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This is to certify that the

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A NOVEL THERMOBALANCE FOR HIGH PRESSURE
GAS-SOLID REACTION STUDIES

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Dennis J. Miller
Major professor

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A NOVEL THERMOBALANCE FOR HIGH PRESSURE GAS-SOLID REACTION STUDIES

By

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ABSTRACT

A NOVEL THERMOBALANCE FOR HIGH PRESSURE GAS-SOLID REACTION STUDIES

By

Michael H. Treptau

A thermobalance has been developed which is capable of operating up to 900°C and 600 psi. The apparatus, a variation of the hanging reactor thermobalance, has several advantages over traditional hanging basket designs. The primary advantages are operation as a packed bed reactor, and the placement of thermocouples directly in the sample.

Several reactions have been run at 825°C and 300 psi, and the apparatus has shown the proper trends of weight loss vs. time. The accuracy, however, has thus far been unsatisfactory. The error in calculated reaction rates has been in the range of 0.1-0.2 grams/hour for reactions whose rates are 0.2-0.3 grams/hour based on actual total weight loss.

The primary source of error appears to be a baseline weight shift which occurs when switching from purge gas to reactant gas. This shift is assumed to be due to the difference in the thermal conductivities of the two gases.

To my parents, who have continuously supported and encouraged me throughout my education.

ACKNOWLEDGEMENTS

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INTRODUCTION

1.1 General Introduction

One of the most straightforward methods for the analysis of the kinetics of gas-solid reactions is to directly measure the change in weight of the solid sample as it reacts. The technology for accomplishing this is highly developed for reactions which take place at room temperature and atmospheric pressure or lower. Only in the past few decades, however, have advances been made in the development of laboratory scale equipment (thermobalances) to study the many important reactions which take place at high temperatures and pressures.

1.2 Current Thermobalance Technology

Most efforts in thermobalance technology to date have focused on modifying commercially available microbalances by enclosing them in pressure containment vessels and isolating the balance mechanism from the high temperature zone by various means. Dobner, et al. [1] review the early chronology of developments in this area. Gardner, et al. [2] review some of these devices in more detail and discuss some of their associated problems and limitations. In order to understand the rationale behind this project, however, it is beneficial to review again some of the thermobalances currently in use.

The most prevalent thermobalance is currently the hanging basket thermobalance developed by Feldkirchner and Johnson [3], and later modified by Gardner, et al. [4]. With this configuration, the sample is placed in a small basket which is then suspended into a tube surrounded by a furnace. A sketch of the apparatus is shown in Figure 1. The balance assembly is isolated from the high temperature environment, and is protected from corrosive reactant gases by a counterflow of purge gas through the chamber containing the assembly. The force on the balance arm is measured by a small mass transducer. The apparatus shown in Figure 1 is designed to operate up to 1000°C and 2000 psi.

A second, somewhat more complex thermobalance has been developed by modifying the DuPont 950 or 951 Thermogravimetric Analyzer [1,5]. This configuration eliminates the long tube necessary to isolate the balance from the high temperature zone by using a cooling coil underneath the balance assembly and a purge stream of cold nitrogen. A sketch of the DuPont 950 TG is shown in Figure 2, and the pressure containment modification is shown in Figure 3. The particular apparatus shown in Figure 3 is designed to operate up to 1100°C and 880 psi.

Forgac and Angus [6] attempted to avoid the problems with mechanical weakening of the metal wall which occur when the pressure vessel is externally heated. They constructed a cold wall thermobalance, in which an induction heating coil was placed inside the pressure vessel, and

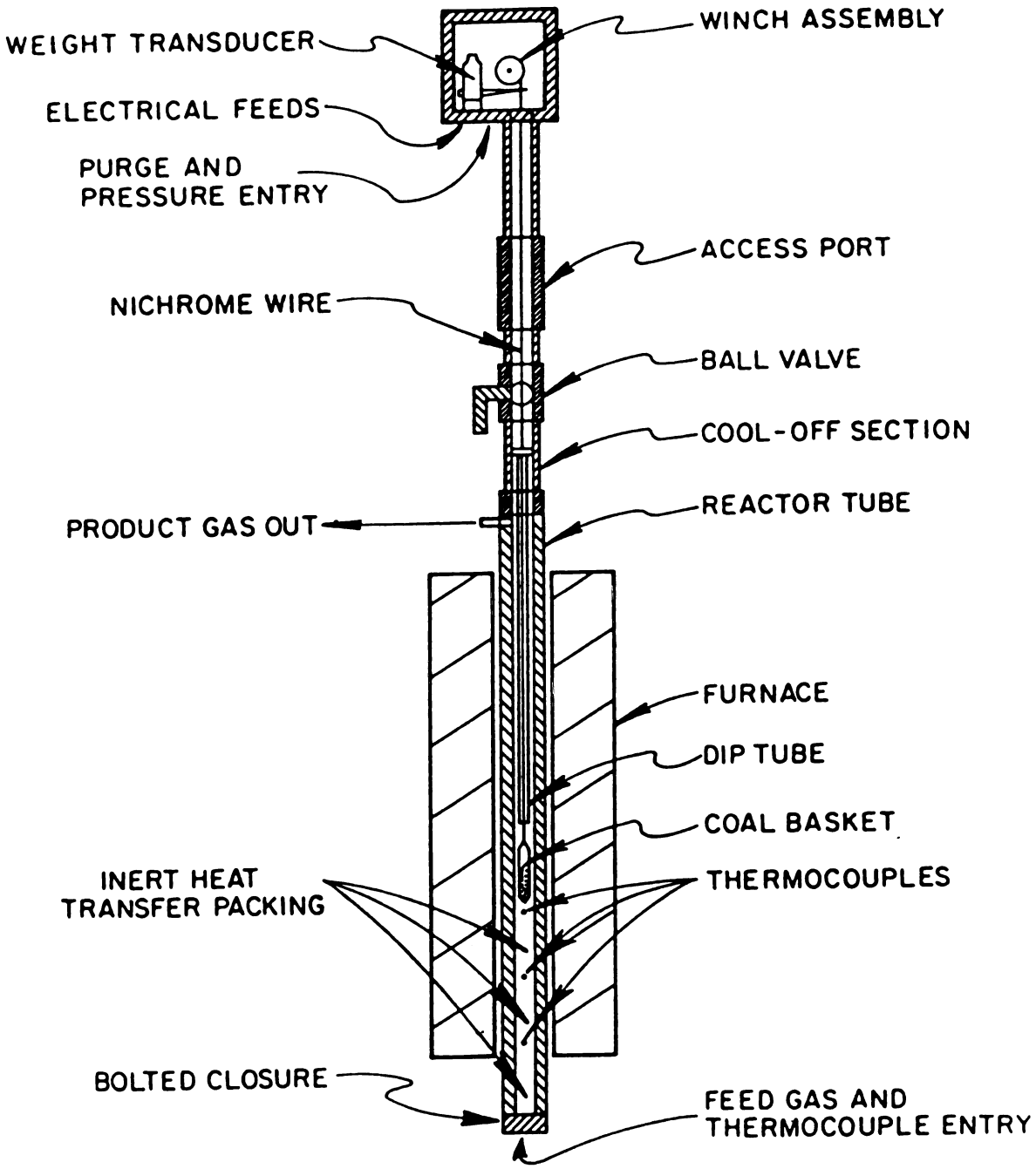

Figure 1. Hanging Basket Thermobalance
(Taken from Reference 2)

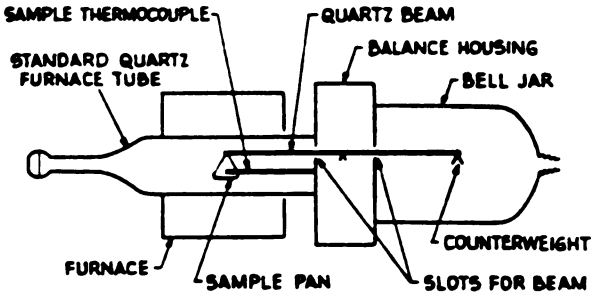

Figure 2. DuPont 950 TG (Taken from Reference 1)

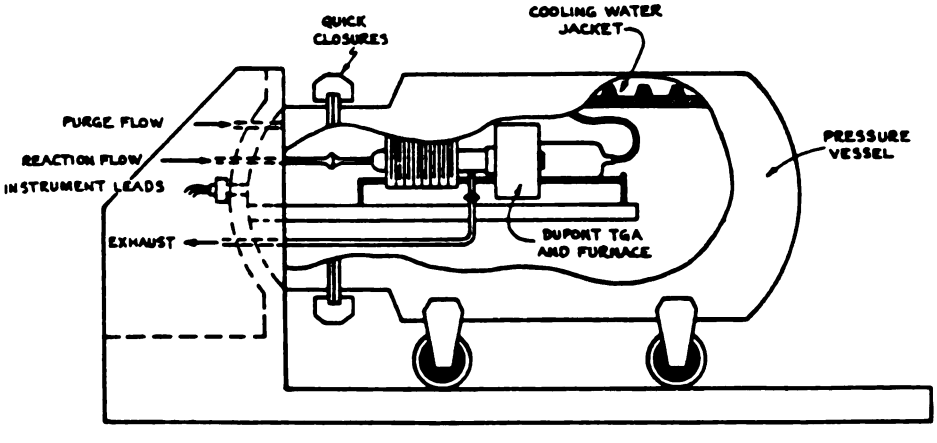

Figure 3. DuPont 950 TG with High Pressure Modification (Taken from Reference 1)

cooling water flowed through grooves in the walls of the pressure vessel, thus keeping it at or near room temperature. Again, the balance assembly is protected by a water cooled plate. Temperature measurements were made using a two-color pyrometer which was focused on the sample through a viewing port in the lid of the pressure vessel. Figure 4 shows a sketch of the reactor. The particular apparatus shown was designed to operate up to 1700°C and 2000 psi in reducing atmospheres.

Most recently, Sears, et al. [7] attempted to improve on the cold wall design by placing high temperature insulation between the heating elements and the pressure vessel walls. A sketch of the apparatus is shown in Figure 5. This apparatus can achieve temperatures of 1700°C at atmospheric pressure and pressures up to 1000 psi at lower temperatures. The maximum attainable simultaneous conditions were 1300°C and 450 psi due to altered heat transfer characteristics at high pressures. Operation in both reducing and oxidizing conditions is possible with this design.

1.3 Limitations of Hanging Basket Designs

Differences in pressure containment, mass measurement, and heater type notwithstanding, all of the previously discussed thermobalances fall into the category of hanging basket reactors. While they all are able to measure changes

Figure 4. Cold Wall High Pressure Termobalance

1. Lid
2. Viewing Port
3. 316 Stainless Steel Outer Shell
4. A286 Inner Steel Liner
5. Cooling Water Grooves
6. Electronic Balance
7. Water Cooled Plate
8. Gas Inlet Tube
9. Induction Heating Coil
10. Water Cooling Inlet
11. Be-Cu Insert
12. Teflon Liner

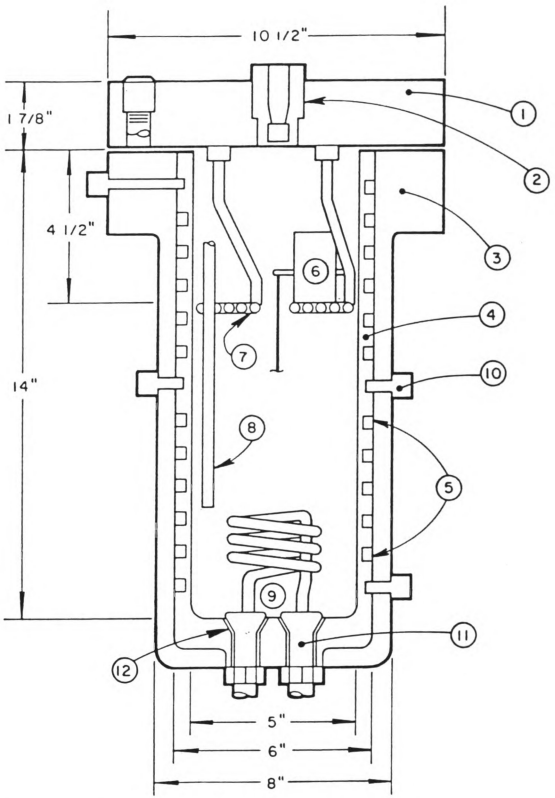

Figure 4. Cold Wall High Pressure Thermobalance
(Taken from Reference 6)

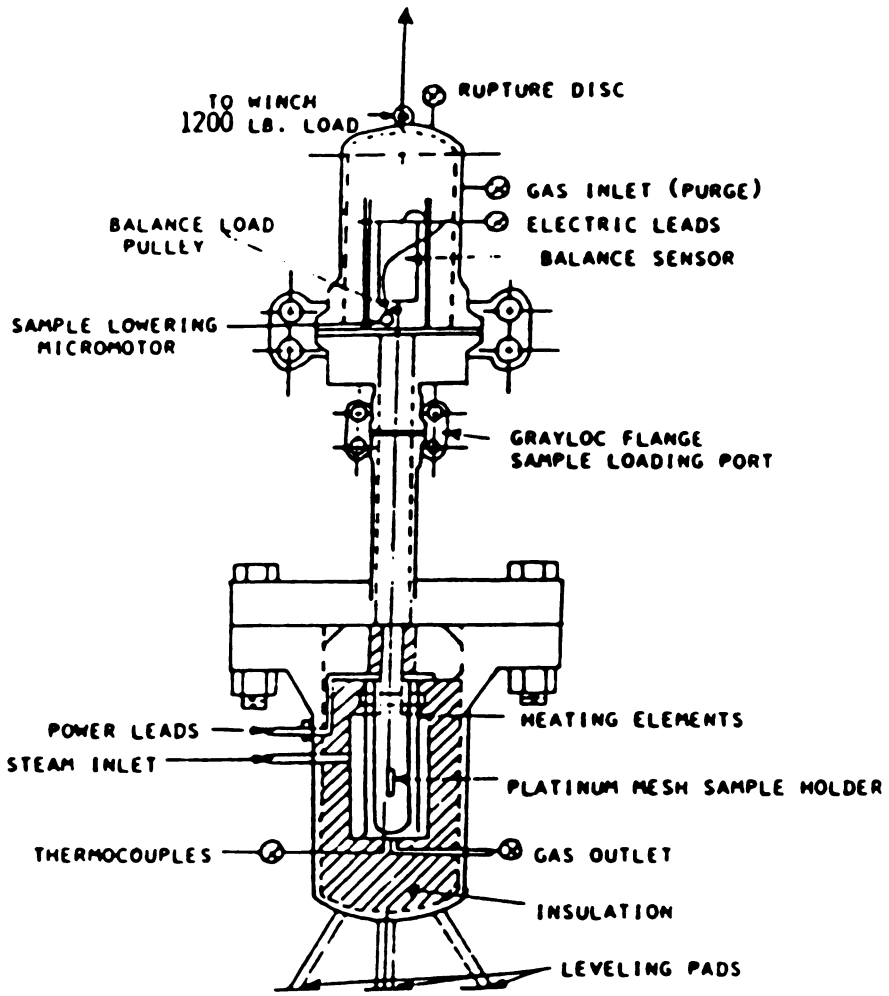

Figure 5. Thermobalance of Sears, et al. [7]
(Taken from Reference 7)

in weight of a small sample very accurately, they nevertheless have some limitations inherent to the design concept.

First, it is very difficult to directly measure the sample temperature. Because of the nature of the hanging basket design, a thermocouple clearly can never be placed directly into the solid sample undergoing reaction. Either a thermocouple is placed very close to the sample or optical pyrometry is used. In most cases, pyrometry is not a viable option from either a technical or economic standpoint, due to the difficulty in designing a high pressure reactor with an optical path to the sample. The other alternative, placing a thermocouple very close to the sample, does not always give an accurate indication of the sample temperature, as was shown by Dobner, et al. [1]. Inaccuracy can result when the gas stream is colder than the sample, or when radiative hotspots are present. An elevated sample temperature could occur either from inadequate gas preheating, or if the solid is reacting rapidly in an ignited state because of the exothermicity of the reaction.

The second major limitation of the hanging basket design is the fact that the reactant gas flows around the basket and the sample, rather than directly through the sample. This means that one has little control over the magnitude of the external mass transfer resistances between the flowing gas and the sample basket. In addition, the reactor cannot be operated as either an integral or

differential bed reactor, since the majority of the gas bypasses the sample without ever coming near it.

The third concern, which has received very little attention, is the possibility of catalytic effects from the basket itself. Baskets have been made of stainless steel or platinum, but often the material of construction is not even mentioned. Both platinum and many of the components of stainless steel, such as nickel, are catalysts. Their effects on many reactions cannot be neglected.

1.4 Hanging Reactor Thermobalance

Gardner, et al. [2] attempted to overcome some of the limitations of the hanging basket design by constructing a hanging reactor thermobalance. With this design, the entire reactor and its contents are weighed during the course of a reaction. A schematic of the system constructed by Gardner, et al. [2] is shown in Figure 6. Their reactor weighs 300 pounds, the bulk of which is tared off by a counterweight suspended from the other end of a balance arm. A 0-1 lb. force transducer measures the difference in weight between the reactor and the counterweight. The sample size is therefore limited to around one pound. A large furnace surrounds and heats the entire reactor, and the apparatus is designed to operate up to 1100°C and 1470 psi. This design presents a new set of problems, since the reactor must be weighed with the gas lines and thermocouple wires attached, but it

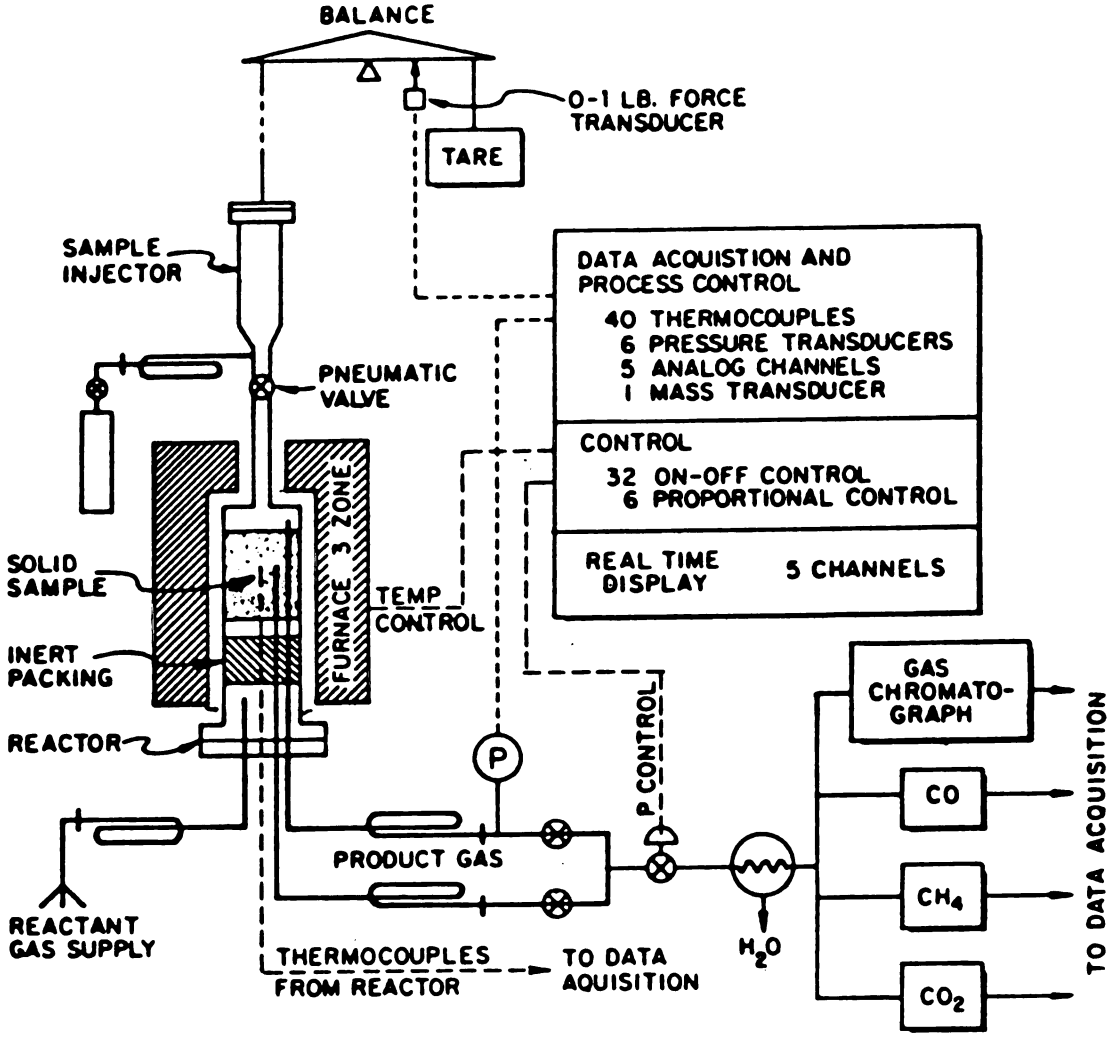

Figure 6. Hanging Reactor Thermobalance
(Taken from Reference 2)

also eliminates many of the problems associated with the hanging basket design.

First, thermocouples can now be placed directly into the solid sample without adversely affecting the weight measurement, providing accurate sample temperature measurement with fast response time. This is only true, of course, if the external thermocouple wires also do not affect the weight measurement. Secondly, the reactor is now essentially a packed bed tubular reactor. All reactant gas must flow directly through the sample, which means external mass transfer resistances can be reduced by increasing the gas flow rate. Also, product gas analysis can be performed to extract kinetic information on the basis that the system is functioning either as a differential bed reactor or a plug flow reactor.

1.5 Limitations of the Hanging Reactor Thermobalance

As previously stated, a new set of problems arises with the type of design presented by Gardner, et al. [2]. Most of the problems are associated either with the large size of the reactor or the fact that the entire reactor, including the sample, the gas in the reactor, and the vessel itself, must be weighed. The combination of these conditions means that a small change in weight of a large mass must be measured to extract kinetic information. As Gardner mentioned, this requires a balance with high sensitivity at large loads. With all gas lines and thermocouple wires

attached, the hanging reactor thermobalance of Gardner, et al. [2] can detect a change in mass of approximately 0.5 gram. This means that for adequate accuracy, a large sample must be used. The large sample size leads to problems with concentration and temperature gradients within the sample, as well as prohibitive materials costs when expensive reactants are used.

Also as a result of the large reactor volume, fluctuations in gas density, especially at high pressure, become important. This is because all the gas contained within the reactor is being weighed. Gardner and coworkers [2] reported that for CO_2 at 500°C and 1000 psi, a fluctuation of one psi caused an apparent mass change of 20 mg, and a fluctuation of 1°C caused an apparent mass change of 40 mg. This problem is clearly directly related to the reactor volume, and means that the system requires very precise temperature and pressure control to avoid large disturbances in the weight measurement.

The combination of external heating and weighing the entire reactor also leads to problems. Oxidation of the hot reactor walls which are exposed to air could cause significant errors in the weight measurement, although proper material selection should keep this effect under control. Gardner and coworkers used Inconel 617 with an aluminum/chromium diffusion coating.

Noise in the mass measurement due to convective currents between the furnace and the hot walls of the reactor

is also a problem. Data from Gardner, et al. [2] show noise with magnitude on the order of one gram. Care must be taken to provide appropriate baffling in order to eliminate weight fluctuations from convective currents.

The final problem is the possibility of either contamination or catalytic effects from the reactor walls for certain reactions. As was the case with the baskets used in hanging basket thermobalances, the reactor walls in Gardner's balance may not be inert to all the types of reactions one might wish to study. Gardner and coworkers assumed that the large sample size would make wall effects negligible, but it is not clear whether they were referring to the fluid mechanics at the wall or its catalytic effects.

This summarizes the various thermobalances currently in use, along with their associated strengths and limitations. Looking at the features of these balances leads to the conclusion that the potential exists for combining some of these strengths along with some new ideas into a new thermobalance which will eliminate the major difficulties associated with collecting solid reaction rate data at elevated temperatures and pressures.

CHAPTER II

NEW DESIGN CONCEPT

2.1 Conceptual Design of Reactor

It was desired to design an apparatus to carry out reactions simultaneously at 600 psi and 1000°C and accurately measure the sample weight. The new thermobalance design set forth in this work is intended to improve upon the design of Gardner, et al. [7] by accomplishing the following:

A) Reducing the size of the reactor and the balance arm necessary to support it, thus increasing sensitivity and reducing the amount of solid sample needed.

B) Employing a cold wall design by using internal heating surrounded by high temperature insulation to protect the pressure vessel.

C) Providing inert reactor walls of nonporous alpha-alumina.

A sketch of the reactor is shown in Figure 7. Materials of construction were chosen with the K_2CO_3-catalyzed carbon hydrogasification reaction specifically in mind, and are discussed in Chapter III. The gas flows in through fittings located on the cover of the pressure vessel. The gas then flows down through an annular space between the heater and insulation, where it is preheated. It then turns around and flows up through the alumina reactor tube containing the solid sample. The gas flows out through another set of fittings in the cover. The inside diameter of the reactor

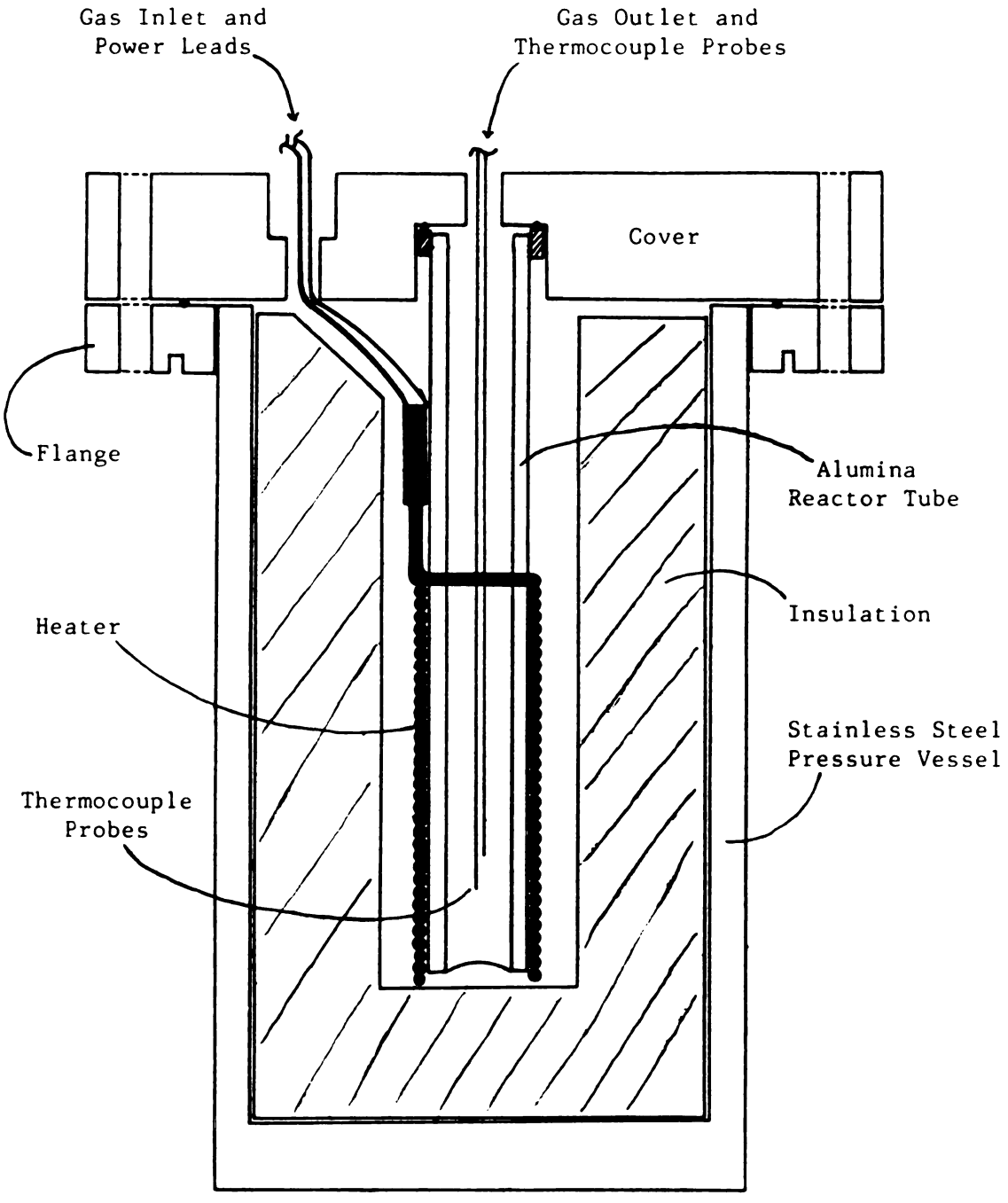

Figure 7. Sketch of Reactor

tube is 1/2 inch and the heated zone is three inches long, allowing samples of up to three grams to be heated uniformly.

The size of the reactor is limited by the capacity of the balance arm used in the weighing system. This was set at 30 pounds. On the other hand, the need for adequate insulation between the heater and pressure vessel walls limited the minimum size of the reactor. One inch of insulation was used, thus setting the outside diameter of the pressure vessel to be four inches. At steady state, this amount of insulation keeps the walls of the pressure vessel at a sufficiently low temperature so that significant weakening of 316 stainless steel does not occur (see Appendix B for heat transfer calculations).

2.2 Evolution of Balance System Design

2.2.1 Configuration I

Initially, the balance system was set up as shown in Figure 8. The reactor rested on a Mettler electronic balance, and the difference in weight between the reactor and counterweights was measured directly. This design had two difficulties, both associated with the fact that the counterweights were hanging.

First, it was difficult to make fine adjustments to the amount of weight which was tared off by the counterweights. Since they were hanging, it was not possible to simply move them closer or farther away from the fulcrum of the balance

arm. Fine adjustment could only be accomplished by adding or removing small weights from the counterweight.

The second and more serious problem was that slight swinging of the counterweights caused undamped oscillations in the balance. This eventully resulted in the reactor bouncing rather violently on the Mettler balance.

2.2.2 Configuration II

The problems from Configuration I were solved by setting up the balance system as shown in Figure 9. The counterweights rested on a plate mounted on the balance arm, rather than being suspended freely underneath the balance arm. This design gave accurate and stable weight readings until the reactor was heated. As the the pressure vessel walls heated slowly to a steady state temperature of about 190°C, they also expanded slightly, causing an apparent mass shift of up to 15 grams over the course of the four hour heatup time. The pan of the Mettler balance also became quite warm, even with a 3/8 inch thick piece of insulation between the pan and the reactor.

After the weight stabilized as the reactor walls reached a steady state temperature, and the reactant gas was fed to the reactor, the weight reading began shifting again, causing large errors in the results of the experiment being run. The weight shift was on the order of 1-2 grams/hour. This phenomenon was assumed to be due to the difference in heat capacity and thermal conductivity

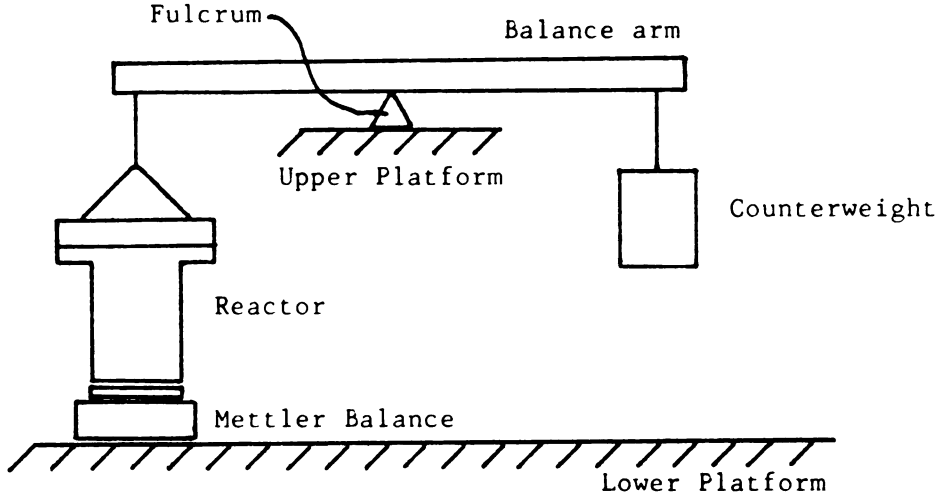

Figure 8. Schematic of Configuration I

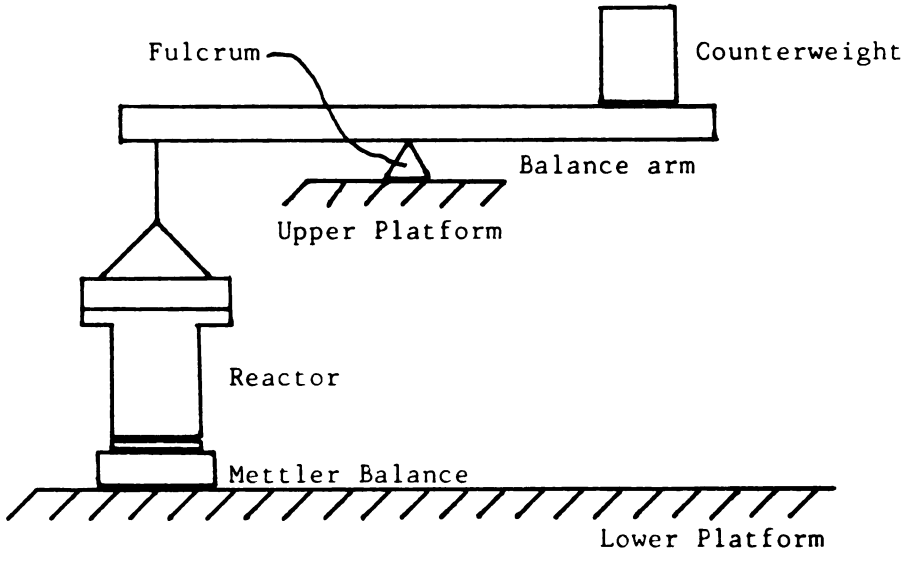

Figure 9. Schematic of Configuration II

between the purge gas (helium) and reactant gas (hydrogen or carbon dioxide). Switching gases was thus suspected to cause the temperature of pressure vessel walls and top to shift to a new steady state value.

2.2.3 Configuration III

The next step in the search for a workable balance configuration, in which the difficulties associated with Configuration II were eliminated, was to interchange the positions of the reactor and counterweights. This configuration is shown in Figure 10. The reactor was isolated from the Mettler balance, preventing it from overheating, and the reactor could expand without exerting a force directly on the Mettler balance.

With the tubing and wiring attached, however, expansion of the reactor was still detected by the balance. Two to three hours were required for the weight to stabilize after the heat was turned on. This was undesirable, since K_2CO_3 reacts with the carbon at elevated temperatures. When the reactor was switched from purge gas to reactant gas, a fairly reproducible weight shift occured. This apparent weight shift was much less severe than for the previous balance configuration, on the order of 0.1-0.2 grams per hour.

A problem associated with placing the counterweights on the Mettler balance, rather than the reactor itself, is that a change in weight registered on the Mettler balance does not correspond directly to the change in weight of the

reactor and its contents. This is due to the fact that the two arms of the balance have different lengths. Correcting for this difference can be accomplished by placing a series of small weights on the reactor and noting the corresponding weight change registered by the Mettler balance. A scaling factor can then be obtained for converting the apparent weight change data into an actual weight change. The scaling factor for this configuration is 0.6-0.7 (apparent change/actual change), depending on the exact position of the reactor on the balance arm.

2.2.4 Configuration IV

In an effort to eliminate the error due to weight shifts which occured during heatup and the switching of gases, the reactor was suspended underneath the balance arm. This configuration is shown in Figure 11. This configuration allowed the reactor to expand freely as the temperature of the pressure vessel walls increased. Unfortunately, a new problem developed with this configuration which made this goal difficult to achieve.

The problem was similar to that of the first configuration, in that a slight disturbance introduced undamped oscillations into the system. The system appeared to have a resonant frequency of about 2 Herz, with an amplitude of several grams. At room temperature, this effect was controlled by placing a foam rubber pad between the weights and the Mettler balance. At elevated temperatures, however,

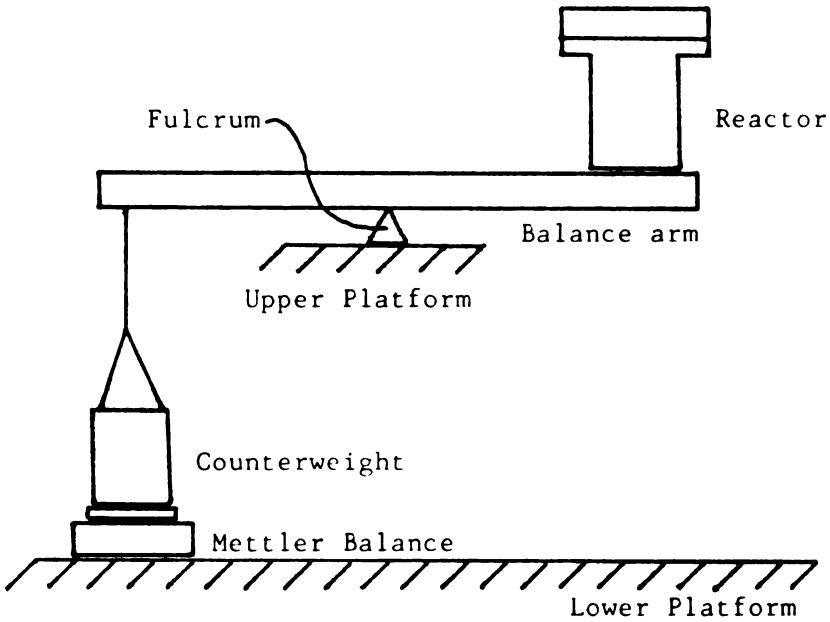

Figure 10. Schematic of Configuration III

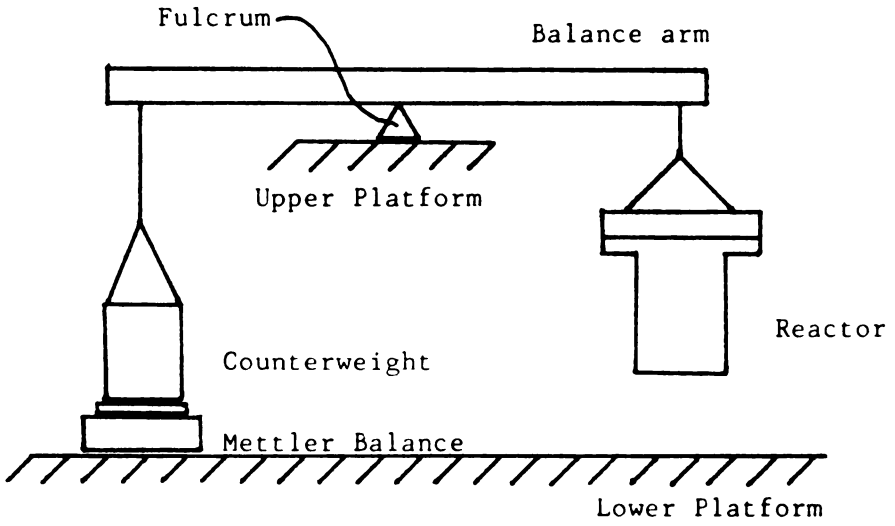

Figure 11. Schematic of Configuration IV

convective currents made it extremely difficult to prevent oscillations, even with the use of baffles around the reactor.

It was then decided that the most promising method for operating the thermobalance successfully was to use Configuration III, where the reactor rests on the balance arm. The photograph in Figure 12 shows the reactor in this configuration, and gives a clearer idea of the spatial arrangement of the various components.

In this configuration, a correction must be made for the shift in the baseline weight which occurs when switching from purge gas to reactant gas. The magnitude of this shift can be determined by running a blank experiment using a sample of inert alpha-alumina rather than carbon. The results of the experiments performed with this configuration are presented in Chapter V.

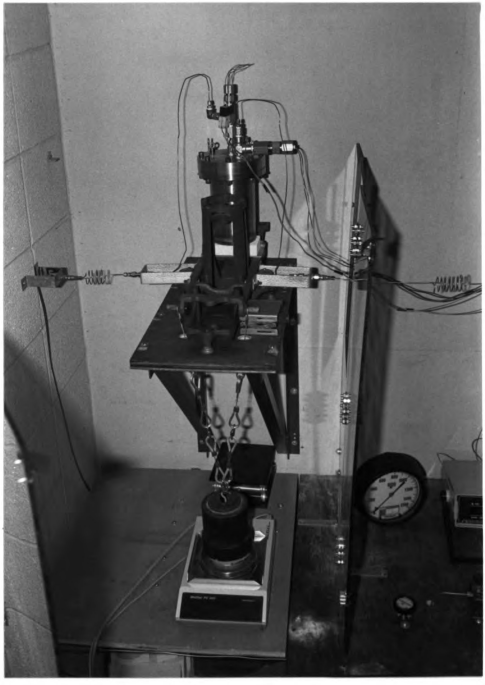

Figure 12. Photograph of Thermobalance

CHAPTER III

DETAILED DESCRIPTION OF APPARATUS

3.1 Reactor Design

A sketch of the reactor is shown in Figure 7. Important dimensions will be mentioned as each particular component is discussed. Fully assembled, the reactor weighs 22 pounds.

3.1.1 Pressure Vessel

The external pressure vessel consists of a seamless cylindrical shell, a bottom piece and flange welded on to the shell, and a cover which is bolted to the flange and sealed with an o-ring. The material of construction was chosen to be 316 stainless steel. This was preferred over other stronger and more expensive alloys because of its resistance to attack by hydrogen and its lower nickel content than alloys such as Inconel. The mechanical strength of 316 stainless steel decreases rapidly with increasing temperature [8], but this problem was mitigated by the use of proper insulation, to be described shortly.

The design pressure of the pressure vessel, excluding the flange, is 1100 psi for a 200°C wall temperature. The required wall thickness can be determined by using the design equation for thin-walled cylindrical shells [8].

$$t = \frac{PR}{SE - 0.6P} \qquad (1)$$

where

t = shell thickness, in.

P = pressure, psi

S = allowable stress, psi. S=13300 psi at 200°C [8].

E = joint efficiency, assumed to be 1.0 for a seamless tube.

R = inside radius, 1.75 in.

Therefore,

$$t = \frac{(1100)(1.75)}{13300 - 0.6(1100)} = 0.15 \text{ in.}$$

A 1/4 inch wall thickness was chosen for its commercial availability.

The required thickness for the bottom of the pressure vessel is given by [8]

$$t = d\sqrt{\frac{0.13P}{SE}} \qquad (2)$$

where

d = inner diameter, 3.5 in.

Therefore,

$$t = 3.5\sqrt{\frac{(0.13)(1100)}{(13300(0.8)}} = 0.40 \text{ in.}$$

Here, the joint efficiency E was assumed to be 0.8 in order to take the welds into account. A bottom thickness of 1/2 inch was chosen for its commercial availability and to provide an adequate safety factor.

The flange has a thickness of 1/2 inch and is one inch wide. The design calculations are quite involved and are shown in detail in Appendix A. Due to an error in

calculation, the maximum operating pressure of the flange, and thus the pressure vessel, is 600 psi.

For design purposes, the cover can also be considered a type of flange. The minimum thickness, however, was not set by stress considerations, but rather by the need for adequate thickness in order to properly install the various fittings. For this reason, a one inch cover was chosen. Based on the relative size of the cover to the bottom of the pressure vessel and the flange, this is clearly thick enough to withstand the maximum operating pressure of 1100 psi.

The cover is fastened down by eight 5/16 inch high-strength bolts. The calculations for the bolt load are shown in Appendix A. The seal for the vessel is provided by a 4 3/8" ID x 1/8 inch thick Viton o-ring.

All gas and electrical feed-throughs are located on the cover. A Conax multiple probe seal is used for the thermocouple probe feed-through, and a Conax power lead gland provides the feed-through for the heater power wires. Swagelock fittings of various sizes and types provide access for gas flow. Due to the stringent space constraints, the various fittings are stacked on top of one another such that the electrical leads enter the pressure vessel through the same holes in the cover as the gas inlet and outlet flows. Figure 13 shows a schematic of the cover assembly.

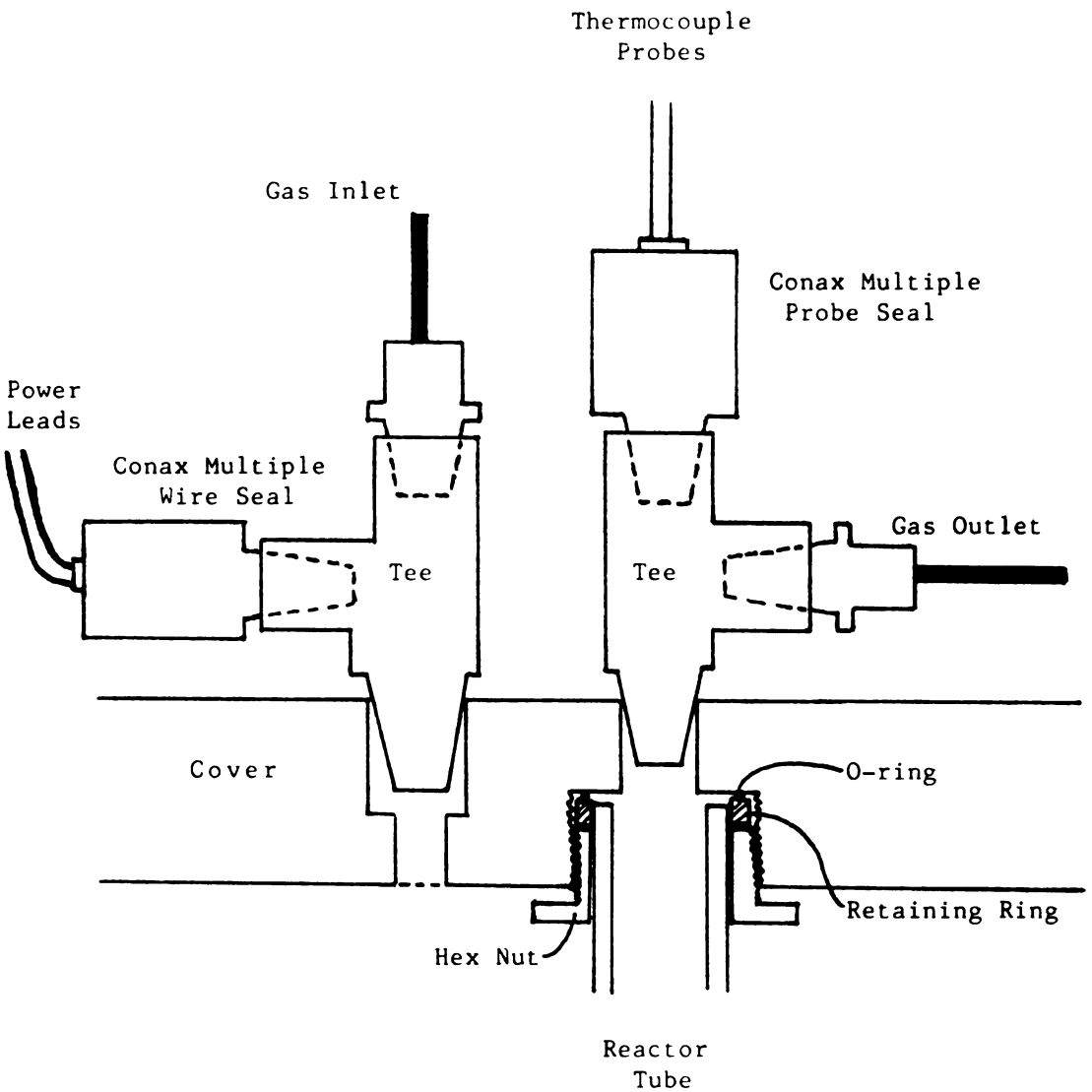

Figure 13. Schematic of Cover Assembly

3.1.2 Insulation

A suitable insulating material for this application must have very low thermal conductivity in order to meet the space and power requirements, while maintaining the stainless steel pressure vessel at a moderately low temperature. It must also hold its shape and be able to withstand temperatures to 1000°C.

The insulating material which meets these requirements is standard K-20 firebrick, suitable for use to 1100°C. It can be easily formed to fit snugly into the pressure vessel, and has been coated with a high temperature ceramic impregnant manufactured by Aremco Products, Inc. This provides a durable, nonporous surface for the otherwise fragile and dusty firebrick. The coating also prevents absorption of gases into the insulation.

The heat transfer calculations shown in Appendix B indicate that for a reactor temperature of 750°C and one inch of insulation, the surface of the pressure vessel can be expected to reach approximately 130°C at steady state. Experimental results using a foil thermocouple for measuring surface temperatures show that the surface temperature actually approaches 175-190°C, depending on ambient conditions.

3.1.3 Reactor Tube

The reactor tube is a 5 1/2 inch cylinder made of 99.7% pure, nonporous alpha-alumina. The inner diameter is 1/2 inch and the outer diameter is 3/4 inch. Alumina was chosen

for its inertness to a wide variety of materials and conditions, including attack by molten alkali metal salts. Conclusive literature on the latter, however, is difficult to find.

A stainless steel ring is cemented to the top of the tube with a high temperature ceramic adhesive, also from Aremco. The ring with tube attached is then pressed up against the bottom of the pressure vessel cover by a nut which slips over the outside of the tube and is screwed into the threaded hole on the underside of the cover (see Figure 13). A Kalrez o-ring provides a seal between the ring and cover, preventing short-circuiting of the gas flow.

Inside the tube, the solid sample rests on a bed of 28-48 mesh alpha-alumina powder. A plug of quartz wool supports the contents of the tube. The height of the alumina layer can be adjusted to assure that the thermocouple probes lie directly within the reacting sample. The alumina also prevents molten alkali metal salts from coming into contact and reacting with the quartz wool. In order to prevent entrainment of fine particles, another layer of alumina is placed on top of the sample.

3.1.4 Heater

The heater is a 750 watt Mightyband heater manufactured by the Tempco Electric Heater Corporation. It is three inches long with an inner diameter of 3/4 inch, thus

fitting snugly over the reactor tube walls. Heat transfer calculations given in Appendix B show that at steady state, roughly 100 watts are required to maintain the reactor at 750°C. Thus, the 750 watt heater was deemed to have adequate power to provide reasonably rapid heatup without being oversized from a temperature control standpoint. Experimental results show that the time required to heat a sample to 825°C is approximately four minutes, and the power required at steady state is about 200 watts.

3.1.5 Temperature Measurement

Two Chromel-Alumel thermocouple probes are used inside the reactor to measure the sample temperature. Each is protected by a .040 inch sheath made of Inconel 600, which has a maximum operating temperature of 1150°C. The probes are placed at differing depths within the sample in order to determine whether axial temperature gradients exist. It is also possible, although more difficult, to control the radial position of the probes with the use of alumina guide tubes inserted into the sample.

3.2 Balance System

The photograph in Figure 12 shows the spatial arrangement of the balance system with the reactor in place and all lines attached.

3.2.1 Balance Arm

The balance arm is part of a two-pan Toledo Scale with a 30 lb. capacity for each pan. All excess parts were removed until only the balance arm and its base remained. The balance arm is arrested by placing a small jack under each end of the balance arm. An aluminum plate is fastened to one end of the arm, on which the reactor or counterweights can be placed. Weights can also be suspended from either end of the arm through holes in the platform. Brackets extend from the fulcrum on both sides of the arm and contain fittings to bring the gas lines in at the fulcrum and then along the balance arm to the reactor. The balance assembly rests on a platform consisting of a 1/2 inch wooden plate on top of a 1/4 inch steel plate bolted onto two triangular brackets of 1 1/4 inch angle iron. The entire assembly is mounted on a one foot thick concrete wall (part of the radiation shielded chamber in room 265) in order to minimize the effect of building vibrations.

3.2.2 Weight Measurement

A Mettler PE 360 top-loading electronic balance is used to measure the change in weight of the reactor's contents with time. It has a capacity of 360 grams with measurement to the nearest 0.01 gram. Within this capacity, it also has a fine (Delta) range of 0-60 grams with a sensitivity of 0.001g.

The Mettler balance has a digital output which is interfaced to the RS-232 port of a Zenith personal computer. The BASIC program shown in Appendix C samples the weight at a user-specified time interval and writes the data to a disk file for future plotting and analysis. The maximum sampling rate with this program is 1 Hz, which is sufficiently rapid to provide an essentially continuous weight vs. time curve of slow gas-solid reactions.

In order to minimize the effect of air currents, the thermobalance is located in the corner of a small, 8 1/2 x 7 1/2 foot room. The two exposed sides and the top are enclosed with clear Lexan sheets which also serve as projectile shields.

3.3 Gas Flow System

Figure 14 shows a schematic of the gas flow system. The reactant gas is Grade 5 Hydrogen, with a maximum impurity level of 10 ppm. The hydrogen flows through a bed of 1/8 inch Linde 3A molecular sieves in order to further purify it. The pressure is controlled by the cylinder regulator, and the flowrate is controlled by a Linde Mass Flow Controller with a flow capacity of 200 SCCM. The flow controller has the capability to be interfaced with a personal computer for remote control and data aquisition. It also has four channels for blending applications.

For rapid pressure relief, there is a line upstream of the reactor which leads directly to a vent. During normal

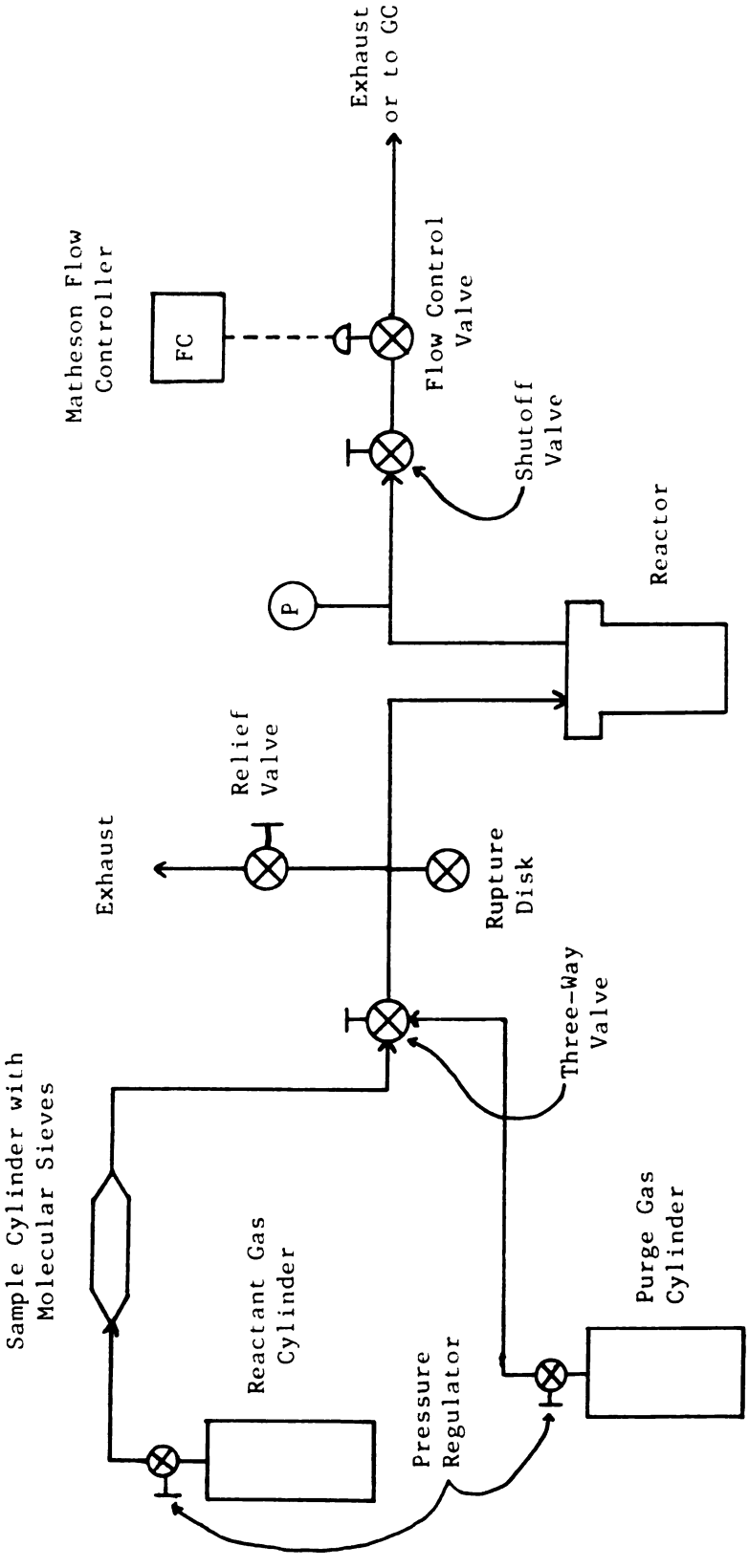

Figure 14. Gas Flow System

operation, this line is closed by a shutoff valve. The system also contains a Fike rupture disk upstream of the reactor to protect the system from excess pressure due to regulator failure or operator error.

Currently, the exit gas from the reactor flows directly to the vent, but this can be easily routed to a gas collection system for analysis by gas chromatography.

3.4 Attachment of Tubes and Wires to Reactor

The most critical factor in the successful operation of this particular thermobalance is the manner in which the tubes and wires are attached to the reactor. Since the tubes and wires will inevitably exert forces on the balance arm, it is essential that the system be designed in such a manner that these forces remain constant during operation.

The gas lines, power, and thermocouple wires never come in contact with the balance arm itself, only with the bracket which extends from the fulcrum. One-sixteenth inch copper or stainless steel tubing is used in this portion of the system in order to minimize the force that the tubing may exert on the balance arm. The inlet tube, supported by a bracket located two feet from the balance arm, travels along the axis of the fulcrum until it reaches the bracket attached to the balance arm. There, it passes through fittings and then continues on to balance arm. The tube then makes a right angle turn down the length of the arm and then another turn up to the inlet of the reactor. The

outlet tubing follows a similar pattern on the other side of the balance. The power and thermocouple wires are attached in basically the same manner as the tubing, resting only on the bracket and not touching the balance arm itself.

3.5 Temperature Monitoring and Control

The temperature of the sample is controlled by an Omega CN-2013 programmable temperature controller with PID control. A schematic of the temperature control and electrical system is shown in Figure 15. The output of the controller is a one amp solid state relay. This relay operates a 10 amp solid state relay which switches the heater on and off. When the controller is properly tuned (see Chapter IV for typical tuning parameters), it is able to maintain the temperature within ±1°C of the setpoint temperature.

In order to conveniently display both temperatures within the reactor, the thermocouples are connected to an Omega Model 199 ten channel thermocouple thermometer. This model has built-in cold junction compensation, as does the temperature controller. This eliminates the need for any ice bath reference junctions. The thermocouple thermometer has an accuracy of ±1°C.

For data acquisition purposes, the thermocouples are also connected to a Tecmar Lab Master multiple-channel interface board installed in an expansion slot of the Zenith personal computer. The board has 12 bit resolution, and

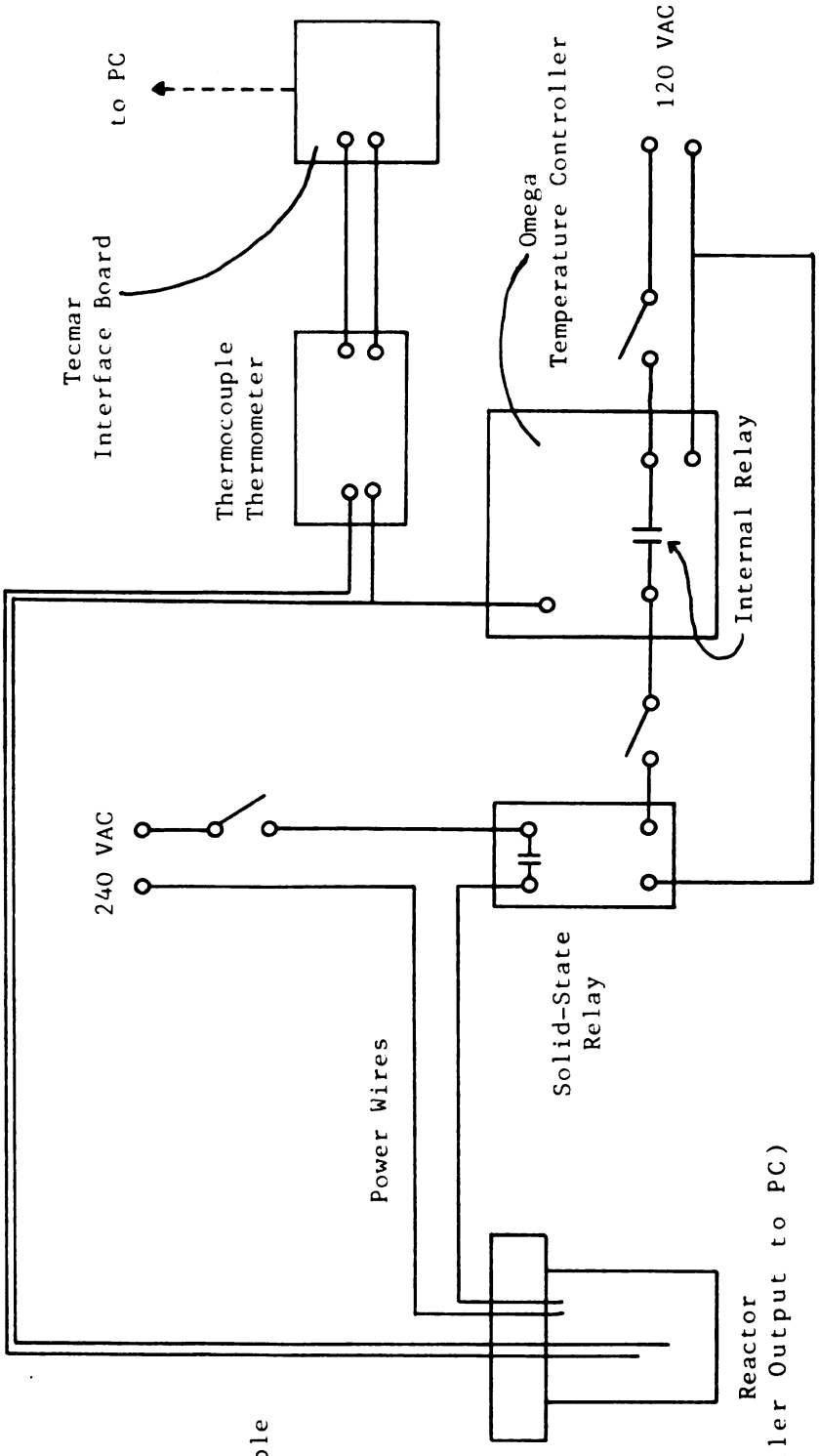

Figure 15. Temperature Control System

converts the thermocouple voltage to a digital signal, which is then sampled and converted to a temperature with software (see program in Appendix C).

This summarizes the various components of the thermobalance and their functions. A detailed operating procedure for the apparatus is presented in Chapter IV.

CHAPTER IV

OPERATING PROCEDURE

The detailed operating procedure for the thermobalance is outlined in the following set of steps. Also refer to the operating manuals of the various components.

4.1 Sample Loading and Preparatory Steps

With the reactor vessel removed from the balance assembly, open the vessel and place the cover assembly on a ring stand. Remove the heater from the reactor tube, and then remove the reactor tube from underneath the cover. Support the tube on a stand using a test tube clamp. Weigh and load the quartz wool plug (0.06-0.1g), bottom alumina layer (1.5-2.5g), sample (2-3g), and top alumina layer (~2.0g), respectively. The quartz wool is loaded from the bottom, while all other materials are loaded from the top with a long-stem funnel. The bottom layer of alumina is added to a depth such that the tips of the thermocouple probes will be located within the reacting sample. Reattach the tube to the underside of the cover, sliding the thermocouple probes into the powder sample. Make sure that the o-ring is in place, and firmly tighten the nut finger tight. Do not twist the reactor tube, since the thermocouple probes are now imbedded in the solid sample. Replace the heater, rotating it into position such that the power wires are coiled underneath the nut. Close the pressure vessel, making sure that the top end of the heater is aligned into

the slot in the insulation to allow the cover to close freely. At the same time, make sure that the bolt holes are aligned. If not, rotate the heater until alignment is achieved.

Make sure that the balance arm is fully arrested on both ends. Place the reactor assembly onto the balance arm and attach all gas lines, power and thermocouple wires. Make sure that no wires touch anything until they reach the bracket two feet from the apparatus.

Turn on the Mettler balance. Remove the jack from under the counterweights and replace it with the Mettler balance, making sure that the Mettler is centered under the counterweights. Lower the weights onto the Mettler balance very slowly in order to prevent bouncing. This is very important, since only very small oscillations are damped. If the Mettler does not register 250 ±20 grams, raise the weights off of the Mettler balance and arrest the balance arm. Adjust the position of the reactor by sliding it away from or toward the fulcrum so that when the weights are lowered, the Mettler registers 250 ±20 grams.

After the weight reading has stabilized sufficiently, switch the Mettler into Delta Range. Close the draft shield and begin the helium purge flow at 5.0 SCCM and 50 psig, following the instructions found on page 18 of the operating manual for the Linde Flow Controller.

Make sure the reactant gas pressure at the regulator is below the intended operating pressure. If not, partially

open the relief valve shown in Figure 14. Switch the three-way valve to reactant gas in order to bleed down the pressure, then switch back to helium and close the relief valve. Allow the system to sit overnight for 12-18 hours in order to purge all oxygen and stabilize the weight.

After the weight has stabilized, place a 100 mg calibration weight on the reactor and remove it several times to determine the average weight change detected by the Mettler balance. This is the calibration factor to be used in conjuction with the data collected from this run.

4.2 Experimental Procedure

Turn on the power to the temperature controller and allow it to warm up. Make sure that the output is turned off and the controller is in AUTO mode. Also turn on the power to the thermocouple thermometer. The flow controller and Mettler balance will already have been running during the stabilization period.

Set the purge gas flow rate to the desired value, typically 100-200 SCCM. Raise the pressure at the regulator to approximately 100 psi below the intended operating pressure to allow for pressure rise during heatup.

If the setpoint temperature is to be different from that of the previous experiment, it can be changed by pressing either the up or down arrow on the keypad of the temperature controller. If the setpoint is much different from that of the previous run, the controller may have to

be retuned. This can be accomplished by following the directions found in Section 3.10 of the operating manual for the temperature controller. Tuning parameters for 825°C are: proportional band, 8%; reset, 0.87 R/M; rate, 0.18 M; cycle time, 15 sec. Since tuning is accomplished experimentally, this should be performed before a reacting sample is loaded into the reactor.

Turn on the heater by pressing the START/STOP key and turning on the OUTPUT switch. After the temperature reaches the setpoint, raise the pressure at the regulator the rest of the way to the desired operating pressure. Also make sure the reactant gas is at the desired operating pressure. It will now take 2 1/2 to 3 hours for the weight to stabilize.

During this period, prepare the computer for data aquisition. Turn on the computer and load the BASIC program "GRAPHM". Choose the data aquisition mode from the initial menu and follow the instructions given.

After the weight has stabilized, begin data aquisition by pressing the "Return" key and switch to reactant gas by turning the three-way valve. Change the flow rate if desired. If the pressure begins to drop, adjust the regulator accordingly. Allow the experiment to run until the desired time or extent of reaction has been reached.

4.3 Shutdown

After the experiment has been completed, terminate data aquisition by pressing capital "S" on the keyboard. Turn off the heater by following the reverse procedure for turning it on, and switch back to purge gas. When the reactor has cooled and the pressure has been relieved, the reactor can be removed from the balance by using the reverse procedure for placing it on the balance. It can then be opened and the contents removed and weighed. After the sample has been removed, reattach the reactor tube and heat in air to approximately 750°C in order to remove the sample residue sticking to the walls of the tube. The reactor is now ready for the next experiment.

CHAPTER V

RESULTS AND DISCUSSION

The following results, with the exception of the sensitivity tests, were obtained while the balance was in Configuration III, which was determined to be the best configuration of the four tested. The reasons for this choice will be discussed in the next chapter.

5.1 Balance Behavior

5.1.1 Sensitivity

Sensitivity tests were performed while the thermobalance was still in Configuration II (see Chapter II for a description). The results should also be applicable to Configuration III, however, since the positions of the reactor and counterweights were simply interchanged.

The sensitivity tests were performed by placing a calibration weight on the reactor and recording the change in weight detected by the Mettler balance. The calibration weight was then removed, and the change in weight recorded once again. Since there was a considerable amount of noise present, the procedure was repeated several times and the results averaged.

The results for the sensitivity tests performed with all tubing and wiring attached to the reactor are summarized in Table 1. These results show that the intrinsic error involved in detecting weight changes with this system is less than 5% for changes as small as 20 mg. In

Configuration III, part of this error is taken into account when the scaling factor to account for unequal balance arm lengths is determined.

Table 1. Sensitivity Test Results

Actual Weight	Average Apparent Weight	Std. Deviation
100 mg	94.5 mg	3.5 mg
50 mg	49.9 mg	1.8 mg
20 mg	19.1 mg	1.0 mg

5.1.2 Noise

On a short time scale on the order of seconds, the magnitude of noise in the data is on the order of 20 mg for a sample temperature of 825°C. This is noticeably more noise than is encountered at room temperature, and is probably due to convective currents set up by the relatively hot walls (~180°C) of the reactor.

On a longer time scale, the data exhibit noise of a larger magnitude. Excursions of 0.1-0.2 grams over the course of several minutes occasionally occur in what would otherwise be a fairly smooth curve. These excursions can be seen clearly in the data to be dicussed shortly. During the course of many tests, the thermobalance has shown itself to be very sensitive to small fluctuations in room temperature. This was most dramatically demonstrated when the air conditioning was turned off during a run, and the reading on the Mettler balance shifted several tenths of a gram

over the course of the next half hour. This is assumed to be the primary cause of the long term noise.

5.2 Reactions

5.2.1 Description of Reaction Conditions and Sample

Three reactions were performed at 825°C, 300 psi and a hydrogen flow rate of 100 SCCM while the thermobalance was in Configuration III. In each case, the solid sample consisted of 2.00 grams of 50-200 mesh activated cocoanut charcoal impregnated with 22.96% by weight K_2CO_3. For clarity, the three reactions are referred to as Runs 1, 2, and 3, in the order in which they were performed. Runs 1 and 2 lasted two hours, while Run 3 lasted three hours. The actual total weight loss of sample during a reaction was determined by weighing the carbon sample, alumina powder, and quartz wool plug before and after the reaction. All runs were performed according to the procedure detailed in Chapter IV.

5.2.2 Reactor Heatup

Before switching to hydrogen, the reactor was heated in helium at 825°C and 300 psi for 2 1/2 hours to allow the reading on the Mettler balance to reach a steady state value. The helium flow rate was approximately 100 SCCM. A typical heatup curve showing the reading of the Mettler balance vs. time is shown in Figure 16.

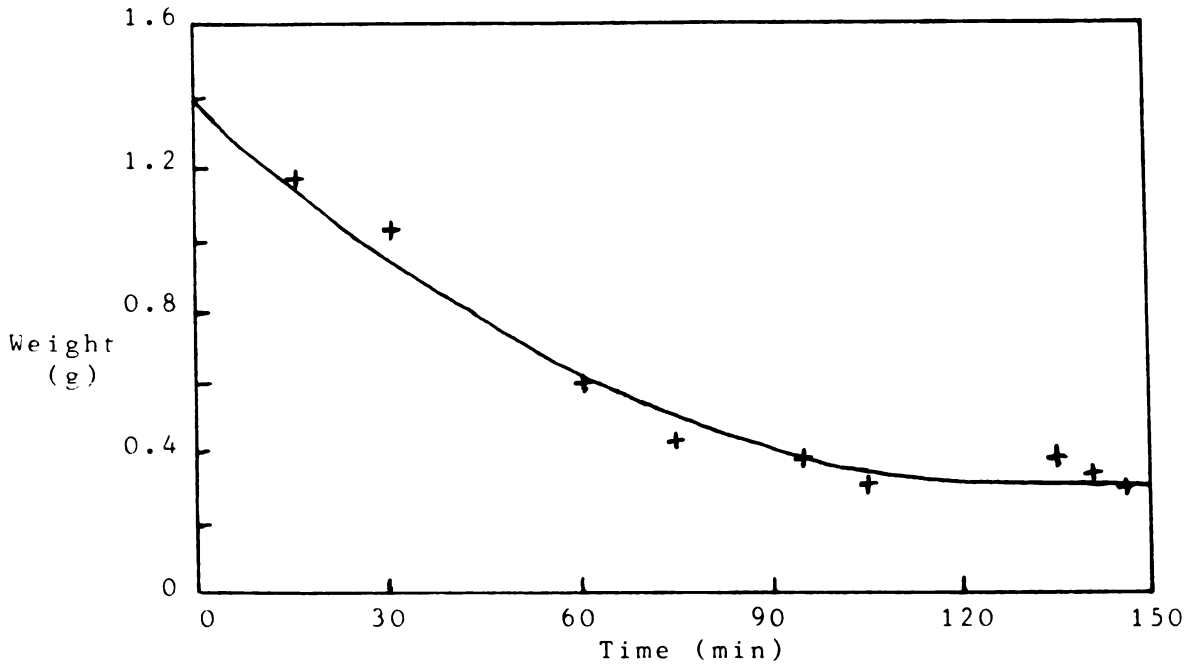

Figure 16. Typical Heatup Curve

In order to know the initial weight of the sample at the time hydrogen is introduced into the reactor, it is necessary to determine how much of the sample is lost during the heatup period. To accomplish this, experiments were carried out where the reaction was stopped after the heatup period, rather than switching to hydrogen. The sample was then removed and weighed. It was found that 0.43 grams of the sample is lost during this time.

The following mechanisms are assumed to contribute to the weight loss which occurs during the heatup period:

A) Reaction of the catalyst with the carbon by the reaction

$$K_2CO_3 + 2C = 2K + 3CO. \qquad (3)$$

If all the catalyst reacts, and all the free potassium deposits on the reactor tube walls, the maximum measured weight loss due to this mechanism would be 0.28 grams. This is not believed to occur, however, because cooled samples which are exposed to air after reaction become quite hot. This heating is most likely caused by the reaction of free potassium with oxygen, and is evidence that some potassium remains on the carbon.

B) Volatilization of the water of hydration present in the potassium carbonate. This amounts to 0.06 grams per water molecule of hydration, or a maximum of 0.18 grams if the K_2CO_3 is all in the form of $K_2CO_3 \cdot 3H_2O$ (the highest hydrate form).

C) Reaction of carbon with the water and oxygen present as impurities in the helium purge gas. Theoretically, this should be negligible, since the maximum amount of water plus oxygen in Grade 5 helium is 2.5 ppm.

D) Vaporization of adsorbed water or other impurities from the activated carbon.

E) Vaporization of adsorbed water from the alumina bed.

The weight loss due to the last two mechanisms was determined by heating an uncatalyzed sample in helium at

30 psi for 3 1/2 hours. The helium flow rate was 100 SCCM. Under these conditions, it was found that 0.09 grams were lost. Assuming that a negligible amount of carbon reacts with impurities, this amount corresponds to the devolatilization of water or other impurities adsorbed on the carbon and the alumina.

5.2.3 Raw Data

The raw data for the three reactions are shown in Figures 17-19. The absolute values of the weights are arbitrary, and depend on the exact time after placing the reactor on the balance arm that the Mettler balance was switched into Delta Range. Since the reactor rests on the opposite end of the balance arm from the Mettler balance, positive slope indicates weight loss in the reactor.

5.2.4 Baseline Shift

In addition to the reactions, blank runs were performed in order to determine the shape of the baseline shift caused by the switch from purge gas to reactant gas. Inert alpha-alumina was used in place of the K_2CO_3/carbon sample for these runs.

Figure 20 shows a typical baseline shift which occurs when switching from purge gas to reactant gas. This blank run is one of four which were performed between Runs 2 and 3. All of them were nearly identical in shape. By a

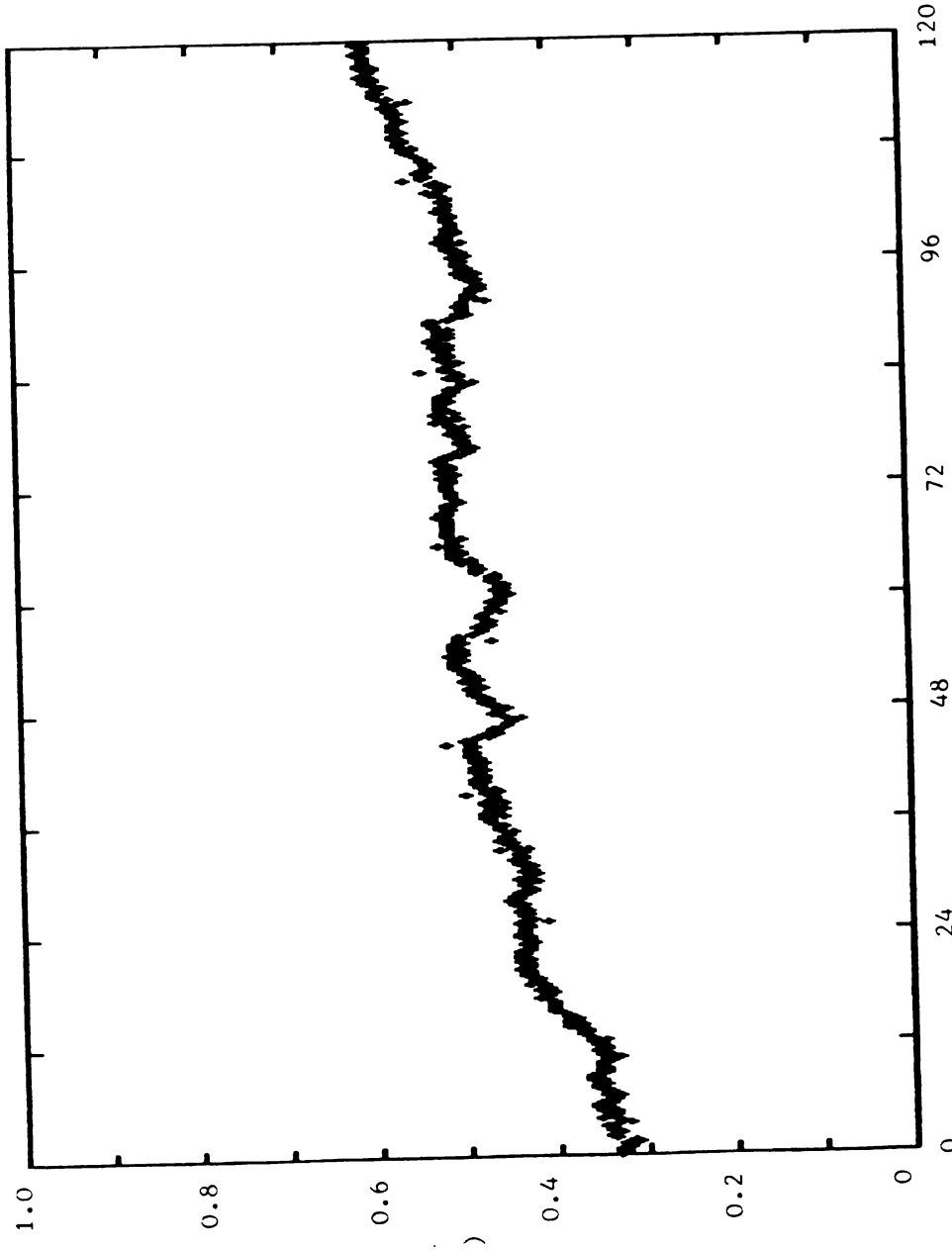

Figure 17. Raw Data for Run 1

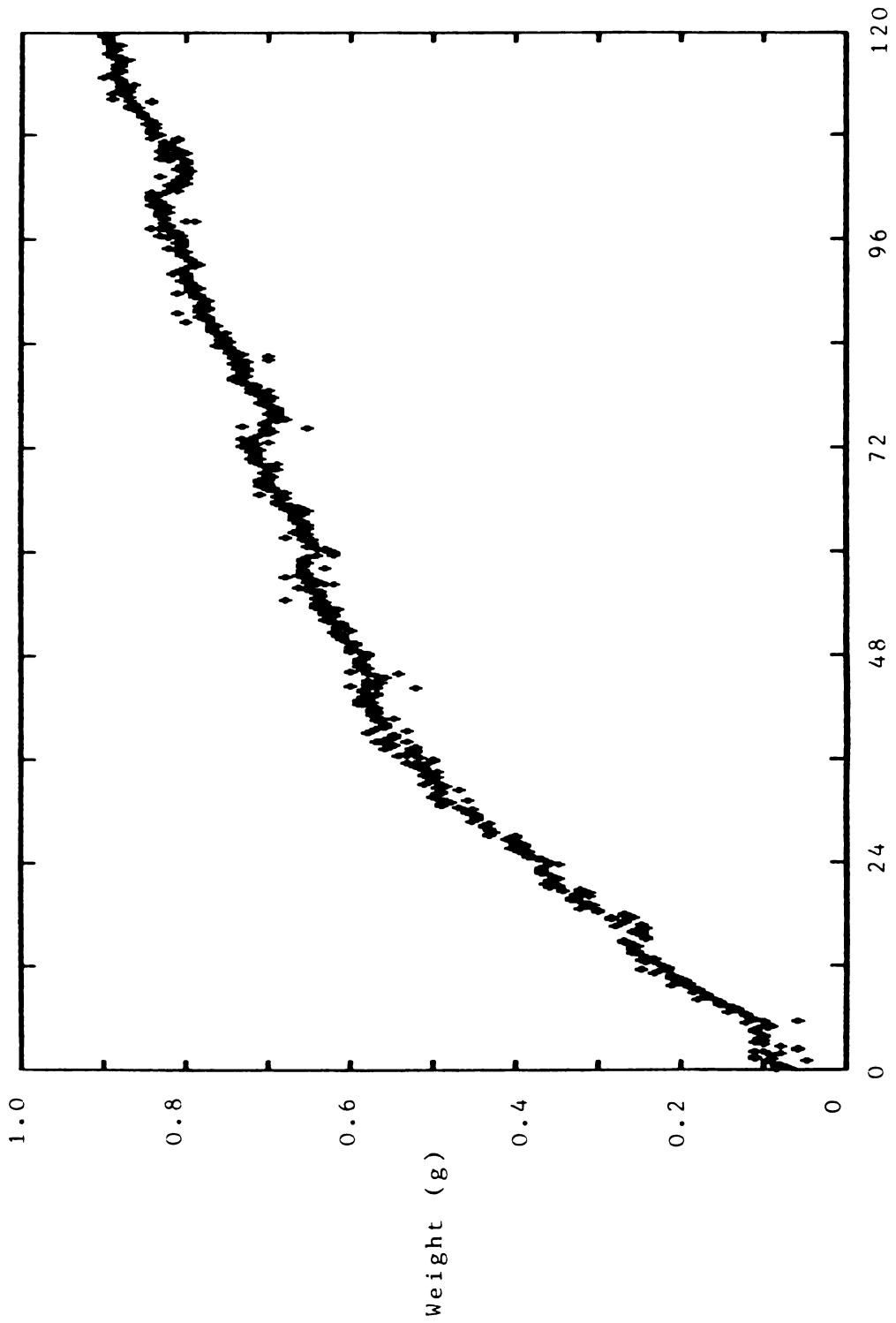

Figure 18. Raw Data for Run 2

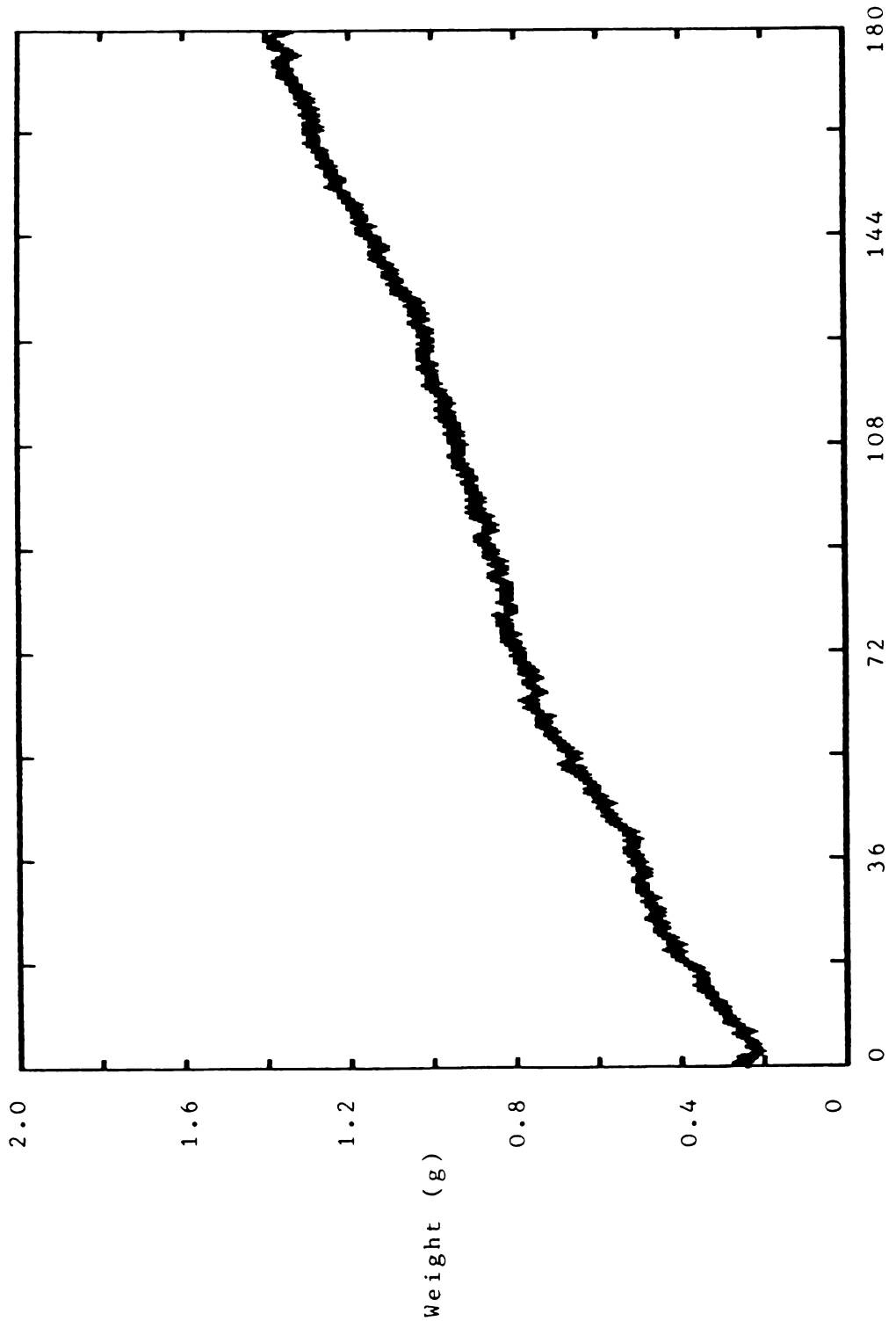

Figure 19. Raw Data for Run 3

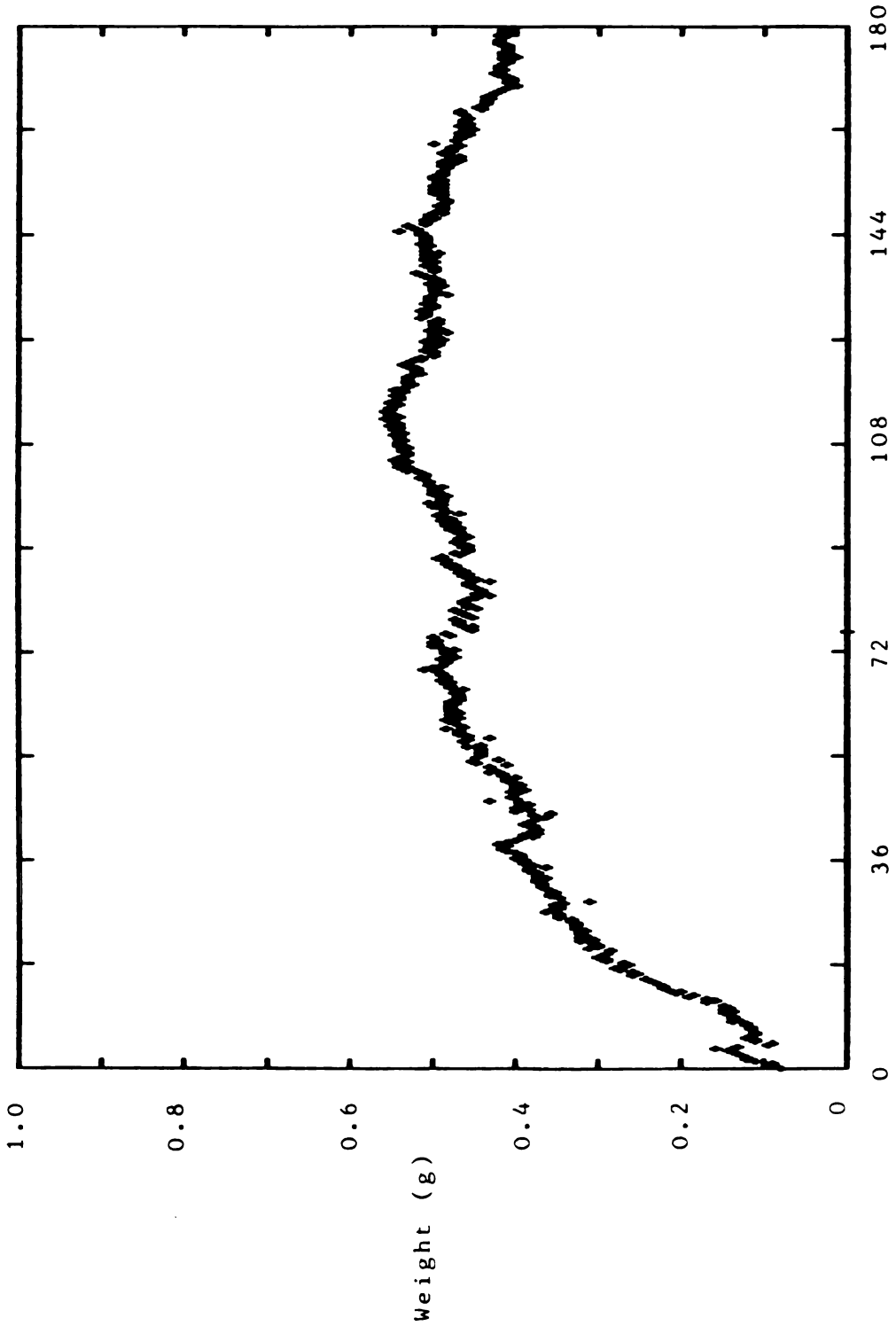

Figure 20. Typical Baseline Shift

least-squares fit, the third order polynomial which best fits this curve was determined to be

$$w = 9.0\times10^{-7}t^3 - 1.06\times10^{-4}t^2 + 9.23\times10^{-3}t - 0.0334 \quad (4.a)$$

Equation 4.a was then subtracted point by point from the data of Runs 2 and 3.

A different polynomial was used as the baseline shift of Run 1. In this case, a polynomial was constructed which reflected the average baseline shift of three blank runs which were performed prior to Run 1. This polynomial is

$$w = 3.28\times10^{-7}t^3 - 1.06\times10^{-4}t^2 + 9.23\times10^{-3}t - 0.0334 \quad (4.b)$$

As mentioned in Chapter II, this baseline shift, which appears to be the major source of error, is apparently caused by differences in the physical properties of the two gases being used. Degassing of some sort from the reactor or insulation can be ruled out, since the magnitude of the shift is larger than the maximum weight of gas which the reactor could contain. In Chapter VI, the majority of conclusions and recommendations will be focused on minimizing this shift and making it more reproducible.

5.2.5 Final Results

In order to arrive at a final plot of weight vs. time, several steps of data manipulation are required. These steps are outlined as follows:

A) The ordinate is shifted vertically so the curve begins at the point (0,0).

B) From the resulting curve, the polynomial which corresponds to the baseline shift caused by switching from purge gas to reactant gas is subtracted.

C) The ordinate is rescaled to account for the fact that the actual weight change is ~68% of the recorded weight change (see Chapter II for discussion).

D) The weight values are subtracted from the initial weight of 1.57 grams in order to convert to a curve which represents weight of sample vs. time. The value of 1.57 grams is the initial weight of the sample after subtracting the amount lost during heatup (0.43 grams).

The final results of the three reactions are presented in Figures 21-23 and summarized in Table 2.

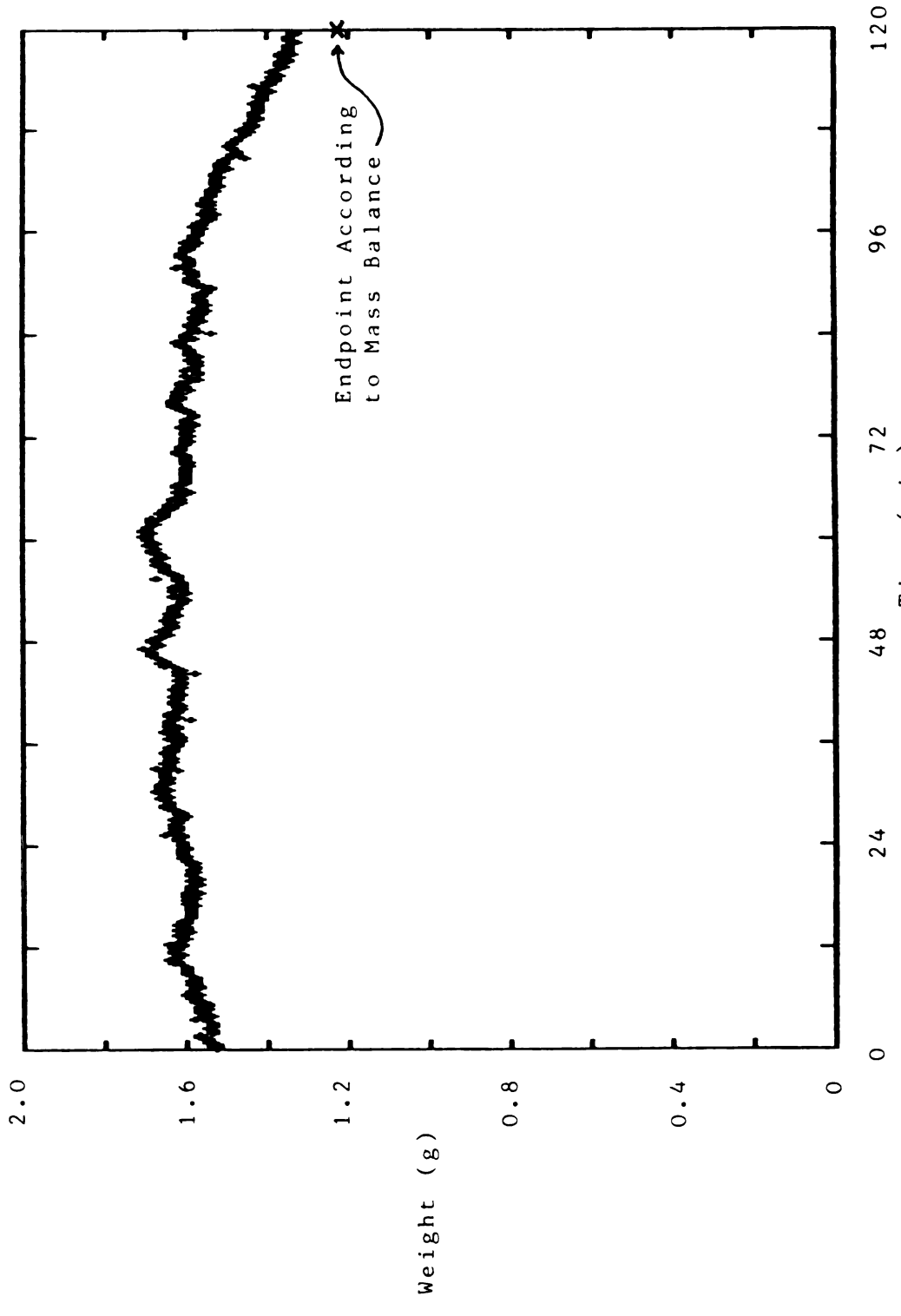

Figure 21. Final Results of Run 1

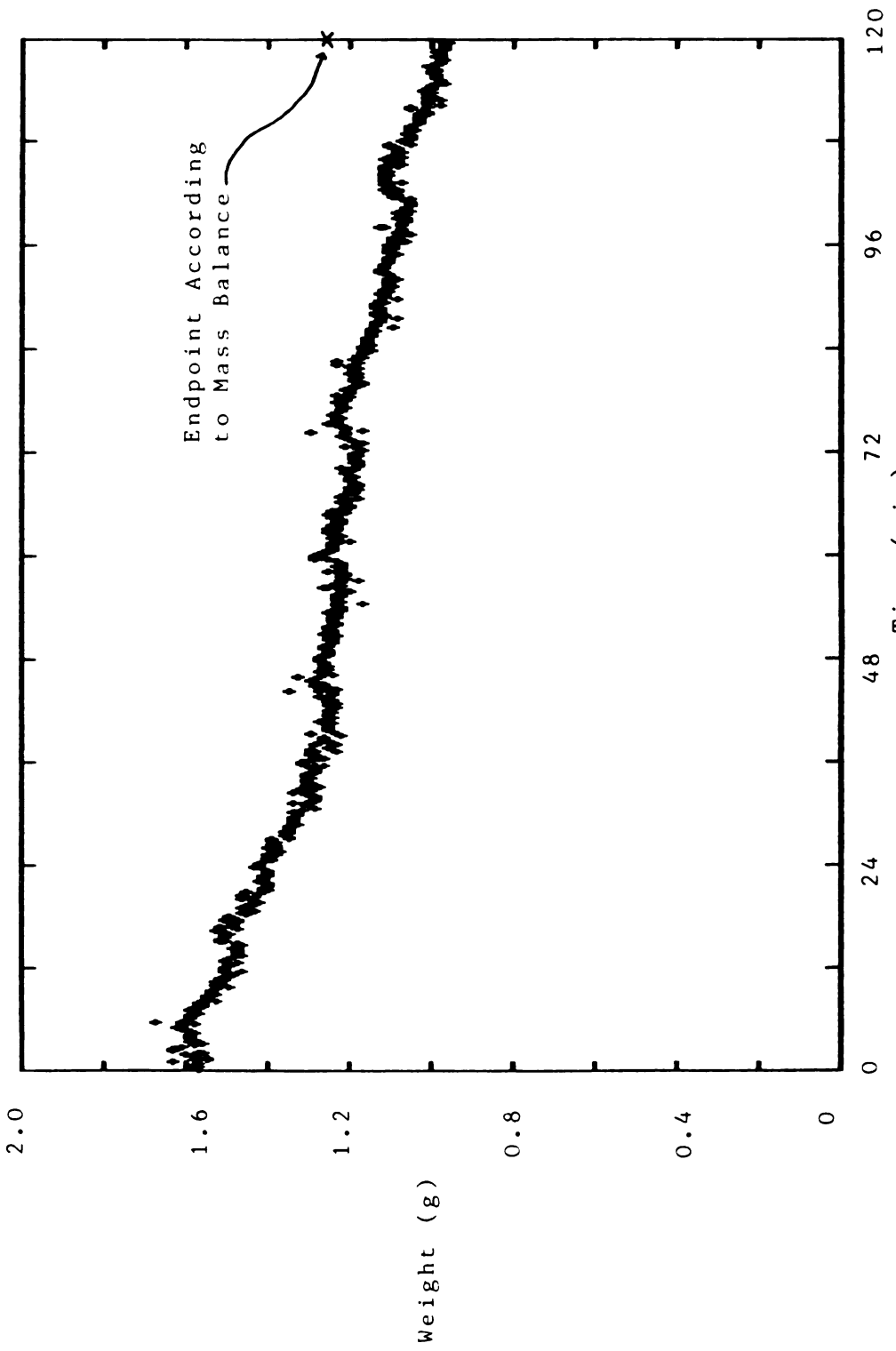

Figure 22. Final Results of Run 2

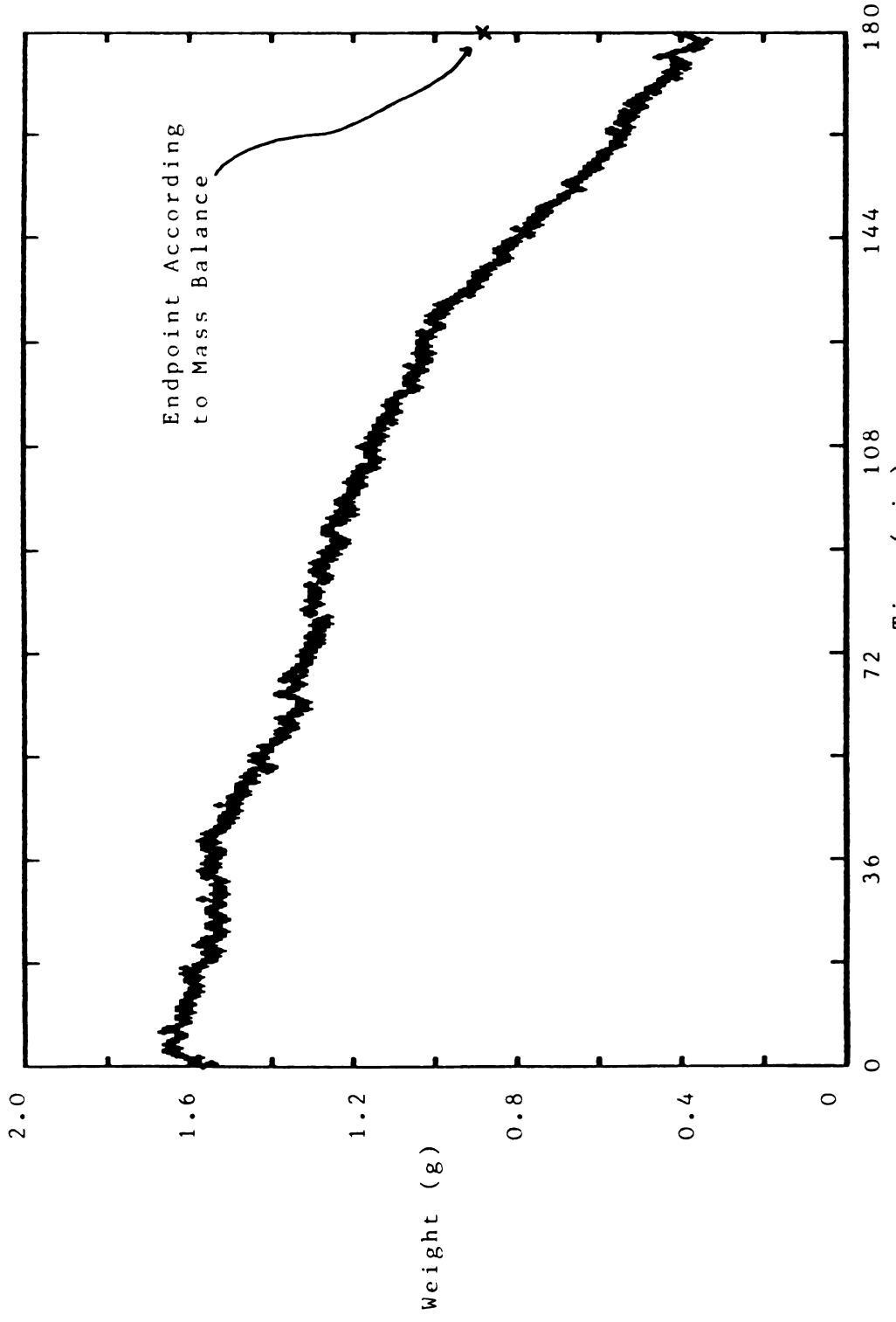

Figure 23. Final Results of Run 3

Table 2. Summary of Reaction Results

Run	1	2	3
Sample weight (g)	2.00	2.00	2.00
Heatup time (min)	145	144	150
Weight loss during heatup (g)	0.43	0.43	0.43
Reaction time (min)	120	119	180
Total weight loss (g)	0.78	0.75	1.11
Actual amount reacted (g) (Total weight loss - Loss during heatup)	0.35	0.32	0.68
Apparent amount reacted according to thermobalance (g)	0.2	0.6	1.2

5.2.6 Discussion of Final Results

The weight vs. time curve of Run 1 is virtually flat for the first 90 minutes, but this does not necessarily mean that no reaction occured until 90 minutes had elapsed. A more plausible conclusion is that the baseline shift which was subtracted from the raw data differs significantly in shape from the baseline shift which actually occured. In fact, the curve for Run 1 looks quite reasonable after simply applying the scaling factor of 0.68 to the data.

The curves for Runs 2 and 3 are virtually identical for the first two hours, the entire length of Run 2. The actual total weight loss for two hours, however, is about 0.32 grams, or a factor of two less than the weight loss indicated by the curves. After three hours, the error in

the total weight loss increases to 0.57 grams for Run 3. Again, the magnitude of these errors suggests that they are caused by the uncertainty in the magnitude of the baseline shift.

CHAPTER VI

CONCLUSIONS AND RECOMMENDATIONS

6.1 Conclusions

Several reactions have been successfully carried out at 825°C and 300 psi, and the reactor has proven to be very durable. The thermobalance has recorded the proper trends in weight loss, but the accuracy has thus far been unsatisfactory.

In spite of its disadvantages, Configuration III appears to provide the best opportunity for success among the various configurations tested. This configuration has the shortest heatup time and the smallest baseline shift, and there is no chance of overheating the Mettler balance. The primary disadvantage is that the reactor must be removed from the balance arm in order to open it and change samples. The reason that this is such a problem will be discussed shortly.

The 2 1/2 hour wait required to allow the weight to stabilize during heatup is unacceptable for reactions such as the primary one for which this reactor was designed, namely, K_2CO_3 catalyzed hydrogasification of carbon. At elevated temperatures, K_2CO_3 reacts with carbon, meaning the catalyst loading and weight of carbon at the point where the gasification reaction is begun will not be the same as their initial values.

While the thermobalance is sensitive enough to detect weight changes on the order of 10 mg, the mass vs. time

trace of an actual reaction still has a large amount of error associated with it. The error in the overall mass balance is on the order of 0.1-0.2 grams over the course of a two hour reaction, giving an uncertainty in the reaction rate on the order of 0.1-0.2 grams/hour.

The bulk of this error is assumed to be due to the uncertainty in the shape of of the baseline shift which occurs after switching from purge gas to reactant gas. This can only be measured by performing blank runs before the sample is loaded and after the sample has been removed following a reaction. Unfortunately, all gas lines, power and thermocouple wires must be detached from the reactor, and the reactor must be removed from the balance arm each time a sample is changed. This means that for any new sample, the baseline shift cannot be determined exactly, since the exact position of the reactor and attached lines cannot be consistently reproduced. Fluctuations in room temperature also seem to affect the shape of the baseline shift.

6.2 Recommendations for Modifications

Work will continue in an effort to improve the accuracy of the thermobalance. The following recommendations, where feasible, will be implemented. Most of these recommendations are directed at reducing the magnitude of the baseline shift and increasing its reproducibility.

A) The thermocouple and power wires should be made of the smallest guage wire allowable for the current involved. This includes using uninsulated wire wherever possible in order to minimize the force the wires exert on the balance. The fine wires would have to travel directly from the wall bracket to the reactor in order to avoid touching anything.

B) The 1/16 inch stainless steel tubing leading away from the fulcrum of the balance on both sides should be replaced with more flexible copper tubing.

C) The temprature and air flow in the room where the thermobalance is located must be more precisely controlled in order to eliminate some of the long term fluctuations in the weight reading.

D) In order to maintain the walls of the pressure vessel at a lower temperature, the outer portion of the high temperature insulation should be replaced with an insulation which has a much lower thermal conductivity.

E) The placement of radiation shields on the inner surface of the insulation and the underside of the cover may help to reduce the outer wall temperature of the reactor.

F) The reactor should be longer in order to provide a longer cool-off section between the top of the reactor and the heated zone. This would of course require the construction of a new pressure vessel. The benefits of doing this may not be as pronounced as expected, however, because at

the low gas flow rates involved, the dominant mode of heat transfer appears to be conduction.

G) Using nitrogen instead of helium as a purge gas may reduce the magnitude of the baseline shift since, like hydrogen, nitrogen is a diatomic molecule.

6.3 Recommendations for Suitable Reactions

The accuracy of the thermobalance would be improved considerably for the case of faster reactions. Assuming the error in guessing the shape of the baseline shift remains unchanged, the relative error in the measured rate of a reaction which goes to 100% conversion over the course of half an hour or so would be an order of magnitude less than for the reaction which was used for testing this apparatus.

Reactions which take place at lower temperatures should also be more suitable for this apparatus. Lower operating temperatures imply lower steady state wall temperatures, which would tend to reduce the magnitude of the baseline shift.

Since such a long heatup time is involved, reactions whose reactants are stable at high temperature in an inert atmosphere are more suitable for study with this apparatus. If K_2CO_3-catalzyed carbon hydrogasification is performed, a sample which has been placed in helium for the length of the heatup period should be analyzed to determine the actual amount of catalyst remaining at the time the switch to reactant gas is made. This can be accomplished by

radioactive labelling of the potassium. If it is found that nearly all of the K_2CO_3 reacts with the carbon during the heatup period, it would be pointless to further study this particular reaction with this apparatus.

In conclusion, this thermobalance has not yet produced results with an acceptable amount of accuracy. With further modifications, however, the potential still exists for extracting accurate kinetic information for certain gas-solid reactions.

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APPENDIX A

APPENDIX A

PRESSURE VESSEL DESIGN CALCULATIONS

Bolt Load Calculations

The bolt load is the sum of the load due to pressure and the load required to seal the o-ring. The load due to pressure is the pressure multiplied by the exposed area.

Load due to pressure

$$= \pi(2.25 \text{ in})^2 \ (1100 \text{ psi}) = 17500 \text{ lb}_f. \quad \text{(A1.a)}$$

The load to seal the o-ring equals the o-ring circumference multiplied by the force per unit length required to seal the o-ring.

Load to seal

$$= \pi(4.5 \text{ in})(100 \text{ lb}_f/\text{in}) = 1410 \text{ lb}_f. \quad \text{(A1.b)}$$

Therefore, the total bolt load is 18900 lb_f.

The proof strength of high strength (Grade 8) bolts is 120 kpsi [9]. To provide a safety factor, use 50000 psi as the working strength.

$$\text{Total bolt area required} = \frac{18900 \text{ lb}_f}{50000 \text{ psi}} = 0.378 \text{ in}^2. \quad \text{(A1.c)}$$

The tensile strength area for a 5/16 inch (nominal) diameter bolt is 0.052 in^2 [9]. Therefore, eight 5/16 inch bolts are sufficient.

Flange Calculations

The bolt circle diameter was chosen to be 5 1/4 inches, in order to ensure that the bolt holes are at least 2 1/4 diameters apart [10]. The width of the flange was then chosen to be one inch, in order to allow full seating of the bolt heads and nuts.

The flange thickness was chosen to be 1/2 inch, but due to an error in the calculation, the thickness was underdesigned. The following calculations were redone in order to determine the maximum operating pressure using the 1/2 inch thick flange.

Method of Waters and Taylor [10]:

$$p_{to} = \frac{2.858Wb}{t^2(r_1-r_o)}\left(\frac{K_1^2 \ln K_1}{K_1^2-1} + 0.1169\right) \qquad (A2)$$

where

b = Radial distance from bolt circle to center of o-ring, 3/8 in.

$K_1 = r_1/r_o = 1.5$.

p_{to} = Maximum stress in flange, psi.

r_o, r_1 = Inside and outside radius of flange, respectively, in.

t = Thickness of flange, 1/2 inch.

W = Bolt load, lb.

In the development of Equation A2, the modulus of elasticity was taken as 3×10^7 psi, and Poisson's ratio as 0.3.

According to this equation, in order for the maximum stress in the flange to be less than the working strength of stainless steel at 200°C, which is 13300 psi [8], the bolt load must not exceed 7150 lbs. This corresponds to a maximum pressure of 450 psi.

Holmberg and Axelson [11] showed that the maximum stress actually occurs at the junction of the flange with the cylinder wall. Since the flange is welded on, the fillet will distribute some of this stress, allowing a higher maximum pressure. The following calculations determine the stresses in the flange at 600 psi:

$$s = \sqrt{at} = 0.9682 \qquad \text{(A3.a)}$$

$$T_1 = \frac{t^3(3a^2 + 5d^2)}{h^3(d^2-a^2)} = 1.266 \qquad \text{(A3.b)}$$

$$T_2 = \frac{3.858t^3}{h^3(d^2-a^2)}\left[\frac{d^2}{3}\ln\left(\frac{b}{a}\right) + 0.1(b^2-a^2)\right] = 0.1099 \qquad \text{(A3.c)}$$

$$R = \frac{(s^2-\frac{h^3}{2t}T_1)(t+0.2325sT_1)p-2T_2(h+0.5377s)P}{1.86st + T_1\left[h^2(2+0.116\frac{s}{t}T_1)+1.6103sh + 0.866s^2\right]} = -586.5 \text{ lb/in} \qquad \text{(A3.d)}$$

$$K = \frac{(h^2T_1+1.86st)R+hT_2P-0.5tp(s^2-\frac{h^3}{2t}T_1)}{1.5hT_1-3.464t} \qquad \text{(A3.e)}$$

Stresses in cylinder wall at junction with flange:

$$\sigma = 6K/t^2 = 32350 \text{ psi} \qquad \text{(A3.f)}$$

$$\sigma_\ell = ap/2 = 1125 \text{ psi} \qquad \text{(A3.g)}$$

Total stress in cylinder wall = $\sigma + \sigma_\ell$ **= 33470 psi.** **(A3.h)**

Stresses in flange:

Radial:

$$\sigma_{ro} = \frac{6}{h^2}\left(K - \frac{h}{2}R\right) = 11610 \text{ psi} \qquad \text{(A3.i)}$$

$$\sigma_{rR} = R/h + p = -573 \text{ psi} \qquad \text{(A3.j)}$$

Therefore, the total radial stress is 11040 psi.

Tangential:

$$\sigma_{to} = \sigma_{ro} + \frac{0.8}{h^2(d^2-a^2)}\left[d^2\left(-1.5K + 7.5hR + 1.492P\ln\left(\frac{b}{a}\right)\right) + 0.4775P(b^2-a^2)\right] = 7082 \text{ psi} \qquad \text{(A3.k)}$$

$$\sigma_{tR} = \frac{h^2}{4t^3}T_1(R + hp) = -1451 \text{ psi} \qquad \text{(A3.}\ell\text{)}$$

Therefore, the total tangential stress is 5631 psi.

These calculations show that the stresses in the flange itself do not exceed the working stress of stainless steel. The stress in the cylinder wall is reduced by the presence of the fillet.

In conclusion, the maximum operating pressure of the reactor is now 600 psi at elevated operating temperatures.

Nomenclature for the calculations of Holmberg and Axelson

a = Mean diameter of cylinder wall, 3.75 in.

b = Diameter of bolt circle, 5.24 in.

d = Outside diameter of flange, 6.0 in.

h = Thickness of flange, 0.5 in.

K = Couple acting at junction of flange and cylinder per inch of circumference, in·lb/in.

p = Internal pressure, 600 psi.

P = Total bolt load, 9543 lbs.

R = Shearing force acting at junction of flange and cylinder per inch of circumference, in·lb/in.

σ = Stress, psi.

t = Thickness of cylinder wall, 0.25 in.

APPENDIX B

APPENDIX B

HEAT TRANSFER CALCULATIONS

Rough estimates of the power requirements and steady state wall temperature of the reactor were obtained as follows, with the following simplifying assumptions:

A) One-dimensional radial conduction through a cylindrical shell of insulation with internal temperature, T.

B) Heat is transferred away from the reactor by free convection and radiation into the surrounding air which is at ambient conditions.

C) For free convection calculations, the walls of the reactor are assumed to be a vertical cylinder, and the top is assumed to be a horizontal block of dimensions L_H and L_V.

D) The temperature of the external surface of the reactor is uniform.

E) As a first approximation, the following equations will be used:

Conduction equation:

$$q = \frac{2\pi k L(T-T_w)}{\ln(r_2/r_1)} \qquad \text{(B1)}$$

Convection + radiation:

$$q = h_w A_w (T_w - T_a) + h_t A_t (T_w - T_a) + \epsilon\sigma A_{t+w} (T_w^4 - T_a^4) \qquad \text{(B2)}$$

Since q is the same in both equations, they can be solved for T_w by substituting Equation B1 into Equation B2. Equation B1 can then be solved for q.

Estimates of hw and ht

The Grashof numbers at the reactor walls and on the top plate are evaluated for an average air temperature of 80°C and wall temperature of 120°C. This is actually the final step in an iteration procedure, since these values must be guessed initially in order to evaluate the Grashof numbers.

Walls:

$$Gr = \frac{g\beta}{\nu}(T_w - T_a)L^3 = 1.17\times10^7 \qquad \text{(B3.a)}$$

$$Gr \cdot Pr = (1.17\times10^7)(0.71) = 8.31\times10^6 \qquad \text{(B3.b)}$$

$$Nu = 0.59(Gr \cdot Pr)^{.25} = 31.7 \quad [12] \qquad \text{(B3.c)}$$

$$h_w = \frac{k_a(Nu)}{L} = 7.23\ W/m^2 \cdot K \qquad \text{(B3.d)}$$

Top - Approximate the top as a 5" x 1 1/2" block.

$$1/L = 1/L_h + 1/L_v \quad [12] \qquad \text{(B4.a)}$$

Therefore, L = 2.9 cm.

$$Gr_L = \frac{g\beta}{\nu}(T_w - T_a)L^3 = 1.39\times10^5 \qquad \text{(B4.b)}$$

$$Gr \cdot Pr = (1.39\times10^5)(0.71) = 9.87\times10^4 \qquad \text{(B4.c)}$$

$$Nu = 0.6(Gr \cdot Pr)^{.25} = 10.6 \quad [12] \qquad \text{(B4.d)}$$

$$h_t = \frac{k_a(Nu)}{L} = 10.6\ W/m^2 \cdot K \qquad \text{(B4.e)}$$

Substituting in the known and calculated constants, Equations B1 and B2 simplify to

$$q = 0.16(T-T_w) \qquad (B5)$$

and

$$q = 0.55(T_w-T_a) + 2.59\times10^{-9}(T_w^4-T_a^4). \qquad (B6)$$

Solving these equations gives

$$q = 99 \text{ watts}$$

and

$$T_w = 132^\circ C.$$

Since this approximation did not take into account conduction through the top and bottom, the values obtained are low compared to the experimental results of 180°C and 200 watts.

Nomenclature for Appendix B

A = Characteristic area.

β = Coefficient of thermal expansion for air, 0.00268 K^{-1}.

ϵ = Emissivity of stainless steel, 0.62.

Gr = Grashof number, defined as $\frac{g\beta}{\nu}(T_w-T_a)L^3$.

h_t = Heat transfer coefficient for the top of the reactor.

h_w = Heat transfer coefficient for the reactor walls.

k = Thermal conductivity of K-20 firebrick, 0.17 W/m·K.

k_a = Thermal conductivity of air, 0.03 W/m·K.

L = Characteristic length.

Nu = Nusselt number, defined as hL/k_a.

ν = Kinematic viscosity of air, 2.36×10^{-5} m^2/s.

q = Rate of heat transfer, watts.

r_1, r_2 = Inner and outer radius of insulation, respectively.

σ = Stefan-Boltzmann constant, 5.67×10^{-8} $W/m^2 \cdot K^4$.

T = Internal temperature of the reactor, 1023 K.

T_a = Temperature of the surrounding ambient air, 298 K.

T_w = Temperature of the reactor walls.

Subscripts:

a - Air.

h - Horizontal dimension.

t - Top of reactor.

v - Vertical dimension.

w - Walls of reactor.

Physical property data were obtained from reference 12.

APPENDIX C

APPENDIX C

COMPUTER PROGRAMS

Data Aquisition and Manipulation

The BASIC program summarized in Table C1 and listed in Table C2 contains subroutines for collecting data from the thermobalance, manipulating the data, fitting curves to it, and plotting the data.

Temperature Monitoring

The BASIC program listed in Table C3 operates the Tecmar interface board, which has a thermocouple voltage for an input signal. The program signals for an analog-to-digital conversion to be performed at a user-specified time interval. See the documentation in the program and in the users' guide for an explanation of the program steps. This program will be incorporated into the main data aquisition program as a subroutine in order to allow simultaneous collection of weight and temperature data.

Table C1. Summary of data aquisition program functions

Line numbers	Function
1-210	Controls the flow of the program into the various subroutines.
220-590	Data aquisition subroutine: Collects data from the Mettler balance at a user-specified time interval and stores it in an array and on a floppy disk.
600-890	Subroutine to set up a graph on the screen.
1020-1130	Subroutine to plot the values in the array on the graph. The scaling of the graph is user-specified.
1140-2020	"Simul" subroutine of Carnahan, et al. [13] translated into BASIC. Solves simultaneous equations.
2030-2550	Nonlinear regression subroutine of Carnahan, et al. [13] translated into BASIC.
2560-2720	Subroutine to plot the curve found by the previous two subroutines on the graph.
2730-2830	Subroutine to load a data file from the floppy disk into an array.
2840-2900	Subroutine to print out the contents of the array.
2910-2970	Subroutine to remove points from the array which fall outside the specified limits of the graph.
4000-4220	Subroutine to subtract a user-specified baseline shift from any data file and write the results to a new file.

Table C2. Data Aquisition and Graphics Program

```
1 'DATA AQUISITION AND GRAPHICS PROGRAM
2 '
10 DIM X(1000),Y(1000),IROW(50),JCOL(50),YZ(50),A(10,10),B(10),SX(20)
20 DIM SYX(10),CYX(10),XT(1000),YT(1000),XCOR(1000),YCOR(1000)
30 KEY OFF:SCREEN 2,1,0,0,2
40 CLOSE:CLS
45 PRINT "Data Aquisition and Graphics Program, Initial Choices:":PRINT
50 PRINT "1- Collecting New Data.":PRINT
52 PRINT "2- Reading a File.":PRINT
54 PRINT "3- Subtracting Baseline Shift from Data.":PRINT
56 INPUT "Enter initial choice:",M:PRINT
60 IF M<1 OR M>3 THEN 50
70 ON M GOSUB 220,2730,4000
100 GOSUB 600
110 GOSUB 1030
120 IF FLAG=0 THEN GOSUB 2030:GOSUB 2560
140 Z$=INKEY$
150 IF Z$="S" THEN 170
160 GOTO 140
170 CLS:INPUT "RESCALE GRAPH";B$
180 IF B$="Y" THEN 100
190 INPUT "RETURN TO MENU";B$
200 IF B$="N" THEN SCREEN 0,0,0,0,0:END
210 GOTO 40
220 REM DATA AQUISITION SUBROUTINE
225 CLS:PRINT "Data aquisition subroutine for Mettler PE360 balance.":PRINT
230 TI=0:N=0:INPUT "ENTER SAMPLING INTERVAL IN SECONDS:",TINTS
235 TINT=TINTS/60
240 INPUT "ENTER NAME OF DATA FILE AND PRESS RETURN TO BEGIN.",FILE$
250 OPEN FILE$ FOR OUTPUT AS #2
260 TIME$="00:00"
270 ON ERROR GOTO 280
280 CLOSE 1
290 OPEN "COM1:2400,E,7,1,LF" AS #1
300 INPUT #1,S$
310 IF LEN(S$)<14, THEN 300
320 CLOSE 1
330 IF N>0 THEN 350
340 TIME=VAL(LEFT$(TIME$,2))*60 + VAL(MID$(TIME$,4,2)) + VAL(RIGHT$(TIME$,2))/60
350 WEIGHT=VAL (MID$(S$,5,12))
360 PRINT RIGHT$(S$,10),TIME$
370 PRINT #2, WEIGHT;",";TIME
380 N=N+1
390 XT(N)=TIME
400 YT(N)=WEIGHT
410 IF N=1000 THEN 480
420 TI=TI+TINT
430 TIME=VAL(LEFT$(TIME$,2))*60 + VAL(MID$(TIME$,4,2)) + VAL(RIGHT$(TIME$,2))/60
440 IF TIME>=TI THEN 290
450 X$=INKEY$
460 IF X$="S" THEN 480
470 GOTO 430
480 CLOSE
490 BEEP:PRINT "DATA AQUISITION TERMINATED":PRINT
500 PRINT N;"DATA POINTS COLLECTED":PRINT
510 INPUT "PRINT LIST OF DATA";A$
520 IF A$="Y" THEN GOSUB 2840:PRINT
540 INPUT "PLOT DATA";A$
550 IF A$="Y" THEN 590
560 GOTO 190
590 RETURN
```

Table C2 (cont'd).

```
600 REM SET UP GRAPH
610 BEEP
620 INPUT "ENTER XMIN, XMAX, YMIN, YMAX TO SET UP AXES.",XMIN,XMAX,YMIN,YMAX
625 INPUT "ENTER ORDER OF CURVE FIT (PRESS RETURN FOR NO CURVE FIT):",M
624 IF M=0 THEN FLAG=1 ELSE FLAG=0
630 GOSUB 2910
650 CLS
700 LINE (59,0) - (59,190): LINE (59,0) - (639,0)
710 LINE (59,190) - (639,190): LINE (639,0) - (639,190)
720  FOR I = 0 TO 10
730 LINE (59,19*I) - (64,19*I)
740 LINE (634,19*I) - (639,19*I)
750 LINE (59+I*58,0) - (59+I*58,3)
760 LINE (59+I*58,187) -(59+I*58,190)
770 NEXT I
890 RETURN
1020 RETURN
1030 'plot x - y values
1040 FOR I=1 TO N
1070 X1%=((X(I)-XMIN)/(XMAX-XMIN))*580+59
1080 Y1%=190-((Y(I)-YMIN)/(YMAX-YMIN))*190
1090 PSET(X1%,Y1%):PSET(X1%+1,Y1%):PSET(X1%-1,Y1%):PSET(X1%,Y1%+1)
1100 PSET(X1%,Y1%-1)
1120 NEXT I
1130 RETURN
1140 'simul routine page 290 carnahan
1150 MAX=M
1160 IF INDIC=0 THEN MAX=M+1
1170 IF M<=50 THEN GOTO 1210
1180 PRINT "m too big"
1190 SIMUL=0
1200 RETURN
1210 DETER=1
1220 FOR K=1 TO M
1230 KM1=K-1
1240 PIVOT=0
1250 FOR I= 1 TO M
1260 FOR J= 1 TO M
1270 IF K=1 GOTO 1340
1280 FOR ISCAN = 1 TO KM1
1290 FOR JSCAN = 1 TO KM1
1300 IF I = IROW(ISCAN) THEN GOTO 1380
1310 IF J=JCOL(JSCAN) THEN GOTO 1380
1320 NEXT JSCAN
1330 NEXT ISCAN
1340 IF  ABS(A(I,J))<= ABS(PIVOT) THEN GOTO 1380
1350 PIVOT = A(I,J)
1360 IROW(K)=I
1370 JCOL(K)=J
1380 NEXT J
1390 NEXT I
1400 IF  ABS(PIVOT)>EPS THEN GOTO 1430
1410 SIMUL=0
1420 RETURN
1430 IROWK = IROW(K)
1440 JCOLK =JCOL(K)
1450 DETER=DETER*PIVOT
1460 FOR J=1 TO MAX
1470 A(IROWK,J)=A(IROWK,J)/PIVOT
1480 NEXT J
```

Table C2 (cont'd).

```
1490 A(IROWK,JCOLK)=1/PIVOT
1500 FOR I=1 TO M
1510 AIJCK=A(I,JCOLK)
1520 IF  I=IROWK THEN GOTO 1570
1530 A(I,JCOLK)=-AIJCK/PIVOT
1540 FOR J=1 TO MAX
1550 IF J<>JCOLK THEN A(I,J)=A(I,J)-AIJCK*A(IROWK,J)
1560 NEXT J
1570 NEXT I
1580 NEXT K
1590 FOR I=1 TO M
1600 IROWI=IROW(I)
1610 JCOLI=JCOL(I)
1620 JORD(IROWI)=JCOLI
1630 IF INDIC >=0 THEN B(JCOLI)=A(IROWI,MAX)
1640 NEXT I
1650 IC%=0
1660 NM1=M-1
1670 FOR I=1 TO NM1
1680 IP1=I+1
1690 FOR J=IP1 TO M
1700 IF JORD(J)>=JORD(I) THEN GOTO 1750
1710 JTEMP=JORD(J)
1720 JORD(J)=JORD(I)
1730 JORD(I)=JTEMP
1740 IC%=IC%+1
1750 NEXT J
1760 NEXT I
1770 IF INT(IC%/2)*2 <> IC% THEN DETER=-DETER
1780 IF INDIC<=0 THEN GOTO 1810
1790 SIMUL=DETER
1800 RETURN
1810 FOR J=1 TO M
1820 FOR I = 1 TO M
1830 IROWI=IROW(I)
1840 JCOLI=JCOL(I)
1850 YZ(JCOLI)=A(IROWI,J)
1860 NEXT I
1870 FOR I = 1 TO M
1880 A(I,J)=YZ(I)
1890 NEXT I
1900 NEXT J
1910 FOR I=1 TO M
1920 FOR J=1 TO M
1930 IROWJ=IROW(J)
1940 JCOLJ=JCOL(J)
1950 YZ(IROWJ)=A(I,JCOLJ)
1960 NEXT J
1970 FOR J=1 TO M
1980 A(I,J)=YZ(J)
1990 NEXT J
2000 NEXT I
2010 SIMUL=DETER
2020 RETURN
```

Table C2 (cont'd).

```
2030 'regr page 580 carnahan
2040 MTWO=2*M
2050 MP1=M+1
2060 SY=0
2070 SYY=0
2080 FOR I=1 TO M
2090 MPI=M+I
2100 SX(I)=0
2110 SX(NPI)=0
2120 SYX(I)=0
2130 NEXT I
2140 FOR I=1 TO N
2170 SY = SY + Y(I)
2180 SYY = SYY + Y(I)^2
2190 DUM=1
2200 FOR J= 1 TO M
2210 DUM = DUM*X(I)
2220 SX(J)=SX(J) + DUM
2230 SYX(J)=SYX(J)+Y(I)*DUM
2240 NEXT J
2250 FOR J= MP1 TO MTWO
2260 DUM = DUM*X(I)
2270 SX(J)=SX(J) + DUM
2280 NEXT J
2290 NEXT I
2300 FM=N
2310 CYY = SYY - SY*SY/FM
2320 FOR I = 1 TO M
2330 CYX(I)=SYX(I)-SY*SX(I)/FM
2340 A(I,MP1)=CYX(I)
2350 FOR J= 1 TO M
2360 IPJ=I + J
2370 A(I,J)=SX(IPJ)-SX(I)*SX(J)/FM
2380 NEXT J
2390 NEXT I
2400 PPS=9.999999E-21
2410 INDIC=1
2420 GOSUB 1140
2430 IF DETER <> 0 THEN GOTO 2460
2440 REGR=0
2450 RETURN
2460 DUM = SY
2470 TEMP = CYY
2480 FOR I = 1 TO M
2490 DUM = DUM - B(I)*SX(I)
2500 TEMP = TEMP - B(I)*CYX(I)
2510 NEXT I
2520 AA=DUM/FM
2530 DENOM=N-M-1
2540 REGR=S
2550 RETURN
```

Table C2 (cont'd).

```
2560 'plot fitted line
2570 XX2=XMIN:YY2=AA:FOR J= 1 TO M:YY2=B(J)*XX2^J+YY2:NEXT J
2580 FOR I = 1 TO 580
2590 XX1=XX2
2600 XX2=I*(XMAX-XMIN)/580
2610 YY1=YY2
2620 YY2=AA
2630 FOR J=1 TO M
2640 YY2=YY2+B(J)*XX2^J
2650 NEXT J
2660 Y1%=190-(YY1-YMIN)*190/(YMAX-YMIN)
2670 IF Y1%<0 THEN Y1%=0
2680 Y2%=190-(YY2-YMIN)*190/(YMAX-YMIN)
2690 IF Y2%<0 THEN Y2%=0
2700 LINE (58+I,Y1%) - (59+I,Y2%)
2710 NEXT I
2720 RETURN
2730 REM FILE READING SUBROUTINE
2740 CLS:PRINT "Subroutine to load file from disk into memory and plot data."
2750 PRINT:INPUT "INPUT NAME OF FILE TO BE READ.",FILE$
2760 INPUT "PRINT LIST OF DATA";D$
2770 OPEN FILE$ FOR INPUT AS #1
2775 N=1
2780 INPUT #1, WEIGHT,TIME
2790 YT(N)=WEIGHT: XT(N)=TIME
2800 IF D$="Y" THEN PRINT WEIGHT,TIME
2810 IF EOF(1) THEN CLOSE: NTOT=N:RETURN
2820 N=N+1
2830 GOTO 2780
2840 REM PRINTING SUBROUTINE
2850 PRINT "WEIGHT(g)          TIME(MIN)":PRINT
2860 FOR I=1 TO N
2880 PRINT XT(I),YT(I)
2890 NEXT I
2900 RETURN
2910 REM SUBROUTINE TO REMOVE POINTS OUTSIDE SET LIMITS.
2920 J=0
2930 FOR I=1 TO NTOT
2940   IF XT(I)>=XMIN AND XT(I)<=XMAX THEN J=J+1:X(J)=XT(I):Y(J)=YT(I)
2950 NEXT I
2960 N=J
2970 RETURN
4000 'Subroutine to subtract baseline shift from raw data.
4010 '
4030 CLS
4035 PRINT "Subroutine to subtract baseline shift from raw data.":PRINT
4040 INPUT "Enter coefficients of third order polynomial:",AA,B1,B2,B3
4050 INPUT "Name of data file";FILE$
4060 INPUT "Name of output file";NFILE$
4070 OPEN FILE$ FOR INPUT AS #1
4080 FOR N=1 TO 1000
4090   INPUT #1, YT(N),XT(N)
4100   IF EOF(1) THEN CLOSE: GOTO 4115
4110 NEXT N
4115   OPEN NFILE$ FOR OUTPUT AS #2
4117 SHIFT = YT(1)
4120 FOR I=1 TO N
4180   YCOR(I)=(YT(I)-(AA+B1*XT(I)+B2*XT(I)^2+B3*XT(I)^3)-SHIFT)/.68
4190   WRITE #2, YCOR(J),XCOR(J)
4200 NEXT I
4210 CLOSE
4220 RETURN
```

Table C3. Data Aquisition Program for Tecmar Interface Board

```
10 ' Data Aquisition Program for TECMAR Interface Board.
20 KEY OFF:CLS
30 PRINT "Data Aquisition Program for TECMAR Interface Board.":PRINT
40 'Coefficients of third order polynomial used to convert voltage to
   temperature.
50 AA=15.985:B1=25.704:B2=-.0994:B3=.001567
60 INPUT "ENTER GAIN(1,10,100,500):",GAIN
70 IF GAIN=1 THEN B=128:GOTO 130
80 IF GAIN=10 THEN B=129:GOTO 130
90 IF GAIN=100 THEN B=130:GOTO 130
100 IF GAIN=500 THEN B=131:GOTO 130
110 GOTO 60
120 'Input starting address of board in decimal and the number of the channel to
    convert.
130 ADDRESS=1808!:CHANNEL=1
140 'Disable auto-incrementing, external start conversions, and all interrupts.
150 OUT ADDRESS+4, B
160 'Output channel number.
170 OUT ADDRESS+5, CHANNEL
180 'Reset counter.
190 SUM=0:N=0
200 'Start a conversion.
210 OUT ADDRESS+6, 0
220 'Wait until bit 7 of status byte is a 1 signaling done converting.
230 IF INP(ADDRESS+4) < 128 THEN 230
240 'Read in the data.
250 LOW=INP(ADDRESS+5)
260 HIGH=INP(ADDRESS+6)
270 'Convert from twos complement to a number between -10 and 10.
280 X=256*HIGH+LOW
290 IF X>32767 THEN X=X-65536!
300 VOLTAGE=X*1000/(204.8*GAIN)   'CONVERTS TO mV
310 SUM=SUM + VOLTAGE
320 N=N+1
330 IF N<50 THEN 210
340 VAV=SUM/N
345 ' Convert voltage to temperature
350 TEMP=AA+B1*VAV+B2*VAV^2+B3*VAV^3
360 'Set up online display on screen.
370 LOCATE 12,30,0:PRINT "VOLTAGE ="
380 LOCATE 14,26,0:PRINT "TEMPERATURE = "
390 LOCATE 12,39:PRINT USING "##.##_mV";VAV
400 LOCATE 14,39:PRINT USING "###_ C  ";TEMP
410 X$=INKEY$:IF X$="S" THEN END ELSE GOTO 190
```