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Full Name of Author — Nom complet de l'auteur: NOLAN ARIE VAN GAALEN

Date of Birth — Date de naissance SEPTEMBER 16, 1955	Country of Birth — Lieu de naissance CANADA

Permanent Address — Résidence fixe: C/o 2217 8A AVE, SOUTH, LETHBRIDGE, ALTA TIJ-157

Title of Thesis — Titre de la thèse: THE DEVELOPMENT OF A BOIL-OFF FLOW CALORIMETER

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The Development of a Boil-off Flow Calorimeter

by

© Nolan A. Van Gaalen

A THESIS

SUBMITTED TO THE FACULTY OF GRADUATE STUDIES AND RESEARCH
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(SIGNED) Nolan Van Gaalen

PERMANENT ADDRESS:
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CANADA T1J-1S7

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The undersigned certify that they have read, and recommend to the Faculty of Graduate Studies and Research, for acceptance, a thesis entitled The Development of a Boil-off Flow Calorimeter submitted by Nolan A. Van Gaalen in partial fulfilment of the requirements for the degree of Master of Science in Chemical Engineering.

........A.E. Mather........
Supervisor

........D.B. Robinson........

........J.D. Dale........

Date....April 13, 1983....

Abstract

A boil-off flow calorimeter designed for use with corrosive gas mixtures at temperatures up to 400 °C and pressures up to 20 MPa is described. Results of tests with N_2 and CO_2, up to 10 MPa and 200 °C, indicate a continuing problem with heat leaks, despite changes in the construction and operation of the calorimeter in order to reduce these heat leaks. Some test runs were made for a gas mixture of CO_2, H_2, and CH_4, with the results comparing reasonably well with predictions based on the Peng-Robinson equation of state.

A survey of heat leak correction calculations made by other workers suggests that a heat leak correction correlation dependent on reciprocal process fluid flow rate may be appropriate if further improvements are realized as a result of the recommendations given here.

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I sincerely thank Dr. Alan E. Mather for his guidance, insights, and support (both financial and moral), as well as his patience, without which this work could not have been initiated or completed.

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I will remember with appreciation the rest of the staff and graduate students, from whom I also learned much and with whom I enjoyed working.

I wish to thank the community of friends in which I live, who have helped me develop a broader perspective on this and other involvements, for support and insights which have enriched my experience here more than I can say.

Table of Contents

Chapter Page

List of Tables

List of Figures

1. Introduction

Calorimetry involves the measurement of energy property changes or energy interactions which occur in some processes of interest. While some of these thermophysical properties can be predicted accurately for an increasing number of substances and their mixtures, their accurate measurement remains important, either for successful process design and operation, or for assessment and improvement of the quality of the available predictive methods. Today's need for enthalpy data, as well as other data, on coal gasification plant streams, synthetic natural gas streams, and processes involving steam and hydrocarbon mixtures at the associated higher temperatures and pressures (Yen et al., 1977; Wormald, 1982), demands that facilities be built for the purpose of determining these properties.

The work described here was initiated in order to meet some of these perceived needs, and consequently a calorimeter was designed and constructed for use with corrosive mixtures at high temperatures and pressures. A survey of previous work, a discussion of the relevant thermodynamics and the various modes of fluid calorimetry lead to a consideration of the important design criteria for comparative boil-off calorimeters. A description of the apparatus as currently operating is given. What is not contained here is an extensive review of the prior development work that was invested into the achievement of an operational device. The design of the calorimeter is

similar in some respects to that of Sahgal et al. (1965). Freon-11 was used as the reference fluid in the device described here. Nitrogen and carbon dioxide, two gases whose thermal properties are well known, were used as standards in order to calculate the heat leaks. A third fluid, composed of a mixture of CH_4 (61.7%), CO_2 (28.6%), and H_2 (9.7%), was also tested. When it became apparent that heat leaks were a major problem, efforts were made to modify the construction and operation of the apparatus in order to improve its performance.

The method and precision of the calculations involved are summarised, and certain of the processed results are compared in an attempt to note trends, as well as to find probable causes of any variation in the results of similar runs. The heat leak correction methods of other workers were surveyed and their applicability to this work is examined.

It is concluded that correlation of the measured property (in this case, enthalpy change from ambient), and hence heat leak, with the reciprocal flow rate would be appropriate if the present performance could be sufficiently improved. Recommendations which should result in some improvements are given.

2. Context

2.1 Survey of the Literature

A literature survey in the general area of calorimetry readily reveals the existence of previous surveys which, combined, form a rather extensive history and context for present work.

These surveys are those of Partington and Shilling (1924), reviewing the calorimetric work done and reported prior to 1924; Masi (1954), who summarized "all of the literature reports of determinations of heat capacity of gases in the period 1924-1952, inclusive"; Faulkner (1959), who reviewed methods of constant volume calorimetry and isenthalpic expansion in addition to a survey of flow calorimetric methods up to that time; Yesavage et al. (1967), who briefly discussed experimental methods, then surveyed much of the data for fluid mixtures with examples of how it may be analysed, and finally treated methods of prediction of certain thermal properties; and Mather (1978), who reviewed recent experimental and predictive methods for the determination of fluid enthalpies.

Since the 1978 survey by Mather, the work of Barry et al. (1982), Christensen et al. (1981), Clarke et al. (1979), Cunningham and Wilson (1978), Eubank et al. (1982), Judd et al. (1980), Kidnay and Yesavage and co-workers (McConnell (1976), Sharma (1977), Yesavage et al. (1977), Andrew (1978), Omid (1978)), Lammers and co-workers (Lammers et al. (1978), van Kasteren and Zeldenrust (1979)), Miyazaki et al. (1980), and Wormald (1979, 1982) and co-workers (Hutchings et al. (1978), Wormald et al. (1979), Richards and Wormald (1981), Richards et al. (1981)) has been documented.

Focusing the survey on comparative calorimetric methods, one finds at least two means of employing a reference fluid. The first involves the use of a reference liquid which undergoes a temperature change. Subsequent calculations require knowledge of the heat capacity of this liquid in order to determine the enthalpy change occurring in the process under study.

The other main technique is the use of a vapourising reference fluid (at constant temperature). The external information required in this case is the data on enthalpy of vapourisation of the reference fluid. The first reported use of this technique appears to be the work of Nelson and Holcomb (1953), who used Freon-11 in the determination of enthalpies of hydrocarbon mixtures. Since then, as indicated in Table 2.1, many other workers have constructed equipment using the same principle in other designs and applications. (Some of the information in the table is extracted from Mather's survey (1978) and from McConnell's thesis (1976).)

Table 2.1 : **Previous Boil-off Calorimetry Reported**

WORKERS	YEAR	REFERENCE FLUID	SYSTEM	CONDITIONS P in MPa	CONDITIONS T in K
Nelson and Holcomb	1953	Freon-11	HC Mixtures	≤ 5.5	388 - 478
Jenkins and Berwaldt	1963	Liq. N2, F-11, other	Air, N2, N2-CH4	≤ 10.1	78 - 295
Sahgal et al.	1965	Liq. N2	N2, CH4	≤ 20.7	88 - 311
Wiener	1966	Freon-11	H2-CH4 Mixtures	≤ 6.9	148 - 311
Dolan et al.	1968	Freon-11	N2, Natural Gas	≤ 13.7	78 - 311
Laverman and Selcukoglu	1969	Liq. N2	Natural Gas Mix	≤ 17.2	83 - 311
Lenoir et al.	1967, 1970	Freon-11	C5 - HC Mixtures C5 - Tetralin	≤ 4.8 ≤ 9.7	311 - 455 322 - 622
Sood and Haselden	1972	Liq. O2	Natural Gas	≤ 3.6	188 - 373
Thinh et al.	1973	Freon-11	n-Pentanol n-Hexanol	≤ 10.3	422 - 617
Yesavage and co-workers	1976	Freon-11	Coal Derived Liquids	≤ 10.3	291 - 561
Sagara et al.	1977	Liq. N2	H2-CH4, H2-C2H6 H2-C3H8	≤ 13.6	120 - 210
Eubank et al.	1982	Freon-11	Steam	≤ 16.0	348 - 574

2.2 Thermodynamic Considerations

Because the enthalpy of a pure substance or mixture in a single homogeneous phase is a state function, changes in enthalpy are dependent only on the initial and final states and independent of the process or path along which the change was effected. Thus, the change in enthalpy is an exact differential and may be expressed as follows:

$$dh = \left(\frac{\partial h}{\partial T}\right)_{P,x} dT + \left(\frac{\partial h}{\partial P}\right)_{T,x} dP + \sum_{i=1}^{n}\left[\left(\frac{\partial h}{\partial x_i}\right)_{P,T,x_j} dx_i\right] \tag{2.1}$$

For pure fluids or mixtures of constant composition, the thermal effects of composition changes, i.e. mixing, are not present and equation (2.1) may be more simply written:

$$dh = \left(\frac{\partial h}{\partial T}\right)_{P,x} dT + \left(\frac{\partial h}{\partial P}\right)_{T,x} dP \tag{2.2}$$

For convenience, the partial derivatives above are, of course, defined respectively as Cp, the constant pressure heat capacity, and Ø, the isothermal expansion coefficient. Sufficient knowledge of these quantities for a certain substance or mixture is necessary for the construction of a pressure - enthalpy diagram[1] and allows calculation of an (integral) change of enthalpy corresponding to a change of state specified by changes in temperature and/or pressure.

---

[1] Necessary and sufficient would be knowing *either* Cp at one pressure and all temperatures, with Ø at all temperatures and pressures, *or* Ø at one temperature and all pressures, with Cp at all temperatures and pressures.

Since temperature and pressure are more easily observed in many situations than are other properties, equation (2.2) is a convenient form. However, the more fundamental relationship for the enthalpy property is the following, derived from the first and second laws of thermodynamics and the definition of enthalpy:

$$dh = T\,ds + v\,dP \quad . \tag{2.3}$$

One can show that Ø, the isothermal expansion coefficient, is fundamentally expressed as follows:

$$\phi = \left(\frac{\partial h}{\partial P}\right)_T = T\left(\frac{\partial s}{\partial P}\right)_T + v \tag{2.4}$$

Use of the fourth Maxwell relation, derived from the definition of the Gibbs free energy,

$$-\left(\frac{\partial s}{\partial P}\right)_T = \left(\frac{\partial v}{\partial T}\right)_P \tag{2.5}$$

allows the expression of Ø as a function of pressure, volume, and temperature only:

$$\phi = \left(\frac{\partial h}{\partial P}\right)_T = v - T\left(\frac{\partial v}{\partial T}\right)_P \tag{2.6}$$

Thus, it is seen that precise P-v-T data (physical property measurements) might be used as a check on this type of calorimetric data (thermal property measurements).

Precision in the P-v-T data is necessary since the derivative involved will tend to emphasize any errors which are present.

The foregoing is in contrast to the case of the constant pressure heat capacity, Cp. It is not possible by similar methods to express Cp as a function of P-v-T only and hence calorimetric methods for its determination are necessary.

In these methods, it is necessary to exchange energy with the substance in some way, while noting the resultant change in state of the substance by accurately observing temperatures and pressures. The first law of thermodynamics may be written as an energy balance:

$$[h + \frac{u^2}{2} + gz]\delta m_{in} - [h + \frac{u^2}{2} + gz]\delta m_{out} + \delta Q - \delta W = d([e + \frac{u^2}{2} + gz]m_{sys}) \qquad \ldots .(2.7)$$

For a steady state flow situation, changes in overall system mass and energy are zero. In flow calorimetry, no shaft work is done on or by the system (any electrical energy being considered as a heat input) and potential and kinetic energy changes as the material moves through are made negligible by proper design and the use of small pressure drops, so that velocity changes are not large. Given all of this, equation (2.7) can be greatly simplified to the form below:

$$h_{in} - h_{out} + q = 0 \qquad (2.8)$$

where q is the amount of heat transfer per mole of process fluid. Further elaboration on this flow calorimetry equation appears in the following section.

2.3 Methodology

Major Methods. As has been previously pointed out, there are published several extensive surveys of the general field of calorimetry which span at least the last 80 years of work in the area. What follows is a brief review of main flow calorimetry methods and their relative merits.

Despite the comments of Masi (1954):

> "Obtaining and measuring a constant flow rate to the desired precision, measuring temperatures which accurately represent the state of the gas and reducing the heat leak of the calorimeter to manageable proportions are only three of the problems which are encountered, and which tend to make extremely complicated any calorimeter built for precision work";

and the words of Barieau (1965), who decided against the direct measurement of enthalpies, concluding that

> "methods that require no mass flow rate or energy measurements are to be preferred [over direct methods, since]... it appears that the most difficult quantity to determine in reducing data so that an enthalpy diagram can be constructed is the determination of the flow rate",

it seems that the basic methods of flow calorimetry have survived. The reasons for this include the directness with which Cp or other thermal properties can be calculated (since, for steady-state operation, the heat capacity of the apparatus itself does not enter the calculation), the wide range of temperatures and pressures for which the methods can be applied, and their applicability to mixtures. With modifications to increase its accuracy and applicability, flow calorimetry will continue as an important technique for gathering reliable thermal property data. Under the general category of flow calorimetry, there are several main types used and discussed in the literature:

1. differential flow micro-calorimetry
2. "resistive heating" (direct)
3. boil-off (comparative)
4. matching boil-off

The first of these is not subject to the criticisms of Masi, Barieau, and others about the precision of flow measurements, since it is actually a comparative method using the fluid at one set of conditions as its own reference for another set of conditions, all at the same flow rate. A ratio of heat capacities is obtained from the data and, therefore, a knowledge of ideal gas heat capacity, attainable from calorimetry or from spectroscopic determinations, is needed in addition to the experimental data to calculate actual values for Cp.

The second type, "resistive heating" flow calorimetry, has the longest history of the remaining three. It involves the use of a carefully measured amount of electrical energy to restore or maintain certain flow conditions in the calorimeter. This energy is transferred to the fluid through dissipation from some resistive element either insulated from or in contact with the fluid. Thus the heat exchanged can be calculated as

$$q_{meas} = \frac{VI}{\dot{m}_f} = \frac{I^2R}{\dot{m}_f} \qquad (2.9)$$

and, since q equals the measured heat transfer plus the transfer due to heat leaks, all per mole of process fluid, equation (2.8) can be rewritten:

$$h_{out} - h_{in} = \frac{VI}{\dot{m}_f} + \frac{\dot{Q}_L}{\dot{m}_f} \qquad (2.10)$$

Note that this method requires accurate measurement of the process fluid flow rate. If this is done volumetrically, it is necessary to know the density of the fluid precisely in order to reduce the uncertainty in the resulting mass flow rate. This basic method has been used extensively, as surveyed by Mather (1978) with more recent examples being Cunningham and Wilson (1978) and Clarke et al. (1982), to determine enthalpies and heat capacities of many fluids at a wide range of conditions and also for excess enthalpy (heat of mixing) experiments, as recently documented by Barry et al. (1982), Christensen et al. (1981), and Wormald et al. (1977) and many of his later co-workers.

The third method, somewhat more recent than the second, is the comparative method of boil-off flow calorimetry, in which the fluid of interest undergoes a change of state and cools. The energy released is transferred to the reference fluid at its boiling point. Liquid nitrogen, Freon-11 (trichlorofluoromethane), and water are the most commonly used reference fluids. The choice depends on operating conditions and also on the precision with which the enthalpy of vapourisation is known at the conditions used.

Generally, the fluid is caused to boil at ambient pressure. In this technique, the measured heat exchange is given by:

$$q_{meas} = -\frac{\lambda_r \dot{m}_r}{\dot{m}_f} \tag{2.11}$$

and the resulting operating equation becomes:

$$h_{out} - h_{in} = -\frac{\lambda_r \dot{m}_r}{\dot{m}_f} + \frac{\dot{Q}_L}{\dot{m}_f} \tag{2.12}$$

Here it is seen that two flow rates must be measured. However, in the work that has been reported to date, the accuracies appear to be comparable to those of the resistive heating method.

McConnell (1976) has summarised the reasons which justify the choice of this method for certain applications. These include the ability to measure changes in enthalpy over larger ranges of conditions, the possibility of simpler construction, especially for higher temperature and pressure applications, and the adequacy of its accuracy for engineering design purposes. Another advantage may be that, rather than heating the process fluid as is done in the direct method, possibly causing its thermal decomposition, in the boil-off method the fluid is cooled, preventing further decomposition.

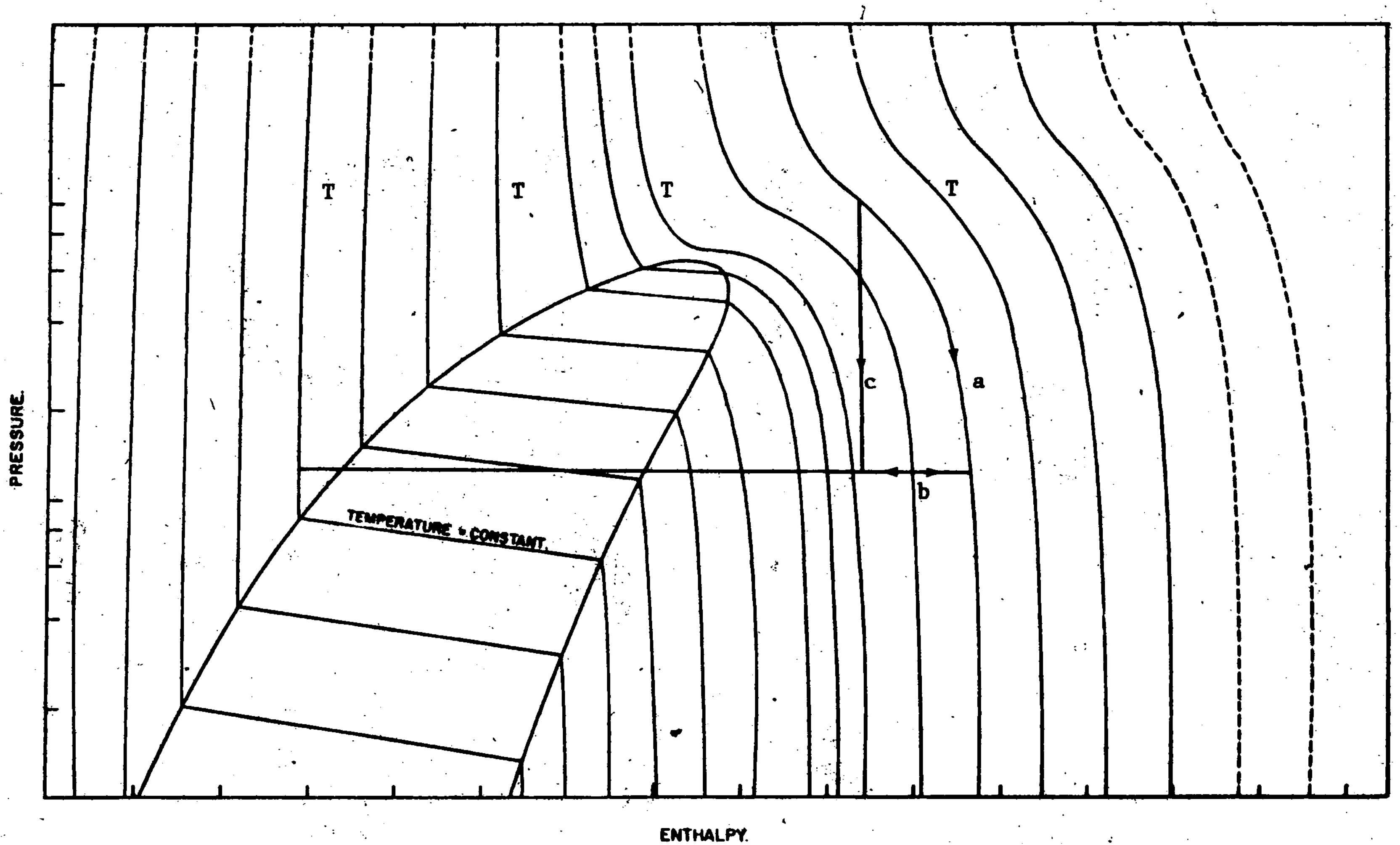
The fourth method listed is a recent modification of the boil-off method which seeks to minimise possible problems of reference flow rate measurements, accuracy of value of enthalpy of vapourisation, and heat leaks by attempting to duplicate the boil-off rate observed in "regular" operation by electrically heating the reference fluid under conditions which match the expected heat leaks. Eubank et al. (1982) recently presented details of this method.

This method still requires knowledge of the process fluid flow rate, but the actual measurement of the reference fluid boil-off rate is replaced by a measurement of the amount of electrical energy required to produce an equivalent boil-off rate. Thus, precision and repeatability of reference fluid flow rates is required, but the need for accurate calibration is eliminated. The operating equation is therefore as follows:

$$h_{out} - h_{in} = \frac{(I^2R)_{matching}}{\dot{m}_f} + \frac{\dot{Q}_L}{\dot{m}_f} \qquad (2.13)$$

<u>Modes of Calorimeter Operation.</u> A general pressure-enthalpy diagram for some mixture of constant composition is given in Figure (2.1). A number of process paths are also shown, indicating several modes of calorimetric investigation.

Mode (a) is an expansion in which energy must be added to the fluid in order to maintain isothermal conditions. Any deviation from the temperature of interest must be corrected by the term on the far right of equation (2.14a) if the results are to be reported as isothermal. The values



a) $[h(P_2)-h(P_1)]_{T,x} = q - \int c_p dT$ [Eq. 2.14a]

b) $[h(T_2)-h(T_1)]_{P,x} = q - \int \phi dP$ [Eq. 2.14b]

c) $[h(T_2,P_2)-h(T_1,P_1)] = 0$ [Eq. 2.14c]

Figure 2.1 Modes of Calorimeter Operation of 0 may be calculated from these data if the pressure drops involved are not too great.

Mode (b) is the isobaric cooling or heating of the process fluid, possibly involving phase change. A small correction must usually be made for pressure drop since flow of the fluid cannot occur if inlet pressure exactly equals outlet pressure, but this adjustment may be negligible, especially where the isotherm is nearly vertical. The "resistive heating" method is generally used to increase the enthalpy of the incoming fluid, but could be used to restore the enthalpy of a fluid having cooled from its initial temperature. Boil-off methods involve only cooling of the process fluid. If sufficient data for small increments in temperature are taken, determination of the values of Cp is possible.

Mode (c) is the Joule-Thomson experiment where a pressure drop under adiabatic flow conditions results in a temperature change. This change is often a decrease, but not necessarily, depending on the local slope of the isotherms. The Joule-Thomson coefficient, defined as follows:

$$\mu_{JT} = \left(\frac{\partial T}{\partial P}\right)_h \qquad (2.15)$$

may be determined from the data if sufficiently small pressure drops are used.

The thermodynamic consistency of the data gathered in the above three modes of experimentation may be examined using the relation

$$\phi = -\mu C_p \qquad (2.16)$$

if all of these properties have been measured at the states of interest.

A fourth mode, not shown, might be some path in which changes in both pressure and temperature are significant. Such a process might be a simple expansion from some higher temperature and pressure to ambient conditions. The work on hydrocarbon enthalpies reported by Wiener (1966), who used a boil-off method, is an example of this. The data thus produced are enthalpy values based on a zero reference value at the ambient or near ambient conditions (eg., 25 °C and 100 kPa) and might represent overall heating or cooling loads although they would be of little use in calculation of local values of C_p, ϕ, and μ.

3. Description of Equipment and Procedure

3.1 Design Considerations

The basic boil-off flow calorimeter equation was previously shown to be:

$$h_{out} - h_{in} = -\frac{\lambda_r \dot{m}_r}{\dot{m}_f} + \frac{\dot{Q}_L}{\dot{m}_f} \qquad (2.12)$$

An examination of the terms of this equation provides the most direct indications of what the primary design considerations for this calorimeter should be.

1. Heat Leaks. Minimisation of heat leaks by radiation, convection, and conduction is of great importance in the accuracy of the experimental results for enthalpy changes. As can be seen upon rearrangement of equation (2.12), the heat leak term is positive when an excess of reference fluid boils off:

$$\dot{Q}_L = \dot{m}_f(h_{out} - h_{in}) + \lambda_r \dot{m}_r \qquad (3.1)$$

If the magnitude of the (positive) boil-off term is greater than the magnitude of the (negative) "cooling" term, then heat is leaking into the boil-off chamber and Q is positive. Q is negative when too little reference fluid boils off.

If the heat leak cannot be eliminated by modification of design and/or operation, it must be evaluated and corrected, if possible, as a function of flow rate and/or temperature. This evaluation involves the use of a "standard" process fluid and use of the known values of outlet and inlet enthalpies in equation (3.1). If such a correction correlation for heat leak is attainable, the calorimeter may be profitably used for enthalpy determinations, despite the existence of a measurable heat leak.

2. Steady-state Operation and Capacity. The rapid achievement of steady-state operation requires that the "thermal inertia" or heat capacity of the calorimeter vessel be relatively low while the volume still contains enough of the boil-off (reference) fluid to handle anticipated heat flows. Thus the rapid "rise-time" may be seen in a trade-off relationship to "capacity".

The mass of the reference fluid vessel and the exchanger (whether that be a simple coil of tubing or some more complex apparatus) will be of importance in keeping the time to reach steady-state to a minimum. The hot process fluid must heat at least the tubing walls to their steady-state temperature before constancy of inlet temperature is achieved. Shortly thereafter, given a constant flow rate, the reference fluid boil-off rate will "level off". Despite the variation of flows that occur during experimental work, it is necessary to design around some figure for capacity, given by a residence time multiplied by an "average" flow rate of the reference fluid.

3. Adequate Flow Rates. Theoretically, the significance of the heat leak term can be reduced by the use of an adequate flow rate of the process fluid. As well, the amount of heat lost by the fluid due to any of the heat transfer mechanisms should be lower per unit mass since the fluid has less total contact time with the inlet tubing. From a practical point of view, it is also desirable to measure a significant boil-off flow rate and thus a larger process fluid flow rate is better. A constraint on this is, of course, that the higher the flow rate, the higher the pressure drop for a given restriction in the exchanger coil. This pressure drop effect must be considered if one wishes to report "isobaric" results.

4. Accurate Pressure, Temperature, and Flow Rate Measurements. Pressures and temperatures must be accurately determined in order to accurately specify the state of the fluid at inlet and outlet. Pressure is perhaps the easiest to determine, using calibrated Heise gauges or pressure differential cells with the associated dead weight equipment. The measurement of temperature in a flow situation in small tubes presents a larger possibility of error. One must decide either to measure temperature "in-line" or on the exterior of the line. If the thermocouple or platinum resistance thermometer being used is in contact with the tubing wall, there may be a small conduction effect cooling the device below the bulk fluid temperature. Also, depending on the way the device is

inserted into the line, there will be some conduction down the probe itself, tending to heat or cool the operative part of the probe. For in-line measurement, there is some influence of flow rate due to convection. If the temperature is not taken in-line, there may also be radiation effects in addition to the above.

Flow rate measurement errors probably comprise the second largest sources of inaccuracy after the heat leak problems, and, with the heat leaks minimised by proper design and/or correlated and corrected for, may actually represent the largest source of error in the value of enthalpy change for most calorimetric methods requiring values of flow rate. Thus, accurately calibrated flow meters are necessary for both process fluid and reference fluid.

5. Variety of Process Fluids. The design should include consideration of a variety of fluids at a reasonably wide range of temperatures and pressures. As new processes become important in such areas as coal gasification and other SNG techniques, for example, increasingly more extreme process conditions of temperature, pressure, and corrosivity are being encountered. A calorimetric facility for handling these same process fluids must be able to withstand the same conditions using tough, corrosion resistant alloys in parts and tubing with which the process fluid comes into contact.

6. Reference Fluid. Other design considerations relate to the handling and type of reference fluid. A fluid

with a boiling point near the ambient temperature should make possible significant reductions of heat leak in from the surroundings. The desired endpoint temperature for the process fluid enthalpy determinations will also be an important factor in deciding upon the best boiling temperature. Operation complexity will be reduced if an appropriate fluid with a normal boiling point near the temperature desired can be found. Increasing the boiling pressure above ambient will, of course, allow greater boiling temperatures for the same fluid, but this may bring further difficulties. The values of the enthalpy of vapourisation for the fluid should be well-known and documented for various conditions, since the boiling pressures will vary somewhat, even from day to day. A decision must be made as to how the flow rate will be measured, and whether the fluid will be vented or condensed and recirculated. The cost and availability of the fluid is important here, as well as its toxicity in the environment.

7. Miscellaneous. Adequate provision must be made for preheating the process fluid and for flow and pressure control. Ease of repetition of the experiments will depend to some extent on the manner in which especially flow rates are controlled and adjusted.

3.2 Description of Apparatus

The basic design of the calorimeter is similar to that described by Sahgal et al. (1965) used for low temperature work. Others have used modifications of this design for other work. (See the summarization of work by Sagara et al., Sood and Haselden, and Laverman and Selcukoglu in Table 2.1.) One basic modification used in this work was the use of Freon-11 as the reference fluid and the consequent use of a higher range of temperatures than those used by Sahgal et al. Another was the recirculation of the F-11 rather than condensing and collecting the liquid for weighing, as other workers have generally done.

The following description begins by tracing the path of the process fluid through the system, and will conclude with a description of the F-11 circulation system. A schematic illustration is given in Figure 3.1 and a detailed layout of the calorimeter vessel and internals appears in Figure 3.2. Appendix 1 contains some of the design calculations that were done prior to construction and reconstruction of certain components of the system.

From the supply cylinder, the process fluid was passed through a high pressure Tescom regulator and into the preheating section (not shown in Figure 3.1). In the case of CO_2, the supply cylinder was wrapped with "Heat-By-The-Yard" (HBTY) heating tape to raise the vapour pressure and so achieve higher inlet pressures for the CO_2 runs. Preheating of the process fluid was achieved simply

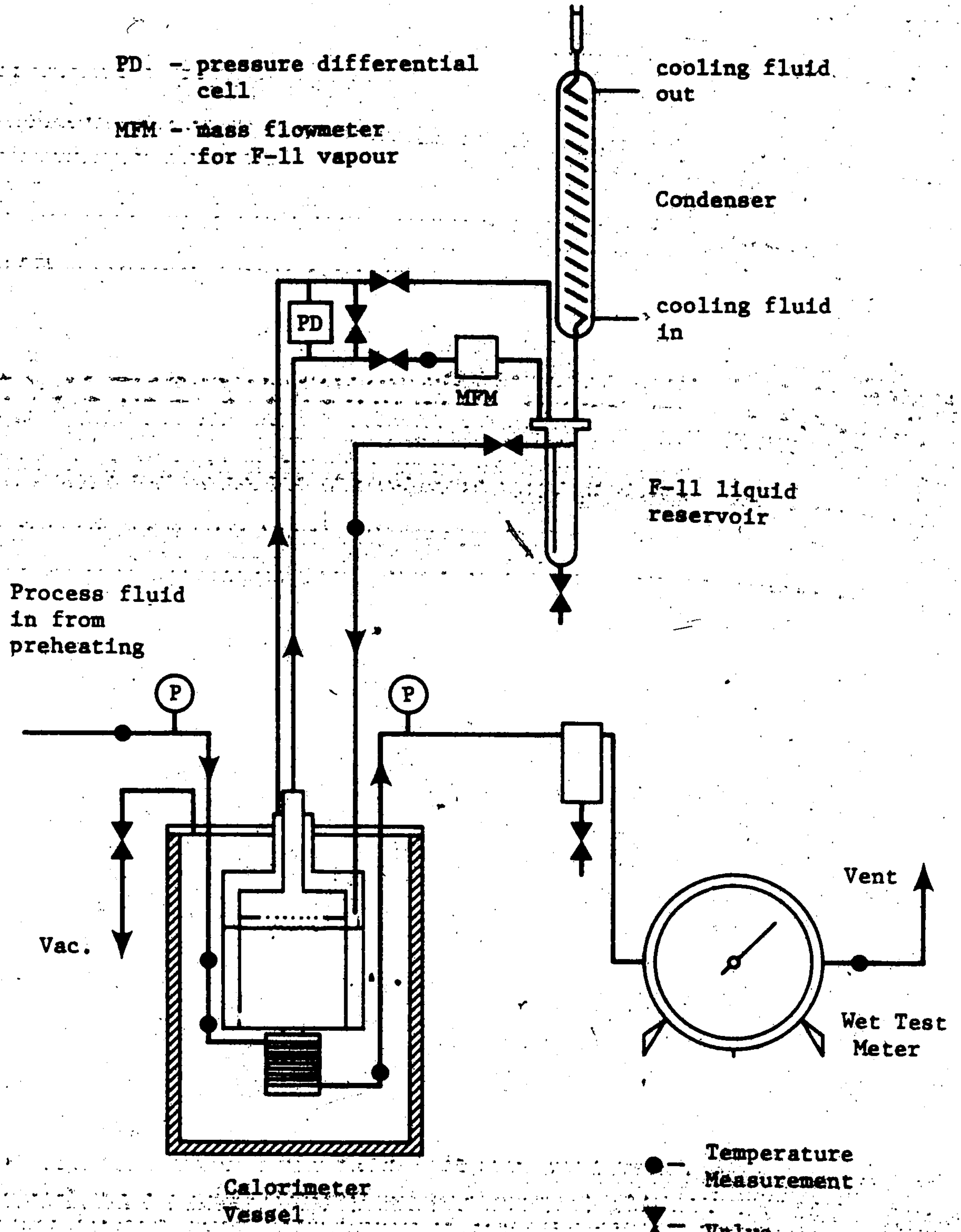
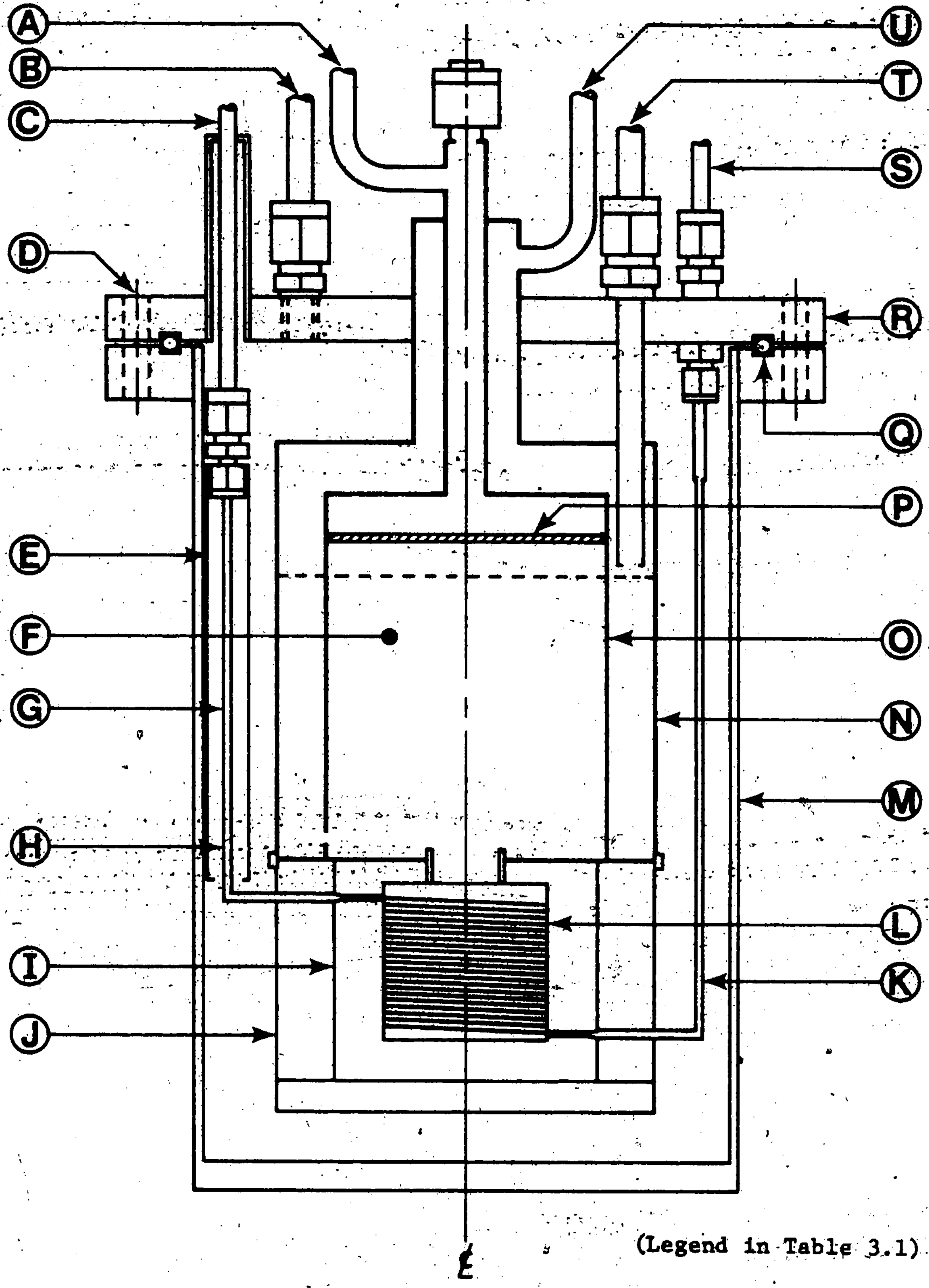


Figure 3.1 Schematic Diagram of Calorimeter

Table 3.1 : Legend for Figure 3.2 (Calorimeter Detail)

A - Inner vessel vapour exit line
B - Vacuum line
C - Process fluid inlet line
D - Bolt holes (8)
E - Inlet line radiation shield
F - Vapourising Freon-11
G - Approx. position of upper inlet T/C
H - Approx. position of lower inlet T/C
I - Inner radiation shield
J - Guard radiation shield
K - Approx. position of outlet T/C
L - Heat exchanger core and coil
M - Outer calorimeter vessel (evacuated)
N - Guard vessel
O - Inner vessel
P - Screen vapour demister
Q - Teflon wrapped aluminum ring seal
R - Calorimeter lid (support plate)
S - Process fluid outlet line
T - Liquid Freon-11 return line
(glass float level indicator inserted here)
U - Guard vessel vapour exit line
Scale: O.D. of Outer Vessel - 5 in (12.7 cm)



(Legend in Table 3.1)

Figure 3.2 Calorimeter Internal Detail

by tightly wrapping the section of 1/8 in (3.18 mm) O.D. stainless steel tubing[2] with HBTY heating tape. This section also included a Nupro sintered steel 60 micron filter element as a precaution against particulate build-up in downstream locations, and an in-line thermocouple.

After preheating, the fluid flowed past the capped connection (not shown) for future attachment of a gas chromatograph (G.C.) sample line, the inlet pressure gauge connection, and through the top of the calorimeter into the evacuated outer calorimeter shell where the line size is reduced from 1/8 in (3.18 mm) to 1/16 in (1.59 mm). Two Thermoelectric 0.020 in (0.51 mm) dia. Type J thermocouples are inserted into the inlet line through a Conax ceramic multihole sealing gland which is teed into the line just after the preheating section. The junctions of these thermocouples are 2 in (51 mm) apart in order to provide an indication of the temperature gradient, if any, along the inlet line. The upper thermocouple was later used as an in-line resistance heater to raise the inlet temperature above that achievable by the preheater. An aluminum foil radiation shield was placed around the inlet line inside the calorimeter in an attempt to decrease any radiative heat transfer from the inlet line to the calorimeter internals.

The fluid next passed into the exchanger composed of some 30 feet (approx. 9 m) of 0.018 in O.D. by 0.00425 in

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[2] Type 316 stainless steel was used for all process fluid tubing.

wall (0.46 mm O.D. by 0.11 mm wall) stainless steel tubing coiled around a 1.25 in (31.8 mm) copper core which is in thermal contact with the F-11 in the inner chamber of the calorimeter vessel. A radiation shield assembly was also constructed in order to minimise radiative heat losses from the exchanger coil to the outer vessel wall.

Upon leaving the exchanger, the fluid had undergone a small pressure drop and was 2 to 3 °C above the boiling temperature of the F-11. This temperature was measured by a 0.020 in (0.51 mm) dia. Thermoelectric iron-Constantan thermocouple inserted into the line in a manner similar to that of the inlet line. The outlet line leaves the evacuated outer vessel of the calorimeter through the top plate. At this point, a second Heise gauge was connected for outlet pressure observation. A capped connection for future attachment of a G.C. sample line was also provided here.

The fluid then passed through a Nupro fine metering valve and a Whitey shut-off valve to a knock-out drum from which any entrained condensed fluid may be collected[1], and on to a Precision Scientific wet test meter for volumetric measurement. A mercury thermometer located in the wet test meter indicated the meter temperature. The fluid leaving

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[1] It should be noted that passage through the needle valve into the knock-out drum is, in effect, an atmospheric flash. For runs in which a mixture undergoes some condensation, the amounts and compositions of the phases at this point will be different from the amounts and compositions of the phases leaving the calorimeter. The mixture used in this work, however, did not change phase in the calorimeter.

the wet test meter was vented to the air purge system of the laboratory.

Freon-11 was chosen as the reference fluid since its boiling point is near room temperature at the ambient pressure, which is near 93 kPa in this location. As such, the heat leak was reduced from that possible for fluids of lower boiling points. The vapourising F-11 was contained in the inner and guard boil-off chambers located within the calorimeter vessel. The guard vessel surrounds the inner chamber and was intended to be the sink for any heat leaking into the calorimeter from the surroundings. Since liquid F-11 flowed into the inner chamber from the guard chamber and the pressure differential between the chambers was monitored and maintained at a small value, the levels of F-11 in each were about the same and the height of the glass float in the the guard vessel was thus an indication of the level in the inner vessel. This float assembly replaced the unsuccessful thermistor-based level sensing system which was initially installed. Level changes during a run can cause changes in relative heat leak and small changes in enthalpy of vapourisation and boiling temperature. Because the F-11 was condensed and circulated back to the calorimeter, the liquid level did not change appreciably once steady operation was achieved, but the float was necessary to gauge the extent of filling.

The F-11 vapourised in the inner cell passed through a simple demister and left the inner chamber, flowing by the pressure differential cell, in-line thermocouple, and shut-off valve to a calibrated Brooks linear mass flow meter assembly and then to the reflux condenser through the vapour space in the liquid reservoir. The vapour boiled off in the guard vessel normally passed by the pressure differential cell, through a needle valve and into the reflux condenser, also by way of the liquid reservoir. After some unsuccesful attempts to operate an in-line condenser, an all glass reflux condenser was installed to operate at ambient pressure and use cooling water at 2 to 3 °C. At this temperature the vapour pressure of F-11 is 45 kPa, and very little was lost to the surroundings. Approximately 100 in (2.5 m) of 0.25 in O.D. by 0.0625 in wall (6.35 mm O.D. by 1.59 mm wall) glass tubing was formed into a coil of about 1.5 in (38 mm) in diameter and 20 in (0.5 m) high to provide the surface area for condensation. A calcium chloride dessicant was used to trap any water vapour that otherwise may have entered the top of the condenser. Vapour entered and liquid left the condenser through the connection to the reservoir immediately below the condenser.

From the reservoir the liquid F-11 returned to the guard vessel of the calorimeter through a liquid leg, shut-off valve, and past an in-line thermocouple for observation of the return temperature. The liquid leg was meant to serve as a vapour lock forcing all vapour flow through the lines described earlier and thus preventing the "short-circuiting" of guard vapour (and possibly some of the inner chamber boil-off) through to the condenser.

3.3 Instrumentation

Thermocouples. The thermocouples used were iron-Constantan (type J) grounded thermocouples. The three of these which were located in the fluid inlet and outlet lines inside the calorimeter were 0.020 in (0.51 mm) in diameter. The two thermocouples in the inlet line were manufactured by Omega, and the one in the outlet line by Thermoelectric. All others were 0.0625 in (1.59 mm) in diameter, also manufactured by Thermoelectric. Each thermocouple was connected to a separate ice-point reference junction and the resulting e.m.f. was read through a Thermoelectric multipoint switch by a Hewlett-Packard Model 3450A Digital Multi-Function Meter, which was also used to monitor signal voltages generated by other instruments described in the following sections.

All thermocouples were calibrated in the Chemical Engineering Instrument Shop against a platinum resistance thermometer. The process fluid thermocouples were calibrated for the temperatures from room temperature (approx. 22 °C) to about 400 °C. For calculation purposes, these calibration data were correlated by a least squares fit of equations of the following form:

$$Y = A + B*M + C/M + D*\ln(M) \tag{3.2}$$

where M is the thermocouple e.m.f. in millivolts and Y is one of the following functions of temperature:

1) $Y = T$ (T in °C) (3.3a)

2) $Y = T$ (T in K) (3.3b)

3) $Y = \ln(T)$ (T in °C) (3.3c)

4) $Y = \ln(T)$ (T in K) (3.3d)

From a comparison of the above four function types for each thermocouple, type (1) generally appeared to be the most accurate, based on the sum of the squared residual errors in temperature. The type (1) correlation was used for all thermocouples except those manufactured by Omega, for which the type (3) correlations were more accurate. It was estimated that the resulting precision of the temperature observations was ±0.05 °C. The accuracy of any temperature determined by these correlations in the range used in this work was ±0.1 °C.

The calibration charts, accompanied by the correlations and corresponding parameter valu~~es,~~ appear in Appendix 2.

Pressure Measurements. The calorimeter inlet and outlet pressures were observed using two Heise gauges with 316 stainless steel Bourdon tubes. The specifications of each are given below:

	Inlet gauge	Outlet gauge
Full scale	3000 psig (20.7 MPa)	10 MPa (1450 psig)
Accuracy	better than 0.1% F.S.	better than 0.1% F.S.
Hysteresis	minimal below 1500 psig	minimal
Precision	1 psig (6.9 kPa)	5 kPa (0.7 psig)

These gauges could be individually isolated from the flow line and compared to check mutual accuracies, if desired. If necessary, they could be vented rapidly to the laboratory air purge system via a shut-off valve in the event of accidental over-pressuring. The absolute pressures were calculated by adding these gauge pressures to the barometric pressure, which was read from a Fisher Scientific mercury barometer. Calibration details for the Heise gauges are provided in Appendix 2.

The calibrations of the 3 in (76 mm) Matheson gauges used in the regulator assembly were not extensively checked since these gauges provided approximate indications of the supply and upstream feed pressures.

A Celesco Transducer Products differential pressure cell was situated between the guard vessel and inner vessel vapour lines. It was calibrated to provide a 0 to 10 volt signal corresponding to a 0 to 0.1 psig (0 to 0.7 kPa) differential pressure range. This range was adequate for the operating conditions encountered.

Flow Meters. After some initial false starts involving dry test meters for the measurement of the F-11 vapour flow, a Brooks linear mass flow meter (Model No. 5810 B1F2E5A) was installed and was successfully employed for measurements of low to moderate vapour flow rates. For moderate to high vapour rates, a Brooks Model No. 5810 B1J2E5A flow meter was used. An attempt was made to shield the flow meter in use from room air currents, but it was not insulated and operated near room temperature. The actual vapour temperature was measured by an in-line thermocouple just prior to entry into the flow meter.

These flow meters produce a full scale linear signal ranging from 0 to 5 volts corresponding to 0 to 500 standard cubic centimeters of N_2 per minute (SCCM N_2) and 0 to 5000 SCCM N_2 for Models 5810 B1F2E5A and 5810 B1J2E5A, respectively. This signal was recorded on a Hewlett Packard Model No. 7127A Strip Chart Recorder and monitored through the Thermoelectric multipoint switch by the H.P. digital multimeter described earlier. Their calibration for F-11 was accomplished by vapourising the F-11 at certain rates, passing this vapour through the meters, condensing the vapour, and weighing the condensed liquid collected in a certain period of time. Efforts were made to prevent vapour losses. The data thus gathered for each meter were correlated by a linear least squares fit. The correlations and the figures comparing the fit with the calibration data appear in Appendix 2. The calibration accuracies were better than 25 mg/min for the smaller capacity meter (flow meter A) and 100 mg/min for the larger capacity meter (flow meter B). For each, the measurement precision was ±0.01 volt, or ±0.2 % of full scale, corresponding to 2 mg/min (0.015 mmol/min) for flow meter A and 16.9 mg/min (0.12 mmol/min) for flow meter B.

A Precision Scientific 0.1 cubic foot (2.83 l) wet test meter using distilled water was used to measure the volume of process fluid passing through the calorimeter during the period of the run. The meter could be read to the nearest 0.0005 cubic foot (14.1 ml) and the accuracy was ±0.5%. This meter operated at the room temperature, indicated by an internal mercury thermometer, and at the ambient barometric pressure. The process fluid mass flow rate during a run was calculated from this volume, temperature, and ambient pressure using appropriate values of the compressibility factor and the duration of the run. The meter was calibrated with a Precision Scientific 0.1 cubic foot (2.83 l) prover at several different flow rates.

Power Supplies. For all applications of the Heat-By-The-Yard heating tape, the 120 volt Variac power supplies were used. A Heathkit Model IP-27 regulated power supply was used to maintain a 300 mA current through the iron-Constantan in-line process fluid heater, which provided a resistance of approximately 35 ohms.

3.4 Experimental Procedure

Three different fluids were used in a series of test runs for the approximate inlet pressures of 1400, 1000, and 500 psig (9.7, 6.9, and 3.4 MPa). At each of these nominal test pressures, three or four flow rates were used, resulting in different inlet temperatures for a given preheater duty. The fluids were nitrogen, carbon dioxide, and a mixture of H_2 (9.7%), CO_2 (28.6%), and CH_4 (61.7%).

A general procedure for the test runs may be itemised as follows:

1. The refrigeration unit for condenser cooling water was started and maintained at 1 to 2 °C.

2. The coolant circulation pump was started to cool the F-11 condenser.

3. F-11 was charged to the calorimeter and the liquid reservoir through the chilled reflux condenser.

4. The process fluid preheater power supply was turned on.

5. The vacuum in the calorimeter outer vessel was checked and the vessel was re-evacuated if necessary.

6. The Heise gauges were zeroed and the thermocouple reference ice-water dewar was refilled.

7. The supply cylinder valve was opened and the regulator was set to provide the desired inlet pressure.

8. The power supply for the in-line process fluid heater in the calorimeter inlet line was activated.

9. The F-11 vapour flow rate recorder was turned on, as was the digital multimeter used to monitor the thermocouples and the mass flow meter.

10. The outlet needle valve was adjusted concurrently with the adjustment of the regulator to set the process fluid flow rate by setting the inlet pressure and the pressure drop across the exchanger. This was continued as necessary during startup to enhance achievement of steady operation, which was assumed when the inlet temperature, inlet pressure, outlet pressure, and F-11 vapour flow rate were constant.

11. The timer was started simultaneously with observation of the volume indicated on the wet test meter. Shortly thereafter, the F-11 vapour flow meter signal, and all thermocouple and pressure gauge readings were recorded.

12. Readings were repeated and recorded every ten to fifteen minutes.

13. Some gradual pressure changes, if any, were corrected by minor adjustments of the regulator and the needle valve.

14. After a sufficient period of steady operation was maintained and recorded, the process fluid flow rate was altered by adjustments of the outlet needle valve to cause a different pressure drop. This occasionally required some adjustment of the regulator as well.

15. Steps 10 through 14 were repeated as often as required to generate the desired data, then step 16 was undertaken.

16. The process fluid heater power supplies, F-11 vapour flow rate recorder, and digital multimeter were shut off. The supply cylinder valve was closed. The refrigeration unit and the condenser coolant circulation pump remained running to prevent excessive losses of F-11, especially if the series of runs was not yet completed.

There were several deviations from this procedure for runs with CO_2:

- Prior to step 7, the CO_2 supply cylinder was heated with HBTY heating tape to increase the supply pressure above the normal room temperature vapour pressure of CO_2 to about 1700 psig (11.7 MPa).

- There was a large cooling effect at the outlet needle valve which tended to alter the original setting of the pressure drop as the run continued. A short section of HBTY heating tape was wrapped around the valve and activated to warm it. This resulted in a much more steady operation.

4. Results and Discussion

4.1 Calculation Procedure

The experimental data collected during a series of runs were entered into data files similar to the sample given in Appendix 5. The program CALOR, written specifically for the purpose, and given in Appendix 3, read these files and converted the data to the more useful units for temperature, pressure, and flow rate. With these values and a data file providing F-11 enthalpy of vapourisation values dependent on the saturation pressure, the program calculated the experimental results for the process fluid enthalpy change.

Further, for N_2 and CO_2, which were used as standards, the program CALOR calculated predicted values of enthalpy change by using the inlet and outlet conditions of temperature and pressure. This was done by taking the difference of the values predicted for each state using a two-dimensional linear interpolation of enthalpy data extracted from the I.U.P.A.C. tables on N_2 (Angus et al., 1979) and on CO_2 (Angus et al., 1973), and provided to the program in appropriate data files of which copies appear in Appendix 4. In order to check the validity of linear interpolation for these data, the fitting and interpolation algorithms of Forsythe et al. (1977) were later used in a two-dimensional cubic spline interpolation in the same data files. The accuracy of these interpolations will be discussed later.

The program compared the experimental values with their associated predicted values of enthalpy change to calculate the heat leak at the particular experimental conditions, after the manner of equation (3.1).

As previously mentioned, the third process fluid used in the calorimeter was a mixture of CO_2, H_2, and CH_4. No standard thermodynamic data are available for this mixture and thus it is typical of fluids for which accurate calorimetric data may yet be needed. The experimental results in this case were compared to the predictions of the EQUI-PHASE program. This program is based on the "two-constant" Peng-Robinson equation of state (Peng and Robinson, 1976)) and is used extensively to predict the behaviour and properties of many mixtures of interest to the hydrocarbon processing industries.

The experimental conditions and results, which comprise the output of the program CALOR, are listed in Appendix 6.

4.2 Precision Analysis

Before the discussion of the accuracy of the calorimeter as a function of heat leaks, operational methods, and design decisions, it is appropriate to examine the effects of the precision of the experimental measurements on the certainty of the calculated results for enthalpy change. An examination of the precision of the corresponding predicted values of enthalpy change is also necessary in order to determine the limits of uncertainty for the estimation of heat leaks in the calorimeter.

The total derivative of the measured heat exchange, or, more specifically, the derivative of equation (2.11) is:

$$d\ \Delta h_{meas} = dq_{meas} = \frac{-\dot{m}_r}{\dot{m}_f}\, d\lambda_r + \frac{-\lambda_r}{\dot{m}_f}\, d\dot{m}_r + \frac{\lambda_r \dot{m}_r}{(\dot{m}_f)^2}\, d\dot{m}_f \qquad (4.1)$$

The precision of the experimental value of enthalpy change for a given run may be calculated conservatively by approximation of the differentials in equation (4.1) by the uncertainties in the corresponding variables. Since some of these experimental variables were themselves determined from other measured values, calculations of their precision were done in a similar fashion. Since especially the reference and process fluid flow rates change from run to run, a fair estimate of the precision of the measured enthalpy change must consider several different sets of experimental conditions.

Calculations for a number of representative runs with both N_2 and CO_2, are given in Appendix 7. The precision of the experimental values of enthalpy change for this work was thus determined to be better than ±485 J/mol for N_2 at the lower flow rate (approx 10 mmol/min), changing to ±140 J/mol as flow rate increased to 35 mmol/min, and ±485 J/mol for CO_2 changing to as much as ±575 J/mol when the higher capacity mass flow meter was used at the larger flow rates.

The corresponding predicted value of the enthalpy change was calculated from a difference of outlet and inlet values of enthalpy which were determined using a two-dimensional linear interpolation as previously indicated. This interpolation scheme is summarised in the equations below to clearly indicate how the calculation of precision proceeded.

$$h_{T^-,P} = \left[\frac{h_{P^+} - h_{P^-}}{P^+ - P^-}\right]_{T^-} \cdot (P - P^-) + h_{P^-,T^-} \qquad (4.2)$$

$$h_{T^+,P} = \left[\frac{h_{P^+} - h_{P^-}}{P^+ - P^-}\right]_{T^+} \cdot (P - P^-) + h_{P^-,T^+} \qquad (4.3)$$

$$h_{T,P} = \left[\frac{h_{T^+} - h_{T^-}}{T^+ - T^-}\right]_{P} \cdot (T - T^-) + h_{T^-,P} \qquad (4.4)$$

The question of the accuracy of linear interpolation is momentarily left aside. Calculation of the precision was done by evaluating the total derivative of equation (4.4), and this involved similar evaluations of the derivatives of equations (4.2) and (4.3). If it can be assumed that the values of enthalpy given in the I.U.P.A.C. tables are exact, or at least much more precise than the experimental measurements of temperature and pressure, then the precision of a predicted value of enthalpy depends upon the precisions of temperature and pressure in the following way, derived in Appendix 7.

$$dh = (j+fP)dT + (k+fT)dP \qquad (A7.4)$$

where the constants are calculated from only the tabulated values.

The parallel of equation (A7.4) with equation (2.2) is obvious and can, indeed, be used to judge whether the linear method of interpolation is appropriate for the data set. If comparisons of C_p with $(j+fP)$ and Ø with $(k+fT)$ at any condition (T,P) do not indicate near equality, then the assumptions of linearity of enthalpy with temperature and pressure are not good in the vicinity of that state and the linear interpolation method is unsuitable for use at that point. A sample of these comparisons is given in Appendix 7 and is representative of the bulk of the points tested. The conclusion drawn was that two-dimensional linear interpolation was a sufficiently accurate method for the N_2 data set and much of the CO_2 data set.

This conclusion was tested further by comparing the results of a more versatile interpolation method which does a better fit of any curvature present in the data. A run by run comparison of results using a two-dimensional cubic spline interpolation with the results of the two-dimensional linear interpolation was done for both N_2 and CO_2. For the N_2 runs, the enthalpy changes calculated from the spline interpolations were, on average, only 0.034 % larger than the changes calculated using linearly interpolated values of enthalpy. Similarly for CO_2 runs, the calculated enthalpy changes were only an average of 0.014 % larger when calculated using the spline interpolation. It seems clear, then, that the major contribution to the uncertainty of the predicted enthalpy change was due to the precision of the experimental measurements of temperature and pressure, and not, at least in this work, to the method of representation of the tabular data. This uncertainty in the enthalpy change values was ±7.0 J/mol for all N_2 runs and from ±55 to ±18 J/mol, decreasing as run pressure decreased, for CO_2.

Finally, the precision of the values of the heat leak for a certain run may be determined as follows:

$$dq_L = d(\Delta h_{pred}) + dq_{meas} \tag{4.5}$$

The precision of the calculated heat leak values ranged from ±490 J/mol to ±150 J/mol for N_2, as flow rate increased, and from ±510 J/mol to ±630 J/mol for CO_2, depending on which flow meter was used. Since the magnitude of uncertainty in the measured enthalpy change is larger than that of the predicted value, it appears that the first attempts to improve the precision must be in the experimental measurements, especially those of flow rate. The words of Masi (1954), quoted earlier, appear to be at least partially borne out.

4.3 Heat Leaks and Calorimeter Accuracy

Examination of the results of the experiments, given in Appendix 6, and as illustrated in Figures 4.1, 4.2, and 4.3, reveals that the performance of the calorimeter was rather severely hampered by the presence of heat leaks which ranged from approximately 0 to ±35 % of the measured boil-off heat exchange. In other words, the predicted enthalpy changes were anywhere from 35 % below to 35 % above the experimentally determined values.

Effects of design decisions and changes. The discussion on precision in the preceding section indicates that there was a sizeable uncertainty in the values of the heat leak, but given the size of the heat leaks calculated, it is quite clear that these results were not merely artifacts of the precision of the experimental measurements. Attempts have been made throughout the experimental work to modify the operation and also the construction of the calorimeter to reduce these heat leaks, and these attempts have met some, albeit limited, success. These attempts, briefly described, involved the redesign and reconstruction of the inlet line to the calorimeter in order to increase the resistance to conductive heat transfer from the hotter inlet line to the calorimeter internals prior to the exchanger core, the discarding of the heated air fluidised bed thermostat as a surrounding medium for the calorimeter vessel in favour of insulating the vessel and operating it at room temperature with the process fluid preheating being

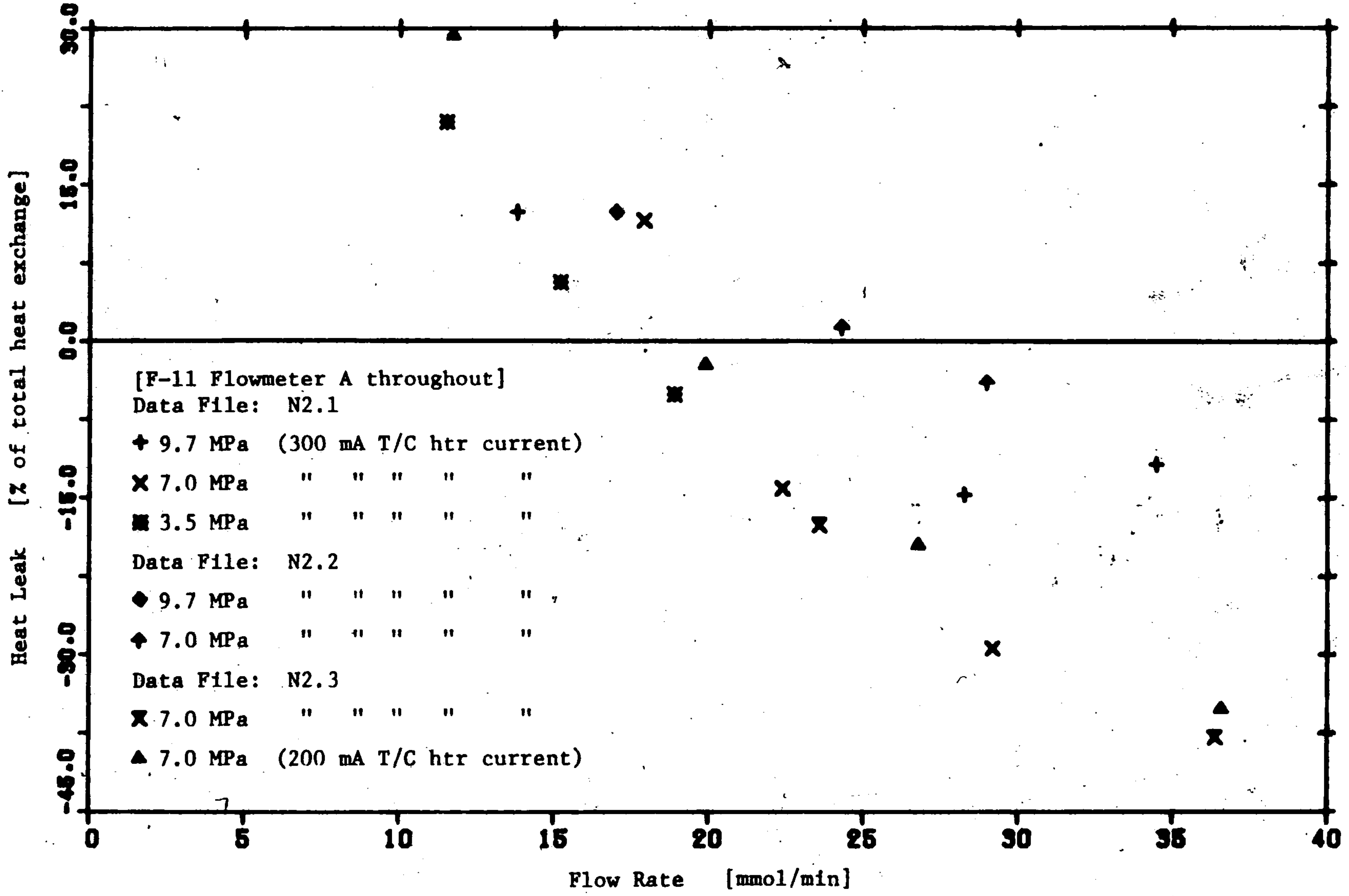

Figure 4.1 Effect of Flow Rate on Heat Leak (N_2 Runs)

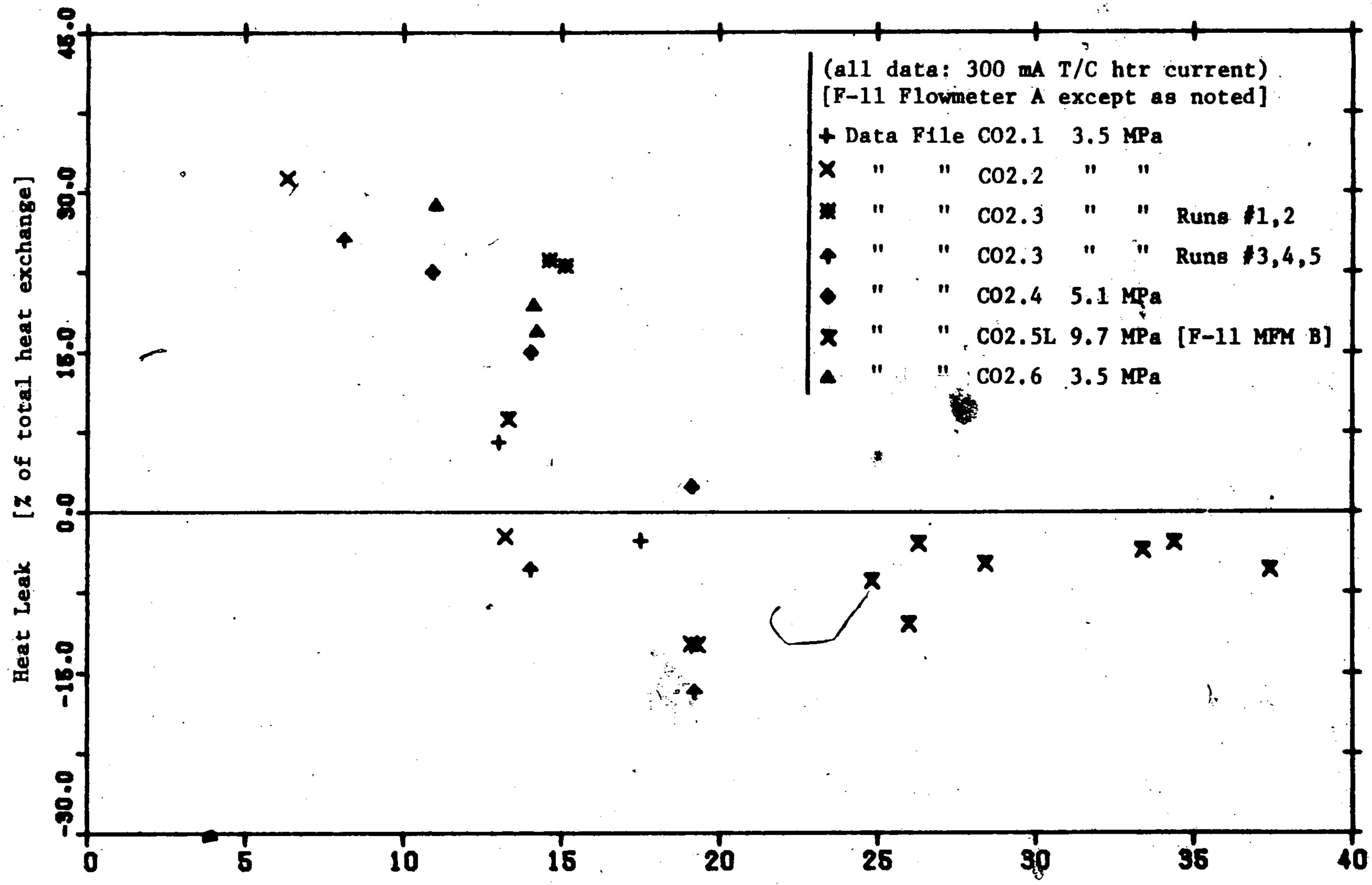

Figure 4.2 Effect of Flow Rate on Heat Leak (CO_2 Runs)

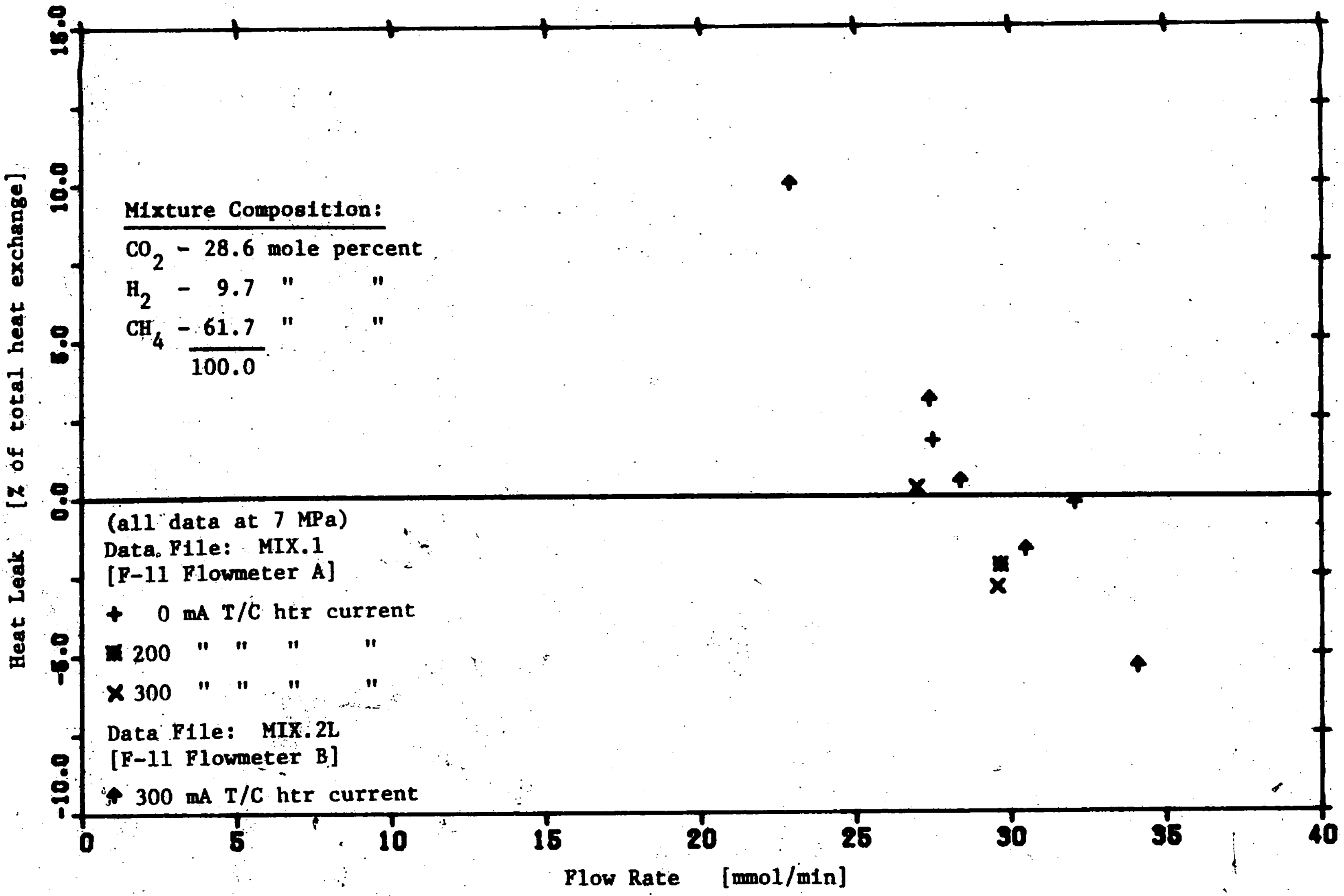

Figure 4.3 Effect of Flow Rate on Heat Leak (Mixture Runs)

done with HBTY heating tape, and installation of an aluminum foil radiation shield around the inlet line in the calorimeter vessel to reduce possible radiative heat transfer.

The primary effect of these modifications was a sizeable decrease in the amount of "background" heat leak, i.e., a reduction in the amount of heat leak into the calorimeter boil-off chamber under non-flow conditions. Determinations of background heat leaks were done and an example is included in Appendix 6 (Run CO2.1 #1). However, since the temperature profile changed from the non-flow profile when fluid was flowing through the calorimeter, these background values were of limited usefulness in correlating the actual heat leak. Eubank et al. (1982), whose work has been discussed earlier, did use non-flow values of heat leak to correct for heat leaks during flow conditions, since their calorimeter was appropriately equipped to adequately reproduce, during non-flow conditions, the temperature profile of the corresponding run with fluid flow.

A further design or development decision which appears to be detrimental to the eventual correlation of heat leaks is the decision not to include sufficiently automatic control of flow or inlet temperature of the process fluid. While such equipment may not be necessary if heat leaks were completely negligible, and may not be of any significance if the heat leaks are large, analysis of the mechanisms of heat leak may be easier if, for example, the temperature change could be independently controlled while flow rates and pressures were varied through a series of runs. Additionally, the reproducibility of the experimental conditions and results would be enhanced.

Because the reproduction of experimental conditions of temperature and pressure and, hence, flow rates was not easily achieved, it was not possible to directly investigate the effects of probable changes in other variables, such as small changes in reference fluid level from one run to another, due to the necessary periodic recharging of fluid to the system. Other examples are changes in atmospheric pressure with the corresponding small changes in saturation temperature, enthalpy of vapourisation, and resultant changes in temperature profile. Tables 4.1 and 4.2 summarise experimental work, the parameters of which, though not exactly identical, are perhaps similar enough to permit some comparison and analysis of difference in the results. A first impression was that calorimeter performance changed after recharging of F-11. Since F-11 levels were carefully monitored during the runs, these performance differences should not be due to differences in reference fluid levels. Casual checks of the F-11 for discoloration or dissolved oils indicated minimal change from the state of the original F-11 charged to the system, though it may be argued that even minimal changes in composition over time could cause substantial differences in the enthalpy of vapourisation of

Table 4.1 : Comparison of Run Results for N_2

Run #	$\dot{m}_f$	P_{in}	P_{out}	T_{in}	T_{out}	%L	Δh_{calc}
N2.1 #5	22.4	6.975	6.694	390.0	300.5	-14	-2793.5
N2.2 #1	24.3	7.006	6.704	395.6	300.3	+1	-2975.9

Basis of Comparison: inlet, outlet conditions similar
External Changes: see next set

N2.1 #6	29.2	6.988	6.594	403.3	302.3	-29	-3141.5
N2.2 #2	29.1	6.988	6.599	405.7	301.5	-4	-3242.9

Basis of Comparison: inlet, outlet conditions similar
External Changes: reconfiguration of vapour lines
- Calorimeter recharged with F-11

N2.3 #4	23.6	6.989	6.694	394.4	301.1	-18	-2910.0
N2.1 #5		as above					
N2.2 #1		as above					

Basis of Comparison: inlet, outlet conditions similar
External Changes: possible loss of F-11 over several days

N2.3 #5	36.4	6.996	6.387	418.1	304.9	-38	-3505.3
N2.3 #6	36.5	6.996	6.389	394.7	303.5	-34	-2831.0

Basis of Comparison: flow rates and pressures similar
External Changes: in-line htr current reduced from
300 mA to 200 mA

N2.3 #2	19.9	6.837	6.604	359.1	299.1	-2	-1882.7
N2.2 #2		as above					

Basis of Comparison: outlet pressures similar
External Changes: inlet pressure reduced
in-line heater current reduced to 200 mA

Table 4.2 : Comparison of Run Results for CO_2

Run #	$\dot{m}_f$	P_{in}	P_{out}	T_{in}	T_{out}	%L	Δh_{calc}
CO2.1 #2	17.5	3.499	3.118	398.5	300.2	-3	-4496.3
CO2.2 #1	19.1	3.527	3.088	404.7	301.6	-12	-4680.2
Basis of Comparison:	inlet, outlet pressures similar						
External Changes:	calorimeter recharged with F-11						
CO2.1 #3	13.0	3.551	3.333	383.9	298.6	+7	-4018.0
CO2.2 #3	13.1	3.527	3.316	385.4	299.5	-2	-4037.8
Basis of Comparison:	flow rates, pressures similar						
External Changes:	as above F-11 level slightly high in CO2.2 #3						
CO2.3 #1	15.1	3.537	3.270	387.7	299.0	+23	-4143.4
CO2.2 #3		as above					
Basis of Comparison:	pressures similar						
External Changes:	as above						
CO2.3 #2	14.6	3.526	3.280	386.9	298.9	+24	-4118.5
CO2.3 #1		as above					
CO2.2 #3		as above					
Basis of Comparison:	attempt to repeat CO2.3 #1						
External Changes:	none						
CO2.3 #3	14.0	3.568	3.328	392.4	300.2	-5	-4315.5
CO2.3 #1		as above					
CO2.2 #3		as above					
Basis of Comparison:	pressures similar						
External Changes:	calorimeter recharged with F-11 prior to CO2.3 #3						
CO2.3 #4	8.1	3.555	3.446	366.1	298.0	+25	-3293.6
CO2.2 #4	6.8	3.510	3.461	353.3	297.5	+31	-2751.2
Basis of Comparison:	outlet pressures, flowrates similar						
External Changes:	inlet pressure set higher in CO2.3 #4 one run prior to CO2.3 #4, F-11 was added						
CO2.3 #5	19.2	3.548	3.113	407.1	302.2	-17	-4773.0
CO2.2 #1	19.1	3.527	3.088	404.7	301.6	-12	-4680.2
CO2.2 #2	19.3	3.520	3.093	407.3	301.8	-12	-4796.8
Basis of Comparison:	flowrates and pressures similar						
External Changes:	probable higher F-11 levels in CO2.2 runs						

Run #	$\dot{m}_f$	P_{in}	P_{out}	T_{in}	T_{out}	%L	Δh_{calc}
CO2.5 #4	28.4	9.745	9.563	438.1	310.4	-5	-12576.6
CO2.5 #5	26.3	9.752	9.570	433.1	309.7	-3	-12502.8

Basis of Comparison: #5 is attempt to repeat #4
External Changes: none

CO2.5 #6	26.0	9.755	9.577	432.6	309.6	-10	-12486.4
CO2.5 #5	as above						
CO2.5 #4	as above						

Basis of Comparison: attempt to repeat after add'n of some F-11
External Changes: addition of some "make-up" F-11

CO2.5 #7	34.4	9.727	9.405	451.9	313.7	-3	-12029.7
CO2.5 #3	37.4	9.759	9.403	458.4	314.6	-5	-12025.2

Basis of Comparison: pressures similar
External Changes: F-11 added one run prior to CO2.5 #7

CO2.5 #8	24.8	9.728	9.408	430.4	309.2	-6	-12352.9
CO2.5 #7	as above						
CO2.5 #3	as above						

Basis of Comparison: pressures similar
External Changes: possible operational or reading errors?

CO2.6 #1	14.2	3.536	3.295	391.5	299.4	+17	-4311.5
CO2.3 #1	15.1	3.537	3.270	387.7	299.0	+23	-4143.4

Basis of Comparison: flowrates, pressures similar
External Changes: none

CO2.6 #2	14.1	3.515	3.260	390.5	299.4	+19	-4246.5
CO2.3 #2	14.6	3.526	3.280	386.9	298.9	+24	-4118.5

Basis of Comparison: flowrates, pressures similar
External Changes: none

CO2.6 #3	11.0	3.628	3.467	379.8	298.5	+29	-3896.2
CO2.3 #4	8.1	3.555	3.446	366.1	298.0	+25	-3293.6

Basis of Comparison: pressures similar
External Changes: none

the reference fluid. Other runs showed performance variations which cannot be linked to possible variations in F-11 level or composition. External causes for these are difficult to postulate.

Corrections for heat leaks. Keeping in mind that the reproducibility of these experimental results is limited and hence may lead to a correlation, or heat leak correction function, that is suspect of some error or at least incompleteness, it remains appropriate to attempt a correlation or partial explanation, in at least a qualitative sense, of the apparent behaviour of the calorimeter. In the literature, one finds a variety of techniques employed, depending on the type of calorimeter or the author's own preference.

One method, used successfully with glass calorimeters measuring Cp of organic vapours (Montgomery and DeVries, 1942), is a linear correlation of the measured property at some condition with the reciprocal of either the flow rate or the square of the flow rate, and subsequent extrapolation to zero reciprocal flow rate. This implies a similarly linear relationship between the heat leak per mole of process fluid at a given condition and the reciprocal of the flow rate, or of the flow rate squared. The method has an intuitive appeal in terms of equation 2.12 in which this heat leak term approaches zero as the process fluid flow rate increases, so long as the actual heat leak itself does not increase faster than the flow rate. Of course, variation in flow rates dictates variation of at least the pressure drop and these variations from the nominal conditions must also be corrected for in the determination of the measured value of the property at a given flow rate.

Judd et al. (1980) used a resistive heating method to measure enthalpies of mixing of condensable vapours. A correction for heat exchange with the surroundings was made in a manner similar to that of Montgomery and DeVries. Since the calorimetric method was completely different, it is interesting to note that a linear correlation of the measured property value, at a given temperature and pressure, with reciprocal flow rate was also successfully used here for extrapolation to the "corrected" value at zero reciprocal flow rate. Thus, this method appears to have broader applicability.

A correction for heat losses is also extensively discussed by van Kesteren and Zeldenrust (1979) who used a resistive heating method to measure enthalpies of condensable natural gas mixtures. These workers analysed heat losses more explicitly, in terms of conduction and radiation due to temperature differences in the calorimeter. It can be shown that this correlation of the heat leak per mole of process fluid for a given temperature profile involves a proportionality with reciprocal flow rate similar to those discussed earlier. Additionally, this proportionality can be calculated from a knowledge of the temperature profile. There is however, a certain non-zero value of the heat leak at infinite flow rate for which a correction must also be made.

Sagara et al. (1977) used a calorimeter also patterned after that of Sahgal et al. and thus similar in some respects to the one developed and tested in this work. The correlation used is one in which the heat leak is a function of the flow rate of the reference fluid and not of the process fluid as was used in the techniques discussed above. The reference fluid used was liquid N_2. This correlation, given in equation (4.6), can be shown to be similar to those described above which relate the he [illegible] to the reciprocal of the process fluid flow rate.

$$\frac{\dot{Q}_L}{\dot{m}_r} = \text{[illegible]} + \frac{10.7}{\dot{m}_r} \qquad (4.6)$$

If the heat leak is relatively small compared to the total measured heat exchange, then the reference fluid flow rate is approximately proportional to the process fluid flow rate. Thus the heat leak is approximately proportional to the process fluid flow rate and, following equation (4.6), the percentage heat leak is proportional to the reciprocal process fluid flow rate. The limits to this correlation are that it was said to hold only for boil-off heat exchanges below 243 cal/min (101[illegible] J/min corresponding to approx. 180 mmol N_2/min), where the heat leak percentage is equal to zero, and also that it can hold only above 10.3 cal/min (43 J/min corresponding to approx. [illegible] mmol N_2/min) at which point the heat leak percentage would be 100. It is to be noted that there was no mention of dependence on inlet temperature, though the data for which this correlation was used included inlet temperatures ranging from 110 K to 280 K.

Applicability of the correlations to this work. In the experimental work presented here, significant changes in flow rate at a certain nominal pressure could only be accomplished with sizeable changes in pressure drop, and large changes in the temperature profile were also observed. While the process fluid heaters could have been manually adjusted to minimise the change in temperature profile as flow rates changed, this was not done. The heat leak analysis of a similar calorimeter by Sagara et al. appears to imply that this is not necessary. In addition, the major concern through most of the work was to determine the size of the heat leak and to make changes to the calorimeter or recommendations for such changes in order to reduce the heat leaks to the point where questions of reproducibility really become important. As well, the run time that would be expended and the change of the base setting of the process fluid heater that would be required, if restoration to the original inlet temperature were attempted, were considerations that discouraged this.

Hence, the new flow resulted in new conditions so that the heat leak could be determined there. Since the conditions were not restored to the original nominal values, correlation of the measured property values or heat leak with only flow rate varying is not possible here. However, following the lead of such a correlating technique, the experimental heat leak percentages are plotted versus flow rates of N_2, CO_2, and the CH_4-H_2-CO_2 mixture, respectively, in Figures 4.1, 4.2, 4.3. It must be pointed out here that, as flow rate increased, so did the inlet temperature (due to better heat transfer from the in-line heater as well as in the preheating section) in a roughly linear way (Figure 4.4), regardless of the pressure. Thus temperature was a "hidden" variable here, and the value of these plots is not to be found in a directly extracted heat leak versus flow rate relationship. They do, however, serve to illustrate the magnitude of the problem and the trend of the problem with flow rate, given the operating strategy used.

It is encouraging to note in Figure 4.3 that, for the gas mixture, the agreement of the experimental results with the predictions of the Peng-Robinson equation of state is somewhat better than what could be expected based on the experience with the N_2 and CO_2 tests.

Most of the methods described above deal with positive heat leaks which approach zero at high flow rates. Though, as previously stated, the functionality of heat leaks with flow rate cannot be determined from Figures 4.1, 4.2, and 4.3 alone, the decreasing trend of heat leak with flow rate appears to be borne out in this work too. However, as seen in these figures, rather than approaching zero at higher

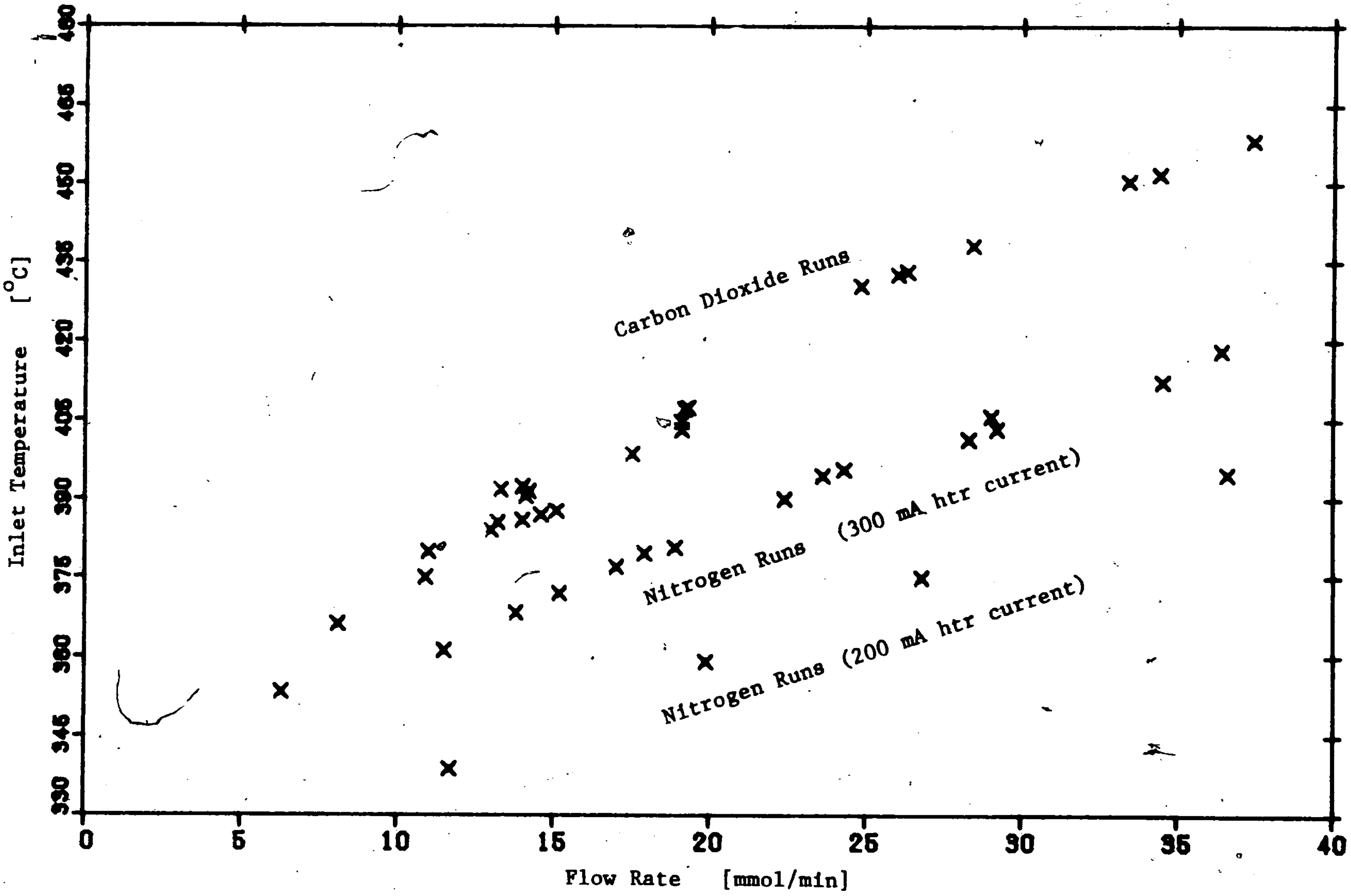

Figure 4.4 Flow Characteristics

flow rates, the heat leak passes through zero and becomes negative for many runs. The reasons for this behaviour are the subject of the following discussion and of possible future correction by design and reconstruction.

Phenomenology. Obviously, there must be an operating mechanism for heat loss from the fluid after the inlet temperature is measured, which increasingly negates the (positive) heat leak into the calorimeter boil-off chamber as flow (and with it, inlet temperature) increases. It may be useful then to regard the total leak as a combination of a relatively constant positive heat leak into the calorimeter and a negative term (heat loss from fluid not transferred to the vapourising reference fluid) which increases with flow rate, at least in the range of conditions studied. (It may be that at much larger flow rates, some steady negative value will be attained.) Here again it should be stated that, had there been more data in which the temperature was controlled independently of flow rate, the mechanism might have been easier to deduce and quantify. However, some discussion which may serve to shed light on the problem follows.

As the process fluid flow rate increases, the positive contribution to heat leak probably increases slightly, since the temperature gradient down the inlet line coincidentally increases. Thus, conduction down the line to the heat exchanger core and eventually to the inner vessel may be increased, even though the tubing walls are very thin and provide minimal conduction of heat flow. This is a common heat leak path to which others have also referred (eg. Eubank et al., 1982). Obviously though, if there is an increase in heat leak into the boil-off chamber, it appears to be more than compensated for by some larger increase in the heat loss terms.

It is felt that one of these terms is actually more properly described as a matter of accuracy of the measurement of the bulk fluid temperature at the inlet. Because of the small diameter of the tubing relative to the diameter of the thermocouple, it may be that the thermocouple is in partial contact with the wall which is at a slightly lower temperature than the bulk fluid at that point. Hence both the inlet temperature used and the prediction of the enthalpy change will be too low. Because the actual fluid enthalpy at inlet is higher, the actual measured enthalpy change is higher and a positive heat leak is calculated. As flow rates increase, however, the wall temperature will approach the bulk fluid temperature more closely, and the thermocouple temperature will more closely approximate the bulk temperature so that the positive "heat leak" calculated due to this effect will be reduced. This contribution will approach zero as flow rate increases and will never be negative.

Perhaps more important is the increase of heat loss from the heat exchange core due to radiation and conduction as the inlet temperature (and with it, flow rate) increases.

If the radiation shields used are not effective, the radiative heat transfer to the surroundings increases proportionally with the fourth power of the absolute temperature, and this could account for some of the increasingly negative heat leak with flow rate. Calculations done for the inlet line above the inlet thermocouple, given in Appendix 1, indicated that radiative losses were more significant than conduction down the line, but the installation of a foil radiation shield around the inlet line produced little noticeable increase in inlet temperature. The inlet temperature was obviously not so high that radiation had a large effect, so the radiation shields may not be that important at the temperatures used in this work, except in so far as improper construction could actually increase the heat loss.

A second loss mechanism could be the conductive heat transfer through the dead air space, if the vacuum was not sufficiently high to be effective. However, this effect is smaller than the radiation effect, especially at the temperatures used here.

To summarise the previous discussion, it suffices to say that the accuracy of the calorimeter is severely affected by heat leaks, the primary mechanisms of which appear to change with flow rate and temperature. For this calorimeter, then, the correction correlation does not depend on flow rate alone. This is not the implication of the correlation used by Sagara et al. and thought to be applicable to this work as well. As stated very early in the section on design considerations, a calorimeter with measurable heat leak may still be usefully accurate if an adequate correlation for the correction of this heat leak is attainable. At this point, such a correction correlation does not appear possible based on flow rate alone and further work would have to be done to examine independently the effect of inlet temperature and of flow rate on the heat leak. The advisability of this is doubtful, however, given the magnitude of the heat leaks still occurring, since in severe cases the correction term might be as high as one third of the measured value of the enthalpy change. It would be best to further improve the performance of the calorimeter to the point where, for example, the heat leaks are only ±5 % of total measured heat exchange, and once that has been achieved, to seek to correlate the remaining "error". Recommendations for further improvements are given in the next section.

4.4 Recommendations for Further Work

Since the calorimeter continues to suffer from sizeable heat leaks, further changes will be required for satisfactory performance. The size of the heat leaks should be reduced before installation of equipment for better overall control would be advisable. Should this not be achievable, work on a complete redesign of the apparatus should be begun, assuming that it is still deemed desirable to obtain this type of data in this department. The recommendations, then, which follow are in these three areas: improvement of performance of the existing apparatus; improvement of control and range of operation; and design and construction of a new device, if necessary.

Improvement of performance.

1. The insulation around the outer vessel may be removed and the calorimeter submerged in a larger dewar filled with F-11 at the same conditions as are present in the inner vessel. This will reduce heat leaks from the surroundings, which can vary slightly due to variations in room temperature. This will involve some changes to the inlet line preheating system as well, but these changes are discussed further in the next section.

2. Professional advice on radiation shielding construction should be obtained prior to reconstruction of the shields. Reconstruction will become increasingly necessary as the inlet temperatures increase.

3. A vacuum diffusion pump should be installed in order to determine if very high vacuum in the outer vessel causes any change.

4. A thinner top supporting plate may reduce heat leak by conduction from the hot inlet line. This would involve a great deal of alteration and would not be necessary if recommendation (1) above were acted upon.

Improvement of control and range of operation. The following steps should be taken if the actions recommended above are successful. They should also be considered during the design of a new device if that is deemed necessary or desirable.

1. Process fluid heating should be done by a preheating section and a final trim heating section composed of an in-line heater controlled to a desired temperature by an automatic temperature controller. (A small diameter iron-Constantan thermocouple may be used as an in-line trim heater.) Heating capability should be sufficient to reach at least 400 °C for all flow rates desired. The inlet line should be well insulated, particularly where it runs through the surrounding F-11 bath recommended earlier.

2. Tighter control of process fluid flow rate would be possible with either a back pressure controller or a flow controller. A larger range of flow rates, for pressure drops below a given maximum, would be possible if larger diameter tubing were used in the exchanger coil. This would allow for more nearly isobaric results at higher flow rates.

3. A mass flow meter could be installed in place of the wet test meter for the process fluid flow rate determination. This would give instantaneous flow rate readings and may be more accurate in some cases but would be more difficult to calibrate for some of the mixtures for which data are desired.

4. A Ruska pump, providing positive displacement of fluids at variable speeds, might be installed in the system along with an appropriate heated mixing manifold with pressure ballast tank to allow preparation of mixtures of gases and steam. In addition, another pump may be used, in lieu of high pressure supply cylinders, to achieve the higher pressures when desired.

5. The existing sampling tees may be connected to a G.C. in order to analyse for any composition changes between inlet and outlet due to coking as a result of high inlet temperatures or due to condensation during cooling in the coil.

<u>Design and construction of a new apparatus.</u> It may well be found that even after further work to improve performance of the existing apparatus, the behaviour remains unacceptable for high quality work. If so, the following suggestions relative to the calorimeter redesign might be considered in addition to those above.

1. The boil-off matching idea of Eubank et al. (1982) for comparative calorimetry appears to be successful, and its application to a future apparatus should be examined.

2. The volume of reference fluid in the calorimeter should be increased above that of the existing device, especially if the higher operating conditions are to be investigated accurately. A more directly visual method of liquid level assessment would be helpful, but not absolutely necessary.

3. The F-11 circulation system, with its reflux condenser and liquid reservoir, is effective as currently operated. For larger vapour flow rates, the vapour line diameter should be increased to 1/2 inch (12.7 mm) to minimise any pressure drops.

4. Further radiation losses may be avoided by immersing the exchanger coil directly in the reference fluid within the boil-off chamber. However, this would result in the loss of ability to easily control the outlet temperature at a value other than the reference fluid boiling point. This may be of concern if heat capacity values, for example, rather than enthalpy changes, were desired.

Nomenclature

A, B, C, D - correlation coefficients for a thermocouple

Cp - constant pressure heat capacity, J/mol K

e - internal energy, J/mol

f, j, k - interpolation constants determined solely from the values of h, T, and P given in data tables for N_2 and CO_2

g - acceleration of gravity, 9.8 m/s^2

h - specific enthalpy, J/mol

I - electric current, amp

m - mass, kg

$\dot{m}_f$ - molar flow rate of process fluid, mol/min

$\dot{m}_r$ - molar flow rate of reference fluid, mol/min

M - thermocouple e.m.f., millivolts

P - pressure, MPa

P^+, P^- - pressure entries in the data tables, just greater than and just less than the pressure of interpolation, MPa

q - heat transferred per mole of process fluid, J/mol

$\dot{Q}_L$ - heat leak rate, J/min

R - electrical resistance, ohm

s - specific entropy, J/mol K

T - temperature, K

T^+, T^- - temperature entries in the data tables, just greater than and just less than the temperature of interpolation, K

u - velocity, m/s

v - specific volume, m^3/mol

V - electrical potential, volt

x - composition, mol fraction

Y - a correlative function of temperature

z - displacement, m

$\%L$ - heat leak as a percent of the total heat exchange

δm - change of mass, kg

δQ - heat flow into system, J

δt - change in time, s

δW - work out of system, J

Δh difference in enthalpies of the outlet stream and inlet stream, denoted by subscript as either predicted by interpolation of each in tables or by P-R equation of state, or measured by experiment, J/mol

λ_r - enthalpy of vapourisation of reference fluid (for F-11 at 93 kPa, λ_r = 24872 J/mol)

μ - Joule-Thomson coefficient, K/MPa

ϕ - isothermal expansion coefficient, J/mol MPa

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Appendix 1 - Design Calculations

Some design calculations are included here as examples of the types of calculations necessary for the construction of this device.

A. Calorimeter Size.

The calorimeter inner vessel was sized to allow a "residence time" of about 30 minutes, based on a heat exchange rate of approximately 2000 J/min, corresponding to a F-11 circulation rate of 11 g/min. The actual heat exchange rates ranged from 2% to 25% of the design heat exchange rate.

$$\dot{Q} \doteq 2000 \text{ J/min} \qquad \dot{m}_r = \dot{Q}/\lambda_r \doteq \frac{2000}{24850} \text{ mol/min} \doteq 0.080 \text{ mol/min}$$

$$\text{Capacity} = \dot{m}_r \times 30 \text{ min residence time}$$

$$\doteq 2.5 \text{ mol F-11 (sat. liq. at 0.093 MPa)}$$

$$\text{Volume required} \doteq 2.5 \text{ mol} \times 92.5 \text{ cc/mol} \doteq 230 \text{ cc}$$

A cylindrical volume of at least 2.5 in (6.4 cm) high and 1.35 in (3.4 cm) in radius will contain this amount of F-11. Surrounding this inner vessel is a "guard" vessel of 1.75 in (4.4 cm) in radius containing F-11 at similar conditions.

B. Process fluid line size.

Based on some initial calculations using a high process fluid flow rate which would produce a (high) velocity of approx. 100 ft/sec (30.5 m/sec) at 750 °F (400 °C) and 2000 psia (13.9 MPa), the heat exchange coil tubing size selected was 0.018 in O.D. by 0.00425 in wall (0.46 mm O.D. by 0.11 mm wall). The wall thickness is sufficient for the conditions as seen from the following calculation as outlined in the Chemical Engineers' Handbook (Perry and Chilton, 1973), Chapter 6.

$$\text{min. wall thickness} = t_m = \frac{P * O.D.}{2*(S * E + P * Y)}$$

where P is the pressure, O.D. is the outside diameter, and

S is the maximum allowable stress as per A.S.M.E. Code at 750 °F : S = 9100 psi

E is the weld joint efficiency (Perry, Table 6-5)

E = 0.8

Y is the coefficient for stress redistribution due to creep at higher temperatures (Perry, Table 6-6)

Y = 0.4

Thus, the minimum wall thickness = 0.0031 in (0.079 mm). The actual wall thickness is 0.00425 in (0.11 mm).

Other tubing sizes used in the process fluid line were checked in a similar fashion.

Due to the flow rates actually used experimentally, the velocities in the coil ranged from approx. 10 to 30 ft/s (0.3 to 0.9 m/s). Higher velocities could be achieved with the use of larger pressure drops, if necessary to ensure entrainment of condensation occurring during cooling.

The exit line tubing was sized so that, as nearly as possible, the velocity of the process stream out of the coil was maintained until out of the whole calorimeter assembly. Tubing of 0.0355 in O.D. by 0.00625 in wall (0.90 mm O.D. by 0.159 mm wall) provided an inside diameter of 0.023 in (0.58 mm) allowing the insertion of a 0.020 in (0.51 mm) dia. thermocouple, leaving an annular area similar to the inside cross-sectional area of the heat exchange coil.

C. Heat transfer calculations.

The process fluid line entrance to the calorimeter was reconstructed based on the following calculations. The calculations below are done in British units with S.I. units given where appropriate.

Thermal conductivity

Stainless steel: $k_{ss} = 113 \frac{\text{Btu}\cdot\text{[illegible]}}{\text{hr}\cdot\text{ft}^2\cdot{}^0\text{F}} = 16.3\ \text{W/m}\cdot{}^0\text{C}$

Nylon: $k_n = 1.68 \frac{\text{Btu}\cdot\text{in}}{\text{hr}\cdot\text{ft}^2\cdot{}^0\text{F}} = 0.24\ \text{W/m}\cdot{}^0\text{C}$

Temperatures

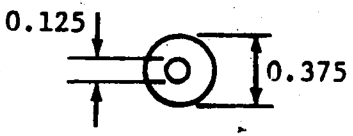
let line temp. be $T_h = 320\,^0\text{F} = 160\,^0\text{C}$

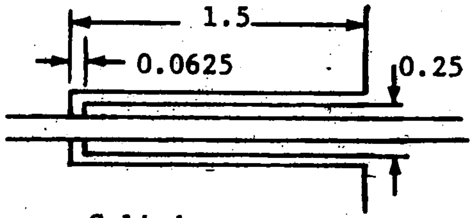
" calorimeter lid temp. be $T_b = 77\,^0\text{F} = 25\,^0\text{C}$

Inlet constructions

A) "Capped Cylinder" - well insulated and evacuated

Dimensions: (in inches)





Disk

$$\dot{q} = -k_{ss}\cdot 2\pi\cdot r\cdot t\cdot\frac{dT}{dr}$$

$$\dot{q}\cdot\int_{r_c}^{r_t}\frac{dr}{r} = -k_{ss}\cdot 2\cdot\pi\cdot t\cdot\int_{T_c}^{T_h} dT$$

$$\dot{q}\cdot(\ln r_t - \ln r_c) = -k_{ss}\cdot 2\cdot\pi\cdot t\cdot(T_h - T_c)$$

Cylinder

$$\dot{q} = -k_{ss}\cdot A_x\cdot\frac{dT}{dx}$$

$$\dot{q}\cdot\int_{L}^{0} dx = -k_{ss}\cdot A_x\cdot\int_{T_b}^{T_c} dT$$

$$\frac{\dot{q}\cdot L}{k_{ss}\cdot A_x} = T_c - T_b$$

(1) $\dot{q} = 0.280499(320 - T_c)$ Btu/hr (2) $\dot{q}\cdot 31.1523 = T_c - 77\,^0\mathrm{F}$

Substitute (1) into (2) to solve for T_c, then solve for $\dot{q}$.

$\rightarrow T_c \doteq 295.05\,^0\mathrm{F}$; $\dot{q} \doteq 7.0$ Btu/hr $\doteq 2.05$ Watt

---

If poorly insulated, this construction may be analysed as a fin with uniform cross section and uniform k and h_c $\rightarrow T = T(x)$ only

For this case, $\frac{d^2T}{dx^2} = m^2(T - T_\infty)$ where $m^2 = \frac{h_c P}{kA_x}$ $\left[\begin{array}{l} P = \pi(\mathrm{O.D.}) \\ A_x = \frac{\pi}{4}(\mathrm{O.D.}^2 - \mathrm{I.D.}^2) \end{array}\right.$

General sol'n: $T - T_\infty = C_1 e^{(m)0} + C_2 e^{mx}$

For $T_{x=0} = 77\,^0\mathrm{F}$ and $T_{x=1.5} = 320\,^0\mathrm{F}$ with $h_c = 2\,\frac{\mathrm{Btu}}{\mathrm{hr}\cdot\mathrm{ft}^2\cdot{}^0\mathrm{F}}$;

the constants are: $m = 0.34\ \mathrm{in}^{-1}$, $C_1 = -365\,^0\mathrm{F}$, and $C_2 = 365\,^0\mathrm{F}$

Thus, $T - T_\infty = 365(e^{0.34x} - 1)\ ^0\mathrm{F}$

Then, $\dot{q}_{fin} = \int_0^{1.5} h_c P(T - T_\infty)dx = \int_0^{1.5}\frac{2}{144}(\ \cdot 0.375)365(e^{0.34x} - 1)dx$

$$\dot{q}_{fin} = 6.0\left[\frac{e^{0.34x}}{0.34} - x\right]_0^{1.5}\cdot\frac{1054}{3600}\ \text{Watt} = \underline{0.8\ \text{Watt}}$$

For a more severe estimate, use $h_c = 20\,\frac{\mathrm{Btu}}{\mathrm{hr}\cdot\mathrm{ft}^2\cdot{}^0\mathrm{F}}$.

Then $T - T_\infty = 60(e^{1.08x} - 1)\,^0\mathrm{F} \rightarrow \underline{\dot{q}_{fin} = 6.6\ \text{Watt}}$

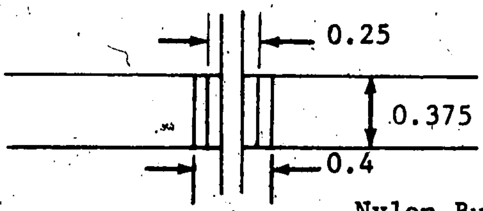
However, this value is a high estimate since the construction is insulated.

Inlet constructions (cont'd)

B) Bulkhead Reducing Union in Nylon Bushing in Lid

Dimensions:

(in inches)



St. St. Union

$$\dot{q} = -k_{ss}\cdot 2\cdot\pi\cdot r\cdot t\cdot\frac{dT}{dr}$$

$$\dot{q}\cdot\int_{r_s}^{r_t}\frac{dr}{r} = -k_{ss}\cdot 2\cdot\pi\cdot t\cdot\int_{T_s}^{T_h} dT$$

$$\dot{q}\cdot(\ln r_t - \ln r_s) = -k_{ss}\cdot 2\cdot\pi\cdot t\cdot(T_h - T_s)$$

Nylon Bushing

$$\dot{q} = -k_n\cdot 2\cdot\pi\cdot r\cdot t\cdot\frac{dT}{dr}$$

$$\dot{q}\cdot\int_{r_b}^{r_s}\frac{dr}{r} = -k_n\cdot 2\cdot\pi\cdot t\cdot\int_{T_b}^{T_s} dT$$

$$\frac{-\dot{q}\cdot(\ln r_s - \ln r_b)}{k_n\cdot 2\cdot\pi\cdot t} = T_s - T_b$$

(1) $\dot{q} = 2.6675(320 - T_s)$ Btu/hr   (2) $\dot{q}\cdot 17.098 = T_s - 77^0F$

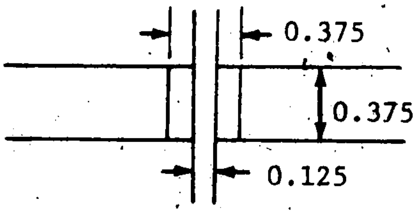
Substitute (1) into (2) to solve for T_s and $\dot{q}$.

$\rightarrow T_s = 314.79^0F$; $\dot{q} \doteq 13.91$ Btu/hr $\doteq 4.07$ Watt

C) Bulkhead Union in Lid

Dimensions:

(in inches)



St. St. Union only

$$\dot{q} = -k_{ss}\cdot 2\cdot\pi\cdot r\cdot t\cdot\frac{dT}{dr}$$

Similar to the above,

$$\dot{q}\cdot(\ln r_t' - \ln r_b) = -k_{ss}\cdot 2\cdot\pi\cdot t\cdot(T_h - T_b)$$

$$\dot{q} = 1.683\cdot(243)\ \text{Btu/hr} \doteq 409\ \text{Btu/hr} \doteq 120\ \text{Watt}$$

Thus, of the inlet constructions examined, construction A appears to provide the minimum heat loss to the calorimeter lid.

Some inlet line heat leak calculations

The estimated radiative heat losses from a section of the inlet line inside the calorimeter are compared to the corresponding conductive heat losses for two relatively low temperature cases. It is found that even for these cases radiation is more important than conduction, and this will be even more true at higher operating temperatures.

Dimensions: Tube O.D. = 0.065 in = 0.1651 cm
I.D. = 0.047 in = 0.1194 cm
Tube section length (distance between measuring thermocouples) = 2 in = 5.1 cm

Physical Constants and Properties:

$k_{ss} = 113$ Btu·in/hr·ft^2·°F $\doteq$ 16.3 W/m·K

$\sigma = 1.355 \times 10^{-12}$ cal/s·cm^2·K^4 = 5.669×10^{-8} W/m^2·K^4

$\varepsilon = 1$; $F = 1$ (conservative values for emissivity and view factor)

A. Conductive heat transfer

i) $T_1 = 312$ K, $T_2 = 308.5$ K ; $\Delta T = -3.5$ K

$$\dot{Q} = -k_{ss} \cdot A_x \cdot \frac{dT}{dx} \doteq -k_{ss} \cdot A_x \cdot \frac{\Delta T}{\Delta x}$$

$$= -16.3 \cdot \pi/4 \cdot (0.001651^2 - 0.001194^2) \cdot (-3.5/0.051) \text{ Watt}$$

$$\dot{Q} \doteq 1.14 \times 10^{-3} \text{ Watt}$$

ii) $T_1 = 333.6$ K, $T_2 = 329.6$ K ; $\Delta T = -4.0$ K

Similar to the above,

$$\dot{Q} \doteq 1.31 \times 10^{-3} \text{ Watt}$$

B. Radiative heat transfer (without shield)

i) let average tube wall temp be $T_t \doteq 310$ K ; $T_{surr} = 295$ K

$$\dot{Q} = \pi \cdot D \cdot \Delta x \cdot \varepsilon \cdot F \cdot \sigma \cdot (T_t^4 - T_{surr}^4)$$

$$= \pi \cdot 0.001651 \cdot 0.051 \cdot 1 \cdot 1 \cdot 5.669\times10^{-8} \cdot (310^4 - 295^4) \text{ Watt}$$

$$\dot{Q} \doteq 2.49 \times 10^{-2} \text{ Watt}$$

ii) let $T_t \doteq 330$ K; similar to the above, $\dot{Q} \doteq 6.43 \times 10^{-2}$ Watt

Appendix 2 - Instrument Calibrations

A. Calibration of Thermocouples.

The thermocouples were calibrated in the Instrument Shop and the calibration results for each thermocouple were correlated by least squares fit to an equation of the following form:

$$Y = A + B*M + C/M + D*\ln(M) \tag{3.2}$$

where Y is one of the following functions of temperature:

$$1)\ Y = T \qquad (T \text{ in } ^\circ C) \tag{3.3a}$$

$$2)\ Y = T \qquad (T \text{ in } K) \tag{3.3b}$$

$$3)\ Y = \ln(T) \qquad (T \text{ in } ^\circ C) \tag{3.3c}$$

$$4)\ Y = \ln(T) \qquad (T \text{ in } K) \tag{3.3d}$$

A summary of T/C locations and correlations follows:

Sw. No.	T/C No.	Location	Y	Correlation Coefficients A	B	C	D
1	1	Mass Flow Meter	1	4.093	21.998	-6.543	-8.278
2	6	Freon Return	1	1.721	18.051	-0.085	1.713
3	8	Proc. Fluid Supply	1	0.866	17.817	0.939	2.682
4	n/a	Cal. Inlet - Upper	3	3.035	0.003	-0.064	0.936
5	23	Cal. Inlet - Lower	3	3.004	0.002	-0.026	0.955
6	4	Cal. Outlet	1	0.732	17.856	1.077	2.730
7	5	Proc. Fluid Prht.	1	2.806	18.044	-1.268	1.116
8	-	Not in use			n/a		
9	9	Refrigeration Unit	[illegible]	3.038	0.002	-0.064	0.942
10	-	Not in use			n/a		
11	n/a	M.F.M. Signal			n/a		
12	n/a	P.D. Trans. Signal			n/a		

The maximum uncertainties in temperature arise from the correlation and are at most ±0.1 K in the range of temperatures used.

B. Calibration of Pressure Gauges.

In the calibration of the two Heise gauges used, any deviations greater than 0.1% of full scale were noted. In the range of pressures used in this work, there were no such deviations noted. Thus, inlet pressures could be reported with a maximum uncertainty of 20 kPa and the outlet pressures could be reported with a maximum uncertainty of 10 kPa.

C. Calibration of Flow Meters.

The mass flow meter calibration curves appear in Figures A2.1 and A2.2. The linear least squares correlation of the calibration data for each meter are as follows:

BROOKS Model No. 5810 B1F2E5A - (Flow meter A)

Flow rate (g/min) = 0.0062 + 0.2019*V

BROOKS Model No. 5810 B1J2E5A - (Flow meter B)

Flow rate (g/min) = 0.0611 + 1.6988*V

In the ranges used, the maximum uncertainties due to the correlation are 25 mg/min (0.18 mmol/min) for flow meter A, and 100 mg/min (0.73 mmol/min) for flow meter B.

The Precision Scientific wet test meter was calibrated and checked periodically. The accuracy is 0.5% or 0.0005 cu ft per 0.1 cu ft of gas passed through the meter during a test.

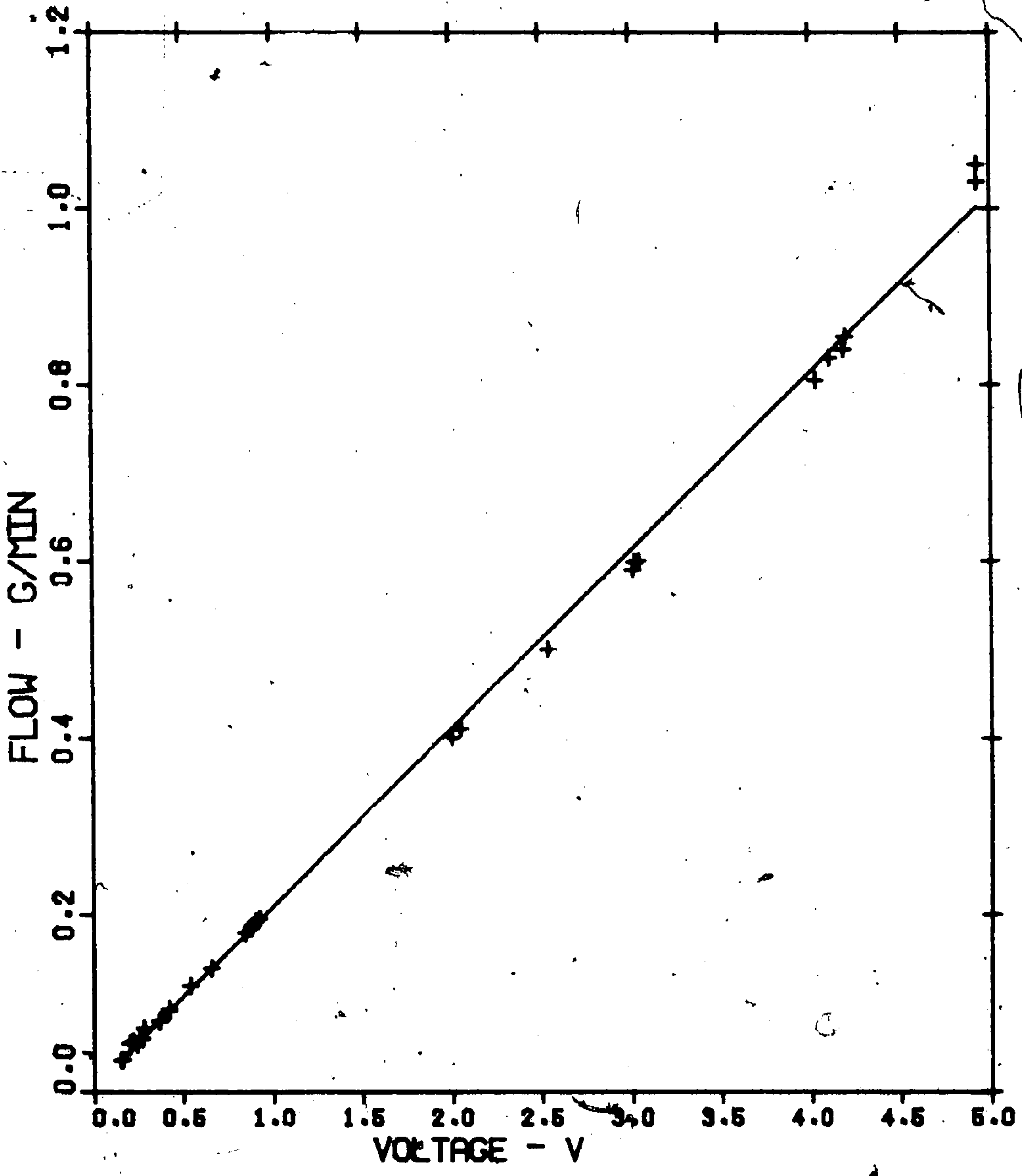


FIG A2.1 : CALIBRATION OF FLOW METER A : F-11

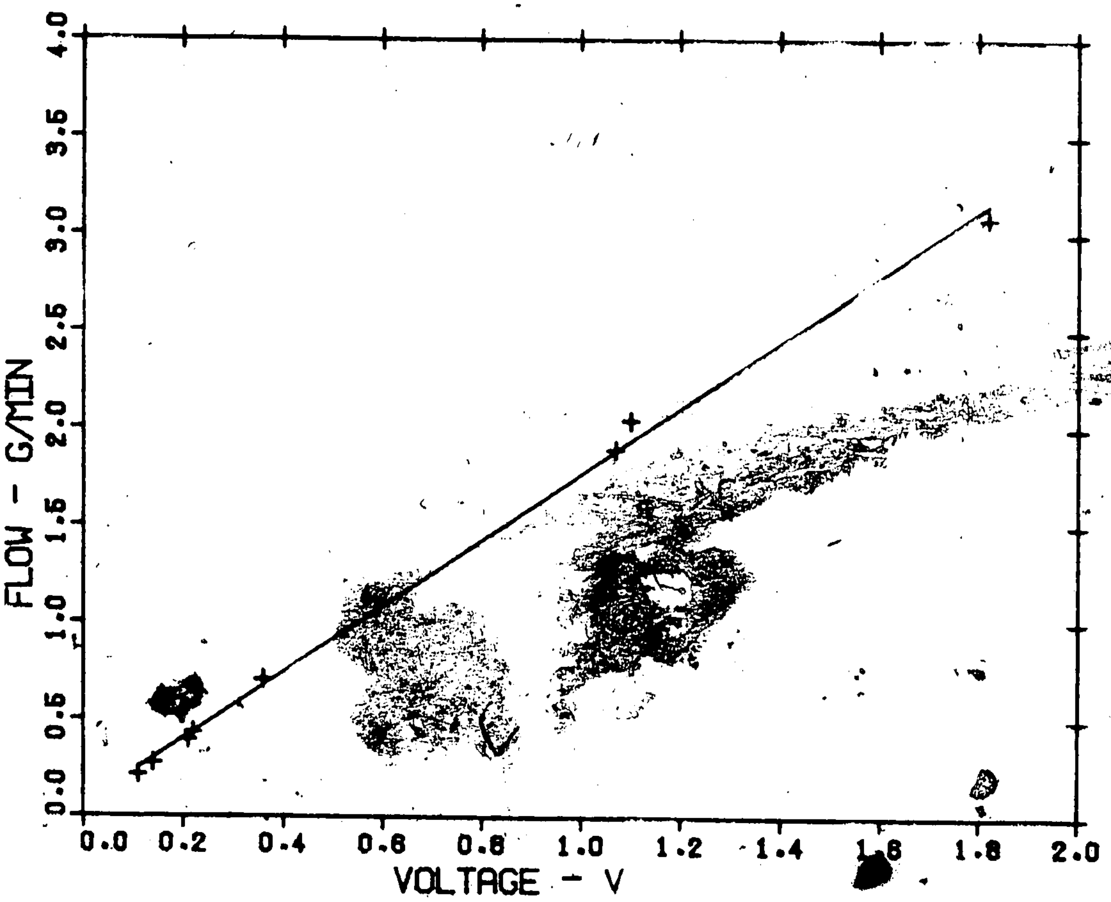


FIG A2.2 : CALIBRATION OF FLOW METER B : F-11

Appendix 3 - Calculation Routines

```
C CONTENTS OF FILE CALOR
C
C CALOR READS DATA FOR TEST GAS (IF ANY) AND FOR F-11
C AS WELL AS EXPERIMENTAL DATA FOR THE CALORIMETER.
C ENTHALPY CHANGES ARE CALCULATED FOR EACH TEST INTERVAL AND
C THE RESULTS AND EXPERIMENTAL INFO STORED IN ARRAY "DHCAL".
C MAIN PROGRAM READS DATA AND CALLS SUBROUTINE CALC TO DO
C NECESSARY CALCULATIONS AFTER WHICH MAIN WRITES RESULTS
C INTO OUTPUT FILE OR TO THE TERMINAL.
C LOGICAL UNIT NOS. ARE:    1 - N2 ENTHALPY DATA
C                           2 - CO2 ENTHALPY DATA
C                           3 - FREON DATA
C                           4 - EXPERIMENTAL DATA
C                           5 - TERMINAL
C                           6 - TERMINAL OR RESULTS FILE
C
C
      REAL HT(20),HP(40),HH(20,40),FRD(10,4)
      REAL EX(10,20),DHCAL(10,10,20)
      INTEGER GAS(3)/'N2','CO2','TEST'/,MSG(20)
      COMMON HT,HP,HH,FRD,EX,DHCAL,NPTS,NPF
      DO 13 I=1,10
      DO 13 J=1,10
      DO 13 K=1,17
   13 DHCAL(I,J,K)=0.0
      READ(3,101) NPF
      READ(3,103)((FRD(I,J),J=1,4),I=1,NPF)
      READ(4,101) KGAS
      IF (KGAS.EQ.3) GO TO 10
      READ(KGAS,101) NTH,NPH
      READ(KGAS,102) (HT(I),I=1,NTH)
      READ(KGAS,102) (HP(J),J=1,NPH)
      READ(KGAS,102) ((HH(I,J),I=1,NTH),J=1,NPH)
   10 READ(4,101) NRUNS
      DO 100 KRUN=1,NRUNS
      READ(4,101) NPTS,KTYP
      DO 15 J=1,17
      READ(4,102) (EX(I,J),I=1,NPTS)
C     WRITE(6,102) (EX(I,J),I=1,NPTS)
      DO 14 I=1,NPTS
      IF (EX(I,J).EQ.0.0.AND.I.GT.1)  EX(I,J)=EX(I-1,J)
   14 CONTINUE
   15 CONTINUE
C     READ(4,130)(MSG(J),J=1,20)
C130  FORMAT(20A4)
      CALL CALC(NTH,NPH,KRUN,KGAS,KTYP)
      NINT=NPTS-1
      IF(KTYP.EQ.0) GO TO 90
      IF(KGAS.EQ.3) GO TO 90
      WRITE(6,105) KRUN,GAS(KGAS),(J,J=1,NINT)
      WRITE(6,107)(DHCAL(KRUN,J,8),J=1,NINT)
      WRITE(6,108)(DHCAL(KRUN,J,9),J=1,NINT)
      WRITE(6,109)(DHCAL(KRUN,J,10),J=1,NINT)
      WRITE(6,110)(DHCAL(KRUN,J,11),J=1,NINT)
      WRITE(6,111)(DHCAL(KRUN,J,13),J=1,NINT)
      WRITE(6,124)(DHCAL(KRUN,J,16),J=1,NINT)
      WRITE(6,129)(DHCAL(KRUN,J,17),J=1,NINT)
      WRITE(6,131)(DHCAL(KRUN,J,12),J=1,NINT)
      WRITE(6,112)(DHCAL(KRUN,J,1),J=1,NINT)
      WRITE(6,113)(DHCAL(KRUN,J,2),J=1,NINT)
      WRITE(6,114)(DHCAL(KRUN,J,3),J=1,NINT)
      WRITE(6,115)(DHCAL(KRUN,J,6),J=1,NINT)
      WRITE(6,116)(DHCAL(KRUN,J,4),J=1,NINT)
      WRITE(6,128)
      WRITE(6,117) GAS(KGAS),(DHCAL(KRUN,J,5),J=1,NINT)
      WRITE(6,118)(DHCAL(KRUN,J,7),J=1,NINT)
      WRITE(6,119)(DHCAL(KRUN,J,15),J=1,NINT)
      WRITE(6,120)(DHCAL(KRUN,J,14),J=1,NINT)
      WRITE(6,125)(DHCAL(KRUN,J,18),J=1,NINT)
      WRITE(6,126)(DHCAL(KRUN,J,19),J=1,NINT)
      WRITE(6,127)(DHCAL(KRUN,J,20),J=1,NINT)
C     WRITE(6,132)(MSG(J),J=1,20)
      GO TO 100
   90 WRITE(6,105) KRUN,GAS(KGAS),(J,J=1,NINT)
      WRITE(6,107)(DHCAL(KRUN,J,8),J=1,NINT)
      WRITE(6,108)(DHCAL(KRUN,J,9),J=1,NINT)
      WRITE(6,109)(DHCAL(KRUN,J,10),J=1,NINT)
      WRITE(6,110)(DHCAL(KRUN,J,11),J=1,NINT)
      WRITE(6,111)(DHCAL(KRUN,J,13),J=1,NINT)
      WRITE(6,124)(DHCAL(KRUN,J,16),J=1,NINT)
      WRITE(6,133)(DHCAL(KRUN,J,2),J=1,NINT)
      WRITE(6,134)(DHCAL(KRUN,J,6),J=1,NINT)
      WRITE(6,116)(DHCAL(KRUN,J,4),J=1,NINT)
      WRITE(6,135)
      WRITE(6,117) GAS(KGAS),(DHCAL(KRUN,J,5),J=1,NINT)
      WRITE(6,118)(DHCAL(KRUN,J,7),J=1,NINT)
      WRITE(6,119)(DHCAL(KRUN,J,15),J=1,NINT)
      WRITE(6,120)(DHCAL(KRUN,J,14),J=1,NINT)
      WRITE(6,125)(DHCAL(KRUN,J,18),J=1,NINT)
      WRITE(6,126)(DHCAL(KRUN,J,19),J=1,NINT)
      WRITE(6,127)(DHCAL(KRUN,J,20),J=1,NINT)
C     WRITE(6,132)(MSG(J),J=1,20)
      GO TO 100
```

```
 90   WRITE(6,121)KRUN,(J,J=1,NINT)
      WRITE(6,122)(DHCAL(KRUN,J,1),J=1,NINT)
      WRITE(6,107)(DHCAL(KRUN,J,5),J=1,NINT)
      WRITE(6,108)(DHCAL(KRUN,J,6),J=1,NINT)
      WRITE(6,123)(DHCAL(KRUN,J,9),J=1,NINT)
      WRITE(6,119)(DHCAL(KRUN,J/8),J=1,NINT)
      WRITE(6,120)(DHCAL(KRUN,J,10),J=1,NINT)
      WRITE(6,125)(DHCAL(KRUN,J,18),J=1,NINT)
      WRITE(6,126)(DHCAL(KRUN,J,19),J=1,NINT)
      WRITE(6,127)(DHCAL(KRUN,J,20),J=1,NINT)
C     WRITE(6,132)(MSG(J),J=1,20)
C132  FORMAT(/5X,'COMMENTS: ',20A4)
 100  CONTINUE
 101  FORMAT(2I4)
 102  FORMAT(10F7.0)
 103  FORMAT(4F10.0)
 105  FORMAT(//5X,'RUN',I3,T16,'TEST GAS - ',A4,/
     &5X,'CONDITIONS FOR TEST INTERVAL',9(4X,I1,4X))
 106  FORMAT(/5X,'TEMPERATURE',6X,'Upper Inlet',9F9.2)
 107  FORMAT(/5X,'TEMPERATURE',6X,'Lower Inlet',9F9.2)
 108  FORMAT(T6,'(K)',T23,'Outlet',T34,9F9.2)
 109  FORMAT(5X,'PRESSURE',T23,'Inlet',T34,9F9.3)
 110  FORMAT(T5,'(MPa)',T23,'Outlet',T34,9F9.3)
 111  FORMAT(T23,'Atmospheric',9F9.3)
 124  FORMAT(/,5X,'HEAT EXCHANGE (J/min)',T34,9F9.3)
 112  FORMAT(5X,'ENTHALPY CHANGE  Predicted  ',9F9.2)
 113  FORMAT(T6,'(J/mol)',T23,'Measured   ',9F9.2)
 114  FORMAT(/,5X,'FLOW RATE  F-11  Pred. Mol. ',9F9.6)
 115  FORMAT(T5,'(l/min) or',T23,'Meas. Mol. ',9F9.5)
 116  FORMAT(T6,'(mmol/min)',T23,'Calc. Vol. ',9F9.5)
 117  FORMAT(T17,A4,T23,'Meas. Vol. ',9F9.5)
 118  FORMAT(T23,'Calc. Mol. ',9F9.5)
 119  FORMAT(/,5X,'Freon Sat''n Temp. (K)',T34,9F9.2)
 120  FORMAT(5X,'Freon Meter Temp. (K)',T34,9F9.2,//)
 121  FORMAT(//5X,'RUN',I3,T16,'BACKGROUND TEST (NO FLOW)'/
     &5X,'CONDITIONS FOR TEST INTERVAL',9(4X,I1,4X))
 122  FORMAT(/5X,'TEST HEAT LEAK (J/min)',T34,9F9.3)
 123  FORMAT(5X,'Atmospheric Pressure (MPa)',T34,9F9.3)
 125  FORMAT(5X,'Freon Return Temp. (K)',T34,9F9.2)
 126  FORMAT(5X,'Heat Exch. Corr. (J/min)',T34,9F9.3)
 127  FORMAT(5X,'Ambient Air Temp. (K)',T34,9F9.2)
 128  FORMAT(T32,'----------------------')
 129  FORMAT(5X,'HEAT LEAK (J/min)',T34,9F9.3)
 131  FORMAT(5X,'RATIO: LEAK OVER EXCHANGE',T34,9F9.4)
 133  FORMAT(5X,'MEAS. ENTH. CHANGE (J/mol)  ',9F9.2)
 134  FORMAT(/,5X,'FLOW RATE  F-11  Meas. Mol. ',9F9.5)
 135  FORMAT(T5,'or (l/min)',T32,'----------------------')
      STOP
      END
C
      SUBROUTINE CALC(NTH,NPH,KRUN,KGAS,KTYP)
      REAL ZN(3)
      REAL HT(20),HP(40),HH(20,40),FRD(10,4)
      DIMENSION EX(10,20),DHCAL(10,10,20)
      COMMON HT,HP,HH,FRD,EX,DHCAL,NPTS,NPF
      ZN(1)=0.9998
      ZN(2)=0.9951
      ZN(3)=0.9977
      NINT=NPTS-1
      DO 200 I=1,NINT
      DT=EX(I+1,1)-EX(I,1)
      TA=(EX(I,6)+EX(I+1,6))/2.
      CT1=3.0043+.002*TA-.0255/TA+.9548*ALOG(TA)
      T1=EXP(CT1)+273.15
      TB=(EX(I,7)+EX(I+1,7))/2.
      T2=.7321+17.8555*TB+1.0788/TB+2.7288*ALOG(TB)+273.15
      TC=(EX(I,8)+EX(I+1,8))/2.
      TS=2.8064+18.0442*TC-1.2877/TC+1.1168*ALOG(TC)+273.15
      TD=(EX(I,2)+EX(I+1,2))/2.
      TFM=4.0929+21.9982*TD-6.5430/TD-5.2786*ALOG(TD)+273.15
      TE=(EX(I,3)+EX(I+1,3))/2.
      TRLIQ=1.7214+18.0513*TE-0.0846/TE+1.7133*ALOG(TE)+273.15
      TAMB=((EX(I,17)+EX(I+1,17))/2-32.)*5/9+273.15
      FTCOR=1.
      PA=EX(I,15)*0.1/750.062*FTCOR
      JPROP=2
      CALL PROP1(PA,HVAP,NPF,FRD,JPROP)
      HVAP=HVAP+24000.0
      JPROP=3
      CALL PROP1(PA,VSP,NPF,FRD,JPROP)
      VSPF=VSP*0.137369
      VOLT=EX(I+1,11)
C     FOR LARGE MFM  B=0.0611 , M=1.8888
C     FOR SMALL MFM  B=0.0062 , M=0.2019
      RFMOL=(.0062+.2019*VOLT)/137.37
      RFVOL=RFMOL*VSPF
      JPROP=4
      CALL PROP1(PA,TSAT,NPF,FRD,JPROP)
      TSAT=TSAT+273.15
      FHTCAP=120.7
      SATCOR=RFMOL*(TSAT-TRLIQ)*FHTCAP
C     SATCOR TO BE ADDED TO DHCAL(16) TO CORRECT FOR DIFF FROM
C     SAT'N TEMP IN CALORIMETER
```

```
      IF (KTYP.EQ.0) GO TO 210
      P1=(EX(I,13)+EX(I+1,13))/2.*0.1/14.5038 +PA
      P2=(EX(I,14)+EX(I+1,14))/2./1000. +PA
      IF (KGAS.EQ.3) GO TO 198
      CALL PROP2(T1,P1,H1,NTH,NPH,HT,HP,HH)
      CALL PROP2(T2,P2,H2,NTH,NPH,HT,HP,HH)
      DHCAL(KRUN,I,1)=H2-H1
      GO TO 199
 198  DHCAL(KRUN,I,1)=-99999.
 199  TWTM=((EX(I,17)+EX(I+1,17))/2.-32.)*5./9.+273.15
      R=8.314E-6
      VSPN=ZN(KGAS)*R*TWTM/PA
      FWTM=1.
      RNVOL=(EX(I+1,12)-EX(I,12))*0.0283168*FWTM/DT
      RNMOL=RNVOL/VSPN
      DHCAL(KRUN,I,2)=-RFMOL*HVAP/RNMOL
      IF (KGAS.NE.3) DHCAL(KRUN,I,3)=RNMOL*(H1-H2)/HVAP*1000.
      DHCAL(KRUN,I,4)=RFVOL*1000.
      DHCAL(KRUN,I,5)=RNVOL*1000.
      DHCAL(KRUN,I,6)=RFMOL*1000.
      DHCAL(KRUN,I,7)=RNMOL*1000.
      DHCAL(KRUN,I,8)=T1
      DHCAL(KRUN,I,9)=T2
      DHCAL(KRUN,I,10)=P1
      DHCAL(KRUN,I,11)=P2
      DHCAL(KRUN,I,13)=PA
      DHCAL(KRUN,I,14)=TFM
      DHCAL(KRUN,I,15)=TSAT
      DHCAL(KRUN,I,16)=RFMOL*HVAP
      IF (KGAS.NE.3) DHCAL(KRUN,I,17)=RFMOL*HVAP-RNMOL*(H1-H2)
      IF (KGAS.NE.3) DHCAL(KRUN,I,12)=DHCAL(KRUN,I,17)/DHCAL(KRUN,I,16)
      DHCAL(KRUN,I,18)=TRLIQ
      DHCAL(KRUN,I,19)=SATCOR
      DHCAL(KRUN,I,20)=TAMB
      GO TO 202
 210  DHCAL(KRUN,I,1)=RFMOL*HVAP
      DHCAL(KRUN,I,2)=RFMOL*1000.
      DHCAL(KRUN,I,3)=RFVOL*1000.
      DHCAL(KRUN,I,4)=TS
      DHCAL(KRUN,I,5)=T1
      DHCAL(KRUN,I,6)=T2
      DHCAL(KRUN,I,8)=TSAT
      DHCAL(KRUN,I,9)=PA
      DHCAL(KRUN,I,10)=TFM
      DHCAL(KRUN,I,18)=TRLIQ
      DHCAL(KRUN,I,19)=SATCOR
      DHCAL(KRUN,I,20)=TAMB
 202  CONTINUE
      DO 201 K=1,20
 201  DHCAL(KRUN,10,K)=DHCAL(KRUN,10,K)+DHCAL(KRUN,I,K)*DT
 200  CONTINUE
      DO 205 K=1,20
 205  DHCAL(KRUN,10,K)=DHCAL(KRUN,10,K)/EX(NPTS,1)
      RETURN
C     DT in min., P1,P2,PA in MPa., T1,T2,TFM,TWTM,etc. in K
C     R in MJ/mol*K, VSPN,VSPF in cu.m/mol
C     RFVOL,RNVOL in cu.m/min, RFMOL,RNMOL in mol/min
C     H1,H2,HVAP in J/mol
      END
C
      SUBROUTINE PROP2(X,Y,Z,NX,NY,AX,AY,ZZ)
      REAL AX(20),AY(40),ZZ(20,40)
      DO 300 I=1,NX
      IF (AX(I).LT.X) GO TO 300
      K=I
      GO TO 301
 300  CONTINUE
 301  DO 302 J=1,NY
      IF (AY(J).LT.Y) GO TO 302
      L=J
      GO TO 303
 302  CONTINUE
 303  R=(ZZ(K,L)-ZZ(K,L-1))/(AY(L)-AY(L-1))
      A=R*(Y-AY(L-1))+ZZ(K,L-1)
      R=(ZZ(K-1,L)-ZZ(K-1,L-1))/(AY(L)-AY(L-1))
      B=R*(Y-AY(L-1))+ZZ(K-1,L-1)
      R=(A-B)/(AX(K)-AX(K-1))
      Z=R*(X-AX(K-1))+B
      RETURN
      END
C
      SUBROUTINE PROP1(X,Y,L,YY,JP)
      REAL YY(10,4)
      DO 400 I=1,L
      IF (YY(I,1).LT.X) GO TO 400
      M=I
      GO TO 401
 400  CONTINUE
 401  R=(YY(M,JP)-YY(M-1,JP))/(YY(M,1)-YY(M-1,1))
      Y=R*(X-YY(M,1))+YY(M,JP)
      RETURN
```

```
C
C      THESE SUBROUTINES ARE USED IN PLACE OF SUBROUTINE PROP2
C      IN THE PROGRAM "CALOR" IF SPLINE INTERPOLATION OF THE
C      ENTHALPY DATA IS DESIRED
C
       SUBROUTINE PROP2(X,Y,Z,NX,NY,AX,AY,AZ)
       REAL AX(20),AY(40),AZ(20,40),B(4),C(4),D(4),XX(4),YY(4)
       REAL ZZ(4),S(4)
       INTEGER INDX(4),INDY(4)
       DO 300 I=1,NX
       IF (AX(I).LT.X) GO TO 300
       K=I
       GO TO 301
 300   CONTINUE
 301   DO 302 J=1,NY
       IF (AY(J).LT.Y) GO TO 302
       L=J
       GO TO 303
 302   CONTINUE
 303   IF (K.LT.3) GO TO 305
       IF (K.EQ.NX) GO TO 307
       DO 304 I=1,4
 304   INDX(I)=K-3+I
       GO TO 310
 305   DO 306 I=1,4
 306   INDX(I)=I
       GO TO 310
 307   DO 308 I=1,4
 308   INDX(I)=K-4+I
C
 310   IF (L.LT.3) GO TO 315
       IF (L.EQ.NY) GO TO 317
       DO 314 J=1,4
 314   INDY(J)=L-3+J
       GO TO 320
 315   DO 316 J=1,4
 316   INDY(J)=J
       GO TO 320
 317   DO 318 J=1,4
 318   INDY(J)=L-4+J
C
 320   DO 340 J=1,4
       DO 330 I=1,4
       XX(I)=AX(INDX(I))
       ZZ(I)=AZ(INDX(I),INDY(J))
C      CORRECTION FOR TWO VALUES IN CO2 TABLES IN LIQ. PHASE
C      IF (INDY(J).EQ.7.AND.INDX(I).EQ.1) XX(I)=291.55
C      IF (INDY(J).EQ.7.AND.INDX(I).EQ.1) ZZ(I)=31448.
C      IF (INDY(J).EQ.8.AND.INDX(I).EQ.1) XX(I)=288.26
C      IF (INDY(J).EQ.8.AND.INDX(I).EQ.1) ZZ(I)=31088.
 330   CONTINUE
       N=4
       CALL SPLINE(N,XX,ZZ,B,C,D)
       S(J)=SEVAL(N,X,XX,ZZ,B,C,D)
 340   YY(J)=AY(INDY(J))
       CALL SPLINE(N,YY,S,B,C,D)
       Z=SEVAL(N,Y,YY,S,B,C,D)
       RETURN
       END
C
C       SPLINE INTERPOLATION FROM FORSYTHE,MALCOLM&MOLER
C       MASLIYAH.... CHE. 574  JAN. 1978
       SUBROUTINE SPLINE (N,X,Y,B,C,D)
       REAL X(N),Y(N),T,B(N),C(N),D(N)
       INTEGER  IB, I
       NM1=N-1
       IF ( N .LT. 2 ) RETURN
       IF ( N .LT. 3 ) GO TO 550
       D(1) = X(2) - X(1)
       C(2) = (Y(2) -Y(1))/D(1)
       DO 510 I = 2, NM1
          D(I) = X(I+1) - X(I)
          B(I) = 2.*(D(I-1) + D(I))
          C(I+1) = (Y(I+1) -Y(I))/D(I)
          C(I) = C(I+1) - C(I)
 510   CONTINUE
       B(1) = -D(1)
       B(N) = -D(N-1)
       C(1) = 0.
       C(N)=0.
       IF ( N .EQ. 3 ) GO TO 515
       C(1) = C(3)/(X(4)-X(2)) - C(2)/(X(3)-X(1))
       C(N) = C(N-1)/(X(N)-X(N-2)) - C(N-2)/(X(N-1)-X(N-3))
       C(1) = C(1)*D(1)**2/(X(4)-X(1))
       C(N) = -C(N)*D(N-1)**2/(X(N)-X(N-3))
 515   DO 520 I = 2, N
         T = D(I-1)/B(I-1)
         B(I) = B(I) - T*D(I-1)
         C(I) = C(I) - T*C(I-1)
 520   CONTINUE
```

```
      C(N) = C(N)/B(N)
      DO 530 IB = 1, NM1
        I = N-IB
        C(I) = (C(I) - D(I)*C(I+1))/B(I)
 530  CONTINUE
      B(N) = (Y(N) -Y(NM1))/D(NM1) + D(NM1)*(C(NM1) + 2.*C(N))
      DO 540 I = 1, NM1
         B(I) = (Y(I+1) -Y(I))/D(I) - D(I)*(C(I+1) + 2.*C(I))
         D(I) = (C(I+1) - C(I))/D(I)
         C(I) = 3.*C(I)
 540  CONTINUE
      C(N) = 3.*C(N)
      D(N) = D(N-1)
      RETURN
 550  B(1) = (Y(2)-Y(1))/(X(2)-X(1))
      C(1) = 0.
      D(1) = 0.
      B(2) = B(1)
      C(2) = 0.
      D(2) = 0.
      RETURN
      END
C
C
      FUNCTION SEVAL(N,U,X,Y,B,C,D)
      REAL U,X(N),Y(N),B(N),C(N),D(N)
      DOUBLE PRECISION DX
      DATA I/1/
      IF ( I .GE. N ) I = 1
      IF ( U .LT. X(I) ) GO TO 410
      IF ( U .LE. X(I+1) ) GO TO 430
 410  I = 1
      J = N+1
 420  K = (I+J)/2
      IF ( U .LT. X (K) ) J = K
      IF ( U .GE. X (K) ) I = K
      IF ( J .GT. I + 1 ) GO TO 420
 430  DX=U-X(I)
      SEVAL = Y(I)+DX*(B(I)+DX*(C(I)+DX*D(I)))
      DERIV=B(I)+DX*(2.0*C(I)+DX*(3.0*D(I)))
      RETURN
      END
```

Appendix 4 - Fluid Properties

Table A4.1 contains the relevant properties of Freon-11, in the range of conditions occurring in the experiments. Values of enthalpy as a function of temperature and pressure for nitrogen and carbon dioxide (used as standards) are given in Tables A4.2 and A4.3.

Enthalpy values for the test gas were estimated using the Equi-Phase program which employs the Peng-Robinson equation of state.

```
TABLE A4.1 : FREON-11 DATA CONVERTED FROM ASHRAE FUNDAMENTALS 1977

STORED IN SIMILAR FORM IN FILE DFREON

P IN MPA, HVAP (ENTHALPY OF VAP'N) IN J/MOL
T IN C, V (SP. VOL. VAPOUR) IN CU.M./KG

    P          HVAP          V         T (SAT)

0.088384    24943.326    0.19368     20.0

0.090183    24915.440    0.19006     20.556

0.092010    24887.416    0.18655     21.111

0.093865    24859.256    0.18311     21.667

0.095747    24830.820    0.17974     22.222

0.097684    24802.072    0.17646     22.778

0.099606    24775.048    0.17324     23.333

0.10158     24746.613    0.17010     23.889

0.10358     24718.178    0.16703     24.444

0.10562     24689.742    0.16403     25.0
```

TABLE A4.2 : NITROGEN ENTHALPY DATA FROM I.U P.A.C.
(ANGUS ET AL., 1979) STORED IN FILE N2DATA
ENTHALPIES GIVEN IN J/MOL

TEMPERATURE IN K

PRESSURE IN MPA	250	300	350	400	450	500	550	600	650	700
1.00	-1492	-8	1468	2942	4418	5899	7391	8894	10411	11944
1.50	-1537	-38	1447	2928	4409	5894	7388	8894	10414	11948
2.00	-1582	-68	1427	2914	4400	5889	7386	8894	10416	11952
2.50	-1626	-98	1406	2900	4391	5884	7384	8895	10418	11956
3.00	-1670	-128	1386	2887	4382	5879	7382	8895	10421	11960
3.50	-1713	-157	1367	2873	4374	5874	7381	8896	10424	11965
4.00	-1756	-185	1347	2860	4366	5870	7379	8897	10426	11969
4.50	-1799	-213	1328	2846	4358	5866	7378	8898	10429	11974
5.00	-1841	-241	1309	2835	4350	5861	7376	8899	10432	11978
5.50	-1883	-269	1291	2823	4342	5857	7375	8900	10435	11983
6.00	-1924	-296	1273	2811	4335	5854	7374	8901	10438	11987
6.50	-1965	-322	1255	2799	4328	5850	7373	8902	10441	11992
7.00	-2005	-348	1238	2788	4321	5846	7372	8904	10444	11997
7.50	-2044	-374	1220	2777	4314	5843	7372	8905	10448	12002
8.00	-2083	-399	1204	2766	4307	5840	7371	8907	10451	12007
8.50	-2121	-424	1187	2755	4301	5837	7371	8908	10455	12012
9.00	-2158	-448	1171	2745	4295	5834	7370	8911	10458	12017
9.50	-2195	-471	1155	2735	4289	5831	7370	8912	10462	12022
10.00	-2231	-495	1140	2725	4283	5828	7370	8915	10466	12027
15.00	-2545	-700	1006	2640	4236	5811	7377	8941	10508	12083
20.00	-2774	-855	905	2581	4206	5808	7395	8975	10558	12146

TABLE A4.3 : CARBON DIOXIDE ENTHALPY DATA FROM I.U.P.A.C.
(ANGUS ET AL., 1973)   STORED IN FILE CO2DATA
VAPOUR ENTHALPIES GIVEN IN J/MOL

PRESSURE IN MPA

TEMPERATURE IN K	2.5	3.0	3.5	4.0	4.5	5.0	5.5	6.0	8.5	9.0	9.5	10.0	15.0
290	34080	33775	33437	33084	32602	32030	31258	30207	23734	23697	23663	23632	23423
300	34561	34295	34008	33698	33350	32955	32469	31900	25089	24975	24886	24828	24426
310	35027	34791	34540	34274	33987	33678	33330	32942	27908	26992	26620	26388	25510
320	35484	35270	35047	34813	34568	34304	34023	33721	31898	30930	30128	29264	26718
330	35934	35740	35538	35329	35110	34882	34643	34390	32848	32458	32032	31568	28090
340	36381	36202	36018	35828	35632	35429	35218	35000	33748	33458	33182	32830	29804
350	36825	36660	36490	36316	36135	35955	35767	35573	34504	34267	34028	33789	31058
360	37268	37114	36957	36797	36633	36466	36295	36120	35181	34978	34772	34560	32298
370	37710	37566	37420	37272	37121	36967	36810	36651	35809	35632	35451	35267	33334
380	38152	38018	37881	37743	37603	37460	37316	37169	36405	36246	36085	35922	34232
390	38595	38469	38341	38211	38080	37946	37814	37679	36978	36834	36686	36541	35036
400	39039	38920	38800	38678	38556	38432	38307	38181	37535	37402	37268	37135	35777
410	39484	39371	39258	39144	39029	38913	38796	38678	38078	37958	37833	37710	36471
420	39930	39824	39717	39609	39501	39392	39282	39172	38612	38498	38385	38271	37131
430	40378	40277	40176	40074	39972	39869	39766	39662	39138	39032	38926	38820	37765
440	40827	40732	40636	40539	40443	40346	40248	40151	39658	39559	39460	39361	38379
450	41279	41188	41097	41006	40914	40822	40730	40637	40174	40081	39988	39895	38976
460	41732	41645	41559	41472	41385	41298	41211	41123	40686	40598	40511	40423	39583
470	42186	42104	42022	41939	41857	41774	41692	41609	41196	41112	41030	40947	40138
480	42643	42565	42487	42408	42330	42251	42172	42094	41702	41624	41546	41468	40705

** NOTE: THE VALUES OF H AT 290 K AND 5.5 MPA AND AT 290 K AND 6.0 MPA
** HAVE BEEN "BACK-EXTRAPOLATED" FROM THE APPROPRIATE VALUES AT
** THE SATURATION TEMPERATURES (290 < T(SAT) < 300 K AT THESE PRESSURES)

Appendix 5 - Sample Experimental Data Files

```
1.                                  C KGAS = 1 FOR N2, 2 FOR CO2, 3 FOR OTHER GASES
3.                                  C KRUNS = NO. OF EXPT'L RUNS IN DATA FILE
7,1,                                C NPTS = NO. OF DATA PTS IN RUN; KTYP = 1 FOR NORMAL CALC'N
   0.00   10.00   20.00   30.00   40.00   50.00   51.00    C TIME OF READINGS
  1.201           1.204           1.206   1.208
  1.105                           1.108           1.104
  1.177           1.179   1.180   1.182   1.183            C EXPERIMENTAL DATA : T/C, PRESSURE, FLOWMETER,
                                                           C AND OTHER READINGS GIVEN HERE
  5.556   5.575   5.589   5.524   5.552   5.548   5.550
  1.387   1.388   1.388   1.385   1.383   1.384   1.385    C BLANK ENTRIES AT ANY TIME INDICATE THAT
 12.467  12.454  12.410  12.438  12.453  12.470  12.448    C READING IS UNCHANGED FROM PREVIOUS VALUE

   .083                                                    C THE TWO BLANK LINES IN EACH RUN DATA SET
   1.99    1.98    1.98    1.98    1.97                    C ARE AVAILABLE FOR STORAGE OF OTHER
  2.535   2.763  2.9895   3.218   3.438   3.684   3.811    C EXPERIMENTAL DATA, IF REQUIRED
  1003.   1002.   1003.                           1002.
  8805.                   8810.
  702.2
    23.
   72.5
6,1,
   0.00   10.00   20.00   30.00   40.00   50.00
  1.217           1.214                   1.210
  1.103                           1.107  1.1022
  1.181                           1.180

  7.083   7.080   7.104   7.111   7.104   7.147
  1.450                   1.451   1.450   1.453
 12.701  12.704  12.721  12.727  12.736  12.768

   .084
   2.46    2.47    2.46    2.46    2.46    2.46
  5.875   6.145   6.413  6.6825  6.9515   7.222
  1000.
  8805.   8810.   8805.           8810.   8800.
  702.7
   23.5
    73.
5,1,
   0.00   10.00   21.00   30.00   40.00
  1.211   1.215   1.219           1.220
  1.106
  1.181   1.182   1.183   1.184   1.185

  5.486   5.470   5.518   5.519   5.502
  1.282           1.296
 12.045  12.002  12.038  12.006  12.000

   .073
   1.27                   1.31
  3.031  3.1835  3.3575  3.4995   3.657
  1397.           1395.   1397.   1398.
  8520.   8525.   8510.
  702.4
   23.5
    73.
CCCCC N2 DATA JULY 21,23 T/C HTR (300 MA)
CCCCC DATA FILE N2.2
```

```
2,
3,                                  C SEE EXPLANATORY COMMENTS ON PREVIOUS PAGE
2,0,                                C KTYP = 0 HERE FOR "BACKGROUND" HEAT LEAK CALC'N
   0.00  10.00
  1.176  1.180
  1.088
  1.163  1.168

  1.238  1.240
  1.198  1.201
  9.850  9.852

   .230   .128
   .148   .148


  688.1
   22.
   72.
5,1,
   0.00  10.00  25.00  35.00  45.00
  1.180                1.187  1.189
  1.083
  1.160

  6.718  6.704  6.717  6.709  6.714
  1.383  1.382         1.381
 12.707 12.657 12.638 12.604 12.588

   .080
  2.12   2.05   2.07   2.08   2.06
  5.485  5.647  5.480  6.053  6.215
   494.   495.   484.
  3030.  3035.  3035.  3025.  3025.
  687.3
   22.
   72.5
5,1,
   0.00  12.00  20.00  40.00  50.00
  1.178         1.182
  1.077                1.076  1.074
  1.157

  6.106  6.085  6.067  5.930  5.861
  1.317  1.315  1.313  1.302  1.297
 12.800 12.812 12.800 12.716 12.748

   .058
  1.65   1.64   1.62   1.52   1.50
  7.120  7.276  7.380  7.532  7.753
   500.          501.          502.
  3210.         3220.  3230.  3250.
  687.7
   23.
   73.
CCCCC CO2 DATA JUNE 30
CCCCC DATA FILE CO2.1
```

```
3,
6,                        C SEE COMMENTS ON PREVIOUS PAGES
7,1,
    0.00   11.00   20.00   30.00   40.00   50.00   60.00
   1.182   1.182   1.183           1.188
   1.083                           1.081
   1.113   1.114                   1.112

   7.787   7.773   7.787   7.838   7.879   7.889   8.000
   1.515   1.518   1.522   1.528   1.544   1.553   1.558
  13.163  13.188  13,176  13.188  13.229  13.230  13.264

    .069    .070    .070    .071    .072    .073    .074
    .40     .39     .40     .41     .42     .44     .44
   1.036   1.308  1.5285   1.776   2.037   2.298  2.5815
    985.           1000.   1004.   1002.   1004.   1006.
   6650.           6655.   6690.   6645.   6665.   6680.
   701.0           700.9           700.8
    21.5
    71.5
5,1,
    0.00   10.00   20.00   30.00   40.00
   1.184   1.191
   1.083
   1.112

   8.501   8.551   8.572   8.583   8.616
   1.667   1.664   1.667   1.671   1.674
  13.443  13.376  13.358  13,446  13.536

    .077    .078    .079            .081
    .53     .53     .54     .53     .54
   3.825   4.137   4.482   4.787   5.083
   1000.
   6620.           6615.           6610.
   700.8
     21.
    71.5
4,1,
    0.00   10.00   30.00   40.00
   1.185           1.178   1.180
   1.082                   1.083
   1.110           1.117   1.115

   8.243   8.247   8.205   8.200
   1.612   1.611   1.606
  13.283  13.264  13.318  13.313

    .084    .101    .083    .084
    .48     .47     .47     .475
   6.550   6.835   7.405   7.687
   1006.   1005.   1006.   1006.
   6625.   6620.   6626.
   701.0
     22.
    71.5
6,.1,
    0.00   10.00   20.00   30.00   40.00
   1.202                   1.214
   1.091                   1.083
   1.153           1.155   1.152

   7.679   7.702   7.740   7.783   7.743
   1.521   1.525   1.527   1.531   1.531
  13.180  13.188  13.156  13.078  13.077

    .058    .062            .060    .059
    .40             .41             .42
   9.735  9.9865  10.238  10.482  10.745
   1002.                   1001.
   6670.                   6665.   6670.
   700.7
     22.
     72.
8,1,
    0.00   11.00   20.00   30.00   40.00
   1.215   1.223   1.221           1.220
   1.081                           1.080
   1.159   1.160                   1.163

   7.172   7.176   7.180   7.188   7.188
   1.450           1.451
  12.868  12.840  12:881  12.848  12.861

    .081    .080
    .33     .34     .345    .345    .34
   2.415   2.649   2.840  3.0525   3.285
   1007.
   6760.
   700.4
     22.
    72.3
CCCCC  MIX DATA AUG 30,31 (USING LMFM)
CCCCC  DATA FILE MIX.2L
```

Appendix 6 - Experimental Results

The following pages contain all of the results of the later experimental work. The results of earlier experimental runs have not been included because changes in equipment configuration and operation have caused at least some improvement in the later performance of the calorimeter over that of earlier work.

The results as shown are in a form similar to the actual output of the program CALOR. Minor additional notes are given for explanatory purposes.

```
DATA FROM FILE N2.1

INTERVALS MARKED WITH ** WERE MOST STABLE IN THE RUN

RUN  1    TEST GAS - N2                                         **          **                                **
CONDITIONS FOR TEST INTERVAL          1           2           3           4           5           6           7

TEMPERATURE          Lower Inlet    370.98      369.57      369.21      369.07      368.38      367.89      368.18
  (K)                Outlet         298.50      298.41      298.35      298.31      298.28      298.28      298.24
PRESSURE             Inlet            9.701       9.701       9.694       9.691       9.691       9.691       9.691
  (MPa)              Outlet           9.611       9.614       9.608       9.606       9.611       9.611       9.606
                     Atmospheric      0.094       0.094       0.094       0.094       0.094       0.094       0.094

HEAT EXCHANGE (J/min)                39.128      38.397      37.686      37.300      36.204      36.204      36.473
HEAT LEAK (J/min)                     5.052       5.373       4.821       4.795       4.717       4.885       4.483
RATIO: LEAK OVER EXCHANGE             0.1291      0.1399      0.1280      0.1285      0.1303      0.1348      0.1229
ENTHALPY CHANGE      Predicted    -2341.28    -2303.12    -2290.62    -2287.30    -2286.01    -2281.94    -2281.03
  (J/mol)            Measured     -2888.38    -2877.62    -2826.88    -2824.70    -2805.45    -2803.21    -2800.85

FLOW RATE  F-11      Pred. Mol.     1.37048     1.32818     1.32088     1.30734     1.28840     1.28861     1.25000
  (l/min) or         Meas. Mol.     1.57368     1.54428     1.51489     1.50019     1.45610     1.45610     1.42670
  (mmol/min)         Calc. Vol.     0.03870     0.03886     0.03822     0.03788     0.03874     0.03874     0.03800
                                 ----------------------
             N2      Meas. Vol.     0.38227     0.37881     0.37881     0.37327     0.36497     0.36620     0.36104
                     Calc. Mol.    14.55428    14.33878    14.33878    14.21128    13.89884    13.90746    13.74584

Freon Sat'n Temp. (K)               294.73      294.73      294.73      294.73      294.73      294.73      294.73
Freon Meter Temp. (K)               296.83      296.86      296.92      296.99      297.08      297.12      297.15
Freon Return Temp. (K)              294.74      294.72      294.72      294.73      294.74      294.74      294.74
Heat Exch. Corr. (J/min)             -0.002       0.002       0.002       0.000      -0.001      -0.001      -0.001
Ambient Air Temp. (K)               295.85      295.85      295.85      295.85      295.85      295.85      295.85


RUN  2    TEST GAS - N2                                         **          **                      **
CONDITIONS FOR TEST INTERVAL          1           2           3           4           5           6

TEMPERATURE          Lower Inlet    401.21      400.00      399.93      399.83      400.68      401.38
  (K)                Outlet         301.79      301.74      301.72      301.74      301.78      301.80
PRESSURE             Inlet            9.738       9.746       9.745       9.738       9.739       9.738
  (MPa)              Outlet           9.499       9.508       9.506       9.489       9.485       9.494
                     Atmospheric      0.094       0.094       0.094       0.094       0.094       0.094

HEAT EXCHANGE (J/min)                78.229      77.132      76.980      77.133      77.894      78.595
HEAT LEAK (J/min)                   -10.785     -11.698     -11.024     -10.690     -10.686     -11.535
RATIO: LEAK OVER EXCHANGE            -0.1379     -0.1517     -0.1433     -0.1386     -0.1372     -0.1468
ENTHALPY CHANGE      Predicted    -3181.87    -3144.77    -3114.82    -3138.96    -3184.88    -3184.72
  (J/mol)            Measured     -2798.09    -2730.65    -2724.19    -2756.91    -2783.03    -2777.12

FLOW RATE  F-11      Pred. Mol.     3.58019     3.57288     3.53833     3.53216     3.56130     3.52486
  (l/min) or         Meas. Mol.     3.14831     3.10222     3.09487     3.10222     3.13161     3.18101
  (mmol/min)         Calc. Vol.     0.07938     0.07827     0.07806     0.07827     0.07901     0.07975
                                 ----------------------
             N2      Meas. Vol.     0.73824     0.74332     0.74332     0.73824     0.73824     0.74473
                     Calc. Mol.    27.97809    28.24708    28.24708    27.97809    27.97809    28.30083

Freon Sat'n Temp. (K)               294.73      294.73      294.73      294.73      294.73      294.73
Freon Meter Temp. (K)               297.48      297.60      297.[illegible]      297.82      297.78      297.72
Freon Return Temp. (K)              294.74      294.75      294.75      294.75      294.75      294.75
Heat Exch. Corr. (J/min)             -0.003      -0.007      -0.010      -0.010      -0.011      -0.011
Ambient Air Temp. (K)               296.21      296.21      296.21      296.21      296.21      296.21


RUN  3    TEST GAS - N2                                                     **          **
CONDITIONS FOR TEST INTERVAL          1           2           3           4           5           6

TEMPERATURE          Lower Inlet    411.14      411.74      412.02      412.25      412.38      412.61
  (K)                Outlet         302.81      302.88      302.86      302.86      302.87      302.86
PRESSURE             Inlet            9.743       9.743       9.753       9.760       9.760       9.760
  (MPa)              Outlet           9.384       9.379       9.384       9.389       9.384       9.384
                     Atmospheric      0.094       0.094       0.094       0.094       0.094       0.094

HEAT EXCHANGE (J/min)               104.904     106.732     106.732     107.482     106.732     107.087
HEAT LEAK (J/min)                   -14.447     -13.326     -13.155     -12.681     -12.633     -13.448
RATIO: LEAK OVER EXCHANGE            -0.1377     -0.1249     -0.1233     -0.1180     -0.1184     -0.1256
ENTHALPY CHANGE      Predicted    -3457.49    -3467.15    -3473.28    -3460.43    -3465.03    -3462.11
  (J/mol)            Measured     -3033.88    -3062.31    -3061.81    -3113.08    -3116.19    -3102.90

FLOW RATE  F-11      Pred. Mol.     4.80028     4.82868     4.82219     4.83213     4.80082     4.84834
  (l/min) or         Meas. Mol.     4.21923     4.29272     4.29272     4.32211     4.29272     4.30742
  (mmol/min)         Calc. Vol.     0.10842     0.10828     0.10828     0.10802     0.10828     0.10865
                                 ----------------------
             N2      Meas. Vol.     0.90897     0.91160     0.90897     0.90897     0.90169     0.90897
                     Calc. Mol.    34.51981    34.62708    34.51981    34.51981    34.25070    34.51981

Freon Sat'n Temp. (K)               294.74      294.74      294.74      294.74      294.74      294.74
Freon Meter Temp. (K)               297.88      297.83      297.81      297.82      297.82      297.77
Freon Return Temp. (K)              294.73      294.74      294.74      294.74      294.74      294.74
Heat Exch. Corr. (J/min)              0.005       0.0         0.0         0.0         0.0         0.0
Ambient Air Temp. (K)               296.48      296.48      296.48      296.48      296.48      296.48
```

```
RUN  4 .  TEST GAS - N2                                         **        **
CONDITIONS FOR TEST INTERVAL         1         2         3         4         5         6

TEMPERATURE       Lower Inlet     380.78    379.88    379.29    379.47    379.46    379.48
  (K)             Outlet          298.89    298.83    298.86    298.83    298.80    298.78
PRESSURE          Inlet            5.981     5.981     5.978     5.974     5.974     5.974
  (MPa)           Outlet           5.781     5.783     5.783     5.783     5.783     5.783
                  Atmospheric      0.093     0.093     0 093     0.093     0.093     0.093

HEAT EXCHANGE (J/min)             54.481    52.653    51.192    51.192    51.192    51.987
HEAT LEAK (J/min)                  7.205     6.686     5.783     5.927     6.185     6.539
RATIO: LEAK OVER EXCHANGE         0.1322    0.1270    0.1130    0.1158    0.1208    0.1257
ENTHALPY CHANGE   Predicted     -2563 06  -2536.28  -2520.48  -2527.52  -2528.19  -2529.39
  (J/mol)         Measured      -2953.68  -2906.31  -2841.45  -2856.47  -2876.69  -2888.21

FLOW RATE  F-11   Pred. Mol.     1.90122   1.84853   1.82613   1.82034   1.80982   1.81079
  (l/min) or      Meas. Mol.     2.19087   2.11748   2.05868   2.05868   2.05868   2.07339
  (mmol/min)      Calc. Vol.     0.05536   0.05350   0.05202   0.05202   0.05202   0.05239
                             --------------------
            N2    Meas. Vol.     0.48705   0.47855   0.47572   0.47289   0.47006   0.47006
                  Calc. Mol.    18.44802  18.12318  18.01598  17.90872  17.80145  17.80154

Freon Sat'n Temp. (K)             294.68    294.68    294.68    294.68    294.68    294.68
Freon Meter Temp  (K)             297.39    297.38    297.45    297 55    297.53    297.49
Freon Return Temp. (K)            294.80    294.80    294.80    294.80    294.80    294.80
Heat Exch. Corr. (J/min)           0.023     0.022     0.021     0.021     0.021     0.021
Ambient Air Temp. (K)             296 78    296.78    *** 78    296.79    296.78    296 78



RUN  5    TEST GAS - N2                                                   **
CONDITIONS FOR TEST INTERVAL         1         2         3         4         5

TEMPERATURE       Lower Inlet     390.90    390.47    390.39    390.18    389.97
  (K)             Outlet          300.59    300.57    300.56    300.54    300.51
PRESSURE          Inlet            5.985     5.982     5.978     5.975     5.975
  (MPa)           Outlet           5.594     5.594     5.594     5.594     5.594
                  Atmospheric      0.094     0.094     0.094     0.094     0.094

HEAT EXCHANGE (J/min)             55.200    55.200    55.200    55.200    54.835
HEAT LEAK (J/min)                -10.839    -9.630   -10.495    -8.848    -7.723
RATIO: LEAK OVER EXCHANGE        -0.1964   -0.1744   -0.1901   -0.1603   -0.1408
ENTHALPY CHANGE   Predicted     -2819.86  -2807.02  -2806.15  -2799.38  -2793.51
  (J/mol)         Measured      -2357.03  -2390.08  -2357.03  -2412.62  -2446.63

FLOW RATE  F-11   Pred. Mol.     2.85635   2.80770   2.64250   2.57628   2.51633
  (l/min) or      Meas. Mol.     2.22037   2.22037   2.22037   2.22037   2.20557
  (mmol/min)      Calc. Vol.     0.05591   0.05591   0.05591   0.05591   0 05554
                             ---------------------
            N2    Meas. Vol.     0.61447   0.60588   0.61447   0.60032   0.58757
                  Calc. Mol.    23.41945  23.09187  23.41945  22.87984  22.39416

Freon Sat'n Temp  (K)             294.78    294.78    294.78    294.78    294.78
Freon Meter Temp. (K)             296.93    297.00    296.99    296.99    296.99
Freon Return Temp. (K)            294.81    294.81    294.81    294.81    294.81
Heat Exch. Corr. (J/min)          -0.008    -0.008    -0.008    -0.008    -0.008
Ambient Air Temp. (K)             295 93    295 93    295.93    295 93    295.93



RUN  6    TEST GAS - N2                        **
CONDITIONS FOR TEST INTERVAL         1         2         3         4

TEMPERATURE       Lower Inlet     403.25    403.26    403.86    404.06
  (K)             Outlet          302.33    302.35    302.36    302.37
PRESSURE          Inlet            5.988     5.988     5.985     5.985
  (MPa)           Outlet           5.594     5.594     5.589     5.594
                  Atmospheric      0.094     0.094     0.094     0.094

HEAT EXCHANGE (J/min)             70.915    70.915    71.646    71.280
HEAT LEAK (J/min)                -21.497   -20.827   -21.371   -21.160
RATIO: LEAK OVER EXCHANGE        -0.3031   -0.2937   -0.2983   -0.2969
ENTHALPY CHANGE   Predicted     -3141.24  -3141.47  -3161.63  -3165.41
  (J/mol)         Measured      -2410.53  -2428.31  -2435.37  -2440.83

FLOW RATE  F-11   Pred. Mol.     3.71701   3.68007   3.74137   3.71615
  (l/min) or      Meas. Mol.     2.85236   2.85236   2.88176   2.86706
  (mmol/min)      Calc. Vol.     0.07187   0.07187   0.07261   0.07224
                             ---------------------
            N2    Meas. Vol.     0.77305   0.76739   0.77305   0.76738
                  Calc. Mol     29.41876  29.20337  29.41876  29.20326

Freon Sat'n Temp. (K)             294.77    294.77    294.77    294.77
Freon Meter Temp. (K)             297.05    297.05    297.07    297.11
Freon Return Temp. (K)            294.79    294.79    294.79    294.79
Heat Exch. Corr. (J/min)          -0.008    -0.008    -0.008    -0.008
Ambient Air Temp. (K)             296.21    296.21    296.21    296.21
```

```
RUN  7    TEST GAS - N2                                                    **          **
CONDITIONS FOR TEST INTERVAL      1          2          3          4          5          6

TEMPERATURE       Lower Inlet   381.30     381.28     381.13     381.07     381.06     381.12
  (K)             Outlet        297.73     297.76     297.77     297.77     297.77     297.77
PRESSURE          Inlet          3.520      3.517      3.520      3.524      3.520      3.520
  (MPa)           Outlet         3.309      3.308      3.309      3.311      3.308      3.309
                  Atmospheric    0.094      0.094      0.094      0.094      0.094      0.094

HEAT EXCHANGE (J/min)           31.085     31.086     28.528     27.797     27.797     27.797
HEAT LEAK (J/min)                8.872      9.102      6.457      5.630      5.853      5.867
RATIO: LEAK OVER EXCHANGE       0.2854     0.2928     0.2263     0.2025     0.2108     0.2111
ENTHALPY CHANGE   Predicted   -1922.93   -1920.90   -1916.49   -1914.65   -1914.55   -1915.21
  (J/mol)         Measured    -2890.90   -2716.28   -2477.18   -2601.21   -2426.38   -2428.91

FLOW RATE  F-11   Pred. Mol.   0.88348    0.88421    0.88773    0.88158    0.88221    0.88205
  (l/min) or      Meas. Mol.   1.25033    1.25033    1.14745    1.11805    1.11805    1.11805
  (mmol/min)      Calc  Vol.   0.03150    0.03150    0.02881    0.02817    0.02817    0.02817
                             ----------------------
           N2     Meas. Vol.   0.30298    0.30016    0.30205    0.30382    0.30047    0.30016
                  Calc. Mol.  11.55204   11.44414   11.51614   11.57615   11.45814   11.44414

Freon Sat'n Temp. (K)           294.77     294.77     294.77     294.77     294.77     294.77
Freon Meter Temp. (K)           298.64     298.68     298.70     298.72     298.72     298.75
Freon Return Temp. (K)          294.77     294.77     294.77     294.77     294.77     294.77
Heat Exch. Corr. (J/min)        -0.001     -0.001     -0.001     -0.001     -0.001     -0.001
Ambient Air Temp. (K)           295.65     295.65     295.65     295.55     295.65     295.65
```

```
RUN  8    TEST GAS - N2                          **         **         **
CONDITIONS FOR TEST INTERVAL      1          2          3          4

TEMPERATURE       Lower Inlet   371.42     371.95     371.93     371.81
  (K)             Outlet        298.47     298.50     298.53     298.54
PRESSURE          Inlet          3.527      3.527      3.527      3.524
  (MPa)           Outlet         3.184      3.186      3.189      3.189
                  Atmospheric    0.094      0.094      0.094      0.094

HEAT EXCHANGE (J/min)           35.470     35.470     35.653     35.653
HEAT LEAK (J/min)                1.895      1.424      2.007      2.020
RATIO: LEAK OVER EXCHANGE       0.0534     0.0401     0.0563     0.0567
ENTHALPY CHANGE   Predicted   -2197.76   -2212.97   -2211.67   -2207.63
  (J/mol)         Measured    -2321.63   -2308.54   -2343.58   -2340.23

FLOW RATE  F-11   Pred. Mol.   1.35047    1.35942    1.35333    1.35280
  (l/min) or      Meas. Mol.   1.42670    1.42670    1.43405    1.43405
  (mmol/min)      Calc. Vol.   0.03595    0.03595    0.03613    0.03613
                             ----------------------
           N2     Meas. Vol.   0.40068    0.40351    0.39901    0.39958
                  Calc. Mol.  15.27590   15.38481   15.21303   15.23485

Freon Sat'n Temp. (K)           294.77     294.77     294.77     294.77
Freon Meter Temp. (K)           298.88     298.91     298.93     298.93
Freon Return Temp. (K)          294.89     294.89     294.89     294.89
Heat Exch. Corr. (J/min)        -0.022     -0.022     -0.022     -0.022
Ambient Air Temp  (K)           295.65     295.65     295.65     295.65
```

```
RUN  9    TEST GAS - N2                          **
CONDITIONS FOR TEST INTERVAL      1          2          3          4

TEMPERATURE       Lower Inlet   380.68     380.55     380.42     380.34
  (K)             Outlet        299.32     299.34     299.35     299.34
PRESSURE          Inlet          3.541      3.541      3.541      3.541
  (MPa)           Outlet         3.094      3.094      3.096      3.101
                  Atmospheric    0.094      0.094      0.094      0.094

HEAT EXCHANGE (J/min)           43.510     43.875     43.875     43.510
HEAT LEAK (J/min)               -2.700     -2.238     -2.033     -2.265
RATIO: LEAK OVER EXCHANGE      -0.0621    -0.0510    -0.0463    -0.0521
ENTHALPY CHANGE   Predicted   -2444.11   -2439.67   -2435.40   -2433.80
  (J/mol)         Measured    -2301.30   -2321.23   -2327.28   -2313.36

FLOW RATE  F-11   Pred. Mol.   1.85665    1.85479    1.84678    1.84114
  (l/min) or      Meas. Mol.   1.75005    1.76474    1.76474    1.75005
  (mmol/min)      Calc. Vol.   0.04411    0.04448    0.04448    0.04411
                             ----------------------
           N2     Meas. Vol.   0.48898    0.49883    0.48554    0.49436
                  Calc. Mol.  18.90669   18.90178   18.85257   18.80798

Freon Sat'n Temp. (K)           294.78     294.78     294.78     294.78
Freon Meter Temp. (K)           297.24     297.29     297.31     297.35
Freon Return Temp. (K)          294.83     294.83     294.83     294.83
Heat Exch. Corr. (J/min)        -0.016     -0.016     -0.016     -0.016
Ambient Air Temp. (K)           296.21     296.21     296.21     296.21
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DATA FROM FILE N2.2

INTERVALS MARKED WITH ** WERE MOST STABLE IN RUN

RUN  1    TEST GAS - N2                  **        **                            **        **
CONDITIONS FOR TEST INTERVAL              1         2         3         4         5         6

TEMPERATURE        Lower Inlet       395.90    395.94    395.81    395.44    395.63    395.61
  (K)              Outlet            300.34    300.35    300.31    300.28    300.26    300.27
PRESSURE           Inlet              7.008     7.005     7.009     7.009     7.009     7.008
  (MPa)            Outlet             6.698     6.699     6.701     6.704     6.704     6.704
                   Atmospheric        0.094     0.094     0.094     0.094     0.094     0.094

HEAT EXCHANGE (J/min)                73.836    73.470    73.470    73.105    73.105    73.105
HEAT LEAK (J/min)                     0.377     0.475     0.766     1.540     0.747     0.920
RATIO: LEAK OVER EXCHANGE            0.0051    0.0065    0.0105    0.0211    0.0102    0.0428
ENTHALPY CHANGE    Predicted       -2962.81  -2963.34  -2971.37  -2970.77  -2976.89  -2975.90
  (J/mol)          Measured        -2997.83  -3002.74  -3002.75  -3034.70  -3007.74  -3013.81

FLOW RATE  F-11    Pred. Mol.       2.95478   2.93616   2.92435   2.87860   2.91048   2.90358
  (l/min) or       Meas. Mol.       2.96994   2.95524   2.95524   2.94055   2.94055   2.94055
  (mmol/min)       Calc. Vol.       0.07479   0.07442   0.07442   0.07405   0 07405   0.07405
                                  --------------------
            N2     Meas. Vol.       0.54562   0.54138   0.54137   0.53147   0.53713   0.53584
                   Calc. Mol.      24.62967  24.46773  24.46762  24.08963  24.30557  24 25661

Freon Sat'n Temp. (K)                294.78    294.78    294.78    294.78    294.78    294 78
Freon Meter Temp. (K)                296.70    296.73    296.76    296.80    296.85    296.88
Freon Return Temp. (K)               294.91    294.91    294.91    294.94    294.97    294.93
Heat Exch. Corr. (J/min)             -0.048    -0.046    -0.046    -0.056    -0.057    -0.053
Ambient Air Temp. (K)                295.85    295.85    295.85    295.85    295.85    295.85



RUN  2    TEST GAS - N2                            **        **
CONDITIONS FOR TEST INTERVAL              1         2         3         4         5

TEMPERATURE        Lower Inlet       405.38    405.46    405.74    405 74    406.07
  (K)              Outlet            301.53    301.53    301.54    301.54    301.56
PRESSURE           Inlet              6.988     6.988     6.988     6.988     6.988
  (MPa)            Outlet             6.601     6.601     6.598     6.601     6.599
                   Atmospheric        0.094     0.094     0.094     0.094     0 094

HEAT EXCHANGE (J/min)                91.378    91.013    90.647    90.647    91.013
HEAT LEAK (J/min)                    -2.723    -2.479    -3.606    -3.435    -4.218
RATIO: LEAK OVER EXCHANGE           -0.0298   -0.0272   -0.0398   -0.0379   -0.0463
ENTHALPY CHANGE    Predicted       -3231.63  -3234.88  -3242.89  -3243.01  -3252.28
  (J/mol)          Measured        -3138.13  -3148.81  -3118.81  -3124.80  -3108.32

FLOW RATE  F-11    Pred. Mol.       3.78494   3.78045   3.79108   3.78420   3.83028
  (l/min) or       Meas. Mol.       3.87542   3.85072   3.84803   3.84803   3.85072
  (mmol/min)       Calc. Vol.       0.09282   0.09225   0.09188   0.09188   0.09225
                                  --------------------
            N2     Meas. Vol.       0.76455   0.75889   0.75314   0.76172   0.76680
                   Calc  Mol.      29.11873  28.90295  29.06473  29.01085  29.28040

Freon Sat'n Temp. (K)                294.76    294.76    294.76    294.76    294.76
Freon Meter Temp. (K)                297.01    296.98    296.95    296.95    296.91
Freon Return Temp. (K)               294.87    294.87    294.87    294.91    294.91
Heat Exch. Corr. (J/min)             -0.049    -0.049    -0.048    -0.056    -0.066
Ambient Air Temp. (K)                295.93    295 93    295.93    295.93    295.93



RUN  3    TEST GAS - N2                                      **
CONDITIONS FOR TEST INTERVAL              1         2         3         4

TEMPERATURE        Lower Inlet       376.12    376.40    376.55    376.71
  (K)              Outlet            298.48    298.52    298.55    298.56
PRESSURE           Inlet              5.726     5.719     5.719     5 722
  (MPa)            Outlet             5.616     5.611     5.604     5.604
                   Atmospheric        0.094     0.094     0.094     0.094

HEAT EXCHANGE (J/min)                47.530    47.530    48.992    48.992
HEAT LEAK (J/min)                     6.364     4.702     6.061     6.217
RATIO: LEAK OVER EXCHANGE            0.1339    0.0989    0.1237    0.1269
ENTHALPY CHANGE    Predicted       -2804.06  -2811.81  -2824.04  -2819.34
  (J/mol)          Measured        -2891.20  -2787.35  -2880.42  -2885.52

FLOW RATE  F-11    Pred. Mol.       1.65574   1.72260   1.72671   1.72045
  (l/min) or       Meas. Mol.       1.81172   1.81172   1.87051   1.87051
  (mmol/min)       Calc. Vol.       0.04818   0.04819   0.04968   0.04968
                                  --------------------
            N2     Meas. Vol.       0.43183   0.44782   0.44678   0.44589
                   Calc. Mol.      16.43885  17.05211  17.00851  16.97856

Freon Sat'n Temp. (K)                294.75    294.75    294.75    294.75
Freon Meter Temp. (K)                296.93    297.01    297.05    297.06
Freon Return Temp. (K)               294.93    294.93    294.93    294.93
Heat Exch. Corr. (J/min)             -0.042    -0.042    -0.043    -0.043
Ambient Air Temp. (K)                295.93    295.93    295.93    295.93
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DATA FROM FILE N2.3

INTERVALS MARKED WITH ** WERE MOST STABLE IN RUN

RUN  1    TEST GAS - N2                **
CONDITIONS FOR TEST INTERVAL           1         2

TEMPERATURE         Lower Inlet    338.77    338.73
  (K)               Outlet         297.58    297.59
PRESSURE            Inlet           5.883     5.883
  (MPa)             Outlet          5.794     5.794
                    Atmospheric     0.094     0.094

HEAT EXCHANGE (J/min)              21.576    21.941
HEAT LEAK (J/min)                   6.316     6.701
RATIO: LEAK OVER EXCHANGE          0.2928    0.3054
ENTHALPY CHANGE     Predicted    -1302.75  -1301.29
  (J/mol)           Measured     -1842.24  -1873.45

FLOW RATE  F-11     Pred. Mol.    0.61395   0.61325
  (l/min) or        Meas. Mol.    0.86818   0.88289
  (mmol/min)        Calc. Vol.    0.02173   0.02210
                                 --------------------
            N2      Meas. Vol.    0.30582   0.30582
                    Calc. Mol    11.71178  11.71166

Freon Sat'n Temp. (K)              294.97    294.97
Freon Meter Temp. (K)              297.61    297.57
Freon Return Temp. (K)             294.97    294.97
Heat Exch. Corr. (J/min)           -0.000    -0.000
Ambient Air Temp  (K)              298.48    298.48



RUN  2    TEST GAS - N2                          **
CONDITIONS FOR TEST INTERVAL           1         2         3

TEMPERATURE         Lower Inlet    359.40    359.07    356.82
  (K)               Outlet         299.06    299.05    299.02
PRESSURE            Inlet           5.837     5.837     5.837
  (MPa)             Outlet          5.604     5.604     5.604
                    Atmospheric     0.094     0.094     0.094

HEAT EXCHANGE (J/min)              36.552    36.552    36.187
HEAT LEAK (J/min)                  -1.668    -0.848    -0.888
RATIO: LEAK OVER EXCHANGE         -0.0456   -0.0232   -0.0245
ENTHALPY CHANGE     Predicted    -1892.87  -1882.77  -1868.83
  (J/mol)           Measured     -1810.18  -1840.10  -1825.02

FLOW RATE  F-11     Pred. Mol.    1.53782   1.50490   1.48184
  (l/min) or        Meas. Mol.    1.47079   1.47079   1.45610
  (mmol/min)        Calc. Vol.    0.03682   0.03682   0.03645
                                 --------------------
            N2      Meas. Vol.    0.52772   0.51914   0.51820
                    Calc. Mol.   20.19246  19.88414  19.82808

Freon Sat'n Temp. (K)              294.96    294.96    294.96
Freon Meter Temp. (K)              298.11    298.11    298.14
Freon Return Temp. (K)             294.88    294.88    294.88
Heat Exch. Corr. (J/min)            0.012     0.012     0.012
Ambient Air Temp. (K)              298.85    298.86    298.85



RUN  3    TEST GAS - N2                          **
CONDITIONS FOR TEST INTERVAL           1         2

TEMPERATURE         Lower Inlet    374.91    374.87
  (K)               Outlet         300.72    300.69
PRESSURE            Inlet           5.968     5.968
  (MPa)             Outlet          5.634     5.634
                    Atmospheric     0.094     0.094

HEAT EXCHANGE (J/min)              51.894    51.894
HEAT LEAK (J/min)                 -10.037   -10.100
RATIO: LEAK OVER EXCHANGE         -0.1934   -0.1946
ENTHALPY CHANGE     Predicted    -2317.28  -2317.04
  (J/mol)           Measured     -1941.71  -1939.57

FLOW RATE  F-11     Pred. Mol.    2.48195   2.49447
  (l/min) or        Meas. Mol.    2.08809   2.08809
  (mmol/min)        Calc. Vol     0.05228   0.05229
                                 --------------------
            N2      Meas. Vol.    0.69942   0.70020
                    Calc. Mol.   26.72806  26.75560

Freon Sat'n Temp. (K)              294.95    294.95
Freon Meter Temp. (K)              298.48    298.50
Freon Return Temp. (K)             294.97    294.97
Heat Exch. Corr. (J/min)           -0.008    -0.008
Ambient Air Temp  (K)              298.93    298.93
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RUN  4     TEST GAS - N2                                             **
CONDITIONS FOR TEST INTERVAL       1          2          3          4

TEMPERATURE        Lower Inlet   385.47     385.32     384.62     384.39
  (K)              Outlet        301.32     301.29     301.21     301.15
PRESSURE           Inlet           5.989      5.989      5.989      5.989
  (MPa)            Outlet          5.684      5.684      5.694      5.694
                   Atmospheric     0.094      0.094      0.094      0.094

HEAT EXCHANGE (J/min)             58.200     59.300     58.470     58.470
HEAT LEAK (J/min)                -12.159    -11.488    -11.243    -10.307
RATIO: LEAK OVER EXCHANGE        -0.2089    -0.1937    -0.1923    -0.1763
ENTHALPY CHANGE  Predicted     -2837.82   -2834.02   -2815.27   -2810.05
  (J/mol)        Measured      -2436.04   -2457.95   -2448.10   -2473.94

FLOW RATE   F-11   Pred. Mol.     2.87240    2.84341    2.80804    2.76737
  (l/min) or       Meas. Mol.     2.38204    2.38204    2.35264    2.35264
  (mmol/min)       Calc. Vol.     0.05866    0.05866    0.05893    0.05893
                            ---------------------
            N2     Meas. Vol.     0.63571    0.63006    0.62564    0.61828
                   Calc. Mol.    24.30177   24.08518   23.91289   23.63414

Freon Sat'n Temp. (K)            294.94     294.94     294.94     294.94
Freon Meter Temp. (K)            298.18     298.19     298.22     298.25
Freon Return Temp. (K)           294.87     294.87     294.87     294.87
Heat Exch. Corr. (J/min)           0.021      0.021      0.020      0.020
Ambient Air Temp. (K)            296.76     296.76     296.76     296.76



RUN  5     TEST GAS - N2          **                                 **
CONDITIONS FOR TEST INTERVAL       1          2          3          4

TEMPERATURE        Lower Inlet   417.83     417.84     418.01     418.09
  (K)              Outlet        304.78     304.84     304.86     304.86
PRESSURE           Inlet           5.996      5.996      5.996      5.996
  (MPa)            Outlet          5.387      5.384      5.379      5.387
                   Atmospheric     0.094      0.094      0.094      0.094

HEAT EXCHANGE (J/min)             90.614     91.344     91.709     92.440
HEAT LEAK (J/min)                -36.317    -36.284    -35.678    -35.053
RATIO: LEAK OVER EXCHANGE        -0.4008    -0.3972    -0.3890    -0.3792
ENTHALPY CHANGE  Predicted     -3500.27   -3498.62   -3502.44   -3506.32
  (J/mol)        Measured      -2488.78   -2503.98   -2521.48   -2541.55

FLOW RATE   F-11   Pred. Mol.     5.10732    5.13539    5.12673    5.12996
  (l/min) or       Meas. Mol.     3.64602    3.67542    3.69012    3.71951
  (mmol/min)       Calc. Vol.     0.08132    0.08206    0.08243    0.08318
                            ---------------------
            N2     Meas. Vol.     0.94861    0.95428    0.95144    0.95145
                   Calc. Mol.    36.26314   36.47961   36.37132   36.37141

Freon Sat'n Temp. (K)            294.94     294.94     294.94     294.94
Freon Meter Temp. (K)            298.32     298.37     298.45     298.50
Freon Return Temp. (K)           294.87     294.87     294.87     294.87
Heat Exch. Corr. (J/min)           0.032      0.032      0.032      0.032
Ambient Air Temp. (K)            296 76     296.76     296.76     296.76



RUN  6     TEST GAS - N2                                  **         **
CONDITIONS FOR TEST INTERVAL       1          2          3          4

TEMPERATURE        Lower Inlet   384.37     384.76     384.74     384.70
  (K)              Outlet        303.43     303 48     303.47     303.48
PRESSURE           Inlet           5.995      5.996      5.996      5.996
  (MPa)            Outlet          5.384      5.384      5.389      5.388
                   Atmospheric     0.094      0 094      0 094      0.094

HEAT EXCHANGE (J/min)             76.001     76.732     77.087     76.732
HEAT LEAK (J/min)                -26.939    -26.568    -25.741    -26.973
RATIO: LEAK OVER EXCHANGE        -0.3545    -0.3462    -0.3339    -0.3515
ENTHALPY CHANGE  Predicted     -2822.15   -2831.98   -2831.95   -2830.53
  (J/mol)        Measured      -2083.61   -2103.64   -2123.09   -2084.33

FLOW RATE   F-11   Pred. Mol.     4.14208    4.18848    4.13800    4.17285
  (l/min) or       Meas. Mol.     3.05812    3.08752    3.10222    3.08752
  (mmol/min)       Calc. Vol.     0.07688    0.07731    0.07768    0.07731
                            ---------------------
            N2     Meas. Vol.     0.95559    0.95559    0.95144    0.95994
                   Calc. Mol.    36.47566   36.47577   36.31384   36.63798

Freon Sat'n Temp. (K)            294.95     294.95     294.95     294.95
Freon Meter Temp. (K)            298.98     299.01     299.00     298.94
Freon Return Temp. (K)           294.89     294.89     294.89     294.89
Heat Exch. Corr. (J/min)           0.022      0.022      0.022      0.022
Ambient Air Temp. (K)            297.32     297.32     297.32     297.32
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DATA FROM FILE CO2.1

INTERVALS MARKED WITH ** WERE MOST STABLE IN RUN


RUN  1     BACKGROUND TEST (NO FLOW)
CONDITIONS FOR TEST INTERVAL     1

TEST HEAT LEAK (J/min)              6.533

TEMPERATURE        Lower Inlet    287.44
  (K)              Outlet         296.70
Atmospheric Pressure (MPa)          0.083

Freon Sat'n Temp. (K)             294.58
Freon Meter Temp. (K)             296.25
Freon Return Temp. (K)            294.60
Heat Exch. Corr. (J/min)           -0.001
Ambient Air Temp. (K)             296.37




RUN  2     TEST GAS - CO2                                     **          **
CONDITIONS FOR TEST INTERVAL      1          2          3          4

TEMPERATURE        Lower Inlet    388.52     388.54     388.56     388.55
  (K)              Outlet         300.23     300.22     300.21     300.20
PRESSURE           Inlet           3.502      3.502      3.498      3.498
  (MPa)            Outlet          3.126      3.128      3.123      3.118
                   Atmospheric     0.083      0.083      0.083      0.083

HEAT EXCHANGE (J/min)             76.081     76.782     78.426     76.426
HEAT LEAK (J/min)                 -2.405     -1.722     -2.651     -2.034
RATIO: LEAK OVER EXCHANGE        -0.0316    -0.0224    -0.0338    -0.0266
ENTHALPY CHANGE    Predicted    -4486.53   -4498.29   -4489.84   -4486.23
  (J/mol)          Measured     -4358.71   -4400.63   -4352.81   -4379.88

FLOW RATE   F-11   Pred. Mol.    3.15482    3.16874    3.17852    3.15459
  (l/min) or       Meas. Mol.    3.05812    3.08752    3.07282    3.07282
  (mmol/min)       Calc. Vol.    0.07786    0.07831    0.07793    0.07793
                               ---------------------
            CO2    Meas. Vol.    0.45873    0.45873    0.46156    0.45873
                   Calc. Mol.   17.45026   17.45018   17.55791   17.45018

Freon Sat'n Temp. (K)             294.57     294.57     294.57     294.57
Freon Meter Temp. (K)             296.29     296.29     296.36     296.44
Freon Return Temp. (K)            294.48     294.48     294.48     294.48
Heat Exch. Corr. (J/min)           0.034      0.034      0.034      0.034
Ambient Air Temp. (K)             295.85     295.85     295.85     295.85




RUN  3     TEST GAS - CO2                          **                     **
CONDITIONS FOR TEST INTERVAL      1          2          3          4

TEMPERATURE        Lower Inlet    387.36     387.01     385.58     383.90
  (K)              Outlet         298.95     298.91     298.75     298.53
PRESSURE           Inlet           3.540      3.544      3.547      3.551
  (MPa)            Outlet          3.303      3.308      3.318      3.333
                   Atmospheric     0.083      0.083      0.083      0.083

HEAT EXCHANGE (J/min)             61.074     60.343     56.687     55.956
HEAT LEAK (J/min)                  3.096      2.536      1.385      3.650
RATIO: LEAK OVER EXCHANGE         0.0507     0.0420     0.0244     0.0652
ENTHALPY CHANGE    Predicted    -4145.41   -4133.20   -4078.84   -4018.05
  (J/mol)          Measured     -4366.77   -4314.48   -4181.80   -4208.42

FLOW RATE   F-11   Pred. Mol.    2.33106    2.32419    2.22348    2.10300
  (l/min) or       Meas. Mol.    2.45553    2.42813    2.27916    2.24875
  (mmol/min)       Calc. Vol.    0.06228    0.06155    0.05782    0.05707
                               ---------------------
            CO2    Meas. Vol.    0.36812    0.36812    0.35670    0.34283
                   Calc  Mol.   13.98811   13.98811   13.55575   13.01787

Freon Sat'n Temp  (K)             294.56     294.56     294.56     294.56
Freon Meter Temp. (K)             296.25     296.29     296.32     296.32
Freon Return Temp. (K)            294.38     294.36     294.35     294.32
Heat Exch. Corr. (J/min)           0.060      0.058      0.058      0.058
Ambient Air Temp. (K)             295.93     295.93     295.93     295.93
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DATA FROM FILE CO2.2

INTERVALS MARKED WITH ** WERE MOST STABLE IN RUN

RUN  1    TEST GAS - CO2                                                                       **
CONDITIONS FOR TEST INTERVAL       1          2          3          4          5          6          7

TEMPERATURE       Lower Inlet    406.08     406.73     405.80     403.94     403.07     403.77     404.88
  (K)             Outlet         301.80     301.73     301.77     301.88     301.57     301.59     301.64
PRESSURE          Inlet           3.520      3.530      3.530      3.520      3.513      3.520      3.527
  (MPa)           Outlet          3.088      3.108      3.093      3.103      3.106      3.093      3.088
                  Atmospheric     0.083      0.083      0.083      0.083      0.083      0.083      0.083

HEAT EXCHANGE (J/min)            82.628     81.531     80.800     79.338     77.577     78.973     78.704
HEAT LEAK (J/min)                -7.343    -10.814    -12.175     -8.883     -9.584    -10.882     -9.808
RATIO: LEAK OVER EXCHANGE       -0.0889    -0.1328    -0.1507    -0.1094    -0.1231    -0.1378    -0.1230
ENTHALPY CHANGE   Predicted    -4744.54   -4734.88   -4727.88   -4854.98   -4828.32   -4848.32   -4880.23
  (J/mol)         Measured     -4387.32   -4180.40   -4108.77   -4185.80   -4118.48   -4083.18   -4187.51

FLOW RATE  F-11   Pred. Mol.    3.81795    3.71348    3.73877    3.53958    3.51698    3.61371    3.58841
  (l/min) or      Meas. Mol.    3.32288    3.27858    3.24918    3.19040    3.13181    3.17571    3.20810
  (mmol/min)      Calc. Vol.    0.08406    0.08294    0.08220    0.08071    0.07922    0.08034    0.08108
                          --------------------
            CO2   Meas. Vol.    0.49895    0.51112    0.51837    0.48854    0.48854    0.50887    0.50121
                  Calc. Mol.   18.88287   19.50323   18.88533   18.90888   18.90898   19.34114   19.12806

Freon Sat'n Temp. (K)            294.85     294.85     294.85     294.85     294.85     294.85     294.85
Freon Meter Temp. (K)            296.84     296.84     296.76     296.98     297.15     297.28     297.28
Freon Return Temp. (K)           294.70     294.70     294.70     294.70     294.70     294.70     294.70
Heat Exch. Corr. (J/min)         -0.019     -0.019     -0.019     -0.019     -0.018     -0.019     -0.019
Ambient Air Temp. (K)            295.54     295.54     295.54     295.54     295.54     295.54     295.54


RUN  2    TEST GAS - CO2                   **
CONDITIONS FOR TEST INTERVAL       1          2          3          4

TEMPERATURE       Lower Inlet    407.20     407.28     407.74     408.21
  (K)             Outlet         301.73     301.78     301.80     301.88
PRESSURE          Inlet           3.523      3.520      3.523      3.523
  (MPa)           Outlet          3.081      3.093      3.083      3.088
                  Atmospheric     0.083      0.083      0.083      0.083

HEAT EXCHANGE (J/min)            81.895     82.280     82.826     83.357
HEAT LEAK (J/min)               -10.496    -10.174     -9.419    -11.052
RATIO: LEAK OVER EXCHANGE       -0.1282    -0.1237    -0.1140    -0.1326
ENTHALPY CHANGE   Predicted    -4784.51   -4788.79   -4817.06   -4831.59
  (J/mol)         Measured     -4249.85   -4268.82   -4324.12   -4286.98

FLOW RATE  F-11   Pred. Mol.    3.71836    3.71711    3.70148    3.79981
  (l/min) or      Meas. Mol.    3.28329    3.30798    3.32288    3.35208
  (mmol/min)      Calc. Vol.    0.08328    0.08385    0.08402    0.08477
                          --------------------
            CO2   Meas. Vol.    0.50846    0.50846    0.50121    0.51283
                  Calc. Mol.   19.27007   19.27007   19.10811   19.53890

Freon Sat'n Temp. (K)            294.85     294.85     294.85     294.85
Freon Meter Temp. (K)            297.17     297.26     297.28     297.32
Freon Return Temp. (K)           294.70     294.70     294.70     294.70
Heat Exch. Corr. (J/min)         -0.015     -0.015     -0.015     -0.015
Ambient Air Temp. (K)            295.93     295.93     295.93     295.93


RUN  3    TEST GAS - CO2        **                                          **
CONDITIONS FOR TEST INTERVAL       1          2          3          4          5          6

TEMPERATURE       Lower Inlet    384.53     385.84     385.15     384.61     385.39     383.71
  (K)             Outlet         289.41     289.48     289.53     289.47     289.45     289.40
PRESSURE          Inlet           3.523      3.530      3.530      3.527      3.527      3.520
  (MPa)           Outlet          3.318      3.318      3.322      3.318      3.316      3.321
                  Atmospheric     0.083      0.083      0.083      0.083      0.083      0.083

HEAT EXCHANGE (J/min)            51.825     53.021     53.752     51.825     51.825     51.825
HEAT LEAK (J/min)                -0.471     -2.044     -0.131      0.901     -1.205      3.136
RATIO: LEAK OVER EXCHANGE       -0.0091    -0.0385    -0.0024     0.0173    -0.0232     0.0604
ENTHALPY CHANGE   Predicted    -4002.88   -4048.43   -4028.02   -3987.91   -4037.78   -3988.05
  (J/mol)         Measured     -3866.88   -3884.19   -4019.24   -4066.50   -3948.21   -4223.12

FLOW RATE  F-11   Pred. Mol.    2.10703    2.21438    2.15864    2.05188    2.13854    1.86187
  (l/min) or      Meas. Mol.    2.06808    2.13218    2.16758    2.06808    2.06808    2.06808
  (mmol/min)      Calc. Vol.    0.05280    0.05392    0.05480    0.05280    0.05280    0.05280
                          --------------------
            CO2   Meas. Vol.    0.34385    0.35711    0.35139    0.33508    0.34848    0.32281
                  Calc. Mol.   13.08989   13.50188   13.38377   12.76271   13.15819   12.20541

Freon Sat'n Temp. (K)            294.85     294.85     294.85     294.85     294.85     294.85
Freon Meter Temp. (K)            296.94     297.12     297.21     297.27     297.31     297.32
Freon Return Temp. (K)           294.70     294.70     294.70     294.70     294.70     294.70
Heat Exch. Corr. (J/min)         -0.008     -0.009     -0.010     -0.008     -0.008     -0.009
Ambient Air Temp. (K)            296.21     296.21     296.21     296.21     296.21     296.21
```

```
RUN  4    TEST GAS - CO2                                          **
CONDITIONS FOR TEST INTERVAL       1          2          3          4

TEMPERATURE       Lower Inlet    355.04     355.88     355.31     353.27
  (K)             Outlet         297.48     297.46     297.46     297.48
PRESSURE          Inlet           3.506      3.510      3.513      3.510
  (MPa)           Outlet          3.456      3.453      3.456      3.461
                  Atmospheric     0.093      0.093      0.093      0.093

HEAT EXCHANGE (J/min)            26.341     25.875     25.610     25.246
HEAT LEAK (J/min)                 9.554      8.663      8.907      7.900
RATIO: LEAK OVER EXCHANGE         0.3627     0.3335     0.3478     0.3129
ENTHALPY CHANGE   Predicted   -2832.11   -2888.70   -2843.78   -2791.17
  (J/mol)         Measured    -4443.56   -4304.05   -4380.33   -4004.17

FLOW RATE  F-11   Pred. Mol.     0.57506    0.55621    0.57167    0.55750
  (l/min) or      Meas. Mol.     1.05526    1.04456    1.02557    1.01517
  (mmol/min)      Calc. Vol.     0.02574    0.02641    0.02604    0.02567
                             ----------------------
             CO2  Meas. Vol.     0.15574    0.15657    0.15433    0.15555
                  Calc. Mol.     5.92730    6.03501    5.87334    6.30449

Freon Sat'n Temp. (K)            294.55     294.55     294.55     294.55
Freon Meter Temp. (K)            295.95     295.95     295.95     295.89
Freon Return Temp. (K)           294.70     294.70     294.70     294.70
Heat Exch. Corr. (J/min)         -0.004     -0.004     -0.004     -0.004
Ambient Air Temp. (K)            296.48     296.48     296.48     296.46
```

```
DATA FROM FILE CO2.3

INTERVALS MARKED WITH ** WERE MOST STABLE IN RUN

RUN  1    TEST GAS - CO2                                              **
CONDITIONS FOR TEST INTERVAL        1         2         3         4         5

TEMPERATURE       Lower Inlet    383.88    386.07    387.81    387.74    387.35
  (K)             Outlet         298.59    298.74    298.91    298.97    298.86
PRESSURE          Inlet           3.547     3.584     3.557     3.537     3.537
  (MPa)           Outlet          3.318     3.313     3.288     3.270     3.278
                  Atmospheric     0.093     0.093     0.093     0.093     0.093

HEAT EXCHANGE (J/min)            74.808    78.285    80.824    81.190    81.190
HEAT LEAK (J/min)                19.331    18.287    18.204    18.740    20.731
RATIO: LEAK OVER EXCHANGE        0.2581    0.2337    0.2252    0.2308    0.2553
ENTHALPY CHANGE   Predicted    -4011.41  -4098.43  -4154.73  -4143.44  -4128.28
  (J/mol)         Measured     -5414.27  -5345.43  -5362.53  -5288.81  -5545.23

FLOW RATE  F-11   Pred. Mol.    2.22220   2.41115   2.51738   2.51083   2.43047
  (l/min) or      Meas. Mol.    2.99934   3.14831   3.24819   3.26388   3.26389
  (mmol/min)      Calc. Vol.    0.07874   0.07898   0.08289   0.08287   0.08287
                              --------------------
          CO2     Meas. Vol.    0.38245   0.38811   0.39844   0.39843   0.38811
                  Calc. Mol.   13.78005  14.54145  15.07201  15.07194  14.54135

Freon Sat'n Temp. (K)            294.50    294.50    294.50    294.50    294.50
Freon Meter Temp. (K)            295.28    295.18    295.15    295.13    295.15
Freon Return Temp. (K)           294.54    294.54    294.54    294.54    294.54
Heat Exch. Corr. (J/min)         -0.013    -0.013    -0.014    -0.014    -0.014
Ambient Air Temp. (K)            295.[illegible]    295.09    295.09    295.09    295.09


RUN  2    TEST GAS - CO2                                              **
CONDITIONS FOR TEST INTERVAL        1         2         3         4

TEMPERATURE       Lower Inlet    385.85    385.87    385.53    385.86
  (K)             Outlet         298.85    298.85    298.85    298.86
PRESSURE          Inlet           3.530     3.526     3.526     3.526
  (MPa)           Outlet          3.278     3.276     3.283     3.280
                  Atmospheric     0.093     0.093     0.093     0.093

HEAT EXCHANGE (J/min)            78.380    78.380    78.895    78.895
HEAT LEAK (J/min)                18.526    19.840    18.886    18.878
RATIO: LEAK OVER EXCHANGE        0.2480    0.2475    0.2391    0.2394
ENTHALPY CHANGE   Predicted    -4115.77  -4107.89  -4104.09  -4116.48
  (J/mol)         Measured     -5458.85  -5458.85  -5393.77  -5393.77

FLOW RATE  F-11   Pred. Mol.    2.40844   2.40084   2.41838   2.42483
  (l/min) or      Meas. Mol.    3.19040   3.19040   3.17571   3.17571
  (mmol/min)      Calc. Vol.    0.08108   0.08108   0.08071   0.08071
                              --------------------
          CO2     Meas. Vol.    0.38228   0.38228   0.38811   0.38811
                  Calc. Mol.   14.53793  14.53793  14.84558  14.84558

Freon Sat'n Temp. (K)            294.51    294.51    294.51    294.51
Freon Meter Temp. (K)            295.13    295.17    295.21    295.39
Freon Return Temp. (K)           294.82    294.82    294.82    294.82
Heat Exch. Corr. (J/min)         -0.041    -0.041    -0.041    -0.041
Ambient Air Temp. (K)            295.09    295.09    295.09    295.09


RUN  3    TEST GAS - CO2                                    **        **
CONDITIONS FOR TEST INTERVAL        1         2         3         4

TEMPERATURE       Lower Inlet    393.35    392.79    392.55    392.37
  (K)             Outlet         300.26    300.22    300.19    300.18
PRESSURE          Inlet           3.562     3.562     3.562     3.568
  (MPa)           Outlet          3.323     3.323     3.323     3.328
                  Atmospheric     0.093     0.093     0.093     0.093

HEAT EXCHANGE (J/min)            57.769    57.769    57.403    57.403
HEAT LEAK (J/min)                -3.282    -2.716    -2.947    -3.085
RATIO: LEAK OVER EXCHANGE       -0.0568   -0.0470   -0.0513   -0.0537
ENTHALPY CHANGE   Predicted    -4355.57  -4322.97  -4322.27  -4316.50
  (J/mol)         Measured     -4121.46  -4137.41  -4111.21  -4098.38

FLOW RATE  F-11   Pred. Mol.    2.45822   2.43285   2.42707   2.43283
  (l/min) or      Meas. Mol.    2.32325   2.32325   2.30855   2.30855
  (mmol/min)      Calc. Vol.    0.05888   0.05888   0.05831   0.05831
                              --------------------
          CO2     Meas. Vol.    0.38870   0.38528   0.38529   0.38870
                  Calc. Mol.   14.01858  13.88247  13.88258  14.01858

Freon Sat'n Temp. (K)            294.70    294.70    294.70    294.70
Freon Meter Temp. (K)            295.35    295.35    295.32    295.31
Freon Return Temp. (K)           294.79    294.79    294.79    294.79
Heat Exch. Corr. (J/min)         -0.028    -0.028    -0.028    -0.028
Ambient Air Temp. (K)            295.54    295.54    295.54    295.54
```

```
RUN  4    TEST GAS - CO2                                        **
CONDITIONS FOR TEST INTERVAL      1          2          3          4

TEMPERATURE        Lower Inlet   385.79     386.11     386.09     386.43
  (K)              Outlet        297.86     298.02     298.04     298.06
PRESSURE           Inlet           3.566      3.556      3.558      3.566
  (MPa)            Outlet          3.461      3.448      3.446      3.456
                   Atmospheric     0.093      0.093      0.093      0.093

HEAT EXCHANGE (J/min)             35.111     35.477     35.842     35.842
HEAT LEAK (J/min)                  8.804      9.232      9.130      9.109
RATIO: LEAK OVER EXCHANGE          0.2508     0.2602     0.2547     0.2541
ENTHALPY CHANGE  Predicted     -3287.80   -3296.02   -3293.64   -3311.03
  (J/mol)        Measured      -4387.88   -4455.39   -4419.46   -4438.16

FLOW RATE  F-11  Pred. Mol.       1.05793    1.05545    1.07421    1.07508
  (l/min) or     Meas. Mol.       1.41200    1.42870    1.44140    1.44140
  (mmol/min)     Calc. Vol.       0.03568    0.03605    0.03643    0.03643
                              ----------------------
           CO2   Meas. Vol.       0.20854    0.20852    0.21238    0.21143
                 Calc. Mol.       8.00188    7.96283    8.11007    8.07405

Freon Sat'n Temp. (K)            294.68     294.68     294.68     294.68
Freon Meter Temp. (K)            295.55     295.61     295.55     295.60
Freon Return Temp. (K)           294.70     294.70     294.70     294.70
Heat Exch. Corr. (J/min)          -0.003     -0.003     -0.003     -0.003
Ambient Air Temp. (K)            295.85     295.85     295.85     295.85
```

```
RUN  5    TEST GAS - CO2                             **
CONDITIONS FOR TEST INTERVAL     1          2          3

TEMPERATURE        Lower Inlet  407.12     407.13     407.13
  (K)              Outlet       302.10     302.18     302.22
PRESSURE           Inlet          3.548      3.548      3.548
  (MPa)            Outlet         3.113      3.113      3.113
                   Atmospheric    0.093      0.093      0.093

HEAT EXCHANGE (J/min)            76.410     77.872     78.603
HEAT LEAK (J/min)               -14.987    -13.428    -13.168
RATIO: LEAK OVER EXCHANGE        -0.1959    -0.1725    -0.1675
ENTHALPY CHANGE  Predicted    -4778.38   -4775.43   -4773.00
  (J/mol)        Measured     -3996.55   -4073.01   -4088.14

FLOW RATE  F-11  Pred. Mol.      3.57470    3.57168    3.59058
  (l/min) or     Meas. Mol.      3.07282    3.13181    3.16101
  (mmol/min)     Calc. Vol.      0.07768    0.07915    0.07988
                             ----------------------
           CO2   Meas. Vol.      0.50121    0.50121    0.50404
                 Calc. Mol.     19.11803   19.11803   19.22710

Freon Sat'n Temp. (K)           294.68     294.68     294.68
Freon Meter Temp. (K)           297.05     297.06     297.06
Freon Return Temp. (K)          294.72     294.72     294.72
Heat Exch. Corr. (J/min)         -0.015     -0.015     -0.015
Ambient Air Temp. (K)           295.93     295.93     295.93
```

```
DATA FROM FILE CO2.4

INTERVALS MARKED WITH ** WERE MOST STABLE IN RUN

RUN  1    TEST GAS - CO2          **                              **
CONDITIONS FOR TEST INTERVAL       1          2          3          4

TEMPERATURE          Lower Inlet   374.50     375.25     375.20     374.92
  (K)                Outlet        298.16     298.17     298.17     298.16
PRESSURE             Inlet           5.123      5.119      5.105      5.102
  (MPa)              Outlet          5.033      5.020      5.010      5.008
                     Atmospheric     0.093      0.093      0.093      0.093

HEAT EXCHANGE (J/min)               61.080     61.811     61.448     61.811
HEAT LEAK (J/min)                   12.940     13.847     13.584     13.902
RATIO: LEAK OVER EXCHANGE            0.2119     0.2240     0.2211     0.2249
ENTHALPY CHANGE      Predicted   -4388.54   -4425.86   -4418.38   -4398.00
  (J/mol)            Measured    -5580.84   -5703.59   -5668.86   -5675.51

FLOW RATE   F-11     Pred. Mol.      1.83532    1.82824    1.82412    1.82802
  (l/min) or         Meas. Mol.      2.45553    2.48492    2.47022    2.48492
  (mmol/min)         Calc. Vol.      0.06228    0.06314    0.06277    0.06314
                                 ----------------------
            CO2      Meas. Vol.      0.28863    0.28800    0.28800    0.28741
                     Calc. Mol.     10.94458   10.83725   10.83725   10.89058

Freon Sat'n Temp. (K)              294.52     294.52     294.52     294.52
Freon Meter Temp. (K)              297.79     297.83     297.91     298.01
Freon Return Temp. (K)             294.56     294.56     294.56     294.56
Heat Exch. Corr. (J/min)            -0.013     -0.013     -0.013     -0.013
Ambient Air Temp. (K)              298.21     298.21     298.21     298.21



RUN  2    TEST GAS - CO2                                                 **
CONDITIONS FOR TEST INTERVAL       1          2          3          4

TEMPERATURE          Lower Inlet   385.77     385.84     385.57     385.94
  (K)                Outlet        299.19     299.20     299.19     299.18
PRESSURE             Inlet           5.095      5.092      5.088      5.081
  (MPa)              Outlet          4.945      4.943      4.938      4.928
                     Atmospheric     0.093      0.093      0.093      0.093

HEAT EXCHANGE (J/min)               78.373     78.373     78.828     78.985
HEAT LEAK (J/min)                   10.721     10.869     11.594     11.871
RATIO: LEAK OVER EXCHANGE            0.1368     0.1387     0.1470     0.1503
ENTHALPY CHANGE      Predicted   -4780.65   -4792.23   -4776.81   -4788.48
  (J/mol)            Measured    -5549.81   -5583.87   -5586.78   -5638.59

FLOW RATE   F-11     Pred. Mol.      2.71873    2.71376    2.69634    2.69846
  (l/min) or         Meas. Mol.      3.15072    3.15072    3.15101    3.17571
  (mmol/min)         Calc. Vol.      0.08007    0.08007    0.08033    0.08071
                                 ----------------------
            CO2      Meas. Vol.      0.37378    0.37284    0.37185    0.37095
                     Calc. Mol.     14.12179   14.08812   14.04149   14.01485

Freon Sat'n Temp. (K)              294.51     294.51     294.51     294.51
Freon Meter Temp. (K)              298.38     298.40     298.42     298.42
Freon Return Temp. (K)             294.52     294.52     294.52     294.52
Heat Exch. Corr. (J/min)            -0.003     -0.003     -0.003     -0.003
Ambient Air Temp. (K)              297.04     297.04     297.04     297.04



RUN  3    TEST GAS - CO2          **                              **
CONDITIONS FOR TEST INTERVAL       1          2          3          4

TEMPERATURE          Lower Inlet   404.72     404.43     403.48     403.10
  (K)                Outlet        301.35     301.32     301.29     301.27
PRESSURE             Inlet           5.118      5.112      5.102      5.091
  (MPa)              Outlet          4.883      4.858      4.848      4.843
                     Atmospheric     0.093      0.093      0.093      0.093

HEAT EXCHANGE (J/min)              106.789    106.789    105.328    105.328
HEAT LEAK (J/min)                    1.437      1.726      2.311      2.418
RATIO: LEAK OVER EXCHANGE            0.0135     0.0162     0.0219     0.0230
ENTHALPY CHANGE      Predicted   -5473.61   -5458.58   -5408.54   -5391.50
  (J/mol)            Measured    -5548.28   -5546.24   -5529.88   -5518.24

FLOW RATE   F-11     Pred. Mol.      4.23498    4.22335    4.14103    4.13688
  (l/min) or         Meas. Mol.      4.29272    4.29272    4.23393    4.23393
  (mmol/min)         Calc. Vol.      0.10924    0.10924    0.10774    0.10774
                                 ----------------------
            CO2      Meas. Vol.      0.50870    0.50870    0.50439    0.50546
                     Calc. Mol.     19.24728   19.24734   19.04681   19.08883

Freon Sat'n Temp. (K)              294.47     294.47     294.47     294.47
Freon Meter Temp. (K)              297.58     297.57     297.62     297.64
Freon Return Temp. (K)             294.56     294.56     294.56     294.56
Heat Exch. Corr. (J/min)            -0.045     -0.045     -0.044     -0.044
Ambient Air Temp. (K)              298.76     298.76     298.76     298.76
```

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DATA FROM FILE CO2.SL

INTERVALS MARKED WITH ** WERE MOST STABLE IN RUN

RUN  1    TEST GAS - CO2                                   **         **         **
CONDITIONS FOR TEST INTERVAL    1          2          3          4          5          6

TEMPERATURE         Lower Inlet  396.26     395.15     393.72     392.45     391.63     391.39
  (K)               Outlet       302.73     302.58     302.38     302.18     302.07     301.99
PRESSURE            Inlet          9.735      9.726      9.731      9.731      9.728      9.728
  (MPa)             Outlet         9.673      9.673      9.683      9.683      9.678      9.673
                    Atmospheric    0.093      0.093      0.093      0.093      0.093      0.093

HEAT EXCHANGE (J/min)            180.278    177.189    171.045    169.506    167.970    167.970
HEAT LEAK (J/min)                 11.185     15.517     11.888     14.085     14.625     18.420
RATIO: LEAK OVER EXCHANGE          0.0620     0.0876     0.0695     0.0831     0.0871     0.1097
ENTHALPY CHANGE     Predicted  -11658.53-11618.39-11573.61-11532.48-11512.13-11489.31
  (J/mol)           Measured   -12429.73-12734.49-12435.79-12577.54-12610.05-12615.70

FLOW RATE   F-11    Pred. Mol.   5.75880    5.49604    5.39673    5.24743    5.16389    5.01132
  (l/min) or        Meas. Mol.   7.24541    7.12274    6.87541    6.81356    6.75175    6.75175
  (mmol/min)        Calc. Vol.   0.18456    0.18140    0.17510    0.17353    0.17185    0.17185
                             --------------------
            CO2     Meas. Vol.   0.38389    0.38612    0.36357    0.35653    0.35238    0.34405
                    Calc. Mol.  14.50380   13.81491   13.75435   13.47703   13.32031   13.00508

Freon Sat'n Temp. (K)            294.45     294.45     294.45     294.45     294.45     294.45
Freon Meter Temp. (K)            295.12     295.09     295.13     295.17     295.17     295.15
Freon Return Temp. (K)           294.36     294.36     294.36     294.36     294.36     294.36
Heat Exch. Corr. (J/min)           0.075      0.074      0.072      0.071      0.070      0.070
Ambient Air Temp. (K)            296.21     296.21     296.21     296.21     296.21     296.21



RUN  2    TEST GAS - CO2                                              **         **
CONDITIONS FOR TEST INTERVAL    1          2          3          4          5

TEMPERATURE         Lower Inlet  448.04     448.64     448.64     450.56     450.63
  (K)               Outlet       312.89     312.97     313.15     313.29     313.29
PRESSURE            Inlet          9.726      9.742      9.745      9.732      9.732
  (MPa)             Outlet         9.526      9.545      9.545      9.523      9.520
                    Atmospheric    0.093      0.093      0.093      0.093      0.093

HEAT EXCHANGE (J/min)            387.912    390.986    397.141    392.526    394.064
HEAT LEAK (J/min)                -12.855    -12.975    -13.719    -17.779    -14.357
RATIO: LEAK OVER EXCHANGE         -0.0331    -0.0332    -0.0345    -0.0453    -0.0364
ENTHALPY CHANGE     Predicted  -12228.69-12247.19-12237.75-12221.23-12223.61
  (J/mol)           Measured   -11837.41-11853.80-11829.13-11691.66-11793.90

FLOW RATE   F-11    Pred. Mol.  16.11056   16.23909   16.51631   16.49405   16.41632
  (l/min) or        Meas. Mol.  15.58286   15.71751   15.96466   15.77935   15.84118
  (mmol/min)        Calc. Vol.   0.39891    0.39976    0.40604    0.40133    0.40290
                             --------------------
            CO2     Meas. Vol.   0.86646    0.87215    0.88773    0.88773    0.88348
                    Calc. Mol.  32.79896   32.98415   33.57307   33.57318   33.41262

Freon Sat'n Temp. (K)            294.49     294.49     294.49     294.49     294.49
Freon Meter Temp. (K)            295.80     295.83     295.90     295.87     295.93
Freon Return Temp. (K)           294.42     294.42     294.42     294.42     294.43
Heat Exch. Corr. (J/min)           0.126      0.127      0.129      0.128      0.110
Ambient Air Temp. (K)            296.46     296.46     296.46     296.46     296.46



RUN  3    TEST GAS - CO2        **                               **
CONDITIONS FOR TEST INTERVAL    1          2          3          4

TEMPERATURE         Lower Inlet  458.33     458.90     458.62     458.40
  (K)               Outlet       314.86     314.94     314.73     314.83
PRESSURE            Inlet          9.763      9.763      9.759      9.759
  (MPa)             Outlet         9.405      9.415      9.406      9.403
                    Atmospheric    0.093      0.093      0.093      0.093

HEAT EXCHANGE (J/min)            430.858    429.417    426.341    426.341
HEAT LEAK (J/min)                -22.440    -23.389    -22.784    -23.123
RATIO: LEAK OVER EXCHANGE         -0.0521    -0.0545    -0.0534    -0.0542
ENTHALPY CHANGE     Predicted  -11964.56-11990.82-12006.50-12025.24
  (J/mol)           Measured   -11372.40-11371.28-11389.32-11406.56

FLOW RATE   F-11    Pred. Mol.  18.22729   18.20357   18.05560   18.06926
  (l/min) or        Meas. Mol.  17.32516   17.26334   17.13966   17.13966
  (mmol/min)        Calc. Vol.   0.44023    0.43869    0.43552    0.43552
                             --------------------
            CO2     Meas. Vol.   1.00100    0.99752    0.98794    0.98731
                    Calc. Mol.  37.89462   37.76331   37.40059   37.37674

Freon Sat'n Temp. (K)            294.52     294.52     294.52     294.52
Freon Meter Temp. (K)            295.83     295.85     295.86     295.82
Freon Return Temp. (K)           294.36     294.37     294.36     294.36
Heat Exch. Corr. (J/min)           0.281      0.301      0.319      0.316
Ambient Air Temp. (K)            296.46     296.46     296.46     296.46
```

```
RUN  4   -TEST GAS - CO2            **                   **
CONDITIONS FOR TEST INTERVAL        1          2          3

TEMPERATURE        Lower Inlet   436.43     436.26     436.06
   (K)             Outlet        310.45     310.44     310.41
PRESSURE           Inlet           9.749      9.745      9.745
  (MPa)            Outlet          9.565      9.563      9.563
                   Atmospheric     0.083      0.083      0.083

HEAT EXCHANGE (J/min)            341.128    341.789    340.212
HEAT LEAK (J/min)                -18.758    -16.619    -16.809
RATIO: LEAK OVER EXCHANGE        -0.0550    -0.0485    -0.0494
ENTHALPY CHANGE   Predicted    -12582.74-12576.64-12570.81
   (J/mol)        Measured     -11826.68-11863.42-11864.40

FLOW RATE   F-11  Pred. Mol.   14.46815 14.40695 14.35278
   (1/min) or     Meas. Mol.   13.71413 13.73669 13.67702
   (mmol/min)     Calc. Vol.    0.34882  0.34815  0.34758
                           --------------------
             CO2  Meas. Vol.    0.75606  0.75323  0.75040
                  Calc. Mol.   28.60187 28.49481 28.38774

Freon Sat'n Temp. (K)            294.81     294.81     294.81
Freon Meter Temp. (K)            298.64     298.62     298.60
Freon Return Temp. (K)           294.24     294.22     294.22
Heat Exch. Corr. (J/min)           0.446      0.478      0.476
Ambient Air Temp. (K)            298.65     298.65     298.65
```

```
RUN  5    TEST GAS - CO2                                                  **
CONDITIONS FOR TEST INTERVAL    1          2          3          4          5

TEMPERATURE       Lower Inlet  431.25     431.86     432.24     432.70     433.07
   (K)            Outlet       309.34     309.41     309.54     309.63     309.69
PRESSURE          Inlet          9.741      9.745      9.748      9.752      9.752
  (MPa)           Outlet         9.567      9.570      9.570      9.567      9.570
                  Atmospheric    0.082      0.082      0.082      0.082      0.082

HEAT EXCHANGE (J/min)          311.120    314.198    316.737    318.814    318.814
HEAT LEAK (J/min)               -8.488     -9.628     -8.338     -9.510     -9.720
RATIO: LEAK OVER EXCHANGE      -0.0273    -0.0306    -0.0264    -0.0298    -0.0305
ENTHALPY CHANGE  Predicted   -12486.94-12475.29-12484.59-12494.48-12502.83
   (J/mol)       Measured    -12134.83-12104.32-12163.55-12132.96-12132.82

FLOW RATE  F-11  Pred. Mol,   12.84321 13.01280 13.02286 13.18361 13.20184
   (1/min) or    Meas. Mol.   12.50220 12.62587 12.68770 12.81137 12.81137
   (mmol/min)    Calc. Vol.    0.31993  0.32308  0.32466  0.32784  0.32784
                         --------------------
            CO2  Meas. Vol.    0.68243  0.69083  0.69083  0.69842  0.69843
                 Calc. Mol.   25.93540 25.95747 25.95758 26.27868 26.27875

Freon Sat'n Temp. (K)          294.30     294.30     294.30     294.30     294:30
Freon Meter Temp. (K)          299.08     299.08     298.99     298.97     299.02
Freon Return Temp. (K)         294.32     294.32     294.32     294.32     294.32
Heat Exch. Corr. (J/min)        -0.027     -0.028     -0.028     -0.024     -0.028
Ambient Air Temp. (K)          298.48     298.48     298.48     298.48     298.48
```

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RUN  6    TEST GAS - CO2                                             **
CONDITIONS FOR TEST INTERVAL    1          2          3          4

TEMPERATURE       Lower Inlet  433.32     433.10     432.73     432.56
   (K)            Outlet       309.80     309.77     309.71     309.64
PRESSURE          Inlet          9.755      9.752      9.752      9.755
  (MPa)           Outlet         9.587      9.582      9.587      9.577
                  Atmospheric    0.082      0.082      0.082      0.082

HEAT EXCHANGE (J/min)          303.437    303.437    300.358    294.204
HEAT LEAK (J/min)              -26.512    -24.953    -24.547    -30.868
RATIO: LEAK OVER EXCHANGE      -0.0874    -0.0822    -0.0817    -0.1049
ENTHALPY CHANGE  Predicted   -12496.20-12488.34-12476.96-12480.38
   (J/mol)       Measured    -11491.18-11527.55-11537.10-11300.57

FLOW RATE  F-11  Pred. Mol.   12.28438 13.18570 13.06573 13.08241
   (1/min) or    Meas. Mol.   12.18304 12.18304 12.06937 11.82204
   (mmol/min)    Calc. Vol.    0.31218  0.31218  0.30902  0.30269
                         --------------------
            CO2  Meas. Vol.    0.70367  0.70084  0.69376  0.69376
                 Calc. Mol.   26.40607 26.29988 26.03416 26.03418

Freon Sat'n Temp. (K)          294.28     294.28     294.28     294.28
Freon Meter Temp. (K)          299.13     299.15     299.14     299.16
Freon Return Temp. (K)         294.38     294.38     294.38     294.38
Heat Exch. Corr. (J/min)        -0.137     -0.137     -0.138     -0.133
Ambient Air Temp. (K)          298.65     298.65     298.65     298.65
```

```
RUN  7    TEST GAS - CO2                                   **
CONDITIONS FOR TEST INTERVAL     1          2          3

TEMPERATURE       Lower Inlet   451.49     451.78     451.88
  (K)             Outlet        313.63     313.68     313.69
PRESSURE          Inlet           8.721      8.724      8.727
  (MPa)           Outlet          8.407      8.405      8.405
                  Atmospheric     0.092      0.092      0.092

HEAT EXCHANGE (J/min)           400.350    401.826    401.828
HEAT LEAK (J/min)               -11.643    -14.300    -11.587
RATIO: LEAK OVER EXCHANGE       -0.0291    -0.0356    -0.0288
ENTHALPY CHANGE  Predicted   -12033.04-12026.43-12028.70
  (J/mol)        Measured    -11693.00-11615.16-11682.62

FLOW RATE  F-11  Pred. Mol.   16.55637 16.72483 16.61692
  (l/min) or     Meas. Mol.   16.09862 16.16934 16.15028
  (mmol/min)     Calc. Vol.    0.41208  0.41367  0.41367
                            --------------------
           CO2   Meas. Vol.    0.91322  0.92287  0.91676
                 Calc. Mol.   34.24182 34.80371 34.37454

Freon Sat'n Temp. (K)           294.28     294.28     294.28
Freon Meter Temp. (K)           298.34     298.38     298.34
Freon Return Temp. (K)          284.42     284.42     284.42
Heat Exch. Corr. (J/min)         -0.281     -0.282     -0.282
Ambient Air Temp. (K)           298.78     296.79     298.78
```

```
RUN  8    TEST GAS - CO2                                   **
CONDITIONS FOR TEST INTERVAL     1          2          3

TEMPERATURE       Lower Inlet   430.88     430.77     430.42
  (K)             Outlet        309.28     309.22     309.20
PRESSURE          Inlet           8.732      8.732      8.728
  (MPa)           Outlet          8.416      8.418      8.408
                  Atmospheric     0.093      0.093      0.093

HEAT EXCHANGE (J/min)           291.010    288.472    287.534
HEAT LEAK (J/min)               -12.213    -13.603    -18.568
RATIO: LEAK OVER EXCHANGE       -0.0420    -0.0470    -0.0646
ENTHALPY CHANGE  Predicted   -12380.89-12374.78-12382.92
  (J/mol)        Measured    -11882.20-11819.34-11803.88

FLOW RATE  F-11  Pred. Mol.   12.18934 12.18338 12.32186
  (l/min) or     Meas. Mol.   11.88838 11.83864 11.57470
  (mmol/min)     Calc. Vol.    0.29757  0.29800  0.28443
                            --------------------
           CO2   Meas. Vol.    0.84862  0.84862  0.86412
                 Calc. Mol.   24.48129 24.49138 24.81358

Freon Sat'n Temp. (K)           294.48     294.48     294.48
Freon Meter Temp. (K)           297.77     297.80     297.71
Freon Return Temp. (K)          294.54     294.54     294.54
Heat Exch. Corr. (J/min)         -0.078     -0.077     -0.077
Ambient Air Temp. (K)           295.54     295.54     295.54
```

```
RUN  9    TEST GAS - CO2                        **
CONDITIONS FOR TEST INTERVAL     1          2

TEMPERATURE       Lower Inlet   390.51     390.97
  (K)             Outlet        296.86     296.63
PRESSURE          Inlet           3.564      3.576
  (MPa)           Outlet          3.494      3.504
                  Atmospheric     0.093      0.093

HEAT EXCHANGE (J/min)            54.133     52.303
HEAT LEAK (J/min)                41.852     40.013
RATIO: LEAK OVER EXCHANGE         0.7684     0.7684
ENTHALPY CHANGE  Predicted    -2871.07  -2868.49
  (J/mol)        Measured    -11585.02-11067.14

FLOW RATE  F-11  Pred. Mol.    0.50173   0.51815
  (l/min) or     Meas. Mol.    2.17511   2.12864
  (mmol/min)     Calc. Vol.    0.05838   0.05410
                            --------------------
           CO2   Meas. Vol.    0.12318   0.42801
                 Calc. Mol.    4.57268   4.78018

Freon Sat'n Temp. (K)           294.48     294.48
Freon Meter Temp. (K)           298.80     298.86
Freon Return Temp. (K)          294.53     294.54
Heat Exch. Corr. (J/min)         -0.012     -0.014
Ambient Air Temp. (K)           295.54     296.54
```

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DATA FROM FILE C02.6

INTERVALS MARKED WITH ** WERE MOST STABLE IN RUN

RUN  1    TEST GAS - C02                           **
CONDITIONS FOR TEST INTERVAL         1             2

TEMPERATURE        Lower Inlet    391.26     391.53
  (K)              Outlet         298.31     298.35
PRESSURE           Inlet           3.536      3.536
  (MPa)            Outlet          3.288      3.289
                   Atmospheric     0.095      0.095

HEAT EXCHANGE (J/min)             73.401     73.584
HEAT LEAK (J/min)                 12.822     12.402
RATIO: LEAK OVER EXCHANGE         0.1747     0.1685
ENTHALPY CHANGE  Predicted      -4302.57   -4311.64
  (J/mol)        Measured       -5213.25   -5185.52

FLOW RATE  F-11  Pred. Mol.      2.43800    2.46326
  (l/min) or     Meas. Mol.      2.85524    2.85258
  (mmol/min)     Calc. Vol.      0.07328    0.07348
                                 --------------------
           C02   Meas. Vol.      0.38104    0.38387
                 Calc. Mol.     14.07872   14.19018

Freon Sat'n Temp. (K)             295.24     295.24
Freon Meter Temp. (K)             297.42     297.42
Freon Return Temp. (K)            295.29     295.29
Heat Exch. Corr. (J/min)          -0.017     -0.017
Ambient Air Temp. (K)             298.37     [illegible]


RUN  2    TEST GAS - C02                           **
CONDITIONS FOR TEST INTERVAL         1             2

TEMPERATURE        Lower Inlet    390.57     390.47
  (K)              Outlet         298.40     298.38
PRESSURE           Inlet           3.532      3.515
  (MPa)            Outlet          3.258      3.280
                   Atmospheric     0.095      0 095

HEAT EXCHANGE (J/min)             74.888     74.138
HEAT LEAK (J/min)                 14.053     14.278
RATIO: LEAK OVER EXCHANGE         0.1877     0.1926
ENTHALPY CHANGE  Predicted      -4243.96   -4246.54
  (J/mol)        Measured       -5232.07   -5259 48

FLOW RATE  F-11  Pred. Mol       2.44827    2.40981
  (l/min) or     Meas. Mol.      3.01403    2.98484
  (mmol/min)     Calc  Vol       0.07483    0.07410
                                 --------------------
           C02   Meas. Vol.      0.38898    0.38151
                 Calc  Mol.     14.30907   14.08589

Freon Sat'n Temp. (K)             295.21     295.21
Freon Meter Temp. (K)             297.14     298.87
Freon Return Temp. (K)            295.21     295.21
Heat Exch. Corr. (J/min)           0.000      0.000
Ambient Air Temp. (K)             298 08     298.09


RUN  3    TEST GAS - C02                           **
CONDITIONS FOR TEST INTERVAL         1             2

TEMPERATURE        Lower Inlet    380.21     379.84
  (K)              Outlet         298.56     298.49
PRESSURE           Inlet           3.535      3.526
  (MPa)            Outlet          3.475      3.467
                   Atmospheric     0.095      0.095

HEAT EXCHANGE (J/min)             60.277     60.277
HEAT LEAK (J/min)                 16.929     17.325
RATIO: LEAK OVER EXCHANGE         0.2808     0.2874
ENTHALPY CHANGE  Predicted      -3811.71   -3898.18
  (J/mol)        Measured       -5439.41   -5487.81

FLOW RATE  F-11  Pred. Mol.      1.74473    1.72878
  (l/min) or     Meas. Mol       2.42813    2.42813
  (mmol/min)     Calc. Vol       0.06046    0.06046
                                 --------------------
           C02   Meas. Vol.      0.28558    0.28411
                 Calc. Mol.     11.06150   11.02385

Freon Sat'n Temp. (K)             295.10     295.10
Freon Meter Temp. (K)             297.22     297.15
Freon Return Temp. (K)            295.21     295.21
Heat Exch. Corr. (J/min)          -0.032     -0.032
Ambient Air Temp. (K)             295.37     295.37
```

```
DATA FROM FILE MIX.1

INTERVALS MARKED WITH ** WERE MOST STABLE IN RUN

RUN  1    TEST GAS - TEST                                          **         **
CONDITIONS FOR TEST INTERVAL      1          2          3          4          5

TEMPERATURE         Lower Inlet   384.73     384.87     384.99     385.09     385.17
  (K)               Outlet        301.89     301.93     301.95     301.96     301.97
PRESSURE            Inlet          7.005      7.012      7.015      7.016      7.015
  (MPa)             Outlet         6.863      6.865      6.870      6.873      6.873
                    Atmospheric    0.083      0.083      0.083      0.083      0.083

HEAT EXCHANGE (J/min)             112.837    113.734    113.734    113.734    114.486
MEAS. ENTH. CHANGE (J/mol)      -3499.38   -3533.45   -3546.19   -3546.20   -3587.98

FLOW RATE  F-11   Meas  Mol.     4.52788    4.57197    4.57197    4.57197    4.60137
  (mmol/min)      Calc  Vol      0.11519    0.11631    0.11631    0.11631    0.11706
  or (l/min)                    --------------------
             TEST  Meas. Vol     0.85516    0.85517    0.85234    0.85233    0.85234
                   Calc. Mol    32.18784   32.18774   32.08122   32.08112   32.08122

Freon Sat'n Temp. (K)             294.48     294.48     294.48     294.48     294.48
Freon Meter Temp. (K)             297.93     297.94     297.94     297.94     297.90
Freon Return Temp. (K)            294.56     294.58     294.56     294.58     294.58
Heat Exch. Corr. (J/min)          -0.043     -0.044     -0.044     -0.044     -0.044
Ambient Air Temp  (K)             297.04     297.04     297.04     297.04     297.04


RUN  2    TEST GAS - TEST                     **         **
CONDITIONS FOR TEST INTERVAL      1          2          3          4

TEMPERATURE         Lower Inlet   372.53     372.78     373.88     375.11
  (K)               Outlet        300.47     300.47     300.53     300.58
PRESSURE            Inlet          7.067      7.070      7.070      7.074
  (MPa)             Outlet         6.828      6.833      6.828      6.823
                    Atmospheric    0.083      0.083      0.083      0.083

HEAT EXCHANGE (J/min)              88.140     87.044     89.888     91.431
MEAS. ENTH. CHANGE (J/mol)      -3272.08   -3231.37   -3268.75   -3263.71

FLOW RATE  F-11   Meas. Mol.     3.84314    3.49905    3.61883    3.87542
  (mmol/min)      Calc. Vol.     0.09014    0.08802    0.08201    0.09350
  or (l/min)                    --------------------
             TEST  Meas. Vol.    0.71500    0.71500    0.73057    0.73907
                   Calc. Mol.   28.83718   28.83706   27.52380   27.84387

Freon Sat'n Temp. (K)             294.48     294.48     294.48     294.48
Freon Meter Temp. (K)             297.03     297.23     297.38     297.48
Freon Return Temp. (K)            294.57     294.54     294.54     294.54
Heat Exch. Corr. (J/min)          -0.036     -0.025     -0.028     -0.026
Ambient Air Temp. (K)             296.78     296.78     296.78     296.78


RUN  3    TEST GAS - TEST         **
CONDITIONS FOR TEST INTERVAL      1          2          3          4

TEMPERATURE         Lower Inlet   419.13     418.28     418.17     419.87
  (K)               Outlet        303.07     303.05     303.09     303.23
PRESSURE            Inlet          7.029      7.029      7.032      7.038
  (MPa)             Outlet         6.780      6.783      6.778      6.778
                    Atmospheric    0.083      0.083      0.083      0.083

HEAT EXCHANGE (J/min)             137.134    133.477    135.671    138.231
MEAS. ENTH. CHANGE (J/mol)      -5080.82   -4925.87   -4910.18   -4946.53

FLOW RATE  F-11   Meas. Mol.     5.51281    5.38564    5.45382    5.58871
  (mmol/min)      Calc. Vol.     0.13024    0.13680    0.13875    0.14197
  or (l/min)                    --------------------
             TEST  Meas. Vol.    0.71842    0.71828    0.73340    0.74190
                   Calc. Mol.   25.89048   27.09720   27.82062   27.85059

Freon Sat'n Temp. (K)             294.48     294.48     294.48     294.48
Freon Meter Temp. (K)             297.20     297.18     297.13     297.07
Freon Return Temp. (K)            294.56     294.58     294.58     294.58
Heat Exch. Corr. (J/min)          -0.052     -0.051     -0.052     -0.053
Ambient Air Temp. (K)             296.78     296.78     296.78     296.78
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RUN  4  - TEST GAS - TEST                                  **
CONDITIONS FOR TEST INTERVAL           1          2

TEMPERATURE        Lower Inlet      421.83     422.42
   (K)             Outlet           303.65     303.71
PRESSURE           Inlet             6.974      6.977
   (MPa)           Outlet            6.883      6.885
                   Atmospheric       0.083      0.083

HEAT EXCHANGE (J/min)              185.769    147.728
MEAS. ENTH. CHANGE (J/mol)       -6243.88   -4888.39

FLOW RATE  F-11  Meas. Mol.        6.26216    5.93664
  (mmol/min)     Calc. Vol.        0.15910    0.15069
  or (1/min)                    ---------------------
            TEST Meas. Vol.        0.78438    0.78198
                 Calc. Mol.       29.70453   29.61388

Freon Sat'n Temp. (K)              294.52     294.52
Freon Meter Temp. (K)              296.47     296.50
Freon Return Temp. (K)             294.82     294.82
Heat Exch. Corr. (J/min)           -0.074     -0.070
Ambient Air Temp. (K)              295.65     295.65
```

```
RUN  5    TEST GAS - TEST                                **         **                    **
CONDITIONS FOR TEST INTERVAL       1          2          3          4          5

TEMPERATURE        Lower Inlet   399.73     399.55     399.57     399.55     399.55
   (K)             Outlet        302.50     302.51     302.51     302.51     302.51
PRESSURE           Inlet          7.001      7.001      7.001      7.001      7.001
   (MPa)           Outlet         6.713      6.713      6.713      6.710      6.706
                   Atmospheric    0.083      0.083      0.083      0.083      0.083

HEAT EXCHANGE (J/min)           122.134    122.134    122.500    123.331    121.789
MEAS. ENTH. CHANGE (J/mol)    -4114.55   -4114.54   -4141.61   -4151.50   -4102.22

FLOW RATE  F-11  Meas. Mol.     4.91001    4.91001    4.92471    4.95411    4.89831
  (mmol/min)     Calc. Vol.     0.12478    0.12478    0.12514    0.12588    0.12438
  or (1/min)                 ---------------------
            TEST Meas. Vol.     0.78437    0.78438    0.78154    0.78437    0.78436
                 Calc. Mol.    29.66350   29.66356   29.57637   29.66348   29.66356

Freon Sat'n Temp. (K)           294.52     294.52     294.52     294.52     294.52
Freon Meter Temp. (K)           296.84     296.82     296.82     296.81     296.81
Freon Return Temp. (K)          294.58     294.58     294.58     294.58     294.58
Heat Exch. Corr. (J/min)        -0.025     -0.025     -0.025     -0.025     -0.025
Ambient Air Temp. (K)           295.82     295.82     295.82     295.82     295.82
```

```
DATA FROM FILE MIX.2L

INTERVALS MARKED WITH ** WERE MOST STABLE IN RUN

RUN  1    TEST GAS - TEST          **                                          **         **
CONDITIONS FOR TEST INTERVAL        1         2         3         4         5          6

TEMPERATURE         Lower Inlet    417.67    418.03    418.62    420.27    421.73    421.92
  (K)               Outlet         302.81    302.87    302.97    303.18    303.42    303.54
PRESSURE            Inlet          .6.981     6.985     7.002     7.009     7.009     7.023
  (MPa)             Outlet          6.743     6.751     6.771     6.761     6.748     6.788
                    Atmospheric     0.093     0.093     0.093     0.093     0.093     0.093

HEAT EXCHANGE (J/min)              130.985   134.060   137.136   140.211   146.362   146.362
MEAS. ENTH. CHANGE (J/mol)        -4835.77  -5038.18  -5084.60  -4869.47  -5188.42  -5188.58

FLOW RATE  F-11   Meas. Mol.      5.26775   5.38142   5.51509   5.63875   5.88608   5.88608
  (mmol/min)      Calc. Vol.      0.13306   0.13617   0.13931   0.14244   0.14871   0.14871
  or (l/min)                  --------------------
             TEST Meas. Vol.      0.68505   0.69691   0.70650   0.73907   0.74190   0.74332
                  Calc. Mol.     26.53786  26.80872  26.97136  28.21446  28.31857  28.37280

Freon Sat'n Temp. (K)               294.70    294.70    294.69    294.69    294.69    294.68
Freon Meter Temp. (K)               298.32    298.33    298.34    298.37    298.40    298.40
Freon Return Temp. (K)              294.68    294.68    294.68    294.66    294.64    294.64
Heat Exch. Corr. (J/min)             0.012     0.012     0.010     0.024     0.036     0.036
Ambient Air Temp  (K)               295.09    295.09    295.09    295.09    295.09    295.09



RUN  2    TEST GAS - TEST                    **                  **
CONDITIONS FOR TEST INTERVAL        1         2         3         4

TEMPERATURE         Lower Inlet    432.46    432.18    432.47    432.87
  (K)               Outlet         305.66    305.66    305.73    305.79
PRESSURE            Inlet           6.988     6.988     6.988     6.988
  (MPa)             Outlet          6.613     6.611     6.606     6.606
                    Atmospheric     0.093     0.093     0.093     0.093

HEAT EXCHANGE (J/min)              174.038   177.113   174.038   177.113
MEAS. ENTH. CHANGE (J/mol)        -5160.81  -5201.89  -5111.66  -5155.52

FLOW RATE  F-11   Meas. Mol.      6.99807   7.12274   6.99807   7.12274
  (mmol/min)      Calc. Vol.      0.17582   0.17895   0.17582   0.17895
  or (l/min)                  --------------------
             TEST Meas. Vol.      0.88348   0.89194   0.89196   0.89481
                  Calc. Mol.     33.72290  34.04710  34.04720  34.15823

Freon Sat'n Temp. (K)               294.69    294.68    294.69    294.69
Freon Meter Temp. (K)               298.53    298.50    298.50    298.50
Freon Return Temp. (K)              294.68    294.68    294.68    294.68
Heat Exch. Corr. (J/min)             0.008     0.009     0.009     0.009
Ambient Air Temp. (K)               295.09    295.09    295.09    295.09



RUN  3    TEST GAS - TEST                              **
CONDITIONS FOR TEST INTERVAL        1         2         3

TEMPERATURE         Lower Inlet    426.35    425.02    425.59
  (K)               Outlet         304.63    304.57    304.52
PRESSURE            Inlet           7.026     7.028     7.028
  (MPa)             Outlet          6.718     6.718     6.718
                    Atmospheric     0.093     0.093     0.093

HEAT EXCHANGE (J/min)              155.585   155.585   157.122
MEAS. ENTH. CHANGE (J/mol)        -5049.27  -5049.27  -5153.41

FLOW RATE  F-11   Meas. Mol.      6.25708   6.25708   6.31892
  (mmol/min)      Calc. Vol.      0.15804   0.15804   0.15960
  or (l/min)                  --------------------
             TEST Meas. Vol.      0.80703   0.80703   0.79853
                  Calc. Mol.     30.61332  30.61332  30.48804

Freon Sat'n Temp. (K)               294.70    294.70    294.70
Freon Meter Temp. (K)               298.35    298.31    298.27
Freon Return Temp. (K)              294.66    294.66    294.67
Heat Exch. Corr. (J/min)             0.029     0.038     0.022
Ambient Air Temp. (K)               295.09    295.09    295.09
```

```
RUN  4    TEST GAS - TEST                                             **
CONDITIONS FOR TEST INTERVAL      1          2          3           4

TEMPERATURE.      Lower Inlet   418.32     418.87     417.42      417.45
  (K)             Outlet        302.93     302.99     303.05      303.08
PRESSURE          Inlet          7.002      7.002      6.989       6.985
  (MPa)           Outlet         6.763      6.763      6.761       6.761
                  Atmospheric    0.093      0.093      0.093       0.093

HEAT EXCHANGE (J/min)          134.083    137.138    137.138     140.213
MEAS. ENTH. CHANGE (J/mol)    -4937.10   -5030.34   -5020.42    -5112.75

FLOW RATE  F-11   Meas. Mol.   5.39142    5.51509    5.51509     5.63875
  (mmol/min)      Calc. Vol.   0.13523    0.13835    0.13835     0.14248
  or (l/min)                ----------------------
             TEST Meas. Vol.   0.71217    0.71500    0.71541     0.71925
                  Calc. Mol.  27.15424   27.26225   27.31509    27.42421

Freon Sat'n Temp. (K)           294.88     294.88     294.88      294.88
Freon Meter Temp. (K)           296.72     296.72     296.44      296.95
Freon Return Temp. (K)          294.64     294.64     294.66      294.68
Heat Exch. Corr. (J/min)         0.030      0.031      0.018       0.005
Ambient Air Temp. (K)           295.37     295.37     295.37      295.37
```

```
RUN  5    TEST GAS - TEST         **                                  **
CONDITIONS FOR TEST INTERVAL      1          2          3           4

TEMPERATURE       Lower Inlet   406.95     407.02     407.11      407.18
  (K)             Outlet        301.53     301.54     301.55      301.55
PRESSURE          Inlet          7.036      7.036      7.036       7.036
  (MPa)           Outlet         6.853      6.853      6.853       6.853
                  Atmospheric    0.093      0.093      0.093       0.093

HEAT EXCHANGE (J/min)          115.615    117.153    117.153     115.615
MEAS. ENTH. CHANGE (J/mol)    -5038.76   -5117.93   -5111.26    -5044.14

FLOW RATE  F-11   Meas. Mol.   4.64942    4.71128    4.71128     4.64942
  (mmol/min)      Calc. Vol.   0.11753    0.11909    0.11909     0.11753
  or (l/min)                ----------------------
             TEST Meas. Vol.   0.80238    0.80095    0.80173     0.80173
                  Calc. Mol.  22.94522   22.88072   22.82064    22.82075

Freon Sat'n Temp. (K)           294.87     294.87     294.87      294.87
Freon Meter Temp. (K)           297.05     297.11     297.09      297.08
Freon Return Temp. (K)          294.64     294.64     294.64      294.63
Heat Exch. Corr. (J/min)         0.019      0.020      0.020       0.025
Ambient Air Temp. (K)           295.54     295.54     295.54      295.54
```

Appendix 7 - Error Analysis

The precision of the results and the calculated heat leaks are estimated using the accuracies of the quantities which enter into these calculations. A summary of the primary equations involved in this estimation follows:

$$d(q_{leak}) = d(\Delta h_{pred}) - d(\Delta h_{meas}) \tag{A7.1}$$

$$d(\Delta h_{meas}) = dq_{meas} = d(-\lambda_r \cdot \dot{m}_r/\dot{m}_f) \tag{A7.2}$$

$$d(\Delta h_{meas}) = (-\dot{m}_r/\dot{m}_f)d\lambda_r - (\lambda_r/\dot{m}_f)d\dot{m}_r + (\lambda_r\dot{m}_r/\dot{m}_f^2)d\dot{m}_f \tag{4.1}$$

$$d(\Delta h_{pred}) = dh_{out} - dh_{in} \tag{A7.3}$$

$$dh = (j+fP)dT + (k+fT)dP \tag{A7.4}$$

[f,j, and k are well defined in terms of the]
[tabular values used in the interpolation]

The differential quantities in the equations above are approximated by small values representing the uncertainties in the corresponding variables. It should be noted that the uncertainties are "plus or minus" and use must be made of the case which gives the larger (i.e., more conservative) estimate of the uncertainty of the quantity of interest. Calculation of these uncertainties for several cases is presented below.

A. Determination of the uncertainty in the experimental value of enthalpy change - $d(\Delta h_{meas})$:

Examine equation (4.1) term by term.

1/ $$d\lambda_r = d\left[\frac{\lambda_r^+ - \lambda_r^-}{P^+ - P^-}\right](P_{sat} - P^-) + \lambda_r^-$$

$$= KdP_{sat} \quad \text{where } K = \left[\frac{\lambda_r^+ - \lambda_r^-}{P^+ - P^-}\right] = 15180 \text{ J/mol·MPa}$$

for ambient conditions

Since $dP_{sat} = 6.7 \times 10^{-6}$ MPa (0.05 torr) ,

$d\lambda_r = \pm 0.1$ J/mol

This interpolation is at least as accurate as linear interpolation of λ_r with the reciprocal of saturation pressure in the narrow range of ambient conditions. An approximate value for λ_r is 24850 J/mol.

2/ $d\dot{m}_r$ is equal to $d(a+bV) = bdV$ or to the maximum correlation error, whichever is larger. $dV = \pm 0.01$ volt.

For flow meter A, $b = 0.2019$ $g·min^{-1}·volt^{-1}$
Thus, $bdV = \pm 0.002$ g/min, but the maximum correlation error in the range used is about ±0.025 g/min.
Hence, for flow meter A,

$d\dot{m}_r = \pm 0.025$ g/min $= \pm 1.8 \times 10^{-4}$ mol/min

For flow meter B, $b = 1.6988$ $g·min^{-1}·volt^{-1}$
Thus, $bdV = \pm 0.017$ g/min, but the maximum correlation error is about ±0.1 g/min in the range used.
Hence, for flow meter B,

$d\dot{m}_r = \pm 0.1$ g/min $= \pm 7.3 \times 10^{-4}$ mol/min

3/ $$d\dot{m}_f = d(P\Delta V/zRTt)$$

$$= \frac{Pd(\Delta V) + \Delta V dP - P\Delta V dT/T - P\Delta V dt/t}{z \cdot R \cdot T \cdot t}$$

$d(\Delta V) = \pm 0.001\ ft^3 = 2.8 \times 10^{-5} m^3$ $\qquad$ $\Delta V = 5.5 \times 10^{-3} m^3$
$dP = \pm 6.7 \times 10^{-6}$ MPa $\qquad$ $P = 0.0933$ MPa
$dT = \pm 0.25\,^0F = \pm 0.14$ K $\qquad$ $T = 296.15$ K
$dt = \pm 0.01$ min $\qquad$ $t = 10.0$ min
$R = 8.314 \times 10^{-6}$ MJ/mol K $\qquad$ $z = 0.998$

Thus, $d\dot{m}_f = \pm 1.4 \times 10^{-4}$ mol/min

Having the above uncertainty estimates, $d(\Delta h_{meas})$ can be calculated from equation (4.1) for a number of cases:

a) $\dot{m}_f = 10$ mmol/min ; $\dot{m}_r = 1$ mmol/min (using N_2 and flow meter A)

$\underline{d(\Delta h_{meas}) = \pm 482 \text{ J/mol}}$

b) $\dot{m}_f = 35$ mmol/min ; $\dot{m}_r = 5$ mmol/min (using N_2 and flow meter A)

$\underline{d(\Delta h_{meas}) = \pm 142 \text{ J/mol}}$

c) $\dot{m}_f = 10$ mmol/min ; $\dot{m}_r = 1$ mmol/min (using CO_2 and meter A)

$\underline{d(\Delta h_{meas}) = \pm 482 \text{ J/mol}}$

d) $\dot{m}_f = 35$ mmol/min ; $\dot{m}_r = 20$ mmol/min (using CO_2 and meter B)

$\underline{d(\Delta h_{meas}) = \pm 575 \text{ J/mol}}$

It is clear that the use of higher process fluid flow rates results in lower uncertainties of the measured enthalpy changes. Case d) shows that this is negated by the use of flow meter B, made necessary by the higher flow rates of the reference fluid. Flow meter B has a higher range but also a higher correlation uncertainty.

B. Determination of the uncertainty in the predicted value of enthalpy change – $d(\Delta h_{pred})$:

Examine equations (A7.3) and (A7.4) term by term after deriving equation (A7.4). This equation arises as a result of a two - dimensional linear interpolation as follows:

$$h_{T^-,P} = \left[\frac{h_{P^+} - h_{P^-}}{P^+ - P^-}\right]_{T^-} \cdot (P - P^-) + h_{P^-,T^-} = a(P - P^-) + b$$

$$h_{T^+,P} = \left[\frac{h_{P^+} - h_{P^-}}{P^+ - P^-}\right]_{T^+} \cdot (P - P^-) + h_{P^-,T^+} = c(P - P^-) + d$$

$$h_{T,P} = \left[\frac{h_{T^+} - h_{T^-}}{T^+ - T^-}\right]_{P} \cdot (T - T^-) + h_{T^-,P} \qquad \left[\begin{array}{l}\text{a, b, c, d, and e are for}\\ \text{convenience only}\end{array}\right]$$

$$= \left[\frac{(c-a)(P-P^-) + (d-b)}{e}\right](T - T^-) + a(P - P^-) + b$$

Continuing from the previous page, two more convenience terms may be defined:

Let $f = \frac{c-a}{e}$ and $g = \frac{d-b}{e}$.

The enthalpy at some condition may then be expressed:

$$h_{T,P} = (f(P-P^-) + g)(T-T^-) + a(P-P^-) + b$$

Upon rearrangement,

$$h_{T,P} = (g-fP^-)T + (a-fT^-)P + fPT + (fP^-T^- - gT^- - aP^- + b)$$

With another three convenience terms defined:

$$j = (g-fP^-) \quad ; \quad k = (a-fT^-) \quad ; \quad l = (fP^-T^- - gT^- - aP^- + b)$$

the predicted value of enthalpy is written

$$h_{T,P} = jT + kP + fPT + l$$

and the derivative may be taken:

$$\underline{dh = (j+fP)dT + (k+fT)dP} \qquad \text{(A7.4)}$$

Applying this to both inlet and outlet conditions as given in equation (A7.3),

$$d(\Delta h_{pred}) = dh_{out} - dh_{in} \qquad \text{(A7.3)}$$

$$= [(j+fP)_{out} - (j+fP)_{in}]dT + [(k+fT)_{out} - (k+fT)_{in}]dP$$

Thus $d(\Delta h_{pred})$ is expressed as a function of the measurement uncertainties dT and dP. Several cases are now examined.

Case 1. Nitrogen Inlet : $T_1 = 401.36$ K ; $P_1 = 9.739$ MPa
Outlet : $T_2 = 301.80$ K ; $P_2 = 9.494$ MPa

Now $(j+fP) = g + f(P-P^-) = \frac{c-a}{e}(P-P^-) + \frac{d-b}{e}$

and $(k+fT) = a + f(T-T^-) = \frac{c-a}{e}(T-T^-) + a$

where $a = \left[\frac{h_{P^+} - h_{P^-}}{P^+ - P^-}\right]_{T^-}$; $b = h_{P^-,T^-}$;

$c = \left[\frac{h_{P^+} - h_{P^-}}{P^+ - P^-}\right]_{T^+}$; $d = h_{P^-,T^+}$; and $e = T^+ - T^-$

The calculations for case 1 follow:

Inlet

$a = \dfrac{2725 - 2735}{0.5} = -20\ \dfrac{\text{J/mol}}{\text{MPa}}$; $b = 2735$ J/mol

$c = \dfrac{4283 - 4289}{0.5} = -12\ \dfrac{\text{J/mol}}{\text{MPa}}$; $d = 4289$ J/mol

and $e = 450 - 400 = 50$ K

Thus, $(j+fP)_{in} = 31.1$ J/mol·K and $(k+fT)_{in} = -19.8\ \dfrac{\text{J/mol}}{\text{MPa}}$

Outlet

$a = \dfrac{-471 - (-448)}{0.5} = -46\ \dfrac{\text{J/mol}}{\text{MPa}}$; $b = -448$ J/mol

$c = \dfrac{1156 - 1171}{0.5} = -30\ \dfrac{\text{J/mol}}{\text{MPa}}$; $d = 1171$ J/mol

and $e = 350 - 300 = 50$ K

Thus, $(j+fP)_{out} = 32.5$ J/mol·K and $(k+fT)_{out} = -45.4\ \dfrac{\text{J/mol}}{\text{MPa}}$

Hence, $d(\Delta h_{pred}) = \pm(31.1+32.5)dT \pm (19.8+45.4)dP$

$= \pm 63.6\ dT \pm 65.2\ dP$

Since $dT = \pm 0.1$ K and $dP = \pm 0.01$ MPa (see App. 2),

$\underline{d(\Delta h_{pred}) \doteq \pm 7.0\ \text{J/mol}}$ for this case.

Case 2. Nitrogen Inlet : $T_1 = 389.57$ K ; $P_1 = 6.975$ MPa

Outlet : $T_2 = 300.51$ K ; $P_2 = 6.694$ MPa

Following the method of Case 1,

$(j+fP)_{in} = 31.0$ J/mol·K ; $(k+fT)_{in} = -26.5\ \dfrac{\text{J/mol}}{\text{MPa}}$;

$(j+fP)_{out} = 31.6$ J/mol·K ; and $(k+fT)_{out} = -51.8\ \dfrac{\text{J/mol}}{\text{MPa}}$

Thus, $\underline{d(\Delta h_{pred}) \doteq \pm 62.6\ dT \pm 78.3\ dP \doteq \pm 7.0\ \text{J/mol}}$.

Case 3. Nitrogen Inlet : $T_1 = 371.81$ K ; $P_1 = 3.524$ MPa

Outlet : $T_2 = 298.54$ K ; $P_2 = 3.199$ MPa

Similar to the above,

$(j+fP)_{in} = 30.1$ J/mol·K ; $(k+fT)_{in} = -33.9\ \dfrac{\text{J/mol}}{\text{MPa}}$;

$(j+fP)_{out} = 31.0$ J/mol·K ; and $(k+fT)_{out} = -58.8\ \dfrac{\text{J/mol}}{\text{MPa}}$

Thus, $\underline{d(\Delta h_{pred}) \doteq \pm 61.1\ dT \pm 92.7\ dP \doteq \pm 7.0\ \text{J/mol}}$.

The following cases are for CO_2:

Case 4. Inlet : $T_1 = 458.40$ K ; $P_1 = 9.759$ MPa

Outlet : $T_2 = 314.63$ K ; $P_2 = 9.403$ MPa

Similar to the above,

$(j+fP)_{in} = 52.6$ J/mol·K ; $(k+fT)_{in} = -177.6 \frac{J/mol}{MPa}$;

$(j+fP)_{out} = 359$ J/mol·K ; and $(k+fT)_{out} = -1144 \frac{J/mol}{MPa}$

Thus, $\underline{d(\Delta h_{pred}) \doteq \pm 411.6\ dT \pm 1321.6\ dP \doteq \pm 54.8\ J/mol.}$

Case 5. Inlet : $T_1 = 374.92$ K ; $P_1 = 5.102$ MPa

Outlet : $T_2 = 298.19$ K ; $P_2 = 5.008$ MPa

As above,

$(j+fP)_{in} = 49.6$ J/mol·K ; $(k+fT)_{in} = -301.2 \frac{J/mol}{MPa}$;

$(j+fP)_{out} = 93.0$ J/mol·K ; and $(k+fT)_{out} = -1043 \frac{J/mol}{MPa}$

Thus, $\underline{d(\Delta h_{pred}) \doteq \pm 142.6\ dT \pm 1344\ dP \doteq \pm 27.7\ J/mol.}$

Case 6. Inlet : $T_1 = 398.56$ K ; $P_1 = 3.499$ MPa

Outlet : $T_2 = 300.20$ K ; $P_2 = 3.118$ MPa

As above,

$(j+fP)_{in} = 45.9$ J/mol·K ; $(k+fT)_{in} = -242.3 \frac{J/mol}{MPa}$;

$(j+fP)_{out} = 50.4$ J/mol·K ; and $(k+fT)_{out} = -570.6 \frac{J/mol}{MPa}$

Thus, $\underline{d(\Delta h_{pred}) \doteq \pm 96.3\ dT \pm 812.9\ dP \doteq \pm 17.8\ J/mol.}$

Summarizing the above, it appears that the uncertainty in the prediction of the enthalpy changes is relatively constant for the cases using N_2, but for CO_2, the uncertainty increases with pressure of the run.

The appropriateness of the use of (two-dimensional) linear interpolation can be judged by comparing (j+fP) with C_p and (k+fT) with ϕ for a number of conditions T and P. Some comparisons are given below.

Nitrogen :

Conditions	(j+fP)	C_p	(k+fT)	ϕ
	[J/mol·K]		[J/mol·MPa]	
Case 1: Inlet	31.1	31.3	-19.8	-19.7
Case 2: Outlet	31.6	32.1	-51.8	-51.8
Case 3: Inlet	30.1	30.2	-33.9	-33.3

For carbon dioxide, the I.U.P.A.C. tables (Angus et al., 1973) give only the values of C_p and comparisons are made below.

Carbon dioxide	Conditions	$(j+fP)$ [J/mol·K]	C_p (from tables by interpolation) [J/mol·K]
	Case 4: Inlet	52.6	52.3
	Case 5: Outlet	93.0	85.1
	Case 6: inlet	45.9	45.9

It appears that linear interpolation is generally accurate for both N_2 and CO_2 in the regions of use. This was also tested by comparing the result of a cubic spline fit with the results of linear interpolation. Differences were negligible.

C. Determination of the uncertainty in the calculated values of the heat leak term - dq_{leak}:

Use equation (A7.1) and examine several cases.

$$dq_{leak} = d(\Delta h_{pred}) - d(\Delta h_{meas}) \qquad (A7.1)$$

For N_2, low flow rate : $dq_{leak} \doteq \pm 7 \pm 482 \doteq \pm 490$ J/mol
" " high " " " $\doteq \pm 7 \pm 142 \doteq \pm 150$ J/mol

For CO_2, low flow rate : $dq_{leak} \doteq \pm 30 \pm 482 \doteq \pm 510$ J/mol
" " high " " " $\doteq \pm 55 \pm 575 \doteq \pm 630$ J/mol

It should be noted that two different F-11 flow meters are used for the two CO_2 cases given above. This, unfortunately, obscures the trend seen in the N_2 cases showing that the uncertainty in this calculation decreases as process fluid flow rate increases.