instrumentation Skills
Submitted to the National Science Foundation, November, 1987.
Instrumentation and Laboratory Improvement Program, 1 year duration.
Amount requested - $19,381 (+ $19,381 matching)