

DEVELOPMENT OF DYNAMIC MODELS OF REACTIVE DISTILLATION  
COLUMNS FOR SIMULATION AND DETERMINATION OF CONTROL  
CONFIGURATIONS

A Thesis

by

ARNAB CHAKRABARTY

Submitted to the Office of Graduate Studies of  
Texas A&M University  
in partial fulfillment of the requirements for the degree of

MASTER OF SCIENCE

December 2004

Major Subject: Chemical Engineering

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December 2004

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## ABSTRACT

Development of Dynamic Models of Reactive Distillation Columns for  
Simulation and Determination of Control Configurations. (December 2004)

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Chair of Advisory Committee: Dr. Juergen Hahn

Dynamic models of a reactive distillation column have been developed and implemented in this work. A model describing the steady state behavior of the system has been built in a first step. The results from this steady state model have been compared to data provided from an industrial collaborator and the reconciled model formed the basis for the development of a dynamic model. Four controlled and four manipulated variables have been determined in a subsequent step and step tests for the manipulated variables were simulated. The data generated by the step responses was used for fitting transfer functions between the manipulated and the controlled variables. RGA analysis was performed to find the optimal pairing for controller design. Feedback controllers of PID type were designed between the paired variables found from RGA and the controllers were implemented on the column model. Both servo and regulatory problems have been considered and tested.

To my parents and sister

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## CHAPTER I

### INTRODUCTION

#### **Introduction to Reactive Distillation**

Even after so many years distillation still remains one of the most important separation process in chemical industry. All around the globe, in almost all the chemical industries a significant fraction of capital investment and operating cost involves distillation. As a result any kind of improvement of distillation operation can be very beneficial economically.

Reactive distillation is one major step in the history of distillation in achieving these goals. Reactive distillation processes couple chemical reactions and physical separations into a single unit operation. These processes as a whole are not a new concept as the first patent dates back to the 1920s<sup>1</sup>. The initial publications dealt with homogeneous self-catalyzed reactions such as esterifications and hydrolysis. Heterogeneous catalysis in reactive distillation is a more recent development and was first described by Spes<sup>2</sup>. While the concept existed much earlier, the first real- world implementation of reactive distillation took place in 1980s.

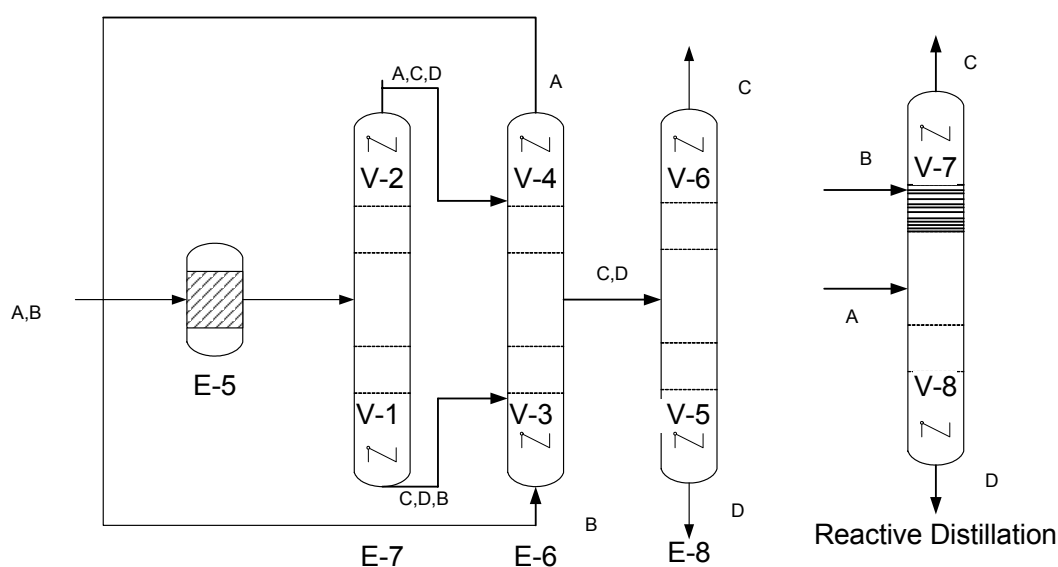
The relatively large amount of new interest in reactive distillation is due to the numerous advantages it has over ordinary distillation. Enhanced reaction rates, increased conversion, enhanced reaction selectivity, heat integration benefits and reduced operating costs are just to name a few. All these factors contribute to the growing commercial importance of reactive distillation. However, since heat transfer, mass transfer, and reactions are all occurring simultaneously, the dynamics which can be exhibited by catalytic distillation columns can be considerably more complex than found in regular columns. This results in an increase in the complexity of process operations and the control structure installed to regulate the process.

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This thesis follows the style and format of the *Journal of Physical Chemistry*.

The principle of building a reactive distillation column is quite simple. A distillation column having a catalyst zone strategically placed in the column to carry out the desired reaction is a reactive distillation column. The catalyst may be in the same phase as that of the reacting species or it can be in the solid phase. The feed for the process is fed either above or below the reactive zone depending upon the volatility of the components and to carry out the desired reaction. The reaction occurs mainly in the liquid phase, in the catalyst zone.

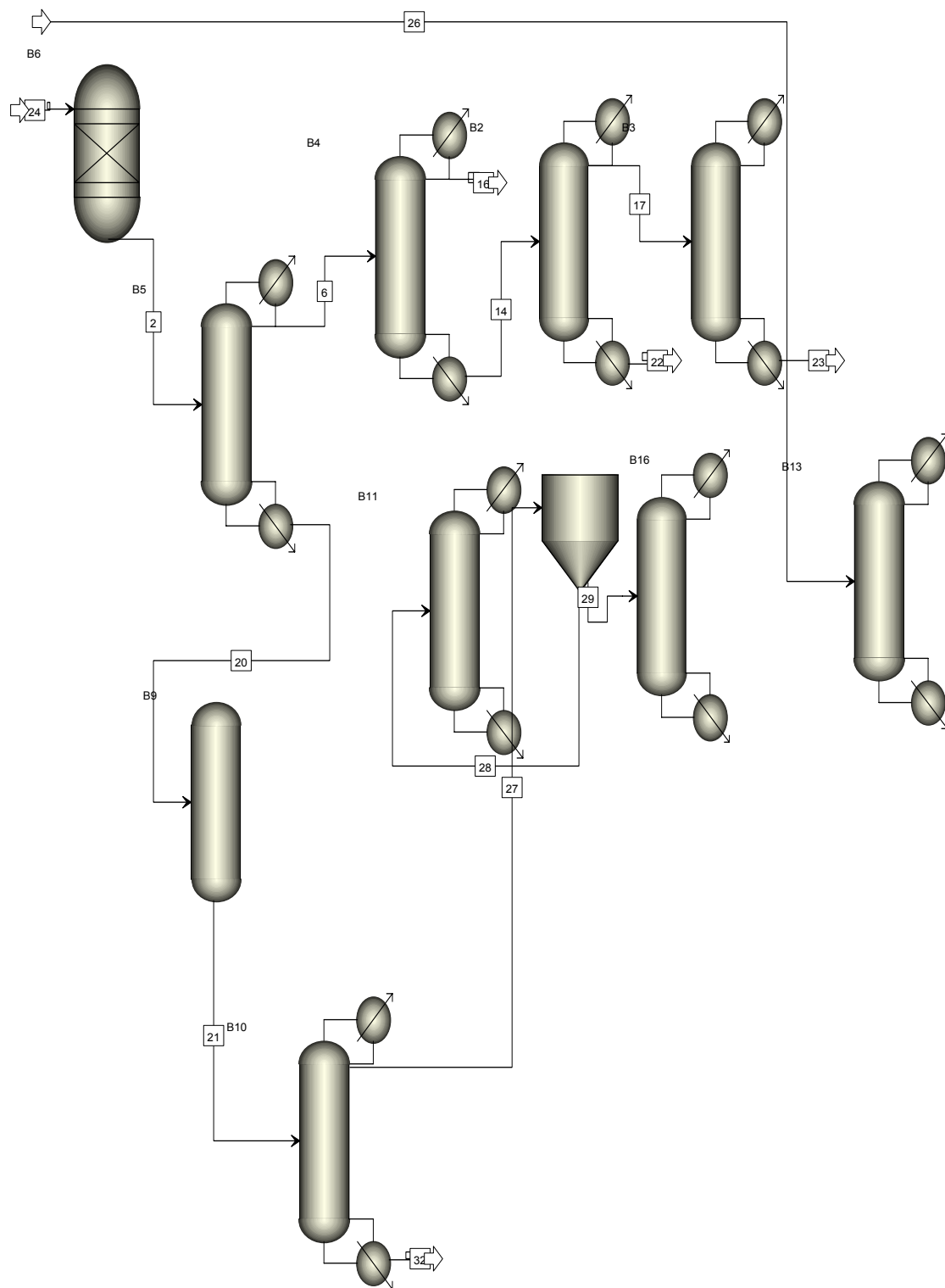
A simple example <sup>3</sup> considering a reversible reaction can explain some of the mentioned properties. Consider a reaction  $A + B \leftrightarrow C + D$ , where the boiling points of the components follow the order A, C, D and B.



**Figure 1: Comparison of conventional scheme and reactive distillation for a generic system**

The traditional flowsheet can be a series of distillation columns as shown in Figure 1 (a) where the mixture of A and B is fed to the reactor, where the reaction takes place in the presence of a catalyst. The mixture of reactant and product is passed through a series of distillation columns and the unreacted components A and B are recycled back to the reactor. The alternative reactive distillation approach consists of a reactive section in the middle of the column. The task of the rectifying section is to recover reactant B from the product stream C. In the stripping section the reactant A is stripped from the product stream D. The relative volatility between the components has been used more effectively in the later case.

An excellent example <sup>3</sup> of the benefits of reactive distillation is the production of methyl acetate. The acid catalyzed reaction  $\text{MeOH} + \text{AcOH} \leftrightarrow \text{MeOAc} + \text{H}_2\text{O}$  was traditionally carried out using the processing scheme shown in Figure 2, which consists of one reactor and a train of nine distillation columns. In the reactive distillation set up however only one column is required and nearly 100% conversion of the reactant is achieved. This results in reducing the capital and operating cost significantly.



**Figure 2: Methyl acetate production**

## **Benzene Hydrogenation**

Gasoline is a complex mixture of hydrocarbons<sup>3</sup>, generally in the range of C6-C10, and different mixtures have different octane ratings. Gasoline reforming is the process of altering the composition of gasoline to achieve a higher octane rating for fuels. A reforming process generally produces high octane aromatics one of which is benzene. Studies show increased incidence of leukemia in humans exposed to benzene and the EPA has classified benzene as a human carcinogen. As a consequence most petroleum refineries are facing the challenge of producing motor gasoline having all the desirable properties, i.e. high octane rating, while also complying with the ever increasing environmental regulations and health restrictions on automotive emissions, one of the main thrust being on benzene control. Since 60-70 percent of the total benzene in gasoline is produced in the reforming unit benzene reduction in the reformat has a major effect on meeting the specification imposed.

To address these points, refineries world wide have adopted three basic approaches for benzene reduction.

- Limitation of benzene production in the reformer by diminishing benzene precursors, C6 content in the reformer feed.
- Adjustment of reformer operating conditions.
- Down stream removal of the benzene produced by the reformer. Various new processes have been developed for benzene reduction namely:
  1. Reformate benzene saturation: benzene is hydrogenated to cyclo-hexane
  2. Reformate splitting and benzene extraction
  3. Alkylation

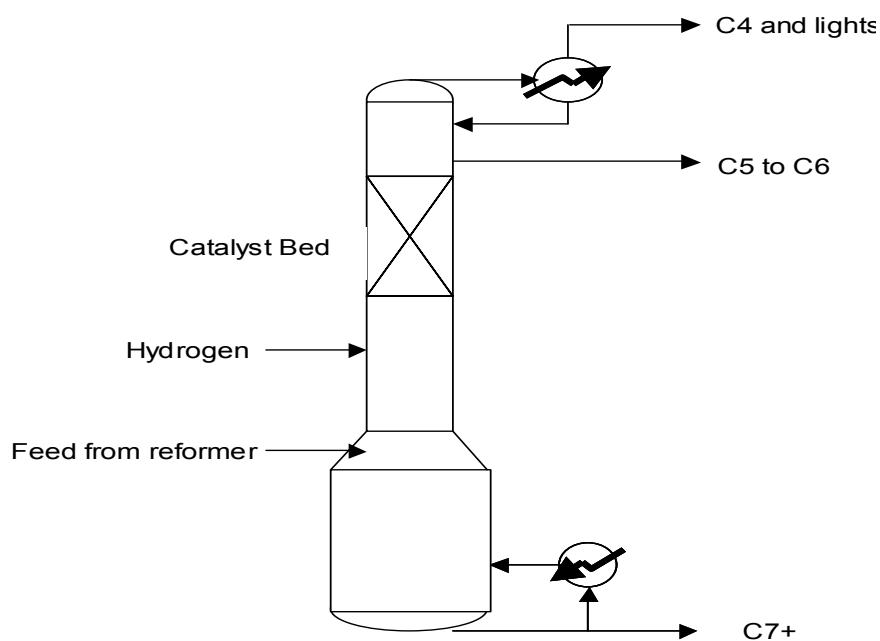
The focus of this work is on the down stream removal of benzene via benzene saturation. However, the feed from the reformer also contains components like toluene which contributes to the high octane number of the stream. This gives rise to a challenging problem as the hydrogenation of benzene can also lead to hydrogenation of toluene which is undesirable. The objective of this work is to study a reactive distillation

column to determine control configurations maximizing the hydrogenation of benzene, while at the same time avoiding saturation of the toluene.

### **Description of the Column Under Study**

The specific process under investigation is a reactive distillation column used for the hydrogenation of benzene in a reformate stream. The column diagram is given in Figure 3. The column is a tray tower and consists of 70 trays, a condenser and a reboiler. The feed from the reformer enters the column as a liquid above tray 25 where the trays are numbered from bottom to top. Hydrogen is required for the reaction and is fed as a separate stream above tray 25. There are three products coming out of the column. The vapor stream leaving the condenser mainly contains the unreacted hydrogen. The top liquid product contains a roughly 30% of cyclohexane produced from the hydrogenation of benzene. The bottom product is also liquid and constitutes the main product stream as it has the desired octane properties due to the high percentage of toluene and at the same time only a small amount of benzene complying with the EPA regulation. The feed flow rate is 200,000 lb/hr and its temperature is 456 K at a pressure of 115 psia. The temperature and pressure associated with the hydrogen stream is 300 K and 14.7 psia respectively. The hydrogen feed flow rate at nominal operating condition is 2000 lb/hr.





**Figure 3: Process under study**

The reactive zone occupies trays 51 through 65 in the column and is filled with catalyst. The location of the reactive zone is important in the process. Essentially it has to be placed somewhere above the feed trays since toluene is heavier than most of the other hydrocarbons present in the feed. At the same time benzene is lighter and will primarily be found above the feed tray.

However it is not a good idea to design the reactive zone to be immediately on top of the feed trays, as some of the toluene will also be found in this part of the column. While it is theoretically possible that the reaction can take place outside of the reactive zone, the reaction will proceed with such a slow rate without the catalyst that this effect can be ignored for this investigation. Two reactions take place in the reactive zone: hydrogenation of benzene to cyclo-hexane and hydrogenation of toluene to methylcyclo-hexane. Both reactions are exothermic and as a result the reboiler heat duty is significantly smaller than the condenser duty. However, while it is the purpose of this process to hydrogenate the benzene, the reaction involving toluene is unwanted and should be suppressed. The easiest way of achieving this is to operate the column in such a way that as little as toluene will enter the reactive zone and the majority of toluene will therefore not be hydrogenated.

The rate of reaction for both the components is assumed to be first order with respect to the reactants. The rate of the reaction for both the cases and can be expressed as follows:

- $r_1 = k_1 x_{Bz} P_{H_2}$
- $r_2 = k_2 x_{To} P_{H_2}$

The reformate component composition by weight percent is given below in Table 1. There is no hydrogen, cyclo-hexane or methylcyclo-hexane in the feed.

**Table 1: Feed Composition**

Feed Components	Weight %
C4s	1
C5s	8
DiMe-Butane	1
Me-Pentane	5
Hexane	3
Benzene	8
Me-hexane	2
DiMe-Pentane	2
Hepatne	1
Toluene	30
Xylene	22
Cumene	17

The tower specifications are:

- 2 ft spacing between the trays
- Reflux enters above tray 70
- Diameter Tray 1 – 25 = 12 ft
- Diameter Tray 26-70 = 8 ft
- Condenser Duty=-64.8MMBtu/hr
- Reboiler Duty = 40.1MMBtu/hr

## **Objective**

As already discussed the main challenge is to hydrogenate the benzene keeping the toluene intact. Even under the influence of different disturbances it has to be ensured that the undesired reactant should be kept out of the catalyst zone and the desired reactant is primarily found in this zone. The disturbances associated with the process can be large as the amount of benzene in the feed can range from 3% to 15% weight fraction. The fraction of heavier components can range from 40% to 80%. The main disturbance investigated in this work is the benzene mass fraction in the feed which changes at regular 24 hours intervals. Due to this disturbance the column moves away from its operating point and normally reaches to a new steady state after 2 to 3 hours. Since the disturbance can occur every day and since it takes a significant amount of time to reach the new steady state, there is a strong need for effective control of the process. However controllers can only be designed if a dynamic model is available and therefore developing a dynamic process model is one of the objectives of this work.

## **Past Work**

Many papers have analyzed both regular and reactive distillation columns operating at steady state. Skogestad and Morari<sup>4</sup> presented a systematic approach to distillation column control configuration design. The main focus was on the steps to be carried out before designing the actual controller for the distillation column as the authors selected a control configuration prior to controller design. Skogestad and Lundstrom<sup>5</sup> investigated the opportunities and challenges of a 5 X 5 control structure of a distillation column having one feed and two products. In later work Skogestad<sup>6</sup> summarized some of the important dynamic and control aspects of continuous distillation column. Kumar and Doutidis<sup>7</sup> studied the control structure of a reactive distillation column with three controlled variables. The process was the formation of ethylene glycol from the reaction of ethylene oxide and water. This work was extended to ethyl acetate reactive distillation afterwards by Vora and Daoutidis<sup>8</sup>. Sneesby<sup>9</sup> investigated a two point control scheme for an ethyl tert-butyl ether reactive distillation column. The controlled variables in this

study were conversion and bottom product purity. Arfaj and Luyben<sup>10</sup> investigated different control structures for closed loop control of an ideal two product reactive distillation column where the reactants are intermediate boilers between light product C and the heavy product D. Later they<sup>11</sup> investigated the design and control of one reactant and two product reactive distillation column system. Lextrait<sup>12</sup> focused on 5 X 5 control structure of a TAME packed reactive distillation column with PI controllers. Tade et al.<sup>13</sup> summarized the current status of reactive distillation column modeling and their significance on process development where control of the column comes into the picture. Peng et al.<sup>14</sup> compared modeling of a packed reactive distillation column via rate-based model and equilibrium-based model. While they did not investigate the control structure of the system, the study itself is very important for the same. In all these studies the investigated system is rather small in nature in terms of the number of components and the investigated reaction chemistries are not very complex. No work on benzene hydrogenation is available in the open literature despite the fact that dozens of units all over the world are operating in chemical plants.

## CHAPTER II

### DYNAMIC MODELING AND CONTROL OF DISTILLATION COLUMN

#### **Building a Dynamic Model**

In order to determine control strategies, it is necessary to gain a quantitative understanding of the dynamic behavior that the process will exhibit. This is best achieved by having a model of the process. Such a model can be based upon first-principles or it could be derived from past data collected from the process. In a first step it is sufficient to build a steady state model which can then be modified to include the dynamic components.

A steady state model of a distillation column can either be rate-based or equilibrium-based. In an equilibrium based model it is assumed that the bulk vapor and the bulk liquid phase are in chemical equilibrium to each other. This means that the vapor and liquid stream exiting from such control volumes will be in equilibrium to each other. There will be no temperature gradient within the region where the equilibrium assumption is valid. In a rate-based model, however, the liquid and vapor interface are assumed to be in equilibrium. There is a temperature gradient in the phases and mass transfer takes place between the bulk and the interface of two phases.

The justification of using an equilibrium-based model in building a distillation column model has often been questioned in many articles<sup>15</sup>. The fact that the streams leaving a tray are never in equilibrium to each other has initiated the use of efficiency in equilibrium based model. However the difficulty and the uncertainty associated with determining the efficiency of each tray is also a concern. Lee et al.<sup>16</sup> compared simulation result of an equilibrium based and non equilibrium based model of a multicomponent reactive distillation column. The conclusion drawn was to prefer generalized non equilibrium model for the simulation as opposed to an equilibrium based model because of the difficulty associated with the prediction of tray efficiencies. Later Taylor et al.<sup>15</sup> compared the two approaches and pointed out that “with ever

increasing computing power these simulations are not only feasible, but in some circumstances they should be regarded as mandatory". In another study however Rouzineau, Prevost and Meyer<sup>17</sup> showed that by taking reasonable values for the Murphee efficiencies one can get similar simulation results in equilibrium models and rate-based models. They also pointed out that if obtaining reasonable Murphee efficiencies is difficult then it will also be problematic to predict some of the rate-based model parameters. While the critical factor is to obtain a good description of vapor-liquid equilibria most real distillation columns, both trayed and packed, can be modeled via stage equilibrium models.<sup>6</sup>

Using rate-based or equilibrium-based models is only one of the assumptions for modeling a process. Other assumptions will also have to be made which has a direct impact on numerical solvability of a topic. Since a dynamic model is a set of DAE (Differential Algebraic Equations) some sets of assumptions might not lead to a solvable model as there may be index (See Appendix for definition) problem.

The index problem was first identified by Petzold (1982), followed by Gear (1988). The problems of solving of a dynamic process model arises with DAE of index 2 or higher. Brenan, Campbell and Petzold<sup>18</sup> pointed out, that the numerical solution of these types of systems has been the subject of intense research in the past few years.

While dealing with high index DAE systems has been a topic of intensive research since index problem was first determined, it is possible to completely avoid this problem by proper modeling and nobody wants a high index DAE system in the first place.

Ponton and Gawthrop<sup>19</sup> outlined a systematic approach for building VLE systems while avoiding index problems. They pointed out that while building a model for the same system, some form might lead to high index problems while others may not. The work presented here also ensured to use the set of assumptions so that the resulting system is not higher index. The approach taken by them suggests that the extensive balance equations should be combined to eliminate interphase flow variables.

Stadtherr and Lefkopoulos<sup>20</sup> developed an algorithm for choosing the independent variables and equations from the given set of equations and assumptions that will

eventually lead to formation of an index one DAE systems. Assuming an efficient maximum transversal finding algorithm is available they try to find out appropriate nonsingular subset of equations and variables leading to index one DAE. In part two of the same<sup>21</sup> the authors presented strategies of predicting the overall index of a system given the indices of the building blocks of the system.

Moe in his Ph.D. dissertation<sup>22</sup> presented his study on modeling and index reduction. His work mainly focused on formulating solvable process models and manipulating models into a more manageable form. A modeling method for developing low index models was presented along with two index reduction algorithms. In the same year Moe, Hauan, Lien and Hertzberg<sup>23</sup> applied a modeling method for modeling a system having both phase and reaction equilibrium which guaranteed the resulting model to be semi-explicit index one. They also looked into the initialization methods of the DAE system.

Hangos and Cameron<sup>24</sup> presented a formal way of representing modeling assumptions in order to analyze the effect of modeling assumptions on a model in a more rigorous way. Their idea was to define the ‘smallest indivisible syntactical element which is regarded as an assumption’ as an assumption atom and so an assumption is a combination of assumption atoms. A set of assumptions is then the combination of different assumptions. They proposed that by decomposing a set of assumptions one can check that whether the given modeling assumptions contains any formal contradiction, i.e. contradicting pair of assumption-atoms or contains any assumption-atom more than once. Later<sup>25</sup> the authors came up with few theorems based on the same ideas. They talked about assumption transformation which allows one model of an equivalence class to move to another model equivalence class. A hierarchy in modeling assumptions was also defined by them from which one can have an idea, qualitatively, about the degree of influence, a change in assumption of different level can have on the model.



## **Control Configuration of a Distillation Column**

### **1. Overview**

As pointed out<sup>26</sup> distillation is probably the most studied unit operation in terms of control. Control of distillation columns refers to the ability of keeping certain variables at or near their setpoints whenever there is a disturbance or set point change in the plant. Many papers and books have been devoted to the investigation and exploration of different aspects of distillation column control over the last half century<sup>26</sup>. The procedure for determining which process variables should be controlled by manipulating certain values is called is control strategy design<sup>27</sup>. Dynamic simulations can be used to provide a picture of how the plant it will behave when there is a set point change and disturbances.

Controller system design can be broken into following steps.

- Formulate control objective
- Identify controlled and manipulated variables
- Choose a control strategy and structure
- Specify controller settings.

The control objective can generally be formulated based on safety concerns, environmental regulations, and economic objectives. In this particular work it is mainly driven by environmental regulations and economic considerations.

Of the steps involved in designing a control strategy for a distillation column identifying controlled and manipulated variables is one of the first steps. The controlled and the manipulated variables are paired based upon the sensitivity between them. Conventionally PID controllers are implemented between these controlled and manipulated variables to get the closed loop plant. Though they tend to work satisfactorily, more complex and advanced control strategies like cascade control, feedforward control are also sometimes used. The selection of variables also depends on the control strategy to be used.

## **2. Simulation of the column for variety of inputs**

To determine the appropriate control structure of a distillation column as discussed the first step is to determine the controlled and manipulated variables. As Moore (1992) pointed out this selection fundamentally defines the structure of the control system and is critical to the success of the same. Choosing the controlled variables depends upon the system and its objective and generally it has to do with ensuring product quality. Selection of manipulated variables depends upon deciding which valves will be used for inventory control and which ones should be used for controlling the separation<sup>28</sup>. Flow control of material and energy streams comes under inventory control. This control mostly deals with the degree of separation taking place in the column.

Once the set of controlled and manipulated variables has been chosen it is necessary to run a number of simulations of the dynamic column via step changes in the manipulated variables. The responses of the controlled variables due to the change in manipulated variables will be recorded. The output from this step serves as an input to the next step, as the data is taken and analyzed to determine the range for which a linear fit is appropriate. In fact this is the main reason for simulating step changes of different magnitude.

## **3. Limitation of linear transfer function**

In order to determine the transfer function relationships between input and output, step changes in the input/manipulated variables in both directions ( $\pm$ ) and of different magnitudes are simulated and the response of the output variables are used to determine the range in which changes in the manipulated variables can be fit by linear relationships. For example for a similar amount of change in a manipulated variable but of different directions can result in a response of the controlled variable in one direction which is approximately the mirror image of the other. In this case fitting a linear transfer function is appropriate. However if the two responses are completely different in nature from each other then this is not a good assumption. For large changes in the manipulated variables such a case can often be observed. Another way to judge this could be to

determine if the superposition principle holds in the response of the controlled variables. For example for a step change of magnitude 3, the response has to be the same as that of the addition of the two different responses with change of magnitude 1 and change of magnitude 2 of the manipulated variable for a linear model. This step identifies the range in which fitting the transfer functions will be valid.

#### 4. Identification of input-output relationships via transfer function models

The transfer function is an expression which dynamically relates the input and the output in a process model.  $Y(s) = G(s) U(s)$  where  $Y$  is the output,  $U$  is the input and  $G$  is the transfer function relating them. So if a transfer function is known between one input and one output, the change in the output can be computed for a change in the input. One important property of the transfer function is that one can calculate the steady-state change in output given a change in input by directly setting  $s = 0$  in  $G(s)$ . Another important property of transfer functions is that they can be added. A single process output variable can be influenced by more than one input variable. The total output change is calculated by summing up the changes of the output if only one of the inputs were changed at a time.

$$Y_i(s) = \sum_j G_{ij}(s) U_j(s)$$

where  $Y_i(s)$  is the  $i^{\text{th}}$  controlled variable,  $U_j(s)$  is the  $j^{\text{th}}$  manipulated variable and  $G_{ij}(s)$  is the transfer function between the  $i^{\text{th}}$  controlled variable and the  $j^{\text{th}}$  manipulated variable.

#### 5. RGA analysis

A systematic approach for the analysis of multivariable process control problems was developed by Bristol<sup>29</sup>. His approach provides two important pieces of information.

- A measure of process interaction.
- A recommendation concerning the most effective pairing of controlled and manipulated variables can be done based on the information so that feedback controller can be designed accordingly.

A process where  $n$  controlled variables and  $n$  manipulated variables are present, the relative gain  $\lambda_{ij}$  between a controlled variable  $y_i$  and a manipulated variable  $u_j$  is defined as the dimensionless ratio of two steady-state gains:

$$\lambda_{ij} \triangleq \frac{(\partial y_i / \partial u_j)_u}{(\partial y_i / \partial u_j)_y} = \frac{\text{open-loop-gain}}{\text{closed-loop-gain}}$$

for  $i = 1, 2, \dots, n$  and  $j = 1, 2, \dots, n$ .

The relative gains are then arranged in a convenient way to construct the relative gain array (RGA) denoted by  $\wedge$ .

$$\wedge = \begin{pmatrix} \lambda_{11} & \dots & \lambda_{1n} \\ \vdots & \ddots & \vdots \\ \lambda_{n1} & \dots & \lambda_{nn} \end{pmatrix}$$

After simulating the step tests and fitting the transfer functions, they can be used to find the RGA.

$$\wedge = \lim_{s \rightarrow 0} \begin{pmatrix} G_{11}(s) & \dots & G_{1n}(s) \\ \vdots & \ddots & \vdots \\ G_{n1}(s) & \dots & G_{nn}(s) \end{pmatrix} X \left( \begin{pmatrix} G_{11}(s) & \dots & G_{1n}(s) \\ \vdots & \ddots & \vdots \\ G_{n1}(s) & \dots & G_{nn}(s) \end{pmatrix}^{-1} \right)^T$$

where X refers to element by element multiplication. After the RGA has been computed, the recommended pairing of controlled variables and manipulated variables are computed so that the corresponding relative gains are positive and as close to one as possible. It should also be kept in mind that there should be one to one pairing of controlled variables to manipulated variables.

## 6. Controller settings

Once the optimal pairing has been found, and transfer functions have been fitted between the paired variables, the controller can be designed. Designing feedback controllers based on IMC tuning is commonly performed in practice. There are also other ways of designing controllers based on available process model like direct synthesis method or tuning relations based on criteria like IAE (Integral Absolute Error), ISE (Integral squared error) or ITAE (Integral time-weighted absolute-error).

## CHAPTER III

### DEVELOPMENT OF DYNAMIC MODEL

A steady state model was derived at first from first principles of an equilibrium-based model and was implemented in gPROMS<sup>30-34</sup> and the physical properties of the fluid streams were computed by MULTIFLASH<sup>35</sup>. This gPROMS/ MULTIFLASH combination simulates equation based models which makes it a very flexible and a powerful software modeling software package. Based on the steady state model properties a dynamic model of the column was derived. For the process under investigation the dynamic model consisted of more than 1000 differential variables and 10,000 algebraic variables. UNIQUAC was used for calculating the liquid properties. The weir dimensions are given as they are generally found<sup>36</sup> in a regular distillation column. The set of equations for a single tray is provided in the Appendix.

#### **Assumptions for the Model**

As discussed in the last chapter making the right assumptions are important for generating a model with desired properties.

The assumptions for modeling the steady state column were chosen to be:

- Equilibrium based model.
- Ideal gas law to describe gas properties.
- Murphee efficiency has been assumed to be equal to 1.
- The reaction rate constant follows an Arrhenius equation.

The steady state model was then implemented one tray at a time. Because of large number of equations in the steady state model writing the whole model at one go and then trying to run it was not a good idea. A single tray of a particular section of the model was implemented. After the simulation of the tray for the given input streams, a tray was added to the top of it. The input streams to this combination of two trays were the same as that for the single tray simulated previously. The simulated values obtained

from the first run were made guess values for both the trays. The whole model was built in this fashion by adding new trays on top of the simulated ones and giving guess values. The guess values given were the simulated values of the tray immediate next to the new tray. Once the steady state model was up and running, it helped to rule out some of the conventional assumptions of building a dynamic model. The couple of conventional assumptions<sup>37</sup> which were ruled out based upon steady state simulated values are:

- Equimolal overflow. This assumption means that whenever one mole of vapor condenses, it vaporizes a mole of liquid. It implies that the vapor and liquid rates through the stripping and rectifying section will be constant under steady state condition. However since the dynamic model will essentially reach steady state the validity of this assumption was checked from the values obtained from the steady state model and was found was far from being valid. The reason can be attributed to the presence of the reactive zone in the column. Investigation of the molar liquid and vapor flow values at those zone big discontinuities of the assumption was observed. Similar point was noted in the feed tray.
- Negligible vapor hold up. This assumption means that the molar vapor rate through each tray in the column is same. Investigating the steady state model vapor flow rate values, it was observed that again the values were far from being inline with the assumption. Big discontinuities were observed in vapor molar flow rates near the reactive zone and feed trays.

The additional assumptions from the steady state model for modeling the dynamic column were chosen to be:

- All liquid phases are modeled with dynamic balances. However, in the condenser however no dynamic component balance was included as only the liquid height in the reflux drum was modeled dynamically.
- No dynamics in the vapor phase in the condenser due to negligible vapor holdup in the condenser. All 70 trays and the reboiler are modeled as having dynamic vapor holdup.
- Liquid flow from the trays are governed by Francis weir formula

- Vapor flow rate is governed by the pressure drop between two consecutive trays.

### Simulation Results

Once the model had been implemented, it has been validated in simulation and compared against available data. As discussed first a steady model was built in gPROMS and based upon the results a dynamic model was built. The steady state values were computed to be as given in Table 2.

**Table 2: Steady State Results**

Stream Name Stream Description	Distillate Draw	BTM	Vent
Phase	Liquid	Liquid	Vapor
Temperature (F)	68.46	431.33	68.46
Pressure (psia)	115	115	115
Total Mass Rate (lb/hr)	60,318	139,672	2,012
N-BUT-01	0.0290	0.0000	0.1202
N-PEN-01	0.2550	0.0000	0.2938
2,3-D-01	0.0320	0.0000	0.0169
3-MET-01	0.1630	0.0000	0.0759
N-HEX-01	0.0980	0.0000	0.0321
BENZE-01	0.0004	0.0000	0.0001
CYCLO-01	0.2830	0.0000	0.0613
3-MET-02	0.0658	0.0001	0.0090
2,4-D-01	0.0658	0.0000	0.0147
N-HEP-01	0.0058	0.0118	0.0005
TOLUE-01	0.0000	0.4287	0.0000
M-XYL-01	0.0000	0.3150	0.0000
ISOPR-01	0.0000	0.2434	0.0000
HYDRO-01	0.0000	0.0000	0.3755
METHYLCYCLOHEXANE	0.0001	0.0009	0.0000

Condenser Duty (MM BTU/HR) = - 64.35

Reboiler Duty (MM BTU/HR) = 40.94

The results closely match the steady state simulation results computed from ASPEN and data provided by CD Tech. The results show that the benzene mass fraction from the

reboiler liquid product is 0.0, and the fraction in the condenser liquid product is 0.0004. Since both of these values are very low, the objective of removing benzene from the reformat stream can be achieved at steady state. At the same time the results show that the toluene mass fraction in the liquid product from the reboiler is 0.4287, as virtually no toluene is found in the top product stream. Since the steady state simulation met the goal, the next step was to implement a dynamic model. The feed composition and temperature are considered to be disturbances of the process as it is inline with a real situation.

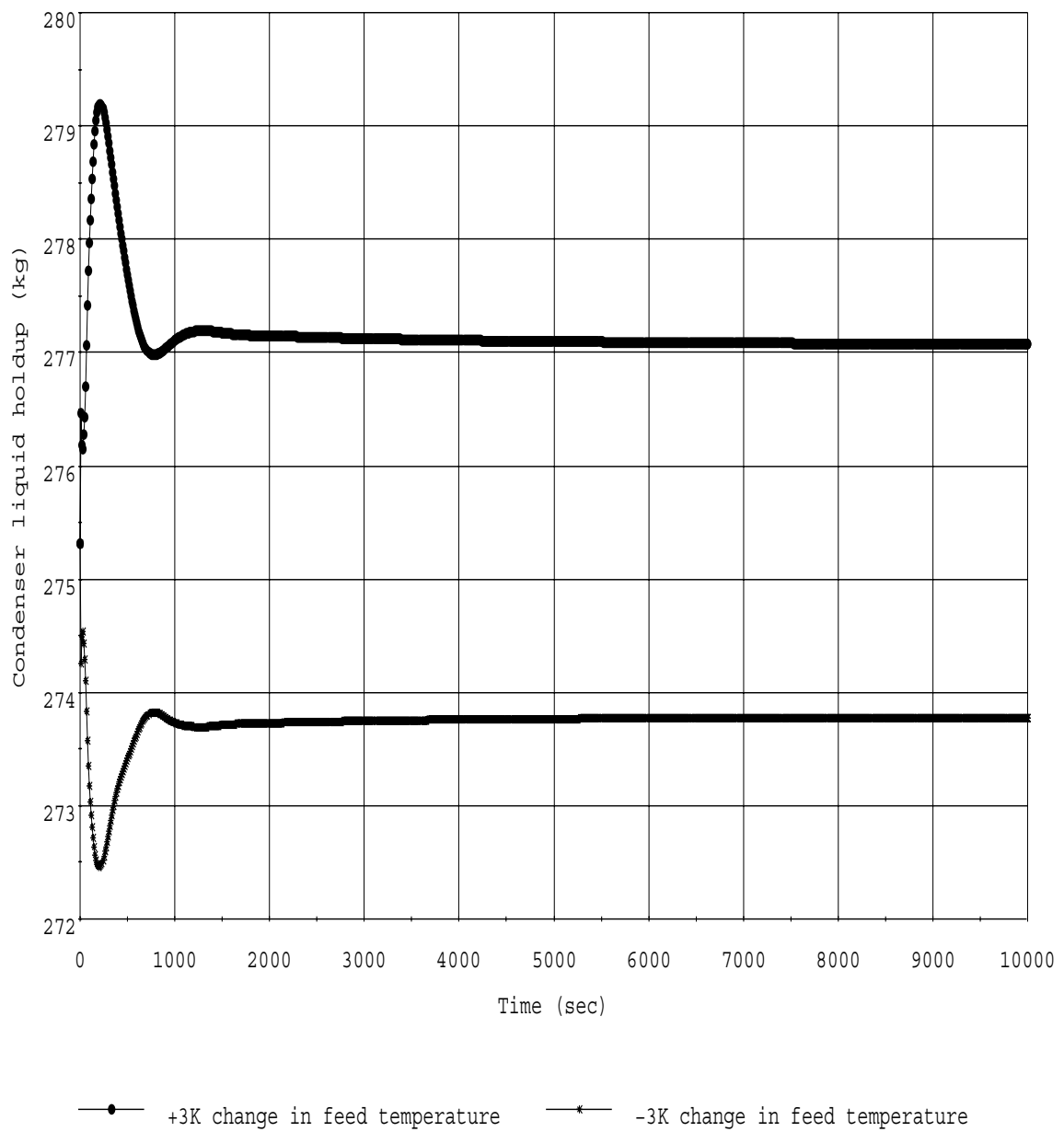
The dynamic model of the reactive distillation column was then simulated and different disturbance in the feed were simulated. Also it gives an idea how much time it takes to reach to the new steady state for the given disturbance. It was observed that different variables take different amount of times to reach the new steady state. Also it was observed that not every disturbance results in a linear response. Figures showing the responses of the four different process variables for load changes are given below. The variables whose responses are shown

- Liquid holdup in the condenser
- Liquid holdup in the reboiler
- Benzene mass fraction in the reboiler
- Temperature of the first tray from top

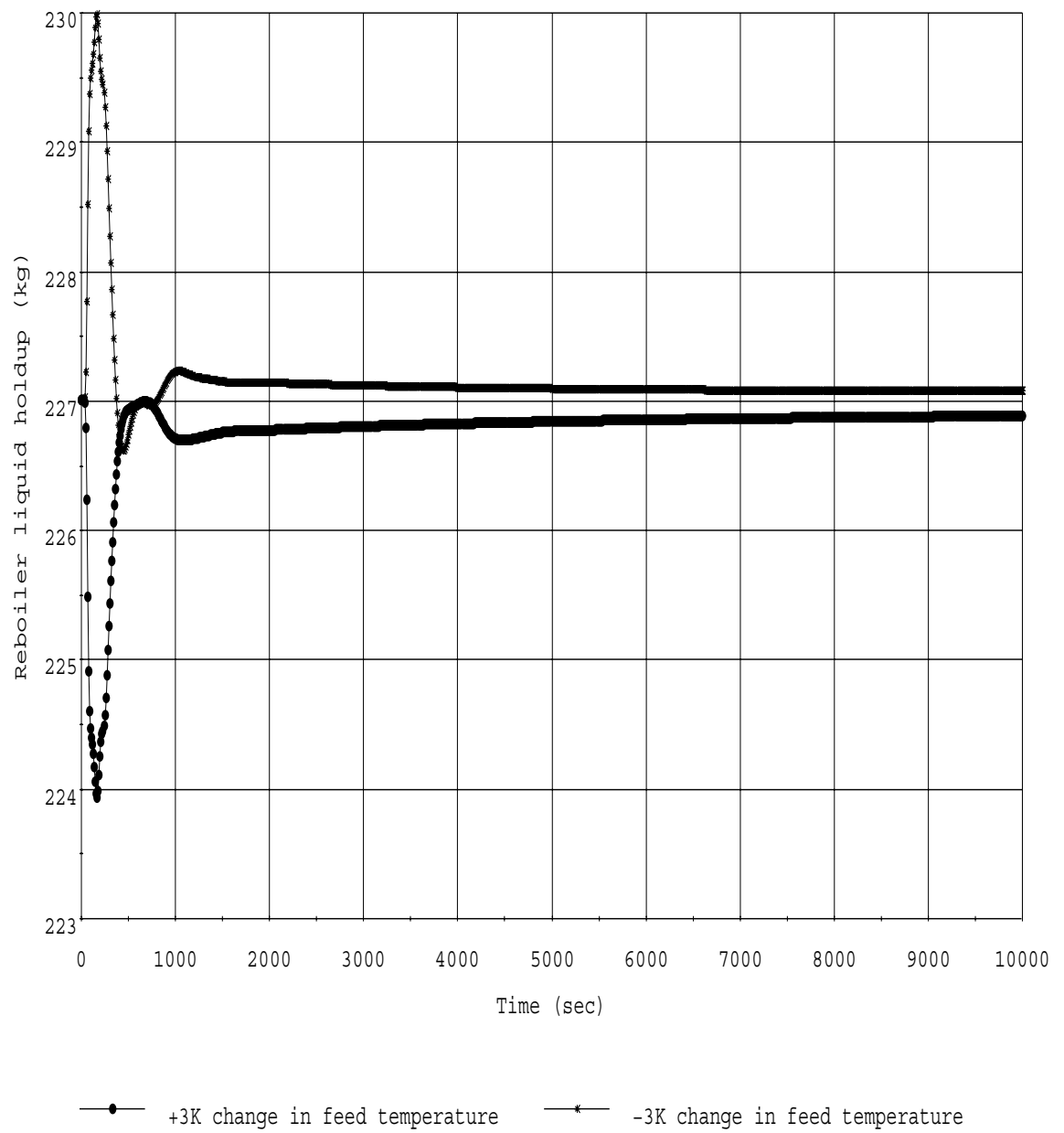
### **1. Change in feed temperature**

The column was simulated given a  $\pm 3\text{K}$  change in the feed temperature. Figures 4 to 7 shows the effect on the different variables considered here. In the first figure an increase in feed temperature should result in an increase in condenser temperature and should result in more vapors flow in the vent and the liquid holdup should decrease.

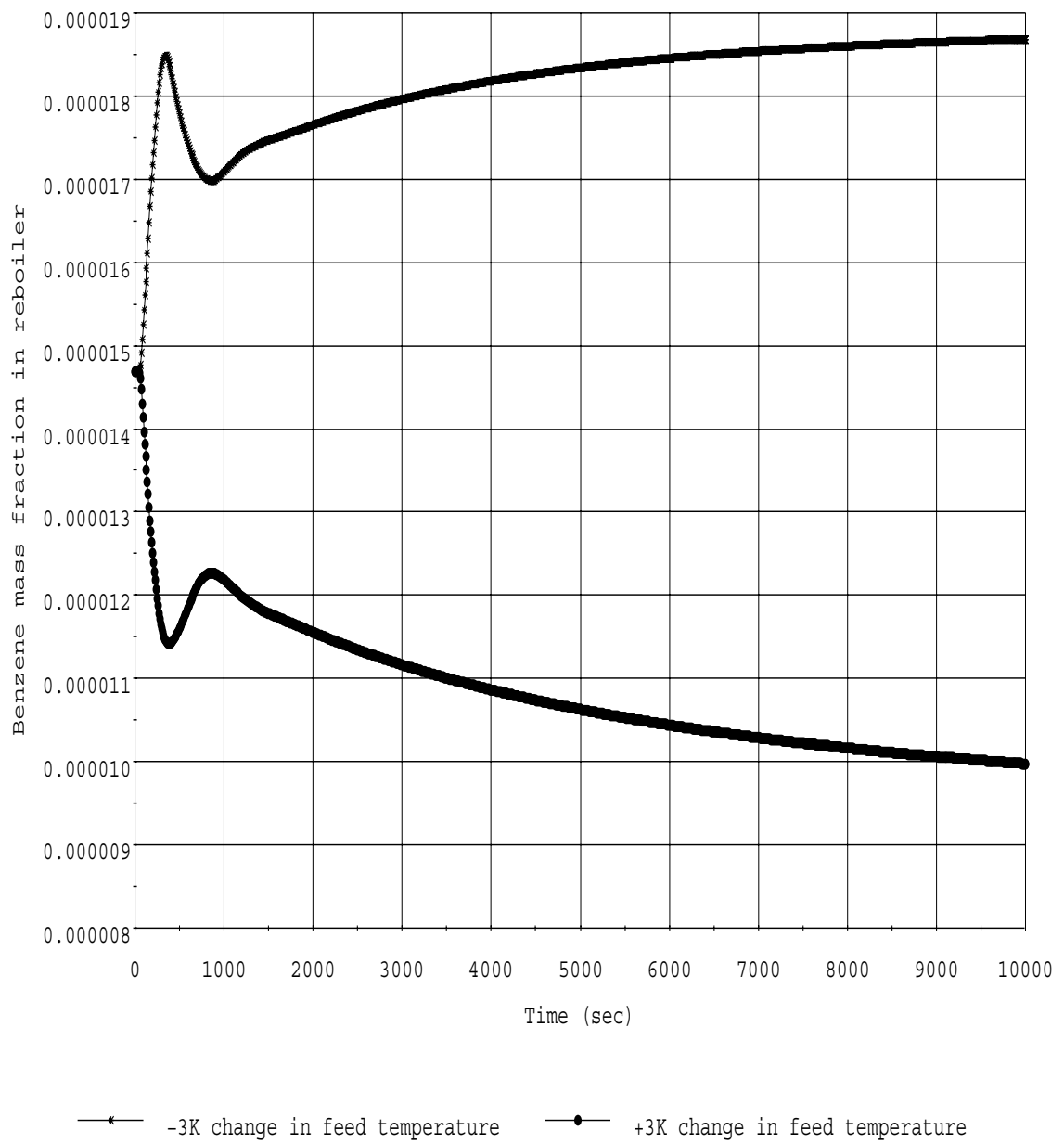




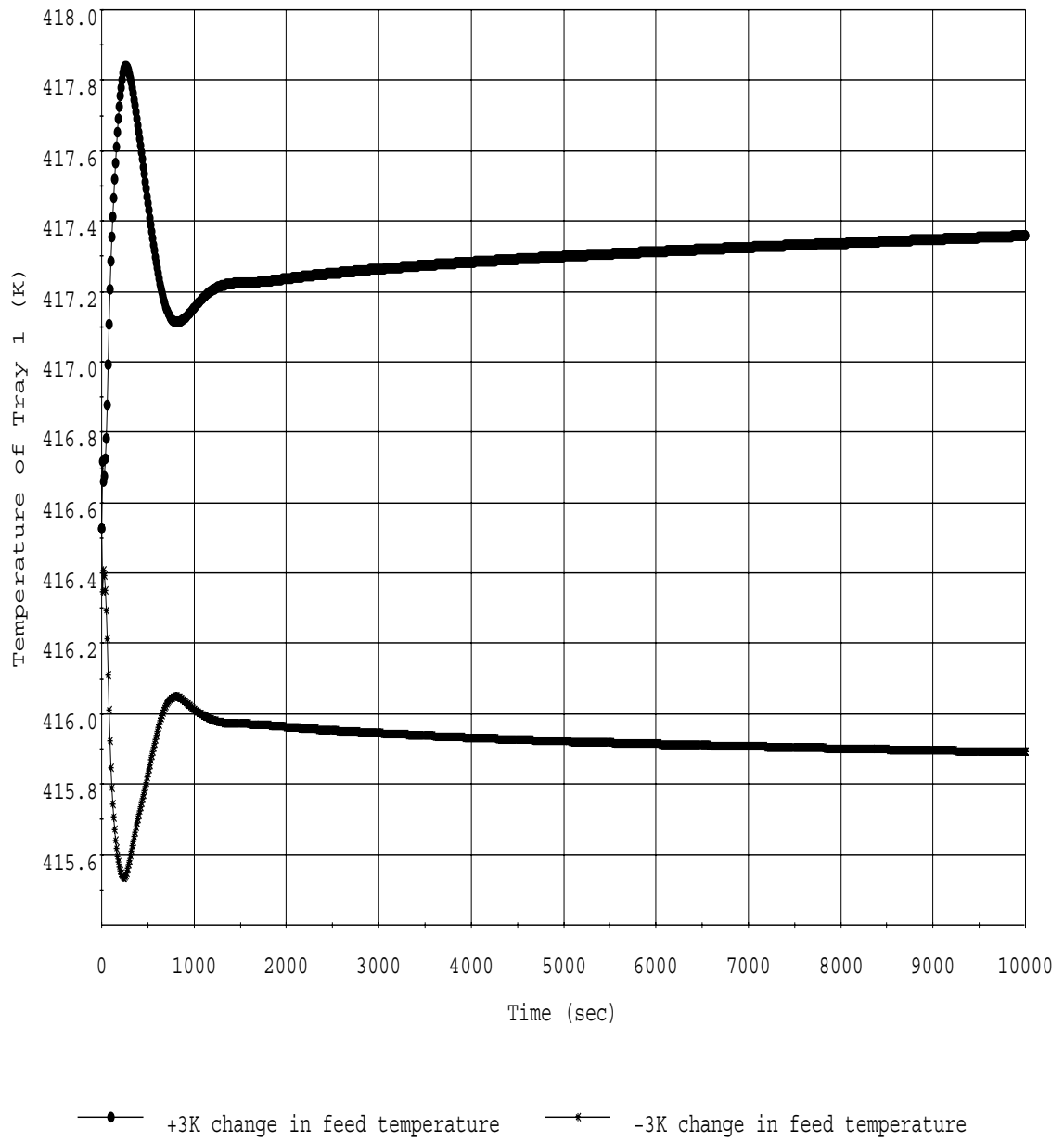
**Figure 4: Response of condenser liquid holdup due to feed temperature change**



**Figure 5: Response of reboiler liquid holdup due to feed temperature change**



**Figure 6: Response of reboiler benzene mass fraction due to feed temperature change**

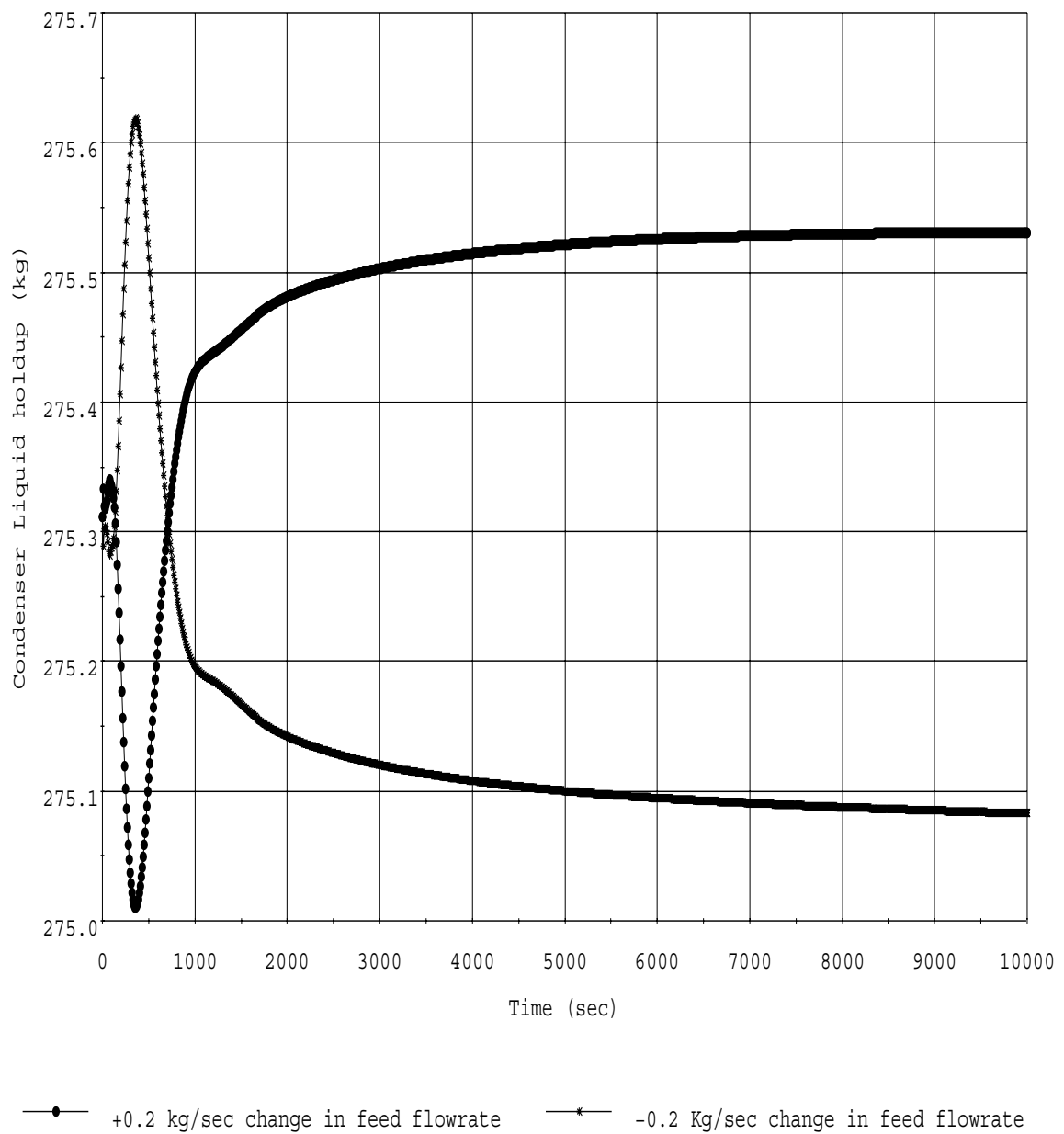


**Figure 7: Response of temperature of tray 1 due to feed temperature change**

A slightly nonlinear effect between the load variable and output variable has been observed. It is observed that though there is some effect of disturbance in feed temperature to condenser liquid holdup, there is hardly any effect of the same on reboiler liquid holdup. The time required to reach a new steady state is around 3 to 4 hours.

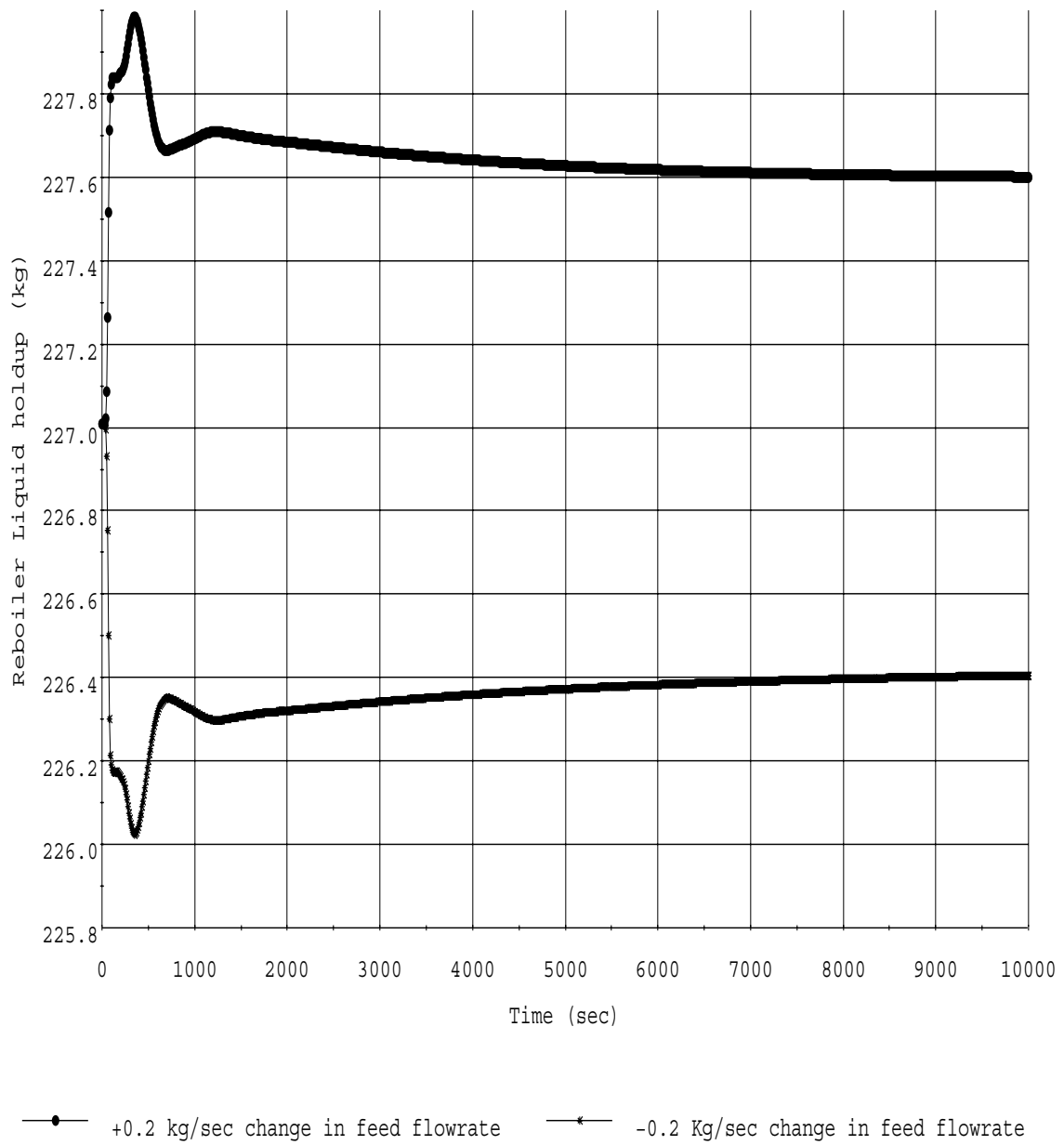
## **2. Change in feed flowrate**

The column was simulated given  $\pm 0.2\text{Kg/sec}$  change in the feed flowrate which means that the column was simulated from a value of 25.0 kg/sec to 25.4 kg/sec for the feed flowrate. Figures 8 to 11 shows the effect on the different variables considered here. Figure 8 shows the response of condenser liquid holdup, figure 9 shows that of the reboiler liquid holdup, figure 10 that of the benzene mass fraction in the product stream coming out of the reboiler and figure 11 shows the response of temperature of tray 1 due to the change in feed flowrate.

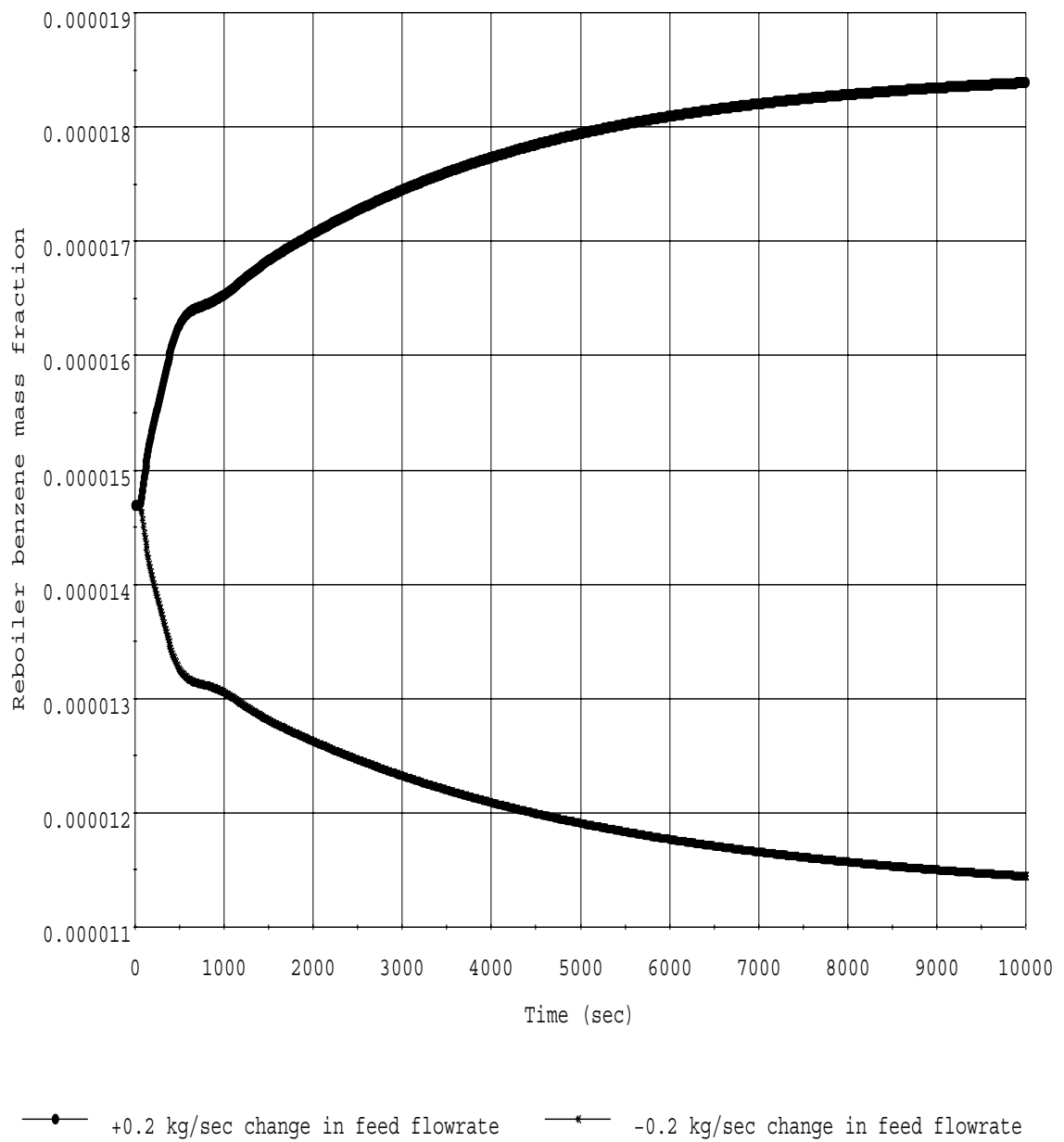


**Figure 8: Response of condenser liquid holdup due to feed flowrate change**

The feed flowrate has an effect on the reboiler liquid holdup as observed in figure 9.

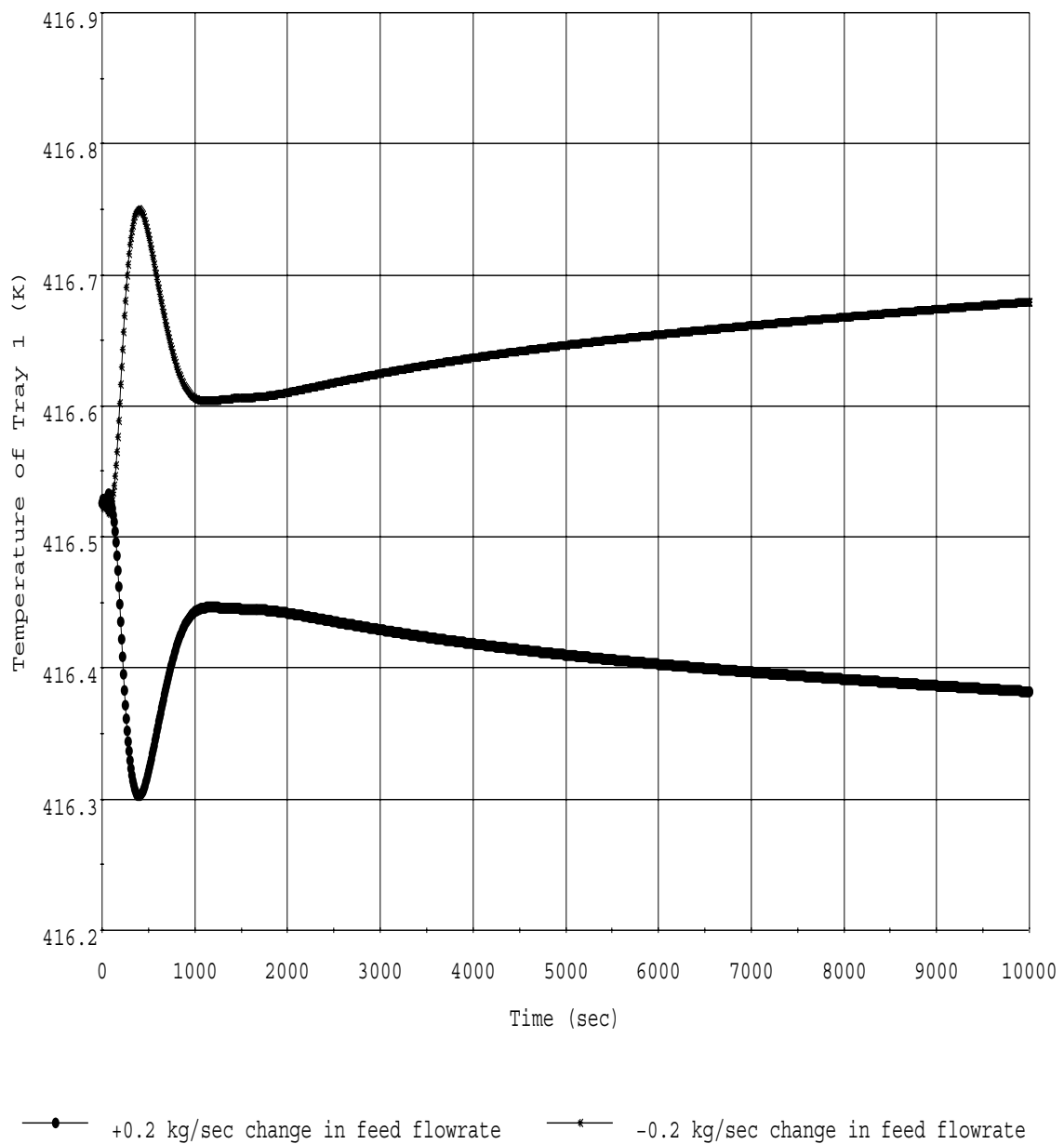


**Figure 9: Response of reboiler liquid holdup due to feed flowrate change**



**Figure 10: Response of reboiler benzene mass fraction due to feed temperature change**





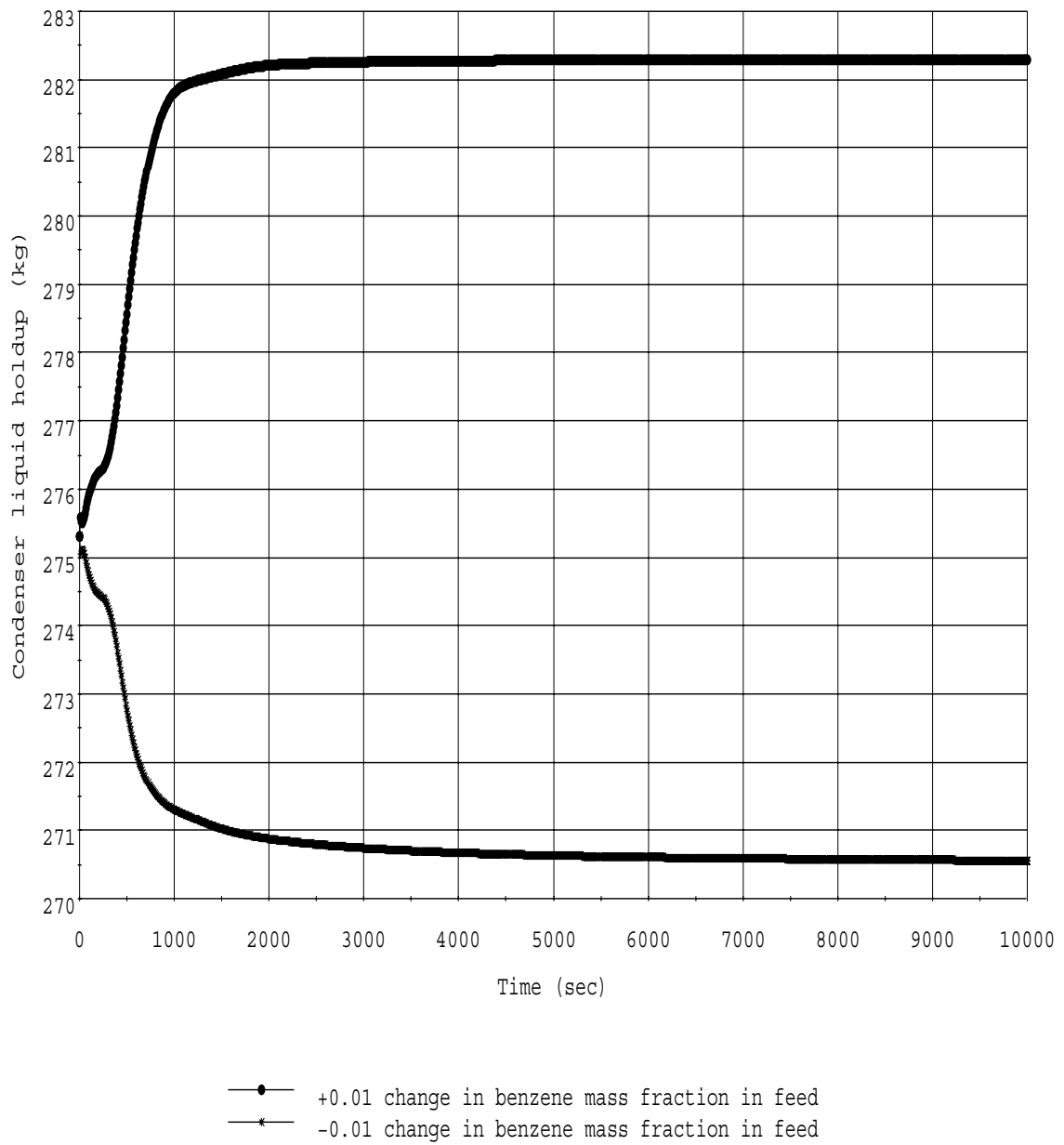
**Figure 11: Response of temperature of tray 1 due to feed temperature change**

### **3. Change in feed composition**

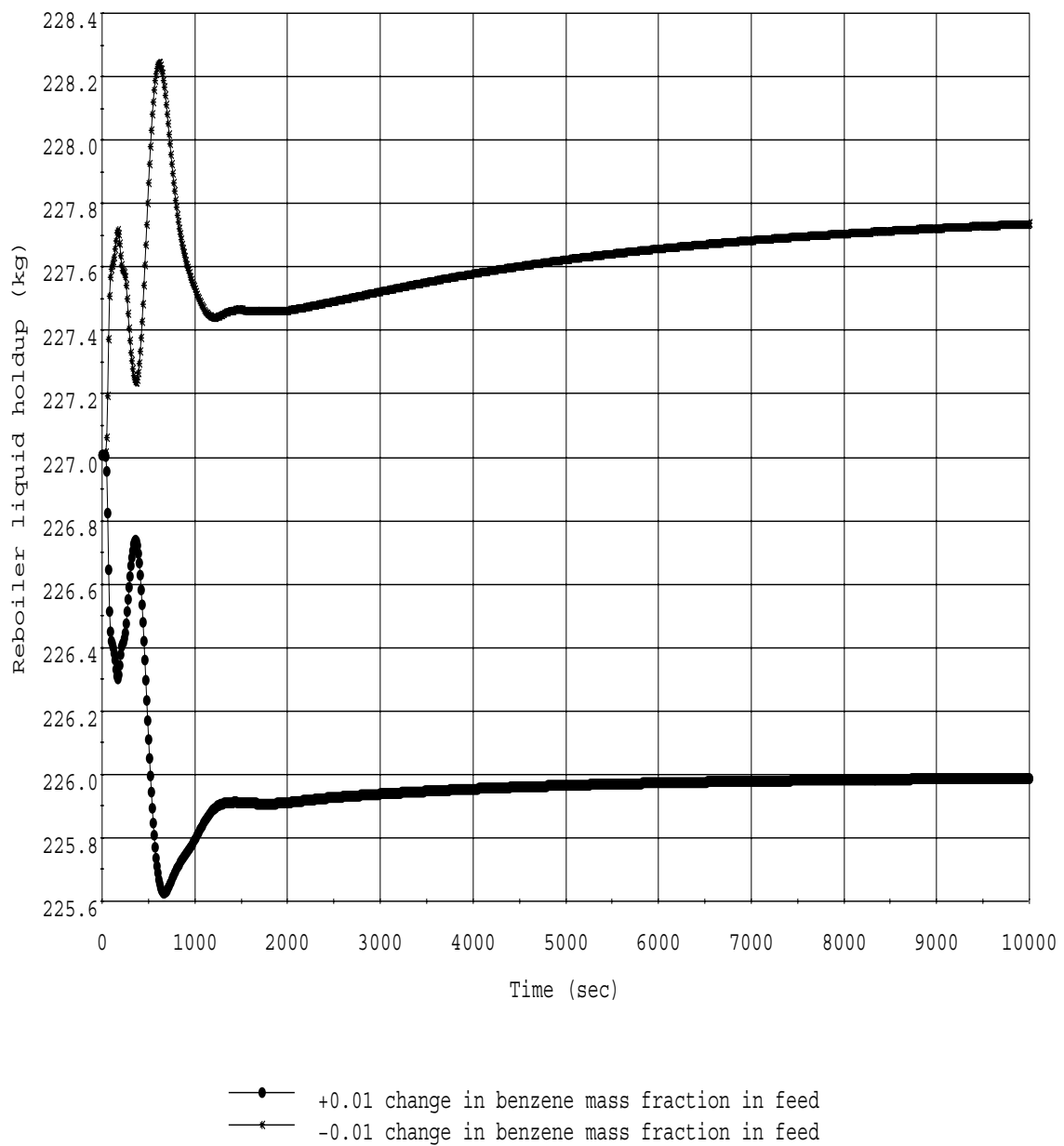
A change in feed composition was implemented and the response was recorded for the same four variables. The benzene mass fraction was changed by  $\pm 0.01$  amounts and the column was simulated. Figures 12 to 15 depict the responses. The two holdups showed linear relationship as opposed to the other two variables. Changing benzene composition also requires change in composition in one or many other components in the feed in order to maintain the sum of the mass fraction equal to one. In this case it was only adjusted with 2-methylhexane (decreasing benzene mass fraction by 0.01 leads to increase in toluene mass fraction by 0.01). Other adjustments in feed composition might lead to different responses and a different set of steady state values for the process variables.

### **4. Comments**

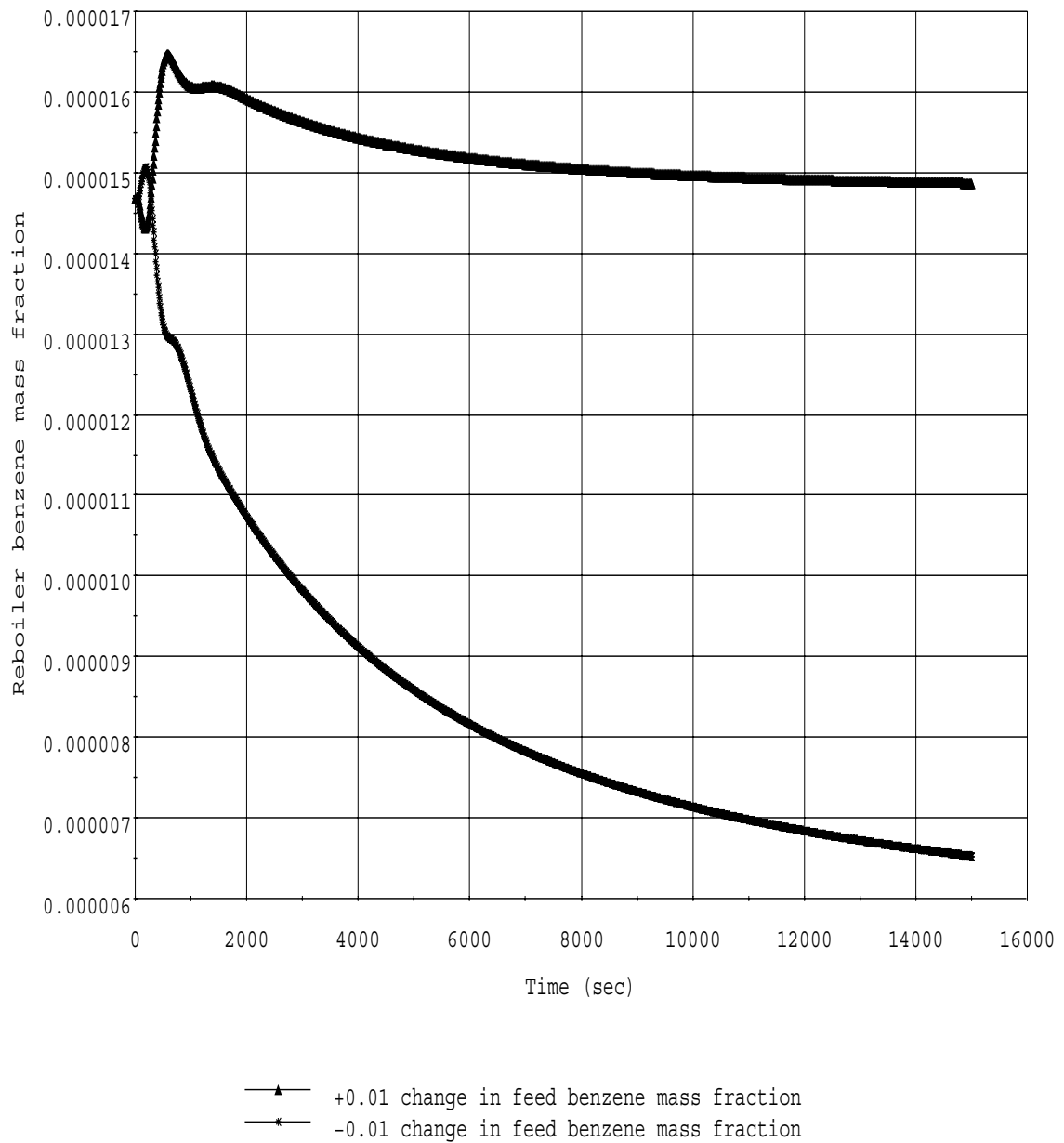
For all three types of disturbances it is observed that the column requires few hours to reach a new steady state. All the variables do not reach the new steady state at the same time and in general it is observed the temperature of tray 1 and the reboiler benzene mass fraction takes longer. From the figures it can also be seen, that the relationship between the output variable and the load variable is not always linear.



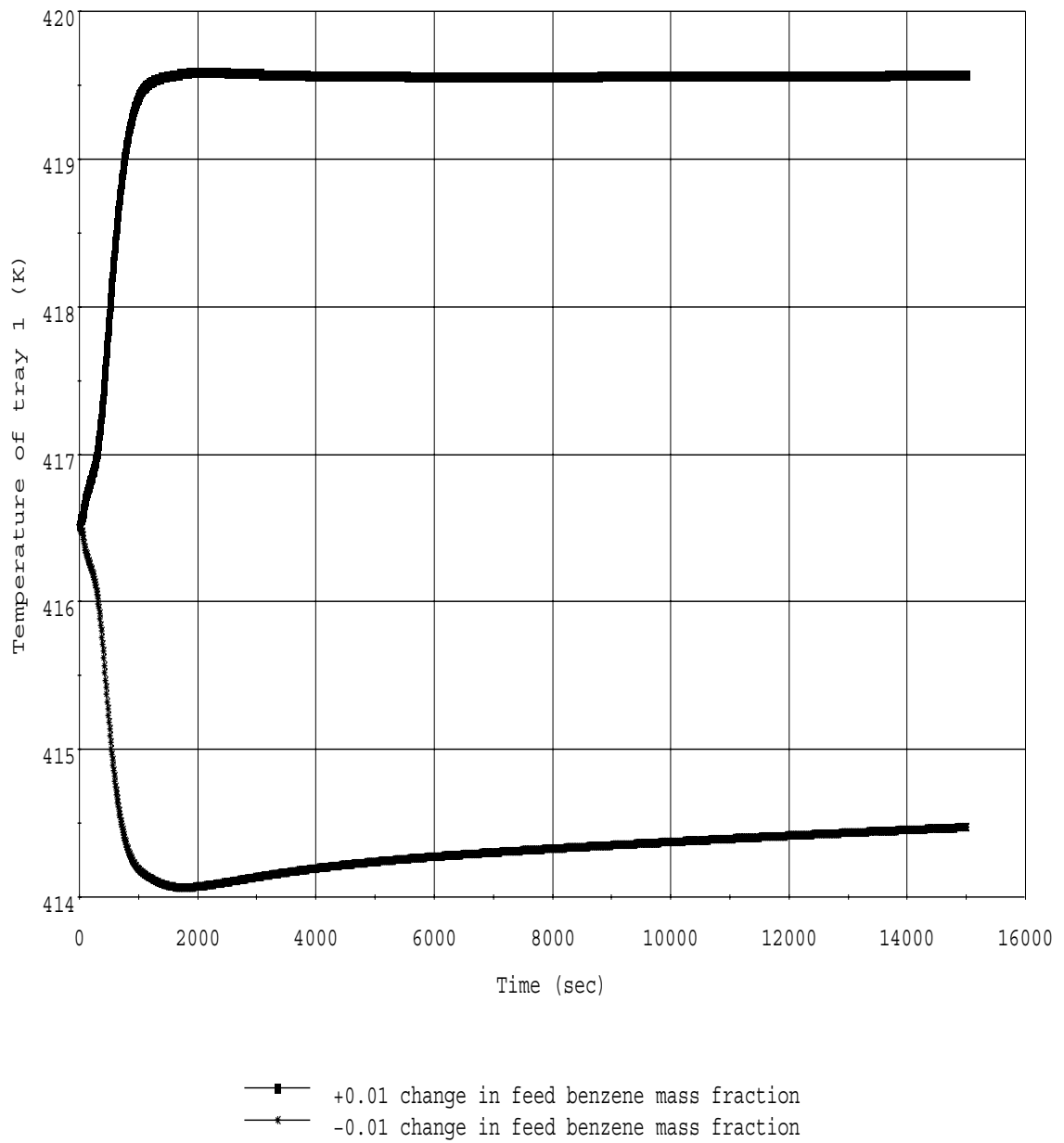
**Figure 12: Response of condenser liquid holdup due to feed composition change**



**Figure 13: Response of reboiler liquid holdup due to feed composition change**



**Figure 14: Response of reboiler benzene mass fraction due to feed composition change**



**Figure 15: Response of temperature of tray 1 due to feed composition change**

## CHAPTER IV

### CONTROLLER DESIGN

#### **Simulation of the Column for Variety of Inputs**

One of the major steps involved before going ahead with changing the input is to choose the controlled and manipulated variables from all available variables. A 5 X 5 control structure is often used in a distillation column, where the potential manipulated variables are the reflux flow or reflux rate, the distillate flow rate, the bottom flow rate from the reboiler, the reboiler boil up rate and the inert flow rate. The potential controlled variables for the same case are the reflux drum liquid volume, the reboiler liquid volume, the mass fraction of monitored component from reboiler and the mass fraction of another monitored component from distillate. For the present study reflux flow, distillate flow, reboiler liquid product flow rate and reboiler heat duty were chosen to be the manipulated variables as summarized in Table 3. The column pressure has been eliminated from the study as it is not effectively controlled by any of the other variables. The reflux drum liquid volume, reboiler liquid volume mass fraction of benzene in the bottom product from the reboiler and the temperature in the last tray in the reactive zone from top were chosen as controlled variables.

**Table 3: Controlled and Manipulated Variables**

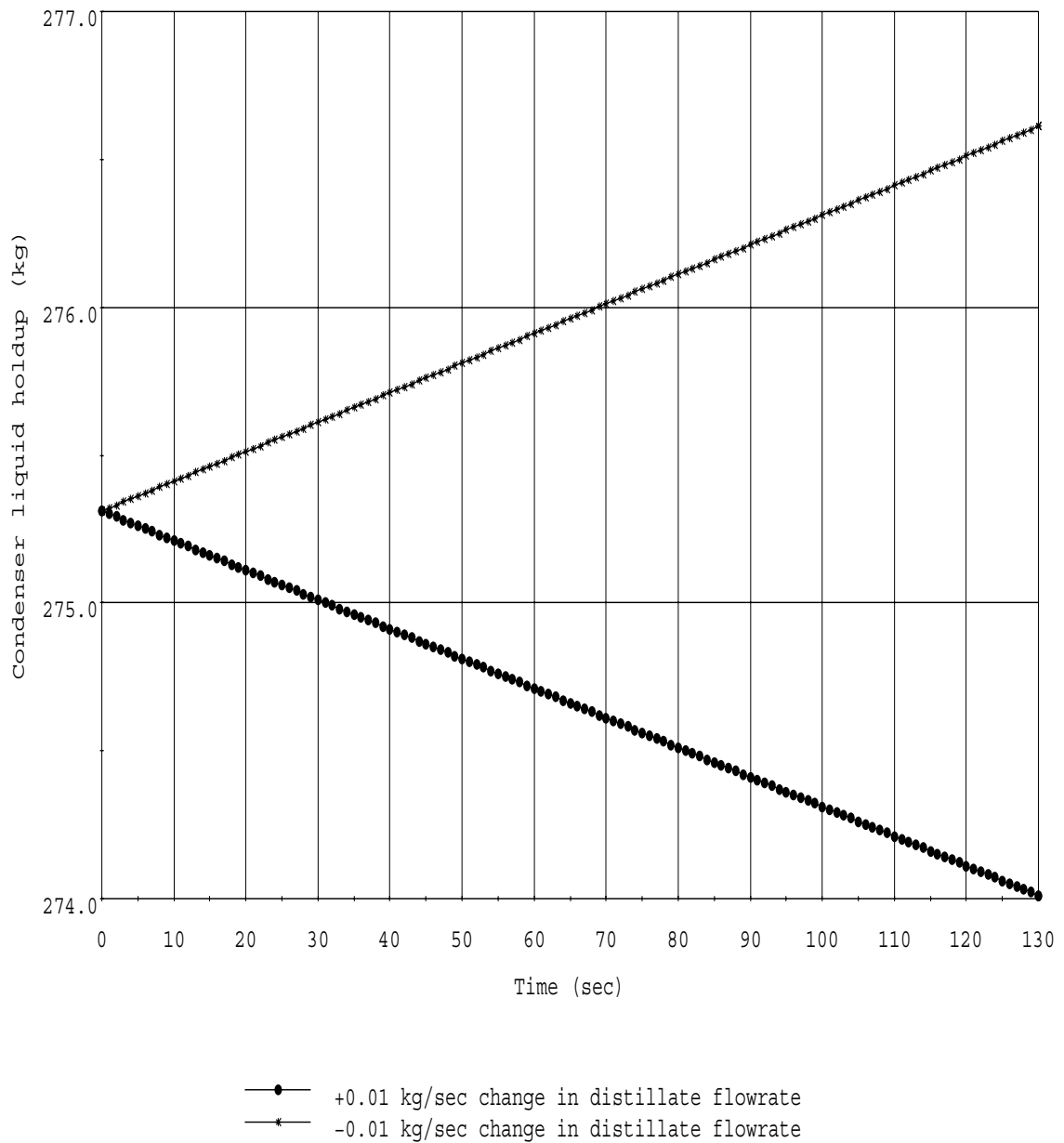
	Manipulated Variables	Controlled variables
1	Reflux flow rate	Reboiler liquid holdup
2	Distillate flow rate	Condenser liquid holdup
3	Reboiler heat duty	Mass fraction of benzene in bottom flow from reboiler
4	Bottom flow rate from reboiler	Temperature in the first tray of the column from top

The mass fraction of benzene was chosen because it is directly related to the objective of this work. The model will be given different step changes in the inputs, which are the manipulated variables. The changes in the manipulated variables will be in different directions and of different magnitude. The responses of the controlled variables due to the change in manipulated variables will be recorded. The output from this step serves as an input to the next step, as data is recorded and analyzed to determine the range for which a linear fit is appropriate. In fact this is the main reason for simulating step changes of different magnitude.

### **Limitations of Linear Transfer Functions**

In order to design the controller the transfer function fit should be done in a region where linear relationship holds between the manipulated and controlled variables. As discussed in Chapter II, this means that the two responses obtained for the same controlled variable for a given change in the manipulated variable in different direction should be approximately mirror image of one another. Figures 16 and 17 show two such cases. It can be seen that in both cases one plot is mirror image to the other which it should if the change give in the input is equal and opposite in nature. The distillate flow rate was changed by  $\pm 0.01$  Kg/sec.

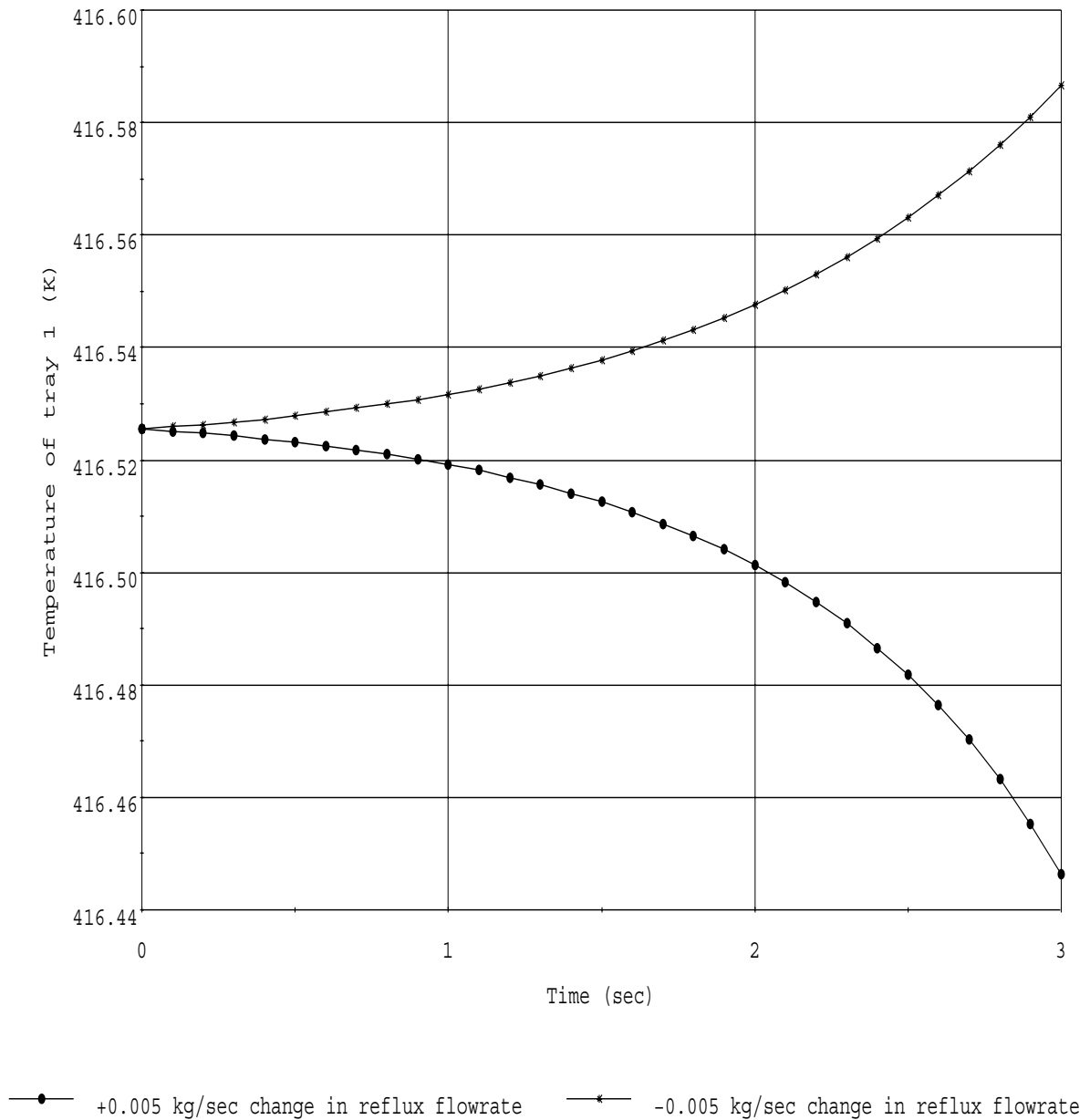




**Figure 16: Response of condenser liquid holdup due to change in distillate flow**

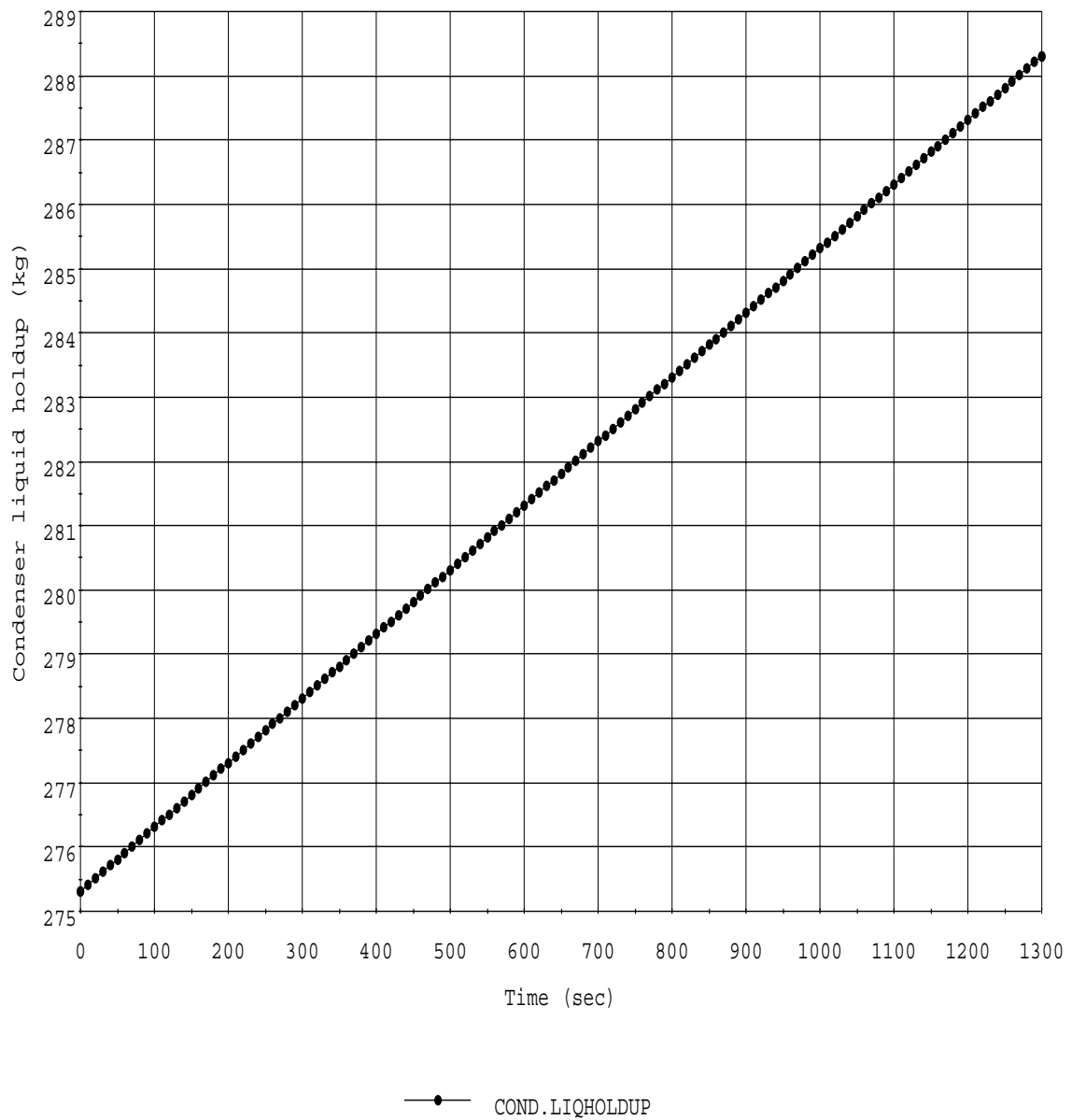
In Figure 17 the reflux flowrate was changed by  $\pm 0.005$  Kg/sec. These values however do not imply the limit for which linear relationship holds. In this particular case the

simulation could run only for a very short period of time as there were convergence problems.

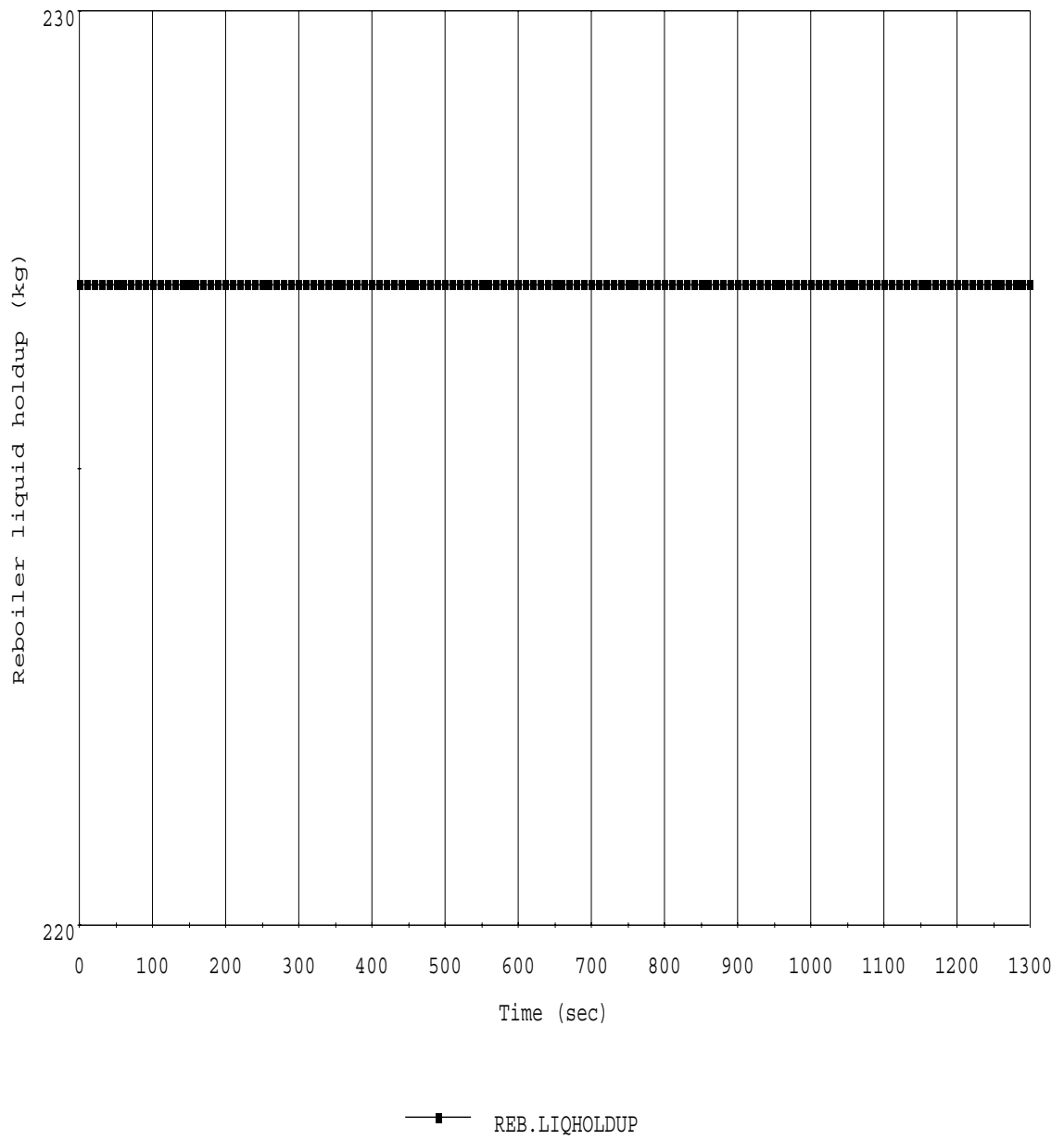


**Figure 17: Response of temperature of tray 1 due to change in reflux flow rate**

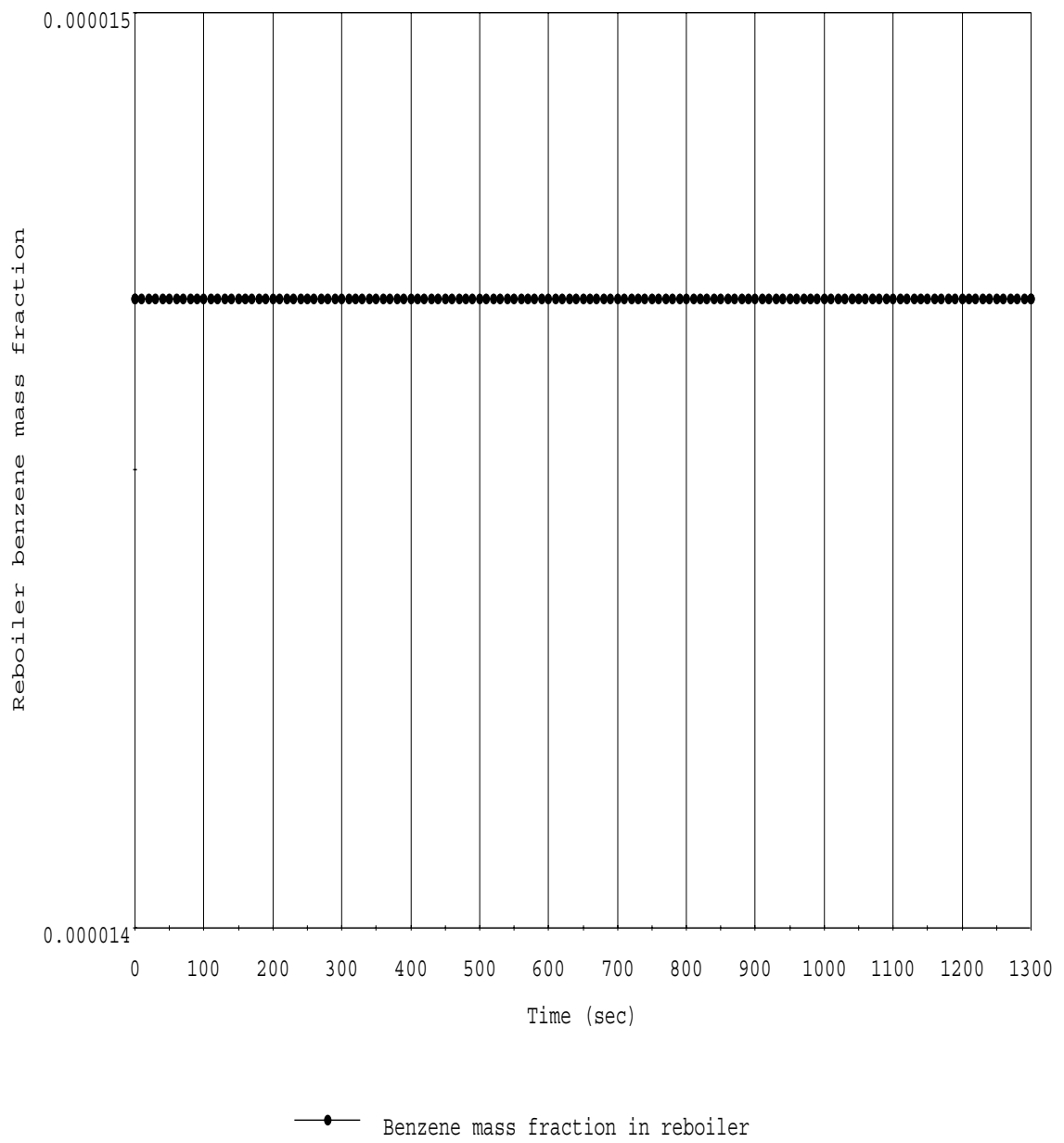
The same procedure was applied for all the other input/output variables to ensure that transfer function fitted correctly. Below are the responses of the different controlled variables due to change in different manipulated variables. Figures 18 to 21 show the response of the four controlled variables due to changes in the distillate flow rate.



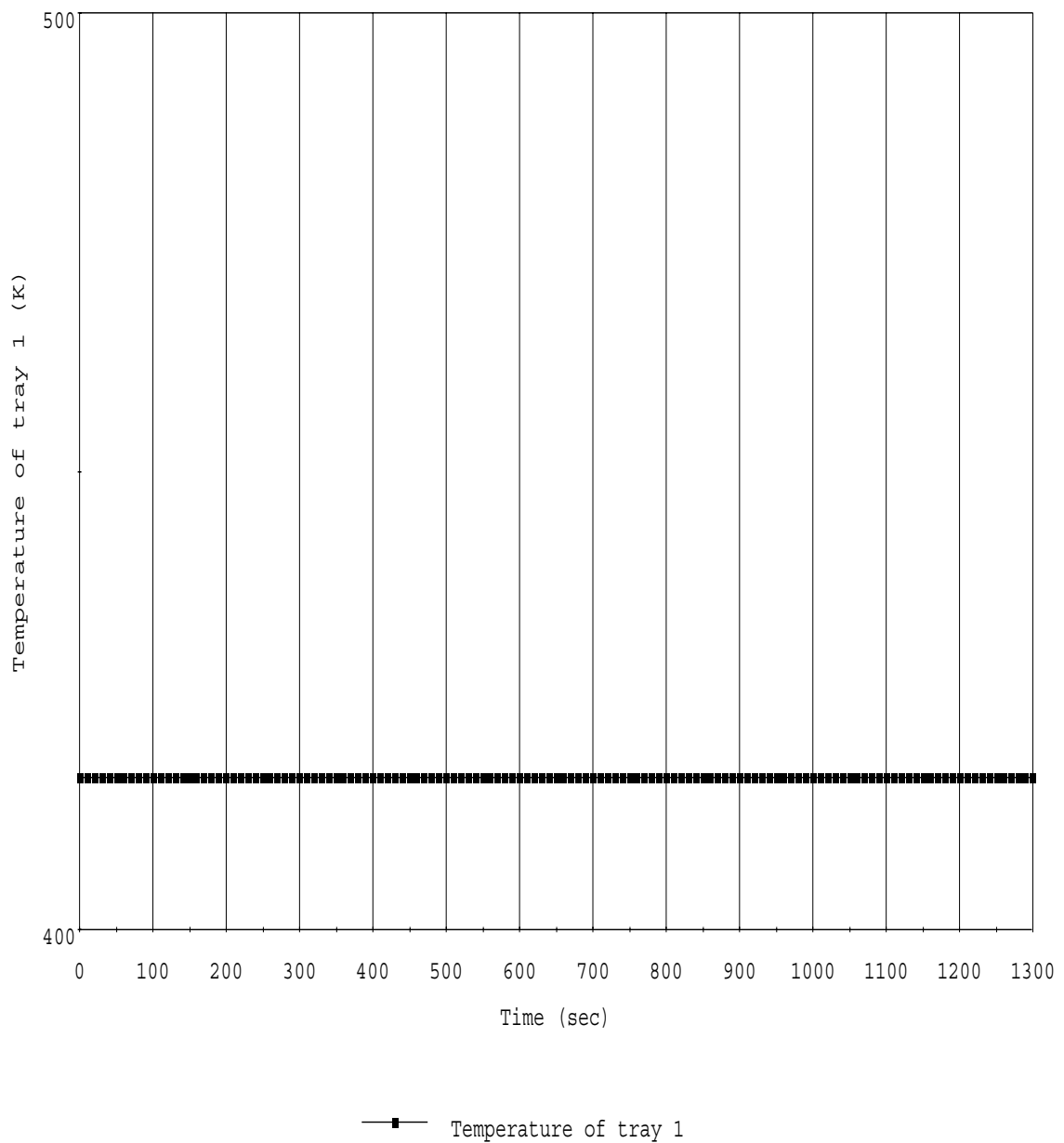
**Figure 18: Condenser liquid holdup (change in distillate flowrate)**



**Figure 19: Reboiler liquid holdup (change in distillate flowrate)**



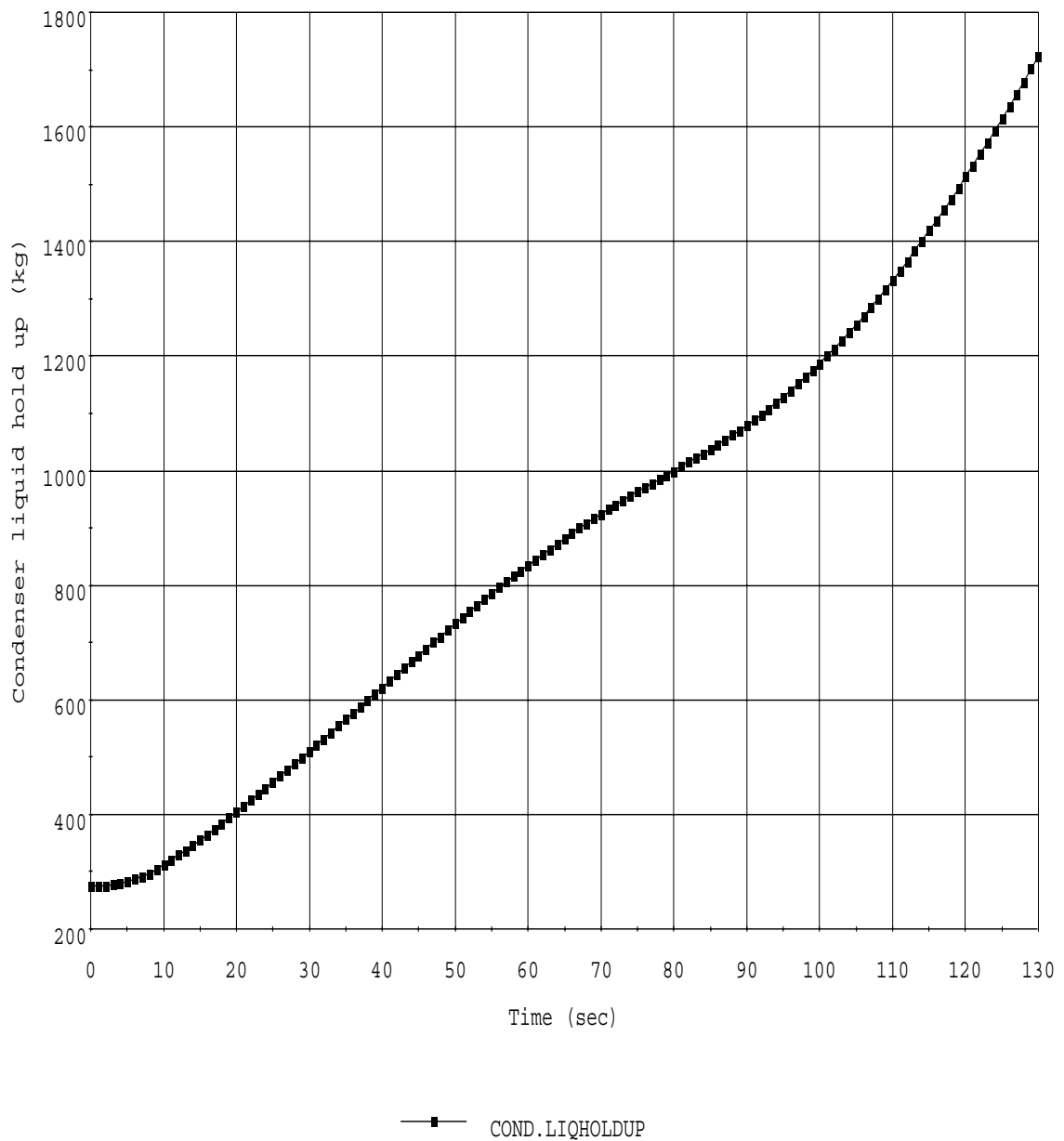
**Figure 20: Reboiler benzene mass fraction (change in distillate flowrate)**



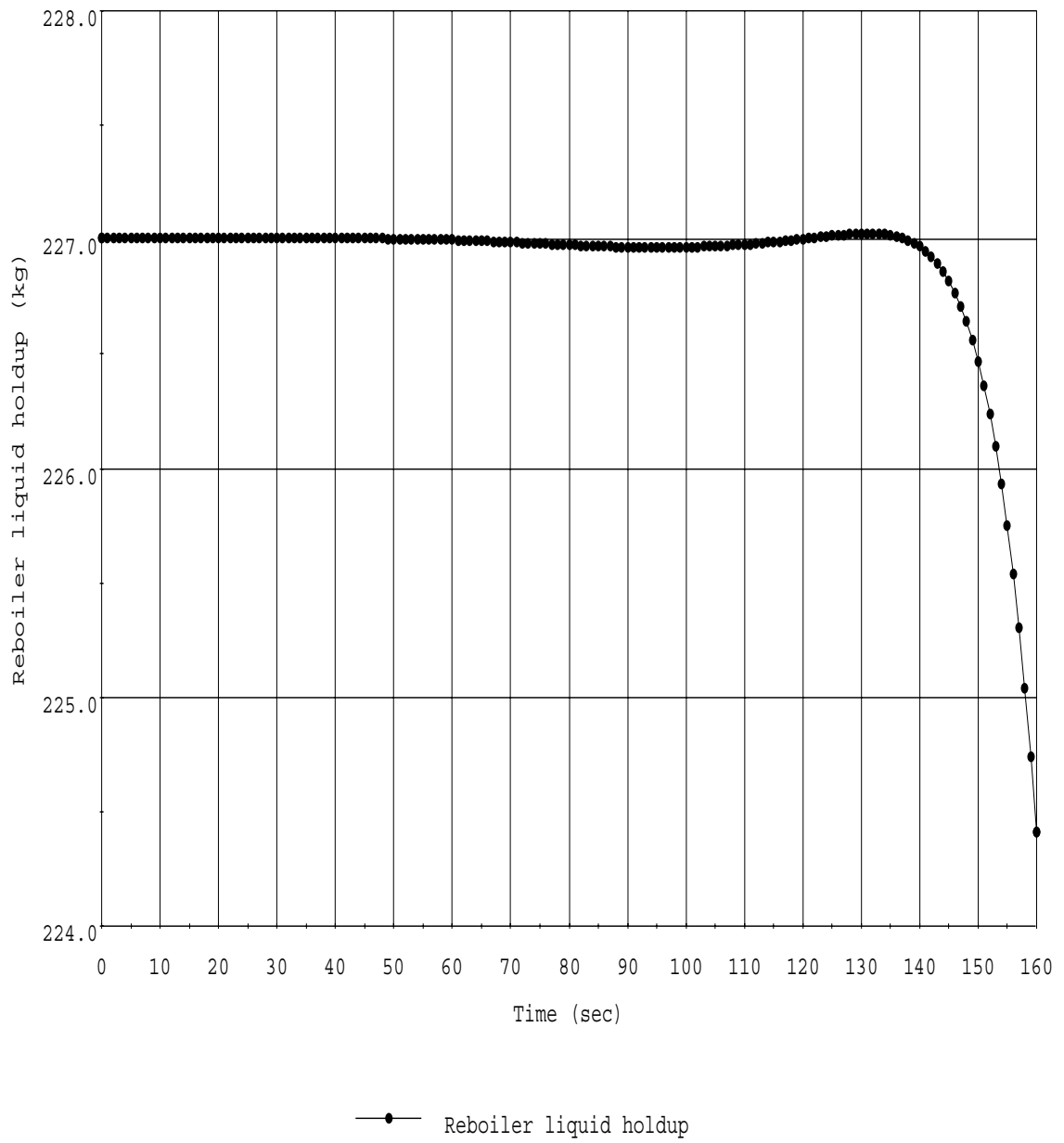
**Figure 21: Temperature of tray 1 (change in distillate flowrate)**

As observed the distillate flow rate only has an effect on the condenser liquid hold up. The other controlled variables remain constant. Figures 22 to 25 show the effect of the

reboiler duty to that of the controlled variables. It is observed that it has effect on all the four controlled variables.

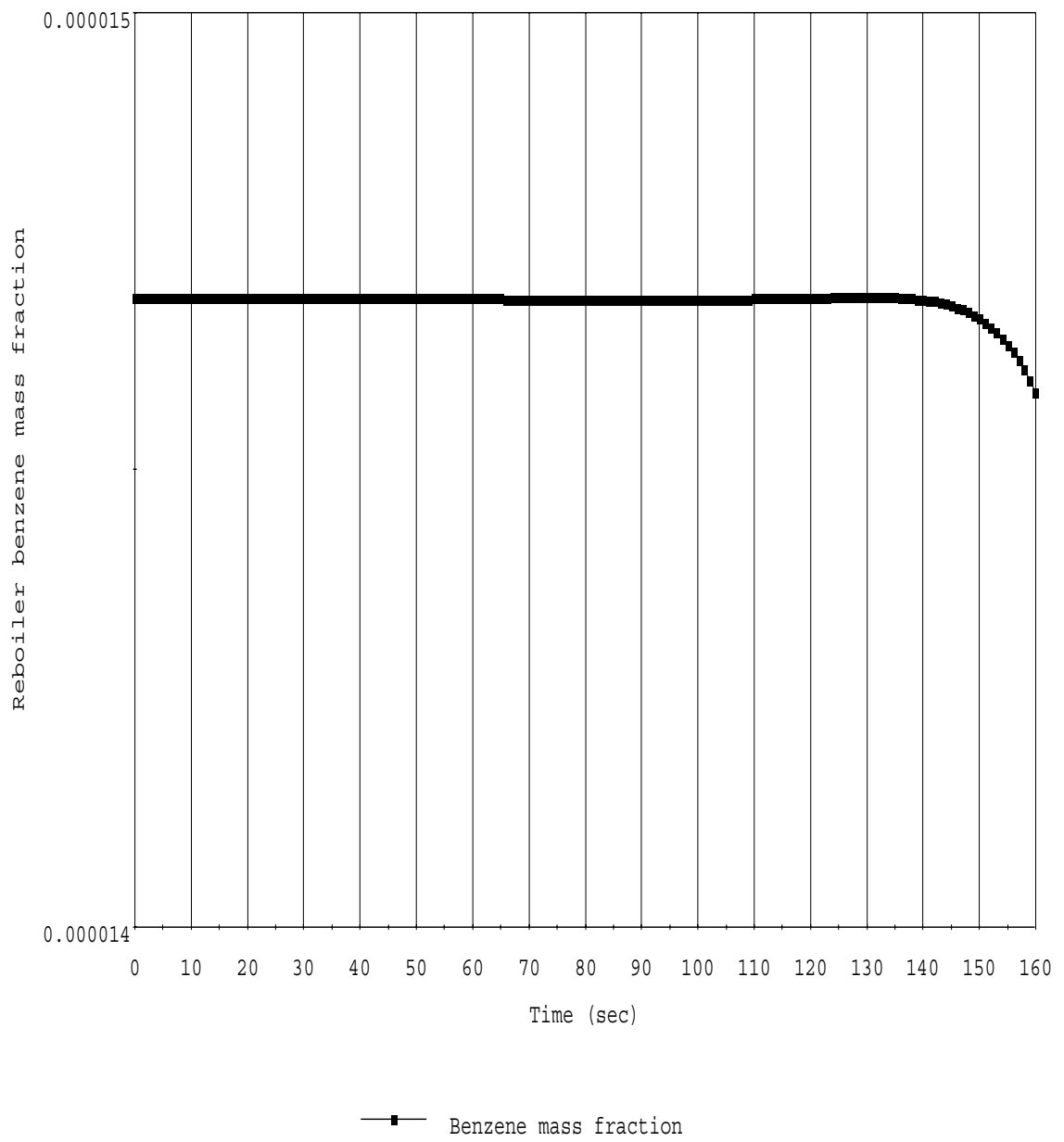


**Figure 22: Condenser liquid holdup (change in reboiler duty)**

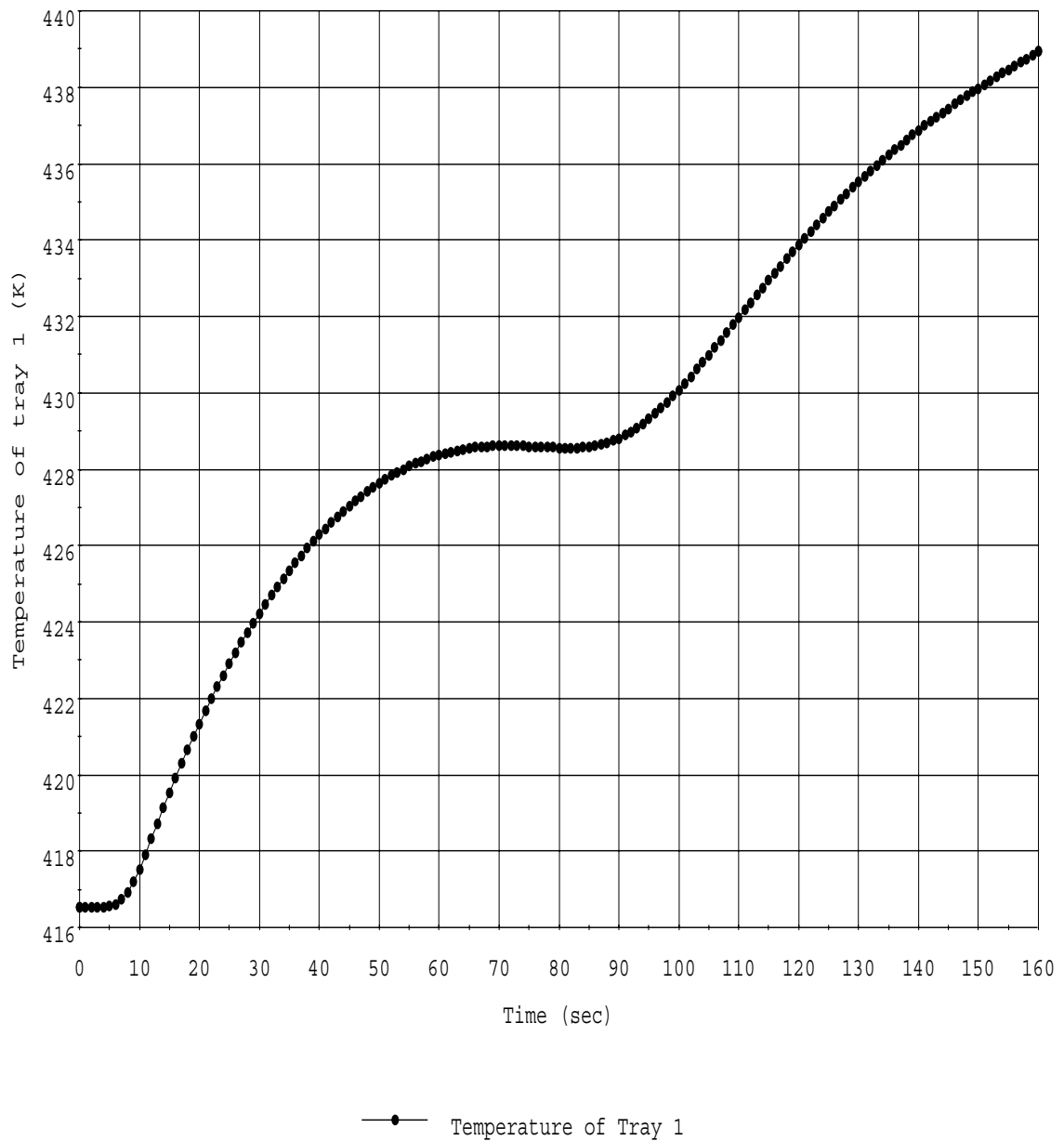


**Figure 23: Reboiler liquid holdup (change in reboiler duty)**



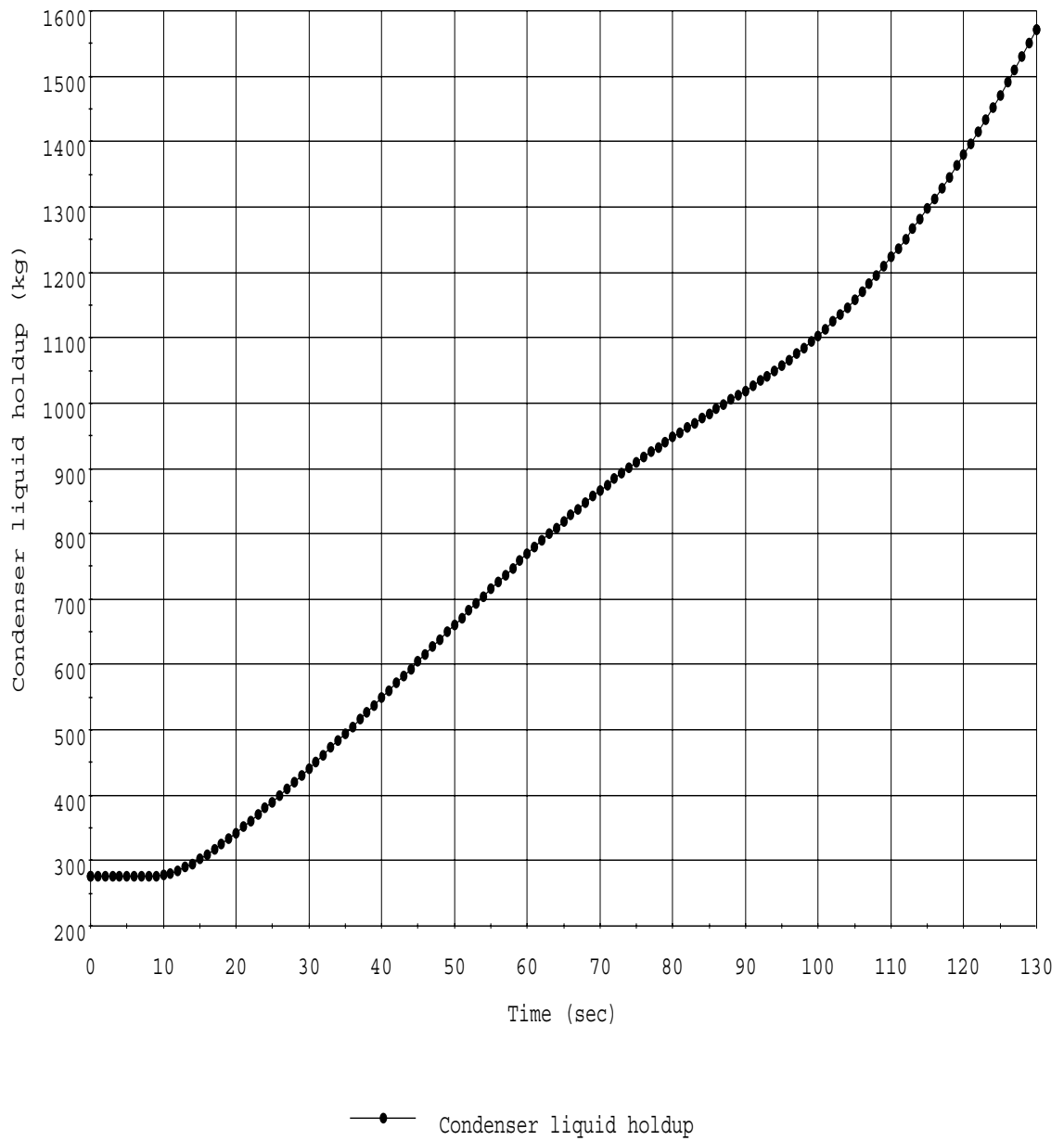


**Figure 24: Reboiler benzene mass fraction (change in reboiler duty)**

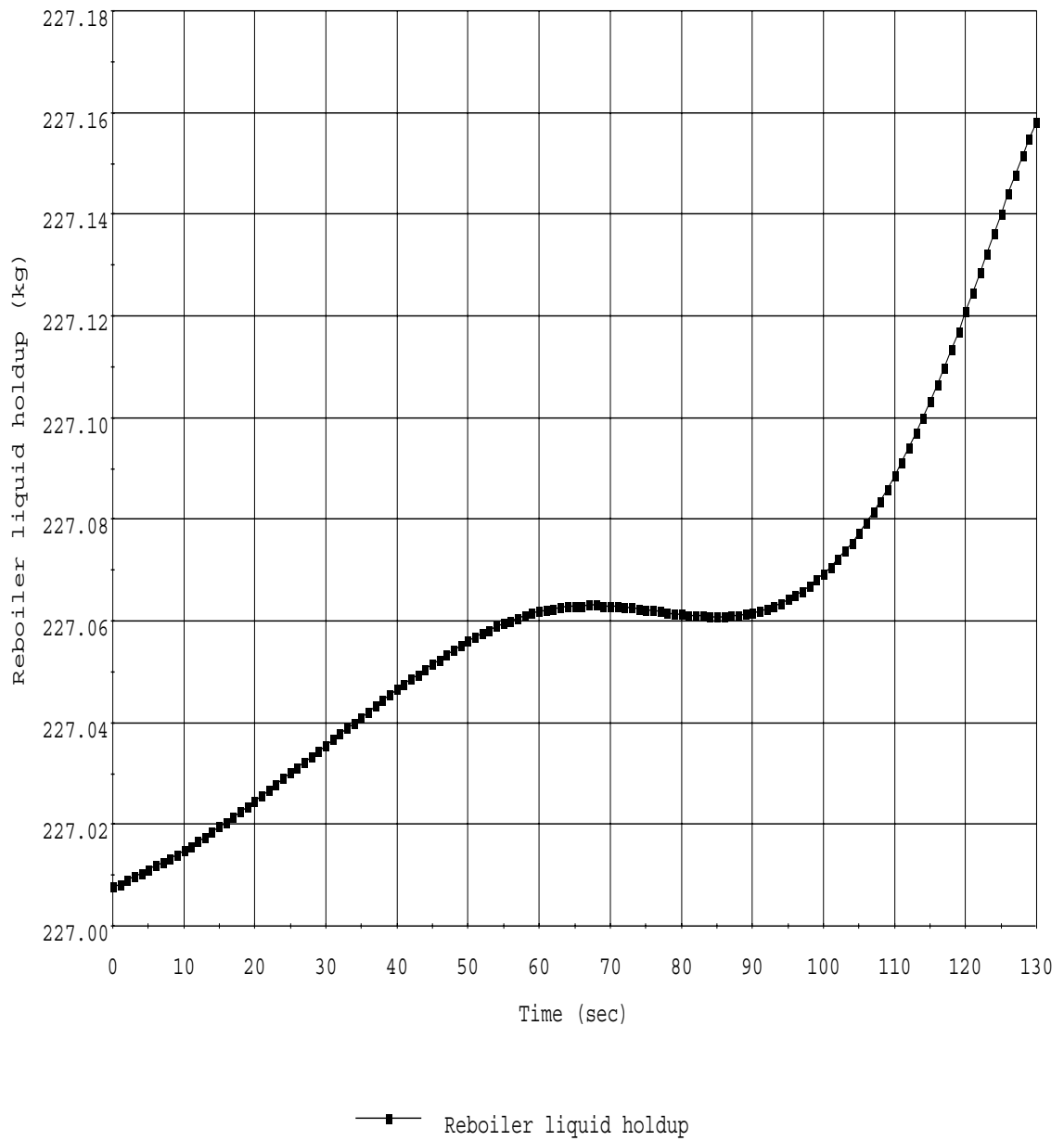


**Figure 25: Temperature of tray 1 (change in reboiler duty)**

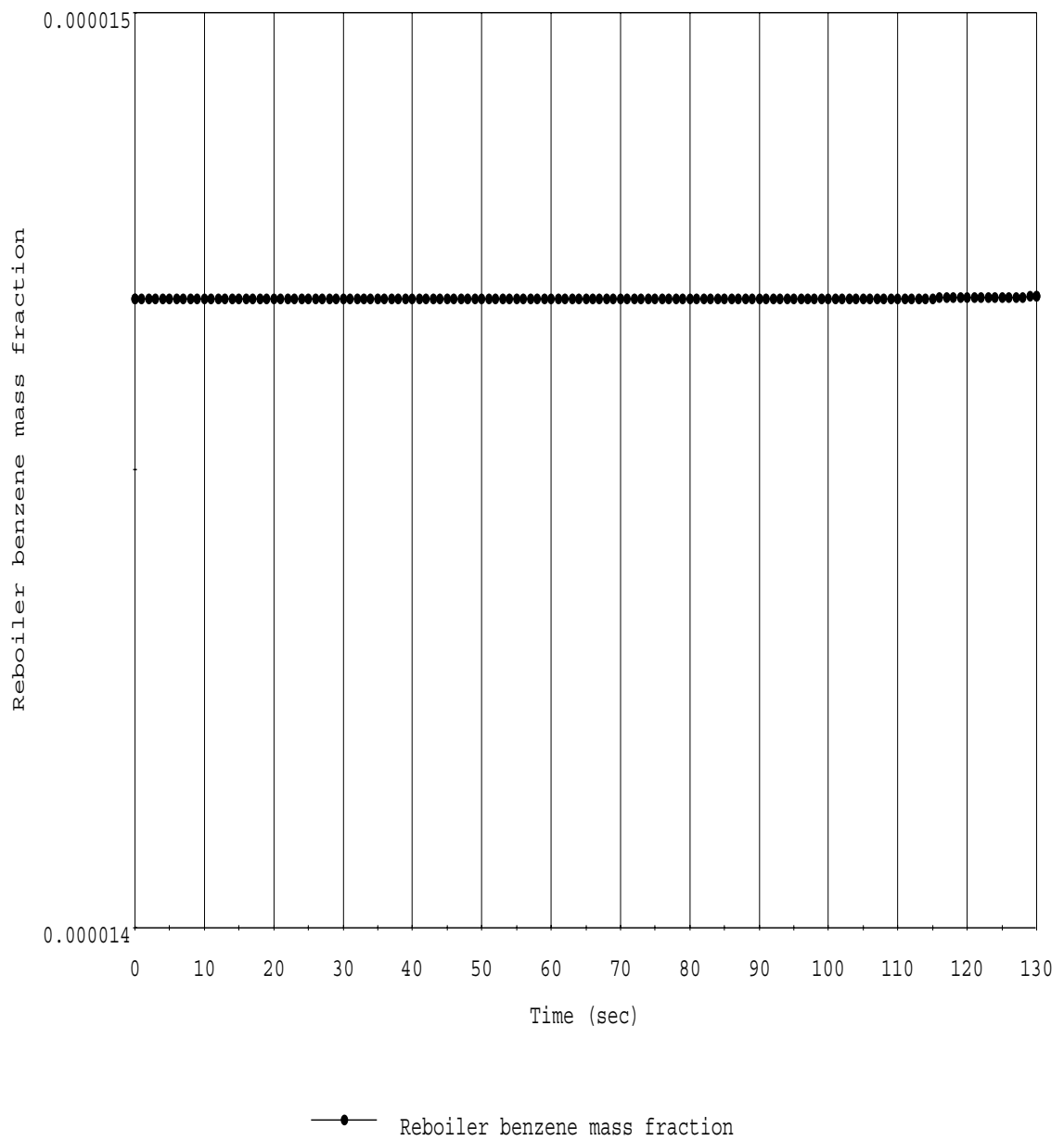
Figures 26 to 29 show the effect of bottoms flow rate from the reboiler on the controlled variables.



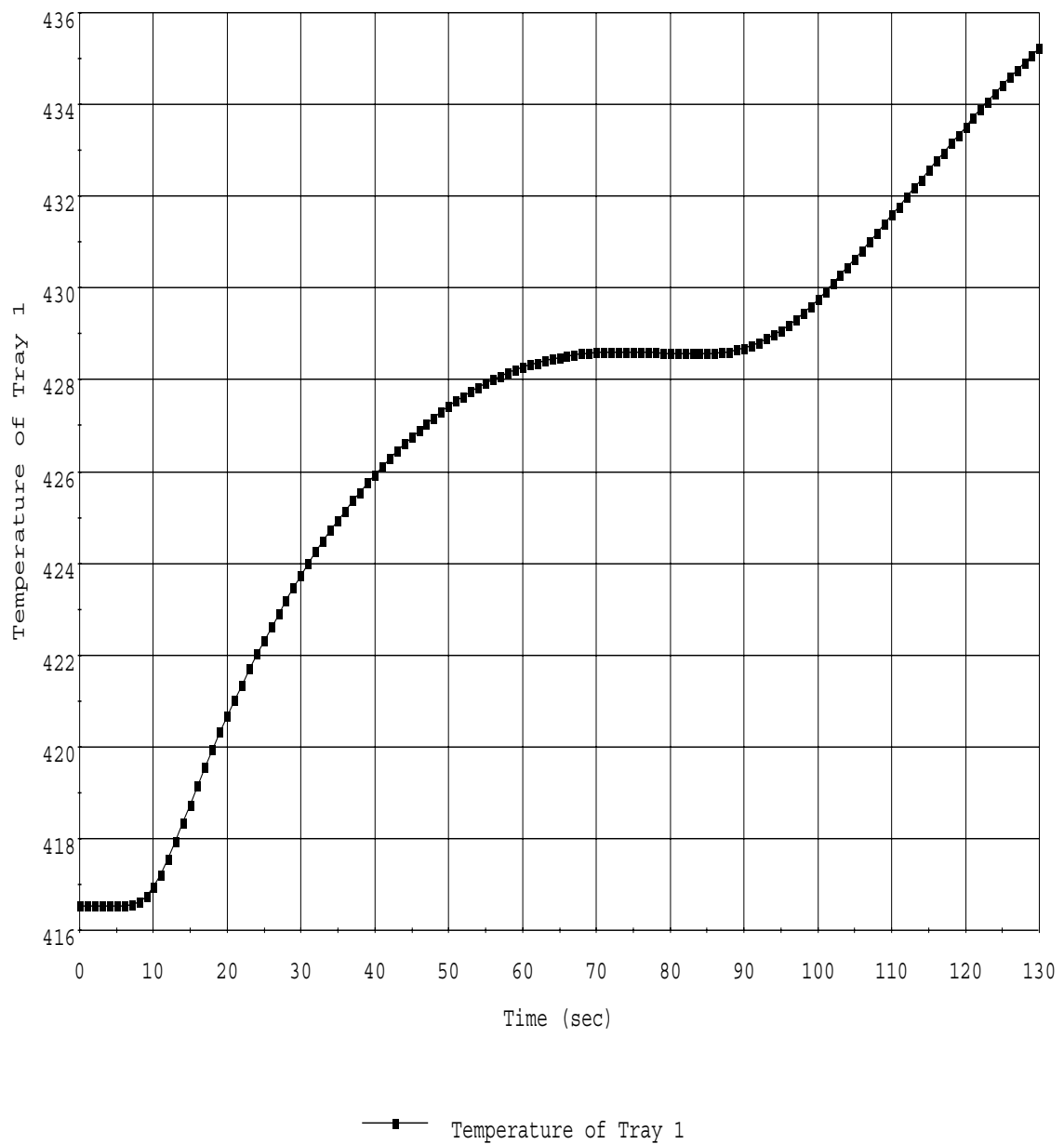
**Figure 26: Condenser liquid holdup (change in bottom flowrate)**



**Figure 27: Reboiler liquid holdup (change in bottom flowrate)**

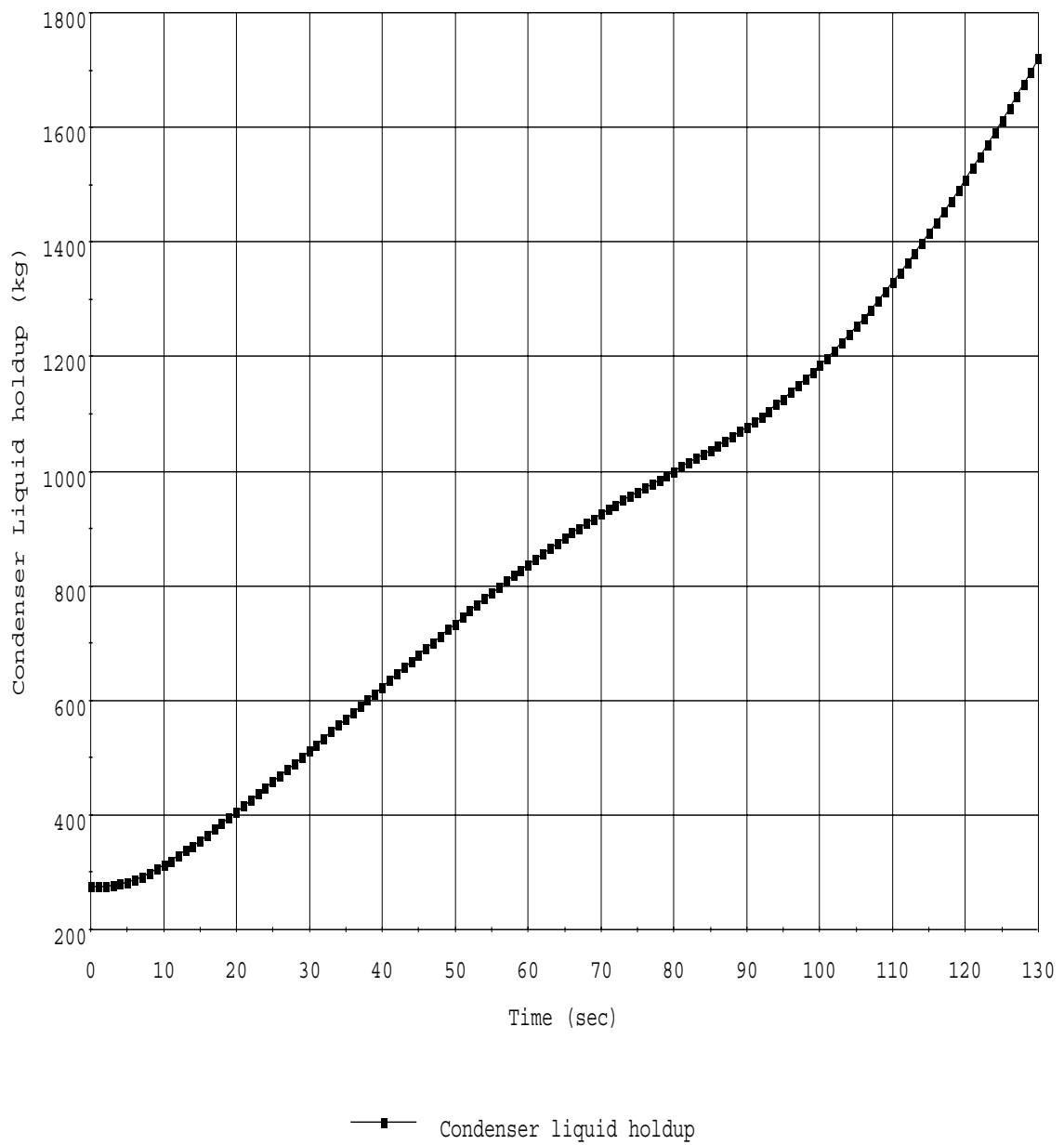


**Figure 28: Reboiler benzene mass fraction (change in bottom flowrate)**

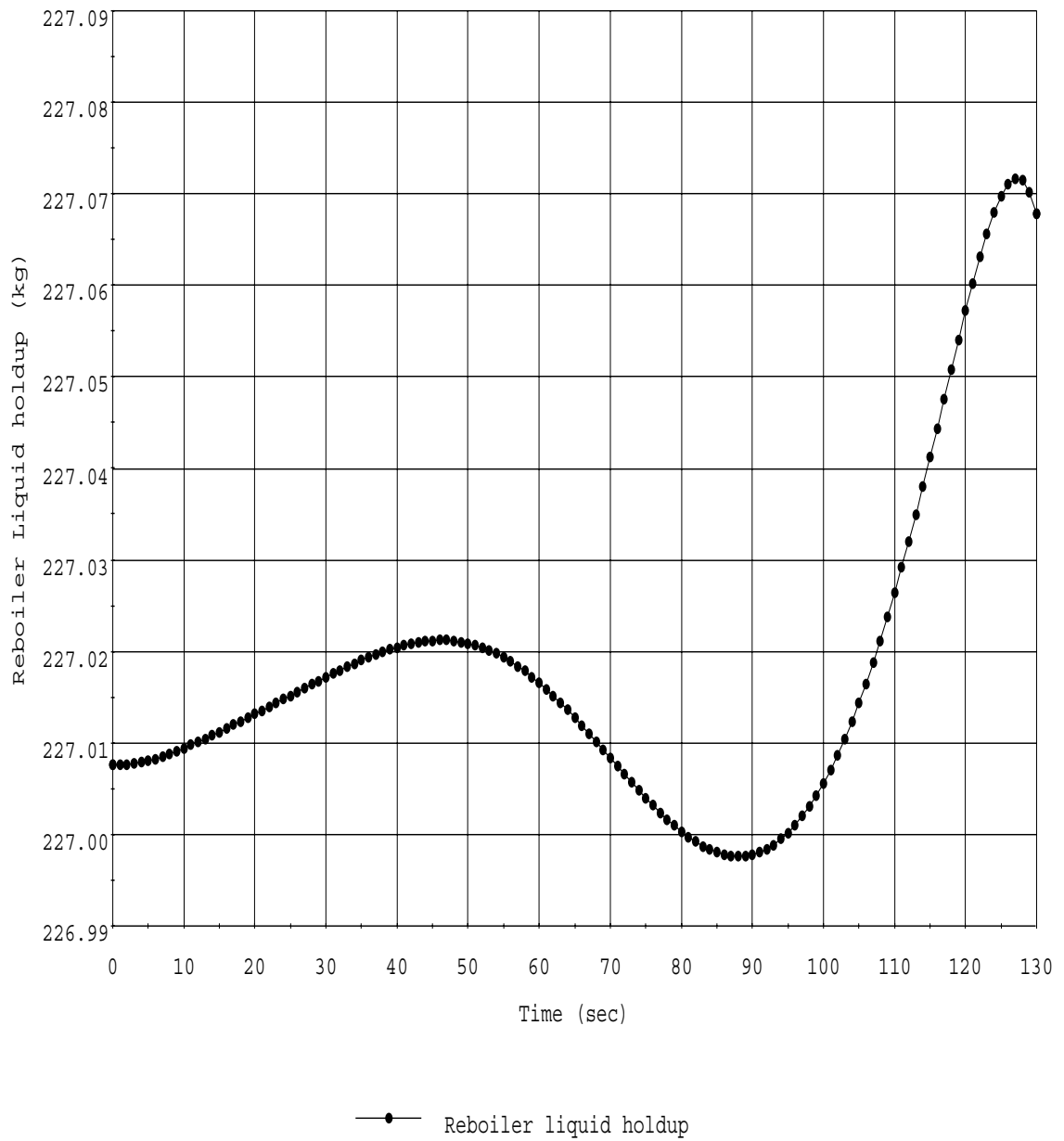


**Figure 29: Temperature of tray 1 (change in bottom flowrate)**

Figures 30 to 33 show the effect of reflux flow rate on the controlled variables.

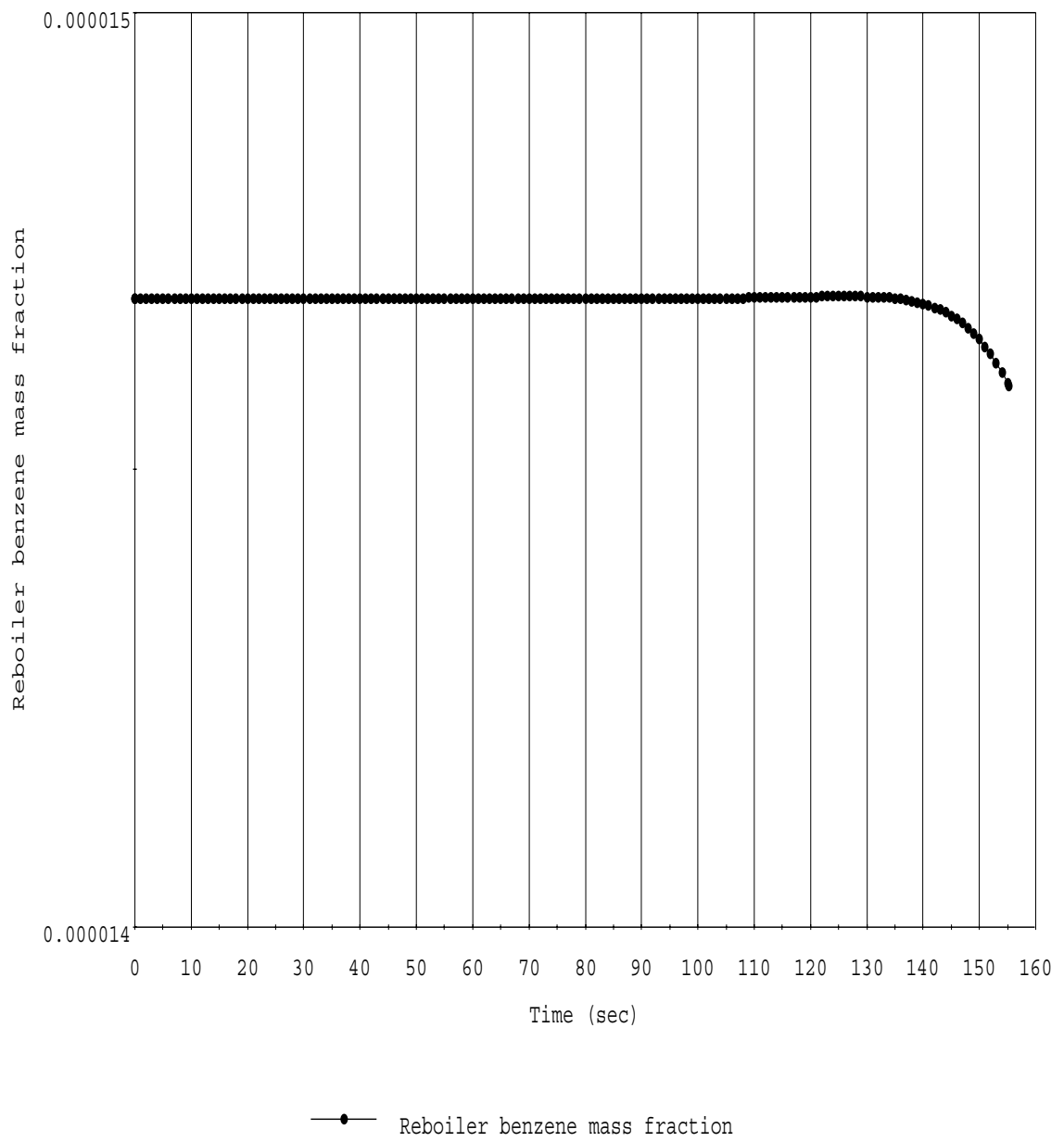


**Figure 30: Condenser liquid holdup (change in reflux flowrate)**

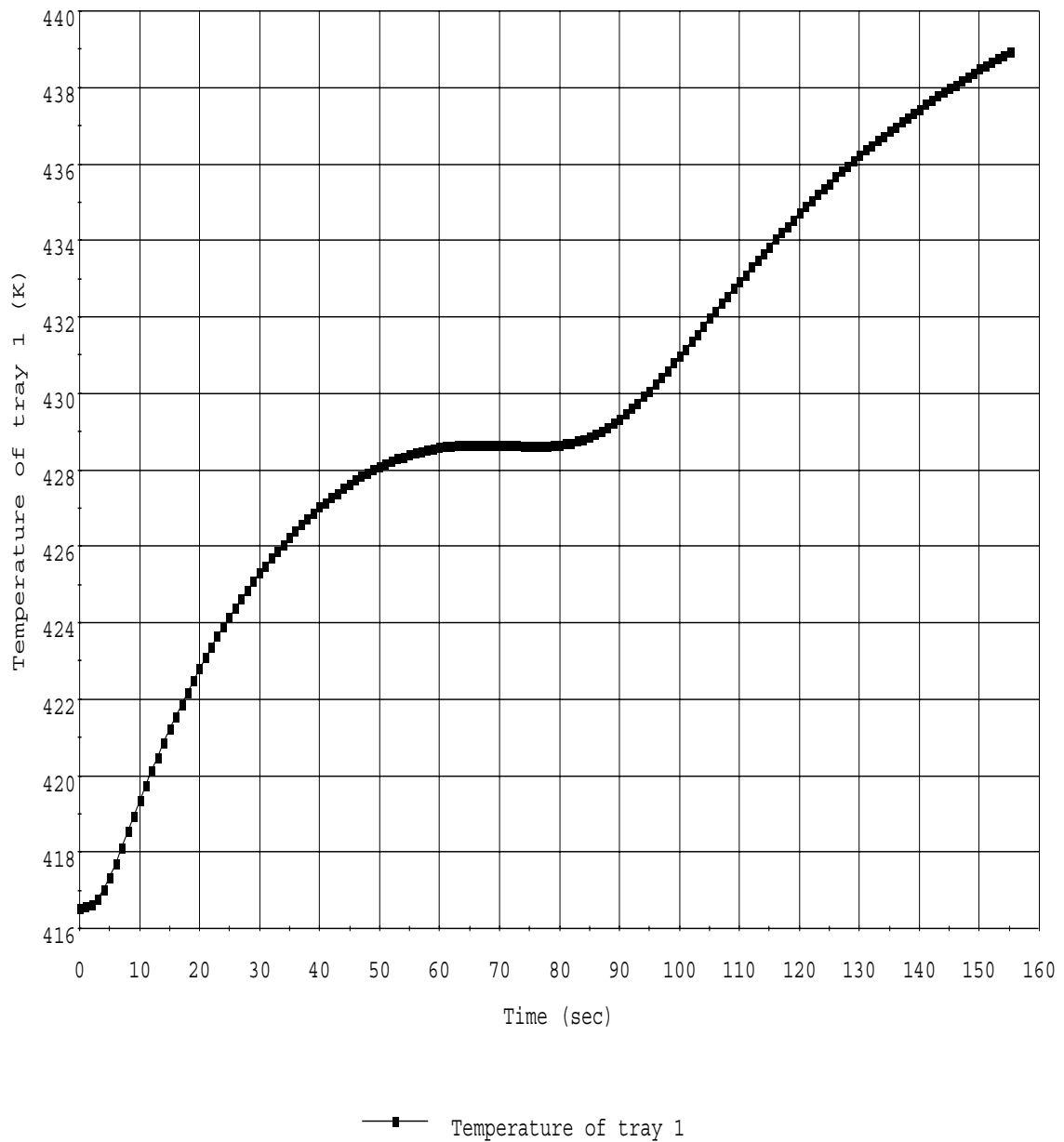


**Figure 31: Reboiler liquid holdup (change in reflux flowrate)**





**Figure 32: Reboiler benzene mass fraction (change in reflux flowrate)**



**Figure 33: Temperature of tray 1 (change in reflux flowrate)**

Many types of responses are observed in all the 16 figures depicting the effects of the manipulated variables on the controlled variables. Transfer functions are fitted to the generated responses in order to determine a control structure.

### Fitting of Transfer Function and RGA Analysis

A transfer function is an expression which dynamically relates the input and the output of a process model.  $Y(s) = G(s) U(s)$  where Y is the output, U is the input and G is the transfer function relating them. Accordingly if a transfer function is known between one input and output, the change in the output can be computed for a change in the input. In this particular study there are four input variables which are the manipulated variables and four output variables which are the controlled variables. As a result of this a 4 X 4 control structure there will have 16 transfer functions since each combination of input-output variables results in one transfer function. Transfer functions were fitted<sup>35,38</sup> based on the response generated in section. Table 4, shows the fitted transfer function between different pairs of input and output.

**Table 4: Fitted Transfer Function**

	Condenser Liquid holdup	Reboiler Liquid Holdup	Reboiler benzene mass fraction	Tray temperature
Distillate flowrate	$\frac{1}{s}$	0	0	0
Bottoms flowrate	$-e^{-10s} \frac{0.02635}{s}$	$-e^{-5.37s} \frac{1.113}{s}$	0	$-e^{-4.3s} \frac{283.4}{s}$
Reboiler heat duty	$\frac{1473}{33.12s^2 + s}$	$-e^{-2.29s} \frac{3.451}{s}$	$-e^{-151s} \frac{0.0005}{s}$	$e^{-2.5s} \frac{28.3}{s}$
Reflux flowrate	$\frac{1.005e08s - 6.645e06}{s}$	$-e^{-107s} \frac{197.1}{4.343s^2 + s}$	$e^{-148s} \frac{0.0003}{s}$	$-e^{-4.49s} \frac{404.4}{s}$

The RGA was calculated (putting  $s = 1e-12$ ) from the above and variables were paired for control purposes. PI type controllers were designed and tested both for regulatory

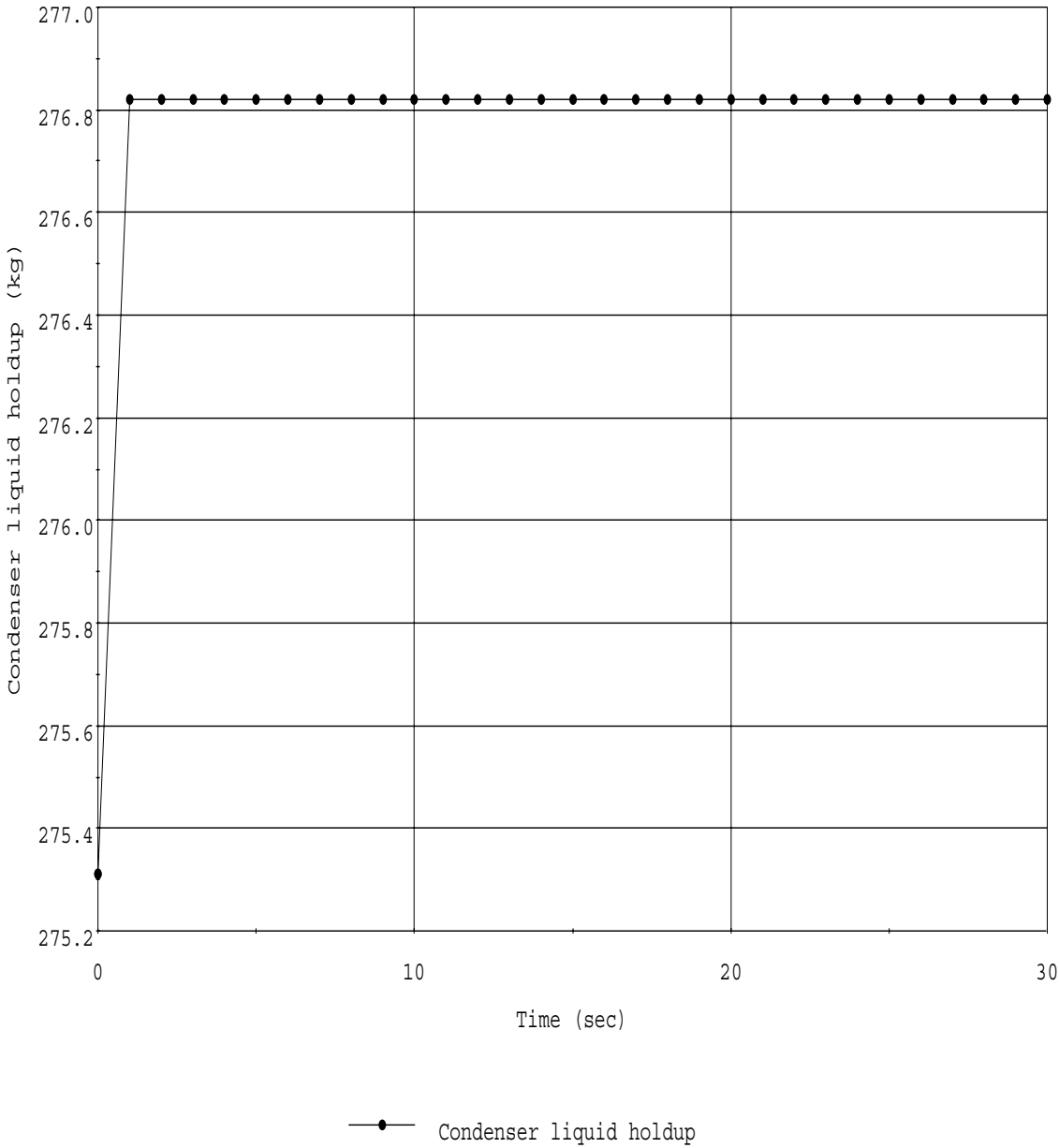
problem and servo problem. The parameters of the controller were found by IMC design method and tuned by trial and error to get the desired result. The paired variables are given in Table 5.

**Table 5: Paired Controlled and Manipulated Variables for Control Structure**

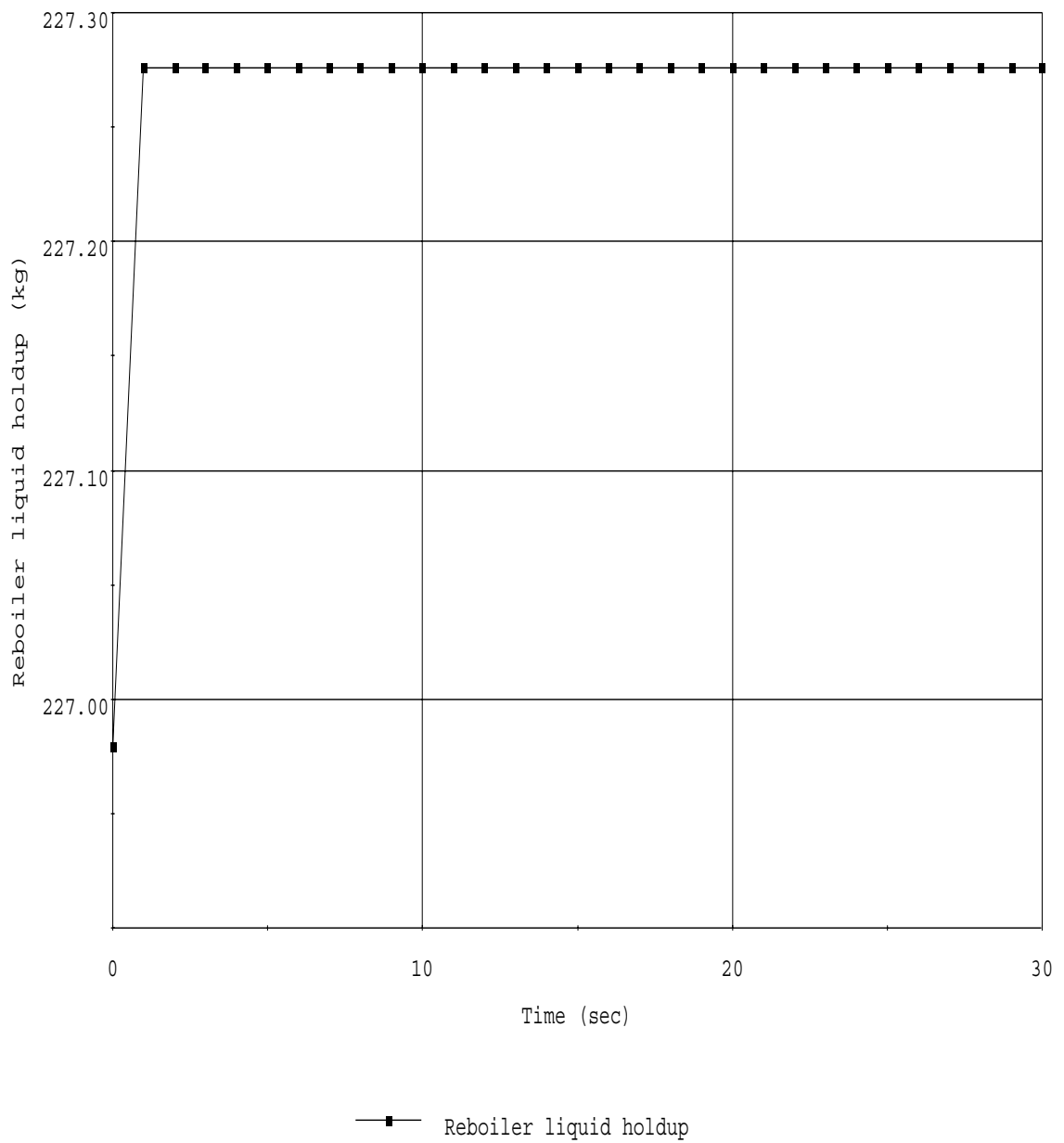
	Manipulated Variables	Controlled variables
1	Reflux flow rate	Temperature in the first tray of the column from top
2	Distillate flow rate	Condenser liquid holdup
3	Reboiler heat duty	$x_{Benzene}$ in bottom flow from reboiler
4	Bottom flow rate from reboiler	Reboiler liquid holdup

### Servo Problem

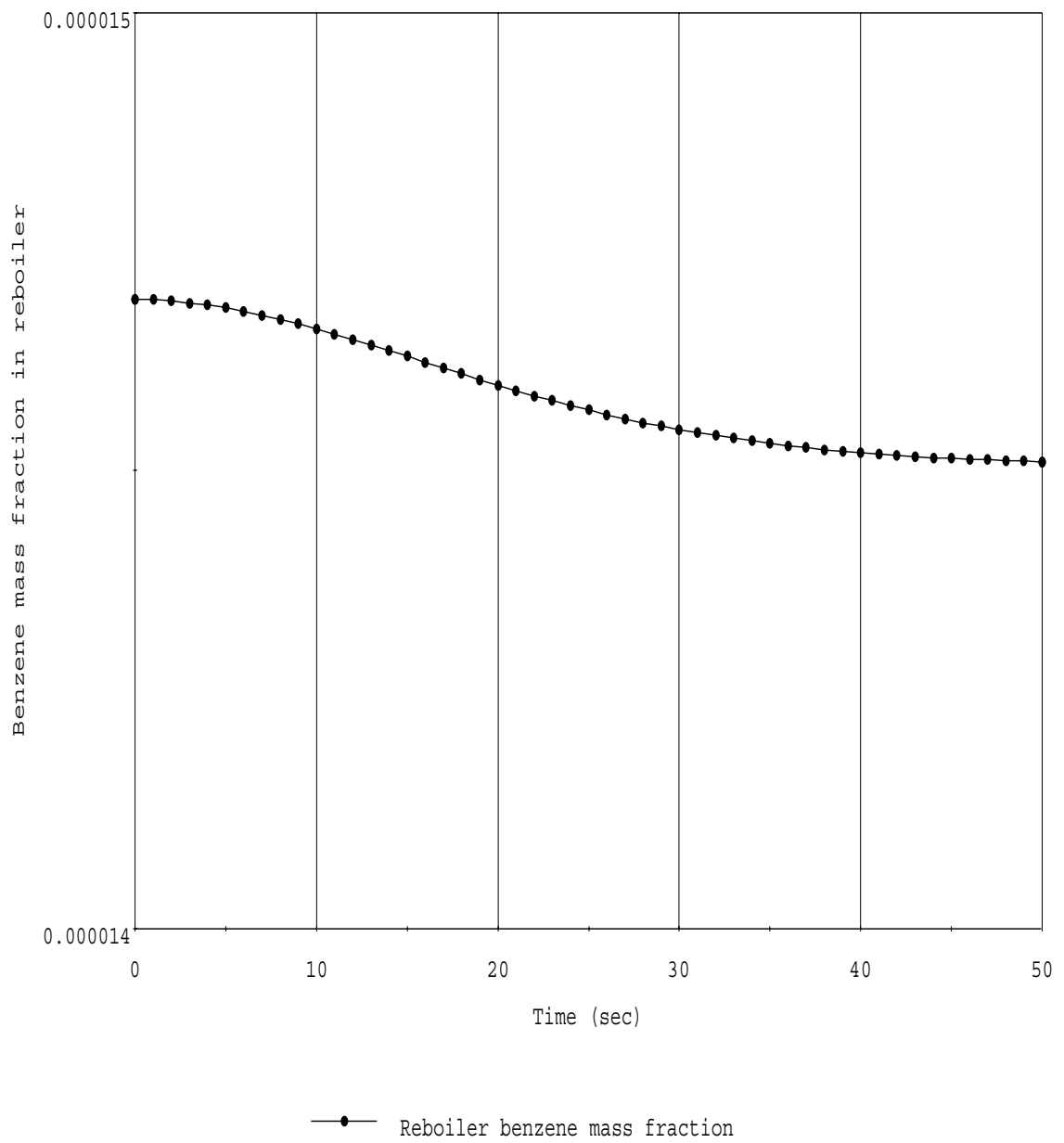
Figures 34 to 37 show the response of the controlled variables in the closed loop system when there is a set point change.



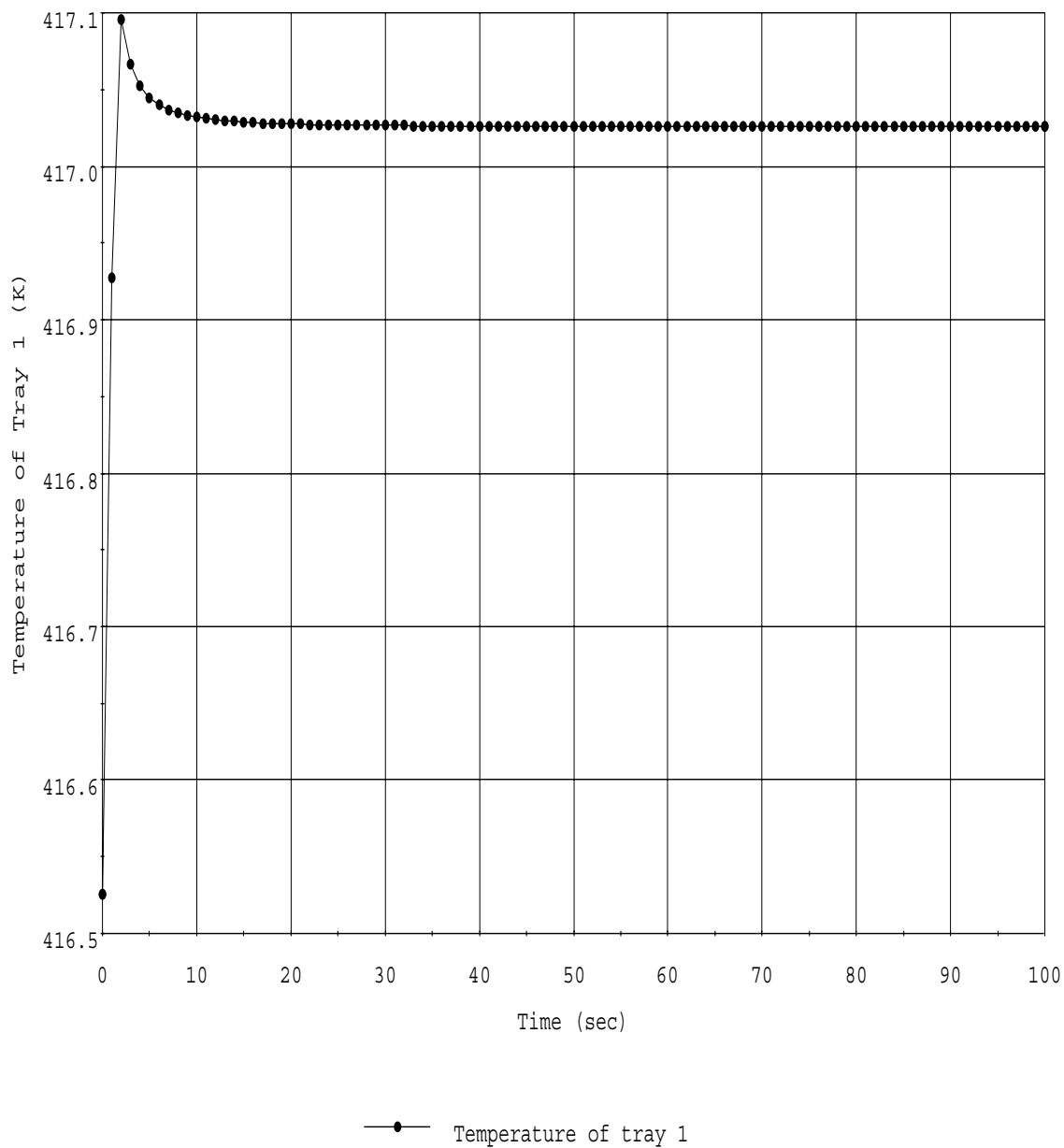
**Figure 34: Set point change for condenser liquid holdup**



**Figure 35: Set point change for reboiler liquid holdup**



**Figure 36: Set point change for reboiler benzene mass fraction**



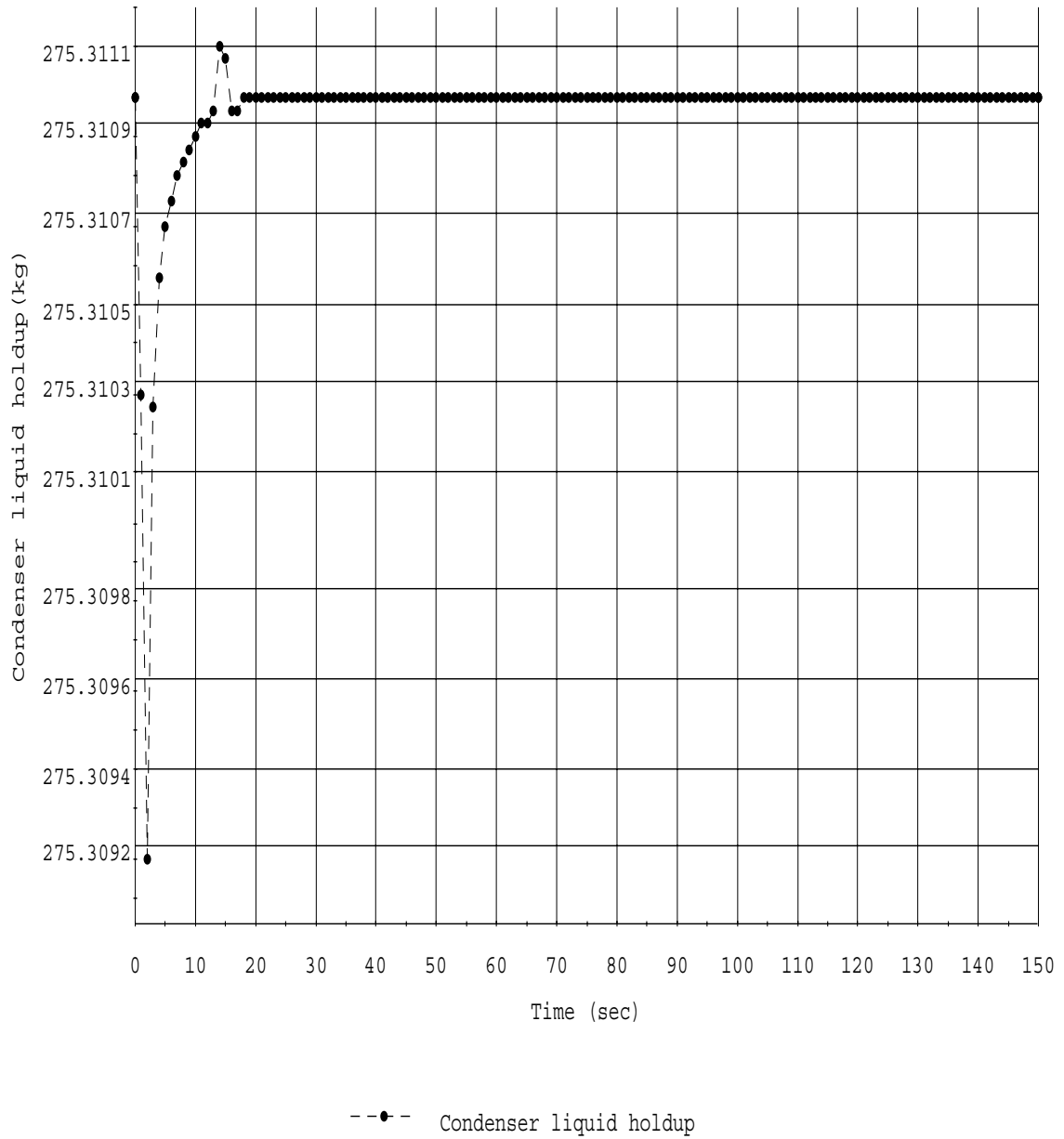
**Figure 37: Set point change for temperature of tray 1**

All the four controlled variables reach the new set point within reasonable time. The mass fraction of benzene takes longer to reach the new set point.

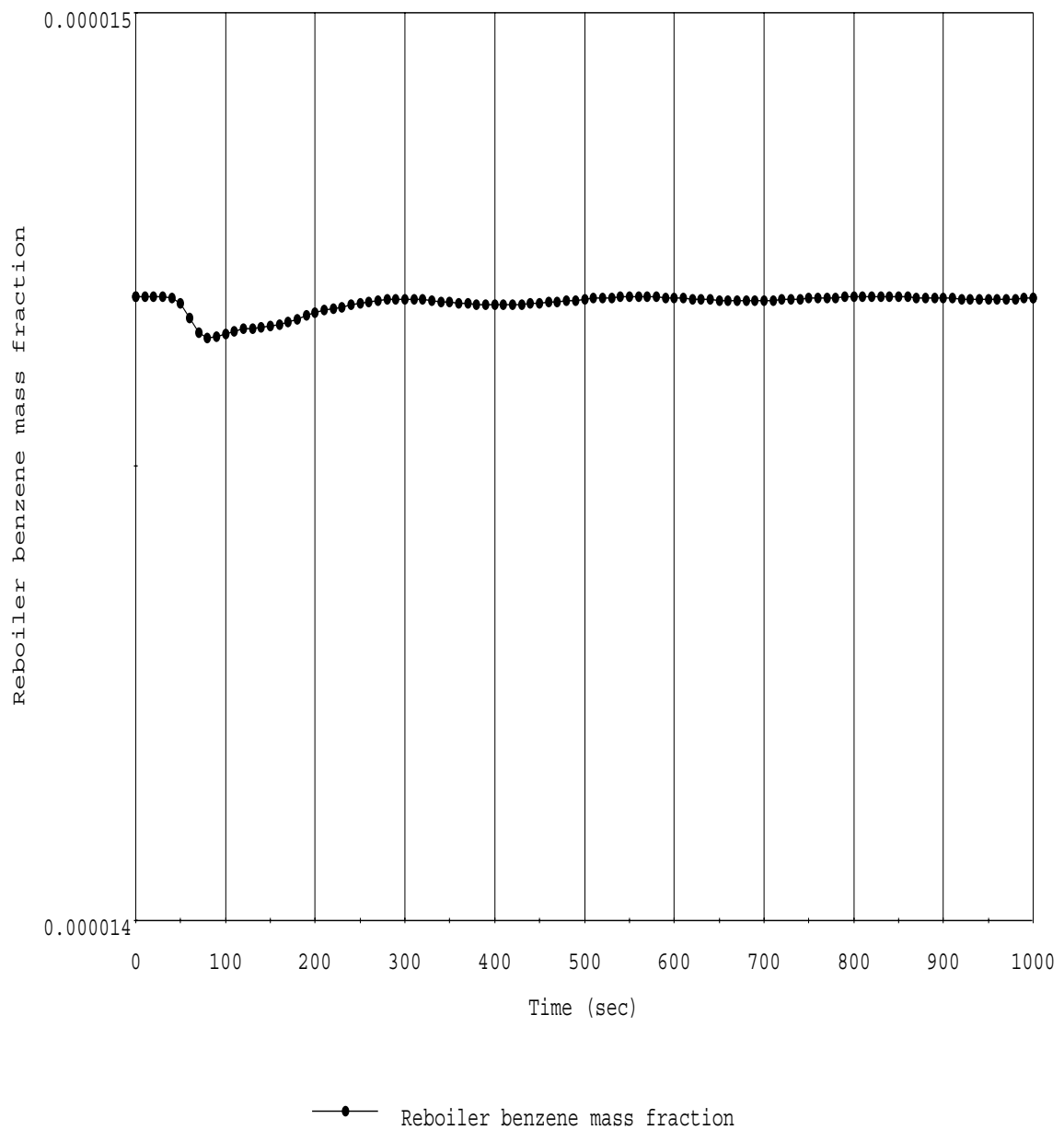


### Regulatory Problem

Figures 38 to 45 show the effect of disturbances on the controlled variables.

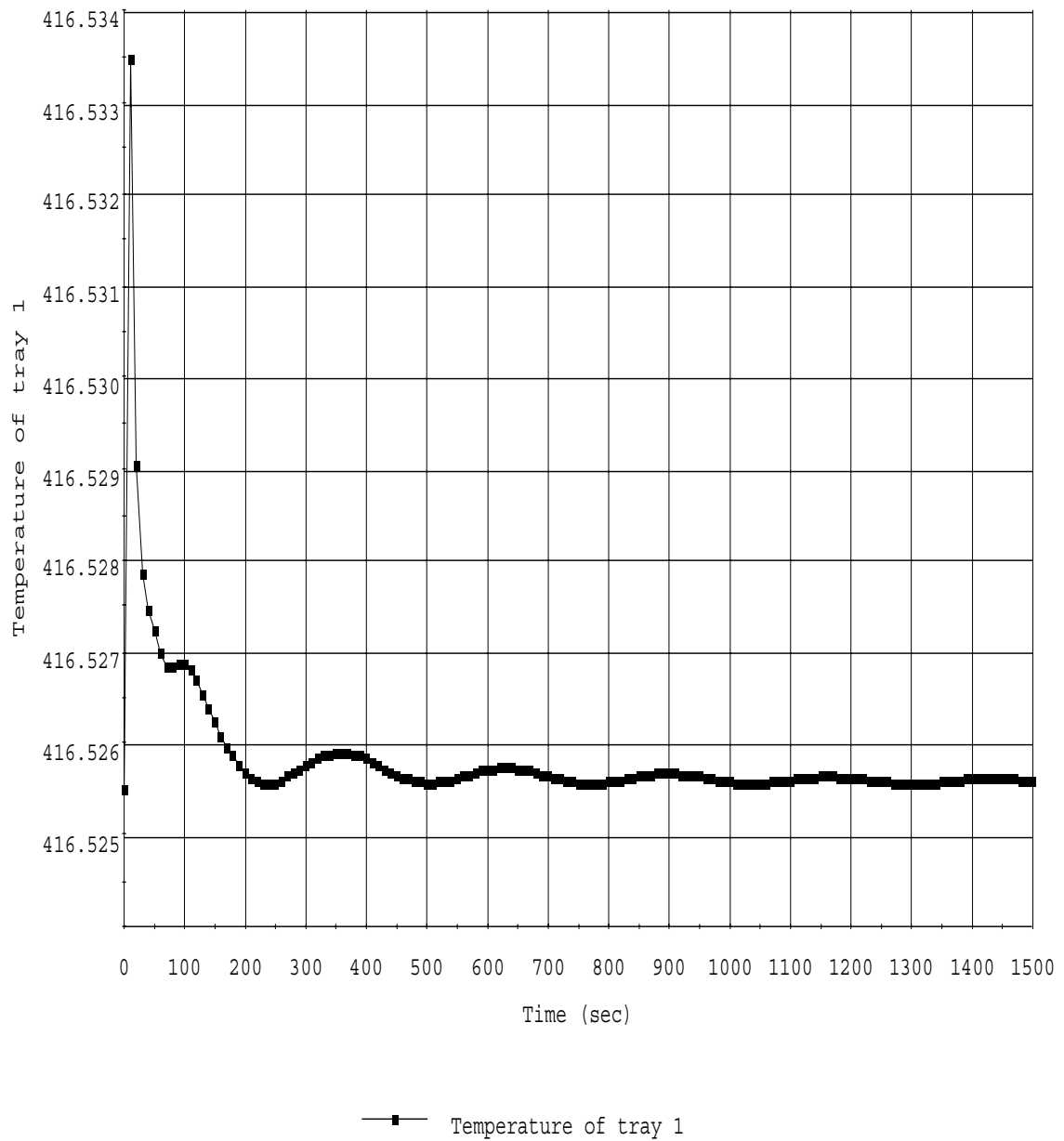


**Figure 38: Response of condenser liquid hold up due to change in feed temperature**

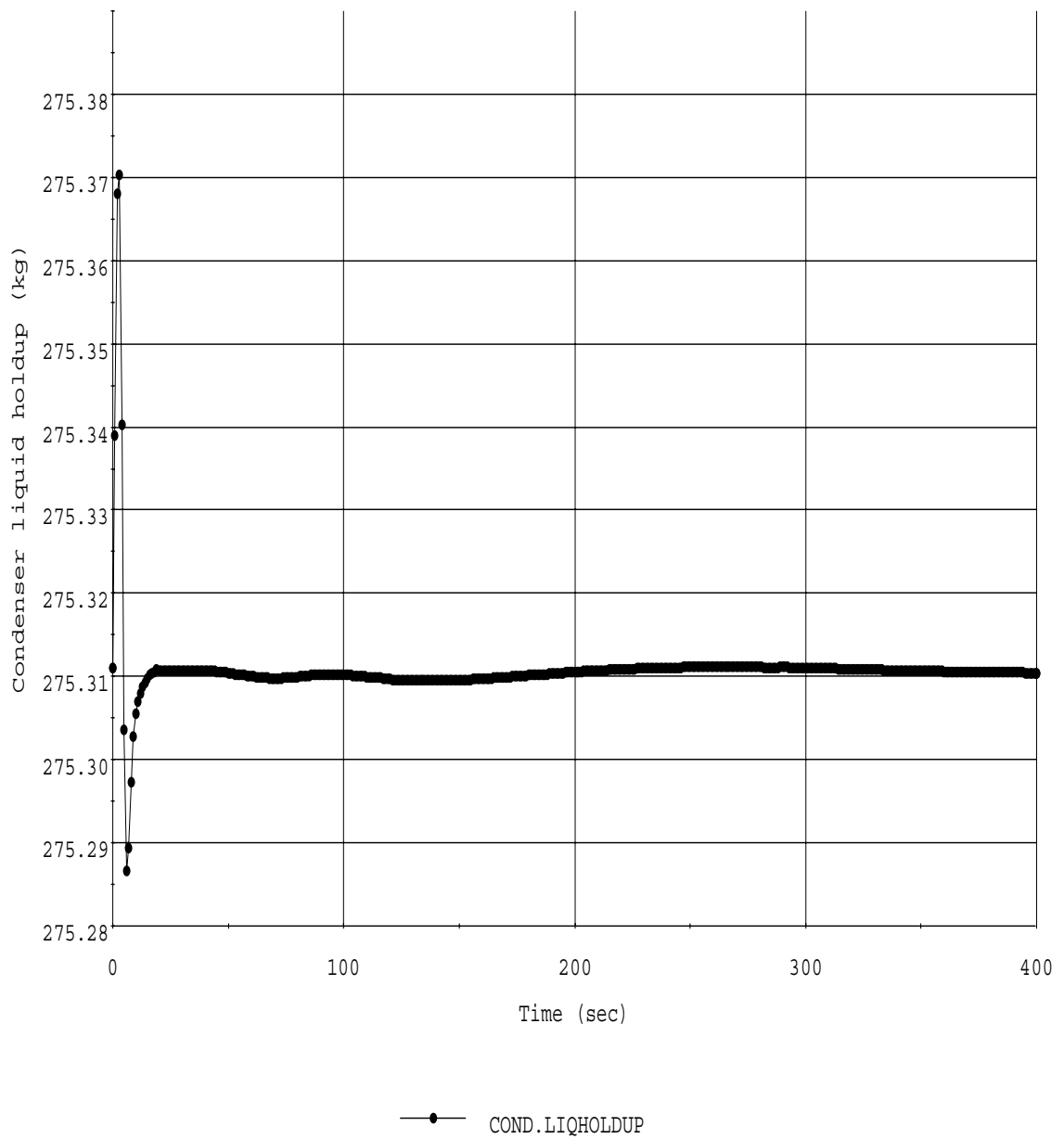


**Figure 39: Response of reboiler benzene mass fraction due to change in feed temperature**

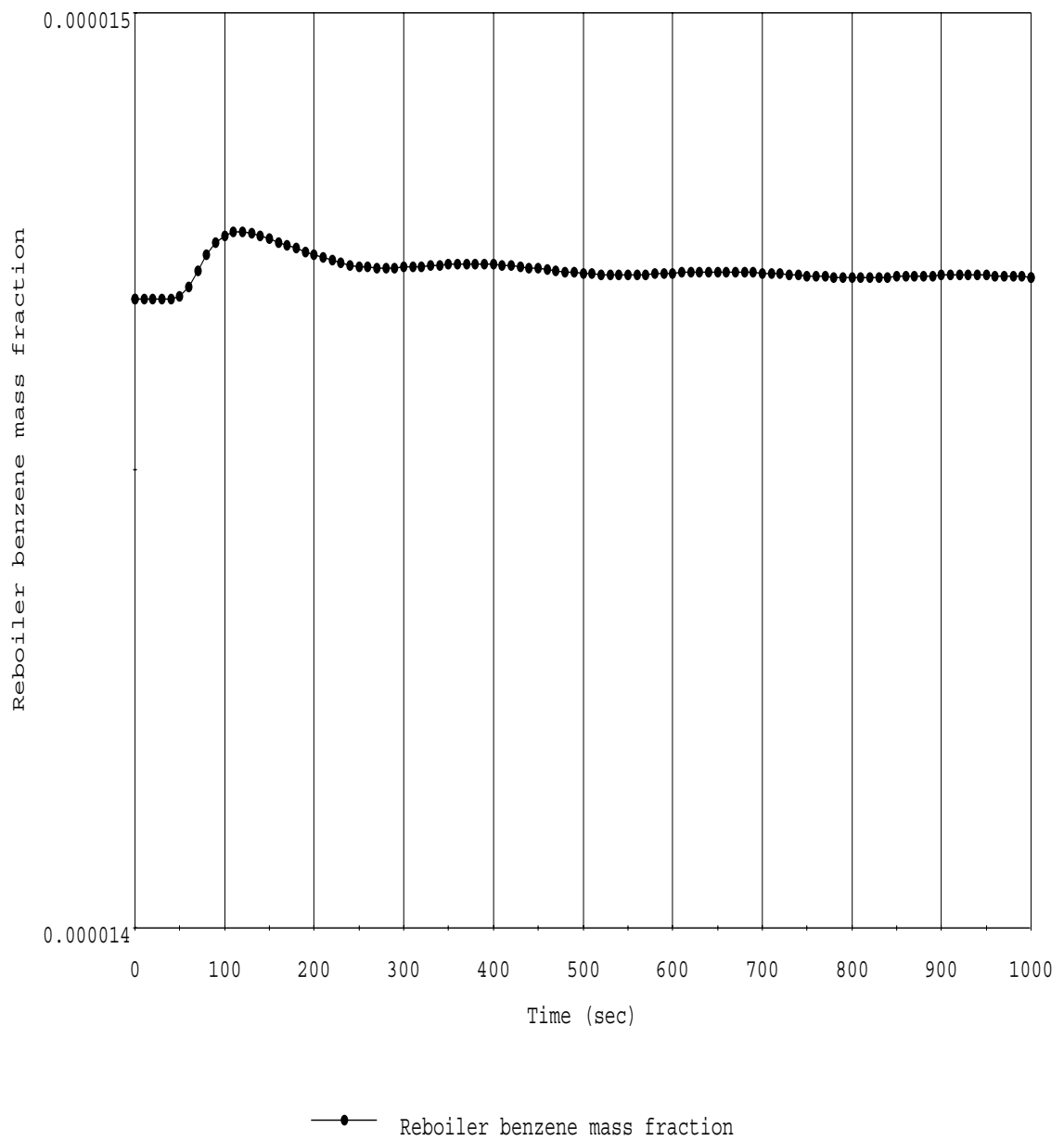
The disturbance in feed temperature apparently does not affect the reboiler liquid holdup much in the closed loop system and hence no graph is shown here.



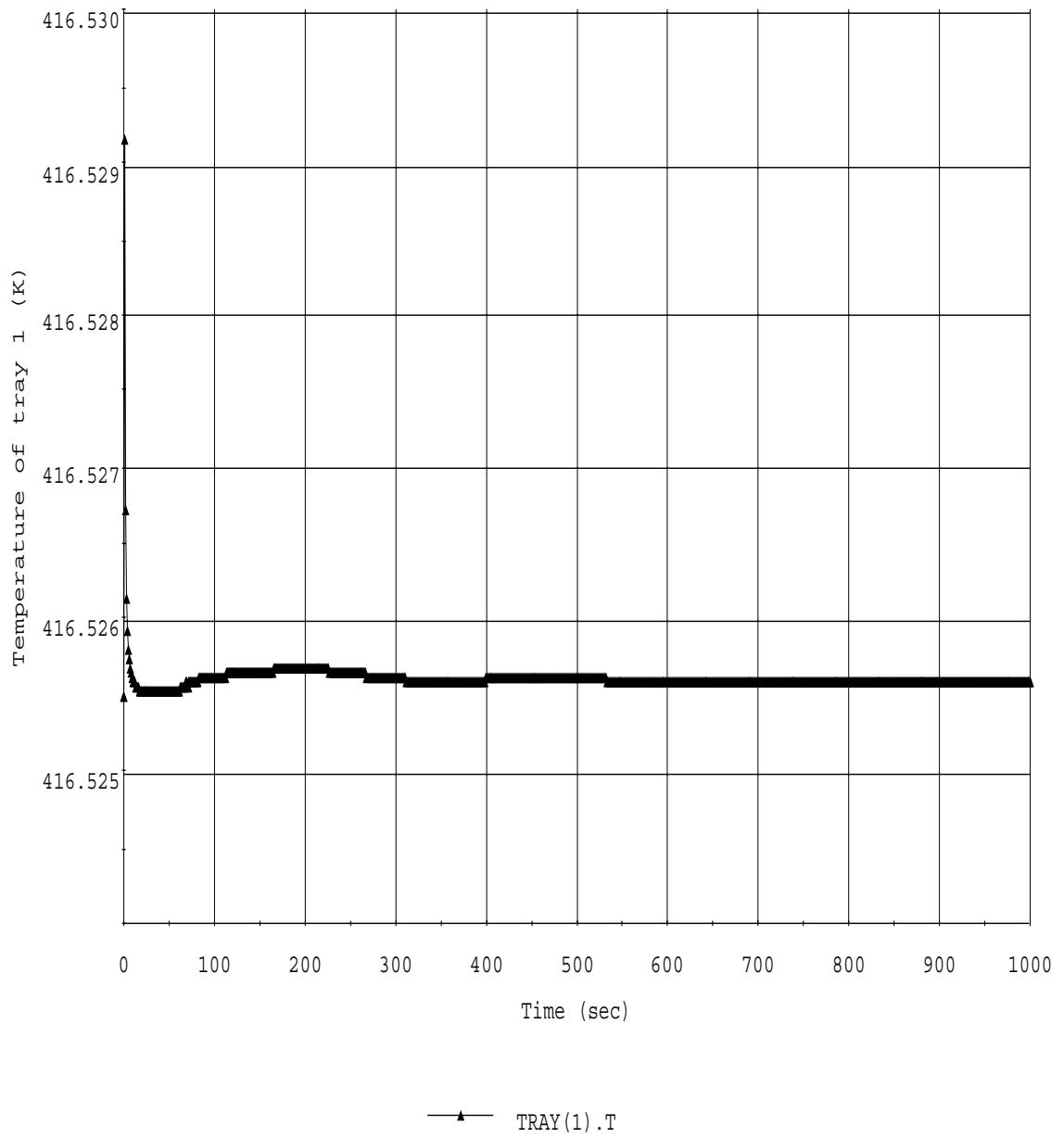
**Figure 40: Response of temperature of tray 1 due to change in feed temperature**



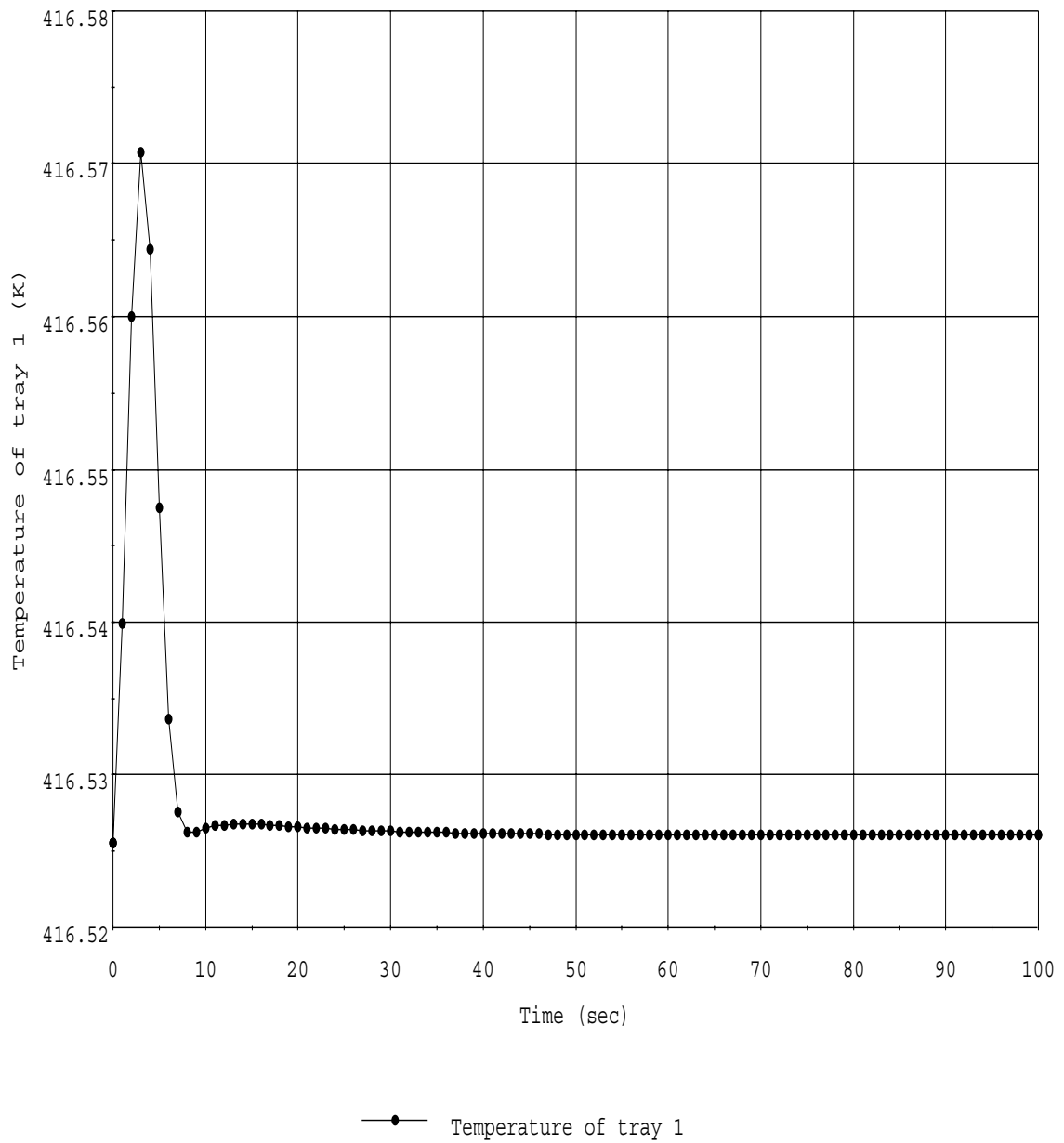
**Figure 41: Response of condenser liquid holdup due to change in feed flowrate**



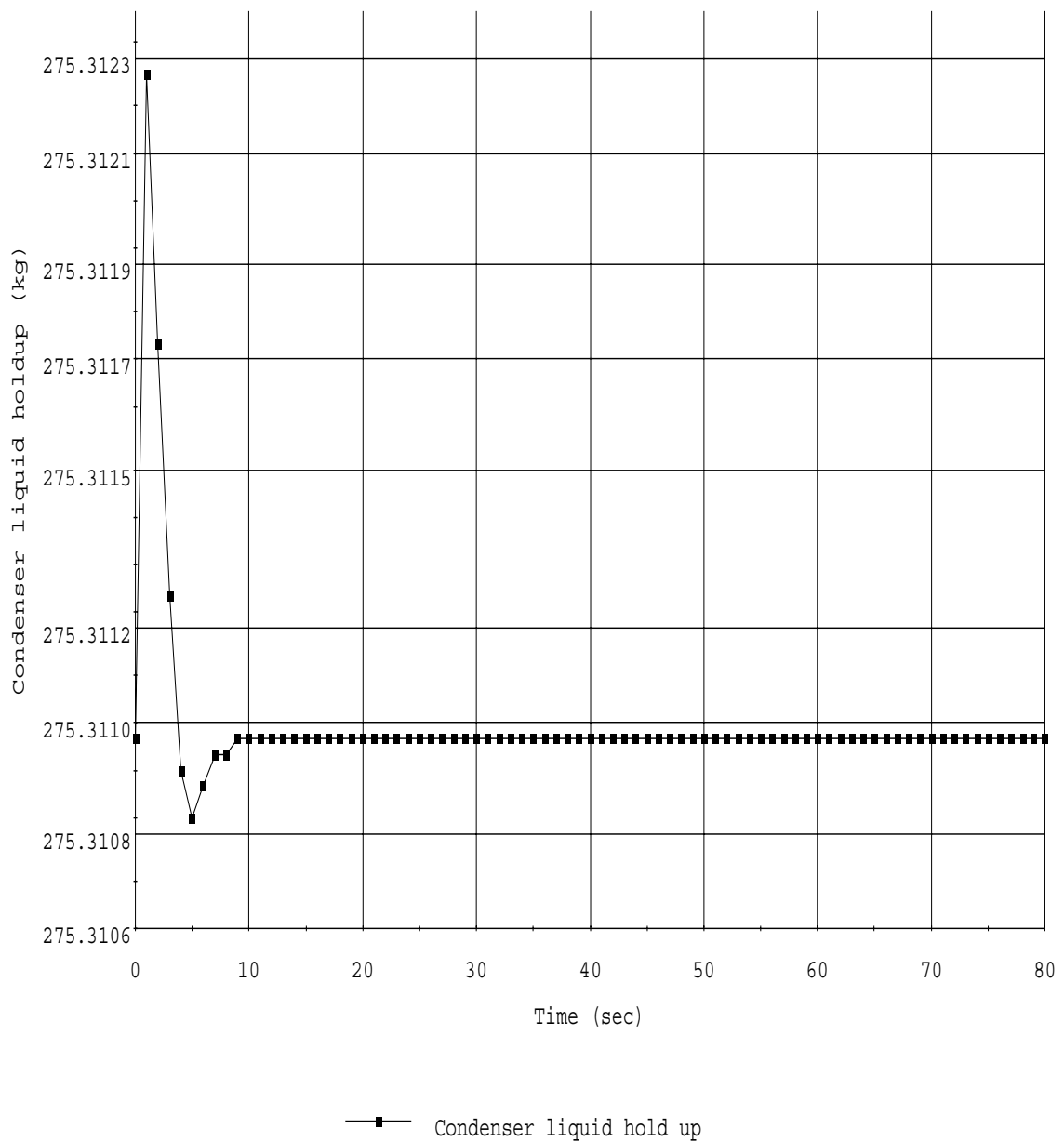
**Figure 42: Response of reboiler benzene mass fraction to change in feed flowrate**



**Figure 43: Response of temperature of tray 1 due to change in feed flowrate**



**Figure 44: Response of temperature of tray 1 due to change in feed composition**



**Figure 45: Response of condenser liquid holdup due to change in feed composition**

It should be noted that the disturbances have little impact on the reboiler liquid holdup. The reboiler benzene mass fraction requires a long time to get back to its original state.



## CHAPTER V

### CONCLUSION AND RECOMMENDATIONS

#### **Conclusion**

A dynamic model of a reactive distillation with 70 trays and 15 components has been implemented. The feed to the system comes from a reformer and may change on a regular basis which presents a need for investigating the dynamic behavior of the process. EPA regulations require that the benzene content in the streams leaving the column is below a certain threshold value even under the influence of large disturbances in the feed. Accordingly, a control system for this process has to take the environmental regulations into account in addition to economic considerations. As dynamic modeling of such large-scale processes is a non-trivial task, a steady state model for a single tray had to be developed in a first step and the results were used as initial guesses for further simulations. This was then followed by the addition of trays at the top and bottom of the already implemented tray where each time the model had to be simulated and new initial guesses for the variables had to be computed. This process was carried out until the model for the entire column had been built. The column was compared to data provided by an industrial collaborator and some parameters in the model were modified to correctly represent the industrial data. This steady state model was then extended to also include dynamics as any control study can only be performed on a dynamic model.

A set of controlled and manipulated variables had to be chosen to determine which controlled variable should be paired with which manipulated variable. The column was simulated in “open-loop” for different disturbances and was determined that upset conditions persists for 3-4 hours, which clearly indicates a need for further control studies for this system. Open loop step tests were performed by changing the manipulated variables and the response of the controlled variables were recorded. Transfer functions were fitted to represent the relationship between the manipulated and the controlled variables. As a transfer function can only represent a system within a

certain operating region, the size of the region in which the process is correctly represented by a transfer function was investigated. RGA analysis was performed on the transfer functions to determine the optimal loop pairing for a 4 X 4 feedback control structure. PI controllers were designed based on IMC tuning. The controllers were implemented and simulations representing the servo and the regulatory problems were carried out.

Contributions specific to this work are:

- A dynamic model was built step by step via a steady state model. This method of building the column can be used for any chemical system as such where the physics of the system is known.
- A 4 X 4 controller structure was designed for the system to take care of both servo and regulatory problems.

In available literature no modeling and control analysis of benzene hydrogenation has been done. As such this work represents the first model developed for benzene hydrogenation.

### **Future Work**

This work forms the foundation for simulation and control studies of reactive distillation processes involving benzene hydrogenation. As such there are a variety of possibilities of extending this work:

- There process under investigation is a potential candidate for feed forward control studies. With the feedback controllers in place for the model, these controllers can be implemented and improve plant operations.
- Introduction of cascade control can also be another area which can be investigated.
- Investigation can also be done in finding out if choosing different set of controlled and manipulated variables gives any possible advantage over the present work.

## NOMENCLATURE

F	Feed total mass flow rate
$h_L$	Unit mass Enthalpy of Liquid
$h_V$	Unit mass Enthalpy of Vapor
$k_i$	Equilibrium constant for $i^{\text{th}}$ species
L	Liquid outlet total mass flow rate
$M_L$	Liquid mass holdup
$M_V$	Vapor mass holdup
P	Pressure
$R_i$	Rate of generation of component i
$\rho_L$	Liquid density
$\rho_V$	Vapor density
T	Temperature
$V_T$	Total volume of the container
$x_i$	Mass fraction of $i^{\text{th}}$ species in the liquid
$y_i$	Mass fraction of $i^{\text{th}}$ species in the vapor
$z_i$	Mass fraction of $i^{\text{th}}$ species in the feed
$r_1$	Rate of hydrogenation of benzene
$r_2$	Rate of hydrogenation of toluene
e	energy generated
q	Heat input from outside

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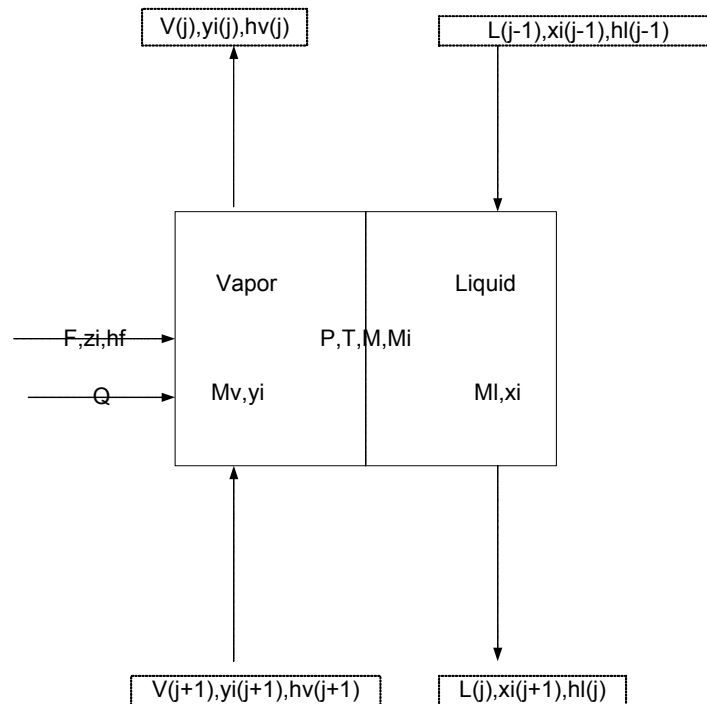
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## APPENDIX

**I. What is an index?**

The index of a DAE system indicates the degree of difficulty associated with the numerical solution of the system<sup>39</sup>. Mathematically it is the ‘minimum number of differentiations with respect to time that the algebraic system of equations has to undergo to convert the system into a set of ODE’s’<sup>40</sup>.

**II. Dynamic model of a single tray**

$$\frac{dM_i}{dt} = F_j z_i + L_{j-1} x_i + V_{j+1} y_i + R_i - L_j x_i - V_j y_i \quad (1)$$

$$\frac{dM}{dt} = F + L_{j-1} + V_{j+1} - L_j - V_j \quad (2)$$

$$\frac{dE}{dt} = F h_f + L_{j-1} h_{l,j-1} + V_{j+1} h_{v,j+1} - L_j h_{l,j} - V_j h_{v,j} + e + q \quad (3)$$

$$M = M_L + M_V \quad (4)$$

$$M_i = x_i M_L + y_i M_V \quad (5)$$

$$y_i = \mathbf{K}_i(x_i, y_i, T, P) x_i \quad (6)$$

$$\sum x_i = 1 \quad (7)$$

$$\sum y_i = 1 \quad (8)$$

$$\frac{M_L}{\rho_L} + \frac{M_V}{\rho_V} = V_T \quad (9)$$

$$M_L = f(T, P, x_i) \quad (10)$$

$$M_V = f(T, P, y_i) \quad (11)$$

$$h_L = f(T, P, x_i) \quad (12)$$

$$h_V = f(T, P, y_i) \quad (13)$$

$$E = h_V M_V + h_L M_L \quad (14)$$

$$L = f\left(\frac{M_L}{\rho_L}\right) \quad (15)$$

$$V = f(P_j, P_{j-1}) \quad (16)$$

$$\rho_L = f(T, P, x_i) \quad (17)$$

$$\rho_V = f(T, P, y_i) \quad (18)$$

Subscript i stand for  $i^{\text{th}}$  component.

Subscript  $j$  stands for  $j^{\text{th}}$  tray



## VITA

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