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THE DEVELOPMENT OF A PERFORMANCE TEST PROCEDURE AND MEASUREMENT TECHNIQUE IN A BATCH MIXING SYSTEM

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NOMENCLATURE

Bpr = probe response intercept

Bsa = signal amplitude intercept

C = height of the impeller from the bottom of the tank

C/T = ratio of impeller height to tank diameter

CSSD = cumulative successive squared difference

CV = weight of liquid in the calibration loop

Ct = concentration at time t

D = diameter of the impeller

D/T = ratio of impeller diameter to tank diameter

Den = salt solution density

Df = degrees of freedom

Hp = horsepower

Ki = change in intercept/°C change in temperature

Ks = change in slope/°C change in temperature

MR = meter reading

Mpr = probe response slope

Msa = signal amplitude slope

N = impeller rotation speed (when in conjunction with Np)

N = the number of salt additions to be made between changes of the tank fluid

Np = power number (dimensionless)

P = power

PR = probe response

PRT = theoretical probe response at a given concentration, voltage (SA) and experimental temperature (T)

S = the amount of NaCl in grams per run

S(t) = CSSD

SA = signal amplitude

T = diameter of the tank

T = temperature of the experiment

Tbase = base calibration temperature (24 °C)

V = weight of liquid in the mixing vessel

W = the weight percent of NaCl in the mixing vessel after N additions

Z = height of liquid

Z/T = ratio of liquid height to tank diameter

b = intercept for point concentration calculation

d = depth of an impeller blade

g = gravitational constant

m = slope for point concentration calculation

p = liquid density

ssd = sum of standard deviation squared

w = width of an impeller blade

The Development of a Performance Test Procedure and Measurement Technique in a Batch Mixing System

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A performance test procedure and measurement technique for a batch mixing system is described using conductivity probes. The design of the automated experimental apparatus is described, and experimental procedures are given. Data collected from the experiments are explained, and a mixtime analysis of the data is performed. Conductivity probe response curves provide a good representation of the system dynamics. Mixtime analysis allows for probe response comparisons to evaluate system geometry and probe location.

Key words: batch mixing; calibration techniques; conductivity probes; experimental apparatus; mixtime analysis; torque measurements

INTRODUCTION

Mixing is one of the most important and one of the most frequently performed operations that occurs in industrial processes for material conversion. Surprisingly, it is also one of the least clearly understood processes. The diversity of mixing systems and the variety of techniques used for analysis of the mixing process make communication and cooperative efforts between interested parties difficult. Much work needs to be done to establish standards for performance and analysis in the mixing field.

The purpose of this project is to develop performance test procedures and measurement techniques for batch mixing systems. The performance tests are to be designed such that they will enhance understanding of the mixing process, aid in facility design, and be useful for in-plant trouble-shooting of batch mixing systems.

Work for this project was carried out in three basic areas. First, an investigation of the literature was made to determine the scope of work to be done, the mixing system to be used, the components to be mixed, and the type of measurements to be made. Second, based upon knowledge gained from the literature, an experimental apparatus was designed and built. Third, experiments were designed, performed, and analyzed in such a manner as to fulfill the goal of developing a performance test procedure and measurement technique.

Due to the complexity of mixing systems, and the large number and variety of parameters, limitations had to be set regarding the scope of work to be

done. A single standard geometry was specified for the laboratory scale equipment, and research was carried out in only the simplest mixing system (i.e., liquid blending). Because of limited resources, the experimental work was limited to the laboratory scale. The variables that were allowed to change were the impeller type (pitch and flat blade turbines), impeller speed (50, 150, and 300 rpm), impeller height from the tank bottom ($C/T = 0.13, 0.33$ and 0.50), and the concentration of NaCl in the system (<0.10 wt percent). The data collected were analyzed to determine the viability of using the measurement technique as a means for evaluating the mixing process. The data were not compared to other work in the field, as this project was not designed or intended to be a comprehensive research analysis and comparison effort.

The following conclusions were formulated based upon the work done:

- 1) A performance test using conductivity probes was developed which can be used to enhance understanding of the mixing process, aid in facility design, and be useful for in-plant trouble-shooting for a batch mixing system.
- 2) A standard geometry has been determined for use in laboratory testing.
- 3) Conductivity probes are flexible, accurate, reliable, and give reproducible results.
- 4) Probe response curves are a viable means of observing the dynamics of the mixing process in a qualitative manner.
- 5) Mixtime analysis provides useful insights into the mixing process, and can be used to enhance understanding of the mixing process, aid in facility design, and be useful for in-plant trouble-shooting of batch mixing processes.
- 6) The torque-arm/weight-scale technique used for these experiments to measure torque provides accurate, reliable, and reproducible information needed for power calculations.

The following recommendations were formulated based upon the work done:

- 1) The work done in this project is incomplete, and further testing is necessary for a better understanding of the mixing process.
- 2) Further work should be done using an expanded parameter list and a broader range of testing for each parameter.
- 3) A moderate size facility (approximately $0.379-0.757 \text{ m}^3$ (100-200 gallons)) is needed for comparison testing of work done in the laboratory scale equipment. All experiments run on the laboratory scale should in turn be run on the moderate scale equipment.

- 4) Work should be expanded to include other mixing systems. The system which should be worked on after miscible liquids is most likely a solid-liquid system.
- 5) The information collected should be more rigorously analyzed, removing qualitative analysis as much as possible from the tabulated results.

BACKGROUND

Mixing, as one of the key chemical processes in most industrial chemical plants, has been studied seriously for the last 35 to 40 years. Many advances have been made in the understanding of mixing and the mechanisms which surround it. Much work has already been carried out by the likes of Rushton, Oldshue, Brodkey, Gray and Uhl, Nagata, and many others. In just the last decade, approximately 100 papers have been published annually concerning the field of mixing. All of the work was meant to help define mixing patterns, methods for enhancing mixing operations, and methods for improving mixing efficiencies. However, much is still unknown, and there are many areas of disagreement yet to be resolved. One major problem is the lack of standardization in experimental procedures, design criteria, and design correlations. Standardization in these areas would allow the various individuals in the mixing field to communicate more effectively with one another.

The subject of mixing covers many different areas such as pipe mixing, continuous stirred tank mixing, and batch mixing. This paper will investigate only the area of batch mixing. In studying batch mixing, one must select the system, materials in the system, and measurement technique(s) to be used.

In 1981, Hilby stated that because of the number of possible variables in the mixing system, the simplest system possible should be used as a basis for initial studies (Hilby, 1981). To this end, a large number of studies deal with blending of miscible liquids. This is as opposed to immiscible liquids, gas-liquid, or solid-liquid systems. If blending of miscible liquids is selected as the system to be studied, then it only remains to determine the materials and measurement technique most suitable for the given operating conditions.

Measurement techniques and materials used are related to one another as Kappel (1979) described in his survey of appropriate techniques for measuring changes in a batch mixing system (Kappel, 1979). He concluded that a tracer-type measurement would be most effective and convey the most information in a blending-type operation. To this end, photographic tracers, and/or color

detectable tracers were considered best. However, as is often the case in the mixing field, disagreement about tracers is quite apparent. Many feel that the use of electrolytic tracers and conductivity probes is a better system to use (Neal, Bankoff, 1963). This is because of the relative degree of sensitivity, and the increased flexibility which conductivity probes provide over other types of tracers. The difficulty with conductivity probes revolves around the construction and operation of the probe circuitry which is needed to obtain meaningful responses. It was felt that our research could be best carried out by the use of conductivity probes, which are more flexible and more likely to be used in the field than other similar tracer techniques.

The idea for and use of conductivity probes as a measurement technique is not new. In 1935, Jones and Bollinger evaluated the notion of using resistance as a way to measure ion concentration (Jones, Bollinger, 1935). They proved that the use of electrodes to measure concentration changes was possible (an NaCl solution was used). Later, in 1956 Prausnitz and Wilhelm developed a usable probe using platinized electrodes in a small tank (Prausnitz, Wilhelm, 1956). The design of the probe, which measured an entire tank's concentration, was substantially improved in 1960 by Cairns and Prausnitz (Cairns, Prausnitz, 1960). Late in 1960, Lamb et al. measured concentration gradients with their version of a platinized conductivity probe (Lamb, Manning, Wilhelm, 1960). Keeler and Gibson in 1963 and 1964 further improved the probe such that volumes as small as $5 \times 10^{-10} \text{ m}^3$ could be measured inside of a tank (Gibson, 1963) (Keeler, 1964). Torrest and Ranz (1969) realized that resolution of the conductivity probe could most properly be enhanced through the design of the circuitry connected to it as opposed to further work on the probe itself (Torrest, Ranz, 1969). They improved the probe response by the use of a filtered ac bridge circuit. In 1975, Khang and Fitzgerald further improved the circuit to the point where there was a linear response to concentration, high and low noise were reduced, and errors caused by cross-talk were removed (Khang, Fitzgerald, 1975). This allowed accurate use of more than one probe at a time. Sheppard and Doddington (1977) improved accuracy even more by adding calibration routines for concentration and temperature to the use of conductivity probes (Sheppard, Doddington, 1977).

In our studies, it was important to determine the system, materials, and measurement techniques most appropriate for fulfilling our objective of creating standard performance tests and measurement techniques for batch mixing systems. Because mixing systems vary so greatly, it was clearly

understood that one technique would not suffice for all systems. Thus, keeping in mind that several techniques would eventually be required, the simplest system was investigated first. That system being blending of low viscosity miscible liquids. The materials chosen were an NaCl solution and deionized water at room temperature. The use of these materials allows for the use of conductivity probes, which are more flexible than most tracer systems, and also are capable of operating in a field situation, such that tests can be performed on-site. Thus, the system, materials to be used, and measurement technique for our experimentation were established based upon previous work done in the fields of mixing and conductivity probe construction.

APPARATUS

The apparatus for the mixing experiments was designed with consideration being given to six specific criteria: safety, automation, reliability, reproducibility, ease of operation, and the ability for the equipment to be used later by industry in a plant environment. Careful consideration was given to creating an efficient unit that would satisfy the criteria and produce results which would fulfill the requirements of the project's stated objective. To insure that work done would be usable by industry, a workshop was held in October 1983 to discuss the research to be attempted and the equipment to be used (see Appendix I).

Members of the workshop represented the manufacturers of mixing equipment, the users of mixing equipment, and academic researchers in the mixing field. This group of experts was called in to help in evaluating the direction of the project's research, and the equipment that should be used to perform the research. It was decided that a standard geometry of some kind should be used such that the largest number of interested parties could correlate the NBS data with their own results in the most direct manner. Thus, the members of the workshop made recommendations for the tank size, baffles, impellers, rotation rates, and horsepower, as well as making suggestions which specified geometric configurations for the mixing equipment. It was left to the researcher to decide which recommendations and suggestions should be put into effect and which should not.

The actual experimental apparatus consists of four major parts. The first is the physical equipment such as the tank, baffles, impeller, and motor. The second is the data collection system which consists of a computer and its accompanying software. The third is the measurement devices for

concentration, tracer addition, and torque. Finally, there are the materials used in the experiments.

Physical Equipment:

The physical equipment was meant to be two specific sizes, such that scale-up information could be obtained. There was to be an approximately 0.30 m (1.0 foot) diameter tank with accompanying attachments, and there was to be an identically equipped 0.91 m (3 foot) diameter facility (see Appendix II). Due to limited resources, the research was restricted to work using the 0.30 m (actually 0.25 m (9.75 in)) diameter facility only.

In the 0.25 m diameter facility, the Z/T ratio was held at 1.0. This was a fairly standard value from the literature inside the specified range between 0.8 and 1.5 for Z/T. The C/T ratio was shifted in the range between 0.00 and 0.50 in three steps (0.13, 0.33, and 0.50). The D/T ratio was to be maintained between 0.25 and 0.50, and was actually maintained at 0.41 for all impellers used. A standard geometric configuration was decided upon using four full length baffles with minimal bottom and side clearance, spaced 90 deg apart. Initial studies used a flat bottomed, clear tank (see fig. 1). There were two impellers used, one a flat, six-blade turbine with a $w/d = 1/6$, and the second a 45 degree pitch, six-blade turbine with a $w/d = 1/5$ (see fig. 2). The use of four-bladed impellers was preferable as it is more common to have four-bladed impellers in industry, but because of the small scale and easy availability of the six-blade turbines (commercially available equipment), it was felt that they would be adequate for initial studies. Only single impeller tests were run on a vertical shaft centrally located. A 186 watt (1/4 hp) variable speed motor was used to insure that up to 2.95 kW/m^3 (15 hp/1000 gal) was available. The combination of all of the aforementioned attributes was deemed to present the best representation of a standard batch mixing geometry (see fig. 1).

Data Collection:

The second part of the experimental apparatus is the data collection system. It consists of two parts, the computer hardware and the computer software. The hardware in this case is a high-speed, scientific computer with a 32-bit internal structure and an accompanying multiprogrammer unit (see fig. 3). The computer, with the multiprogrammer, is designed to send and receive signals to or from external devices (up to approximately

30000/s). In this way, the computer can control the experimental run, reducing the chance for error, as well as collecting data from the experiment. The multiprogrammer consists of five A/D cards, five 4k memory cards, a timing card, and a relay card. The computer, by the use of software, controls the functions and operation of these cards.

The software is truly the heart of the experiment. It is designed such that once the experiment is started, no manual changes need or in fact can be made. The computer program not only controls the experiment, but also controls the timing of the experiment and collects the data. The program is also capable of converting the data into concentrations, and provides a graphical representation of the just completed experiment (see Appendix III for program listing). More information concerning the user input to the program during the experiment will be given in the experimental procedure.

Measurement Equipment:

The third part of the experimental apparatus is the measurement equipment. The measurement equipment consists of the conductivity probes, the probe circuits, a torque measuring device, and an rpm meter. The conductivity probes measure the changes in ion concentration that occur within the system. To gain insight into the workings in the batch mixer, it was necessary to position probes in several locations throughout the tank. Four probes were used. Probe locations were determined based upon general knowledge of flow patterns obtained from the literature. Thus, the probes were positioned in an area of good mixing in the tank, and an area of poor mixing on the surface of the mixing fluid, in front of a baffle and behind a baffle (see fig. 4).

The original experiments were performed with homemade probes (described by Khang and Fitzgerald, 1975) which measured essentially a point volume. These probes proved unreliable, and experimental results were not reproducible. Initial testing of commercial probes indicated they would be much more reliable with more reproducible results. For this reason, commercial probes were used in the experiment. Many types are available, and the one selected was picked based upon its similarity to the homemade probes, its ease of acquisition, and its cost. It must be pointed out that any one of many various commercial probes will work adequately. The probe selected uses two small platinized plate electrodes approximately 2 mm apart, with a measurement volume of about $8 \times 10E-6 \text{ m}^3$ (see fig. 5). This volume was felt to be small enough to be considered a point volume when compared to the volume (10.830 m^3)

of the mixing vessel (less than 0.0001 percent of the mixing vessel volume). The probes were equipped with glass jackets to help reduce extraneous noise. At least in intent, these probes followed the ideals of probe design as suggested by Khang and Fitzgerald (1975), and subsequently were felt to be adequate. They proved reliable, and are readily available to all members of the mixing community.

As was discussed earlier, the resolution of a conductivity probe is significantly increased by the design of its accompanying circuit. This is also the case with the setup used in these experiments. The circuit was designed such that it has a wide range of sensitivity, responds proportionally to changes in concentration, eliminates high and low end noise, and reduces cross-talk to the point where more than one probe can be accurately used simultaneously (modification of Khang and Fitzgerald (1975) design by D. M. Ginley and Nolan Frederick 1984). The use of an oscillator amplifier, several filters and operational amplifiers make this possible (see fig. 6). Because each probe needs its own circuit, four pc-boards were built, each with its own set of external adjusting knobs. The pc-boards were also connected to a common voltmeter so that calibrations and comparisons between probes could be performed reproducibly.

The other parts of the measurement equipment, are the rpm and torque devices. These two devices combine to allow one to calculate the power used for the experiment. The rpm meter is a belt-driven tachometer that is read manually, and used to set the rpm for a given experiment. Torque measurement may be obtained in several different ways. A strain gauge may be placed on the impeller shaft, or the motor may be mounted on a "frictionless" bearing with torque being measured by use of a weigh scale and torque arm, or a plate and strain gauge apparatus may be placed beneath the tank and torque measured by the plate's movement relative to a fixed reference point. All of these methods have their advantages and disadvantages which will be discussed in more detail in a following section. Because of design difficulties, and the need for flexibility, as well as availability of equipment, a "frictionless" bearing was positioned under the motor and gear box, and changes in torque were measured by a weigh scale using a weight connected to a torque arm attached to the gear box (see fig. 7). Although not as sophisticated as some other methods, this torque measuring method proved both reliable and reproducible.

Materials:

The final part of the experimental apparatus is the materials used in the system, and the transport equipment necessary for tracer injection. Although many materials are available, considerations for safety and disposal dictated that simple materials be used. The simplest electrolytic blending system that meets the criteria of safety and easy disposal is most probably a tracer of NaCl solution, and deionized water. In using these materials a 9.62 wt. percent solution of NaCl was added in approximately $0.0107E-3 \text{ m}^3$ amounts to the mixing vessel at the beginning of each run. One pulse only was added for each experimental run. Acetic acid was also tested and found acceptable, but less desirable for use because of problems with the safety and disposal requirements. Use of the probes was also attempted in a two-phase system of immiscible liquids (dichloromethane and deionized water). The probes read both phases at the same time, and as such averaged the concentrations in the separate phases. This made it very difficult to discern what was taking place in the experiment. Because of handling, safety and disposal problems it was felt that further investigation into this behavior of the probes, at this time, was unwarranted (see Appendix IV).

Injection of the tracer into the system is a very important part of the experiment, and if not done properly could negatively affect the results of the experiment. To this end, careful consideration was given to how to inject the tracer, where to inject the tracer and how much of the tracer was to be injected. The amount of tracer injected needed to be small in respect to the volume of the tank (< 0.10 vol. percent). This insures that the injection has the least effect on the mixing flow patterns, and the relative integrity of the system is unchanged. The injection point needed to be a point that could be standardized for all experimental configurations, and insure that the tracer entered the mixing flow pattern as quickly and with the least disturbance possible. Thus, the injection point was selected to be $0.1875 \times D$ above the impeller, and $0.375 \times D$ away from the impeller shaft center (see Figure 8). Other injection points are of course possible, but a study of injection points was felt to be beyond the scope of the current experimental work.

The only remaining question is how to inject a standard amount of tracer reproducibly in a substantially short amount of time ($< 0.75 \text{ s}$). This was accomplished by the use of a tracer addition system (design help from S. J. Khang). This system consisted of two separate loops with a common calibrated tracer loop of $0.0107E-3 \text{ m}^3$ (0.0897 vol. percent of total tank liquid). The

first loop was designed to flush and fill the calibrated tracer loop with the tracer. The second loop ran continually, taking a small amount of liquid out of the tank, and then putting it back in at the injection point (approximately $1.07\text{E-}5 \text{ m}^3/\text{s}$ (0.17 gpm)). The suction point was located in an area of good mixing 180 deg away from the injection point (see fig. 8). At the appropriate time, the tracer was flushed from the calibrated tracer loop, and into the tank at the injection point. The time for complete injection, out of the addition system and into the tank was 0.6 seconds. To insure reproducibility, the addition system was computer controlled, and rigorously tested before use in the experiments. The system only failed once in 588 experimental runs. The design of this system is such, that by replacing one calibrated loop for another, differing amounts of tracer could be added. The system will also work with a variety of tracers and at a variety of injection points with only minor modifications (see fig. 9).

CALIBRATION AND ACCURACY

Developing appropriate equipment is not enough to insure a successful experiment. It is also important to organize the experiment in such a manner that the most and best information possible is obtained (see table 1). For the experiments that will be performed, it is therefore essential that the location of the probes in the mixing vessel be carefully thought out. Probe location should be such that the four probes being used in the vessel give the most coherent picture of what is actually happening in the mixing vessel. In addition, the probes must be properly calibrated and the reproducibility and accuracy of their responses verified.

Probe Location:

Once the system geometry is established, it is important to decide where the probes should be located in the mixing vessel such that the largest amount of information is obtained for an experiment. This can only be done if the flow patterns and mixing vessel geometry are carefully considered. Another point of importance is the physical positioning of the probes. It is essential to establish a technique for reproducibly positioning the probes, such that placement of the probes can be repeated.

Theoretically, placement of the probes should be performed in such a way as to most clearly show what is occurring in the mixing vessel. Because the probes are measuring a very small volume in respect to the total system

volume, probes should be placed at points in the mixing vessel which represent where the mixing will be best, and points where the mixing will be the poorest. These positions in the vessel will be related to the vessel geometry and the flow pattern established (Voncken, Rotte, Houten, 1965). Best and worst locations will vary with changes to the mixing system parameters, and thus probes should be positioned so as to best note changes in the system. Absolute positioning in the best and worst locations is not realistically achievable due to these locations' variability, and approximations of these positions must be used.

Two types of flow patterns are observable while performing flow visualization studies (see Appendix V). These are axial and radial flow. Although it was not possible to create perfect axial or radial flow when performing the studies, the general flow pattern could be established in such a way that it approximated what was desired. Flow patterns were established by using different impellers (flat and pitch (45 deg) blade turbines) at specific C/T values. Flow reversal could be found for a given impeller by varying the C/T value until the flow pattern changed from axial to radial or radial to axial flow. Thus, using a flat blade turbine at a C/T of 0.13 created axial flow, while changing the C/T to 0.33 reversed the flow pattern to a radial one. In the case of the pitch (45 deg) blade turbine, a C/T of 0.33 developed axial flow and a C/T of 0.50 developed radial flow. It is important to consider the possible flow patterns carefully when placing the probes.

In locating the probes for our experiments, the vessel geometry, location of the intake and outlet of the tracer addition system, and the assumption that both axial and radial flow were possible, had to be taken into account. These criteria, coupled with the physical limitations of the probes, led to placement of the four probes in the following manner:

- 1) Probe #3 was placed in front of the baffle which was 290 deg away (in the direction of impeller rotation) from the tracer addition point. It was located close to the wall of the vessel, and 0.28 times the vessel diameter from the bottom. This location was meant to represent an area of good mixing near a baffle which was as far away from the tracer addition point as possible. Closer positioning of the probe to the tracer addition point (i.e., a radial location to the tracer addition point of > 290 deg) would allow the probe to see back-mixing at the time of addition. The back-mixing information would distort information regarding the overall mixing in the vessel.

- 2) Probe #4 was positioned behind the baffle where probe #3 was located, at the same distance from the vessel wall, and the same height from the vessel's bottom. Positioning the probe at this location was meant to represent an area of moderate to poor mixing. It was also meant to show the effect that baffles have on mixing.
- 3) Probe #5 was located approximately 175 deg away from the tracer addition point. It was also located away from all baffles, positioned near the vessel wall, and approximately 0.28 times the vessel diameter away from the bottom. This location was meant to represent an area of good mixing, away from the effects of baffles and positioned such that circulation information (when compared to probe #3) could be obtained.
- 4) Probe #6 was positioned directly above probe #5, near the vessel wall and essentially on the surface. This location represents an area of poor mixing in the vessel. The probe was positioned such that it could be directly compared with probe #5 (an area of good mixing), and such that it would indicate the extreme in regards to the difficulty of mixing in the vessel.

Probe location was determined by the use of a three coordinate measurement system. In this system, each probe was located by its radial location (angle) about the vessel, its distance from the vessel center and its height from the vessel's bottom (see fig. 4). Special measuring tools were constructed to insure accurate placement and replacement of the probes and the tracer addition system's inlet and outlet. The baffles were used as reference points for the radial measurements with the baffles being placed at 90 deg intervals starting at 0 or 360 degrees. The angle increased in the direction of impeller rotation. This system for probe location was quick, simple, and reproducible, and proved accurately repeatable when attempted by a number of different individuals ($\pm 6.4E-3$ m in any direction) (see table 2).

Probe Calibration:

Calibration of the conductivity probes is the next logical step once the geometry of the mixing system has been established. Calibration of the conductivity probes is a relatively simple, although time consuming, process in which probe responses are recorded as the dependent variable against the independent variables of NaCl concentration, pc-board meter reading, and temperature.

The first step in the calibration process is to develop an equation which will relate the pc-board meter reading to the actual voltage of the circuit's output sine wave. This voltage may be observed as the amplitude of the sine wave generated by the oscillator-amplifier part of the circuit. By setting the pc-board meter to read specific values, and using a voltmeter to record amplitude values, a table of data may be generated. A statistical analysis using F-factors can then be used to determine linearity (see fig. 10), and an equation for each pc-board generated. These equations are independent of the probes and should remain constant so long as the circuit components do not change significantly. The equation will have the following form:

$$\text{general form: } SA = (Msa \cdot MR) + Bsa \quad [1]$$

where: SA = signal amplitude, Msa = signal amplitude slope, MR = meter reading, and Bsa = signal amplitude intercept (see table 3).

The next step in the calibration process is to zero the probes in de-ionized water. The actual zero point was specified as +3.5 volts. This was done so that the maximum possible range of response was used for each probe. Meter readings for all pc-boards were set at a value of 50.00. This was thought to be a good average value for the sensitivity range in which the probes would operate. Use of the computer allowed for highly accurate adjustment for probe zeroing (± 0.02 volts).

After the probes were zeroed, standard salt solutions $4.0E-4 \text{ m}^3$ (400 cc) were prepared at three different temperatures. Salt solutions were made with 0.00, 0.02, 0.04, 0.06, 0.08, and 0.10 weight percent NaCl in them. The temperature of these solutions was then regulated to a specific value for the calibration run. The temperatures used were 19, 24, and 29 degrees Celsius. This range of temperatures was felt to cover all possible ambient conditions which might occur in the laboratory environment.

With the salt solutions prepared, all probes being calibrated were placed in one of the thoroughly mixed solutions at the specified temperature. Probe responses were recorded by the computer with the pc-board meter readings being set manually to sequentially different values. An average of 1000 actual points was used to represent the probe response. Meter values were varied from 10 to 160 in steps of 5. The subsequent data obtained could then be analyzed for linearity, and equations generated (see table 4).

Six probes were put through the calibration routine at the six different concentrations and three temperatures. Probes #1 and #2 were homemade probes

which proved less sensitive and less reliable than the commercial probes (#3, #4, #5, and #6). For this reason, the homemade probes were not used in further experimentation, and only the tested and calibrated commercial probes were used. Recalibration of the probes after six weeks of intensive experimentation showed no degradation with less than a 1 percent change in the probe response for probes #3, #4, #5, and #6.

The configuration of probes and pc-boards was also an important consideration. The probe response could vary depending upon the pc-board it was attached to. It had to be determined if a probe would respond with a cleaner, more accurate and more sensitive signal if it were attached to a specific pc-board. Calibration tests were thus run with all the possible configurations of pc-board and probe. The configuration which was selected as the best possible for probe responses, for the commercial probes used, is as follows:

Probe Configuration:

pc-board #	probe #
1	3
2	4
3	5
4	6

With the configuration selected and the data collected, the linear equations for the probe responses could be formulated. Using probe #3 as an example, the following equation was formulated for a 0.04 weight percent solution at 24 deg C.

$$\text{probe response} = -0.273 \cdot \text{meter reading} + 3.84$$

$$\text{degrees of freedom} = 8$$

$$\text{general form: } PR = (Mpr \cdot MR) + Bpr \quad [2]$$

where: PR = probe response, Mpr = probe response slope, MR = meter reading and Bpr = probe response intercept (see table 5).

Because there is a limited range of responses for a given sensitivity, not all values for probe responses were used in formulating the equations. Only those responses which had meaning (i.e., were not outside the range +3.5 to - 10.24 volts) were used. Thus, in the above case, only nine actual responses were used to formulate the equation. This indicates that the maximum useful sensitivity for these conditions corresponds to a maximum meter reading of 50 (10 + 5 · 8) (initial reading + change in reading · degrees of freedom). This also shows the only portion of the responses which fit a linear model (see fig. 11).

Determination of temperature conversion factors was done graphically (see fig. 12). Two conversion factors were obtained (see table 6):

- 1) K_s = change in slope/ $^{\circ}\text{C}$ change in temperature.
- 2) K_i = change in intercept/ $^{\circ}\text{C}$ change in temperature.

These values are necessary if accurate conversion to concentration information is to be obtained from the raw data. Use of temperature conversion factors is done in the following manner:

adding temperature conversion factors to equation [2]

$$PR = M_{pr}[K_s(T - T_{base})] \cdot MR + B_{pr}[K_i(T - T_{base})] \quad [3]$$

where: T = temperature of the experiment ($^{\circ}\text{C}$), T_{base} = base calibration temperature (24°C).

With the calibration information obtained as described, and the equations which were subsequently generated, the concentration can be calculated. This is done by using the following technique:

- 1) A final concentration for a set of experimental runs for the mixing vessel is determined.
 - 2) By a step-wise analysis the meter reading value for the most sensitive pc-board/probe set is determined.
 - 3) The meter reading value determined is converted to voltage based upon the signal amplitude equation [1].
 - 4) Meter reading values for the other probes are backcalculated from the voltage found in step 3 (SA) by use of modified equation [1].
- $$MR = (SA - B_{sa})/M_{sa} \quad [4]$$
- 5) All pc-boards are set to the appropriate meter reading values, and the experiment is run.
 - 6) For conversion to concentration, theoretical probe responses are calculated based upon the voltage (SA) and the temperature at which the experiment was run (T). This is done for all calibration concentrations (i.e., 0.00, 0.02, 0.04, 0.06, 0.08, and 0.10 weight percent).

For any single concentration and any one probe:

Substituting equation [4] into equation [3]:

$$PRT = M_{pr}[K_s(T - T_{base})] \cdot [(SA - B_{sa})/M_{sa}] + B_{pr}[K_i(T - T_{base})] \quad [5]$$

where: PRT = theoretical probe response at a given concentration, voltage (SA) and experimental temperature (T).

7) Once all of the theoretical probe responses for each probe at the six concentrations are calculated, a step-wise analysis is used for conversion of each data point. Each point is fit into the appropriate range between theoretical probe responses and corresponding calibration concentrations, and converted to concentration values. For instance: A point probe response falls into the range between theoretical probe responses 3 and 4.

If this is the case, then the following occurs:

$$m = (\text{calib. conc. 4} - \text{calib. conc. 3}) / (\text{theo. probe response 4} - \text{theo. probe response 3})$$

$$b = \text{calib. conc. 4} - m \cdot \text{theo. probe response 4}$$

and

$$\text{point concentration} = (m \cdot \text{point probe response}) + b$$

For ease of analysis, the starting concentrations for all probes are normalized to a single value. This constitutes a maximum change in the starting concentration curves for any probe of less than 2 percent.

In summary, probe calibration is a time consuming operation that, if done correctly, need not be repeated often. After six weeks of extensive use, no significant change in response of the probes (#3, #4, #5, and #6) was observed. The probe calibration process consists of three distinct parts. These parts are the calibration of the meter reading values for each pc-board, the calibration of the probes for the various concentrations, and the establishment of temperature correction factors for the probes. It is also necessary for experimentation purposes to select and maintain a standard configuration of probe to pc-board. In addition, selection must be made such that operating conditions of final concentration in the mixing vessel and experimental temperature of the mixing fluid are such that they will insure that the calibration conversion equations are usable. In this manner, results are given as concentration. Concentration results are easily comparable to other similar work, and readily understood by members of the mixing community.

Reproducibility And Accuracy

In order to have a successful experiment, the measurement devices should be accurate, reliable, and results should be reproducible. In the case of the conductivity probes, this is doubly important because the measurement technique is not only desired for the specific experiment, but also as a standard method for measuring mixer performance. To this end, the probes' accuracy,

reliability, and ability to reproduce information were severely tested.

Reliability is the ability of a system to perform repeatedly without failure. Prevention of failure is an important aspect to consider in any system that will possibly have an industrial application. The probe system is comprised of three essential components. They are the probe, the probe circuit board, and the wiring connecting them. A failure of any part constitutes a failure of the probe to provide adequate information. Reliability was based upon the 588 experiments run under laboratory conditions throughout the course of the investigation. The rate of failure for probes #3, #4, #5, and #6 are as follows:

Probe #3	5 failures	0.85%
Probe #4	8 failures	1.36%
Probe #5	17 failures	2.89%
Probe #6	3 failures	0.51%
Total	33 failures	5.61%

In any experimental setup there are inevitably going to be failures. The above rates are very low, and well within the realm of acceptability for experimental design (Mendenhall, Scheaffer, 1973). Failures of the probes were generally due to faulty connections between the probe and its accompanying pc-board. No failures were due to the actual probe failing, and only one failure was caused by a circuit malfunction (op-amp burned out).

Accuracy, in experimentation, is the ability to obtain expected or known values. In the blending experiments the known values are the final concentrations of NaCl. These values can be readily calculated by knowing the weight of liquid in the mixing vessel and the amount of NaCl added for each run. The general formula for this calculation is as follows:

$$(S \cdot N / (V + S \cdot N)) \cdot 100 = W$$

where: S = the amount of NaCl/run (1.029 g), N = the number of salt additions, V = weight of the liquid in the mixing vessel and the recirculation loop (10830 g) and W = the weight percent NaCl in the mixing vessel after N additions (see table 7) [there is a maximum 0.7 percent error in the calculations due to volume consideration in the addition loop].

In all, 49 runs were made at each of 7 different concentrations. All probes were used in each run. A correlation matrix shows that only the intended concentration is of consequence when observing end-of-run, probe response concentrations. The variables rpm, impeller type and C/T do not affect final concentrations in the mixing vessel. Variation in response from probe to probe was less than 5 percent, such that all probe responses could be compared to

the expected final concentration by averaging the probe responses first, and then comparing them (see table 7). The percent errors between the observed concentrations and expected concentrations were less than 2 percent except for the case of the first NaCl addition. This error was 3.8 percent, and is attributable to impurities in the addition system at the start of the experiment (see table 7). These low percentage values indicate a higher than average degree of accuracy in the experimental method and the measurement devices.

Reproducibility is the ability to obtain a given result if the experiment is repeated using identical conditions. Reproducibility was tested for the conductivity probes by observing the final concentrations for each run. As in the accuracy tests, 49 runs at each of the 7 concentrations were used to evaluate the probes' response reproducibility. Ranges of responses varied less than 4.5 percent from the calculated sample mean, except for the case of the first NaCl addition where possible impurities in the addition system broadened the range to 11.9 percent. All probe responses fell within a 95 percent confidence interval generated about the sample mean. The combination of a small variation in the range of response and the consistent appearance of probe responses inside of a 95 percent confidence interval are good indications of a reproducible system (Plane, 1977).

As is the case with all experiments, it is important to mention sources of error in the experiments. In the mixing experiments, error sources can be found in the establishment of the liquid level in the mixing vessel, the makeup of the NaCl solutions, the addition of the NaCl solutions, the setting of the impeller rpm, the reading of the voltage output from the probes, and any roundoff errors generated by the computers' manipulation of the data. An additional source of error is in the probe calibration curves, the temperature readings, and the assumptions used concerning calculations when evaluating probe responses. Obviously, human involvement in the placing of probes, filling of the mixing vessel, makeup of the NaCl solutions, and establishment of appropriate rpm values are all error sources. However, it is important to note, that whenever possible, automation was used to reduce error, and the statistical analysis substantiates the success of this practice.

In summary, the use of automation significantly reduced error sources to the point where the experiments proved to be very reproducible with highly improved accuracy. A sound, well-thought-out experimental design, using tested hardware

and solid state electronics, provided probe responses which were highly reliable. Some error sources exist, but these have been kept to a minimum, and results show them not to have a significant effect on the information obtained.

PROCEDURE

The procedure for the experimental runs consists of three parts. They are the preparation of the salt solution, the preparation of the equipment, and the actual running of the experiment. Much of an experimental run is controlled by the computer, but operator responses to computer prompts are also necessary. The following paragraphs will describe the three parts of the procedure.

The salt solution is prepared by first determining the final desired concentration in the mixing vessel. For our experiments this value was 0.06 weight percent. This value was determined to be optimum for probe responses throughout a set of experimental runs. The final concentration in the mixing vessel should not exceed 0.10 weight percent because calibration data was not taken beyond this level. The next step is to decide the desired number of salt additions for the experiment (7 salt additions were used, one addition/run). Now, the weight of salt and the weight of water in each injection must be determined. This is done by solving two equations in two unknowns. The equations are as follows:

$$(1) \quad (S \cdot 7)/(V + (S \cdot 7)) = 0.0006$$

$$(2) \quad S + W = CV \cdot \text{Den}$$

where:

S = weight of salt in the calibration volume in grams,

W = weight of water in the calibration volume in grams,

V = weight of water in the mixing vessel in grams,

CV = Calibration volume in cm³. (10.7 cm³) (0.0107E-3 m³),

Den = salt solution density, and

7 is the number of additions to be made.

Once values for S and W have been determined, a salt solution should be made with 20 times the volume of that which you plan to add to the mixing vessel. This is because there is waste when flushing and refilling the calibration loop.

The preparation of the equipment consists of the following steps:
(see fig. 9 for addition system information)

- 1) Turn the probe circuits on. They must be on for at least one (1) hour prior to the running of an experiment in order to reach an equilibrium state.
- 2) Turn on the oscilloscope, multiprogrammer, salt addition system and weigh scale at least ten (10) minutes prior to the running of an experiment.
- 3) Zero the weigh scale.
- 4) Open the air valves for the salt addition system.
- 5) Make sure that the inlet to the salt-side of the addition system is immersed in the prepared salt solution. The outlet should be positioned over an empty beaker.
- 6) Fill the mixing tank to the indicated level such that the appropriate Z/T ratio is established (usually $Z/T = 1.0$).
- 7) Prepare the salt addition system. Put the computer/manual switch into the 'manual' position. Turn the salt side pump (#1) on with the calibration loop closed. After 15 seconds, operate the four-way valve (#1) and open the calibration loop. Run in this position for 15 seconds, and then close valve #1, isolating the calibration loop with salt solution in it. Continue to run for 15 seconds more before turning off the salt side pump (#1). Return the switch to the 'computer' position.
- 8) Load the computer with the experimental program. This completes preparation for the experiment.

To run the experiment, the following are done:

- 1) Run the experimental program. It will ask you to answer certain questions, and perform certain tasks.
- 2) After prompt: Input the time and date.
- 3) After prompt: Input which mixing vessel is being used.
- 4) After prompt: Input the number of times salt solution will be added before changing the mixing vessel liquid. This values should correspond with that used to make the salt solution (i.e., 7).
- 5) After prompt: Input the experimental run's name. The nomenclature for this is a letter (R-Z), followed by the date (month then day), followed by the salt addition number. [example: first salt addition on May 12 = R5121] You enter only the first letter and date, the computer inputs the salt addition number.

- 6) After prompt: Input the temperature of the liquid in the mixing vessel (degrees C).
- 7) After prompt: Input the incremental amount of salt (S) in grams to be added in each run (1.029 g).
- 8) At this point, the computer will ask you to 'zero' all probes to a value of +3.5 volts with the probe meter set at 50.00 for all probes. The probes should be in deionized water appropriately positioned in the mixing vessel. The value of +3.5 is selected to give the probe resistivity the maximum possible range for response (+3.5 to -10.24 volts). The meter reading of 50.00 is an average value which approximates the possible values which might be used for an experiment. The meter reading is directly proportional to the amplitude of the circuit carrier wave for the probe signals. Fifty is also the value at which the probes were calibrated, and is therefore an essential zeroing point if the conversion routines are to be used.
- 9) After the completion of step 8, the computer will calculate what meter values should be used for all probes to insure maximum sensitivity for the experiment. These meter values are based upon the final mixing vessel concentration. Each probe must be adjusted to have the appropriate meter value. This meter value is different for each probe because each probe is slightly different, as is each probe circuit.
- 10) After prompt: Input any desired changes to the default values given for the geometric parameters (see fig. 13).
- 11) After prompt: Input the number of data points desired (1000 points were taken in our experiments), and the run time in seconds. The run time must be based upon the rpm value chosen for the experiment (i.e., slower rpm values need longer run times {50 rpm - 200 s, 150 rpm - 75 s, 300 rpm - 30 s}).
- 12) After prompt: Input the rotation speed (rpm).
- 13) All the preliminary information has now been entered, and the experimental run is ready to begin. The computer will tell you it is ready to take the 'tare torque' measurement, and ask that the mixer be turned on at zero rpm. This measurement is used to establish a base point from which the power requirement for mixing can be calculated.

- 14) When the 'tare torque' measurement is complete (approximately 10 s), the computer will ask you to set the mixer to the experimental rpm value which you previously specified. It will then ask you to perform several visual checks on the equipment to insure that everything is in order to proceed. Once you verify that the experiment is indeed ready to run, the computer will start the experimental run.
- 15) The computer runs the experiment in the following manner:
 - a) The addition system is activated, and all valves are placed in their start positions. The salt-side pump in the addition system is started, and the salt solution flushes and then is trapped in the calibrated loop. The salt-side pump is then turned off.
 - b) The mixing vessel pump is turned on and allowed to run for 10 seconds until its effect on the flow patterns in the mixing vessel, if any, have reached equilibrium.
 - c) The data collection system (i.e., multiprogrammer) is activated, and data collection is started based upon the number of points desired and the total run time for the experiment.
 - d) Standard conditions data is collected for 1/10 of the total run time. After which time the calibration loop, with the salt solution in it, is flushed into the mixing vessel with fluid from the mixing vessel.
 - e) At the end of the total run time, data collection stops and the addition system is deactivated with all valves being placed in the appropriate positions, and all pumps being turned off.
- 16) After the experiment is completed, the data is first converted to all positive numbers (for ease of calculations), and then displayed for visual inspection. Display consists of a summary of information regarding the experimental setup, and a graphical representation of the experimental run (see fig. 14).
- 17) If there are no problems found with the visual inspection, then the computer will store the resistivity, or raw data, on a floppy disk. The information thus stored may be retrieved at a later date for conversion to concentration data and subsequent analysis.

- 18) The computer now allows for conversion to concentration data. This conversion is based upon calibration data for each probe and takes into account the amplitude of the circuit's carrier wave, the expected final salt concentration, and the temperature in the mixing vessel at the time of the run. It will also allow you to store the concentration data if you desire.
- 19) At this point, one complete run has been made. The computer will return to the start of the program and automatically set values for another salt addition. No rezeroing of the probes should be necessary. The experiment will be run as many times as you specified salt additions, each time following the steps (6-18 excluding steps 8 and 9) as described above.
- 20) After all runs have been made, the mixing vessel should be emptied and refilled with deionized water such that all probes are completely covered. The addition system should also be flushed of all salt solution and filled with deionized water. These precautions are designed to minimize contamination, minimize equipment failure, and promote the highest degree of reproducibility and reliability in the experiments.

PROBE RESPONSE

The objective of these experiments was to select a measurement technique and then prove its viability as a means of observing the mixing process. To this end, conductivity probes were selected for use in simple blending experiments. The conductivity probes were tested for reliability, reproducibility, and accuracy and found satisfactory. Observation of the probe responses can tell much about the mixing process and how such variables as probe noise, probe location, flow pattern, impeller type, C/T, rpm, and concentration relate to the mixing process.

The data displayed in the graphs (see Appendix X) is an average probe response obtained from seven identical experimental runs. The time of injection precedes the first noticeable change in probe #5 by approximately 5 seconds. Responses are initially normalized to a specific starting concentration, and then left alone to show each individual probe's response. Each probe response curve consists of 1000 individual data points, each individually averaged. All probe responses for a given experiment were taken simultaneously so that, should it be desired, a point by point comparison of probe responses is possible. Total experimental run times were predetermined and

kept constant for each of the seven experiments. Seven experiments were run to insure that the average value would be within the 95 percent confidence interval of the true average. The actual needed number of experiments is less than seven based upon a desired standard deviation of the mean of 0.0001 weight percent, and an expected standard deviation of approximately 0.0015 weight percent. The actual standard deviation proved to be less than expected and, as such, the experimental design of seven identical experiments was more than adequate.

Signal Noise and Probe Response Time:

It has been shown that probe responses when related to final concentrations in the mixing vessel following an experimental run, are accurate, reliable, and reproducible. It now remains to examine the ability of the probe to model the dynamic system of the mixing vessel during an experiment. To this end, it is important to first investigate the signal noise and the probe response time.

Probe response time is the measure of how quickly the probe system records a change in concentration. A simple experiment which plunges the probe into a stagnant salt solution and triggers a timer was used to determine a response time. This time must be of a sufficiently short order that changes caused by the dynamics of the mixing system are observed. Although extensive tests were not made to determine probe response time, experimentation shows that the probe response time is approximately 0.0005 seconds or less. This is the time between when an event occurred and when the probe showed the event occurring. For the experiments run, the fastest responses needed were 0.03 seconds. Experiments run to test diffusion rates for the probes showed times in excess of two hours. Therefore, it was felt that the probe response time was sufficiently fast enough to show the events or dynamics occurring within the mixing vessel. This is easily observable in the data which was taken for the experiments (see Appendix X).

Noise in the system can come from several sources. It can come from the experimental apparatus itself, the probe circuits, or the data collection setup. It is important to determine the effect that noise has on the experiment and the data taken if any. Observable noise was recorded both before and after the experiment. The noise level was found to be less than 5 percent of the signal in approximately 97 percent of the cases. This is sufficiently low, such that experimental data taken is not significantly

affected by noise (Mendenhall, Scheaffer, 1973). Several trends were noticeable in the noise with regards to the independent variables used in the experiments. Noise was reduced with increased rotation rates. Signal noise was increased slightly with increased concentrations. The pitch blade turbine experiments had higher noise levels on the average than did the flat blade turbine experiments. And in the case of the flat blade turbine, increasing the C/T reduced the experimental noise level (see Appendix X for an example of noise data).

In summary, the probe response time is easily fast enough for system dynamics to be observed. The overall noise level is sufficiently low, such that it does not affect the experimental results taken. Signal noise is affected by experimental parameters, especially impeller type, concentration and rpm. Further work is needed to verify these results, but time and resources were not available. The observed noise in the experiment is such that it tends to average out to zero. That is, there is as much noise on the positive side of the response curve as there is on the negative side. This can readily be observed in the accuracy of the probe responses when relating final concentrations to expected results.

Probe Location:

The location of the probes is such that if the probes' response truly models the dynamics of the experiment, then differences between probe responses should be observable throughout the experimental runs. This is indeed the case, as can readily be seen in fig. 15. Although the probe response curves begin at the same concentration, and end at the same concentration, the path taken between these concentrations varies according to the probe location (see fig. 15a (1-4)). Thus, probe #5 sees the injection first, when it is most concentrated, and the resulting peak observed is the highest. Probes #3 and #4 are close together and thus have similar curves. However, a noticeable lag is seen in probe #4 because it is behind the baffle that probe #3 is in front of. Probe #6 shows only a slow rise to the final concentration because it is the surface probe and mixing at that point is relatively poor. In summary, the probe location affects the probe response as a function of time, but not the final concentration. Probe location is a very important consideration in the experimental design.

Flow Patterns:

A closer examination of the probe response curves gives information about the flow patterns in the mixing vessel. For example, the response curve for probe #5 shows two distinct peaks approximately 16 seconds apart (see fig. 15). This 16 seconds is a measure of the circulation rate of fluid at the vessel wall. A comparison of probes #3 and #4 shows the effect of a baffle in the flow pattern. Probe #4 lags probe #3 responses because of the presence of the baffle. A comparison of probes #5 and #6 shows areas of theoretically good mixing and theoretically poor mixing respectively.

A comparison of response curves for different sets of parameters for the mixing system can point out the effects of flow patterns on mixing (see figs. 15 and 16). In these cases, only the C/T value was changed. Notice that under the conditions of the experiment which the second graph represents (see fig. 16), the probe response for probe #6 is quite different from the probe response in fig. 15. The difference is an indication that much better mixing is occurring at the surface of the fluid in the second case. The effect of the baffle (comparison of probes #3 and #4) is not so clear for these two examples. The lag is clearly defined in fig. 15, but not in fig. 16. The response of probe #5, its initial peak, is less sharply defined in fig. 16, indicating that the flow pattern at that point in the mixing vessel is not as well defined or is more turbulent than it was in the first case (fig. 15).

Special events can on occasion be observed in the data presented, as is the case for a pitch blade impeller rotating at 50 rpm with a C/T of 0.33 (see fig. 17). In this case, a small peak can be seen in probe #4's response at approximately 10 seconds into the run. This peak shows back-mixing which occurred at the time of injection. Back-mixing is a momentary flow in the opposite direction of the impeller rotation. Back-mixing was originally detected by visual observation during the experiment. On the whole, however, it must be noted that these probes were never intended for observing micro-mixing, and as such are inefficient in doing so. The intent of the experiments was to show that the probes could pick up basic trends in mixing and discern flow reversal (i.e., a change in flow pattern from axial to radial flow).

In summary, by observing the individual probe response curves and comparing them to each other, it is entirely possible to gain an insight into the mixing process. (use of multiple probes is a drawback for industrial applications.) Effects of baffles, areas of good and poor mixing, and circulation rates may all be observed. Although the probes are not meant to

measure micro-mixing, such disturbances as back-mixing at the time of injection may be spotted. In addition, the comparison of separate experiments can demonstrate the effects of different flow patterns on the probe responses, and thus the mixing process.

Impeller Types:

Comparisons of the data for different experimental runs may also be used to observe differences caused by the use of various impeller types. Only two impeller types were used in the experiments; one being a flat blade turbine, and the other being a pitch (45 deg) blade turbine. Under identical conditions of rpm, C/T and concentration, all probes showed observable differences when different impellers were used (see fig. 18). The flat blade turbine showed poorer mixing of the surface of the mixing fluid (probe #6) than did the pitch blade turbine. The flat blade turbine also showed a more pronounced baffle effect (comparison of probes #3 and #4), and sharper peaks for probes #3, #4, and #5. Peak maximum values and circulation rates are essentially the same for both the flat and pitch blade turbines. From these observations it can be concluded that when the impeller type is allowed to change, and all other conditions of the mixing experiment are being held constant, an observable difference is seen in the probe responses.

C/T Values:

Not unlike the impeller type, changes in C/T can also be observed by looking at the probe response curves (see fig. 19). The most apparent difference is probe #6, which shows the effect of changing the C/T on surface mixing. Probe #5 shows a false peak, and less clearly defined peaks and valleys in the case where the C/T value is low (0.13). The higher C/T value (0.33) shows a much better delineation between probes #3 and #4, whose comparison can be related to the effect of the baffle between them. Therefore, it is valid to say that C/T variations do affect the appearance of the probe response curves.

RPM:

Changing the rotation rate of the impeller changes the probe response curves in two different ways (see fig. 20). First, the length of time in which the mixing experiment runs varies inversely with the rpm. That is, as the rpm increases the length of time needed for the mixing experiment to occur

decreases. The second observable change is in the probe response curves themselves. Changes in probe #6 (the surface probe) are most apparent, with mixing improving as the rpm increases, until at 300 rpm, the response curve for probe #6 looks fairly much like those of probes #3, #4, and #5. The probe response for probe #5 becomes less sharply defined with a decreasing maximum first peak value as the rpm increases. This indicates a quicker dispersion of the tracer into the mixing system at higher rpm values. Probes #3 and #4 also show a reduction in maximum first peak values as the rpm increases. They also show an increased separation of peaks as the rpm increases, indicating that baffles have an increased effect at higher rpm values.

In summary, changing the rotation rate through three distinct levels (i.e., 50, 150, and 300 rpm) produces noticeable changes in the probe response curves for all probes with differences in response being greatest for probe #6, but changes being quite observable for probes #3, #4, and #5. Increases in the rotation rate also decrease the time needed to run a mixing experiment and have some affect on the flow pattern.

Concentration:

Another variable which must be considered regarding its effect on probe responses is the average concentration during an experimental run. As has been previously discussed, the concentration in the mixing vessel at the time of an experimental run affects the calibration of the probes and subsequent conversion of raw data into concentration data. It also affects the noise level in the experiment. The only other observable effect of concentration levels in the mixing vessel on probe responses is a quantitative change in the magnitude of the response. That is, as the average concentration in the mixing vessel increases, the probe responses increase proportionally.

MIXTIME ANALYSIS

It has been shown through our experimentation that the dynamics of the mixing system are observable through investigation of probe responses. The information obtained is of a qualitative nature, and is meant only to point out what the probes can and cannot 'see' in the mixing system. A quantitative analysis of the data is useful for demonstrating what information can be gained through the use of the measurement technique and/or the experimental apparatus. A quantitative analysis can be performed using mixtime as a dependent variable, and considering some of the independent variables that affect the mixtime such as the probe location, flow pattern, impeller type, C/T, rpm, power, and concentration (see table 8).

Determination of Mixtime:

Determination of the mixtime as the dependent variable is a very important part of the analysis of the data. There are many techniques used to determine mixtime, and unfortunately most of these are highly qualitative in nature. An effort was made to make the mixtime estimates as accurately and reproducibly as possible. A qualitative, graphical technique was used in this study to determine mixtimes because work on a strictly quantitative technique is beyond the scope of the work for this project. Possible quantitative techniques are discussed in a later section of this report.

The graphical technique which was used selected a mixtime within a range of approximately 10 seconds. This range was found by looking at the probe response curves for each probe and estimating visually what the mixtime was likely to be. The mixtime is the point in time at which the response curve ceases to change with respect to concentration. After the range was determined, this area of the graph was expanded, and the final estimate of the mixtime was obtained by visual observation (see fig. 21). Use of this graphical technique provided mixtimes which were consistent with one another to within ± 5 percent in 92 percent of all the experimental cases. This consistency only held for probes #3, #4, and #5, with probe #6 being an exception. Probe #6 mixtime variations were not solely a function of the technique used but, rather, a function of the experiments themselves (i.e., in some experiments, mixing was never completed for probe #6). Overall, it was possible to reproduce mixtimes to within ± 10 percent for all of the experiments run (see Appendix VI).

In summary, the mixtime was determined graphically by visual observation for each probe. The values obtained were consistent to within ± 5 percent in most cases, excluding probe #6. Probe #6 mixtime determinations varied considerably due to the experiments and not the technique for determination. Mixtime determination was reproducible for all probes to within ± 10 percent for all experimental runs. A strictly controlled technique of qualitative mixtime determination provides adequate information, and allows for further analysis of the effect of various independent variables on the mixtime.

Probe Location:

As has been previously noted through observation of the probe response curves, probe location is an important factor in the information received for an experiment. It is quite apparent that discernable mixing equilibrium is not completed at all locations in the tank simultaneously but varies with location in the vessel.

Because of this, probe location can be seen to have a strong effect on mixtime. Much can be learned concerning the flow patterns and areas of both good and poor mixing by a comparison of the mixtimes for various probes.

A comparison of mixtimes for probes #3 and #4 shows the effect of a baffle by measuring the mixtimes (time to uniformity) for a location in front of and behind a baffle respectively. Mixtimes for probe #4 (behind the baffle) lagged those of probe #3 by approximately 5 percent in 89 percent of the applicable cases. Cases which were not applicable were those where $C/T = 0.13$ and the flat blade turbine was used. This combination of C/T and impeller type produced a condition of extreme axial flow, which was observed in the flow visualization experiments. (Appendix V explains flow visualization.) Under extreme conditions of axial flow, probe #4 did not lag probe #3 significantly. Baffle effects were apparently removed, at the level from the tank bottom of probes #3 and #4, for rotation rates in the range of 50 to 300 rpm when extreme axial flow conditions existed.

A comparison of probe #5 and probe #3 mixtimes shows probe #5 lagging probe #3 in 67 percent of all cases. In cases where the flow pattern was axial, 67 percent of the time probe #5 did not lag probe #3 irrespective of impeller type used or rotation rate. From this information it can be concluded that turbulence caused by a baffle promote better local mixing in the area in front of the baffle. The effectiveness of the baffle is reduced under axial flow conditions, and circulation rates become less important than flow patterns when extreme axial flow is present.

Examination of probe #6 mixtimes is very useful in gaining insight about the mixing process. A comparison of probe #6 mixtimes to mixtimes for probes #3, #4, and #5 shows probe #6 mixtimes lagging all other probe mixtimes significantly in 75 percent of all cases, and 100 percent of the cases where radial flow was not involved. It can be concluded, based upon mixtimes, that under most conditions of rpm, impeller type and C/T , surface mixing is not as good as the mixing at probe locations elsewhere in the mixing vessel.

In summary, probe location has a strong effect on the measured mixtime. The effectiveness of a baffle can be determined by comparison of mixtimes for probes in front of and behind a baffle. Baffle effects are minimized under conditions of extreme axial flow. Circulation rates become less important than flow patterns when determining mixtimes under conditions of extreme axial flow. Surface mixing is generally poorer than mixing elsewhere in the tank, with the exception of extreme radial flow in which surface mixtimes compare well with mixtimes at other locations in the mixing vessel.

Flow Patterns:

Further investigation of probe #6 mixtimes can provide insight and information concerning flow patterns in the mixing system. Flow reversal can be inferred for some cases based upon probe #6 responses. This is because both radial and axial flow conditions may be observed by using the surface probe. With only the C/T varying to create conditions of either axial or radial flow, and all else being held constant, mixtimes changed thusly:

TABLE A
PROBE #6 MIXTIMES IN SECONDS

impeller flow	flat		pitch	
	axial	radial	axial	radial
C/T	0.13	0.33	0.33	0.50
rpm	mixtimes			
50	172	174	122	82
150	42	44	54	30
300	21.6	20.3	16.4	15.5

Flow visualization experiments were used to determine impeller positions in which either radial or axial flow conditions existed. For the pitch (45 deg) blade turbine, axial flow was observed at a C/T of 0.33, and radial flow was observed at a C/T of 0.50. For the flat blade turbine, C/T values of 0.13 and 0.33 created conditions of axial and radial flow respectively. Insufficient energy was put into the flat blade turbine system until the 300 rpm level for a significant difference in mixtimes between axial and radial flow to be observed at the probe #6 location. Radial and axial flow differences were easily observable from probe #6 mixtimes for the pitch blade turbine at all rpm values tested (see table A).

Impeller Types:

Useful information was also gained concerning the effectiveness of one type of impeller over another. A comparison of probe #6 mixtimes with all system parameters except impeller type held constant was made (see table A).

The C/T value was kept at 0.33 and the flat and pitch blade turbine experimental runs were observed. The flat blade turbine at a C/T of 0.33 produces a radial flow pattern, and has shorter mixtimes at probe #6 than does the pitch blade turbine when the rotation rate is 150 rpm. When the rotation rate is 50 rpm, mixtimes are shorter for the flat blade turbine for probes #3, #4, and #5, but longer for probe #6 (see Appendix VI). At the 300 rpm rate, the difference between the flat and pitch blade turbines is slight, with the pitch blade mixtime being slightly less. This is possibly due to the fact that with a C/T of 0.33 and the pitch blade turbine, the flow pattern is axial not radial.

In summary, with a C/T of 0.33 and rotation rates of 50 and 150 rpm, the flat blade turbine produces shorter mixtimes than does the pitch blade turbine for all probes with the exception of probe #6 at 50 rpm. At the higher rate of 300 rpm, the difference in mixtimes is less well-defined with the axial flow pattern of the pitch blade impeller giving a slightly shorter mixtime for probe #6. It is important to note that power levels differ depending upon which impeller is used.

C/T Values:

As has been previously stated and observed, with all else held constant, a change in the C/T can produce a change in the flow pattern. Probe #6 is most strongly affected by impeller location changes because of its position on the surface of the mixing fluid. Probes #3, #4, and #5 are affected to a much lesser extent due to their close proximity to the impeller regardless of the C/T value chosen, provided this C/T value is between 0 and 0.50. A change in C/T may be used to change the flow pattern from radial to axial or axial to radial. The other technique for changing the flow pattern is to use a different type of impeller.

RPM:

Mixtime as a dependent variable is most strongly affected by the rotation rate of the impeller. The rotation rate is directly related to the energy input into the system. If the energy to the system is increased with all else held constant, it is reasonable to assume that up to a point, the mixtimes in the vessel will be correspondingly shortened. This indeed can be seen in the mixtimes for all probes, but especially for probe #6 (see table A). Probe #6, as the probe on the surface, is in the position where the mixing is poorest (considering all probe locations used) and, as such, changes at this location are more dramatic and thus more easily observable than elsewhere. It can also

be seen that, at low rpm values, the rotation rates effect on mixtime is strongly influenced by the type of impeller used.

Concentration:

The last variable for consideration in its effect on mixtimes is the average concentration in the mixing vessel during an experimental run. Analysis of mixtimes as a function of concentration show that there is no significant dependence on concentration in determining mixtimes (see fig. 22). Concentrations affect the probe response curves in the manner previously stated, but in no way affect the mixtimes. This is a wholly reasonable conclusion as no reaction is occurring within the vessel, and physical parameters of the system should indeed have the greatest influence on mixtimes.

It must be understood that not all possible parameters were investigated for their effect on mixtime. Also, those parameters tested were only evaluated under a very small set of specific conditions. The intent was to show that probe response curves could be analyzed for mixtimes, and these mixtimes could in turn be analyzed to determine the effect of various independent variables on mixtime. From this analysis, conclusions could be drawn regarding the mixing process. The methodology used for experimentation and the technique used for analysis did positively show that information gained in this fashion is appropriate for enhanced engineering design, trouble shooting, and general understanding of the mixing process.

TORQUE MEASUREMENTS

In mixing experiments it is important to measure the torque or, more accurately, the power input into the system. Measuring the power input is important for two reasons. First, any design of batch mixing equipment must take into account the operational costs of running the equipment. Trying to develop the most efficient design that fully performs the desired work demands knowledge of the energy requirements for the system. Second, the use of power, or of the power number^{*}, to develop correlations has become one of the few standards about which members of the mixing community can communicate. Thus, torque measurements, as a measure of power^{**}, are taken so that the

^{*} Power number: $N_p = P \cdot g / (p \cdot N^3 \cdot D^5)$ where: N_p = power number, P = power, g = gravity constant, p = liquid density, N = impeller rotation speed, D = impeller diameter (Oldshue, 1983).

^{**} Torque to power: $H_p = [(\text{torque (in-lbs)}) \cdot (\text{rpm})] / 63000$
where: H_p = horsepower (Chemineer-Kenics, 1981)

ability to create practical, energy-efficient designs is possible, and information gained through experimentation may be properly communicated to interested parties in the mixing community.

Power measurement in a batch mixing system is done in essentially five different ways. First, a wattmeter may be used to measure the current and voltage (power being derived from this information) for the mixing motor. The problem with this technique is that readings at low levels are difficult, and system losses such as the bearings and gearbox are not accounted for. The advantage of a wattmeter is that it is portable and may readily be used at an on-site facility.

A second method for determining the power is done by using a weigh scale (micro-balance) and a torque arm. In this method, a weight is hung from a torque arm on the motor, with the motor mounted and balanced on a "frictionless" bearing. Variations in the weight, as accurately read by the electronic weigh scale are easily interpreted as torque values. These values are then converted into power by knowing the rpm value for the motor (see fig. 7). The advantage of this system is that small torque values are easily obtained, the accuracy is high (within 7.77 percent for the flat blade turbine, and 5.25 percent for the pitch (45 deg) blade turbine), gearbox losses are accounted for, and the equipment for taking the measurements is simple and relatively trouble free. The disadvantage is that the setup is impractical for field use, and sensitivity is limited by the ability to choose the correct hanging weight.

Another technique for measuring torque is the use of a hollow torque ring. In this case, the motor is mounted to a hollow ring which in turn is mounted on a rigid structure. The impeller shaft runs through the center of the ring, and torque is calculated by using a strain gauge to measure the torsional deformation in the ring. This technique gives accurate responses (approximately ± 5 percent as stated by manufacturers), and is usable on fairly large size units. However, the range for any torque ring is small, so that careful sizing is necessary if realistic results are to be obtained. Use of a torque ring in the field would be difficult but not impossible.

A fourth technique for measuring power is the use of strain gauges mounted on the impeller shaft. Both torsional and vertical deformation of the shaft are measured, with torque values being calculated from this information. This

is a generally accepted technique for measuring torque. This method does remove errors caused by bearing and gearbox losses. The difficulty with shaft mounted strain gauges is that the reading must be taken off a rotating object, the range for any one strain gauge is small, and calibration is difficult.

The final method used to measure torque is one in which the mixing vessel or the mixer drive is mounted on a flat plate. The plate is in turn mounted on a specially built cylinder attached to a rigid structure. Strain gauges measure the torsional deformation of the cylinder as the top plate rotates relative to the rigid structure. Torque values are then calculated from this information. This technique gives very accurate information (within 5.98 percent of the expected value for the flat blade turbine, and 5.29 percent for the pitch (45 deg) blade turbine) under a limited set of conditions. The values obtained approximate more closely the actual energy put into mixing than most other systems for torque measurement (i.e., taking into account bearing and gearbox losses). However, a particular plate device will only work for limited weights and tank sizes, and the absolute largest size is still small on an industrial scale. The setup would also be difficult to use in the field as most mixing vessels are secured to fixed structures, and most vessels are not specially balanced about the center of the vessel.

Because the work being performed was of a laboratory nature, it was felt that accuracy of reading, ease of operation, and reliability of the equipment were of prime importance. For this reason, two methods for measuring the torque were used. First, the weigh scale approach was used because of its reliability (only 6 failures in 588 uses) and ease of use. Second, a torque plate was installed because of its ability to obtain accurate information. Calibration of the torque plate is easy, and the torque plate and the weigh scale method may both be used at the same time. Thus comparison of techniques is possible. The most realistic method for measuring torque in the field is probably the wattmeter. However, testing of wattmeters on laboratory scale equipment is inappropriate. It was planned to test wattmeters on the larger scale 0.379-0.757 m³ (100-200 gallon) facility when it was built.

Testing of the two types of torque measurement devices was performed with both flat and pitch (45 deg) blade turbines at different C/T values (0.13-0.50) and three different rotation rates (50, 150, and 300 rpm) (see Appendix VII). The change in power at different C/T values was minimal (< 2.5 percent) for both the torque plate and the weigh scale techniques (see fig. 23). An initial

comparison of the theoretical (given by manufacturer), torque plate and weigh scale horsepowers versus rpm showed that the weigh scale values were off by a factor of ten (see fig. 24). Examination of the calculations used to determine the horsepower from the weigh scale readings showed that the calculations had been set up for a 2000 gram hanging weight, when in fact a 200 gram hanging weight had actually been used. A replot of the horsepower versus rpm for the theoretical, torque plate and weigh scale values, after correcting the weigh scale horsepower, shows that the torque plate and weigh scale readings are within 4 percent of each other, and both are approximately 17 percent lower than the theoretical values (see fig. 25 and fig. 26). This 17 percent difference corresponds to the approximate expected loss in the bearings and gearbox. An additional plot of the power versus rpm shows that at any given rpm, the flat blade turbine requires more power input than does the pitch (45 deg) blade turbine (see fig. 27 and fig. 28). Experimental data proved to be highly linear, so only the linear plots through the data points are shown.

In summary, two different methods were investigated to determine the torque in a given experiment. One method used a torque arm attached to the mixing motor, with a hanging weight attached to the torque arm and positioned on a weigh scale. The other method used a torque plate centered beneath the mixing vessel, with information being obtained from strain gauges. Both techniques proved accurate and reproducible over the range of rpm values (50-300 rpm), C/T values (0.13-0.5) and impeller types (flat and pitch (45 deg) blade turbines) used in the experiments. Variation between methods of torque measurement were less than 4 percent. Variation between these values and those theoretical values provided by the manufacturer were on the order of 17 percent. This latter difference being attributable to bearing and gearbox losses.

FUTURE WORK

As is the case with most research work, there is always more to be done. Mixing research is no exception. Although the experiments run were very useful and provided a great deal of information, they only scratched the surface of the mixing field. Mixing is an extremely complicated subject with a great deal of diversity. To properly study even a small aspect of mixing demands that a significant commitment be made both in terms of time and money. The approach for the work done here was different from similar work done elsewhere because the desired result was a methodology and analysis technique, instead of a

correlation or body of data. This work is useful and should be continued with emphasis being put on improving the existing experiments, doing further experimentation, and developing better analysis techniques.

Experimentation:

The experimental apparatus for the set of experiments run was designed for flexibility, reliability, and reproducibility. Experiments showed that the equipment met all these criteria. Further work needs to be done in the calibration of the addition loop, the torque measurement technique, and the makeup of NaCl solutions. Most of the work that needs to be done is in carrying out additional experiments and data analysis.

The experiments that have been run are useful but not complete. Further experiments need to be run for all process variables. Different types of impellers with different numbers of blades should be tested. A greater spectrum of rotation rates should also be investigated. Impeller positions should be changed at smaller intervals to gain greater insight into flow pattern generation. Probe locations should be varied to prove whether or not the selected probe locations truly give the best representation of the mixing system. The tracer injection point should also be changed to note its effect on the response curves. Alternate concentrations should be tried to determine if the probes respond well outside of the tested range.

Additional experiments should be run at different viscosities, using different materials for mixing with the same basic parameter set. Experimentation should also be performed in different sized vessels to determine the difficulties in data collection and analysis caused by changes of scale. After a sufficient amount of information has been obtained for miscible liquid blending, immiscible liquid mixing should be investigated. Other mixing systems of liquid-liquid, liquid-solid, and gas-liquid should also be investigated. Further work will definitely include the use of probes other than conductivity probes, and may include systems where chemical reactions and changes in viscosity and temperature occur.

It is of course important to remember that the mixing field is very large, and a research program must be carefully planned and organized if useful information is to be obtained, and the project may be accomplished in a reasonable amount of time.

A reasonable step forward for this project would be to build a moderate size 0.379-0.757 m³ (100-200 gallon) facility, perform the identical

experiments in the larger facility that have been performed in the laboratory scale facility, and expand the experiments in the laboratory scale equipment to include a wider variation of the parameters of probe location, impeller type, C/T and rpm. Additional variables that should be looked at are Z/T, D/T, baffles, and viscosities. A new set of experiments should be designed for the solid-liquid mixing system. All experiments run on the laboratory scale should later be run on the moderate size facility, with some testing being done at plant sites with the large scale equipment that exists there.

Data Analysis:

The data generated in the mixing experiments is typical of data generated in many different kinds of experiments. The data is in the general form of a decaying sine wave but displays extensive non-linear type behavior. The analysis of this type of data is difficult at best. For truly useful information to be obtained from the mixing experiments, it will be necessary to devise a much more quantitative method for analysis than the visual observation of graphical data. Throughout the course of experimentation, many different techniques have been tried to analyze the data. Most of these techniques were designed specifically to calculate a mixtime. Straight observation of the data in a graphical format is one such method. Fitting the peak points of the curves to a decaying exponential and then calculating the rate of decay is another. Looking for deviation from the final concentration value is another alternative, and is possible in several different ways. An integration to determine the rate of change of the area under the curve is also a possibility. In all, many methods were attempted, and the simple graphical technique proved most reproducible, even though it is more qualitative than the others. Strange perturbations in the data as well as the necessity to make qualifying assumptions, detract from the other techniques tried. More work is needed in developing a quantitative system of analysis for this type of data. To this end, some work has been started.

One possible technique for analyzing the data is to use a strictly statistical analysis technique. In this technique, one would, as before, obtain a set of average probe response curves in the form of time versus concentration for each probe. Each individual probe response curve would then be looked at on a point by point basis. A sum of the standard deviation squared (SSD) would be calculated for the curve and then normalized to a value of one (1), such that at each point in time along the curve a fraction of the total SSD

would be represented. The resultant point for point statistical information can then be plotted against time (see fig. 29). Observation of the slope of the curve generated, as well as perturbations in the curve, should give much insight into the mixing system. Mixtimes would be calculated by starting at the end of the experiment and backtracking the slope value until it varied significantly for a statistically significant number of points. The use of this technique sounds simple, but there are assumptions to be made regarding the noise in the experiment, the effect that concentration has during the experiment, what constitutes significance, etc. It is, nevertheless, an interesting analysis technique which bares further scrutiny (see Appendix VIII).

In summary, the experiments investigated to this point demonstrate a viable approach for determining measurement techniques for mixing processes. However, further work is necessary for a better understanding of the mixing process and development of future measurement techniques. This work should involve the use of an expanded parameter list with a broader range of testing for each parameter. Scale-up facilities are needed as is testing of different mixing systems (i.e., solid-liquid, etc.). Finally, analysis of the data collected must be improved if the measurement technique is to provide truly useful information.

SUMMARY

Work for this project was carried out in three basic areas. First, an investigation of the literature was made to determine the scope of work to be done, the mixing system to be used, the components to be mixed, and the type of measurements to be made. Second, based upon knowledge gained from the investigation of the literature, an experimental apparatus was built, keeping in mind the criteria of safety, automation, reliability, reproducibility, ease of operation, and the ability of the equipment to be used later by industry in a plant environment. Third, experiments were designed, performed, and analyzed in such a manner as to fulfill the goals of the project and present meaningful information to the mixing community.

The objective of the project was to develop performance test procedures and measurement techniques for batch mixing systems. The performance tests were to be designed such that they would enhance understanding of the mixing process, aid in facility design, and be useful for in-plant trouble-shooting of batch mixing systems. Due to the complex nature and variety of the mixing

process, and the limitations imposed upon the project by time, money, and manpower constraints, it was necessary to carefully select which type of mixing system was to be investigated, what parameters would be considered, what equipment would be used, and what analysis of the data would be performed.

The performance test which was established through the investigation of the literature, and the help of a panel of experts (see Appendix I) is as follows:

- 1) Mixing system - miscible liquid blending with no reactions carried out at ambient condition.
- 2) Mixing components = deionized water as the mixing fluid and an approximately 10 wt percent NaCl solution as the tracer.
- 3) Parameters = impeller type, C/T, RPM, concentration, probe location, and power (torque).
- 4) Measurement technique = measurement of ion concentration by the use of conductivity probes, measurement of power by the use of a tachometer and torque arm/weight scale combined apparatus (see Appendix IX).

The second part of the project was the design of the apparatus. The apparatus consists of three parts: the physical equipment, the data collection system, and the measurement equipment.

- 1) Geometric configuration (to be considered as a standard for laboratory testing) is as follows:

Tank diameter (T) = 0.25 m (9.75 in) (range = 0.23-0.38 m (9-15 in))

Z/T = 1.0 (range = 0.8 to 1.5)

C/T = 0.13, 0.33, 0.50 (range = 0 to 0.50)

Baffles = four baffles, 90 deg spacing, minimal bottom and side clearance

Tank bottom = flat

Impellers = flat blade turbine with four blades w/d = 1/6,
pitch (45 deg) blade turbine with four blades
w/d = 1/5 {note: six bladed turbines were used
in the experiments}

Shaft = vertical shaft centrally located

Power = motor to establish 2.95 kW/m³ (15 hp/1000 gal)

- 2) Data collection system:

Hardware = high-speed, scientific computer with 32-bit internal structure and an accompanying multiprogrammer with 5 A/D, 5 memory (4k), 1 timing and 1 relay cards

Software = interactive program for controlling the experiment,
collecting and manipulating data

3) Measurement equipment:

Probe type = conductivity (commercially available)

Probe circuit = for linearity, sensitivity, noise reduction and
the elimination of crosstalk (see fig. 6)

Probe number = four

Probe locations: (see table 2)

Probe #3 = in front of baffle

Probe #4 = behind baffle (same baffle as probe #3)

Probe #5 = area of good mixing (away from baffles)

Probe #6 = on surface above probe #5

RPM = tachometer attached by belt to impeller shaft

Torque = hanging weight on electronic weigh scale, tension from
torque arm on mixing motor

Injection = automated addition of tracer from $0.0107E-3 \text{ m}^3$ (10.7 cc)
calibrated loop, injection located in area of good
mixing (see fig. 8)

The third portion of this project was the design, running, and analysis of the actual experiments. A matrix type design of experiments was developed such that the most information could be gained given the constraints of time, money, and manpower (see table 1). Running of the experiments consisted of first calibrating the probes, and then performing the experiments as specified in the matrix design. Analysis consisted of first determining the accuracy, reliability, and reproducibility of the probes, then analyzing the probe response curves and finally performing a mixtime comparison analysis with the independent variables in the system. Work was also done on alternate torque measurement techniques.

Calibration and accuracy for the probes entail the positioning of the probes, calibration of the probes for both temperature and concentration, and a verification of the accuracy, reliability, and reproducibility of the probes. It is not possible to position the probes in absolute locations of best and worst mixing as these locations change dependent upon the system parameters. Probes were positioned in locations where it was felt the most information could be gained. Thusly, a probe was placed in front of a baffle and behind the same one. A probe was placed in an area where good mixing should generally occur, and in an area of poor mixing (see fig. 4 and

table 2). Probe locations were carefully determined using a three dimensional coordinate system.

Probe calibration is a time consuming operation that, if done correctly, need not be repeated often. After six weeks of extensive use, all probe responses were within 1 percent of their previous values. The probe calibration process consists of three distinct parts. They are: 1) Calibration of meter reading values for each pc-board (i.e., the oscillator-amplifier sine wave amplitude). 2) Calibration of the probe responses at different concentrations (up to 0.10 wt percent). 3) Establishment of temperature correction factors for the probes in the range of ambient conditions (19 to 29°C). In addition to these three parts, it is also necessary to maintain a standard probe to pc-board configuration, and run experiments in the range where conversion to concentration values is possible (i.e., ranges of concentration and temperature).

In order to have a successful experiment, the conductivity probes should be accurate, reliable and reproducible. In all, probes failed in only 5.61 percent of the runs, with probe #5 failing the most often (51.5 percent of all failures). A probe failure is considered a failure of the probe, pc-board or connecting wiring. In all, 97 percent of the failures were due to failures in the connecting wiring. Accuracy of results was high, with nearly all observed concentrations falling within 2 percent of the expected values. Reproducibility was also high, with all experimental results falling within a 95 percent confidence interval about the sample mean.

Probe responses were examined for their ability to detect changes in the mixing system. To this end, probe responses were observed and related to the variables of probe noise, probe location, flow pattern, impeller type, C/T, RPM, and concentration. The purpose of this phase of the analysis was to qualitatively prove the viability of using conductivity probes as a means of observing the mixing process.

A summary of findings for probe responses show that:

- probe response time is easily fast enough for system dynamics to be observed
- probe response noise is sufficiently low and of an averaging nature such that it does not significantly affect information gained from the experiment
- probe response noise is affected by experimental parameters, especially impeller type, concentration, and rpm

- probe location affects the probe response as a function of time, but not the final concentration in the system
- comparison of individual probe response curves can show the effect of baffles, indicate areas of good and poor mixing, show circulation rates and indicate back-mixing occurrences
- probe responses are not an effective measure of micro-mixing
- comparisons of separate experiments demonstrate the effects of different flow patterns on the probe responses
- variations caused by different impeller types can be observed in the probe responses when comparing separate experiments
- changes in the C/T change the flow pattern, and as such change the probe response, as observed through comparisons of separate experiments
- probe responses are strongly affected by changes in rpm, both in curve shape and response as a function of time
- probe responses are not significantly affected by changes in the average run concentration, other than the obvious change in magnitude

Once it was established that probe response curves did indeed model the dynamics of the mixing system, it was important to determine a method for analyzing the data other than in a qualitative manner. To this end, mixtimes were determined as the dependent variable, and subsequently related to the independent variables of probe location, flow pattern, impeller type, C/T, RPM, power, and concentration.

A summary of findings related to the mixtime analysis are as follows:

- mixtime was determined graphically by visual observation for each probe to within ± 5 percent of the sample mean
- probe #6 mixtime determinations varied considerably due to the nature of the experiments and the location of this probe
- mixtime determinations were reproducible to within ± 10 percent
- graphical mixtime determination, as described, provides adequate information for further analysis
- mixtime varies according to probe location and experimental conditions
- probe #4 lagged probe #3 mixtimes by 5 percent in 89 percent of the applicable cases (applicable cases did not have extremely axial flow)
- mixing generally takes longer behind a baffle than in front of it
- probe #5 mixtimes lags probe #3 mixtimes when axial flow was not present, this is an indication that under the right conditions, mixing in front of the baffle is better than in the free area between baffles

- in cases of extreme axial flow, baffle effects are minimized and probe #5 and probe #3 mixtimes are comparable
- probe #6 (surface) mixtimes lagged all other probe mixtimes in all cases where extreme radial flow did not exist
- probe #6 responses are more dramatic for system changes
- probe #6 mixtimes can be used to show flow pattern changes, indicating whether a flow is predominately axial or radial
- flow pattern reversal was most easily observed in the probe #6 mixtimes when a pitch blade turbine was used
- the flat blade turbine gives shorter mixtimes at 50 and 150 rpm for all probes except probe #6 at 50 rpm than does the pitch blade turbine ($C/T = 0.33$)
- the two impellers produce similar mixtimes when the rotation rate is 300 rpm ($C/T = 0.33$)
- C/T can be used to change the flow pattern from axial to radial or radial to axial
- increasing rpm decreases mixtimes for all probes
- the average concentration of an experimental run does not affect the mixtime
- power is directly related to rpm, and thus its effect on mixtime is the same

A final area of consideration is the measurement of the torque. Two different methods were investigated to determine the torque in a given experiment. One method used a torque arm attached to the mixing motor, with a hanging weight attached to the torque arm and positioned on a weigh scale (this method was used for the experiments). The other method used a torque plate centered beneath the mixing vessel, with information being obtained from strain gauges. Both techniques provided accurate and reproducible results over the range of rpm values, C/T values, and impeller types used in the experiments. Variation between methods was less than 4 percent, and variations with the theoretical values as given by the manufacturer was on the order of 17 percent. This latter difference is attributable to bearing and gearbox losses.

CONCLUSIONS

- 1) A performance test using conductivity probes was developed which can be used to enhance understanding of the mixing process, aid in facility design, and may be useful for in-plant trouble-shooting for a batch mixing system.
- 2) A standard geometry has been determined for use in laboratory testing.
- 3) Conductivity probes are flexible, accurate, reliable, and give reproducible results.
- 4) Probe response curves are a viable means of observing the dynamics of the mixing process in a qualitative manner.
- 5) Mixtime analysis provides useful insights into the mixing process and can be used to enhance understanding of the mixing process, aid in facility design, and be useful for in-plant trouble-shooting of batch mixing processes.
- 6) The torque-arm/weigh-scale technique used for these experiments to measure torque provides accurate, reliable, and reproducible information needed for power calculations.

RECOMMENDATIONS

- 1) The work done in this project is incomplete, and further testing is necessary for a better understanding of the mixing process.
- 2) Further work should be done using an expanded parameter list, and a broader range of testing for each parameter.
- 3) A moderate size facility 0.379-0.757 m³ (100-200 gallons) is needed for comparison testing of work done in the laboratory scale equipment. All experiments run on the laboratory scale should in turn be run on the moderate scale equipment.
- 4) Work should be expanded to include other mixing systems. The system which should be worked on after miscible liquids is most likely a solid-liquid system.
- 5) The information collected should be more rigorously analyzed, removing qualitative analysis as much as possible from the tabulated results.

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EXPERIMENTAL DESIGN

TABLE 1

RPM	IMPELLER	C/T	CONCENTRATION *
50	PITCH	0.33	TO 0.06 WT% (7 STEPS)
50	PITCH	0.50	"
50	FLAT	0.13	"
50	FLAT	0.33	"
150	PITCH	0.33	"
150	PITCH	0.50	"
150	FLAT	0.13	"
150	FLAT	0.33	"
300	PITCH	0.33	"
300	PITCH	0.50	"
300	FLAT	0.13	"
300	FLAT	0.33	"

* SEVEN CONCENTRATIONS ARE AS FOLLOWS (WT%): 0.00949, 0.01896, 0.02833, 0.03784, 0.04725, 0.05664, 0.06601 (THESE ARE EXPECTED VALUES)

NOTE: AT EACH SET OF CONDITIONS SEVEN IDENTICAL EXPERIMENTS WERE RUN.

PROBE AND INJECTION
LOCATION COORDINATES *

TABLE 2

	ANGLE (DEG)	X m (in.)	Y m (in.)
	-----	-----	-----
PROBE #3	195	0.121 (4.75)	0.057 (2.25)
PROBE #4	175	0.121 (4.75)	0.057 (2.25)
PROBE #5	300	0.121 (4.75)	0.057 (2.25)
PROBE #6	300	0.083 (3.25)	0.216 (8.50)
INJEC. POINT **	125	0.038 (1.50)	0.076 (3.00)
RECIRC. POINT	350	0.095 (3.75)	0.051 (2.00)

* REFERENCE BAFFLE IS AT 0 DEG., ANGLE TAKEN FROM THIS POINT AROUND THE CIRCUMFERENCE OF THE TANK. X=DISTANCE FROM CENTER OF TANK. Y=DISTANCE FROM BOTTOM OF TANK.

** INJECTION POINT DEPENDS UPON IMPELLER LOCATION. RECIRCULATION POINT IS FIXED AND DOES NOT DEPEND UPON IMPELLER LOCATION.

SIGNAL AMPLITUDE

EQUATIONS

TABLE 3

GENERAL FORM: $SA = (M_{sa} * MR) + B_{sa}$

PC-BOARD -----	M_{sa} -----	B_{sa} -----
#1	0.00582	-0.00115
#2	0.00563	-0.03046
#3	0.00541	-0.00542
#4	0.00559	-0.01045

WHERE: SA=SIGNAL AMPLITUDE, M_{sa} =SIGNAL AMPLITUDE SLOPE,
MR=METER READING, B_{sa} =SIGNAL AMPLITUDE INTERCEPT

CALIBRATION DESIGN **
TABLE 4

TEMPERATURE (DEGREE C)	CONCENTRATION (WT% NaCl)	METER READING * (PROBE CIRCUIT)
19	0.00	10-160 IN STEPS OF 5
19	0.02	"
19	0.04	"
19	0.06	"
19	0.08	"
19	0.10	"
24	0.00	"
24	0.02	"
24	0.04	"
24	0.06	"
24	0.08	"
24	0.10	"
29	0.00	"
29	0.02	"
29	0.04	"
29	0.06	"
29	0.08	"
29	0.10	"

* METER READINGS = 10, 15, 20, 25, 30, 35, 40, 45, 50, 55, 60, 65, 70, 75, 80, 85, 90, 95, 100, 110, 120, 130, 140, 150, 160

** CALIBRATION DESIGN IS FOR PROBES #3, #4, #5 AND #6 WHEN THEY ARE CONNECTED TO PC-BOARDS 1, 2, 3 AND 4 RESPECTIVELY.

PROBE RESPONSE
EQUATIONS
TABLE 5

GENERAL FORM: $PR = (M_{pr} * MR) + B_{pr}$

TEMP. (DEG. C)	CONC. (WT% NaCl)	PROBE #	M_{pr}	B_{pr}	Df
19	0.00	3	-.0047	3.619	24
19	0.02	3	-.1246	3.627	19
19	0.04	3	-.2492	3.692	9
19	0.06	3	-.3495	3.661	6
19	0.08	3	-.4833	3.808	3
19	0.10	3	-.5919	3.833	2
19	0.00	4	-.0039	3.594	24
19	0.02	4	-.1185	4.168	19
19	0.04	4	-.2247	4.512	11
19	0.06	4	-.3398	5.403	7
19	0.08	4	-.4604	6.035	5
19	0.10	4	-.5641	6.666	4
19	0.00	5	-.0042	3.607	24
19	0.02	5	-.1232	3.675	19
19	0.04	5	-.2376	3.558	10
19	0.06	5	-.3506	3.884	6
19	0.08	5	-.4723	3.894	4
19	0.10	5	-.4388	3.438	2
19	0.00	6	-.0042	3.607	24
19	0.02	6	-.1186	3.839	20
19	0.04	6	-.2294	3.925	11
19	0.06	6	-.3391	4.423	6
19	0.08	6	-.4627	4.748	4
19	0.10	6	-.5672	5.037	3
24	0.00	3	-.0063	3.815	24
24	0.02	3	-.1415	3.805	18
24	0.04	3	-.2732	3.839	8
24	0.06	3	-.4020	3.842	5
24	0.08	3	-.5331	3.932	3
24	0.10	3	-.6631	3.989	2
24	0.00	3	-.0054	3.770	24
24	0.02	4	-.1346	4.417	19
24	0.04	4	-.2612	5.124	9
24	0.06	4	-.3858	5.813	6
24	0.08	4	-.5128	6.549	4
24	0.10	4	-.6418	7.299	3
24	0.00	5	-.0057	3.786	24
24	0.02	5	-.1403	3.881	18
24	0.04	5	-.2738	3.992	8
24	0.06	5	-.4012	4.090	5
24	0.08	5	-.5304	4.191	3
24	0.10	5	-.6486	4.303	2
24	0.00	6	-.0056	3.780	24
24	0.02	6	-.1368	4.097	18
24	0.04	6	-.2650	4.402	9
24	0.06	6	-.3923	4.734	5
24	0.08	6	-.5083	4.916	4
24	0.10	6	-.6417	5.387	2

TABLE 5
(CONTINUED)

TEMP. (DEG. C)	CONC. (WT% NaCl)	PROBE #	M _{pr}	B _{pr}	Df
29	0.00	3	-.0075	3.879	24
29	0.02	3	-.1553	3.879	16
29	0.04	3	-.3035	4.071	7
29	0.06	3	-.4532	4.149	4
29	0.08	3	-.5993	4.001	2
29	0.10	3	-.6970	3.589	2
29	0.00	4	-.0065	3.824	24
29	0.02	4	-.1506	4.571	18
29	0.04	4	-.2915	5.348	8
29	0.06	4	-.4362	6.213	5
29	0.08	4	-.5694	6.830	4
29	0.10	4	-.7164	7.752	3
29	0.00	5	-.0068	3.849	24
29	0.02	5	-.1559	3.991	16
29	0.04	5	-.3018	4.070	7
29	0.06	5	-.4496	4.257	4
29	0.08	5	-.5898	4.268	2
29	0.10	5	-.7312	4.404	2
29	0.00	6	-.0067	3.837	24
29	0.02	6	-.1528	4.203	17
29	0.04	6	-.2911	4.533	8
29	0.06	6	-.4327	4.862	5
29	0.08	6	-.4359	4.917	3
29	0.10	6	-.7216	5.645	2

WHERE: PR=PROBE RESPONSE, M_{pr}=PROBE RESPONSE SLOPE,
MR=METER READING, B_{pr}=PROBE RESPONSE INTERCEPT

Df=DEGREES OF FREEDOM: AS THE DEGREES OF FREEDOM DECREASE
THE ERROR IN VALUES INCREASES

TEMPERATURE
CONVERSION FACTORS

TABLE 6

PROBE	K_s	K_i
-----	-----	-----
3	0.0211	0.0096
4	0.0185	0.0133
5	0.0237	0.0103
6	0.0238	0.0090

BASE TEMPERATURE (T_{base}) IS 24 DEGREE C

K_s = CHANGE IN SLOPE/DEGREE C CHANGE IN TEMPERATURE

K_i = CHANGE IN INTERCEPT/DEGREE C CHANGE IN TEMPERATURE

CONCENTRATION ACCURACY

STATISTICS

TABLE 7

EXPECTED CONC. (WT% NaCl)	OBSERVED CONC. (WT% NaCl)	ERROR (%)	PROBE-TO-PROBE VARIATION (%)	RANGE OF RESPONSE (%)
0.00949	0.00913	3.8	4.4	11.9
0.01896	0.01873	1.2	2.2	3.2
0.02833	0.02813	0.7	2.2	3.2
0.03784	0.03743	1.1	1.6	3.4
0.04725	0.04665	1.3	1.8	4.2
0.05664	0.05583	1.4	1.0	3.4
0.06601	0.06478	1.9	0.4	3.3

CORRELATION COEFFICIENTS

TABLE 8

```

*****
*                               SUMMARY STATISTICS                               *
*                               ON DATA SET:                                       *
*                               MIXTIMES AND CONCENTRATIONS (WT %)                 *
*****

```

(SUBFILES IGNORED)

CORRELATION MATRIX

	MIXTIME #4	MIXTIME #5	MIXTIME #6	RPM	IMPELLER	CONC.
MIXTIME #3	.9984560	.9902375	.7943061	-.8812963	.1343965	-.0624895
MIXTIME #4		.9926423	.7874389	-.8809342	.1410659	-.0675428
MIXTIME #5			.8088981	-.8952735	.1054441	-.0789490
MIXTIME #6				-.8185558	-.2317648	-.0425129
RPM					.0138727	.0209695
IMPELLER						-.0184337

	C/T
MIXTIME #3	.0628910
MIXTIME #4	.0815206
MIXTIME #5	.0740451
MIXTIME #6	-.2265783
RPM	.0188891
IMPELLER	.7016182
CONC.	-.0250995

FIGURES

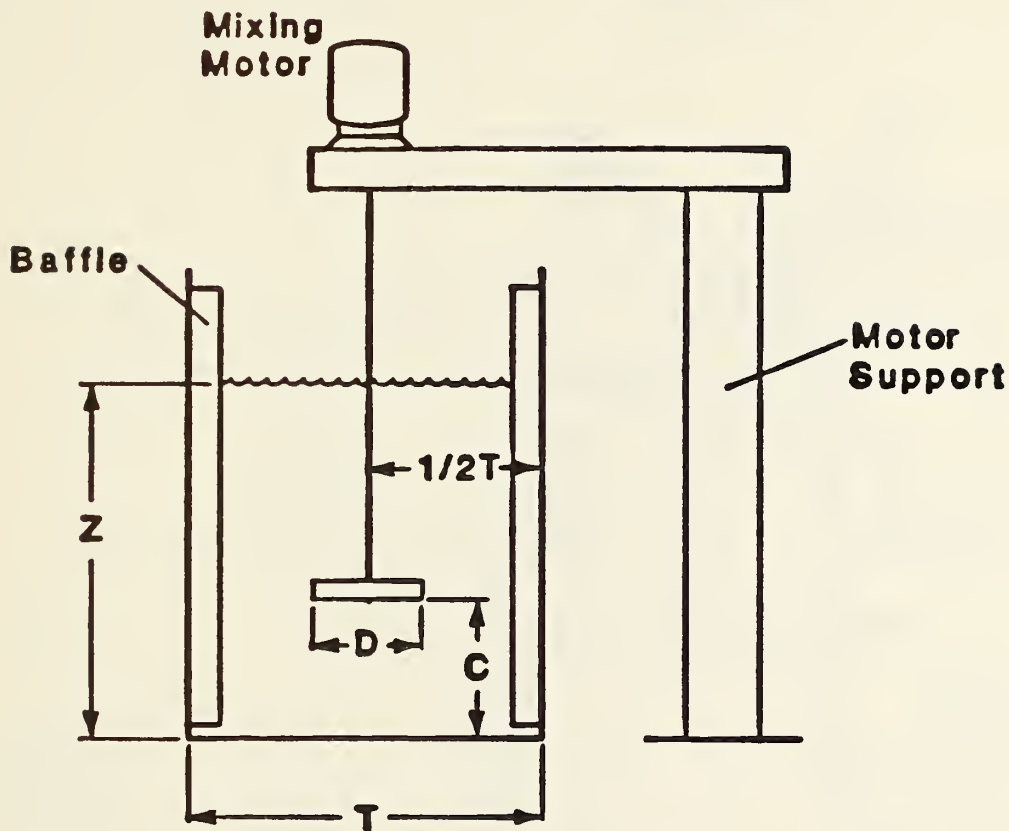
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FIGURES
(continued)

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FIGURE 1

Mixing Vessel Specifications



Specifications:

Glass Tank ($1.89E-2 \text{ m}^3$) (5 gallon)

0.25 m I.D. X 0.44 m high (9.75 in I.D. X 17.5 in high)

$C/T = 0 \text{ to } 0.50$ $D/T = 0.41$ $Z/T = 1.0$

Baffles = 4 baffles (0.083 X I.D. width)/90 degree spacing
minimal bottom and side spacing ($< 3.2E-3 \text{ m}$)

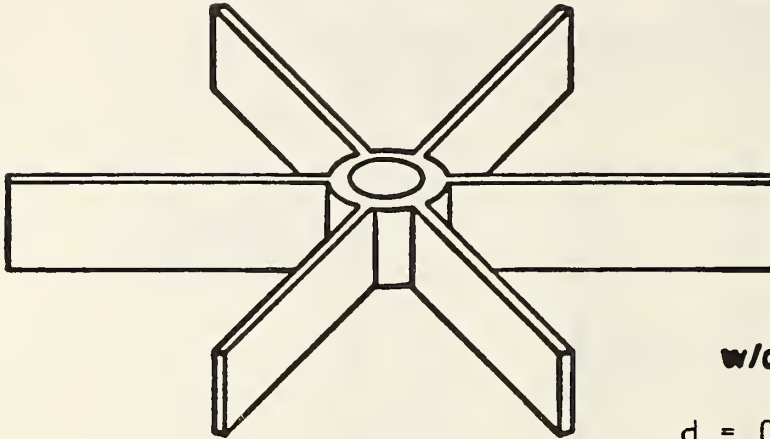
Impeller = flat blade turbine (6 blades) + pitch (45 deg.)
blade turbine (6 blades)

Mixing Motor = variable speed (0-1100 rpm), 186 W (1/4. hp)

Tank bottom = flat

FIGURE 2

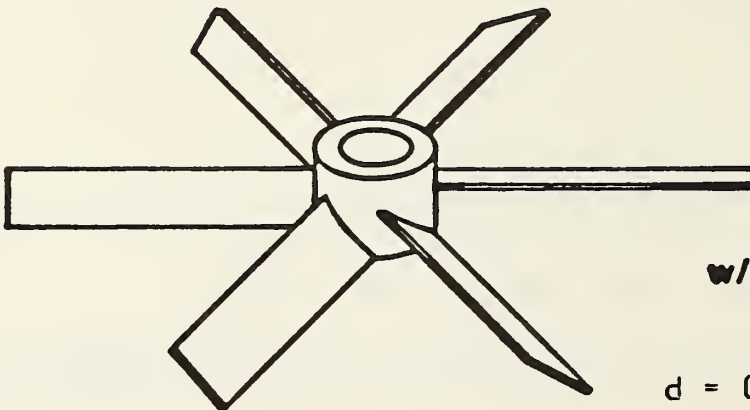
IMPELLERS



$w/d = 1/6$

$d = 0.102 \text{ m (4 in)}$

Flat Blade Turbine



$w/d = 1/5$

$d = 0.102 \text{ m (4 in)}$

Pitch Blade Turbine

Figure 3
Laboratory Experimental Design

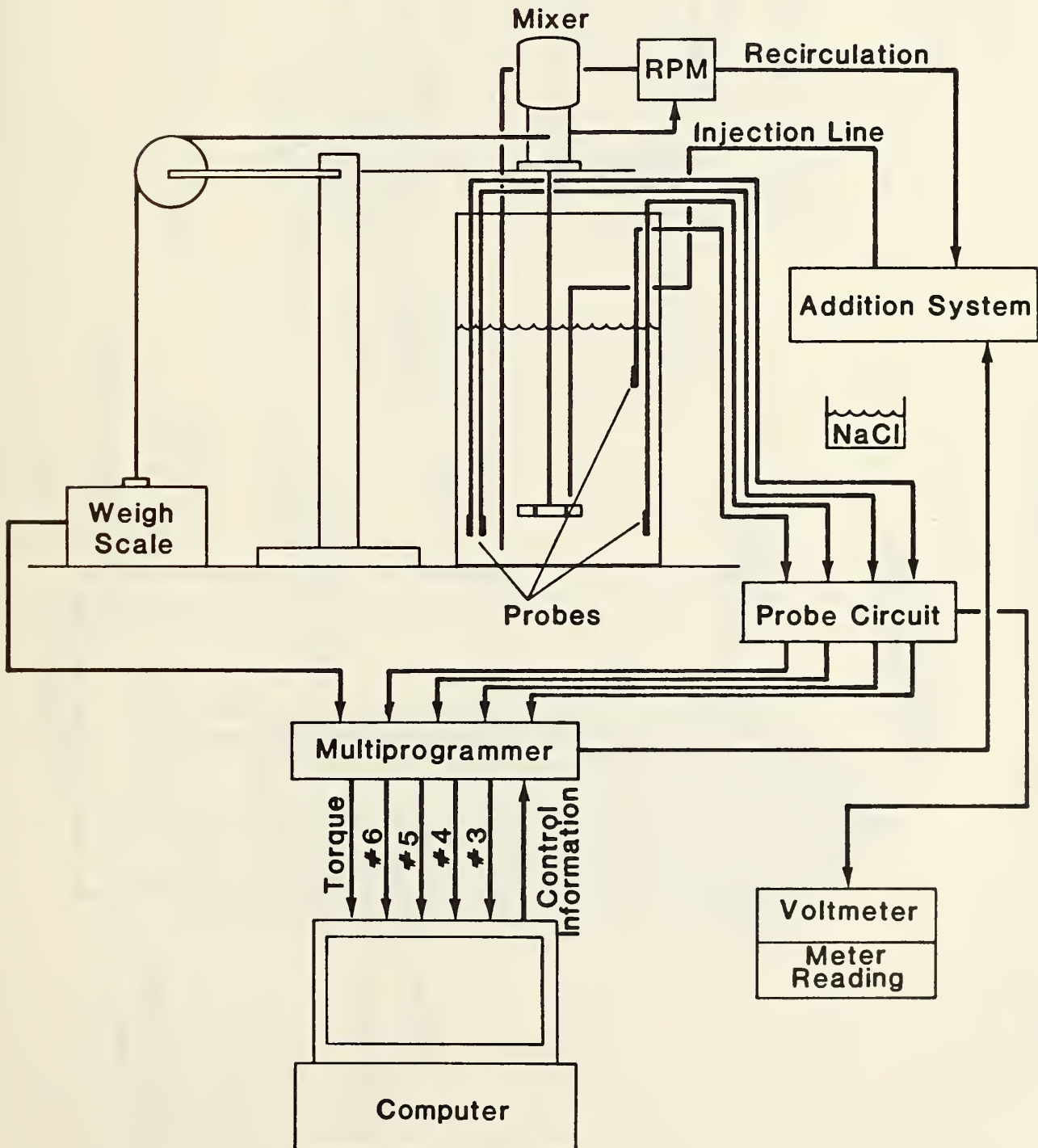
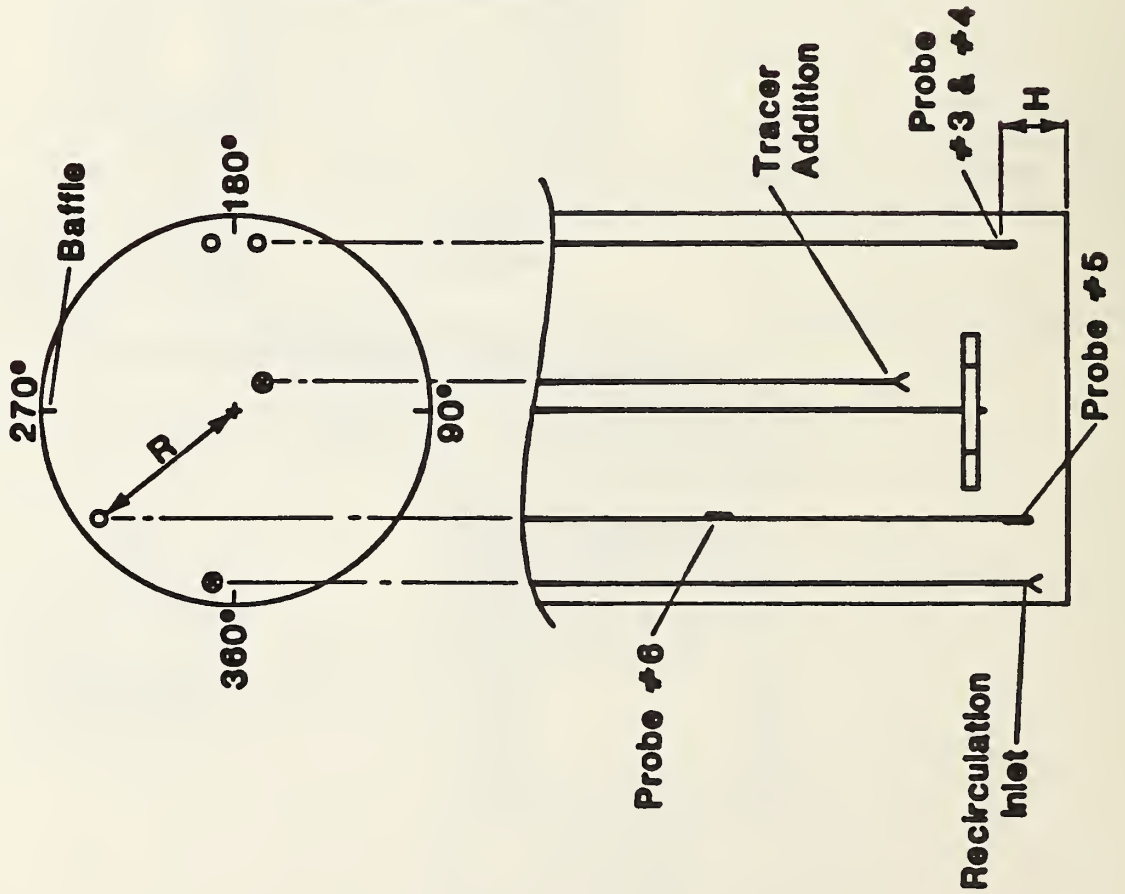


FIGURE 4



PROBE LOCATIONS

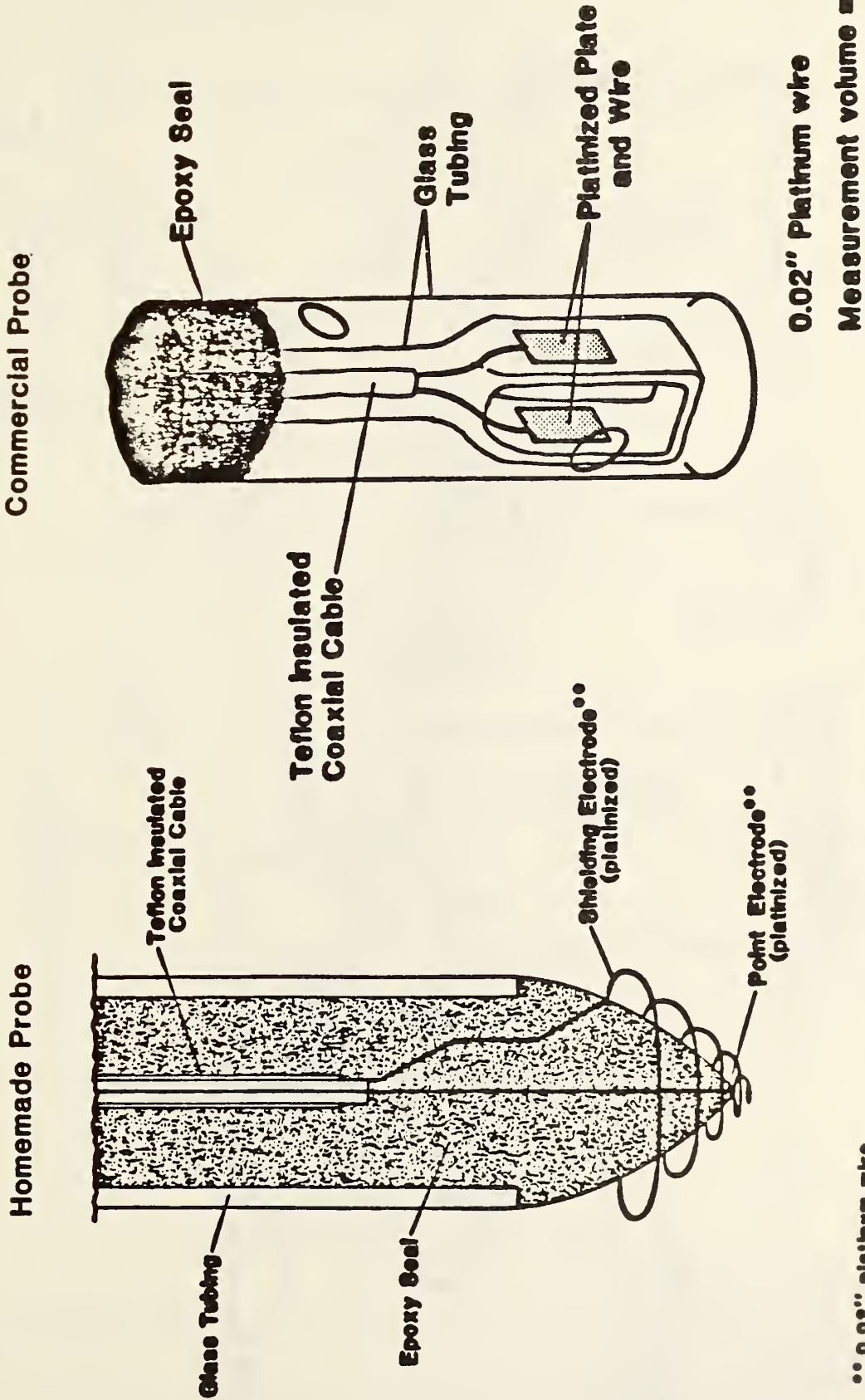
Probe ϕ	Angle, $^{\circ}$	Radius, R	Height, H
3	195	0.121	0.057
4	175	0.121	0.057
5	300	0.121	0.057
6	300	0.083	0.216
Inlet	350	0.095	0.051
Addition	125	0.038	0.076*

Radius and Height are in meters.

* Varies with C/T

FIGURE 5

Conductivity Probes



** 0.02" platinum wire

o Design from-Soon Jai Khang and Thomas J. Fitzgerald.

"A New Probe Circuit for Measuring Electrolyte Conductivity",
Ind. Eng. Chem. Fundam. Vol. 14, No. 3, 1975.

FIGURE 6

NBS Probe Circuit

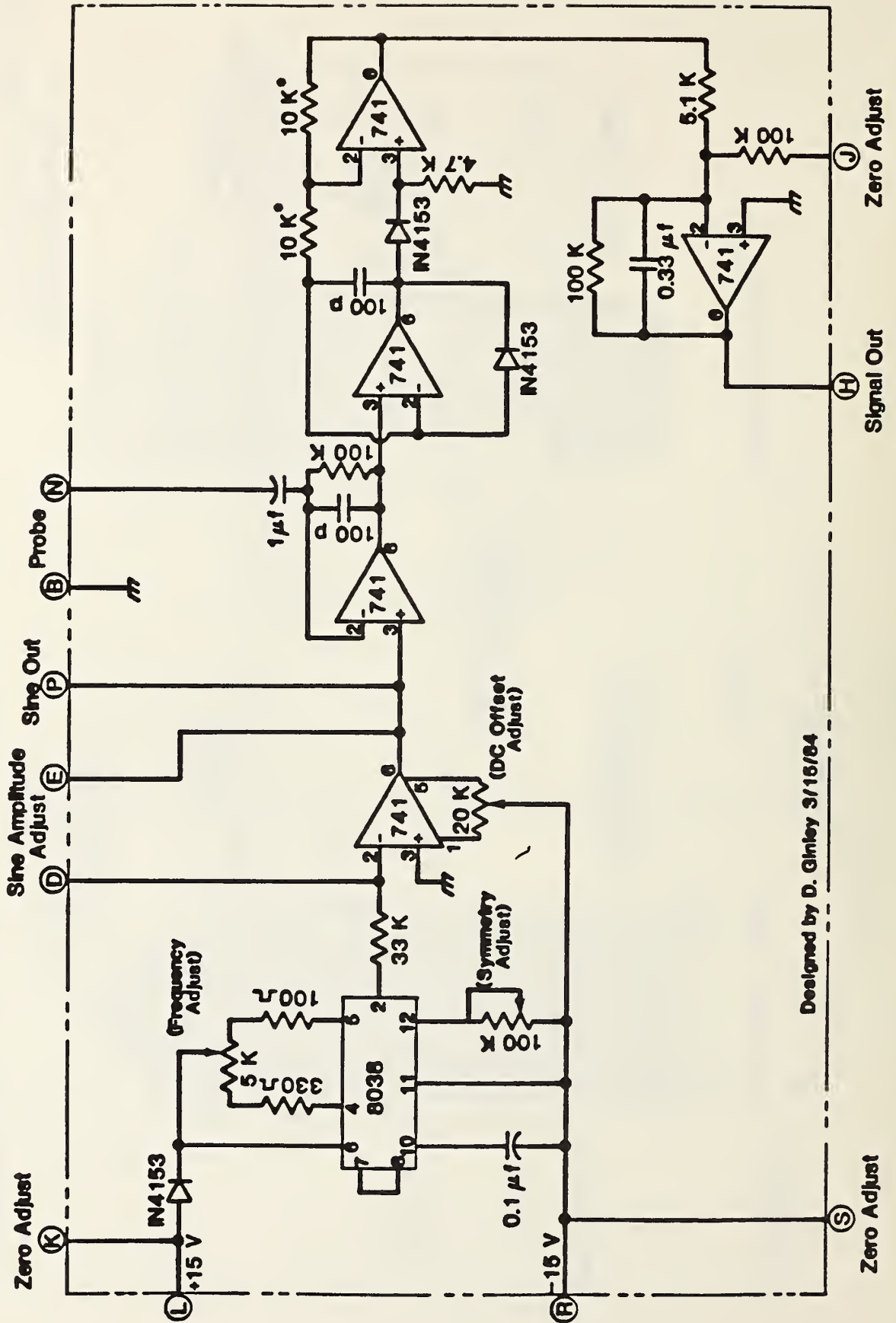
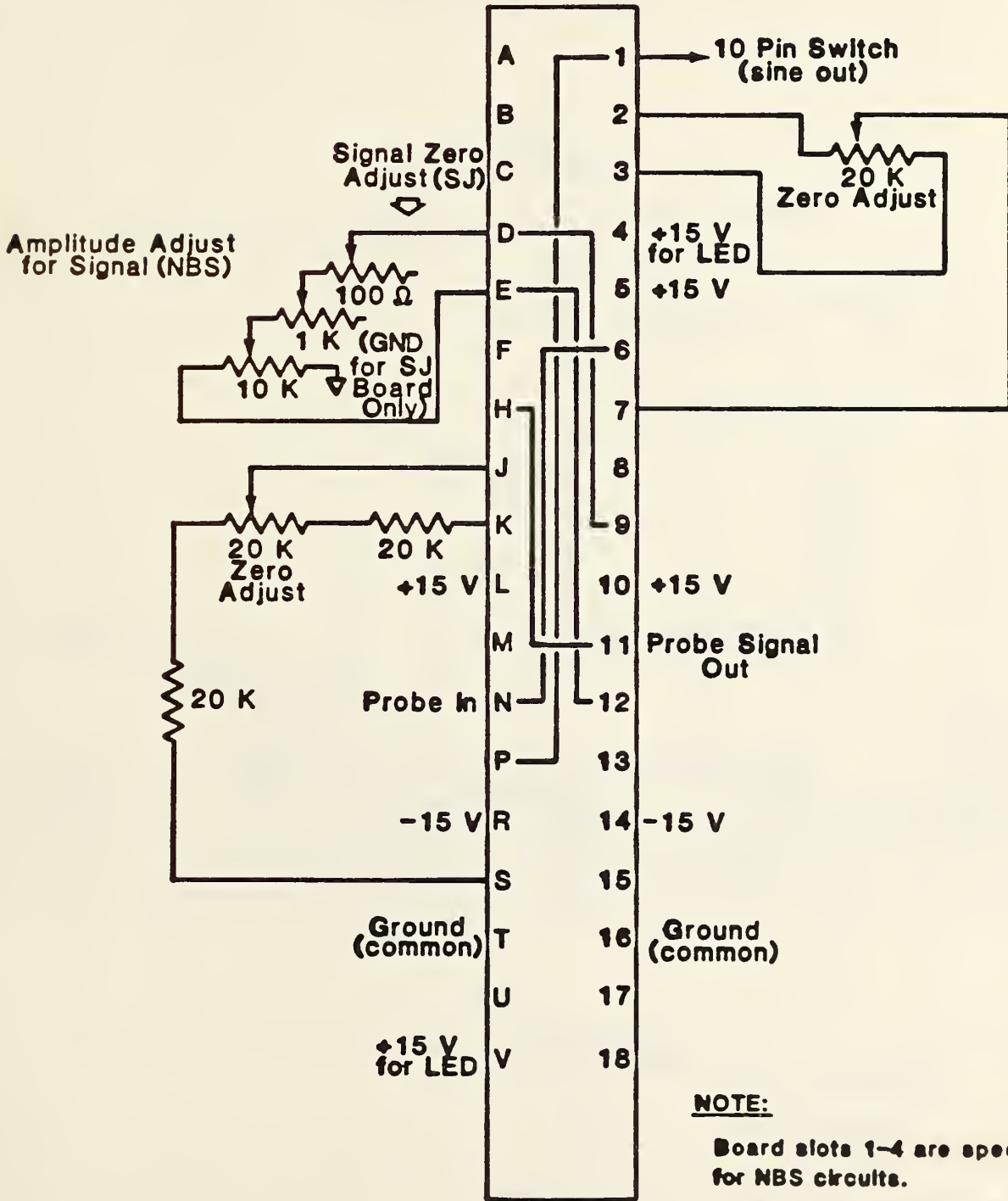


FIGURE 6A

18 Pin Connector for Probe Circuits



NOTE:

Board slots 1-4 are specifically for NBS circuits.

SJ is an alternate probe circuit designed by SJ Khang and TJ Fitzgerald (1975)

Figure 7

Torque Measurement

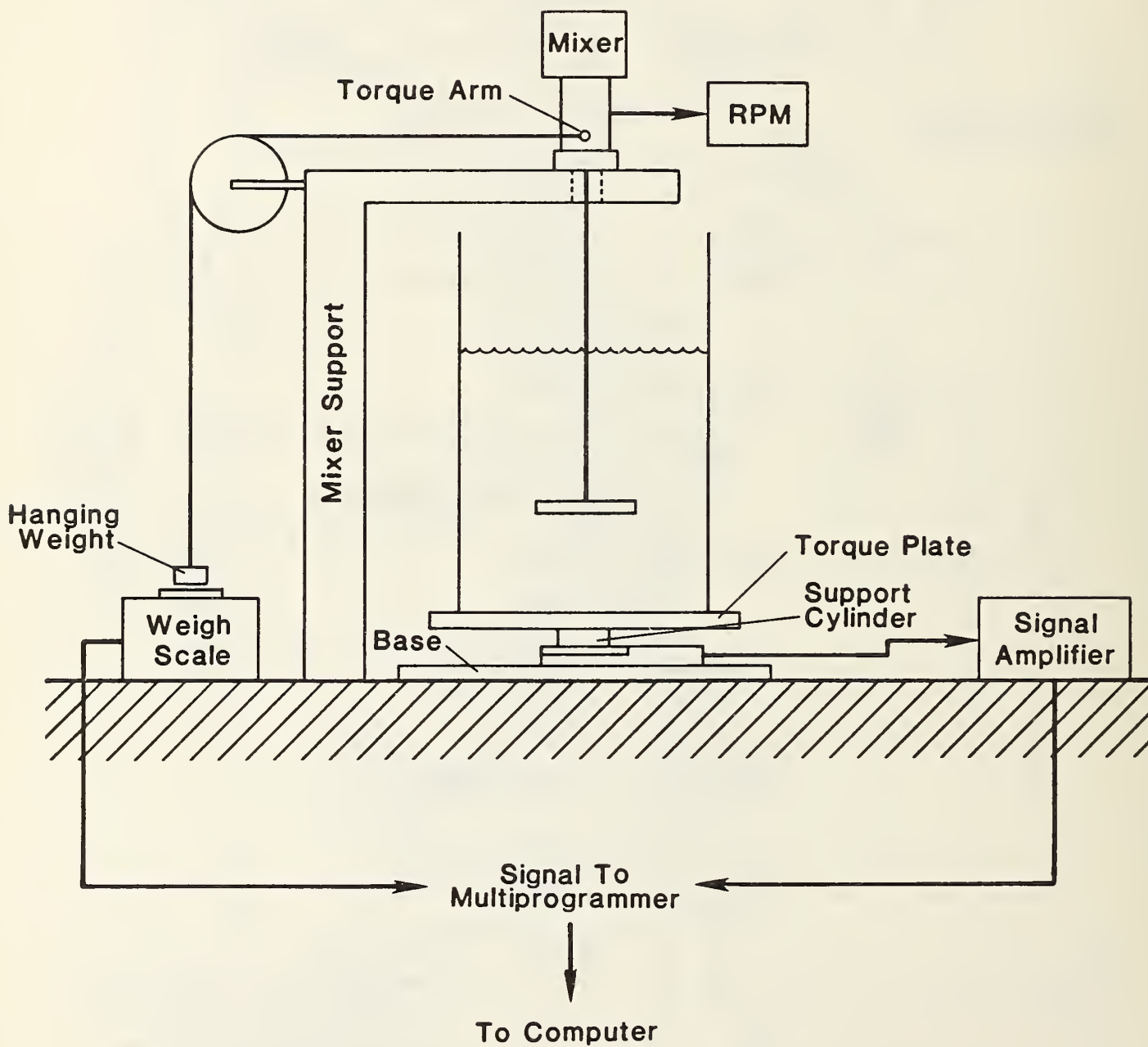
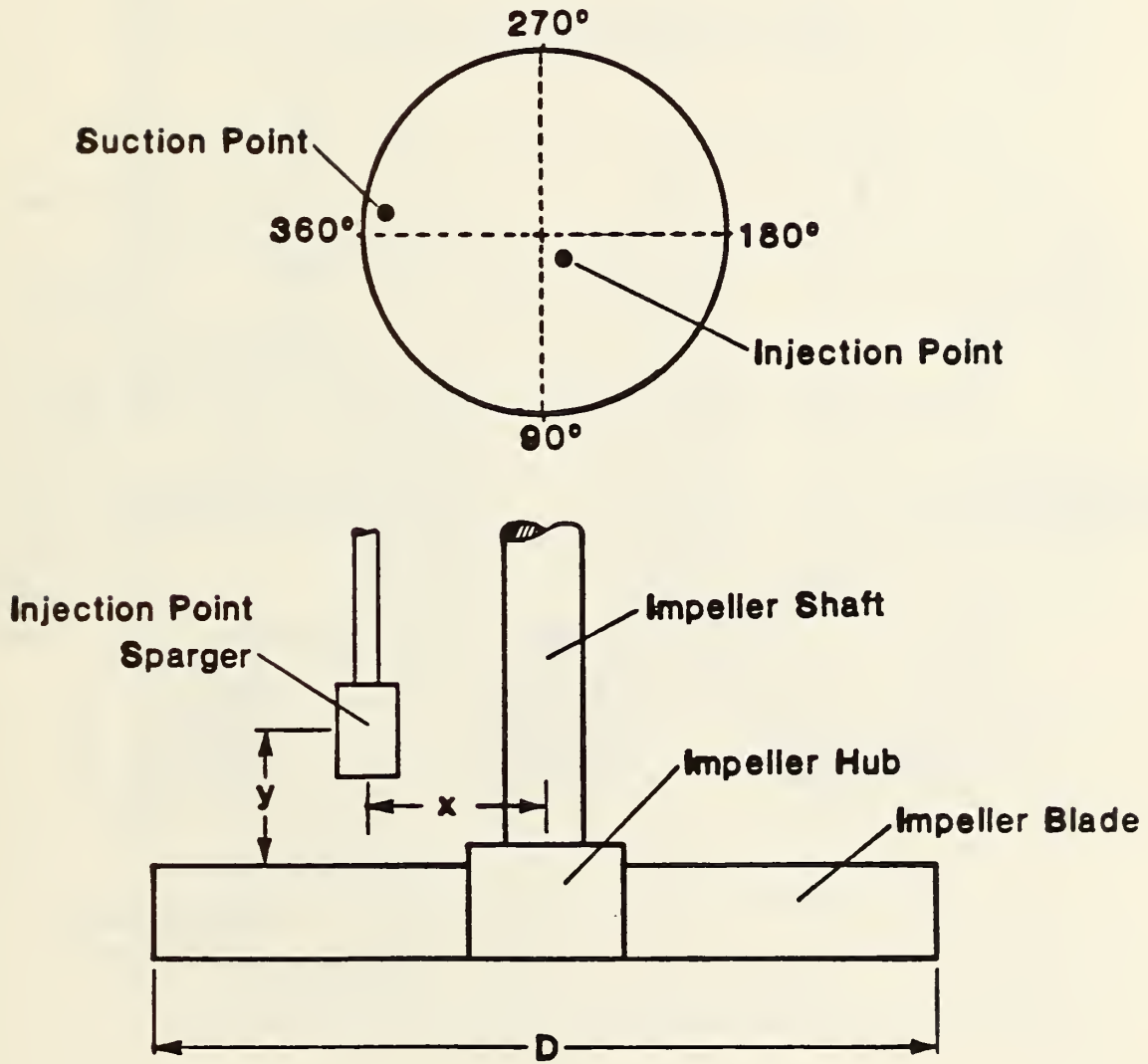


FIGURE 8

Injection Point



$$y = 0.1875(D)$$

$$x = 0.375(D)$$

FIGURE 9

Tracer Addition System

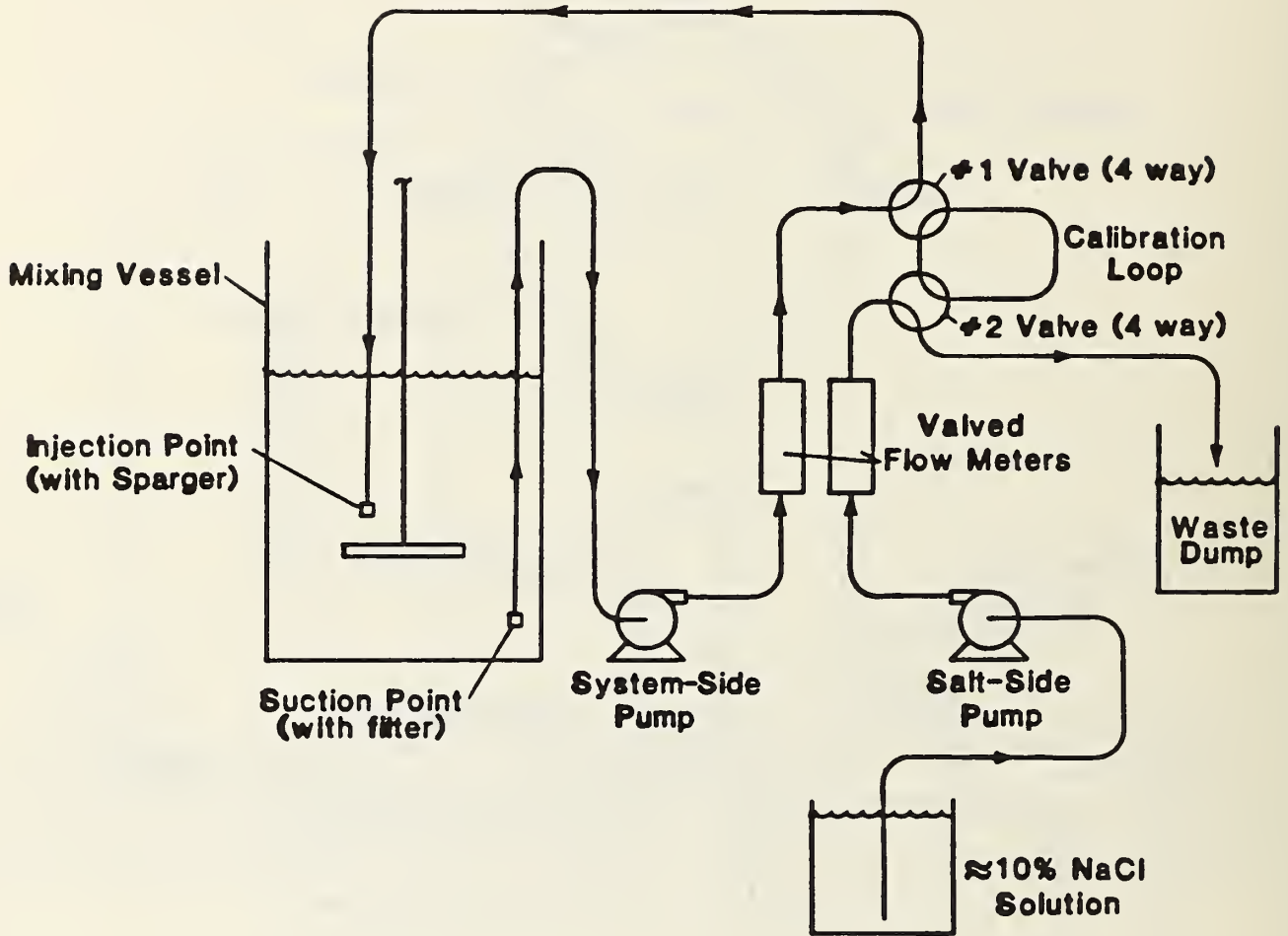


FIGURE 10

PC-BOARD 1

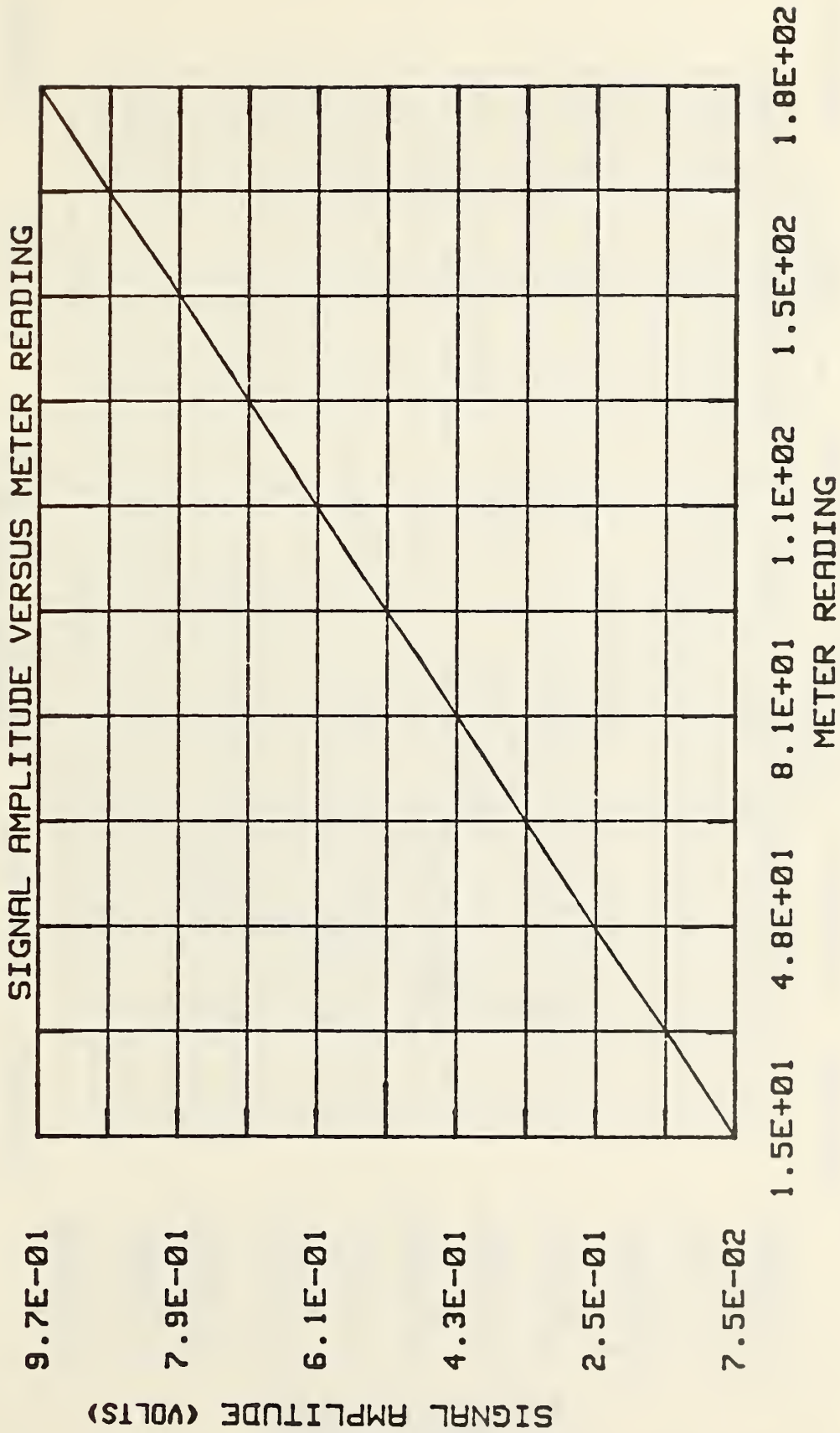


FIGURE 11

PC-BOARD 4 WEIGHT % = .0999124
 READING PROBE 4 VERSUS METER READING

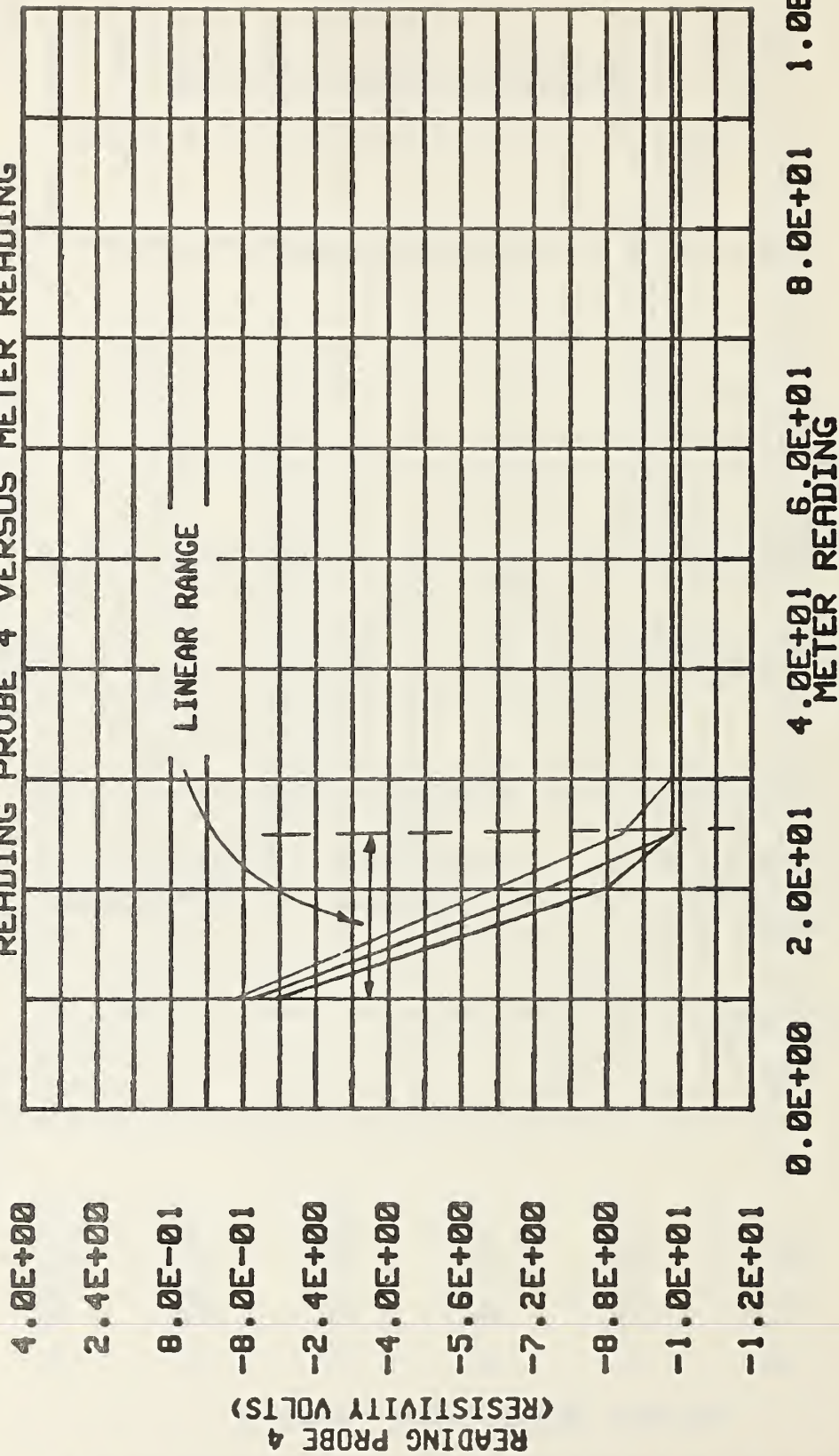
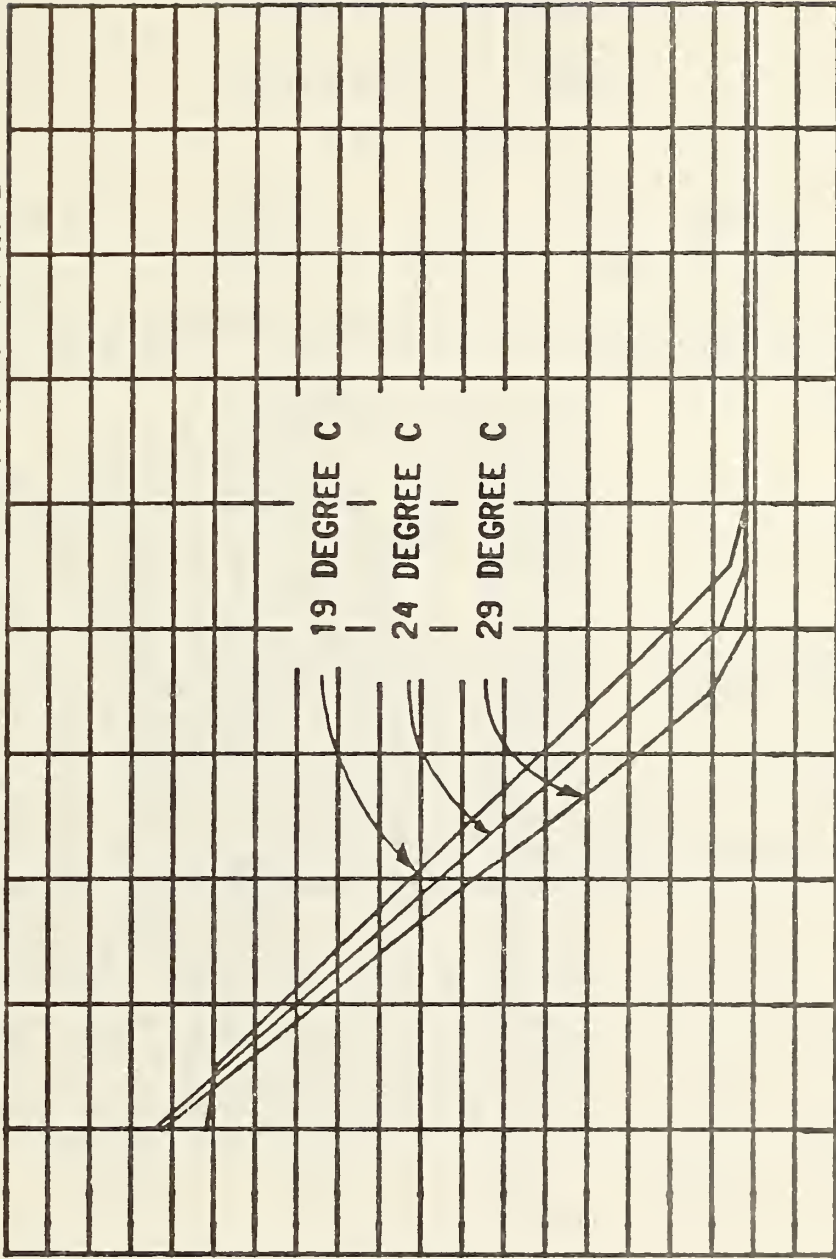


FIGURE 12

PC-BOARD 1 WEIGHT % - .04007
 READING PROBE 1 VERSUS METER READING

READING PROBE 1
 (RESISTIVITY VOLTS)

4.0E+00
 2.4E+00
 0.0E-01
 -0.0E-01
 -2.4E+00
 -4.0E+00
 -5.6E+00
 -7.2E+00
 -8.8E+00
 -1.0E+01
 -1.2E+01



0.0E+00 2.0E+01 4.0E+01 6.0E+01 8.0E+01 1.0E+02
 METER READING

DEFAULT LIST FOR
GEOMETRIC PARAMETERS

FIGURE 13

***** ASSIGNMENT SECTION FOR DEFAULT PARAMETERS. *****

Tp\$="PITCH"	!TYPE OF IMPELLER
Psz=4	!DIAMETER OF IMPELLER
Nblade=6	!NUMBER OF IMPELLER BLADES
Ic=3.250	!CLEARANCE IMPELLER TO TANK BOTTOM (IN.)
Ag1=195	!PROBE 3 ANGLE LOCATION (DEGREES)
X1=4.75	!PROBE 3 RADIAL LOCATION (IN.)
Y1=2.25	!PROBE 3 CLEARANCE FROM TANK BOTTOM (IN.)
Ag2=175	!PROBE 4 ANGLE LOCATION (DEG.)
X2=4.75	!PROBE 4 RADIAL LOCATION (IN.)
Y2=2.250	!PROBE 4 CLEARANCE FROM TANK BOTTOM (IN.)
Ag3=300	!PROBE 5 ANGLE LOCATION (DEG.)
X3=4.75	!PROBE 5 RADIAL LOCATION (IN.)
Y3=2.250	!PROBE 5 CLEARANCE FROM TANK BOTTOM (IN.)
Ag4=300	!PROBE 6 ANGLE LOCATION (DEG.)
X4=3.25	!PROBE 6 RADIAL LOCATION (IN.)
Y4=8.5	!PROBE 6 CLEARANCE FROM TANK BOTTOM (IN.)
Ag5=125	!LIQUID INPUT ANGLE LOCATION (DEG.)
Xs=1.50	!LIQUID INPUT RADIAL LOCATION (IN.)
Ys=5.00	!LIQUID INPUT VERTICAL LOCATION (IN.)
Bafw=.75	!BAFFLE WIDTH (IN.)
Bafc=2.625	!BAFFLE CLEARANCE FROM TANK BOTTOM (IN.)
Nbaf=4	!NUMBER OF BAFFLES
Bspace=90	!ANGLE LOCATION OF BAFFLES (DEG.)

EXPERIMENTAL RUN
FACT SHEET AND
GRAPHICAL REPRESENTATION

FIGURE 14

13:41:59 23 Aug 1984 50 RPM RUN #:S8231 NUMBER OF POINTS = 1000
TIME PER POINT = .2
TANK: VOL= 11.939 L , DIAMETER= 24.765 CM IMPELLOR:PITCH(4 IN)

SALT START= 0 GM, SALT FINISH= 1.029 GM

WATTS OF STIRRING ENERGY = .008284

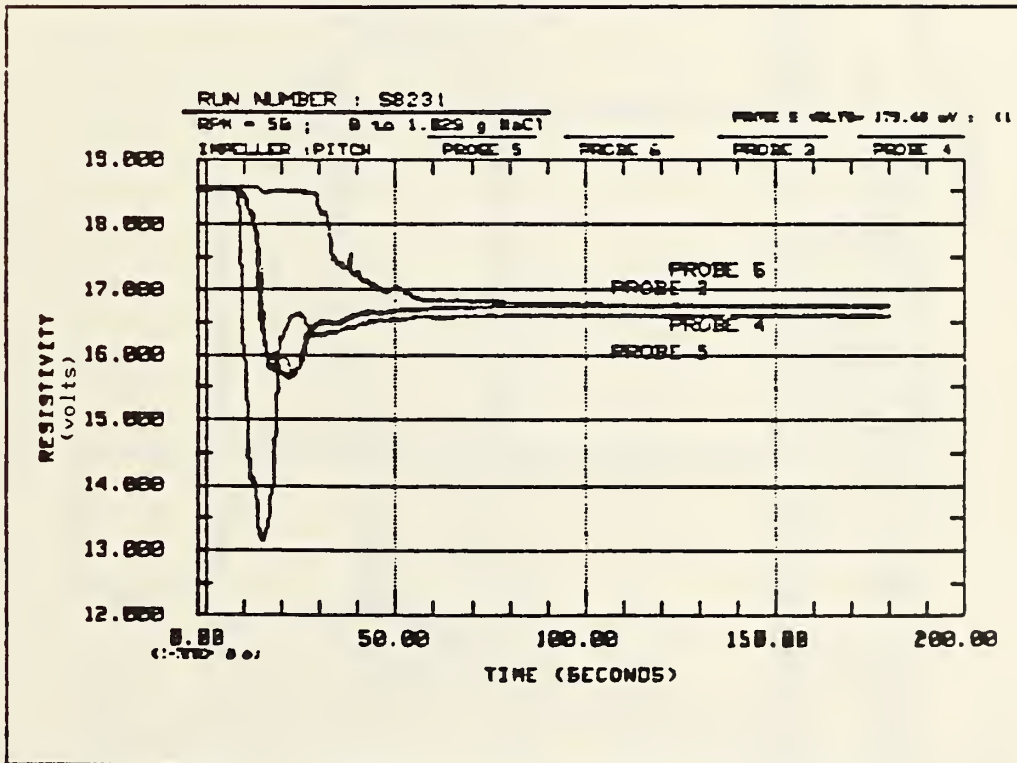


FIGURE 15

AVERAGE DATA FILE # AVG50P12

RPM = 50 ; 0 to 1.029 g NaCl

IMPELLER : PITCH

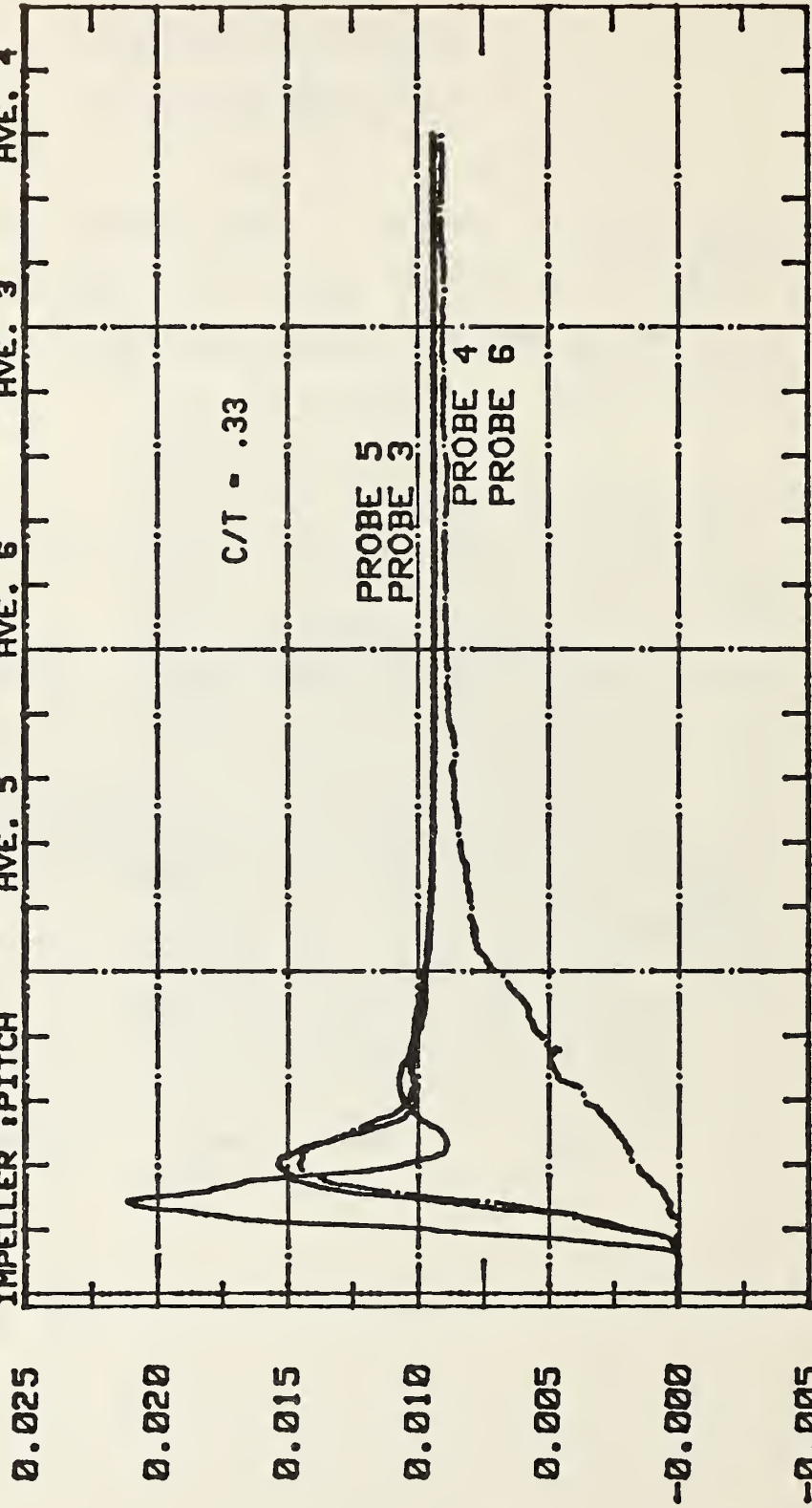
AVE. 5

AVE. 6

AVE. 3

AVE. 4

CONCENTRATION NaCl (wt. percent)



C/T = .33

PROBE 5
PROBE 3
PROBE 4
PROBE 6

0.00
(1-ZERO 0 0)

TIME (SECONDS)

FIGURE 15A (1 OF 4)

AVERAGE DATA FILE # AVGSØP12

RPM = 50 ; Ø to 1.029 g NaCl

IMPELLER iPITCH

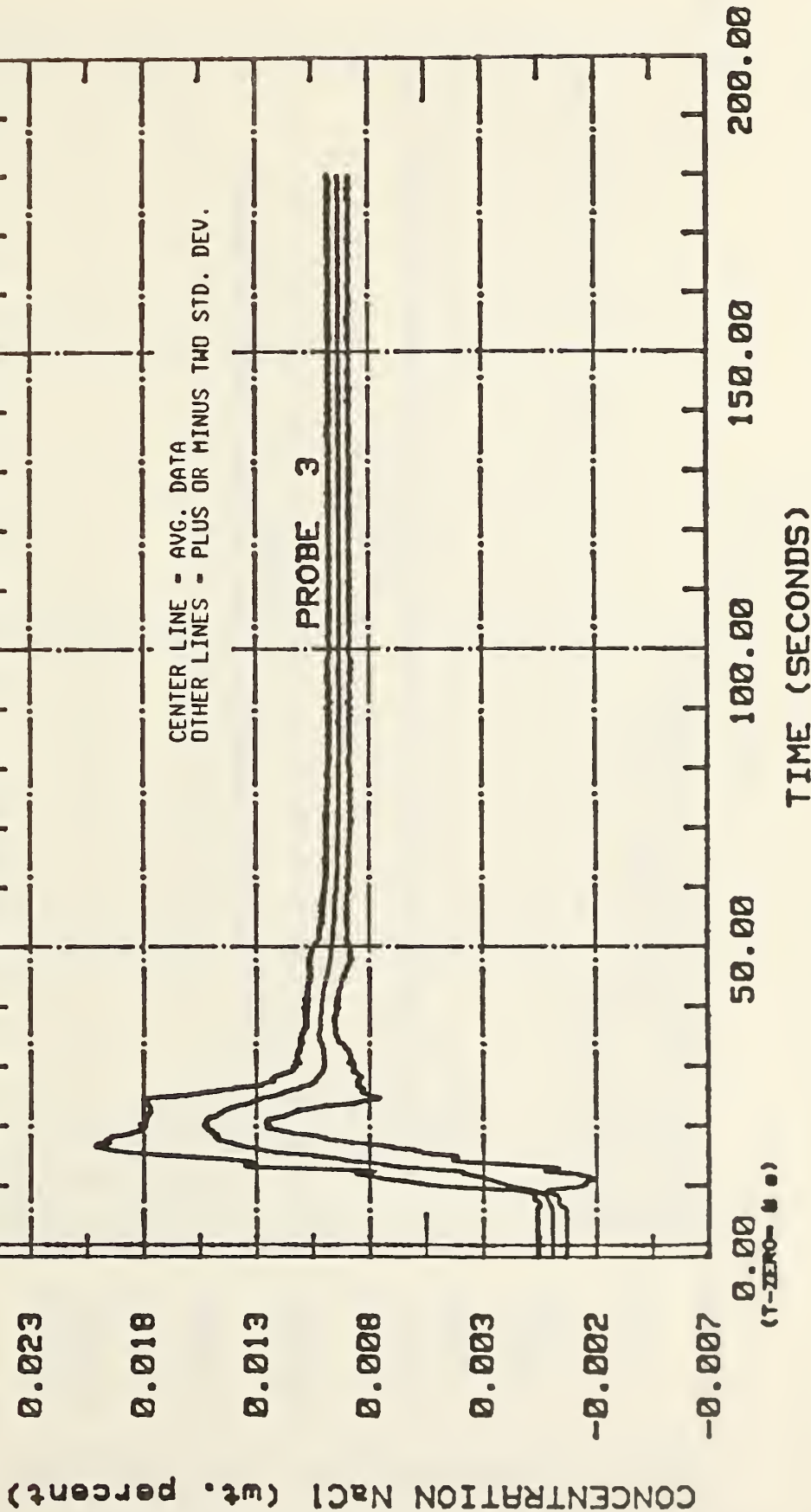


FIGURE 15A (2 OF 4)

AVERAGE DATA FILE # AVG50P12

RPM - 50 ; 0 to 1.029 g NaCl

IMPELLER : PITCH

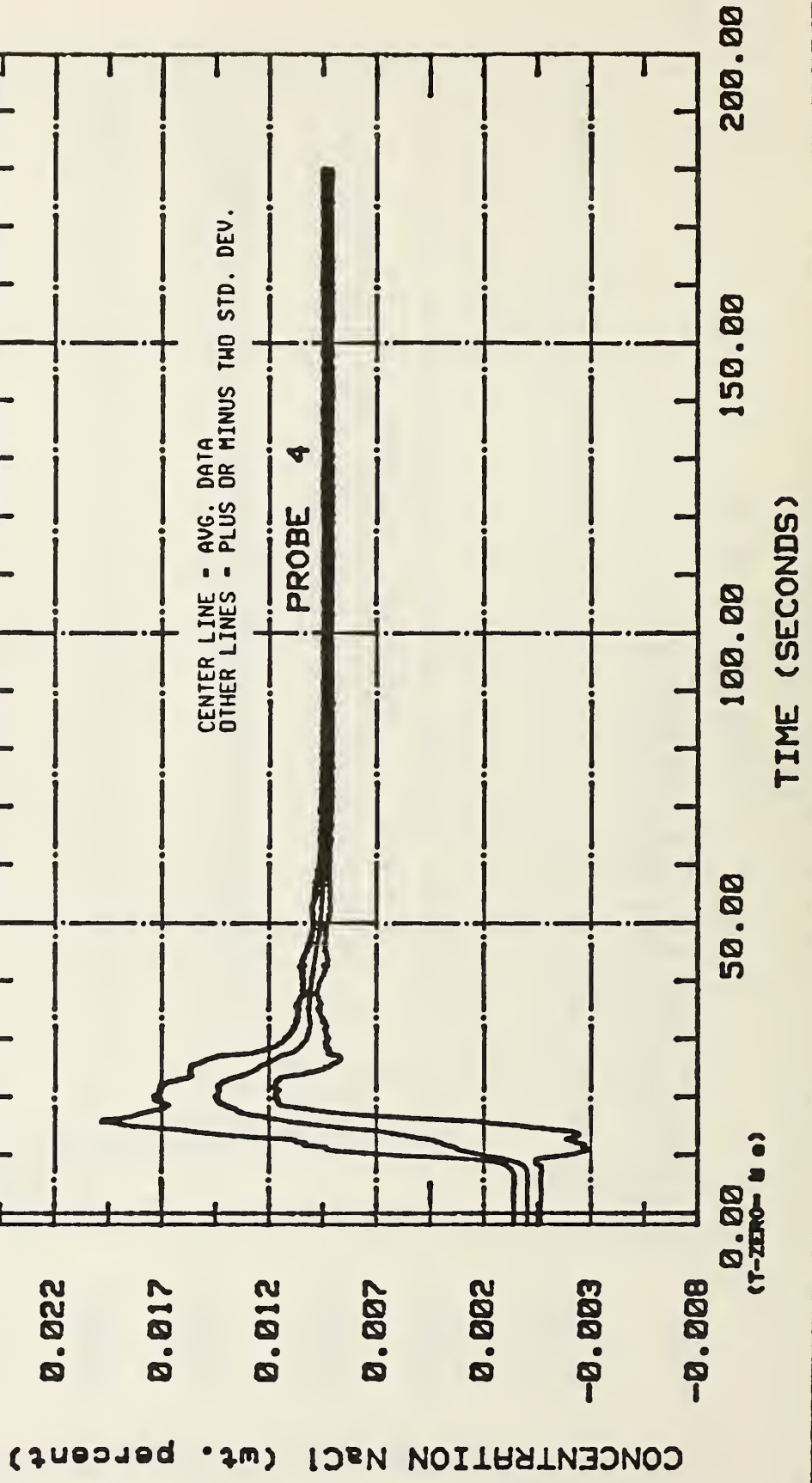


FIGURE 15A (3 OF 4)

AVERAGE DATA FILE + AVG50P12

RPM = 50 ; 0 to 1.029 g NaCl

IMPELLER : PITCH

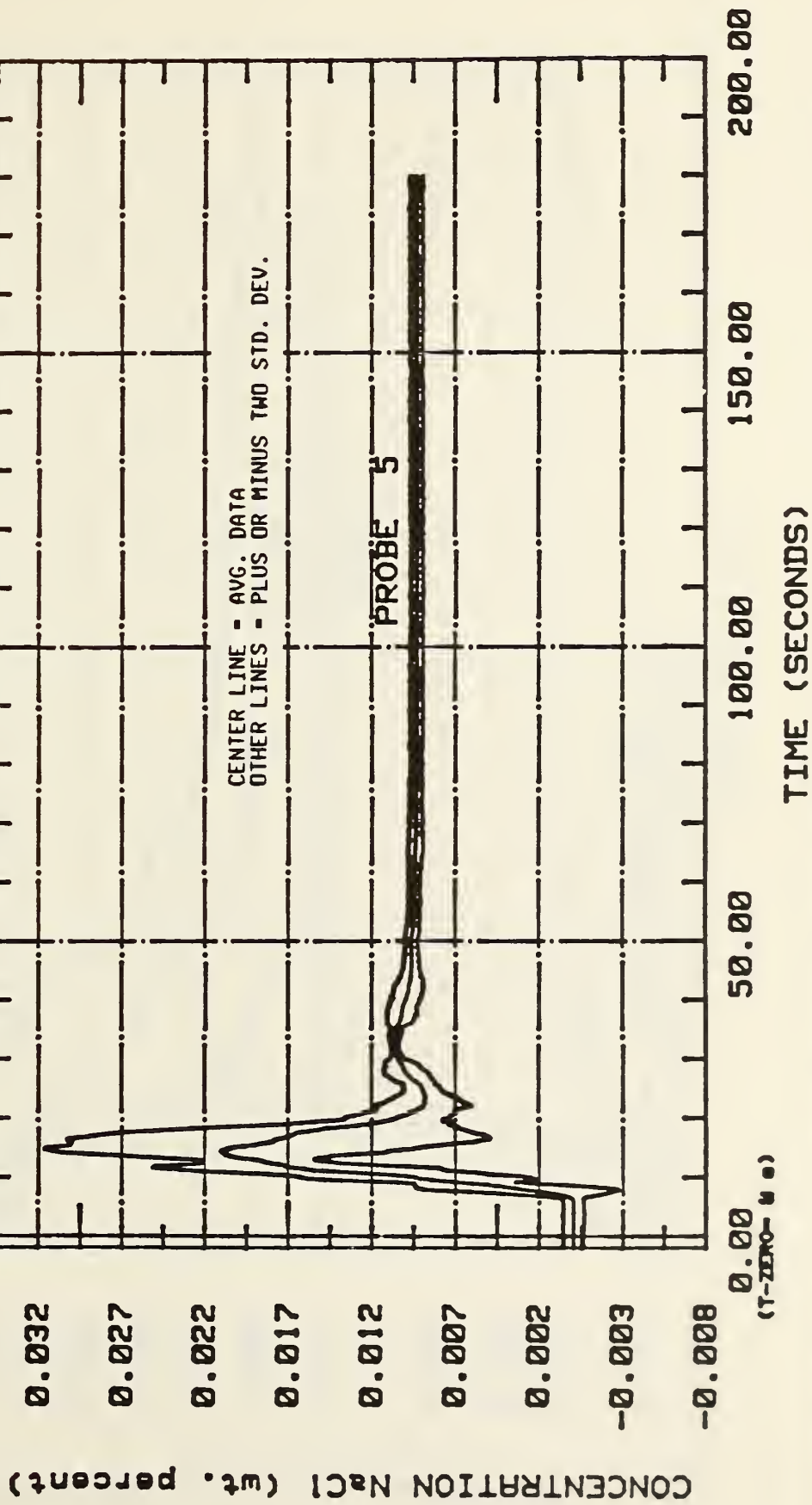


FIGURE 15A (4 OF 4)

AVERAGE DATA FILE # AVG50P12

RPM = 50 ; 0 to 1.029 g NaCl

IMPELLER : PITCH

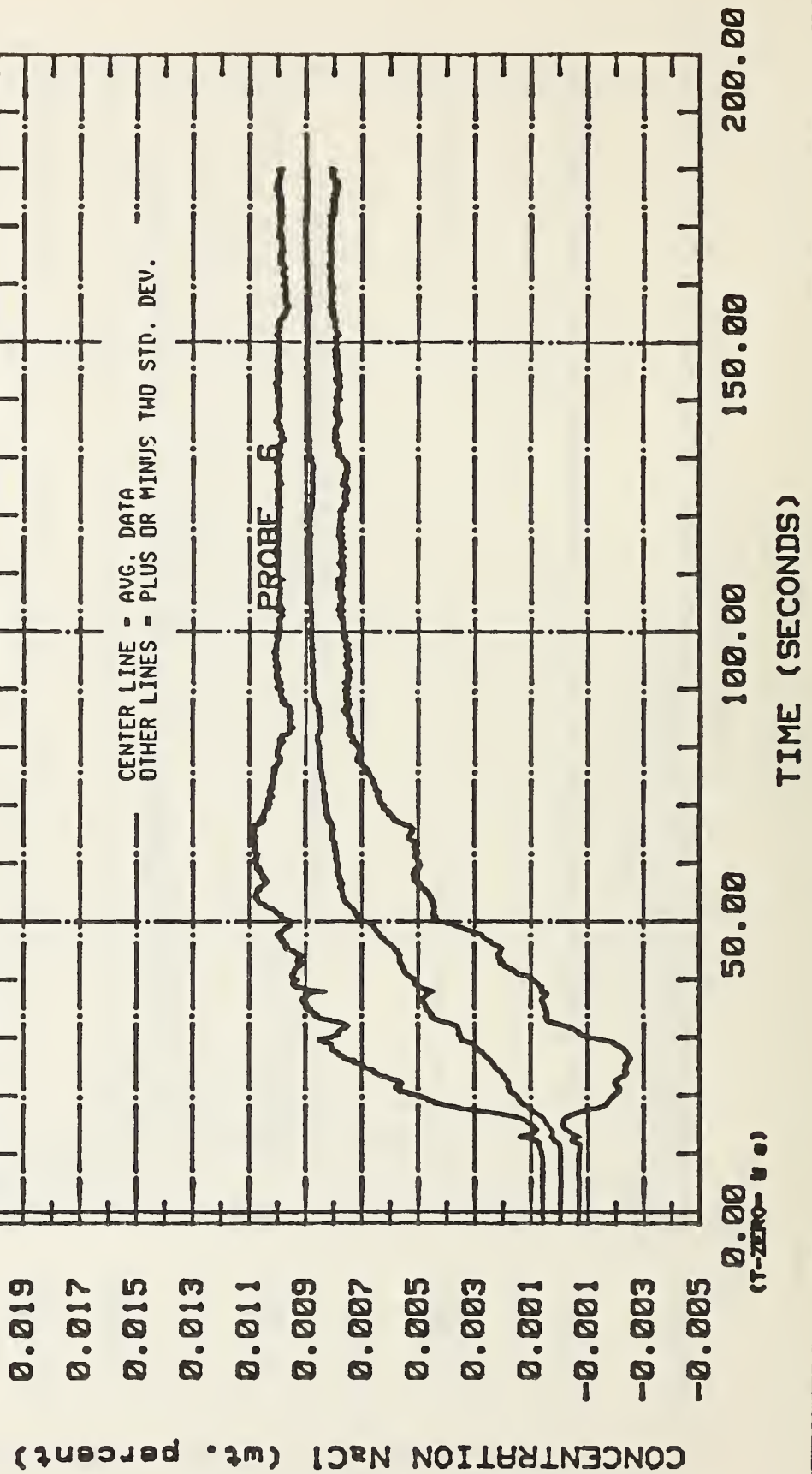


FIGURE 16

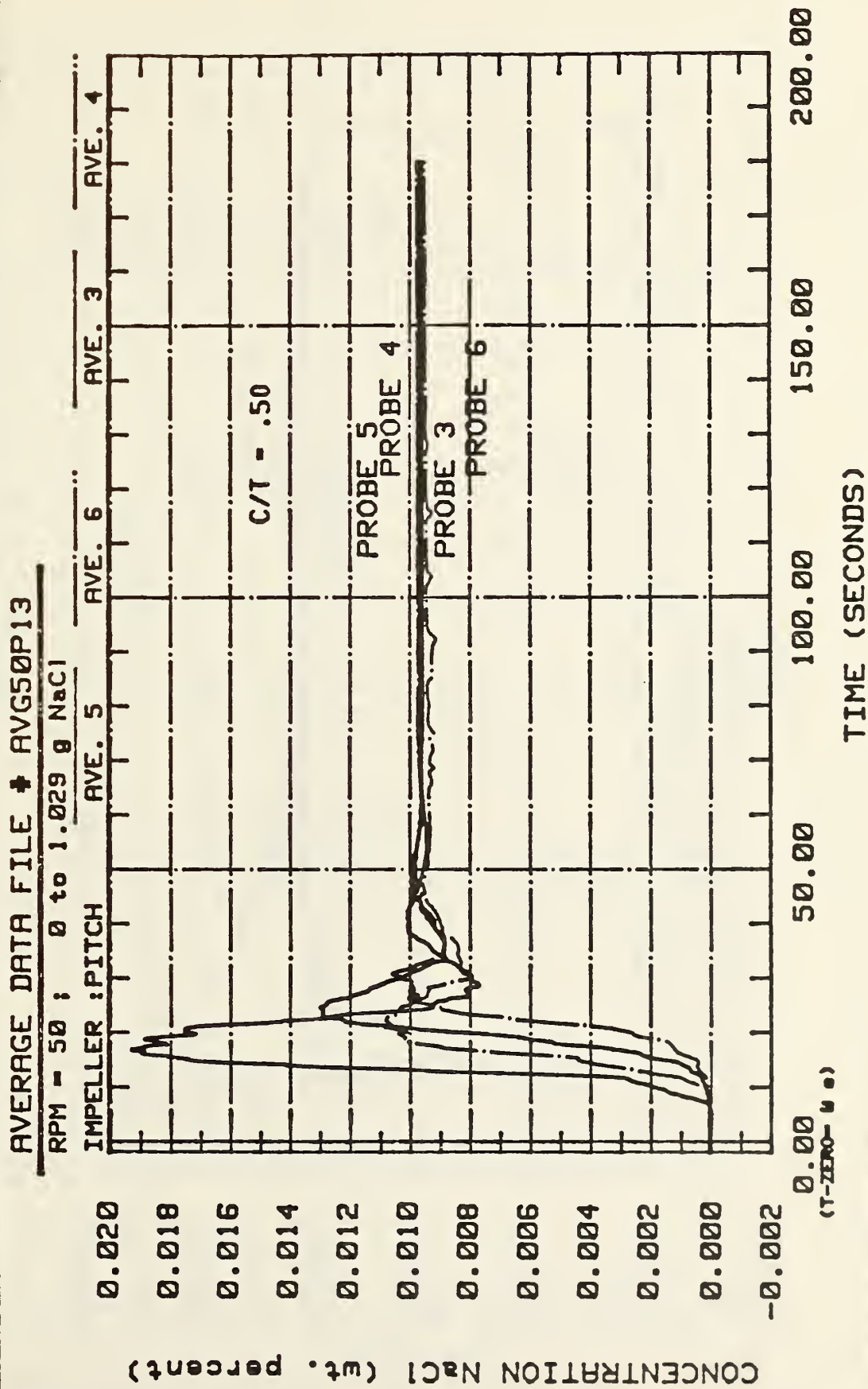


FIGURE 17

AVERAGE DATA FILE # AVG50P62

RPM = 50 ; 5.145 to 6.174 g NaCl

IMPELLER : PITCH

AVE. 5

AVE. 3

AVE. 4

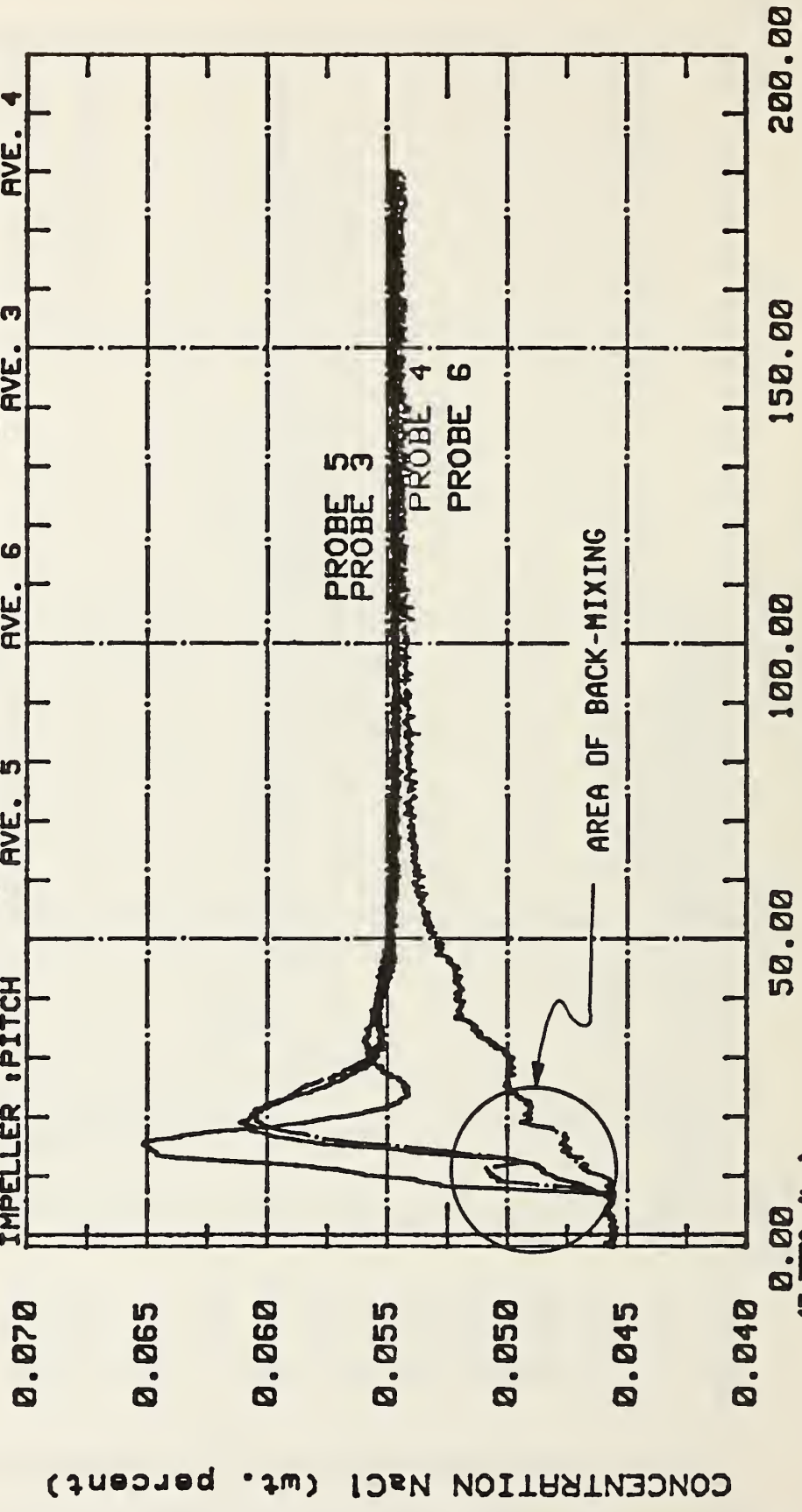


FIGURE 18

COMPARISON OF IMPELLER TYPES

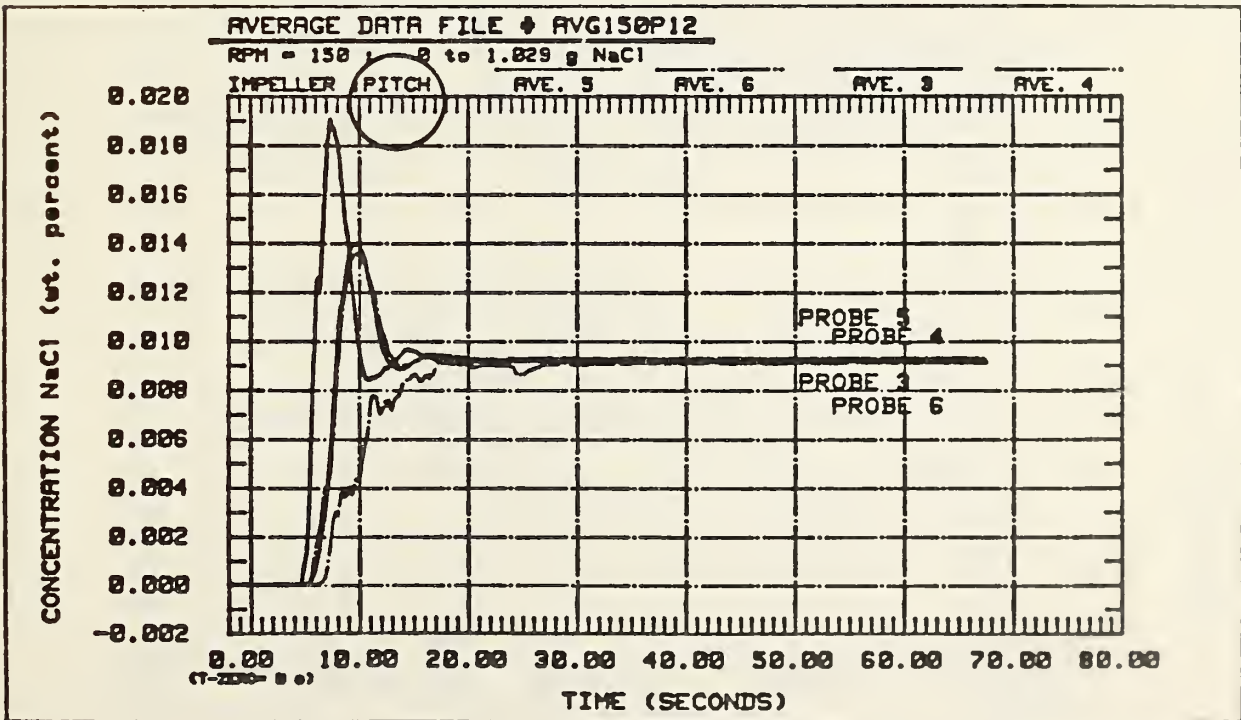
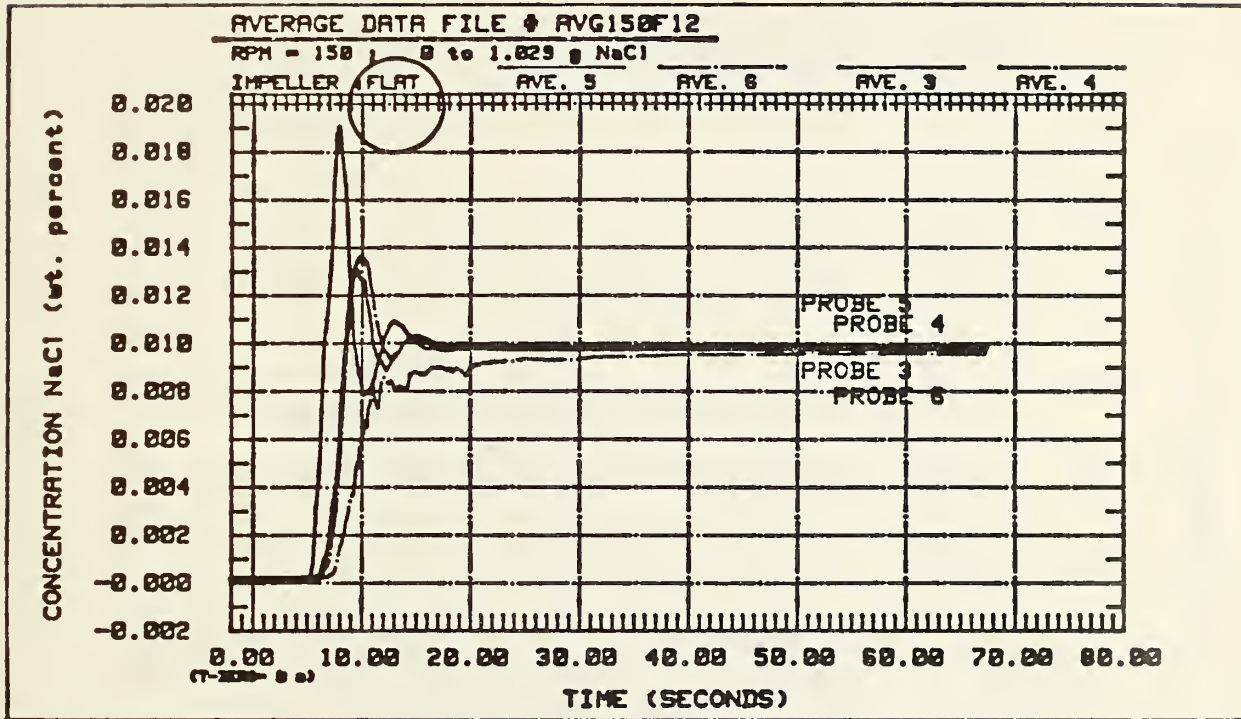


FIGURE 19

COMPARISON OF C/T VALUES

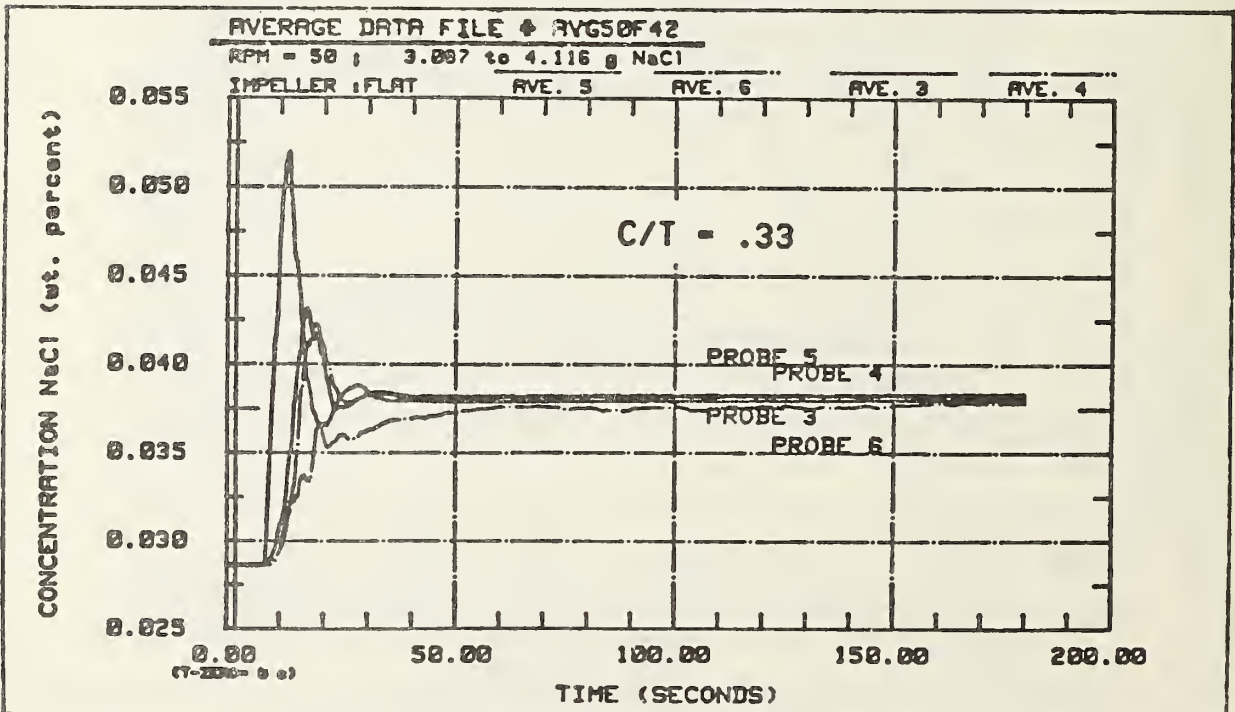
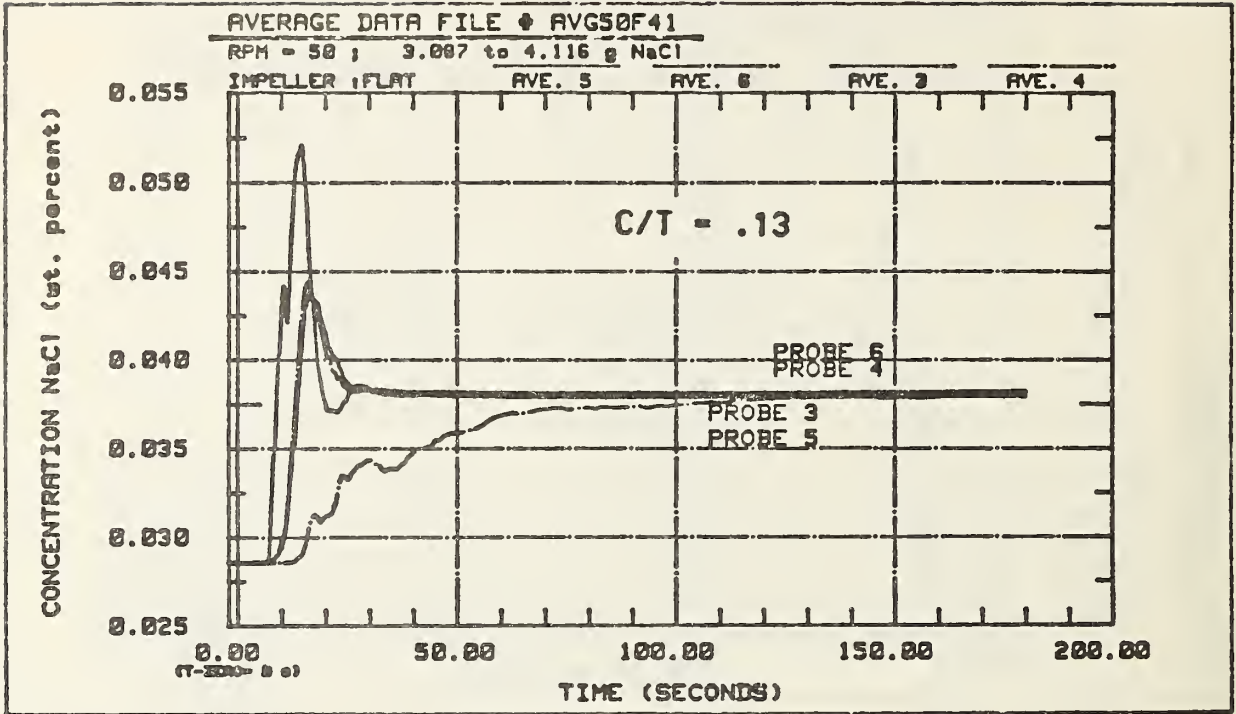


FIGURE 20

COMPARISON OF ROTATION RATES (RPM)

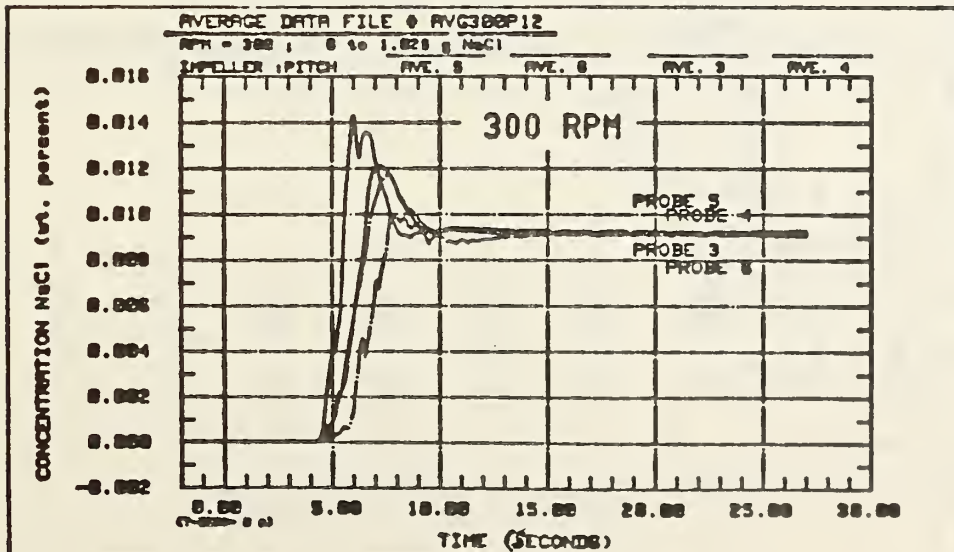
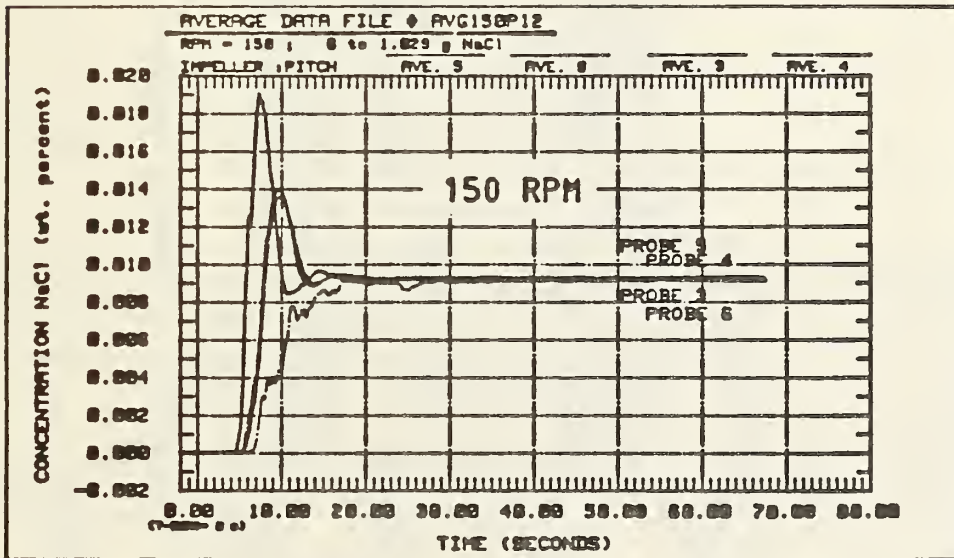
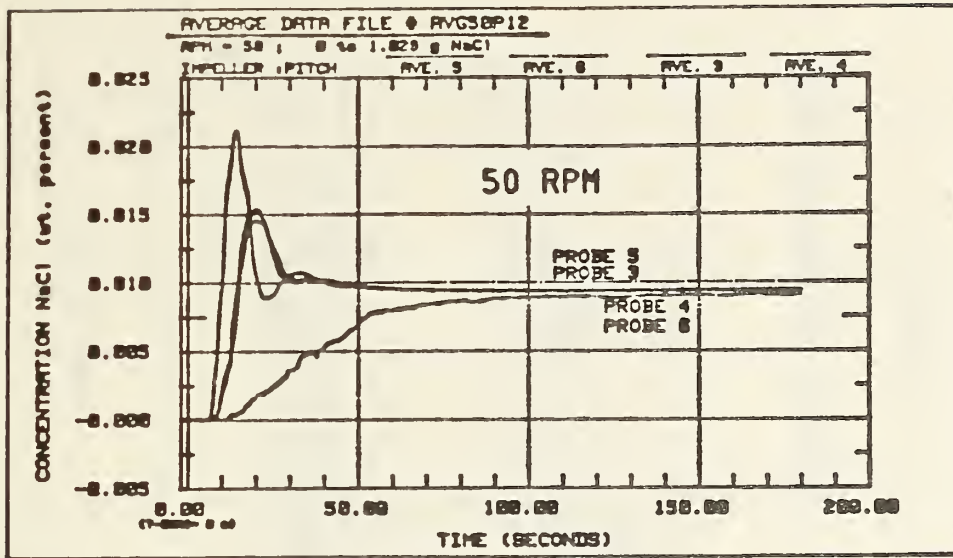


FIGURE 21

MIXTIME DETERMINATION

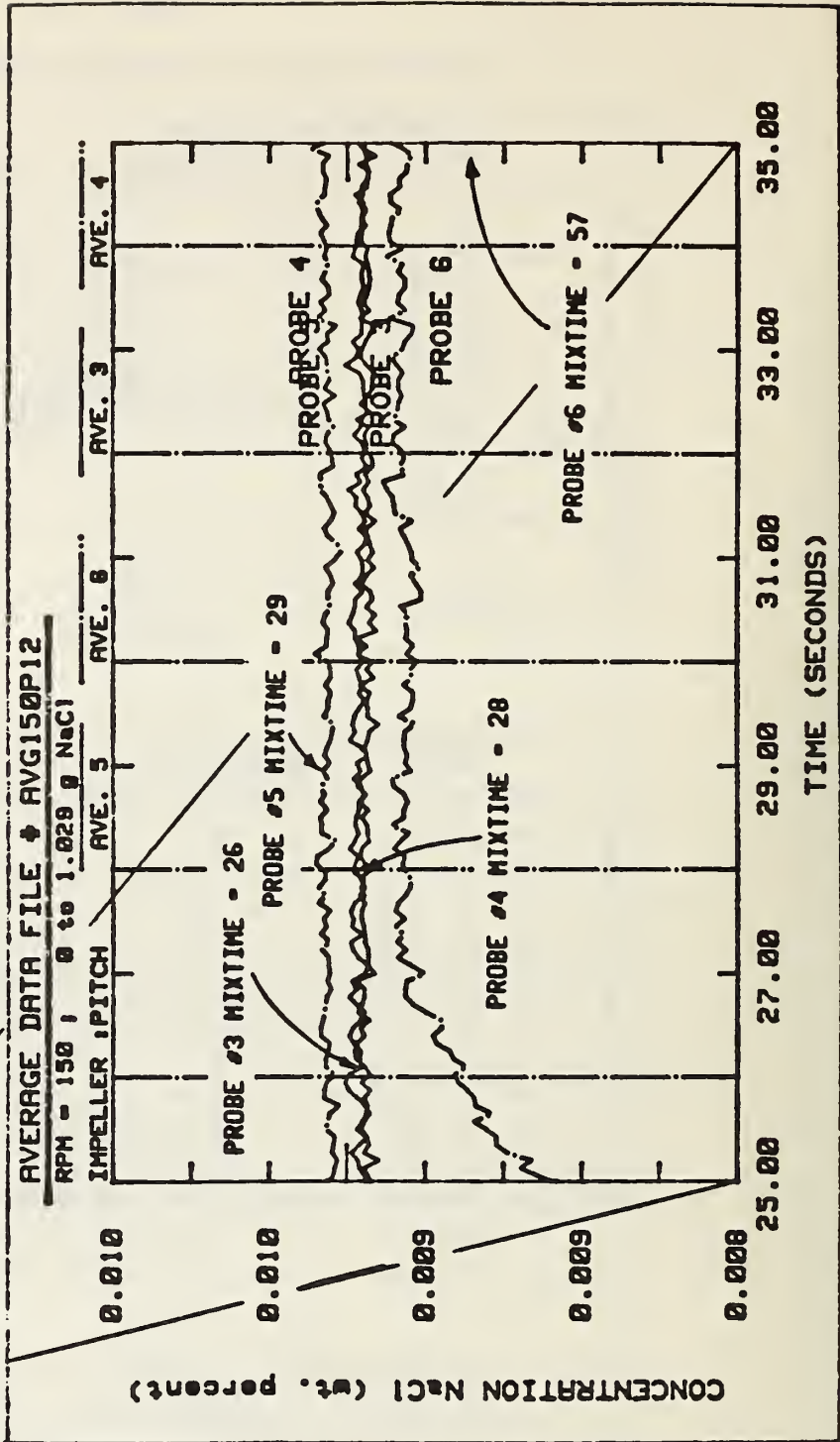
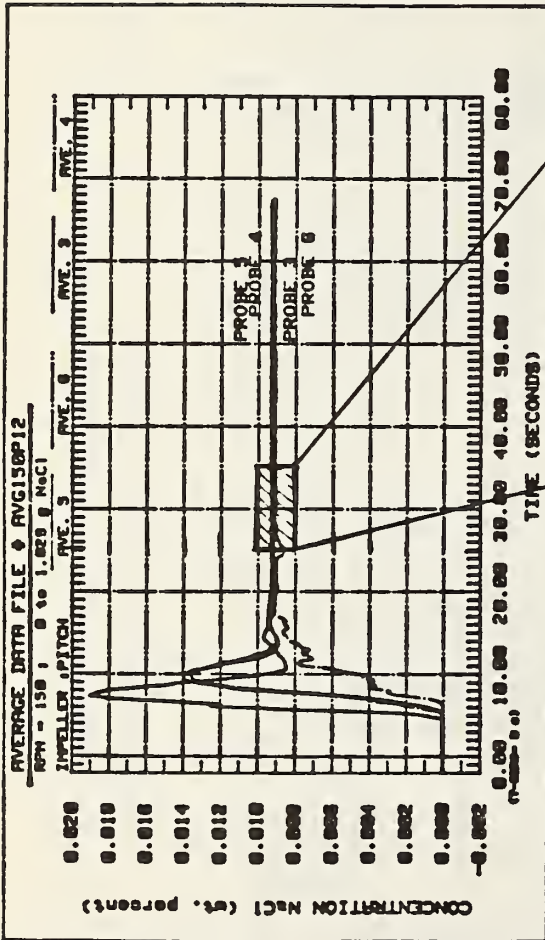
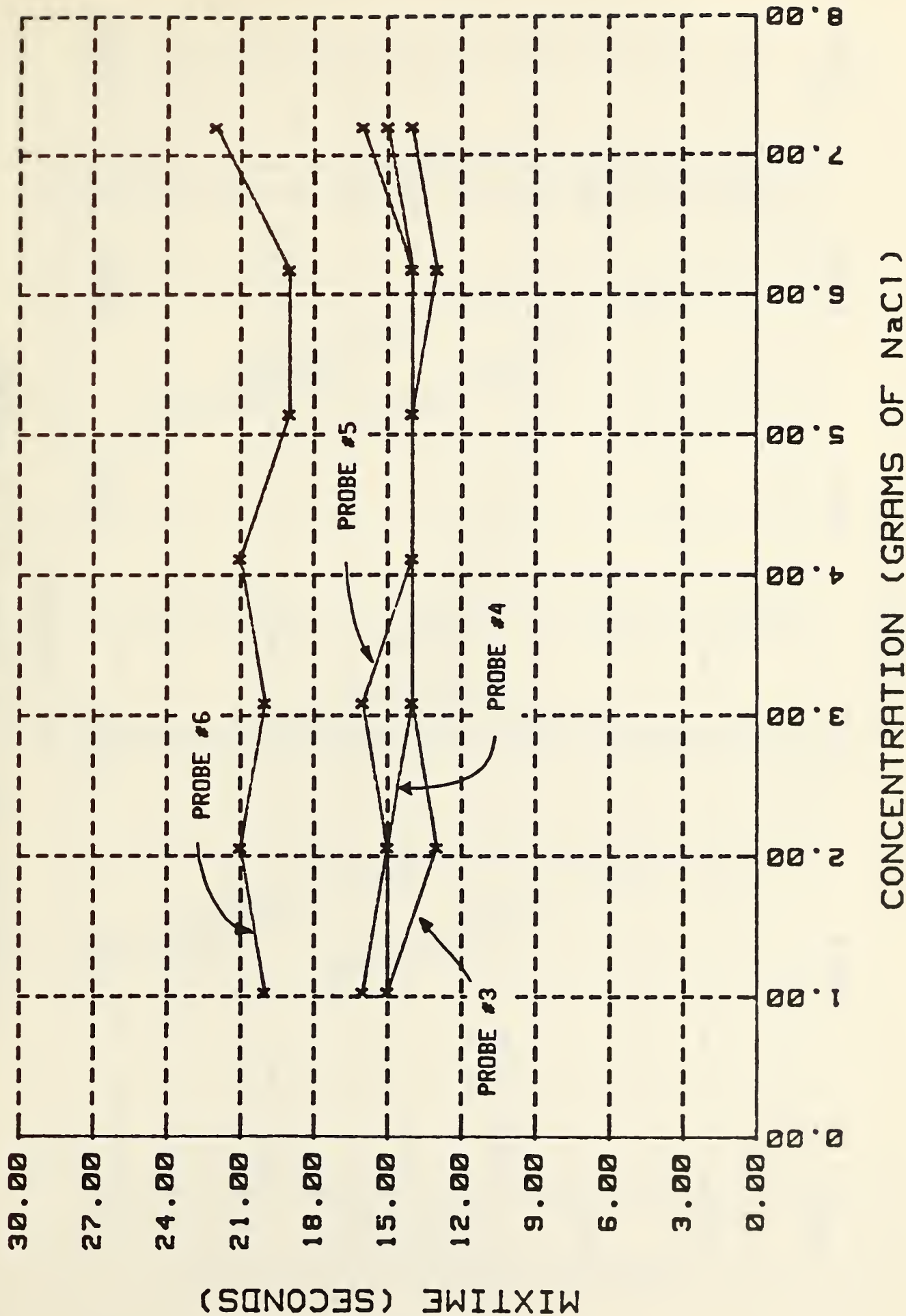


FIGURE 22

CONC. VS. MIXTIME (EXAMPLE)



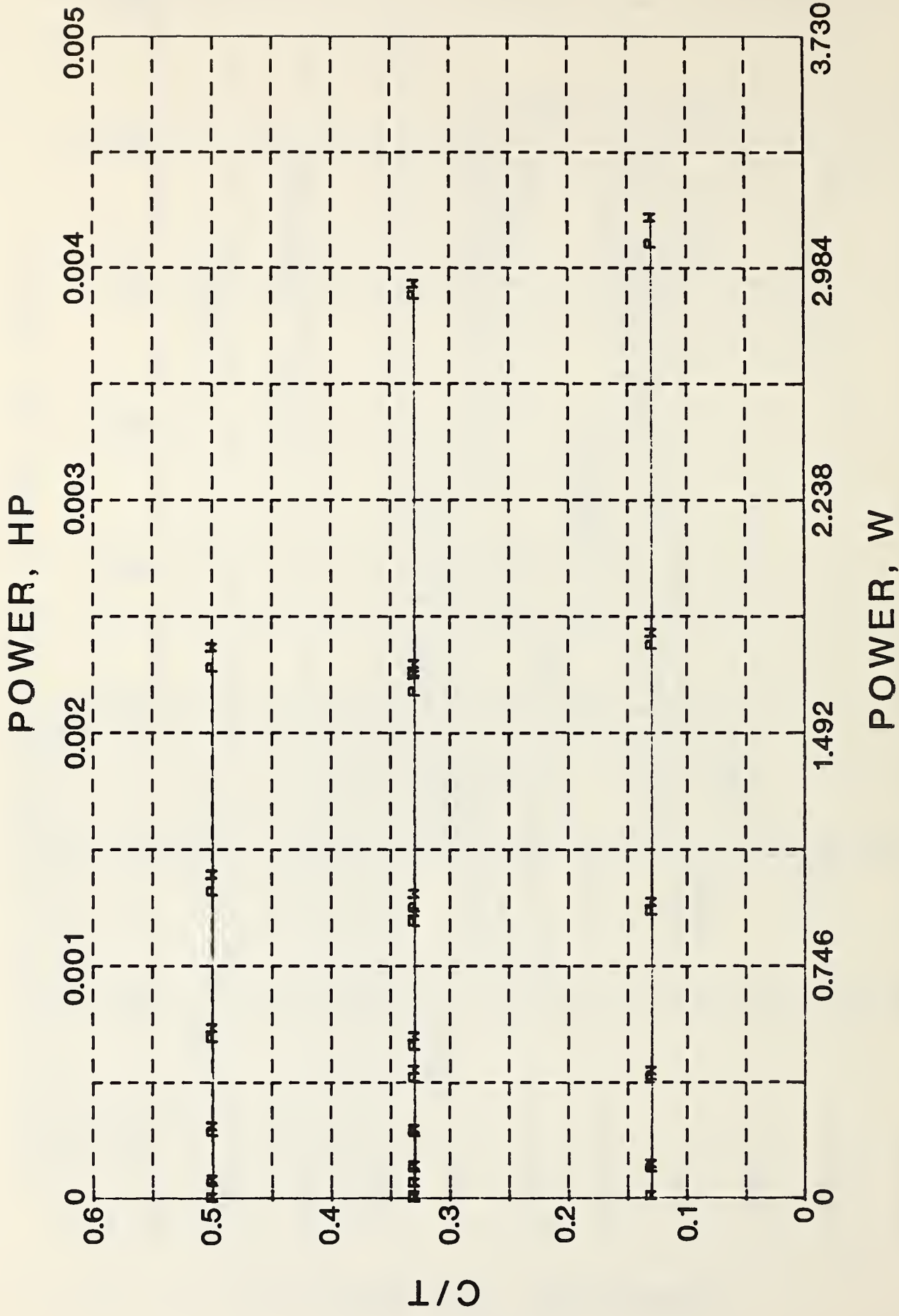


Figure 23: Power versus C/T (P = torque plate, W = weigh scale)

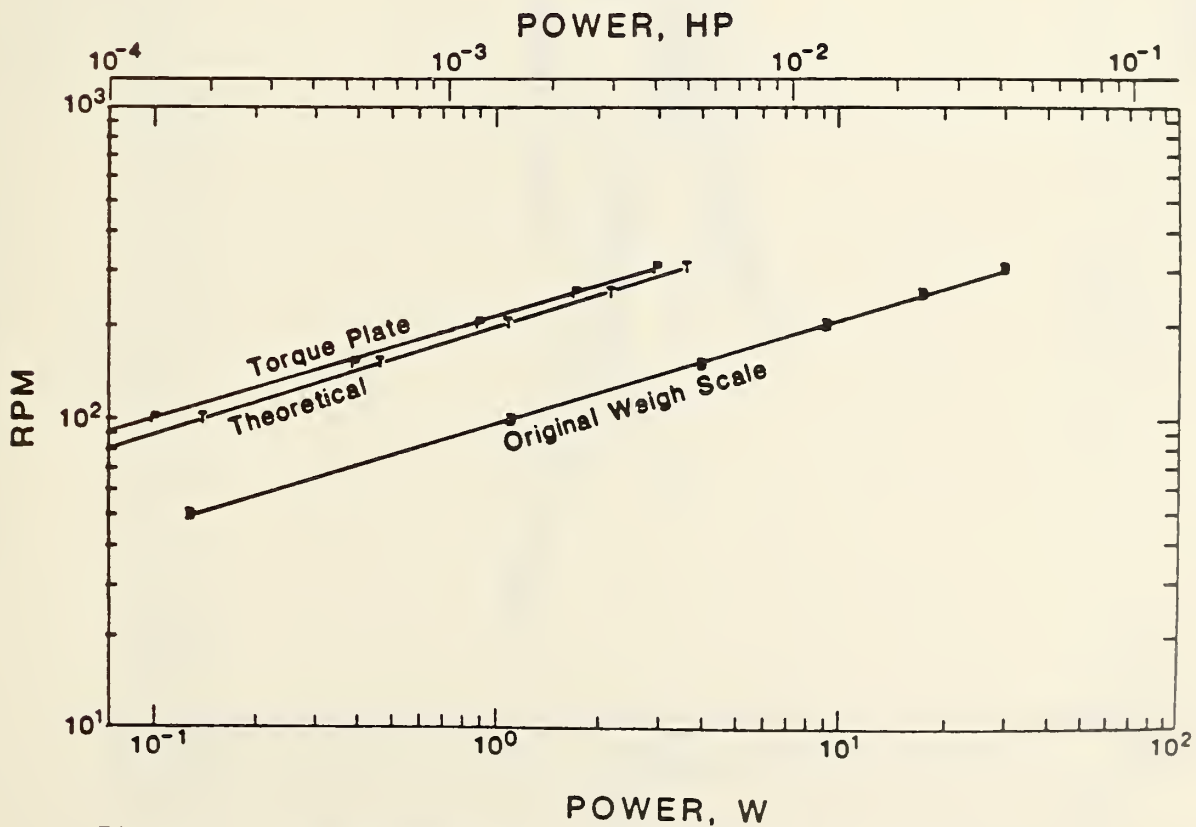
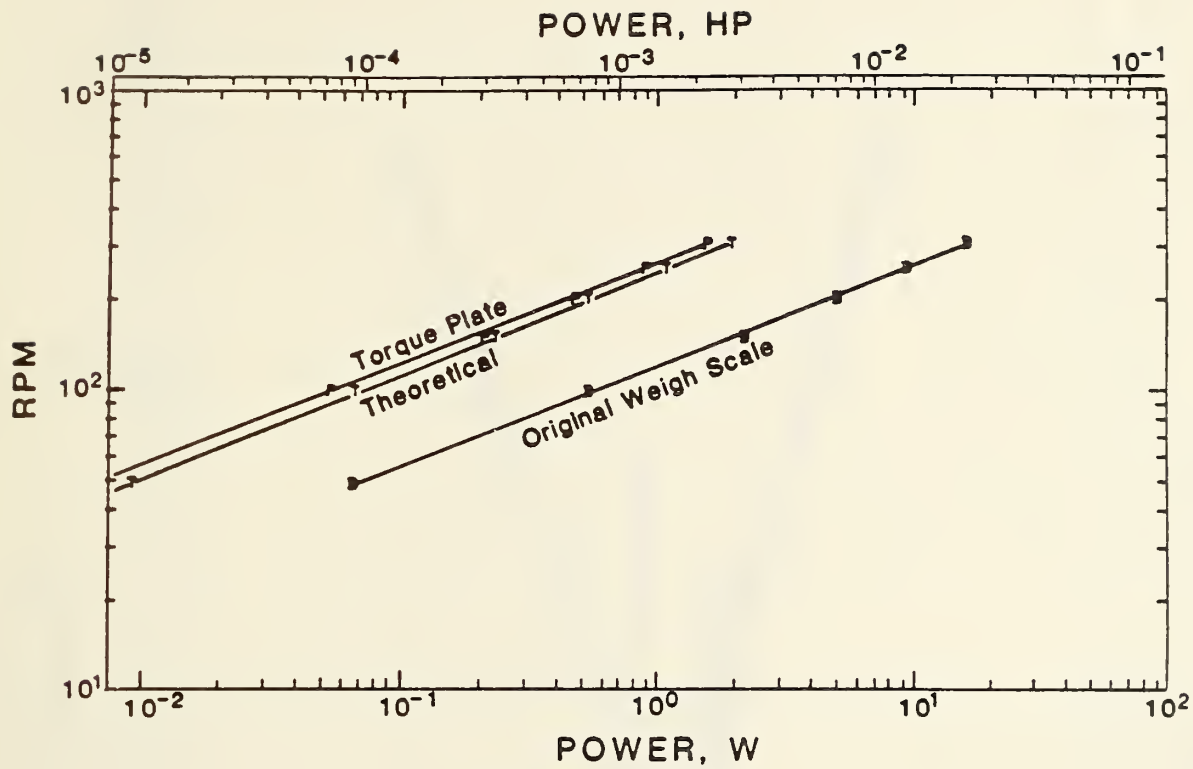


Figure 24: Power comparison. Upper graph is the Pitch Blade Impeller and the lower graph is the Flat Blade Impeller.

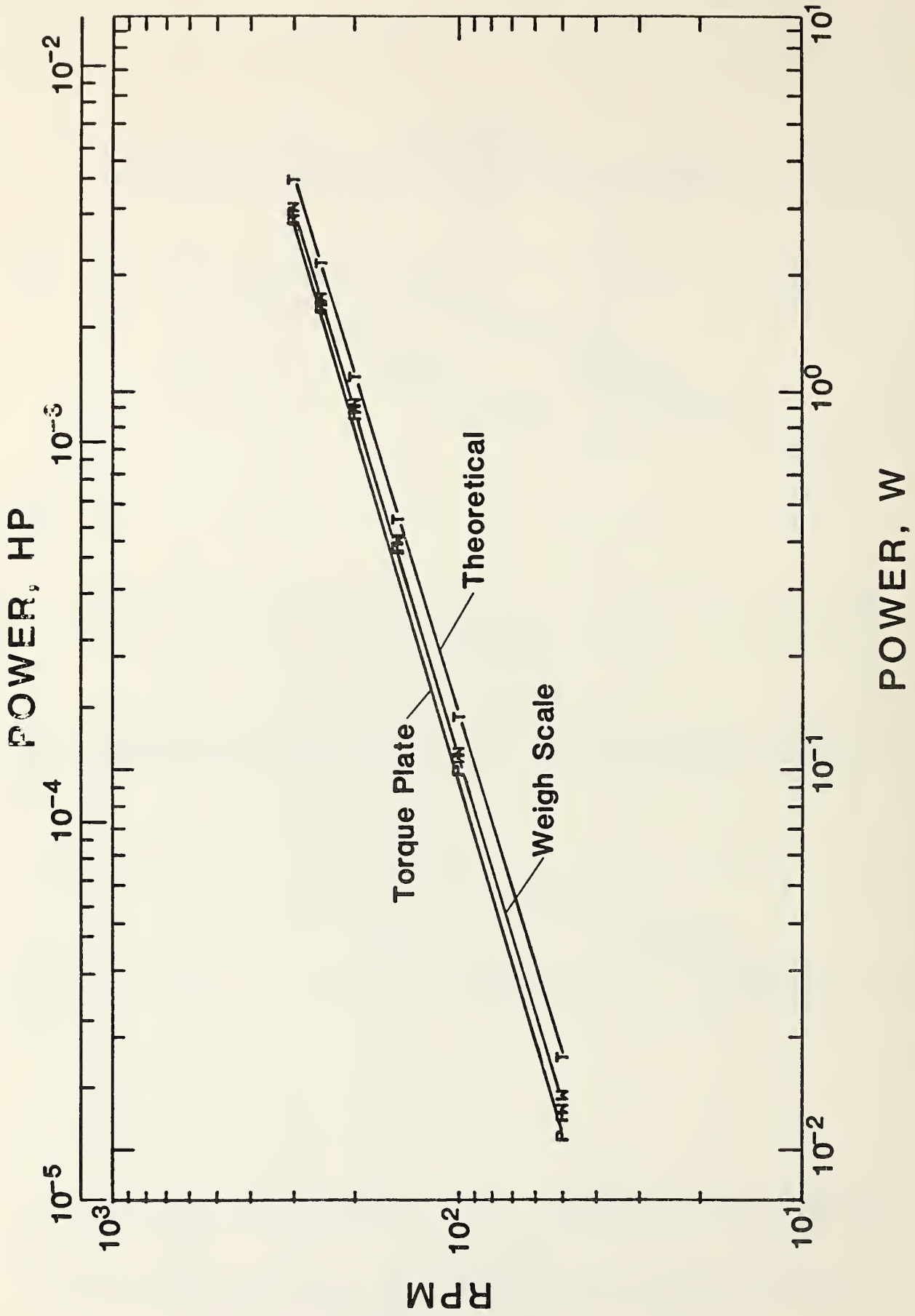


Figure 25: Power comparison Flat Blade Impeller.

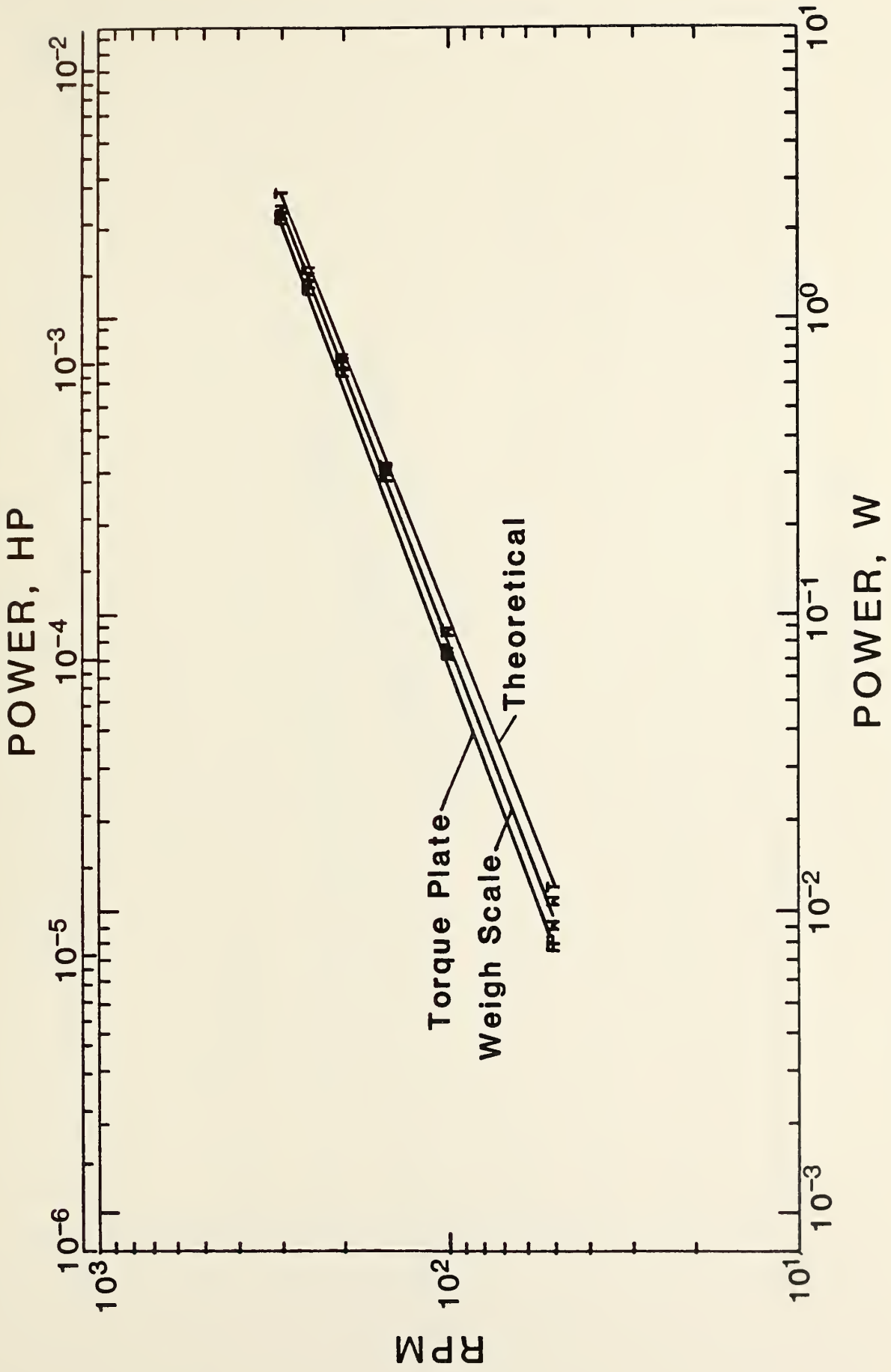


Figure 26: Power comparison Pitch Blade Impeller.

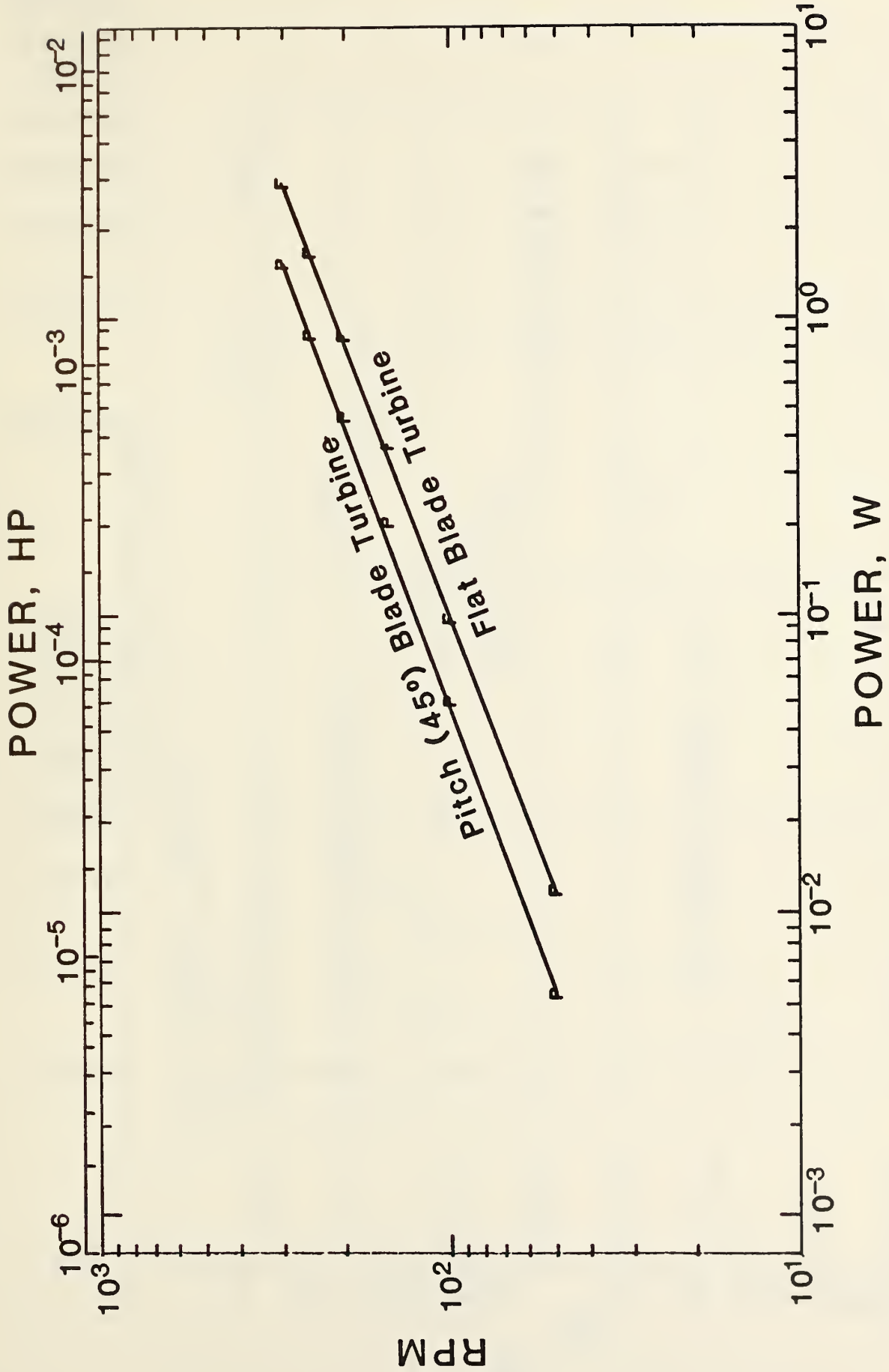


Figure 27: Power (Flat versus Pitch Blade Impeller).

FIGURE 28
 Re VS. N_p COMPARISON

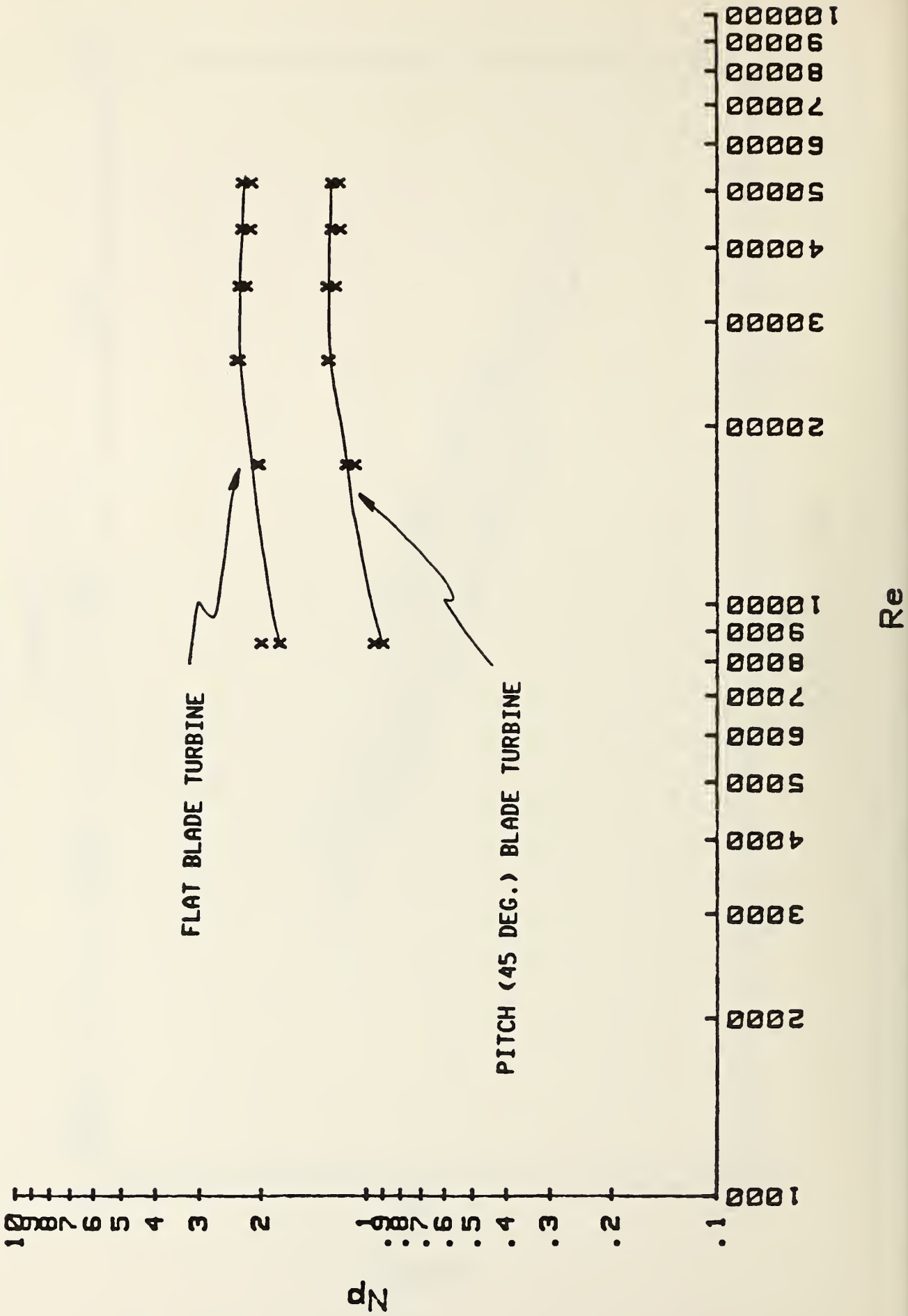
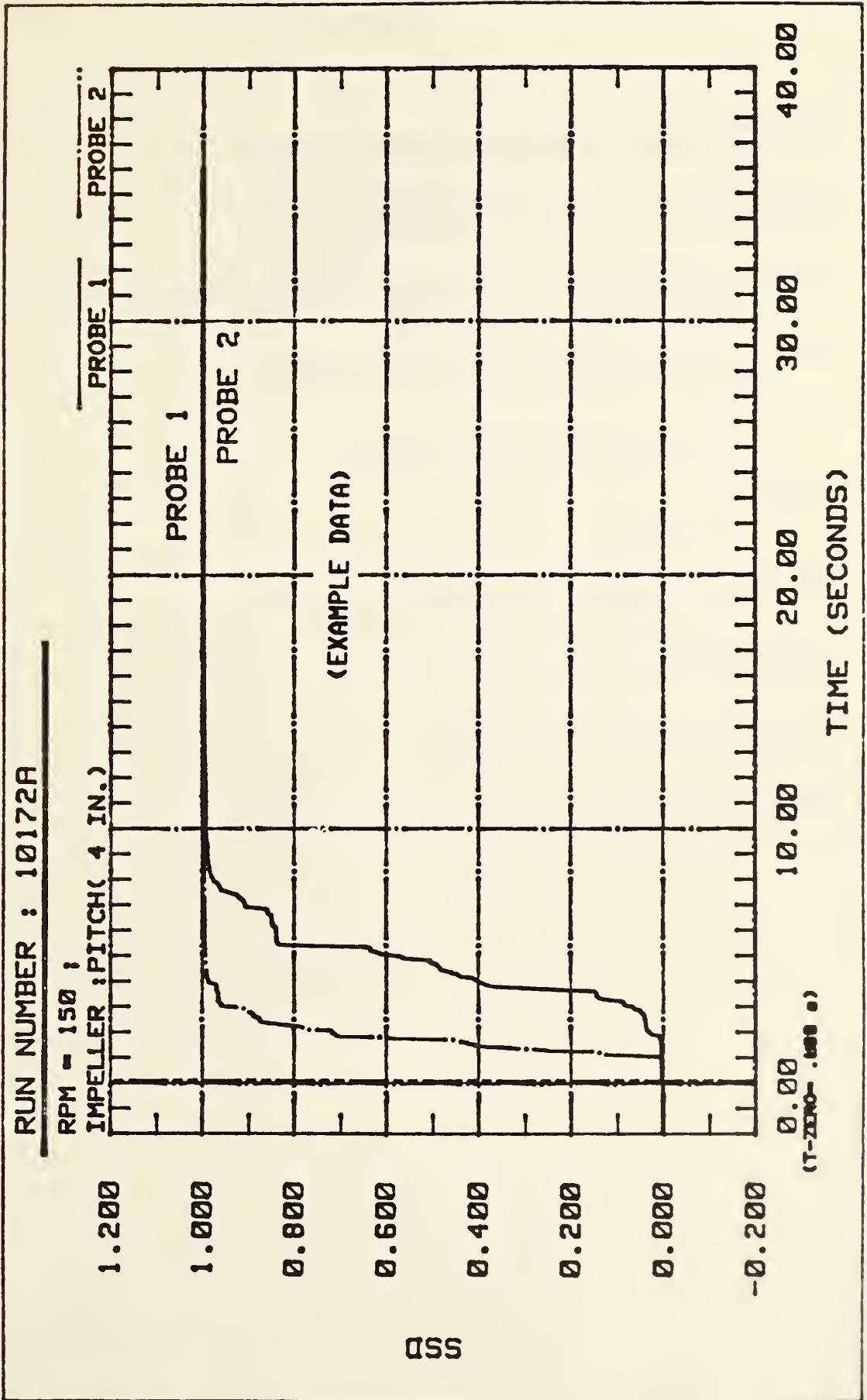


FIGURE 29

MIXTIME ANALYSIS TECHNIQUE



APPENDICES

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Appendix I
NBS Workshop on
Mixer Performance
Standards

Workshop Summary

Goal:

To develop standard test methods and measurement techniques for evaluating the performance of impeller-type mixing equipment.

Systems to be Studied in Order of Preference:

- 1) Blending (conductivity probes)
- 2) Liquid-Solid
- 3) Liquid-Liquid
- 4) Liquid-Gas

Experimental Equipment:

- 1) Existing laboratory scale equipment.
- 2) 3' diameter tank with standard type geometry ($Z/T \approx 1.0$, $C/T \approx .33$, etc.)
- 3) Large scale (to be supplied by industry).

Recommendations:

- 1) Establish Steering Committee.
- 2) Keep experiments simple.
- 3) Publish information on test methods and measurement techniques as investigated; not performance standards.

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October 27, 1983

Reply to the attention of:

773.10

David S. Dickey
Technical Director
Chemineer, Inc.
P. O. Box 1123
Dayton, OH 45401

Dear Dave:

As a result of the recent workshop, I am transmitting herein the collective views of the National Bureau of Standards-Boulder staff relative to the planned stirred tank mixing program.

Goal: To develop performance test procedures and measurement techniques for batch mixing systems.

Objective: The performance test should enhance understanding of fundamentals of mixing processes by improving mixing measurement techniques, and enabling more intelligent troubleshooting of ailing systems.

Performance Test: Performance tests should be designed so as to be repeatable by different individuals in the field. Tests should specify:

- 1) Definition of mixing system (i.e., liquid-liquid, liquid-solid, etc.)
- 2) System geometry
- 3) Probe type, number and location
- 4) Procedure for data collection
- 5) Method(s) for data analysis (including the possibility of experimentally verified modeling)
- 6) Components to be mixed

Physical System at NBS-Boulder:

Emphasis should be on inexpensive simple fixed geometries.

- 1) Two transparent tank sizes (1' and 3' diameters)*
- 2) Z/T = .8 to 1.5
- 3) C/T = approximately 0 to .5

- 4) D/T = .25 to .5 (in three steps)
- 5) Four baffles, 90° spacing, minimal bottom and side clearance
- 6) Tank bottom to be flat or dished (possibility for one of each)
- 7) Impellers = one flat, one pitch (45°) four blades w/d = 1/5 for pitch and 1/6 for flat
- 8) Vertical shaft centrally located for single impeller (dual impellers to be considered at a later date)
- 9) Motor to establish 15 HP/1000 gal (i.e., approximately 5 HP motor for three foot diameter tank)

* Most tests to be performed on three foot diameter tanks, then one foot diameter tanks with an occasional spot check on a one liter tank and very large diameter tank. Large tests to be performed cooperatively with industry.

Mixing System at

NBS-Boulder:

- 1) Initially single phase fluids will be used. Viscosity and density will be varied such that the range of the Reynolds number will be between 200 and 20000 at three fixed RPM values.
- 2) Multiphase systems will be as simple as possible, including macro mixing for solid-liquid, immiscible liquids, and then gas-liquid systems. These experiments may be qualitative (i.e., designed for general understanding).
- 3) The selection and use of probes in the above mentioned systems will satisfy the goal and objective of the program.

Possible Probes

to be

Investigated:

- 1) Conductivity
- 2) PH
- 3) Fiber Optics
- 4) Fluorescence
- 5) Viscosity
- 6) Temperature
- 7) Hot-wire anemometers
- 8) Redox reactions

Independent Variables

for Data Analysis

and Scaling:

- 1) Torque measurement
- 2) Viscosity
- 3) Density
- 4) RPM
- 5) Impeller geometry
- 6) Injection location

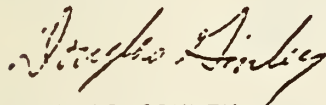
Immediate Suggested

Plan for NBS:

- 1) Continue work with conductivity probes keeping in mind the new goal agreed upon at the workshop.
- 2) Develop a three foot diameter tank facility as quickly as possible for further testing.
- 3) Establish a Steering Committee for annual progress reviews.
- 4) Develop a communications system for relating information, data, and problems to the Steering Committee.

If you have any questions or comments, please feel free to call me at (303)497-3521.

Sincerely,



DOUGLAS GINLEY
Chemical Engineering Science Division
Center for Chemical Engineering

Appendix II
Moderate Scale
Facility Design

MIXER SPECIFICATIONS

- 1) Tank sizes are 24" to 36" diameters
- 2) Mixer is to be mounted on an A-frame type structure
- 3) Mixer should be capable of being used with a reaction torque ring
- 4) Shaft is to be a large diameter (1.5-2"), with Jacobson drill-chuck on the end. Shaft should be capable of having a strain guage mounted on it, and RPM values read from it.
- 5) A smaller shaft (.5-1") for tank entry should be designed. It should have an appropriately sized impeller hub designed for changable blades. A number of different impellers is also acceptable.
- 6) Two types of impellers will be used, each with four blades. A) Flat blade turbine ($w/d=1/6$) B) 45 Pitch blade turbine ($w/d=1/5$)
- 7) Motor and drive design criteria:
 - RPM range - approximately 50 to 1000
 - viscosity - 1 to 20000 cp
 - D/T - .25 to .5 (3 steps)
 - Z/T - .8 to 1.5 (1.0 for 36" tank)
 - Power - to at least 15 Hp/1000 gal
 - C/T - 0 to .5
 - motor - prefer AC (possibility of adjustable frequency drive), but is not essential

TANK DESIGN

36" Plexiglass Tank

44" high plexiglass to form 36" ID tank (1/2" wall thickness)

3/4" thick bottom plate notched for 36" ID tank, to be glued and screwed to plate, offcenter drain hole

3/4" thick top ring, notched for 36" ID tank, screwed into place

all seams to be reinforced

MIXER SPECIFICATIONS

- 1) Tank sizes are 18" to 24" diameters
- 2) Mixer is to be mounted on an A-frame type structure
- 3) Mixer should be capable of being used with a reaction torque ring
- 4) Shaft is to be a large diameter (1.5-2"), with Jacobson drill-chuck on the end. Shaft should be capable of having a strain gauge mounted on it, and RPM values read from it.
- 5) A smaller shaft (.5-1") for tank entry should be designed. It should have an appropriately sized impeller hub designed for changable blades. A number of different impellers is also acceptable.
- 6) Two types of impellers will be used, each with four blades. A) Flat blade turbine ($w/d=1/6$) B) 45 Pitch blade turbine ($w/d=1/5$)
- 7) Motor and drive design criteria:
 - RPM range - approximately 50 to 1000
 - viscosity - 1 to 20000 cp
 - D/T - .25 to .5 (3 steps)
 - Z/T - .8 to 2.0 (1.5 for 24" tank)
 - Power - to at least 15 Hp/1000 gal
 - C/T - 0 to .5
 - motor - prefer AC (possibility of adjustable frequency drive), but is not essential

TANK DESIGN

18" Plexiglass Tank

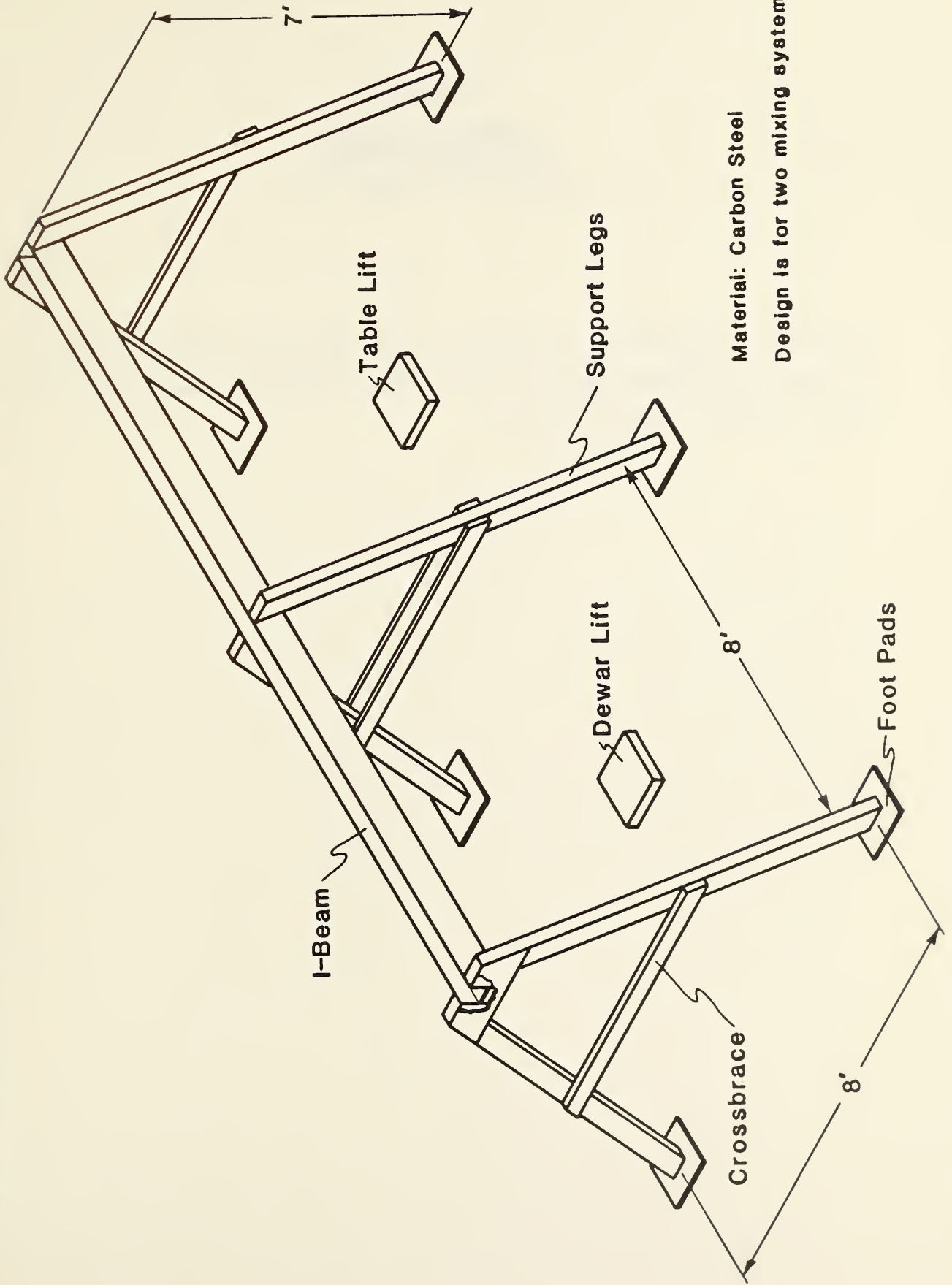
36" length of 18" ID plexiglass pipe (1/4-3/8" wall thickness)

1/2" thick bottom plate notched for 18" ID pipe, offcenter drain hole, plate to be glued and screwed to pipe

1/2" thick top ring notched for 18" ID pipe (screwed into place)

1/2" thick additional ring for midposition up pipe length (if necessary)

Preliminary Structural Design for Mixing Facility



Material: Carbon Steel

Design is for two mixing systems

Appendix III

Computer Program for Laboratory Scale Batch Mixing Experiments

The computer program's use is outlined in the procedure section of this paper. For further information concerning the computer program or a listing of the program, please contact the author.

Appendix IV
Immiscible Liquid
Dispersion Experiment

Immiscible Liquid Dispersion Experiment

Materials:

Organic Phase: Methylene Chloride
Surfactant: Sodium Dodecyl Sulfate

Purpose:

To determine the affect of different power levels of mixing on an organic phase dispersed in an aqueous phase. Further, to test the affect of a surfactant on that dispersion.

Procedure:

- 1) Fill the tank with a 50% organic solution ($Z/T \approx 1.0$).
- 2) Place impeller in the organic phase ($CH_2 Cl_2$ spgr.= 1.33).
- 3) Photograph the dispersion at various RPM values and different tank levels.
- 4) Repeat using a surfactant.

Analysis:

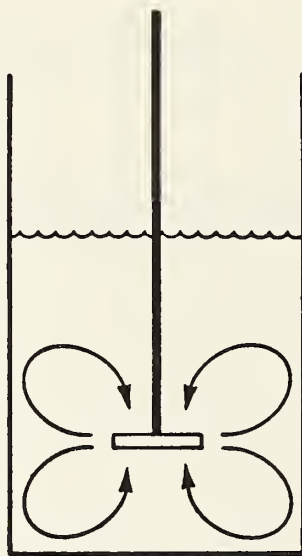
Enlarge photographs and manually determine droplet size and number/area being studied.

Appendix V
Flow Visualization
Experimentation

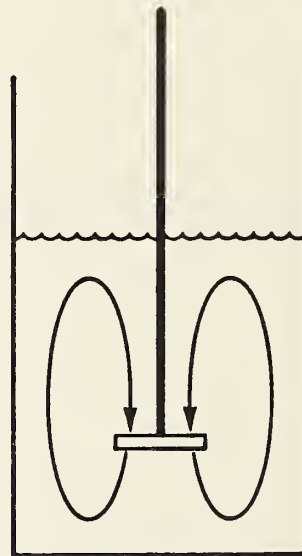
FLOW VISUALIZATION

Flow visualization studies were performed in the laboratory equipment under various conditions of rpm, C/T and impeller type. The purpose of the study was to gain a basic understanding of the mixing process, determine conditions that produced either axial or radial flow, and enhance general mixing knowledge needed for appropriate experimental design.

Flow visualization tests were done using a red acrylic modeling powder in water. Rotation rates from 50 to 600 rpm were observed. A flat blade turbine and a pitch (45 deg) blade turbine were used at C/T values ranging from 0.13 to 0.50. Observation of the flow patterns were observed visually and recorded with video recording equipment. Knowledge gained from these tests was used to help design the experiments.



Radial Flow



Axial Flow

Appendix VI
Mixtime Data

MIXTIME DATA
(SECONDS)

IMPELLER	C/T	RPM	CONCENTRATION (GRAMS OF NaCl)	MIXTIME						
				PROBE #1	PROBE #2	PROBE #3	PROBE #4	PROBE #5	PROBE #6	
1-FLAT										
2-PITCH										
1	.13000	50.00000	1.02900	70.00000	70.00000	70.00000	70.00000	70.00000	70.00000	167.00000
2	.13000	50.00000	2.05800	55.00000	55.00000	55.00000	55.00000	55.00000	55.00000	180.00000
3	.13000	50.00000	3.08700	57.00000	57.00000	57.00000	57.00000	57.00000	57.00000	180.00000
4	.13000	50.00000	4.11600	56.00000	56.00000	56.00000	56.00000	56.00000	56.00000	180.00000
5	.13000	50.00000	5.14500	57.00000	57.00000	57.00000	57.00000	57.00000	57.00000	170.00000
6	.13000	50.00000	6.17400	57.00000	57.00000	57.00000	57.00000	57.00000	57.00000	152.00000
7	.13000	50.00000	7.20300	52.00000	52.00000	52.00000	52.00000	52.00000	52.00000	180.00000
8	.33000	50.00000	1.02900	54.00000	54.00000	54.00000	54.00000	54.00000	54.00000	180.00000
9	.33000	50.00000	2.05800	52.00000	52.00000	52.00000	52.00000	52.00000	52.00000	180.00000
10	.33000	50.00000	3.08700	54.00000	54.00000	54.00000	54.00000	54.00000	54.00000	180.00000
11	.33000	50.00000	4.11600	50.00000	50.00000	50.00000	50.00000	50.00000	50.00000	168.00000
12	.33000	50.00000	5.14500	52.00000	52.00000	52.00000	52.00000	52.00000	52.00000	180.00000
13	.33000	50.00000	6.17400	49.00000	49.00000	49.00000	49.00000	49.00000	49.00000	150.00000
14	.33000	50.00000	7.20300	50.00000	50.00000	50.00000	50.00000	50.00000	50.00000	180.00000
15	.13000	150.00000	1.02900	26.00000	26.00000	26.00000	26.00000	26.00000	26.00000	42.00000
16	.13000	150.00000	2.05800	28.00000	28.00000	28.00000	28.00000	28.00000	28.00000	44.00000
17	.13000	150.00000	3.08700	27.00000	27.00000	27.00000	27.00000	27.00000	27.00000	45.00000
18	.13000	150.00000	4.11600	24.00000	24.00000	24.00000	24.00000	24.00000	24.00000	43.00000
19	.13000	150.00000	5.14500	25.00000	25.00000	25.00000	25.00000	25.00000	25.00000	35.00000
20	.13000	150.00000	6.17400	25.00000	25.00000	25.00000	25.00000	25.00000	25.00000	41.00000
21	.13000	150.00000	7.20300	28.00000	28.00000	28.00000	28.00000	28.00000	28.00000	44.00000
22	.33000	150.00000	1.02900	26.00000	26.00000	26.00000	26.00000	26.00000	26.00000	43.00000
23	.33000	150.00000	2.05800	26.00000	26.00000	26.00000	26.00000	26.00000	26.00000	45.00000
24	.33000	150.00000	3.08700	23.00000	23.00000	23.00000	23.00000	23.00000	23.00000	43.00000
25	.33000	150.00000	4.11600	23.00000	23.00000	23.00000	23.00000	23.00000	23.00000	39.00000
26	.33000	150.00000	5.14500	23.00000	23.00000	23.00000	23.00000	23.00000	23.00000	44.00000
27	.33000	150.00000	6.17400	25.00000	25.00000	25.00000	25.00000	25.00000	25.00000	47.00000
28	.33000	150.00000	7.20300	23.00000	23.00000	23.00000	23.00000	23.00000	23.00000	23.00000
29	.13000	300.00000	1.02900	15.00000	15.00000	15.00000	15.00000	15.00000	15.00000	18.00000
30	.13000	300.00000	2.05800	17.00000	17.00000	17.00000	17.00000	17.00000	17.00000	21.00000
31	.13000	300.00000	3.08700	16.00000	16.00000	16.00000	16.00000	16.00000	16.00000	22.00000
32	.13000	300.00000	4.11600	17.00000	17.00000	17.00000	17.00000	17.00000	17.00000	20.00000
33	.13000	300.00000	5.14500	16.00000	16.00000	16.00000	16.00000	16.00000	16.00000	20.00000
34	.13000	300.00000	6.17400	15.00000	15.00000	15.00000	15.00000	15.00000	15.00000	22.00000
35	.13000	300.00000	7.20300	15.00000	15.00000	15.00000	15.00000	15.00000	15.00000	22.00000
36	.33000	300.00000	1.02900	13.00000	13.00000	13.00000	13.00000	13.00000	13.00000	21.00000
37	.33000	300.00000	2.05800	14.00000	14.00000	14.00000	14.00000	14.00000	14.00000	20.00000
38	.33000	300.00000	3.08700	14.00000	14.00000	14.00000	14.00000	14.00000	14.00000	21.00000
39	.33000	300.00000	4.11600	14.00000	14.00000	14.00000	14.00000	14.00000	14.00000	19.00000
40	.33000	300.00000	5.14500	14.00000	14.00000	14.00000	14.00000	14.00000	14.00000	22.00000
41	.33000	300.00000	6.17400	14.00000	14.00000	14.00000	14.00000	14.00000	14.00000	131.00000
42	.33000	300.00000	7.20300	15.00000	15.00000	15.00000	15.00000	15.00000	15.00000	126.00000
43	.33000	50.00000	1.02900	77.00000	77.00000	77.00000	77.00000	77.00000	77.00000	
44	.33000	50.00000	2.05800	76.00000	76.00000	76.00000	76.00000	76.00000	76.00000	
45	.33000	50.00000	3.08700	73.00000	73.00000	73.00000	73.00000	73.00000	73.00000	

MIXTIME DATA
(SECONDS)

(CONTINUED)

IMPELLER	C/T	RPM	CONCENTRATION (GRAMS OF NaCl)	MIXTIME		MIXTIME		MIXTIME	
				PROBE #3	PROBE #4	PROBE #5	PROBE #6		
1-FLAT									
2-PITCH									
46	.33000	50.00000	4.11600	72.00000	69.00000	66.00000	122.00000		
47	.33000	50.00000	5.14500	67.00000	67.00000	71.00000	125.00000		
48	.33000	50.00000	6.17400	71.00000	71.00000	69.00000	109.00000		
49	.33000	50.00000	7.20300	70.00000	70.00000	68.00000	113.00000		
50	.50000	50.00000	1.02900	70.00000	72.00000	75.00000	138.00000		
51	.50000	50.00000	2.05800	73.00000	75.00000	80.00000	69.00000		
52	.50000	50.00000	3.08700	73.00000	77.00000	76.00000	71.00000		
53	.50000	50.00000	4.11600	76.00000	74.00000	78.00000	74.00000		
54	.50000	50.00000	5.14500	67.00000	73.00000	77.00000	69.00000		
55	.50000	50.00000	6.17400	71.00000	73.00000	78.00000	72.00000		
56	.33000	150.00000	1.02900	26.00000	28.00000	29.00000	57.00000		
57	.33000	150.00000	2.05800	28.00000	27.00000	28.00000	51.00000		
58	.33000	150.00000	3.08700	27.00000	28.00000	30.00000	50.00000		
59	.33000	150.00000	4.11600	27.00000	28.00000	30.00000	49.00000		
60	.33000	150.00000	5.14500	25.00000	25.00000	27.00000	58.00000		
61	.33000	150.00000	6.17400	28.00000	28.00000	28.00000	55.00000		
62	.33000	150.00000	7.20300	26.00000	26.00000	27.00000	56.00000		
63	.50000	150.00000	1.02900	27.00000	28.00000	30.00000	31.00000		
64	.50000	150.00000	2.05800	29.00000	30.00000	29.00000	29.00000		
65	.50000	150.00000	3.08700	27.00000	29.00000	29.00000	28.00000		
66	.50000	150.00000	4.11600	28.00000	29.00000	31.00000	35.00000		
67	.50000	150.00000	5.14500	27.00000	28.00000	30.00000	28.00000		
68	.50000	150.00000	6.17400	28.00000	27.00000	28.00000	28.00000		
69	.50000	150.00000	7.20300	31.00000	31.00000	30.00000	30.00000		
70	.33000	300.00000	1.02900	13.00000	13.00000	12.50000	17.00000		
71	.33000	300.00000	2.05800	14.00000	13.00000	14.50000	16.50000		
72	.33000	300.00000	3.08700	13.00000	13.00000	12.50000	16.50000		
73	.33000	300.00000	4.11600	13.00000	14.00000	12.50000	16.00000		
74	.33000	300.00000	5.14500	12.50000	13.00000	12.50000	16.50000		
75	.33000	300.00000	6.17400	13.00000	14.50000	12.50000	16.50000		
76	.33000	300.00000	7.20300	13.00000	13.00000	12.50000	16.00000		
77	.50000	300.00000	1.02900	14.50000	15.00000	16.50000	16.00000		
78	.50000	300.00000	2.05800	17.00000	17.00000	17.00000	15.50000		
79	.50000	300.00000	3.08700	17.00000	17.50000	18.00000	16.00000		
80	.50000	300.00000	4.11600	16.50000	17.00000	17.50000	15.00000		
81	.50000	300.00000	5.14500	17.00000	17.50000	18.00000	15.00000		
82	.50000	300.00000	6.17400	18.00000	18.00000	17.50000	15.50000		
83	.50000	300.00000	7.20300	18.00000	18.00000	17.00000	15.50000		

Appendix VII
Torque Comparison Power Data

TORQUE COMPARISON

(WATTS)

IMPELLER	C/T	RPM	WEIGH SCALE		TORQUE PLATE	WEIGH SCALE		THEORETICAL
			ORIGINAL	CORRECTED		CORRECTED		
1-FLAT								
2-PITCH								
1	.13000	50.00000	.13045	.01249	.01249	.01305	.01790	.01790
2	.13000	100.00000	1.14443	.10266	.10266	.11444	.14168	.14168
3	.13000	150.00000	4.12611	.39747	.39747	.41261	.46979	.46979
4	.13000	200.00000	9.52930	.92474	.92474	.95293	1.11855	1.11855
5	.13000	250.00000	18.14437	1.77544	1.77544	1.81444	2.23710	2.23710
6	.13000	300.00000	31.47823	3.06363	3.06363	3.14782	3.72850	3.72850
7	.33000	50.00000	.14189	.01107	.01107	.01419	.01790	.01790
8	.33000	100.00000	1.10177	.10197	.10197	.11018	.14168	.14168
9	.33000	150.00000	4.16548	.39195	.39195	.41655	.46979	.46979
10	.33000	200.00000	9.19374	.88865	.88865	.91937	1.11855	1.11855
11	.33000	250.00000	17.13246	1.68752	1.68752	1.71325	2.23710	2.23710
12	.33000	300.00000	29.33360	2.89809	2.89809	2.93336	3.72850	3.72850
13	.33000	50.00000	.06623	5.616836E-03	5.616836E-03	6.622860E-03	8.948400E-03	8.948400E-03
14	.33000	100.00000	.55556	.05422	.05422	.05556	.06711	.06711
15	.33000	150.00000	2.27364	.21732	.21732	.22736	.23862	.23862
16	.33000	200.00000	5.23481	.49338	.49338	.52348	.55182	.55182
17	.33000	250.00000	9.78731	.93481	.93481	.97873	1.11855	1.11855
18	.33000	300.00000	16.79316	1.62555	1.62555	1.67932	2.01339	2.01339
19	.50000	50.00000	.07997	5.925407E-03	5.925407E-03	7.996887E-03	8.948400E-03	8.948400E-03
20	.50000	100.00000	.65078	.05698	.05698	.06508	.06711	.06711
21	.50000	150.00000	2.35112	.21783	.21783	.23511	.23862	.23862
22	.50000	200.00000	5.49775	.51881	.51881	.54977	.55182	.55182
23	.50000	250.00000	10.40028	.98664	.98664	1.04003	1.11855	1.11855
24	.50000	300.00000	17.66041	1.70169	1.70169	1.76604	2.01339	2.01339

TORQUE COMPARISON

(HORSEPOWER)

IMPELLER

	IMPELLER	1-FLAT	2-PITCH	C/T	RPM	WEIGH SCALE		TORQUE PLATE	WEIGH SCALE		THEORETICAL
						ORIGINAL	TORQUE PLATE		CORRECTED		
1		1.00000		.13000	50.00000	1.749400E-04	1.675100E-05	1.749400E-05	1.749400E-05	2.400000E-05	
2		1.00000		.13000	100.00000	1.534700E-03	1.376700E-04	1.534700E-04	1.534700E-04	1.900000E-04	
3		1.00000		.13000	150.00000	5.533200E-03	5.330100E-04	5.330100E-04	5.333200E-04	6.300000E-04	
4		1.00000		.13000	200.00000	.01278	1.240100E-03	1.277900E-03	1.277900E-03	1.500000E-03	
5		1.00000		.13000	250.00000	.02433	2.380900E-03	2.380900E-03	2.433200E-03	3.000000E-03	
6		1.00000		.13000	300.00000	.04221	4.108400E-03	4.108400E-03	4.221300E-03	5.000000E-03	
7		1.00000		.33000	50.00000	1.902800E-04	1.484900E-05	1.484900E-05	1.902800E-05	2.400000E-05	
8		1.00000		.33000	100.00000	1.477500E-03	1.367500E-04	1.367500E-04	1.477500E-04	1.900000E-04	
9		1.00000		.33000	150.00000	5.586000E-03	5.256200E-04	5.256200E-04	5.586000E-04	6.300000E-04	
10		1.00000		.33000	200.00000	.01233	1.191700E-03	1.191700E-03	1.232900E-03	1.500000E-03	
11		1.00000		.33000	250.00000	.02298	2.263000E-03	2.263000E-03	2.297500E-03	3.000000E-03	
12		1.00000		.33000	300.00000	.03934	3.886400E-03	3.886400E-03	3.933700E-03	5.000000E-03	
13		2.00000		.33000	50.00000	8.881400E-05	7.532300E-06	7.532300E-06	8.881400E-06	1.200000E-05	
14		2.00000		.33000	100.00000	7.450200E-04	7.270700E-05	7.270700E-05	7.450200E-05	9.000000E-05	
15		2.00000		.33000	150.00000	3.049000E-03	2.914300E-04	2.914300E-04	3.049000E-04	3.200000E-04	
16		2.00000		.33000	200.00000	7.020000E-03	6.616300E-04	6.616300E-04	7.020000E-04	7.400000E-04	
17		2.00000		.33000	250.00000	.01313	1.253600E-03	1.253600E-03	1.312500E-03	1.500000E-03	
18		2.00000		.33000	300.00000	.02252	2.179900E-03	2.179900E-03	2.252000E-03	2.700000E-03	
19		2.00000		.50000	50.00000	1.072400E-04	7.946100E-06	7.946100E-06	1.072400E-05	1.200000E-05	
20		2.00000		.50000	100.00000	8.727100E-04	7.641400E-05	7.641400E-05	8.727100E-05	9.000000E-05	
21		2.00000		.50000	150.00000	3.152900E-03	2.921100E-04	2.921100E-04	3.152900E-04	3.200000E-04	
22		2.00000		.50000	200.00000	7.372600E-03	6.957300E-04	6.957300E-04	7.372600E-04	7.400000E-04	
23		2.00000		.50000	250.00000	.01395	1.323100E-03	1.323100E-03	1.394700E-03	1.500000E-03	
24		2.00000		.50000	300.00000	.02368	2.282000E-03	2.282000E-03	2.368300E-03	2.700000E-03	

Appendix VIII
Fluid Mixing Statistics

FLUID MIXING STATISTICS

D. Ginley and D. Friday (12/84)

The fluid mixing problem is, from a statistical perspective a non-standard application. Most of existing statistical methodology is non-applicable due to the time dependent nature of the experiment. Furthermore, almost all of the time series methodology, the statistical area most relevant to the mixing problems, assumes stationarity and linearity or very specific forms of non-stationarity or non-linearity. As a consequence very little of existing methodology is applicable to the analysis of the laboratory data. It was therefore necessary to adapt existing statistical concepts as far as possible and then to develop new procedures specifically for the mixing problems. The results, which are only partially described here, provide a thorough and efficient description of the experimental results and minimize the use of the subjective reasoning which is characteristic of past work in this area.

There were three basic objectives in the statistical analysis. The first is to provide an effective statistical summary of the large amounts of data which result from mixing experiments. The second objective is to provide an estimate of the mixing time which is both statistically and physically sound. That is, an estimate uniquely determined from the data, which has optimal statistical properties, which is applicable to data from a wide variety of experimental conditions, which is compatible with mixing theories, and is in agreement with visual observation of the mixing waveform. The only investigated previous attempt to replace human judgement required the assumption of a very restrictive sinusoidal-exponential model (Khang, Levenspiel, 1976). Finally we hoped to obtain a general statistical model which could adequately describe the time dependence of the experimental results and thereby provide another parallel approach to achieving the first two objectives.

Statistical Summary of Experimental Results:

The summary of the data combined statistical concepts and graphics, utilizing to advantage the high resolution graphics hardware/software which was available. The simplest graph is a time plot which exhibits the central location and the dispersion of a set of replications of an experiment under identical experimental conditions at each point in time. The only underlying assumptions are that all the replications fluctuate about some "norm" for the

given experimental conditions and that their deviations from this norm are random and independent of the outcomes of the other replications. These assumptions appear to be reasonable. Note that there is a clear time dependence in the random component but this property does not affect the interpretation of the plots. These plots can be implemented in various ways. The most common is to use the arithmetic average for each epoch of time, average across replications, as the location component of the plot. The dispersion component of the plot then gives information on how variable the repetitions of the experiment are, with respect to the location estimate, at each point in time. The most common such estimate is the sample variance. A plot of the average with upper and lower bounds (avg. \pm variance) exhibiting the time pattern of the changing variance proved to very very informative (see fig. A).

In traditional statistical applications the mean and variance estimate is used to develop confidence intervals. In this case not enough repetitions were obtained of a single experiment to ascertain distributional properties over time. This is a necessary assumption in developing confidence intervals and we therefore did not develop confidence time plots. They are a possible future extension, however, given sufficient data on a single experimental configuration. The mean-variance plot is by no means the only such plot. Three-sigma bounds may be plotted, for example, as long as it is clear what is plotted and that they are not used as confidence intervals. Another possibility which was used is the median-extremes plot. The median is used for the location plot and the maximum end minimum values are used as the upper and lower bound. This plot has the advantage that the location plot is more robust to outliers than the average and the extremes are directly influenced by the outliers. Therefore the typical mixing path is estimated clearly and any extreme behavior is also apparent. Other possibilities are also reasonable such as mean \pm range, median \pm order statistics, etc. These plots proved to be a very useful way of summarizing the data and we advocate use of several of them for exploratory purposes (see fig. B).

Another plot which proved useful is one we developed from some statistical considerations called the cumulative successive squared difference (CSSD). It involves taking successive difference on the data, then squaring this series of differences and developing the partial sums. If c_t is the concentration at time $t = 0, 1, \dots, N$ then $S(t)$ the CSSD at time t is given by (see fig. C):

$$s(t) = \sum_{i=1}^t (c_i - c_{i+1})^2 ; \quad t = 1, 2, \dots, N-1$$

The reasoning behind such plots will be developed in a later publication. Their interpretation, however, is to exhibit the dissipation over time of the mixing energy in the experiment. Energy here is analogous to turbulence or variation in the observed data. It established another way of observing the "quiet" periods before and after mixing with the additional advantage of providing quantiles over time. That is the percentage of energy in the mixing experiment expended in any time interval can be measured and used for comparison with other experiments. It is conditional on the total energy in the experiment however. If an experiment is run with a longer tail after the mixing is completed then this small residual variation biases the estimates. We have developed methods to compensate for this, however, for brevity we will not discuss them at this time.

The location-dispersion plots and CSSD plots were not the only ones we found useful. Others exhibited variance decomposition, and structural information. These will be reported on later. A statistic which proved useful in discriminating between different experimental conditions, such as probe location baffle position relative to probe location and stirring conditions, was derived. It is related to the CSSD and showed marked differences between different configurations. This will also be discussed further in a paper in preparation.

Statistical studies also focused on mixtime estimation. All of these approaches are non-parametric or model free. The intention was that they be applicable to a wide range of mixing experiments and not be dependent on a particular, restrictive model. Two approaches to mixtime proved reasonable. One is based on the CSSD plots and defines mixtime as the epoch after initiation at which the energy change falls into the background noise level. The other approach is not related to the CSSD plots but to exceedance of the background noise level in reverse time. While both methods are reasonable statistically some practical problems were encountered due to the fact that the background noise level is in the quantization region of the A/D converters.

The CSSD method is less affected by the quantizing and the effects of the quantizing on the reverse-time method are being studied. When complete, the details of the methods will be presented. The effort to remove the subjective judgement from mixtime estimation we feel has been successful.

The final objective of modeling the sample paths from mixing experiments and doing it in a very flexible and general way is in the research stage. Some progress has been made and it is considered a difficult problem. We are not prepared to report on this work at the present time.

FIGURE A.

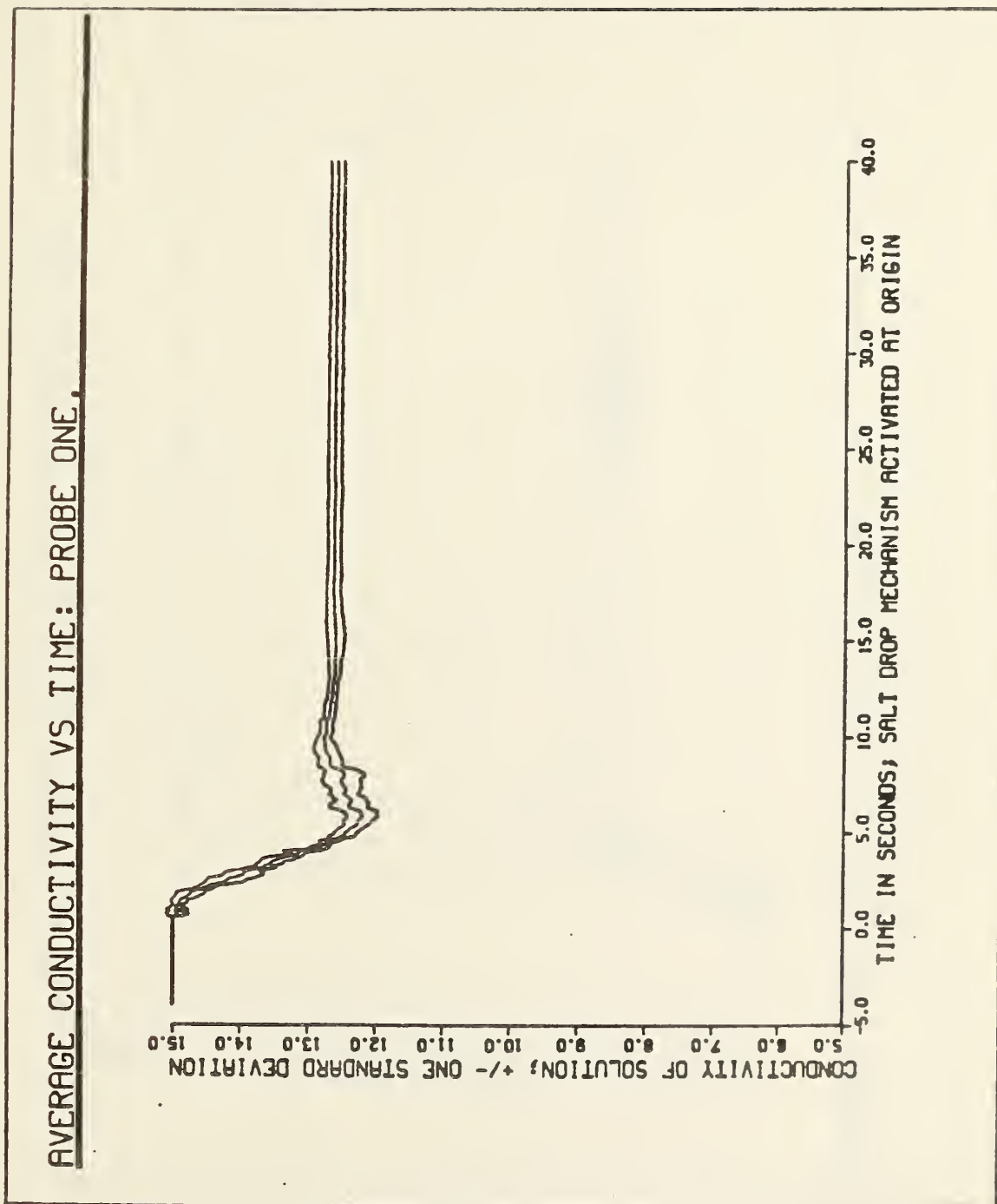


FIGURE B.

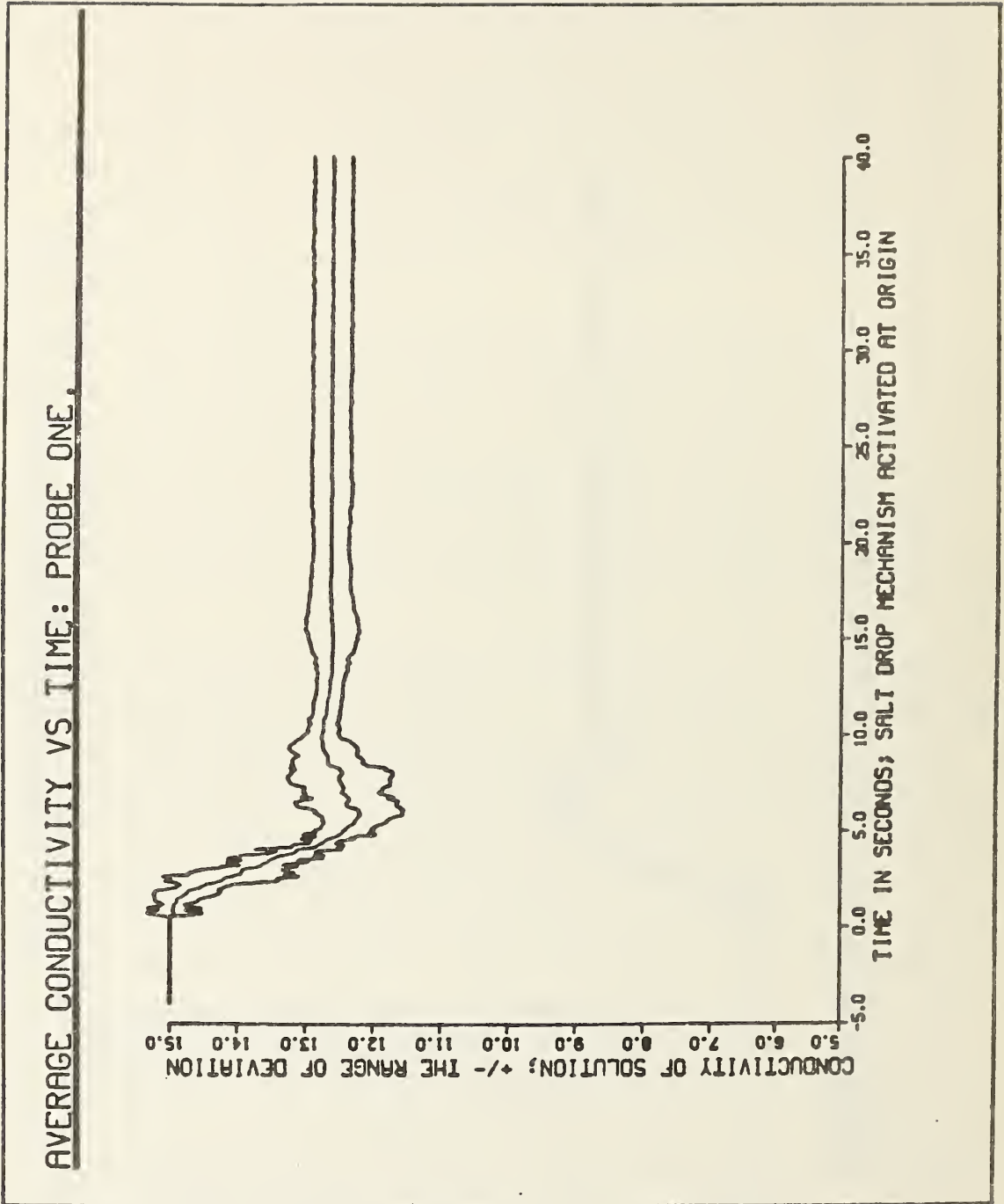
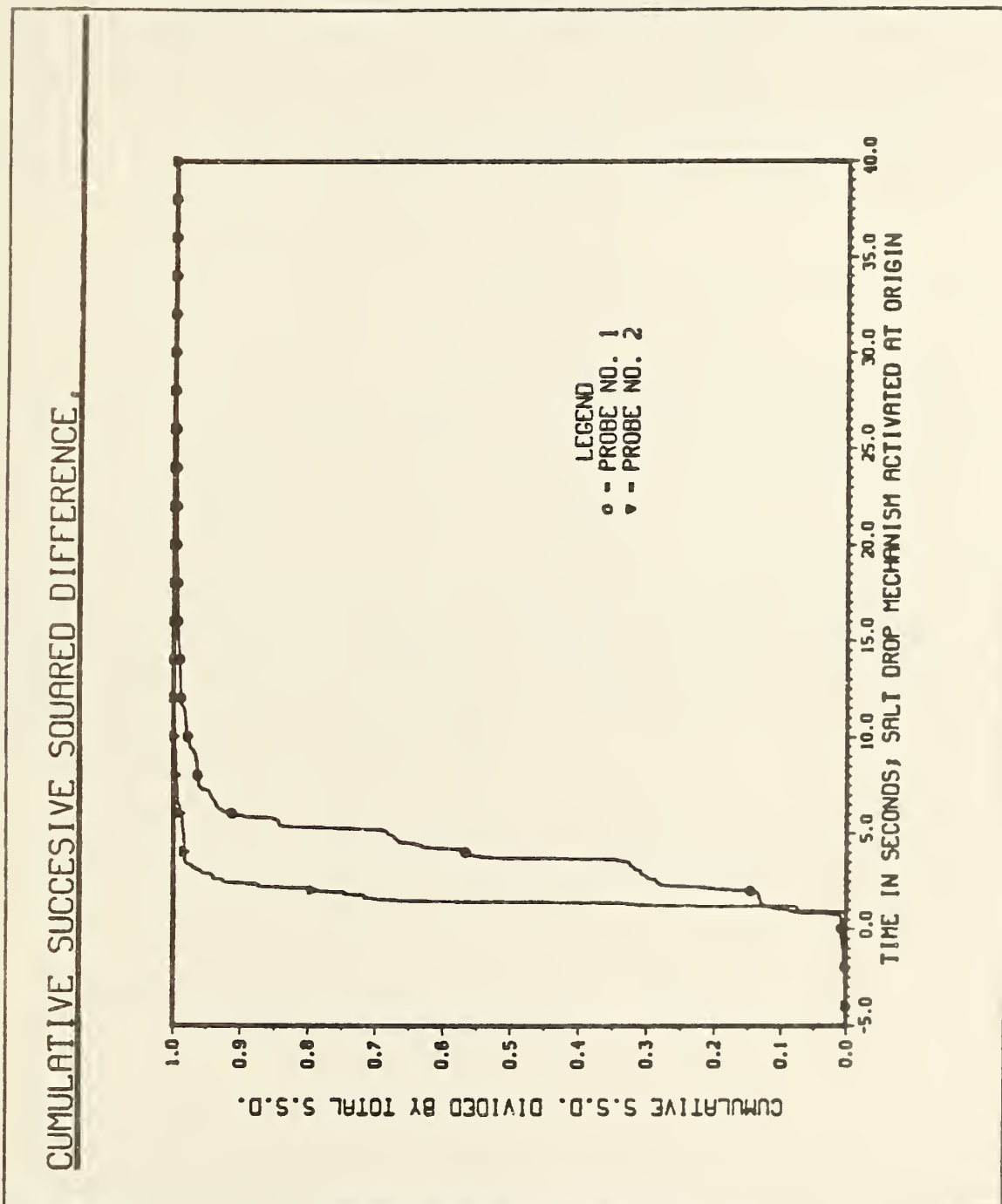


FIGURE C.



Appendix IX
Experimental Test Procedure

TEST PROCEDURE

- 1) Select mixing vessel to be tested, and determine its ability to be filled with deionized water, and to have a tracer of NaCl solution injected.
- 2) Determine the number of conductivity probes to be used (minimum of two). Place these probes, one at the surface of the mixing fluid, and one away from baffles at approximately the height of the impeller. Both probes should be located near the vessel walls, with the surface probe above the other one. Additional probes may be added to enhance information gained from the test.
- 3) Fill the vessel with deionized water ($Z/T = 1.0$).
- 4) Record resistivity information from the conductivity probes. Data points should be taken at no greater than 0.10 second intervals.
- 5) Inject the NaCl solution in the most realistic manner for the process being studied (i.e., surface injection, injection by the impeller, etc.).
- 6) Record the data before the NaCl injection, and for a sufficiently long time after the injection to insure that complete mixing has occurred.
- 7) For comparison to other work done, and to established correlations it is necessary to measure the power. This may be done directly with a wattmeter, or by measuring the torque and rpm for the test performed. Analysis of the data at this time is left to the individual. Further details regarding experimental procedures and equipment may be found in the 'apparatus' section of this report.

Appendix X Experimental Data

The following information is a representative example of the data obtainable from the described experiments. For further information regarding specific experimental setups and/or a more complete data set, please feel free to contact Douglas Ginley at the National Bureau of Standards (phone: (303)497-3521, FTS 320-3521).

FINAL CONCENTRATION DATA (WEIGHT PERCENTS)

IMPELLER 1-FLAT 2-PITCH	C/T	RPM	CONCENTRATION (GRAMS OF NaCl)	PROBE #3 (WT %)	PROBE #4 (WT %)	PROBE #5 (WT %)	PROBE #6 (WT %)
1	.13000	50.00000	1.02900	7.488000E-03	7.520000E-03	7.528000E-03	7.470000E-03
2	.13000	50.00000	2.05800	.01905	.01905	.01908	.01877
3	.13000	50.00000	3.08700	.02873	.02873	.02853	.02842
4	.13000	50.00000	4.11600	.03819	.03819	.03795	.03798
5	.13000	50.00000	5.14500	.04744	.04744	.04743	.04717
6	.13000	50.00000	6.17400	.05679	.05679	.05682	.05663
7	.13000	50.00000	7.20300	.06610	.06598	.06598	.06620
8	.33000	50.00000	1.02900	9.455000E-03	9.539000E-03	9.631000E-03	9.084000E-03
9	.33000	50.00000	2.05800	.01920	.01922	.01918	.01890
10	.33000	50.00000	3.08700	.02880	.02884	.02854	.02832
11	.33000	50.00000	4.11600	.03828	.03833	.03802	.03784
12	.33000	50.00000	5.14500	.04763	.04763	.04755	.04725
13	.33000	50.00000	6.17400	.05691	.05694	.05689	.05653
14	.33000	50.00000	7.20300	.06621	.06612	.06604	.06608
15	.13000	150.00000	1.02900	8.850000E-03	8.829000E-03	8.983000E-03	8.902000E-03
16	.13000	150.00000	2.05800	.01843	.01839	.01856	.01848
17	.13000	150.00000	3.08700	.02814	.02809	.02807	.02811
18	.13000	150.00000	4.11600	.03766	.03763	.03753	.03760
19	.13000	150.00000	5.14500	.04707	.04704	.04714	.04701
20	.13000	150.00000	6.17400	.05649	.05649	.05645	.05648
21	.13000	150.00000	7.20300	.06579	.06562	.06578	.06600
22	.33000	150.00000	1.02900	9.343000E-03	9.493000E-03	9.445000E-03	9.173000E-03
23	.33000	150.00000	2.05800	.01888	.01888	.01887	.01858
24	.33000	150.00000	3.08700	.02842	.02845	.02821	.02814
25	.33000	150.00000	4.11600	.03778	.03784	.03755	.03749
26	.33000	150.00000	5.14500	.04714	.04714	.04707	.04684
27	.33000	150.00000	6.17400	.05635	.05638	.05635	.05604
28	.33000	150.00000	7.20300	.06555	.06545	.06541	.06539
29	.13000	300.00000	1.02900	9.023000E-03	8.987000E-03	9.104000E-03	9.133000E-03
30	.13000	300.00000	2.05800	.01849	.01843	.01856	.01858
31	.13000	300.00000	3.08700	.02803	.02797	.02797	.02797
32	.13000	300.00000	4.11600	.03740	.03737	.03722	.03730
33	.13000	300.00000	5.14500	.04665	.04661	.04661	.04655
34	.13000	300.00000	6.17400	.05586	.05581	.05593	.05574
35	.13000	300.00000	7.20300	.06509	.06493	.06503	.06518
36	.33000	300.00000	1.02900	9.024000E-03	9.047000E-03	9.112000E-03	9.101000E-03
37	.33000	300.00000	2.05800	.01856	.01855	.01863	.01859
38	.33000	300.00000	3.08700	.02812	.02813	.02799	.02797
39	.33000	300.00000	4.11600	.03757	.03757	.03759	.03736
40	.33000	300.00000	5.14500	.04685	.04687	.04688	.04671
41	.33000	300.00000	6.17400	.05619	.05618	.05627	.05592
42	.33000	300.00000	7.20300	.06544	.06531	.06537	.06547
43	.33000	50.00000	1.02900	9.288000E-03	9.269000E-03	9.280000E-03	8.966000E-03
44	.33000	50.00000	2.05800	.01850	.01856	.01849	.01827
45	.33000	50.00000	3.08700	.02785	.02796	.02765	.02760

FINAL CONCENTRATION DATA
(WEIGHT PERCENTS)
(CONTINUED)

IMPELLER 1-FLAT 2-PITCH	C/T	RPM	CONCENTRATION (GRAMS OF NaCl)	PROBE #3 (WT %)	PROBE #4 (WT %)	PROBE #5 (WT %)	PROBE #6 (WT %)
46	2.00000	50.00000	4.11600	.03695	.03707	.03671	.03665
47	2.00000	50.00000	5.14500	.04594	.04599	.04578	.04565
48	2.00000	50.00000	6.17400	.05477	.05477	.05479	.05440
49	2.00000	50.00000	7.20300	.06356	.06353	.06345	.06337
50	2.00000	50.00000	1.02900	9.61200E-03	9.67200E-03	9.53900E-03	9.44600E-03
51	2.00000	50.00000	2.05800	.01929	.01952	.01928	.01921
52	2.00000	50.00000	3.08700	.02892	.02920	.02870	.02870
53	2.00000	50.00000	4.11600	.03840	.03865	.03814	.03820
54	2.00000	50.00000	5.14500	.04782	.04806	.04774	.04763
55	2.00000	50.00000	6.17400	.05709	.05729	.05707	.05689
56	2.00000	150.00000	1.02900	9.22500E-03	9.32200E-03	9.19400E-03	9.14900E-03
57	2.00000	150.00000	2.05800	.01829	.01835	.01826	.01822
58	2.00000	150.00000	3.08700	.02737	.02746	.02716	.02720
59	2.00000	150.00000	4.11600	.03638	.03644	.03610	.03618
60	2.00000	150.00000	5.14500	.04626	.04626	.04160	.04604
61	2.00000	150.00000	6.17400	.05389	.05395	.05387	.05367
62	2.00000	150.00000	7.20300	.06256	.06257	.06249	.06244
63	2.00000	150.00000	1.02900	9.40800E-03	9.56200E-03	9.32100E-03	9.11000E-03
64	2.00000	150.00000	2.05800	.01899	.01913	.01902	.01888
65	2.00000	150.00000	3.08700	.02846	.02864	.02826	.02822
66	2.00000	150.00000	4.11600	.03786	.03803	.03764	.03763
67	2.00000	150.00000	5.14500	.04706	.04721	.04699	.04679
68	2.00000	150.00000	6.17400	.05640	.05653	.05641	.05615
69	2.00000	150.00000	7.20300	.06553	.06552	.06541	.06549
70	2.00000	300.00000	1.02900	9.26700E-03	9.37000E-03	9.23700E-03	9.18700E-03
71	2.00000	300.00000	2.05800	.01816	.01822	.01812	.01808
72	2.00000	300.00000	3.08700	.02717	.02726	.02697	.02699
73	2.00000	300.00000	4.11600	.03591	.03596	.03569	.03567
74	2.00000	300.00000	5.14500	.04465	.04470	.04453	.04444
75	2.00000	300.00000	6.17400	.05331	.05333	.05327	.05309
76	2.00000	300.00000	7.20300	.06191	.06190	.06183	.06173
77	2.00000	300.00000	1.02900	9.63700E-03	9.82000E-03	9.64500E-03	9.60500E-03
78	2.00000	300.00000	2.05800	.01907	.01921	.01907	.01903
79	2.00000	300.00000	3.08700	.02854	.02870	.02833	.02834
80	2.00000	300.00000	4.11600	.03783	.03801	.03760	.03762
81	2.00000	300.00000	5.14500	.04701	.04717	.04694	.04679
82	2.00000	300.00000	6.17400	.05629	.05643	.05631	.05618
83	2.00000	300.00000	7.20300	.06556	.06554	.06544	.06556

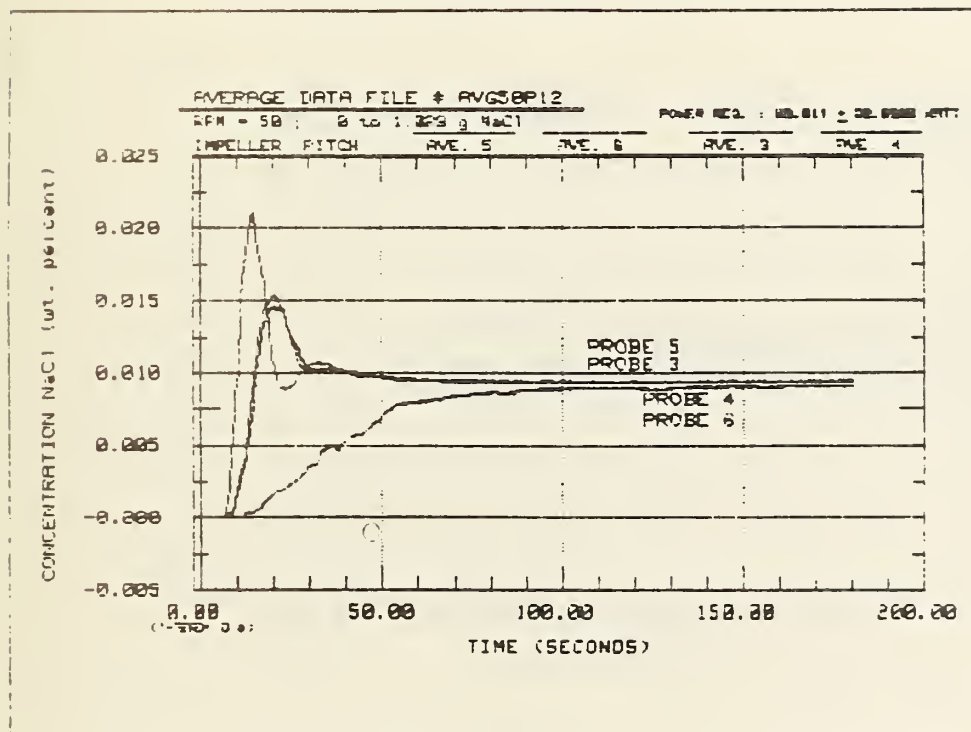
XRPMS0XR #AVG50P12XP 50 RPM RUN #:AVG50P12 NUMBER OF POINTS = 1000
 TIME PER POINT = .2
 TANK: VOL= 11.939 L , DIAMETER= 24.765 CM IMPELLOR:PITCH(4 IN)
 SALT START= 0 GM, SALT FINISH= 1.029 GM
 WATTS OF STIRRING ENERGY = .011143

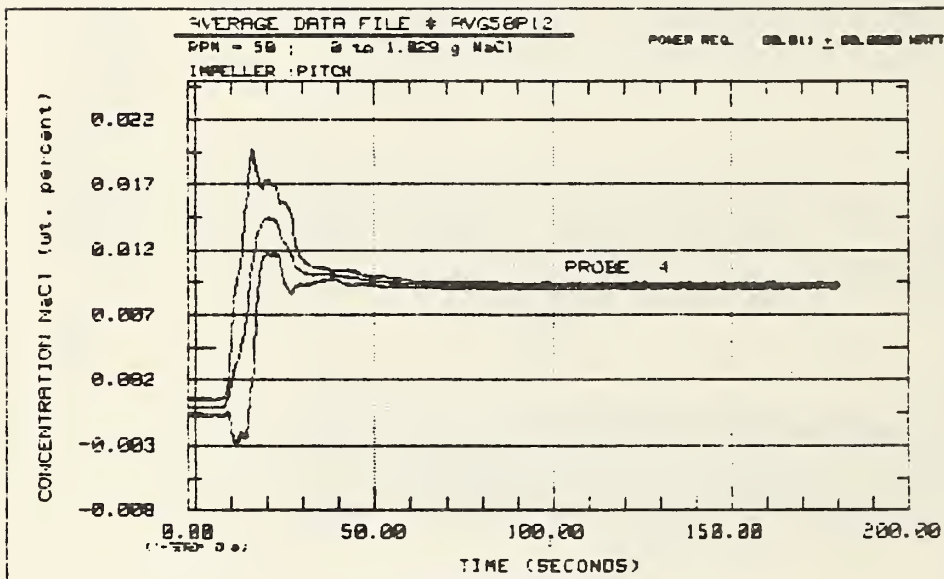
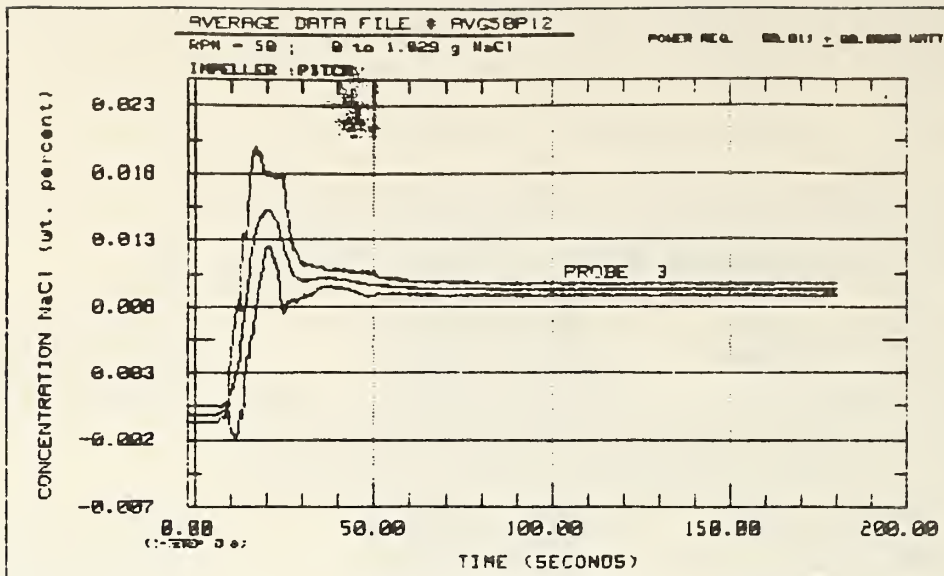
THE FOLLOWING IS INFORMATION CONCERNING THE NOISE FOR EACH PROBE:
 START OF RUN

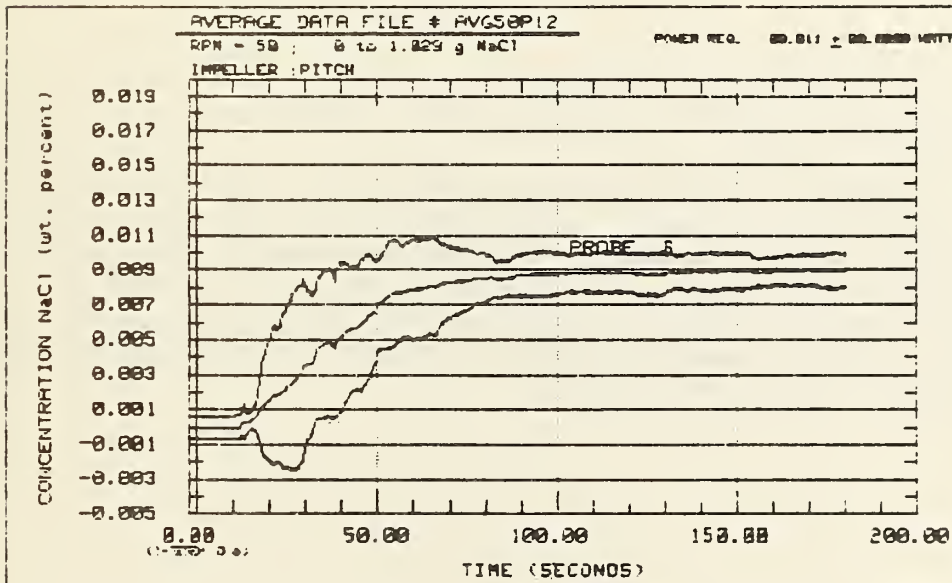
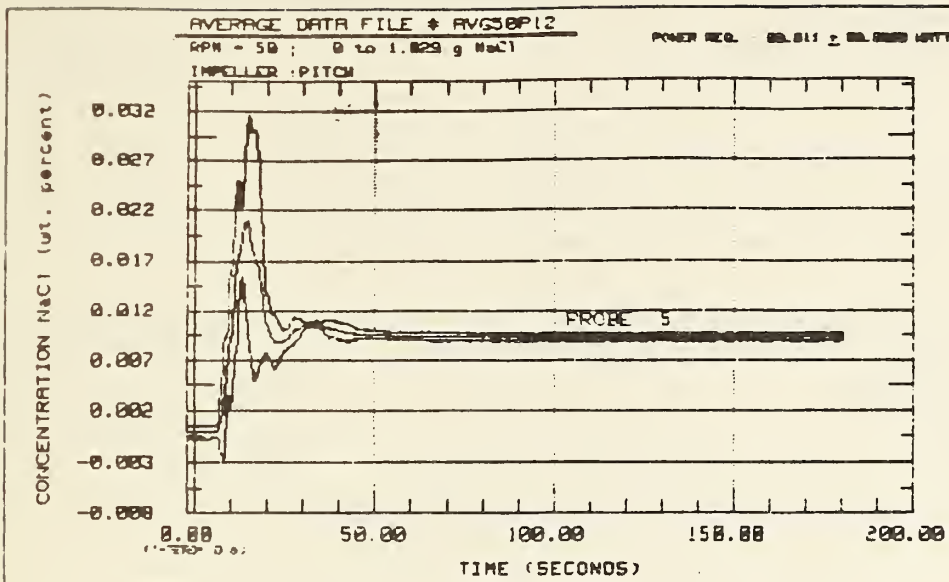
PROBE #3 :	.000314304217115	+-	1.14856402679E-5	FOR 95% CONFIDENCE
PROBE #4 :	.000314811378328	+-	1.51136669544E-5	FOR 95% CONFIDENCE
PROBE #5 :	.000314303221825	+-	1.4868657205E-5	FOR 95% CONFIDENCE
PROBE #6 :	.000314338002131	+-	1.41507338855E-5	FOR 95% CONFIDENCE

END OF RUN

PROBE #3 :	.000220006410492	+-	3.00595229976E-5	FOR 95% CONFIDENCE
PROBE #4 :	8.49510724091E-5	+-	3.37740140687E-5	FOR 95% CONFIDENCE
PROBE #5 :	.000174849351197	+-	3.56991696551E-5	FOR 95% CONFIDENCE
PROBE #6 :	.000455250696519	+-	7.36037863441E-5	FOR 95% CONFIDENCE







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11. ABSTRACT (A 200-word or less factual summary of most significant information. If document includes a significant bibliography or literature survey, mention it here) <p>A performance test procedure and measurement technique for a batch mixing system is described using conductivity probes. The design of the automated experimental apparatus is described, and experimental procedures are given. Data collected from the experiments are explained, and a mixtime analysis of the data is performed. Conductivity probe response curves provide a good representation of the system dynamics. Mixtime analysis allows for probe response comparisons to evaluate system geometry and probe location.</p>			
12. KEY WORDS (Six to twelve entries; alphabetical order; capitalize only proper names; and separate key words by semicolons) batch mixing; calibration techniques; conductivity probes; experimental apparatus; mixtime analysis; torque measurements			
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