



Sorption of water vapor from an ethanol-water mixture using barley as the sorbent  
by Glenn Kenneth Graham

A thesis submitted in partial fulfillment of the requirements for the degree of Master of Science in  
Chemical Engineering  
Montana State University  
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**Abstract:**

Experiments were performed to study the use of Shabet barley grain as a sorbent for drying ethanol-water vapor mixtures.

A central composite design was used to set up experiments to study the effects of initial grain temperatures (86-100°C), feed mass flux (0.025-0.250 gm./cm. -min.), and feed composition (73-87 wt. % ethanol in water). Effects of initial grain moisture content (0-2.3 wt. % water), grain particle size (0.208-2.363 mm), and grain regeneration (one desiccation as compared with two) were also studied. One experiment was performed using barley straw. Ethanol-water vapors were pumped through a fixed bed containing ground grain.

Under the conditions used, the amount of water sorbed and amount of ethanol concentrated were inversely related to the initial grain temperature, feed mass flux, initial grain moisture content, and grain particle size (for the larger size range tested). The amount of water sorbed was also inversely related to the feed composition. Over the range of 73-87 wt. % ethanol, the amount of ethanol concentrated was not affected by the feed composition. A maximum of 0.0999 gm. water was sorbed per gm. grain for a combined effluent sample of 99 wt. % ethanol. A maximum of 0.326 ml. ethanol was concentrated per gm. grain for a combined sample of 99 wt. % ethanol. Grain regeneration had little effect on its performance. For grain desiccated once, 0.138 gm. ethanol was concentrated per gm. grain for a combined sample of 99 wt. % ethanol.

Grain desiccated twice had a corresponding value of 0.143. Grain yielded better results than straw. Grain concentrated 0.318 ml. of ethanol per gm. of grain for a combined sample of 99 wt. % ethanol. The corresponding value for straw was 0.182.

The results indicate that adsorption occurs, as well as absorption. About 5-20% of the feed ethanol was sorbed by the grain.

A mathematical model was developed that expresses the amount of ethanol concentrated as a function of feed mass flux and initial grain temperature.

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APPROVAL

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Glenn Kenneth Graham

This thesis has been read by each member of the thesis committee and has been found to be satisfactory regarding content, English usage, format, citations, bibliographic style, and consistency, and is ready for submission to the College of Graduate Studies.

September 7, 1983  
Date

Harvey P. Searnal  
Chairperson, Graduate Committee

Approved for the Major Department

Sept. 7, 1983  
Date

John T. Sears  
Head, Major Department

Approved for the College of Graduate Studies

9-9-83  
Date

Michael D. Mabe  
Graduate Dean

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## ABSTRACT

Experiments were performed to study the use of Shabet barley grain as a sorbent for drying ethanol-water vapor mixtures.

A central composite design was used to set up experiments to study the effects of initial grain temperatures (86-100°C), feed mass flux (0.025-0.250 gm./cm.-min.), and feed composition (73-87 wt. % ethanol in water). Effects of initial grain moisture content (0-2.3 wt. % water), grain particle size (0.208-2.363 mm), and grain regeneration (one desiccation as compared with two) were also studied. One experiment was performed using barley straw. Ethanol-water vapors were pumped through a fixed bed containing ground grain.

Under the conditions used, the amount of water sorbed and amount of ethanol concentrated were inversely related to the initial grain temperature, feed mass flux, initial grain moisture content, and grain particle size (for the larger size range tested). The amount of water sorbed was also inversely related to the feed composition. Over the range of 73-87 wt. % ethanol, the amount of ethanol concentrated was not affected by the feed composition. A maximum of 0.0999 gm. water was sorbed per gm. grain for a combined effluent sample of 99 wt. % ethanol. A maximum of 0.326 ml. ethanol was concentrated per gm. grain for a combined sample of 99 wt. % ethanol. Grain regeneration had little effect on its performance. For grain desiccated once, 0.138 gm. ethanol was concentrated per gm. grain for a combined sample of 99 wt. % ethanol. Grain desiccated twice had a corresponding value of 0.143. Grain yielded better results than straw. Grain concentrated 0.318 ml. of ethanol per gm. of grain for a combined sample of 99 wt. % ethanol. The corresponding value for straw was 0.182.

The results indicate that adsorption occurs, as well as adsorption. About 5-20% of the feed ethanol was sorbed by the grain.

A mathematical model was developed that expresses the amount of ethanol concentrated as a function of feed mass flux and initial grain temperature.

## INTRODUCTION

The continued depletion of our crude oil and natural gas supplies has turned our attention to other forms of energy. Considering the renewable forms of energy, ethanol is currently being looked at as a possible cure for part of our energy problems.

Currently, in Brazil ethanol is mixed with gasoline in concentrations of up to 30 volume % ethanol (1). It is projected that by 1985, five billion liters of ethanol will be produced annually or enough ethanol to combine all gasoline consumed into 25 volume % ethanol mixtures (2).

In the United States the annual production of ethanol is expected to increase from 300 million liters in 1980 to 5.7 billion liters in 1990 (3). The 1990 figure amounts to enough ethanol to produce 10 volume % ethanol mixtures with 20% of the gasoline consumed (4). This quantity of ethanol is not enough to become a major solution to our energy needs, but it could help to extend our oil reserves a little longer and allow more time for development of other forms of energy.

(2)

Pure ethanol can be burned in an engine designed for gasoline use, but there are two main disadvantages to this:

- (1) ethanol has only about two-thirds of the energy content of gasoline (5)
- (2) engine adjustments (mostly related to the carburetor) must be made (1)

Another alternative is to combine ethanol and gasoline into mixtures of up to 20 volume % ethanol. Some of the advantages of this alternative are:

- (1) although ethanol only has two-thirds of the energy of gasoline, it has a blending octane value of approximately 120, which tends to compensate for the lower energy content of the ethanol
- (2) engine modifications are not necessary (6)

Although no engine adjustments are necessary for ethanol-gasoline mixtures of up to 20 volume % ethanol, Brazilian studies have shown that the optimum mixture contains 10-15 volume % ethanol (7). In the United States, the standard ethanol-gasoline mixture contains 10 volume % ethanol and is called Gasohol.

One disadvantage to blending ethanol with gasoline to form Gasohol is that even small amounts of water will separate out of the mixture and form two phases. A 25 volume % ethanol in gasoline mixture can only tolerate about 1% water at 70°F without separation occurring (8).

At lower temperatures and lower ethanol concentrations the amount of water able to remain in solution becomes even less. The main problems, with this separation phenomenon, are that 1) the separated water phase will cause the engine to function improperly, and 2) a relatively large amount of ethanol will go into the water phase, thus decreasing the ethanol content of the combustible gasoline phase.

The majority of the ethanol produced for Gasohol will be made by the fermentation process. The only other feasible way to produce ethanol uses ethylene as the main feedstock and is not a very good process if the goal is to reduce petroleum consumption. During the fermentation process, water and six-carbon sugars (such as glucose) from some form of biomass, such as corn or barley, are converted by yeast to ethanol and carbon dioxide. The fermentation process takes about 36 to 48 hours and the product is a solution of 6 to 12 volume % ethanol in water (9).

The standard method for separating ethanol and water is by distillation. Ethanol and water mixtures form what is known as an azeotrope and can only be concentrated to 97.25 volume % ethanol by binary (only ethanol and water

present) distillation (10). If another component, such as benzene, is added to the azeotropic mixture, a ternary system is then created and the ethanol can be completely separated from the remaining water. Some of the problems associated with this method are:

- 1) Three distillation columns are conventionally needed:
  - a) one for concentrating the 6-12% water-ethanol mixture to 97+ volume % ethanol in water,
  - b) another column for the ternary distillation stage in which 97+ volume % ethanol in water is concentrated to 100% ethanol, and
  - c) a third column to regenerate the benzene (11).
- 2) 1.4 to 1.6 times more energy is required just to obtain 97+ volume % ethanol in water than can be generated by burning the ethanol. This does not include the energy required for ternary distillation (12).

The last major point, when considering the use of distillation in the production of ethanol for fuel, is that the majority of the heat required for distillation is the heat needed to concentrate ethanol to concentrations higher than about 93.5 volume % ethanol in water. Studies show that when ethanol is concentrated from 15 volume % to 93.5 volume %, the energy content of the ethanol will be 11 times the amount required for

distillation (13). As higher ethanol concentrations are approached, this ratio drops rapidly and becomes less than 1 at the azeotrope.

As a result of this problem, an alternative to distillation for dehydrating ethanol in the 93.5 to 100 volume % range is needed. Ladisch and Dyck found, in addition to a number of other dehydrating agents, corn was able to remove water from ethanol-water mixtures (14). It was shown that shelled and cracked corn could concentrate ethanol-water vapors from 77 wt. % to 97.7 wt. % ethanol (see Table 71, Appendix G, for percent conversion). I will discuss their research in greater detail in a later part of this thesis.

The vast availability of barley in Montana makes this grain desirable to use in ethanol production, as opposed to corn in the Ladisch and Dyck experiments. Barley is Montana's second largest grain crop, with an annual production of 40.5 million bushels. Montana also ranks fifth in the United States for barley production (15).

The mechanism by which the ethanol dehydration process takes place is selective sorption. In this case, selective sorption means water has a greater affinity

for the barley grain than ethanol does, and consequently, water is sorbed in larger amounts than ethanol. As the ethanol-water mixture passes through the grain, more water will be sorbed than ethanol, which will increase the ethanol concentration of the part of the ethanol-water mixture that is not sorbed. This essentially dehydrates the ethanol.

In order to understand how this dehydration process occurs, a study of the sorption phenomenon is necessary.

### Sorption

The general term "sorption" refers to the process by which a gas, vapor or liquid is taken up by a solid. Sorption is a general category that includes two more specific subjects (16):

- 1) Adsorption, which can be further broken down to:
  - a) Physical Adsorption
  - b) Chemisorption
- 2) Absorption

Adsorption refers to the process in which a gas, vapor or liquid collects on the surface of a solid, which may be very porous; therefore, some of the surface may be in the interior of the solid (16). The adsorbed molecules may not penetrate the field of force that exists between the atoms, ions, or molecules of the solid.

Absorption, on the other hand, refers to the actual penetration of the field of force that exists between the atoms, ions, or molecules of the solid by the absorbed molecules. In this case, a solid solution is actually formed between the solid and the absorbed material, whereas in adsorption, a solid solution is not formed.

An example of the difference between adsorption and absorption can be made using alumina and calcium chloride (17). The alumina will take up moisture at room temperatures, but can easily be regenerated, or dried, by simply raising the temperature and distilling off the water. When the surface of the alumina becomes saturated with water, no more water is taken up and no further changes occur.

However, if calcium chloride is used as a desiccant, a hydrate is formed and reaches a saturation point at  $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$ . Additional water added to the calcium chloride after this point will cause the crystalline structure of the desiccant to lose its shape and actually dissolve in the absorbed water. Throughout this example, calcium chloride is an absorbent while alumina is an adsorbent.

It is necessary for a sorbed molecule to attach to the solid surface of a sorbent before it can penetrate

the field of force between the molecules of the solid sorbent. Since this is true, adsorption can occur without absorption being present, but absorption must always be preceded by adsorption. Adsorption can be seen to be very important in all sorption processes and therefore warrants further discussion.

### Adsorption

As mentioned previously, adsorption is the main category for two subcategories: physical adsorption and chemisorption (18). Physical adsorption is due to the same weak van der Waals forces that are responsible for condensation. Chemical bonds are not formed and the adsorbed molecules may be easily removed from the adsorbent if the temperature is increased or the pressure is decreased. Chemisorption, on the other hand, has associated with it the formation of surface chemical bonds that may vary in their strength (19).

During the process of adsorption, there is heat generated which is known as the "heat of adsorption" (20). Adsorption is similar to condensation and heat is released during the phase change. Therefore, just as condensation is an exothermic process, so is adsorption. The heat of physical adsorption is approximately 1-1/2 to

3 times as much as the latent heat of condensation and is actually closer to the latent heat of sublimation. Heats of chemisorption are generally much higher than that of physical adsorption and usually chemisorption occurs at higher temperatures than physical adsorption.

It has been found that as the amount of adsorbed material increases, the average heat of adsorption decreases (21). Brunauer, Emmett, and Teller assumed that in the adsorption process the adsorbed material is laid down on the adsorbent in layers and that all layers, except the first, had the normal heat of condensation associated with them (See Figure 1). The first layer has a much higher heat of condensation; therefore, as the first amount of adsorbate is adsorbed, a large quantity of heat is released. As additional amounts of material, corresponding to the second and higher layers, is adsorbed, the normal heat of adsorption (condensation) for the upper layers averages out with that of the first layer. As the amount of adsorbed material increases, the average heat of adsorption decreases.

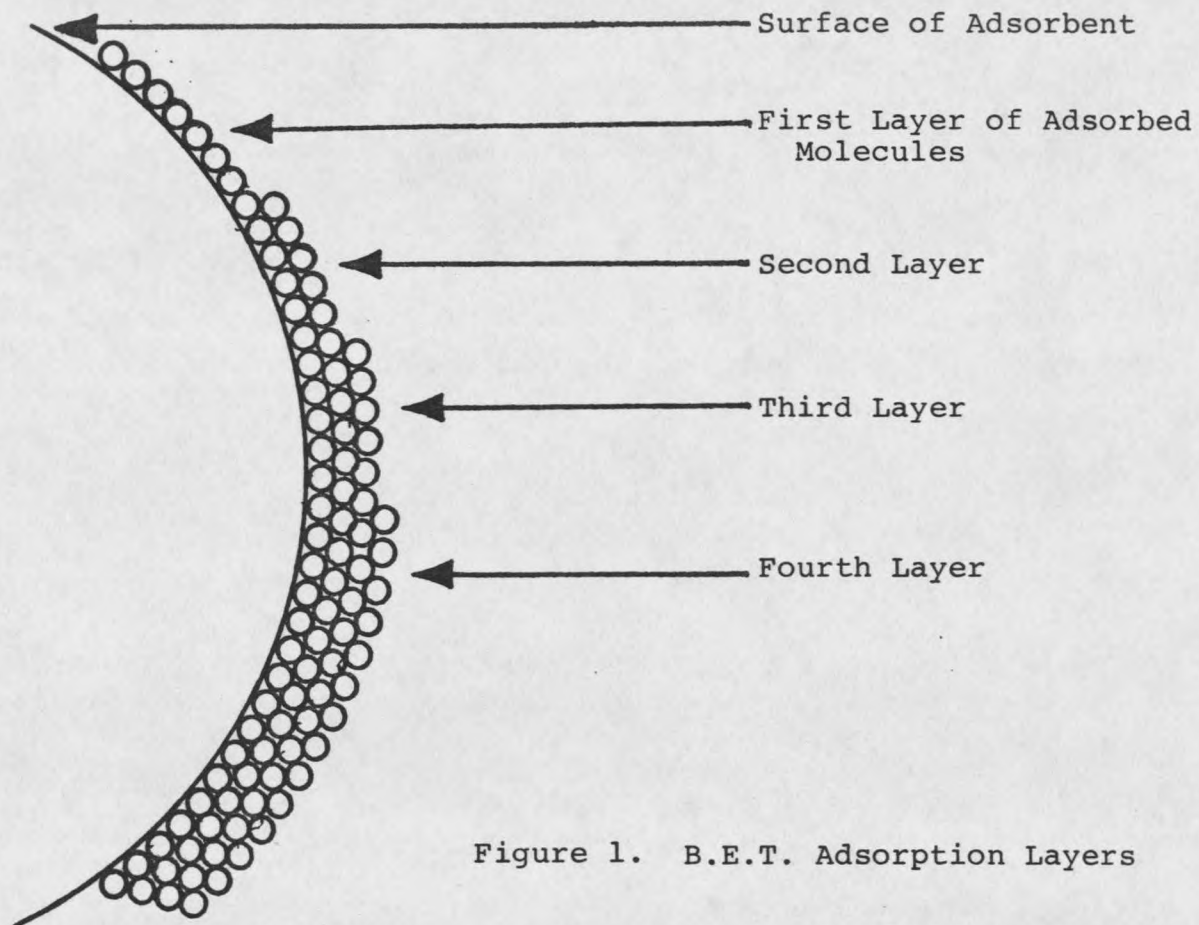


Figure 1. B.E.T. Adsorption Layers

### Theory and History

C. W. Steele, in 1773, and Abbé F. Fontana, in 1777, were the first to study adsorption. Their studies were with the adsorption of gases by charcoal (22). Since that time, many efforts have been made to understand the theory behind this phenomenon.

In 1915, a theory was proposed by Langmuir that said adsorption was a process that was a type of chemical combination and that the absorbed layer was unimolecular (23). Near this time, Polanyi thought adsorption was a physical process, electrical in nature, and that the absorbed phases could have many layers (23). Zsigmondy's studies in 1911, led him to believe that the adsorption of water by silica gel was a function of the capillary properties of the gel (23). During the 1920's Patrick suggested that all physical adsorption was the result of capillary condensation (23).

Brunauer, Emmett, and Teller, during the year 1938, proposed a theory for physical adsorption in which they assumed that the same forces which are effective in condensation are also largely responsible for the binding energy of multimolecular adsorption (23). The assumptions that they used to develop their mathematical model

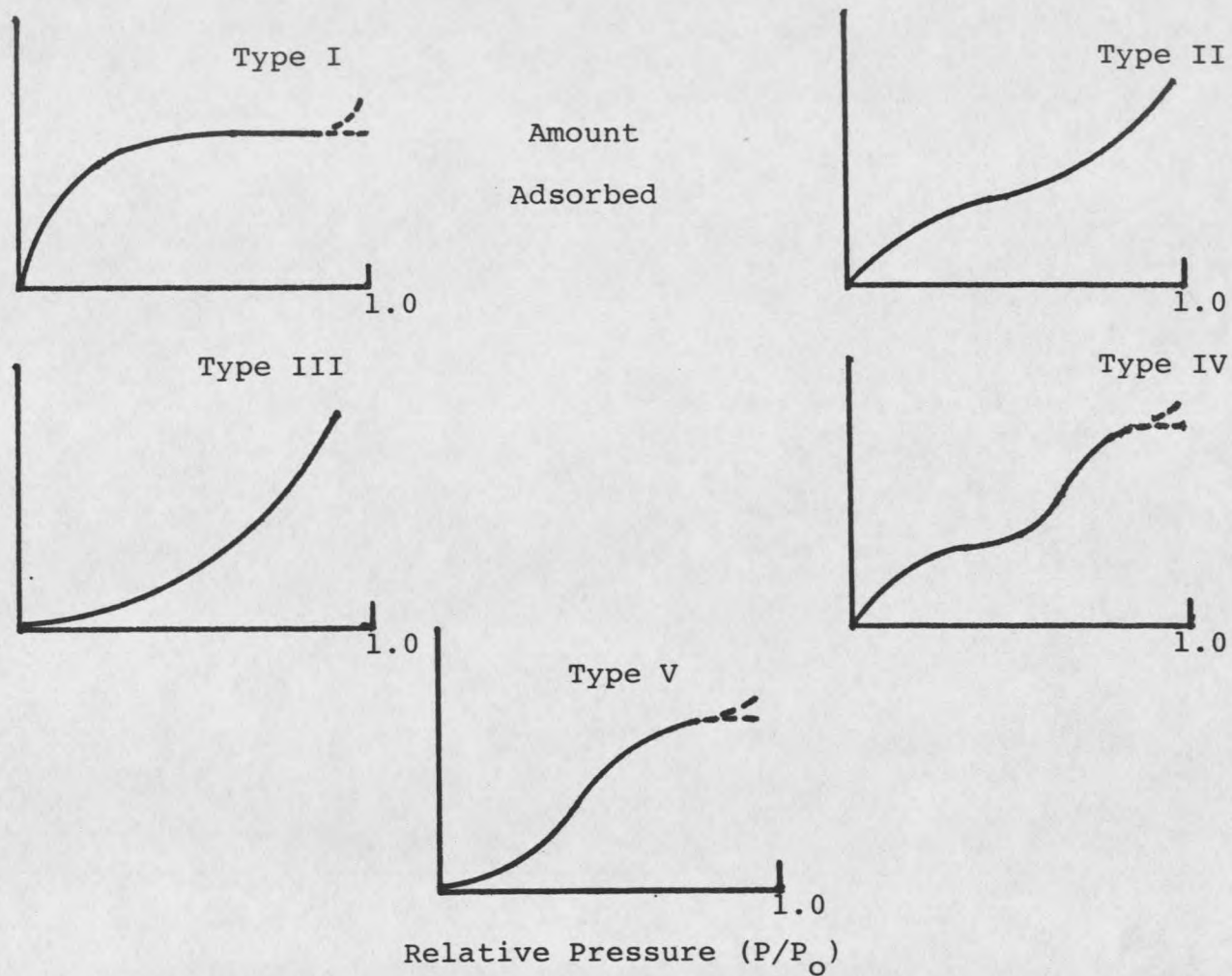
(12)

are essentially the same as the assumptions made by Langmuir, except for the concept of multilayers. The equation that they developed is as follows (24):

$$V = \frac{V_m C P}{(P_0 - P)(1 + (C - 1)(P/P_0))} \quad \text{(Eqn. 1)}$$

V = volume of gas adsorbed  
V<sub>m</sub> = volume of gas necessary to cover  
the available adsorbent surface  
C = a constant  
P = partial pressure of gas  
P<sub>0</sub> = saturation pressure of gas

An adsorption isotherm is a plot of the amount of material adsorbed vs. the pressure with temperature constant (25). Brunauer, Demming, Demming and Teller proposed a classification in which adsorption isotherms were divided into five classes. These are shown in Figure 2. Type I represents single layer chemisorption. Type II represents multiple layer physical adsorption in which there is no pore volume limitations on the adsorbent. This is the most common kind of physical adsorption and also describes sorption in biological materials, as is discussed in the next section. Type IV represents multiple layer physical adsorption with pore volume limitations. Types III and V are representative of



(13)

Figure 2. Five Adsorption Classifications of B.E.T. (23)

physical adsorption systems with very weak attractive forces. The above equation has been shown to give a close approximation of actual isotherms in these five classes. This equation is still used extensively to describe actual isotherms (26).

A better understanding of how the sorption process works in the system of this project is needed. Thus, it is important to study adsorption, and sorption in general, as related to biological materials, such as grain.

#### Sorption in Biological Materials

Adsorption in biological materials is almost completely physical adsorption and the isotherms generally are of the Type II classification of Brunauer, Emmett, and Teller (B.E.T.) (26) (see Figure 2). An explanation of the different parts of this sigmoidal shape curve was proposed by Babbitt from studies of spruce wood and cotton (27). In his studies, the equilibrium moisture contents of wood and cotton were plotted against the relative humidity of air used in the experiments. Babbitt thought that the low relative humidity region of the isotherm could be explained in Langmuir's equation

for a monolayer of adsorbate. Gradual, rather than abrupt, deviations from the Langmuir equation at around 20% relative humidity were explained by the concept that the second layer of adsorbed material begins building before the first layer is complete. The 20% to 80% relative humidity range was explained by the polymolecular layer B.E.T. equation. Deviations of the B.E.T. equation, from the experimental isotherms at relative humidities greater than 80% were explained by the theory of capillary adsorption in which water vapor is believed to condense in very minute capillaries of the adsorbent.

Most of the theoretical equations that are derived to describe adsorption in biological materials are based on one or more of the following basic theories (26):

- 1) Polanyi's adsorption potential theorem.
- 2) The B.E.T. polymolecular layer adsorption theory.
- 3) Zsigmondy's "capillary condensation" theory.

An attempt was made by Ngoddy and Bakker-Arkema (26) to combine all three of these theories in the development of an equation to correlate adsorption data for raw freeze-dried beef and corn. Excellent correlations were obtained between calculated and experimental isotherms.

Desorption is the process by which the adsorbate is removed from the adsorbent. There is a hysteresis effect that is present between the adsorption and desorption isotherms of biological products. During desorption, more water is retained by the adsorbent at a given pressure than during adsorption. Young and Nelson made an attempt at explaining this phenomenon. They assumed that there are three types of water sorption that occur in biological materials (21): 1) there is one layer of water that is adsorbed directly onto the outer surface of each biological cell, 2) several layers of normally condensed water adhere to the first layer, and 3) water from the first layer passes through the cell wall and becomes absorbed water in the interior of the cell. Ngoddy and Bakker-Arkema theorized that a second layer of water on the exterior of the cell was necessary before any water could be pushed into the interior of the cell during adsorption. During desorption, all water from the exterior of the cell must be removed before any absorbed water could be released. Therefore, a cell would retain more moisture at a given water vapor pressure during desorption than during adsorption which,

in turn, would explain the hysteresis effect. Very good agreement was obtained between the theoretical equation and the observed moisture contents at 30°C.

Several investigators have shown that starches from different origins (different plants) have varying sorptive capacities, with respect to water, whereas cellulose sorptive capacity shows little change with respect to origin (28,29,30,31,32). Sair and Fetzer demonstrated that tuber starch (potato) had the largest sorptive capacity (33). Pith or root starches (sago, arrowroot, and sweet potato) were next best for water sorption (33). Cereal starches (wheat, corn, and rice) had the least ability to sorb water (33).

It has been suggested by a number of investigators that for both cellulose and starches, the hydroxyl groups on these sorbents play a major roll in water sorption (34,35,36,37). Sair and Fetzer found that severe desiccation in vacuo at 100°C and 135°C reduced the ability of starch to retain water (38). It was proposed that the high heat causes mutual bonding of the hydroxyl groups present in starch and in turn reduces its sorptive capacity. The loss of sorptive capacity is greatest in

the starches with the largest ability to retain water, such as tuber starch. These starches would presumably have the highest number of hydroxyl groups and therefore, more mutual bonding of these groups could occur.

Studies done by Udani, Nelson, and Steinberg showed that in wheat flour, the rate of moisture adsorption was directly proportional to protein content, which shows that protein in wheat is hygroscopic (39).

Most of the equations that are derived to model biological material hygroscopicity deal with equilibrium moisture content as a function of water vapor partial pressure. Few equations have been developed that will calculate moisture sorption rates. Udani, Nelson, and Steinberg developed an equation for the adsorption rate as a function of the difference in equilibrium moisture content and actual moisture content of wheat flour (39). Very good results were obtained from this equation which is as follows:

$$\frac{dW}{dt} = \frac{1}{r} (E-W) \quad (\text{Eqn. 2})$$

W = the flour weight at time t

t = the time from the beginning of adsorption

(19)

$E$  = the equilibrium weight of the flour corresponding to equilibrium moisture content

$r$  = the absorption rate characteristic and is a constant

Babbitt correlated adsorption rate data, for wheat adsorbing water vapor, with the following equation (40):

$$Q_t = -(C_2 - C_0) \frac{4\pi a^3}{3} \left( 1 - \frac{6}{\pi^2} \sum_{n=1}^{\infty} \frac{1}{n^2} e^{-D \frac{2\pi n t}{a^2}} \right) \quad (\text{Eqn. 3})$$

The assumptions involved here are: 1) a kernel of wheat is a sphere of homogeneous material, and 2) diffusion of water vapor into the sphere follows Fick's law. The variables in the above equation have the following meanings:

$Q_t$  = the amount of water that has diffused into the sphere in time  $t$

$t$  = the time from the start of diffusion

$a$  = the radius of a sphere

$C_0$  = the initial concentration of the moisture in the sphere

$C_2$  = the moisture concentration at the surface of the sphere

$D$  = the true diffusion coefficient

$n$  = 1 through  $\infty$  (infinity) for the summation

Fairly good results were obtained between the calculated and experimental moisture contents. However, the experimental rate of adsorption was slower than the calculated

rate at the beginning and becomes faster as the grain approaches equilibrium.

Udani, Nelson, and Steinberg found that rates of adsorption in wheat flour are independent of particle size (39).

#### Previous Work on Ethanol Dehydration by Sorption

It was discovered, over 40 years ago, that calcium oxide is capable of drying ethanol of associated water (41).

Ladisich and Dyck found that certain materials high in cellulose and starch are also capable of dehydrating ethanol (13). Some of these materials are cornstarch, cracked and shelled corn, corn residue, and Buckeye CM cellulose. The experimental procedure for these experiments was as follows. A 20 ml. (milliliter) column was placed in the neck of a 300 ml. flask. The column was kept at a constant temperature by a heated water jacket and the flask was heated by an electric mantle. Aqueous ethanol was boiled in the flask while the entire system was heated to steady state. The preheated dehydrating agent was quickly poured into the column and vapor samples were taken at various time intervals. Plots of Distillate Composition vs. Volume of Distillate were obtained. The

cracked and shelled corn was able to concentrate the ethanol from 77.0 wt. % to 97.7 wt. % and Buckeye CM cellulose dried ethanol from 84.8 wt. % to 99.8 wt. %.

Calculations done by Ladisch and Dyck show that to concentrate ethanol from 12 wt. % to 99+ wt. % gives an ethanol combustion energy-to-distillation energy ratio of 8 for calcium oxide and 10 for cellulose, using binary distillation from 12 wt. % to 89 wt. % and 12 wt. % to 84.8 wt. %, respectively.

#### Fixed Bed Adsorbers

Using fixed bed adsorbers, the adsorbent material is fixed in place in what is usually a cylindrically-shaped vessel. A liquid- or gas-phase, non-sorbable or slightly sorbable fluid will act as a carrier for the sorbable component.

When the mixture first enters the adsorption column, the adsorbent is at an initial loading condition in which the adsorbent contains very little of the sorbable component. As the mixture of carrier and sorbable component flows through the adsorber, the sorbable component is removed from the feed stream by the adsorbent, which becomes increasingly saturated with the sorbable component

until an equilibrium loading level is reached. As the adsorbent becomes increasingly loaded, the sections of the adsorber that are at the equilibrium loading level will no longer remove the sorbable component from the feed. When the entire column is saturated, the effluent will exit from the adsorber with the same concentration of sorbable component as the entering feed contains.

Figure 3 shows graphically how the adsorbent becomes loaded after the feed stream first enters the adsorber.

The nomenclature used is as follows:

- $x$  = amount of adsorbent loading
- $x_0$  = initial loading condition
- $x_e$  = equilibrium loading condition  
(adsorbent is saturated)
- $Y$  = concentration of sorbable component in effluent
- $Y_0$  = sorbable component concentration in effluent before breakthrough occurs
- $Y_f$  = concentration of sorbable component in feed
- $T$  = time
- $T_0$  = time when feed stream first enters absorber
- $T_b$  = breakthrough time (time at which no adsorbent remains in the initial loading condition)
- $T_e$  = time when all adsorbent is at equilibrium loading condition
- $L$  = length of adsorber

Diagram (a) shows the initial loading condition of the adsorbent before any feed has entered the adsorber. Diagram (e) shows the initial concentration of the

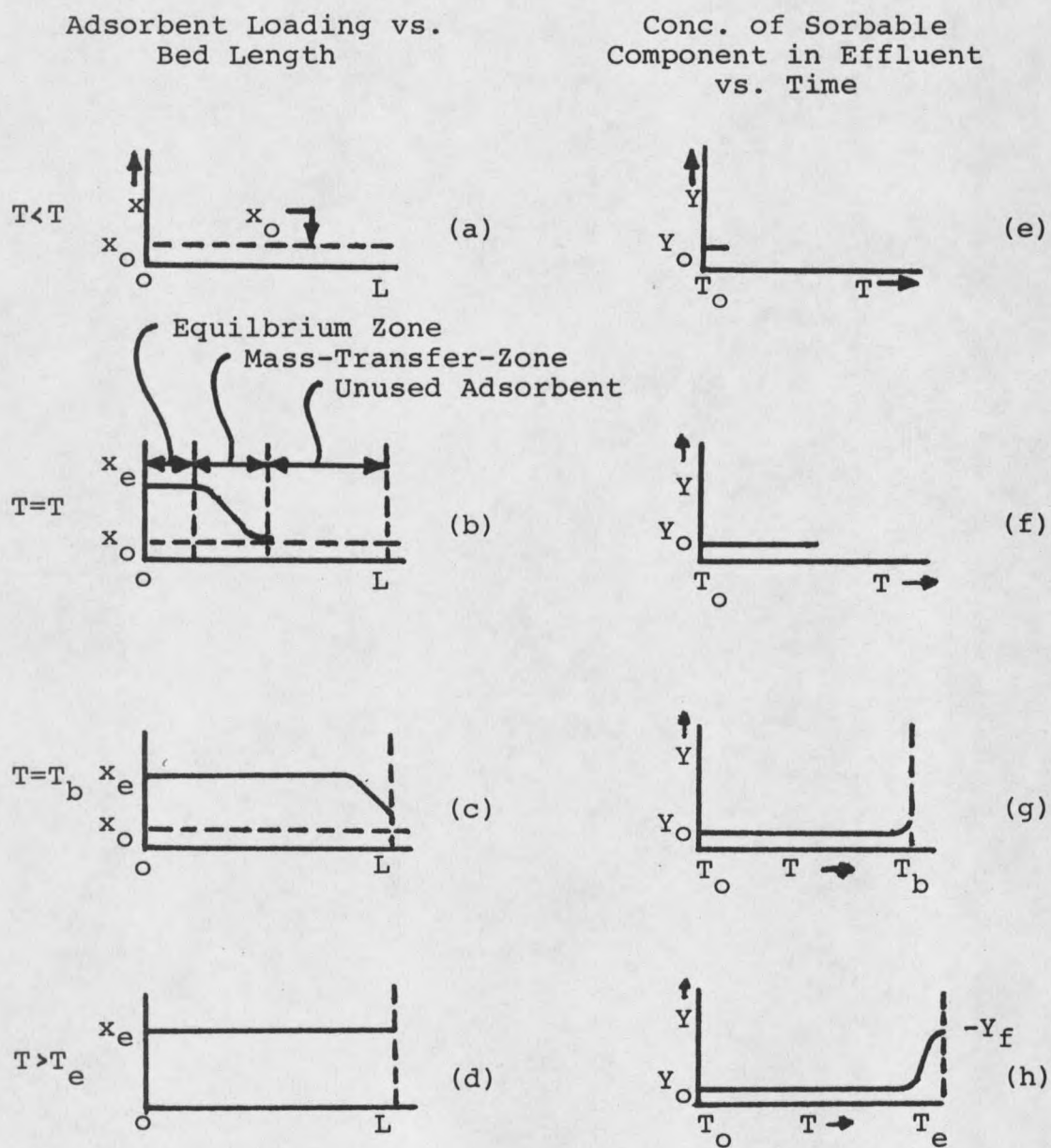


Figure 3. Mass-Transfer Zone Concept (42)

sorbable component in the effluent. Diagram (b) shows the condition of the adsorber after some of the adsorbent has become loaded to the equilibrium loading condition. An area called the Mass-Transfer-Zone (M.T.Z.) exists which is the region where the adsorption process is actually occurring. At the tail-end of the M.T.Z., the adsorbent has become loaded to the equilibrium loading condition. At the leading edge of the M.T.Z., no loading has occurred yet and the adsorbent is in the initial loading condition. The adsorbent is in various loading conditions between these two regions. Diagram (f) shows the concentration of the sorbable component in the effluent, which is still very low.

As time progresses, the M.T.Z. curve will maintain its shape and move down the adsorber toward the exit. Diagram (c) shows the condition called breakthrough, which occurs at time  $T_b$ . Breakthrough refers to the condition when the leading edge of the M.T.Z. reaches the exit. When this occurs, there is no adsorbent that remains in the initial loading condition. At this time, the concentration of the sorbable component in the effluent would begin to increase, as can be seen in diagram (g). At some time after this, all adsorbent is saturated and the concentration of the sorbable component in the effluent

becomes equal to the concentration of the sorbable component in the feed.

The previous discussion applies to situations in which there is little or no heat of adsorption generated, corresponding to a low concentration of the sorbable component in the feed. If this concentration is high, the large amounts of heat produced during the adsorption process could severely distort the shape of the Mass-Transfer-Zone curve. Under these conditions, a temperature wave will also progress down the column corresponding to the M.T.Z.

## RESEARCH OBJECTIVES

The objective of this project was to study the possibility of using barley as a sorbent for removing water from ethanol and water mixtures. The following parameters were considered to be important factors:

- 1) grain temperature
- 2) feed composition
- 3) feed mass flux per unit cross sectional area
- 4) initial grain moisture content
- 5) grain particle size
- 6) grain variety
- 7) adsorber column length
- 8) adsorber column diameter
- 9) pressure
- 10) amount of desiccation which the grain is exposed to

Parameters 1-5 and 10 were varied in the experiments performed and parameters 6-9 were held constant at the following values:

- |                    |                                                                 |
|--------------------|-----------------------------------------------------------------|
| 6) grain variety   | = Shabet barley                                                 |
| 7) column length   | = 30.48 centimeters (cm.)                                       |
| 8) column diameter | = 2.07 cm.                                                      |
| 9) pressure        | = atmospheric (approx.<br>640 millimeters of<br>mercury (mmHg)) |

Table 7 (Appendix A) shows the run numbers for runs used to study the effects of initial grain moisture content, grain particle size, and regeneration of the grain

(redesiccation). Runs No. 34 and 35 were also performed in order to compare barley straw with grain.

A central composite design (43) was used to study the effects of initial grain temperature, feed composition, and feed mass flux. Since the temperature of the grain varied during each experiment, it would have been too difficult to correlate the effect of temperature, at each point in time, with the results obtained. Therefore, the initial temperature of the grain was recorded. This was the temperature of the grain just prior to starting the feed. See Figure 4 and Table 1 for the data points used.

The following parameters were held constant for the central composite design:

- 4) initial grain moisture content = 0%
- 5) grain particle size (See Table 2)
- 10) amount of desiccation which the grain is exposed to = 96 hours at 120 C° and 640 mmHg

In the central composite design, two of the runs used conditions that did not fit the exact design because of lower limits on the temperature and feed mass flux. The effects of this will be discussed later. The lower temperature limit of 86°C was used to prevent vapors from condensing at cold points in the equipment. The lower feed mass flux limit of 0.025 gm./cm.<sup>2</sup>-min.

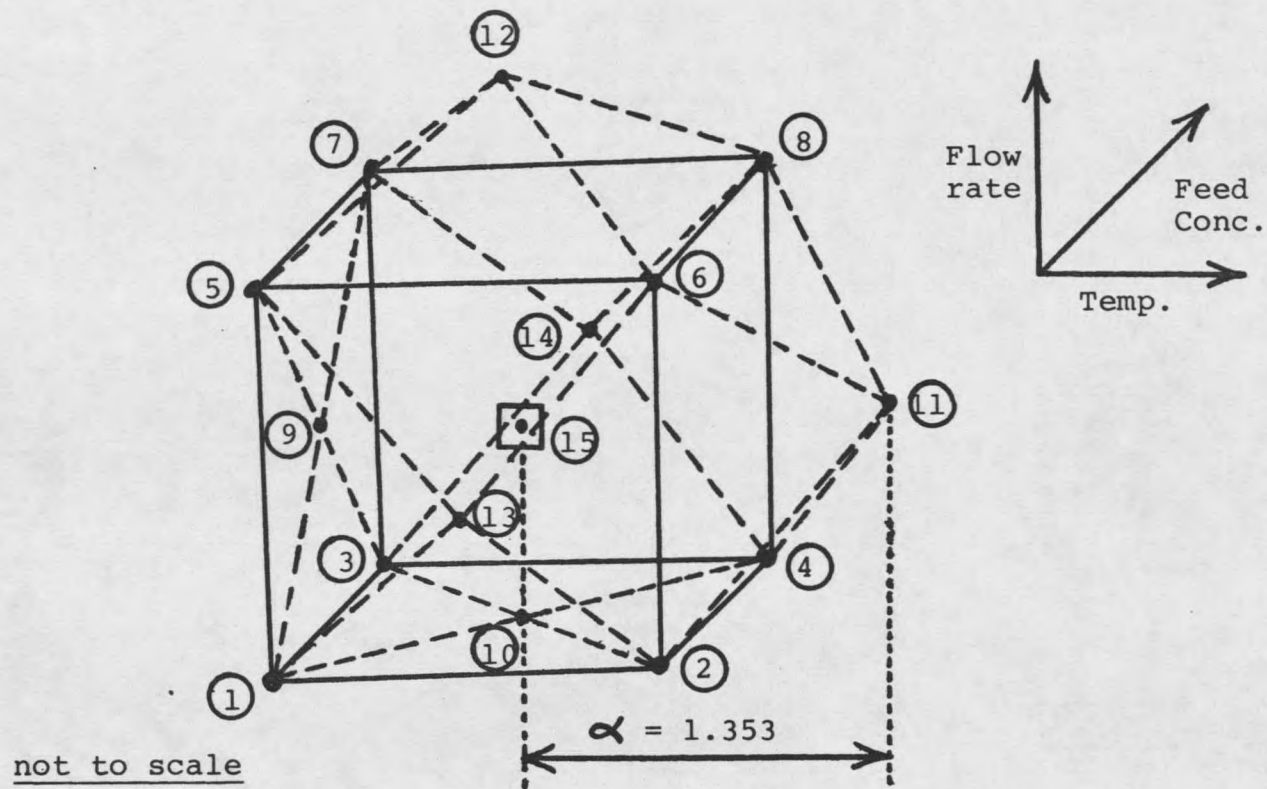


Figure 4. Graphical Representation of Central Composite Design

Table 1. Central Composite Design

| Point<br>No. | Run<br>No.   | Initial<br>Temp.<br>(°C) | Feed Mass<br>Flux<br>(gm./cm. <sup>2</sup> -min.) | Approx.<br>Feed Conc.<br>(wt.%eth.) |
|--------------|--------------|--------------------------|---------------------------------------------------|-------------------------------------|
| 1            | 26,50        | 86                       | 0.025                                             | 75                                  |
| 2            | 30           | 100                      | 0.025                                             | 75                                  |
| 3            | 37           | 86                       | 0.025                                             | 85                                  |
| 4            | 38           | 100                      | 0.025                                             | 85                                  |
| 5            | 28           | 86                       | 0.250                                             | 75                                  |
| 6            | 29           | 100                      | 0.250                                             | 75                                  |
| 7            | 36           | 86                       | 0.250                                             | 85                                  |
| 8            | 33,39        | 100                      | 0.250                                             | 85                                  |
| 9            | 44           | 86*                      | 0.140                                             | 80                                  |
| 10           | 47           | 93                       | 0.025*                                            | 80                                  |
| 11           | 43           | 103                      | 0.140                                             | 80                                  |
| 12           | 45           | 93                       | 0.290                                             | 80                                  |
| 13           | 48           | 93                       | 0.140                                             | 73                                  |
| 14           | 49           | 93                       | 0.140                                             | 87                                  |
| 15           | 40,41,<br>46 | 93                       | 0.140                                             | 80                                  |

\*Lower Limit

was used to keep the required time for a run at less than 12 hours, including set-up and clean-up.

Two correlations were developed to describe the effect that initial grain temperature and feed mass flux had on the amount of ethanol concentrated per gram of barley for a combined sample concentration of 99 wt. % ethanol.

## EXPERIMENTAL SYSTEM

When this project was started, very little of the necessary equipment had been built. A great deal of trial-and-error was involved in the development and construction of the final experimental set-up used to collect the required data. A flow diagram of this final set-up can be seen in Figure 5. The experimental system essentially consisted of:

- 1) Feed Burette
- 2) Pump
- 3) Evaporator
- 4) Adsorber
- 5) Sample Collection Tubes

The feed burette had a capacity of 50 ml. and was accurate to 0.1 ml. The first burette used for this purpose was made of plastic which slowly reacted with the ethanol and became opaque. It was replaced with a feed burette made of pyrex glass.

Two different pumps were used at different times. Up through Rkn No. 37 a high pressure positive displacement pump was used. Proceeding with Run No. 38, and after, a low pressure, peristaltic pump was used. The reason for the pump change was that the high pressure

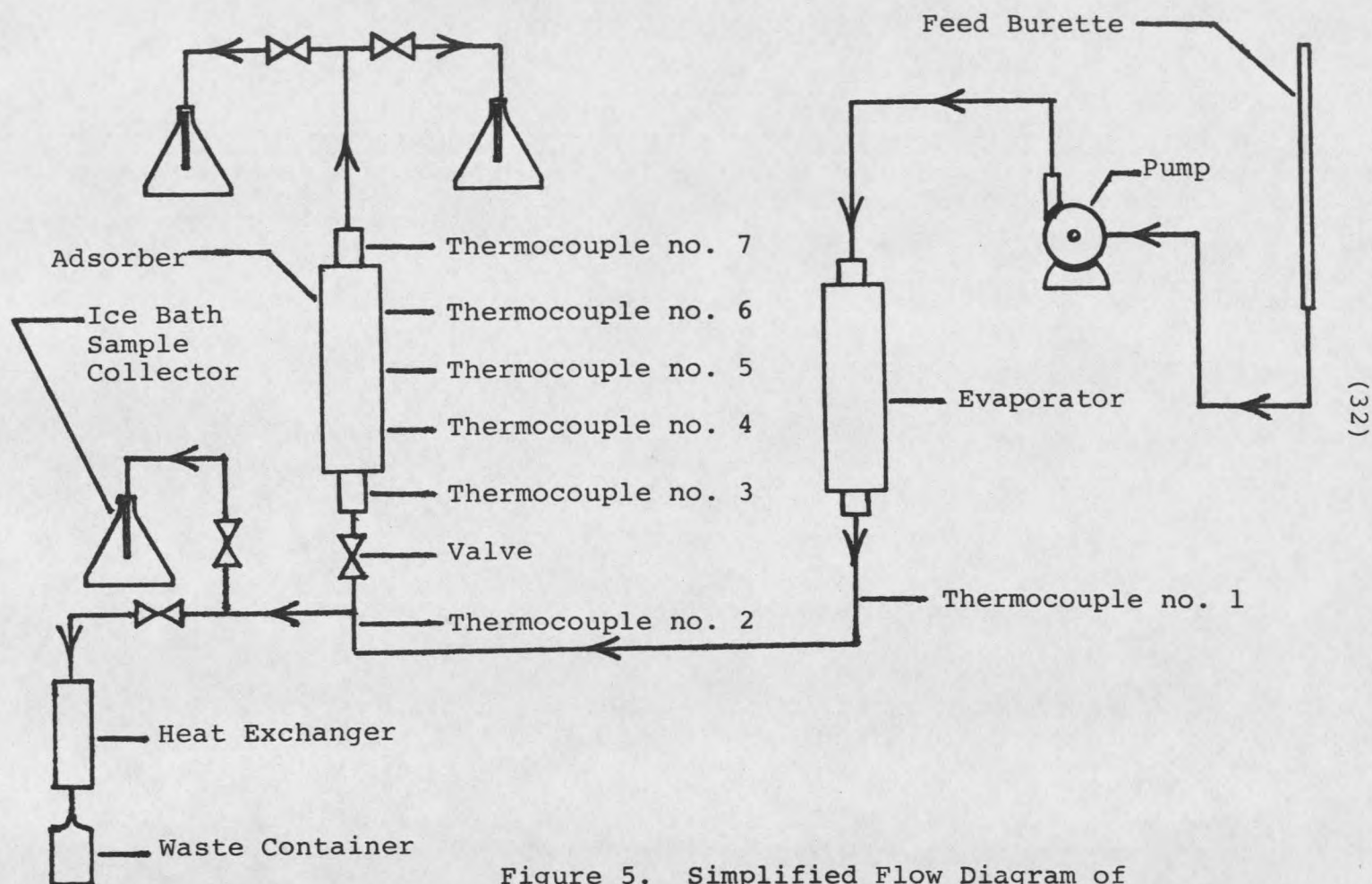


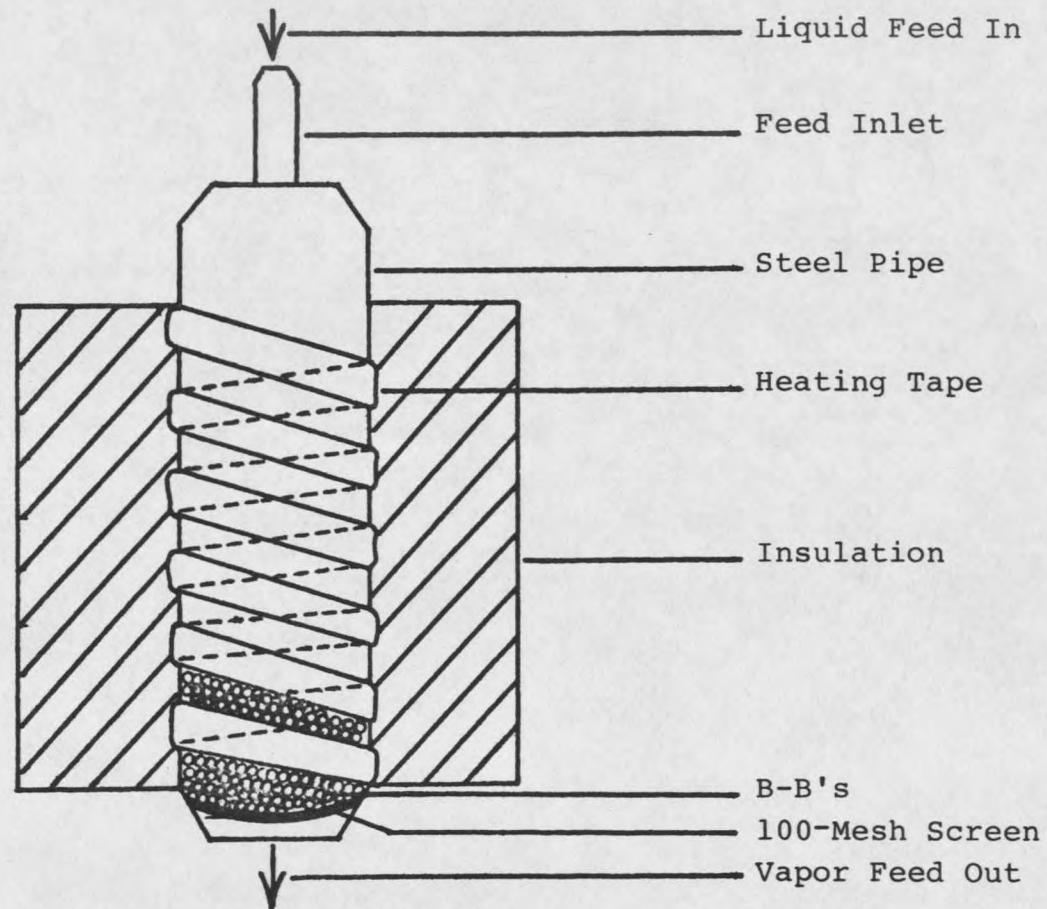
Figure 5. Simplified Flow Diagram of the Experimental System

pump was needed for another experiment. The change of pumps was not due to any problems with the pump's mechanism. The maximum flow rate of both pumps was approximately 2 milliliters per minute. Tygon tubing of 1.59 millimeters (mm.) inside diameter (I.D.) was used in the peristaltic pump and worked very well.

The 0.79 mm. I.D. plastic tubing used to transfer feed from the pump to the evaporator experienced numerous leaks where it was connected to the evaporator feed inlet. Since the evaporator was kept very hot to insure complete vaporization of the feed, the feed inlet on the evaporator was also hot, though not as hot as the evaporator's main body. The plastic tubing had a tendency to come loose and had to be tightened regularly throughout each run. The high temperature also caused the threads on the plastic tubing to wear out rather quickly, and the tubing had to be replaced several times.

Plastic tubing of 0.79 mm. I.D. was used to transfer aqueous ethanol from the feed burette to the pump and from the pump to the evaporator.

The purpose of the evaporator was to vaporize the aqueous ethanol (See Figure 6 for details). The evaporator consisted of a 2.54 cm. diameter pipe filled with B-B's. Heating tape was wrapped around the outside of the pipe and the entire piece of equipment was



(34)

Figure 6. Evaporator

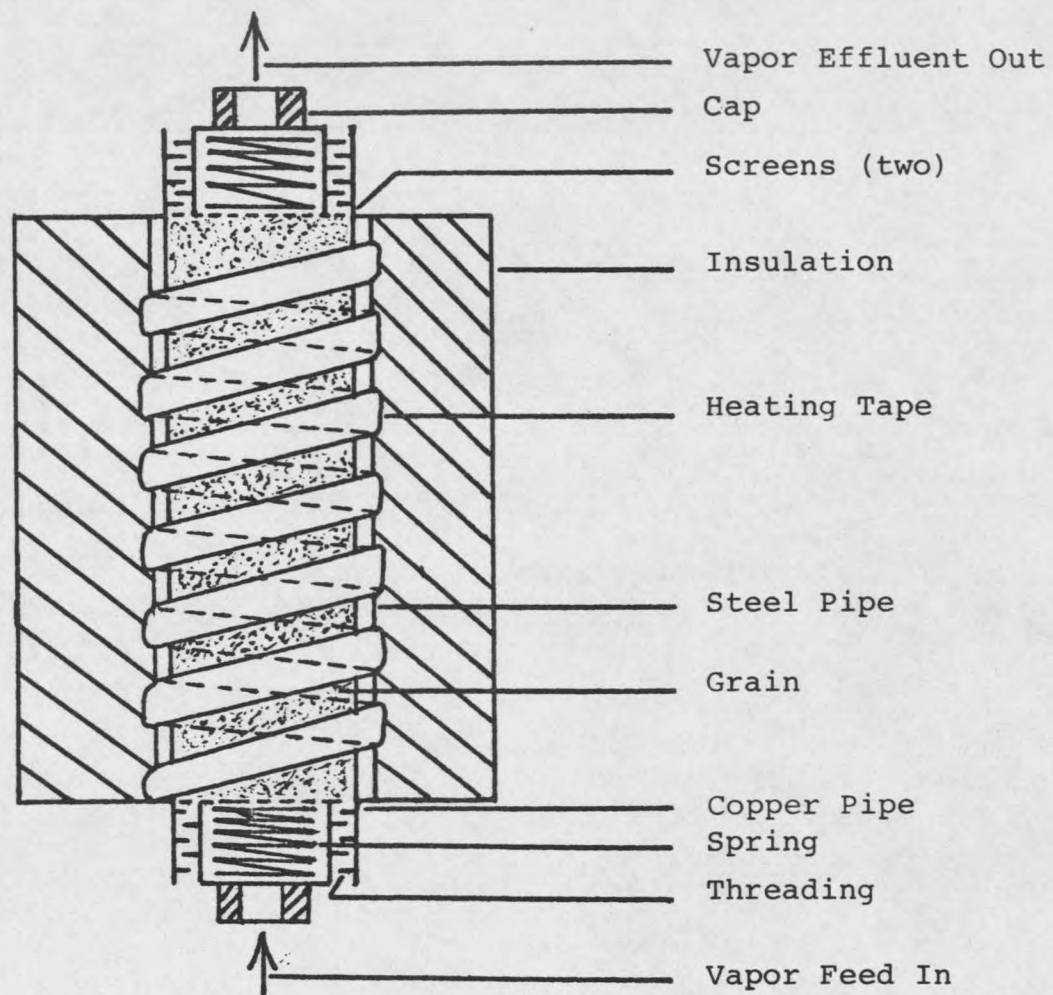
insulated. Feed entered from the top of the vertical evaporator and was discharged from the bottom. The purpose of the B-B's was to create a large, heated surface to assure that all of the entering liquid was vaporized. Extreme care was taken to insure that all the feed was vaporized since this was a vapor-phase process and any remaining liquid would have a different composition than the entering feed. This also would mean that the vapor composition would be different from that of the entering feed. The purpose of having the liquid feed enter from the top of the evaporator was to insure liquid would continue to trickle down through the evaporator until complete vaporization was obtained. The heat output from the heating tape of this piece of equipment and all others was controlled by a Powerstat.

The vapor would then flow through a heating tape-covered and insulated section of 6.35 mm. outside diameter (O.D.) copper tubing before entering the adsorber. The purpose of this part of the experimental system was to insure that all of the feed was vaporized and allowed the flowing vapor to attain steady state before entering the adsorber.

Between the section of tubing just discussed and the adsorber were a set of valves and additional heating

tape-covered copper tubing. This allowed for the vapor to bypass the adsorber and be condensed by the water-jacketed heat exchanger, while the vapor flow and temperature came to steady state. This made it possible, from this system of valves, to take a sample of vapor just before it entered the adsorber. This allowed the composition of the vapor to be checked to see if it was the same as the composition of the feed just before it entered the adsorber. Vapor samples were condensed in test tubes immersed in an ice bath.

The purpose of the vertically-positioned adsorber was to hold the ground barley in a fixed position and to allow for changes in the temperature of the barley (See Figure 7 for details). The main body of the adsorber was a 30.48 centimeter (cm.) long by 2.073 cm. I.D. piece of copper pipe. A steel pipe with a wall thickness of 0.35 cm. was placed around the copper pipe to act as a heat sink and eliminate temperature variances in the axial direction of the adsorber. Heating tape was wrapped around the steel pipe and the entire adsorber was insulated. Grain was held in place by four 100-mesh screens, two in each end of the adsorber. A spring in each end of the adsorber was fitted between the cap and the screens and thereby held the screens in place. The outside end of each cap was a Swagelok fitting and was

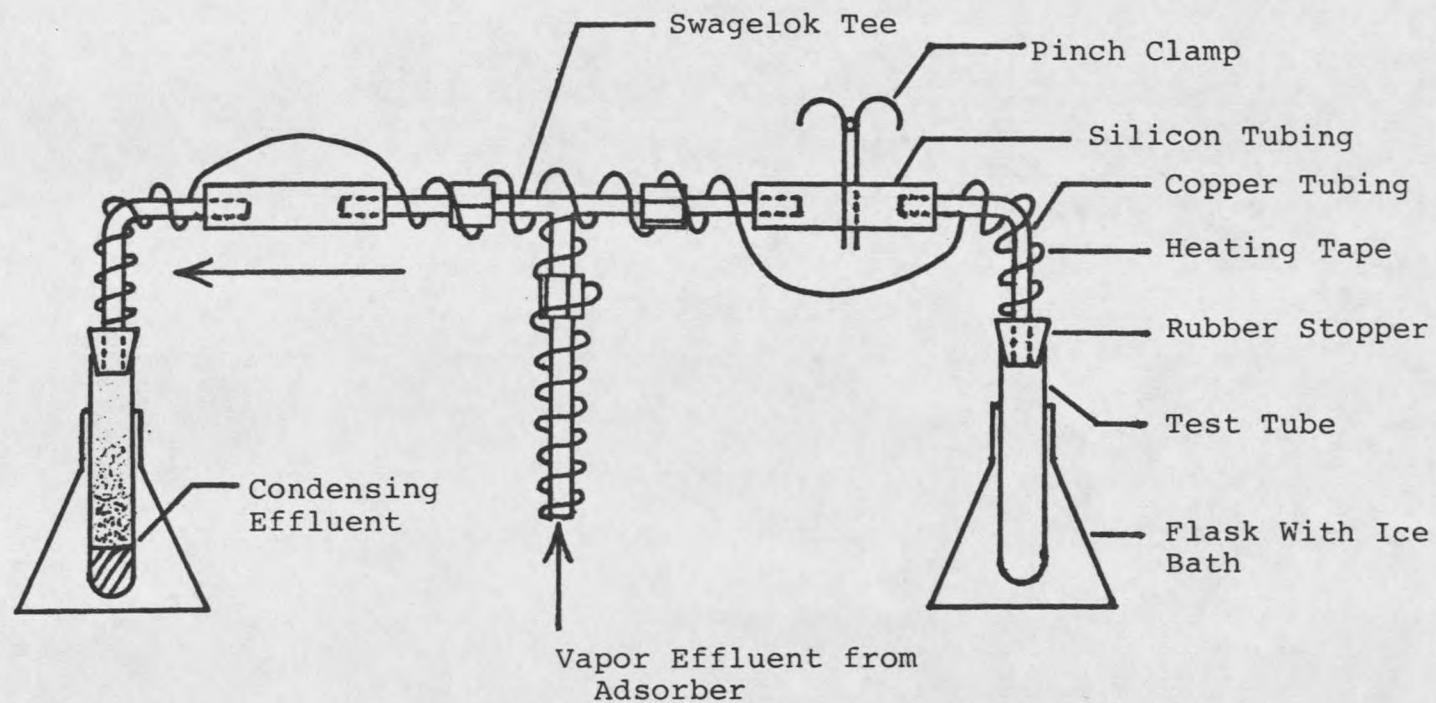


(37)

Figure 7. Adsorber

connected to copper tubing at each end of the adsorber. All copper tubing connections were made by Swagelok fittings. Vapor entered the bottom and left through the top of the adsorber. Temperature indication in the adsorber will be discussed later.

Vapor leaving the top of the adsorber entered a Swagelok tee (See Figure 8 for details). Two valves were used to control which side of the tee the vapor left from. At each exit of the tee was a test tube immersed in an ice bath. The purpose of this system was to allow for continuous collection of vapor from the adsorber for mass balance and sample collection purposes. The effluent from the adsorber was kept in the vapor phase until it entered the sample collection tubes so that the problem of excessive lag times from the adsorber to the sample tubes could be kept to a minimum. Thus, the problem of effluent sticking to the inside of the tubing was eliminated. During runs a test tube would be in place at each exit of the tee, but one of the exit valves would be open and the other closed. This would allow for all of the vapor leaving the adsorber to be collected in only one test tube. When it was time to change to the next test tube for the next sample, the valves' positions were simply reversed and vapor was immediately directed to the other test tube.



(39)

Figure 8. Vapor Effluent Collection Equipment

The valves that are being discussed were actually pieces of silicon tubing placed between two pieces of copper tubing with a pinch clamp to close off the vapor flow when necessary.

Iron-constantan thermocouples were used for temperature measurement. A Leeds and Northrup Speedomax temperature recorder was connected to these thermocouples. The position of the five thermocouples used in the adsorber are shown in Figures 5 and 9. Thermocouples No. 1 and 2 were used to detect the temperature of the vapor leaving the evaporator and before entering the adsorber, respectively. These two thermocouples were each positioned in one of the three openings of a tee, with the other two openings connected into the main flow of vapor. The thermocouples were held in place by fast-drying epoxy glue.

Thermocouples No. 3 and 7 were used to detect the temperatures of the inlet and outlet vapor, respectively. They were placed directly in the vapor flow, next to the 100-mesh screens. These thermocouples, No. 3 and 7, were placed on the opposite side of the screens from the barley. A hole was drilled in the side of the adsorber at the top and also one at the bottom. These were located just above and below the top and bottom screens, respectively. Pieces of small-diameter copper tubing,

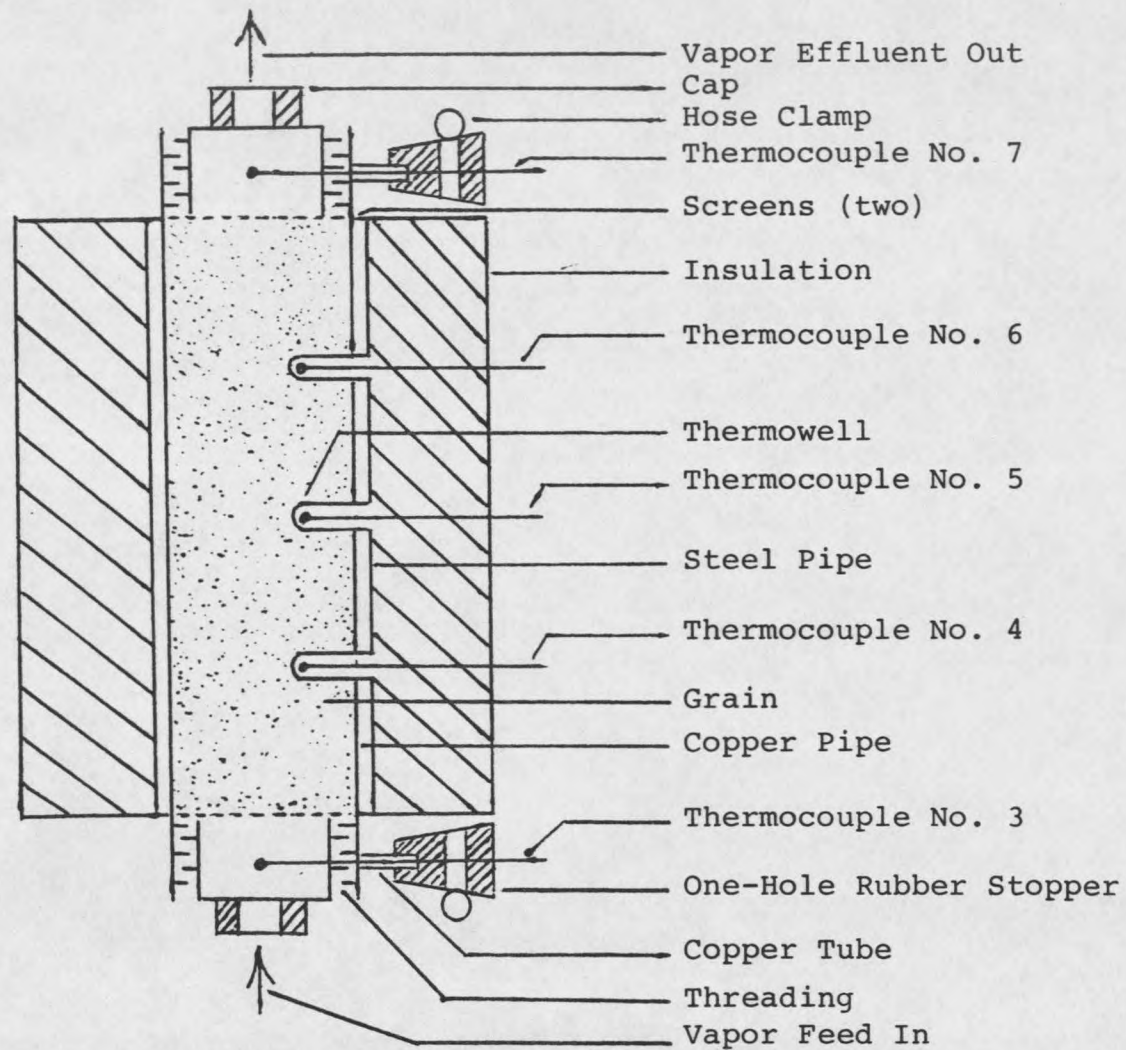


Figure 9. Thermocouple Placement  
in Adsorber

2.54 cm. long, were soldered into these holes. The thermocouple wires could then be pushed through these tubes and positioned in front of the respective screens. One-holed rubber stoppers with hose clamps placed around them were placed around the copper tubes and thermocouple wires. The clamps were tightened and sealed so that no vapor could escape.

Thermocouples No. 4, 5, and 6 were used to detect the barley temperature at 7.6 cm., 15.2 cm., and 22.9 cm. from the inlet, respectively. Holes were drilled in the side of the adsorber at the indicated positions. Sealed thermowells, made from small diameter copper tubing sealed with solder at one end, were placed in these holes. The thermowells extended in from the adsorber wall about 0.7 cm. The thermocouples were then pushed into the thermowells as far as possible.

## EXPERIMENTAL PROCEDURE

Barley Preparation

The following discussion pertains to only the barley used for Runs 26-59 since Runs 1-25 did not provide useful data and were mainly important only for equipment development.

All barley grain used was first ground in a hammer mill with a 1.59 mm. screen. In all runs, except the barley straw and specific-particle-size runs, ground barley was used. Grinding and sizing of the straw was done by the Biochemistry Department. Two runs were made using barley straw and three runs were used using grain of specific particle sizes. The particle sizes were measured in millimeters (mm). The particle sizes of the barley for the specific-particle-size runs were 8-12 mesh, 20-30 mesh, and 50-70 mesh for Runs No. 54, 56, and 57, respectively. The exact opening size for these meshes can be seen in Table 2.

The grain was screened when its moisture content was about 9.0 wt. %. The size screens used can be seen in Table No. 2 and were round Ro-Tap screens. Ro-Tap

Table 2. Particle Size Distribution of Grain After  
Grinding in Hammermill with 1.59 mm  
Screen, but Before Drying

| U.S. Series                            | Screen Size   | Percent of total over that screen size |          |          |
|----------------------------------------|---------------|----------------------------------------|----------|----------|
| Mesh                                   | Opening (mm.) | Sample 1                               | Sample 2 | Sample 3 |
| 8                                      | 2.362         | 4.3                                    | 4.5      | 4.9      |
| 12                                     | 1.651         | 12.8                                   | 12.9     | 13.3     |
| 16                                     | 1.168         | 27.6                                   | 27.6     | 28.0     |
| 20                                     | 0.833         | 48.8                                   | 49.1     | 49.2     |
| 30                                     | 0.589         | 64.8                                   | 64.9     | 65.0     |
| 40                                     | 0.417         | 77.3                                   | --       | 77.0     |
| 50                                     | 0.295         | 86.9                                   | 86.7     | 86.9     |
| 70                                     | 0.208         | 92.4                                   | 92.7     | 92.7     |
| 100                                    | 0.147         | 96.8                                   | 97.1     | 96.9     |
| 140                                    | 0.107         | 98.4                                   | 98.9     | 98.8     |
| 200                                    | 0.074         | 99.8                                   | 99.9     | 99.9     |
| 270                                    | 0.053         | 99.9                                   | 100.0    | 100.0    |
| Total sample wt. after screening (gm)  |               | 97.70                                  | 113.60   | 124.45   |
| Total sample wt. before screening (gm) |               | 99.35                                  | 115.25   | 126.10   |
| Wt. loss (gm)                          |               | 1.65                                   | 1.65     | 1.65     |
| Percent loss of total wt.              |               | 1.7                                    | 1.4      | 1.3      |

screens were stacked on top of each other in order of decreasing screen size from top to bottom. The choice of these specific sizes was based on a screen scale ratio of 1.414, which means that, starting with the small screen, every other size was twice as big as the last. The in-between screen sizes fit into the progression according to the 1.414 ratio. The range of screen sizes was chosen according to the approximate maximum and minimum sizes of the barley grain particles. The weight of grain for each size was added cumulatively, beginning with the largest screen size and progressing to the smallest. This means that the percent of the total grain above a certain screen size was found.

Since the screens were stacked together as discussed earlier, grain could then be sifted through the screens with the largest particles remaining on the larger top screens and the smaller particles sifted to the bottom. A special machine called a Ro-Tap was used to shake and tap this stack of screens in order to assist in the sifting process.

Obtaining samples for the particle size distribution of all the grain used for Runs 26-59 was done by shaking the grain in a large grocery sack and then pouring it onto a table and splitting the pile using a wide piece

of cardboard. The main pile was first split in half, then into fourths and eighths, and so on until the piles were of the correct sample size.

Three different Ro-Tap runs were made using separate samples from the same bag of ground grain in order to obtain an estimate of error. In Table No. 2, a comparison can be seen of the percent of the total sample weight that is above each screen size for each sample. A comparison can also be seen of the weight loss and percent loss of total weight for each of the three samples.

Drying the barley was accomplished by the use of a large oven in which hot air at 120°C was blown through. Samples to be dried for moisture determination were placed in small aluminum pans with a sample depth of 0.3-0.4 cm. and dried for 24 hours. A Mettler balance, accurate to within 0.1 milligrams, was used to weigh barley before and after being placed in the oven.

Barley that was used for the runs was dried to zero percent by being placed in large aluminum pans, at a depth of 0.5-0.8 cm., and dried in the same oven for 96 hours. The reason for this longer drying time was because of the thicker layers of grain in the drying

pans. The only exception to the 96 hour drying time was the grain that was used for Runs 58 and 59, in which only partially dried grain was used. Runs 58 and 59 were runs in which the grain was dried for 1 hour and 1/2 hour, respectively, in very thin layers (0.2 cm.).

Hart, Feinstein, and Golumbic suggest that samples of barley should be dried at 130°C for 20 hours (44). The oven that was available for drying the samples discussed in this paper was at a fixed 120°C and could not be adjusted. As a result, longer drying times than 20 hours were used to compensate for this difference.

#### Procedure for Runs

The adsorber was blown out before each run with compressed air to insure that it was clean. Teflon tape was wrapped around the threads of the top and bottom caps (See Figure 7). The bottom two screens were put into place and a spring was placed between the screens and the cap. The cap was then tightened. The purpose of the springs was to hold the screens in place.

Barley was weighed on a Mettler balance accurate to within 0.1 gram (gm.). The barley (usually 60 gm.) was weighed in a pan and then poured through a funnel

placed on the top of the adsorber and the adsorber was filled to within 1 cm. of the top. The other two screens were put in place at the top of the column. The top cap was then screwed on and tightened, with the other spring between the cap and the screens.

The adsorber was then fitted into place and the thermocouples connected (See Figures 5 and 9). The heating tape was connected and turned on. Powerstats were used to control the power input to the heating tape. All six Powerstats, for the six sections of heating tape, were turned on and the system was allowed to warm up for about one hour. During this warm-up period, the adsorber and evaporator Powerstats were adjusted to give a higher heat output than was needed during the actual run. This caused the adsorber and evaporator to heat up to the required temperatures more quickly.

When the adsorber temperature had come within about 4°C of the temperature necessary for the run, the adsorber Powerstat was turned down to a setting that was estimated to be close to the correct setting. Trial-and-error was then used to zero-in the adsorber Powerstat to a setting at which there was virtually no temperature

change in the adsorber with respect to time. After the run began, the adsorber Powerstat was left at the constant temperature setting achieved before beginning the run. By doing this, the adsorber could be run under conditions similar to those inside a large-scale adsorber.

While the adsorber was coming to pre-run steady state, liquid feed was pumped into the evaporator. Vapor-phase feed leaving the evaporator would then bypass the adsorber and be condensed into the waste container (See Figure 5). Feed continued to flow through this part of the system until steady state was attained with respect to the temperature and flow rate.

Before the run began, vapor that was going into the waste container was directed instead to enter the pre-adsorber ice bath sample collector (See Figure 5). As soon as the feed sample was collected, all valves, except the one at the entrance of the adsorber, were closed and feed vapors would then enter the adsorber. This was the initial start of the run and the time and level of liquid in the feed burette were noted. All

seven temperatures were recorded first before, and then after the run began (See Figure 9).

Effluent samples from the adsorber were condensed in the post-adsorber ice bath sample collectors (see Figure 8). Samples were collected for specific time periods which varied depending on the feed rate. The slow flow rate (0.1 ml. liquid feed/minute) samples were collected for 20 minute time periods. The fast flow rate (1.0 ml./minute) samples were collected for 5 minute time periods. Using 0.1 ml./minute as an example for the following discussion, the feed burette level would be noted at exactly 20, 40, 60, etc. minutes. One minute after the burette level was noted, flow of effluent was directed to the other ice bath sample collector. The one minute lag time was consistent for all flow rates. During the one minute lag time, all seven temperatures were recorded.

Deciding when to stop the run was done by analyzing effluent samples with the aid of a gas chromatograph, which will be discussed later. After the run had been going on for a period of time, dependent upon the conditions, the ethanol concentration would decrease from a

value close to 100% down to a value close to that of the feed. At this point, the barley approached saturation. The equipment was allowed to run for a variable period of time after this, depending upon the conditions and what data was needed.

The run was completed by closing all valves directly connected to the adsorber. This sealed off the adsorber and allowed feed vapor to be directed to the pre-adsorber ice bath sample collector. Another feed sample was collected at this time and the final burette level was recorded. A feed sample from the pump was taken and the settings of the Powerstats were recorded.

#### Analysis of Samples

Effluent sample tubes were weighed on the Mettler balance which is accurate to within 0.1 milligrams (mg.). The effluent from these tubes was then poured individually into smaller glass sample containers which were later taken to the gas chromatograph. The original sample tubes were dried and weighed again. This gave the mass of each sample and total mass of all effluent.

Barley samples from the top and bottom of the column were placed on pans at the end of the run, weighed, and

dried as discussed earlier. Each sample weighed 10-15 grams. The dried samples were reweighed and a post-run percent moisture was obtained. The center portion of barley in the adsorber was usually not dried. The average moisture percent of all barley in the adsorber was assumed to be the same as the average moisture percent of the two samples that were dried. This estimation can be justified as follows. In Run No. 53, the center portion of grain was dried as was the top and bottom portions. The average of top and bottom samples, estimated that the center sample should be 14.0 wt. % moisture. The actual moisture content of the center sample was 13.3% wt. %. The percent moisture of the top sample was 7.2 wt. % and the bottom sample was 20.8 wt. %. Since this was the greatest difference between the top and bottom of any of the runs, estimates of the moisture content of the center portion of barley would have been even closer on other runs than it was for this run.

Before starting some of the runs, a sample of the barley was dried and weighed in order to make sure that the barley being used for the run had the correct initial moisture.

The gas chromatograph used for effluent sample analysis contained a Poropak R packed column operated at

190°C. A calibration curve of Area Percent Ethanol vs. Weight Percent Ethanol was obtained by making up samples of a known weight percent and comparing this percentage with the area percent of the peaks from the chromatograph for these samples. An integrator, on the chromatograph recorder, aided in finding the peak areas.

## RESULTS AND DISCUSSION

The experiments performed can be catagorized as follows:

- \* Effects of temperature, mass flux, and feed composition.
- \* Effects of grain particle size.
- \* Effects of initial moisture content of grain.
- \* Effects of grain regeneration.
- \* Comparison of grain and straw.

Before these catagories are discussed, some general points about the experiments that were performed should be mentioned.

During the first few runs, large temperature drops were experienced in the vapor feed, measured at Thermocouple No. 2 (See Figure 5), when the valve was opened that allowed feed to enter the adsorber. I believe this was caused by the feed vapors contacting cool surfaces at the base of the adsorber, therefore causing condensed feed to drop down on Thermocouple No. 2. This problem was solved by placing heating tape around the base of the adsorber and by opening the valve to the adsorber

slowly. The amount of cool surface at the base of the adsorber was therefore reduced. Also, by opening the valve slowly, any feed that condensed was readily re-vaporized by the main vapor feed stream.

An interesting phenomenon was noted when analyzing the feed stream before and after the evaporator. As stated previously, a gas chromatograph was used to analyze the ethanol-water samples. Samples leaving the evaporator had a peak on the chromatograph that was not present in the feed entering the evaporator. This peak exactly matched that of methanol with regard to retention time. The magnitude of the peak increased as the temperature of the evaporator was increased. A thermodynamic analysis that was done indicates that the conversion of ethanol and water to methanol has a very low yield and probably some other compound is being formed. Another possibility of the unknown compound is diethyl ether, but the retention time of diethyl ether on the gas chromatograph is much different than the unknown compound. In order to reduce the conversion to the unknown compound, the temperature of the evaporator had to be kept as low as possible while still evaporating the feed.

As discussed in the Experimental System section, problems were experienced obtaining a good seal where the plastic tubing from the pump connected to the

evaporator feed inlet. This problem occurred throughout all of the runs and was expressed, at its worst, as a small hissing sound which would stop temporarily when the plastic tubing was tightened more. Attempts were made to correct this problem, but it could not be eliminated. For this reason, any differences between the amount of ethanol-water entering the adsorber and that leaving it are assumed to be due to losses of the feed at the evaporator inlet.

The results of Runs 26-59, which are in Appendix C, indicate that, although water was primarily sorbed by the barley, some ethanol was also removed from the feed stream. Throughout most of the runs, it appears that 5-20 wt. % of the ethanol was removed from the feed stream. This sorbed ethanol would constitute about 20-30 wt. % of all liquid sorbed. The accuracy of my data does not allow for strong reliance on the exact values obtained but it does give a rough estimate of the amount of ethanol removed from the entering feed stream.

Figure 10 shows a profile of effluent sample concentration vs. time for Run No. 30. The shape of this curve was typical of all of the runs. Note that at the beginning of the run, the effluent concentration was in the 99 to 100 wt. % range and dropped off very fast after about 100 minutes and decreased very slowly after approximately

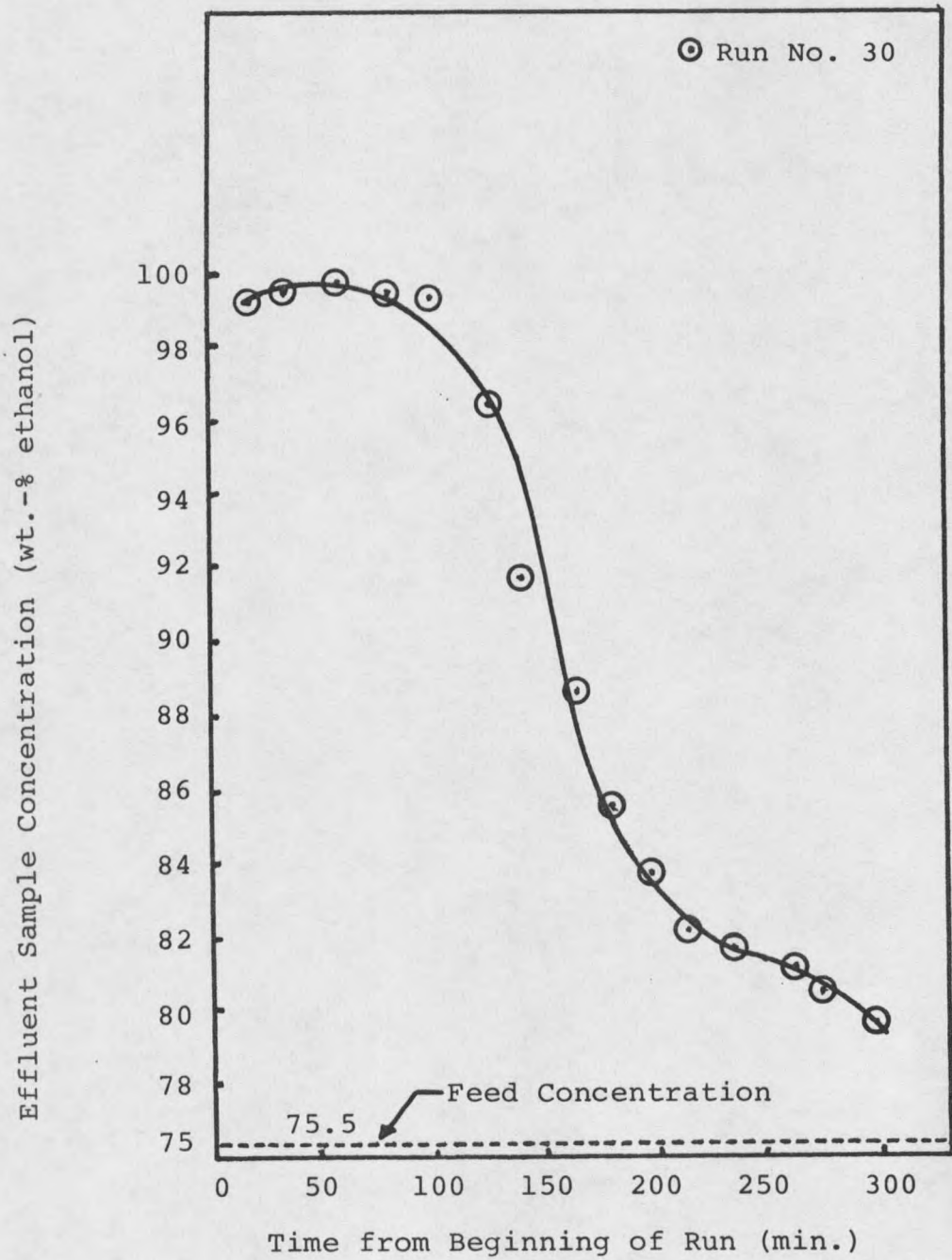


Figure 10. Effluent Concentration vs. Time

220 minutes. At this point, the effluent concentration was still 5 wt. % above the inlet feed concentration. A possible explanation for this is that water and ethanol were adsorbed onto the surface of the barley very quickly and then the adsorbed water and ethanol were slowly adsorbed into the cells of the barley, consequently making more surface available for further adsorption. According to this theory, water and ethanol would be removed very quickly from the feed stream by adsorption until all of the available barley surface was covered. At this point, the effluent ethanol concentration would decrease because water could no longer be removed from the feed at a fast enough rate. The slow adsorption process would become predominant and continue to make more surface available for adsorption. During this time, the adsorption process would occur at a much slower rate. Water would be removed from the feed stream at a rate just fast enough to keep the effluent ethanol concentration a few percentage points above that of the entering feed.

Since barley cells normally contain water and the barley was desiccated before each run to remove this water, it seems reasonable to assume that water would re-enter these cells during the run by the process described above. Young and Nelson (21) developed an

equation to describe isotherms for water sorption by wheat which was based on essentially the same ideas as were discussed above and they obtained very good correlation with the data from a number of investigators.

It should be noted that the thermocouples that were used to measure the barley temperature (4, 5, and 6) for all runs except Run No. 60, were inserted into thermowells which would tend to slow down the response time of the thermocouples.

The process of adsorption releases heat, as was discussed in the Introduction, and therefore causes the temperature of the barley to increase. Tables 63 and 64 (Appendix D) show the barley-temperature increases that occurred during a run with a low mass flux and a run with a high mass flux, respectively. Note that there was a much greater temperature increase in the barley (approximately  $10^{\circ}\text{C}$  vs.  $1^{\circ}\text{C}$ , as shown by temperatures 4, 5, and 6) during the run with the high mass flux than during the run with the low mass flux. This was because at the beginning of a run with a high mass flux, more water vapor per unit time was available for adsorption, the adsorption rate was faster, more heat was produced, and the temperature, therefore, increased to a higher value than for the low mass flux.

At this higher temperature, the barley could no longer retain as much water as it could when it was cooler and adsorbed water was released. Tables 12 and 17 (Appendix B) display that Sample 2 contained almost twice as much mass as Samples 1, 3, 4, or 5, even though Samples 3, 4, and 5 collected effluent for the same period of time as Sample 2, and Sample 1 collected effluent for a longer period of time. This phenomenon only occurred during runs with high mass fluxes and correspondingly high temperature increases. It, therefore, seems reasonable to assume that after the temperature increased, originally adsorbed water was released and became part of Sample 2. The results were similar for all high-mass-flux runs, but the two runs discussed displayed the phenomenon the best. The overall effect of high mass flux was to reduce the amount of water sorbed, as will be discussed later.

Run No. 60 was performed to find out if radial temperature gradients were present during runs. Tables 35 (Appendix B) and 65 (Appendix D) show the conditions and results of this run, respectively. The conditions used for this run gave the greatest temperature increases ( $23^{\circ}\text{C}$ ) of any conditions for any of the runs. Thermocouple 7 was placed in the center of the adsorber and about 5 cm. from the adsorber entrance. Thermocouple 6

was placed in the same axial position as Thermocouple 7, but next to the wall on the inside of the adsorber. It can be seen in Table 65 that the largest radial temperature difference at any time was 3°C. All other runs that were performed would have had this much, or less, depending upon the conditions used.

#### Effects of Temperature, Mass Flux, and Feed Composition

As mentioned in the Research Objective section, a central composite experimental design was set up to determine what conditions to use for each of Runs 26-50 (excluding 34 and 35). The two main quantities to be discussed here are: 1) the Grams of Water Sorbed per Gram of Dry Barley for a Combined Sample of 99 wt. % Ethanol, and 2) the Milliliters of Pure Ethanol Concentrated per Gram of Dry Barley for a Combined Sample of 99 wt. % Ethanol. A "Combined Sample of 99 wt. % Ethanol" has the following meaning. At the beginning of a run, the ethanol concentration of the effluent from the adsorber was usually above 99 wt. %. As water was removed from the feed stream, the barley in the adsorber would eventually become saturated and the ethanol concentration in the adsorber effluent would decrease. This concentration would drop below 99 wt. % ethanol and approach that of the feed to the adsorber. If all the

effluent from the adsorber was sent to the same container, the concentration in this container would start at 99+ wt. % ethanol, but it would begin to decrease as the barley became saturated and at some point would be exactly equal to 99 wt. % ethanol. If no more effluent were added to this container, it would then contain a "Combined Sample of 99 wt. % Ethanol." In this project, samples were collected for 2-20 minute time periods, depending on the feed mass flux, and these samples were "mathematically combined." In other words, the amount of ethanol and water was calculated for each sample from the concentration and sample weight, and the total number of samples that would have to be added together to give a "combined sample of 99 wt. % ethanol" was mathematically computed.

Figure 11 shows a plot of Grams of Water Sorbed per Gram of Grain for a Combined Sample of 99 wt. % Ethanol vs. Initial Temperature. Runs 26, 30, and 50 had a constant feed composition of 76.5 wt. % ethanol and feed mass flux of  $0.025 \text{ gram/minute-centimeter}^2$  ( $\text{gm./min.-cm.}^2$ ). The initial temperature was the only parameter that was varied. Runs 40, 41, 43, 44, and 46 had a different set of constant values: the feed mass flux for

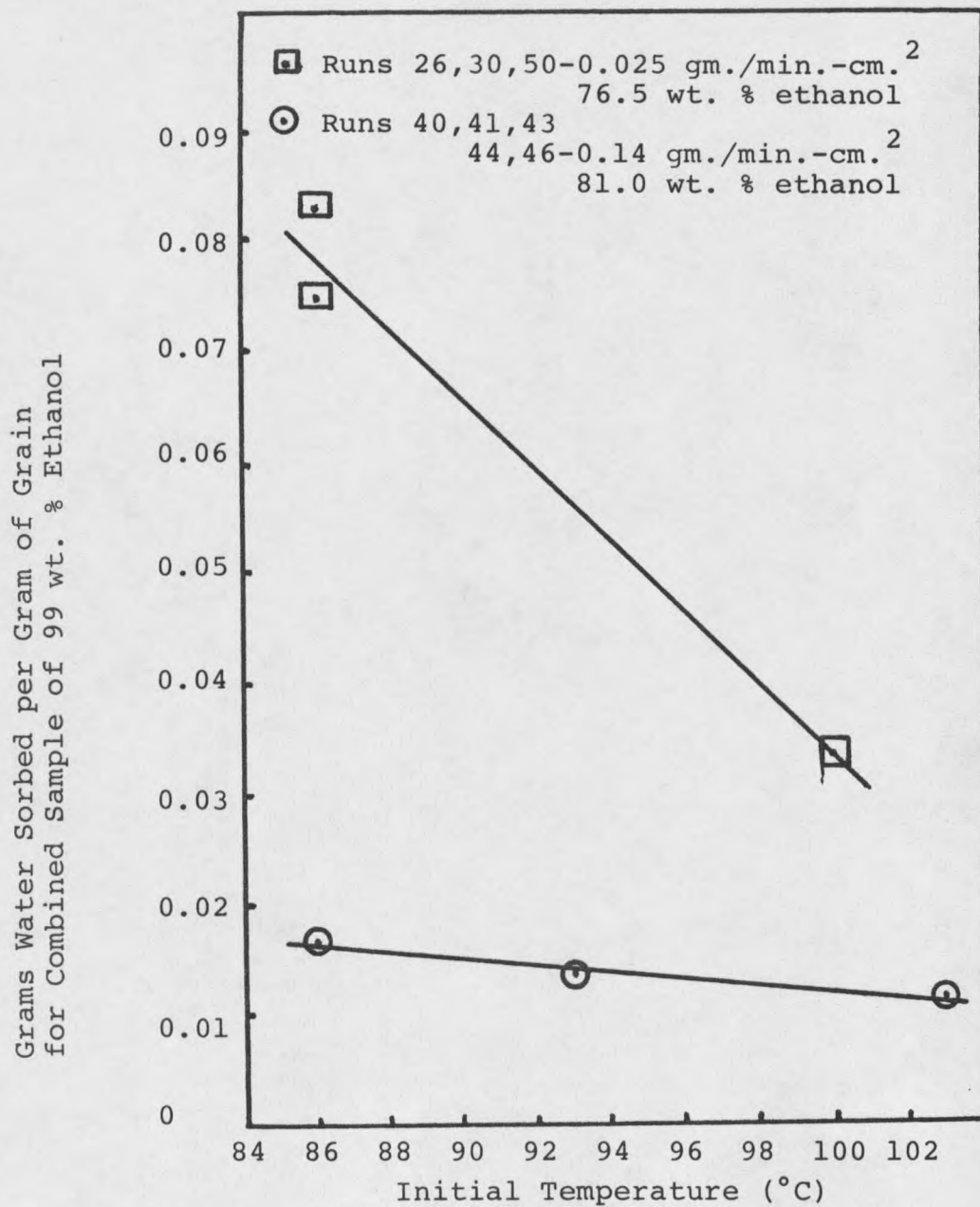


Figure 11. Amount of Water Sorbed  
vs. Initial Temperature

these runs was 0.14 gm./min.-sq. cm. and the feed composition was 81.0 wt. % ethanol. Here again, the only variable was the initial temperature of the grain. It can be seen that for both constant sets of feed mass flux and feed composition, the Gram of Water Sorbed per Gram of Grain for a Combined Sample of 99 wt. % Ethanol is inversely related to the initial temperature. This seems reasonable considering that as the temperature of a liquid increases, its vapor pressure increases, which seems likely to cause any adsorbed liquid to be released from the barley. Babbitt (45) showed that during desorption of water by wheat, the moisture content of the wheat was inversely related to the temperature.

Figure 12 shows a plot of Grams of Water Sorbed per Gram of Grain for a Combined Sample of 99 wt. % Ethanol vs. Feed Mass Flux. Here, Runs 26, 28, and 50 were held constant at an initial temperature of 86°C and feed composition of ~77.0 wt. % ethanol while the feed mass flux was varied and Runs 41, 45, and 47 were held constant at an initial temperature of 93°C and feed composition of ~81.5 wt. % ethanol while the feed mass flux was varied. For the exact conditions used for these

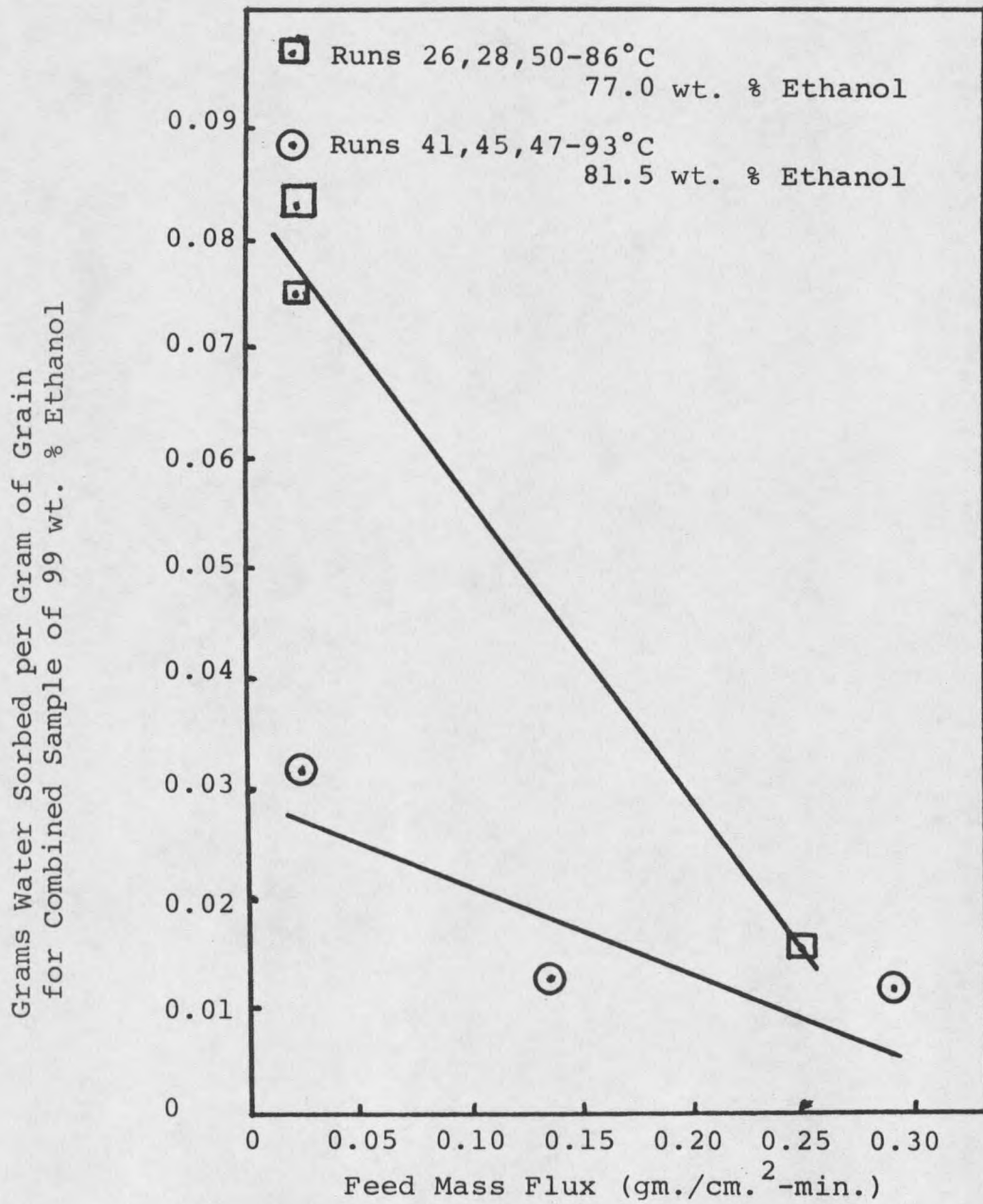


Figure 12. Amount of Water Sorbed vs. Feed Mass Flux

runs, see Appendix B. Note here that for the conditions used, the amount of water sorbed is inversely related to the feed mass flux. In earlier discussions (see pages 58 and 59), it was noted that an increase in feed flow decreases the amount of sorbed water. It would seem that this would explain the main process in which feed mass flux affects the amount of water sorbed.

An additional explanation might be found from the previous discussion on fixed-bed adsorbers. For a high mass flux, the mass transfer zone would be wider and when breakthrough occurred, there would be a higher percentage of unsaturated barley and, therefore, less water would be sorbed before the effluent concentration began to decrease. This would give a lower value of the Gram of Water Sorbed per Gram of Grain for a Combined Sample of 99 wt. % Ethanol.

Figure 13 shows a plot of Grams of Water Sorbed per Gram of Grain for a Combined Sample of 99 wt. % Ethanol vs. Ethanol Feed Concentration. Here, Runs 26, 37, 50, and 53 were held constant at an initial temperature of 86°C and Feed Mass Flux of 0.025 gm./min.-sq. cm. while the ethanol feed concentration was varied and Runs 46, 48, and

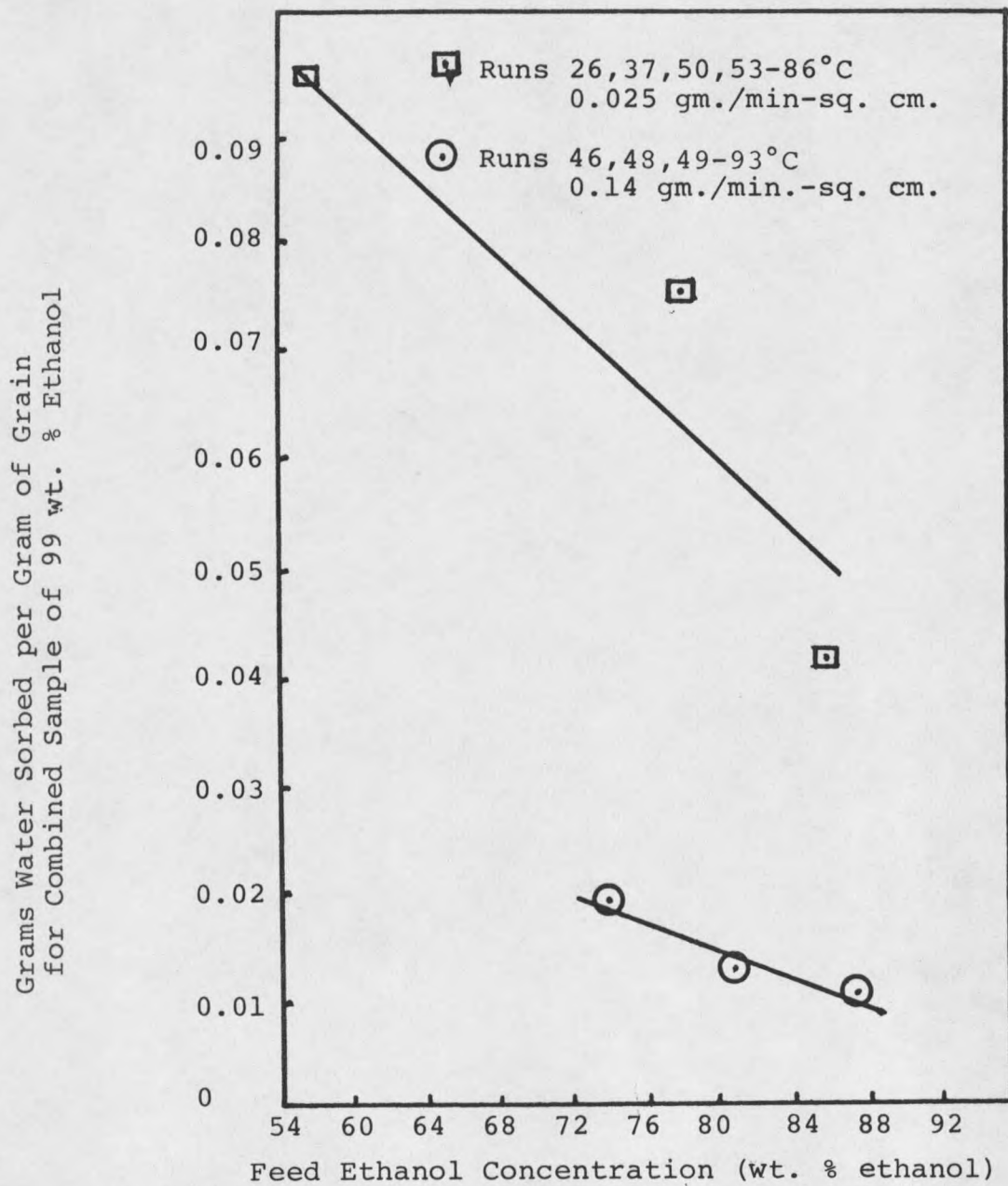


Figure 13. Amount of Water Sorbed vs.  
Feed Ethanol Concentration

49 were held constant at an initial temperature of 93°C and feed mass flux of 0.14 gm./min.-sq. cm. while the ethanol feed concentration was varied. Note the exact conditions used for these runs in Appendix B. Although Run 53 was not part of the central composite design, it was used here in order to show how feed ethanol concentrations that were lower than 75 wt. % ethanol affected the amount of water sorbed. It can be seen here that the amount of water sorbed is inversely related to the ethanol feed concentration. This would seem to be reasonable since a decrease in the ethanol feed concentration corresponds to an increase in water feed concentration. Therefore, the amount of water sorbed is actually directly related to the water feed concentration. Since the concentration of water in the feed is the main driving force for sorption of the water, it would seem reasonable that an increase in the water concentration in the feed would increase the amount of water sorbed. Studies done by Sair and Fetzer (38) demonstrated that as the relative humidity of air increased at constant temperature and atmospheric pressure, the amount of water sorbed increased for corn, potato, arrowroot, and cotton. If ethanol is considered as the

water-carrying medium instead of air, a similarity can be seen between the experiments of Sair and Fetzer and the experiments presented in this paper.

Data from Runs 26-50 (excluding 34 and 35) was used to develop two correlations for the Milliliters of Pure Ethanol Concentrated per Gram of Barley for Combined Samples of 99 wt. % Ethanol with respect to initial temperature, ethanol feed concentration and feed mass flux. Feed ethanol concentration was found to have a F-value of well below 2.99 which is the minimum F-value for a variable to have any effect within 90% confidence. See Tables 66 and 67 (Appendix F) for specific values. Ethanol feed concentration was therefore not included in the correlations. Both correlations used the same equation, but with different constants. The first correlation was developed using a Step-wise Forward Elimination statistical method which is one of the statistics packages developed by Dr. Lund that is in the M.S.U. computer library. (See Appendix F):

$$C = 0.0411 / ((T-80)^{0.2603} F_n^{0.6282}) \quad (\text{Eqn. 4})$$

The second correlation was developed using an optimization technique called the Complex Method of Box (See Appendix F):

(70)

$$C = 0.0729 / ((T-80)^{0.529} F_n^{0.6439}) \quad (\text{Eqn. 5})$$

C = milliliters of ethanol concentrated  
per gram of barley for a combined  
sample of 99 wt.-% ethanol

T = initial temperature (°C)

$F_n$  = feed mass flux (gm./min.-cm<sup>2</sup>)

The R-squared value for the statistically derived correlation is 0.879 (See Table 7.0 in Appendix F for details). A least squares technique was used by both methods in order to find the value of the 3 constants. The equation used was:

$$OBJ = \sum_{i=1}^n (EXC_i - CALCC_i)^2 \quad (\text{Eqn. 6})$$

n = number of experimental points

i = the ith experimental point

OBJ = the total sum of the least squares

EXC = the experimental value of C

CALCC = the calculated value of C

The value of OBJ for the statistically derived equation was 0.0169. The value of OBJ for the optimization technique was 0.0084. This would tend to suggest that the optimization technique produced a better correlation than the statistical method. A comparison of the

experimental value of C and the calculated values of C for both correlations can be seen in Table 3.

Both correlations show that the amount of ethanol concentrated had an inverse relationship with respect to the initial temperature and the feed mass flux. During earlier discussions, it was noted that the amount of water sorbed was also inversely related to the initial temperature and the feed mass flux. Since the process by which the ethanol was concentrated was by having water removed from the feed stream, it seems reasonable that the amount of ethanol concentrated should have a similar relationship to the initial temperature and feed mass flux as the amount of water sorbed does.

A probable explanation for why the feed composition had no effect on the amount of ethanol concentrated for the experiments performed is that as the feed ethanol concentration increased, there was less water that needed to be removed in order to concentrate the same amount of ethanol. During earlier discussions, it was noted that as the feed ethanol concentration increased, the amount of water sorbed decreased. Therefore, as the feed ethanol concentration increased, the amount of water

Table 3. Experimental and Calculated Values of C  
 (C = ml. of ethanol concentrated per gm.  
 of barley for a combined sample of  
 99 wt. % ethanol)

| Run<br>No. | Experimental<br>Value of<br>C | Calculated<br>Optimization<br>Value of C<br>(Eqn. 5) | Calculated<br>Statistical<br>Value of C<br>(Eqn. 4) |
|------------|-------------------------------|------------------------------------------------------|-----------------------------------------------------|
| 26         | 0.3260                        | 0.3081                                               | 0.2616                                              |
| 28         | 0.0555                        | 0.0699                                               | 0.0616                                              |
| 29         | 0.0302                        | 0.0373                                               | 0.0450                                              |
| 30         | 0.1320                        | 0.1645                                               | 0.1911                                              |
| 33         | 0.0602                        | 0.0373                                               | 0.0450                                              |
| 36         | 0.0405                        | 0.0699                                               | 0.0615                                              |
| 37         | 0.3180                        | 0.3081                                               | 0.2616                                              |
| 38         | 0.2020                        | 0.1645                                               | 0.1911                                              |
| 39         | 0.0728                        | 0.0373                                               | 0.0450                                              |
| 40         | 0.0708                        | 0.0679                                               | 0.0724                                              |
| 41         | 0.0725                        | 0.0679                                               | 0.0724                                              |
| 43         | 0.0587                        | 0.0504                                               | 0.0625                                              |
| 44         | 0.0842                        | 0.1016                                               | 0.0886                                              |
| 45         | 0.0528                        | 0.0424                                               | 0.0459                                              |
| 46         | 0.0721                        | 0.0679                                               | 0.0724                                              |
| 47         | 0.1690                        | 0.2059                                               | 0.2137                                              |
| 48         | 0.0640                        | 0.0679                                               | 0.0724                                              |
| 49         | 0.0942                        | 0.0679                                               | 0.0724                                              |
| 50         | 0.3020                        | 0.3081                                               | 0.2616                                              |

sorbed decreased, but the amount of water that needed to be sorbed also decreased. It would appear, then, that these two effects cancelled each other out.

#### Effect of Grain Particle Size

Figures 14 and 15 show the effect that grain particle size had on the amount of ethanol concentrated and the amount of water sorbed. For all three runs, the initial temperature was 86°C, the feed mass flux was 0.025 gm./min.-cm<sup>2</sup>, and the feed concentration was about 76 wt. % ethanol. It can be seen that the largest particle size did not work as well as the two smaller particle sizes. This seems reasonable since the largest particle size would not have had as much surface area for adsorption of water as the smaller particles did.

It can be seen that Runs 56 and 57 (see Figures 14 and 15) had similar results. One possibility is that the amount of water sorbed and amount of ethanol concentrated are not dependent upon the barley particle size with particles smaller than 0.833 mm. Another possibility is that the data for these two runs contains too much experimental error to make any conclusions with respect to particle size for this particle size range.

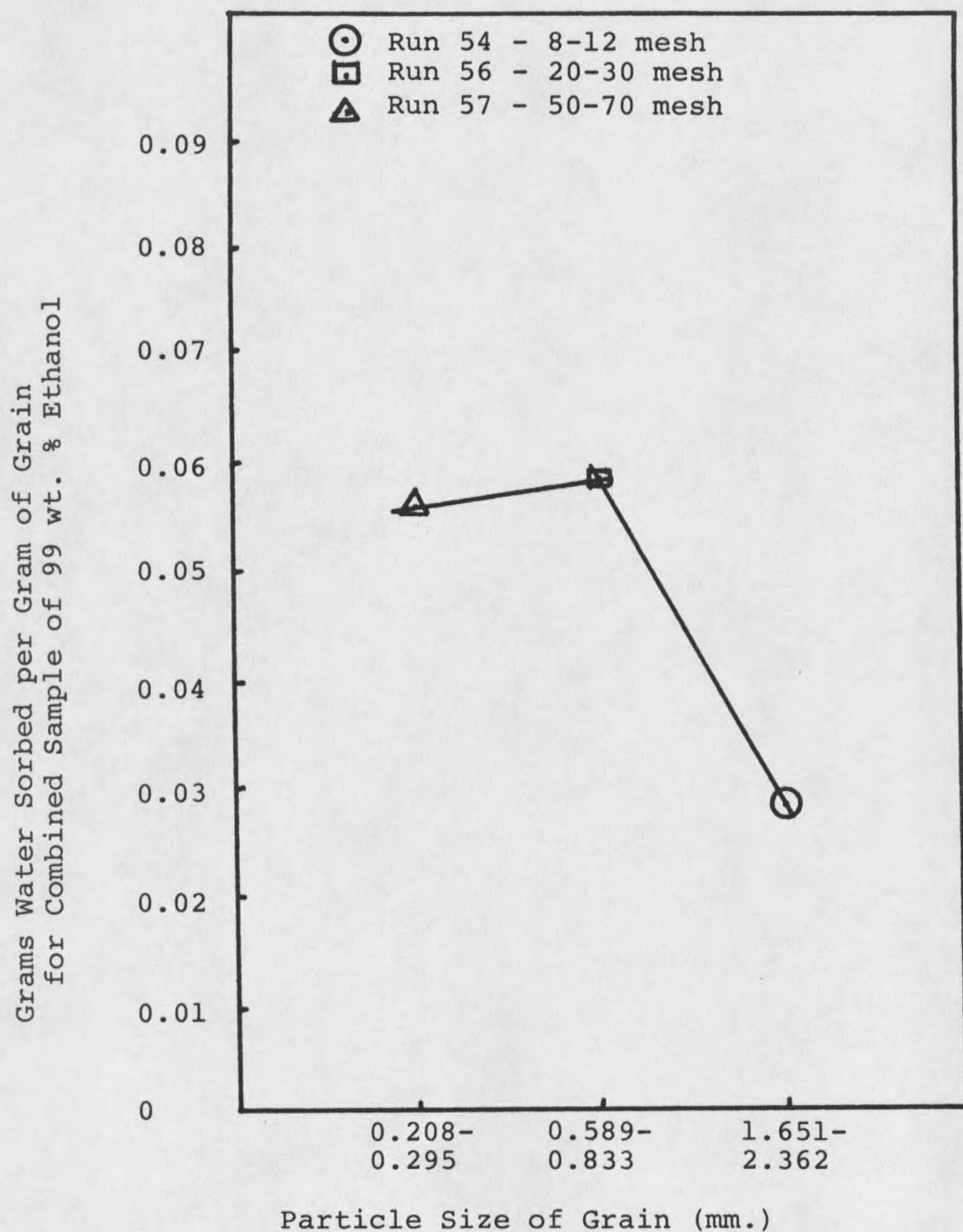


Figure 14. Amount of Water Sorbed vs. Grain Particle Size

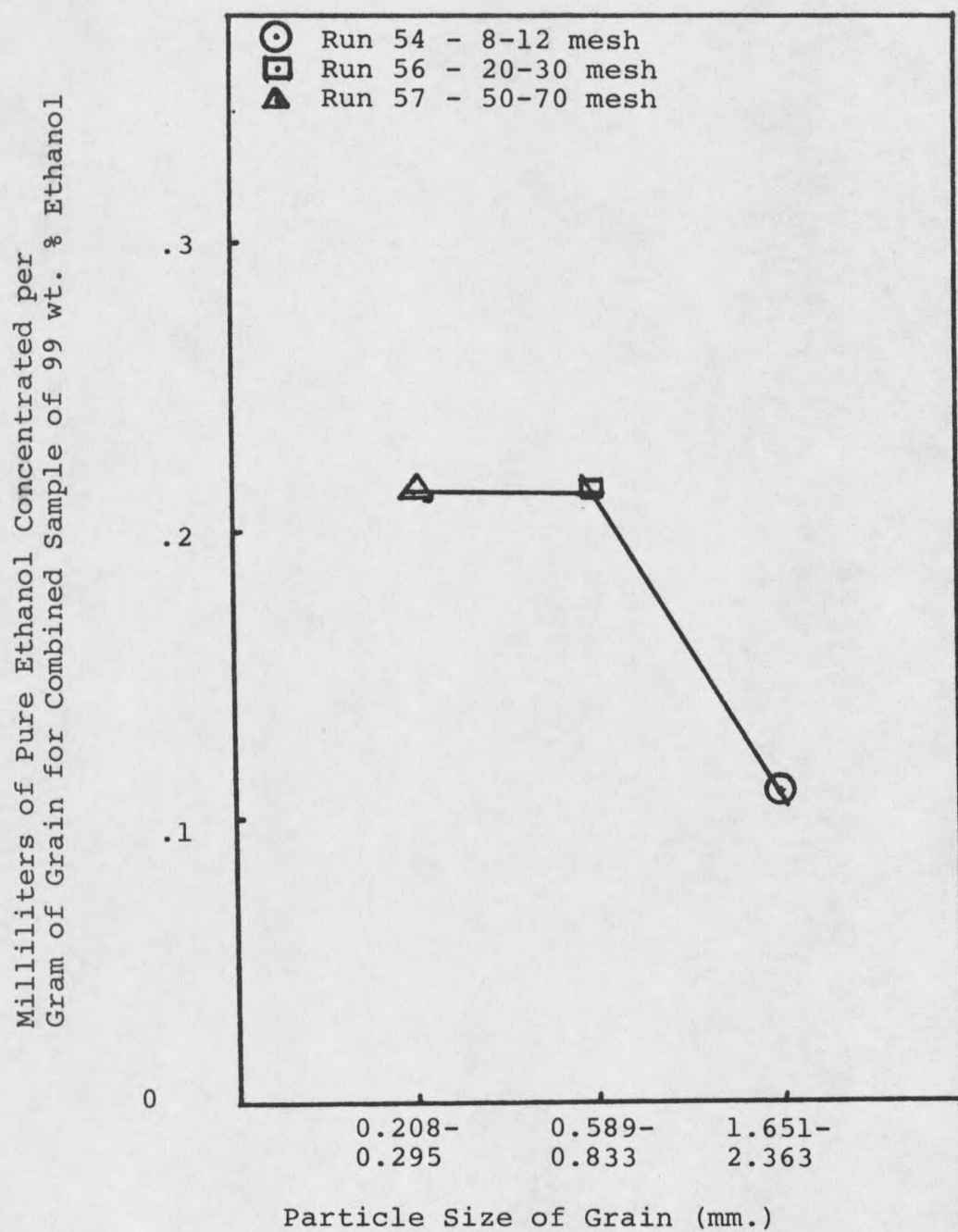


Figure 15. Amount of Ethanol Concentration vs. Grain Particle Size

### Effect of Initial Moisture Content of Grain

Figures 16 and 17 show the effect that the initial moisture content of the grain had on the amount of ethanol concentrated and the amount of water sorbed. For all three runs, the initial temperature was 86°C, the feed mass flux was 0.025 gm./min.-cm.<sup>2</sup>, and the feed composition was about 76 wt. % ethanol. It can be seen that as the initial moisture content was increased, the amount of ethanol concentrated and the amount of water sorbed decreased. This seems reasonable, since the barley will only sorb a specific amount of water for a given set of conditions. If there was water in the barley before the run began, there was that much less room in the barley for additional water.

### Effects of Grain Regeneration

Grain used for Run No. 53 was re-dried and used again for Run No. 55. For both runs, the initial temperature was 86°C, the feed mass flux was about 0.026 gm./min.-cm.<sup>2</sup>, and the feed composition was about 55 wt. % ethanol. Run No. 53 had 0.0967 gm. water sorbed per gram of grain while Run No. 55 had 0.0999 gm. water sorbed per gram of grain. This contradicts the results of Sair and Fetzer (38) in which they found that the

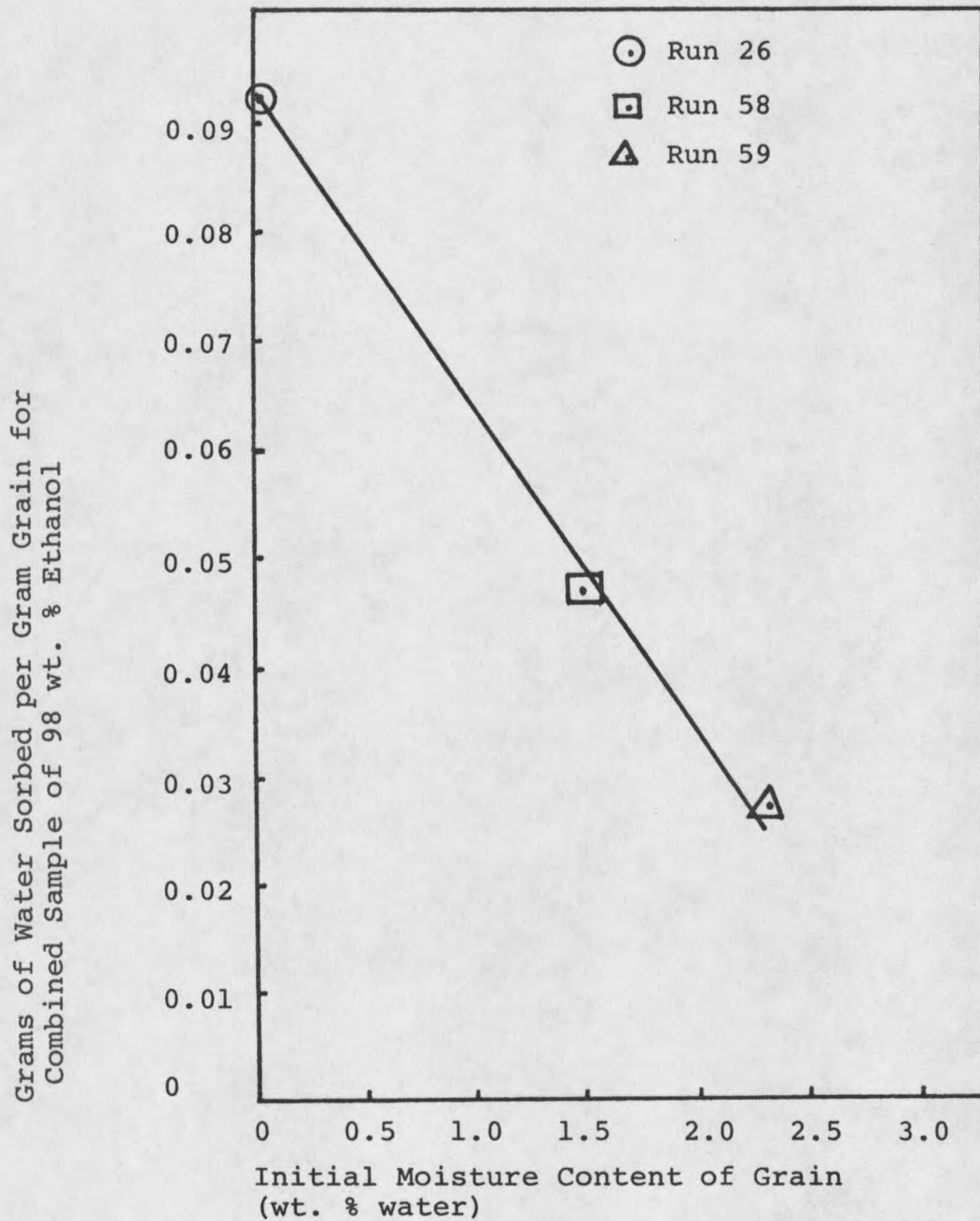


Figure 16. Amount of Water Sorbed vs.  
Initial Moisture Content of  
Grain

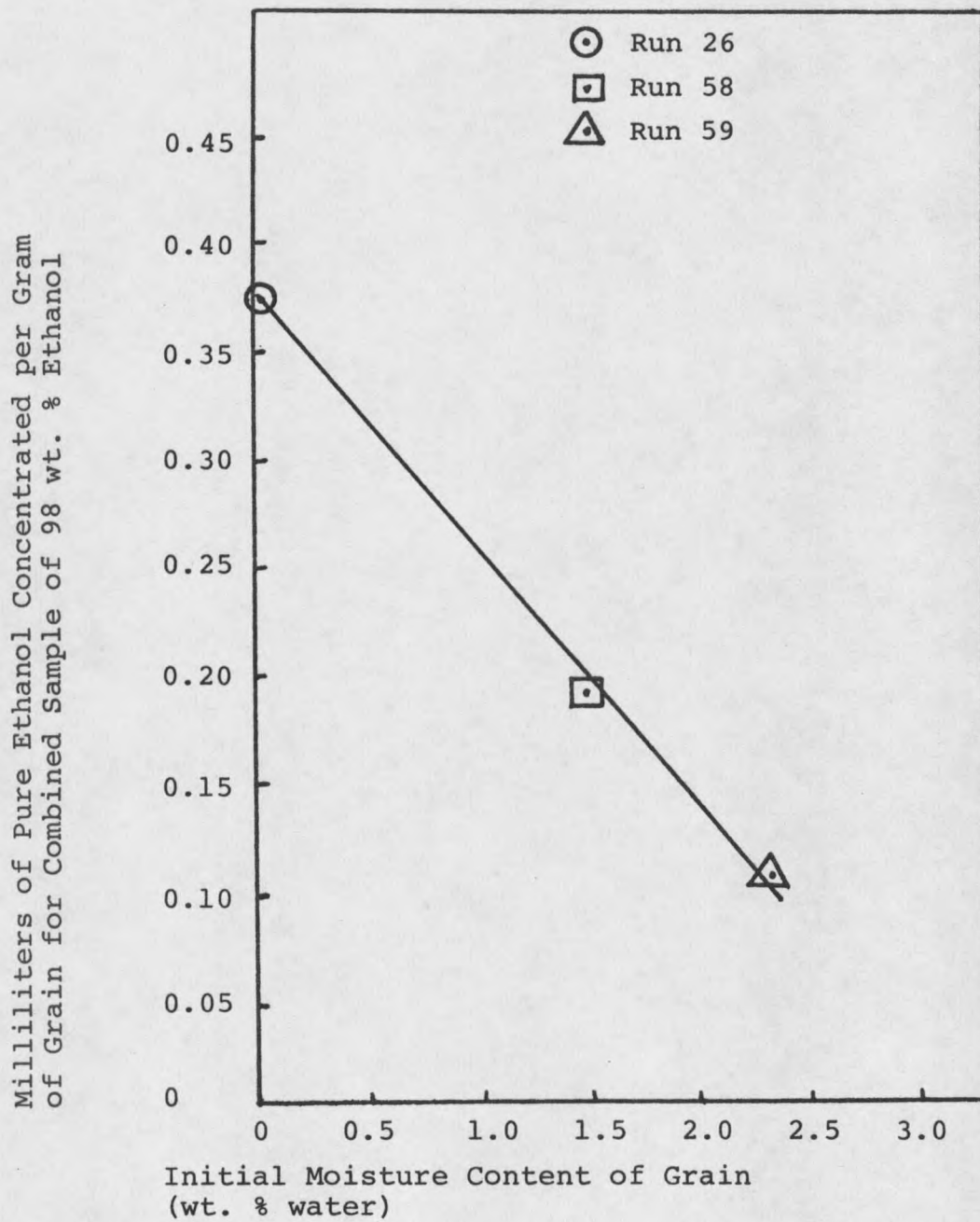


Figure 17. Amount of Ethanol Concentrated vs. Initial Moisture Content of Grain

sorptive ability of starches decreased when starches were dried at temperatures between 100°C and 150°C. There were two factors that were different between the Sair and Fetzer results and the results presented in this paper. Sair and Fetzer desiccated their starches in vacuo, whereas the grains used in the runs of this paper were not. Secondly, Sair and Fetzer compared starch that had not been desiccated with starch that had undergone 100 hours of desiccation. The comparison done in this paper was such that the grain for Run No. 53 was desiccated for a first period of 96 hours at 120°C and then desiccated again, under the same conditions, for Run No. 55.

One possibility may be that the initial desiccation decreases the sorptive ability of the grain, but further desiccation has no effect. Additional studies would be necessary in order to more completely determine the effect of repeated desiccation periods.

#### Comparison of Grain and Straw

Tables 41 and 43 (Appendix C) show a comparison between using dried barley straw for Run 35 and dried grain for Run 37 as the adsorbent. For both runs, the initial temperature was 86°C, the feed mass flux was 0.025 gm./min.-cm.<sup>2</sup>, and the feed concentration was

about 84% ethanol. The straw (variety BBIN) was able to sorb 0.0290 gram water per gram straw for a combined sample of 99 wt. % ethanol while the Shabet grain had a corresponding value of 0.0432. The initial temperature of the adsorbent was 86°C for both runs and the feed mass flux was 0.025 gm./min.-cm.<sup>2</sup> for both runs. The feed concentration for Run 35 with straw was 83.0 wt. % ethanol while it was 85.5 wt. % ethanol for Run 37 with grain. If these concentrations had been the same, the difference in results would have been even greater (see the discussion on pages 65-67). Urquhart and Williams (32) showed that cellulose hygroscopicity had very little variability with respect to its origin (what plant it was from). Therefore, even though the barley straw, which is largely cellulose, did not originate from the same barley species that the grain did, it seems reasonable to assume that Shabet barley grain (which was used in Run 26) would give much better results than Shabet straw would.

### CONCLUSIONS

Within the range of conditions reported here, it is therefore concluded that:

- 1) Absorption, in addition to adsorption, of water vapor by grain occurs when ethanol and water vapors are passed through the grain.
- 2) Ethanol vapor, in addition to water vapor is sorbed by grain, but to a lesser degree.
- 3) The sorption of water vapor by grain releases heat which increases the temperature of the grain and since the rate of sorption is related to the feed mass flux, the temperature rise is directly related to the feed mass flux.
- 4) The amount of water sorbed by grain is inversely related to the initial grain temperature, feed mass flux, and ethanol feed concentration.
- 5) The amount of ethanol concentrated by the grain is inversely related to the initial grain temperature of the feed mass flux.
- 6) Over the range of 73-87 wt. % ethanol, the ethanol feed concentration has no effect on the amount of ethanol concentrated by the grain.
- 7) Between grain particle size ranges of 0.589-0.833 and 1.651-2.362 millimeters, the amount of ethanol concentrated and

amount of water sorbed is inversely related to the particle size, whereas there is little difference between smaller size ranges (between 0.208-0.295 and 0.589-0.833 millimeters).

- 8) The amount of water sorbed and amount of ethanol concentrated is inversely related to the initial grain moisture content.
- 9) The single redrying of the grain appears to slightly increase its sorptive capacity as compared to the initial capacity of fresh grain.
- 10) Grain has a much higher sorptive capacity than straw.

RECOMMENDATIONS FOR FUTURE RESEARCH

The following are suggestions for future research:

- 1) Experimentation with conditions outside of the range of conditions discussed in this paper (different grain types, particle sizes, equipment sizes, etc.).
- 2) Combination of a distillation of adsorption column and optimization of the relative equipment sizes.

(84)

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Literature Cited

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## APPENDICES

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APPENDIX A

PURPOSE OF RUNS

Table 4. Purpose of Runs 1-17

| Run No. | Purpose of Run        | Comments                                                        |
|---------|-----------------------|-----------------------------------------------------------------|
| 1       | equipment development |                                                                 |
| 2       | "                     |                                                                 |
| 3       | "                     |                                                                 |
| 4       | "                     |                                                                 |
| 5       | "                     |                                                                 |
| 6       | "                     |                                                                 |
| 7       | "                     | no separation                                                   |
| 8       | "                     | "                                                               |
| 9       | "                     | "                                                               |
| 10      | "                     | "                                                               |
| 11      | "                     | "                                                               |
| 12      | "                     | "                                                               |
| 13      | "                     | "                                                               |
| 14      | "                     | eth-H <sub>2</sub> O separated, no individual samples collected |
| 15      | "                     | flow rate too high, temp. too low                               |
| 16      | "                     | effluent eth. concentration too low                             |
| 17      | "                     | "                                                               |

Table 5. Purpose of Runs 18-33

| Run No. | Purpose of Run           | Comments                            |
|---------|--------------------------|-------------------------------------|
| 18      | equipment development    | effluent eth. concentration too low |
| 19      | "                        | total mass not accounted for        |
| 20      | "                        | post-run barley moisture incorrect  |
| 21      | "                        | feed condensed                      |
| 22      | "                        | temp. too low                       |
| 23      | "                        | "                                   |
| 24      | "                        | "                                   |
| 25      | "                        | "                                   |
| 26      | central composite design | obtained usable data                |
| 27      | "                        | effluent eth. concentration too low |
| 28      | "                        | obtained usable data                |
| 29      | "                        | "                                   |
| 30      | "                        | "                                   |
| 31      | "                        | feed temp. varied too much          |
| 32      | "                        | incomplete data collection          |
| 33      | "                        | obtained usable data                |

Table 6. Purpose of Runs 34-50

| Run No. | Purpose of Run           | Comments                                          |
|---------|--------------------------|---------------------------------------------------|
| 34      | test barley straw        | undried straw no eth.-H <sub>2</sub> O separation |
| 35      | "                        | dried straw eth.-H <sub>2</sub> O separated       |
| 36      | central composite design | obtained usable data                              |
| 37      | "                        | "                                                 |
| 38      | "                        | "                                                 |
| 39      | "                        | "                                                 |
| 40      | "                        | "                                                 |
| 41      | "                        | "                                                 |
| 42      | "                        | pump malfunctioned                                |
| 43      | "                        | obtained usable data                              |
| 44      | "                        | "                                                 |
| 45      | "                        | "                                                 |
| 46      | "                        | "                                                 |
| 47      | "                        | "                                                 |
| 48      | "                        | "                                                 |
| 49      | "                        | "                                                 |
| 50      | "                        | "                                                 |

Table 7. Purpose of Runs 51-60

| Run No. | Purpose of Run                   | Comments                     |
|---------|----------------------------------|------------------------------|
| 51      | feed concentration effects       | Temp. recorder malfunctioned |
| 52      | "                                | feed condensed               |
| 53      | "                                | usable data obtained         |
| 54      | particle size effects            | "                            |
| 55      | regeneration of barley (run #53) | "                            |
| 56      | particle size effects            | "                            |
| 57      | "                                | "                            |
| 58      | pre-run grain moisture           | "                            |
| 59      | "                                | "                            |
| 60      | radial temp. gradients           | "                            |

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## APPENDIX B

### RUN CONDITIONS AND DATA

Table 8. Run No. 26 Conditions and Data

Date: 3-11-81

type of barley ..... Shabet grain

initial moisture content ..... 0 wt. % water

size distribution ..... See Table 1

feed concentration ..... 76.5 wt. % eth.

feed mass flux ..... 0.025 gm./min.-cm<sup>2</sup>

weight of barley used ..... 60.0 gm.

feed additions ..... 40.3 ml.

initial barley temperature ..... 86 °C

top          bottom

post run barley moisture content .. 9.3          16.1 wt. %

| SAMPLE<br>NUMBER | TIME<br>(MIN.) | BURETTE<br>(ML.) | EFFLUENT<br>SAMPLE<br>WT. (GM.) | EFFLUENT<br>% ETHANOL<br>(WT. %) |
|------------------|----------------|------------------|---------------------------------|----------------------------------|
| 1                | 20             | 2.0              | 0.49                            | 99.8                             |
| 2                | 40             | 4.1              | 0.85                            | 99.9                             |
| 3                | 60             | 6.1              | 1.03                            | 99.9                             |
| 4                | 80             | 8.1              | 1.09                            | 99.9                             |
| 5                | 100            | 10.2             | 1.12                            | 99.9                             |
| 6                | 120            | 12.2             | 1.02                            | 99.9                             |
| 7                | 140            | 14.2             | 1.08                            | 99.9                             |
| 8                | 160            | 16.2             | 1.18                            | 99.9                             |
| 9                | 180            | 18.3             | 0.99                            | 99.9                             |
| 10               | 200            | 20.3             | 1.11                            | 99.9                             |
| 11               | 220            | 22.3             | 1.13                            | 99.9                             |
| 12               | 240            | 24.3             | 1.20                            | 99.9                             |
| 13               | 260            | 26.3             | 1.20                            | 97.8                             |
| 14               | 280            | 28.3             | 1.19                            | 95.4                             |
| 15               | 300            | 30.2             | 1.20                            | 93.6                             |
| 16               | 320            | 32.2             | 1.30                            | 92.0                             |
| 17               | 340            | 34.2             | 1.26                            | 90.5                             |
| 18               | 360            | 36.2             | 1.34                            | 89.0                             |
| 19               | 380            | 38.2             | 1.28                            | 88.0                             |
| 20               | 400            | 40.2             | 1.34                            | 87.5                             |

Table 9. Run No. 28 Conditions and Data

Date: 3-20-81

type of barley ..... Shabet grain

initial moisture content ..... 0 wt. % water

size distribution ..... See Table 1.

feed concentration ..... 76.5 wt. % eth.

feed mass flux ..... 0.25 gm./min.-cm<sup>2</sup>

weight of barley used ..... 60.0 gm.

feed additions ..... 61.3 ml.

initial barley temperature ..... 86 °C

top      bottom

post run barley moisture content .. 6.6      9.2 wt. %

| SAMPLE<br>NUMBER | TIME<br>(MIN.) | BURETTE<br>(ML.) | EFFLUENT<br>SAMPLE<br>WT. (GM.) | EFFLUENT<br>% ETHANOL<br>(WT. %) |
|------------------|----------------|------------------|---------------------------------|----------------------------------|
| 1                | 4              | 4.0              | 2.41                            | 99.8                             |
| 2                | 10             | 9.9              | 3.91                            | 91.3                             |
| 3                | 15             | 14.9             | 3.57                            | 85.0                             |
| 4                | 20             | 19.9             | 3.82                            | 83.0                             |
| 5                | 25             | 24.9             | 3.83                            | 81.0                             |
| 6                | 30             | 29.9             | 3.82                            | 81.0                             |
| 7                | 35             | 35.0             | 3.86                            | 80.5                             |
| 8                | 40             | 40.0             | 3.86                            | 80.0                             |
| 9                | 45             | 45.1             | 3.93                            | 80.0                             |
| 10               | 50             | 50.1             | 4.02                            | 80.0                             |
| 11               | 55             | 55.2             | 3.86                            | 79.5                             |
| 12               | 60             | 60.3             | 4.12                            | 79.5                             |
| 13               |                |                  |                                 |                                  |
| 14               |                |                  |                                 |                                  |
| 15               |                |                  |                                 |                                  |
| 16               |                |                  |                                 |                                  |
| 17               |                |                  |                                 |                                  |
| 18               |                |                  |                                 |                                  |
| 19               |                |                  |                                 |                                  |
| 20               |                |                  |                                 |                                  |

Table 10. Run No. 29 Conditions and Data

Date: 3-23-81

type of barley ..... Shabet grain

initial moisture content ..... 0 wt. % water

size distribution ..... See Table 1

feed concentration ..... 75.5 wt. % eth.

feed mass flux ..... 0.25 gm./min.-cm<sup>2</sup>

weight of barley used ..... 60.0 gm.

feed additions ..... 60.8 ml.

initial barley temperature ..... 100 °C

top            bottom

post run barley moisture content .. 4.3            5.0 wt. %

| SAMPLE<br>NUMBER | TIME<br>(MIN.) | BURETTE<br>(ML.) | EFFLUENT<br>SAMPLE<br>WT. (GM.) | EFFLUENT<br>% ETHANOL<br>(WT. %) |
|------------------|----------------|------------------|---------------------------------|----------------------------------|
| 1                | 3.7            | 3.5              | 1.37                            | 99.6                             |
| 2                | 10             | 9.7              | 5.46                            | 87.5                             |
| 3                | 15             | 14.7             | 3.83                            | 82.0                             |
| 4                | 20             | 19.7             | 3.98                            | 80.0                             |
| 5                | 25             | 24.7             | 3.93                            | 79.5                             |
| 6                | 30             | 29.7             | 4.05                            | 78.0                             |
| 7                | 35             | 34.7             | 3.98                            | 78.0                             |
| 8                | 40             | 39.8             | 4.09                            | 78.0                             |
| 9                | 45             | 44.8             | 4.03                            | 77.5                             |
| 10               | 50             | 49.8             | 4.13                            | 77.0                             |
| 11               | 55             | 54.8             | 4.08                            | 76.5                             |
| 12               | 60             | 59.8             | 4.12                            | 77.0                             |
| 13               |                |                  |                                 |                                  |
| 14               |                |                  |                                 |                                  |
| 15               |                |                  |                                 |                                  |
| 16               |                |                  |                                 |                                  |
| 17               |                |                  |                                 |                                  |
| 18               |                |                  |                                 |                                  |
| 19               |                |                  |                                 |                                  |
| 20               |                |                  |                                 |                                  |

Table 11. Run No. 30 Conditions and Data

Date: 3-24-81

type of barley ..... Shabet grain

initial moisture content ..... 0 wt. % water

size distribution ..... See Table 1

feed concentration ..... 75.5 wt. % eth.

feed mass flux ..... 0.025 gm./min.-cm<sup>2</sup>

weight of barley used ..... 60.0 gm.

feed additions ..... 29.8 ml.

initial barley temperature ..... 100 °C

top            bottom

post run barley moisture content... 6.1            6.3 wt. %

| SAMPLE<br>NUMBER | TIME<br>(MIN.) | BURETTE<br>(ML.) | EFFLUENT<br>SAMPLE<br>WT. (GM.) | EFFLUENT<br>% ETHANOL<br>(WT. %) |
|------------------|----------------|------------------|---------------------------------|----------------------------------|
| 1                | 20             | 1.4              | 0.21                            | 99.2                             |
| 2                | 40             | 3.5              | 1.05                            | 99.6                             |
| 3                | 60             | 5.7              | 1.53                            | 99.8                             |
| 4                | 80             | 7.7              | 1.15                            | 99.7                             |
| 5                | 100            | 9.8              | 1.15                            | 99.5                             |
| 6                | 120            | 11.8             | 1.15                            | 96.6                             |
| 7                | 140            | 13.8             | 1.22                            | 91.7                             |
| 8                | 160            | 15.8             | 1.42                            | 88.5                             |
| 9                | 180            | 17.8             | 1.32                            | 85.5                             |
| 10               | 200            | 19.8             | 1.39                            | 83.5                             |
| 11               | 220            | 21.8             | 1.36                            | 82.0                             |
| 12               | 240            | 23.8             | 1.49                            | 81.5                             |
| 13               | 260            | 25.7             | 1.44                            | 81.0                             |
| 14               | 280            | 27.7             | 1.53                            | 80.5                             |
| 15               | 300            | 29.7             | 1.44                            | 79.5                             |
| 16               |                |                  |                                 |                                  |
| 17               |                |                  |                                 |                                  |
| 18               |                |                  |                                 |                                  |
| 19               |                |                  |                                 |                                  |
| 20               |                |                  |                                 |                                  |

Table 12. Run No. 33 Conditions and Data

Date: 4-9-81

type of barley ..... Shabet grain

initial moisture content ..... 0 wt. % water

size distribution ..... See Table 1

feed concentration ..... 82.5 wt. % eth.

feed mass flux ..... 0.25 gm./min.-cm<sup>2</sup>

weight of barley used ..... 60.0 gm.

feed additions ..... 86.1 ml.

initial barley temperature ..... 100 °C

top      bottom

post run barley moisture content .. 4.4      5.5 wt. %

| SAMPLE<br>NUMBER | TIME<br>(MIN.) | BURETTE<br>(ML.) | EFFLUENT<br>SAMPLE<br>WT. (GM.) | EFFLUENT<br>% ETHANOL<br>(WT. %) |
|------------------|----------------|------------------|---------------------------------|----------------------------------|
| 1                | 2              | 2.0              | 0.54                            | 99.5                             |
| 2                | 4              | 3.9              | 2.07                            | 99.4                             |
| 3                | 6              | 5.8              | 1.26                            | 94.9                             |
| 4                | 8              | 7.8              | 1.39                            | 90.3                             |
| 5                | 10             | 9.7              | 1.38                            | 89.0                             |
| 6                | 15             | 14.6             | 3.74                            | 86.5                             |
| 7                | 20             | 19.6             | 3.77                            | 86.0                             |
| 8                | 25             | 24.6             | 3.90                            | 85.0                             |
| 9                | 30             | 29.6             | 3.97                            | 84.5                             |
| 10               | 35             | 34.7             | 3.99                            | 84.0                             |
| 11               | 40             | 39.7             | 3.97                            | 83.5                             |
| 12               | 45             | 44.8             | 3.99                            | 83.5                             |
| 13               | 50             | 49.9             | 3.97                            | 83.5                             |
| 14               | 55             | 54.9             | 4.02                            | 83.5                             |
| 15               | 60             | 59.9             | 3.94                            | 83.0                             |
| 16               | 65             | 65.0             | 4.00                            | 83.5                             |
| 17               | 70             | 70.0             | 3.99                            | 83.5                             |
| 18               | 75             | 75.0             | 4.03                            | 83.5                             |
| 19               | 80             | 80.1             | 4.02                            | 83.5                             |
| 20               | 85             | 85.1             | 4.09                            | 83.0                             |

Table 13. Run No. 35 Conditions and Data

Date: 4-17-81

type of barley ..... Straw BBIN

initial moisture content ..... 0      wt. % water

size distribution ..... < 1 mm.

feed concentration ..... 83.0 wt. % eth.

feed mass flux ..... 0.025 gm./min.-cm<sup>2</sup>

weight of barley used ..... 14.1 gm.

feed additions ..... 7.7 ml.

initial barley temperature ..... 86 °C

top      bottom

post run barley moisture content .. 7.3      9.5   wt. %

| SAMPLE<br>NUMBER | TIME<br>(MIN.) | BURETTE<br>(ML.) | EFFLUENT<br>SAMPLE<br>WT. (GM.) | EFFLUENT<br>% ETHANOL<br>(WT. %) |
|------------------|----------------|------------------|---------------------------------|----------------------------------|
| 1                | 5              | 0.5              | 0.00                            | --                               |
| 2                | 10             | 1.0              | 0.02                            | --                               |
| 3                | 15             | 1.5              | 0.30                            | 99.3                             |
| 4                | 20             | 2.1              | 0.26                            | 99.4                             |
| 5                | 40             | 3.9              | 1.04                            | 99.3                             |
| 6                | 60             | 5.6              | 0.99                            | 97.9                             |
| 7                | 80             | 7.6              | 1.42                            | 90.8                             |
| 8                |                |                  |                                 |                                  |
| 9                |                |                  |                                 |                                  |
| 10               |                |                  |                                 |                                  |
| 11               |                |                  |                                 |                                  |
| 12               |                |                  |                                 |                                  |
| 13               |                |                  |                                 |                                  |
| 14               |                |                  |                                 |                                  |
| 15               |                |                  |                                 |                                  |
| 16               |                |                  |                                 |                                  |
| 17               |                |                  |                                 |                                  |
| 18               |                |                  |                                 |                                  |
| 19               |                |                  |                                 |                                  |
| 20               |                |                  |                                 |                                  |

Table 14. Run No. 36 Conditions and Data

Date: 4-24-81

type of barley ..... Shabet grain

initial moisture content ..... 0.1 wt. % water

size distribution ..... See Table 1

feed concentration ..... 86.0 wt. % eth.

feed mass flux ..... 0.25 gm./min.-cm<sup>2</sup>

weight of barley used ..... 60.0 gm.

feed additions ..... 60.2 ml.

initial barley temperature ..... 86 °C

top      bottom

post run barley moisture content .. 4.8      7.6 wt. %

| SAMPLE<br>NUMBER | TIME<br>(MIN.) | BURETTE<br>(ML.) | EFFLUENT<br>SAMPLE<br>WT. (GM.) | EFFLUENT<br>% ETHANOL<br>(WT. %) |
|------------------|----------------|------------------|---------------------------------|----------------------------------|
| 1                | 2.8            | 2.8              | 0.97                            | 99.7                             |
| 2                | 9.5            | 9.6              | 5.32                            | 98.3                             |
| 3                | 15             | 15.0             | 3.79                            | 92.6                             |
| 4                | 20             | 20.0             | 3.52                            | 90.9                             |
| 5                | 25             | 25.0             | 3.53                            | 89.5                             |
| 6                | 30             | 30.0             | 3.85                            | 89.9                             |
| 7                | 35             | 34.9             | 3.58                            | 88.5                             |
| 8                | 40             | 39.7             | 3.59                            | 88.0                             |
| 9                | 45             | 44.3             | 3.45                            | 88.0                             |
| 10               | 50             | 49.3             | 3.72                            | 88.0                             |
| 11               | 55             | 54.3             | 3.74                            | 88.0                             |
| 12               | 60             | 59.2             | 3.72                            | 87.5                             |
| 13               |                |                  |                                 |                                  |
| 14               |                |                  |                                 |                                  |
| 15               |                |                  |                                 |                                  |
| 16               |                |                  |                                 |                                  |
| 17               |                |                  |                                 |                                  |
| 18               |                |                  |                                 |                                  |
| 19               |                |                  |                                 |                                  |
| 20               |                |                  |                                 |                                  |

Table 15. Run No. 37 Conditions and Data

Date: 4-25-81

type of barley ..... Shabet grain

initial moisture content ..... 0.1 wt. % water

size distribution ..... See Table 1

feed concentration ..... 85.5 wt. % eth.

feed mass flux ..... 0.025 gm./min.-cm<sup>2</sup>

weight of barley used ..... 60.0 gm.

feed additions ..... 36.4 ml.

initial barley temperature ..... 86 °C

top      bottom

post run barley moisture content ... 6.0      8.9 wt. %

| SAMPLE<br>NUMBER | TIME<br>(MIN.) | BURETTE<br>(ML.) | EFFLUENT<br>SAMPLE<br>WT. (GM.) | EFFLUENT<br>% ETHANOL<br>(WT. %) |
|------------------|----------------|------------------|---------------------------------|----------------------------------|
| 1                | 20             | 2.0              | 0.59                            | 99.5                             |
| 2                | 40             | 4.0              | 1.06                            | 99.7                             |
| 3                | 60             | 6.0              | 1.06                            | 99.8                             |
| 4                | 80             | 8.0              | 1.12                            | 99.8                             |
| 5                | 100            | 10.0             | 1.22                            | 99.8                             |
| 6                | 120            | 12.0             | 1.14                            | 99.8                             |
| 7                | 140            | 14.0             | 1.12                            | 99.8                             |
| 8                | 160            | 16.0             | 1.30                            | 99.9                             |
| 9                | 180            | 18.1             | 1.30                            | 99.9                             |
| 10               | 200            | 20.2             | 1.25                            | 99.6                             |
| 11               | 220            | 22.3             | 1.17                            | 98.6                             |
| 12               | 240            | 24.3             | 1.36                            | 97.0                             |
| 13               | 260            | 26.3             | 1.21                            | 95.6                             |
| 14               | 280            | 28.3             | 1.30                            | 94.4                             |
| 15               | 300            | 30.1             | 1.17                            | 93.6                             |
| 16               | 320            | 32.0             | 1.22                            | 93.1                             |
| 17               | 340            | 34.1             | 1.48                            | 92.1                             |
| 18               | 360            | 36.3             | 1.67                            | 91.1                             |
| 19               |                |                  |                                 |                                  |
| 20               |                |                  |                                 |                                  |

Table 16. Run No. 38 Conditions and Data

Date: 4-26-81

type of barley ..... Shabet grain

initial moisture content ..... 0.1 wt. % water

size distribution ..... See Table 1

feed concentration ..... 85.5 wt. % eth.

feed mass flux ..... 0.025 gm./min.-cm<sup>2</sup>

weight of barley used ..... 60.0 gm.

feed additions ..... 26.4 ml.

initial barley temperature ..... 100 °C

top      bottom

post run barley moisture content .. 4.3      5.0 wt. %

| SAMPLE<br>NUMBER | TIME<br>(MIN.) | BURETTE<br>(ML.) | EFFLUENT<br>SAMPLE<br>WT. (GM.) | EFFLUENT<br>% ETHANOL<br>(WT. %) |
|------------------|----------------|------------------|---------------------------------|----------------------------------|
| 1                | 20             | 2.0              | 0.62                            | 99.5                             |
| 2                | 40             | 4.0              | 1.17                            | 99.5                             |
| 3                | 60             | 6.0              | 1.10                            | 99.5                             |
| 4                | 80             | 7.9              | 1.24                            | 99.6                             |
| 5                | 100            | 10.1             | 1.40                            | 99.6                             |
| 6                | 120            | 12.2             | 1.33                            | 99.6                             |
| 7                | 140            | 14.3             | 1.21                            | 99.3                             |
| 8                | 160            | 16.3             | 1.34                            | 96.7                             |
| 9                | 180            | 18.3             | 1.25                            | 93.9                             |
| 10               | 200            | 20.3             | 1.42                            | 91.8                             |
| 11               | 220            | 22.3             | 1.44                            | 90.1                             |
| 12               | 240            | 24.3             | 1.41                            | 88.7                             |
| 13               | 260            | 26.3             | 1.43                            | 87.4                             |
| 14               |                |                  |                                 |                                  |
| 15               |                |                  |                                 |                                  |
| 16               |                |                  |                                 |                                  |
| 17               |                |                  |                                 |                                  |
| 18               |                |                  |                                 |                                  |
| 19               |                |                  |                                 |                                  |
| 20               |                |                  |                                 |                                  |

Table 17. Run No. 39 Conditions and Data

Date: 5-1-81

type of barley ..... Shabet grain

initial moisture content ..... 0.1 wt. % water

size distribution ..... See Table 1

feed concentration ..... 86.5 wt. % eth.

feed mass flux ..... 0.25 gm./min.-cm<sup>2</sup>

weight of barley used ..... 60.0 gm.

feed additions ..... 84.2 ml.

initial barley temperature ..... 100 °C

top            bottom

post run barley moisture content .. 3.8            4.8 wt. %

| SAMPLE<br>NUMBER | TIME<br>(MIN.) | BURETTE<br>(ML.) | EFFLUENT<br>SAMPLE<br>WT. (GM.) | EFFLUENT<br>% ETHANOL<br>(WT. %) |
|------------------|----------------|------------------|---------------------------------|----------------------------------|
| 1                | 2              | 2.0              | 0.23                            | 99.2                             |
| 2                | 4              | 4.0              | 2.31                            | 99.5                             |
| 3                | 6              | 6.0              | 1.18                            | 97.8                             |
| 4                | 8              | 7.9              | 1.80                            | 93.4                             |
| 5                | 10             | 10.0             | 1.30                            | 91.1                             |
| 6                | 15             | 15.0             | 4.00                            | 89.5                             |
| 7                | 20             | 20.1             | 3.80                            | 89.0                             |
| 8                | 25             | 25.1             | 3.90                            | 88.0                             |
| 9                | 30             | 30.1             | 3.78                            | 88.0                             |
| 10               | 35             | 35.0             | 3.83                            | 87.5                             |
| 11               | 40             | 40.0             | 3.89                            | 87.5                             |
| 12               | 45             | 44.9             | 3.91                            | 86.5                             |
| 13               | 50             | 49.9             | 3.92                            | 86.5                             |
| 14               | 55             | 55.0             | 4.01                            | 86.5                             |
| 15               | 60             | 60.2             | 4.09                            | 86.5                             |
| 16               | 65             | 65.3             | 3.96                            | 86.5                             |
| 17               | 70             | 70.3             | 3.96                            | 86.5                             |
| 18               | 75             | 75.3             | 3.93                            | 86.5                             |
| 19               | 79             | 79.2             | 3.19                            | 86.5                             |
| 20               | 85             | 85.2             | 4.70                            | 86.5                             |

Table 18. Run No. 40 Conditions and Data

Date: 5-3-81

type of barley ..... Shabet grain

initial moisture content ..... 0.3 wt. % water

size distribution ..... See Table 1

feed concentration ..... 81.0 wt. % eth.

feed mass flux ..... 0.14 gm./min.-cm<sup>2</sup>

weight of barley used ..... 60.0 gm.

feed additions ..... 60.8 ml.

initial barley temperature ..... 93 °C

top      bottom

post run barley moisture content .. 5.3      6.6 wt. %

| SAMPLE<br>NUMBER | TIME<br>(MIN.) | BURETTE<br>(ML.) | EFFLUENT<br>SAMPLE<br>WT. (GM.) | EFFLUENT<br>% ETHANOL<br>(WT. %) |
|------------------|----------------|------------------|---------------------------------|----------------------------------|
| 1                | 2              | 1.2              | 0.01                            | --                               |
| 2                | 4              | 2.3              | 1.18                            | 99.6                             |
| 3                | 6              | 3.4              | 0.59                            | 99.6                             |
| 4                | 8              | 4.5              | 0.66                            | 99.7                             |
| 5                | 10             | 5.6              | 0.62                            | 99.6                             |
| 6                | 20             | 11.1             | 3.65                            | 93.3                             |
| 7                | 30             | 16.6             | 4.00                            | 87.5                             |
| 8                | 40             | 22.0             | 4.17                            | 86.0                             |
| 9                | 50             | 27.5             | 4.04                            | 85.0                             |
| 10               | 60             | 32.9             | 4.20                            | 84.0                             |
| 11               | 70             | 38.3             | 4.13                            | 83.0                             |
| 12               | 80             | 43.8             | 4.34                            | 82.5                             |
| 13               | 90             | 49.2             | 4.26                            | 82.5                             |
| 14               | 100            | 54.7             | 4.34                            | 82.5                             |
| 15               | 110            | 60.2             | 4.34                            | 82.5                             |
| 16               |                |                  |                                 |                                  |
| 17               |                |                  |                                 |                                  |
| 18               |                |                  |                                 |                                  |
| 19               |                |                  |                                 |                                  |
| 20               |                |                  |                                 |                                  |

Table 19. Run No. 41 Conditions and Data

Date: 5-13-81

type of barley ..... Shabet grain

initial moisture content ..... 0.3 wt. % water

size distribution ..... See Table 1

feed concentration ..... 81.5 wt. % eth.

feed mass flux ..... 0.14 gm./min.-cm<sup>2</sup>

weight of barley used ..... 60.0 gm.

feed additions ..... 40.0 ml.

initial barley temperature ..... 93 °C

top      bottom

post run barley moisture content .. 4.4      6.0 wt. %

| SAMPLE<br>NUMBER | TIME<br>(MIN.) | BURETTE<br>(ML.) | EFFLUENT<br>SAMPLE<br>WT. (GM.) | EFFLUENT<br>% ETHANOL<br>(WT. %) |
|------------------|----------------|------------------|---------------------------------|----------------------------------|
| 1                | 2              | 1.1              | 0.04                            | --                               |
| 2                | 4              | 2.2              | 1.25                            | 99.5                             |
| 3                | 6              | 3.3              | 0.59                            | 99.6                             |
| 4                | 8              | 4.5              | 0.65                            | 99.6                             |
| 5                | 10             | 5.5              | 0.62                            | 99.6                             |
| 6                | 20             | 11.0             | 3.68                            | 94.0                             |
| 7                | 30             | 16.3             | 3.87                            | 87.0                             |
| 8                | 40             | 21.6             | 4.06                            | 86.0                             |
| 9                | 50             | 26.8             | 3.96                            | 84.0                             |
| 10               | 60             | 32.1             | 4.20                            | 84.5                             |
| 11               | 70             | 37.5             | 4.10                            | 83.0                             |
| 12               |                |                  |                                 |                                  |
| 13               |                |                  |                                 |                                  |
| 14               |                |                  |                                 |                                  |
| 15               |                |                  |                                 |                                  |
| 16               |                |                  |                                 |                                  |
| 17               |                |                  |                                 |                                  |
| 18               |                |                  |                                 |                                  |
| 19               |                |                  |                                 |                                  |
| 20               |                |                  |                                 |                                  |

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Table 20. Run No. 43 Conditions and Data

Date: 6-11-81

type of barley ..... Shabet grain

initial moisture content ..... 0.2 wt. % water

size distribution ..... See Table 1

feed concentration ..... 81.0 wt. % eth.

feed mass flux ..... 0.14 gm./min.-cm<sup>2</sup>

weight of barley used ..... 60.0 gm.

feed additions ..... 38.9 ml.

initial barley temperature ..... 103 °C

top      bottom

post run barley moisture content .. 3.5      4.1 wt. %

| SAMPLE<br>NUMBER | TIME<br>(MIN.) | BURETTE<br>(ML.) | EFFLUENT<br>SAMPLE<br>WT. (GM.) | EFFLUENT<br>% ETHANOL<br>(WT. %) |
|------------------|----------------|------------------|---------------------------------|----------------------------------|
| 1                | 2              | 1.1              | 0.06                            | --                               |
| 2                | 4              | 2.2              | 1.09                            | 99.4                             |
| 3                | 6              | 3.4              | 0.68                            | 99.4                             |
| 4                | 8              | 4.5              | 0.66                            | 99.2                             |
| 5                | 10             | 5.6              | 0.72                            | 96.8                             |
| 6                | 20             | 11.1             | 3.89                            | 90.0                             |
| 7                | 30             | 16.6             | 4.13                            | 85.0                             |
| 8                | 40             | 22.1             | 4.25                            | 84.0                             |
| 9                | 50             | 27.5             | 4.23                            | 83.0                             |
| 10               | 60             | 32.9             | 4.28                            | 82.0                             |
| 11               | 70             | 38.3             | 4.17                            | 83.0                             |
| 12               |                |                  |                                 |                                  |
| 13               |                |                  |                                 |                                  |
| 14               |                |                  |                                 |                                  |
| 15               |                |                  |                                 |                                  |
| 16               |                |                  |                                 |                                  |
| 17               |                |                  |                                 |                                  |
| 18               |                |                  |                                 |                                  |
| 19               |                |                  |                                 |                                  |
| 20               |                |                  |                                 |                                  |

Table 21. Run No. 44 Conditions and Data

Date: 6-12-81

type of barley ..... Shabet grain

initial moisture content ..... 0.2 wt. % water

size distribution ..... See Table 1

feed concentration ..... 81.0 wt. % eth.

feed mass flux ..... 0.14 gm./min.-cm<sup>2</sup>

weight of barley used ..... 60.0 gm.

feed additions ..... 45.0 ml.

initial barley temperature ..... 86 °C

top            bottom

post run barley moisture content .. 5.1            8.0 wt. %

| SAMPLE<br>NUMBER | TIME<br>(MIN.) | BURETTE<br>(ML.) | EFFLUENT<br>SAMPLE<br>WT. (GM.) | EFFLUENT<br>% ETHANOL<br>(WT. %) |
|------------------|----------------|------------------|---------------------------------|----------------------------------|
| 1                | 2              | 1.1              | 0.01                            | --                               |
| 2                | 4              | 2.2              | 1.26                            | 99.6                             |
| 3                | 6              | 3.3              | 0.61                            | 99.6                             |
| 4                | 8              | 4.5              | 0.68                            | 99.5                             |
| 5                | 10             | 5.6              | 0.63                            | 99.6                             |
| 6                | 20             | 11.1             | 3.58                            | 96.8                             |
| 7                | 30             | 16.2             | 3.87                            | 90.0                             |
| 8                | 40             | 22.3             | 4.12                            | 87.0                             |
| 9                | 50             | 27.8             | 4.11                            | 86.0                             |
| 10               | 60             | 33.4             | 4.22                            | 85.0                             |
| 11               | 70             | 38.9             | 4.10                            | 85.0                             |
| 12               | 80             | 44.4             | 4.22                            | 84.0                             |
| 13               |                |                  |                                 |                                  |
| 14               |                |                  |                                 |                                  |
| 15               |                |                  |                                 |                                  |
| 16               |                |                  |                                 |                                  |
| 17               |                |                  |                                 |                                  |
| 18               |                |                  |                                 |                                  |
| 19               |                |                  |                                 |                                  |
| 20               |                |                  |                                 |                                  |

Table 22. Run No. 45 Conditions and Data

Date: 6-12-81

type of barley ..... Shabet grain

initial moisture content ..... 0 wt. % water

size distribution ..... See Table 1

feed concentration ..... 82.0 wt. % eth.

feed mass flux ..... 0.29 gm./min.-cm<sup>2</sup>

weight of barley used ..... 60.0 gm.

feed additions ..... 46.8 ml.

initial barley temperature ..... 93 °C

top      bottom

post run barley moisture content .. 3.5      5.6 wt. %

| SAMPLE<br>NUMBER | TIME<br>(MIN.) | BURETTE<br>(ML.) | EFFLUENT<br>SAMPLE<br>WT. (GM.) | EFFLUENT<br>% ETHANOL<br>(WT. %) |
|------------------|----------------|------------------|---------------------------------|----------------------------------|
| 1                | 2              | 2.3              | 0.08                            | --                               |
| 2                | 4              | 4.6              | 2.21                            | 99.5                             |
| 3                | 6              | 6.9              | 2.14                            | 95.5                             |
| 4                | 8              | 9.2              | 1.58                            | 88.5                             |
| 5                | 10             | 11.5             | 1.55                            | 88.0                             |
| 6                | 15             | 17.3             | 4.24                            | 86.0                             |
| 7                | 20             | 23.0             | 4.23                            | 85.0                             |
| 8                | 25             | 38.7             | 4.41                            | 84.0                             |
| 9                | 30             | 34.3             | 4.35                            | 83.5                             |
| 10               | 35             | 39.9             | 4.47                            | 83.5                             |
| 11               | 40             | 45.6             | 4.46                            | 83.5                             |
| 12               |                |                  |                                 |                                  |
| 13               |                |                  |                                 |                                  |
| 14               |                |                  |                                 |                                  |
| 15               |                |                  |                                 |                                  |
| 16               |                |                  |                                 |                                  |
| 17               |                |                  |                                 |                                  |
| 18               |                |                  |                                 |                                  |
| 19               |                |                  |                                 |                                  |
| 20               |                |                  |                                 |                                  |

Table 23. Run No. 46 Conditions and Data

Date: 6-16-81

type of barley ..... Shabet grain

initial moisture content ..... 0 wt. % water

size distribution ..... See Table 1

feed concentration ..... 81.0 wt. % eth.

feed mass flux ..... 0.14 gm./min.-cm<sup>2</sup>

weight of barley used ..... 60.0 gm.

feed additions ..... 33.5 ml.

initial barley temperature ..... 93 °C

top      bottom

post run barley moisture content .. 4.3      5.3 wt. %

| SAMPLE<br>NUMBER | TIME<br>(MIN.) | BURETTE<br>(ML.) | EFFLUENT<br>SAMPLE<br>WT. (GM.) | EFFLUENT<br>% ETHANOL<br>(WT. %) |
|------------------|----------------|------------------|---------------------------------|----------------------------------|
| 1                | 2              | 1.1              | 0.00                            | --                               |
| 2                | 4              | 2.2              | 1.27                            | 99.5                             |
| 3                | 6              | 3.3              | 0.63                            | 99.5                             |
| 4                | 8              | 4.4              | 0.66                            | 99.5                             |
| 5                | 10             | 5.5              | 0.62                            | 99.4                             |
| 6                | 20             | 11.0             | 3.71                            | 93.2                             |
| 7                | 30             | 16.5             | 4.01                            | 87.0                             |
| 8                | 40             | 22.0             | 4.21                            | 85.0                             |
| 9                | 56             | 27.5             | 4.15                            | 84.0                             |
| 10               | 60             | 33.0             | 4.26                            | 83.5                             |
| 11               |                |                  |                                 |                                  |
| 12               |                |                  |                                 |                                  |
| 13               |                |                  |                                 |                                  |
| 14               |                |                  |                                 |                                  |
| 15               |                |                  |                                 |                                  |
| 16               |                |                  |                                 |                                  |
| 17               |                |                  |                                 |                                  |
| 18               |                |                  |                                 |                                  |
| 19               |                |                  |                                 |                                  |
| 20               |                |                  |                                 |                                  |

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Table 24. Run No. 47 Conditions and Data

Date: 6-22-81

|                                     |                     |                          |       |
|-------------------------------------|---------------------|--------------------------|-------|
| type of barley .....                | <u>Shabet grain</u> |                          |       |
| initial moisture content .....      | <u>0</u>            | wt. %                    | water |
| size distribution .....             | <u>See Table 1</u>  |                          |       |
| feed concentration .....            | <u>81.5</u>         | wt. %                    | eth.  |
| feed mass flux .....                | <u>0.025</u>        | gm./min.-cm <sup>2</sup> |       |
| weight of barley used .....         | <u>60.0</u>         | gm.                      |       |
| feed additions .....                | <u>24.2</u>         | ml.                      |       |
| initial barley temperature .....    | <u>93</u>           | °C                       |       |
|                                     | <u>top</u>          | <u>bottom</u>            |       |
| post run barley moisture content .. | 4.7                 | 6.4                      | wt. % |

| SAMPLE<br>NUMBER | TIME<br>(MIN.) | BURETTE<br>(ML.) | EFFLUENT<br>SAMPLE<br>WT. (GM.) | EFFLUENT<br>% ETHANOL<br>(WT. %) |
|------------------|----------------|------------------|---------------------------------|----------------------------------|
| 1                | 20             | 2.0              | 0.67                            | 99.5                             |
| 2                | 40             | 4.0              | 1.06                            | 99.6                             |
| 3                | 60             | 6.1              | 1.27                            | 99.7                             |
| 4                | 80             | 8.2              | 1.20                            | 99.6                             |
| 5                | 100            | 10.1             | 1.04                            | 99.6                             |
| 6                | 120            | 11.9             | 1.09                            | 99.5                             |
| 7                | 140            | 14.2             | 1.41                            | 99.6                             |
| 8                | 160            | 16.3             | 1.37                            | 94.0                             |
| 9                | 180            | 18.2             | 1.29                            | 91.5                             |
| 10               | 200            | 20.1             | 1.31                            | 89.5                             |
| 11               | 220            | 22.0             | 1.32                            | 89.0                             |
| 12               | 240            | 24.1             | 1.51                            | 87.0                             |
| 13               |                |                  |                                 |                                  |
| 14               |                |                  |                                 |                                  |
| 15               |                |                  |                                 |                                  |
| 16               |                |                  |                                 |                                  |
| 17               |                |                  |                                 |                                  |
| 18               |                |                  |                                 |                                  |
| 19               |                |                  |                                 |                                  |
| 20               |                |                  |                                 |                                  |

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Table 25. Run No. 48 Conditions and Data

Date: 6-23-81

type of barley ..... Shabet grain

initial moisture content ..... 0 wt. % water

size distribution ..... See Table 1

feed concentration ..... 74.0 wt. % eth.

feed mass flux ..... 0.14 gm./min.-cm<sup>2</sup>

weight of barley used ..... 60.0 gm.

feed additions ..... 38.8 ml.

initial barley temperature ..... 93 °C

post run barley moisture content .. top bottom  
5.3 7.4 wt. %

| SAMPLE<br>NUMBER | TIME<br>(MIN.) | BURETTE<br>(ML.) | EFFLUENT<br>SAMPLE<br>WT. (GM.) | EFFLUENT<br>% ETHANOL<br>(WT. %) |
|------------------|----------------|------------------|---------------------------------|----------------------------------|
| 1                | 2              | 1.1              | 0.02                            | --                               |
| 2                | 4              | 2.2              | 1.10                            | 99.6                             |
| 3                | 6              | 3.3              | 0.61                            | 99.6                             |
| 4                | 8              | 4.4              | 0.61                            | 99.7                             |
| 5                | 10             | 5.5              | 0.60                            | 99.4                             |
| 6                | 20             | 11.0             | 3.77                            | 87.0                             |
| 7                | 30             | 16.4             | 3.94                            | 82.0                             |
| 8                | 40             | 21.8             | 4.14                            | 80.0                             |
| 9                | 50             | 27.2             | 4.07                            | 79.5                             |
| 10               | 60             | 32.8             | 4.40                            | 78.0                             |
| 11               | 70             | 38.2             | 4.18                            | 78.0                             |
| 12               |                |                  |                                 |                                  |
| 13               |                |                  |                                 |                                  |
| 14               |                |                  |                                 |                                  |
| 15               |                |                  |                                 |                                  |
| 16               |                |                  |                                 |                                  |
| 17               |                |                  |                                 |                                  |
| 18               |                |                  |                                 |                                  |
| 19               |                |                  |                                 |                                  |
| 20               |                |                  |                                 |                                  |

Table 26. Run No. 49 Conditions and Data

Date: 6-23-81

type of barley ..... Shabet grain

initial moisture content ..... 0 wt. % water

size distribution ..... See Table 1

feed concentration ..... 87.5 wt. % eth.

feed mass flux ..... 0.14 gm./min.-cm<sup>2</sup>

weight of barley used ..... 60.0 gm.

feed additions ..... 28.0 ml.

initial barley temperature ..... 93 °C

top          bottom

post run barley moisture content .. 2.4          4.2 wt. %

| SAMPLE<br>NUMBER | TIME<br>(MIN.) | BURETTE<br>(ML.) | EFFLUENT<br>SAMPLE<br>WT. (GM.) | EFFLUENT<br>% ETHANOL<br>(WT. %) |
|------------------|----------------|------------------|---------------------------------|----------------------------------|
| 1                | 2              | 1.1              | 0.01                            | --                               |
| 2                | 4              | 2.2              | 1.27                            | 98.5                             |
| 3                | 6              | 3.2              | 0.69                            | 99.5                             |
| 4                | 8              | 4.3              | 0.66                            | 99.5                             |
| 5                | 10             | 5.4              | 0.67                            | 99.6                             |
| 6                | 20             | 11.0             | 3.78                            | 98.6                             |
| 7                | 30             | 16.5             | 3.85                            | 93.5                             |
| 8                | 40             | 22.0             | 4.12                            | 91.6                             |
| 9                | 50             | 27.4             | 4.04                            | 90.7                             |
| 10               |                |                  |                                 |                                  |
| 11               |                |                  |                                 |                                  |
| 12               |                |                  |                                 |                                  |
| 13               |                |                  |                                 |                                  |
| 14               |                |                  |                                 |                                  |
| 15               |                |                  |                                 |                                  |
| 16               |                |                  |                                 |                                  |
| 17               |                |                  |                                 |                                  |
| 18               |                |                  |                                 |                                  |
| 19               |                |                  |                                 |                                  |
| 20               |                |                  |                                 |                                  |

Table 27. Run No. 50 Conditions and Data

Date: 6-24-81

type of barley ..... Shabet grain

initial moisture content ..... 0 wt. % water

size distribution ..... See Table 1

feed concentration ..... 77.5 wt. % eth.

feed mass flux ..... 0.025 gm./min.-cm<sup>2</sup>

weight of barley used ..... 60.0 gm.

feed additions ..... 30.1 ml.

initial barley temperature ..... 86 °C

top      bottom

post run barley moisture content .. 7.1      15.9 wt. %

| SAMPLE<br>NUMBER | TIME<br>(MIN.) | BURETTE<br>(ML.) | EFFLUENT<br>SAMPLE<br>WT. (GM.) | EFFLUENT<br>% ETHANOL<br>(WT. %) |
|------------------|----------------|------------------|---------------------------------|----------------------------------|
| 1                | 20             | 2.1              | 0.78                            | 99.6                             |
| 2                | 40             | 4.0              | 0.65                            | 99.6                             |
| 3                | 60             | 6.1              | 1.14                            | 99.8                             |
| 4                | 80             | 8.0              | 1.06                            | 99.7                             |
| 5                | 100            | 10.0             | 1.03                            | 99.8                             |
| 6                | 120            | 12.1             | 1.00                            | 99.8                             |
| 7                | 140            | 14.1             | 1.05                            | 99.8                             |
| 8                | 160            | 16.0             | 1.10                            | 99.8                             |
| 9                | 180            | 18.0             | 1.04                            | 99.8                             |
| 10               | 200            | 20.0             | 1.10                            | 99.8                             |
| 11               | 220            | 22.0             | 1.00                            | 99.5                             |
| 12               | 240            | 24.0             | 1.14                            | 98.6                             |
| 13               | 260            | 26.0             | 0.93                            | 96.7                             |
| 14               | 280            | 28.0             | 1.25                            | 95.4                             |
| 15               | 300            | 30.0             | 1.01                            | 94.0                             |
| 16               |                |                  |                                 |                                  |
| 17               |                |                  |                                 |                                  |
| 18               |                |                  |                                 |                                  |
| 19               |                |                  |                                 |                                  |
| 20               |                |                  |                                 |                                  |

Table 28. Run No. 53 Conditions and Data

Date: 7-1-81

type of barley ..... Shabet grain

initial moisture content ..... 0 wt. % water

size distribution ..... See Table 1

feed concentration ..... 55.5 wt. % eth.

feed mass flux ..... 0.027 gm./min.-cm<sup>2</sup>

weight of barley used ..... 60.0 gm.

feed additions ..... 24.2 ml.

initial barley temperature ..... 86 °C

top      bottom

post run barley moisture content... 7.2      20.8 wt. %

| SAMPLE<br>NUMBER | TIME<br>(MIN.) | BURETTE<br>(ML.) | EFFLUENT<br>SAMPLE<br>WT. (GM.) | EFFLUENT<br>% ETHANOL<br>(WT. %) |
|------------------|----------------|------------------|---------------------------------|----------------------------------|
| 1                | 20             | 2.0              | 0.34                            | 99.6                             |
| 2                | 40             | 4.0              | 0.84                            | 99.5                             |
| 3                | 60             | 6.0              | 0.68                            | 99.6                             |
| 4                | 80             | 8.1              | 0.98                            | 99.6                             |
| 5                | 100            | 10.2             | 0.80                            | 99.6                             |
| 6                | 120            | 12.0             | 0.73                            | 99.6                             |
| 7                | 140            | 14.0             | 0.83                            | 99.6                             |
| 8                | 160            | 16.1             | 0.88                            | 98.9                             |
| 9                | 180            | 18.1             | 0.84                            | 93.4                             |
| 10               | 200            | 19.9             | 0.81                            | 89.0                             |
| 11               | 220            | 22.0             | 1.09                            | 85.0                             |
| 12               | 240            | 24.1             | 1.11                            | 82.5                             |
| 13               |                |                  |                                 |                                  |
| 14               |                |                  |                                 |                                  |
| 15               |                |                  |                                 |                                  |
| 16               |                |                  |                                 |                                  |
| 17               |                |                  |                                 |                                  |
| 18               |                |                  |                                 |                                  |
| 19               |                |                  |                                 |                                  |
| 20               |                |                  |                                 |                                  |

Table 29. Run No. 54 Conditions and Data

Date: 7-2-81

type of barley ..... Shabet grain

initial moisture content ..... 0 wt. % water

size distribution ..... 1.651-2.362 mm.

feed concentration ..... 76.5 wt. % eth.

feed mass flux ..... 0.025 gm./min.-cm<sup>2</sup>

weight of barley used ..... 77.75 gm.

feed additions ..... 24.1 ml.

initial barley temperature ..... 86 °C

top      bottom

post run barley moisture content .. 3.5      9.1 wt. %

| SAMPLE<br>NUMBER | TIME<br>(MIN.) | BURETTE<br>(ML.) | EFFLUENT<br>SAMPLE<br>WT. (GM.) | EFFLUENT<br>% ETHANOL<br>(WT. %) |
|------------------|----------------|------------------|---------------------------------|----------------------------------|
| 1                | 20             | 2.0              | 0.84                            | 99.6                             |
| 2                | 40             | 4.0              | 1.07                            | 99.7                             |
| 3                | 60             | 6.0              | 1.10                            | 99.7                             |
| 4                | 80             | 8.0              | 1.11                            | 99.7                             |
| 5                | 100            | 10.1             | 1.13                            | 98.9                             |
| 6                | 120            | 12.0             | 1.13                            | 96.9                             |
| 7                | 140            | 14.0             | 1.27                            | 95.3                             |
| 8                | 160            | 16.1             | 1.29                            | 93.7                             |
| 9                | 180            | 18.1             | 1.22                            | 92.6                             |
| 10               | 200            | 20.1             | 1.31                            | 91.7                             |
| 11               | 220            | 22.0             | 1.27                            | 90.9                             |
| 12               | 240            | 24.0             | 1.22                            | 90.5                             |
| 13               |                |                  |                                 |                                  |
| 14               |                |                  |                                 |                                  |
| 15               |                |                  |                                 |                                  |
| 16               |                |                  |                                 |                                  |
| 17               |                |                  |                                 |                                  |
| 18               |                |                  |                                 |                                  |
| 19               |                |                  |                                 |                                  |
| 20               |                |                  |                                 |                                  |

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Table 30. Run No. 55 Conditions and Data

Date: 7-4-81

type of barley ..... Shabet grain

initial moisture content ..... 0 wt. % water

size distribution ..... See Table 1

feed concentration ..... 55.0 wt. % eth.

feed mass flux ..... 0.025 gm./min.-cm<sup>2</sup>

weight of barley used ..... 58.7 gm.

feed additions ..... 22.1 ml.

initial barley temperature ..... 86 °C

post run barley moisture content .. 5.7 top bottom 20.9 wt. %

| SAMPLE<br>NUMBER | TIME<br>(MIN.) | BURETTE<br>(ML.) | EFFLUENT<br>SAMPLE<br>WT. (GM.) | EFFLUENT<br>% ETHANOL<br>(WT. %) |
|------------------|----------------|------------------|---------------------------------|----------------------------------|
| 1                | 20             | 2.0              | 0.29                            | 99.0                             |
| 2                | 40             | 4.0              | 0.73                            | 99.4                             |
| 3                | 60             | 6.0              | 0.64                            | 99.4                             |
| 4                | 80             | 8.0              | 0.86                            | 99.5                             |
| 5                | 100            | 10.0             | 0.80                            | 99.3                             |
| 6                | 120            | 12.1             | 1.02                            | 99.5                             |
| 7                | 140            | 14.1             | 0.86                            | 99.5                             |
| 8                | 160            | 16.0             | 0.88                            | 99.0                             |
| 9                | 180            | 18.0             | 0.84                            | 95.4                             |
| 10               | 200            | 20.0             | 1.12                            | 89.5                             |
| 11               | 220            | 22.0             | 0.81                            | 85.0                             |
| 12               |                |                  |                                 |                                  |
| 13               |                |                  |                                 |                                  |
| 14               |                |                  |                                 |                                  |
| 15               |                |                  |                                 |                                  |
| 16               |                |                  |                                 |                                  |
| 17               |                |                  |                                 |                                  |
| 18               |                |                  |                                 |                                  |
| 19               |                |                  |                                 |                                  |
| 20               |                |                  |                                 |                                  |

Table 31. Run No: 56 Conditions and Data

Date: 7-6-81

type of barley ..... Shabet grain

initial moisture content ..... 0 wt. % water

size distribution ..... 0.589-0.833 mm.

feed concentration ..... 75.0 wt. % eth.

feed mass flux ..... 0.025 gm./min.-cm<sup>2</sup>

weight of barley used ..... 45.0 gm.

feed additions ..... 26.2 ml.

initial barley temperature ..... 86 °C

top      bottom

post run barley moisture content .. 8.4      10.1 wt. %

| SAMPLE<br>NUMBER | TIME<br>(MIN.) | BURETTE<br>(ML.) | EFFLUENT<br>SAMPLE<br>WT. (GM.) | EFFLUENT<br>% ETHANOL<br>(WT. %) |
|------------------|----------------|------------------|---------------------------------|----------------------------------|
| 1                | 20             | 2.1              | 0.64                            | 99.3                             |
| 2                | 40             | 4.0              | 1.03                            | 99.5                             |
| 3                | 60             | 6.0              | 1.03                            | 99.5                             |
| 4                | 80             | 8.1              | 1.08                            | 99.5                             |
| 5                | 100            | 10.1             | 0.94                            | 99.5                             |
| 6                | 120            | 12.0             | 1.15                            | 99.5                             |
| 7                | 140            | 14.1             | 1.10                            | 98.8                             |
| 8                | 160            | 16.0             | 1.19                            | 94.5                             |
| 9                | 180            | 18.0             | 1.26                            | 89.5                             |
| 10               | 200            | 20.0             | 1.29                            | 86.0                             |
| 11               | 220            | 22.0             | 1.43                            | 83.5                             |
| 12               | 260            | 26.1             | 3.14                            | 81.0                             |
| 13               |                |                  |                                 |                                  |
| 14               |                |                  |                                 |                                  |
| 15               |                |                  |                                 |                                  |
| 16               |                |                  |                                 |                                  |
| 17               |                |                  |                                 |                                  |
| 18               |                |                  |                                 |                                  |
| 19               |                |                  |                                 |                                  |
| 20               |                |                  |                                 |                                  |

Table 32. Run No. 57 Conditions and Data

Date: 7-7-81

type of barley ..... Shabet grain

initial moisture content ..... 0.6 wt. % water

size distribution ..... 0.208-0.295 mm.

feed concentration ..... 76.0 wt. % eth.

feed mass flux ..... 0.025 gm./min.-cm<sup>2</sup>

weight of barley used ..... 35.0 gm.

feed additions ..... 30.1 ml.

initial barley temperature ..... 86 °C

top      bottom

post run barley moisture content .. 9.8      11.3 wt. %

| SAMPLE<br>NUMBER | TIME<br>(MIN.) | BURETTE<br>(ML.) | EFFLUENT<br>SAMPLE<br>WT. (GM.) | EFFLUENT<br>% ETHANOL<br>(WT. %) |
|------------------|----------------|------------------|---------------------------------|----------------------------------|
| 1                | 20             | 2.1              | 0.47                            | 98.9                             |
| 2                | 40             | 4.1              | 1.03                            | 99.2                             |
| 3                | 60             | 6.1              | 1.05                            | 99.4                             |
| 4                | 80             | 8.1              | 1.01                            | 99.3                             |
| 5                | 100            | 9.9              | 0.96                            | 99.4                             |
| 6                | 120            | 12.0             | 1.25                            | 98.8                             |
| 7                | 140            | 14.1             | 1.34                            | 89.0                             |
| 8                | 160            | 16.2             | 1.47                            | 84.0                             |
| 9                | 180            | 18.0             | 1.24                            | 82.0                             |
| 10               | 200            | 19.9             | 1.40                            | 81.0                             |
| 11               | 220            | 21.9             | 1.61                            | 80.0                             |
| 12               | 240            | 24.1             | 1.65                            | 78.8                             |
| 13               | 260            | 26.2             | 1.52                            | 77.5                             |
| 14               | 280            | 28.1             | 1.51                            | 77.5                             |
| 15               | 300            | 30.0             | 1.34                            | 77.5                             |
| 16               |                |                  |                                 |                                  |
| 17               |                |                  |                                 |                                  |
| 18               |                |                  |                                 |                                  |
| 19               |                |                  |                                 |                                  |
| 20               |                |                  |                                 |                                  |

Table 33. Run No. 58 Conditions and Data

Date: 7-8-81

type of barley ..... Shabet grain

initial moisture content ..... 1.5 wt. % water

size distribution ..... See Table 1

feed concentration ..... 76.0 wt. % eth.

feed mass flux ..... 0.025 gm./min.-cm<sup>2</sup>

weight of barley used ..... 60.0 gm.

feed additions ..... 26.1 ml.

initial barley temperature ..... 86 °C

top      bottom

post run barley moisture content .. 7.8      10.9 wt. %

| SAMPLE<br>NUMBER | TIME<br>(MIN.) | BURETTE<br>(ML.) | EFFLUENT<br>SAMPLE<br>WT. (GM.) | EFFLUENT<br>% ETHANOL<br>(WT. %) |
|------------------|----------------|------------------|---------------------------------|----------------------------------|
| 1                | 20             | 2.1              | 0.36                            | 98.3                             |
| 2                | 40             | 4.2              | 0.96                            | 98.2                             |
| 3                | 60             | 6.1              | 0.93                            | 98.8                             |
| 4                | 80             | 8.0              | 0.98                            | 98.9                             |
| 5                | 100            | 10.0             | 1.08                            | 98.9                             |
| 6                | 120            | 12.0             | 1.12                            | 98.8                             |
| 7                | 140            | 14.0             | 1.14                            | 98.7                             |
| 8                | 161            | 16.0             | 1.16                            | 98.0                             |
| 9                | 180            | 18.0             | 1.10                            | 95.2                             |
| 10               | 200            | 20.1             | 1.37                            | 91.4                             |
| 11               | 220            | 22.1             | 1.25                            | 89.0                             |
| 12               | 240            | 24.0             | 1.28                            | 87.0                             |
| 13               | 260            | 26.0             | 1.28                            | 86.0                             |
| 14               |                |                  |                                 |                                  |
| 15               |                |                  |                                 |                                  |
| 16               |                |                  |                                 |                                  |
| 17               |                |                  |                                 |                                  |
| 18               |                |                  |                                 |                                  |
| 19               |                |                  |                                 |                                  |
| 20               |                |                  |                                 |                                  |

Table 34. Run No. 59 Conditions and Data

Date: 7-9-81

```
type of barley ..... Shabet grain
```

initial moisture content ..... 2.4 wt. % water

size distribution ..... See Table 1

feed concentration ..... 76.0 wt. % eth.

feed mass flux ..... 0.025 gm./min.-cm<sup>2</sup>

weight of barley used ..... 60.0 gm.

feed additions ..... 20.2 ml.

initial barley temperature .....86 °C

top      bottom

|                                     |     |            |
|-------------------------------------|-----|------------|
| post run barley moisture content... | 6.9 | 10.6 wt. % |
|-------------------------------------|-----|------------|

| SAMPLE<br>NUMBER | TIME<br>(MIN.) | BURETTE<br>(ML.) | EFFLUENT<br>SAMPLE<br>WT. (GM.) | EFFLUENT<br>% ETHANOL<br>(WT. %) |
|------------------|----------------|------------------|---------------------------------|----------------------------------|
| 1                | 20             | 2.0              | 0.27                            | 98.0                             |
| 2                | 40             | 4.0              | 0.96                            | 98.2                             |
| 3                | 60             | 6.0              | 1.01                            | 98.1                             |
| 4                | 80             | 8.0              | 1.13                            | 98.0                             |
| 5                | 100            | 10.1             | 1.10                            | 97.9                             |
| 6                | 120            | 12.1             | 1.17                            | 97.6                             |
| 7                | 140            | 14.0             | 1.03                            | 96.5                             |
| 8                | 160            | 16.1             | 1.20                            | 93.8                             |
| 9                | 180            | 18.0             | 1.23                            | 90.5                             |
| 10               | 200            | 20.1             | 1.43                            | 88.0                             |
| 11               |                |                  |                                 |                                  |
| 12               |                |                  |                                 |                                  |
| 13               |                |                  |                                 |                                  |
| 14               |                |                  |                                 |                                  |
| 15               |                |                  |                                 |                                  |
| 16               |                |                  |                                 |                                  |
| 17               |                |                  |                                 |                                  |
| 18               |                |                  |                                 |                                  |
| 19               |                |                  |                                 |                                  |
| 20               |                |                  |                                 |                                  |

Table 35. Run No. 60 Conditions and Data

Date: 7-16-81

type of barley ..... Shabet grain

initial moisture content ..... 0 wt. % water

size distribution ..... See Table 1

feed concentration ..... 75.0 wt. % eth.

feed mass flux ..... 0.25 gm./min.-cm<sup>2</sup>

weight of barley used ..... 60.0 gm.

feed additions ..... -- ml.

initial barley temperature ..... 86 °C

top      bottom

post run barley moisture content .. --      -- wt. %

| SAMPLE<br>NUMBER | TIME<br>(MIN.) | BURETTE<br>(ML.) | EFFLUENT<br>SAMPLE<br>WT. (GM.) | EFFLUENT<br>% ETHANOL<br>(WT. %) |
|------------------|----------------|------------------|---------------------------------|----------------------------------|
| 1                | 2              | 2.1              | --                              | --                               |
| 2                | 4              | 4.2              | --                              | --                               |
| 3                | 6              | 6.2              | --                              | --                               |
| 4                | 8              | 8.3              | --                              | --                               |
| 5                | 10             | 10.4             | --                              | --                               |
| 6                | 12             | 12.5             | --                              | --                               |
| 7                | 14             | 14.5             | --                              | --                               |
| 8                | 16             | 16.5             | --                              | --                               |
| 9                |                |                  |                                 |                                  |
| 10               |                |                  |                                 |                                  |
| 11               |                |                  |                                 |                                  |
| 12               |                |                  |                                 |                                  |
| 13               |                |                  |                                 |                                  |
| 14               |                |                  |                                 |                                  |
| 15               |                |                  |                                 |                                  |
| 16               |                |                  |                                 |                                  |
| 17               |                |                  |                                 |                                  |
| 18               |                |                  |                                 |                                  |
| 19               |                |                  |                                 |                                  |
| 20               |                |                  |                                 |                                  |

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## APPENDIX C

### RESULTS OF RUNS

Table 36. Results of Run No. 26

percent of feed accounted for ..... 90.9 wt. %  
total ethanol sorbed ..... 2.28 gm.  
total water sorbed ..... 6.45 gm.  
total water sorbed per gm. barley ..... 0.108 gm./gm.

| ETH. CONC.<br>OF COMBINED<br>EFFLUENT<br>SAMPLES | GM. WATER<br>SORBED PER<br>GM. GRAIN | ML. PURE<br>ETH. CONC.<br>PER GM.<br>GRAIN | % OF ETH.<br>SORBED | % ETH. IN<br>SORBED<br>LIQUID | TIME<br>REQUIRED<br>FOR THIS<br>(MINUTES) |
|--------------------------------------------------|--------------------------------------|--------------------------------------------|---------------------|-------------------------------|-------------------------------------------|
| 99                                               | 0.0838                               | 0.326                                      | 8.58                | 22.4                          | 296.3                                     |
| 98                                               | 0.0921                               | 0.373                                      | 7.69                | 21.1                          | 334.8                                     |
| 97                                               | 0.0984                               | 0.416                                      | 7.06                | 20.2                          | 369.4                                     |
| 96                                               | 0.105                                | 0.461                                      | 6.62                | 19.8                          | 406.1                                     |
| 95                                               |                                      |                                            |                     |                               |                                           |
| 94                                               |                                      |                                            |                     |                               |                                           |
| 92                                               |                                      |                                            |                     |                               |                                           |
| 90                                               |                                      |                                            |                     |                               |                                           |
| 88                                               |                                      |                                            |                     |                               |                                           |
| 86                                               |                                      |                                            |                     |                               |                                           |
| 85                                               |                                      |                                            |                     |                               |                                           |
| 84                                               |                                      |                                            |                     |                               |                                           |
| 82                                               |                                      |                                            |                     |                               |                                           |
| 80                                               |                                      |                                            |                     |                               |                                           |

Table 37. Results of Run No. 28

percent of feed accounted for ..... 96.3 wt. %  
total ethanol sorbed ..... 1.094 gm.  
total water sorbed ..... 4.055 gm.  
total water sorbed per gm. barley ..... 0.676 gm./gm.

| ETH. CONC.<br>OF COMBINED<br>EFFLUENT<br>SAMPLES | GM. WATER<br>SORBED PER<br>GM. GRAIN | ML. PURE<br>ETH. CONC.<br>PER GM.<br>GRAIN | % OF ETH.<br>SORBED | % ETH. IN<br>SORBED<br>LIQUID | TIME<br>REQUIRED<br>FOR THIS<br>(MINUTES) |
|--------------------------------------------------|--------------------------------------|--------------------------------------------|---------------------|-------------------------------|-------------------------------------------|
| 99                                               | 0.0146                               | 0.0555                                     | 10.48               | 26.0                          | 5.38                                      |
| 98                                               | 0.0160                               | 0.0632                                     | 9.74                | 25.2                          | 5.99                                      |
| 97                                               | 0.0178                               | 0.0735                                     | 8.98                | 24.3                          | 6.81                                      |
| 96                                               | 0.0204                               | 0.0882                                     | 8.19                | 23.3                          | 7.99                                      |
| 95                                               | 0.0245                               | 0.111                                      | 7.36                | 22.2                          | 9.80                                      |
| 94                                               | 0.0280                               | 0.133                                      | 7.05                | 22.1                          | 11.53                                     |
| 92                                               | 0.0317                               | 0.167                                      | 6.14                | 21.4                          | 14.21                                     |
| 90                                               | 0.0364                               | 0.217                                      | 4.97                | 19.8                          | 18.02                                     |
| 88                                               | 0.0412                               | 0.285                                      | 3.74                | 17.5                          | 23.15                                     |
| 86                                               | 0.0474                               | 0.390                                      | 3.00                | 16.7                          | 31.17                                     |
| 85                                               |                                      |                                            |                     |                               |                                           |
| 84                                               |                                      |                                            |                     |                               |                                           |
| 82                                               |                                      |                                            |                     |                               |                                           |
| 80                                               |                                      |                                            |                     |                               |                                           |

Table 38. Results of Run No. 29

percent of feed accounted for ..... 95.6 wt. %  
total ethanol sorbed ..... 0.119 gm.  
total water sorbed ..... 2.84 gm.  
total water sorbed per gm. barley ..... 0.0473 gm./gm.

| ETH. CONC.<br>OF COMBINED<br>EFFLUENT<br>SAMPLES | GM. WATER<br>SORBED PER<br>GM. GRAIN | ML. PURE<br>ETH. CONC.<br>PER GM.<br>GRAIN | % OF ETH.<br>SORBED | % ETH. IN<br>SORBED<br>LIQUID | TIME<br>REQUIRED<br>FOR THIS<br>(MINUTES) |
|--------------------------------------------------|--------------------------------------|--------------------------------------------|---------------------|-------------------------------|-------------------------------------------|
| 99                                               | 0.0099                               | 0.0302                                     | 23.9                | 43.0                          | 3.76                                      |
| 98                                               | 0.0102                               | 0.0327                                     | 22.1                | 41.7                          | 3.95                                      |
| 97                                               | 0.0106                               | 0.0358                                     | 20.2                | 40.2                          | 4.17                                      |
| 96                                               | 0.0111                               | 0.0396                                     | 18.1                | 38.4                          | 4.45                                      |
| 95                                               | 0.0117                               | 0.0444                                     | 15.9                | 36.2                          | 4.80                                      |
| 94                                               | 0.0125                               | 0.0716                                     | 8.2                 | 25.0                          | 6.78                                      |
| 92                                               | 0.0218                               | 0.126                                      | --                  | --                            | 10.74                                     |
| 90                                               | 0.0255                               | 0.168                                      | --                  | --                            | 13.87                                     |
| 88                                               | 0.0294                               | 0.228                                      | --                  | --                            | 18.42                                     |
| 86                                               |                                      |                                            |                     |                               |                                           |
| 85                                               |                                      |                                            |                     |                               |                                           |
| 84                                               |                                      |                                            |                     |                               |                                           |
| 82                                               |                                      |                                            |                     |                               |                                           |
| 80                                               |                                      |                                            |                     |                               |                                           |

Table 39. Results of Run No. 30

percent of feed accounted for ..... 89.0 wt. %  
total ethanol sorbed ..... 0.509 gm.  
total water sorbed ..... 3.46 gm.  
total water sorbed per gm. barley ..... 0.0576 gm./gm.

| ETH. CONC.<br>OF COMBINED<br>EFFLUENT<br>SAMPLES | GM. WATER<br>SORBED PER<br>GM. GRAIN | ML. PURE<br>ETH. CONC.<br>PER GM.<br>GRAIN | % OF ETH.<br>SORBED | % ETH. IN<br>SORBED<br>LIQUID | TIME<br>REQUIRED<br>FOR THIS<br>(MINUTES) |
|--------------------------------------------------|--------------------------------------|--------------------------------------------|---------------------|-------------------------------|-------------------------------------------|
| 99                                               | 0.0328                               | 0.132                                      | 16.9                | 53.4                          | 122.2                                     |
| 98                                               | 0.0365                               | 0.151                                      | --                  | --                            | 138.7                                     |
| 97                                               | 0.0390                               | 0.169                                      | --                  | --                            | 151.9                                     |
| 96                                               | 0.0414                               | 0.186                                      | --                  | --                            | 165.9                                     |
| 95                                               | 0.0436                               | 0.204                                      | --                  | --                            | 180.7                                     |
| 94                                               |                                      |                                            |                     |                               |                                           |
| 92                                               |                                      |                                            |                     |                               |                                           |
| 90                                               |                                      |                                            |                     |                               |                                           |
| 88                                               |                                      |                                            |                     |                               |                                           |
| 86                                               |                                      |                                            |                     |                               |                                           |
| 85                                               |                                      |                                            |                     |                               |                                           |
| 84                                               |                                      |                                            |                     |                               |                                           |
| 82                                               |                                      |                                            |                     |                               |                                           |
| 80                                               |                                      |                                            |                     |                               |                                           |

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Table 40. Results of Run No. 33

percent of feed accounted for ..... 96.8 wt. %  
 total ethanol sorbed ..... 0.929 gm.  
 total water sorbed ..... 2.231 gm.  
 total water sorbed per gm. barley ..... 0.0372 gm./gm.

| ETH. CONC.<br>OF COMBINED<br>EFFLUENT<br>SAMPLES | GM. WATER<br>SORBED PER<br>GM. GRAIN | ML. PURE<br>ETH. CONC.<br>PER GM.<br>GRAIN | % OF ETH.<br>SORBED | % ETH. IN<br>SORBED<br>LIQUID | TIME<br>REQUIRED<br>FOR THIS<br>(MINUTES) |
|--------------------------------------------------|--------------------------------------|--------------------------------------------|---------------------|-------------------------------|-------------------------------------------|
| 99                                               | 0.0104                               | 0.0602                                     | 7.09                | 25.9                          | 5.43                                      |
| 98                                               | 0.0129                               | 0.0789                                     | 6.99                | 26.6                          | 6.92                                      |
| 97                                               | 0.0141                               | 0.0905                                     | 7.25                | 28.3                          | 7.80                                      |
| 96                                               | 0.0154                               | 0.105                                      | 6.66                | 27.8                          | 8.91                                      |
| 95                                               | 0.0166                               | 0.122                                      | 5.93                | 26.7                          | 10.18                                     |
| 94                                               | 0.0179                               | 0.140                                      | 5.80                | 27.6                          | 11.58                                     |
| 92                                               | 0.0199                               | 0.187                                      | 4.35                | 25.2                          | 15.01                                     |
| 90                                               | 0.0238                               | 0.275                                      | 3.87                | 26.9                          | 21.41                                     |
| 88                                               | 0.0275                               | 0.429                                      | 2.46                | 23.7                          | 32.36                                     |
| 86                                               |                                      |                                            |                     |                               |                                           |
| 85                                               |                                      |                                            |                     |                               |                                           |
| 84                                               |                                      |                                            |                     |                               |                                           |
| 82                                               |                                      |                                            |                     |                               |                                           |
| 80                                               |                                      |                                            |                     |                               |                                           |

Table 41. Results of Run No. 35

percent of feed accounted for ..... 82.9 wt. %  
total ethanol sorbed ..... 0.553 gm.  
total water sorbed ..... 0.738 gm.  
total water sorbed per gm. barley ..... 0.0523 gm./gm.

| ETH. CONC.<br>OF COMBINED<br>EFFLUENT<br>SAMPLES | GM. WATER<br>SORBED PER<br>GM. GRAIN | ML. PURE<br>ETH. CONC.<br>PER GM.<br>GRAIN | % OF ETH.<br>SORBED | % ETH. IN<br>SORBED<br>LIQUID | TIME<br>REQUIRED<br>FOR THIS<br>(MINUTES) |
|--------------------------------------------------|--------------------------------------|--------------------------------------------|---------------------|-------------------------------|-------------------------------------------|
| 99                                               | 0.0290                               | 0.182                                      | 3.50                | 15.2                          | 50.01                                     |
| 98                                               | 0.0387                               | 0.252                                      | 4.59                | 19.8                          | 64.90                                     |
| 97                                               | 0.0408                               | 0.290                                      | 2.07                | 10.6                          | 71.45                                     |
| 96                                               | 0.0438                               | 0.343                                      | --                  | --                            | 80.55                                     |
| 95                                               |                                      |                                            |                     |                               |                                           |
| 94                                               |                                      |                                            |                     |                               |                                           |
| 92                                               |                                      |                                            |                     |                               |                                           |
| 90                                               |                                      |                                            |                     |                               |                                           |
| 88                                               |                                      |                                            |                     |                               |                                           |
| 86                                               |                                      |                                            |                     |                               |                                           |
| 85                                               |                                      |                                            |                     |                               |                                           |
| 84                                               |                                      |                                            |                     |                               |                                           |
| 82                                               |                                      |                                            |                     |                               |                                           |
| 80                                               |                                      |                                            |                     |                               |                                           |

Table 42. Results of Run No. 36

percent of feed accounted for ..... 94.7 wt. %  
 total ethanol sorbed ..... 1.534 gm.  
 total water sorbed ..... 2.440 gm.  
 total water sorbed per gm. barley ..... 0.0407 gm./gm.

| ETH. CONC.<br>OF COMBINED<br>EFFLUENT<br>SAMPLES | GM. WATER<br>SORBED PER<br>GM. GRAIN | ML. PURE<br>ETH. CONC.<br>PER GM.<br>GRAIN | % OF ETH.<br>SORBED | % ETH. IN<br>SORBED<br>LIQUID | TIME<br>REQUIRED<br>FOR THIS<br>(MINUTES) |
|--------------------------------------------------|--------------------------------------|--------------------------------------------|---------------------|-------------------------------|-------------------------------------------|
| 99                                               | 0.0076                               | 0.0405                                     | 34.5                | 68.8                          | 4.98                                      |
| 98                                               | 0.0172                               | 0.143                                      | 5.8                 | 28.7                          | 11.38                                     |
| 97                                               | 0.0194                               | 0.173                                      | 5.4                 | 29.3                          | 13.67                                     |
| 96                                               | 0.0222                               | 0.216                                      | 5.3                 | 30.1                          | 16.84                                     |
| 95                                               | 0.0250                               | 0.266                                      | 5.1                 | 33.4                          | 24.86                                     |
| 94                                               | 0.0327                               | 0.508                                      | 3.4                 | 30.1                          | 38.26                                     |
| 92                                               |                                      |                                            |                     |                               |                                           |
| 90                                               |                                      |                                            |                     |                               |                                           |
| 88                                               |                                      |                                            |                     |                               |                                           |
| 86                                               |                                      |                                            |                     |                               |                                           |
| 85                                               |                                      |                                            |                     |                               |                                           |
| 84                                               |                                      |                                            |                     |                               |                                           |
| 82                                               |                                      |                                            |                     |                               |                                           |
| 80                                               |                                      |                                            |                     |                               |                                           |

(130)

Table 43. Results of Run No. 37

|                                         |      |         |
|-----------------------------------------|------|---------|
| percent of feed accounted for .....     | 88.1 | wt. %   |
| total ethanol sorbed .....              | 1.63 | gm.     |
| total water sorbed .....                | 3.23 | gm.     |
| total water sorbed per gm. barley ..... | --   | gm./gm. |

| ETH. CONC.<br>OF COMBINED<br>EFFLUENT<br>SAMPLES | GM. WATER<br>SORBED PER<br>GM. GRAIN | ML. PURE<br>ETH. CONC.<br>PER GM.<br>GRAIN | % OF ETH.<br>SORBED | % ETH. IN<br>SORBED<br>LIQUID | TIME<br>REQUIRED<br>FOR THIS<br>(MINUTES) |
|--------------------------------------------------|--------------------------------------|--------------------------------------------|---------------------|-------------------------------|-------------------------------------------|
| 99                                               | 0.0432                               | 0.318                                      | 6.65                | 29.3                          | 266.3                                     |
| 98                                               | 0.0490                               | 0.387                                      | 6.15                | 29.0                          | 323.3                                     |
| 97                                               | 0.0522                               | 0.452                                      | 4.17                | 23.0                          | 365.3                                     |
| 96                                               |                                      |                                            |                     |                               |                                           |
| 95                                               |                                      |                                            |                     |                               |                                           |
| 94                                               |                                      |                                            |                     |                               |                                           |
| 92                                               |                                      |                                            |                     |                               |                                           |
| 90                                               |                                      |                                            |                     |                               |                                           |
| 88                                               |                                      |                                            |                     |                               |                                           |
| 86                                               |                                      |                                            |                     |                               |                                           |
| 85                                               |                                      |                                            |                     |                               |                                           |
| 84                                               |                                      |                                            |                     |                               |                                           |
| 82                                               |                                      |                                            |                     |                               |                                           |
| 80                                               |                                      |                                            |                     |                               |                                           |

(131)

Table 44. Results of Run No. 38

percent of feed accounted for ..... 88.2 wt. %  
total ethanol sorbed ..... 0.912 gm.  
total water sorbed ..... 2.04 gm.  
total water sorbed per gm. barley ..... 0.0341 gm./gm.

| ETH. CONC.<br>OF COMBINED<br>EFFLUENT<br>SAMPLES | GM. WATER<br>SORBED PER<br>GM. GRAIN | ML. PURE<br>ETH. CONC.<br>PER GM.<br>GRAIN | % OF ETH.<br>SORBED | % ETH. IN<br>SORBED<br>LIQUID | TIME<br>REQUIRED<br>FOR THIS<br>(MINUTES) |
|--------------------------------------------------|--------------------------------------|--------------------------------------------|---------------------|-------------------------------|-------------------------------------------|
| 99                                               | 0.0262                               | 0.202                                      | 2.99                | 15.8                          | 164.8                                     |
| 98                                               | 0.0291                               | 0.239                                      | 2.88                | 16.1                          | 193.6                                     |
| 97                                               | 0.307                                | 0.273                                      | 2.13                | 13.3                          | 218.7                                     |
| 96                                               | 0.0318                               | 0.307                                      | 1.82                | 12.4                          | 244.5                                     |
| 95                                               |                                      |                                            |                     |                               |                                           |
| 94                                               |                                      |                                            |                     |                               |                                           |
| 92                                               |                                      |                                            |                     |                               |                                           |
| 90                                               |                                      |                                            |                     |                               |                                           |
| 88                                               |                                      |                                            |                     |                               |                                           |
| 86                                               |                                      |                                            |                     |                               |                                           |
| 85                                               |                                      |                                            |                     |                               |                                           |
| 84                                               |                                      |                                            |                     |                               |                                           |
| 82                                               |                                      |                                            |                     |                               |                                           |
| 80                                               |                                      |                                            |                     |                               |                                           |

Table 45. Results of Run No. 39

percent of feed accounted for ..... 97.9 wt. %  
total ethanol sorbed ..... 1.29 gm.  
total water sorbed ..... 1.41 gm.  
total water sorbed per gm. barley ..... 0.0235 gm./gm.

| ETH. CONC.<br>OF COMBINED<br>EFFLUENT<br>SAMPLES | GM. WATER<br>SORBED PER<br>GM. GRAIN | ML. PURE<br>ETH. CONC.<br>PER GM.<br>GRAIN | % OF ETH.<br>SORBED | % ETH. IN<br>SORBED<br>LIQUID | TIME<br>REQUIRED<br>FOR THIS<br>(MINUTES) |
|--------------------------------------------------|--------------------------------------|--------------------------------------------|---------------------|-------------------------------|-------------------------------------------|
| 99                                               | 0.0103                               | 0.0728                                     | 17.7                | 54.5                          | 6.61                                      |
| 98                                               | 0.0115                               | 0.0923                                     | 12.3                | 47.1                          | 7.83                                      |
| 97                                               | 0.0128                               | 0.115                                      | 8.9                 | 41.1                          | 9.17                                      |
| 96                                               | 0.0143                               | 0.137                                      | 10.1                | 46.0                          | 10.93                                     |
| 95                                               | 0.0150                               | 0.161                                      | 8.6                 | 44.4                          | 12.50                                     |
| 94                                               | 0.0160                               | 0.194                                      | 7.0                 | 41.8                          | 14.73                                     |
| 92                                               | 0.0193                               | 0.311                                      | 5.7                 | 43.6                          | 22.81                                     |
| 90                                               | 0.0299                               | 0.499                                      | 5.0                 | 49.7                          | 36.02                                     |
| 88                                               |                                      |                                            |                     |                               |                                           |
| 86                                               |                                      |                                            |                     |                               |                                           |
| 85                                               |                                      |                                            |                     |                               |                                           |
| 84                                               |                                      |                                            |                     |                               |                                           |
| 82                                               |                                      |                                            |                     |                               |                                           |
| 80                                               |                                      |                                            |                     |                               |                                           |

(133)

Table 46. Results of Run No. 40

percent of feed accounted for ..... 94.8 wt. %  
total ethanol sorbed ..... 0.997 gm.  
total water sorbed ..... 2.84 gm.  
total water sorbed per gm. barley ..... 0.0473 gm./gm.

| ETH. CONC.<br>OF COMBINED<br>EFFLUENT<br>SAMPLES | GM. WATER<br>SORBED PER<br>GM. GRAIN | ML. PURE<br>ETH. CONC.<br>PER GM.<br>GRAIN | % OF ETH.<br>SORBED | % ETH. IN<br>SORBED<br>LIQUID | TIME<br>REQUIRED<br>FOR THIS<br>(MINUTES) |
|--------------------------------------------------|--------------------------------------|--------------------------------------------|---------------------|-------------------------------|-------------------------------------------|
| 99                                               | 0.0144                               | 0.0708                                     | 12.0                | 34.7                          | 11.94                                     |
| 98                                               | 0.0163                               | 0.0850                                     | 10.8                | 33.2                          | 13.91                                     |
| 97                                               | 0.0193                               | 0.107                                      | 9.5                 | 31.4                          | 16.94                                     |
| 96                                               | 0.0234                               | 0.139                                      | 8.2                 | 29.5                          | 21.35                                     |
| 95                                               | 0.0247                               | 0.155                                      | 7.5                 | 28.8                          | 23.63                                     |
| 94                                               | 0.0263                               | 0.177                                      | 6.8                 | 28.0                          | 26.62                                     |
| 92                                               | 0.0302                               | 0.240                                      | 4.7                 | 23.8                          | 34.99                                     |
| 90                                               | 0.0354                               | 0.338                                      | 3.7                 | 22.6                          | 48.28                                     |
| 88                                               | 0.0402                               | 0.487                                      | 2.6                 | 20.3                          | 68.47                                     |
| 86                                               |                                      |                                            |                     |                               |                                           |
| 85                                               |                                      |                                            |                     |                               |                                           |
| 84                                               |                                      |                                            |                     |                               |                                           |
| 82                                               |                                      |                                            |                     |                               |                                           |
| 80                                               |                                      |                                            |                     |                               |                                           |

Table 47. Results of Run No. 41

percent of feed accounted for ..... 90.2 wt. %  
total ethanol sorbed ..... 1.01 gm.  
total water sorbed ..... 2.32 gm.  
total water sorbed per gm. barley ..... 0.0387 gm./gm.

| ETH. CONC.<br>OF COMBINED<br>EFFLUENT<br>SAMPLES | GM. WATER<br>SORBED PER<br>GM. GRAIN | ML. PURE<br>ETH. CONC.<br>PER GM.<br>GRAIN | % OF ETH.<br>SORBED | % ETH. IN<br>SORBED<br>LIQUID | TIME<br>REQUIRED<br>FOR THIS<br>(MINUTES) |
|--------------------------------------------------|--------------------------------------|--------------------------------------------|---------------------|-------------------------------|-------------------------------------------|
| 99                                               | 0.0137                               | 0.0725                                     | 9.19                | 29.7                          | 11.98                                     |
| 98                                               | 0.0159                               | 0.0897                                     | 7.26                | 25.9                          | 14.34                                     |
| 97                                               | 0.0195                               | 0.118                                      | 5.21                | 20.9                          | 18.26                                     |
| 96                                               | 0.0222                               | 0.146                                      | 2.90                | 13.5                          | 22.08                                     |
| 95                                               | 0.0232                               | 0.163                                      | 2.58                | 12.8                          | 24.41                                     |
| 94                                               | 0.0245                               | 0.184                                      | 2.25                | 12.0                          | 27.40                                     |
| 92                                               | 0.0273                               | 0.244                                      | --                  | --                            | 35.76                                     |
| 90                                               | 0.0299                               | 0.332                                      | --                  | --                            | 48.02                                     |
| 88                                               |                                      |                                            |                     |                               |                                           |
| 86                                               |                                      |                                            |                     |                               |                                           |
| 85                                               |                                      |                                            |                     |                               |                                           |
| 84                                               |                                      |                                            |                     |                               |                                           |
| 82                                               |                                      |                                            |                     |                               |                                           |
| 80                                               |                                      |                                            |                     |                               |                                           |

Table 48. Results of Run No. 43

percent of feed accounted for ..... 93.4 wt. %  
total ethanol sorbed ..... 0.555 gm.  
total water sorbed ..... 1.87 gm.  
total water sorbed per gm. barley ..... 0.0312 gm./gm.

| ETH. CONC.<br>OF COMBINED<br>EFFLUENT<br>SAMPLES | GM. WATER<br>SORBED PER<br>GM. GRAIN | ML. PURE<br>ETH. CONC.<br>PER GM.<br>GRAIN | % OF ETH.<br>SORBED | % ETH. IN<br>SORBED<br>LIQUID | TIME<br>REQUIRED<br>FOR THIS<br>(MINUTES) |
|--------------------------------------------------|--------------------------------------|--------------------------------------------|---------------------|-------------------------------|-------------------------------------------|
| 99                                               | 0.0113                               | 0.0587                                     | 7.88                | 25.9                          | 10.05                                     |
| 98                                               | 0.0129                               | 0.0713                                     | 6.03                | 21.9                          | 11.77                                     |
| 97                                               | 0.0138                               | 0.0807                                     | 5.45                | 21.0                          | 13.04                                     |
| 96                                               | 0.0151                               | 0.0932                                     | 4.84                | 19.9                          | 14.73                                     |
| 95                                               | 0.0168                               | 0.0111                                     | 4.22                | 18.6                          | 17.09                                     |
| 94                                               | 0.0194                               | 0.137                                      | 3.57                | 17.1                          | 20.64                                     |
| 92                                               | 0.0212                               | 0.174                                      | 2.63                | 14.9                          | 25.67                                     |
| 90                                               | 0.0236                               | 0.234                                      | 1.72                | 12.1                          | 33.71                                     |
| 88                                               | 0.0261                               | 0.332                                      | --                  | --                            | 46.80                                     |
| 86                                               | 0.0291                               | 0.515                                      | --                  | --                            | 71.65                                     |
| 85                                               |                                      |                                            |                     |                               |                                           |
| 84                                               |                                      |                                            |                     |                               |                                           |
| 82                                               |                                      |                                            |                     |                               |                                           |
| 80                                               |                                      |                                            |                     |                               |                                           |

(136)

Table 49. Results of Run No. 44

|                                         |               |         |
|-----------------------------------------|---------------|---------|
| percent of feed accounted for .....     | <u>94.2</u>   | wt. %   |
| total ethanol sorbed .....              | <u>0.999</u>  | gm.     |
| total water sorbed .....                | <u>3.22</u>   | gm.     |
| total water sorbed per gm. barley ..... | <u>0.0537</u> | gm./gm. |

| ETH. CONC.<br>OF COMBINED<br>EFFLUENT<br>SAMPLES | GM. WATER<br>SORBED PER<br>GM. GRAIN | ML. PURE<br>ETH. CONC.<br>PER GM.<br>GRAIN | % OF ETH.<br>SORBED | % ETH. IN<br>SORBED<br>LIQUID | TIME<br>REQUIRED<br>FOR THIS<br>(MINUTES) |
|--------------------------------------------------|--------------------------------------|--------------------------------------------|---------------------|-------------------------------|-------------------------------------------|
| 99                                               | 0.0165                               | 0.0842                                     | 9.14                | 28.9                          | 13.37                                     |
| 98                                               | 0.0251                               | 0.142                                      | 3.99                | 15.7                          | 21.23                                     |
| 97                                               | 0.0268                               | 0.160                                      | 3.48                | 14.5                          | 23.76                                     |
| 96                                               | 0.0292                               | 0.185                                      | 2.95                | 13.2                          | 27.13                                     |
| 95                                               | 0.0335                               | 0.217                                      | 5.45                | 22.8                          | 31.50                                     |
| 94                                               | 0.0354                               | 0.246                                      | 4.82                | 21.7                          | 35.25                                     |
| 92                                               | 0.0404                               | 0.328                                      | 3.47                | 18.7                          | 46.24                                     |
| 90                                               | 0.0467                               | 0.457                                      | 2.51                | 16.6                          | 63.39                                     |
| 88                                               |                                      |                                            |                     |                               |                                           |
| 86                                               |                                      |                                            |                     |                               |                                           |
| 85                                               |                                      |                                            |                     |                               |                                           |
| 84                                               |                                      |                                            |                     |                               |                                           |
| 82                                               |                                      |                                            |                     |                               |                                           |
| 80                                               |                                      |                                            |                     |                               |                                           |

(137)

Table 50. Results of Run No. 45

percent of feed accounted for ..... 93.1 wt. %  
total ethanol sorbed ..... 0.981 gm.  
total water sorbed ..... 1.99 gm.  
total water sorbed per gm. barley ..... 0.0331 gm./gm.

| ETH. CONC.<br>OF COMBINED<br>EFFLUENT<br>SAMPLES | GM. WATER<br>SORBED PER<br>GM. GRAIN | ML. PURE<br>ETH. CONC.<br>PER GM.<br>GRAIN | % OF ETH.<br>SORBED | % ETH. IN<br>SORBED<br>LIQUID | TIME<br>REQUIRED<br>FOR THIS<br>(MINUTES) |
|--------------------------------------------------|--------------------------------------|--------------------------------------------|---------------------|-------------------------------|-------------------------------------------|
| 99                                               | 0.0121                               | 0.0528                                     | 27.1                | 56.1                          | 5.30                                      |
| 98                                               | 0.0140                               | 0.0733                                     | 16.0                | 44.2                          | 6.25                                      |
| 97                                               | 0.0160                               | 0.0947                                     | 9.3                 | 32.8                          | 7.35                                      |
| 96                                               | 0.0167                               | 0.106                                      | 8.9                 | 32.9                          | 8.13                                      |
| 95                                               | 0.0179                               | 0.121                                      | 8.5                 | 33.2                          | 9.14                                      |
| 94                                               | 0.0194                               | 0.140                                      | 8.4                 | 34.2                          | 10.44                                     |
| 92                                               | 0.0219                               | 0.185                                      | 7.2                 | 34.2                          | 13.42                                     |
| 90                                               | 0.0281                               | 0.392                                      | 3.4                 | 27.8                          | 26.85                                     |
| 88                                               |                                      |                                            |                     |                               |                                           |
| 86                                               |                                      |                                            |                     |                               |                                           |
| 85                                               |                                      |                                            |                     |                               |                                           |
| 84                                               |                                      |                                            |                     |                               |                                           |
| 82                                               |                                      |                                            |                     |                               |                                           |
| 80                                               |                                      |                                            |                     |                               |                                           |

(138)

Table 51. Results of Run No. 46

percent of feed accounted for ..... 94.3 wt. %  
total ethanol sorbed ..... 0.777 gm.  
total water sorbed ..... 2.25 gm.  
total water sorbed per gm. barley ..... 0.0375 gm./gm.

| ETH. CONC.<br>OF COMBINED<br>EFFLUENT<br>SAMPLES | GM. WATER<br>SORBED PER<br>GM. GRAIN | ML. PURE<br>ETH. CONC.<br>PER GM.<br>GRAIN | % OF ETH.<br>SORBED | % ETH. IN<br>SORBED<br>LIQUID | TIME<br>REQUIRED<br>FOR THIS<br>(MINUTES) |
|--------------------------------------------------|--------------------------------------|--------------------------------------------|---------------------|-------------------------------|-------------------------------------------|
| 99                                               | 0.0142                               | 0.0721                                     | 9.69                | 30.1                          | 11.74                                     |
| 98                                               | 0.0161                               | 0.0863                                     | 8.51                | 28.3                          | 13.69                                     |
| 97                                               | 0.0189                               | 0.108                                      | 7.28                | 26.1                          | 16.64                                     |
| 96                                               | 0.0232                               | 0.141                                      | 6.08                | 23.7                          | 21.21                                     |
| 95                                               | 0.0243                               | 0.157                                      | 5.59                | 23.2                          | 23.39                                     |
| 94                                               | 0.0258                               | 0.178                                      | 5.10                | 22.7                          | 26.19                                     |
| 92                                               | 0.0291                               | 0.235                                      | 3.96                | 20.8                          | 33.81                                     |
| 90                                               | 0.0325                               | 0.315                                      | 2.84                | 18.3                          | 44.62                                     |
| 88                                               |                                      |                                            |                     |                               |                                           |
| 86                                               |                                      |                                            |                     |                               |                                           |
| 85                                               |                                      |                                            |                     |                               |                                           |
| 84                                               |                                      |                                            |                     |                               |                                           |
| 82                                               |                                      |                                            |                     |                               |                                           |
| 80                                               |                                      |                                            |                     |                               |                                           |

Table 52. Results of Run No. 47

percent of feed accounted for ..... 89.0 wt. %  
total ethanol sorbed ..... 0.94 gm.  
total water sorbed ..... 2.62 gm.  
total water sorbed per gm. barley ..... 0.0437 gm./gm.

| ETH. CONC.<br>OF COMBINED<br>EFFLUENT<br>SAMPLES | GM. WATER<br>SORBED PER<br>GM. GRAIN | ML. PURE<br>ETH. CONC.<br>PER GM.<br>GRAIN | % OF ETH.<br>SORBED | % ETH. IN<br>SORBED<br>LIQUID | TIME<br>REQUIRED<br>FOR THIS<br>(MINUTES) |
|--------------------------------------------------|--------------------------------------|--------------------------------------------|---------------------|-------------------------------|-------------------------------------------|
| 99                                               | 0.0309                               | 0.164                                      | 5.75                | 20.9                          | 146.4                                     |
| 98                                               | 0.0344                               | 0.201                                      | 4.13                | 16.6                          | 170.8                                     |
| 97                                               | 0.0371                               | 0.229                                      | 3.60                | 15.4                          | 193.4                                     |
| 96                                               | 0.0396                               | 0.260                                      | 3.18                | 14.6                          | 218.2                                     |
| 95                                               | 0.0415                               | 0.291                                      | 2.66                | 13.2                          | 240.5                                     |
| 94                                               |                                      |                                            |                     |                               |                                           |
| 92                                               |                                      |                                            |                     |                               |                                           |
| 90                                               |                                      |                                            |                     |                               |                                           |
| 88                                               |                                      |                                            |                     |                               |                                           |
| 86                                               |                                      |                                            |                     |                               |                                           |
| 85                                               |                                      |                                            |                     |                               |                                           |
| 84                                               |                                      |                                            |                     |                               |                                           |
| 82                                               |                                      |                                            |                     |                               |                                           |
| 80                                               |                                      |                                            |                     |                               |                                           |

(140)

Table 53. Results of Run No. 48

percent of feed accounted for ..... 94.5 wt. %  
 total ethanol sorbed ..... 0.681 gm.  
 total water sorbed ..... 3.459 gm.  
 total water sorbed per gm. barley ..... 0.057 gm./gm.

| ETH. CONC.<br>OF COMBINED<br>EFFLUENT<br>SAMPLES | GM. WATER<br>SORBED PER<br>GM. GRAIN | ML. PURE<br>ETH. CONC.<br>PER GM.<br>GRAIN | % OF ETH.<br>SORBED | % ETH. IN<br>SORBED<br>LIQUID | TIME<br>REQUIRED<br>FOR THIS<br>(MINUTES) |
|--------------------------------------------------|--------------------------------------|--------------------------------------------|---------------------|-------------------------------|-------------------------------------------|
| 99                                               | 0.0190                               | 0.0640                                     | 8.95                | 20.7                          | 11.38                                     |
| 98                                               | 0.0198                               | 0.0691                                     | 8.40                | 20.2                          | 12.12                                     |
| 97                                               | 0.0208                               | 0.0752                                     | 7.82                | 19.5                          | 13.01                                     |
| 96                                               | 0.0220                               | 0.0827                                     | 7.23                | 18.8                          | 14.09                                     |
| 95                                               | 0.0235                               | 0.0921                                     | 6.62                | 18.0                          | 15.44                                     |
| 94                                               | 0.0255                               | 0.104                                      | 5.98                | 17.0                          | 17.19                                     |
| 92                                               | 0.0302                               | 0.136                                      | 4.30                | 13.8                          | 21.84                                     |
| 90                                               | 0.0335                               | 0.167                                      | 3.84                | 13.6                          | 26.30                                     |
| 88                                               | 0.0379                               | 0.212                                      | 2.95                | 11.9                          | 32.95                                     |
| 86                                               | 0.0426                               | 0.276                                      | 1.93                | 9.2                           | 42.09                                     |
| 85                                               |                                      |                                            |                     |                               |                                           |
| 84                                               |                                      |                                            |                     |                               |                                           |
| 82                                               |                                      |                                            |                     |                               |                                           |
| 80                                               |                                      |                                            |                     |                               |                                           |

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Table 54. Results of Run No. 49

percent of feed accounted for ..... 92.2 wt. %  
 total ethanol sorbed ..... 0.475 gm.  
 total water sorbed ..... 1.59 gm.  
 total water sorbed per gm. barley ..... 0.0265 gm./gm.

| ETH. CONC.<br>OF COMBINED<br>EFFLUENT<br>SAMPLES | GM. WATER<br>SORBED PER<br>GM. GRAIN | ML. PURE<br>ETH. CONC.<br>PER GM.<br>GRAIN | % OF ETH.<br>SORBED | % ETH. IN<br>SORBED<br>LIQUID | TIME<br>REQUIRED<br>FOR THIS<br>(MINUTES) |
|--------------------------------------------------|--------------------------------------|--------------------------------------------|---------------------|-------------------------------|-------------------------------------------|
| 99                                               | 0.0105                               | 0.0942                                     | 5.81                | 30.3                          | 14.22                                     |
| 98                                               | 0.0174                               | 0.174                                      | 2.70                | 18.0                          | 24.49                                     |
| 97                                               | 0.0202                               | 0.221                                      | 2.35                | 17.3                          | 30.75                                     |
| 96                                               | 0.0220                               | 0.270                                      | 1.27                | 11.1                          | 36.80                                     |
| 95                                               | 0.0240                               | 0.336                                      | --                  | --                            | 45.25                                     |
| 94                                               |                                      |                                            |                     |                               |                                           |
| 92                                               |                                      |                                            |                     |                               |                                           |
| 90                                               |                                      |                                            |                     |                               |                                           |
| 88                                               |                                      |                                            |                     |                               |                                           |
| 86                                               |                                      |                                            |                     |                               |                                           |
| 85                                               |                                      |                                            |                     |                               |                                           |
| 84                                               |                                      |                                            |                     |                               |                                           |
| 82                                               |                                      |                                            |                     |                               |                                           |
| 80                                               |                                      |                                            |                     |                               |                                           |

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Table 55. Results of Run No. 50

percent of feed accounted for ..... 89.1 wt. %  
total ethanol sorbed ..... 2.80 gm.  
total water sorbed ..... 5.00 gm.  
total water sorbed per gm. barley ..... 0.0833 gm./gm.

| ETH. CONC.<br>OF COMBINED<br>EFFLUENT<br>SAMPLES | GM. WATER<br>SORBED PER<br>GM. GRAIN | ML. PURE<br>ETH. CONC.<br>PER GM.<br>GRAIN | % OF ETH.<br>SORBED | % ETH. IN<br>SORBED<br>LIQUID | TIME<br>REQUIRED<br>FOR THIS<br>(MINUTES) |
|--------------------------------------------------|--------------------------------------|--------------------------------------------|---------------------|-------------------------------|-------------------------------------------|
| 99                                               | 0.0755                               | 0.302                                      | 10.9                | 28.0                          | 285.2                                     |
| 98                                               |                                      |                                            |                     |                               |                                           |
| 97                                               |                                      |                                            |                     |                               |                                           |
| 96                                               |                                      |                                            |                     |                               |                                           |
| 95                                               |                                      |                                            |                     |                               |                                           |
| 94                                               |                                      |                                            |                     |                               |                                           |
| 92                                               |                                      |                                            |                     |                               |                                           |
| 90                                               |                                      |                                            |                     |                               |                                           |
| 88                                               |                                      |                                            |                     |                               |                                           |
| 86                                               |                                      |                                            |                     |                               |                                           |
| 85                                               |                                      |                                            |                     |                               |                                           |
| 84                                               |                                      |                                            |                     |                               |                                           |
| 82                                               |                                      |                                            |                     |                               |                                           |
| 80                                               |                                      |                                            |                     |                               |                                           |

Table 56. Results of Run No. 53

percent of feed accounted for ..... 90.4 wt. %  
 total ethanol sorbed ..... 1.53 gm.  
 total water sorbed ..... 8.24 gm.  
 total water sorbed per gm. barley ..... 0.137 gm./gm.

| ETH. CONC.<br>OF COMBINED<br>EFFLUENT<br>SAMPLES | GM. WATER<br>SORBED PER<br>GM. GRAIN | ML. PURE<br>ETH. CONC.<br>PER GM.<br>GRAIN | % OF ETH.<br>SORBED | % ETH. IN<br>SORBED<br>LIQUID | TIME<br>REQUIRED<br>FOR THIS<br>(MINUTES) |
|--------------------------------------------------|--------------------------------------|--------------------------------------------|---------------------|-------------------------------|-------------------------------------------|
| 99                                               | 0.0967                               | 0.138                                      | 10.5                | 11.7                          | 173.8                                     |
| 98                                               | 0.107                                | 0.155                                      | 10.5                | 11.8                          | 195.4                                     |
| 97                                               | 0.115                                | 0.168                                      | 10.6                | 12.0                          | 209.7                                     |
| 96                                               | 0.121                                | 0.181                                      | 10.1                | 11.7                          | 222.9                                     |
| 95                                               | 0.127                                | 0.193                                      | 9.5                 | 11.2                          | 235.7                                     |
| 94                                               |                                      |                                            |                     |                               |                                           |
| 92                                               |                                      |                                            |                     |                               |                                           |
| 90                                               |                                      |                                            |                     |                               |                                           |
| 88                                               |                                      |                                            |                     |                               |                                           |
| 86                                               |                                      |                                            |                     |                               |                                           |
| 85                                               |                                      |                                            |                     |                               |                                           |
| 84                                               |                                      |                                            |                     |                               |                                           |
| 82                                               |                                      |                                            |                     |                               |                                           |
| 80                                               |                                      |                                            |                     |                               |                                           |

Table 57. Results of Run No. 54

percent of feed accounted for ..... 93.6 wt. %  
total ethanol sorbed ..... 1.35 gm.  
total water sorbed ..... 3.88 gm.  
total water sorbed per gm. barley ..... 0.0499 gm./gm.

| ETH. CONC.<br>OF COMBINED<br>EFFLUENT<br>SAMPLES | GM. WATER<br>SORBED PER<br>GM. GRAIN | ML. PURE<br>ETH. CONC.<br>PER GM.<br>GRAIN | % OF ETH.<br>SORBED | % ETH. IN<br>SORBED<br>LIQUID | TIME<br>REQUIRED<br>FOR THIS<br>(MINUTES) |
|--------------------------------------------------|--------------------------------------|--------------------------------------------|---------------------|-------------------------------|-------------------------------------------|
| 99                                               | 0.0274                               | 0.105                                      | 9.82                | 24.8                          | 123.0                                     |
| 98                                               | 0.0336                               | 0.135                                      | 8.50                | 22.8                          | 153.3                                     |
| 97                                               | 0.0394                               | 0.165                                      | 7.84                | 22.0                          | 185.2                                     |
| 96                                               | 0.0449                               | 0.197                                      | 6.75                | 20.1                          | 219.3                                     |
| 95                                               |                                      |                                            |                     |                               |                                           |
| 94                                               |                                      |                                            |                     |                               |                                           |
| 92                                               |                                      |                                            |                     |                               |                                           |
| 90                                               |                                      |                                            |                     |                               |                                           |
| 88                                               |                                      |                                            |                     |                               |                                           |
| 86                                               |                                      |                                            |                     |                               |                                           |
| 85                                               |                                      |                                            |                     |                               |                                           |
| 84                                               |                                      |                                            |                     |                               |                                           |
| 82                                               |                                      |                                            |                     |                               |                                           |
| 80                                               |                                      |                                            |                     |                               |                                           |

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Table 58. Results of Run No. 55

percent of feed accounted for ..... 89.7 wt. %  
total ethanol sorbed ..... 1.29 gm.  
total water sorbed ..... 7.71 gm.  
total water sorbed per gm. barley ..... 0.131 gm./gm.

| ETH. CONC.<br>OF COMBINED<br>EFFLUENT<br>SAMPLES | GM. WATER<br>SORBED PER<br>GM. GRAIN | ML. PURE<br>ETH. CONC.<br>PER GM.<br>GRAIN | % OF ETH.<br>SORBED | % ETH. IN<br>SORBED<br>LIQUID | TIME<br>REQUIRED<br>FOR THIS<br>(MINUTES) |
|--------------------------------------------------|--------------------------------------|--------------------------------------------|---------------------|-------------------------------|-------------------------------------------|
| 99                                               | 0.0999                               | 0.143                                      | 8.81                | 9.82                          | 175.2                                     |
| 98                                               | 0.110                                | 0.161                                      | 7.34                | 8.41                          | 193.6                                     |
| 97                                               | 0.118                                | 0.176                                      | 7.29                | 8.46                          | 210.3                                     |
| 96                                               |                                      |                                            |                     |                               |                                           |
| 95                                               |                                      |                                            |                     |                               |                                           |
| 94                                               |                                      |                                            |                     |                               |                                           |
| 92                                               |                                      |                                            |                     |                               |                                           |
| 90                                               |                                      |                                            |                     |                               |                                           |
| 88                                               |                                      |                                            |                     |                               |                                           |
| 86                                               |                                      |                                            |                     |                               |                                           |
| 85                                               |                                      |                                            |                     |                               |                                           |
| 84                                               |                                      |                                            |                     |                               |                                           |
| 82                                               |                                      |                                            |                     |                               |                                           |
| 80                                               |                                      |                                            |                     |                               |                                           |

Table 59. Results of Run No. 56

percent of feed accounted for ..... 88.3 wt. %  
total ethanol sorbed ..... 0.894 gm.  
total water sorbed ..... 3.72 gm.  
total water sorbed per gm. barley ..... 0.0826 gm./gm.

| ETH. CONC.<br>OF COMBINED<br>EFFLUENT<br>SAMPLES | GM. WATER<br>SORBED PER<br>GM. GRAIN | ML. PURE<br>ETH. CONC.<br>PER GM.<br>GRAIN | % OF ETH.<br>SORBED | % ETH. IN<br>SORBED<br>LIQUID | TIME<br>REQUIRED<br>FOR THIS<br>(MINUTES) |
|--------------------------------------------------|--------------------------------------|--------------------------------------------|---------------------|-------------------------------|-------------------------------------------|
| 99                                               | 0.0583                               | 0.211                                      | 7.55                | 18.9                          | 151.1                                     |
| 98                                               | 0.0647                               | 0.243                                      | 6.74                | 17.7                          | 171.3                                     |
| 97                                               | 0.0687                               | 0.268                                      | 6.29                | 17.1                          | 187.0                                     |
| 96                                               | 0.0719                               | 0.291                                      | 5.98                | 16.9                          | 201.8                                     |
| 95                                               | 0.0740                               | 0.313                                      | 5.29                | 15.7                          | 214.9                                     |
| 94                                               | 0.0759                               | 0.336                                      | 4.84                | 15.1                          | 228.0                                     |
| 92                                               | 0.0786                               | 0.388                                      | --                  | --                            | 257.3                                     |
| 90                                               | 0.0825                               | 0.464                                      | --                  | --                            | 299.7                                     |
| 88                                               |                                      |                                            |                     |                               |                                           |
| 86                                               |                                      |                                            |                     |                               |                                           |
| 85                                               |                                      |                                            |                     |                               |                                           |
| 84                                               |                                      |                                            |                     |                               |                                           |
| 82                                               |                                      |                                            |                     |                               |                                           |
| 80                                               |                                      |                                            |                     |                               |                                           |

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Table 60. Results of Run No. 57

percent of feed accounted for ..... 89.0 wt. %  
total ethanol sorbed ..... 1.02 gm.  
total water sorbed ..... 2.89 gm.  
total water sorbed per gm. barley ..... 0.083 gm./gm.

| ETH. CONC.<br>OF COMBINED<br>EFFLUENT<br>SAMPLES | GM. WATER<br>SORBED PER<br>GM. GRAIN | ML. PURE<br>ETH. CONC.<br>PER GM.<br>GRAIN | % OF ETH.<br>SORBED | % ETH. IN<br>SORBED<br>LIQUID | TIME<br>REQUIRED<br>FOR THIS<br>(MINUTES) |
|--------------------------------------------------|--------------------------------------|--------------------------------------------|---------------------|-------------------------------|-------------------------------------------|
| 99                                               | 0.0562                               | 0.212                                      | 8.77                | 22.2                          | 122.6                                     |
| 98                                               | 0.0593                               | 0.233                                      | 7.81                | 20.8                          | 132.4                                     |
| 97                                               | 0.0627                               | 0.256                                      | 7.36                | 20.4                          | 143.0                                     |
| 96                                               | 0.0643                               | 0.275                                      | 6.56                | 19.2                          | 151.3                                     |
| 95                                               | 0.0662                               | 0.297                                      | 5.74                | 17.7                          | 161.0                                     |
| 94                                               | 0.0675                               | 0.318                                      | 5.06                | 16.6                          | 172.5                                     |
| 92                                               | 0.0712                               | 0.369                                      | 4.54                | 16.3                          | 198.4                                     |
| 90                                               | 0.0737                               | 0.433                                      | 3.38                | 14.0                          | 225.6                                     |
| 88                                               |                                      |                                            |                     |                               |                                           |
| 86                                               |                                      |                                            |                     |                               |                                           |
| 85                                               |                                      |                                            |                     |                               |                                           |
| 84                                               |                                      |                                            |                     |                               |                                           |
| 82                                               |                                      |                                            |                     |                               |                                           |
| 80                                               |                                      |                                            |                     |                               |                                           |

Table 61. Results of Run No. 58

percent of feed accounted for ..... 86.7 wt. %  
total ethanol sorbed ..... 1.37 gm.  
total water sorbed ..... 3.86 gm.  
total water sorbed per gm. barley ..... 0.0653 gm./gm.

| ETH. CONC.<br>OF COMBINED<br>EFFLUENT<br>SAMPLES | GM. WATER<br>SORBED PER<br>GM. GRAIN | ML. PURE<br>ETH. CONC.<br>PER GM.<br>GRAIN | % OF ETH.<br>SORBED | % ETH. IN<br>SORBED<br>LIQUID | TIME<br>REQUIRED<br>FOR THIS<br>(MINUTES) |
|--------------------------------------------------|--------------------------------------|--------------------------------------------|---------------------|-------------------------------|-------------------------------------------|
| 99                                               | --                                   | --                                         | --                  | --                            | --                                        |
| 98                                               | 0.0481                               | 0.190                                      | 7.31                | 19.8                          | 184.4                                     |
| 97                                               | 0.0524                               | 0.219                                      | 5.50                | 16.1                          | 206.4                                     |
| 96                                               | 0.0560                               | 0.245                                      | 4.61                | 14.3                          | 228.1                                     |
| 95                                               | 0.0594                               | 0.271                                      | 4.28                | 13.9                          | 250.3                                     |
| 94                                               |                                      |                                            |                     |                               |                                           |
| 92                                               |                                      |                                            |                     |                               |                                           |
| 90                                               |                                      |                                            |                     |                               |                                           |
| 88                                               |                                      |                                            |                     |                               |                                           |
| 86                                               |                                      |                                            |                     |                               |                                           |
| 85                                               |                                      |                                            |                     |                               |                                           |
| 84                                               |                                      |                                            |                     |                               |                                           |
| 82                                               |                                      |                                            |                     |                               |                                           |
| 80                                               |                                      |                                            |                     |                               |                                           |

Table 62. Results of Run No. 59

percent of feed accounted for ..... 85.9 wt. %  
total ethanol sorbed ..... 1.18 gm.  
total water sorbed ..... 3.03 gm.  
total water sorbed per gm. barley ..... 0.0517 gm./gm.

| ETH. CONC.<br>OF COMBINED<br>EFFLUENT<br>SAMPLES | GM. WATER<br>SORBED PER<br>GM. GRAIN | ML. PURE<br>ETH. CONC.<br>PER GM.<br>GRAIN | % OF ETH.<br>SORBED | % ETH. IN<br>SORBED<br>LIQUID | TIME<br>REQUIRED<br>FOR THIS<br>(MINUTES) |
|--------------------------------------------------|--------------------------------------|--------------------------------------------|---------------------|-------------------------------|-------------------------------------------|
| 99                                               | --                                   | --                                         | --                  | --                            | --                                        |
| 98                                               | 0.0271                               | 0.108                                      | 6.99                | 19.1                          | 111.4                                     |
| 97                                               | 0.0402                               | 0.169                                      | 5.04                | 15.0                          | 163.8                                     |
| 96                                               | 0.0444                               | 0.195                                      | 4.55                | 14.2                          | 184.8                                     |
| 95                                               |                                      |                                            |                     |                               |                                           |
| 94                                               |                                      |                                            |                     |                               |                                           |
| 92                                               |                                      |                                            |                     |                               |                                           |
| 90                                               |                                      |                                            |                     |                               |                                           |
| 88                                               |                                      |                                            |                     |                               |                                           |
| 86                                               |                                      |                                            |                     |                               |                                           |
| 85                                               |                                      |                                            |                     |                               |                                           |
| 84                                               |                                      |                                            |                     |                               |                                           |
| 82                                               |                                      |                                            |                     |                               |                                           |
| 80                                               |                                      |                                            |                     |                               |                                           |

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APPENDIX D

TEMPERATURE DATA FOR RUNS 26, 28, 60

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Table 63. Run No. 26 Temperature Data

Date: 3-11-81

| TIME<br>(MIN) | SAMPLE<br>NO. | BURETTE<br>(ML) | TEMP ( ° C ) |     |    |    |    |    |    |
|---------------|---------------|-----------------|--------------|-----|----|----|----|----|----|
|               |               |                 | 1            | 2   | 3  | 4  | 5  | 6  | 7  |
| 0             | BEFORE VAPOR  |                 | 89           | 94  | 86 | 86 | 86 | 86 | 84 |
| 0             | AFTER VAPOR   |                 | 88           | 96  | 86 | 86 | 86 | 86 | 85 |
| 20            | 1             | 2.0             | 89           | 111 | 86 | 86 | 86 | 86 | 84 |
| 40            | 2             | 4.1             | 86           | 112 | 86 | 87 | 86 | 86 | 84 |
| 60            | 3             | 6.1             | 86           | 112 | 86 | 87 | 87 | 86 | 84 |
| 80            | 4             | 8.1             | 86           | 112 | 86 | 87 | 87 | 85 | 84 |
| 100           | 5             | 10.2            | 88           | 113 | 87 | 87 | 87 | 86 | 84 |
| 120           | 6             | 12.2            | 88           | 111 | 86 | 87 | 87 | 85 | 84 |
| 140           | 7             | 14.2            | 89           | 111 | 84 | 85 | 86 | 85 | 84 |
| 160           | 8             | 16.2            | 86           | 113 | 86 | 87 | 87 | 86 | 83 |
| 180           | 9             | 18.3            | 87           | 111 | 85 | 86 | 87 | 86 | 84 |
| 200           | 10            | 20.3            | 87           | 111 | 86 | 87 | 87 | 86 | 84 |
| 220           | 11            | 22.3            | 87           | 112 | 86 | 87 | 87 | 86 | 85 |
| 240           | 12            | 24.3            | 87           | 112 | 86 | 87 | 87 | 86 | 85 |
| 260           | 13            | 26.3            | 88           | 114 | 86 | 86 | 87 | 86 | 85 |
| 280           | 14            | 28.3            | 89           | 113 | 86 | 86 | 87 | 86 | 85 |
| 300           | 15            | 30.2            | 88           | 113 | 86 | 86 | 87 | 86 | 85 |
| 320           | 16            | 32.2            | 89           | 114 | 86 | 87 | 86 | 86 | 85 |
| 340           | 17            | 34.2            | 88           | 114 | 86 | 86 | 87 | 86 | 85 |
| 360           | 18            | 36.2            | 90           | 114 | 86 | 86 | 86 | 86 | 85 |
| 380           | 19            | 38.2            | 88           | 115 | 86 | 86 | 86 | 86 | 85 |
| 400           | 20            | 40.2            | 89           | 116 | 86 | 87 | 87 | 86 | 86 |

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Table 64. Run No. 28 Temperature Data

Date: 3-20-81

| TIME<br>(MIN) | SAMPLE<br>NO. | BUTETTE<br>(ML) | TEMP ( ° C ) |     |    |    |    |    |    |
|---------------|---------------|-----------------|--------------|-----|----|----|----|----|----|
|               |               |                 | 1            | 2   | 3  | 4  | 5  | 6  | 7  |
| 0             | BEFORE VAPOR  |                 | 95           | 95  | 86 | 86 | 86 | 86 | 85 |
| 0             | AFTER VAPOR   |                 | 82           | 102 | 87 | 87 | 86 | 86 | 85 |
| 4             | 1             | 4.0             | 102          | 82  | 85 | 95 | 93 | 88 | 86 |
| 10            | 2             | 9.9             | 95           | 92  | 88 | 96 | 97 | 95 | 92 |
| 15            | 3             | 14.9            | 96           | 94  | 86 | 94 | 96 | 96 | 93 |
| 20            | 4             | 19.9            | 96           | 93  | 86 | 94 | 96 | 95 | 94 |
| 25            | 5             | 24.9            | 96           | 95  | 86 | 93 | 96 | 95 | 94 |
| 30            | 6             | 29.9            | 96           | 94  | 86 | 93 | 95 | 94 | 94 |
| 35            | 7             | 35.0            | 97           | 94  | 86 | 92 | 94 | 94 | 94 |
| 40            | 8             | 40.0            | 97           | 93  | 86 | 92 | 94 | 94 | 94 |
| 45            | 9             | 45.1            | 95           | 93  | 86 | 93 | 94 | 94 | 93 |
| 50            | 10            | 50.1            | 95           | 93  | 86 | 92 | 93 | 94 | 93 |
| 55            | 11            | 12.2            | 95           | 93  | 86 | 92 | 93 | 94 | 93 |
| 60            | 12            | 17.3            | 96           | 93  | 86 | 91 | 93 | 93 | 92 |
|               | 13            |                 |              |     |    |    |    |    |    |
|               | 14            |                 |              |     |    |    |    |    |    |
|               | 15            |                 |              |     |    |    |    |    |    |
|               | 16            |                 |              |     |    |    |    |    |    |
|               | 17            |                 |              |     |    |    |    |    |    |
|               | 18            |                 |              |     |    |    |    |    |    |
|               | 19            |                 |              |     |    |    |    |    |    |
|               | 20            |                 |              |     |    |    |    |    |    |

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Table 65. Run No. 60 Temperature Data

Date: 7-16-81

| TIME<br>(MIN) | SAMPLE<br>NO. | BURETTE<br>(ML) | TEMP ( °C ) |     |    |   |   |     |     |
|---------------|---------------|-----------------|-------------|-----|----|---|---|-----|-----|
|               |               |                 | 1           | 2   | 3  | 4 | 5 | 6   | 7   |
| 0             | BEFORE VAPOR  |                 | 10          | 10  | 86 |   |   | 86  | 86  |
| 0             | AFTER VAPOR   |                 | 100         | 100 | 86 |   |   | 87  | 87  |
| 2             |               | 2.1             | 100         | 98  | 87 |   |   | 108 | 109 |
| 4             |               | 4.2             | 98          | 98  | 98 |   |   | 105 | 108 |
| 6             |               | 6.2             | 97          | 97  | 86 |   |   | 103 | 106 |
| 8             |               | 8.3             | 97          | 95  | 86 |   |   | 101 | 104 |
| 10            |               | 10.4            | 96          | 94  | 86 |   |   | 100 | 102 |
| 12            |               | 12.5            | 97          | 97  | 86 |   |   | 100 | 102 |
| 14            |               | 14.5            | 96          | 96  | 86 |   |   | 99  | 100 |
| 16            |               | 16.5            | 92          | 96  | 86 |   |   | 98  | 99  |

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## APPENDIX E

### EXAMPLE OF CALCULATIONS

We can see how well the sorption process works at various conditions by calculating the following quantities:

- 1) percent of feed accounted for
- 2) total ethanol sorbed
- 3) total water sorbed
- 4) total water sorbed per gm. grain

The following quantities are for combined samples of a particular concentration, such as 99 wt. % ethanol:

- 5) gm. of water sorbed per gm. of grain
- 6) ml. of pure ethanol concentrated per gm. of grain
- 7) percent of feed ethanol sorbed
- 8) percent of ethanol in sorbed liquid
- 9) time required to accomplish this

The concentration and weight of the sample were used to find the weight of water and ethanol in each sample. The concentration of cumulative samples was then calculated. For example, the concentration of cumulative (combined) samples 1 and 2 was found as follows. The weight of ethanol for samples 1 and 2 was added together. The weight of water for samples 1 and 2 was added together. The cumulative concentration of ethanol was then calculated which would approximate the ethanol concentration that would result if samples 1 and 2 were actually mixed together. This same procedure was used to find the cumulative ethanol concentrations for samples 1-3, 1-4, etc.

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After the above calculations were completed, quantities 1-9 from above were found. Run No. 26 will be used as an example of these calculations. A cumulative ethanol concentration of 99 wt. % will be used.

A) Feed Additions

$$(40.3 \text{ ml.} - 0 \text{ ml.}) \cdot (0.85 \text{ gm./ml.}) = \boxed{34.26 \text{ gm.}}$$

40.3 ml. = final feed burette reading  
0 ml. = initial feed burette reading  
0.85 gm./ml. = specific gravity of feed

B) Total Weight of Liquid Samples

$$= \boxed{22.40 \text{ gm.}}$$

See Run No. 26 of "Run Conditions and Data"

C) Average Post-run Barley Moisture Content

$$(0.093 + 0.161)/2 = \boxed{0.127}$$

0.093 = mass fraction of water at top of column  
0.161 = mass fraction of water at bottom of column

D) Total Liquid Sorbed

$$\begin{aligned} & (60.0 \text{ gm. dry barley}) \\ & \cdot (1/(1-0.127)-1) = \boxed{8.73 \text{ gm.}} \end{aligned}$$

60.0 gm. dry barley = barley used as sorbent

E) Combined Weight of Liquid Samples and Sorbed Liquid

$$22.40 \text{ gm.} + 8.73 \text{ gm.} = \boxed{31.13 \text{ gm.}}$$

1) Percent of Feed accounted for

$$(31.13 \text{ gm.}/34.26 \text{ gm.}) \cdot (100) = \boxed{90.9 \text{ wt. \%}}$$

This means that of all of the feed that entered the system, 90.9 wt. % was collected and measured. The 9.1 wt. % that was not accounted for is assumed to be due to leaks in the system, as discussed earlier. It was assumed that these losses occurred evenly throughout the run and that the ratio of ethanol to water in the unaccounted for liquid was the same as the ratio of ethanol to water in the feed. Throughout the remainder of the calculations it was assumed that the actual amount of feed entering the system was equal to the amount of liquid that was accounted for.

F) Mass of Ethanol Entering the System  
 $(31.13 \text{ gm.}) \cdot (0.765) = \boxed{23.81 \text{ gm.}}$

0.765 = mass fraction of ethanol in feed

G) Mass of Water Entering the System  
 $(31.13 \text{ gm.}) \cdot (0.235) = \boxed{7.31 \text{ gm.}}$

0.235 = mass fraction of water in feed

2) Total Ethanol Sorbed  
 $23.81 \text{ gm.} - 21.534 \text{ gm.} = \boxed{2.28 \text{ gm.}}$

21.534 gm. = total ethanol in liquid samples

3) Total Water Sorbed  
 $7.32 \text{ gm.} - 0.867 \text{ gm.} = \boxed{6.45 \text{ gm.}}$

0.867 gm. = total water in liquid samples

4) Total Water Sorbed per Gram Barley  
 $6.45 \text{ gm.} / 60.0 \text{ gm.} = \boxed{0.108 \text{ gm./gm.}}$

H) Cumulative sample 1-14 had a concentration of 99.3 wt. % ethanol and 1-15 had a concentration of 98.9 wt. % ethanol. It was necessary to calculate how much of sample 15 would need to be added to samples 1-14 in order to have a cumulative sample of 99 wt. %

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ethanol. It was assumed that the change in ethanol concentration from sample 14 to sample 15 was linear and, therefore, interpolation can be used.

$$\begin{aligned} & (14.583 \text{ gm.} + (15.706 \text{ gm.} - 14.583 \text{ gm.}) \cdot Z) / \\ & (14.583 \text{ gm.} + (15.706 \text{ gm.} - 14.583 \text{ gm.}) \cdot Z) \\ & + 0.097 \text{ gm.} + (0.174 \text{ gm.} - 0.097 \text{ gm.})Z = 0.99 \end{aligned}$$

$$Z = \boxed{0.766}$$

14.583 gm. = total ethanol in cumulative sample 1-14  
15.706 gm. = total ethanol in cumulative sample 1-15  
0.097 gm. = total water in cumulative sample 1-14  
0.174 gm. = total water in cumulative sample 1-15  
0.99 = mass fraction of ethanol in desired cumulative sample  
Z = fraction of sample 15 that must be added to cumulative sample 1-14 to obtain a solution containing 99 wt. % ethanol

I) Amount of Water in Cumulative Sample of 99 wt. % Ethanol

$$\begin{aligned} & 0.097 \text{ gm.} + (0.174 \text{ gm.} - 0.097 \text{ gm.}) \\ & \cdot (0.766) = \boxed{0.156 \text{ gm.}} \end{aligned}$$

J) Average Flow Rate of Feed for Samples 1 to 15

$$(30.2 \text{ ml.}/300 \text{ min.}) \cdot (0.909) = \boxed{0.0915 \text{ ml.}/\text{min.}}$$

K) Liquid That was not in Sample 1 Due to Lag Time

$$\begin{aligned} & ((10.6 \text{ gm.}) \cdot (21 \text{ min.})/200 \text{ min.}) \\ & - 0.45 \text{ gm.} = \boxed{0.663 \text{ gm.}} \end{aligned}$$

10.6 gm. = total weight of samples 2 to 11  
21 min. = time during which sample 1 was collected  
200 min. = time during which samples 2 to 11 were collected

0.45 gm. = weight of sample 1

L) Lag Time

$$\begin{aligned} & ((0.663 \text{ gm.})/(0.663 \text{ gm.} + 0.45 \text{ gm.})) \\ & \cdot (21 \text{ min.}) = \boxed{12.51 \text{ min.}} \end{aligned}$$

M) Amount of Feed Entering Adsorber for Samples 1 to 14  

$$\frac{(0.0915 \text{ ml./min.}) \cdot (281 \text{ min.} - 12.51 \text{ min.})}{= 24.57 \text{ ml.}}$$

281 min. = period of time that samples 1 to 14 were collected for

N) Amount of Feed Entering Adsorber for Cumulative Sample Containing 99 wt. % Ethanol  

$$\frac{24.57 \text{ ml.} + (0.0915 \text{ ml./min.}) \cdot (0.766) \cdot (0.85 \text{ gm./ml.})}{= 22.07 \text{ gm.}}$$

O) Weight of Water in this Amount of Feed  

$$\frac{(22.07 \text{ gm.}) \cdot (0.235)}{= 5.19 \text{ gm.}}$$

P) Weight of Ethanol in this Amount of Feed  

$$\frac{(22.07 \text{ gm.}) \cdot (0.765)}{= 16.89 \text{ gm.}}$$

Q) Weight of Water Sorbed  

$$5.19 \text{ gm.} - 0.156 \text{ gm.} = 5.03 \text{ gm.}$$

R) Amount of Ethanol in Cumulative Sample of 99 wt. % Ethanol  

$$14.583 \text{ gm.} + (15.706 \text{ gm.} - 14.583 \text{ gm.}) \cdot (0.766) = 15.44 \text{ gm.}$$

S) Weight of Ethanol Sorbed  

$$16.89 \text{ gm.} - 15.44 \text{ gm.} = 1.45 \text{ gm.}$$

5) Grams of Water Sorbed per Gram of Grain with a Cumulative Sample of 99 wt. % Ethanol  

$$5.03 \text{ gm.}/60.0 \text{ gm.} = 0.0838 \text{ gm./gm.}$$

6) Milliliters of Pure Ethanol Concentrated Per Gram of Grain with a Cumulative Sample of 99 wt. % Ethanol  

$$15.44 \text{ gm.}/((0.79 \text{ gm./ml.}) \cdot (60.0 \text{ gm.})) = 0.326 \text{ ml./gm.}$$

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0.79 gm./ml. = specific gravity of pure ethanol

The amount of ethanol discussed here was the amount leaving the adsorber as samples and not the amount entering as feed.

- 7)  $\frac{\text{Percent of Feed Ethanol Sorbed}}{(1.45 \text{ gm.}) \cdot (100)/(16.89 \text{ gm.})} = \boxed{8.58 \text{ wt. \%}}$
- 8)  $\frac{\text{Percent of Ethanol in Sorbed Liquid}}{(1.45 \text{ gm.}) \cdot (100)/(1.45 \text{ gm.} + 5.03 \text{ gm.})} = \boxed{22.4 \text{ wt. \%}}$
- 9)  $\frac{\text{Time Required to Accomplish this}}{281 \text{ min.} + (20 \text{ min.}) \cdot (0.766)} = \boxed{296.3 \text{ min.}}$

Steps H-9 should be repeated for each cumulative sample concentration that is necessary.

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APPENDIX F

STATISTICAL ANALYSIS OF DATA

The purpose of doing the statistical analysis was:  
 1) to determine which independent variables had a significant effect, and 2) to develop an equation that accurately describes the data, but is relatively simple.

The central composite design shown in Figure 4 and Table 1 was used because 1) it allows the effects of each variable to be completely independent of one another, and 2) it allows for the development of a fully expandable quadratic equation. It can be seen that the design was not exactly a central composite because it had two face points. An analysis of the run conditions for the three independent variables (initial grain temperature, feed composition, and feed mass flux) was done using the following equation:

$$r = \frac{(x - \bar{x}) \cdot (y - \bar{y})}{(N-1) \cdot S_x \cdot S_y} \quad (\text{Eqn. 7})$$

$r$  = the correlation coefficient

$x$  = one of the data points for one of the independent variables

$\bar{x}$  = the average value for all of the  $x$ 's

$y$  = one of the data points for an independent variable different from  $x$

$\bar{y}$  = the average value of all of the  $y$ 's

$S_x$  = sample standard deviation for  $x$

$S_y$  = sample standard deviation for  $y$

$N$  = number of data points

A value of  $r = 0$  indicates that there is no correlation between the two independent variables. A value of  $r = 1$  indicates that the two variables are directly related. This equation was used to determine the value of  $r$  for all three possible binary combinations of the independent variables. If a true central composite design had been used,  $r$  would have been equal to 0 for all cases. Since two of the points were face points,  $r$  was slightly higher than 0. The highest value of  $r$  was  $r = 0.154$  for the combination of initial grain temperature and feed mass flux. For the number of points used (19 points) and a 90% confidence level,  $r$  would have to be 0.370 before there would be any noticeable effect. Therefore, I assumed it was a true central composite design for purposes of analysis.

The following is a brief description of how the first correlation was derived that is shown on page 68. A least squares technique is used as shown in the following equation:

$$S = \sum_{i=1}^N (C - \hat{C})^2 \quad (\text{Eqn. 8})$$

S = sum of least squares

C = the experimental value of C for point  
(C was a function of the independent  
variables initial temperature, feed  
mass flux, and/or feed concentration.)

$\hat{C}$  = the calculated value of C for point using  
the correlation being tested

Initially, each independent variable was tested individually while holding the other independent variables constant. An equation such as the following was then used:

$$C = a + bx \quad (\text{Eqn. 9})$$

a, b = constants

x = the value of one of the independent  
variables at a given data point

Constant b was the important constant with regard to getting a good fit of the equation to the data. The first derivative of S with respect to b was taken and set equal to zero. The resulting equation was then solved for b. This was then the best estimate of b for this correlation.

Two of the main tests considered then were: 1) the F test, and 2) the residuals. The F test was done as follows. The Var(b) (variance of b) was calculated. The following equation was then used:

$$F = \frac{(b)^2}{\text{Var}(b)} \quad (\text{Eqn. 10})$$

F would then be large if b was large with respect to  $\text{Var}(b)$  meaning that the correlation was good. For a 90% confidence interval, F would have been 2.99 or greater for 19 points or the correlation would not have been very good.

The residuals were the differences between the calculated and experimental values of C for each point. These are displayed on Figures 18-22 for the statistical equation developed in this thesis. The data should have been scattered evenly on each side of the line representing the correlation being tested for it to have been a good fit. If all the data were scattered on one side of the equation, then the equation was not very descriptive of the data.

Different equations were tried for each of the independent variables until the above tests gave satisfactory results. The equations developed for each of the independent variables being considered were then combined together and tests were done on the combined equation as on the individual equations. One additional test was R-squared. This represented what fraction of variability of the data was explained by the correlation.

The two following relationships were tried for the effect of the feed concentration of the amount of ethanol concentrated:

$$\ln C = f(\ln(Fe)) \quad (\text{see Table 66; Figure 18})$$

(Eqn. 11)

$$C = f(\ln(Fe)) \quad (\text{see Table 67; Figure 19})$$

(Eqn. 12)

C = amount of pure ethanol concentrated per gm. of barley for a combined sample of 99 wt. % ethanol

Fe = feed ethanol concentration

The highest F-value obtained was 0.025, which is well below the 2.99 necessary for significance at the 90% confidence interval. It was then determined that feed concentration had no noticeable effect on the amount of ethanol concentrated in the range studied.

Tests done for initial temperature and feed mass flux showed that they were related to the amount of ethanol concentrated (see Tables 68 and 69; Figures 20 and 21). The F-value obtained for initial grain temperature was 2.122. Although this is less than 2.99, a decision was made to include the initial temperature in the correlation. The feed mass flux was shown to have a very direct affect on the amount of ethanol concentrated. The F-value obtained was 92.77.

Table 70 and Figure 22 show the combined-equation analysis. R-squared is 0.8793, which indicates a very strong correlation.

#### Complex Method of Box

The correlation developed using this method is essentially the same as the statistical correlation, but with different constants. The same form of equation was used, but the Complex Method was used to find the constants.

The Complex Method of Box is essentially an improved version of the Simplex Method. In the Simplex Method, vertices of a regular geometric figure (such as a triangle in two dimensions) are determined which represent the dependent variable for a specific set of independent variables. The vertex with the worst value will be rejected and a new vertex determined on the other side of the centroid of the remaining points. This process of discarding vertices and finding new ones continues until the optimum set of independent variables is determined. During this entire process, a regular-shaped geometric figure of a constant size is maintained. The Complex Method of Box is very similar to the Simplex Method, except for the following main points:

- 1) the shape of the geometric figure does not remain fixed,

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- 2) the size of the figure can enlarge or contract,
- 3) the number of vertices may be different.

Table 66. Statistical Analysis of Data for Natural Logarithm of C vs. Natural Logarithm of Feed Ethanol

Dependent Variable = natural logarithm of C(ml. of pure ethanol concentrated per gm. of barley for a combined sample of 99 wt. % ethanol)

Independent Variable (7) = natural logarithm of feed ethanol concentration

|      |     |            |         |       |
|------|-----|------------|---------|-------|
| Fit: | VAR | R-PART     | B       | E(B)  |
|      | (7) | 0.3851D-01 | 0.5317  | 3.346 |
|      | VAR | T          | P-VALUE |       |
|      | (7) | 0.1589     | 0.8756  |       |

Intercept = -4.712

R-squared = 0.1483E-02

#### ANALYSIS OF VARIANCE:

| SOURCE   | DF | SS         | M.S.       | F-VALUE    |
|----------|----|------------|------------|------------|
| REGRESS  | 1  | 0.132aE-01 | 0.1321E-01 | 0.2524E-01 |
| RESIDUAL | 17 | 8.895      | 0.5232     |            |
| TOTAL    | 18 | 8.908      |            |            |

|         |         |
|---------|---------|
|         | P-VALUE |
| REGRESS | 0.8756  |

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Table 67. Statistical Analysis of Data for C vs.  
Natural Logarithm of Feed Ethanol  
Concentration

Dependent Variable = C(ml. of pure ethanol concentrated  
per gm. of barley for a combined  
sample of 99 wt. % ethanol)

Independent Variable (7) = natural logarithm of feed  
ethanol concentration

|      |     |             |             |        |
|------|-----|-------------|-------------|--------|
| Fit: | VAR | R-PART      | B           | SE(B)  |
|      | (7) | -0.2488E-01 | -0.4727E-01 | 0.4607 |

|  |     |         |
|--|-----|---------|
|  | VAR | T       |
|  | (7) | -0.1026 |

Intercept = 0.3275

R-squared = 0.6189E-03

ANALYSIS OF VARIANCE:

| SOURCE   | DF | S.S.       | M.S.       | F-VALUE    |
|----------|----|------------|------------|------------|
| REGRESS  | 1  | 0.1044E-03 | 0.1044E-03 | 0.1053E-01 |
| RESIDUAL | 17 | 0.1686     | 0.9918E-02 |            |
| TOTAL    | 18 | 0.1687     |            |            |

|         |         |
|---------|---------|
|         | P-VALUE |
| REGRESS | 0.9195  |

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Table 68. Statistical Analysis of Data for Natural  
Logarithm of C vs. Natural Logarithm of  
Initial Temperature

Dependent Variable = natural logarithm of C(ml. of pure  
ethanol concentrated per gm. of  
barley for a combined sample of  
99 wt. % ethanol)

Independent Variable (6) = natural logarithm of initial  
temperature

|      |     |         |        |       |
|------|-----|---------|--------|-------|
| Fit: | VAR | R-PART  | B      | SE(B) |
|      | (6) | -0.3331 | -3.666 | 2.516 |
|      | VAR | T       |        |       |
|      | (6) | -1.457  |        |       |

Intercept = 14.24  
R-squared = 0.1110

ANALYSIS OF VARIANCE:

| SOURCE   | DF | S.S.   | M.S.   | F-VALUE |
|----------|----|--------|--------|---------|
| REGRESS  | 1  | 0.9885 | 0.9885 | 2.122   |
| RESIDUAL | 17 | 7.919  | 0.4658 |         |
| TOTAL    | 18 | 8.908  |        |         |

|         |         |
|---------|---------|
|         | P-VALUE |
| REGRESS | 0.1634  |

Table 69. Statistical Analysis of Data for Natural  
Logarithm of C vs. Natural Logarithm of  
Feed Mass Flux

Dependent Variable = natural logarithm of C (ml. of pure  
ethanol concentrated per gm. of  
barley for a combined sample of  
99 wt. % ethanol)

Independent Variable (8) = natural logarithm of feed  
mass flux

|      |     |         |         |         |
|------|-----|---------|---------|---------|
| Fit: | VAR | R-PART  | B       | SE(B)   |
|      | (8) | -0.9193 | -0.6531 | 0.06781 |
|      | VAR | T       |         |         |
|      | (8) | -0.9632 |         |         |

Intercept = -3.891

R-squared = 0.8451

#### ANALYSIS OF VARIANCE:

| SOURCE   | DF | S.S.  | M.S.    | F-VALUE |
|----------|----|-------|---------|---------|
| REGRESS  | 1  | 7.528 | 7.528   | 92.77   |
| RESIDUAL | 17 | 1.380 | 0.08115 |         |
| TOTAL    | 18 | 8.908 |         |         |

|         |         |
|---------|---------|
|         | P-VALUE |
| REGRESS | 0       |

Table 70. Statistical Analysis of Data for Natural Logarithm of C vs. Natural Logarithm of Initial Temperature and Natural Logarithm of Feed Mass Flux

Dependent Variable = natural logarithm of C (ml. of pure ethanol concentrated per gm. of barley for a combined sample of 99 wt. % ethanol)

Independent Variables = natural logarithm of initial temperature (6), natural logarithm of feed mass flux (8)

|      |     |         |         |            |
|------|-----|---------|---------|------------|
| Fit: | VAR | R-PART  | B       | SE(B)      |
|      | (6) | -0.4694 | -0.2603 | 0.1224     |
|      | (8) | -0.9285 | -0.6282 | 0.6281E-01 |

|  |     |            |        |
|--|-----|------------|--------|
|  | VAR | P-VALUE    | T      |
|  | (6) | 0.4939E-01 | -2.126 |
|  | (8) | 0.000      | -10.00 |

Intercept = -3.192  
R-squared = 0.8793

#### ANALYSIS OF VARIANCE:

| SOURCE   | DF | S.S.  | M.S.       | F-VALUE | P-VALUE |
|----------|----|-------|------------|---------|---------|
| REGRESS  | 2  | 7.832 | 3.916      | 58.26   | 0.000   |
| RESIDUAL | 16 | 1.075 | 0.6722E-01 |         |         |
| TOTAL    | 18 | 8.908 |            |         |         |

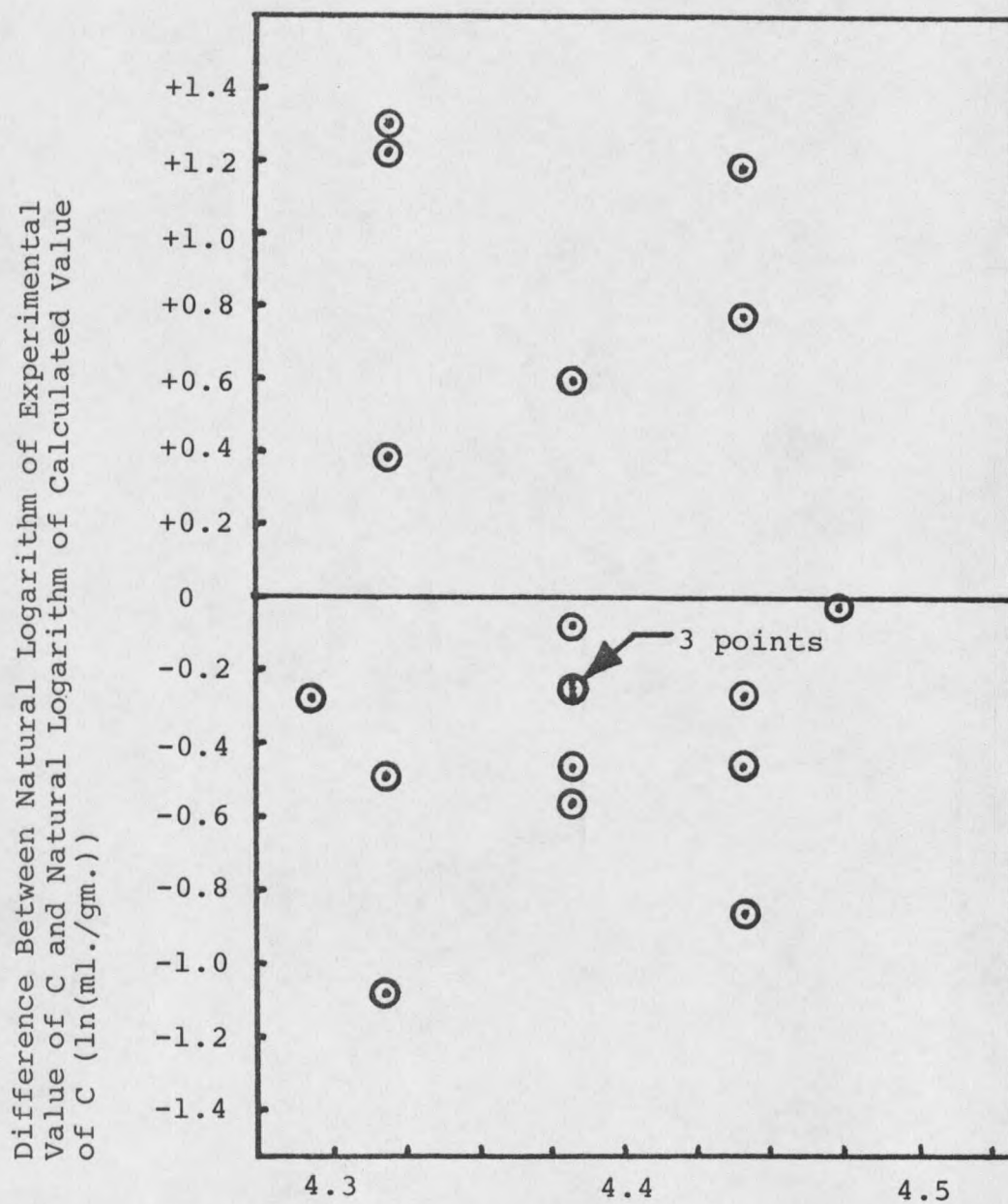


Figure 18. Residuals for ln C vs. ln Feed Concentration



Figure 19. Residuals for C vs. ln Feed Concentration

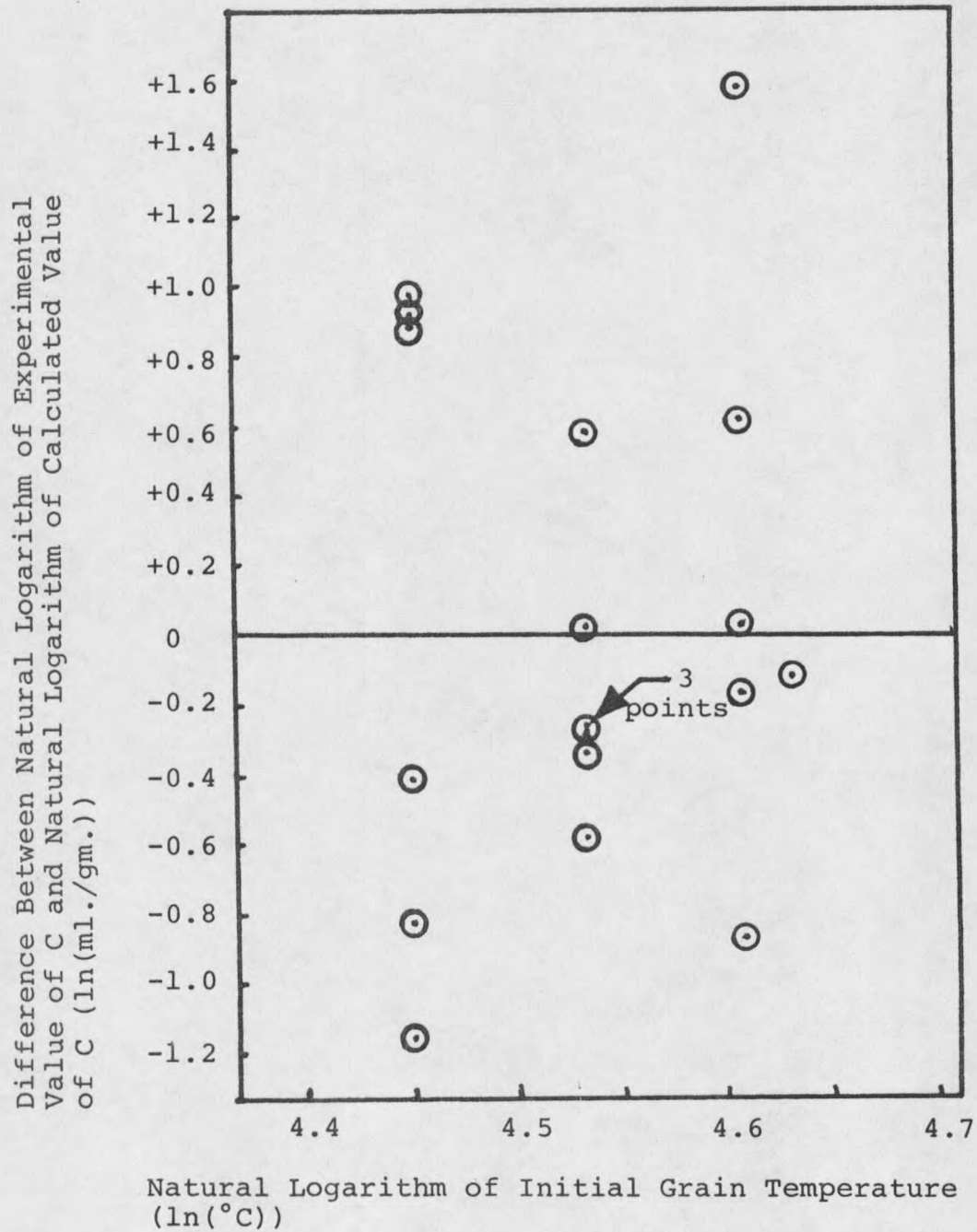


Figure 20. Residuals for ln C vs. ln Initial Grain Temperature

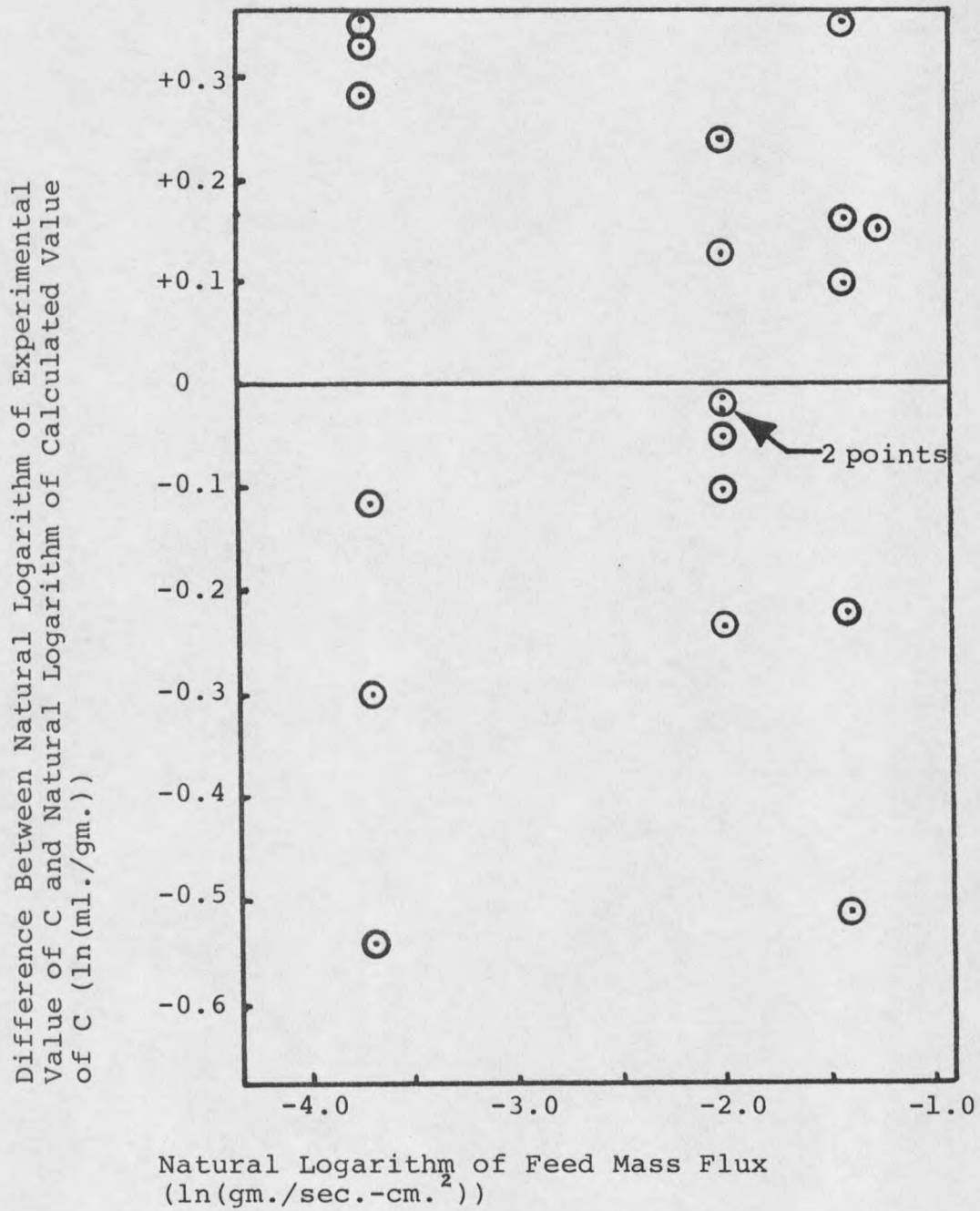
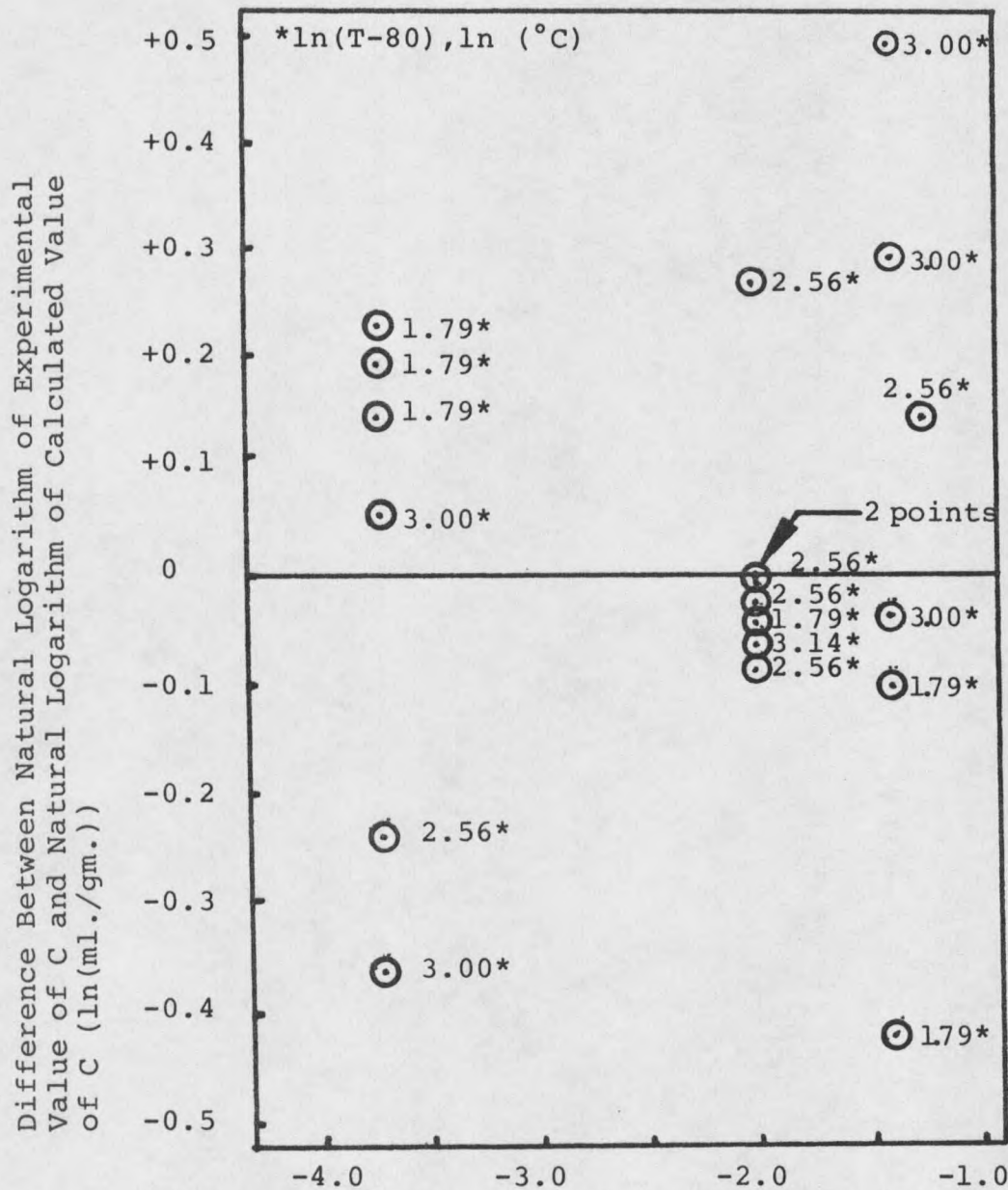


Figure 21. Residuals for  $\ln C$  vs.  $\ln$  Feed Mass Flux



Natural Logarithm Feed Mass Flux (ln(gm./sec.-cm.<sup>2</sup>))

Figure 22. Residuals for ln C with Respect to ln Feed Mass Flux and ln Initial Grain Temperature

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APPENDIX G

ETHANOL-WATER MIXTURE DATA

Table 71. Ethanol-Water Mixture Data

| <u>Vol. %</u> | <u>Wt. %</u> | <u>Mole %</u> |
|---------------|--------------|---------------|
| 5             | 4.0          | 1.6           |
| 10            | 8.1          | 3.3           |
| 15            | 12.2         | 5.1           |
| 20            | 16.5         | 7.2           |
| 25            | 20.8         | 9.3           |
| 30            | 25.3         | 11.7          |
| 35            | 29.8         | 14.2          |
| 40            | 34.5         | 17.1          |
| 45            | 39.2         | 20.1          |
| 50            | 44.1         | 23.5          |
| 55            | 49.1         | 27.4          |
| 60            | 54.2         | 31.6          |
| 65            | 59.4         | 36.4          |
| 70            | 64.8         | 41.8          |
| 75            | 70.3         | 48.0          |
| 80            | 75.9         | 55.2          |
| 85            | 81.7         | 63.5          |
| 90            | 87.7         | 73.6          |
| 95            | 93.7         | 85.3          |

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