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Sensitivity of Process Variables on Rigid Polyurethane Foaming

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SENSITIVITY OF PROCESS VARIABLES ON RIGID POLYURETHANE FOAMING

A Thesis
Presented to the
Department of Mechanical Engineering
Brigham Young University

In Partial Fulfillment
of the Requirements for the Degree
Master of Science

by
Bruce A. Pugsley
December 1976

This thesis, by Bruce A. Pugsley, is accepted in its present form by the Department of Mechanical Engineering of Brigham Young University as satisfying the thesis requirements for the degree of Master of Science.

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ACKNOWLEDGEMENTS

Beginning the evaluation of this process and finding exactly what my part might be in helping solve this problem seemed like an almost impossible task to perform. My worry was that I might be able in some way to help those who so generously contributed to my schooling while I attended graduate school. Whirlpool Corporation supplied me with a sizeable stipend each month and also funds for research. My thanks goes to them for their help.

Dr. Dell K. Allen of the B. Y. U. Manufacturing Technology Department was of valuable assistance in directing my research and in giving me much needed advice and direction.

Dr. Milton Wille of the Mechanical Engineering Department also gave me many valuable suggestions to help me accomplish this task.

My thanks also goes to my wife and family, who went through some hardships because I was attending school, but supported me in all I tried to accomplish.

Finally, thanks goes to my typist Marie Pugsley for her patience and many hours spent in typing this thesis.

DEFINITION OF TERMS

THERMAL INSULATION For the purpose of this study thermal insulation is defined as a material which will retard the flow of heat between media of different temperatures as to reduce the energy loss and cost of equipment needed to maintain the temperature difference to economically acceptable levels.

K FACTOR Thermal conductivity, the reciprocal of thermal insulation, is commonly referred to as the k factor. The k factor is defined as heat flow per unit time, per unit area, per unit temperature difference, per unit thickness of material. The units that are usually used are

$$k = \text{Btu}/(\text{hr})(\text{ft}^2)(^{\circ}\text{F}/\text{in})$$

The lower the k factor the better the insulation quality.

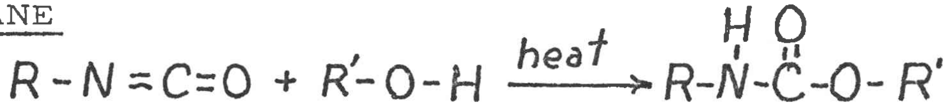
DENSITY The weight of a unit volume of material. In this paper the units will be in pounds per cubic foot. (lbs/ft^3)

CREAM TIME (Initiation time) The time interval between mixing the foam and the visible start of the foaming reaction. The start of the reaction is denoted by the mixture turning a creamy color or the mixture just beginning to rise.

RISE TIME The time from mixing the foam components to the time the foam stops rising in an open container.

TACK FREE TIME The time from the mixing of the foam components to the time a wooden spatula touched to the foam surface just ceases to stick to it.

URETHANE



$$R-N=C=O + R'-O-H \xrightarrow{heat} R-\overset{H}{\overset{|}{N}}-\overset{O}{\overset{\|}{C}}-O-R'$$

SURFACTANT A silicone material usually blended into the "B" component which lowers surface tension and reduces bubble collapse.

CHAPTER I

DEFINITION, SCOPE AND LIMITATIONS

The Problem

Inadequate understanding and/or control of the polyurethane rigid foam process normally requires five to ten percent overpack of cavities to obtain correct fill and foam characteristics. This practice of overpacking has resulted in excessive waste and cost.

Motivation and sponsorship for this research was initiated by Whirlpool Corporation to enable them to reduce the cost of producing refrigerator cabinets.

Economic Importance of Study

Solving of problems associated with overpack of cavities will contribute significantly to lowering industrial cost of rigid foam systems. Two areas of cost savings involve the foam itself and the support equipment.

The excess material required above the theoretical amount to fill refrigerator cavities costs one manufacturing company around $1300.00 per day. "Those industries using rigid foam as an insulator are usually consumer oriented and highly cost competitive. Under such circumstances a difference of 1.0 percent in loss factor may represent many thousands of dollars which could be the difference between a profitable operation and a marginal or loss position."(1)

The other area that overpack affects directly is the cost of fixtures. The cost of fixtures may reach 50 percent of the total cost of a newly installed foam system. (1) The reason for expensive fixtures comes as a result of excessive pressures developed during foaming. In usual overpacking procedures the pressures created by the rising foam may be between two and seven pounds per square inch. When looking at a large area, such as one side of a refrigerator, the total force may reach 15,000 pounds.

Method

To obtain a good feel, perspective, and orientation to the research, it was decided to first visit the Whirlpool plant in Evansville, Indiana, to see firsthand their polyurethane foam system and to become acquainted with and interview those who had experience with the foam process.

The next step was to complete a library research and glean pertinent information on the rigid foam process, experimentation techniques, and results of other researchers.

These two steps helped in identifying process variables, limiting the research, and selecting and designing appropriate experimental techniques and equipment.

Scope of Study

The scope of this research was to determine the sensitivity of the process to pre-mixed viscosities, temperature, mix ratio, and mixing time or blendability of the foam ingredients; and temperature dimensions and surface finish of the mold on the rigid polyurethane foam process. The overall goal will be to decrease the percentage overpack needed while maintaing the correct "k" factor or thermal properties of the foam. Both experimental and literature review will be utilized to accomplish this goal.

Limitations

The formulation used in the experimental tests will be the particular set of ingredients used by Whirlpool to fill their refrigerators. This will be the only foam ingredient system experimented with. Since this formulation is of proprietary nature, the exact manufacturer's name will not be used. The formulation is listed below by percent weight.

Table 1

Foam System Formulation

Masterbatch	Percent by Weight
Resin	67.727%
Surfactant	.756%
Water	.481%
Catalyst	.463%
Catalyst	.072%
Freon R-11	30.501%
	100.000%

The mix ratio for the two ingredients was calculated to be

$$\frac{\text{Masterbatch} = 140 \text{ Parts}}{\text{Isocyanate TDI} \quad 100 \text{ Parts}}$$

The formulations used throughout industry are varied and each company has worked up their own "best" formulation. Some of the well known companies that have rigid foam formulations are: DuPont, Mobay, UpJohn, PPG Industries, Olin, UniRoyal and Union Carbide.

The filling of a refrigerator cavity was simulated during experimental research by using a specially constructed test cell. The changing viscous nature of the foam as it rises up a column was taken into account in designing the test cell. The test cell was constructed to maintain the same volume to surface ratio(2) as foam rises up one side of a refrigerator. Refrigerator cabinets are usually filled laying on their back.

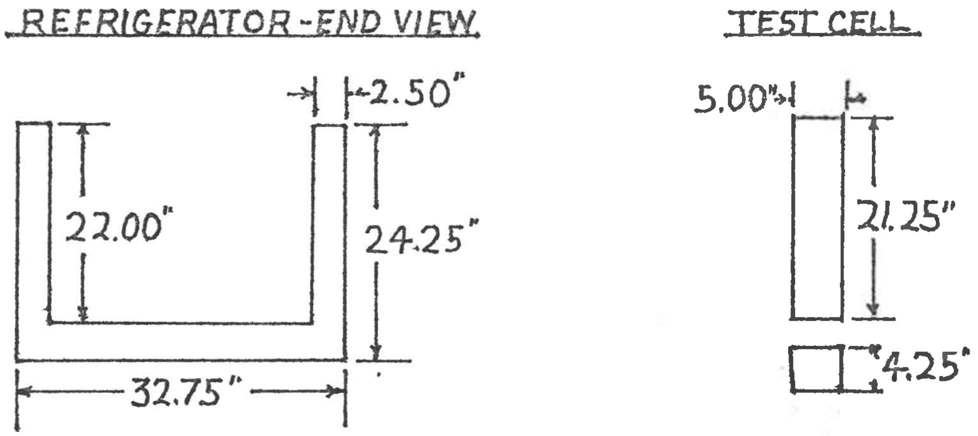


Fig. 1. Relationship between refrigerator cabinet and five inch diameter test sample

The calculations used in maintaining similitude between the refrigerator and test cell are shown below.

VOLUME TO SURFACE RATIO

REFRIGERATOR | TEST CELL

$$\frac{WL}{2(W+L)} = \frac{W}{2} \qquad \frac{\pi D^2}{4\pi D} = \frac{D}{4}$$

SIMILITUDE

$$D = 2W \qquad \text{WHERE } W = 2.5" \quad D = 5.0"$$

W is the width of the refrigerator, L is the length of the side, and D is the diameter of the test cell.

Humidity

It has been found that humidity has some effects upon the final foam characteristics. During the tests, humidity will be monitered and the data will be presented, but no attempt will be made to correlate this data to the final results of the experiments. It is thought that its effects will be neglible with respect to the other variables.

k Factor

Density and foam cell size will be used to determine the final characteristics of the foam instead of using the k factor determination. The relationship between k factor and density is shown in Figure 2 and between k factor and cell size in Figure 3.

At lower densities the k factor is also lower until an optimum point is reached, between 1.5 and 2 lbs/ft^3. The lower density is wanted because of the low conductivity of the gas in the cells, (3) but this only happens to a point as the density decreases the cell size increases. This gives rise to interconnected cells which increases convection currents. The cells are also weak and are easily ruptured. (1) Therefore, the thermal conductivity increases at densities below 1.5 lbs/ft^3.

The relationship between cell size and k factor is almost linear(4). As the cell size increases the efficiency decreases.

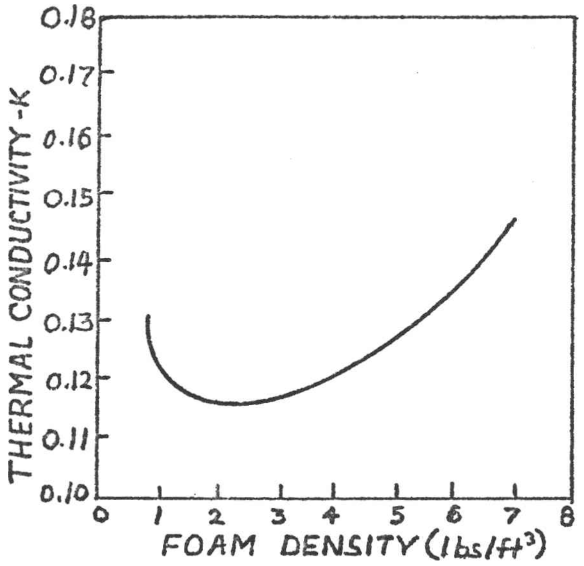


Fig. 2. Relationship between k factor and density

SOURCE: Lane, Roger I. 1965. <u>Processing handbook rigid urethane foam</u>, p. 2. Westport, Conn.: Technomic

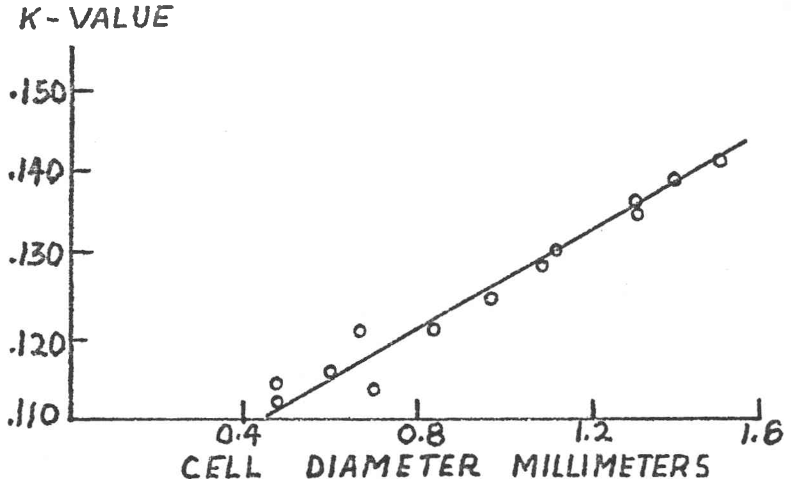


Fig. 3. Relationship between cell size and k factor

SOURCE: Buist, J. M. and Gudgeon H. 1968. <u>Polyurethane technology</u>, p. 212. London: MaClaren.

Actual production equipment will not be used during the experiments. Mixing will be accomplished by using a high speed stirrer with a two inch diameter, three propeller blade, and metering by use of a balance scale for one ingredient and a syringe for the other. All production operations will be simulated using specially designed equipment.

The limitations may be summarized as: (1) A sample test cell will be used to determine flow in a refrigerator cavity; (2) Humidity will be monitored but not correlated with the other data; (3) Density and cell size and structure will be used to determine the foam's thermal qualities; and (4) Actual production equipment will not be used during tests.

CHAPTER II

BACKGROUND

This chapter acquaints the reader with rigid polyurethane foaming. It will contain information on the following: (1) Polyurethane as an insulator; (2) Composition of rigid polyurethane foam; (3) Research of others concerning overpacking; and (4) Explanation of the foaming system.

Polyurethane, an Insulating Material

Polyurethane foam was selected as an insulating material because of its k factor. Donald S. Gilmore Research Laboratories (5) reported on some comparisons carried out between isocyanate based rigid foams and other insulating materials. For each of the materials the k factor was measured at 25°C. The measurements showed that the polyurethane foams have an insulation efficiency about twice that of other common insulating materials. These tests also showed that the k factors of all insulating material decreased as temperature decreased, but the rigid foams decreased at a slower rate.

Table 2

Typical k Factors for Commercial Insulation

Material	k Factor Btu/(hr)(ft^2)(°F/in)
Asbestos	1.08
Powdered Gypsum	0.50
Wool Felt	0.48
Foamed Glass	0.40
Sawdust	0.36
Wallboard, Insulating type	0.34
Rock Wool	0.30
Cork	0.30
Animal Wool and Hair Felt	0.25
Polystyrene Foam	0.25
Fiberglass	0.24
Rigid PVC Foam	0.17
Urethane Foam, CO_2 expanded	0.23
Urethane Foam, Freon 11 expanded	0.11-0.15

Polyurethane foam has been slow coming into use because of its higher cost, but since recent development by the chemical companies which decreased its cost and the increased pressures for better insulation because of the energy shortage, the polyurethane industry is now rapidly expanding. By filling the same refrigerator space that was once filled with fiberous glass, the insulating qualities could be practically doubled using urethane foam.

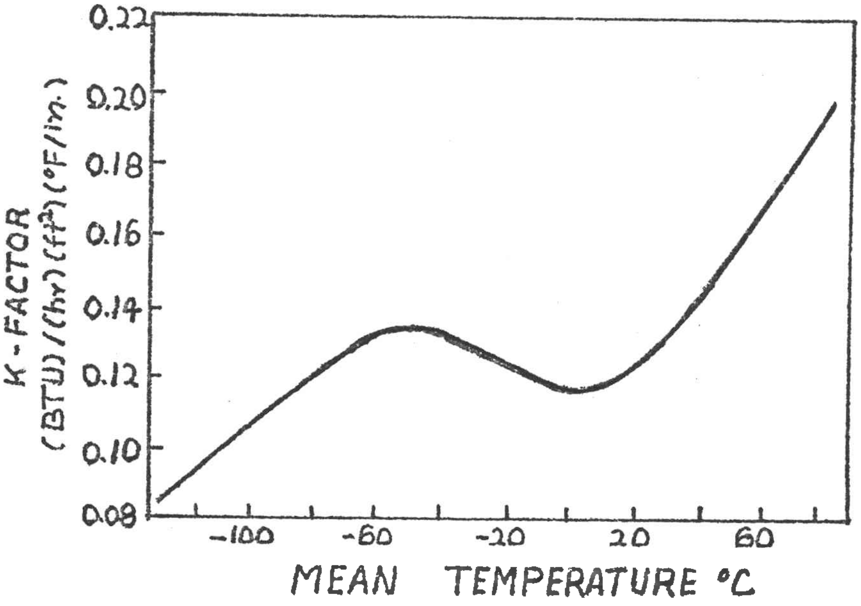


Fig. 4. Effects of temperature on the thermal conductivity of rigid foam blown with trichlorofluoromethane (Freon 11)

SOURCE: Backas, Jack and Gemeinhardt, P. G., Plastic foams, ed. Frish, Kurt and Saunders, James H. 1973. p. 498. New York: Marcel Dekker, Inc.

Composition of Rigid Polyurethane Foam

Rigid polyurethane foam consists usually of two reactive liquid components, each of which is stable by itself. One component is an isocyanate (usually referred to as the "A" component), the other component (called the "B" component) is a blend of polyol, catalyst, surfactant, and blowing agent. The polyol is a hydroxyl

(-OH) bearing material which reacts with the isocyanate. Catalysts are added for control of the rate of reaction and the cure time; and the surfactant is a silicone material that lowers surface tension and reduces bubble collapse. When these two chemicals are mixed together the exotherm (heat generated during the reaction)vaporizes the blowing agent to produce the foam structure. The blowing agents for insulating foams are usually R-11 (Trichloroflouromethane), carbon dioxide, or sometimes R-12 (Dichlorodifloromethane). The foam used for this research was blown by R-11. R-11 is usually used because of the lower k factor that it exhibits.

Table 3

K Factor of Gasses

Gas	k Factor Btu/(hr)(ft^2)(^oF/in)
Air	0.168
Carbon Dioxide	0.102
R-11	0.056

The Procedure of Five to Ten Percent Overpacking

It is a common practice in industry to calculate the amount of polyurethane foam it takes to fill a cavity and then add 5 to 10 percent to insure proper fill. It has been hard to determine why this is needed, but all authors agree that it is a good practice.

Probably the best reason given for the need of overpacking is to apply some back pressure on the foam so as to make the cell more spherical in shape. When the foam rises in a column the cells are elongated in the direction of rise. Following are some quotes on the procedure of overpacking:

> Usually, depending upon the operator (in batch operation), it is found that a 10% to 30% excess over the amount theoretically needed to fill the mold is required for a good fill. Little variation is found in foam densities within these limits. When continuous metering and mixing are employed, lower foam densities are observed. *Apparently, a more efficient utilization of the gas blowing reaction is obtained.* For mechanically dispensed formulations, excesses in the neighborhood of 5% may be used. With this excess, wall pressures up to 2 to 3 psi may be expected. (2) (Italics mine)

M. Kaplan(6) makes the following observation concerning the packing of urethane filled cavities:

> *Packing* To ensure complete fill of mold extremities and optimal foam properties, a foam charge 5-10% in excess of that needed for an unrestrained foam rise should be used for closed molding as described by Jones. *Use of too little results in nonuniformities and slightly soft foam at the top of the panel.* Use of excessive packing results in higher panel densities and higher pressures which, without adequate jigging, may distort the mold. (Italics mine)

In another work by Buist, Hurd and Stafford(4) the reason for overpacking is for dimensional stability.

> Another method of improving the dimensional stability of rigid foam is the technique known as "overpacking" or pressurization. This is a technique whereby more foam is injected into a cavity than that which is strictly

> necessary to fill it. *The effects of this overfilling is to build up internal pressure in the foam system while it is still in the fluid stage.* Since this pressure is equal in all directions, more uniform cells are produced and the anisotropy of the resulting foam is reduced. (Italics mine)

Lane(1) goes into more detail concerning the cost factor and ideal level of overpacking:

> When estimating the cost of a product in which a cavity is to be filled with rigid urethane foam the most difficult cost component to obtain with any degree of confidence is that of the urethane material itself. Calculation of the size of the cavity is, in itself, far from simple when the cavity is of irregular shape. Using again the example of the household refrigerator with urethane foam insulation, the insulation cavity between the outer cabinet and inner liner will vary in thickness as a function of the design and will also vary as a function of component dimensional tolerances. Contained within the insulation cavity will be support brackets, refrigerant carrying tubes, sealing tapes and mastics, pads of glass fibre, and so on all of which make accurate calculations of the volume of the insulation cavity extremely difficult. *For reasons unknown theoretical calculations of the volume of cavities at this stage of product design usually understate the volume.* Only experience with a particular type of product and design will give an indication of the correction factors which should be applied. (Italics mine)

Lane(1) also takes into account other losses such as the amount left in the mixing chamber of the equipment, and what he calls the buoyancy loss. He continues that other losses will occur because of operator error, and through machine calibration and testing. "All of these losses combined should not exceed 10% in an efficiently run operation, 5% may be considered as near ideal level." The purpose of this overpack, he states, ".....is to ensure that the cavity is always completely filled even when the dispensing equipment is operating at the lower limit of its tolerance, to obtain a complete fill on occasions when material, mould or product temperatures are at the lower tolerance limits and to ensure that uniform pressure is exerted on the foam mass just near the top of rise to create a uniform, orthotropic cellular structure and also to ensure that the still flowing foam is forced into all the areas of the cavity into which it is required to penetrate."

To summarize, it is a general feeling by those experienced with urethane foam that it is a good procedure to overfill cavities by 5 to 10 percent to obtain optimum foam characteristics.

The challenge is to see if there is a way to decrease this amount needed by careful examination of process variables, and still obtain optimum foam qualities.

The Foam System

The major disadvantage of using rigid polyurethane foam is that it requires a chemical plant for installation. When installing fiber glass, all the manufacturer has to do is to take it out of the box and place it in the refrigerator cabinet. When installing polyurethane many variables must be taken into account, if these variables are not controlled quite closely, using polyurethane could be a sad and expensive experience. Many types of mixing heads and chemical plants have been developed for this process. Some have worked quite well under given conditions, but have failed miserably under others. Some experienced with urethane foam feel that this process is an art rather than a science.

Rigid polyurethane foam systems are set up to accomodate usually one out of the three types of foaming applications: (1) The intermittent two-component system for the foaming in place of mold cavities; (2) The continuous two component system for producing foam buns and slabs, and (3) the frothing system where R-12 is used as a third stream for pouring molding of cavities. This paper will explain the intermittent process since this is the type of system that Whirlpool uses for the filling of refrigerators and where overpacking is used.

The intermittent foam system consists mainly of four basic components: the tanks or reservoirs where the chemicals are stored, the mixing and metering equipment, the preheat and cure oven, and the fixtures for holding and securing the molds while the foam is rising and curing in the cavity.

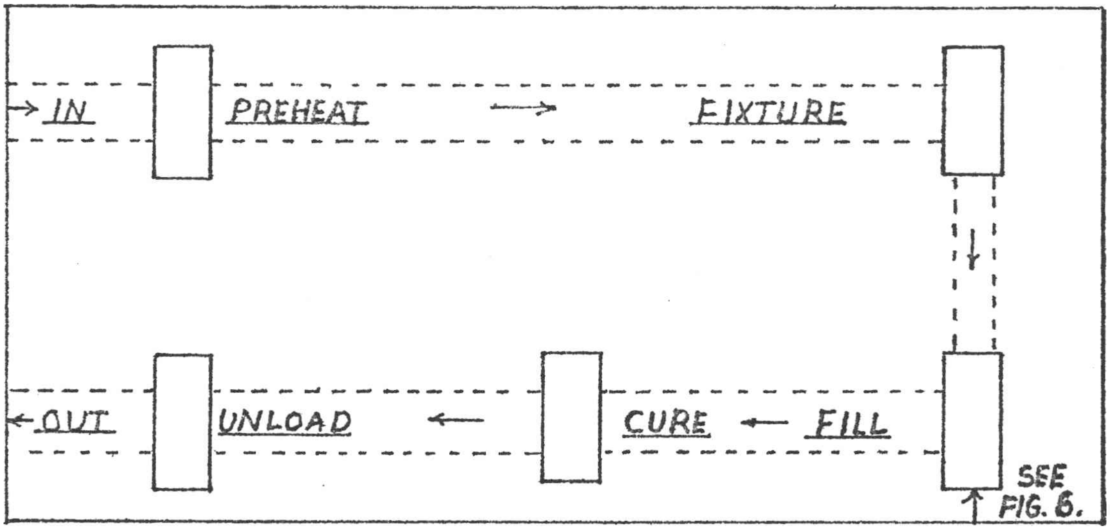


Fig. 5. Foam support system

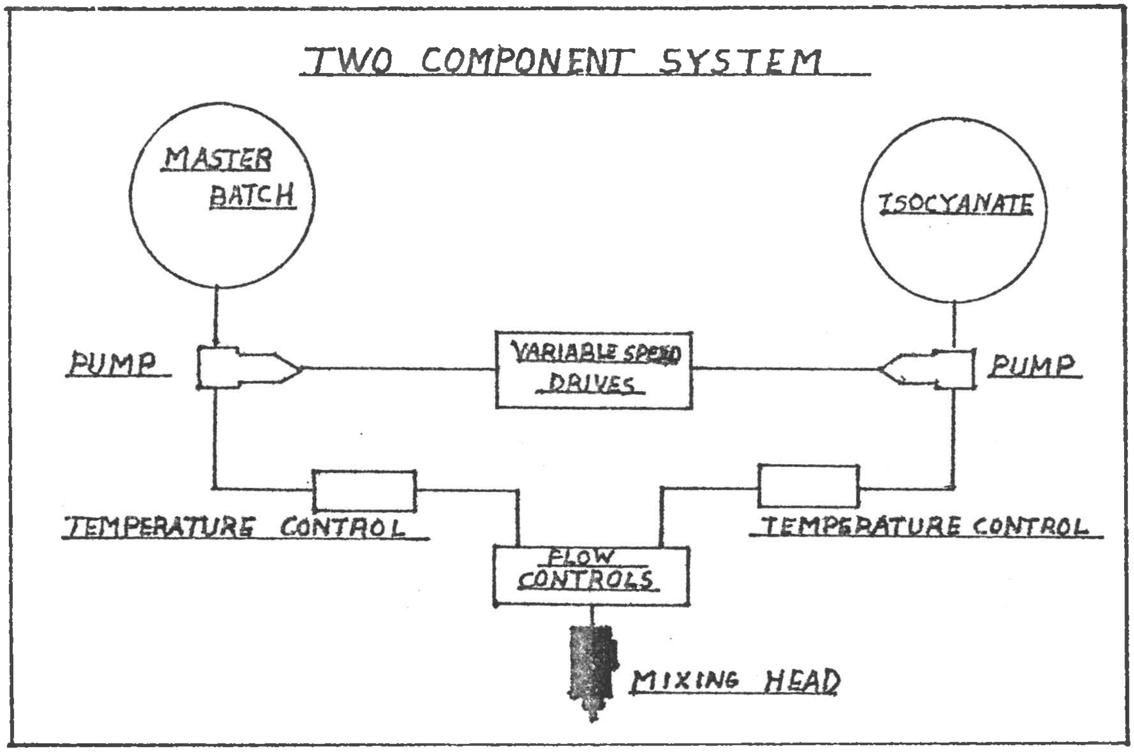


Fig. 6. Foam chemical transfer system

Chemical Holding Tanks. There are many types of chemical holding systems in operation. The basic chemical storage system takes into account the reactivity of the components. The storage tank for the "B" component is usually made of steel or black iron, but the "A" component is more reactive and must be stored in stainless steel or epoxy-phenolic lined tanks. Most tanks are equipped with agitators or stirrers to keep the chemicals mixed and at a constant temperature, and blanketed with nitrogen to prevent the chemicals from reacting with or picking up humidity from the air. To keep the ingredients at certain temperatures, heat controlling devices are either added to tanks or heat exchangers placed on the transfer lines to the mixing and metering equipment. It should be noted that the lines or pipes are also constructed to account for the reactivity of the chemicals.

The Toluene Diisocyanate (Isocyanate) causes health hazards and precautionary measures are taken to assure that only certain amounts are concentrated in the air.

Mixing and Metering Equipment. Initially the foamed in place systems were handled by bathwise stirring due to the lack of equipment. In this process the various foam ingredients were weighed out and placed into a container and blended with spatulas or stirred with mechanized mixing devices. The mix was then quickly poured into the desired cavity for foaming. It was important that the stirring was of a "blending type" and not a "beating" one. (2) Since that time, elaborate mechanized equipment has been developed for this operation. There are many machines available on the market today, and much literature has been written on the different makes and types. A brief review of the machines available on the market today was written by Vincent Johnson(7) and is included in Appendix A.

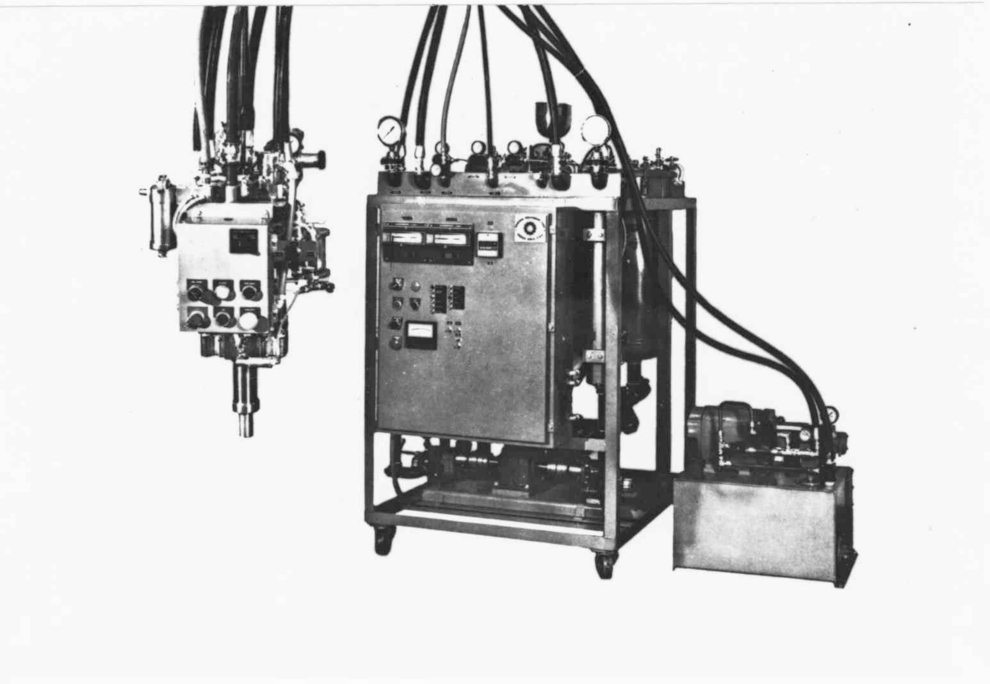


Fig. 7. Admiral Polyurethane Pour Machine

The equipment has two vital functions: (1) to proportion the components accurately to the mixing head, and (2) to properly mix the components there.(8) The importance of the proportioning equipment is then to make sure that correct shot sizes are delivered to the mixing chamber and there be completely mixed.

There are two types of metering pumps used: the fixed displacement gear pumps, and piston pumps with either variable speed drives or adjustable stroke lengths.

"The mixing is just as important as the metering of the foam ingredients since poor mixing nullifies proportioning by the most elaborate pumping and metering devices.(9) There are two main types of intermittent mixers. One uses mechanical means for mixing and the other is an impingement style(10) where mixing is accomplished by using high pressures and the kinetic energy of the two components.

The most common type is the mechanical mixers. The agitator is most usually of helical gear type similar to the one shown in Figure 8. Most mixers provide high shear requiring small clearance between the mixer and housing.

"As a basic concept, a polyurethane foam machine is relatively simple. It delivers two or more liquid feed streams into a mixing device. However, the required accuracy in temperature, feedstream delivery, and ratios has created the need for a precision machine."(11) One should become familiar with the foam equipment available. Some machines have worked extremely well under certain conditions, but have failed miserably under others.

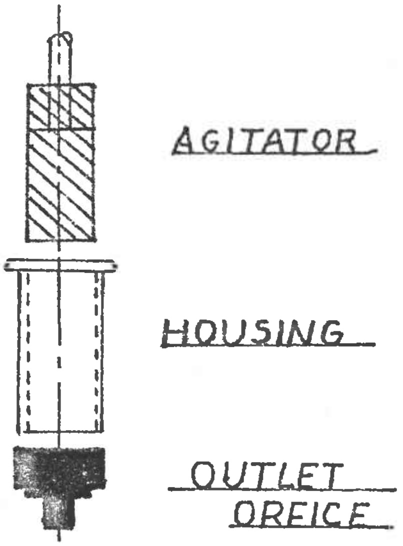


Fig. 8. Typical mixing chamber

<u>Preheat and Cure Ovens.</u> The temperature makes a big difference in the final foam characteristics when filling a product or cavity.

> If the temperature of the mould is too high the foaming reaction will become more active, the rise will be more vigorous and there is a probability that the blowing agent will flash off prematurely causing unacceptable surface conditions in moulded products, and cavities, small voids and irregular cell sizes in all products. When the product or mould is too cold at the time the foam is injected the foaming reaction is suppressed and the heat generated by the reaction between the isocyanate and the polyol may be absorbed too rapidly by the surroundings with the result that the density of the foam near the cold surfaces will be high, the rise of the foam will be sluggish and the mould, or cavity may not be completely filled. (1)

The purpose of the preheat oven is to bring the products to a correct temperature range so as to take advantage of the exothermic reaction. The products to be foamed are passed through the preheat oven just prior to foaming to bring them up to temperature. The preheat oven should be designed to keep the cabinets at an optimum, consistent, and uniform temperature no matter what the production rate.

The purpose of the cure oven is to speed up production. If the foam is postcured the time in the jig is reduced considerably. (12) No postcure oven is needed, however, if this is not a factor since the restraining fixtures will usually retain the heat necessary for the cure. Microwave, dielectric and infrared are being used commercially to help speed up the postcuring procedure. (11)

Fixtures. The fixtures are designed to provide three basic functions: (1) To restrain the pressures developed during foaming; (2) to secure the products to prevent warpage; and (3) to retain the exotherm heat.

Because of the pressures developed during foaming, adequate fixtures are required. If the fixtures are not well built they will need to be replaced in a few years adding additional cost to the manufacturing operation.

If the surface finish of the product is critical, close tolerances are needed between the product wall and the fixture so that warpage or ripples in the metal surfaces are not noticeable.

The temperature of the fixture is also critical. Some companies pass the fixture through a preheated oven to get it to correct temperature before starting a shift. If this is not done, excess material is placed into the product at the start of the shift to allow for the lower temperature. After twenty minutes the exotherm heat generated from the reaction will have the fixture to a correct temperature range. People experienced with urethane foaming(13, 14, 9, 1) feel that the temperature of the fixture is very hard to control and keep constant during a production run because they are continuously heating from the exotherm heat. Since the temperature of the fixture changes, this is a critical area of concern when trying to control the process variables.

The construction materials that are used are also of concern. If the fixtures are made of metal, the metal conducts the heat too rapidly from the reaction causing poor foam characteristics. Most fixtures are made of wood and plastic material to retain the exotherm heat.

Some have experimented with heated jigs, thereby eliminating the need for the preheat and cure ovens. Perhaps more experimentation should be done to find ways to control the temperature of the fixtures. An example might be in using water as a transfer medium to keep the fixtures at constant temperature.

CHAPTER III

PROCESS VARIABLES

The purpose of this chapter was to take a closer look at the variables associated with the rigid foam process by reviewing literature that has been written and results gathered by other researchers. These include variables associated with the foam, equipment, and mold cavity.

Variables Associated With the Foam

The variables associated with the foam would include: (1) the ingredients, (2) the temperatures of premixed ingredients, (3) viscosities of the ingredients, (4) ratio of ingredients, (5) flow characteristics of the rising foam, and (6) environmental factors.

The Ingredients.

The purpose of this study was not to go into the compositions of different makes and types of foam, but to use one specific foam for experimentation and find its sensitivity to process variables. Many excellent articles are available(2, 8, 9, 12, 15, 16, 17, 18, 19, 20) on the different foam ingredients.

The ingredients include the A and B components. The B component, as mentioned earlier, is a combination of polyol, catalysts, surfactants and blowing agent. The catalysts of the system should be balanced so that the top of the rising foam does not solidify before the mold is completely filled. (2) The surfactant is an important part of the mixture in that it helps to form a homogeneous mixture assuring complete and uniform reaction, and also helps control cell size and formation. (12, 18, 21, 22) The amount of blowing agent in the mixture has an influence on the density of the molded part(24). It serves as an expanding agent for the foam and also contributes to the low thermal conductivity of the foam.

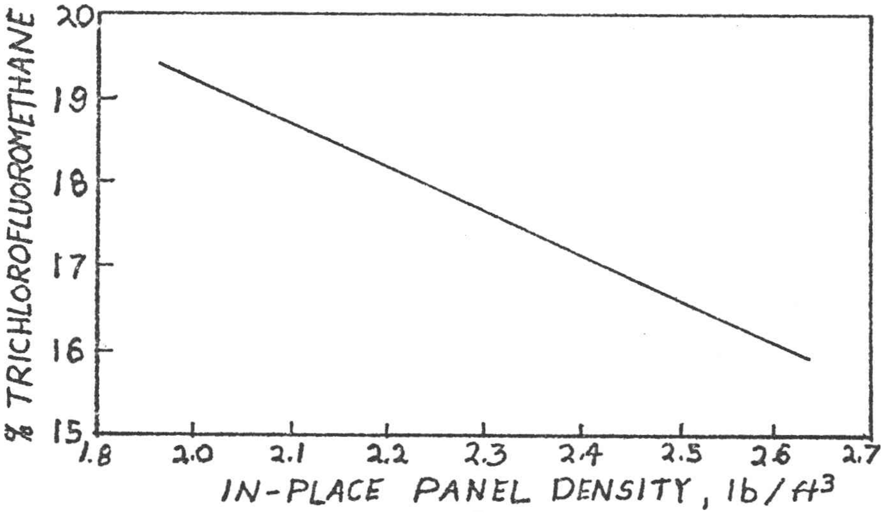


Fig. 9. In place panel density versus percent trichlorofluoromethane for a one-shot rigid foam

SOURCE: Kaplan, M. 1967. Formulation and processing techniques. Rigid Plastics Foams. p. 106. Ferrigno, T. H.

Temperatures of Premixed Ingredients. The component temperatures affect the repeated accuracy of the metering system, the mixers efficiency, the flow characteristics in the cavity and the final quality of the foam. (1)

If the ingredient temperature is increased, the density decreases, but the bad feature is that the foam structure may become "course" and "irregular". (2)

Kaplan(6) mentions that, ". . . . in general, the reaction rate increases with increasing ingredient temperatures, " and that density decreases. Shown below are the results of his experiments.

Table 4

Effects of Ingredient Temperature on Reaction Times in a One-Shot Rigid Urethane Foam System

Premix	Isocyanate	Cream Time Sec.	Rise Time Sec.	Tack free Time Sec.
70	71	17	140	145
78	123	9	100	110

Table 5

Effects of Ingredient Temperature on Panel Density in a One-Shot Rigid Urethane Foam System

Foam	Ingredient Temp. °F Premix	Ingredient Temp. °F Isocyanate	Trichloro-floromethane parts per 100 parts polyester	Overall panel density lbs/ft^3
A	77	77	45	2.3
B	87	125	45	2.0
C	77	77	52	2.0

NOTE: Panel size 24 X 24 X 1.75 inches.
On foam C the temperature was kept the same for ingredients, but Freon level was increased to decrease density.

Viscosities. The main area where the viscosity of the components is critical is in the mixing and metering operations. Doyle(8) mentions that the metering part is generally governed by the viscosities of the foam system used. "The higher the viscosities, the harder it is to handle the components. Where one component is much higher in viscosity than the other, the problem is compounded, and metering can become very irregular with many machines." The viscosities are regulated by changing the temperature. In most operations the A component is chilled because it is of lower viscosity and the B component slightly heated to make them similar in viscosities.

The mixing operation is also affected by the viscosities of the components. The clearance between the agitator and housing provides a high degree of mixing shear. As viscosities increase, so should the clearance. (6) On impingement mixers it is critical that the viscosities be similar for a good mix. (10)

If the viscosities are similar there is also a better chance for complete mixing because of the similarity between the two components.

Viscosities are often measured in industry by use of a Brookfield viscometer. (23) A sample viscosity curve for TDI is shown in Figure 10.

Ingredient Ratio. The ingredient ratio is the ratio of the weights of the two ingredients. The stoichiometry of ratio between the NCO to OH should be from 1.00 to 1.03. This ratio of NCO to active H ions is sometimes called the TDI index. From using this index, the ratio of weights of ingredients can be established. The foam system used in experimentation, as mentioned earlier, is 140 parts Masterbatch to 100 parts TDI. It is of utmost importance that this ratio be kept as close as possible for optimum foaming.

Flow Characteristics. When the two components are mixed the foam begins to expand and to fill the cavity. This expansion is in the range of thirty times the original volume.

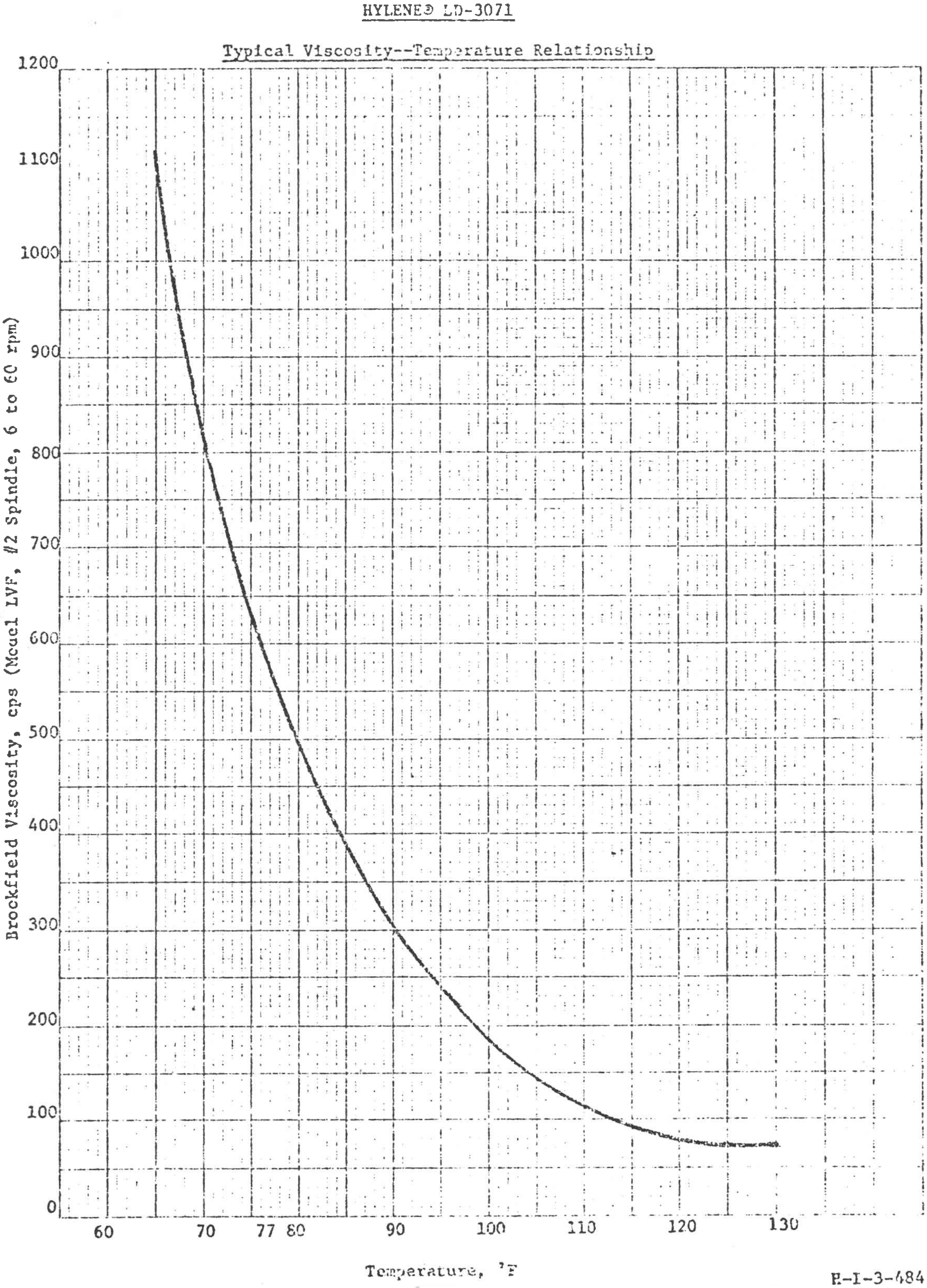


Fig. 10. Typical viscosity versus temperature curve for TDI

"During this filling period the viscosity of the reacting mixture increases markedly from the initial low value of the liquid mixture to the high value of the final polymerized foam structure."(4)

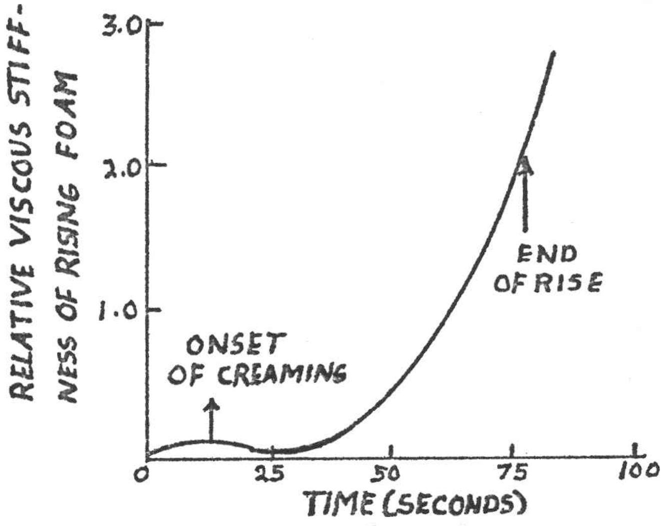


Fig. 11. Variation of relative viscous stiffness with time of a reacting foam from the initial dispensing of the liquid mixture

SOURCE: Buist, J. M., Hurd, R. and Stafford, R. L. 1968. "Rigid foam: Manufacture and properties" Advances in polyurethane technology. ed. by Buist, J. M. and Gudgeon, H. London: MaClaren

Dave Hart(14) mentioned that most mistakes are made in designing for foam because the designer is not familiar with the foam abilities. He mentions the following concerning the reaction:

> As the polymerization proceeds, more heat is given off and the viscosity of the liquid and ensuing foam becomes greater. When the foam is well on the way to complete polymerization, it is extremely viscous and will not flow well. In order to get good foam flow, the foam should flow during the primary polymerization stage. If the polymerization exotherm is contained within the liquid mass, foaming will begin as soon as

> the materials begin to polymerize. If this occurs the area foamed will be filled with foam before the polymerization has proceeded to a point that produces a viscous foam that will not flow. In order to overcome this problem, one must design so that the exotherm remains in the original mass.

Some catalysts are added to the foam to help delay the curing of the foam until the foam has filled the cavity.

The Urethane Foam Flow Test, and ASTM Standard test for Rate-of-Rise (Volume Increase) Properties of Urethane Foaming Systems(23) have been developed to determine the foam's abilities. See Appendix B.

Environmental

Foam is about 97 percent gas by volume. The pressure in the rising foam must exceed atmospheric pressure, otherwise the foam will not rise.(4) "Bubble growth will continue from an area of high pressure to an area of low pressure."(22) After the foam fills the cavity the temperature of the cavity continues to increase until a maximum is reached. This temperature can reach as high as 160°C.(4) The pressure of the gas in the cell can be determined using the gas laws.

From this discussion, it could be inferred that barometric pressure could have some effect on the rising foam.

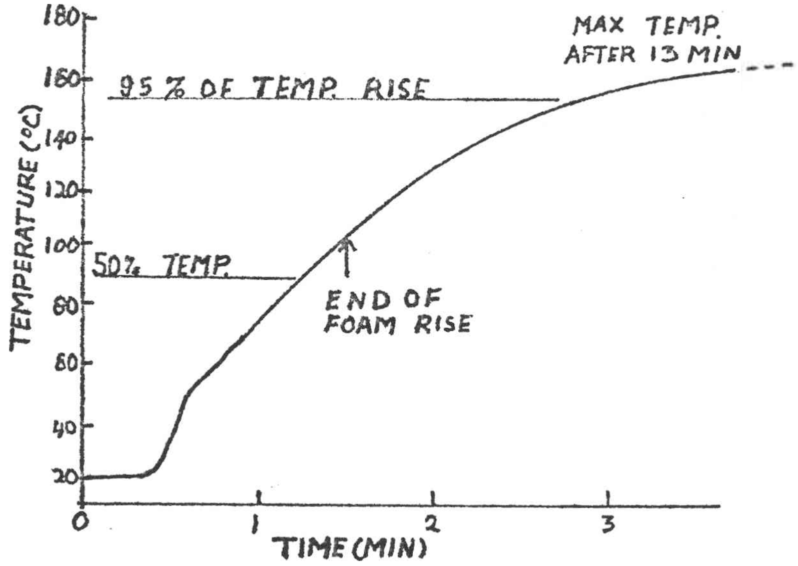


Fig. 12. Increase in internal temperature of a typical foam from the time of dispensing of liquid mixture

SOURCE: Buist, J. M., Hurd R. and Stafford, R. L. 1968. "Rigid foam: manufacture and properties." Advances in polyurethane technology. ed. by Buist, J. M. and Gudgeon, H. p. 205. London: MaClaren

Strickman(22) mentions that one reason for non-uniform urethane foam "is the presence of excessive humidity in the reacting materials due to the entrapment of ambient air. In small amounts the 'air entrapment' enhances the cell structure." Excessive air gives poor foam because excessive moisture interferes with the nucleating agent.

The temperature of the air could also have an effect on the final foam characteristics. Hart(14) reports on an experiment where he foamed a container measuring twelve inches by twelve inches by one and one-half inches on end and laying on its face. One had 144 square inches exposed to the air above the rising foam and the other eighteen square inches. The results showed that 120 grams of two pound per cubic foot density foam was required to fill the container on its end. This same amount only filled 65 percent when on its face. The reason for the decrease in fill was attributed to the escape of the exotherm to the air.

Variables Associated With the Mixing and Metering Equipment

As mentioned earlier, proportioning is critical and is affected by the viscosities of the foam, the pressures of the streams, and the pump speed. Most machines are calibrated at the beginning of the shift to insure that the ratios are correct.

Kaplan(6) makes some excellent observations concerning the variables of the mixing operation:

> The function of the mixing head is to provide sufficient mixing so that the streams are thoroughly blended and the resultant foam has fine uniform cell structure and optimal physical properties. Insufficient mixing will result in a weak foam with coarse, irregular cell structure. The degree of mixing achieved in the head is largely dependent upon *agitator design, clearance between housing and agitator, size of outlet orfice, and agitator speed.* (Italics mine)

He mentions that the "high-shear, helical-gear type" of agitator is preferred because it consistently provides thorough mixing and fine cell structure.

The clearance between the agitator and housing, he states, should be between 0.006 and 0.030 inches. The higher the viscosity the more clearance is needed. The orifice on the outlet of mixing chambers provides back pressure and increases the mixing action. He states, "Excessive restriction increases cell size, which is undesirable. Optimal results have been achieved by using the smallest orifice that does not significantly increase cell size."

Speed affects the degree of mixing. Kaplan mentions that the "normal speed" is usually 4000 to 6000 revolutions per minute.

Polyurethane foaming equipment manufacturers have put much research into making the proper equipment. The company obtaining the equipment should be careful to pick the proper equipment that will be compatible with the foam ingredients they use; and that will have the proper output capacities needed for a given operation.

Variables Associated With the Mold

The variables associated with the mold cavity are the mold temperature and the physical makeup of the mold.

Mold Temperature.

It is generally felt that mold temperatures of 110 to 130°F are the best range for metal molds to insure low panel densities and optimum k factors. (18, 12, 6) Lower temperatures result in higher density due to the loss of exotherm heat. A thicker skin is also formed on the foam that may increase the k factor. (18)

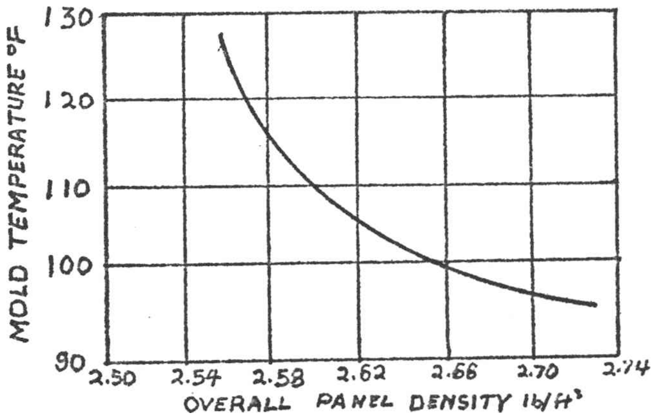


Fig. 13. Mold temperature versus in-place panel density for a one-shot system

SOURCE: Ferrigno, T.H. 1967 Rigid Plastic Foams p. 112 New York: Reinhold

Hart(14) Carried out tests by changing the mold temperature in 10° increments from 80°F to 160°F on two molds made with walls different distances apart. The molds used were made of 3/4 inch plywood covered with 20 guage cold rolled steel. The foam was poured into vertically positioned cabinets and allowed to rise freely with no packing. The dimensions for the molds were 24 inches by 24 inches by two inches and one and one-half inches. The results for the two panels foamed are shown in Figure 14.

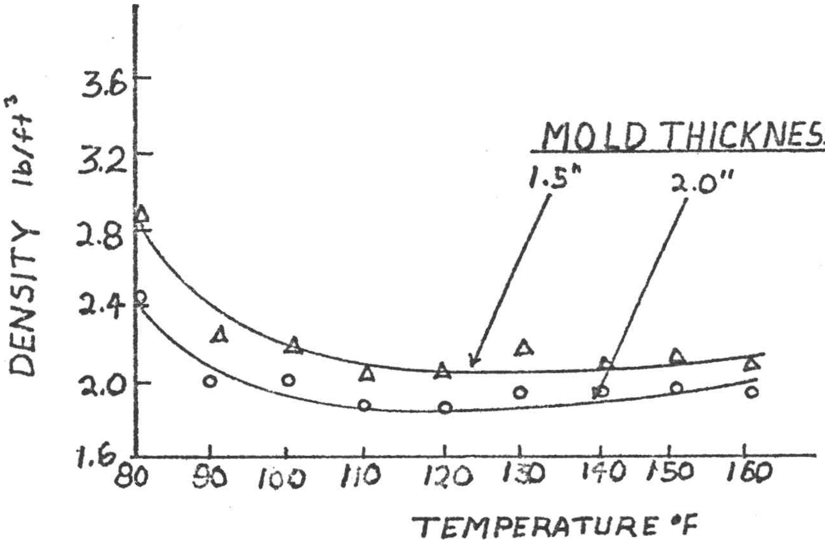


Fig. 14. Panel density versus mold temperature

SOURCE: Hart, David. "Polyurethane foam insulation in household refrigerators design considerations techniques and application." 1973 Unpublished

His results showed that very little density change was obtained after 90°F was reached for the particular foam system he used.

Physical makeup of the mold. Dombrow(2) mentions four factors that should be taken into account while designing the mold: (1) the material the mold is made of, (2) surface to volume ratio, (3) the size of pour needed, and (4) the restraints placed on the rising foam. Hart adds one more factor concerning the shape of the cavity. (14)

> Most mistakes are made in designing for the foam because the designer is most familiar with the foam's abilities. One ability that is most abused is the ability of the foam to rise in a panel and flow to all small concerns of the unit being foamed. The foam will rise nicely in a narrow panel, but avoid overworking the foam if possible. Causing the foam to change directions many times in its upward rise will overwork the foam and it will begin to drag at the top of the rise causing poor physical properties in this area. Attempt to give the foam a smooth rise and its physical properties will be uniform. (14)

<u>Mold Materials.</u> The materials are of two types; the conductors and nonconductors of heat. The insulating type of mold will produce lower-density foams than metal molds. This is because the exotherm heat is not lost through the walls.

Similar to this is a test made on panel thickness by Hart(14). Results shown in Figure 13 showed that as panel thickness increased, density decreased.

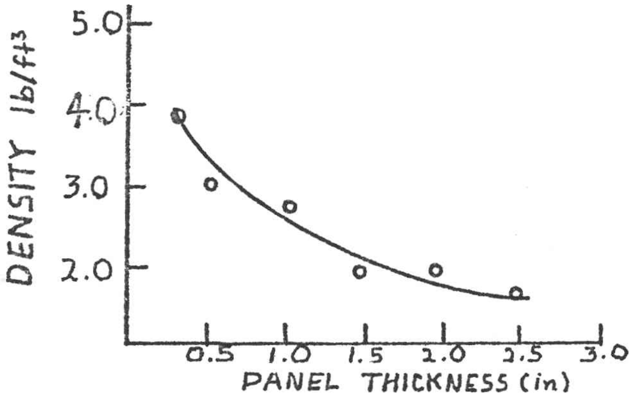


Fig. 15. Density versus panel thickness

SOURCE: Hart, David. "Polyurethane foam insulation in household refrigerators design considerations techniques and application." 1973. Unpublished

Surface to Volume Ratio. Tests have shown that the higher the surface ratio to the volume, the higher the density of the resulting foam. If an attempt is made to foam thin slabs, higher density also should be expected.

Size of Pour. The size of pour also effects the density. The larger the volume of material, the greater the exotherm(8) contained in the system.

Table 6

Effect of Size of Pour

Size of Pour (in^3)	Foam Density (lbs/ft^3)
12	7.6
60	6.0
200	4.7
900	3.9

SOURCE: Dombrow, Bernard A. 1965 Polyurethanes. Second Ed. p. 52 New York: Reinhold

Restraint. Openings should be placed in the cavity for the escape of the air being pushed out by the rising foam. The vents should be designed to prevent escape of the foam. Usually 1/16 inch diameter holes will allow air to escape, but not the foam.(6) On some operations fiberous glass is used as a foam stop, and to allow for the escape of air.

After review of the variables associated with the rigid urethane foam process one can now appreciate the statement made by Kaplan(24):

> The formulating and processing of rigid urethane foam is both a science and an art. It is a science in respect to the theoretical aspects concerning the catalysis and kinetics of the polymorization and cross-link density of the resulting foam. It should, however, be considered an art as well, since trace impurities or only minimal changes in formulation or mechanical processing can result in a foam with a radically different appearance and/or physical properties.

Limitiations of Previous Studies

Most of the studies made on rigid urethane foam have been dealing with one variable at a time while the others are held constant. It would be beneficial if a relationship could be established between the variables so that the rigid foam process could be controlled accurately and become more of a "science" and less of an "art".

CHAPTER IV

EXPERIMENTAL DESIGN AND PROCEDURE

After careful review of the process variables, an experimental design was next established. A foam ingredient system was donated by Whirlpool for research. (See Chapter I) The ratio between the masterbatch and TDI had been calculated as 140 parts masterbatch to 100 parts of TDI.

Test Design

To obtain meaningful data from the experiments, it was important that the actual production system and conditions be simulated as exact as possible. The foam system, as outlined in Chapter II, consisted of four main components: the chemical holding system, the mixing and metering equipment, the preheat and cure ovens, and the fixtures. The variables outlined in Chapter III were taken into account in the design. The preliminary steps associated with the experiments were to insure that the freon level was checked and corrected before each set of test runs, and the viscosity-temperature relationships were found for both ingredients to find the range where they were similar. A chemical storage and materials handling system was devised that would keep the chemicals mixed and at correct temperatures for the experiments. The mixing and metering of the two components was controlled so that correct proportioning and mixing would resemble that of industry. (This portion was difficult because of lack of the proper equipment.) A production oven was constructed to simulate the conditions used in industry. The test cell was designed to simulate the fill of refrigerator walls. Finally a means was devised to monitor the variables and obtain the desired results from the experiments.

Preliminary Tests

Freon Percentage Test. A freon 11 test was run on the A side to insure that the contents remained at the proper specifications. The freon 11 test consisted of the following: Between 10 and 20 grams of the A component was weighed out and placed into a preweighed three necked vessel. A thermometer was placed into one neck of the vessel. Into the other neck was placed a tube through which dry air was bubbled. The vessel was placed on a thermostatically controlled hot plate and maintained at 212°F for a period of 20 minutes. The sample was removed from the hot plate and weighed. The freon loss was calculated and corrected.

Viscosity Tests. To try and take advantage of viscosity similarities viscosity tests were run on both ingredients. To insure better control of mixing and metering the viscosity data is very important to calibrate commercial machines. On impingement mixers the viscosity is critical to insure proper mixing, since it is the kinetic energy that accomplishes the mixing.

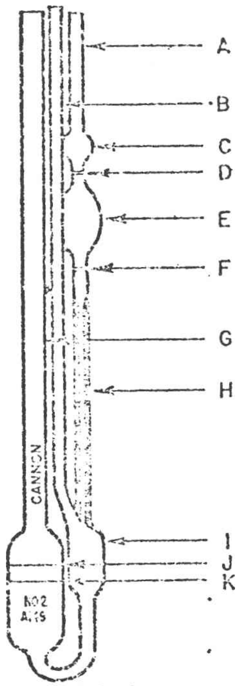
The tests were performed using Ubbelohde Viscometers. Three were selected in the range of the viscosity of the range of viscosity of the TDI. Numbers 2B, 2C, and 3 were used during the tests. The calibration date for these viscometers is located in Appendix C.

> The flow of a fluid in a capillary tube is the basis for viscometers of the Oswald-Cannon-Fenske or Ubbelohde type. In essence the viscometer is a U tube, one leg of which is a fine capillary tub connected to a reservoir above. The tube is held vertically, and a known quantity of fluid is placed in the reservoir and allowed to flow by gravity through the capillary. The time is recorded for the free surface in the reservoir to fall between two scribed marks. A calibration constant for each instrument takes into account the variation of the capillary's bore from the standard, the bore's uniformity, entrance conditions, and the slight unsteadiness due to the falling head during the one to two minute test. Various bore sizes can be obtained to cover a wide range of viscosities. Exact procedures for carrying out the tests are contained in the standards of the American Society for Testing and Materials.(25)

The procedure shown in Figure 16 was followed to get the results obtained by the experiments.

Instructions for the use of The Ubbelohde Viscometer

See also ASTM D 445 and D 2515



Ubbelohde Type
for Transparent Liquids

1. Clean the viscometer using suitable solvents and dry by passing clean, dry filtered air through the instrument to remove the final traces of solvents. Periodically, traces of organic deposits should be removed with chromic acid.

2. If there is a possibility of lint, dust, or other solid material in the liquid sample, filter the sample through a fritted glass filter or fine mesh screen.

3. Charge the viscometer by introducing sample through tube G into the lower reservoir; introduce enough sample to bring the level between lines J and K.

4. Place the viscometer into the holder, and insert it into the constant temperature bath. Vertically align the viscometer in the bath if a self aligning holder has not been used.

5. Allow approximately 20 minutes for the sample to come to bath temperature.

6. Place a finger over tube B and apply suction to tube A until the liquid reaches the center of bulb C. Remove suction from tube A. Remove finger from tube B, and immediately place it over tube A until the sample drops away from the lower end of the capillary into bulb I. Then remove finger and measure the efflux time.

7. To measure the efflux time, allow the liquid sample to flow freely down past mark D, measuring the time for the meniscus to pass from mark D to mark F to the nearest 0.1 second.

8. Calculate the viscosity of the sample by multiplying the efflux time by the viscometer constant.

9. Without recharging the viscometer, make check determinations by repeating steps 6 to 8.

RECOMMENDED VISCOSITY RANGES FOR THE UBBELOHDE VISCOMETERS

Size	*Approximate Constant Centistokes/Second*	*Viscosity Centistokes*
O	0.001	0.3 to 1
OC	0.003	0.6 to 3
OB	0.005	1 to 5
1	0.01	2 to 10
1C	0.03	6 to 30
1B	0.05	10 to 50
2	0.1	20 to 100
2C	0.3	60 to 300
2B	0.5	100 to 500
3	1.0	200 to 1000
3C	3.0	600 to 3000
3B	5.0	1000 to 5000
4	10	2000 to 10000
4C	30	6000 to 30000
4B	50	10000 to 50000
5	100	20000 to 100000

For information for other sizes and viscometers, write to us.

CANNON INSTRUMENT CO. P. O. BOX 16 STATE COLLEGE, PA. 16801

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Fig. 16. Instructions for the use of the Ubbelohde viscometer

The temperature bath used in the experiments was a Magni Whirl made by the Blue M Electric Company, Blue Island, Illinois.

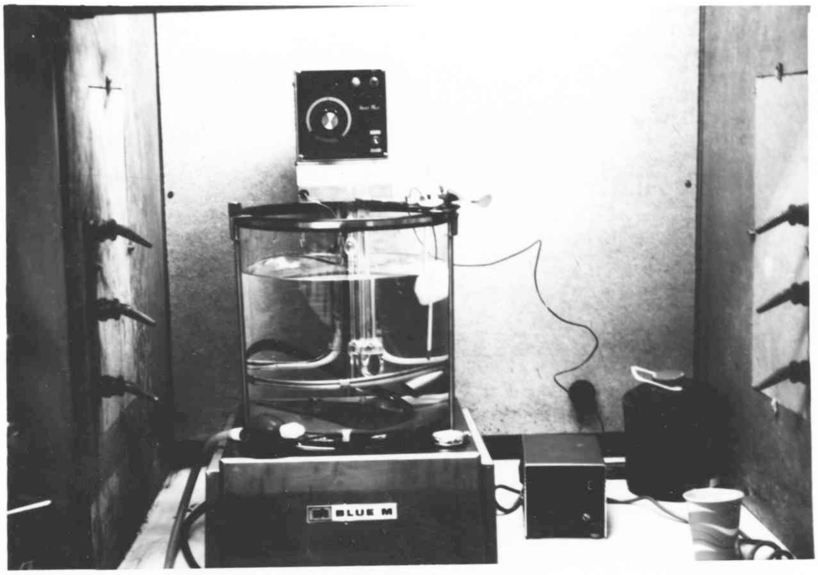


Fig. 17. Bath setup for viscosity measurements

Because freon 11 starts to boil out of solution, accurate measurements could not be obtained above 90°F for the B side. The results are shown in Tables 7 and 8 and the graphs in Figures 18 and 19.

Component Storage and Handling

The storage tanks that were used for the components were the same containers they were shipped in. The masterbatch,

Table 7

Viscosity Data for TDI (Component A)

Temperature °F	Viscosometer #	Constant	1st Run Sec.	2nd Run Sec.	Average Sec.	Viscosity Centistokes
53.5	3	0.947	559.15	543.00	551.08	521.87
60.0	3	0.947	405.17	398.93	402.05	380.74
65.0	3	0.947	326.13	324.30	325.22	307.98
68.0	3	0.947	283.08	283.30	283.19	268.18
72.0	3	0.947	240.82	238.20	239.51	226.81
77.0	3	0.947	195.80	195.78	195.79	185.41
85.0	2B	0.5147	247.20	254.55	250.88	129.13
92.0	2B	0.5147	202.90	202.60	202.75	104.36
100.0	2B	0.5147	155.70	155.60	155.65	80.11
110.0	2C	0.2814	214.07	214.35	214.21	60.28
120.0	2C	0.2814	161.80	162.20	162.00	45.59
130.0	2C	0.2814	130.02	130.02	130.41	36.70

Table 8

Viscosity Data for Masterbatch (Component B)

Temperature °F	Viscosometer #	Constant	1st Run Sec.	2nd Run Sec.	Average Sec.	Viscosity Centistokes
50	3	0.947	1730.50	1680.00	1705.25	1614.87
60	3	0.947	1047.60	990.00	1018.80	964.80
68	3	0.947	728.00	700.60	714.30	676.44
72	3	0.947	609.10	592.05	600.58	568.74
77	3	0.947	508.06	502.05	505.06	478.29
82	3	0.947	442.05	442.23	442.23	418.79
90	3	0.947	337.70	335.40	335.55	317.77

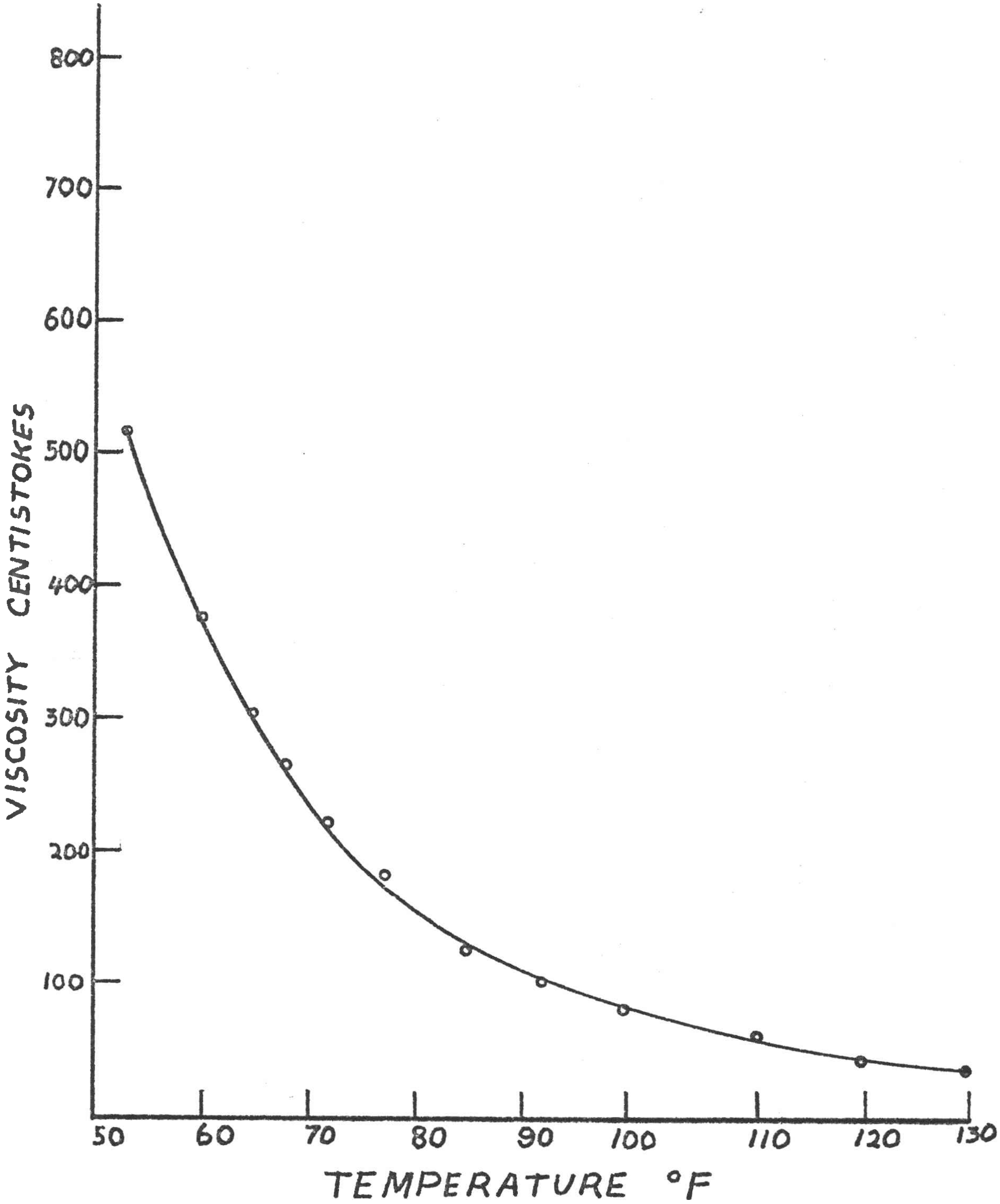


Fig. 18. Viscosity versus density cruve - TDI

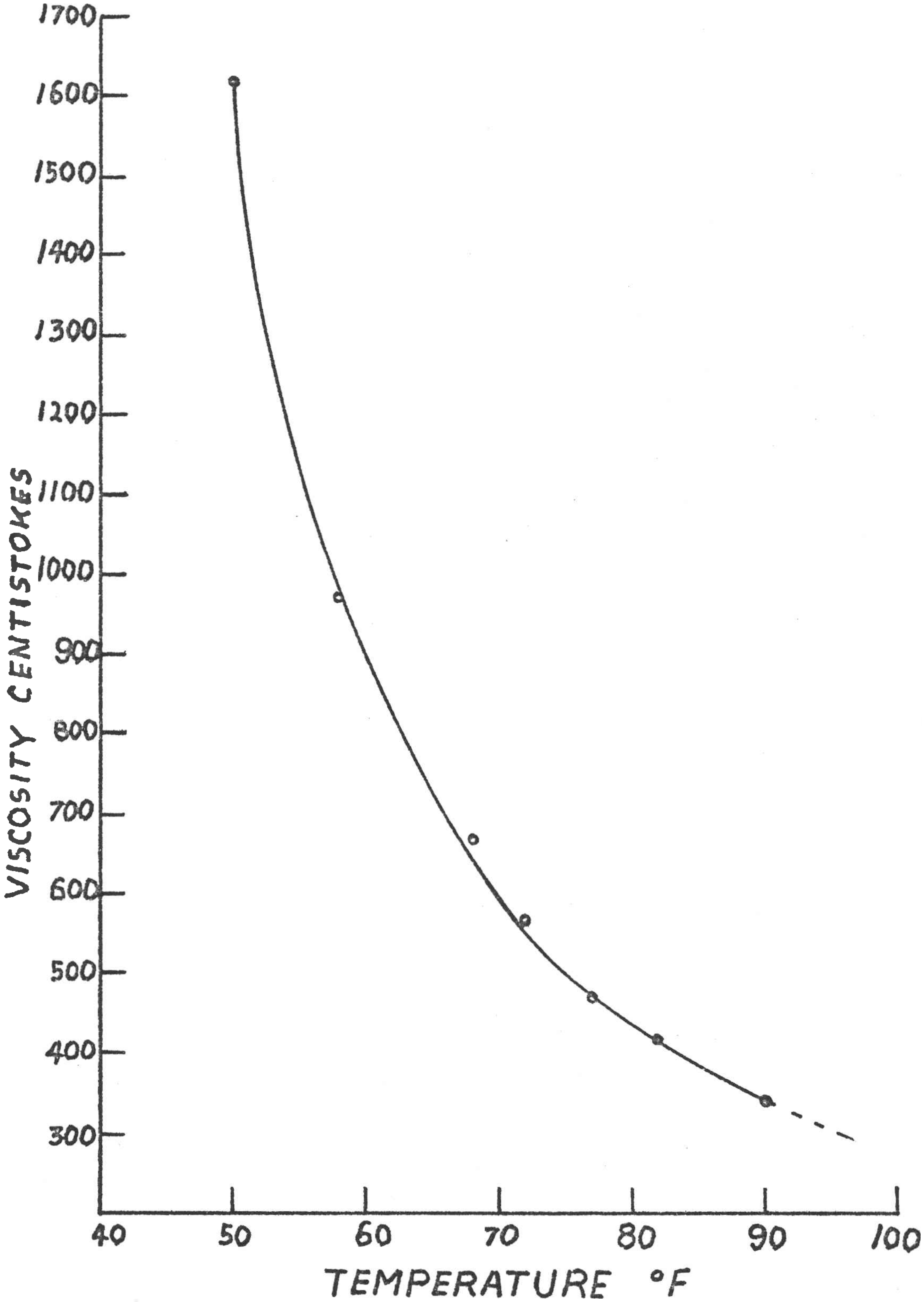


Fig. 19. Viscosity versus density curve - masterbatch

(component B) since it contained freon 11, was shipped in a special pressure containing 55 gallon drum. The isocyanate was shipped in a 55 gallon drum that was coated to prevent reaction with the chemical.

The masterbatch was stored in a walk-in refrigerator and kept near 56°F. To keep the components mixed, a device was constructed to rotate the drum. Two V-belts were placed around the drum and connected to two inch pullies mounted on the shaft of a 3/4 horsepower three phase motor. The motor was geared down to obtain lower mixing speeds.

Fig. 20. Drum rotator for masterbatch

A paddle was contructed out of mild steel and inserted into the large bung hole so that every time the drum made one revolution the paddle would pull through the mixture to keep it mixed. (See Figure 21.)

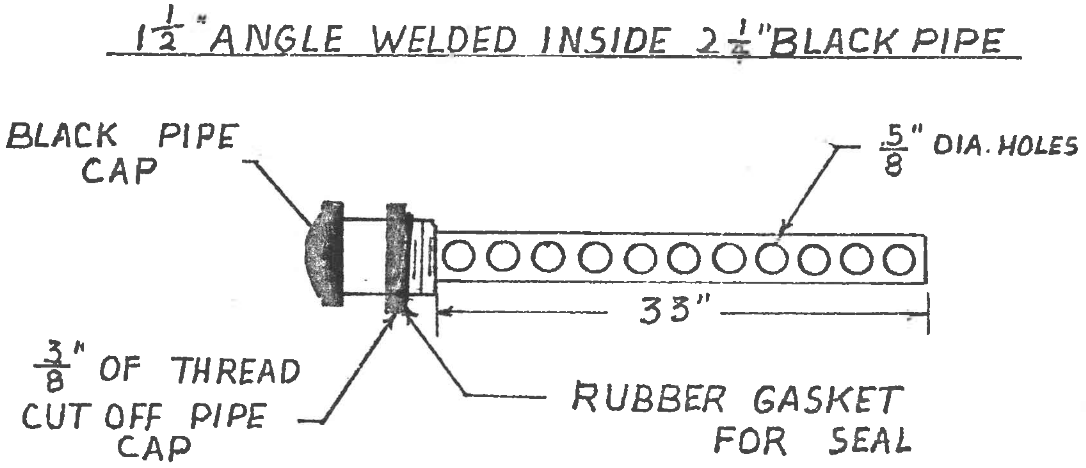


Fig. 21. Design of paddle for masterbatch drum

The isocyanate was kept at room temperature. Mixing was accomplished before the tests by rolling the drum on the floor and by shaking it back and forth within the drum. It was felt that the isocyanate remained fairly stable and did not need elaborate mixing equipment.

To drain the components from the tanks, valves were inserted into the small bung holes. The masterbatch required a special pressure valve to contain the pressure from the freon 11.

Transfer of the ingredients from the drums to the experiment area also required consideration. Special collapsible containers were obtained for the isocyanate. The containers were filled and a valve connected to them. As the component was drained, the containers collapsed. This precaution was taken to insure that the isocyanate did not react with the air before it was used for the experiments.

The masterbatch was transferred to the test area in a pressure vessel. This was to insure that there was no appreciable freon loss between the test runs.

Temperature control of the ingredients was accomplished by placing the components into a water bath one hour before the tests were run. The baths were made by placing a two-gallon container inside a five gallon container and foaming polyurethane between them. A lid was made by using 2-1/2 inch foam cut to the diameter of the large container.

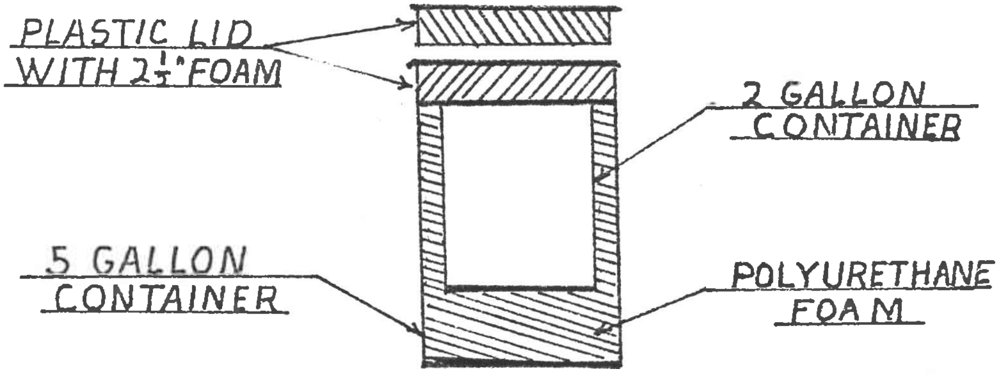


Fig. 22. Water bath design

The collapsible bags containing the isocyanate were placed inside the water bath. For the masterbatch seven holes 2-1/4 inches in diameter were cut into a 1/4 inch plywood circle. This provided the rack to place the samples in. Seven 100 ml tripour disposable beakers were placed into the bath as pictured below.

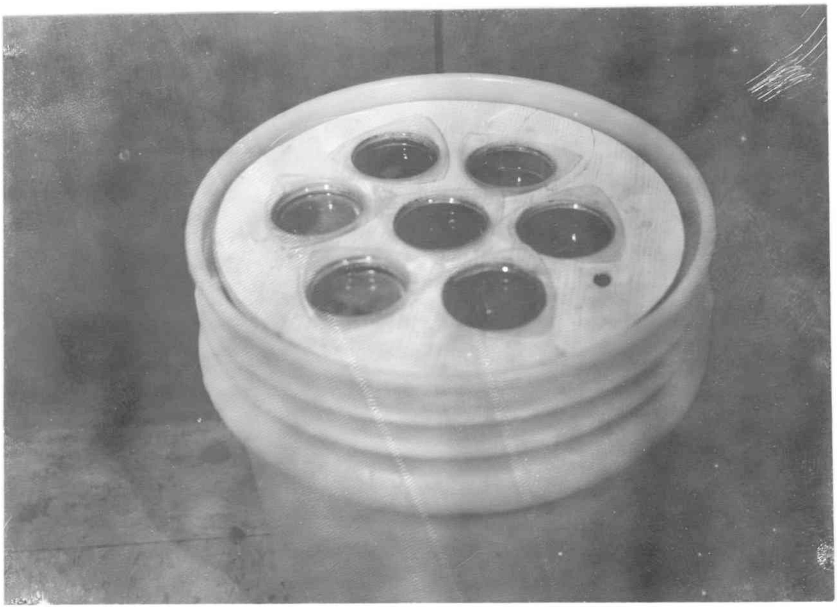


Fig. 23. Bath setup for masterbatch

A test was made on the stability of the freon in the bath. Seven samples were weighed out, each containing near 125 grams. These were left in the bath for seventy-two hours. The temperature was initially at 53°F and after the period of time had risen to 68°F. The samples were taken out and weighed again. The results showed no appreciable loss in freon content. The baths were kept at a constant temperature by using ice as a cooling medium and warm water as a heating.

Mixing and Metering

The proportioning or metering part of the rigid foam process was simulated using laboratory techniques. The masterbatch was weighed into a 32 ounce Dixie cup. The isocyanate was metered into the mixture using a syringe that had been calibrated so that the correct amount was placed in each time. On most tests the amount of masterbatch used was 105 grams and the amount of isocyanate was 75 grams.

Mixing was accomplished by using a 1/10 horsepower Dayton variable speed motor. (Model 2M037 - Maximum spped - 8000 RPM) The motor was connected to a Van Walters and Rogers two-inch diameter three blade propellor. The shaft diameter was 1/4 inch. The stirrer was calibrated to determine the RPM using a Strobotac. The mixing was observed using different speeds.

Simulating the Mold Cavity

To insure that the data obtained through experimentation was simulating actual production conditions, careful consideration was given to the design of the test cell. (1) It should be designed to control temperature and maintain it evenly from 30°C to 60°C. (2)

The surface to volume ratio should be similar to refrigerator fill. (3) The containing of exotherm heat should be similar to the production fixtures.

Temperature Control of Test Cell. Several different methods of keeping the walls at a constant even temperature were explored before the foam was poured into the cavity. The walls of the test cell were made of 20 gauge steel similar to refrigerator walls.

The first method reviewed was to wrap heating tape around the column, but it was felt that hot spots would develop and the cylinder would be unevenly heated.

The next method explored was blowing hot air through the column to obtain even and constant temperature. A Regal Commercial Electric Blower heat gun that had a variable temperature control was obtained and heat was blown through the column. Thermocouples were placed at three different places on the wall of the test cell and temperature readings were taken using Digital Pyrometers (Newport Model 267).

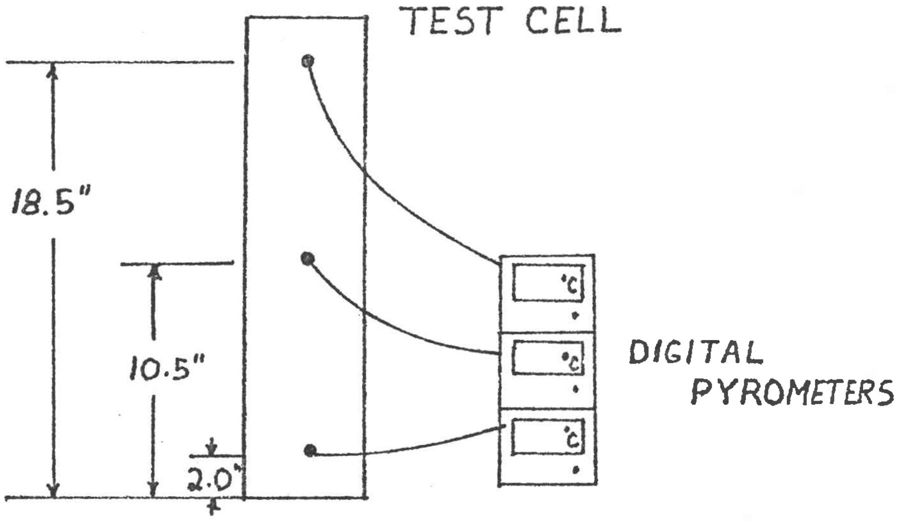


Fig. 24. Position of thermocouples on test cell wall

There was a variation in temperature of 8° to 10°C from the top to the bottom. Fiberous glass insulation was wrapped around the test cell and the test run again, but no better results were obtained.

Next a fire brick oven was built. The heating gun was placed behind a baffel made of asbestos.

It was felt that the heat would flow through the holes in the baffel and heat the tube evenly.

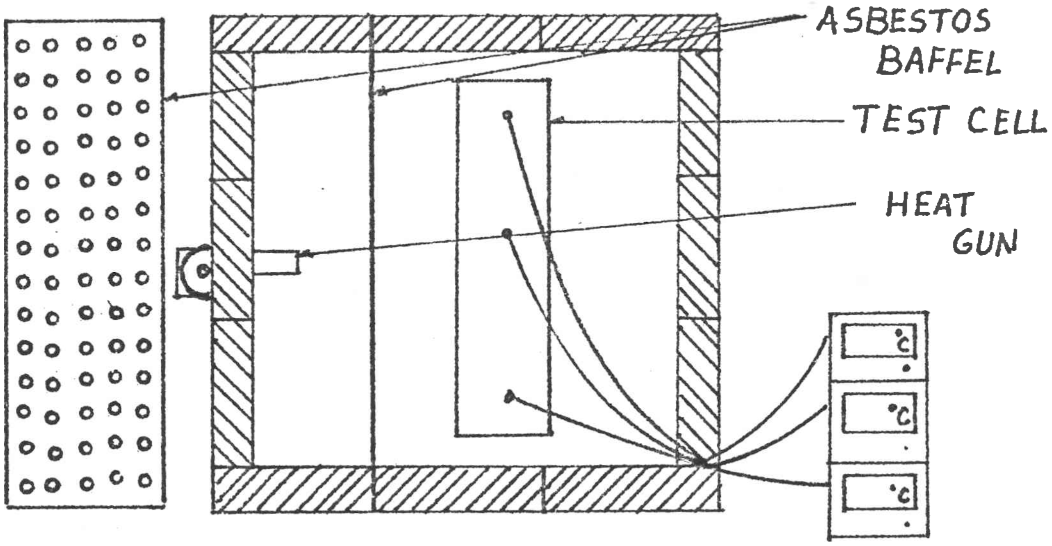


Fig. 25. Fire brick oven setup for temperature testing

The top of the cell in these tests was still between 4^{o} and $6^{o}C$ hotter than the bottom. This method was not used to heat the column, however, it was felt that better results could have been obtained by using a second fan placed on top of the oven to keep the air circulating around the column. A variation of this oven was used in making ambient air tests of the foam.

The method used to heat the column evenly was by using graphite impregnated paper (Temsheet by Armstrong Cork). This resistance paper was said to have a variation in temperature of within 1 percent. The 20 guage test tube was sprayed with crystal clear spray coating to insulate it from the resistance paper. The Temsheet was cut to size and wrapped around the tube.

Conducting silver paint and a three-eighths wide strip of aluminum foil were used to connect the ends of the resistance paper together.

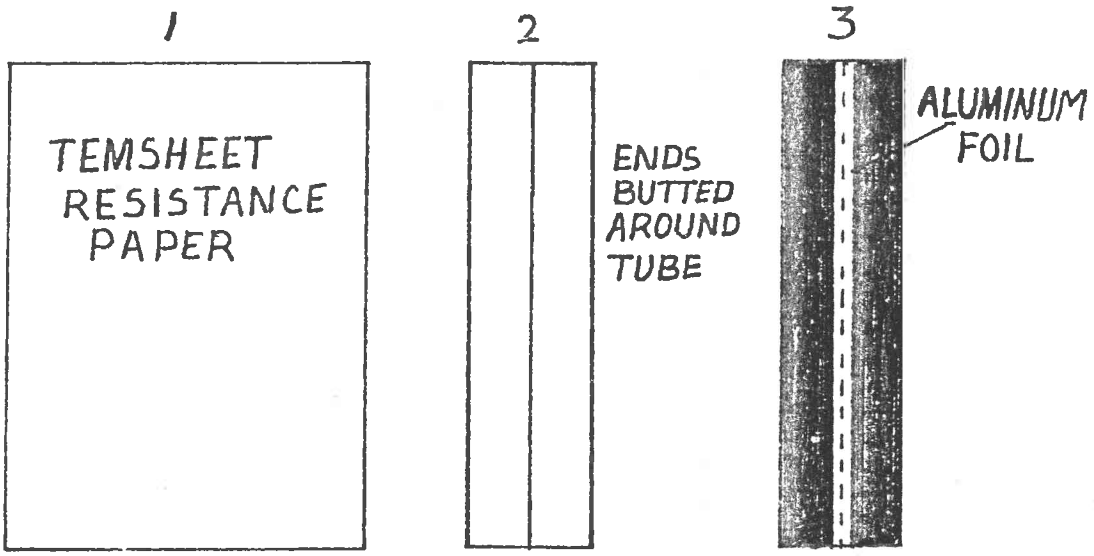


Fig. 26. Connecting the ends of the graphite impregnated paper together

A strip of aluminum foil was also connected on the opposite side of the tube to the resistance paper by using silver conducting paint. The two leads from an adjustable rehostat were connected to the two strips of aluminum foil. The final effect was like having many resistors placed in parallel. The resistance was found to be 42.1 ohms.

Several tests were run on the test cell using the thermocouple setup shown in Figure 24. The temperature was adjusted by changing the voltage on the rehostat. The test cell remained constant to within ± 1°C from top to bottom in the range of 20°C to 60°C.

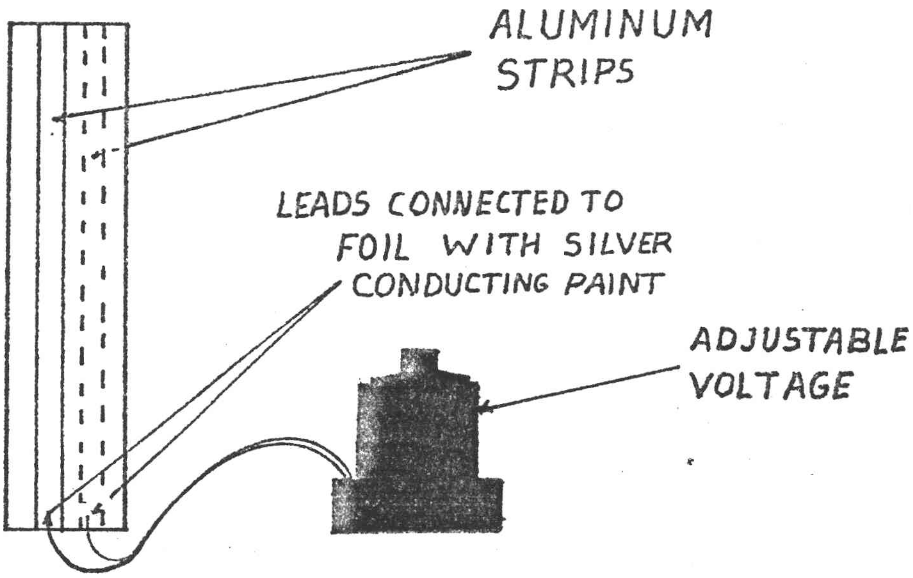


Fig. 27. Test cell setup--temperature control

Test Cell. After temperature control was established the test cell was completed by making a mixing container, simulating the fixture, and finding a means for removing the samples.

The mixing container was a 32 oz. Dixie cup. A 20 guage metal container was devised to fit around the cup. The same procedure was used for heating the cup as for heating the column.

To maintain similarities between the actual process and the test cell a one-quarter inch thick five inch diameter cardboard tube was split on one side and slipped over the test cell. One-fourth inch cardboard was also wrapped around the cup container. This was done to simulate the fixture that holds the refrigerators in industry.

The test cell was lined with aluminum foil so the foamed sample could be removed from the cell after the test. Paper and polyethylene film was experimented with, but it was felt that the aluminum foil would resemble more closely the control of exotherm heat. Paper and plastic would act as insulators to contain exotherm heat unlike the fill of refrigerators in industry.

The outer rim of the 32 oz. cup provided the seal of the test cell.

Fig. 28. Completed test cell

Test Procedure and Monitoring Results

In Chapter III the sensitivity of the different variables associated with the rigid polyurethane foam process were reviewed. In the experiments and in simulating the process some of the variables were already fixed.

The fixtures at Whirlpool are already constructed so the effects of changing the wall thickness were not accounted for in making the test runs. The test cell wall construction was given in the previous section.

The size of the pour was fixed during the test runs similar to refrigerator fill in industry. The humidity and barometric pressure were monitored during test runs. No attempt was made to determine their effects on the process since it was felt that their effects were neglible compared to the other variables. The data collected during the test was included for reference or for further experiments associated with these variables.

With the lack of the proper mixing equipment that is used in industry it was felt that the mixing variables could not be determined for their process. Therefore, no tests were run on the effect of mixing speed except for preliminary tests that were run to insure that proper mixing was being accomplished. The results of improper mixing are shown in Figure 29.

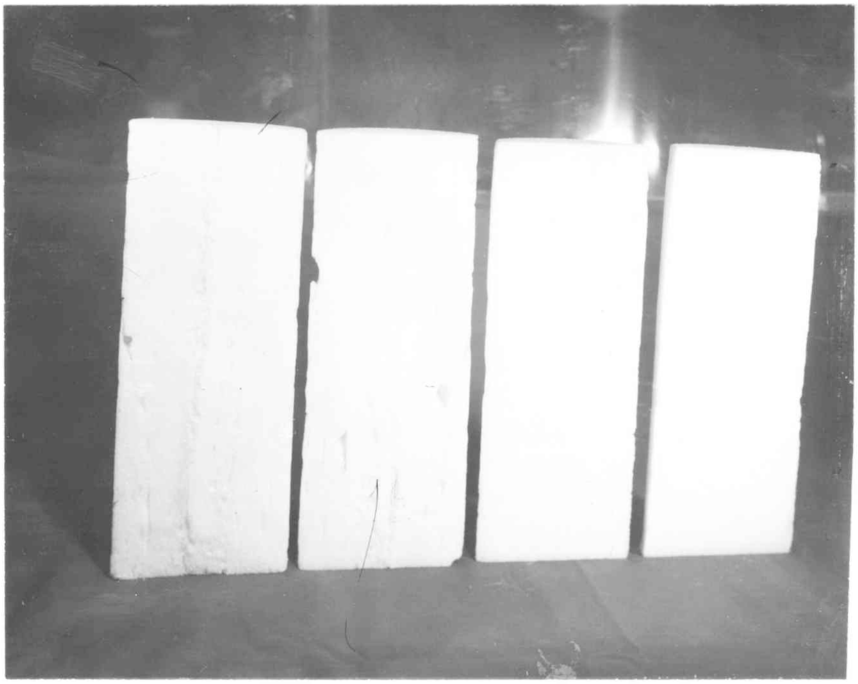


Fig. 29. Test samples on left show results of improper mixing.

Poor mixing not only causes long finger-like voids in the bottom of the rise, but also causes larger cell sizes in the foam and a poorer quality soft foam at the top of the rise.

The mixing speed was set at 2500 RPM for the tests. An industrial timer was used to set the length of time of the mix. The timer was set on six seconds for each test run unless otherwise specified. It was noted that even the position of the cup and moving the cup under the stirrer affected the mixing.

The temperature of the ingredients affects both the metering and mixing operation. If the temperature of the ingredients is varied the viscosity varies. As the viscosity changes, mixing is affected in two ways: (1) the shear required for the mixing changes, and (2) the blendability is not the same. The proportioning equipment varies because there is usually a pressure change. The cream time changes when temperature varies.

The temperature of the cavity and the materials of construction affects the exotherm heat generated by the reaction.

An experiment was devised to determine the relationship between the ingredient temperature and cavity wall temperature. During the tests it was assumed that the fixture walls were already constructed and that the k factor for the fixtures remained constant for the entire range of temperatures used. In the ideal case the k factor would increase with the increase of temperature, but since the range used was small it was assumed that the change was neglible.

The temperature of the ingredients was measured using mercury thermometers. The temperature of the cell walls was monitored using the digital pyrometers and setup explained in the previous section. The temperature of the ingredients was changed by changing the temperature of the A component. The B component was kept at 70°F plus or minus 2°F. The B component was kept at this temperature because at higher temperatures the freon 11 starts to boil off; at lower temperatures the viscosity is too high for the best mixing.

Table 9

Experimental Points Used in Tests

Variables		Range Determined From Literature	Test Points Used
Temperature of Ingredients	A	60 to 90°F	60, 70, 80 and 90°F
	B	60 to 77°F	70°F
Temperature of Cavity Walls		110 to 150°F	68, 86, 104 and 113°F 122, 131 and 140°F
Mixing Speed		1500 to 4000 RPM	2500 RPM

These tests required 4 X 7 = 28 tests. Fifteen of these tests were run over to insure that results were repeatable. The total tests used to determine the relationship between the temperature of ingredients and cavity walls were 28 + 15 = 43 tests.

While the tests were being made on temperatures the following results were monitored. The cream time was determined by noting when the foam first begins to rise. The rise rate was determined using a specially constructed measuring device. A glass tube was connected to a meter stick with epoxy, and suspended above the test cell. A float was made from a 1/4 inch thick, 2-1/2 inch diameter disk of polyurethane foam. The float was connected to a wire that ran up the glass tube. As the foam flowed up the tube the height reached was determined by noting the position of a bulb placed on the other end to the wire. (See Figures 30 and 31)

The tack free time was determined by touching a wooden spatula to the foam after the rise was completed.

The times were recorded using a Hewlett Packard model 55 calculator that had ten memories for storage. An eleventh time could be recorded by using the time stop for the final reading.

The eleven times recorded were: (1) the cream time, (2) time to reach 10 cm on the flow stick, (3) time to reach 15 cm, (4) time to reach 20 cm, (5) time to reach 30 cm, (6) time to reach 40 cm, (7) time to reach 50 cm, (8) time to reach 55 cm, (9) time to reach 60 cm, (10) final height time and (11) tack free time.

Figure 32 shows a sample data sheet used to record the data, and Figures 33 and 34 show the equipment setup for the tests.

To obtain more data points on the rise rate versus time curve five tests were run using a Bolex 16 mm movie camera and an electronic timer. These tests were run mainly for information purposes and to insure that the rise rate versus time curves were approximated by the eight data points used in the experiments.

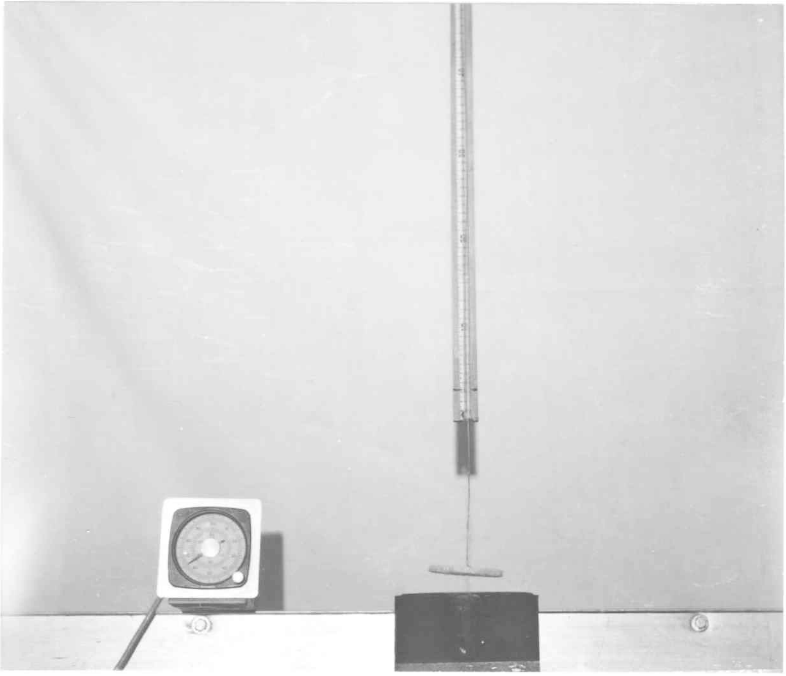


Fig. 30. Foam flow apparatus set up

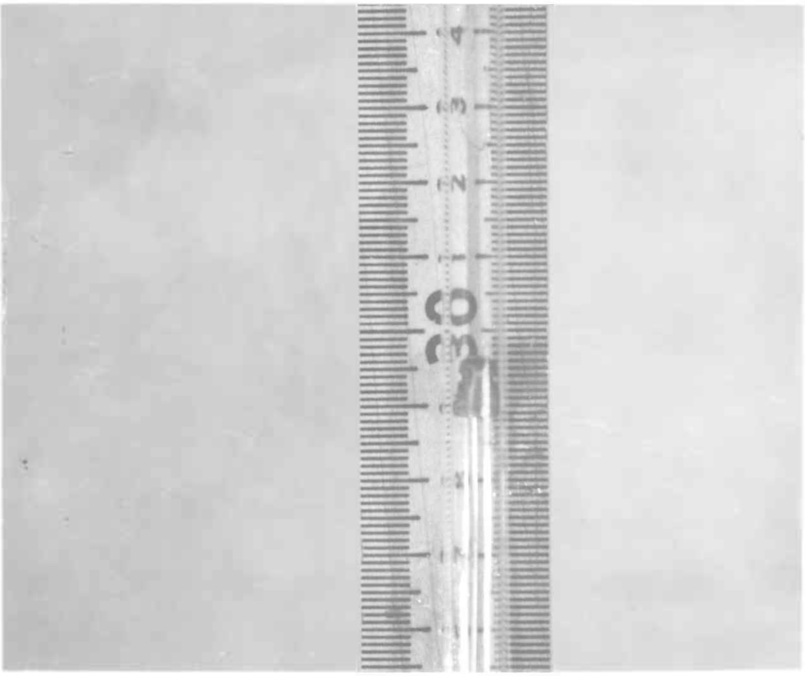
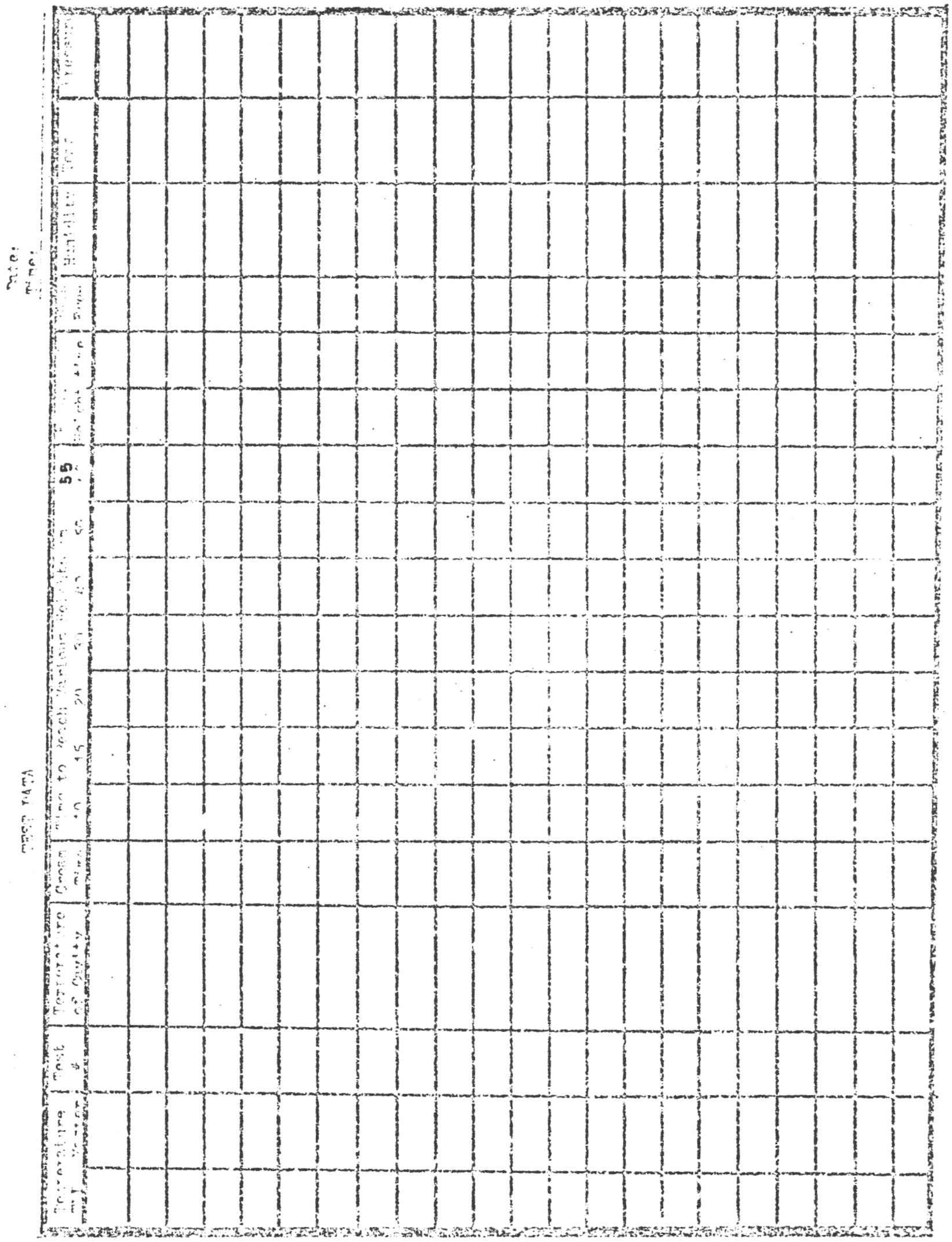


Fig. 31. Obtaining readings from the flow tube



TEST DATA

Date:
Time:

Temperature		Test	Temperature	Cream	Time to Reach Various Heights in							[illegible]		[illegible]	Humidity	Temp	Pressure
Oil	Water	#	of Cavity	Time	10	15	20	30	40	50	55	Height	Time	Room			

Fig. 32. Sample data sheet

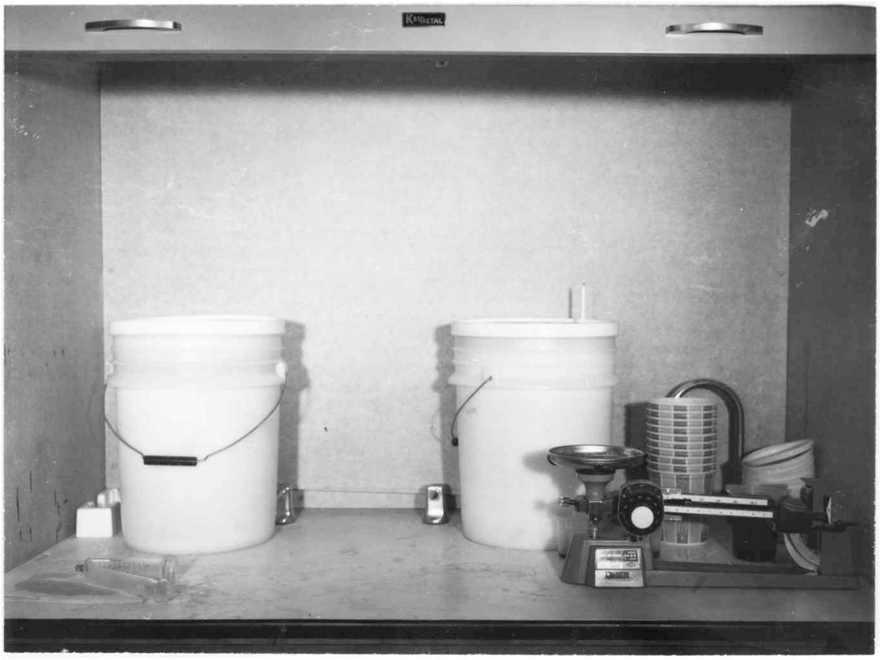


Fig. 33. Temperature baths and proportioning operation

Fig. 34. Mixing operation, test cell setup and monitoring results

To determine the final density of the foamed sample, the sample was divided as shown in Figure 35.

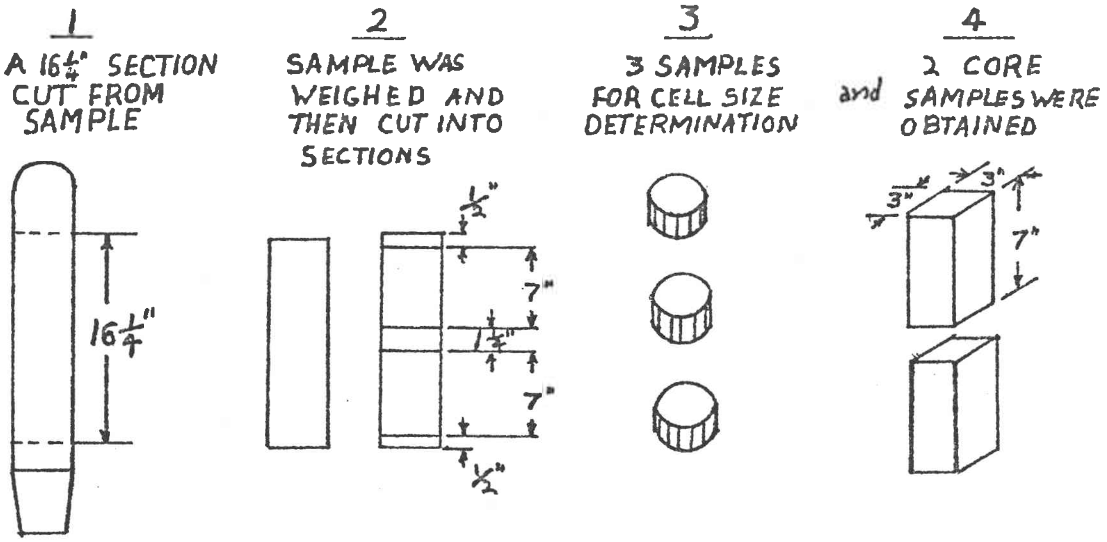


Fig. 35. Sectioning the test sample to obtain density and cell size samples

Figure 36 shows a sample data sheet used to determine the density at various places in the sample.

Test #	Weight of Samples in Grams			Appearance
	16-1/4" Section	Bottom Core	Top Core	

Fig. 36. Sample data sheet used for determining density

CHAPTER V

DATA

The purpose of this chapter was to present the data gathered from the experiments outlined in Chapter IV.

Tables 10 to 13 show the results of the experiments using the heated test cell and the controlled ingredient temperatures.

The actual height of the rising foam was determined by subtracting 5 cm from the height reading on the meter stick. This was done because the initial height of the fluid in the cup was 1.8 cm, and the initial reading on the meter stick was 3.2 cm when there was no fluid in the cup.

The tack free time in some cases was not obtained. This was because the tack free time had been reached before the test was taken after the final rise was reached.

The cream time in theory was to be constant for a set of ingredient temperatures. An average of the first three tests was used to determine the cream time for the foam at certain ingredient temperatures for the first two test runs. Since the variance in some instances was large the cream time was recorded for all tests in the next two test runs.

Table 10

Test Data With Ingredient Temperatures
of 60°F for A and 70°F for B

Test #	1	2	3	4	5	6	7
Temperature of wall	20°C	30°C	40°C	45°C	50°C	55°C	60°C
Cream Time (sec)	8.06	8.06	8.06	8.06	8.06	8.06	8.06
Time to reach 5 cm	15.33	-	25	-	-	-	13.00
" " " 10 cm	19.84	24.59	29	26	-	-	17.39
" " " 15 cm	24.17	-	36	31	-	28.68	23.11
" " " 25 cm	34.93	40.02	45	40	47	38.23	33.47
" " " 35 cm	48.29	53.98	57	52	59	50.79	45.83
" " " 45 cm	68.52	71.87	74	68	76	64.24	58.34
" " " 50 cm	83.20	82.67	84	78	93	74.80	68.05
" " " 55 cm	98.13	100.72	-	-	114	84.93	82.27
Final Height	56.5	57.0	57.5	58.8	60.5	61	63
Final Height Time	111.05	115.34	125.00	120	139	111.20	109.90
Tack Free Time	140.00	142.84	145	140	-	125	114
Humidity %	44	44	42	42	42	42	40
Temperature °F	68	68	74	74	74	69	69
Pressure lbs/ft^3	1800	1800	1800	1800	1800	1794	1794

Table 11

Test Data With Ingredient Temperatures of 70°F for A and 70°F for B

Test #	8	9	10	11	12	13	14
Temperature of wall	20°C	30°C	40°C	45°C	50°C	55°C	60°C
Cream Time	6.88	6.88	6.88	6.88	6.88	6.88	6.88
Time to Reach 5 cm	-	-	12.09	14.25	-	-	15.12
" " " 10 cm	-	15.77	16.50	18.96	-	-	19.48
" " " 15 cm	22.94	18.65	20.62	24.60	-	21.90	25.54
" " " 25 cm	35.57	27.87	30.15	32.29	34.93	31.95	33.02
" " " 35 cm	45.41	38.66	41.49	41.16	41.83	42.60	44.26
" " " 45 cm	62.74	54.26	55.61	54.66	52.92	56.76	58.86
" " " 50 cm	-	65.16	64.77	64.08	61.54	63.93	61.09
" " " 55 cm	-	78.14	78.19	76.65	71.54	73.93	84.59
Final Height	53.0	58.5	58.5	59.6	61.5	60.5	57.5
Final Height Time	112.72	92..86	105.48	101.84	105.68	104.83	103.50
Tack Free Time	-	140	116	115	130	-	-
Humidity %	54	54	52	52	52	52	52
Temperature °F	72	72	72	72	72	72	72
Pressure lbs/ft^3	1800	1800	1800	1800	1800	1800	1800

Table 12

Test Data With Ingredient Temperatures of 80°F for A and 70°F for B

Test #	15	16	17	18	19	20	21
Temperature of wall	20°C	30°C	40°C	45°C	50°C	55°C	60°C
Cream Time	7.93	6.92	4.26	5.63	5.63	5.53	3.52
Time to Reach 5 cm	-	-	13.03	-	-	-	-
" " " 10 cm	-	13.89	17.68	-	-	12.84	12.51
" " " 15 cm	22.90	16.74	21.55	-	-	16.24	17.32
" " " 25 cm	29.35	29.51	30.95	-	-	24.69	23.68
" " " 35 cm	-	40.73	41.96	35.44	-	33.64	33.22
" " " 45 cm	56.72	55.43	56.71	49.63	-	45.99	45.51
" " " 50 cm	-	65.75	67.16	60.42	-	54.43	53.15
" " " 55 cm	-	80.21	80.40	71.85	-	62.45	63.08
Final Height	56.0	58.7	59.0	59.0	60.2	60.8	59.8
Final Height Time	122	103.87	120.73	101.73	-	84.50	81.61
Tack Free Time	-	132	135	108	-	96	107
Humidity %	46	46	46	46	46	40	40
Temperature °F	68	68	68	68	68	70	70
Pressure lbs/ft^3	1800	1800	1800	1800	1800	1794	1794

Table 13

Test Data With Ingredient Temperatures of 90°F for A and 70°F for B

Test #	22	23	24	25	26	27	28
Temperature of wall	20°C	30°C	40°C	45°C	50°C	55°C	60°C
Cream Time	4.69	5.53	4.85	4.30	4.75	4.00	4.69
Time to Reach 5 cm	-	-	10.01	-	-	-	-
" " " 10 cm	-	12.60	12.92	12.83	12.44	9.92	-
" " " 15 cm	13.23	15.00	16.84	16.57	15.15	14.20	12.52
" " " 25 cm	22.43	22.90	25.15	23.57	22.95	20.69	19.68
" " " 35 cm	31.91	33.92	35.38	32.92	32.25	29.43	28.39
" " " 45 cm	46.12	48.46	47.87	45.10	44.25	40.71	38.95
" " " 50 cm	54.67	58.77	57.57	52.28	52.46	47.84	45.62
" " " 55 cm	73.13	77.36	72.68	62.39	64.27	58.33	54.08
Final Height	56.3	56.7	57.3	59.5	59.5	62	63
Final Height Time	92.59	99.98	101.86	100.76	95.65	90.31	85.08
Tack Free TIme	-	-	-	-	-	-	-
Humidity %	50	48	47	46	45	45	45
Temperature °F	72	74	75	78	77	78	78
Pressure lbs/ft^3	1800	1800	1800	1800	1800	1800	1800

Tables 14 to 17 contain the data collected to determine the density of the foamed samples. The overall density was calculated by cutting the top and bottom portion of the sample as shown previously in Figure 35. The volume of this sample was calculated to be:

$$\frac{L\pi D^2}{4} = \frac{16.25''}{}\bigg|\frac{\pi}{4}\bigg|\frac{(5.0\,\text{in})^2}{}\bigg|\frac{\text{ft}^3}{1728\,\text{in}^3} = .1846\,\text{ft}^3$$

The sixteen 1/4 inch section was weighed and then the number of grams was converted to pounds by

$$\text{Pounds} = \text{Grams}/453.5924$$

The overall density was calculated by dividing the pounds by the volume.

$$\text{Density} = \frac{\text{Pounds}}{\text{Volume}}$$

The sixteen 1/4 inch section was then cut into the samples as shown in Figure 35. The two core samples were 3 inches X 3 inches X 7 inches = 63 in^3 = .03645 ft^3 in volume. The density of the core samples was obtained in the same way as the density for the overall sample.

As the core samples were weighed the appearance of the foam was noted. On the bottom of most core samples small voids were apparent near the wall. The foam was mostly of good quality, but this description was noted to help and determine the foam of the best quality.

The data shows very little difference in density of the samples.

Table 14

Density of Samples for Tests 1 through 7

Test #	Weight of Sample	Density of Sample	Weight of Bottom Core	Density of Bottom Core	Weight of Top Core	Density of Top Core	APPEARANCE
1	121.7 grams	1.453 lbs/ft^3	21.10 grams	1.276 lbs/ft^3	20.45 grams	1.237 lbs/ft^3	.5 cm to 1 cm. voids in bottom near edge. Top looks OK
2	118.0 grams	1.409 lbs/ft^3	20.40 grams	1.234 lbs/ft^3	20.35 grams	1.231 lbs/ft^3	long .5cm. Dia. voids in bottom
3	120.5 grams	1.439 lbs/ft^3	21.90 grams	1.324 lbs/ft^3	21.40 grams	1.294 lbs/ft^3	Large void in top .6 cm. x 3 cm x 5cm. 1cm. void in bottom
4	120.6 grams	1.440 lbs/ft^3	21.30 grams	1.288 lbs/ft^3	21.00 grams	1.270 lbs/ft^3	Looks OK, Small pencil lead size holes in bottom
5	118.7 grams	1.417 lbs/ft^3	21.55 grams	1.303 lbs/ft^3	21.40 grams	1.294 lbs/ft^3	Same as above
6	117.6 grams	1.404 lbs/ft^3	21.50 grams	1.300 lbs/ft^3	21.20 grams	1.282 lbs/ft^3	One void in bottom .5 cm. Dia. and 2 cm. long
7	120.5 grams	1.439 lbs/ft^3	21.60 grams	1.306 lbs/ft^3	21.60 grams	1.306 lbs/ft^3	OK

Table 15

Density of Samples for Tests 8 through 14

Test #	Weight of Sample	Density of Sample	Weight of Bottom Core	Density of Bottom Core	Weight of Top Core	Density of Top Core	APPEARANCE
8	121.1 grams	1.446 lbs/ft^3	22.10 grams	1.336 lbs/ft^3	21.45 grams	1.297 lbs/ft^3	1 cm. Dia. x 2 cm void in bottom. .5 cm in middle
9	118.6 grams	1.416 lbs/ft^3	20.75 grams	1.255 lbs/ft^3	20.60 grams	1.246 lbs/ft^3	small .5 xl.5 bottom void near wall
10	120.1 grams	1.434 lbs/ft^3	21.25 grams	1.285 lbs/ft^3	21.20 grams	1.282 lbs/ft^3	Small voids in bottom Top OK
11	120.0 grams	1.433 lbs/ft^3	21.55 grams	1.303 lbs/ft^3	21.25 grams	1.285 lbs/ft^3	Looks OK except for small .5 x 1.5 cm void in bottom
12	118.3 grams	1.412 lbs/ft^3	20.80 grams	1.258 lbs/ft^3	20.80 grams	1.258 lbs/ft^3	Voids 15x 2cm near bottom outside edge- structure OK
13	116.2 grams	1.387 lbs/ft^3	21.15 grams	1.279 lbs/ft^3	21.00 grams	1.270 lbs/ft^3	.5 cm spherical void in bottom Foam OK
14	119.1 grams	1.422 lbs/ft^3	21.33 grams	1.290 lbs/ft^3	21.28 grams	1.287 lbs/ft^3	1cm. x 3 cm void near wall at bottom. Hole through center

Table 16

Density of Samples for Tests 15 Through 21

Test #	Weight of Sample	Density of Sample	Weight of Bottom Core	Density of Bottom Core	Weight of Top Core	Density of Top Core	APPEARANCE
15	121.1 grams	1.445 lbs/ft^3	21.60 grams	1.306 lbs/ft^3	20.85 grams	1.261 lbs/ft^3	Small .5 cm. voids in Bottom Appearance OK
16	117.8 grams	1.407 lbs/ft^3	20.60 grams	1.246 lbs/ft^3	20.33 grams	1.230 lbs/ft^3	Small Voids near surface bottom .5 x 1 cm.
17	120.5 grams	1.439 lbs/ft^3	21.18 grams	1.280 lbs/ft^3	20.68 grams	1.251 lbs/ft^3	Appearance OK .5 cm Dis x 1 cm Void in bottom
18	120.1 grams	1.434 lbs/ft^3	21.35 grams	1.291 lbs/ft^3	20.90 grams	1.264 lbs/ft^3	Edge Voids in Bottom
19	117.7 grams	1.405 lbs/ft^3	21.00 grams	1.270 lbs/ft^3	20.78 grams	1.257 lbs/ft^3	4 voids .5 cm dia x 1 cm joining together along bottom edge
20	115.0 grams	1.373 lbs/ft^3	20.75 grams	1.255 lbs/ft^3	20.52 grams	1.241 lbs/ft^3	OK small edge voids
21	118.0 grams	1.409 lbs/ft^3	21.22 grams	1.283 lbs/ft^3	21.22 grams	1.283 lbs/ft^3	Bad Split 3 cm. long through entire center of cores

Table 17

Density of Samples for Tests 22 Through 28

Test #	Weight of Sample	Density of Sample	Weight of Bottom Core	Density of Bottom Core	Weight of Top Core	Density of Top Core	APPEARANCE
22	128.5 grams	1.534 lbs/ft^3	21.35 grams	1.291 lbs/ft^3	21.25 grams	1.285 lbs/ft^3	.3 dia. x 1 cm void in bottom .5 x 1 cm voids in bottom wall
23	120.5 grams	1.439 lbs/ft^3	20.75 grams	1.255 lbs/ft^3	20.80 grams	1.258 lbs/ft^3	.5cm dia x 2 cm voids in bottom surface looks rough
24	121.0 grams	1.445 lbs/ft^3	21.45 grams	1.297 lbs/ft^3	21.35 grams	1.291 lbs/ft^3	Top OK .5 cm x 1cm voids near surface
25	119.5 grams	1.427 lbs/ft^3	21.50 grams	1.300 lbs/ft^3	21.25 grams	1.285 lbs/ft^3	Tiny .15cm x .75 cm voids towards surfaces all the way up
26	119.6 grams	1.428 lbs/ft^3	21.30 grams	1.288 lbs/ft^3	21.32 grams	1.289 lbs/ft^3	.3 cm holes through center 1.2cm spherical void in bottom
27	116.1 grams	1.386 lbs/ft^3	20.62 grams	1.247 lbs/ft^3	20.43 grams	1.235 lbs/ft^3	.4cm finger like voids running in bottom and along outer edges
28	112.6 grams	1.344 lbs/ft^3	20.10 grams	1.215 lbs/ft^3	19.75 grams	1.193 lbs/ft^3	.5cm dia void through entire center of sample. small voids near walls

To analyze where the excess material goes when overpacking, the data was looked at in three different ways. The excess material could increase the volume of the sample, increase the core density of the sample, or increase the thickness and density of the sample wall.

The sample was allowed to rise freely up the test cell. From the final height reached the total expanded volume could be calculated.

From the analysis of the difference between the overall density and the average core density the amount of material that makes up the skin was calculated.

The urethane also goes into increasing or decreasing the core density of the sample. As shown in the cell cluster in Figure 37, this material could either go into increasing the thickness of the cell walls or the size of the cell. The thickness of the individual polyurethane foam cell will be affected somewhat by the flow of the foam in the primary polymerization stage. Table 18 shows the calculations used in making these determinations.

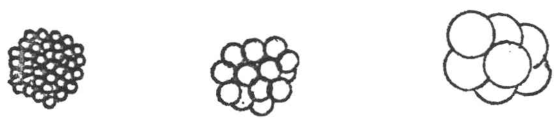


Fig. 37. Cell size affects amount of material

Table 18

Overpack Considerations

Test #	Difference Between Bottom & Top Core	Average Core Density	Difference Between Overall & Average Core Density	Final Height Reached	90% Cell Size (Inch)
1	0.039	1.2565	0.1785	56.5	.015
2	0.003	1.2325	0.1765	57.0	.015
3	0.030	1.3090	1.1300	57.5	.017
4	0.018	1.2790	0.1610	58.8	.015
5	0.009	1.2980	0.1190	60.5	.011
6	0.018	1.2910	0.1130	61.0	.013
7	0.000	1.3060	0.1330	63.0	.010
8	0.039	1.3165	0.1295	53.0	.010
9	0.009	1.2505	0.1655	58.5	.010
10	0.003	1.2835	0.1505	58.5	.011
11	0.018	1.2940	0.1390	59.6	.011
12	0.000	1.2580	0.1540	61.5	.012
13	0.009	1.2745	0.1125	60.5	.015
14	0.003	1.2885	0.1335	57.5	.013
15	0.045	1.2835	0.1615	56.0	.012
16	0.016	1.2830	0.1690	58.7	.011
17	0.029	1.2655	0.1735	59.0	.013
18	0.027	1.2775	0.1565	59.0	.012
19	0.013	1.2635	0.1415	60.2	.010
20	0.014	1.2480	0.1250	60.8	.014
21	0.000	1.2830	0.1260	59.8	.014
22	0.006	1.2880	0.2460	56.3	.013
23	-0.003	1.2565	0.1825	56.7	.012
24	0.006	1.2940	0.1510	57.3	.011
25	0.015	1.2925	0.1345	59.5	.013
26	-0.001	1.2885	0.1395	59.5	.012
27	0.012	1.2410	0.1450	62.0	.011
28	0.022	1.2040	0.1400	63.0	.014

To construct the graphs and to find the best fit for the data a regression analysis was run using a computer program. A sample printout was included in Appendix D.

The three variables used were: (1) density of the cell wall, (2) core density, and (3) final height reached.

A program was run for each range of ingredient temperatures used. (1) 60°F for A and 70°F for B. (2) 70°F for A and 70°F for B. (3) 80°F for A and 70°F for B. (4) 90°F for A and 70°F for B.

The following equations were looked at, where "Temp" equals the cell wall temperature and "var" equals the variable looked at.

(1) $\text{Var} = \text{Temp}$

(2) $\text{Var} = \text{Temp} + \text{Temp}^2$

(3) $\text{Var} = \text{Temp} + \text{Temp}^2 + \text{Temp}^3$

(4) $\text{Var} = \text{Temp} + \text{Temp}^3$

The best fit was determined by finding the equation with the lowest mean and the highest "R". (Percentage of data points in range) The "t" table information presented in the printout was used to determine if the results were significant. However, because of the sample size this information was not as important in the consideration as the other factors.

The results were plotted and included in Figures 38 to 49.

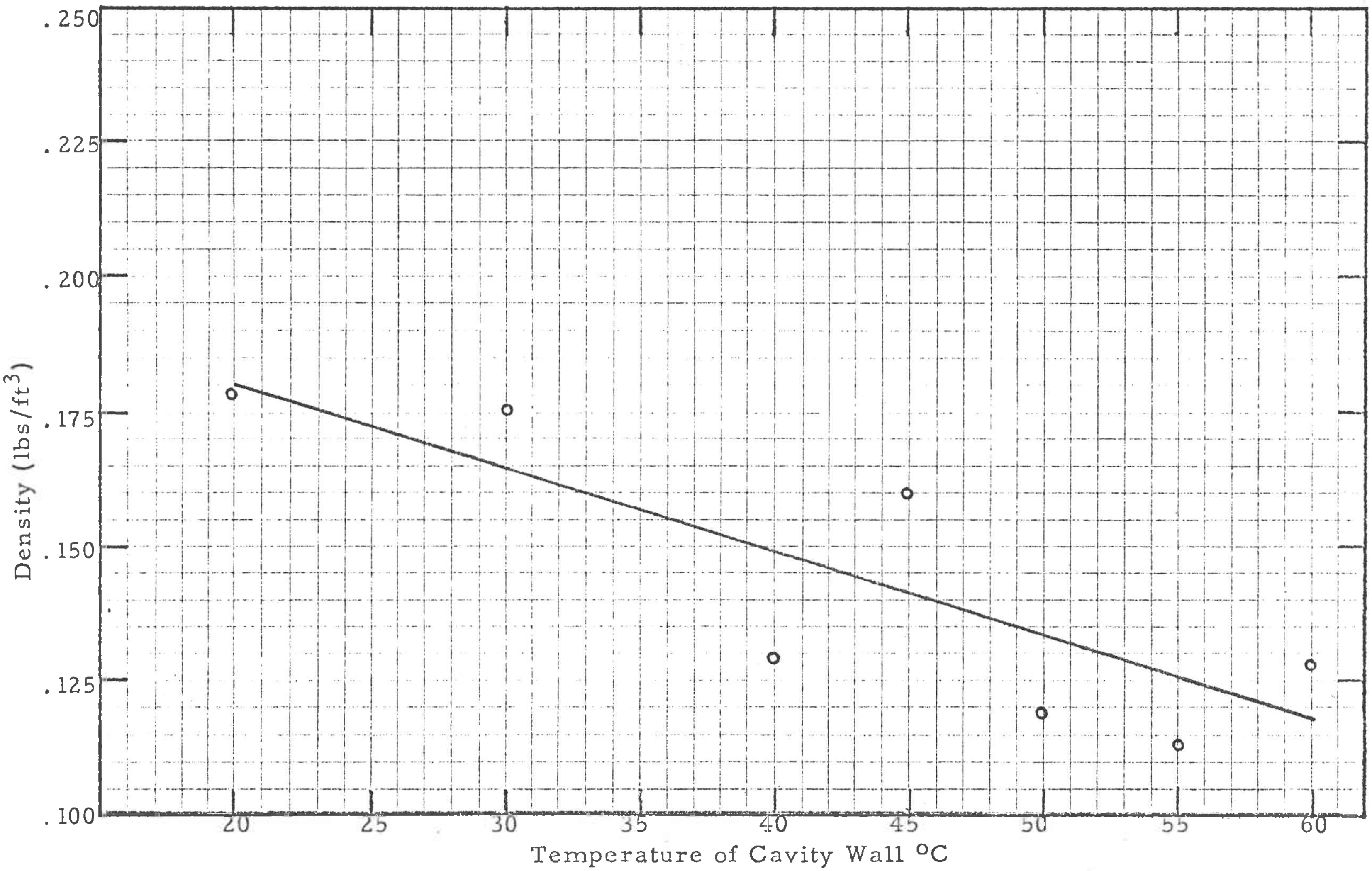


Fig. 38. Difference between overall density and average density A = 60°F B = 70°F

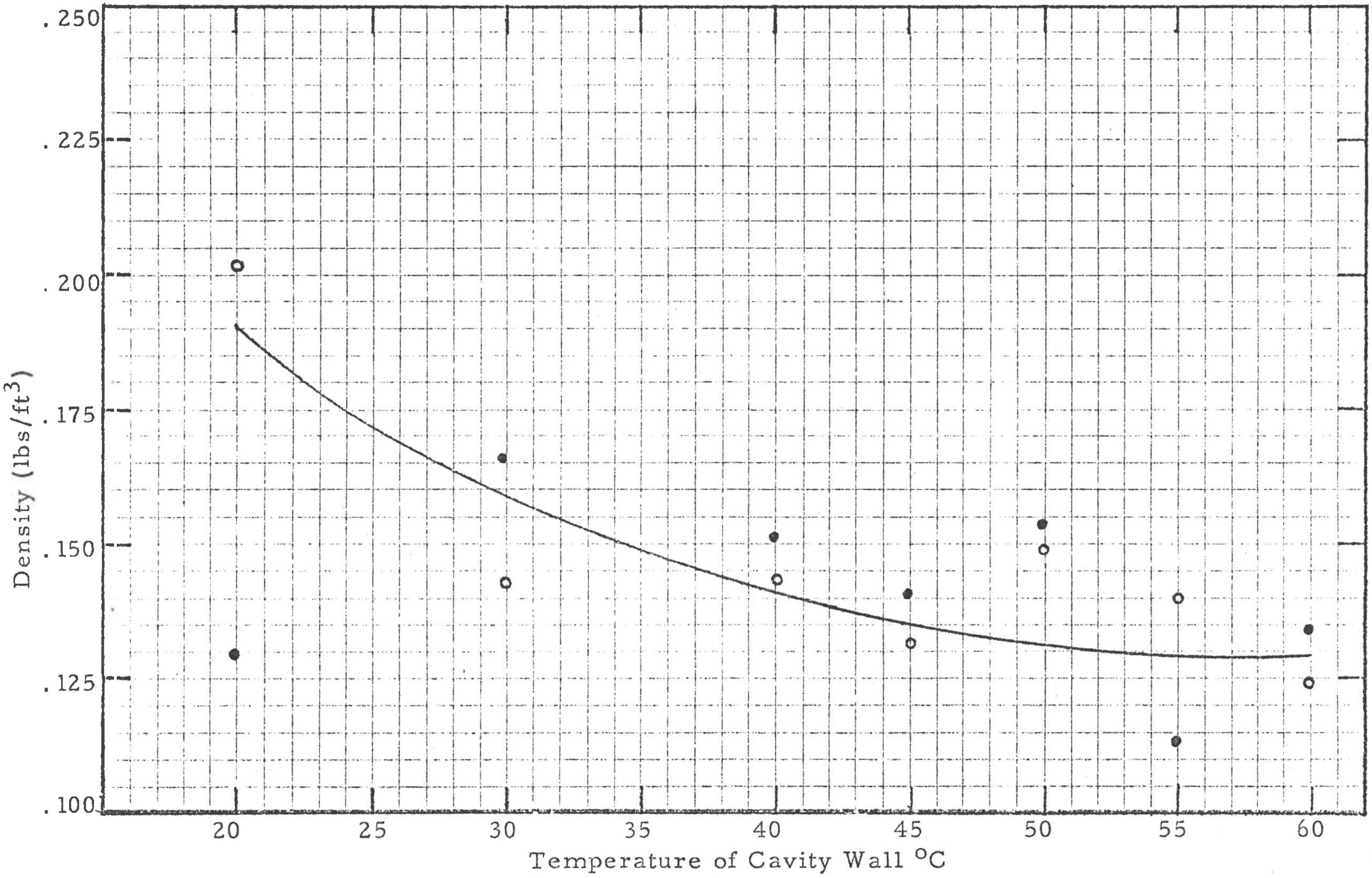


Fig. 39. Difference between overall density and average core density A= 70°F B=70°F

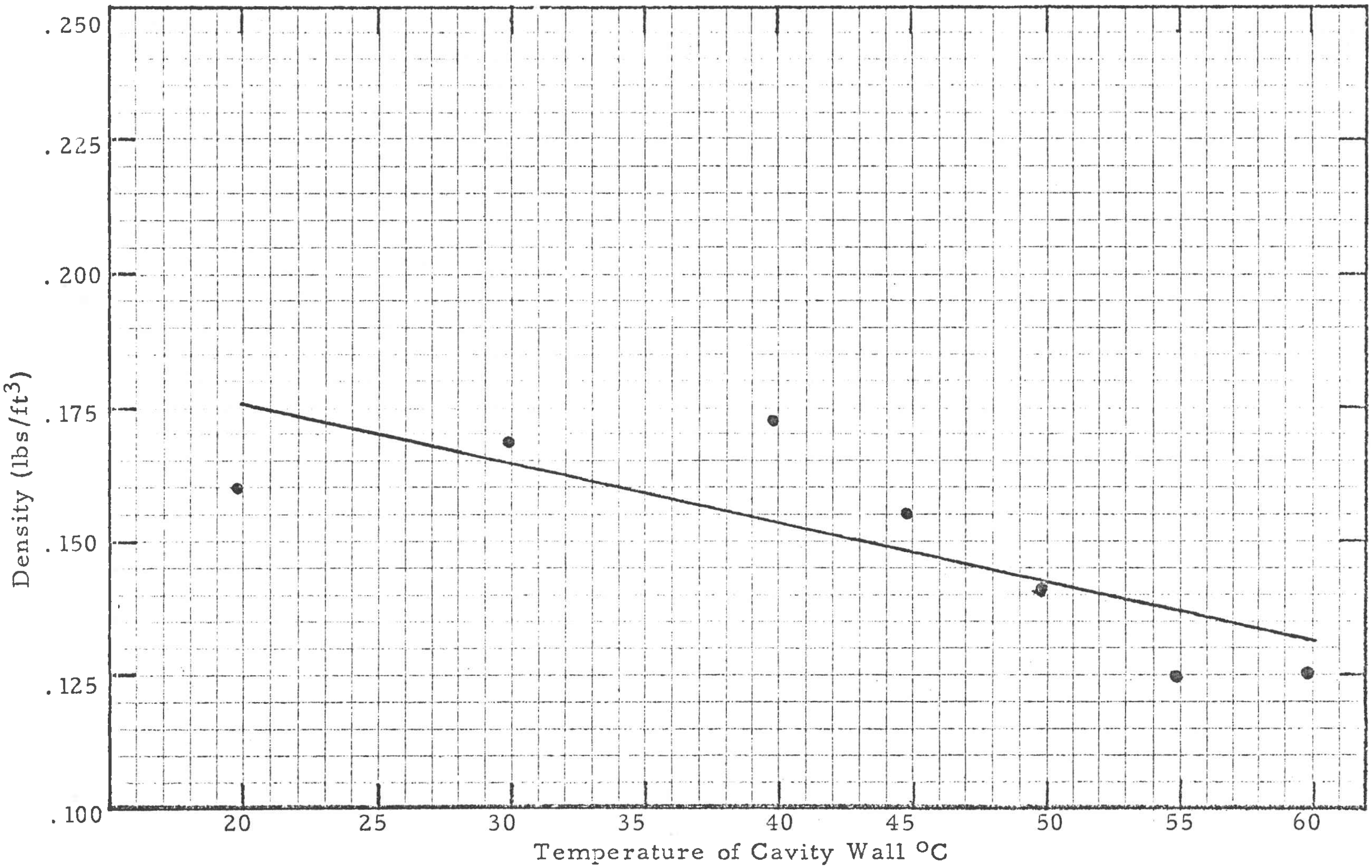


Fig. 40. Difference between overall density and average core density A = 80°F B = 70°F

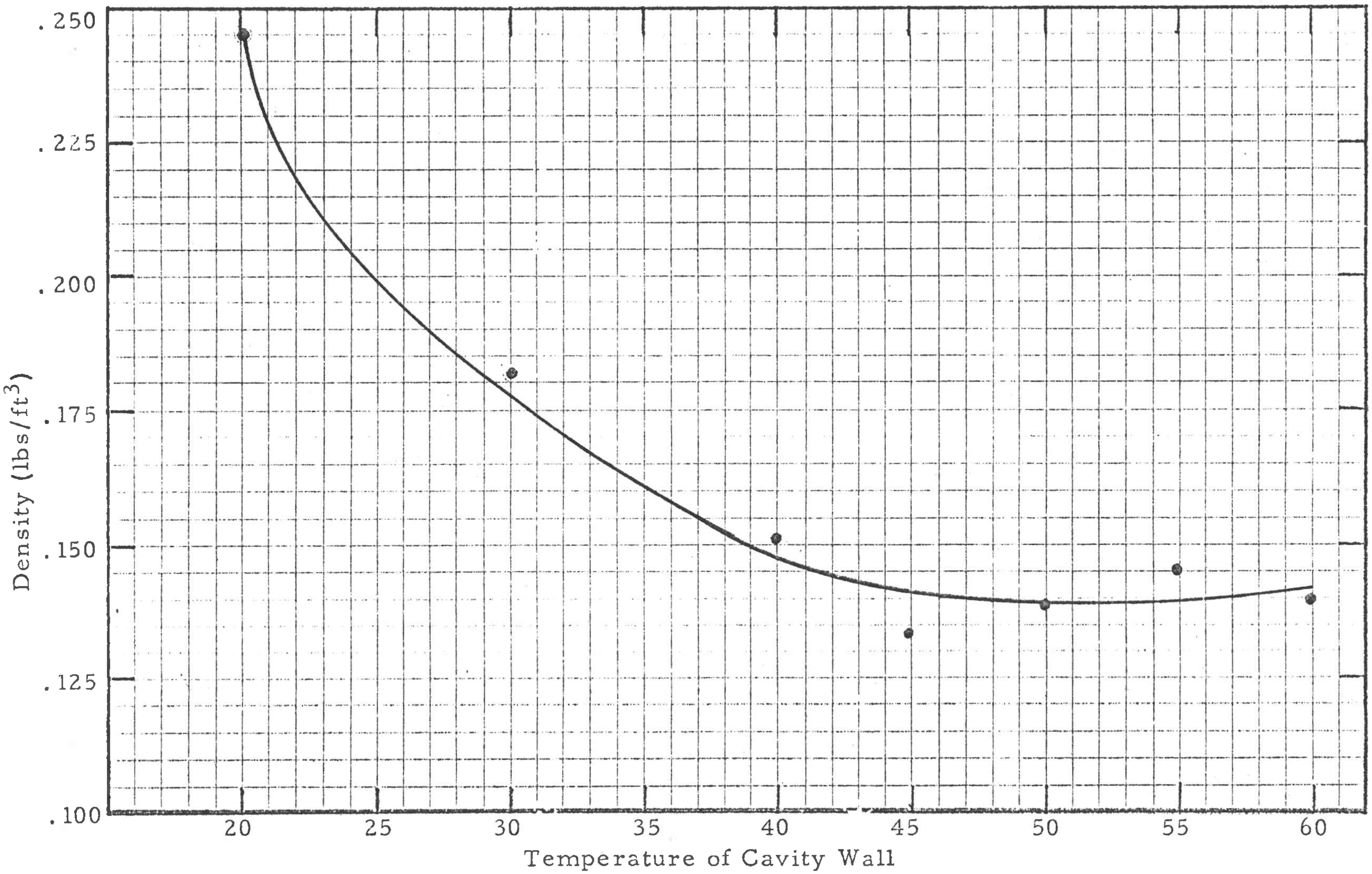


Fig. 41. Difference between overall density and average core density A=90°F B=70°F

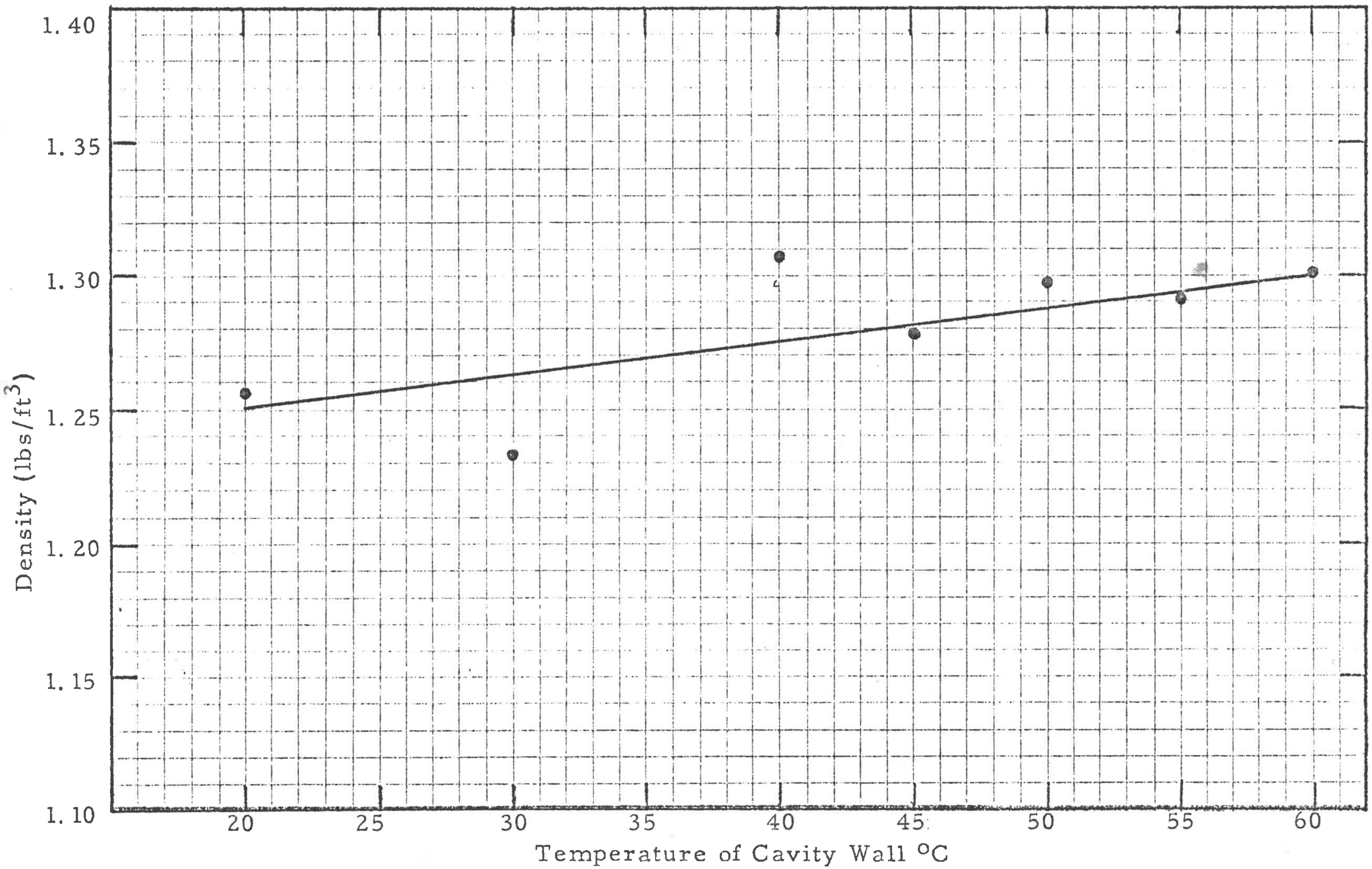


Fig. 42. Average core density for A = 60°F and B = 70°F

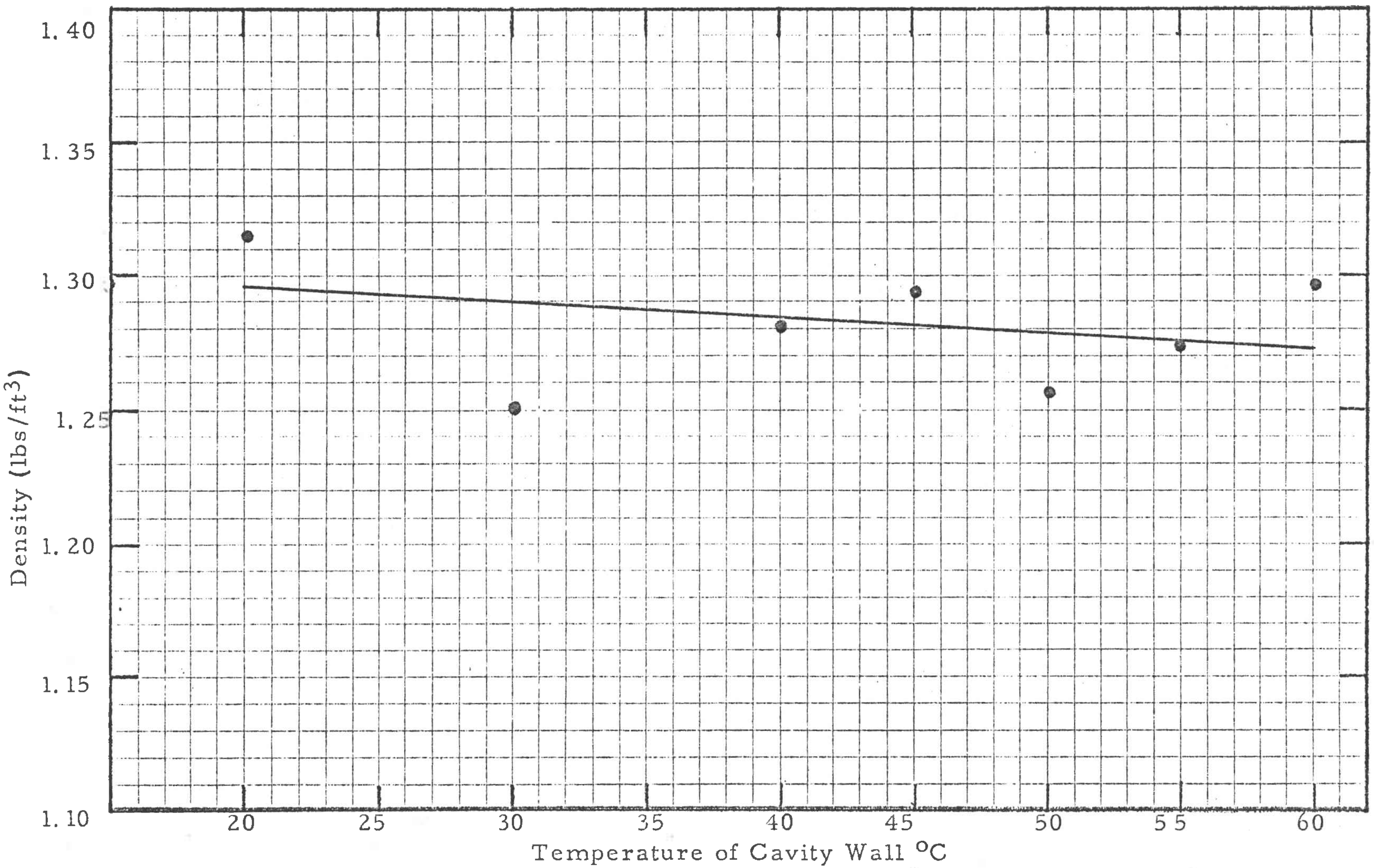


Fig. 43. Average core density for A = 70°F and B = 70°F

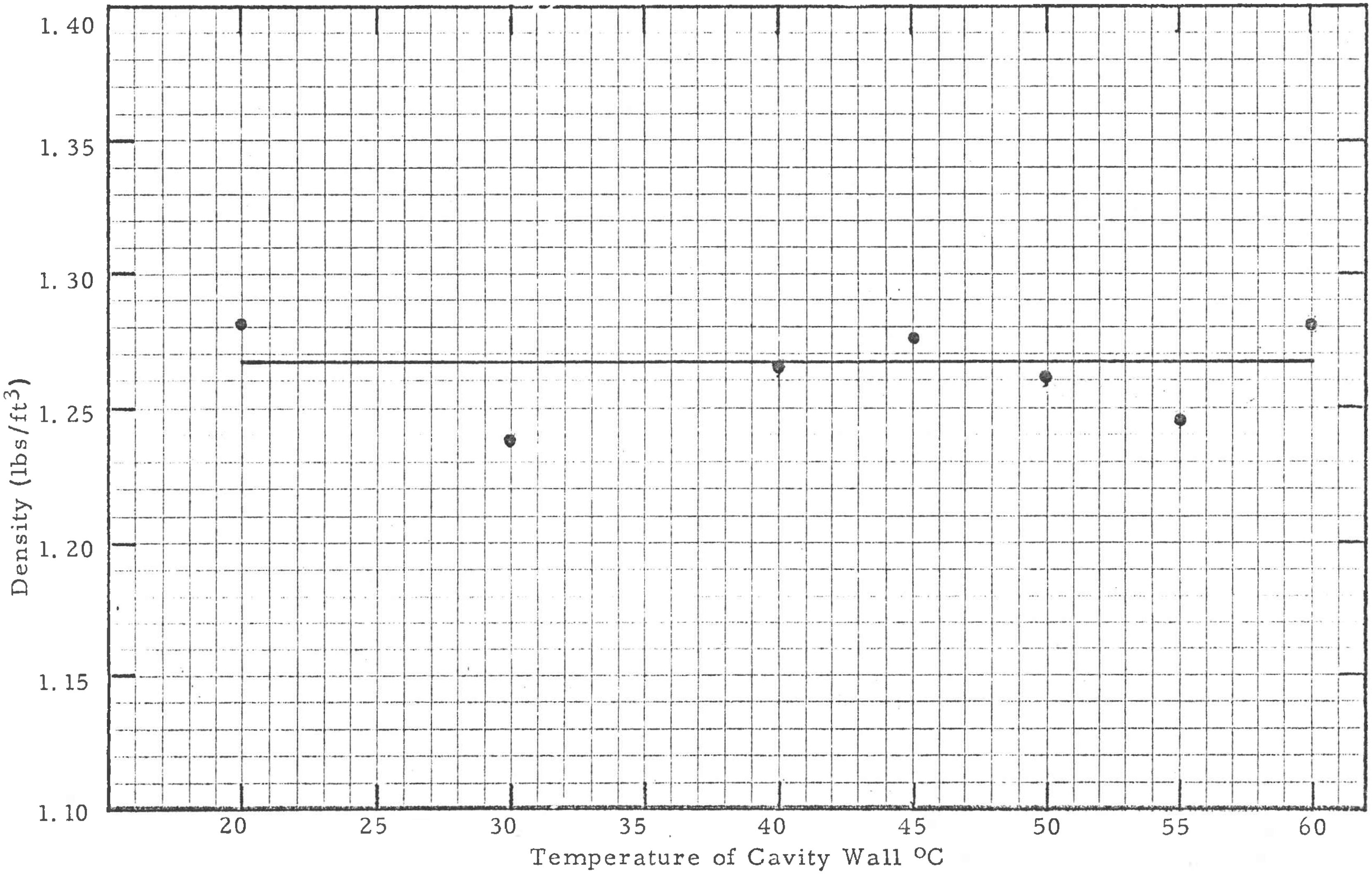


Fig. 44. Average core density for A= 80°F and B= 70°F

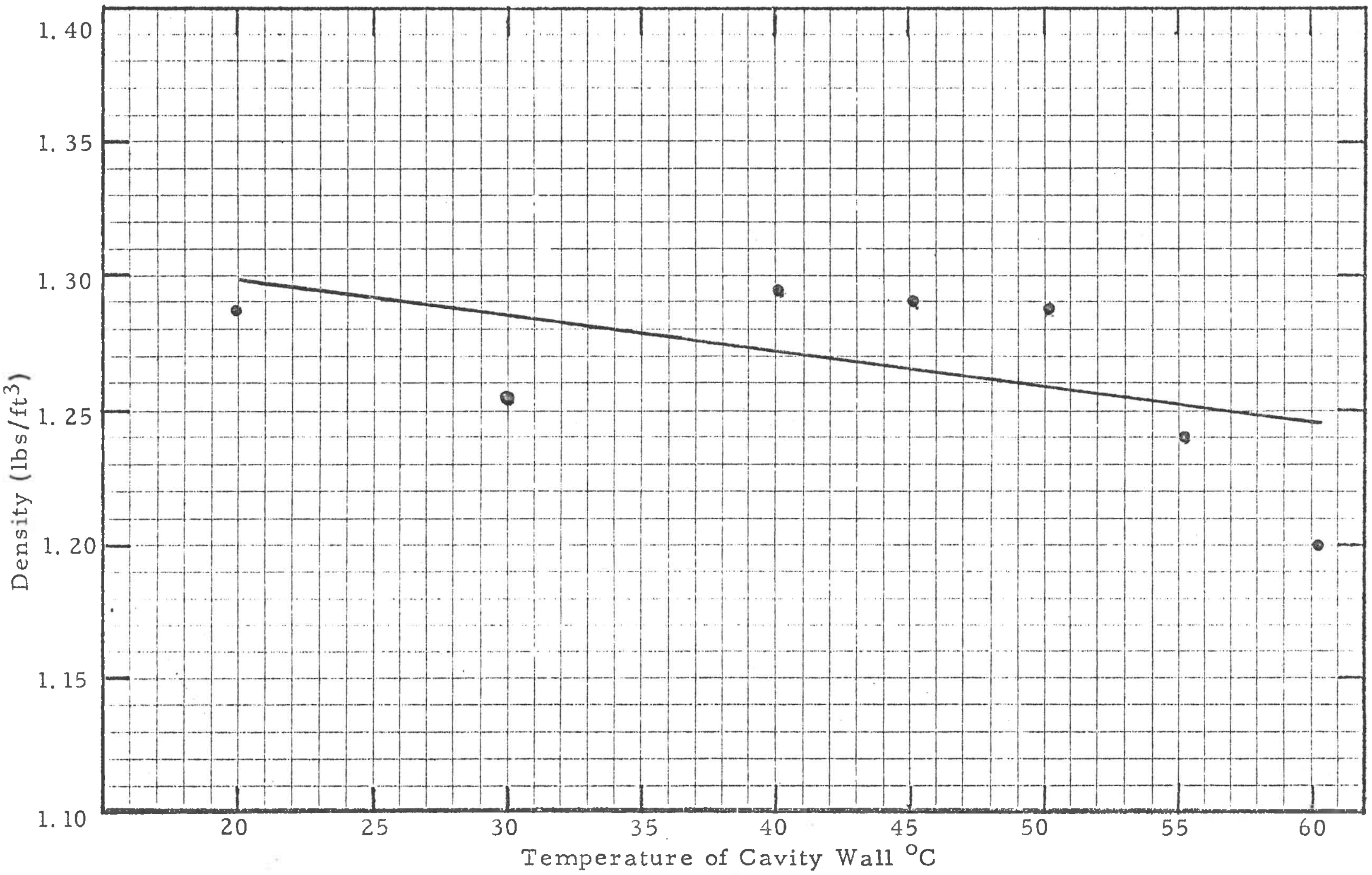


Fig. 45. Average core density for A=90°F and B= 70°F

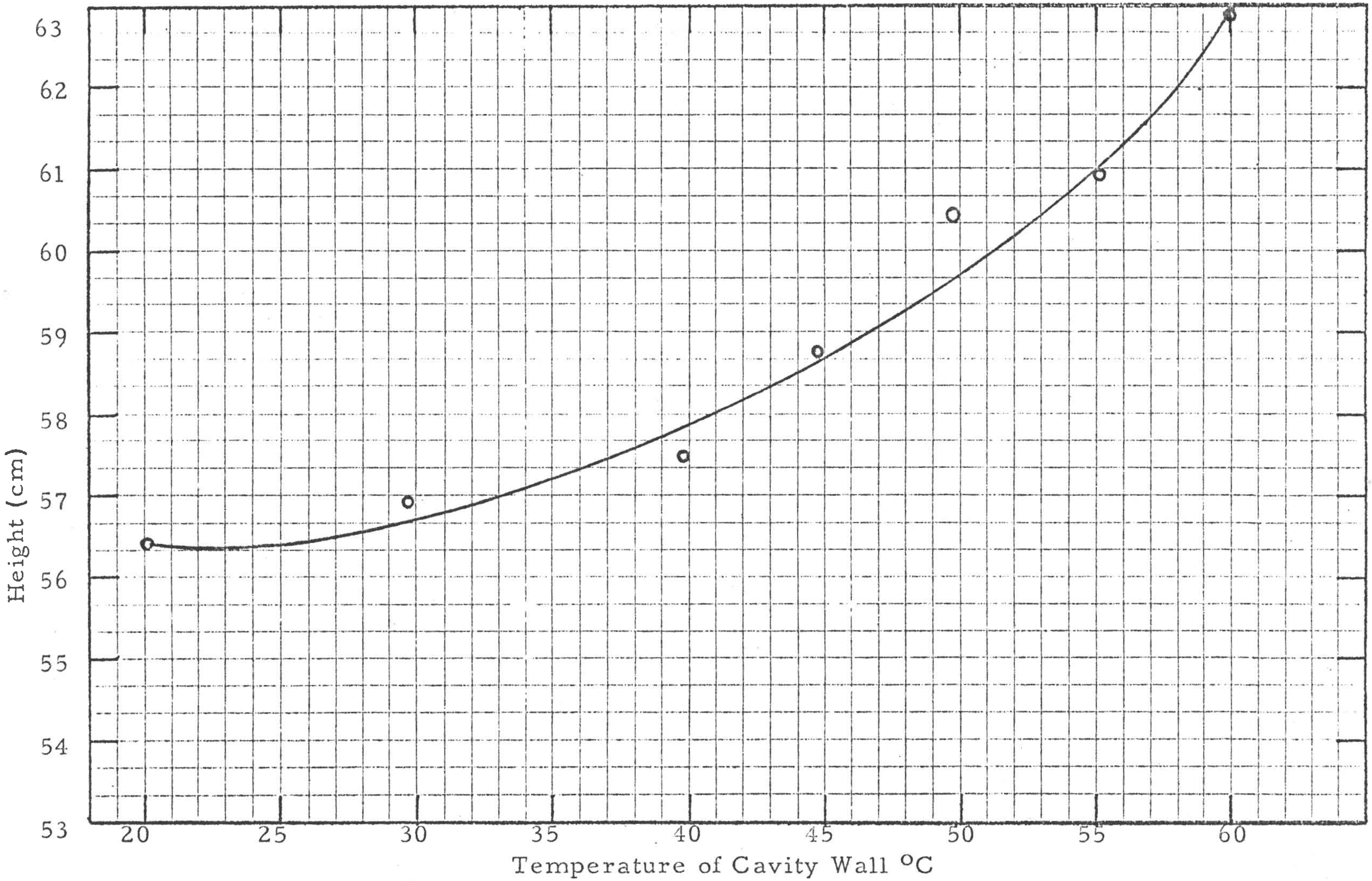


Fig. 46. Height of rise versus wall temperature for A= 60° and B=70°F

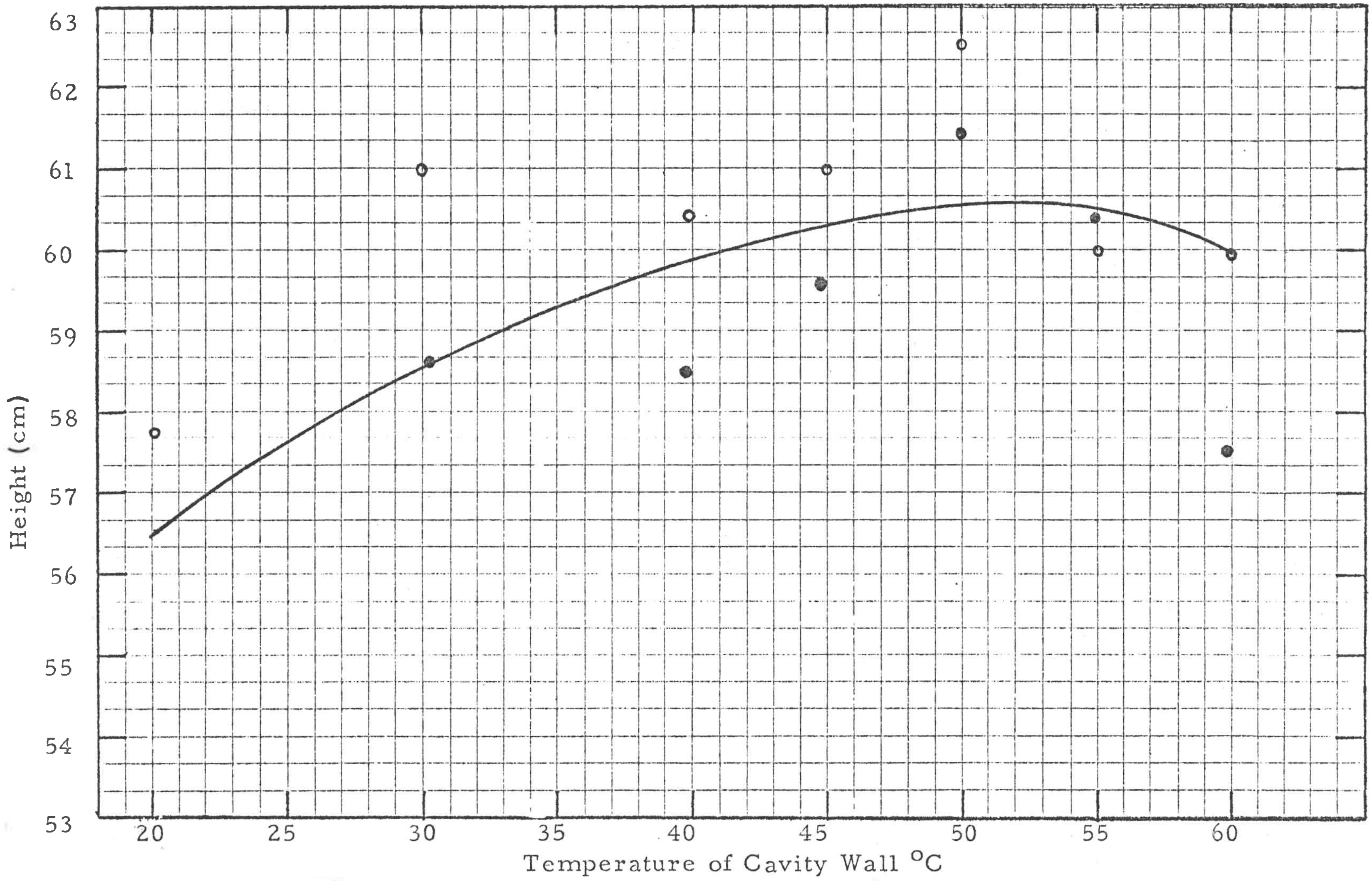


Fig. 47. Height of rise versus wall temperature for A= 70°F and B= 70°F

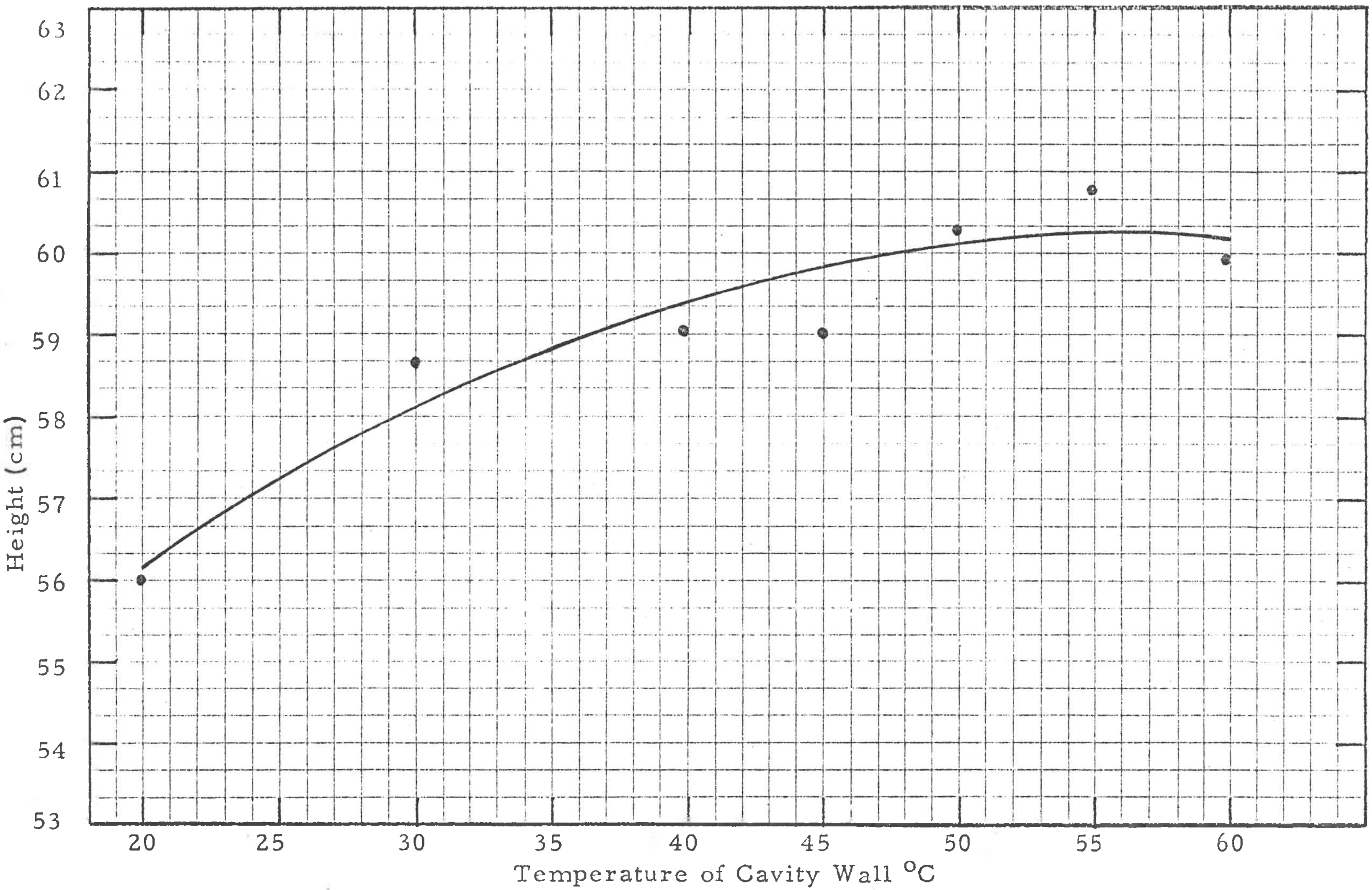


Fig. 48. Height of rise versus wall Temperature for A= 80°F and B=70°F

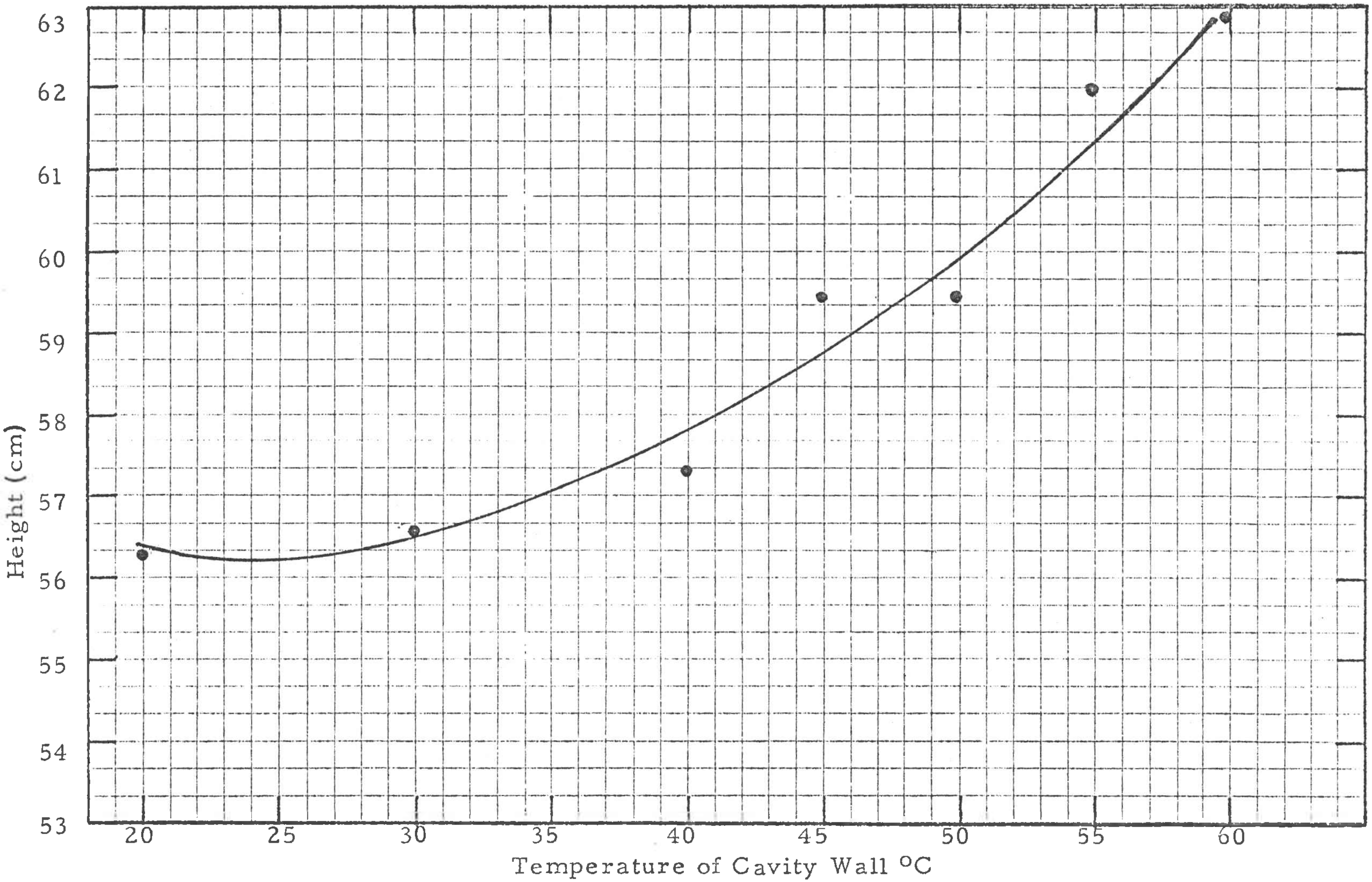


Fig. 49. Height of rise versus wall temperature for A = 90°F and B = 70°F

To get an idea of the repeatability of the experiments, tests 1 through 14 were run more than once. The results in the range where component A was held at 70°F and component B at 70°F were evaluated and plotted on the graphs in Figures 39 and 47. Table 19 shows the results. This data helped to smooth out the curves on the graphs and helped to determine the type of curve obtained.

Table 19

Temperature Sensitivity Tests 8 Through 14 (Repeated) Where A = 70°F and B = 70°F

Test #	Temp. of Cavity	Density of Sample	Density of Bottom Core	Density of Top Core	Average Core Density	Height of Rise	Difference Between Overall and Average Core Density
8a	20°C	1.451 lb/ft^3	1.252 lb/ft^3	1.246 lb/ft^3	1.249 lb/ft^3	57.8cm	.202 lb/ft^3
9a	30°C	1.385 lb/ft^3	1.243 lb/ft^3	1.243 lb/ft^3	1.243 lb/ft^3	61.0cm	.142 lb/ft^3
10a	40°C	1.387 lb/ft^3	1.249 lb/ft^3	1.238 lb/ft^3	1.2435 lb/ft^3	60.5cm	.1435 lb/ft^3
11a	45°C	1.369 lb/ft^3	1.234 lb/ft^3	1.234 lb/ft^3	1.234 lb/ft^3	61.0cm	.135 lb/ft^3
12a	50°C	1.396 lb/ft^3	1.246 lb/ft^3	1.246 lb/ft^3	1.246 lb/ft^3	62.5cm	.150 lb/ft^3
13a	55°C	1.392 lb/ft^3	1.252 lb/ft^3	1.249 lb/ft^3	1.250 lb/ft^3	60.0cm	.142 lb/ft^3
14a	60°C	1.403 lb/ft^3	1.285 lb/ft^3	1.276 lb/ft^3	1.2805 lb/ft^3	60.0cm	.1225 lb/ft^3

To determine if the temperature of the ingredients was significant over the entire range of wall temperatures, an analysis of variance program was run on the computer. (See Appendix E.) This program showed that the temperature of the ingredients was not significant over the entire range of temperatures for the average core density, the difference between overall and average core density and for the height of rise reached.

CHAPTER VI

RESULTS, CONCLUSIONS AND RECOMMENDATIONS FOR FURTHER STUDY

The purpose of this chapter was to arrive at conclusions from the data obtained in Chapter V and the information gleaned from other researchers.

The less sensitive the process is to the variables, the easier it is to control

1. Viscosities. The viscosities of the two components are similar when the B component is held near 72°F and the A component is chilled to 60°F or slightly lower. This range was selected because above the temperature of 72°F there will be freon loss from the masterbatch. The lower values are wanted also because the viscosities are easier to handle and proportion correctly, and at this range extensive heating or cooling was not needed. The mixing will be optimized at this range because the viscosities are similar. This is especially true in impingement mixing.

However, there is a tradeoff between mixing and reaction rate. As the temperature of the A component is lowered the cream time, rise rate and tack time are all decreased. If the reaction rate is slowed too much production rate goes down and any material savings would be lost because of decreased production.

2. Temperature of Ingredients. The results of this study showed that ingredient temperature has little effect on the core density, the skin density or the volume filled when cabinet temperatures are in the range from 35 to 55°C. The ingredient temperature does have effect upon the viscosity and the results are mentioned in the discussion above.

3. Mixing Time and Speed. This study showed that mixing time effects the cream time, the density, the appearance, and the cell size of the final foam product. Because of the equipment used the results gained from this study could not be compared to actual production processes.

4. Wall Temperature. At wall temperatures in the range of 20°C to 40°C the foam is quite sensitive to wall temperature. The slope of this curve was determined to be 2.5×10^{-2} from the graphs and the units used. In the range of 45°C to 60°C the slope is practically flat in this region, and therefore, the sensitivity is low.

At $60^{\circ}C$ and above the sensitivity increases. This was probably the result of a premature release of freon.

Recommendations for Further Study

An investigation into mixing and effects of proper mixing would be a valuable study for helping to model the process. It is recommended that this study be made along with the test cell temperature experiments explained in this paper. No real advances in mixing have been made in the past fifteen years.

Another area requiring further investigation is the relationship of humidity, barometric pressure and ambient air to the process.

A study that would help in optimizing the process would be the development of sensors to monitor process variables. One sensor that would be valuable would be a sensor that would give constant readout of wall temperature of the cavity. If wall temperature could be monitored and coupled with a computer other variables could be changed to compensate to help in decreasing material consumption. This would help only after the relationship between the variables was established.

Since the fixtures are constantly heating up during a production run, an investigation into a way of keeping the fixtures at a constant temperature could also be an area of further research.

APPENDIX A

URETHANE FOAM MACHINERY AVAILABLE TODAY

Specifying

Urethane Foam Machinery

By Vincent Johnson, Admiral Equipment Co., div. of Upjohn Co.

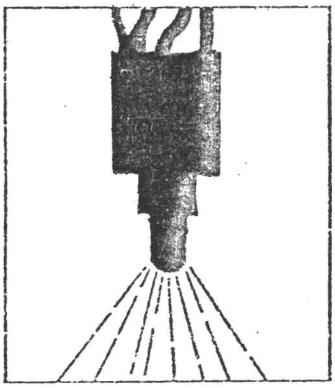
If you're looking to buy equipment for urethane foam applications, important points to keep in mind are the product, process and rate of production. Once you've determined the application, it becomes a lot easier to specify the process (e.g., spraying vs. molding) and choose the chemical system required (e.g., two vs. three components).

Same latitude with process Pour, froth, spray and injection are the four basic dispensing methods governing urethane foam equipment. Particular machines are capable of dispensing urethane by two or more of these approaches, but some modifications are usually required for the conversion from one mode to another.

The various ways of dispensing urethane can each be employed with either of the basic processing techniques—i.e., molding or casting, both generally considered a form of pouring. The particular dispensing method you settle on will be largely dependent on the product being made and chemicals needed to produce it. Particular attention must be paid to dispensing when interfacing your urethane equipment to the production process you desire.

For the purposes of this discussion, we will group dispensing methods into two broad classes—spraying and molding.

Spraying is usually, though not exclusively, a field application. In many respects selecting spray machinery is similar to selecting any other urethane-dispensing equipment: One looks for simplicity, dependability, versatility and value. Other points to be kept in mind include: (1) output range and control, (2) machine weight and portability, (3) electrical and air requirements, (4) material heating capability, (5) weight and dependability of the spray gun (head), (6) solvent flushing or air-purge provisions for cleaning the gun, (7) ease of calibrating, (8) pulsation-free spraying, (9) pumping ratio, (10) maximum pumping pressure and means of adjustment, (11) effect of material viscosity on output.



To specify machinery needed for a molding application, the part weight and characteristics of the chemicals to be used become essential. The weight of the part is directly related to machine output; furthermore, the larger the part, the larger is the machine needed and, consequently, the greater the attendant investment. An important chemical characteristic to bear in mind in a molding operation is the system's cream time (i.e., the interval between dispensing and initiation of reaction, as it is visually detected). It is the cream time that determines how long a period one has to dispense the material.

Besides the points of interest which also apply to spray machinery, factors that determine the selection of equipment for urethane molding applications include: (1) shot or dispensing accuracy and metering precision, (2) mixing action, (3) ease of maintenance for the head and metering system, (4) process control options.

As for the properties and characteristics of the chemical system you're running, bear these in mind:

- Viscosity.
- Reaction temperature.
- Specific gravity.
- Reactivity time—cream time, gel time (or pot life, for elastomers), tack-free time, rise time, cure and demold time.
- Free-rise density.

Frothing machinery is really a type of molding or casting equipment. What it involves is addition of another component (usually fluorocarbon 12) directly into the mixing head. While pumping and metering units are standard for the third component, the mixing head operates under pressure of about 100 psi to keep the frothing agent liquid until it's discharged into the mold. Two types of head are used in this low-pressure process: (1) a conventional mechanical mixer with air-controlled backpressure valve and (2) a helical mixer—it's simpler but generally has a more limited output range.

Determining the right output When you have decided on the dispensing process best suited to your needs, you should next consider machine output (or throughput). This is the weight or volume of the chemicals per unit of time as they are metered, mixed and dispensed. Output is dependent on the pumping rate of the metering system and the mixer size (or capacity). (If output of a machine is given in terms of volume, a conversion to lb/min can be made if you know the specific gravity of the materials being used.) Maximum output is usually reported by calibrating the machine with a low-viscosity material at a 1:1 ratio. Bear in mind that machines have upper and lower output limits that should be defined and established within the framework and accuracy of the metering system and the dispense shot time at a given output. A 100-lb/min machine, for instance, cannot perform adequately at an output of 5 lb/min, but it could produce 5-lb shots with 3-sec dispense times (100 lb of material thus being dispensed in 1 min).

Another point not to be overlooked is the effect of material viscosity on a machine's output range. Remember that the output for each component stream must remain constant at any particular setting; furthermore, machine accuracy is vital with respect to reproducing timed shots.

Your choice of chemicals as well as the product being made will determine whether you should have fixed or variable output ratios. Fixed-ratio machines dispense the same unit weight (or volume) of each chemical component simultaneously, requiring mechanical changes for processing chemicals with different ratios. Of course, this seeming drawback is overcome by using systems specifically formulated to offer 1:1 or fixed ratios. Variable-ratio machines, on the other hand, offer adjustable outputs for each of the chemical components. For example, a 100-lb/min machine set at a 1:1 ratio and maximum output dispenses 50 lb/min of each component. When set at 2:1, the same machine dispenses only 50 lb of the first component and 25 lb of the second, cutting output to 75 lb/min.

Mixing and metering A critical factor in producing good urethane product is the quality of the mixing action. The mixing head is supplied with urethane chemicals under pressure from the metering system or the material reservoir. In most spray operations, these components dead-end at the head. In other cases, the chemicals continuously recirculate, returning each material to its respective reservoir. Valves at the mixing head allow each chemical stream to simultaneously enter the mixing chamber. This is accomplished by balancing the recirculation pressure with the dispensing pressure so that there is no pressure difference between either of the two modes.

Pressure balancing is also important in calibrating machine output. In either the dead-end or the recirculating-feed systems, different component streams may exhibit somewhat different pressures because of dissimilar viscosities or flow characteristics. The head principally functions as a two- or three-way-valve system that controls the entry of material into the mixing chamber.

The three basic types of mixers used on urethane equipment are:

- *High-speed mechanical (dynamic) rotating mixers.* Designs include interrupted helical gears, roto-stators, worm gears, impellers or similar types.
- *Motionless (no-moving-parts) mixers.* These depend upon turbulent flow to induce mixing. The available designs offer regular or irregular helices, split- or flow-reversing ribbons, etc.
- *High-pressure direct- or indirect-stream impingement mixers.* Such devices use the kinetic energy of the material for mixing.

Another point of interest in this area is solvent-free, or self-cleaning, mixers. You might also consider operating your machines on multiple short shots, since doing so requires little or no flushing. While the technique is not widespread and depends on the system formulation as well as mixer configuration, it may be worth your while to take a look into it. Consult your machine supplier to determine which particular type of mixing arrangement best suits the material system and process you have in mind.

There are four basic metering systems used to pump the chemical components of a urethane system to the mixing head:

1. *Rotary gear pumps.* These are either the fixed-speed, variable-displacement types or those with variable speed and fixed displacement. Special shaft seals are recommended for pumps working with materials containing isocyanates since these react with atmospheric moisture to form urea, which is extremely abrasive and a cause of pump-seal failure.
2. ***Positive-displacement cylinders, piston or rod type.*** These may be operated by either a pneumatic or hydraulic master-drive cylinder.
3. ***Annular or axial piston pumps.*** These are multicylinder units used to develop high pressures.
4. ***Flow-control valves.*** These devices meter materials from pressurized vessels; they are not pumps and consequently preclude any recirculation of components.

A word on RIM Reaction injection molding (RIM)—or liquid injection molding (LIM), as it's sometimes called—has recently attracted the interest of plastics processors. The RIM process consists of injecting liquid urethane chemicals into closed molds at very fast rates. The chemicals themselves are highly catalyzed and therefore react extremely fast. Compared to injection molding, RIM offers the following advantages: (1) short demold times, (2) short mold cycle times, (3) low capital investment, (4) large-part capability, (5) elimination of flow and stress lines, (6) low molding pressure, (7) ease in handling difficult mold configurations.

Output capacities for RIM equipment are generally on the order of 100-200 lb/min, though units are available that go as high as 600 lb/min. [See p. 109.] What's more, one foam machine can usually service several molds, the arrangement often involving a manifold system which brings the reactive ingredients to mixing heads positioned at the individual molds.

Liquid reactants are delivered to the mixing head at pressures of about 1500-3000 psi; these pressures are commonly obtained via piston pumps that also serve as metering pumps. While mixing heads can be variously designed, all are self-cleaning cavities of rather small volume (about 0.3 cu in., or 15-30 times smaller than heads on low-pressure equipment).

Most RIM equipment is designed for two components. Other systems use an in-line piston pump that delivers the additional component to the suction side of the polyol stream; pump is designed to run only while the unit is dispensing into the mold. □□

COMPARATIVE SPECIFICATIONS URETHANE MACHINERY

SPRAY

Max Output, lb/min	Air, cfm @ psi	Ratio Output	Electric Power, kw	Mixing Action[a]	Wt. of Head, lb	Pump Type	Pump Pressure, psi	Model Designation	Mfr.
2-4	[illegible]	—	—	—	1.5	—	—	69GW	Binks
2-12	[illegible] @ 100	1:1-4:1	5	Hyd-Static[b]	3	Piston	400-1000	FF	Gusmer
2-16	[illegible]	—	—	—	1.5	—	—	18CAT	Binks
2-20	4-15/70	—	—	—	0.1	—	—	18CAT	
	9-12/70	—	—	—	1.75	—	—	18NC	
2-25	Airless	—	—	—	1.75	—	—	43PL	
	Airless	—	—	—	2	—	—	43PLA	
	Airless	—	—	—	2.1	—	—	Maverick	
	@ 30	—	—	—	0.1	—	—	ACI	
	16 @ 80	—	—	—	2.6	—	—	Renegade	
Up to 8	17	1:1	None	Static	Hand gun	Pneumatic	Up to 60	Foam Master	Ransburg
3-30	7 @ 90-100	1:1-4:1	13	Hyd-Static[b]	3	Piston	400-1000	H-II	Gusmer
16	—	—	—	—	0.1	—	—	18N	Binks
	Minute	—	—	—	0.25	—	—	18N	
	4-12/50	—	—	—	1.5	—	—	18N	
	12-16/70	—	—	—	1.5	—	—	18N	
Up to 20	25	1:1	220-1-3	Static	Hand gun	Pneumatic	Up to 60	Problen	Ransburg
20	Airless	—	—	—	2.25	—	—	15PL	Binks
	11 @ 80	—	—	—		—	—	101-S770	
	22 @ 100	1:1-3:1	10	Static	[illegible]	Piston	800#	200-25	Admiral
40	None	4:1	None	Static	1	None	None	64	MR Plastics
80	8 @ 80	1:1-7:1	[c]	Mech-Helix	100	Rotary	20-150	RD25 RD45 RD75	Ransburg
145	30 @ 90	1:1 to 100:3	—	M.H.S	NA	Pos Dis	Variable	Supershot	Pyles Ind

SPRAY AND POUR

2-10	29 @ 90-100	1:7 to 7:1	4.2	Mech	5	Internal gear	250	78	Martin Sweets
4-14	10 @ 100	1:4	2	Mech	4.5	Gear	—	C-3	Cannon
300	3 @ 10+	2:1	2-3	Mech	85	Vane-Piston	300-1500	Model 300	LME
500	5 @ 10+	1.5:1	5-10	Mech	110	Vane-Piston	300-1500	Model 500	
750	7 @ 10+	1.5:1	15-25	Mech	150	Vane-Piston	300-1500	Model 750	
1000	10 @ 10+	1.5:1	20-30	Mech	200	Vane-Piston	300-1500	Model 1000	

POUR

0-20	5 @ 80	1:1	220	Mech	3	Piston	800	BV0707	Accumetric
2 gm-10 lbs	7-8 400[e]	1:1-100:5	3	Static	10	Piston-Gear	Low Pres	[illegible]	Compo-Desma
50 g-80 lbs	7-8 500[e]	1:1-100:5	7.5	Mech	10	Piston-Gear	Low Pres	[illegible]	
0.37	[illegible] @ 90-100	1:6-6:1	4.2	Mech-Helix	10	Internal Gear	250	[illegible]	Martin Sweets
0.3-10	[illegible] @ 100	5:1-1:5	6	Mech	75	Gear	400	[illegible]	Accuratio
0.5-150	1 @ 100	3:100-100:3	5	Mech-Static	75-200	Piston	400	[illegible]	
1.5	—	1:1-1:3	7	Impingement	50	Axial piston	Up to 3500	575	Compo-Desma
2-10	1 @ 100	5:1-1:5	5	Mech-Static	40	Gear	400	SPD-10	Accuratio
	2 @ 60	1:1 @ 10[f]	8	Mech-Helix	100	Gear	Low	600-210	Stepan
	10 @ 100	5:1-1:5	4	Mech-Static	40	Piston	400	RM-5-10	Accuratio
	10 @ 100	2:1-1:2	3	Mech-Static	40	Piston	600	RM-20-[illegible]	
3-20	2 @ 80	1:1 @ 20[g]	9	Mech-Helix	125	Gear	Low	700-[illegible]	Stepan
4	15 @ 90	Variable	3	Mech	Boom	SPD	120	4	McNeil-Femco
5	—	1:1-100:300	2	Mech	—	Piston	125	600[illegible]	Hardman
5-20	5 @ 90	1:1 to 8:1	3	Mech	35	Gearpumps	20-250	A-5 A-10 A-20	No. American
5-25	5 @ 60	1:5-5:1	20	Mech-Static	25	Pos Disp	30	#575	Reactive Fl.
5-30	1 @ 100	5:1-1:5	5-7	Mech-Static	40	Gear	400	SPD-25[h]	Accuratio
	15 @ 100	2:1-1:2	4	Mech-Static	60	Piston	600	RM-5-30[i]	
5-100	1.5 @ 100	1:1-4:1	5-30	Mech	20-30	Gear	160	[illegible]2P	Admiral
6-40	2 @ 80	1:1 @ 40[k]	10	Mech-Helix	132	Gear	Low	800-640	Stepan
7.5	—	1:1-1:3	7	Impingement	60	Axial piston	Up to 3500	576	Compo-Desma
10	—	1:1-100:300	2	Mech	—	Piston	[illegible]	600IV	Hardman
10-200	2.0 @ 100	3:1-1:3	7.25	Mech	40-80	Piston[l]	to 700	[illegible]2V[l]	Admiral
12	1 @ 100	4:1-1:1	3.5	Mech	20	Gear	180	120-2P	
12-60	2 @ 80	1:1 @ 60[m]	11	Mech-Helix	150	Gear	Low	900-1260	Stepan
14.5	—	1:1-1:3	7	Impingement	80	Axial piston	Up to 3500	577	Compo Desma
20	15 @ 90	Variable	5	Mech	Boom	SPD	120	20	McNeil-Femco
20-60	1 @ 100	5:1-1:5	8	Mech-Static	60	Gear	400	EPD-60	Accuratio
20-100	20 @ 100	2:1-1:2	4	Mech-Static	60	Piston	600	RM-5-100[n]	
25-75	5 @ 60	1:5-5:1	20	Mech-Static	35	Pos Disp	45	2575	Reactive Fl
30	1-2 @ 90-100	1:1-20:1/1:20	5	M.S.H	30-80	Internal gear	10-150	D25	Ransburg
	2 @ 100	10:1	[o]	Mech	[illegible]	Gear	300	30	MR Plastics
	50 @ 90	1:1-100:3	[p]	M.H.S	NA	Piston	600	Super Flo	Pyles Ind
30-100	5 @ 90	1:1-8:1	18-22-24-37	Mech	50-50-50-60	Gear pumps	20-250	E-30[q]	No. American
	2 @ 100	5:1-1:5	9	Mech	60	Gear	400	EPD-100	Accuratio

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[a] Mech. Helix or Static. [b] Mechanically self-cleaning, no air or solvent fluids. [c] Unit's output depends on spray gun selected. [d] 220 VAC/60/3. [e] Liter/Min. [f] 3:1 @ 8. [g] 3:1 @ 15. [h] EPD-30 has similar properties. [i] RM-20-30 has similar properties. [j] Variable output, constant displacement. [k] 3:1 @ 30. [l] Also froth. [m] 3:1 @ 40. [n] HM20-100 has similar properties. [o] 40 amps @ 220 volts, 3 phase. [p] 120 vac control circuits only. [q] Similar properties for E-50, E-70 and E-100.

URETHANE MACHINERY (Continued)

POUR (Continued)

Max Output, lb/min	Air, cfm @ psi	Ratio Output	Electric Power, kw	Mixing Action[a]	Wt. of Head, lb	Pump Type	Pump Pressure, psi	Model Designation	Mfr.
32 liters[d]	—	Variable	16.5	Impingement	15-25	Piston	2500	PU 16	Krauss Maffei
40.8	10 @ 180	Variable	14	Impingement	30	Piston	2000[f]	H 40-2	Cannon
45-100	1 @ 90-100	1:5-5:1	10	Mech-Helix	50	Internal gear	200	Flex 100	Martin Sweets
50	—	1:1-5:1	—	Impingement	—	Radial piston	4000	HP 50	McNeil Femco
	5 @ 90	1:4-4:1	12	Mech	40	Gear	200	K500-2P	Admiral
52	1-2 @ 90-100	1:1-20:1-1:20	7	M S H	30-100	Internal gear	10-150	045	Ransburg
60	2 @ 100	10:1	7	High spd. Impeller	140	Gear	300	60	MR Plastics
	15 @ 90	1:1	6	Mech	—	Gear	200	60	McNeil Femco
60-100[c]	8 700[?]	1:1-100:25	15	Screw	—	Gear	Low Pres	1531[h]	Compo Desma
75-150	60	1:5-5:1	60	Mech-Static	65	Pos Disp	COP	75150	Reactive Fl
80 liters	—	Var	27.5	Impingement	15-25	Piston	2500	PV 40	Krauss-Maffei
80	20 @ 90	Variable	8	Mech	Boom	Gear[i]	120	80	McNeil-Femco
95	1-2 @ 90-100	1:1-1:20-20:1	7	M S H	30-100	Internal gear	10-150	075	Ransburg
100	15 @ 90	Variable	26	Mech	Boom	Gear[i]	120	100	McNeil-Femco
	20-30 @ 100	—	24	Mech	50	Internal gear	200	RFS-30x20-100-3BD	Martin Sweets
100-200	10 @ 180	Variable	20	Impingement	20	Piston	2000	H 100-2	Cannon
100-400	2 @ 100	5:1-1:5	10	Mech	200	Gear	400	SPD-250	Accuratio
120	15 @ —	Variable	6	Mech	Boom	Gear[i]	120	120	McNeil Femco
150-400	60	1:5-5:1	60	Mech-Static	90	Pos Disp	60	150400	Reactive Fl
160 liters		Var	41.5	Impingement	15-25	Piston	2500	PV 80	Krauss-Maffei
180	30-40 @ 100	—	24	Mech	75	Internal gear	200	RFS 64x40-180-3BD[e]	Martin Sweets
200-400	10 @ 180	Variable	30	Impingement	30	Piston	2000	H 200-2	Cannon
222	30 @ 90	Variable	13	Mech	Boom	Gear[i]	120	222	McNeil Femco
310 liters	—	Var	85	Impingement	15-25	Piston	2500	PV 155	Krauss-Maffei
400-800	10 @ 15	Variable	50	Impingement	30	Piston	2000	H 400-2	Cannon
	15 @ 100	4:1-1:4	20	Mech	250	Gear	400	SPD-800	Accuratio
500	40-50 @ 100	—	48	Mech	150	Internal gear	200	FS-84x70-500-5[illegible]D[e]	Martin Sweets

FROTH

Max Output, lb/min	Air, cfm @ psi	Ratio Output	Electric Power, kw	Mixing Action[a]	Wt. of Head, lb	Pump Type	Pump Pressure, psi	Model Designation	Mfr.
0.5-150	1 @ 100	3:100-100:3	5	Mech-Static	75-200	Piston	400	Meter Maid	Accuratio
10	2-3 @ 100	1:1-10:1	5	Helix	15	Rotary Gear	150	LC Series	Wilco
10-100	1.5 @ 100	1:1-4:1	7.22	Helix	40-50	Gear[i]	180	[illegible]F[j]	Admiral
30	15 @ 100	1:1-25:1	—	—	—	—	—	Dec-R-Froth	Ransburg
30-100	5 @ 90	1:1-10:1	10-14-16-24	Helix	40-40-40-50	Gear Pumps	20-250	F-30[k]	No. American

FROTH AND POUR

Max Output, lb/min	Air, cfm @ psi	Ratio Output	Electric Power, kw	Mixing Action[a]	Wt. of Head, lb	Pump Type	Pump Pressure, psi	Model Designation	Mfr.
0.32-2	1 @ 90-100	1:5-5:1	4.2	Mech-Helix	20	Internal Gear	200	Flex-2	Martin Sweets
0.5-150	1 @ 100	3:100-100:3	5	Mech-Static	75-200	Piston	400	Meter-Maid	Accuratio
2-10	1 @ 90-100	1:5-5:1	4.2	Mech-Helix	20	Internal Gear	200	Flex-10	Martin Sweets
	1 @ 90-100	1:6-6:1	4.2	Mech-Helix	15	Internal Gear	250	Mod 2	
2-100	5 @ 100	1:1-10:1	10	Mech-Helix	50-100	Rotary Gear	150	DR Series	Wilco
	5 @ 100	1:1-10:1	20	Mech-Helix	50-100	Rotary Gear	150	S3P Series	
4-20	1 @ 90-100	1:5-5:1	4.2	Mech	30	Internal Gear	200	Flex-20	Martin Sweets
	1 @ 90-100	1:6-6:1	4.2	Mech-Helix	30	Internal Gear	250	Mod 3	
10-25	7 @ 90	1:4	2.5	Mech	40	Gear	—	C-7	Cannon
10-30	1 @ 90-100	1:5-5:1	11.5	Mech-Helix	50	Internal Gear	200	Mod 4	Martin Sweets
10-40	1 @ 90-100	1:5-5:1	10	Mech-Helix	50	Internal Gear	200	Flex 40	
20-55	7 @ 90	1:4	8	Mech	45	Gear	—	C-25	Cannon
20-60	1 @ 90-100	1:4.5-4.5:1	11.5	Mech-Helix	50	Internal Gear	200	Mod 5	Martin Sweets
20-80	7 @ 90	1:4	8	Mech	45	Gear	—	C-35	Cannon
30-140	7 @ 90	1:4	8	Mech	45	Gear	—	C-60	
80-330	7 @ 90	1:4	35	Mech	125	Gear	—	C-150	
140-550	7 @ 90	1:4	35	Mech	125	Gear	—	C-250	

ELASTOMER CASTING

Max Output, lb/min	Air, cfm @ psi	Ratio Output	Electric Power, kw	Mixing Action[a]	Wt. of Head, lb	Pump Type	Pump Pressure, psi	Model Designation	Mfr.
0.25-10	10-35 @ 90	Variable	6.5	Mech-Static	15	Piston	200	Flying wedge	Adv. Machine
0.25-30	10-30 @ 90	Variable	30	Mech-Static	Fixed	Piston	50	Flying wedge	
5-20	20 @ 100	Var 1:4-4:1	6	Mech	80	Gear[i]	180	[illegible]C	Admiral

HIGH PRESSURE

Max Output, lb/min	Air, cfm @ psi	Ratio Output	Electric Power, kw	Mixing Action[a]	Wt. of Head, lb	Pump Type	Pump Pressure, psi	Model Designation	Mfr.
10.5	25 @ 150	2:1	5	[l]	5	Piston	3500	HKA 35C	Mobay Chemical
14	25 @ 150	1:1	5	[l]	5	Piston	3500	HKA 35H	
30	25 @ 150	2:1	10	[l]	5	Piston	3500	HKA 100C	
40	25 @ 150	1:1	10	[l]	5	Piston	3500	HKA 100H	
48	25 @ 150	2:1	15	[l]	5	Piston	3500	HKA 165C	
63	25 @ 150	2:1	20	[l]	5	Piston	3500	HKA 245C	
64	25 @ 150	1:1	15	[l]	5	Piston	3500	HKA 165H	
84	25 @ 150	1:1	20	[l]	5	Piston	3500	HKA 245H	
135	25 @ 150	2:1	25	[l]	5	Axial piston	2000	HKA 500C	
200	25 @ 150	1:1	30	[l]	5	Axial piston	2000	HKA 500H	
300	25 @ 150	2:1	40	[l]	5	Axial piston	2000	HKA 1000C	
400	25 @ 150	1:1	50	[l]	5	Axial piston	2000	HKA 1000H	

[a] Mech, Helix or Static. [b] 50 amps @ 220 volts, 3 phase. [c] Cc/sec. [d] Liter/min. [e] With multistation. [f] Also rotary piston; 5 components standard. [g] Also annular piston. [h] Flexible and rigid urethane foam, continuous slab production line. [i] Variable output, constant displacement. [j] Also pour. [k] Similar properties for F-50, F-70 and F-100. [l] Counter current injection of high velocity streams.

APPENDIX B

FOAM RISE TESTS

Urethane Foam Flow Test
Three Foot Tube

I. Scope & Application

This is a bench scale method for determining the flow properties of urethane foam systems.

II. Principle

A specific batch size of foam mixture is mixed in a cup upon which a funnel and 3' lined tube are placed. The foam rises in the tube to a height dependent upon the flow properties of the systems.

III. Variables

Temperature of ingredients, mixing method, mixing time, batch size and % blowing agent should be accurately controlled when comparing one system with another. No study has been made on the effect of these variables on the test results.

IV. Apparatus

1. 36" cardboard fabric tube, 3" I. D., 1/4" wall thickness
   Sonoco Products Co., Philadelphia, Penna.

2. 12"x36" 5 mil "Mylar" - E. I. du Pont de Nemours & Co., Inc., Film Dept., Wilmington, Delaware.

3. Dixie Cup #2112, 12 fluid oz. Hot Drink Cup
   Dixie Cup Div., American Can Co., Easton, Penna.

4. Tri-Pour Beaker (Plastic) 400 ml.
   Biological Research Inc., ALOE Div.
   Subsidiary of Brunswick Corp.
   12201 New Columbia Pike, Silver Springs, Md.

5. Diagger Dial-Speed Stirrer
   High Speed Model #56540B, 200 to 3000 RPM (A. Diagger Co., 159 W. Kinzie, Chicago, Illinois)

With 3-blade stainless steel propeller 2" dia.
Catalog #9240-K(A. H. Thomas, Philadelphia, Penna.)
On stainless steel stirring shaft Catalog #9240-F
(A. H. Thomas, Philadelphia, Penna.)

V. Procedure

1. Place "Mylar" film in tube.

2. Cut beaker 2.5" from top and place top section upside down in bottom of tube to act as a funnel.

3. Weigh 170 ± 1 gms of total system in Dixie cup. (170 gms required for 2 pcf system; larger amounts for higher densities).

4. Mix for 9 secs. on Diagger Stirrer at high speed, i.e. up to 3000 RPM. (Shorter mix times required for systems that cream faster than 9 secs.)(Figure 1).

V. Proceudre (cont'd.)

5. Quickly place on bench and put tube assembly over cup. Cup should fit tightly into funnel (inverted cut beaker) and funnel should fit tightly into tube to prevent foam leakage. Assembly can be held or taped* until foaming is complete. (Figure 2).

6. After foam has risen and set (Approx. 20 min. for most systems), slide tube off of foam/beaker/cup unit. (Figure 3).

VI. Report

Measure and report the following:

A. Foam height; from bottom of cup to point of maximum rise. (Figure 4).

B. Shear line height; from bottom of cup to point of foam breakdown (Shear) on foam surface. (Figure 4).

C. Core density of foam (skin removed).

*Lay 2 foot length of masking tape on bench, sticky side up. After mixing, quickly place cup in center of tape and put tube assembly over cup. While holding complete assembly with one hand, run tape up one side of tube, then the other.

Designation: D 2237 - 70

AMERICAN SOCIETY FOR TESTING AND MATERIALS
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Standard Method of Test for
RATE-OF-RISE (VOLUME INCREASE) PROPERTIES OF URETHANE FOAMING SYSTEMS[1]

This Standard is issued under the fixed designation D 2237; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval.

1. Scope

1.1 This method covers determination of the rate at which the volume of a foaming system changes under standard conditions. Two quantities, the Extrapolated Initiation Time (EIT_T) and the Extrapolated Rise Time (ERT_T) are obtained by graphical analysis.

1.2 The method and apparatus as presented are applicable to foam systems having Extrapolated Rise Times down to about 1 min. For faster systems, modifications in the apparatus, such as automatic height recording, may be required.

NOTE—The values stated in U.S. customary units are to be regarded as the standard. The metric equivalents of U.S. customary units may be approximate.

2. Summary of Method

2.1 The test apparatus consists of a constant cross-section cylindrical container and a float connected to a height-measuring device as shown in Fig. 1. A freshly mixed foaming system is placed in the bottom of the container and the float placed on the foaming system so that height versus time data may be obtained. The data are plotted on semilogarithmic graph paper as shown in Fig. 2. From this plot, rate-of-rise quantities at a specific average component temperature are obtained. The quantities are adjusted through suitable temperature compensation to correspond to 23 C (73.4 F).

3. Significance

3.1 Rate-of-rise data obtained by this method are applicable to the standard conditions under which they were obtained and are not necessarily the same as obtained under manufacturing conditions. In addition to the shape and cross section of a mold cavity, the data can also be affected by such factors as the method of mixing and the time-temperature relation of the mold surface during foaming. The test is useful for identification and comparison purposes. When properly interpreted, it can be helpful in predicting foaming behavior under nonstandard conditions.

4. Definitions

4.1 *mixing time*—time in seconds for a batch mix during which mechanical agitation is applied to the combined components of a foam system.

4.2 *dispensing time*—time in seconds for a continuous blending process during which mixed material is dispensed into a container.

4.3 *initial height*—theoretical height in inches of mixed liquid components in a given container prior to any gas formation.

4.4 *final height*—height of the foam in inches in a given container after all volume change has ceased.

4.5 *rise rate curve*—plot of change in height as a percentage of the total height change ver-

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[1] This method is under the jurisdiction of ASTM Committee D-20 on Plastics. A list of committee members may be found in the ASTM Yearbook. This standard is the direct responsibility of Subcommittee D-20.22 on Cellular Plastics.

Current edition effective Dec. 24, 1970. Originally issued 1964. Replaces D 2237 - 64 T.

sus time on similogarithmic-coordinates (see Fig. 2).

4.6 *extrapolated initiation time at a specified average component temperature* (EIT_T)—time in minutes, and corresponding to a specific average component temperature, obtained from the intersection of the linear extrapolation of the straight-line section of a rise rate curve with 0 percent height (see Fig. 2). Note that time equals zero at the start of mixing.

4.7 *extrapolated rise time at a specified average component temperature* (ERT_T)—time in minutes, and corresponding to a specific average component temperature, obtained from the intersection of the linear extrapolation of the straight-line section of a rise rate curve with 100 percent height (see Fig. 2). Note that time equals zero at the start of mixing.

5. Apparatus

5.1 *Standard Foaming Container*—The inside dimensions of the standard foaming container shall be 170 ± 3 mm ($6^{5}/_{8} \pm {}^{1}/_{8}$ in.) in diameter and 120 ± 13 mm ($8 \pm {}^{1}/_{2}$ in.) in height. The container should be constructed of low heat flow material such as pasteboard or fiberboard to minimize container effects on the foaming system. The inside surface of the container should be uncoated. Such a container is shown in Fig. 1.[2]

5.2 *Height Gage*[3]—The height gage shall consist of a float, a stem, a glass tube, and a linear scale as shown in Fig. 1. The height of the scale shall be adjusted so that it reads zero with the float resting on the bottom of the standard container. The float shall be a disk (76 ± 3 mm ($3 \pm {}^{1}/_{8}$ in.) in diameter which is constructed of low heat flow material. The total weight of the float and stem should not exceed 10 g. The end of the stem shall have a hook so that it may be suspended from the top of the glass tube. The tube and scale shall be connected to the supporting stand so as to permit them to swing to one side during dispensing.

5.3 *Mixing Components*—The mixing of the components shall be accomplished by mechanical means. This may take place by a continuous blending process in a mixing chamber from which the mixed material is dispensed or by a batch process using a mixing blade. Where the batch process is used, a heavy-duty portable drill having a mixing speed of 1500 to 2000 rpm and a 50-mm (2-in.) diameter mixer[4] is required. The mixing shall be carried out in a paper cup of proper size to permit good agitation. For combined components weighing up to 300 g, a (470-cm^3) paper cup[5] or equivalent is recommended. The accuracy of metering or weighing should be ±1 percent.

5.4 *Thermometer*—ASTM Pensky-Martens, Low-Range Tag Closed Tester Thermometer having a range from −5 to +110 C or +20 to +230 F and conforming to the requirements for Thermometer 9C or 9F as prescribed in ASTM Specification E 1, for ASTM Thermometers.[6]

5.5 *Stop Watch.*

6. Number of Test Pours

6.1 The number of pours tested for a sample can vary widely, depending on the intended use of the data. It is recommended that four random pours be made for the initial sample of the material. Then, depending on the accuracy and degree of certainty required, this sample size may be increased or decreased for subsequent samples.

7. Conditioning

7.1 The initial temperature of the components must be controlled since a change in temperature may also cause a change in the rate of rise. Unless otherwise specified, the temperature of all components at the start of mixing should be 23 ± 2 C (73.4 ± 4 F). In cases of dispute, the entire apparatus and chemical components should be conditioned at 23 ± 1 C (73.4 ± 2 F) prior to mixing and the test conducted in a conditioned room at the same condition.

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[2] A cylindrical container, uncoated, $6^{3}/_{4}$ in. in diameter, 1-gal capacity, available through the Sealright Co., Inc., Fulton, N. Y., has been found satisfactory for this purpose.

[3] Suitable equipment for conducting this test is available from the R. B. Snyder Co., 5739 West 16th St., Cicero, Ill. 60650, and Olin Corp., Plastics Group Specialty Urethane Equipment Dept., 6367 East Land Road, Brook Park, Ohio 44142.

[4] A suitable mixer is available from the Conn Co., Warren, Pa.

[5] Unwaxed 16-oz cups, Catalog No. 4416 S, available from the Lily Tulip Corp., New York, N. Y., have been found satisfactory for this purpose.

[6] *Annual Book of ASTM Standards*, Part 30.

8. Procedure

8.1 Obtain the temperature of the components to the nearest 0.1 C (0.2 F) and combine as specified by the manufacturer.

8.2 Adjust the charge of material empirically so that the final height is approximately equal to the height of the container.

8.3 Start mixing and stop watch simultaneously. If a continuous mixing process is used, dispense directly into the standard container. If a batch mix is used, use a separate cup for mixing for a specified period and the mixed components shall be poured into the standard container. Pour the mixed components into the center of the bottom of the container so that they are distributed symmetrically around the axis of the cylindrical container.

8.4 Move the height gage over the foaming system, unhook the stem from the glass tube, and lower it gently into the container. The float should be centered on the foaming system. Record height versus time data. Sufficient readings should be taken to define the rise rate curve. After the foam rise has apparently stopped, allow an elapse of time about equal to the estimated Extrapolated Rise Time and record the final height of foam.

8.5 Allow the foam to cure and cool for a minimum of 16 h under ambient conditions and then cut a nominal 50-mm (2-in.) cube from the core of the foam. Use this to inspect the cell structure and obtain the density.

9. Calculations

9.1 Determine the density to three significant figures in accordance with ASTM Method D 1622, Test for Apparent Density of Rigid Cellular Plastics.[7]

9.2 Obtain the average component temperature to the nearest 0.1 C (0.2 F). When the temperatures of the components are the same, the average temperature is that of either component. When the temperatures of the components differ, a weighted average based on the weights (or ratios) and, if significantly different, the specific heats of the components, shall be used.

9.3 Plot the rise rate curve and obtain $\mathrm{EIT_T}$ and $\mathrm{ERT_T}$. Calculate the change in height, Δh, as a percentage of the total change in height to three significant figures as follows:

$$\text{Change in height, percent} = [(h_t - h_o)/(h_f - h_o)] \times 100$$

where:

h_t = height at any given time, mm (or in.),

h_f = final height, mm (or in.),

h_o = initial height = $D/D_r \times h_f$, mm (or in.),

D = density of foam core, lb/ft^3, and

D_r = nominal density of raw materials, lb/ft^3 (may be taken as 70).

Plot the rise rate curve (change in height, Δh, versus time) on semi-logarithmic graph paper as shown in Fig. 2. Make a linear extrapolation of the straight-line section of the rise rate curve to intersect with both 0 percent and 100 percent change in height. The 0 percent and 100 percent extrapolation values become the $\mathrm{EIT_T}$ and $\mathrm{ERT_T}$ respectively, and shall be reported to three significant figures.

9.4 *Temperature Coefficients for ERT and EIT*—To maintain the indicated precision of ±5 percent for the ERT and EIT and also allow a reasonable temperature range in which to work, correct the experimental values to 23 C (73.4 F) through the use of temperature coefficients. The step of obtaining the coefficients is normally carried out once on a given foam system. The temperature coefficients obtained may then be used for adjusting all subsequent rise rate determinations. Obtain the temperature coefficients as follows: Plot rise rate curves at several (at least three) points throughout the range of temperature permitted. Then plot the $\mathrm{EIT_T}$ and $\mathrm{ERT_T}$ versus temperature. From the best fitting straight lines obtain the temperature coefficients to two significant figures as follows:

$$I = (\mathrm{EIT}_{T_1} - \mathrm{EIT}_{T_2})/(T_2 - T_1)$$

and

$$R = (\mathrm{ERT}_{T_1} - \mathrm{ERT}_{T_2})/(T_2 - T_1)$$

where:

I = temperature coefficient for EIT, min/deg F,

R = temperature coefficient for ERT, min/deg F, and

T_2 and T_1 = temperatures at two points on line.

9.5 *Standard EIT and ERT*—Calculate the Standard Extrapolated Rise Time as follows

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[7] *Annual Book of ASTM Standards*, Part 26.

(see Fig. 2):

$$ERT_{73.4\,F} = ERT_T + R(T - 73.4)$$

and the Standard Extrapolated Initiation Time as follows (see Fig. 2):

$$EIT_{73.4\,F} = EIT_T + I(T - 73.4)$$

9.6 Calculate the standard deviations (estimated) for the density, Standard Extrapolated Initiation Time, and Standard Extrapolated Rise Time, and report them to two significant figures.

10. Report

10.1 The report shall contain the following:

10.1.1 Complete identification of the material tested, including type, source, code numbers, previous history, etc.

10.1.2 Number of pours tested if different from that specified in Section 6,

10.1.3 Temperature of components and method of averaging temperatures,

10.1.4 Atmospheric conditions in test room if different from those specified in Section 7,

10.1.5 Method of mixing and dispensing time or mixing time,

10.1.6 Final height of foam in standard container if different from that specified in 8.2,

10.1.7 Standard Extrapolated Initiation Time, Standard Extrapolated Rise Time, and density to three significant figures expressed as an average value, and standard deviation,

10.1.8 Description of cell structure including uniformity and average cell size, and

10.1.9 Date of test.

11. Precision

11.1 The precision of this method has been determined to be about ±5 percent.

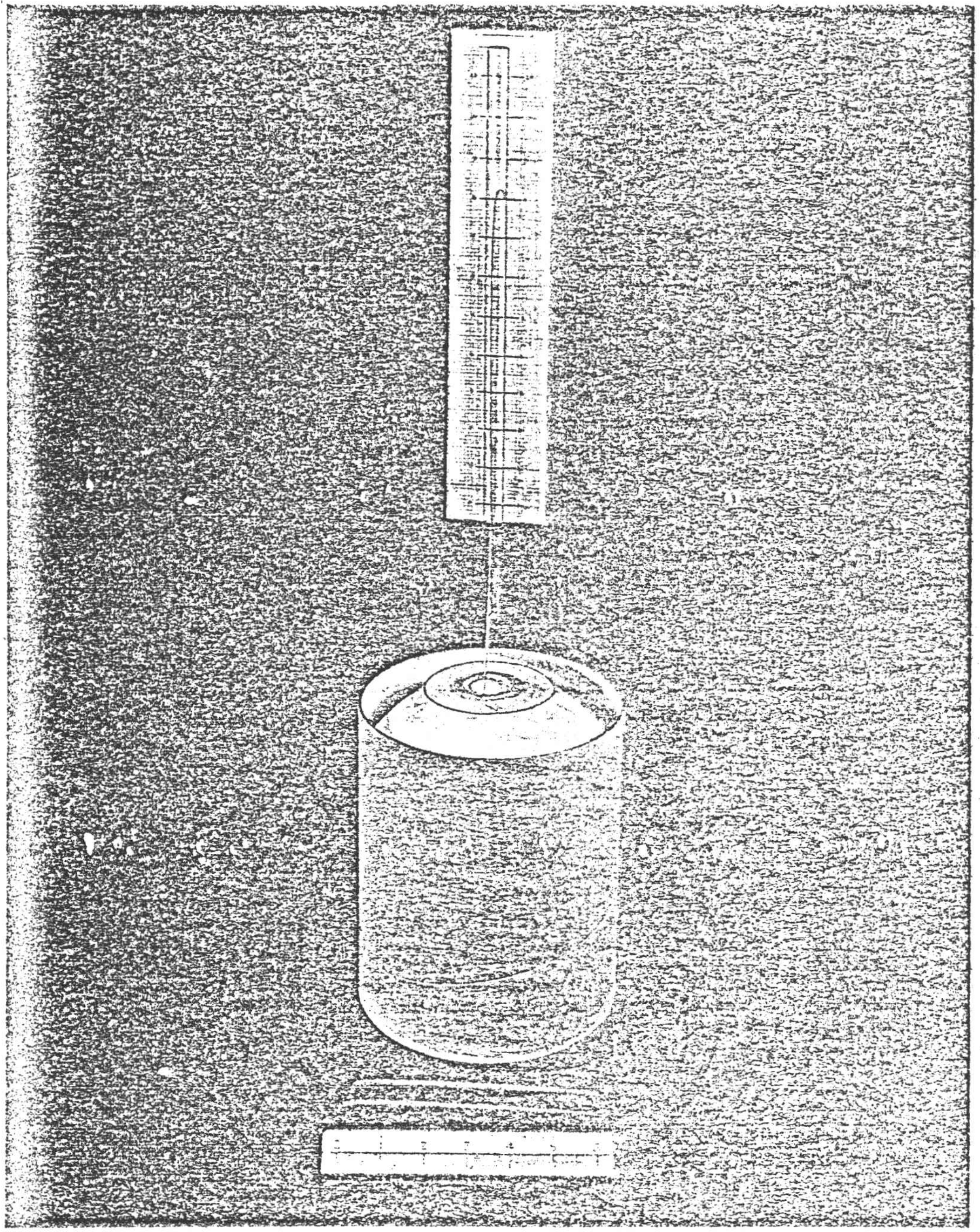

FIG. 1 Rate-of-Rise Measuring Apparatus

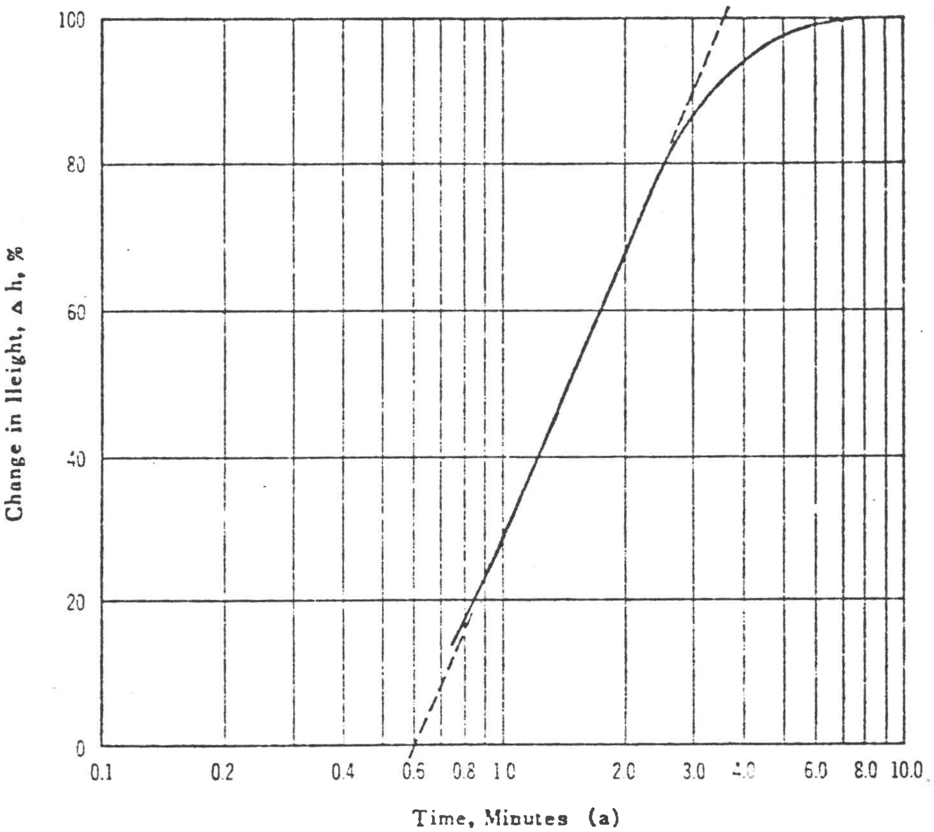

FIG. 2 Typical Rise Rate Curve.

APPENDIX

A1. SAMPLE CALCULATIONS

A1.1 *Substitution in Equation for Δh to Derive Rise Rate Curve Illustrated in Fig. 2*—Taking density, D as 2.18 lb/ft^3 and final height, h_f, as 12.8 in., the initial height becomes

$$h_o = (D/70) \times h_f = (2.18/70) \times 12.8 = 0.40 \text{ in.}$$

For time = 2.00 min, the change in height is as follows:

$$\Delta h = [(h - h_o)/(h_f - h_o)] \times 100 = [(8.83 - 0.40)/(12.8 - 0.40)] \times 100 = 68.0 \text{ percent}$$

A1.2 *Rise Rate Values*—Using an average component temperature of 70.0 F, the rise rate values from the graph are as follows:

$$\text{EIT}_{70.0\text{ F}} = 0.60 \text{ min}$$
$$\text{ERT}_{70.0\text{ F}} = 3.60 \text{ min}$$

Taking the temperature coefficients for this particular foam system to be I = 0.025 min per deg F and R = 0.10 min/deg F, the Standard Rise Rate Values become, using the equations in 9.5,

$$\text{EIT}_{73.4\text{ F}} = 0.60 + 0.025\,(70.0 - 73.4) = 0.52 \text{ min}$$
$$\text{ERT}_{73.4\text{ F}} = 3.60 + 0.10\,(70.0 - 73.4) = 3.26 \text{ min}$$

APPENDIX C

CERTIFICATES OF CALIBRATION FOR VISCOMETERS

Certificate of Calibration

Viscometer No. 3 A869

UBBELOHDE TYPE
(Standard Test ASTM D 445)

Viscometer Constant	0.947	Centistokes/Second

The viscometer constant is the same at all temperatures.

To obtain viscosity in centistokes multiply the efflux time in seconds by the viscometer constant.

CALIBRATION DATA AT 100° F.

Viscosity Standard	Viscosity Centistokes	Efflux Time Seconds	Viscometer Constant Centistokes/Second
1313	352.2	372.2	0.946
1312	213.6	225.5	0.947
		Average =	0.947

Calibrated by RJD–71038 under supervision of

R. E. Manning, Ph.D.
W. A. Lloyd, Ph.D.
Registered Professional Engineers
Cannon Instrument Co.
State College, Pennsylvania

Viscosities of the standards used in calibrating were established in Master Viscometers as described in Ind. Eng. Chem. Anal. Ed. 16, 708 (1944) by M. R. Cannon. This method has been favorably checked at the National Bureau of Standards by Swindells, Hardy and Cottington and their work is published in the Journal of Research of the National Bureau of Standards, Vol. 52, No. 3 March 1954, Research Paper 2479.

Viscosities are based on the value for water adpoted by the National Bureau of Standards and The American Society for Testing Materials July 1, 1953. The viscosity basis is 1.0038 centistokes for water at 68°F. The gravitational constant, g, is 980.1 cm/sec^2 at the Cannon Instrument Company. The gravitational constant varies up to 0.1% in the United States. To make this small correction in the viscometer constant, multiply the above viscometer constant by the factor [g (at your laboratory) /980.1]. The calibration data above are traceable to the National Bureau of Standards.

See instruction for use of the Ubbelohde Viscometer on the reverse side.

Certificate of Calibration

Viscometer No. 2B A700

UBBELOHDE TYPE
(Standard Test ASTM D 445)

Viscometer Constant 0.5147 Centistokes/Second

The viscometer constant is the same at all temperatures.

To obtain viscosity in centistokes multiply the efflux time in seconds by the viscometer constant.

CALIBRATION DATA AT 100° F.

Viscosity Standard	Viscosity Centistokes	Efflux Time Seconds	Viscometer Constant Centistokes/Second
1312	213.60	415.1	0.5146
1311	123.87	240.6	0.5148
		Average =	0.5147

Calibrated by RJD-71022 under supervision of

R. E. Manning, Ph.D.
W. A. Lloyd, Ph.D.
Registered Professional Engineers
Cannon Instrument Co.
State College, Pennsylvania

Viscosities of the standards used in calibrating were established in Master Viscometers as described in Ind. Eng. Chem. Anal. Ed. 16, 708 (1944) by M. R. Cannon. This method has been favorably checked at the National Bureau of Standards by Swindells, Hardy and Cottington and their work is published in the Journal of Research of the National Bureau of Standards, Vol. 52, No. 3 March 1954, Research Paper 2479.

Viscosities are based on the value for water adpoted by the National Bureau of Standards and The American Society for Testing Materials July 1, 1953. The viscosity basis is 1.0038 centistokes for water at 68°F. The gravitational constant, g, is 980.1 cm/sec^2 at the Cannon Instrument Company. The gravitational constant varies up to 0.1% in the United States. To make this small correction in the viscometer constant, multiply the above viscometer constant by the factor [g (at your laboratory) /980.1]. The calibration data above are traceable to the National Bureau of Standards.

See instruction for use of the Ubbelohde Viscometer on the reverse side.

Certificate of Calibration

Viscometer No. 2C B71

UBBELOHDE TYPE
(Standard Test ASTM D 445)

Viscometer Constant 0.2814 Centistokes/Second

The viscometer constant is the same at all temperatures.

To obtain viscosity in centistokes multiply the efflux time in seconds by the viscometer constant.

CALIBRATION DATA AT 100° F.

Viscosity Standard	Viscosity Centistokes	Efflux Time Seconds	Viscometer Constant Centistokes/Second
1311	123.87	440.1	0.2815
1310	65.35	232.2	0.2814
		Average =	0.2814

Calibrated by RJD-71024 under supervision of

R. E. Manning, Ph.D.
W. A. Lloyd, Ph.D.
Registered Professional Engineers
Cannon Instrument Co.
State College, Pennsylvania

Viscosities of the standards used in calibrating were established in Master Viscometers as described in Ind. Eng. Chem. Anal. Ed. 16, 703 (1944) by M. R. Cannon. This method has been favorably checked at the National Bureau of Standards by Swindells, Hardy and Cottington and their work is published in the Journal of Research of the National Bureau of Standards, Vol. 52, No. 3 March 1954, Research Paper 2479.

Viscosities are based on the value for water adpoted by the National Bureau of Standards and The American Society for Testing Materials July 1, 1953. The viscosity basis is 1.0038 centistokes for water at 68°F. The gravitational constant, g, is 980.1 cm/sec^2 at the Cannon Instrument Company. The gravitational constant varies up to 0.1% in the United States. To make this small correction in the viscometer constant, multiply the above viscometer constant by the factor [g (at your laboratory) -/980.1]. The calibration data above are traceable to the National Bureau of Standards.

See instruction for use of the Ubbelohde Viscometer on the reverse side.

APPENDIX D

SAMPLE COMPUTER PRINTOUT FOR REGRESSION ANALYSIS

```
               ****************************************************************************
               *                                                                          *
               *  REGRESSION TNT = 1        NUMBER CF VARIABLES = 6    NUMBER OF INDEPENDENTS = C  *
               *                                                                          *
               *           INCICATORSC  PRINT SIMPLE STATISTICS ..... NO                  *
               *                                                                          *
               *                        PRINT BI-VARIATE STATISTICS . NO                  *
               *                                                                          *
               *                        USE UNCCRRECTED S.S. ........ NO                  *
               *                                                                          *
               *                        CRDER CARDS FOLLOW CATA ..... YES                 *
               *                                                                          *
               *                        PRINT A MATRIX OF CCCLITTLE . NO                  *
               *                                                                          *
               *                        PRINT B MATRIX OF CCCLITTLE . NO                  *
               *                                                                          *
               *                        PRINT C MATRIX OF CCCLITTLE . NO                  *
               *                                                                          *
               *                        PRINT EXPECTED VALUES ....... YES                 *
               *                                                                          *
               ****************************************************************************
               *                                                                          *
               *                        X1 = TEMPERATURE OF CAVITY                        *
               *                                                                          *
               *                        X2 = CELL WALL THICKNESS                          *
               *                                                                          *
               *                        X3 = CORE DENSITY                                 *
               *                                                                          *
               *                        X4 = HEIGHT                                       *
               *                                                                          *
               *                        X5 = TEMP SQUARED                                 *
               *                                                                          *
               *                        X6 = TEMP CUBED                                   *
               *                                                                          *
               ****************************************************************************

 INPUT FORMAT(F2.0,3F9.4,T78,A3)
     20.00000C    C.17850000    1.2565000     56.500000    400.0CCCC     8CCC.CCCC

     30.0C000CC   C.17650000    1.2325000     57.000000    900.0000C     27CCC.CCC

     40.0000CC    C.13000000    1,3090000     57.500000    1600.CCCC     64CCC.CCC

     45.0000CC    C.16100000    1.2790000     58.800000    2025.C0CC     91125.CCC

     50.0C000C    C.11900000    1.2980000     60.500000    2500.CCCC     125CCC.CC

     55.CC00CC    C.11300000    1.2910000     61.000000    3025.C0CC     166375.CC

     60.00000C    C.13300000    1,3060000     63,000000    3600.C0CC     216CCC.CC
```

```
**************************************************************************************
*                                                                                    *
*  ORDER CARD TMT = 1        NUMBER OF VARIABLES = 4    NUMBER OF INDEPENDENTS = 1   *
*                                                                                    *
*  INDEPENDENT VARIABLESO    X 1                                                     *
*                                                                                    *
*    DEPENDENT VARIABLESO    X 2 X 3 X 4                                             *
*                                                                                    *
*             INDICATORSO    USE UNCORRECTED S.S. ........ NO                        *
*                                                                                    *
*                            PRINT A MATRIX OF DOCLITTLE . NO                        *
*                                                                                    *
*                            PRINT B MATRIX OF CCCLITTLE . NO                        *
*                                                                                    *
*                            PRINT C MATRIX OF DOCLITTLE . NO                        *
*                                                                                    *
*                            PRINT EXPECTED VALUES ....... YES                       *
*                                                                                    *
**************************************************************************************
```

```
           ANALYSIS OF VARIANCE TABLE , REGRESSION COEFFICIENTS , AND STATISTICS OF FIT FOR DEPENDENT VARIABLE X 2       TPT = 1

           SOURCE        DF        SUM OF SQUARES        MEAN SQUARE              F VALUE

           REGR           1        0.29427412D-02        0.29427412D-02        9.8454142

           DEVIATIONS     5        0.14944731D-02        0.29889461D-03

           TOTAL          6        0.44372143D-02


           R-SQUARE =              0.66319565            SIGMA =               0.17288569D-01


  SOURCE   SS FOR X(I) ADJ         SS IF X(I) LAST       F FOR HO:B(I)=0            B VALUES          STD ERROR B         STD B VALUES

    X 0                                                                        0.21174251
    X 1      0.29427412D-02        0.29427412D-02       -3.1377403            -0.15706587D-02       0.50057001D-03       -0.81436825

                         SET              EXPECTED             OBSERVED             DIFFERENCE

                           1           0.18032934           0.17850000           0.18293413D-02
                           2           0.16462275           0.17650000          -0.11877246D-01
                           3           0.14891617           0.13000000           0.18916168D-01
                           4           0.14106287           0.16100000          -0.19937126D-01
                           5           0.13320958           0.11900000           0.14209581D-01
                           6           0.12535629           0.11300000           0.12356287D-01
                           7           0.11750299           0.13300000          -0.15497006D-01

           DEVIATIONS*****             0.14944731D-02       0.14944731D-02      -0.22714036D-16
```

```
        ANALYSIS OF VARIANCE TABLE , REGRESSION COEFFICIENTS , AND STATISTICS OF FIT FOR DEPENDENT VARIABLE X 3      TRT = 1

        SOURCE        DF        SUM OF SQUARES         MEAN SQUARE          F VALUE
        REGR           1        0.26285034D-02        0.26285034D-02       6.1922170
        DEVIATIONS     5        0.21224251D-02        0.42448503D-03
        TOTAL          6        0.47509286D-02

        R-SQUARE =              0.55326099             SIGMA =             0.20603034D-01

SOURCE  SS FOR X(I) ADJ     SS IF X(I) LAST    T FOR HO:B(I)=0        B VALUES           STD ERROR B        STD B VALUES
  X C                                                                1.2180958
  X 1   0.26285034D-02      0.26285034D-02       2.4884166          0.14844311D-02      0.59653643D-03      0.74361516

                      SET           EXPECTED           OBSERVED          DIFFERENCE
                       1           1.2477844          1.2565000        -0.87155689D-02
                       2           1.2626287          1.2325000         0.30128743D-01
                       3           1.2774731          1.3090000        -0.31526946D-01
                       4           1.2848952          1.2790000         0.58952096D-02
                       5           1.2923174          1.2980000        -0.56826347D-02
                       6           1.2997395          1.2910000         0.87395210D-02
                       7           1.3071617          1.3060000         0.11616766D-02

        DEVIATIONS*****         0.21224251D-02        0.21224251D-02      -0.18393490D-15
```

```
ANALYSIS OF VARIANCE TABLE , REGRESSION COEFFICIENTS , AND STATISTICS OF FIT FOR DEPENDENT VARIABLE X 4       IPT = 1

          SOURCE        DF        SUM OF SQUARES         MEAN SQUARE            F VALUE

          REGR           1          30.354559            30.354559          36.187974

          DEVIATIONS     5          4.1940120          0.83880240

          TOTAL          6          34.548571


          R-SQUARE =               0.87860534           SIGMA =             0.91586156


  SOURCE    SS FOR X(I) ADJ      SS IF X(I) LAST      T FOR H0:B(I)=0          B VALUES          STD ERROR B        STD B VALUES

    X 0                                                                     52.349102
    X 1       30.354559           30.354559            6.0156441            0.15952096        0.26517685D-01       0.93733950

                             SET            EXPECTED          OBSERVED          DIFFERENCE

                              1            55.539521         56.500000        -0.96047904
                              2            57.134731         57.000000         0.13473054
                              3            58.729940         57.500000         1.2299401
                              4            59.527545         58.800000         0.72754491
                              5            60.325150         60.500000        -0.17485030
                              6            61.122754         61.000000         0.12275449
                              7            61.920359         63.000000        -1.0796407

          DEVIATIONS*****           4.1940120         4.1940120         0.50652815D-11
```

```
****************************************************************************
*                                                                          *
*  ORDER CARD TMT = 1       NUMBER OF VARIABLES = 5   NUMBER OF INDEPENDENTS = 2  *
*                                                                          *
*  INDEPENDENT VARIABLESO   X 1 X 5                                        *
*                                                                          *
*    DEPENDENT VARIABLESO   X 2 X 3 X 4                                    *
*                                                                          *
*            INDICATORSO    USE UNCORRECTED S.S. ........ NO               *
*                                                                          *
*                           PRINT A MATRIX OF DOOLITTLE . NO               *
*                                                                          *
*                           PRINT B MATRIX OF DOOLITTLE . NO               *
*                                                                          *
*                           PRINT C MATRIX OF DOOLITTLE . NO               *
*                                                                          *
*                           PRINT EXPECTED VALUES ....... YES              *
*                                                                          *
****************************************************************************
```

```
            ANALYSIS OF VARIANCE TABLE , REGRESSION COEFFICIENTS , ANC STATISTICS OF FIT FOR CEPENCENT VARIABLE X 2      TPT = 1

            SOLRCE       DF        SUM OF SCUARES         MEAN SQUARE             F VALLE

            RECR          2        0.29946C58D-C2        0.14973029D-02        4.1516540

            DEVIATICNS    4        0.14426085D-02        0.36065213D-03

            TOTAL         6        0.44372143D-02


            R-SQUARE =             0.67488419              SIGMA =             0.18990843D-C1


   SOURCE   SS FOR X(I) ADJ       SS IF X(I) LAST     T FCR H00B(I)=0            B VALLES          STD ERRCR B          STC B VALLES

     X C                                                                0.23667C1C
     X 1     0.29427412D-02       0.22771725D-03       -0.79460942      -0.29751C36D-C2       0.37441C81C-C2       -1.5425566
     X 5     0.51864519D-04       0.51864519D-04        0.37921973       0.17568711C-C4       C.46328579C-04        0.73617035

                         SET             EXPECTED              OBSERVED              DIFFERENCE

                           1           0.18419551            0.17850000            0.56955C98C-C2
                           2           0.16322883            0.17650C00           -C.1327117CC-C1
                           3           0.14577589            0.13000000            0.15775892C-C1
                           4           0.13836708            0.16100000           -0.22632924C-C1
                           5           0.13183670            0.11900000            0.12936697C-C1
                           6           0.12618475            0.11300000            0.13184752C-C1
                           7           0.12141124            0.13300000           -0.11588757C-C1

            DEVIATICNS*****            0.14426085D-02        0.14426085D-02       -0.38597597C-16
```

```
          ANALYSIS OF VARIANCE TABLE , REGRESSION COEFFICIENTS , AND STATISTICS OF FIT FOR DEPENDENT VARIABLE X 3       IPT = 1

          SOURCE       DF       SUM OF SQUARES        MEAN SQUARE           F VALUE

          REGR          2       0.26425593D-02       0.13212797D-02      2.5067330

          DEVIATIONS    4       0.21083692D-02       0.52709231D-03

          TOTAL         6       0.47509286D-02


          R-SQUARE =            0.55621955              SIGMA =          0.22958491D-01


  SOURCE     SS FOR X(I) ADJ    SS IF X(I) LAST    T FOR H0:B(I)=0          B VALUES        STD ERROR B       STD B VALUES

    X C                                                                   1.2051188
    X 1     0.26285034D-02     0.12628802D-03       0.48940314        0.22155586D-02      0.45263431D-02      1.1101718
    X 5     0.14055913D-04     0.14055913D-04      -0.16330000       -0.91460644D-05      0.56007743D-04     -0.37037240

                        SET          EXPECTED           OBSERVED           DIFFERENCE

                          1         1.2457717          1.2565000        -0.10728251D-01
                          2         1.2633544          1.2325000         0.30854403D-01
                          3         1.2791078          1.3090000        -0.29892156D-01
                          4         1.2862986          1.2790000         0.72986102D-02
                          5         1.2930321          1.2980000        -0.49679273D-02
                          6         1.2993082          1.2910000         0.83082320D-02
                          7         1.3051271          1.3060000        -0.87291193D-03

          DEVIATIONS*****   0.21083692D-02     0.21083692D-02     -0.10358285D-15
```

```
          ANALYSIS OF VARIANCE TABLE , REGRESSION COEFFICIENTS , AND STATISTICS OF FIT FOR DEPENDENT VARIABLE X 4       IPT = 1

          SOURCE        DF         SUM OF SQUARES        MEAN SQUARE              F VALUE
          REGR           2          33.922138          16.961069          108.30248
          DEVIATIONS     4         0.62643325         0.15560831
          TOTAL          6          34.548571

          R-SQUARE =               0.98186804               SIGMA =           0.39573768

  SOURCE      SS FOR X(I) ADJ       SS IF X(I) LAST      T FOR HO:B(I)=0              B VALUES          STD ERROR B          STD B VALUES

    X 0                                                                         58.886904
    X 1          30.354559             1.1219103           -2.6765272          -0.20882534         0.78021003D-01         -1.2270503
    X 5          3.5675787             3.5675787            4.7728675           0.46077776D-02     0.96541076D-03          2.1881147

                              SET            EXPECTED              OBSERVED               DIFFERENCE

                               1            56.553508             56.500000            0.53507551D-01
                               2            56.769143             57.000000           -0.23085661
                               3            57.906334             57.500000            0.40633436
                               4            58.820513             58.800000            0.20513163D-01
                               5            59.965081             60.500000           -0.53491915
                               6            61.340037             61.000000            0.34003742
                               7            62.945383             63.000000           -0.54617132D-01

          DEVIATIONS*****          0.62643325            0.62643325             0.51823545D-11
```

```
**************************************************************
*                                                            *
*  ORDER CARD TMT = 1    NUMBER OF VARIABLES = 6    NUMBER OF INDEPENDENTS = 3  *
*                                                            *
*  INDEPENDENT VARIABLESC   X 1 X 5 X 6                      *
*                                                            *
*    DEPENDENT VARIABLESO   X 2 X 3 X 4                      *
*                                                            *
*             INDICATORSO   USE UNCORRECTED S.S. ........ NO *
*                                                            *
*                           PRINT A MATRIX OF DOOLITTLE . NO *
*                                                            *
*                           PRINT B MATRIX OF DOOLITTLE . NO *
*                                                            *
*                           PRINT C MATRIX OF DOOLITTLE . NO *
*                                                            *
*                           PRINT EXPECTED VALUES ....... YES*
*                                                            *
**************************************************************
```

```
          ANALYSIS OF VARIANCE TABLE , REGRESSION COEFFICIENTS , AND STATISTICS OF FIT FOR DEPENDENT VARIABLE X 2       TMT = 1

          SOURCE        DF        SUM OF SQUARES         MEAN SQUARE              F VALUE

          REGR           3        0.32761546D-02       0.10920515D-02       2.8216936

          DEVIATIONS     3        0.11610597D-02       0.38701989D-03

          TOTAL          6        0.44372143D-02

          R-SQUARE =              0.73833590             SIGMA =            0.19672821D-01

  SOURCE     SS FOR X(I) ADJ        SS IF X(I) LAST       T FOR H0:B(I)=0           B VALUES            STD ERROR B          STD B VALUES

    X 0                                                                        0.28369642D-01
    X 1       0.29427412D-02        0.19071504D-03        0.70198176           0.15223213D-01       0.21686052D-01          7.8930588
    X 5       0.51864519D-04        0.25957791D-03       -0.81096854          -0.46549557D-03       0.56839249D-03        -19.505360
    X 6       0.28154886D-03        0.28154886D-03        0.85292392           0.39937797D-05       0.46824577D-05         10.558770

                            SET            EXPECTED             OBSERVED           DIFFERENCE

                             1        0.17858591           0.17850000           0.85911682D-04
                             2        0.17395207           0.17650000          -0.25479278D-02
                             3        0.14810715           0.13000000           0.18107154D-01
                             4        0.13471888           0.16100000          -0.26281124D-01
                             5        0.12501383           0.11900000           0.60138335D-02
                             6        0.12198736           0.11340000           0.85873606D-02
                             7        0.12863479           0.13300000          -0.43652085D-02

          DEVIATIONS*****     0.11610597D-02       0.11610597D-02       0.60932162D-16
```

```
            ANALYSIS OF VARIANCE TABLE , REGRESSION COEFFICIENTS , AND STATISTICS OF FIT FOR DEPENDENT VARIABLE X 3       TMT = 1

            SOURCE      DF        SUM OF SQUARES        MEAN SQUARE          F VALUE

            REGR         3        0.29360224D-02       0.97867415D-03      1.6177269

            DEVIATIONS   3        0.18149061D-02       0.60496871D-03

            TOTAL        6        0.47509286D-02

            R-SQUARE =            0.61798918           SIGMA =            0.24596112D-01


  SOURCE    SS FOR X(I) ADJ     SS IF X(I) LAST     T FOR H0:B(I)=0          B VALUES          STD ERROR B        STD B VALUES

    X 0                                                                   1.4177805
    X 1     0.26285034D-02      0.22036414D-03      -0.60353715       -0.16363806D-01      0.27113171D-01      -8.1995361
    X 5     0.14055913D-04      0.28066412D-03       0.68112527        0.48403318D-03      0.71063754D-03      19.001057
    X 6     0.29346311D-03      0.29346311D-03      -0.69648265       -0.40774061D-05      0.58542825D-05     -10.852004

                          SET          EXPECTED          OBSERVED          DIFFERENCE

                           1          1.2514988         1.2565000       -0.50011924D-02
                           2          1.2524066         1.2325000        0.19906625D-01
                           3          1.2767278         1.3090000       -0.32272232D-01
                           4          1.2900232         1.2790000        0.11023201D-01
                           5          1.2999978         1.2980000        0.19978005D-02
                           6          1.3035935         1.2910000        0.12593513D-01
                           7          1.2977523         1.3060000       -0.82477153D-02

            DEVIATIONS*****       0.18149061D-02    0.18149061D-02    -0.29437173D-14
```

```
         ANALYSIS OF VARIANCE TABLE , REGRESSION COEFFICIENTS , AND STATISTICS OF FIT FOR DEPENDENT VARIABLE X 4       TMT = 1

         SOURCE        DF        SUM OF SQUARES          MEAN SQUARE             F VALUE

         REGR           3          33.922158              11.307386            54.153018

         DEVIATIONS     3        0.62641307             0.20880436

         TOTAL          6          34.548571


         R-SQUARE =              0.98186863               SIGMA =             0.45695115


SOURCE   SS FOR X(I) ADJ       SS IF X(I) LAST      T FOR H0:B(I)=0            B VALUES           STD ERROR B         STD B VALUES

  X 0                                                                         58.831135
  X 1      30.354559           0.34232168D-01       -0.40489967             -0.20395345          0.50371354          -1.1984232
  X 5      3.5675787           0.24026652D-01        0.33921641              0.44784560D-02      0.13202357D-01       2.1267032
  X 6    0.20178371D-04        0.20178371D-04        0.98304473D-02          0.10691786D-05      0.10876195D-03       0.33369556D-01

                      SET            EXPECTED            OBSERVED            DIFFERENCE

                       1            56.552006           56.500000           0.52006200D-01
                       2            56.772014           57.000000          -0.22798588
                       3            57.906958           57.500000           0.40695846
                       4            58.819536           58.800000           0.19536500D-01
                       5            59.963254           60.500000          -0.53674571
                       6            61.338914           61.000000           0.33891373
                       7            62.947317           63.000000          -0.52683309D-01

         DEVIATIONS*****         0.62641307          0.62641307           0.51897930D-11
```

```
*************************************************************************************
*                                                                                   *
*   ORDER CARD TPT = 1         NUMBER OF VARIABLES = 5    NUMBER OF INDEPENDENTS = 2 *
*                                                                                   *
*   INDEPENDENT VARIABLESO    X 1 X 6                                               *
*                                                                                   *
*     DEPENDENT VARIABLESO    X 2 X 3 X 4                                           *
*                                                                                   *
*              INDICATORSO    USE UNCORRECTED S.S. ........ NO                      *
*                                                                                   *
*                             PRINT A MATRIX OF DOOLITTLE . NO                      *
*                                                                                   *
*                             PRINT B MATRIX OF DOOLITTLE . NO                      *
*                                                                                   *
*                             PRINT C MATRIX OF DOOLITTLE . NO                      *
*                                                                                   *
*                             PRINT EXPECTED VALUES ....... YES                     *
*                                                                                   *
*************************************************************************************
```

```
            ANALYSIS OF VARIANCE TABLE , REGRESSION COEFFICIENTS , AND STATISTICS OF FIT FOR DEPENDENT VARIABLE X 2       TPT = 1

            SOURCE        DF        SUM OF SQUARES          MEAN SQUARE              F VALUE

            REGR           2        0.30165767D-02          0.15082884D-02        4.2467928

            DEVIATIONS     4        0.14206376D-02          0.35515940D-03

            TOTAL          6        0.44372143D-02


            R-SQUARE =              0.67983571               SIGMA =              0.18845673D-01


   SOURCE   SS FOR X(I) ADJ       SS IF X(I) LAST      T FOR H0:B(I)=0           B VALUES             STD ERROR B          STD B VALUES

     X 0                                                                      0.23237204
     X 1     0.29427412D-02       0.52829286D-03       -1.2196233            -0.24535144D-02        0.20116985D-02        -1.2721187
     X 6     0.73835472D-04       0.73835472D-04        0.45595384            0.17268825D-06        0.37874064D-06         0.47557913

                           SET            EXPECTED              OBSERVED             DIFFERENCE

                            1          0.18468326            0.17850000            0.61832567D-02
                            2          0.16342919            0.17650000           -0.13070811D-01
                            3          0.14528351            0.13000000            0.15283510D-01
                            4          0.13770011            0.16100000           -0.23299890D-01
                            5          0.13128235            0.11900000            0.12282345D-01
                            6          0.12615975            0.11300000            0.13159753D-01
                            7          0.12246164            0.13300000           -0.10538165D-01

            DEVIATIONS*****            0.14206376D-02        0.14206376D-02       -0.35073940D-16
```

```
            ANALYSIS OF VARIANCE TABLE , REGRESSION COEFFICIENTS , AND STATISTICS OF FIT FOR DEPENDENT VARIABLE Y 3       IPT = 1

            SOURCE        DF        SUM OF SQUARES        MEAN SQUARE           F VALUE

            REGR           2        0.26553583D-02        0.13276792D-02       2.5342585

            DEVIATIONS     4        0.20955703D-02        0.52389256D-03

            TOTAL          6        0.47509286D-02

            R-SQUARE =              0.55891354            SIGMA =              0.22888699D-01


   SOURCE   SS FOR X(I) ADJ    SS IF X(I) LAST    T FOR H0B(I)=0         B VALUES           STD ERROR B         STD B VALUES

     X 0                                                                1.2056544
     X 1    0.26285034D-02     0.35698742D-03     0.42547769          0.20168691D-02     0.24432751D-02       1.0106079
     X 6    0.26854900D-04     0.26854900D-04    -0.22640742         -0.10414586D-06     0.45995316D-06      -0.27718389

                          SET         EXPECTED          OBSERVED          DIFFERENCE

                            1        1.2451586         1.2565000        -0.11341354D-01
                            2        1.2633486         1.2325000         0.30848565D-01
                            3        1.2796639         1.3090000        -0.29336141D-01
                            4        1.2869232         1.2790000         0.79232476D-02
                            5        1.2934797         1.2980000        -0.45203487D-02
                            6        1.2992550         1.2910000         0.82549621D-02
                            7        1.3041711         1.3060000        -0.18289311D-02

            DEVIATIONS*****     0.20955703D-02    0.20955703D-02    -0.14024155D-15
```

```
          ANALYSIS OF VARIANCE TABLE , REGRESSION COEFFICIENTS , AND STATISTICS OF FIT FOR DEPENDENT VARIABLE X 4       IPT = 1

          SOURCE        DF         SUM OF SQUARES          MEAN SQUARE              F VALUE

          REGR           2          33.898132            16.949066           104.23143

          DEVIATIONS     4          0.65043972           0.16260993

          TOTAL          6          34.548571


          R-SQUARE =                0.98117318               SIGMA =            0.40324922


   SOURCE     SS FOR X(I) ADJ      SS IF X(I) LAST      T FOR H0:B(I)=0             B VALUES            STD ERROR B          STD B VALUES

    X 0                                                                      56.860466
    X 1        30.354559           0.10078689          -0.78727867        -0.33888571D-01       0.43045205D-01       -0.19912805
    X 6        3.5435723           3.5435723            4.6681748          0.37831265D-04       0.81040813D-05        1.1807313

                         SET            EXPECTED            OBSERVED            DIFFERENCE

                          1            56.493345           56.500000         -0.66553481D-02
                          2            56.873253           57.000000         -0.12674696
                          3            57.934124           57.500000          0.43412427
                          4            58.790855           58.800000         -0.91454240D-02
                          5            59.902346           60.500000         -0.59765405
                          6            61.258772           61.000000          0.25877183
                          7            63.006706           63.000000          0.67056825D-02

          DEVIATIONS*****          0.65043972          0.65043972          0.51683241D-11
```

APPENDIX E

COMPUTER PRINTOUT FOR ANALYSIS OF VARIANCE

```
A GENERALIZED ANALYSIS PROGRAM DEVELOPED BY THE BRIGHAM YOUNG UNIVERSITY STATISTICS DEPARTMENT
CAPABLE OF ANALYZING UNBALANCED  (AND BALANCED) UNIVARIATE AND MULTIVARIATE ANALYSIS OF VARIANCE PROBLEMS
AS WELL AS UNIVARIATE AND MULTIVARIATE REGRESSION PROBLEMS(FEB, 1972) 12 AUG 1976 11 HR 36 MIN 12.46 SEC AM


      BRUCE PUGSLEY

NUMBER OF VARIABLES READ IN            3

SAMPLE SIZE           0

NUMBER OF NEW VARIABLES ADDED            0

OUTPUT OPTION AS DATA IS READ IN      0

INPUT OPTION      0

DATA ON ANOTHER INPUT DEVICE      0

ZERO SET TO  0.100E-05

THE EXPERIMENTAL DESIGN MODEL IS


Y(IJ)=T(I)+B(J)+E



THE MAIN EFFECT T IS FIXED  WITH SUBSCRIPT I = 1, 4      TEMP 1=60,2=70,3=80,4=90       COL 3
THE MAIN EFFECT B IS FIXED  WITH SUBSCRIPT J = 1, 7      BLOCK 1=20,2=30,3=40,4=45,5=50,6=55,7=60


YOUR FORMAT (I3,2I1,3F3.4)
THE NUMBER OF DATA POINTS IS GREATER THAN THE NUMBER GIVEN
THE NUMBER COUNTED WAS           28
PROCEEDING WITH NEW COUNT




THE ORDER OF THE TERMS IN THE MODEL IS
   1   2

THE ORDER OF THE DEPENDENT VARIABLES IS
   1   2   3
```

```
THE DEPENDENT VARIABLE FOR THIS TABLE IS       1


         ANALYSIS OF VARIANCE TABLE

SOURCE           DF  SUM OF SQUARES  MEAN SQUARES   EXPECTED MEAN SQUARE COEFFICIENTS
                                                    T        B
 MEAN             1    0.62611
 T                3    1.944320D-03    6.48107E-04   7.000    0.0
 B                6    9.93721D-03     1.65654E-03   0.0      4.000

 ERROR           18    8.35128D-03     4.63960E-04

 TOTAL           28    0.64634


 REGRESSION      10    0.63799         6.37990D-02

 R-SQUARED             0.58729
```

```
THE DEPENDENT VARIABLE FOR THIS TABLE IS       2

        ANALYSIS OF VARIANCE TABLE

SOURCE           DF  SUM OF SQUARES  MEAN SQUARES   EXPECTED MEAN SQUARE COEFFICIENTS
                                                     T        B
 MEAN             1     45.419
 T                3    1.640750D-03    5.46917E-04   7.000    0.0
 B                6    5.949680D-03    9.91613E-04   0.0      4.000

 ERROR           18    1.066980D-02    5.92764E-04

 TOTAL           28     45.436

 REGRESSION      10     45.426          4.5426

 R-SQUARED             0.41580
```

```
THE DEPENDENT VARIABLE FOR THIS TABLE IS       3

        ANALYSIS OF VARIANCE TABLE

SOURCE            DF  SUM OF SQUARES  MEAN SQUARES   EXPECTED MEAN SQUARE COEFFICIENTS
                                                    T        B
 MEAN              1     97374.
 T                 3     2.6694        0.88982       7.000    0.0
 B                 6     99.182         16.530       0.0      4.000

 ERROR            18     35.906         1.9948

 TOTAL            28     97511.


 REGRESSION       10     97475.         9747.5

 R-SQUARED              0.73939
```

```
THE DEPENDENT VARIABLE FOR THIS TABLE IS        1

**************************************************************************
*                                                                        *
* THE FOLLOWING TABLE IS ONLY VALID FOR FIXED MODELS WITH NO MISSING CELLS *
*                                                                        *
**************************************************************************


                              ORIGINAL
 SOURCE            VARIABLE  VARIABLE   SUM OF SQUARES   PREDICTION          F-RATIO
                      NO.       NO.      DUE TO BETAS    COEFFICIENTS
                       1         1       0.62611         0.14954              1349.5

T                      2         1       2.43440D-04     1.70238D-03          0.52472

T                      3         2       1.17872D-03     5.29762D-03          2.5407

T                      4         3       5.22161D-04     6.10714D-03          1.1255

B                      5         1       4.01704D-03    -4.38988D-03          8.6585

B                      6         2       3.96175D-03    -5.74583D-03          8.5354

B                      7         3       7.62612D-04    -3.08750D-03          1.6438

B                      8         4       7.60021D-04    -3.97917D-03          1.6382

B                      9         5       2.66667D-04    -3.33333D-03          0.57479

B                     10         6       1.71125D-04     4.62500D-03          0.36885
```

```
THE DEPENDENT VARIABLE FOR THIS TABLE IS       2

**************************************************************************
*                                                                        *
* THE FOLLOWING TABLE IS ONLY VALID FOR FIXED MODELS WITH NO MISSING CELLS *
*                                                                        *
**************************************************************************


                                ORIGINAL
 SOURCE              VARIABLE  VARIABLE   SUM OF SQUARES   PREDICTION          F-RATIO
                        NO.       NO.     EQL TO BETAS    COEFFICIENTS
                         1         1        45.410          1.2736              76621.

T                        2         1       6.13440D-04    -2.70238D-03          1.0349

T                        3         2       1.02515D-03    -4.94048D-03          1.7294

T                        4         3       2.16071D-06     3.92857D-04        3.64515E-03

B                        5         1       7.31251D-04    -2.08631D-03          1.2336

B                        6         2       3.53710D-03     5.42917D-03          5.9671

B                        7         3       6.10512D-04    -2.76250D-03          1.0299

B                        8         4       7.13021D-04    -3.85417D-03          1.2029

B                        9         5       2.66667D-04    -3.33 33D-03        0.44987

B                       10         6       9.11250D-05     3.37500D-03        0.15373
```

```
THE DEPENDENT VARIABLE FOR THIS TABLE IS       3

*****************************************************************************
*                                                                           *
* THE FOLLOWING TABLE IS ONLY VALID FOR FIXED MODELS WITH NO MISSING CELLS *
*                                                                           *
*****************************************************************************

                                 ORIGINAL
 SOURCE              VARIABLE  VARIABLE   SUM OF SQUARES   PREDICTION          F-RATIO
                        NO.        NO.      DUE TO BETAS    COEFFICIENTS
                        1          1        97374.          58.371              48814.

T                       2          1       0.42857        -7.14286D-02        0.21384

T                       3          2        2.1943          0.22857             1.1000

T                       4          3       4.57143D-02     5.71429D-02        2.29167E-02

B                       5          1        57.869          0.58690            29.010

B                       6          2        16.144          0.36667             8.0877

B                       7          3        17.113          0.46250             8.5785

B                       8          4        7.2075          0.38750             3.6131

B                       9          5       0.73500          0.17500            0.36846

B                      10          6       0.12500         -0.12500           6.26628E-02
```

```
                    TABLE OF ESTIMATED MEANS AND VARIANCES

************************************************************************************
*                                                                                  *
* THE VARIANCES OF THE FOLLOWING MEANS ARE VALID ONLY FOR UNIVARIATE FIXED MODELS *
*                                                                                  *
************************************************************************************

                                                                    I     J

                  NUM            EST.             VARIANCE
                                 MEAN             OF MEAN

I
                   7                                                1   ***
      1                        0.14443           0.66277E-04
      2                         1.2817           0.84681E-04
      3                         59.193           0.28497
                   7                                                2   ***
      1                        0.14064           0.66277E-04
      2                         1.2605           0.84681E-04
      3                         55.443           0.28497
                   7                                                3   ***
      1                        0.15043           0.66277E-04
      2                         1.2655           0.84680E-04
      3                         57.071           0.28497
                   7                                                4   ***
      1                        0.16204           0.66277E-04
      2                         1.2504           0.84680E-04
      3                         59.185           0.28497

B
                   4                                              ***     1
      1                        0.17687           0.11599E-03
      2                         1.2601           0.14819E-03
      3                         55.450           0.49870
                   4                                              ***     2
      1                        0.17337           0.11599E-03
      2                         1.2444           0.14817E-03
      3                         57.725           0.49870
                   4                                              ***     3
      1                        0.15125           0.11599E-03
      2                         1.2820           0.14819E-03
      3                         58.075           0.49870
                   4                                              ***     4
      1                        0.14775           0.11599E-03
      2                         1.2657           0.14817E-03
      3                         57.225           0.49870
                   4                                              ***     5
      1                        0.13856           0.11599E-03
      2                         1.2770           0.14819E-03
      3                         59.425           0.49870
                   4                                              ***     6
      1                        0.12387           0.11599E-03
      2                         1.2635           0.14819E-03
      3                         61.075           0.49870
                   4                                              ***     7
      1                        0.13312           0.11599E-03
      2                         1.2704           0.14819E-03
      3                         60.825           0.49870
```

APPENDIX F

LETTERS

Whirlpool
CORPORATION

Evansville Division

EVANSVILLE, INDIANA 47727 • AREA CODE 812 424-7741

February 27, 1976

Mr. Bruce Pugsley
785 North 100 West
Orem, Utah 84507

Dear Bruce,

For your use in your research project, the formulation and ratio for 17 foot thickwall cabinets are:

	Formulation
Resin 6416	67.727%
Silicone 5340	.756%
Water	.481%
Polycat 8	.463%
T-12	.072%
Freon R-11	30.501%
	100.000%

	Ratio
Masterbatch =	140 Parts
TDI	100 Parts

The masterbatch shipped to use is in a special pressure drum that is used for masterbatch. Therefore, the masterbatch should not require reformulation.

If I can assist you further Bruce, please call.

RN:dlh

cc: K. Leimgruber
J. Schaus
K. Werling

EL-1000 REV 10-71

E. I. DU PONT DE NEMOURS & COMPANY
INCORPORATED
WILMINGTON, DELAWARE 19898

ELASTOMER CHEMICALS DEPARTMENT
ELASTOMERS LABORATORY
CHESTNUT RUN

302-774-1000

February 3, 1976

Mr. Bruce A. Pugsley
Brigham Young University
785 N. 100W
Orem, Utah 84057

Dear Mr. Pugsley:

In your recent letter you requested information on the rigid urethane foaming process, particularly sensors which could be coupled with a computer to control the process variables, and equipment used for measuring viscosity of the isocyanate.

The chemistry and manufacture of rigid urethane foam is rather complex as you will see by reading the enclosed literature. We don't know of any particular sensors that can be coupled to computers. Thermocouples can be positioned in the foam and the temperature profile recorded on standard multipoint recorders, but I don't know if this is what you want to do. The viscosity of the isocyanate (LD-3071 or HYLENE TRF) is measured with a Brookfield Viscometer, Model LVF (Brookfield Engineering Labs, Inc., 240 Cushing Street, Stoughton, Massachusetts 02072). In order to study variables in the foam system, ASTM D2237, Rate of Rise Properties of Urethane Foaming Systems, is often used (see enclosure). Other methods, such as the Urethane Foam Flow Test (enclosed), are often employed to study the effect of various changes in formulation on the flow properties of foaming systems.

One of the most integrated systems for controlling the variables in the urethane foaming system is made by Fluidyne Instrumentation, 1631 San Pablo Avenue, Oakland, California 94612. Perhaps they could be of assistance to you in your research project. I have included some additional information on "Du Pont Synthetic Rubbers" which may be of interest to you.

Very truly yours,

RFH:bsa
Encls.

BETTER THINGS FOR BETTER LIVING . . . THROUGH CHEMISTRY

LIST OF REFERENCES

LIST OF REFERENCES

1. Lane, Roger I. 1974. *Rigid urethane foam processing handbook*, pp. 1-18, 32-47. Westport, Conn: Technomic.

2. Dombrow, Bernard A. 1965. *Polyurethanes*, Second Ed. pp. 47-93. New York: Reinhold Publishing Corporation.

3. Benning, Calvin J. 1969. *Plastic Foams: The physics and chemistry of product performance and process technology, vol. 1*, p. 534. New York: Wiley

4. Buist, J. M., Hurd, R., Stafford, R. L. 1968. "Rigid foam: manufacture and properties" *Advances in polyurethane technology*, Ed. by Buist, J. M., Gudgeon, H. pp. 190-215. London:MaClaren

5. Donald S. Gilmore Laboratories, 1974. "Cryogenic News" *Cryogenics*, 14:227, April 1974.

6. Kaplan, M. "Chemical and mechanical factors affecting one-shot rigid urethane foam" 1969 *Polyurethane technology*, Ed. by Paul F. Bruins. pp. 85-92. New York: Wiley

7. Johnson, Vincent, 1975. "Specifying Urethane foam machinery" *Plastics Technology*. 21:104-107.

8. Doyle, E. N. 1971. *The development and use of polyurethane products*, pp. 266-272. New York: McGraw-Hill.

9. Backas, J. K., Gemeinhardt, P. G. 1973. "Rigid urethane foams" *Plastic Foams*, part II, Ed. by Frisch, Kurt C. and Saunders, James H. New York: Marcell Dekker, Inc.

10. Mobay Equipment Report. Mobay Chemical Corporation, Pittsburg, Pa.

11. Knox, R. E. 1969 "New Trends in equipment and application techniques for polyurethane foam manufacture" *Polyurethane Technology*, Ed. by Bruins, Paul F. New York: Interscience

12. Kaplan, Melvin Dr. 1967. "One-shot rigid urethane foam molding reduces cost, improves properties", Plastics technology. 10:38-40.

13. Shaus, John, Personal Interview.

14. Hart, Dave. "Polyurethane foam insulation in household refrigerators design considerations, techniques and application", Unpublished, Presented to University of Evansville, May, 1973.

15. Baumann, G.F. 1969, "Polymetric polyisocyanes in urethane foams", Polyurethane Technology. Ed. by Bruins Paul F. pp. 95-116. New York: Interscience.

16. Benning, Calvin J. 1969. "Physics and chemistry of foam formulation and stability", Plastic foams; the physics and chemistry of product performance and process technology, Vol. II, pp. 1-9. New York: Wiley.

17. Lyon, C.K. Garrett, V.H. and Frankel, E.N. "Rigid urethane foams from hydroxymethylated castor oil, safflower oil, oleic safflower, and polyol esters of castor acids", American Oil Chemists Society Journal, 51:331-334. July-Dec. 1974.

18. Ferrigno, T.H. 1967. Rigid plastic foams. pp. 1-200. New York: Reinhold.

19. Frisch. Kurt C., Sanders, James H. 1973. Plastic Foams, Vol. 1, 2. New York: Marcel Dekker.

20. Hobbs, S.Y. "The application structure-property studies to structural foams", Polymer Engineering and Science, 15:854-861, Dec. 1975.

21. Boudreau, Robert J. 1967. "How silicone surfactants affect polyurethane foams", Plastics Engineering. 44:133-135, 138, 143-147, 234, 239, 240, Jan. 1967.

22. Strickman, Samuel W., "Neucleation-the control of the entrapment of gas in the production of elastomeric or plastic foams..can be carried out so as to permit production of better and more uniform end products", Rubber Age, 106:58-60. Jan-June, 1974.

23. Letter from DuPont, Feb. 3, 1976.

24. Kaplan, 1967, "Formulation and processing techniques", Rigid plastics foams, Second Ed., Ferringo, T. H., pp 94-106. New York: Reinhold.

25. Streeter, Victor L. and Wylie, Benjamin E. 1975. Fluid Mechanics, p. 488. New York: McGraw-Hill.

SENSITIVITY OF PROCESS VARIABLES ON RIGID POLYURETHANE FOAMING

Bruce A. Pugsley

Department of Mechnical Engineering

M. S. Degree, December 1976

ABSTRACT

The scope of this research was to determine the sensitivity of the process to pre-mixed viscosities, temperature, mix ratio, and mixing time or blendability of the foam ingredients; and temperature dimensions and surface finish of the mold on the rigid polyurethane foam process. The overall goal was to decrease the percentage overpack needed while maintaining the correct "k" factor or thermal properties of the foam. Both experimental and literature review were utilized to accomplish this goal.

Findings showed that polyurethane foam was not very sensitive to cavity wall temperatures in the range between 40°C to 55°C.

Ingredient temperature affected the reaction rates and viscosities; but had little effect on final densities.

COMMITTEE APPROVAL: