AN EXPERIMENTAL STUDY OF DISTILLATION COLUMN DYNAMICS

By

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AN EXPERIMENTAL STUDY OF DISTILLATION COLUMN DYNAMICS

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PREFACE

A series of thirteen experimental runs has been made. Five of these runs were made to test the applicability of a first order lumped parameter model for predicting the dynamic behavior of distillation columns. The remaining eight runs were made to test the applicability of the model for feed forward control. These runs were conducted using binary and ternary systems. The data obtained show that the first order model accurately described the behavior of the column products. It can also be used for feed forward control to maintain a constant bottoms product composition during the transient period following a change in feed composition.

I wish to express my sincere thanks to Dr. R. N. Maddox for the guidance and advice that he gave me while serving as my research adviser. I would also like to thank Dr. J. H. Erbar, Dr. J. B. West, and Dr. J. R. Norton for the advice that they gave as my Doctoral Advisory Committee. My thanks go to my research associate, M. D. Burns, for helping me build the equipment that was used in this study and for helping me make my experimental runs.

I am indebted to the Oklahoma State University Computing Center for the use of its computing facilities. I wish to express my sincere thanks to the Dow Chemical Company for their fellowship grant which made this work possible.

Finally, I want to especially thank my wife, Ruth, who has given me inspiration during my graduate study.

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CHAPTER I

INTRODUCTION

Members of the chemical engineering profession are becoming more and more interested in the automatic control of industrial processes. The control of distillation columns has received a large amount of this interest because distilling operations are found in almost every phase of the chemical industry. Also, distillation columns are frequently used in the final step of purification of a product; thus, accurate control of product compositions and/or column performance may be very important.

Stemming from this increasing interest in distillation column control is feed forward control. The use of effective and economical feed forward control of distillation columns can result in increased profits over the conventional feed back schemes because the distillation column can be operated with a constant product composition.

There were three major objectives of this study. The first of these objectives was to obtain experimental data to test the applicability of a first order lumped parameter model for predicting the dynamic performance of a distillation column. The second objective was to determine if this model can be used for feed forward control of a distillation column. The third objective was to determine if the separation parameters in this first order model can be predicted from tray-by-tray data.

The equipment used in this study was a ten-tray twelve-inch diameter distillation column which was operated as a non-refluxed stripper. Experimental data were obtained for both a binary system of benzene and toluene and a ternary system of benzene, toluene, and para-xylene. The column pressure was maintained at ten psig for the binary runs and five psig for the ternary runs.

CHAPTER II

REVIEW OF LITERATURE

Marshall and Pigford (13) proposed the first mathematical model that could be used to describe the dynamic performance of a distillation column in 1947. Their model was based on the equilibrium stage concept where the vapor and liquid leaving a given tray are in equilibrium. According to the equilibrium tray concept, each tray is considered individually, and in order to predict the behavior of a column a separate equation must be written for each component on each tray. The equation for the i^{th} component in the n^{th} tray is

$$\frac{d\delta_n^L x_{n,i}}{dt} + \frac{d\delta_n^V y_{n,i}}{dt} = (L_{n-1}\, x_{n-1,i} + V_{n+1}\, y_{n+1,i}) - (L_n\, x_{n,i} + V_n\, y_{n,i}) \qquad (1)$$

where

$\frac{d\delta_n^L x_{n,i}}{dt}$ = change in liquid holdup of component i on the n^{th} tray with respect to time.

$\frac{d\delta_n^V y_{n,i}}{dt}$ = change in vapor holdup of component i above the n^{th} tray with respect to time.

$L_{n-1}\, x_{n-1,i} + V_{n+1}\, y_{n+1,i}$ = rate at which component i flows to the n^{th} tray.

$L_n\, x_{n,i} + V_n\, y_{n,i}$ = rate at which component i flows from the n^{th} tray.

Although the Marshall-Pigford model is sound, it has several drawbacks. The most significant of these drawbacks is the vast number of simultaneous equations which must be solved. For example, if a column has n trays and i components then there are n times i simultaneous equations plus external heat and material balance equations which must be solved. Another drawback of this model is that trays are not ideal. Thus, some method of estimating the efficiency of each tray is required. High speed computers were not in existence at the time that equation (1) was first presented. Therefore, in order to make their model more useful, Marshall and Pigford made the following assumptions:

(1) constant molal overflow;

(2) negligible vapor holdup above a tray;

(3) approach to equilibrium between the liquid on a tray and the vapor above the respective tray could be represented by a straight pseudo-equilibrium line.

These assumptions enabled Marshall and Pigford to obtain a solution to the differential equations; however, the accuracy of the model was reduced. The assumption of negligible vapor holdup is usually a good assumption, but the assumption of constant molal overflow requires that the molar heats of vaporization of the components be equal. The assumption of a straight pseudo-equilibrium line requires that the concentration of the component be small.

Rose, et al. (20, 21, 22, 23) applied equation (1) as the basic equation to a batch distillation column. They avoided use of the limiting assumptions used by Marshall and Pigford by programming the differential equations on a digital computer. They found that because of the large number of simultaneous equations to be solved, the solution of problems required an excessive amount of computer time.

Robinson and Gilliland (19) developed an approximate graphical method for predicting the approach to steady state of a distillation column. This method is restricted to columns experiencing an upset caused by a change in feed composition. The assumption of ideal trays was also employed.

Voetter (26) was one of the first workers with transient distillation to combine experimental data with a theoretical analysis. He compared experimental data obtained with a single-section sixty-tray Oldershaw column with the Marshall-Pigford equations. He found that the experimental and calculated values were close during the early portion of the transient period; however, they differed considerably as the new steady state was approached.

Wilkinson and Armstrong (27, 28) presented experimental data obtained on a five-tray, four-inch diameter column. This work was performed on a complete column. A few years later, Armstrong and Wood (1) published experimental and calculated results for a 21-tray distillation column. The upset was caused by a change in reflux rate. Their data showed good agreement between experimental and calculated results at the top of the column. However, there was poor agreement at the bottom of the column.

Baber (2, 3, 4) in 1961 presented one of the most extensive experimental and calculated studies of transient distillation column performance. He used an analog computer to program a series of differential equations that were developed by Lamb and Pigford (9), which were based on the Marshall-Pigford equations. Baber compared the results obtained with the computer with his experimental data. The experimental equipment used was a five-tray, single-section distillation column. The column was allowed to reach steady state at total reflux.

One of the operating variables such as reflux rate, reflux composition, or vapor rate was then changed. The flow rates and compositions of the various streams were determined before the step change was made, and the compositions were measured at intervals throughout the transient period. When the column reached steady state, the flow rates and compositions were again measured. Baber obtained good agreement between experimental and computer results for a few of his runs. However, for the majority of his runs he was unable to obtain good agreement.

Marr (12) presented a new concept for predicting the transient behavior of a distillation column in 1962. He proposed that in order to get away from the conventional and complicated tray-by-tray model, some parameter could be used to describe the degree of separation within the column. However, Marr considered all aspects of the mechanics of construction of the column, thus complicating his model. Because of these additions, Marr's model was almost as complex as the tray-by-tray concept.

Reynolds (18) presented a method whereby the degree of separation could be calculated easily thus shortening Marr's concept. Reynolds envisioned a distillation column as being composed of several sections in which there could be any number of trays. According to the section concept, as shown in Figure 1, a section of a distillation column is that part of the column which lies between the points at which either feed streams enter or product streams leave the column. According to Reynolds, the rate at which mass is transferred from the vapor phase to the liquid phase can be expressed by the equation

$$N^V_{n,i} = - J_{n,i} (y^* - y)_{n,i} \tag{2}$$

where $J_{n,i}$ is the parameter which describes the degree of separation

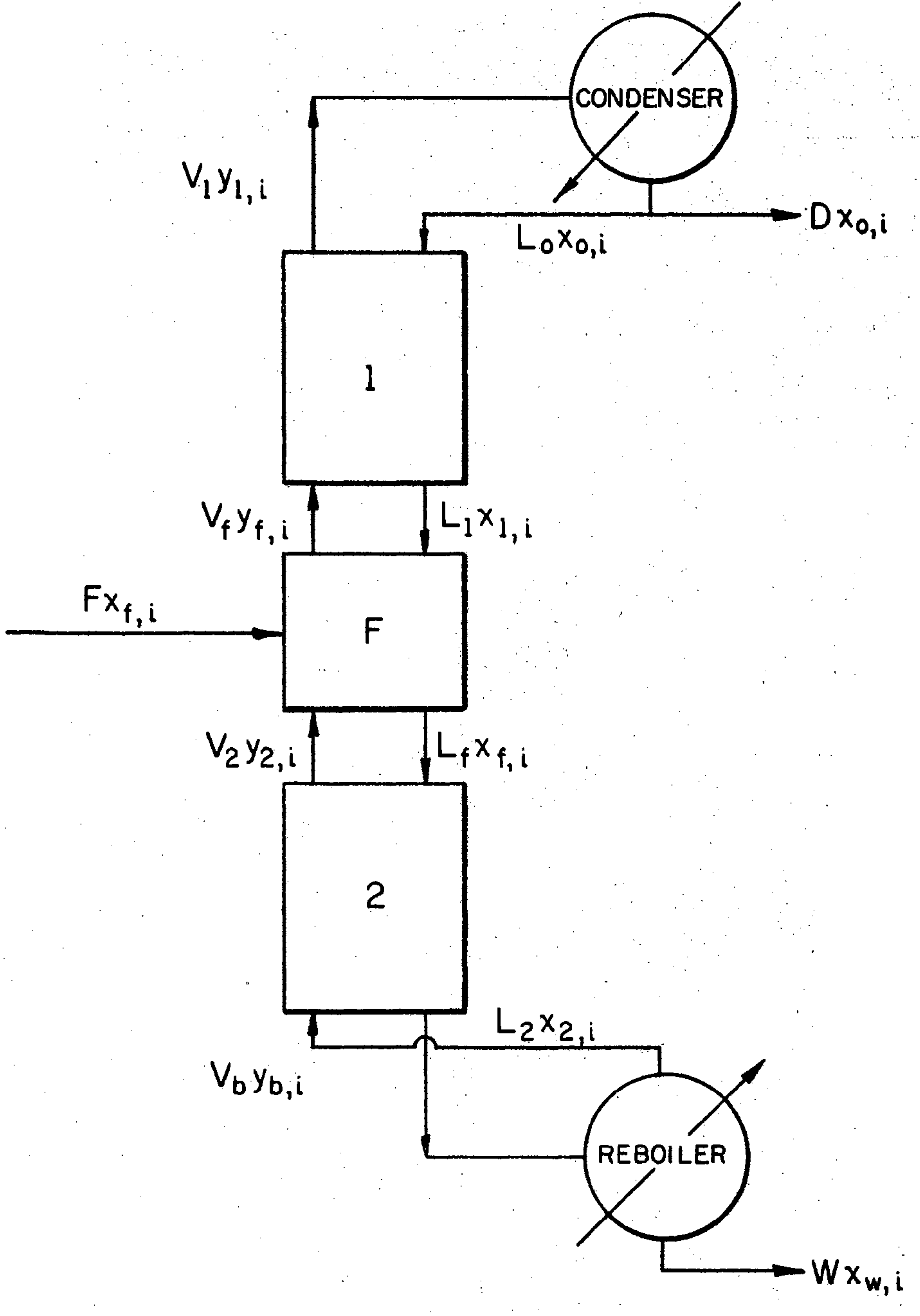

Figure 1. A Distillation Column According to the Section Concept.

occurring in a section. The term $(y^* - y)_{n,i}$ is the driving force for mass transfer in the section.

Although equation (2) appears to be identical in form to the mass transfer equation

$$N = K(y^* - y),$$

there are fundamental differences between the two equations. K is related to the diffusivity in the conventional mass transfer equation. The coefficient in equation (2), however, is not related directly to the physical properties of the component being transferred. The coefficient, $J_{n,i}$, is a parameter which describes the degree of separation occurring in a section and is an empirically determined factor.

Reynolds developed a set of differential equations for the transient behavior of the liquid and vapor streams leaving the section based on the idea of mass transfer in the section. Reynolds made two major assumptions in developing the equations. The first of these assumptions was that $J_{n,i}$ remains constant for small changes in column conditions, and the second was constant molal overflow throughout the section. Reynolds attempted to prove his model by comparing values predicted with the model with experimental data, but he was unable to obtain good agreement.

Osborne (14, 15) and Osborne, et al. (16, 17) felt that Reynolds' concept was basically sound. They extended Reynolds' model to relate the net rate at which mass is transferred from the liquid phase with the net rate at which mass is transferred from the vapor phase. This relationship was found to be

$$N^L_{n,i} = J_{n,i} (y^* - y)_{n,i}. \qquad (3)$$

Osborne also presented a method for evaluating the separation parameter

$J_{n,i}$. The equations presented by Osborne based on the vapor and liquid phase respectively are

$$J_{n,i} = \frac{(V_n y_{n,i} - V_{n+1} y_{n+1,i})}{(Kx)_{n-1,i} - y_{n+1,i}} \tag{4}$$

$$J_{n,i} = - \frac{(L_n x_{n,i} - L_{n-1} x_{n-1,i})}{(Kx)_{n-1,i} - y_{n+1,i}} . \tag{5}$$

Osborne tested his model with data presented by Baber (2) and with data that he obtained experimentally. Osborne reported good agreement between experimental and calculated results for both sets of data. He was also the first person to present experimental ternary data for transient distillation.

There have been only a small number of papers published on feed forward control of distillation columns. The most extensive work in this field of distillation has been presented by Luyben (10, 11). He presented results of an analog simulation of a ten-tray and a forty-tray distillation column. The experimental work was conducted on the ten-tray, two-foot diameter column using the acetone-benzene system. The model used by Luyben was based on an external material balance scheme. The deviation in product compositions was found to be very small during the transient period and was eventually reduced to zero.

Shinskey (25) used a modification of the Fenske equation to develop a feed forward control model. Like Luyben's model, Shinskey's model was based on an over-all material balance control scheme.

Cadman, et al. (7) presented several methods to facilitate the design of feed forward controllers using linear calculational techniques. A steady-state Taylor series expansion of a nonlinear steady state model was used to estimate nonlinearities in the dynamic column performance.

The authors report that this approach is limited to the description of column dynamics for disturbances about the points of linearization.

Distefano, et al. (8) verified a mathematical feed forward control model experimentally using a twelve-tray, ten-inch diameter column with a methanol-tertiary butyl alcohol system. The model that was tested was based on material and energy balances around various sections of the column. The authors report that overhead purity was maintained within 0.5 per cent of the initial steady state value. The model tested was virtually limited to simple binary systems because of the large amount of computer time required to obtain a solution.

Osborne (15, 16) presented a feed forward control model using the first order lumped parameter model described earlier in this chapter. He presented a mathematical solution to a hypothetical example. However, no experimental verification of the model was presented.

An important part of the work presented in this paper was the verification of the feed forward control scheme presented by Osborne. This model lumps column parameters such as the number of trays, tray efficiencies, and holdup into one parameter. Thus, it is one of the least complex of all of the transient distillation models presented. This simplicity makes the model more economical to use than more complex models because less computer time and smaller computer size is required for a program of the model. Data are presented for both binary and ternary systems.

CHAPTER III

EXPERIMENTAL APPARATUS

The experimental apparatus consists primarily of a distillation column, condenser, reboiler, and six tanks for product collection and feed purposes. The distillation column was operated as a non-refluxed stripper. It is twelve inches in diameter and contains ten Nutter float valve trays with twelve-inch spacing. The trays have a two-inch weir height, and a one and one-half-inch downcomer escape height. The downcomer area is 0.0702 square feet. The top tray is equipped with an entrance baffle for the feed stream. A detailed diagram of the column is shown in Figure 2.

The reboiler is a U-tube kettle reboiler manufactured by the Western Supply Company. The tube bundle consists of twelve three-quarter-inch steel tubes with a total surface of twenty-three square feet. The kettle is twenty inches in diameter and six feet in length. Steam at fifty psig was used.

The condenser is a Ross BCF 603 copper and brass heat exchanger. It is mounted vertically with condensation on the shell side. The condenser contains 116-0.625-inch tubes which are 31.5 inches long.

Pumping was provided by two Eastern pumps. A two-stage model 2J-34D cast iron pump was used for a feed pump, and the bottoms product pump was a single-stage model F-34B cast iron pump. The distillate product flowed by gravity, and no pump was needed.

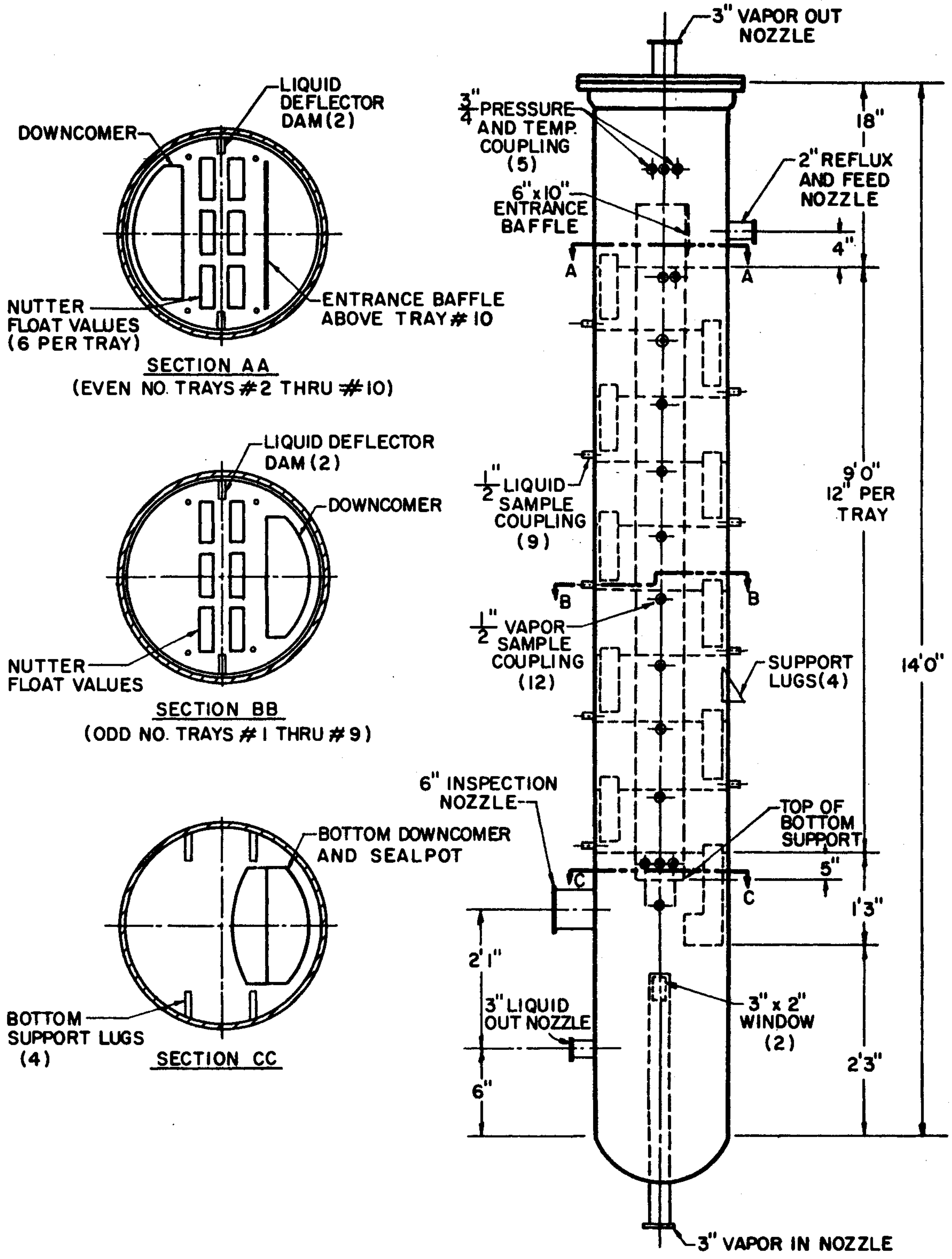

Figure 2. Detailed Diagram of Column.

Tankage was provided by six horizontal cylindrical aluminum tanks with a capacity of 250 gallons each. The tanks were mounted in three vertical rows of two tanks each. The piping to these tanks was arranged so that the distillate and bottoms products could be put into any of the six tanks. Feed could be withdrawn from the three bottoms tanks. In order to obtain a feed capacity greater than 250 gallons, two tanks could be combined to serve as one 500 gallon feed tank.

The flow rates of the liquid stream to the reboiler and the vapor stream from the reboiler were determined by measuring the pressure drop across an orifice. The flow rates of the feed and two products were measured with rotameters.

The temperatures of the feed, products, and trays were measured with copper-constantan thermocouples. These temperatures were recorded with a Honeywell 24-point temperature recorder.

The column pressure was controlled using a Honeywell pressure recorder-controller and an air driven diaphragm control valve. The flow rates of the feed, bottoms product, and steam to the reboiler were controlled manually. The flow rate of the distillate was controlled by the amount of steam to the reboiler.

Samples were taken using Asco Number 8314A-75 Solenoid valves and evacuated sample bombs. The sample bombs have a capacity of 380 cubic centimeters and over-all dimensions are twenty-seven centimeters long with a diameter of five centimeters. These samples were analyzed on an F & M Model 500 Programmed High-Temperature Gas Chromatograph with a Perkin-Elmer D2 Electronic Integrator.

A schematic diagram of the distillation column and auxiliary equipment is shown in Figure 3. Figures 4 and 5 are photographs of the column and equipment respectively. Figure 6 is a schematic diagram, and

Figure 7 is a photograph of the sampling system.

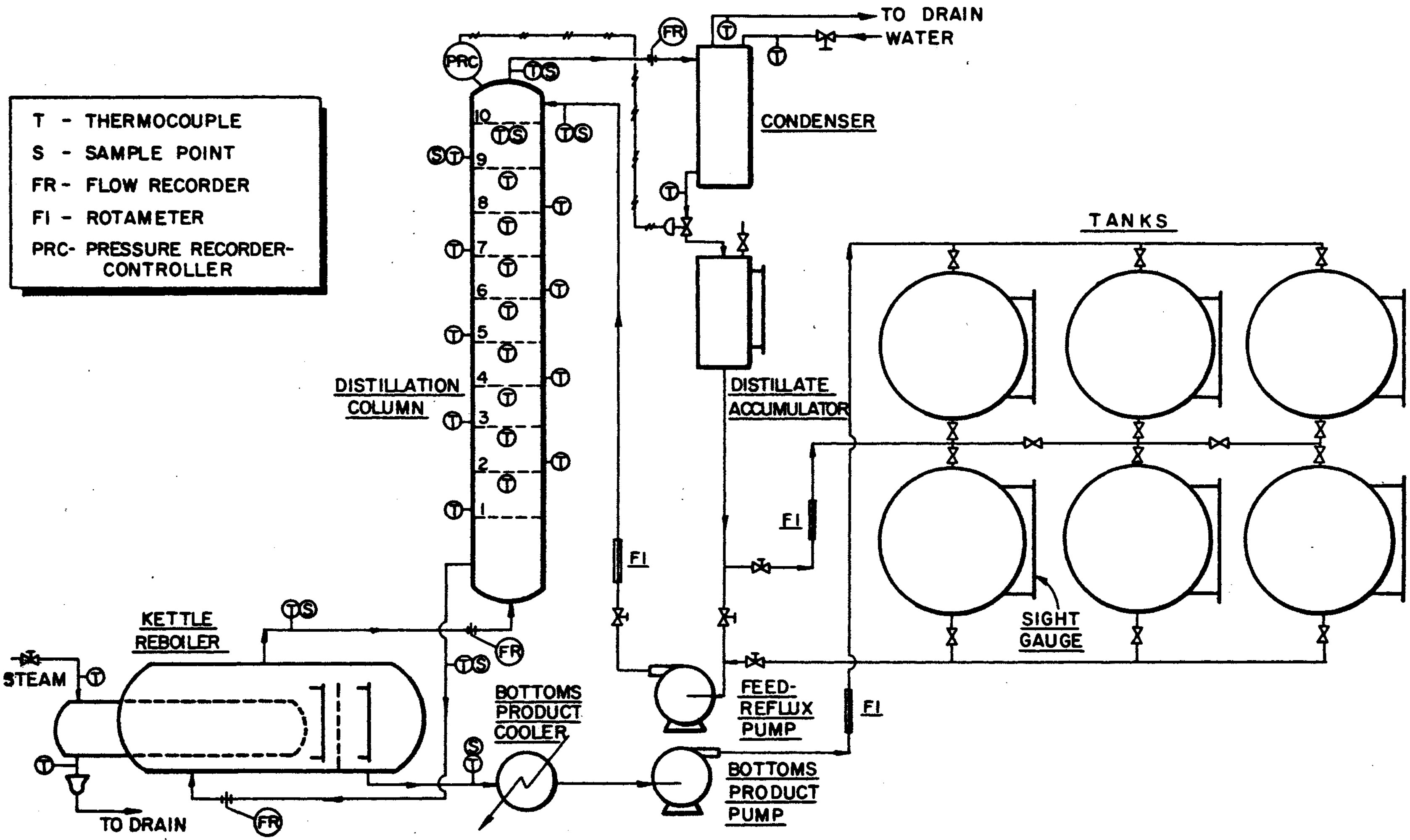

Figure 3. Schematic Flow Diagram.

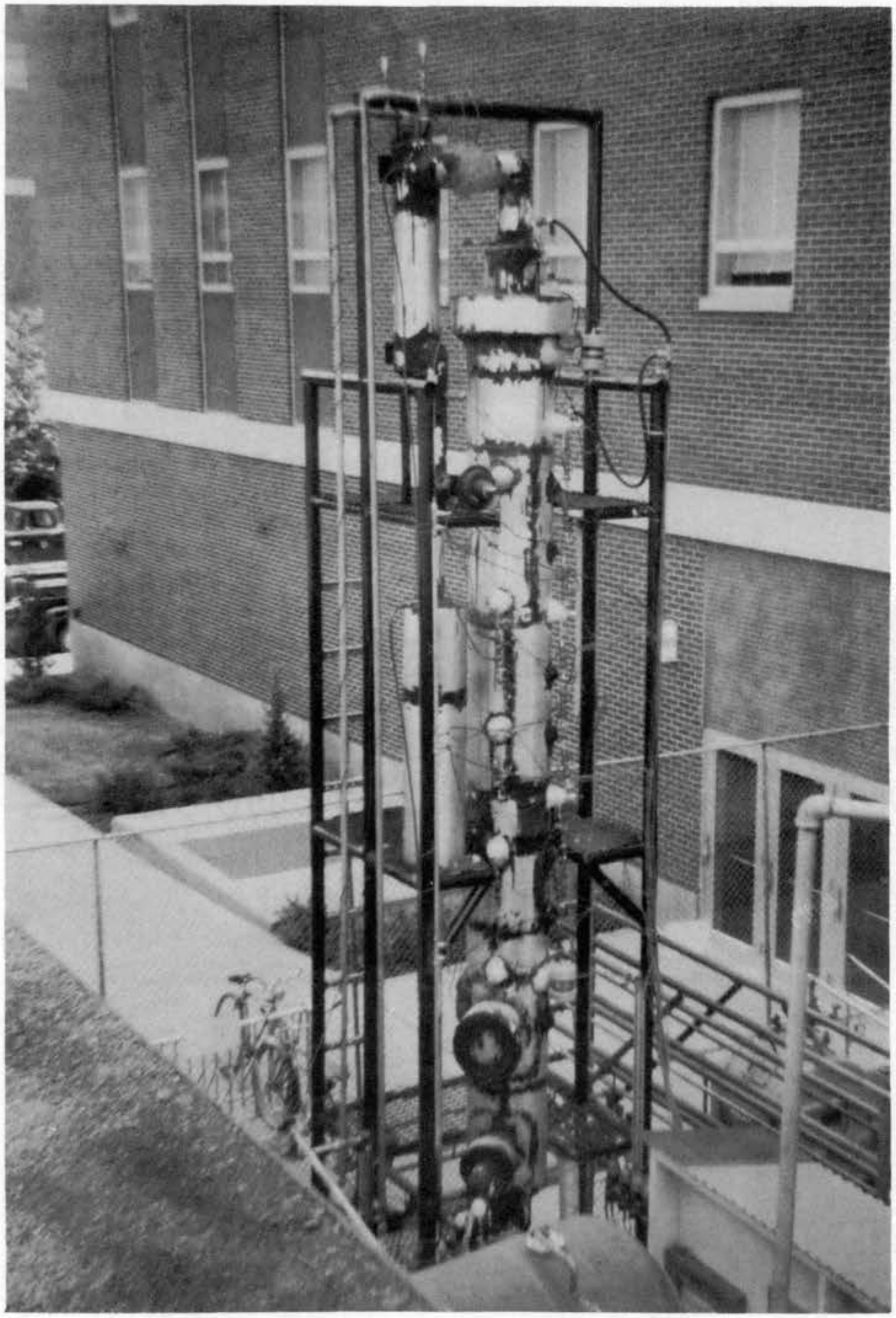

Figure 4. Photograph of Column and Structure.

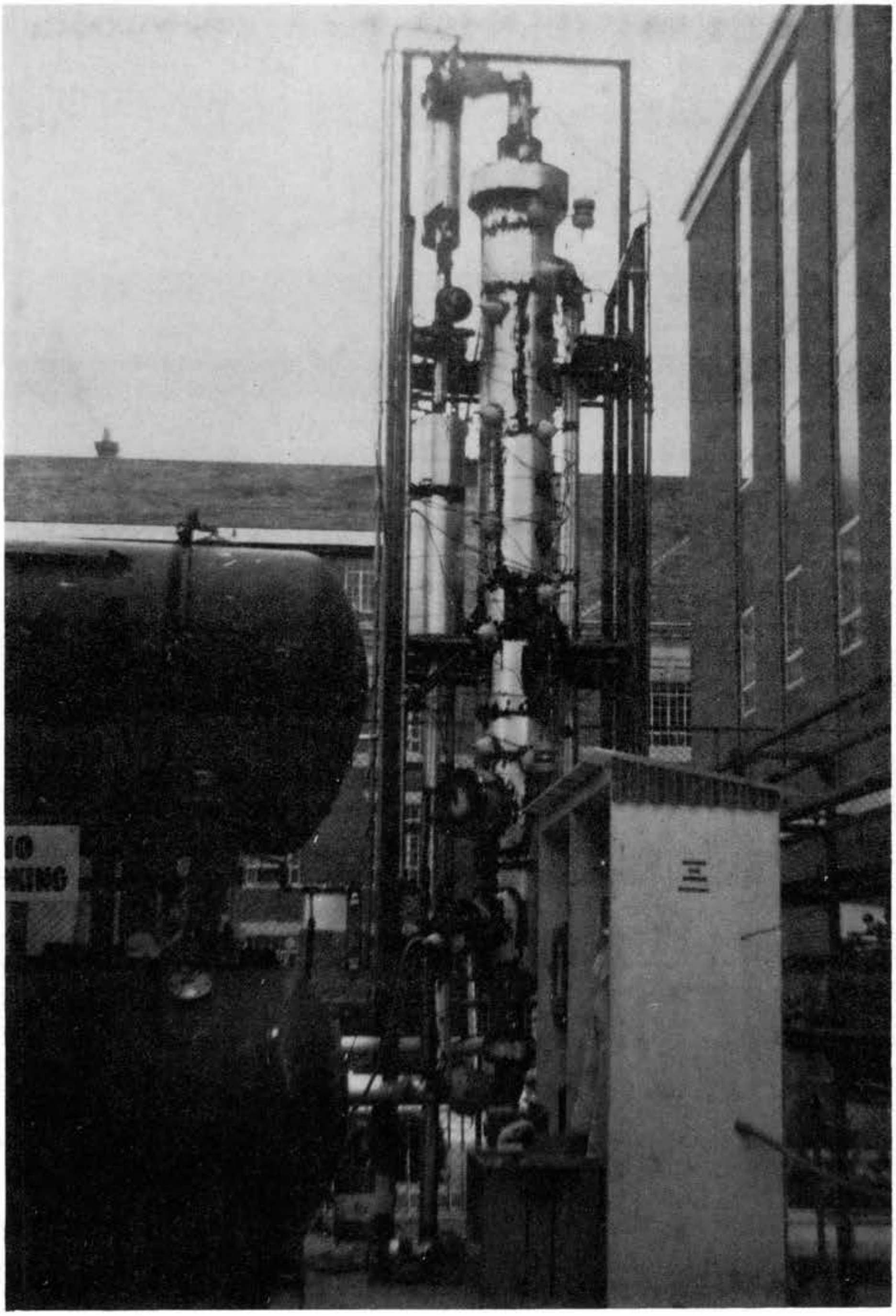

Figure 5. Photograph of Equipment

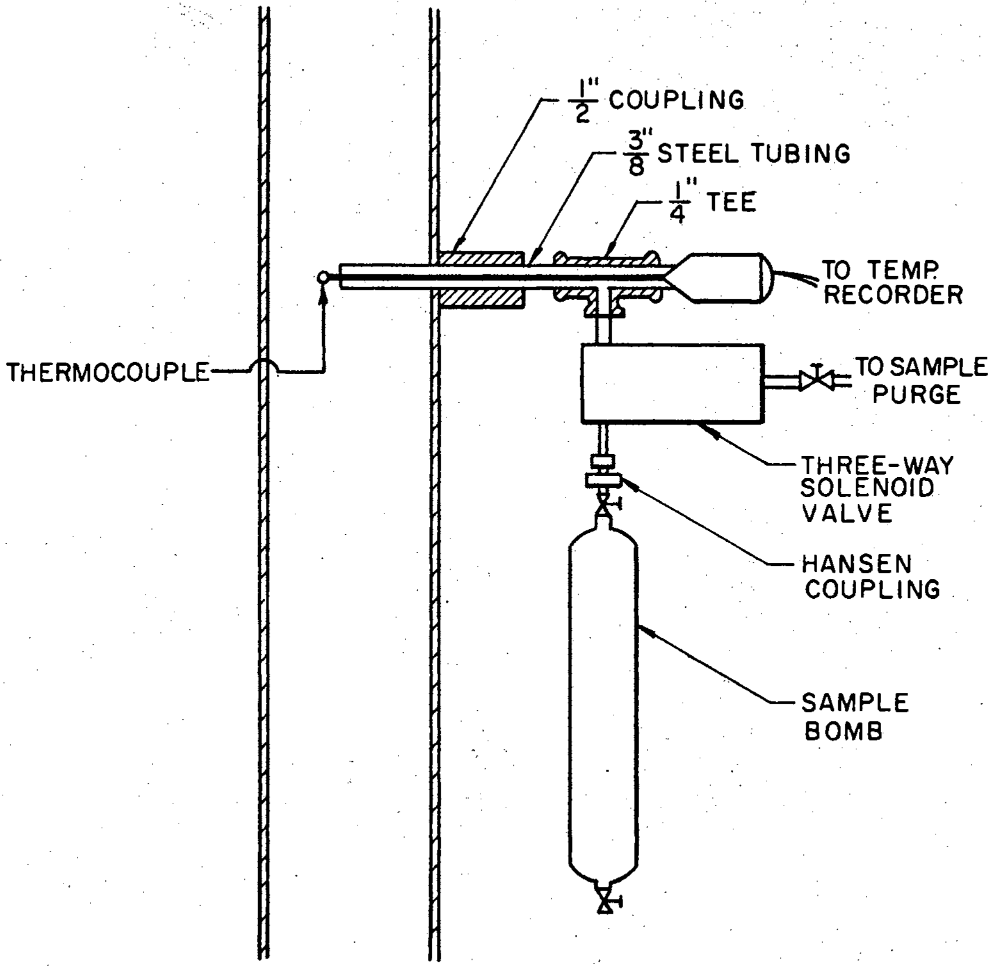

Figure 6. Detail of Sampling System.

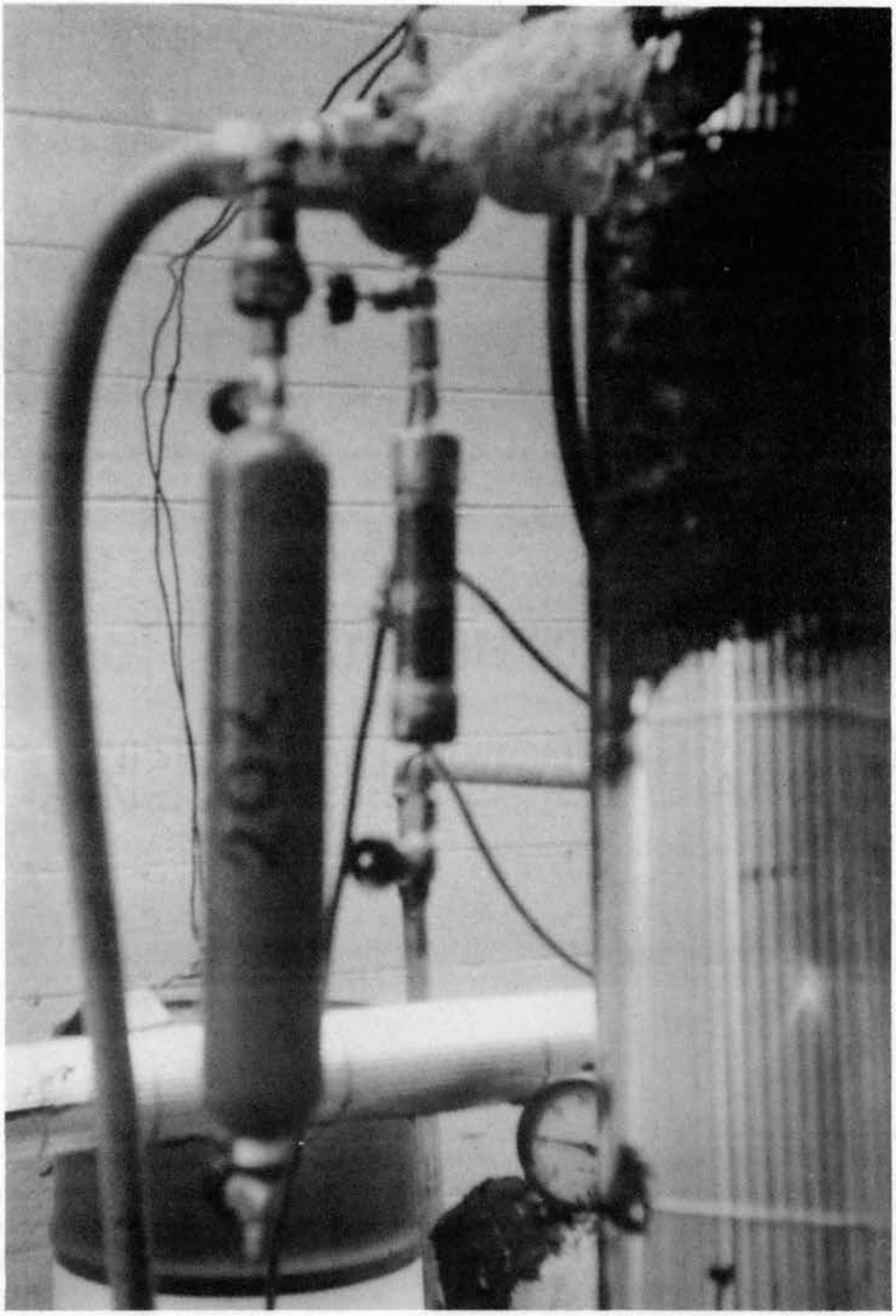

Figure 7. Photograph of Sampling System.

CHAPTER IV

EXPERIMENTAL PROCEDURE

The column was started by filling the reboiler with feed. Steam to the reboiler was then turned on. A bleed valve at the top of the column was opened to allow non-condensable gases to escape from the column. When condensable material first began to flow through the bleed valve, the valve was closed. Pressure then began to build up in the column until the pressure control valve opened. At this point, the feed pump was started with the feed rate set at the desired operating rate. The bottoms product flow was started at its desired operating rate. The distillate rate was then adjusted until the column was in material balance. Distillate rate was adjusted by adjusting the steam rate. Because of a shortage of feed stock, the distillate and bottoms products were recycled to the feed tank. The column was allowed to run two hours after the column pressure, temperatures, and flow rates lined out. Experimental data presented in Appendix G show that changes in product composition with respect to time were insignificant after this two hour running period.

Dynamic Behavior

The procedure for obtaining transient data to test the applicability of the lumped parameter model (15, 16) for predicting the dynamic performance of the column was to start the column and allow it to reach steady state. (Steady state is defined as the point where no measurable change in product and tray compositions and temperatures occurs.) The feed composition was then changed by changing feed tanks. All other column parameters were kept constant.

Samples were taken from both products, the feed, liquid to the reboiler and vapor from the reboiler. These samples were taken starting with the initial steady state and ending when the feed stock in the second tank was depleted. This was an upset period of approximately fifty minutes. The samples were taken at time intervals ranging from about one and one-half minutes at the time of the feed upset to about eight minutes as the column approached a new steady state. In addition to the samples mentioned above, samples were taken from the vapor to the feed section during the initial steady state.

The initial steady state data were used to evaluate the separation parameter in the lumped parameter model. The results from this model were compared with experimental data to determine the reliability of the model for predicting the transient behavior of the column.

Feed Forward Control

The procedure for obtaining feed forward control data was to start the column and allow it to reach steady state. Samples were then taken from the feed and distillate and bottoms products. The respective flow rates were recorded, and the column was shut down. The second or upset feed tank was also sampled. The initial steady state conditions were simulated using tray-by-tray data from the O. S. U. Tray-by-Tray Program (6). These tray-by-tray data and the final feed composition data were used with a feed forward control computer program to predict the rate at which the bottoms flow rate had to be changed in order to maintain a constant bottoms product composition. The column was then restarted and brought to the same initial steady state conditions. Samples were taken from the feed and distillate and bottoms products to assure that the same steady state conditions had been achieved. At this time the feed tanks were changed. A time of one minute was allowed to elapse between the time the feed tanks were changed and the time that the run was started. A timer was then started and the bottoms rate was changed according to the predicted rate. Samples of the bottoms product were taken at time intervals ranging from two minutes at the start of the upset to four minutes as the new steady state was approached to determine if any change in composition occurred. The elapsed time of one minute, which was determined experimentally for the feed rate that was used, was to allow the feed upset to reach the column from the feed tanks. Experimental data showing the feed profile test are presented in Figure 9.

The bottoms flow rate was controlled manually. Therefore, the bottoms rate was changed in steps. The method used to determine the flow rate at each of these steps, which is similar to the procedure used in graphical integration, is illustrated in Figure 8 for run FFC 2-2. A curve of the computed bottoms rate response was divided into time intervals ranging from two minutes at the start of the upset to four minutes at the end of the upset period. The bottoms flow rate for each time increment was determined by a horizontal line which gave an equal amount of area above and below the horizontal line bounded by the bottoms rate response curve, the horizontal line, and the boundaries of the time increment. The bottoms flow rate was changed at the center of each time increment.

Chromatograph Operation

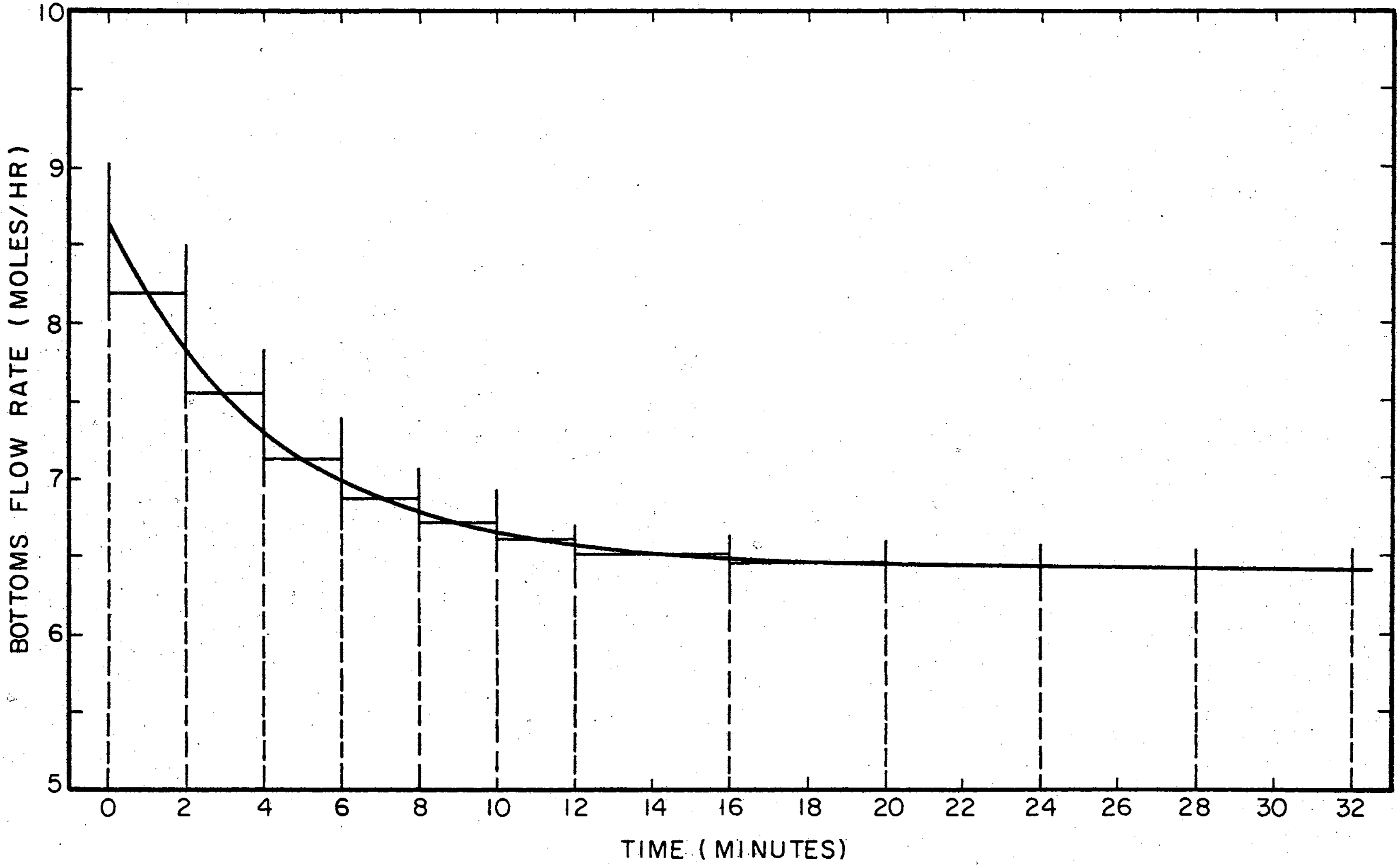

Figure 8. Illustration of Method Used to Determine Bottoms Rate for Feed Forward Control Runs.

In order to analyze the samples on the gas chromatograph, the integrator and chromatograph were both turned on, and the chromatograph was allowed to reach thermal steady state. The balance needle on the integrator was used to determine when the chromatograph reached thermal steady state. Drift of the balance needle stopped when thermal steady state was achieved. The sample bombs were packed in ice prior to withdrawing samples from them. To analyze a sample, a small portion of the sample was extracted from the sample bomb by pouring it into a small pre-cooled glass bottle. A two micro-liter portion was then injected into the chromatograph. The results of the analyses were recorded by a digital printer connected to the integrator. This information was recorded as frequency counts which are proportional to the amount of each component analyzed. The count fraction of each component in a sample was obtained by dividing the number of counts for a given component by the sum of the counts for all of the components in the sample. The count fractions were converted to mass fractions using calibration data. The mass fractions were then converted to mole fraction. Further details on the chromatograph calibration are given in Appendix B.

CHAPTER V

DISCUSSION OF RESULTS

The three major objectives of this project were to obtain transient experimental distillation data to

(1) test the applicability of a first order lumped parameter model for predicting the dynamic behavior of distillation columns;

(2) test the applicability of the lumped parameter model for use in feed forward control;

(3) determine if tray-by-tray data could be substituted for experimental initial steady state data in the lumped parameter model.

All experimental data were taken for a step change in feed composition. A brief study was conducted to determine the type of feed profile actually obtained as the feed stream entered the column. The results of this study are shown in Figure 9. The data show that, for a feed rate of three gallons per minute, a total time of about two minutes is required for the feed composition to completely change. However, only about 0.6 minute is required for a ninety per cent change in feed composition. A feed rate of about three gallons per minute was used in each of the thirteen runs made. Data presented in Appendix G show that the column required approximately sixty minutes to reach a new steady state. Thus, only about 3.4 per cent of the upset period is required for the feed composition to change completely, and the feed upset can be assumed to occur as a step change.

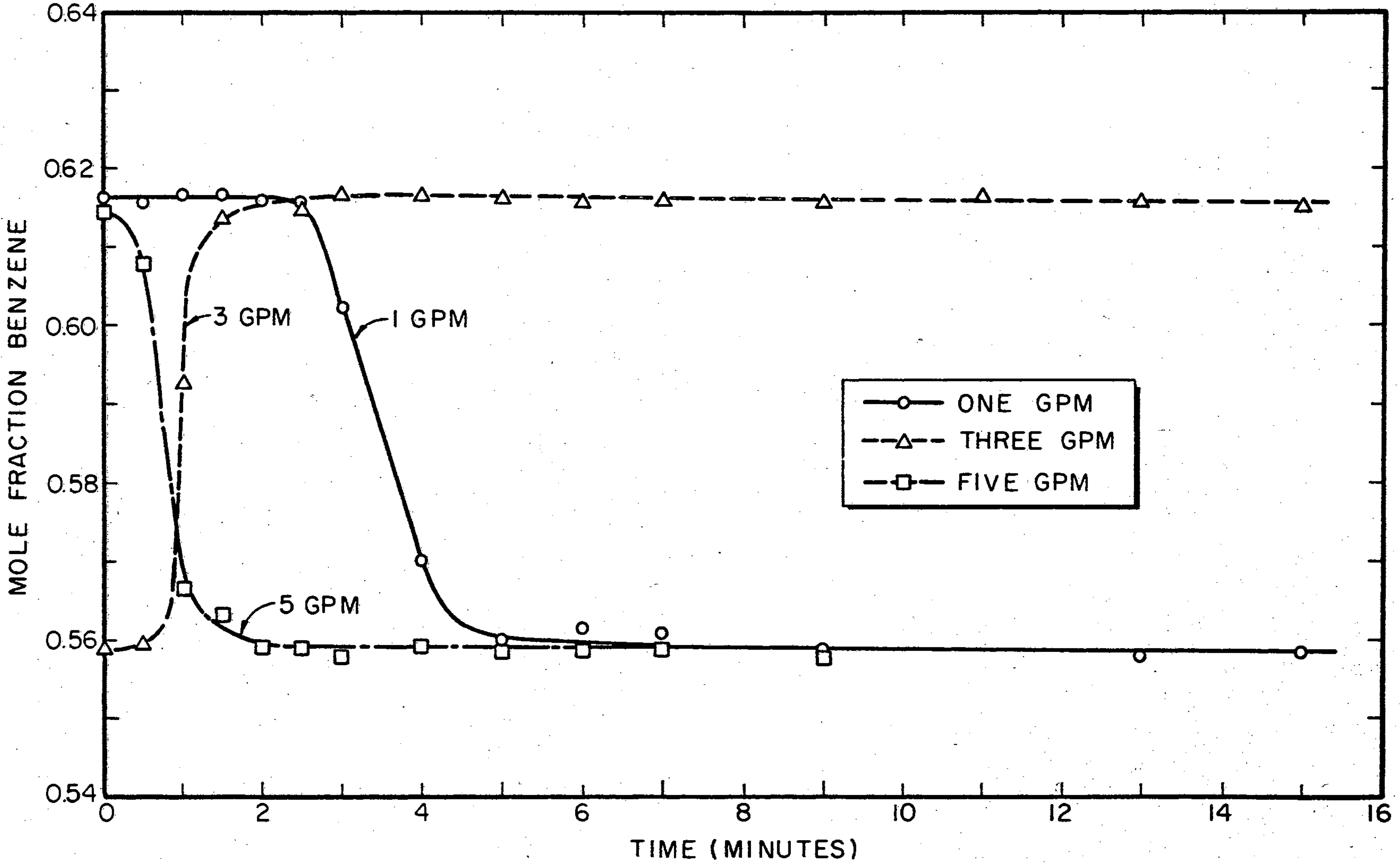

Figure 9. Plot of Feed Profile.

Reproducibility of sample analysis could be obtained within 0.0025 mole fraction as shown in Tables VII and VIII in Appendix B. Thus, reasonable accuracy of analysis was obtained.

A brief study was conducted to determine the accuracy of this sampling procedure. In conducting this study a standard solution of benzene and toluene was prepared and analyzed in triplicate. A sample was taken from the standard using an evacuated sample bomb. The sample bomb was cooled and a sample withdrawn in the same manner used in analyzing all samples. The sample was then analyzed in triplicate. The results of this test are shown in Table I.

These data show that the difference between the average of the three analyses of the standard and the average of the three analyses of the sample was 0.0004 mole fraction benzene. These data also show that the triplicate analyses were accurate to +0.0008 and -0.0006 mole fraction benzene. Thus, the accuracy of the sampling procedure is within the accuracy of the analysis.

Dynamic Behavior

A total of five runs were made to test the applicability of a first order lumped parameter model for predicting the dynamic behavior of distillation columns. Four of these runs were conducted using a binary system. The remaining run was made using a ternary system. A summary of these runs is presented in Table II. Data for two typical runs are presented in Figures 10 and 11. The data for these runs are shown in Tables XI through XVI and Figures 28 through 31 in Appendix G.

The transient response curves obtained from the first order lumped parameter model are shown in Figures 10, 11, and 28, 29, 30, and 31 in Appendix G. These figures show that the column product composition

TABLE I

RESULTS OF SAMPLING TEST

	Compositions, m.f. Benzene	
Analysis	Standard	Sample
1	0.5508	0.5499
2	0.5504	0.5508
3	0.5500	0.5494
Average	0.5504	0.5500
Maximum Error		
(+)	0.0004	0.0008
(-)	0.0004	0.0006

TABLE II

SUMMARY OF DYNAMIC BEHAVIOR RUNS

	Flow Rates, moles/hr.			
	Feed		Distillate	
Run	Initial	Final	Initial	Final
2-1	15.366	15.491	6.076	6.297
2-2	16.139	16.271	7.748	6.982
2-3	16.003	16.364	5.655	6.347
2-4	15.543	16.042	5.900	6.680
3-1	14.048	14.020	7.343	6.922

	Compositions*					
	Feed		Distillate		Bottoms	
Run	Initial	Final	Initial	Final	Initial	Final
2-1	0.5077	0.7231	0.7579	0.8929	0.3859	0.4782
2-2	0.5838	0.6199	0.8021	0.8283	0.4224	0.4591
2-3	0.5380	0.6592	0.7700	0.8500	0.4045	0.4816
2-4	0.5379	0.6729	0.7420	0.8387	0.3775	0.4661
3-1	0.4070-B	0.3687-B	0.6920-B	0.6622-B	0.1072-B	0.0943-B
	0.4920-T	0.5238-T	0.2866-T	0.3128-T	0.7112-T	0.7200-T

*Compositions are given as mole fraction benzene unless specified otherwise.

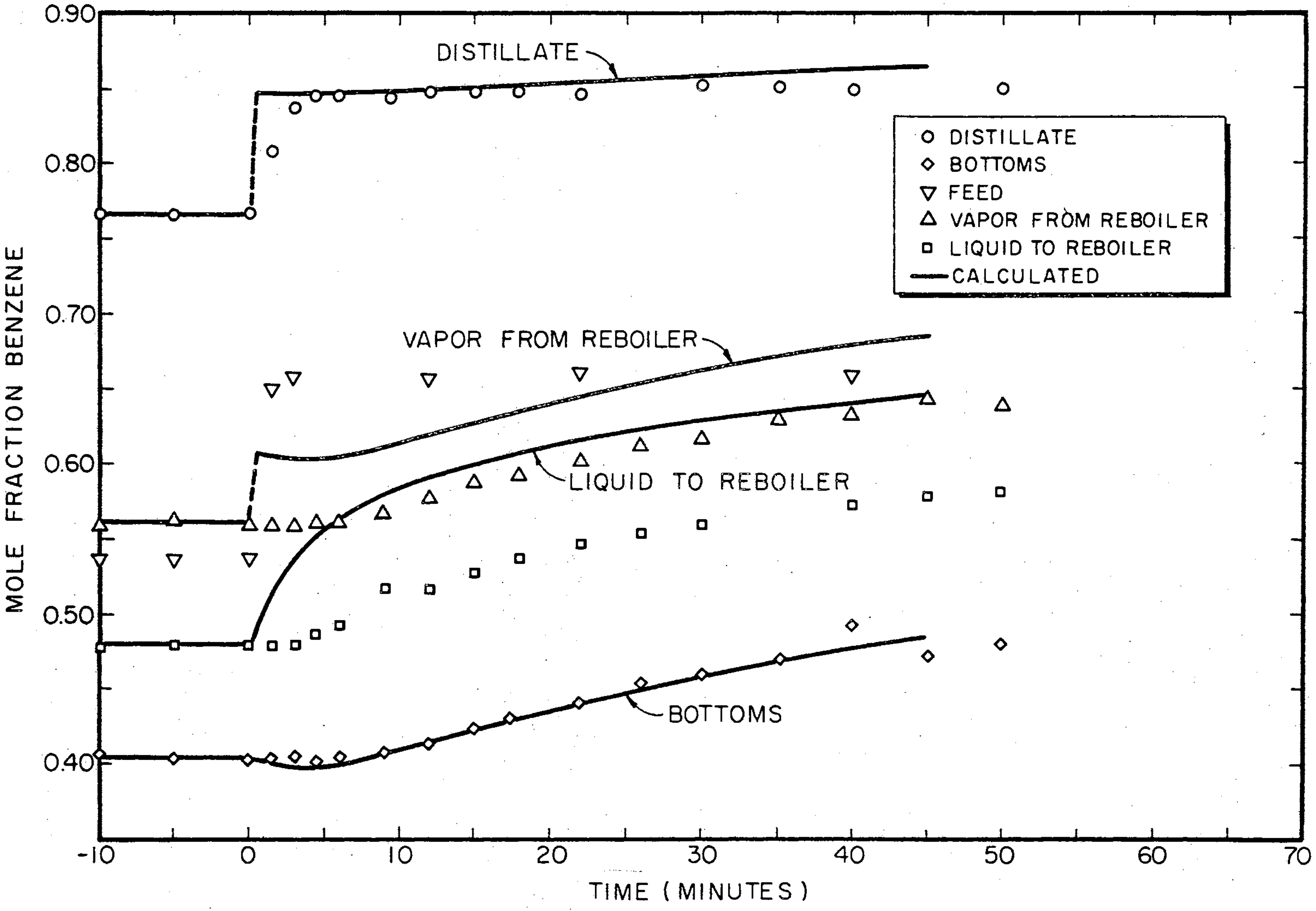

Figure 10. Experimental and Calculated Data for Run 2-3.

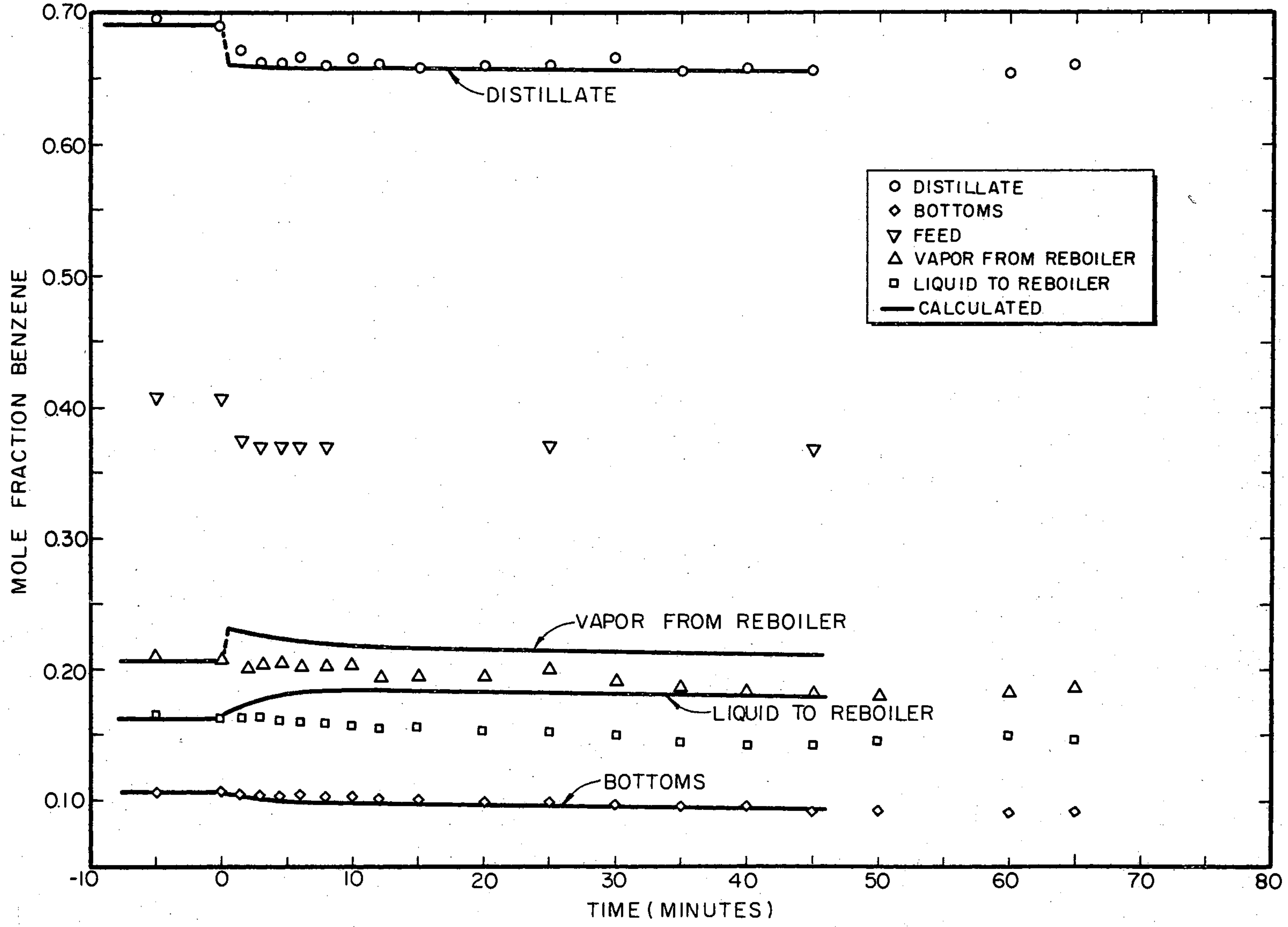

Figure 11. Experimental and Calculated Data for Run 3-1.

could be predicted accurately with the first order model. However, the model did not accurately predict the composition of the vapor from and the liquid to the reboiler. An attempt was made to fit these internal streams. The accuracy for each of the product streams decreased as the accuracy of the fit of the internal streams increased. The results of a typical example are shown in Figure 12 for run 2-4.

The inability of the model to fit the internal streams may be a result of some simplifying assumptions used in developing the model. The model was developed using the assumptions that the feed section and the reboiler were theoretical stages. A comparison, shown in Table III, of experimental compositions of the vapor leaving the reboiler with values calculated for a theoretical stage shows that the reboiler is not a theoretical stage. Thus, the assumption that the reboiler was an ideal stage is not valid.

Work with the experimental equipment which was conducted after this project was completed revealed that samples of the liquid stream leaving the feed section were in error. Thus, no data were obtained to test the assumption that the feed tray was a theoretical stage.

The initial composition of the vapor to the feed section was not obtained for runs 2-1 and 2-2. However, this stream was sampled and analyzed in the remaining three runs. The purpose of analyzing this stream was to obtain the best available value of the separation parameters. The composition of this stream for runs 2-1 and 2-2 was calculated using the assumption that the feed section was ideal. However, the accuracy of the computed transient response curve was not greatly increased when experimental data were used around the feed section. Thus, reasonable results can be obtained by substituting calculated data for the experimental composition of the vapor to the reboiler.

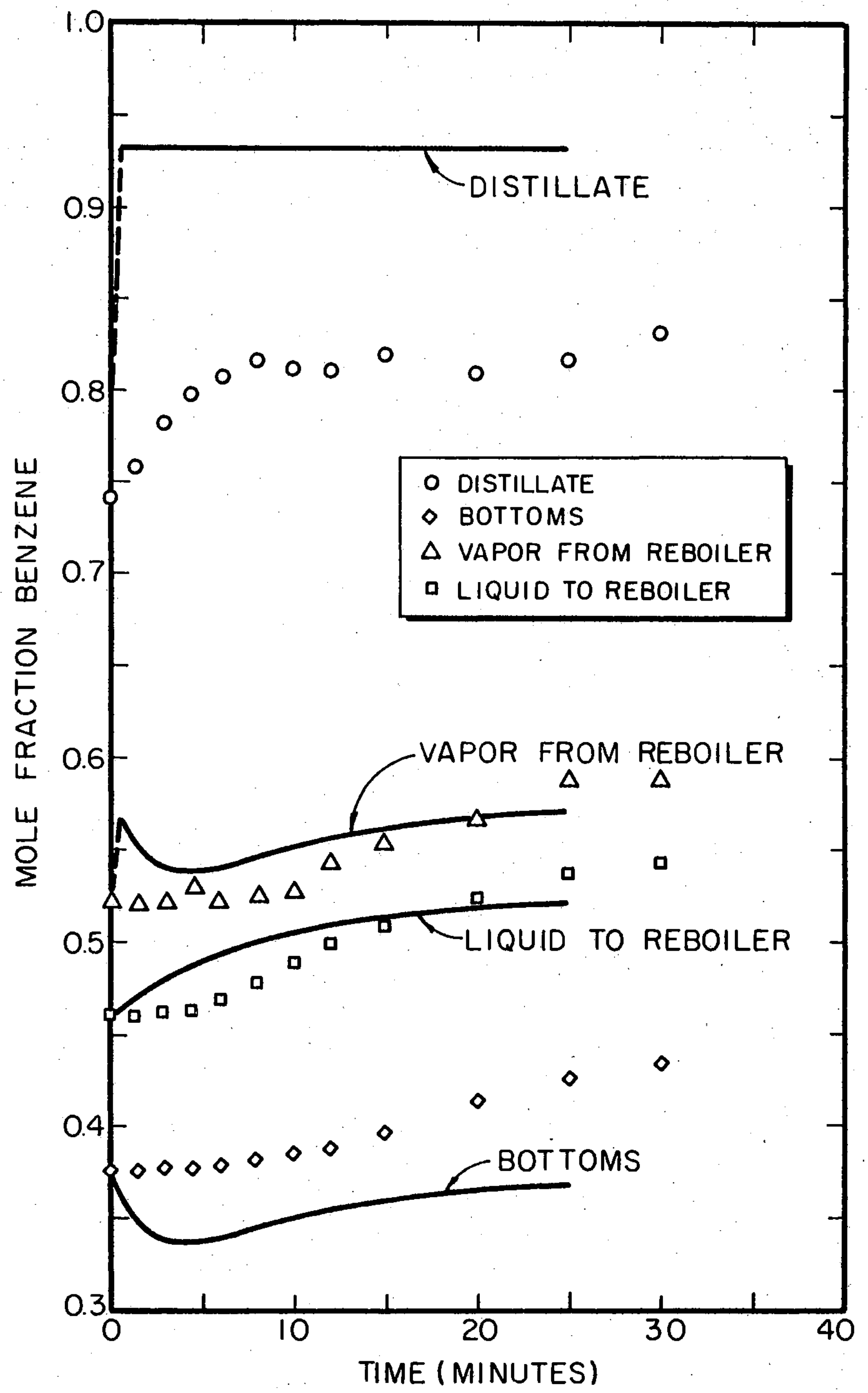

Figure 12. Results of Attempt to Fit Model to Internal Streams for Run 2-4.

TABLE III

RESULTS OF TEST TO DETERMINE IF THE REBOILER IS THEORETICAL STAGE

Run	Composition of Vapor Leaving Reboiler, m.f. Benzene Experimental	Theoretical Stage
2-1	0.5374	0.5858
2-2	0.5858	0.6268
2-3	0.5630	0.6105
2-4	0.5154	0.5805
3-1	0.2071	0.2420

Experimental data, presented in Tables XI to XV, show that the distillate composition changes rapidly with a change in feed composition. However, since the feed entered on the top tray, an almost instantaneous change in distillate composition could be expected.

The bottoms composition was also found to change very rapidly with a change in feed composition. However, the rate at which the bottoms composition changed was slower than the rate at which the distillate composition changed. In addition the bottoms composition in runs 2-1 and 2-2 was found to drop slightly before beginning to rise. Also, the calculated curve of bottoms composition was found to drop slightly in a manner similar to the experimental data. The reason for this drop in composition is not clear. However, the calculated bottoms composition curve is obtained from material balance equations thus indicating that the drop in bottoms composition is a material balancing phenomenon rather than heat balancing.

Feed Forward Control

Eight runs were made to test the applicability of the lumped parameter model for use in feed forward control. Five of these runs were made using the benzene-toluene system. The remaining three runs were made using the benzene-toluene-para-xylene system. A summary of these runs is shown in Table IV. The data for these runs are shown in Tables XVI through XXIII and Figures 32 through 37 in Appendix G. The computed bottoms rate response curves are presented in Figures 38 through 45 in Appendix G. Data for two typical runs are presented in Figures 13 and 14.

The results of this feed forward work show that the bottoms composition could be kept constant for small feed upsets. However, as the

TABLE IV

SUMMARY OF FEED FORWARD CONTROL RUNS

Run	Feed Composition, m.f. Benzene Initial	Final	Difference*
FFC 2-2	0.4279	0.4779	0.0499
FFC 2-3	0.5413	0.3910	0.1503
FFC 2-4	0.4536	0.4661	0.0125
FFC 2-5	0.4577	0.4151	0.0426
FFC 2-6	0.4071	0.4752	0.0681
FFC 3-1	0.3026	0.3738	0.0712
FFC 3-3	0.3858	0.4367	0.0509
FFC 3-4	0.3246	0.4223	0.0977

Run	Per Cent Upset	Initial Bottoms Composition, m.f. Benzene	Maximum Per Cent Error in Bottoms Product, m.f. Benzene	Bottoms** Error
FFC 2-2	11.7	0.2659	3.7	0.0098
FFC 2-3	27.8	0.3764	13.9	0.0523
FFC 2-4	2.8	0.2742	nil	nil
FFC 2-5	9.3	0.2877	3.8	0.0109
FFC 2-6	16.7	0.2553	14.7	0.0376
FFC 3-1	23.5	0.1483	3.0	0.0045
FFC 3-3	13.2	0.2301	1.3	0.0029
FFC 3-4	30.1	0.1769	5.2	0.0092

*Difference = Initial - Final Feed Composition

**Bottoms Error = Maximum deviation from initial bottoms composition in mole fraction benzene.

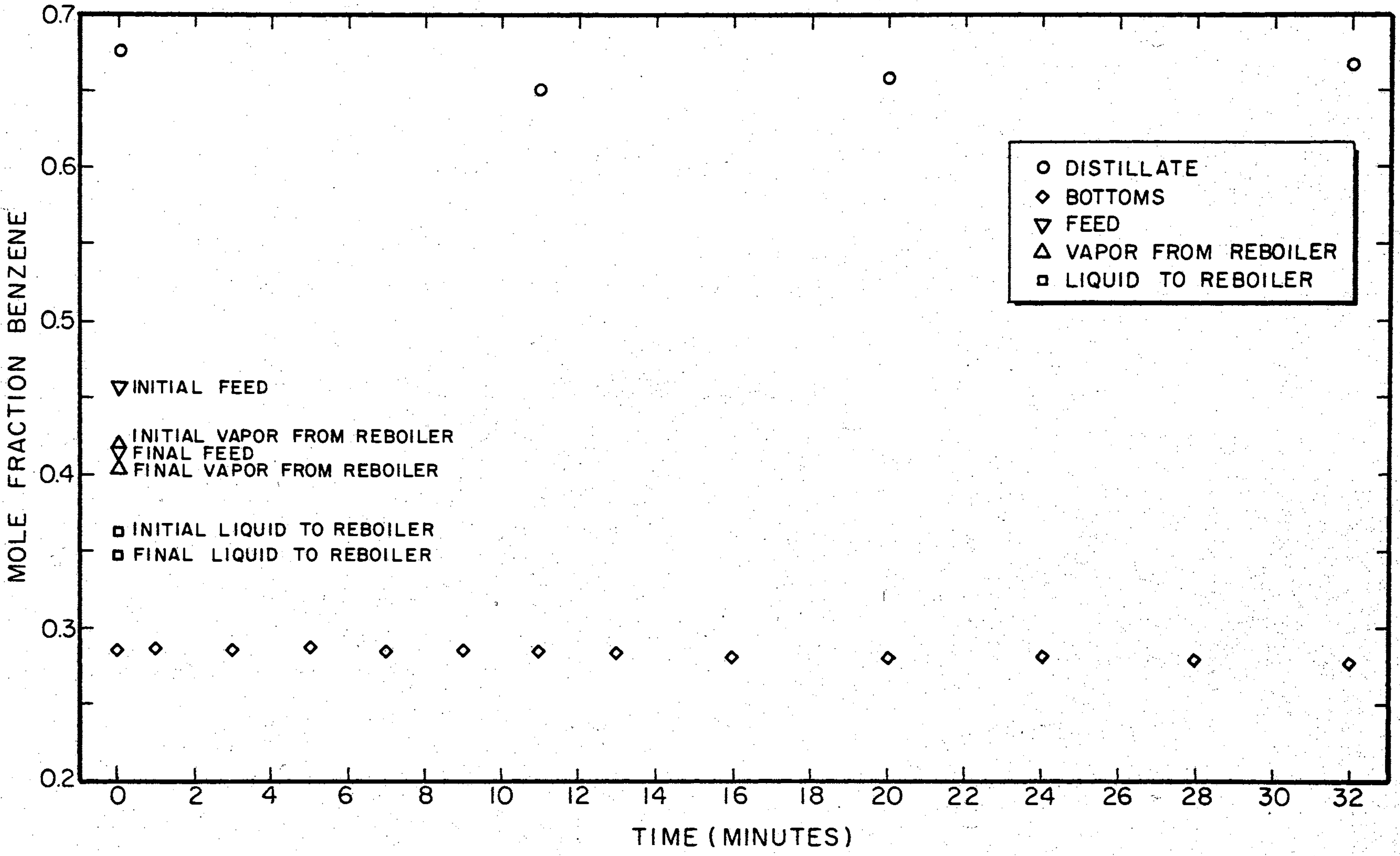

Figure 13. Experimental Data for Run FFC 2-5.

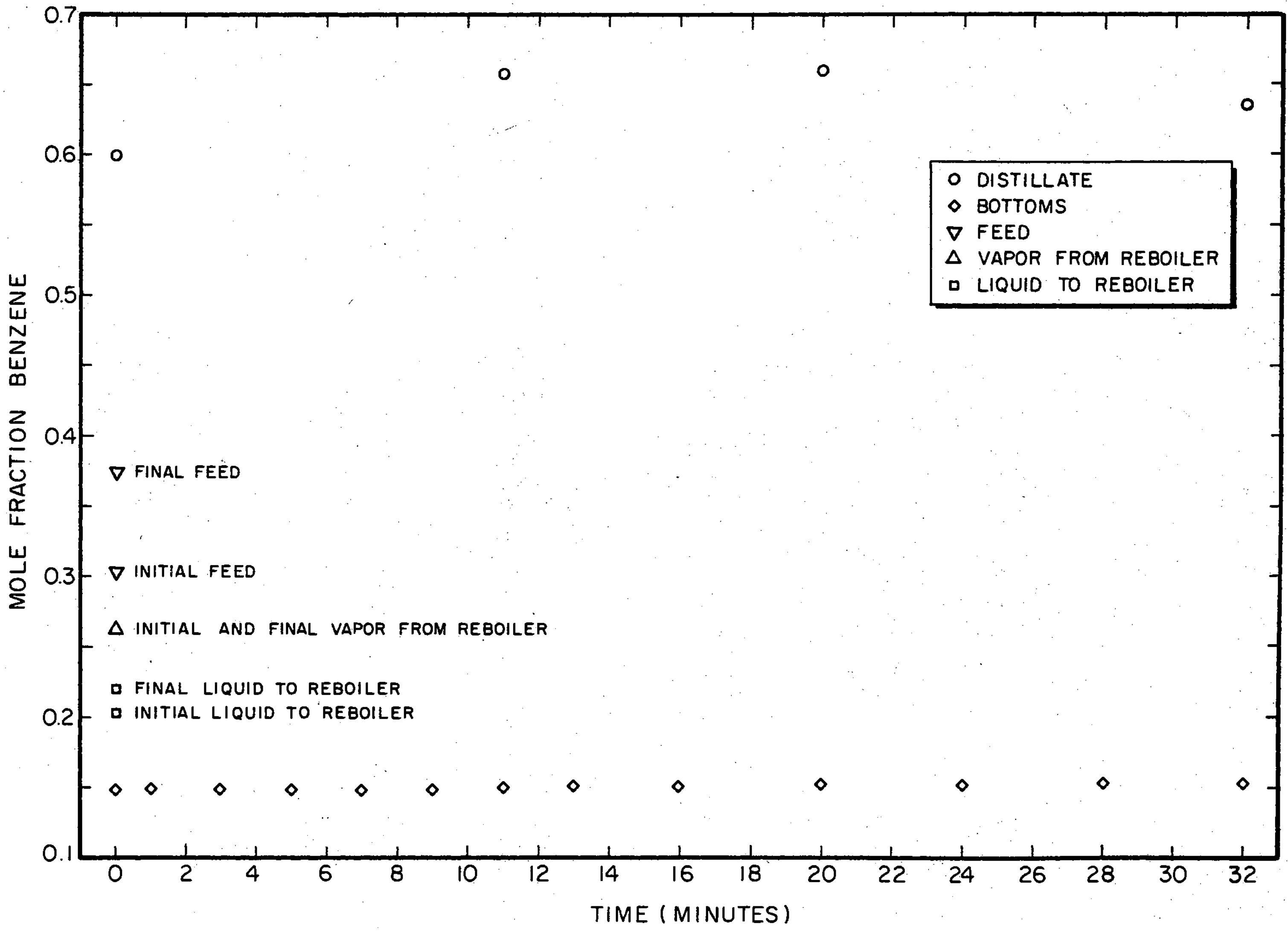

Figure 14. Experimental Data for Run FFC 3-1.

magnitude of the upset increased, the reliability of the model decreased. Figures 15 and 16 show the trend in reliability with the magnitude of the feed upset. Figure 15 is a plot of bottoms error, which is the maximum deviation of the bottoms product composition from the initial steady state composition, versus the per cent change in feed composition. Figure 16 is a plot of bottoms error versus the feed composition change in mole fraction.

Figures 15 and 16 indicate that the feed forward control model is more reliable for ternary systems than for binary systems. However, when para-xylene was added to the system, the over-all benzene concentration decreased. Thus the bottoms rate change was less for a ternary run than for a binary run with the same magnitude of feed upset. This can be further illustrated by comparing the bottoms rate response curves for runs FFC 2-6 and FFC 3-1. These curves are shown in Figures 42 and 43 respectively. The two response curves are almost identical. However, run FFC 2-6 had a feed upset of 16.7 per cent while run FFC 3-1 had a feed upset of 23.5 per cent.

Prediction of Separation Parameters

A study was conducted to determine if tray-by-tray data could be used with the first order lumped parameter model to predict the transient response of distillation columns. A summary of this study is shown in Tables V and VI. Table V shows the separation parameters which were obtained from both experimental and tray-by-tray data. This table shows that the separation parameters for runs 2-1 and 2-2 differed by a factor of about 2.0 for the two types of data. Better agreement was obtained between the two methods of calculation for runs 2-3 to 3-1. The large difference between the two sets of separation parameters for

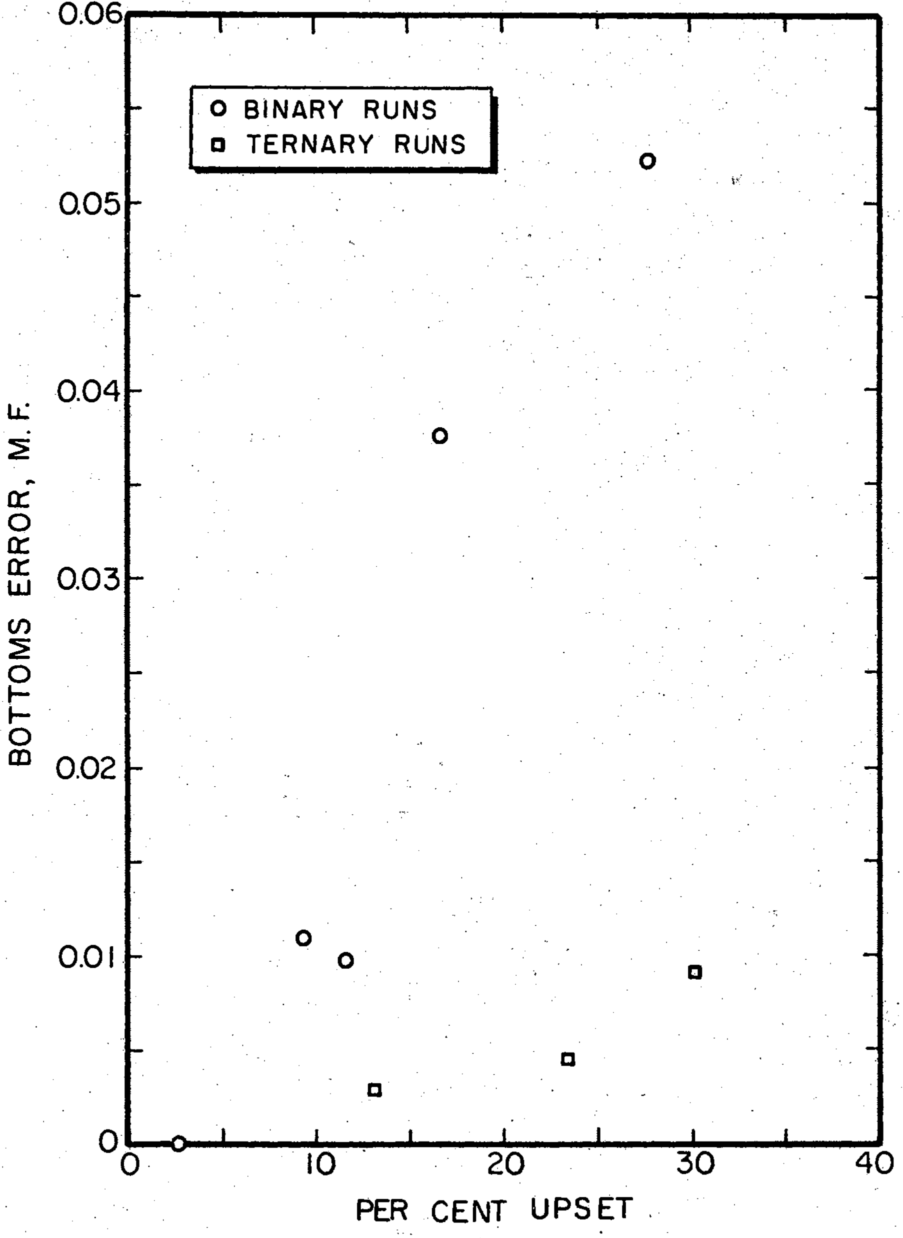

Figure 15. Plot of Bottoms Error Versus Per Cent Upset for Feed Forward Control Runs.

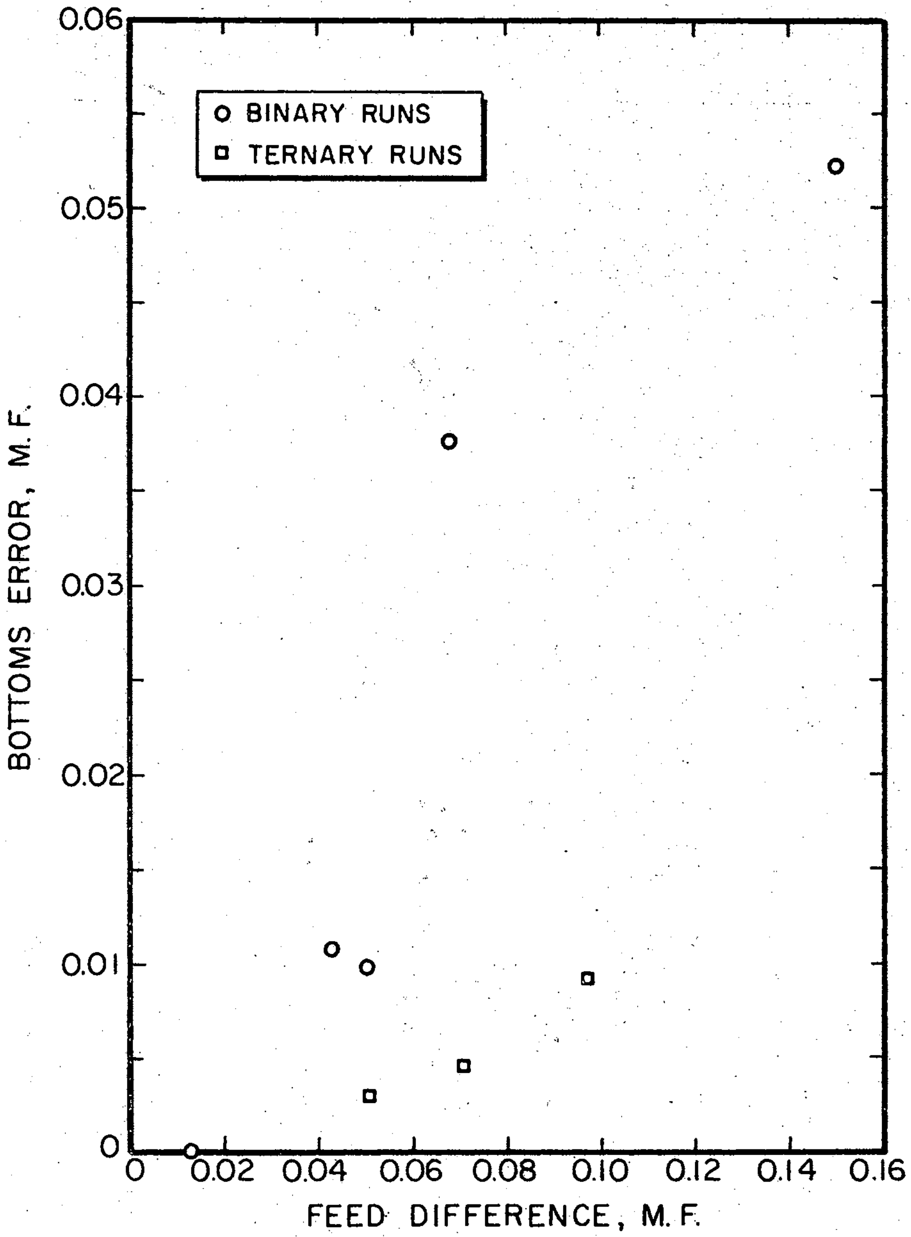

Figure 16. Plot of Bottoms Error Versus Feed Difference for Feed Forward Control Runs.

TABLE V

SEPARATION PARAMETERS

Run	J-Factor From Experimental Data			J-Factor From Tray-by-Tray Data		
	Benzene	Toluene	p-Xylene	Benzene	Toluene	p-Xylene
2-1	21.490	21.488	-	10.405	9.678	-
2-2	20.501	20.509	-	12.240	11.282	-
2-3	14.995	14.996	-	10.259	9.482	-
2-4	7.143	7.143	-	7.703	7.184	-
3-1	15.635	15.443	16.712	11.833	11.251	11.895

runs 2-1 and 2-2 may have been caused by the fact that the vapor to the feed section was not sampled during these two runs. This stream was sampled in runs 2-3 through 3-1. Thus, more accurate values of the separation parameters were obtained for runs 2-3 through 3-1 than for runs 2-1 and 2-2.

Since the separation parameters were not the same, Table VI was prepared to compare the product compositions which were predicted from the two types of data. This table shows the products composition which were calculated using experimental data and tray-by-tray data after twenty-five minutes of run time. It also shows that the product compositions can be compared to two decimal places for most of the runs. The best fit of experimental data was obtained using experimental data as the starting point.

Figures 17 and 18 show the transient response curves obtained using the two types of data for runs 2-1 and 2-4. Poor agreement was obtained between the two types of separation parameters for run 2-1 while good agreement was obtained for run 2-4. The poor agreement for run 2-1 can probably be attributed to the large difference in separation parameters as shown in Table V. Good agreement was obtained between separation parameters for run 2-4.

TABLE VI

CALCULATED PRODUCT COMPOSITIONS* AFTER TWENTY-FIVE MINUTES INTO RUN

Distillate

Run	Separation Parameter Evaluated From Experimental Data	Tray-by-Tray Data
2-1	0.8911	0.8979
2-2	0.8589	0.8601
2-3	0.8594	0.8374
2-4	0.8092	0.8103
3-1	0.6567	0.6596

Bottoms

Run	Separation Parameter Evaluated From Experimental Data	Tray-by-Tray Data
2-1	0.4216	0.4451
2-2	0.4339	0.4467
2-3	0.4663	0.521
2-4	0.4228	0.4237
3-1	0.0969	0.0979

*Compositions are given as mole fraction benzene.

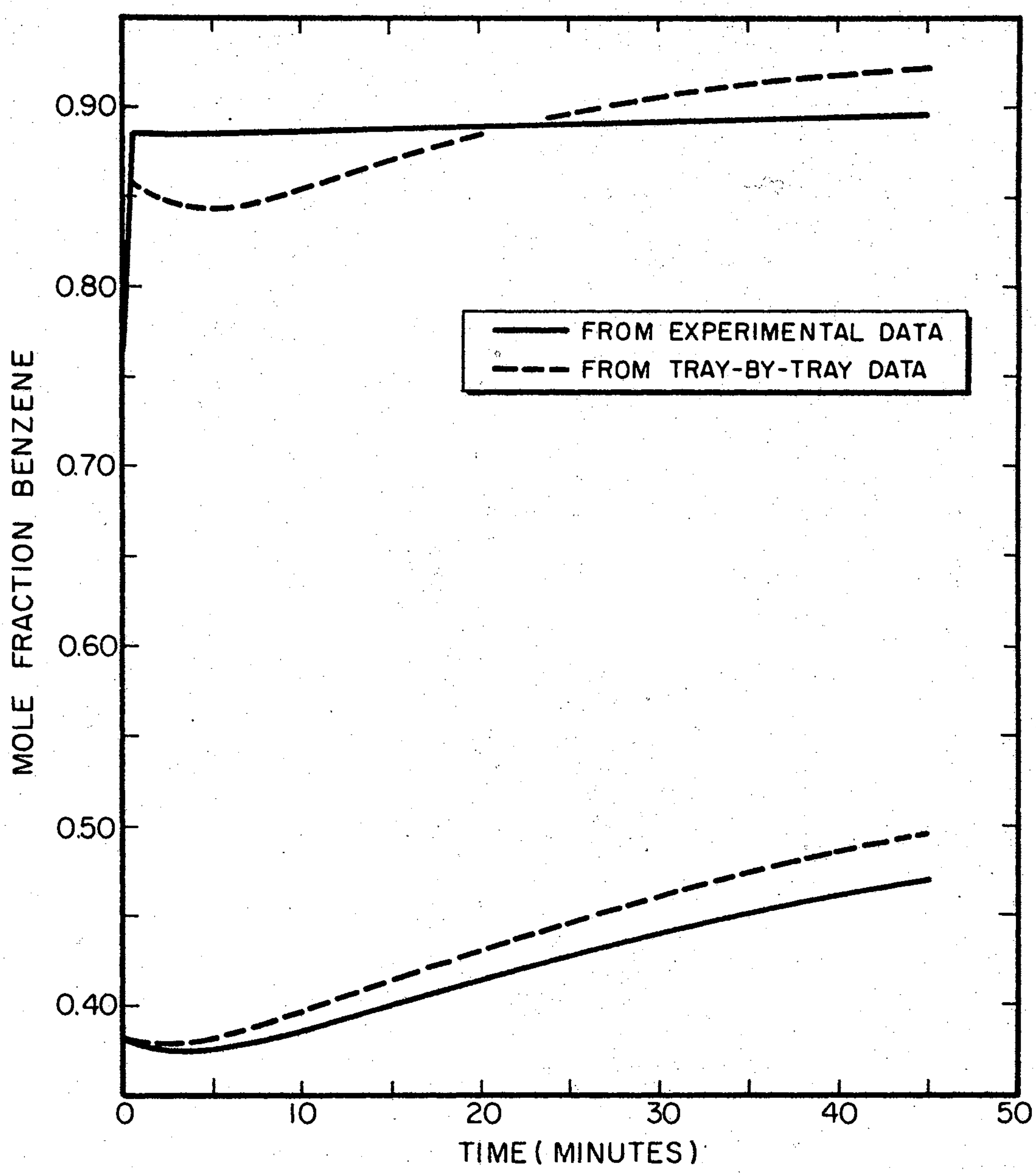

Figure 17. Comparison of Response Curves Obtained from Experimental and Tray-by-Tray Data for Run 2-1.

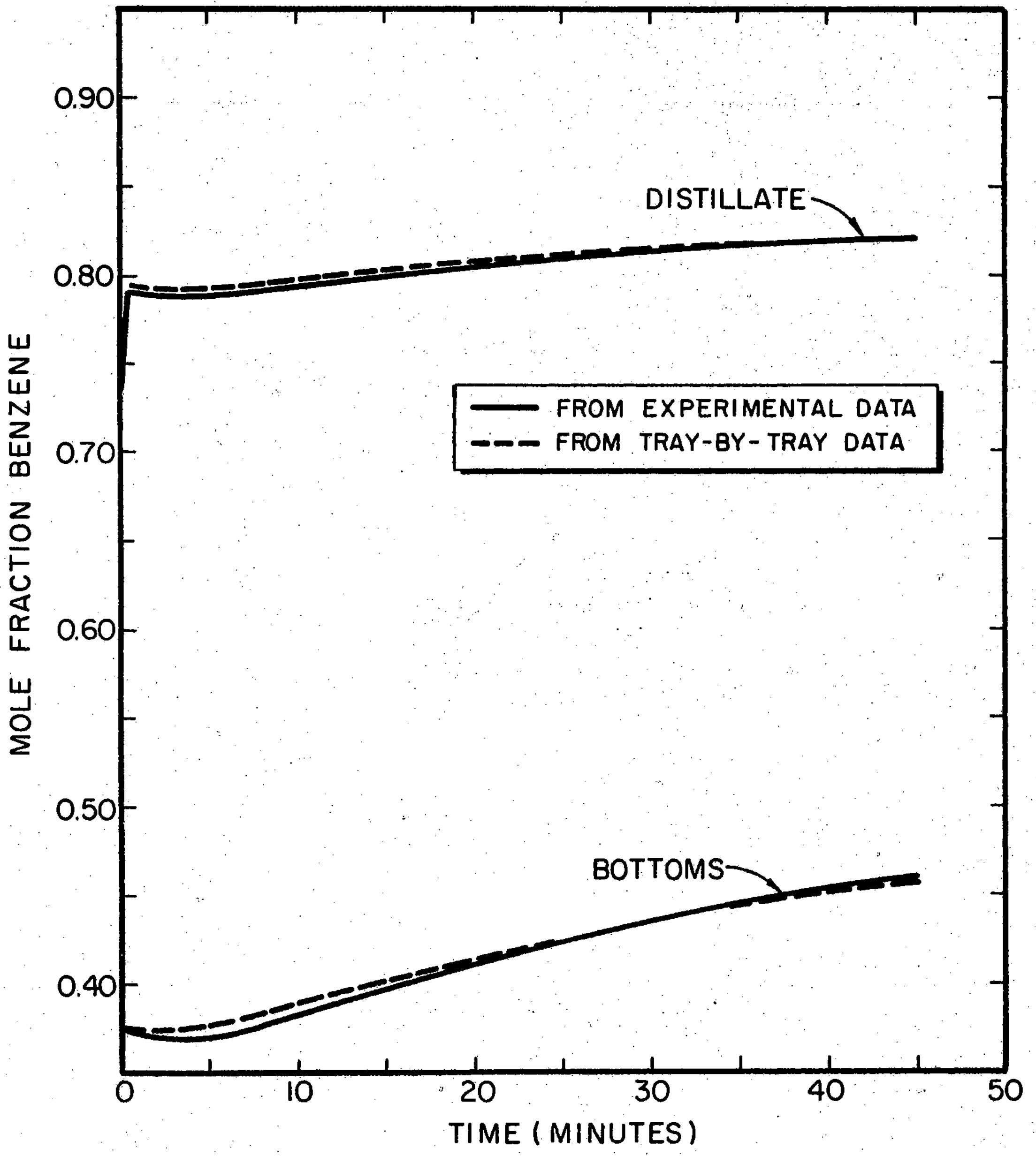

Figure 18. Comparison of Response Curves Obtained from Experimental Data and Tray-by-Tray Data for Run 2-4.

CHAPTER VI

CONCLUSIONS AND RECOMMENDATIONS

Conclusions

The objectives of this research project were to obtain transient experimental data to (1) test the applicability of a first order lumped parameter model for predicting the dynamic performance of distillation columns, (2) test the applicability of the lumped parameter model for use in feed forward control, and (3) determine if tray-by-tray data could be substituted for experimental data with the lumped parameter model.

Five runs were made to determine the applicability of the lumped parameter model for predicting the dynamic behavior of the column. Experimental data from these runs were used to show that the product compositions can be accurately predicted with the lumped parameter model. However, the model cannot be used to predict the compositions of the internal streams.

Eight runs were made to determine the applicability of the lumped parameter model for use in feed forward control. Experimental data show that the bottoms composition could be kept constant for small changes in feed composition. However, as the magnitude of the upset increases, the accuracy of the model decreases. This decrease in accuracy of the model with an increase in the magnitude of the feed upset was expected because the separation parameter is assumed to be constant for small changes in feed composition. As the magnitude of the upset increases the validity of the assumption of constant separation parameter decreases.

The program used to obtain feed forward control data was found to require a large amount of computer time to predict the bottoms rate response. For example, the binary runs required about the same amount of time on an IBM 7040 as actual operating time. The ternary runs required about three times longer to calculate the bottoms rate response than actual run time. Thus, in order to be economically usable, a program of the model which requires less machine time must be written. The author believes that a faster program can be written. One possible approach to this problem is to apply a polynomial curve fit to a number of computed bottoms rate response curves. A cross fit of these equations could then be used to compute the bottoms response curve for a given feed upset and initial bottoms composition.

The five runs which were made to determine the applicability of the lumped parameter model for predicting the dynamic behavior of the column were simulated using tray-by-tray data. These simulation data were used with the dynamic model to determine if tray-by-tray data could be substituted for experimental data. A comparison of the results obtained from the two types of data show that the compositions of the two products were the same to two decimal places for most of the runs after twenty-five minutes of run time. The results of the test show that experimental data give the best reproducibility, shown in Figures 17 and 18; however, tray-by-tray data can be used in the absence of experimental data to obtain reasonable results.

Recommendations

Some changes are recommended for future studies of the dynamic behavior of distillation columns. The dynamic model of the column should be changed to include separation parameters for the feed section of the column and reboiler in order to determine if internal stream compositions can be predicted accurately. A new feed forward control program should be written to determine if the amount of computation time can be reduced. Also, some type of programmed control valve should be placed in the bottoms product line for more accurate study of feed forward control.

NOMENCLATURE

Major Symbols

English Letters

Af = count fraction of chromatograph output.

B = bottoms product flow rate, moles/hour.

D = distillate flow rate, moles/hour.

F = feed flow rate, moles/hour.

g_c = gravitational constant, 32.17 ft.-lbm./lbf.-sec.^{2}

h = enthalpy of liquid, BTU/lb.-mole.

H = enthalpy of vapor, BTU/lb.-mole.

J = parameter which describes the degree of separation occurring in a column section, moles/hour-mole fraction.

K = vapor liquid equilibrium coefficient.

L = liquid flow rate, moles/hour.

N = net rate of mass transfer between phases, moles/hour.

P = pressure, lb./in.^{2} absolute.

Q_r = reboiler heat duty, BTU/hour.

S = cross-sectional area of the column, sq. ft.

T = temperature, ^{o}F.

t = time, hour.

u = flow rate, cu. ft./min.

V = vapor flow rate, moles/hour.

Wf = weight fraction

x = liquid composition, mole fraction.

y = vapor composition, mole fraction.

z = height of the column section, ft.

Greek Letters

β = ratio of orifice diameter to pipe diameter.

δ = holdup in a section or on a tray, moles.

ρ = molar density, moles/cu. ft.

Subscripts

b = bottoms.

d = distillate.

f = feed section.

i = component number.

m = subsection number.

n = section number.

Superscripts

$*$ = equilibrium value.

o = initial condition.

Groups

$\frac{d}{dt}$ = total derivative with respect to time.

$\frac{\partial}{\partial t}$ = partial derivative with respect to time.

$\frac{\partial}{\partial z}$ = partial derivative with respect to distance.

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APPENDIX A

CALCULATION OF PHYSICAL DATA

CALCULATION OF PHYSICAL DATA

Vapor-liquid equilibrium data, K, were calculated from the equation

$$K = \frac{P^o}{P} , \tag{6}$$

where the vapor pressures P^o were calculated from the Antoine equation

$$\log P^o = A - \frac{B}{(C + T)} . \tag{7}$$

A, B, and C are experimentally determined coefficients which were found in Rossini (24).

The heats of vaporization, H_{vap}, were calculated using the Clausius-Clapeyron equation

$$\frac{dP}{dT} = \frac{H_{vap}}{T(V^G - V^L)} . \tag{8}$$

The vapor pressure data needed for the above equation were obtained from the Antoine equation, equation (7). Differentiation of equation (7) gives

$$\frac{dP}{dT} = \frac{2.303 \ BP}{(C + T)^2} . \tag{9}$$

Equations (8) and (9) were combined to obtain an expression for the heat of vaporization

$$H_{vap} = PT \left(\frac{2.303 \ BP}{(C + T)^2} \right) (V^G - V^L) . \tag{10}$$

The liquid molar volume, V^L, is much smaller than the gas molar, V^G, volume, and therefore was neglected. The molar gas volume was calculated from the equation

$$V^G = \frac{RT}{P} + B^o \quad (11)$$

B^o is the second virial coefficient.

The vapor enthalpies were taken from Rossini (24). Liquid enthalpies were calculated by subtracting the calculated heat of vaporization from the vapor enthalpy at a given temperature.

APPENDIX B

CHROMATOGRAPH CALIBRATION

CHROMATOGRAPH CALIBRATION

The samples which were collected in evacuated sample bombs were analyzed on an F & M Model 500 Programmed High-Temperature Gas Chromatograph with a Perkin-Elmer Model D2 Electronic Integrator. The Model D2 Integrator operates on the principle of voltage-to-frequency conversion. The output frequency is proportional to the input voltage. The output pulses are fed into a seven-digit decade counter. The counts are stored in the counter until they are read out and printed by a Kienzle Digital Printer. The sum of the output pulses is proportional to the peak area.

The area or count fraction for each component can be calculated easily. However, the count fraction by itself is not a common indication of composition. Compositions are generally reported on the basis of mole or weight fraction. The purpose of calibration was to convert the count fractions obtained from the chromatograph analysis to weight fraction.

The combination of sample size and column temperature which gave the best reproducibility was determined prior to calibrating the chromatograph. This was done by analyzing a large number of duplicate samples for different sample sizes and chromatograph column temperatures. A column temperature of 135 °C and a sample of two micro-liters gave the lowest standard deviation of any of the combinations of column temperature and sample size used.

Two different sets of calibration data were obtained for the two different systems used. Six samples were carefully prepared to calibrate the chromatograph for the binary system, and eleven samples were used for the ternary system. Both sets of samples covered the full range of weight fractions that were obtained in the experimental data. The compositions of these standard samples are shown in Tables VII and VIII.

The samples were refrigerated before analysis to prevent loss by evaporation. Multiple analyses were made for each sample in order to make the results as accurate as possible. The chromatograph results are shown in Tables VII and VIII.

The next step in the calibration procedure was to correlate the weight fractions with the chromatograph output in terms of count fractions. Two different models were used for this correlation. The first model was the linear equation

$$Wf_i = a_i + b_i \, Af_i \; . \tag{12}$$

The second model was the quadratic equation

$$WF_i = a_i + b_i \, Af_i + c_i \, Af_i^2 \; . \tag{13}$$

The results obtained from these two models are presented in Tables IX and X.

A comparison of the correlations indicate that the quadratic model, equation (13), was the best fit for both the binary and the ternary system. These calibration data were used to convert count fractions to weight fractions which were used to calculate the sample compositions in terms of mole fraction.

TABLE VII

CHROMATOGRAPH CALIBRATION DATA FOR THE BENZENE-TOLUENE SYSTEM

Benzene

Sample	Number of Samples Analyzed	Sample Composition, w.f.	Average Count Fraction of Chromatograph Output	Standard Deviation	Maximum Fraction (+)	Count Error (-)
A	10	0.19613	0.20043	0.00065	0.00086	0.00139
B	13	0.40955	0.41585	0.00125	0.00246	0.00165
C	13	0.50815	0.51516	0.00132	0.00241	0.00237
D	13	0.60500	0.61207	0.00099	0.00181	0.00142
E	12	0.70234	0.70948	0.00122	0.00234	0.00204
F	12	0.79862	0.80489	0.00056	0.00058	0.00145

Toluene

Sample	Number of Samples Analyzed	Sample Composition, w.f.	Average Count Fraction of Chromatograph Output	Standard Deviation	Maximum Fraction (+)	Count Error (-)
A	10	0.80387	0.79957	0.00066	0.00139	0.0086
B	13	0.59045	0.58415	0.00125	0.00165	0.00246
C	13	0.49185	0.48484	0.00131	0.00237	0.00241
D	13	0.39500	0.38793	0.00099	0.00142	0.00181
E	12	0.29766	0.29023	0.00122	0.00204	0.00234
F	12	0.20138	0.19511	0.00055	0.00146	0.00058

TABLE VIII

CHROMATOGRAPH CALIBRATION DATA FOR THE BENZENE-TOLUENE-PARA-XYLENE SYSTEM

Benzene

Sample	Number of Samples Analyzed	Sample Composition, w.f.	Average Count Fraction of Chromatograph Output	Standard Deviation	Maximum Fraction (+)	Count Error (-)
A	5	0.40722	0.41960	0.00092	0.00121	0.00127
B	5	0.33173	0.33855	0.00038	0.00043	0.00053
C	5	0.40692	0.41813	0.00136	0.00123	0.00179
D	5	0.27649	0.28399	0.00057	0.00068	0.00083
E	5	0.21942	0.22560	0.00023	0.00023	0.00034
F	5	0.18246	0.18737	0.00031	0.00026	0.00036
G	5	0.59902	0.61034	0.00158	0.00171	0.00198
H	7	0.74251	0.75304	0.00128	0.00170	0.00139
I	6	0.93405	0.94062	0.00495	0.00856	0.00516
J	5	0.15614	0.16037	0.00037	0.00060	0.00034
K	6	0.07821	0.07821	0.00043	0.00067	0.00044

Toluene

Sample	Number of Samples Analyzed	Sample Composition, w.f.	Average Count Fraction of Chromatograph Output	Standard Deviation	Maximum Fraction (+)	Count Error (-)
A	5	0.38434	0.38392	0.00042	0.00053	0.00061
B	5	0.31652	0.31863	0.00069	0.00104	0.00081
C	5	0.19435	0.19467	0.00040	0.00065	0.00035
D	5	0.50540	0.50558	0.00062	0.00086	0.00064
E	5	0.61415	0.61548	0.00040	0.00030	0.00060
F	5	0.43174	0.43419	0.00037	0.00036	0.00057
G	5	0.21467	0.21225	0.00065	0.00061	0.00103
H	7	0.12396	0.12022	0.00067	0.00099	0.00117
I	6	0.02009	0.01724	0.00151	0.00152	0.00273
J	5	0.71387	0.71530	0.00085	0.00127	0.00107
K	6	0.88471	0.89030	0.00172	0.00262	0.00221

TABLE VIII (continued)

Para-Xylene

Sample	Number of Samples Analyzed	Sample Composition, w.f.	Average Count Fraction of Chromatograph Output	Standard Deviation	Maximum Fraction (+)	Count Error (-)
A	5	0.20844	0.19648	0.00121	0.00124	0.00175
B	5	0.35175	0.34282	0.00060	0.00094	0.00051
C	5	0.39874	0.38920	0.00115	0.00134	0.00108
D	5	0.21811	0.21043	0.00100	0.00094	0.00154
E	5	0.16647	0.15893	0.00050	0.00058	0.00047
F	5	0.38579	0.37845	0.00062	0.00088	0.00059
G	5	0.18631	0.11741	0.00108	0.00147	0.00137
H	7	0.13353	0.12647	0.00082	0.00098	0.00130
I	6	0.04585	0.04214	0.00349	0.00363	0.00583
J	5	0.12999	0.12433	0.00074	0.00112	0.00092
K	6	0.03709	0.03141	0.00146	0.00164	0.00217

TABLE IX

REGRESSION COEFFICIENTS AND STANDARD ERROR FOR BINARY SYSTEM

Linear Model

Component	Regression Coefficients a	Regression Coefficients b	Standard Error of Estimate
Benzene	-0.004386	0.9964	0.00095
Toluene	0.008132	0.9962	0.00098

Quadratic Model

Component	Regression Coefficients a	Regression Coefficients b	Regression Coefficients c	Standard Error of Estimate
Benzene	-0.0007340	0.9790	0.01731	0.00023
Toluene	0.004561	1.1013	-0.01723	0.00038

TABLE X

REGRESSION COEFFICIENTS AND STANDARD ERROR FOR TERNARY SYSTEM

Linear Model

Component	Regression Coefficients a	Regression Coefficients b	Standard Error of Estimate
Benzene	-0.0043307	0.9923	0.00343
Toluene	0.005538	0.9916	0.00168
p-Xylene	0.005241	1.01291	0.00210

Quadratic Model

Component	Regression Coefficients a	Regression Coefficients b	Regression Coefficients c	Standard Error of Estimate
Benzene	0.003480	0.9480	0.04409	0.00117
Toluene	0.003083	0.9911	0.000582	0.00179
p-Xylene	0.003066	1.0413	-0.06490	0.00199

APPENDIX C

FLOW METER CALIBRATIONS

FLOW METER CALIBRATIONS

The meters used to measure the flow rates of the feed, distillate and bottoms products, and vapor from and liquid to the reboiler were calibrated prior to taking experimental data. Rotameters were used to measure the flow rates of the feed and distillate and bottoms products. The remaining two stream flow rates were measured with orifice meters.

The bottoms rotameter was calibrated by collecting and weighing three samples at recorded rotameter readings and time periods. The actual flow rate was calculated and a plot of the actual flow rate versus the rotameter reading in gallons per minute was constructed. The calibration data are presented in Figure 19.

The feed rotameter was calibrated using the bottoms rotameter. Liquid was pumped through both rotameters and the flow rates of the two rotameters were recorded. This procedure was repeated for five different flow rates. The actual flow rates were determined using the bottoms rotameter calibration. The calibration data are presented in Figure 20.

The distillate rotameter was calibrated in the same manner as the bottoms rotameter. Three data points were obtained. The results of this calibration are presented in Figure 21.

The orifice in the liquid line to the reboiler was calibrated, in place, using the bottoms rotameter. A blind orifice was placed in the vapor return line in order to prevent error caused by liquid entering the reboiler through this line. The reboiler was filled completely with

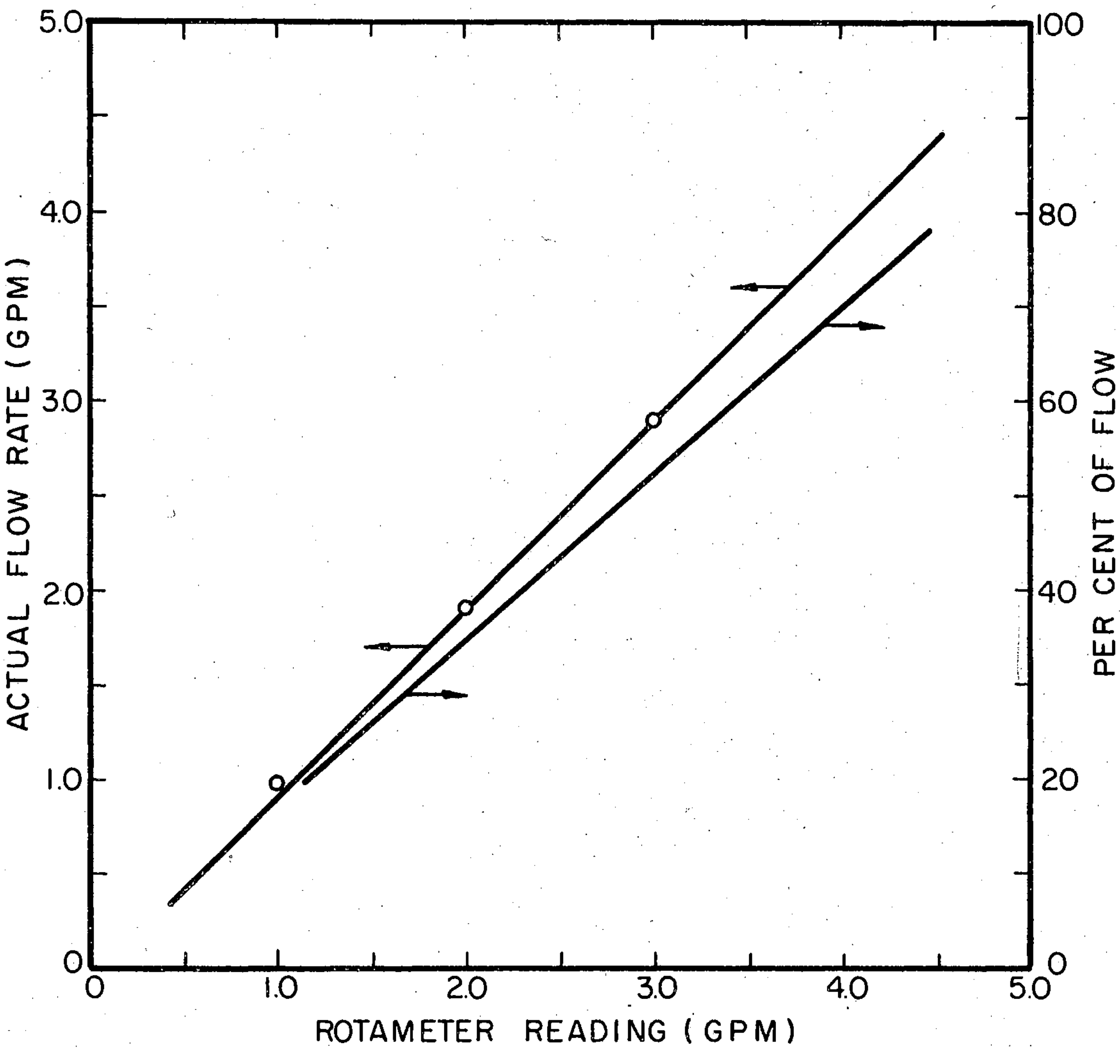

Figure 19. Bottoms Rotameter Calibration.

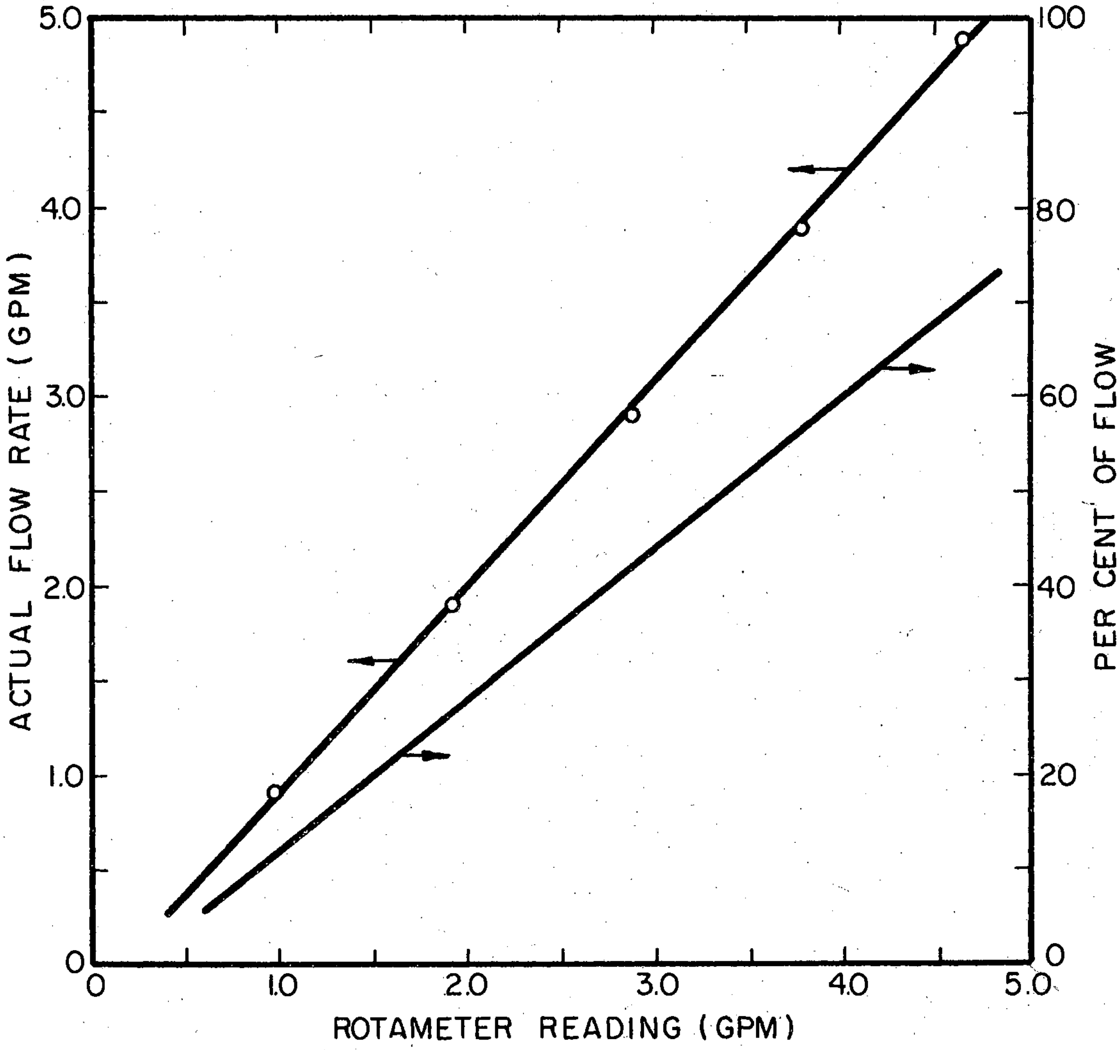

Figure 20. Feed Rotameter Calibration.

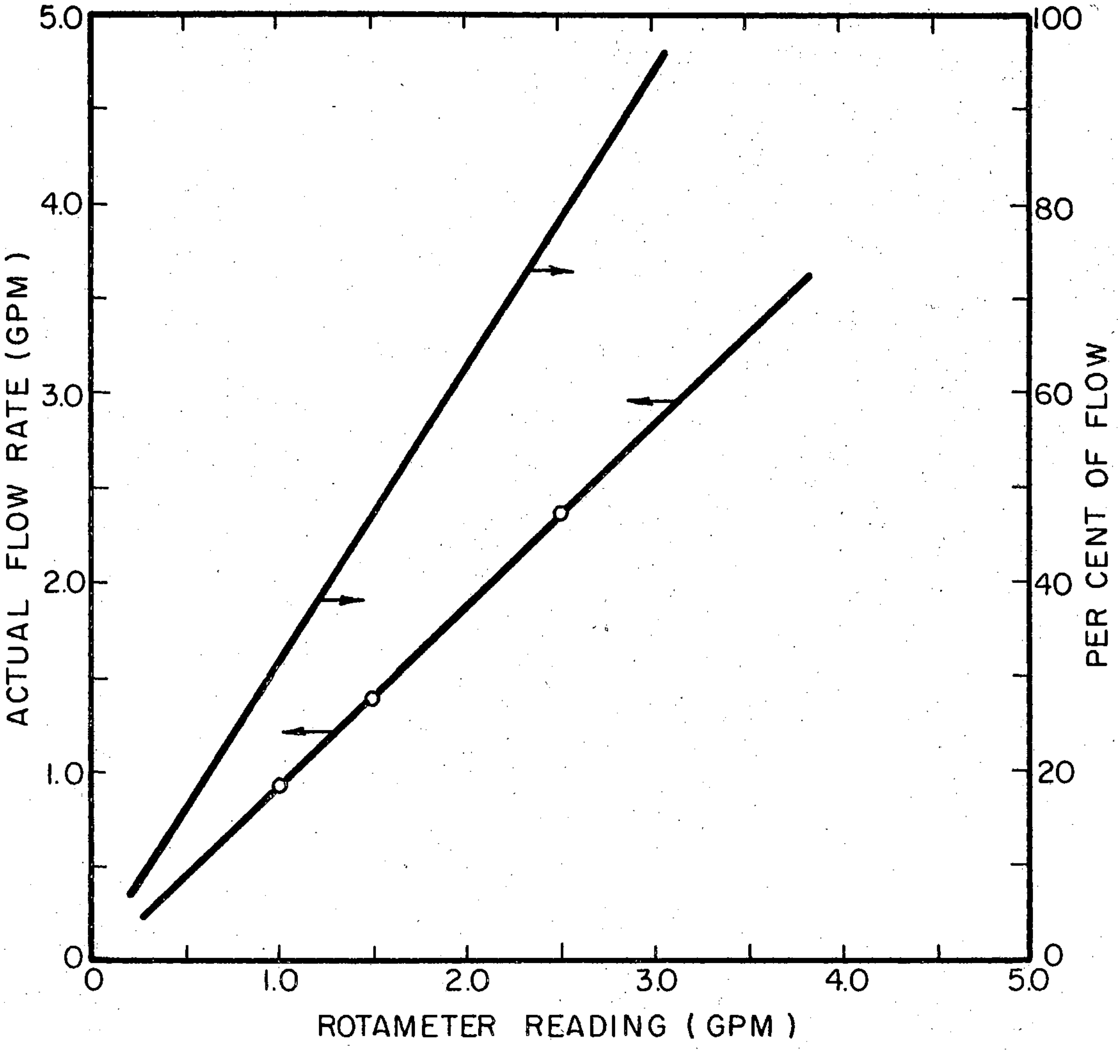

Figure 21. Distillate Rotameter Calibration.

liquid. The feed pump was then started at a set flow rate and liquid was recycled through the column. The rate of the liquid leaving the reboiler through the bottoms product line was recorded using the bottoms rotameter. A set flow rate was maintained until the liquid recorder remained constant for about ten minutes. Readings were then taken from the recorder chart and the rotameter. A plot of chart scale versus flow rate in gallons per minute was prepared.

In order to correct for changes in flow rate caused by temperature difference a density correction factor was employed. This correction was derived from the orifice equation

$$U = C_o \sqrt{\frac{2g_c\ (P_1 - P_2)}{\rho(1 - \beta^4)}} . \tag{14}$$

The orifice coefficient, C_o, can be assumed to be constant for small changes in flow rate. Changes in liquid density caused by pressure can be neglected. Therefore, for a given pressure drop, the density correction factor can be derived by dividing equation (14) evaluated at temperature T_2 by the same equation evaluated at temperature T_1. The following equation is then obtained

$$U\Big|_{T_2} = U\Big|_{T_1} \sqrt{\frac{\rho\big|_{T_1}}{\rho\big|_{T_2}}} . \tag{15}$$

The results of this orifice calibration are presented in Figure 22.

The orifice in the vapor return line was calibrated in place using the liquid orifice and the bottoms rotameter. Five points were at total reflux, and four data points were taken while bottoms product was being removed from the column. The total number of moles of vapor leaving the reboiler was calculated by material balance, and the flow rate in cubic feet per minute was calculated using the ideal gas law. The results of

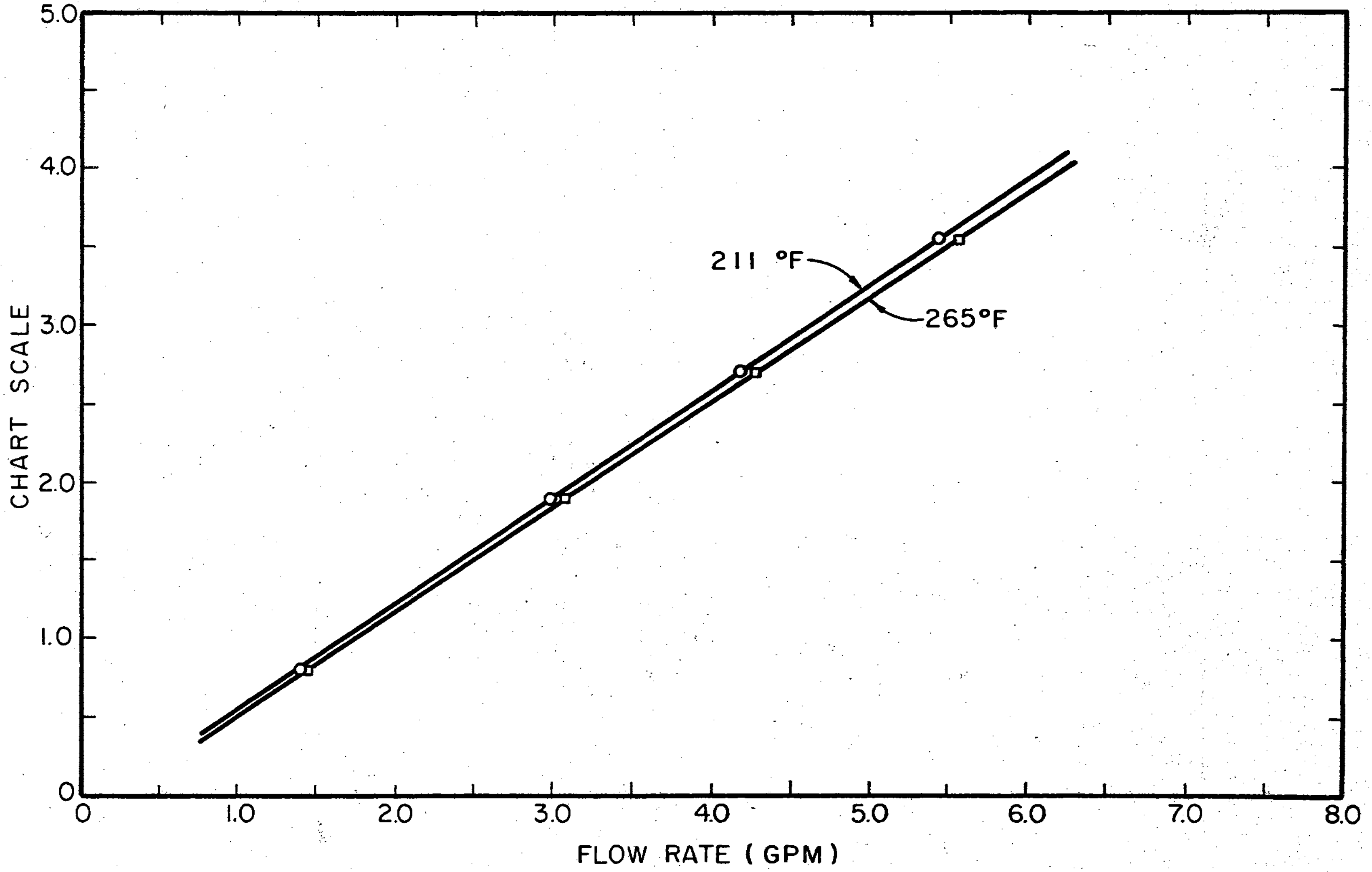

Figure 22. Liquid Orifice Calibration.

this calibration are given in Figure 23.

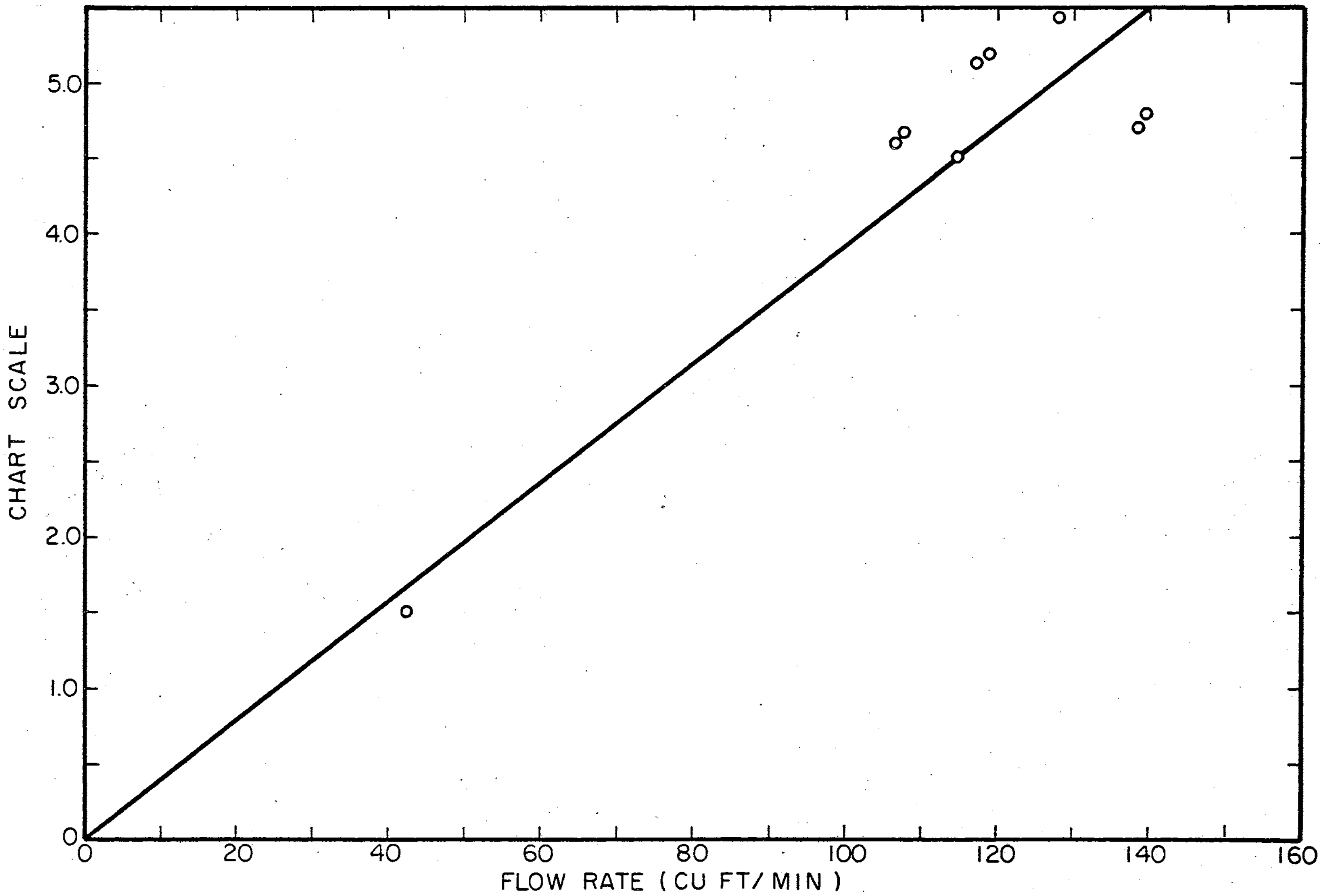

Figure 23. Vapor Orifice Calibration.

APPENDIX D

COLUMN SIMULATION

COLUMN SIMULATION

Tray-by-tray data were used to simulate each feed upset run, in order to determine if this type of data could be used to evaluate the separation parameter in the dynamic model. Also, tray-by-tray simulation data were used to obtain data for the feed forward control program. The O. S. U. Tray-by-Tray Program (6) was used to obtain tray-by-tray data for these simulations.

The tray-by-tray data were obtained for integral numbers of theoretical trays. The experimental column used did not give a separation equivalent to an integral number of ideal trays. Therefore, the tray-by-tray data for a fractional number of trays was obtained graphically.

The procedure used to simulate the column was to obtain experimentally the necessary data, such as feed and distillate rates, and feed temperature and composition, for the O. S. U. Tray-by-Tray Program. Solutions were then obtained for four, five, and six theoretical trays. A plot of composition of the various streams versus the number of trays was constructed using the rigorous data. The fractional number of ideal trays required to produce the experimental separation was found by the point where the distillate composition intersected the distillate line. This number of trays was checked with the bottoms composition. The composition of the internal streams required for the dynamic and feed forward control program was then obtained from the point where a plot of the respective compositions crossed the predetermined number of trays. The flow rates of these respective streams were obtained in a similar manner.

The graphical simulation of run 2-3 is presented in Figures 24, 25, and 26 in order to illustrate this procedure. Figure 24 illustrates the method whereby the fractional number of ideal trays required to give the desired separation was determined. This figure shows that 4.27 ideal stages were required. Figure 25 illustrates the method used to obtain the compositions of internal streams, and Figure 26 illustrates the method used to obtain the flow rates of these internal streams.

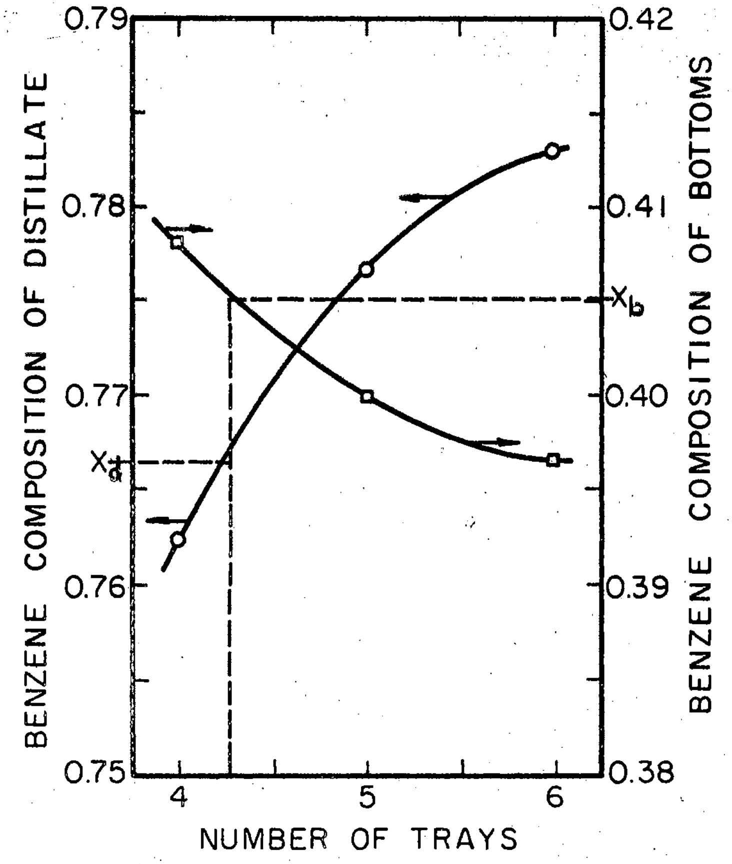

Figure 24. Determination of Number of Ideal Stages Required to Simulate Run 2-3.

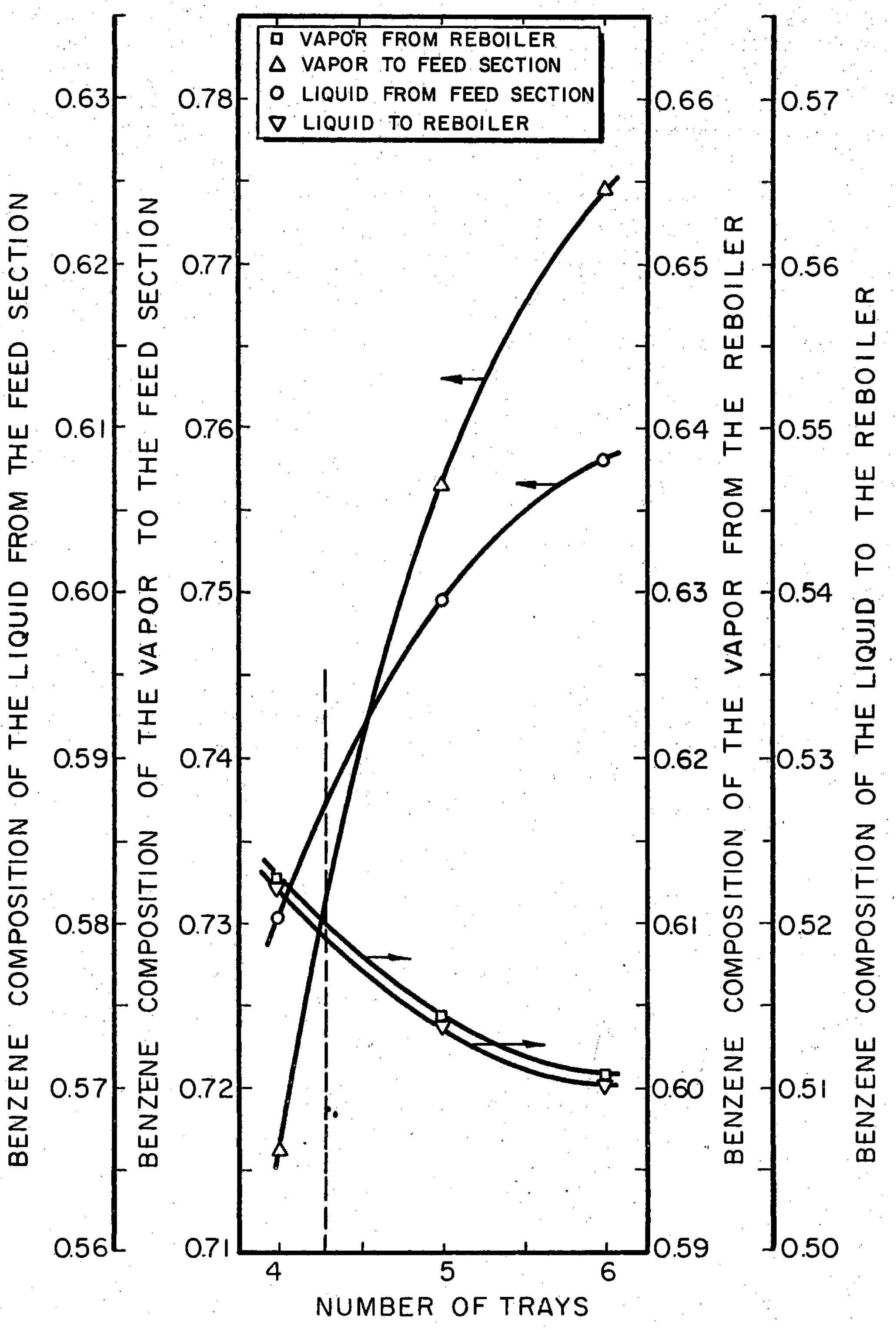

Figure 25. Determination of Composition of Internal Streams for Simulation of Run 2-3.

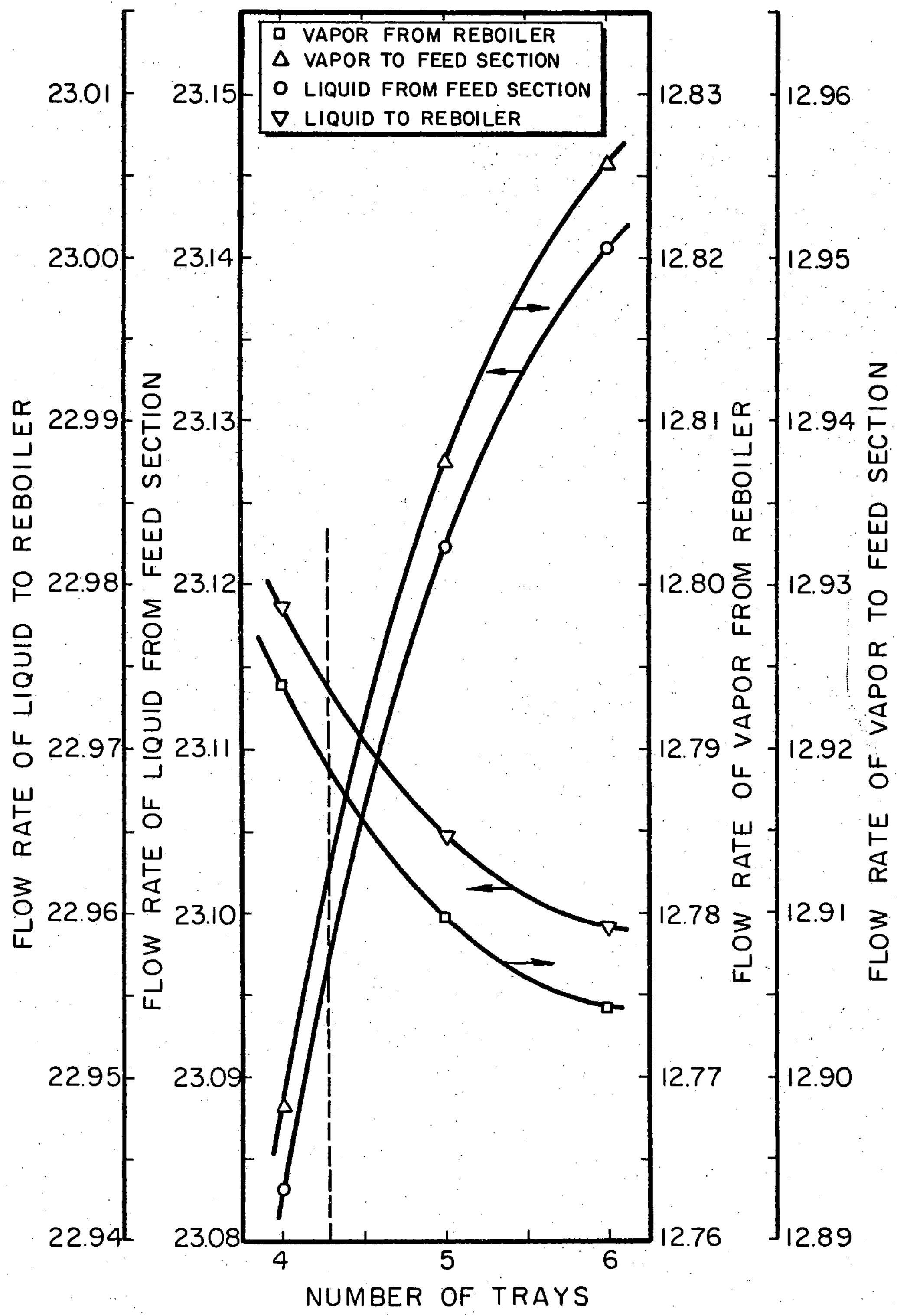

Figure 26. Determination of Flow Rates of Internal Streams for Simulation of Run 2-3.

APPENDIX E

DEVELOPMENT OF THE LUMPED PARAMETER MODEL

DEVELOPMENT OF THE LUMPED PARAMETER MODEL (14, 15, 16, 17)

The development of the lumped parameter model is based on the section concept whereby a distillation column is divided into sections in which there can be any number of trays. According to this concept, a section of a distillation column is that part of the column which lies between the points at which either feed streams enter or product streams leave the column.

A material balance around the n^{th} section for the i^{th} component can be written as

$$\text{input} - \text{output} = \text{accumulation} \tag{16}$$

where

$$\text{input} = V_{n+1}\, y_{n+1,i} \tag{17}$$

$$\text{output} = V_{n+1}\, y_{n+1,i} + \frac{\partial (V_n\, y_{n,i})}{\partial z}\, dz + N^V_{n,i} \tag{18}$$

$$\text{accumulation} = \frac{\partial (\delta^V_n\, y_{n,i})}{\partial t}\,. \tag{19}$$

The output term is the sum of all material that leaves the vapor phase either by flow or mass transfer. The rate at which a component flows from the section is equal to the flow rate in $(V_{n+1}\, y_{n+1,i})$ plus the increase or decrease in the stream flow rate that occurs in the section $\frac{\partial (V_n\, y_{n,i})}{\partial z}\, dz$. The mass transfer from the vapor phase is represented by the term $N^V_{n,i}$.

According to Reynolds (18), the rate at which mass can be transferred from the vapor phase to the liquid phase can be expressed by the equation

$$N^{V}_{n,i} = - J_{n,i} \, (y^{*} - y)_{n,i} \, . \tag{20}$$

The term $J_{n,i}$ is the parameter which describes the degree of separation occurring in a section and $(y^{*} - y)_{n,i}$ is the driving force for mass transfer in the section.

Using equations (17, 18, 19, 20) the mass balance can be written as

$$\frac{\partial(\delta^{V}_{n} \, y_{n,i})}{\partial t} = - \frac{\partial(V_{n} \, y_{n,i})}{\partial z} \, dz + J_{n,i} \, (y^{*} - y)_{n,i} \, . \tag{21}$$

If the change in height Δz is small, the partial derivative, $\frac{\partial(V_{n} \, y_{n,i})}{\partial z}$, can be replaced by the approximation

$$\frac{\partial(V_{n} \, y_{n,i})}{\partial z} = \frac{(V_{n} \, y_{n,i} - V_{n+1} \, y_{n+1,i})}{\Delta z} \, . \tag{22}$$

The term $\delta^{V}_{n} \, y_{n,i}$ is now a function of time only; therefore, the partial derivative with respect to time can be replaced by the total derivative. Assuming constant molal holdup, equation (22) can be used in equation (21) to obtain

$$\delta^{V}_{n} \frac{dy_{n,i}}{dt} = - (V_{n} \, y_{n,i} - V_{n+1} \, y_{n+1,i}) + J_{n,i} \, (y^{*} - y)_{n,i} \, . \tag{23}$$

A similar equation for the liquid stream can be written as

$$\delta^{V}_{n} \frac{dx_{n,i}}{dt} = - (L_{n} \, x_{n,i} - L_{n-1} \, x_{n-1,i}) - N^{L}_{n,i} \, . \tag{24}$$

The derivation of equation (24) is the same as the derivation of equation (23) except that the liquid streams are used.

The term $N^{L}_{n,i}$ can be shown to be equal to $- N^{V}_{n,i}$ by equating equations (23) and (24) at steady-state conditions; thus equation (24) can be written as

$$S_n^L \frac{dx_{n,i}}{dt} = -(L_n x_{n,i} - L_{n-1} x_{n-1,i}) - J_{n,i} (y^* - y)_{n,i} \,. \qquad (25)$$

Equations (23) and (25) are valid for any systems which meet the assumptions that were made in deriving them. However, some method must be developed for determining the parameter $J_{n,i}$. In order to evaluate this parameter, Osborne (14, 15) and Osborne, et al. (16, 17) made the assumption that the separation parameter remained constant for small changes in column conditions. In addition, since the function $J_{n,i} (y^* - y)_{n,i}$, which represents the net rate of mass transfer between phases, is based on passing streams which cannot be measured, some method of approximating the driving force for mass transfer had to be developed. Osborne solved this problem by developing the following expression

$$(y^* - y)_{n,i} = (Kx)_{n-1,i} - y_{n+1,i} \,. \qquad (26)$$

In developing equation (26) Osborne envisioned a section of a column as being subdivided into an infinite number of subsections. The driving force for one of these infinitestimal subsections can be represented by the equation

$$(y^* - y)_{m,i} = (Kx)_{m-1,i} - y_{m+1,i} \,. \qquad (27)$$

Equation (26) is obtained by summing equation (27) over all these subsections.

Expressions for $J_{n,i}$ can now be written by substituting equation (26) into equations (23) and (25) at steady state. These expressions are respectively

$$J_{n,i} = \frac{(V_n y_{n,i} - V_{n+1} y_{n+1,i})}{(Kx)_{n-1,i} - y_{n+1,i}} \qquad (28)$$

and

$$J_{n,i} = - \frac{(L_n x_{n,i} - L_{n-1} x_{n-1,i})}{(Kx)_{n-1,i} - y_{n+1,i}} . \qquad (29)$$

$J_{n,i}$ can now be determined using equations (28) or (29) and steady state data.

APPENDIX F

DEVELOPMENT OF THE FEED FORWARD CONTROL MODEL

DEVELOPMENT OF THE FEED FORWARD CONTROL MODEL (14, 15, 16, 17)

The development of the feed forward control model is an extension of the dynamic model developed in Appendix E. Heat and material balances are solved simultaneously after each time interval, Δt, in order to calculate the time rate of change of the distillate and bottoms product rates required to maintain a constant composition of the i^{th} component in one of these products.

The feed forward control model that is discussed here is for the stripping column that was used to obtain experimental data. Such a column is shown in Figure 27.

The holdup in the feed section is assumed to be negligible. Heat and material balances for the feed section can be written as

$$V_3 + F = D + L_2 \qquad (30)$$

$$V_3\, y_{3,i} + F\, x_{f,i} = D\, y_{d,i} + L_2\, x_{2,i} \quad (i = 1, 2, \ldots, N) \qquad (31)$$

$$V_3\, H_3 + F\, h_f = D\, H_d + L_2\, h_2 \;. \qquad (32)$$

Since holdup in the feed section has been assumed to be negligible, equations (30), (31), and (32) are valid at all times. If the feed section is assumed to be an ideal stage, the distillate is in equilibrium with the liquid leaving the feed section. Thus, the distillate and the liquid leaving the feed section are related according to the equation

$$y_{d,i} = K_{2,i}\, x_{2,i} \quad (i = 1, 2, \ldots, N) \;. \qquad (33)$$

Assuming that the column is at thermal steady-state at all times,

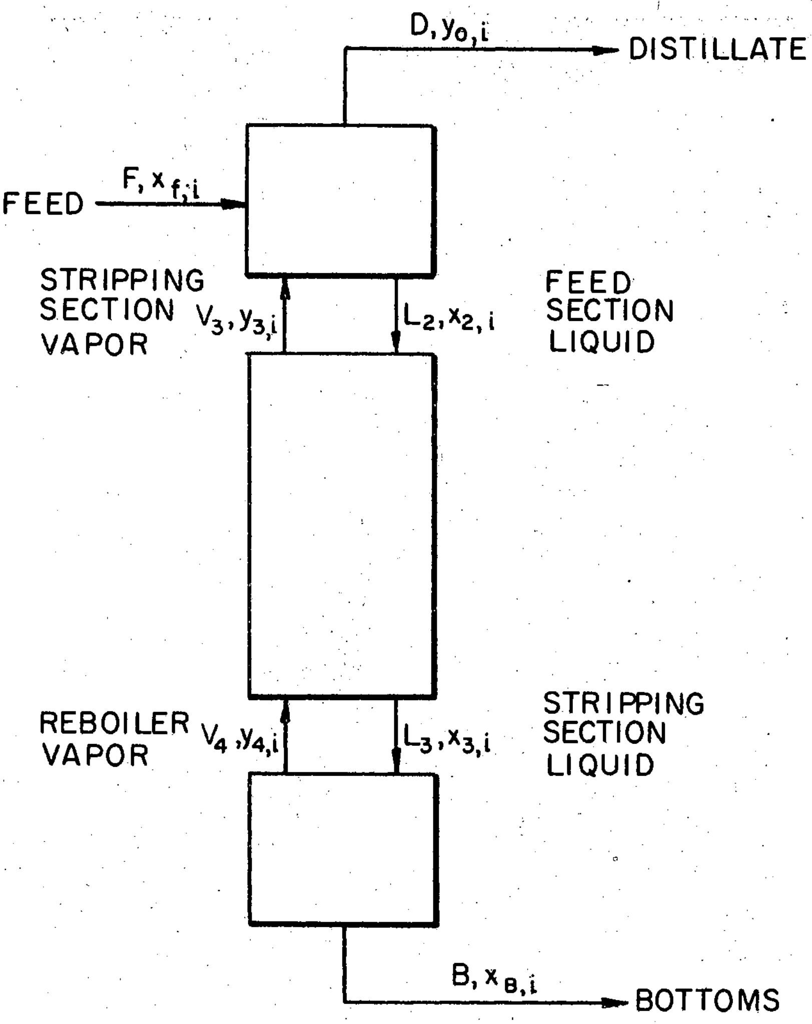

Figure 27. Stripping Column.

the following equations can be written for the stripping section at any time

$$L_2 + V_4 = L_3 + V_3 \tag{34}$$

$$L_2 h_2 + V_4 H_4 = L_3 h_3 + V_3 H_4 . \tag{35}$$

Since the stripping section has appreciable holdup, it will not necessarily be at steady-state with respect to component flow rates. Consequently, a dynamic model must be used to determine the compositions of the streams leaving the stripping section. Accordingly, the compositions can be expressed as

$$x_{3,i} = \int_o^t \left(\frac{dx_{3,i}}{dt}\right)dt + x_{3,i}\Big|_{t=o} \quad (i = 1, 2, \ldots, N) \tag{36}$$

$$y_{3,i} = \int_o^t \left(\frac{dy_{3,i}}{dt}\right)dt + y_{3,i}\Big|_{t=o} \quad (i = 1, 2, \ldots, N) . \tag{37}$$

The first order lumped parameter model is used to evaluate the derivative of the liquid and vapor compositions as

$$\frac{dx_{3,i}}{dt} = \frac{1}{\delta L}\left[L_2\, x_{2,i} - L_3\, x_{3,i} - J_i\, (K_{2,i}\, x_{2,i} - y_{4,i})\right]$$
$$(i = 1, 2, \ldots, N) \tag{38}$$

$$\frac{dy_{3,i}}{dt} = \frac{1}{\delta V}\left[V_4\, y_{4,i} - V_3\, y_{3,i} + J_i\, (K_{2,i}\, x_{2,i} - y_{4,i})\right]$$
$$(i = 1, 2, \ldots, N) . \tag{39}$$

The first step in the calculational procedure is to determine the values of the separation parameter J_i from the steady state operating conditions. Once the separation parameters have been determined, the feed composition is changed to its new value in a step manner. The column is then restored to heat balance. This operation is permissible because the column is always in heat balance.

The new values of $x_{3,i}$ and $y_{3,i}$ are then used to restore the column to heat balance. The vapor boilup rate is changed so as to bring the bottoms composition back to the desired value.

Finally, using the new vapor boilup rate, the column is again restored to heat balance. This procedure of calculating values of $x_{3,i}$ and $y_{3,i}$, heat balancing, adjusting the vapor rate, and heat balancing again is repeated until the column reaches steady state.

The heat and material balance equations used for the reboiler are

$$L_3 = B + V_4 \tag{40}$$

$$x_{b,i} = \int_o^t \left(\frac{dx_{b,i}}{dt}\right)dt + x_{b,i} \Big|_{t=o} \qquad (i = 1, 2, \ldots, N) \tag{41}$$

$$\frac{dx_{b,i}}{dt} = \frac{1}{\delta^{B}} \left[L_3\, x_{3,i} - (B\, x_{b,i} - V_4\, y_{4,i})\right]$$

$$(i = 1, 2, \ldots, N) \tag{42}$$

$$Q_r + L_3\, h_3 = B\, h_b + V_4\, H_4 \tag{43}$$

APPENDIX G

EXPERIMENTAL AND CALCULATED DATA

TABLE XI

EXPERIMENTAL DATA FOR RUN 2-1

Benzene-Toluene

Parameter	Initial Steady State	Final Steady State
Feed Rate, moles/hr.	15.366	15.491
Distillate Rate, moles/hr.	6.076	6.297
Bottoms Rate, moles/hr.	9.290	9.194
Liquid to Reboiler Rate, moles/hr.	34.613	35.442
Vapor from Reboiler Rate, moles/hr.	26.155	27.541

Stream Compositions*

Time, min.	Feed	Distillate	Bottoms	Liquid to Reboiler	Vapor from Reboiler
-10.0	0.5160	0.7567	0.3859	0.4584	0.5305
5.0	0.5123	0.7472	0.3828	0.4650	0.5374
0.0	0.5077	0.7579	0.3770	0.4588	0.5297
1.5	0.5101	0.7543	0.3734	0.4511	0.5223
3.0	0.7061	0.8440	0.3715	0.4515	0.5257
4.5	0.7214	0.8723	0.3731	0.4536	0.5220
6.0	0.7211	0.8686	0.3723	0.4606	0.5177
7.5	0.7226	0.8761	0.3714	0.4738	0.5196
9.5	0.7231	0.8772	0.3745	0.4897	0.5275
11.5	-	0.8786	0.3786	0.5035	0.5390
13.5	-	0.8816	0.3863	0.5153	0.5386
16.5	-	0.8791	0.3946	0.5299	0.5526
21.5	-	0.8900	0.4131	0.5463	0.5619
26.5	-	0.8883	0.4302	0.5627	0.5613
31.5	0.7231	0.8872	0.4475	0.5737	0.6085
36.5	-	0.8834	0.4618	0.5824	0.6209
44.6	-	0.8929	0.4782	0.5900	0.6432

*Compositions are given as mole fraction benzene.

TABLE XII

EXPERIMENTAL DATA FOR RUN 2-2

Benzene-Toluene

Parameter	Initial Steady State	Final Steady State
Feed Rate, moles/hr.	16.139	16.271
Distillate Rate, moles/hr.	7.748	6.982
Bottoms Rate, moles/hr.	9.862	9.469
Liquid to Reboiler Rate, moles/hr.	35.411	35.361
Vapor from Reboiler Rate, moles/hr.	25.236	25.346

Stream Compositions*

Time, min.	Feed	Distillate	Bottoms	Liquid to Reboiler	Vapor from Reboiler
-10.0	0.5769	0.7943	0.4259	0.5046	0.5785
- 5.0	0.5849	0.7983	0.4229	0.5075	0.5858
0.0	0.5838	0.8021	0.4224	0.5047	0.5821
1.5	0.6194	0.8154	0.4162	0.5040	0.5853
3.0	0.6240	0.8294	0.4200	0.5044	0.5878
4.5	0.6241	0.8296	0.4197	0.5072	0.5812
6.0	0.6244	0.8316	0.4180	0.5095	0.5860
7.5	0.6265	0.8319	0.4177	0.5080	0.5848
9.0	0.6282	0.8300	0.4185	0.5165	0.5822
11.0	-	0.8310	0.4211	0.5184	0.5870
13.0	-	0.8312	0.4213	0.5216	0.5826
18.5	-	0.8378	0.4266	-	0.5879
23.0	-	0.8391	0.4300	0.5322	0.5960
28.0	0.6199	0.8326	0.4358	0.5362	0.6004
33.0	-	0.8340	0.4438	0.5407	0.6042
38.0	-	0.8343	0.4464	0.5404	0.6103
43.0	-	0.8403	0.4494	0.5463	0.6165
54.0	-	0.8342	0.4560	0.5533	0.6188
61.0	-	0.8283	0.4596	0.5527	0.6096

*Compositions are given as mole fraction benzene.

TABLE XIII

EXPERIMENTAL DATA FOR RUN 2-3

Benzene-Toluene

Parameter	Initial Steady State	Final Steady State
Feed Rate, moles/hr.	16.003	16.364
Distillate Rate, moles/hr.	5.655	6.347
Bottoms Rate, moles/hr.	9.000	9.123
Liquid to Reboiler Rate, moles/hr.	36.789	37.102
Vapor from Reboiler Rate, moles/hr.	24.640	25.479
Vapor to Top Tray, m.f.*	0.7587	-

Stream Compositions*

Time, min.	Feed	Distillate	Bottoms	Liquid to Reboiler	Vapor from Reboiler
-10.0	0.5370	0.7657	0.4091	0.4801	0.5606
- 5.0	0.5368	0.7664	0.4051	0.4794	0.5630
0.0	0.5380	0.7700	0.4045	0.4802	0.5603
1.5	0.6501	0.8087	0.4041	0.4797	0.5582
3.0	0.6588	0.8378	0.4056	0.4793	0.5576
4.6	-	0.8450	0.4037	0.4866	0.5614
6.0	-	0.8450	0.4043	0.4926	0.5605
9.0	-	0.8444	0.4097	0.5177	0.5670
12.0	0.6562	0.8482	0.4148	0.5156	0.5771
15.0	-	0.8481	0.4240	0.5266	0.5888
18.0	-	0.8478	0.4319	0.5381	0.5934
22.0	0.6605	0.8466	0.4418	0.5470	0.6015
26.0	-	-	0.4526	0.5527	0.6120
30.0	-	0.8521	0.4608	0.5604	0.6181
35.3	-	0.8508	0.4705	-	0.6299
40.0	0.6592	0.8487	0.4944	0.5727	0.6326
45.0	-	-	0.4739	0.5780	0.6431
50.0	-	0.8500	0.4816	0.5811	0.6383

*Compositions are given as mole fraction benzene.

TABLE XIV

EXPERIMENTAL DATA FOR RUN 2-4

Benzene-Toluene

Parameter	Initial Steady State	Final Steady State
Feed Rate, moles/hr.	15.543	16.042
Distillate Rate, moles/hr.	5.900	6.680
Bottoms Rate, moles/hr.	8.884	8.543
Liquid to Reboiler Rate, moles/hr.	33.209	22.698
Vapor from Reboiler Rate, moles/hr.	28.368	31.882
Vapr to Top Tray, m.f.*	0.5908	-

Stream Compositions*

Time, min.	Feed	Distillate	Bottoms	Liquid to Reboiler	Vapor from Reboiler
-10.0	0.5424	-	0.3770	0.4596	-
- 5.0	0.5419	0.7334	0.3762	0.4601	0.5154
0.0	0.5379	0.7420	0.3775	0.4595	0.5218
1.5	0.6517	0.7589	0.3775	0.4604	0.5225
3.0	0.6710	0.7829	0.3781	0.4616	0.5210
4.5	0.6739	0.7993	0.3784	0.4643	0.5298
6.0	0.6744	0.8081	0.3794	0.4689	0.5217
8.0	-	0.8162	0.3820	0.4797	0.5250
10.1	-	0.8125	0.3848	0.4903	0.5276
12.0	-	0.8111	0.3883	0.4981	0.5440
15.0	-	0.8206	0.3969	0.5088	0.5531
20.0	0.6741	0.8103	0.4137	0.5252	0.5676
25.0	-	0.8173	0.4260	0.5376	0.5877
30.0	-	0.8329	0.4348	0.5445	0.5891
35.0	-	0.8413	0.4407	0.5529	0.6024
40.0	0.6729	0.8481	0.4505	0.5629	0.6079
48.0	-	0.8387	0.4661	0.5740	0.6239

*Compositions are given as mole fraction benzene.

TABLE XV

EXPERIMENTAL DATA FOR RUN 3-1

Benzene-Toluene-p-Xylene

Parameter	Initial Steady State	Final Steady State
Feed Rate, moles/hr.	14.048	14.020
Distillate Rate, moles/hr.	7.343	6.922
Bottoms Rate, moles/hr.	6.705	7.098
Liquid to Reboiler Rate, moles/hr.	27.764	27.702
Vapor from Reboiler Rate, moles/hr.	21.059	20.604
Vapor to Top Tray, m.f.		
Benzene	0.6756	-
Toluene	0.2992	-

Stream Compositions, m.f. Benzene

Time, min.	Feed	Distillate	Bottoms	Liquid to Reboiler	Vapor from Reboiler
- 5.0	0.4081	0.6967	0.1072	0.1661	0.2111
0.0	0.4070	0.6920	0.1072	0.1638	0.2071
1.5	0.3736	0.6711	0.1060	0.1632	0.2003
3.0	0.3702	0.6626	0.2053	0.1636	0.2034
4.5	0.3693	0.6620	0.1050	0.1620	0.2048
6.0	0.3691	0.6656	0.1046	0.1613	0.2019
8.0	0.3699	0.6618	0.1035	0.1583	0.2014
10.0	-	0.6651	0.1032	0.1572	0.2039
12.0	-	0.6606	0.1018	0.1546	0.1956
15.0	-	0.6591	0.1016	0.1557	0.1958
20.0	-	0.6597	0.1000	0.1544	0.1949
25.0	0.3691	0.6624	0.0998	0.1530	0.2001
30.0	-	0.6657	0.0978	0.1505	0.1916
35.0	-	0.6561	0.0958	0.1453	0.1864
40.0	-	0.6583	0.0968	0.1443	0.1828
45.0	0.3687	0.6584	0.0930	0.1442	0.1823
50.0	-	-	0.0931	0.1473	0.1806
60.0	-	0.6553	0.0936	0.1504	0.1831
65.0	-	0.6622	0.0943	0.1491	0.1859

TABLE XV (continued)

Stream Compositions, m.f. Toluene

Time, min.	Feed	Distillate	Bottoms	Liquid to Reboiler	Vapor from Reboiler
- 5.0	0.4915	0.2822	0.7104	0.6959	0.6905
0.0	0.4920	0.2866	0.7112	0.6988	0.6919
1.5	0.5202	0.3052	0.7118	0.6994	0.6965
3.0	0.5230	0.3137	0.7114	0.6994	0.6950
4.5	0.5231	0.3142	0.7112	0.6995	0.6958
6.0	0.5235	0.3122	0.7128	0.7010	0.6964
8.0	0.5226	0.3144	0.7132	0.7041	0.6976
10.0	-	0.3126	0.7142	0.7057	0.6982
12.0	-	0.3143	0.7139	0.7059	0.7019
15.0	-	0.3169	0.7141	0.7055	0.7022
20.0	-	0.3163	0.7132	0.7066	0.7040
25.0	0.5233	0.3138	0.7164	0.7083	0.7007
30.0	-	0.3113	0.7169	0.7121	0.7074
35.0	-	0.3190	0.7186	0.7113	0.7110
40.0	-	0.3169	0.7172	0.7160	0.7140
45.0	0.5238	0.3172	0.7207	0.7157	0.7149
50.0	-	-	0.7201	0.7133	0.7158
60.0	-	0.3198	0.7196	0.7106	0.7132
65.0	-	0.3128	0.7200	0.7131	0.7113

TABLE XVI

EXPERIMENTAL DATA FOR RUN FFC 2-2

Benzene-Toluene

Parameter	Initial Steady State	Final Steady State
Feed Rate, moles/hr.	14.893	14.893
Distillate Rate, moles/hr.	6.239	8.57
Bottoms Rate, moles/hr.	8.655	6.43
Feed Composition, m.f.*	0.4279	0.4779
Liquid to Reboiler Composition, m.f.*	0.3394	0.3420
Vapor from Reboiler Composition, m.f.*	0.4101	0.4020

Time, min.	Bottoms Rate, moles/hr.	Stream Compositions* Distillate	 Bottoms
- 5.0	8.655	0.6502	0.2674
0.0	8.655	0.6478	0.2659
1.0	8.20	-	0.2649
3.0	7.55	-	0.2658
5.0	7.12	-	0.2668
7.0	6.87	-	0.2659
9.0	6.72	0.6575	0.2664
11.0	6.61	-	0.2671
14.0	6.51	-	0.2685
18.0	6.46	0.6288	0.2736
22.0	6.45	-	0.2757
26.0	6.43	-	0.2729
30.0	6.43	0.6680	0.2694

*Compositions are given as mole fraction benzene.

TABLE XVII

EXPERIMENTAL DATA FOR RUN FFC 2-3

Benzene-Toluene

Parameter	Initial Steady State	Final Steady State
Feed Rate, moles/hr.	14.779	14.779
Distillate Rate, moles/hr.	6.202	1.59
Bottoms Rate, moles hr.	8.578	12.80
Feed Composition, m.f.*	0.5413	0.3910
Liquid to Reboiler Composition, m.f.*	0.4515	0.3888
Vapor from Reboiler Composition, m.f.*	0.5249	0.4902

Time, min.	Bottoms Rate, moles/hr.	Stream Compositions* Distillate	Bottoms
- 5.0	8.578	0.7599	0.3740
0.0	8.578	0.7563	0.3764
1.0	9.25	-	0.3764
3.0	10.40	-	0.3743
5.0	11.23	-	0.3755
7.0	11.78	-	0.3716
9.0	12.14	0.6398	0.3684
11.0	12.37	-	0.3627
13.0	12.52	-	0.3507
15.0	12.62	-	0.3562
17.0	12.68	-	0.3520
20.0	12.75	0.6389	0.3456
24.0	12.77	-	0.3382
28.0	12.80	-	0.3308
31.5	12.80	0.6444	0.3241

*Compositions are given as mole fraction benzene.

TABLE XVIII

EXPERIMENTAL DATA FOR RUN FFC 2-4

Benzene-Toluene

Parameter	Initial Steady State	Final Steady State
Feed Rate, moles/hr.	14.519	14.519
Distillate Rate, moles/hr.	6.132	7.46
Bottoms Rate, moles/hr.	8.387	7.10
Feed Composition, m.f.*	0.4536	0.4661
Liquid to Reboiler Composition, m.f.*	0.3455	0.3457
Vapor from Reboiler Composition, m.f.*	0.4125	0.3981

Time, min.	Bottoms Rate, moles/hr.	Stream Compositions* Distillate	Stream Compositions* Bottoms
- 5.0	8.387	0.6586	0.2734
0.0	8.381	0.6607	0.2726
1.0	8.13	-	0.2742
3.0	7.76	-	0.2730
5.0	5.53	-	0.2722
7.0	7.37	-	0.2727
9.0	7.27	-	0.2715
11.0	7.21	0.6690	0.2716
13.0	7.16	-	0.2697
16.0	7.14	-	0.2716
20.0	7.12	0.6990	0.2705
24.0	7.11	-	0.2725
28.0	7.10	-	0.2726
32.1	7.10	0.6674	0.2712

*Compositions are given as mole fraction benzene.

TABLE XIX

EXPERIMENTAL DATA FOR RUN FFC 2-5

Benzene-Toluene

Parameter	Initial Steady State	Final Steady State
Feed Rate, moles/hr.	14.591	14.591
Distillate Rate, moles/hr.	6.203	4.92
Bottoms Rate, moles/hr.	8.388	9.51
Feed Composition, m.f.*	0.4577	0.4151
Liquid to Reboiler Composition, m.f.*	0.3622	0.3472
Vapor from Reboiler Composition, m.f.*	0.4248	0.4029

Time, min.	Bottoms Rate, moles/hr.	Stream Compositions* Distillate	Bottoms
- 5.0	8.388	0.6786	0.2877
0.0	8.388	0.6774	0.2877
1.0	8.53	-	0.2874
3.0	8.93	-	0.2864
5.6	9.15	-	0.2866
7.0	9.28	-	0.2850
9.0	9.32	-	0.2855
11.0	9.43	0.6501	0.2849
13.0	9.46	-	0.2837
16.0	9.50	-	0.2823
20.0	9.50	0.6590	0.2815
24.0	9.50	-	0.2809
28.0	9.51	-	0.2794
32.0	9.51	0.6674	0.2768

*Compositions are given as mole fraction benzene.

TABLE XX

EXPERIMENTAL DATA FOR RUN FFC 2-6

Benzene-Toluene

Parameter	Initial Steady State	Final Steady State
Feed Rate, moles/hr.	14.454	14.454
Distillate Rate, moles/hr.	4.924	6.678
Bottoms Rate, moles/hr.	9.530	7.776
Feed Composition, m.f.*	0.4071	0.4752
Liquid to Reboiler Composition, m.f.*	0.3236	0.3729
Vapor from Reboiler Composition, m.f.*	0.3920	0.4184

Time, min.	Bottoms Rate, moles/hr.	Stream Compositions* Distillate	Bottoms
- 5.0	9.53	0.6808	0.2545
0.0	9.53	0.6632	0.2553
1.0	9.15	-	0.2557
3.0	8.60	-	0.2551
5.0	8.28	-	0.2564
7.0	8.08	-	0.2594
9.0	7.97	-	0.2603
11.0	7.88	0.7171	0.2633
13.0	7.83	-	0.2674
16.0	7.81	-	0.2711
20.0	7.79	0.7243	0.2760
24.0	7.78	-	0.2838
28.0	7.78	-	0.2874
32.0	7.78	0.7152	0.2921

*Compositions are given as mole fraction benzene.

TABLE XXI

EXPERIMENTAL DATA FOR RUN FFC 3-1

Benzene-Toluene-p-Xylene

Parameter	Initial Steady State	Final Steady State
Feed Rate, moles hr.	13.891	13.891
Distillate Rate, moles/hr.	4.584	6.264
Bottoms Rate, moles/hr.	9.307	7.627
Feed Composition, m.f.		
Benzene	0.3026	0.3738
Toluene	0.5725	0.5166
Liquid to Reboiler Composition, m.f.		
Benzene	0.2010	0.2196
Toluene	0.6600	0.6486
Vapor from Reboiler Composition, m.f.		
Benzene	0.2617	0.2625
Toluene	0.6387	0.6382

Time, min.	Bottoms Rate, moles/hr.	Product Stream Compositions, m.f.			
		Benzene		Toluene	
		Distillate	Bottoms	Distillate	Bottoms
- 5.0	9.31	0.5989	0.1490	0.3707	0.6780
0.0	9.31	0.5848	0.1483	0.3813	0.6769
1.0	8.93	-	0.1485	-	0.6778
3.0	8.38	-	0.1484	-	0.6778
5.0	8.07	-	0.1474	-	0.6791
7.0	7.87	-	0.1477	-	0.6796
9.0	7.77	-	0.1481	-	0.6792
11.0	7.71	0.6563	0.1490	0.3172	0.6781
13.0	7.67	-	0.1509	-	0.6767
16.0	7.65	-	0.1507	-	0.6763
20.0	7.65	0.6594	0.1522	0.3155	0.6743
24.0	7.64	-	0.1525	-	0.6728
28.0	7.63	-	0.1528	-	0.6723
32.0	7.63	0.6348	0.1523	0.3374	0.6717

TABLE XXII

EXPERIMENTAL DATA FOR RUN FFC 3-3

Benzene-Toluene-p-Xylene

Parameter	Initial Steady State	Final Steady State
Feed Rate, moles/hr.	14.160	14.160
Distillate Rate, moles/hr.	4.692	5.832
Bottoms Rate, moles/hr.	9.468	8.328
Feed Composition, m.f.		
Benzene	0.3858	0.0467
Toluene	0.5152	0.4771
Liquid to Reboiler Composition, m.f.		
Benzene	0.2948	0.3086
Toluene	0.5948	0.5868
Vapor from Reboiler Composition, m.f.		
Benzene	0.3791	0.3796
Toluene	0.5479	0.5476

		Product Stream Compositions, m.f.			
		Benzene		Toluene	
Time, min.	Bottoms Rate, moles/hr.	Distillate	Bottoms	Distillate	Bottoms
- 5.0	9.47	0.6837	0.2303	0.2948	0.6298
0.0	9.47	0.6731	0.2301	0.3037	0.6298
1.0	9.23	-	0.2292	-	0.6305
3.0	8.88	-	0.2293	-	0.6304
5.0	8.68	-	0.2289	-	0.6308
7.0	8.54	-	0.2299	-	0.6299
9.0	8.46	-	0.2306	-	0.6296
11.0	8.41	0.7037	0.2325	0.2685	0.6287
13.0	8.37	-	0.2333	-	0.6279
15.0	8.35	-	0.2343	-	0.6272
18.0	8.33	0.7133	0.2354	0.2685	0.6269
22.0	8.33	-	0.2350	-	0.6276
26.0	8.33	-	0.2347	-	0.6279
32.0	8.33	0.7051	0.2335	0.2750	0.6285

TABLE XXIII

EXPERIMENTAL DATA FOR RUN FFC 3-4

Benzene-Toluene-p-Xylene

Parameter	Initial Steady State	Final Steady State
Feed Rate, moles/hr.	14.833	14.833
Distillate Rate, moles/hr.	4.665	7.183
Bottoms Rate, moles/hr.	9.279	6.761
Feed Composition, m.f.		
Benzene	0.3246	0.4223
Toluene	0.5598	0.4891
Liquid to Reboiler Composition, m.f.		
Benzene	0.2362	0.2628
Toluene	0.6366	0.6205
Vapor from Reboiler Composition, m.f.		
Benzene	0.3080	0.3160
Toluene	0.6058	0.5976

Time, min.	Bottoms Rate, moles/hr.	Product Stream Compositions, m.f. Benzene Distillate	Benzene Bottoms	Toluene Distillate	Toluene Bottoms
- 5.0	9.28	0.6202	0.1765	0.3528	0.6630
0.0	9.28	0.6113	0.1769	0.3617	0.6624
1.0	8.75	-	0.1766	-	0.6635
3.0	7.97	-	0.1753	-	0.6642
5.0	7.46	-	0.1758	-	0.6639
7.0	7.20	-	0.1760	-	0.6640
9.0	7.02	-	0.1755	-	0.6641
11.0	6.91	0.6677	0.1761	0.3107	0.6640
13.0	6.85	-	0.1766	-	0.6630
15.	6.82	-	0.1768	-	0.6627
17.0	6.79	-	0.1772	-	0.6619
20.0	6.77	0.6396	0.1791	0.3336	0.6608
24.0	6.76	-	0.1820	-	0.6589
28.0	6.76	-	0.1843	-	0.6568
32.0	6.76	0.6465	0.1861	0.3279	0.6559

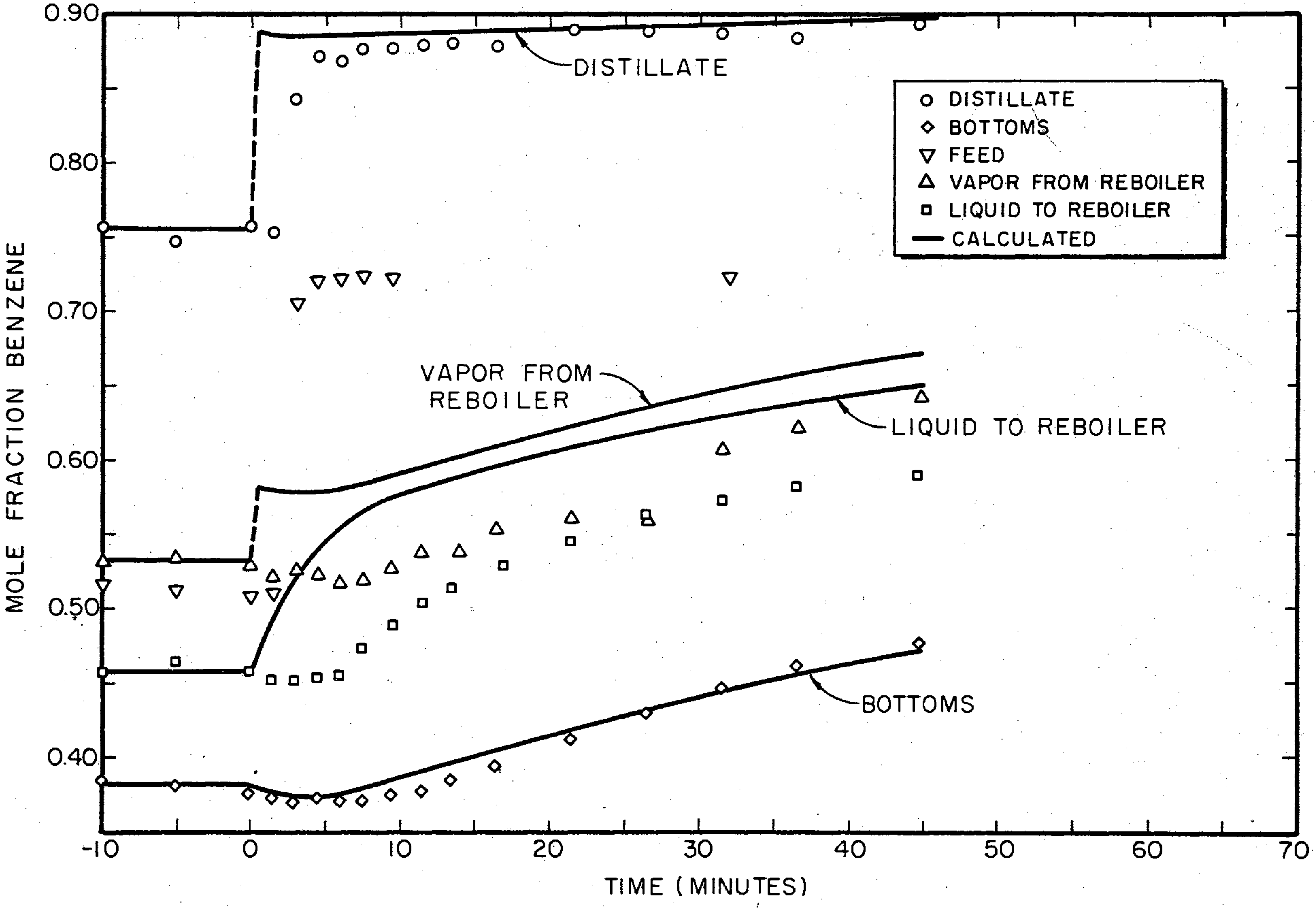

Figure 28. Experimental and Calculated Data for Run 2-1.

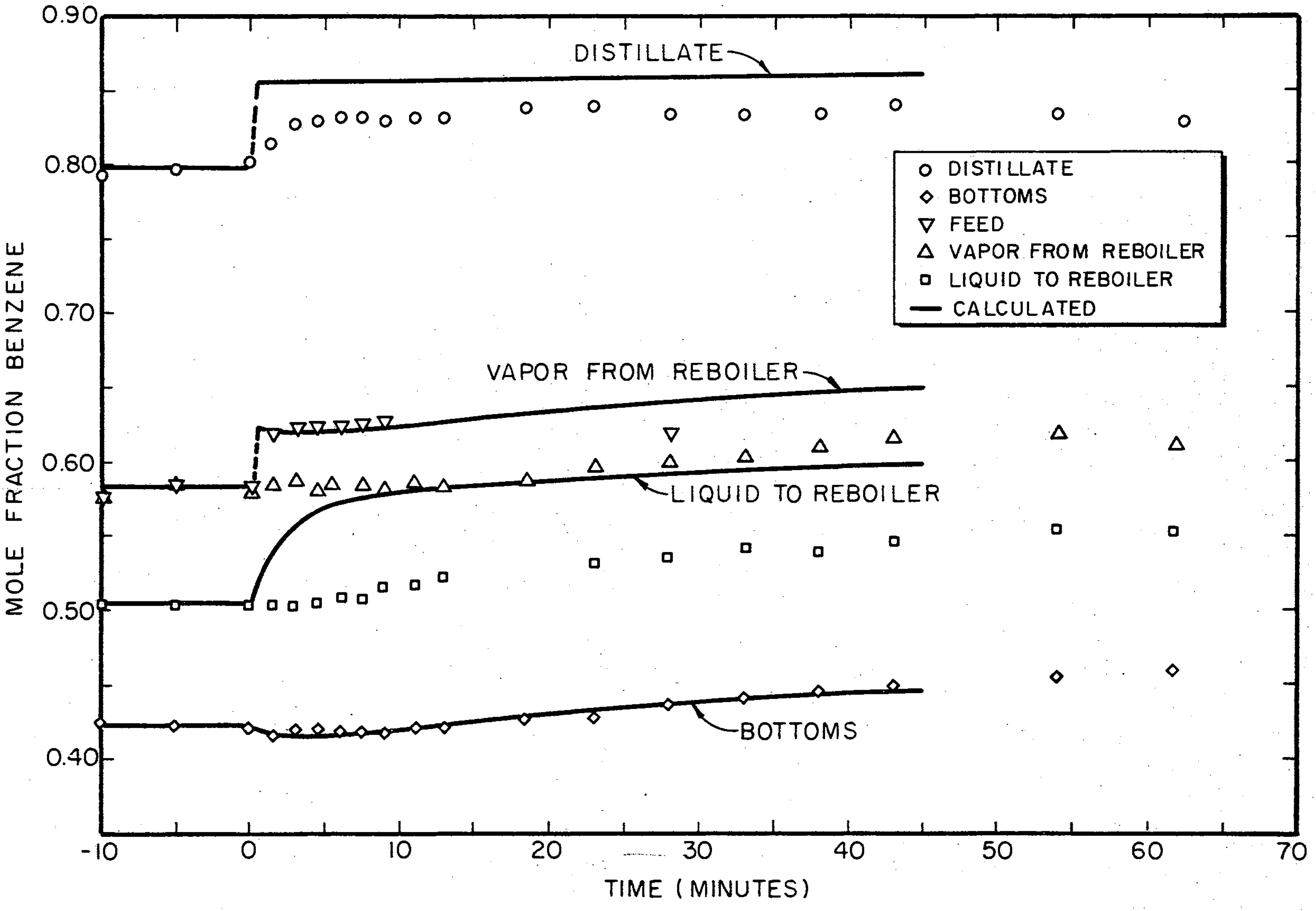

Figure 29. Experimental and Calculated Data for Run 2-2.

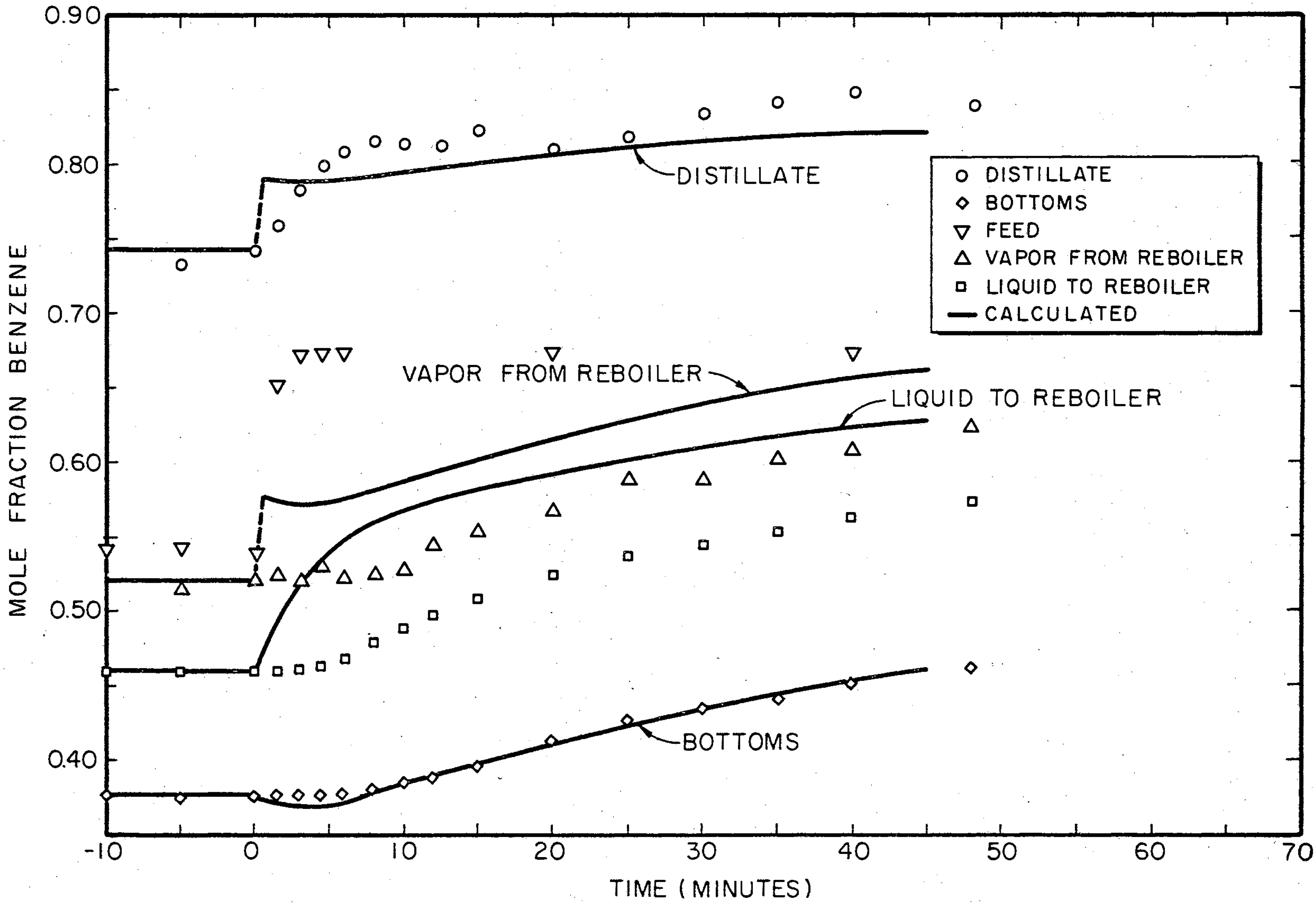

Figure 30. Experimental and Calculated Data for Run 2-4.

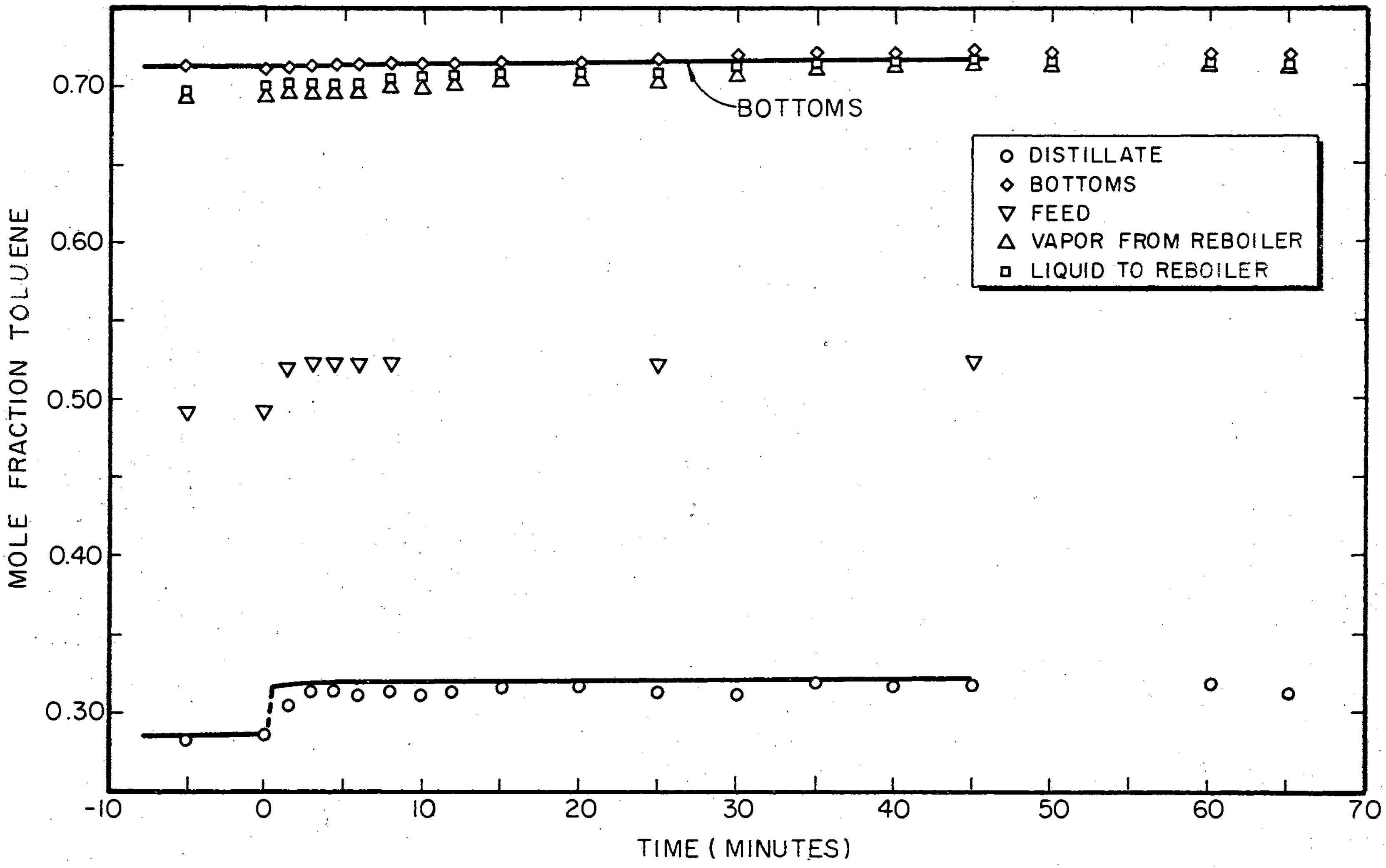

Figure 31. Experimental and Calculated Data for Run 3-1.

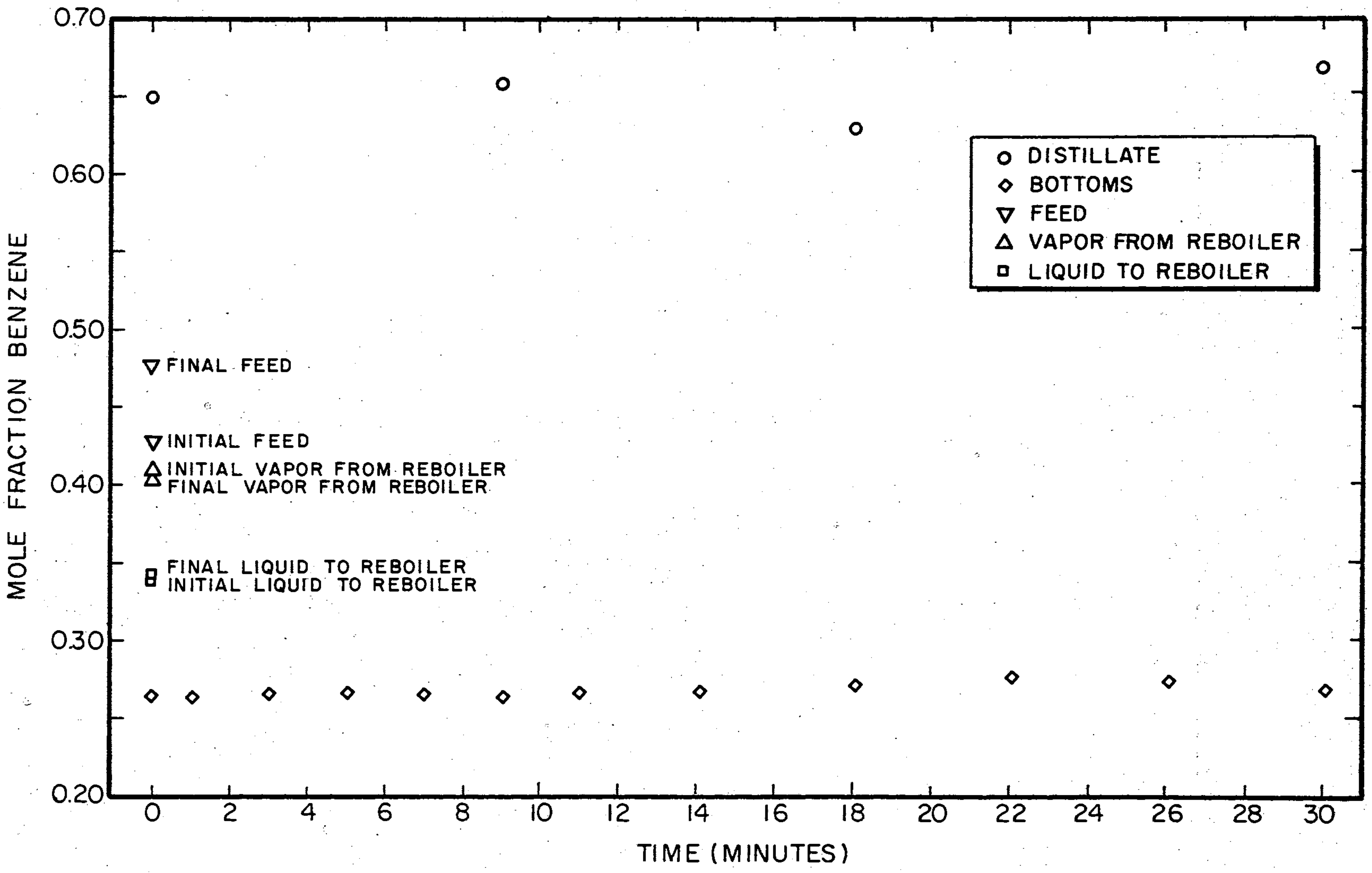

Figure 32. Experimental Data for Run FFC 2-2.

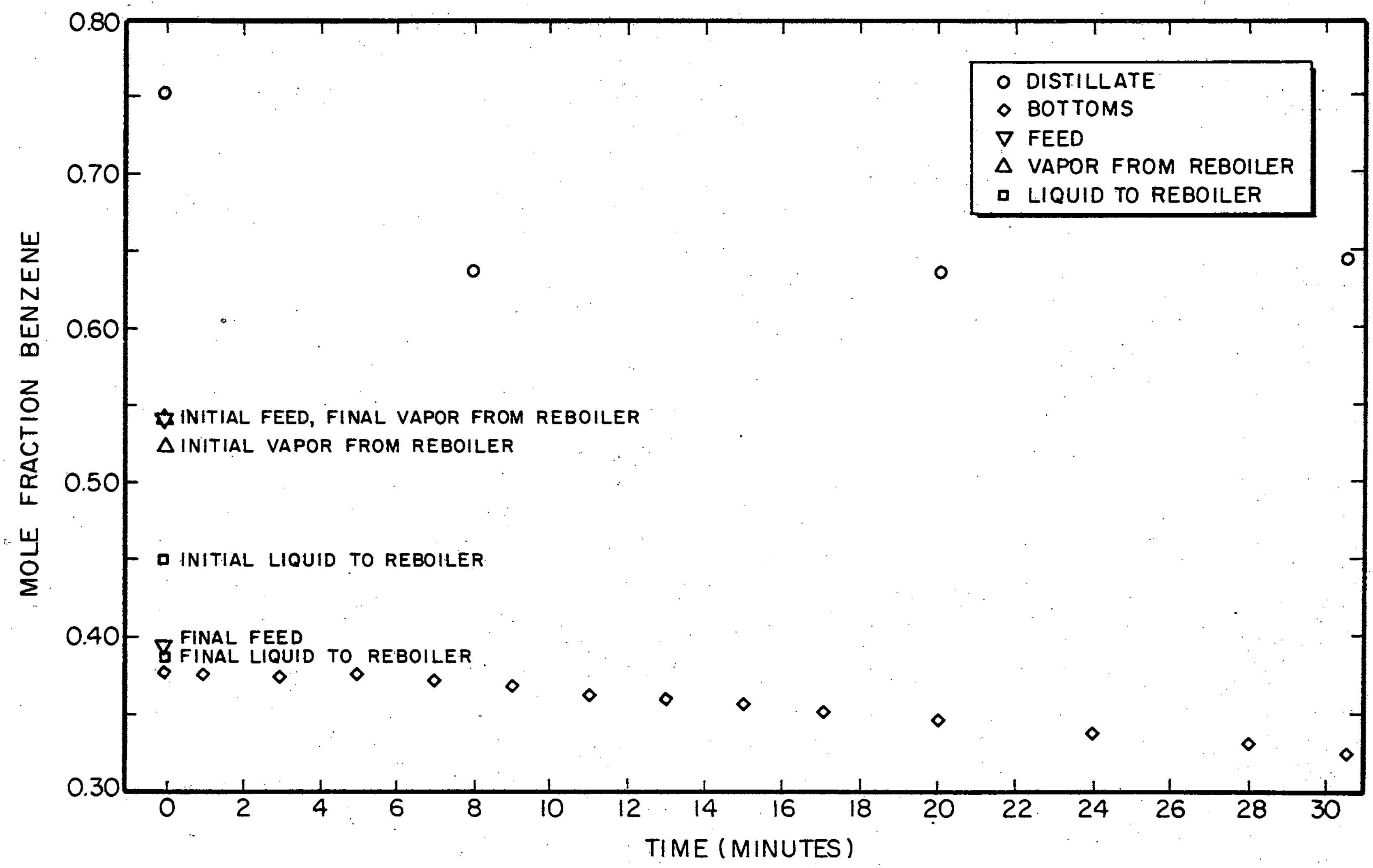

Figure 33. Experimental Data for Run FFC 2-3.

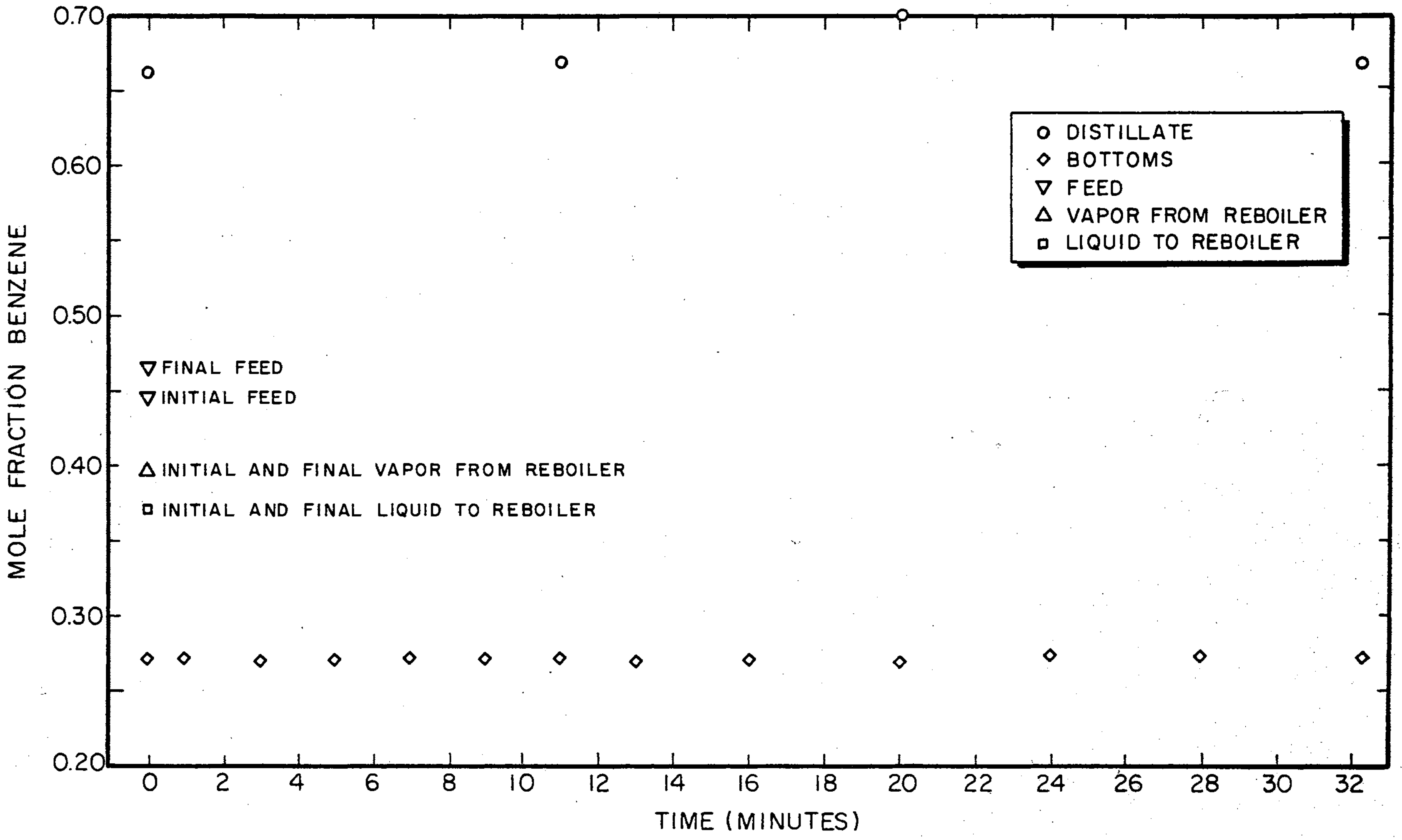

Figure 34. Experimental Data for Run FFC 2-4.

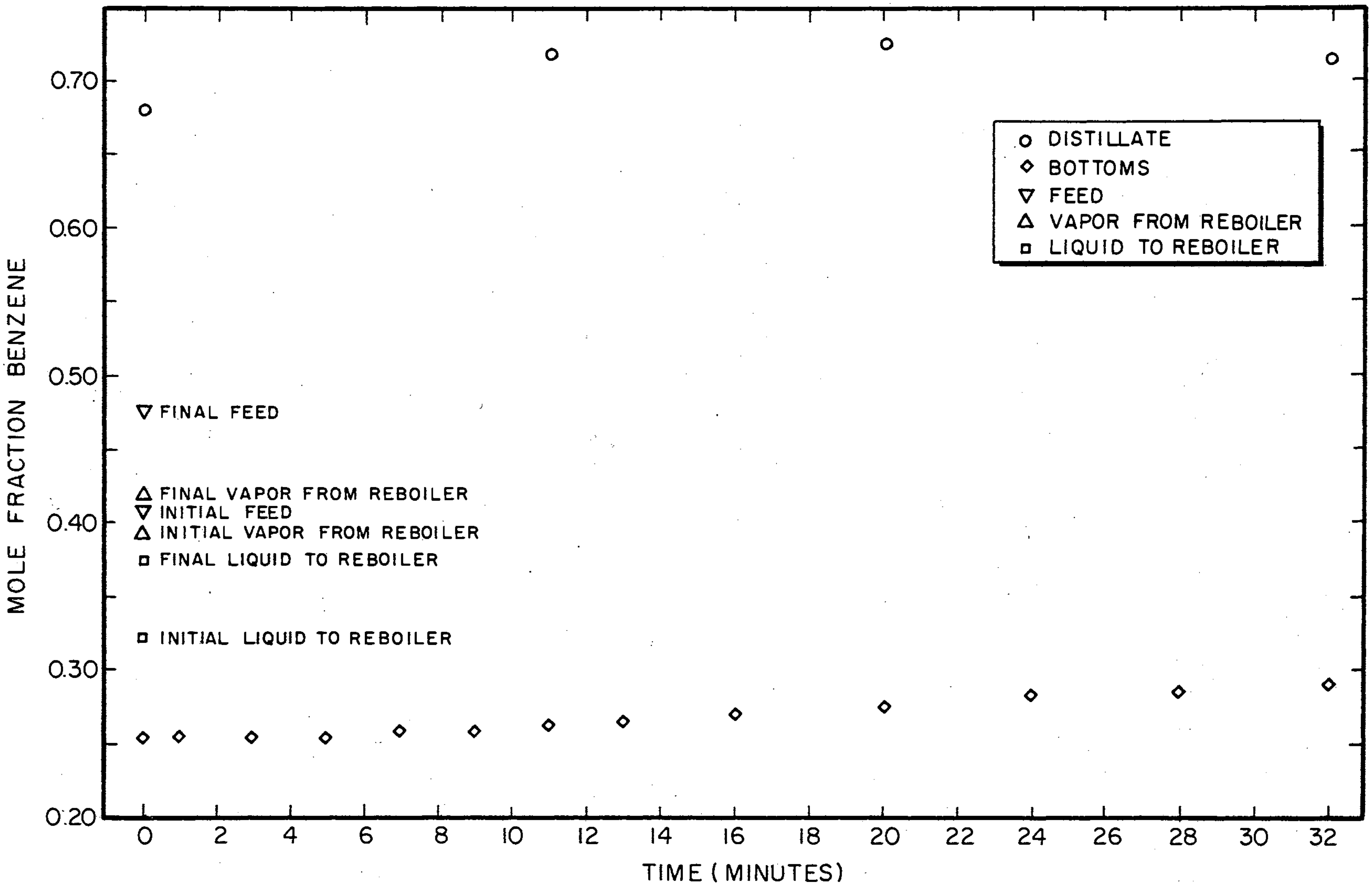

Figure 35. Experimental Data for Run FFC 2-6.

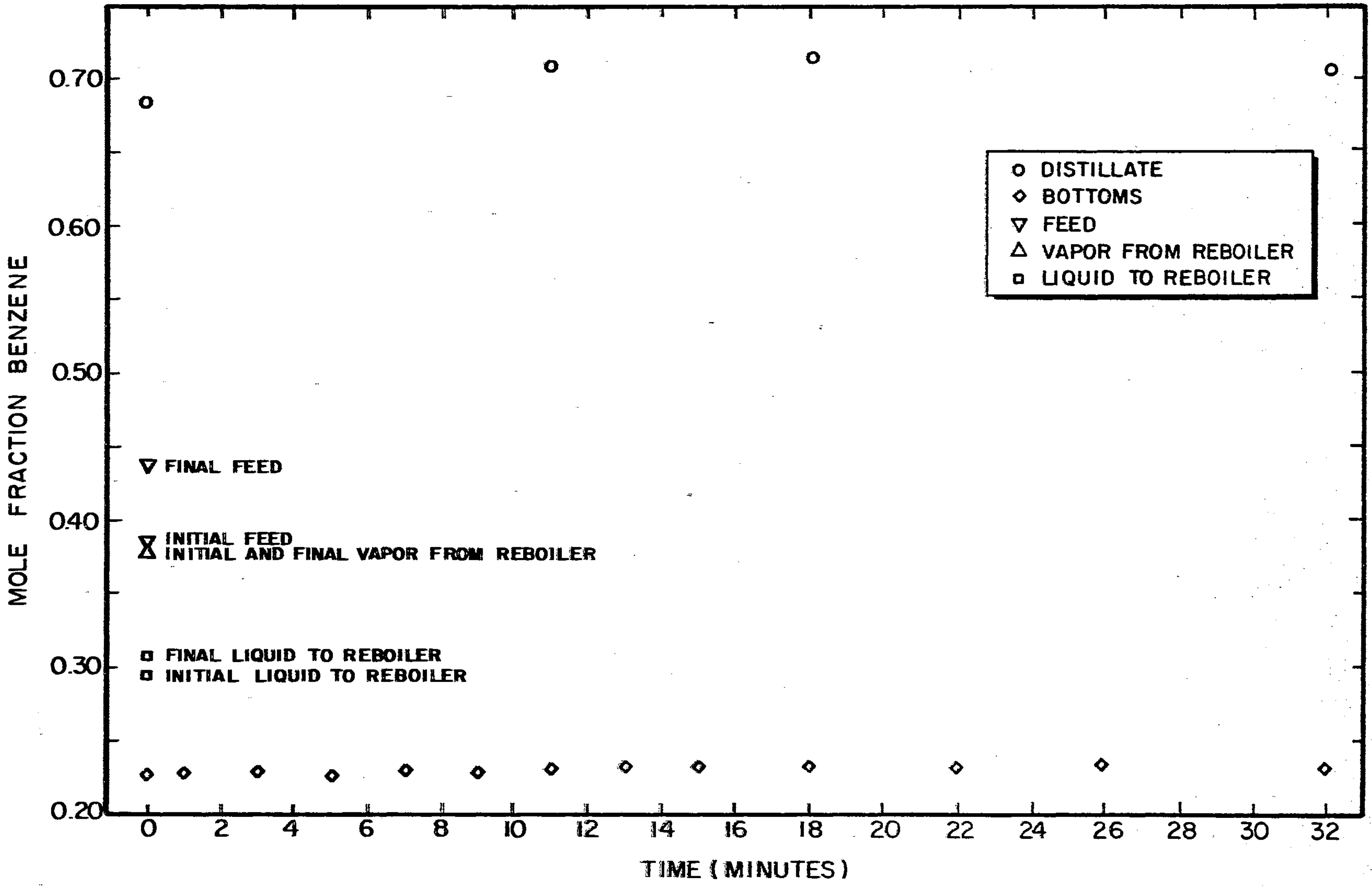

Figure 36. Experimental Data for Run FFC 3-3.

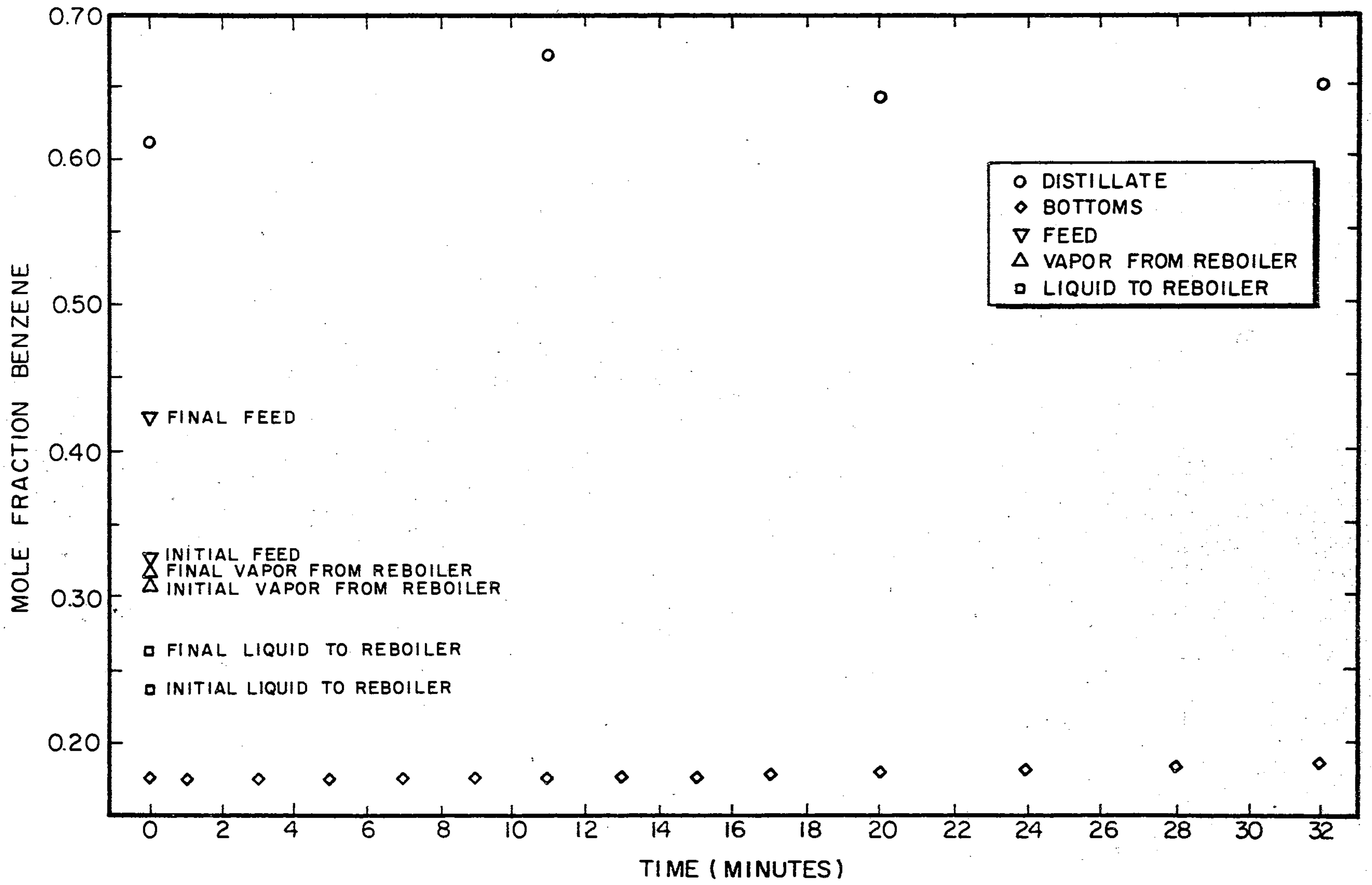

Figure 37. Experimental Data for Run FFC 3-4.

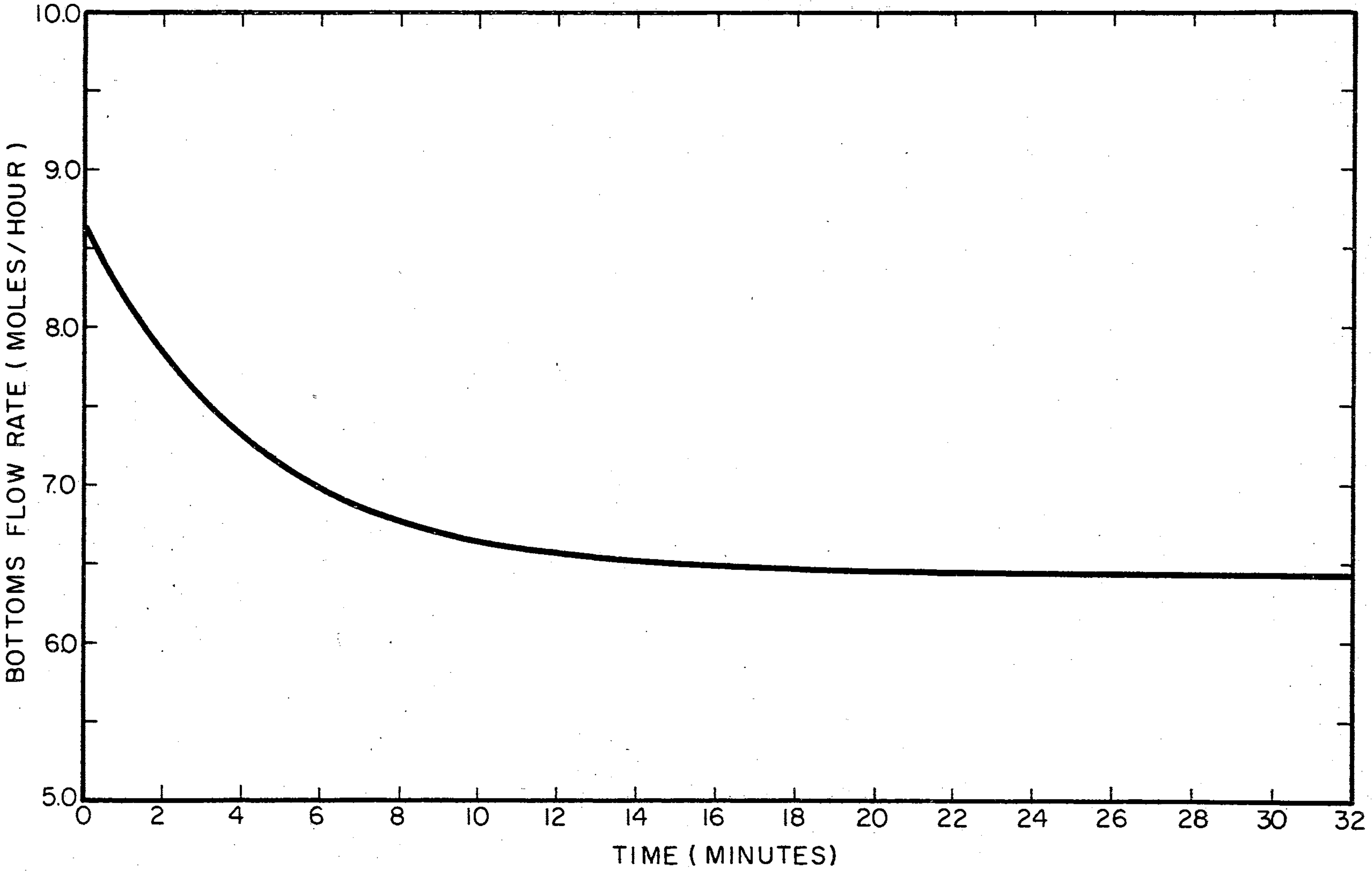

Figure 38. Computed Bottoms Rate Response for Run FFC 2-2.

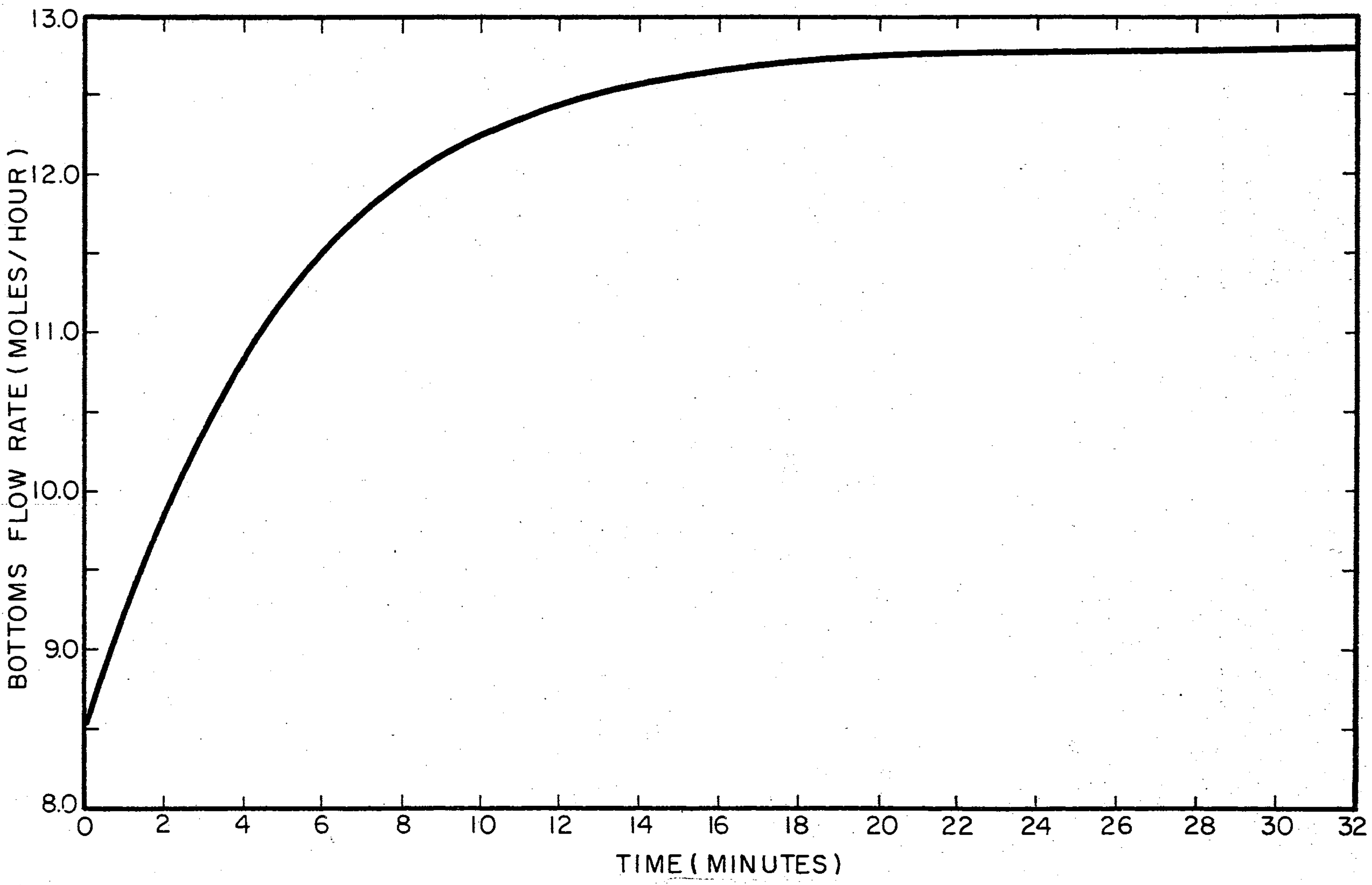

Figure 39. Computed Bottoms Rate Response for Run FFC 2-3.

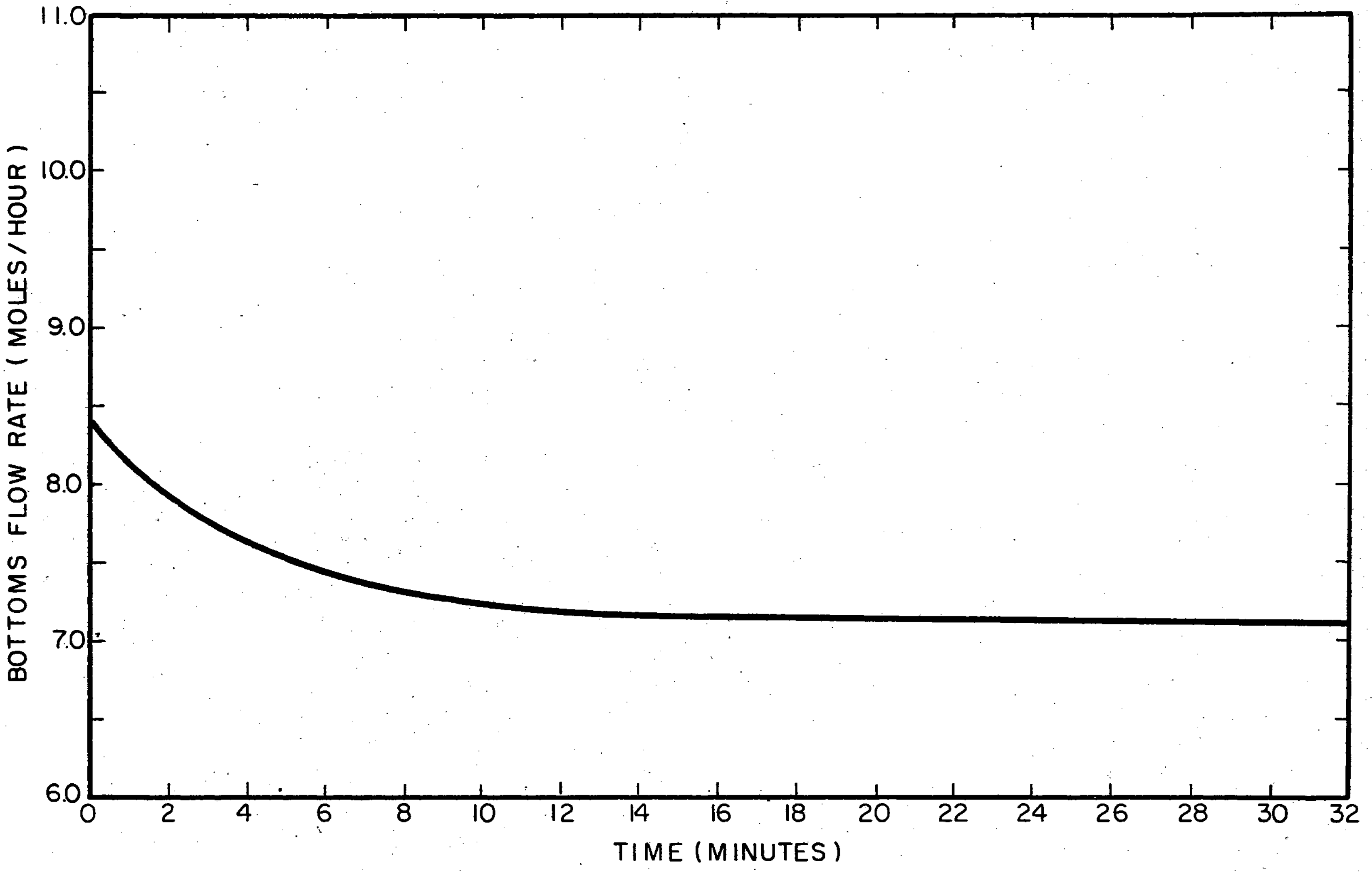

Figure 40. Computed Bottoms Rate Response for Run FFC 2-4.

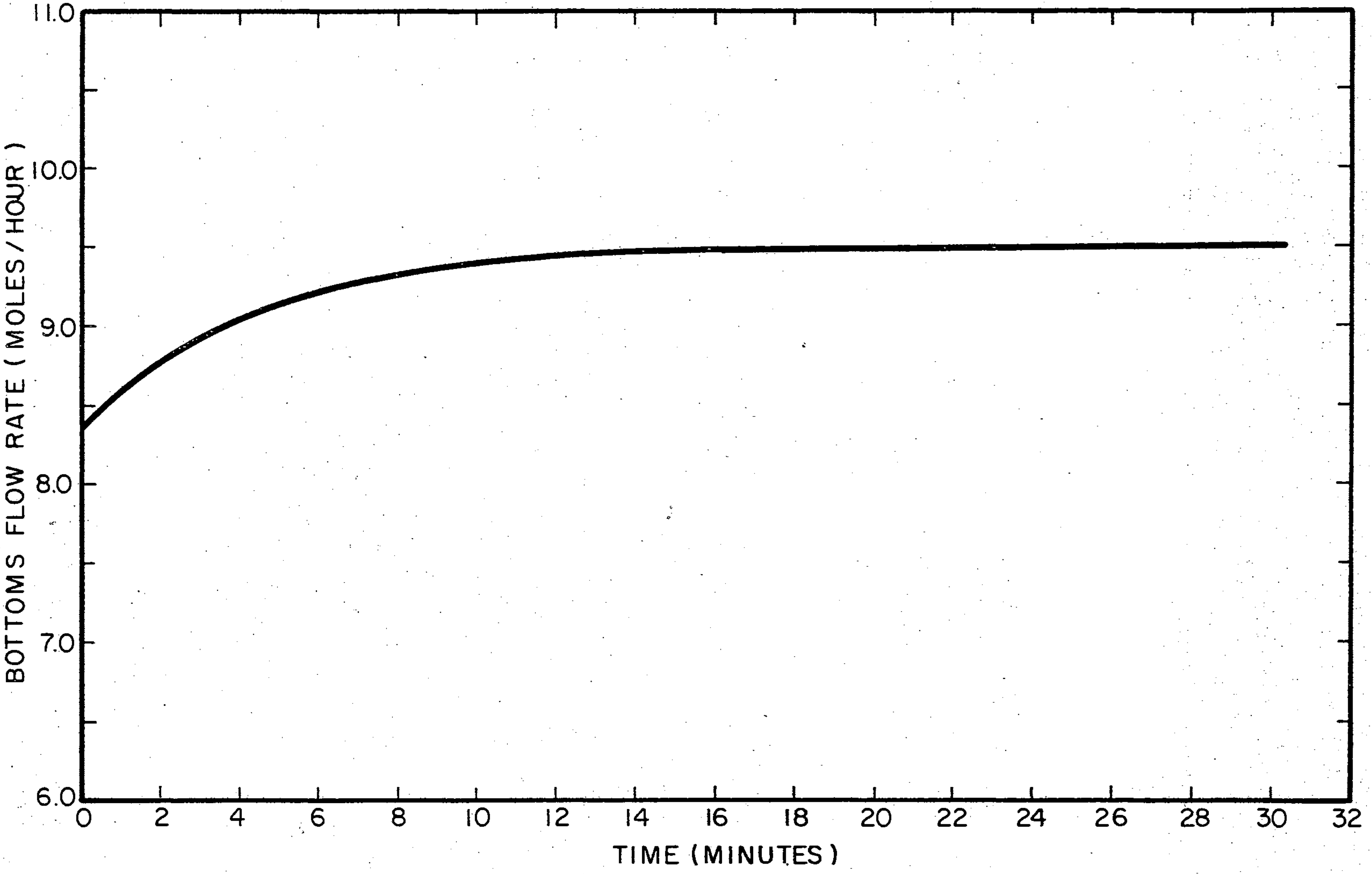

Figure 41. Computed Bottoms Rate Response for Run FFC 2-5.

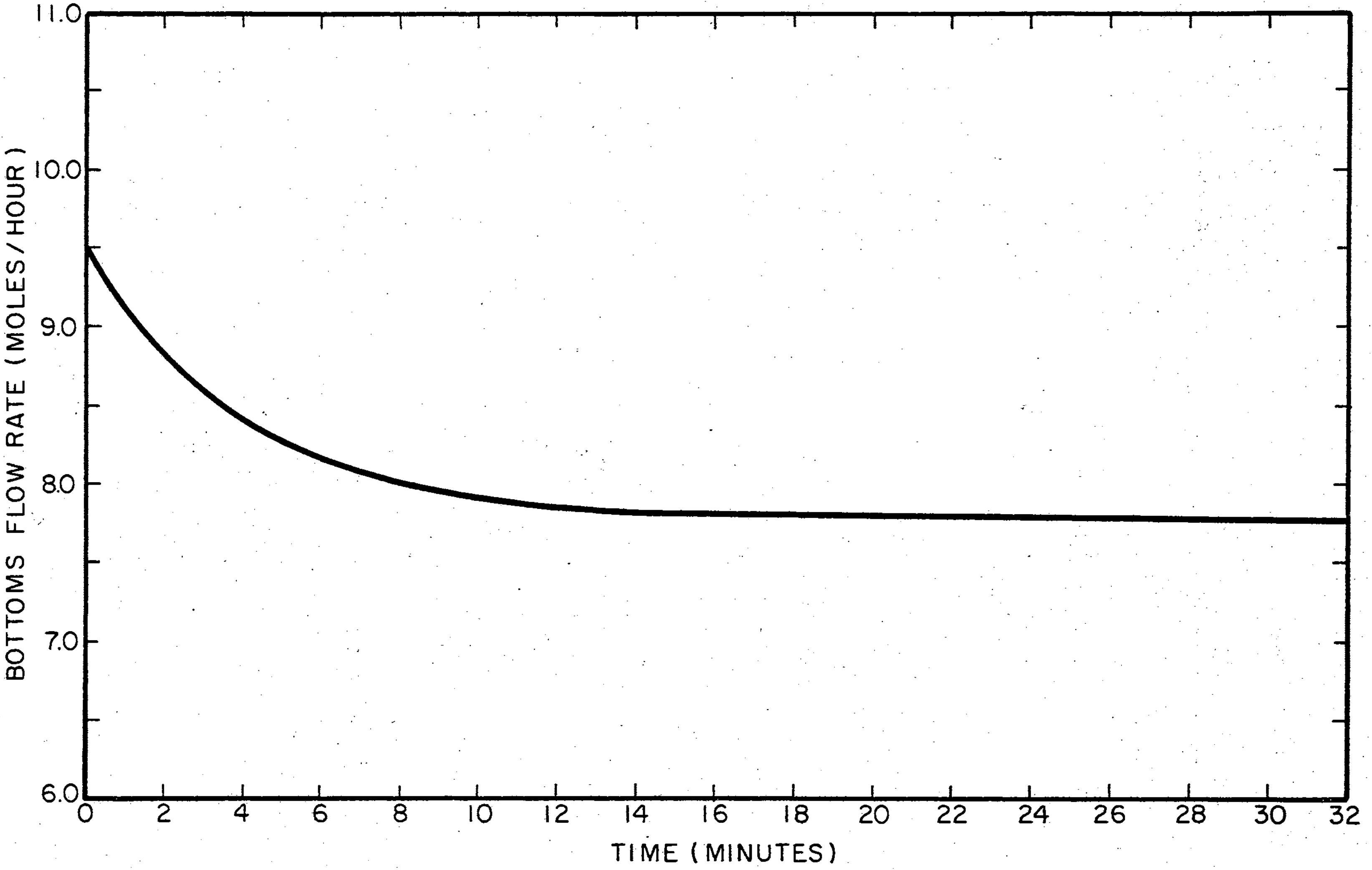

Figure 42. Computed Bottoms Rate Response for Run FFC 2-6.

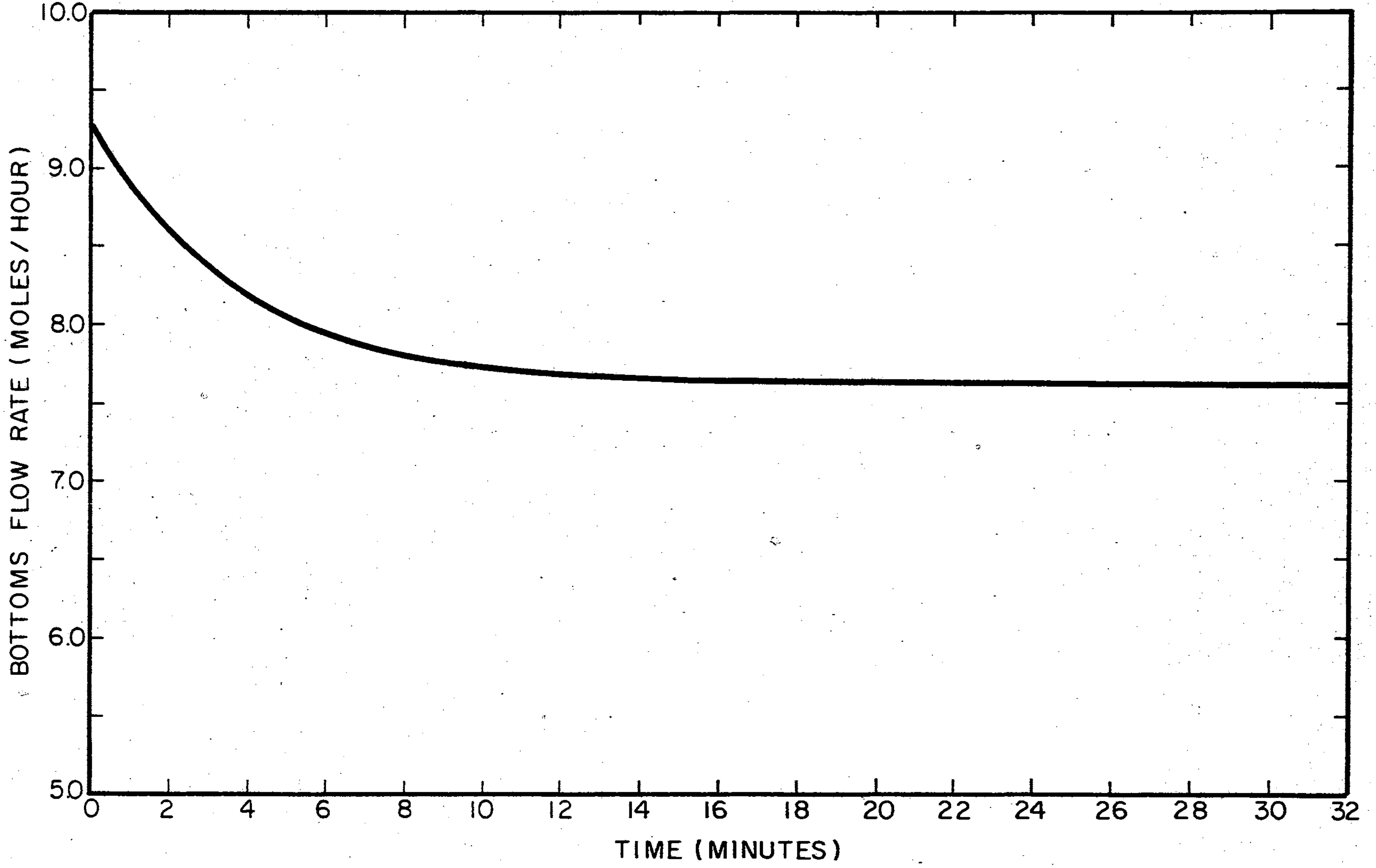

Figure 43. Computed Bottoms Rate Response for Run FFC 3-1.

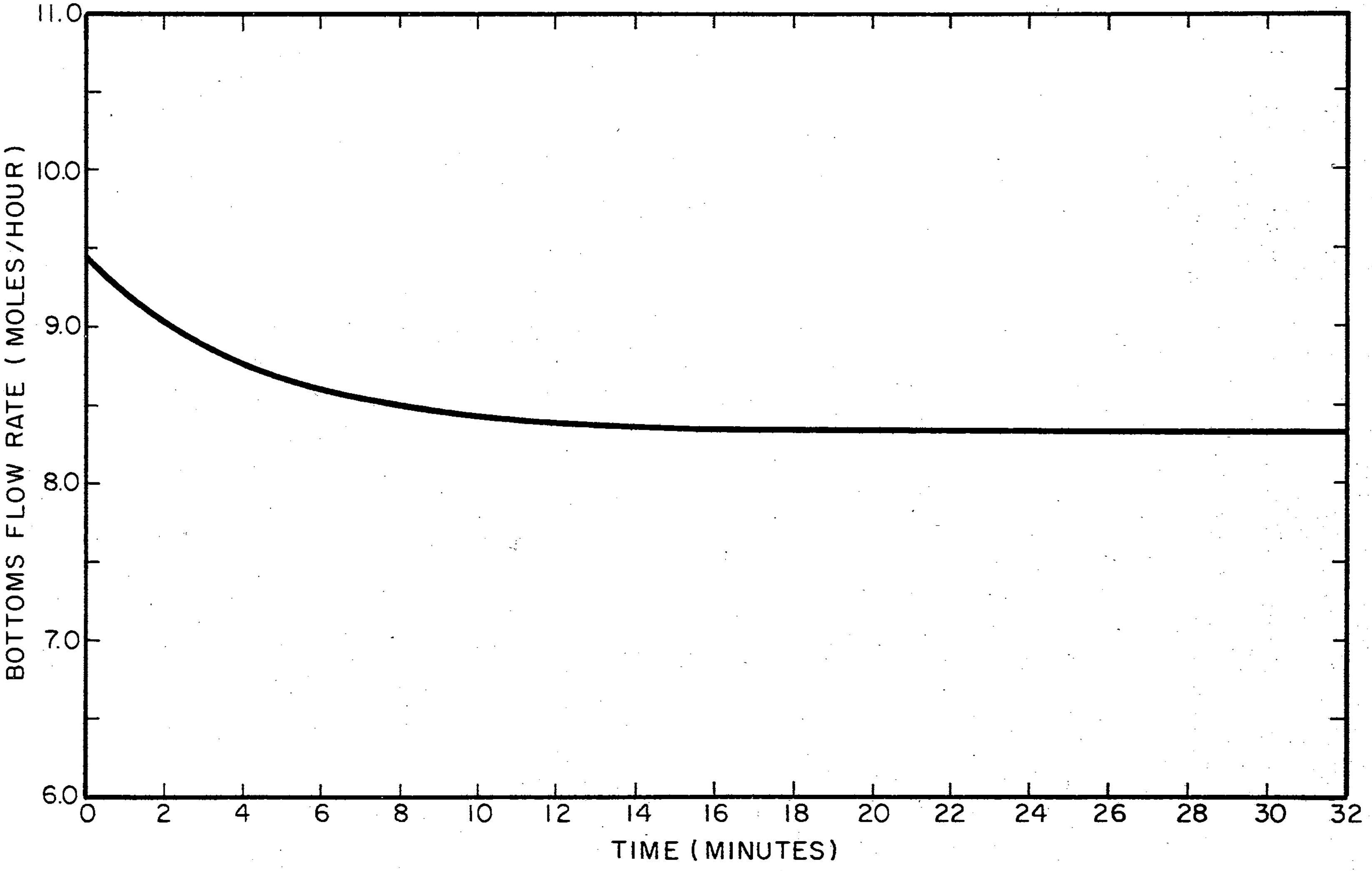

Figure 44. Computed Bottoms Rate Response for Run FFC 3-3.

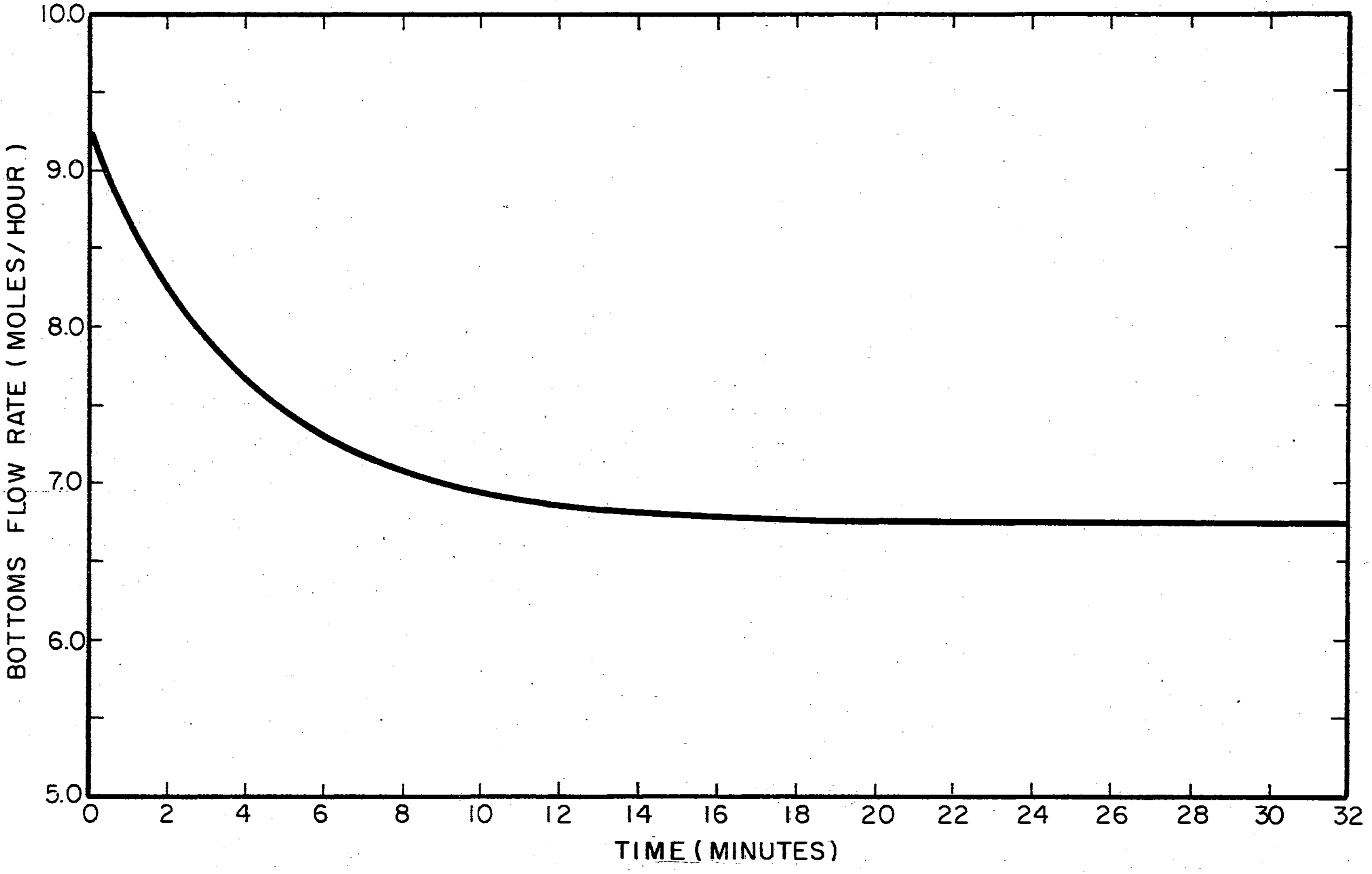

Figure 45. Computed Bottoms Rate Response for Run FFC 3-4.

VITA

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Doctor of Philosophy

Thesis: AN EXPERIMENTAL STUDY OF DISTILLATION COLUMN DYNAMICS

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