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CAPSULE CALORIMETER DEVELOPMENT REPORT

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CAPSULE CALORIMETER DEVELOPMENT REPORTI. INTRODUCTION

The radioisotopes strontium-90 and cesium-137 are being recovered from the high level wastes that are generated during the irradiated fuels reprocessing operations in the 200 East Area of the Hanford complex. These long half-life radioisotopes will be converted to the solid strontium fluoride and cesium chloride salts, doubly encapsulated in metal containers and stored under water for a number of years. The conversion, encapsulation, and storage of these radioisotopes will be accomplished in the High Level Waste Encapsulation and Storage Facility (225-B Building) which is presently under construction.

In order to accurately evaluate the production rate, efficiency and accountability of the encapsulation processes, the amount of strontium-90 or cesium-137 that is contained in each capsule must be determined. To accomplish this task, a capsule calorimeter is needed that will measure the rate of thermal energy generation of each capsule. The energy generated by the radiolytic decay of a long half-life isotope is essentially constant during a measurement period and is directly proportional to the number of curies present. Therefore, the measured thermal energy emitted by a capsule can be used to calculate the total number of curies of an isotope that is contained in that capsule.

The application of calorimetry for radioisotopic assay is not new to the nuclear industry. However, most applications to date have been oriented towards precision laboratory type calorimeters for measuring the specific power of small transuranic and fission product sources (references 1, 2, 3, and 6).

The primary objective of this development program is to design, fabricate, and test a production type calorimeter that is capable of measuring the heat generation of capsule sources ranging from 300 to 1,000 watts of power, with an accuracy of plus or minus five percent on a routine basis. Several modes of operation were evaluated for accuracy and reproducibility of results over this specified range.

The capsule calorimeter development program was initially contracted to Pacific Northwest Laboratories (PNL) in conjunction with other development programs directly associated with the High Level Waste Encapsulation and Storage Facility. PNL designed and fabricated the capsule calorimeter vessel in accordance with the design criteria specified in ARH-1264 (reference 5). However, prior to design verification and testing, the calorimeter development program was transferred to the Atlantic Richfield Hanford Company for completion.

The design verification and testing of the capsule calorimeter was divided into two major segments or development phases. Phase I involved the design, selection, and procurement of auxillary equipment that was needed to complete the calorimeter system, equipment assembly, preliminary testing of the various operating modes, and modification of the calorimeter vessel and auxillaries. The second phase of development (Phase II) was directed towards calibration and evaluation of the different operating modes and towards refinement of the operating parameters and procedures.

II. SUMMARY

A capsule calorimeter has been designed, fabricated, and tested that is capable of measuring the rate of thermal energy generation of a capsule source with an accuracy of plus or minus five percent over a range of 300 watts to 1,000 watts of power. Three operating modes were investigated. The rate of temperature rise mode of operation was found to be the superior operating mode based on accuracy, time cycle, and simplicity of operation. This mode of operation is recommended for use in the encapsulation plant for performing calorimetric determinations on the strontium and cesium capsules that will be produced in the plant. The heat generation data collected by calorimetry will be used for isotopic assay of the capsule sources. The capsule calorimeter is specifically designed for installation into a remotely operated hot cell.

III. DISCUSSION

A. Theory of Radiometric Calorimetry

The application of radiometric calorimetry for radioisotopic assay requires the solution of two separate problems. The first problem is to develop the ability to accurately determine the energy generated by a capsule source, i.e., the fabrication and calibration of an adequate calorimeter. The second problem is to correlate the power output with the radioisotopic content of the capsule source. By knowing the

total weight, the isotopic distribution, the specific power of each isotope, and the total power output of the capsule source, it is then possible to calculate the total curies of each isotope contained in the capsule source.

The objective of this calorimeter development program is to solve the problem of adequately measuring the rate of thermal energy generation (power output) of a capsule source.

1. Modus Operandi

The capsule calorimeter system was designed for three different modes of operation, either of which can be used to perform a calorimetric determination on an unknown capsule source. The system can be operated as: (1) an adiabatic calorimeter where the rate of temperature rise of the calorimeter bath of known heat capacity is measured. The rate of temperature rise is proportional to the power output of the capsule source being investigated; (2) the system can also be operated as a isothermal calorimeter employing the method of steady state temperature measurement of the calorimeter bath which is correlated to the power output of the capsule source; and (3) the third operating mode, labeled the differential power mode, is a variation of the steady state temperature mode of operation. The power output of a capsule is determined by the difference in power output of electric heaters for two independent steady state temperature measurements.

These operating modes differ primarily in operating procedure, accuracy, and time cycle. These differences are emphasized in the following discussions for each of the operating modes.

a. Rate of Temperature Rise Mode of Operation

The rate of temperature rise mode of operation is a simple and straight forward method of performing a calorimetric determination. The capsule source is inserted into a calorimeter bath of known heat capacity. The rate of temperature rise of the bath is measured and the data is used to calculate the rate of thermal energy generation of the capsule source. The controlling equation for this mode of operation,

which relates the rate of temperature rise of the bath to the power generation of the capsule source, is discussed below.

General Energy Balance for the Calorimeter Bath

$$\left[\begin{array}{c} \text{Rate of Accumulation} \\ \text{of Energy} \end{array} \right] = \left[\begin{array}{c} \text{Rate of Energy} \\ \text{Addition} \end{array} \right] - \left[\begin{array}{c} \text{Rate of Energy} \\ \text{Loss} \end{array} \right]$$

Differential Equation:

$$\frac{dE}{d\theta} = Q_c - Q_{\text{loss}}$$

The accumulation of energy in the calorimeter bath is indicated by an increase in temperature and is numerically equivalent to the change in enthalpy of the calorimeter bath. For any specific calorimetric determination, the mass (M) and the specific heat (C_p) of the bath are constant.

$$MC_p \frac{dT}{d\theta} = Q_c - Q_{\text{loss}}$$

The total heat capacity of the calorimeter bath is equal to the summation of the individual heat capacities of the gamma attenuator, capsule basket, capsule source and water that are contained in the calorimeter bath*.

$$MC_p = \sum (MC_p)_i$$

Since the gamma attenuator, basket, and capsule have fixed heat capacities, the important operating variable is the mass of the water contained in the calorimeter bath. The mass of the water can be calculated by determining the volume and density of the water in the bath. The volume of water contained in the bath is determined

* The heat content of a capsule is dependent on the temperature of the capsule, which is a function of the temperature in the calorimeter bath [$Q_c = U A_c (T_c - T_b)$]. Therefore, the heat capacity of the capsule must be included in the controlling equation. Special care must be taken to quench the capsule in water prior to inserting it in the calorimeter bath.

by direct measurement of the liquid level using a float type liquid level indicator (see Figure 1). The temperature of the water in the bath is measured and the density of water as a function of temperature is determined.

The rate of energy loss (Q_{loss}) from the calorimeter bath due to conduction, convection, and radiation is a function of the temperature within the calorimeter bath. For a complicated calorimeter geometry, the heat loss is determined empirically by measuring the rate of cooling in the bath as a function of the temperature difference between the bath temperature and the ambient surroundings (see Figure 2-1). The heat loss is evaluated at the average bath temperature during the measurement period. Figure 2-1 is based on an air sparge rate of 0.5 l/minute, which is adequate for agitating the calorimeter bath and is sufficient for hydrogen dilution requirements. The heat loss contributed by the air sparge can be calculated if the initial and final thermodynamic properties of the air are known (see Figure 2-2). Comparing the Figures 2-1 and 2-2 indicates that the heat loss contributed by the air sparge is a small percentage of the total heat loss from the calorimeter bath.

Knowing the heat capacity and the rate of heat loss from the calorimeter bath, then the rate of thermal energy generation of an unknown capsule source can be directly calculated by measuring the temperature rise in the bath as a function of time (θ).

$$Q_c = \sum MC_p \frac{\Delta T}{\Delta \theta} + Q_{loss}$$

b. Steady State Temperature Mode of Operation

The steady state temperature mode of operation differs from the rate of temperature rise technique in that a cooling water flow system is incorporated for removing the heat that is generated by the capsule source. A dynamic equilibrium is established between the rate of energy generated by the capsule and the rate of energy removed by the cooling water. For any given set of operating parameters of inlet temperature and flow rate of the cooling water, the steady state temperature in the calorimeter bath can be directly correlated to the energy that is generated by the capsule source. The controlling equation for the steady state temperature mode of operation is discussed below.

General Energy Balance for the Calorimeter Bath:

$$\left[\begin{array}{c} \text{Rate of Accumulation} \\ \text{of Energy} \end{array} \right] = \left[\begin{array}{c} \text{Rate of Energy} \\ \text{Generation} \end{array} \right] - \left[\begin{array}{c} \text{Rate of Energy} \\ \text{Loss} \end{array} \right]$$

Differential Equation:

$$\frac{dE}{d\theta} = Q_c - Q_{\text{loss}}$$

The accumulation of energy in the calorimeter bath is indicated by an increase in temperature and is numerically equivalent to the change in enthalpy of the calorimeter bath. The energy removed from the calorimeter bath (Q_{loss}) is expressed in terms of the overall heat transfer coefficient (U_o), the heat transfer area (A_o), and the temperature gradient (ΔT).

$$\frac{d(MC_p T)}{d\theta} = Q_c - U_o A_o \Delta T$$

At steady state, the rate of accumulation of energy is equal to zero. Therefore, the mass of the calorimeter bath does not influence the steady state temperature, but it does influence the time interval required to reach steady state. Hence,

$$Q_c = U_o A_o \Delta T$$

The energy removed from the calorimeter bath is equivalent to the energy absorbed by the cooling water. Therefore,

$$Q_c = U_o A_o (T_{ss} - \overline{T_{cw}}) = \dot{M} C_p (T_{cwo} - T_{cwi})$$

This relationship suggests the development of a calibrated curve for predicting the power output of a capsule by measuring the steady state temperature in calorimeter bath at known parameters of flow rate and inlet temperature of the cooling water.

c. Differential Power Mode of Operation

The differential power mode of operation is a variation of the steady state temperature mode of operation previously discussed in subsection (b) of this report. The

steady state temperature mode of operation requires the development of a calibrated curve for predicting the power generation of an unknown capsule source based on the established steady state temperature. The differential power mode of operation, on the other hand, does not require a calibrated curve. However, the differential power mode does require two dynamic equalibria per calorimetric determination; whereas the steady state temperature mode only requires one dynamic equilibrium or steady state temperature measurement. The controlling equations for both operational modes are identical.

Initially, an unknown capsule source is introduced into the capsule calorimeter bath and a known amount of power is added to the calorimeter bath using the electric heaters. The combined power loads are allowed to attain a steady state temperature in the calorimeter bath. The capsule source is then removed from the calorimeter bath and the procedure is repeated using the electric heaters as the only power source. Power to the electric heaters is increased until the same steady state temperature is attained. Hence, the power output of the capsule source is equal to the difference in electrical power outputs of the two independent steady state temperature measurements. The variable operating parameters such as flow rates and inlet temperature of the cooling water are the same for both measurements.

Steady State No. 1:

$$Q_c + Q_{E_1} = U_o A_o (T_{ss_1} - \overline{T_{cw}}) = \dot{M} C_p (T_{cwo} - T_{cwi})$$

Steady State No. 2:

$$Q_{E_2} = U_o A_o (T_{ss_2} - \overline{T_{cw}}) = \dot{M} C_p (T_{cwo} - T_{cwi})$$

$$\text{Since } T_{ss_1} = T_{ss_2}$$

$$\text{Then } Q_c + Q_{E_1} = Q_{E_2}$$

$$\text{Therefore: } Q_c = Q_{E_2} - Q_{E_1}$$

B. Calorimeter Description

The capsule calorimeter apparatus is a multi-component system consisting of three primary assemblies: (1) the calorimeter vessel, (2) the flow system, and (3) the instrumentation network. The calorimeter apparatus is shown on the engineering flow diagram H-2-36770.

The calorimeter apparatus is specifically designed for remote operation and contact maintenance. The calorimeter vessel and flow system will be located in a man-entry hot cell and the instrumentation network will be located in the operating gallery of the 225-B Building (H-2-36926).

1. Calorimeter Vessel

The calorimeter vessel is 36 inches high, has a maximum outside diameter of 19-1/2 inches and weighs approximately 350 pounds. The calorimeter is assembled in two major sections (see H-2-36403). The inner container or calorimeter bath is inserted into the outer shell of the calorimeter vessel, forming an annular space between the two sections. A copper cooling coil, attached to the bottom flange, is inserted into the annular space.

The calorimeter bath is approximately nine inches in diameter by 30 inches in depth. When filled with water, the calorimeter bath provides a large heat capacity for absorbing the thermal energy that is generated by a capsule source. The large heat capacity tends to stabilize the system, making it less sensitive to rapid changes in temperature.

The calorimeter bath is agitated by an air sparge to maintain a uniform temperature throughout the bath. The air sparge also provides hydrogen dilution air that is required to maintain a non-explosive atmosphere within the calorimeter vessel. Hydrogen gas is evolved during radiolysis of the water at the rate of 4.0 cc's per watt hour of ionizing radiation (reference 8). The original design provided for a magnetic stirrer for agitating the calorimeter bath, but was determined to be inadequate during Phase I testing.

Escaping gamma rays emitted by the cesium capsules will be absorbed by the gamma attenuator that is contained inside the calorimeter bath (reference 7). The gamma attenuator is an annular configuration with a five inch inside diameter by one inch wall thickness. It is fabricated from lead

and weighs approximately 200 pounds. It is supported by a stainless steel collar suspended from the top of the calorimeter vessel.

Two Chromalox electric heater elements are immersed in the calorimeter bath, rated at 1025 watts each. These elements are essential for calibration and operation of the calorimeter system. They are replaceable by removing the lid assembly on the calorimeter vessel.

The calorimeter vessel is equipped with a removable basket which is used for inserting or removing a capsule source from the calorimeter bath. The basket is three and three-eighths inches in diameter by 27-1/2 inches long. It is fabricated from heavy gage wire mesh so as to allow free circulation of the water around the capsule source. The top of the basket has a lifting bail that is easily accessible using the remote manipulators.

2. Flow System

A cooling medium is required for removing heat from the calorimeter bath when employing the steady state temperature mode or the differential power mode of operation. Cooling water is pumped from the constant temperature bath, through a flow control valve and into the calorimeter vessel. The cooling water enters the vessel via the cooling coil and discharges into the annular space between the calorimeter bath and the shell of the vessel, thus providing a uniform temperature around the calorimeter bath. The warm cooling water exists at the top of the calorimeter shell, flows through a secondary heat exchanger and discharges back into the constant temperature bath.

The constant temperature bath (CTB) has a working volume of approximately 10 gallons of water which provides a mean residence time in the bath of approximately seven minutes at a cooling water flow rate of 1.5 gallons per minute. The bath is equipped with an 800 watt electric heater for maintaining the temperature of the cooling water at the set point. The temperature of the bath is measured by a thermocouple and compared to the set point on the temperature indicator-controller. If the temperature of the water is below the set point, power is added to the electric heaters. When the temperature of the bath is at the set point, the electric heaters are off. The CTB is agitated to maintain a uniform temperature of the water in the bath.

The discharge pressure of the pump and the pressure drop characteristics of the flow system as a function of flow rate are illustrated in Figure 4. At the maximum flow rate of 1.8 gallons per minute, the pressure drop through the flow system is equal to the discharge pressure of the pump.

The manual control valve is a calibrated variable orifice type with a manufacturer rating of plus or minus 1.5 percent of the indicated flow rate. However, this degree of accuracy was not demonstrated during the tests. The flow rate varied considerably for any specific set point on the control valve dial, as indicated by a variation in pressure drop through the flow system (see Figure 5). However, using the manual valve for flow control and the pressure gauge as a flow indicator did provide the accuracy of flow control necessary for satisfactory results.

The secondary heat exchanger has a design duty of 5700 Btu per hour. It is a tube and shell design, fabricated from 304-L stainless steel. The exchanger has an outside heat transfer area of 2.445 square feet. The secondary cooling water passes on the shell side of the heat exchanger, counter-current to the primary cooling water which passes on the tube side of the exchanger. The secondary cooling water flow rate is manually adjusted so as to maintain the exit temperature of the primary cooling water slightly below the set point temperature of the CTB.

3. Instrumentation Network

During the design verification and testing of the calorimeter apparatus, the heat generation of a capsule source is simulated using electric heaters that are immersed in the calorimeter bath. The desired power output of the heaters is manually regulated by a variable power supply consisting of a line voltage stabilizer, a variac regulator-controller, a current transformer, a watt transducer, and a watt meter. The watt meter has been calibrated at the standards laboratory and the calibrated curve is shown on Figure 6.

The temperature in the calorimeter bath is measured by three thermocouples of various lengths, which indicate the presence of temperature gradients that could result from insufficient agitation of the bath. Three additional thermocouples are provided for measuring the exit temperature of (1) the primary cooling water from the calorimeter vessel,

(2) the primary cooling water from the secondary heat exchanger, and (3) the calorimeter vent. The thermocouples were type J iron-constantan junctions that are accurate within one degree fahrenheit of the actual temperature. The temperature measurement circuitry also includes an ice-point reference junction, selector switches, two temperature recorders and a precision potentiometer.

All thermocouple signals are transmitted to the six-point recorder, which has a full scale range from zero to 10 millivolts. This recorder is used for preliminary temperature measurements that provide information relative to the operation of the calorimeter. Precision temperature measurements for analytical results are taken with the potentiometer or with the single pen trace recorder which has a full scale range from 1.07 to 3.41 millivolts (70° F - 150° F). The single pen trace recorder is connected to the intermediate thermocouple in the calorimeter bath. Any one of the six thermocouples can be measured with the potentiometer by changing the position of the selector switch.

The instrumentation control network also includes the temperature indicator-controller for the CTB which was discussed in the preceding section. On-off controls for the agitator and pump are also mounted on the instrument panel.

4. Calorimeter Test Apparatus

The calorimeter apparatus used during Phase I testing is illustrated in Figures 7-1 and 7-2. The modified calorimeter apparatus used during Phase II testing is illustrated in Figures 8-1 and 8-2.

Modifications to the calorimeter apparatus were necessary to reduce the rate of heat loss from the calorimeter bath and to provide an apparatus more suitable for remote manipulator operation.

C. Calibration Procedure

Basic differences in the calorimeter apparatus and ambient conditions that will exist in the hot cell will necessitate recalibration of the calorimeter system for each of the operating modes. The following calibration procedures should be followed.

1. Rate of Temperature Rise Mode of Operation

The first requirement in calibrating the rate of temperature rise mode of operation is to determine the rate of heat loss from the calorimeter bath as a function of the temperature difference between the bath temperature and the ambient cell temperature (see Figure 2).

Initially, the calorimeter bath is filled with water to a predetermined liquid level. The permissible operating range of the liquid level is shown on Figure 1. If the initial volume of water is too low, the liquid level will not rise above the gamma attenuator when the capsule is inserted into the bath. This condition will result in insufficient agitation and the formation of thermal gradients in the bath. If the initial volume of water is too high, the water will overflow the calorimeter when the capsule is inserted into the bath. The mass and heat capacity of the water is calculated and added to the known heat capacity of the gamma attenuator and the capsule basket. The use of a dummy capsule for the calibration procedure is optional.

The air sparge is turned on to 0.5 l/minute, and the calorimeter bath is heated to approximately 150° F using the calorimeter heaters. The power to the heaters is turned off, and rate of cooling of the calorimeter bath is measured using the simple pen trace recorder. This temperature data is used to graphically integrate the heat loss (Btu per hour) as a function of the temperature difference (ΔT_1). The controlling equation is indicated below.

$$\Sigma MC_p \frac{\Delta T_b}{\Delta \theta} = Q_E - Q_{loss}$$

$$\text{During Cooling: } Q_E = 0$$

$$\therefore Q_{loss} = -\Sigma MC_p \frac{\Delta T_b}{\Delta \theta}$$

$$\Delta T_1 = T_b - T_a$$

The degree of accuracy of the graphical integration is dependent on the magnitude of the interval $\Delta \theta$.

The degree of accuracy of a calorimetric determination using the rate of temperature rise mode of operation is established by the following procedure. The total heat capacity of the calorimeter bath is determined, as described in the preceding paragraphs. The power output of a capsule is simulated using the electric heaters. A known amount of power (Q_E) is added to the heaters as indicated by the watt meter. The bath is agitated by the air sparge at a rate of 0.5 l/minute. The rate of temperature rise ($\Delta T_b / \Delta \theta$) of the calorimeter bath is measured using the single pen trace recorder (see Figures 3-1 and 3-2). The heat capacity of the calorimeter bath is large as compared to the power output of the heaters (or capsules), so the rate of temperature rise is relatively slow. Therefore, the temperature lag in the thick walled gamma attenuator is negligible.

The ambient cell temperature (T_a) is measured and the temperature difference (ΔT_1) is determined. The heat loss (Q_{loss}) is estimated from the heat loss curve that was developed in the preceding paragraphs (Figure 2). The theoretical power output of the heater can then be calculated.

$$Q_T = \Sigma MC_p \frac{\Delta T_b}{\Delta \theta} + Q_{loss}$$

The degree of accuracy of the rate of temperature rise mode of operation is determined by comparing the theoretical power output of the heaters to the known power output of the heaters as indicated by the watt meter.

$$\% \text{ Error} = \frac{Q_E - Q_T}{Q_E} \times 100$$

2. Steady State Temperature Mode of Operation

The steady state temperature mode of operation requires the development of a calibrated curve for predicting the power output of a capsule source. The calibrated curve is developed by measuring the steady state temperature of the calorimeter bath as a function of power output of the heaters at fixed parameters of flow rate and inlet temperature of the primary cooling water.

Initially, the flow system and the inner bath are filled with demineralized water. The desired inlet temperature of the cooling water is selected on the constant temperature indicator-controller. The agitator and circulation pump are turned on and the desired flow rate of the primary cooling water is adjusted using the manual flow control valve. A known amount of power is added to the electric heaters in the calorimeter bath, as indicated by the watt meter. The sparge air is turned on at a rate of 0.5 l/minute.

When the temperature of the primary cooling water reaches the set point on the constant temperature controller, the cooling water to the secondary heat exchanger is turned on. This flow rate is adjusted until the exit temperature of the primary cooling water from the secondary heat exchanger is slightly below the set point.

The primary cooling water entering the CTB below the set point temperature is warmed by the electric heat until the set point temperature is attained. Optimum control of the CTB is established when the ratio of the heater-time-on to heater-time-off is equal to one. Insufficient cooling water to the secondary heat exchanger will cause the temperature of the primary cooling water to rise above the control set point. The CTB heaters will be off continuously. Excessive cooling water to the secondary heat exchanger will cause the temperature of the primary cooling water to drop well below the control set point. The CTB heater will be on continuously. Either extreme will result in an unstable system.

When the calorimeter bath has reached a steady state temperature, the bath temperature is measured and recorded using the single pen trace recorder or the potentiometer. This data point is plotted against the electrical power output of the heaters to form a calibrated curve as shown in Figure 9. This procedure is repeated for numerous power settings on the watt meter until the desired operating range of the calibrated curve has been investigated. Each calibrated curve that is developed is for a specific set of operating parameter of flow rate and inlet temperature of primary cooling water.

The accuracy of the calibrated curve is determined by performing multiple calibration checks. The power output predicted by the calibrated curve at the measured steady state temperature is compared to the known power output of the heaters, as indicated by the watt meter. The accuracy is determined by the maximum or minimum deviation.

$$\% \text{ Error} = \frac{Q_{wm} - Q_{cc}}{Q_{wm}} \times 100$$

Typical calibration curves are shown on Figure 9 for different parameters of flow rate and inlet temperature of the primary cooling water.

3. Differential Power Mode of Operation

The differential power mode of operation is a variation of the steady state temperature mode of operation. The controlling equations for the two operating modes are identical. Hence, the number of operating variables are also the same. Therefore, the degree of accuracy of the two operating modes should also be the same order of magnitude.

In order to simulate this mode of operation, a second variable power supply source would be required. Therefore, this operating mode was not investigated empirically. The operating procedure is similar to that described for the steady state temperature mode of operation, except a calibrated curve is not required. However, two steady state temperatures must be attained per calorimetric determination; hence, the time cycle would be doubled.

D. Calorimeter Performance

1. Rate of Temperature Rise Mode of Operation

The rate of temperature rise mode of operation is a simple and straight forward method of performing a calorimeter determination on an unknown capsule source. Approximately 45 to 60 minutes are required for this procedure, including preparation efforts. A typical data sheet for this mode of operation is shown in Appendix A. The data sheet and the corresponding strip charts from the temperature recorders will be turned over to the process engineer for evaluation.

Accuracies ranging from 2.5 percent to 5.0 percent have been routinely demonstrated with this mode of operation for heater outputs ranging from 1,000 watts to 300 watts, respectively. The primary sources of error are due to inaccuracies in measuring the following operating variables.

a. Heat Capacity

A 0.5 centimeter error in liquid level measurement is equivalent to 250 ml of water, or an error in heat capacity $(MC_p)_w$ of approximately 0.5 Btu/°F which is approximately one percent of the total heat capacity. The heat capacity of the gamma attenuator was calculated from the drawing details. A direct weight measurement of the lead annulus is required.

b. Temperature Measurement

An error of one degree fahrenheit per hour in measuring the rate of temperature rise could result in a maximum error of approximately 5.0 percent over the specified operating range.

c. Heat Loss

Error is introduced by estimating the rate of heat loss from the calorimeter bath as a function of the average temperature difference between the bath temperature and the ambient cell temperature. This temperature difference should not exceed 40° F so as to minimize the rate of heat loss from the calorimeter bath.

d. Watt Meter Reading

The watt meter dial has a full scale range from zero to 2,000 watts, graduated in increments of 50 watts. An error in reading the watt meter, equal to the width of the needle indicator, is equivalent to five watts or 1.6 percent of a 300 watt source.

e. Sparge Rate

The air sparge contributes to mass and heat loss from the calorimeter bath. The rate of heat loss due to sparging is accountable by empirical measurement. However, no attempt was made in the controlling equation to account for the loss of water from the calorimeter bath due to air sparging. For small sparge rates and moderate temperatures, the change in mass of the water is negligible.

2. Steady State Temperature Mode of Operation

The steady state temperature mode of operation requires approximately two and one-half hours to perform a calorimetric determination on an unknown capsule source. A typical data sheet for this mode of operation is shown in Appendix A. The data sheet and the corresponding strip chart from the temperature recorders are turned over to the process engineer for evaluation.

Accuracies ranging from four percent to eight percent have been routinely demonstrated with this mode of operation for heater outputs ranging from 1,000 watts to 300 watts, respectively. The primary sources of error are due to inaccuracies in measuring the following operating variables.

a. Liquid Level

The liquid level in the calorimeter bath determines the wetted perimeter or inside heat transfer area. A 0.5 centimeter error in liquid level measurement would have a negligible effect on the results.

b. Temperature Measurement

The calibrated curve developed for the steady state temperature mode of operation is based on measuring the absolute temperature in the calorimeter bath. An error in temperature measurement of one degree fahrenheit could result in a maximum error of 20 percent for a 300 watt power source (see Figure 9). Since the thermocouples are rated at plus or minus one degree fahrenheit, it is apparent that a new calibrated curve must be developed each time the intermediate thermocouple in the calorimeter bath is replaced.

c. Flow Rate

The flow rate of primary cooling water through the shellside of the calorimeter vessel effects the shellside film heat transfer coefficient (h_o) and the exit temperature (T_{cwo}), which influences the steady state temperature of the calorimeter bath. The effect of flow rate on the overall heat transfer coefficient (U_o) is shown on Figure 10. The flow rate through the shellside of the calorimeter vessel is highly laminar ($N_{Re} < 1000$). Therefore, the film heat transfer coefficient is slightly dependent on the temperature of the water; as predicted by the Colburn relationship (reference 4).

d. Sparge Rate

The air sparge contributes to the loss of heat from the calorimeter bath. This heat loss is accountable in the empirical determination of the calibrated curve, and is <0.1 percent of the total heat removed from the calorimeter bath.

The rate of air sparging effects the inside film heat transfer coefficient (h_i) in the calorimeter bath, which influences the steady state temperature in the bath. However, the controlling film resistance is the shellside film heat transfer coefficient (h_o). This is demonstrated by increasing the air sparge rate or degree of agitation in the bath with very little change in steady state temperature.

e. Constant Temperature Bath (CTB)

The constant temperature controller on the CTB is rated at plus or minus one degree fahrenheit of the set point temperature. This determines the inlet temperature of the primary cooling water to the calorimeter vessel and influences the steady state temperature in the calorimeter bath.

f. Watt Meter Reading

Inaccurate reading of the watt meter dial during calibration of the steady state temperature mode of operation introduced the same degree of error as was discussed for the rate of temperature rise mode of operation. However, the electric heater in the CTB also influences the power output of the calorimeter bath heaters. Both heaters were connected to the same power source. When the CTB heater was on, power to the calorimeter heater was reduced, as indicated by a slight deflection in the watt meter dial. This resulted in an unstable system that had a minor effect on the steady state temperature reading. Therefore, separate power supplies are needed for the CTB heater and the calorimeter bath heaters.

3. Differential Power Mode of Operation

As discussed in Section C, the differential power mode of operation was not empirically investigated. This mode of operation has the same operating variables and controlling equation as the steady state temperature

mode of operation. Therefore, the degree of accuracy of the two operating modes should be of the same order of magnitude. However, the differential power mode of operation requires two dynamic equilibria which would require approximately four hours to complete a calorimetric determination on an unknown capsule source.

VI. CONCLUSIONS AND RECOMMENDATIONS

Of the three operating modes considered in this development report, the rate of temperature rise mode of operation has demonstrated the highest degree of accuracy in performing a calorimetric determination. It is the only operating mode that consistently performed within the required accuracy of plus or minus five percent over the specified operating range from 300 watts to 1,000 watts of power. The time required to perform a calorimetric determination using this mode of operation (~ one hour) is several orders of magnitude less than the time required by the other operational modes.

The calorimeter apparatus required for the rate of temperature rise mode of operation is greatly simplified as compared to the apparatus required for the steady state temperature mode or the differential power mode of operation; i.e., the flow system described in Section B can be eliminated. The space requirement for the calorimeter apparatus in the cell can be significantly reduced. Therefore, it is recommended that the rate of temperature rise mode of operation be used as the only technique for performing a calorimeter determination, and only the equipment required for this mode of operation be installed in the encapsulation plant.

In the event that a market for the isotopes strontium-90 and cesium-137 becomes a reality, the accuracy of the calorimeter may have to be improved for measuring the power output of small capsule sources. Improvements in the existing calorimeter apparatus could include the installation of a digital watt meter for accurately measuring the power output of the calorimeter heaters and replacement of the thermocouples with thermohms for more accurate temperature measurements. These improvements in the calorimeter apparatus would cost approximately \$1,600.

The solutions of the controlling equations for the operating modes described in this report require rigorous hand calculations to determine the rate of heat generation of a capsule source and ultimately the number of curies of an isotope contained in the capsule source, based on the empirical data collected during the calorimetric determination (see Appendix A). The solutions of

these controlling equations are readily adaptable for computer application. The development of a computer program for the solution of these controlling equations is beyond the scope of this report, but should be considered as a future tool to assist the process control engineer.

V. ACKNOWLEDGEMENT

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APPENDIX ATYPICAL DATA SHEETRATE OF TEMPERATURE RISE MODE OF OPERATION1. General Information

- *a. Date:
- *b. Capsule Number:
- c. Feed Batch Number:
- d. Principle Isotope: Cesium-137

2. Specific Information

a. Capsule:

1. Total weight	16.72#
2. Weight of inner container with caps	4.54#
3. Weight of outer container with caps	5.51#
4. Weight of salt	6.67#

b. b. Ambient Cell Temperature	85° F
c. Initial Liquid Level of the Bath
Without Capsule and Basket	69 cm
d. Initial Temperature of the Bath	75° F
e. Sparge Rate	0.5 L/min
*f. Beginning Temperature (% of chart)
and Time	24.7%, 10:15 a.m.
*g. Final Temperature (% of chart) and
Time	35.2%, 10:45 a.m.

* Information to be marked on both temperature recorders

APPENDIX AEXAMPLE CALCULATIONRATE OF TEMPERATURE RISE MODE OF OPERATION

Part No. 1: To determine power output of capsule source

Controlling Equation:

$$Q_c = \sum MC_p \frac{\Delta T_b}{\Delta \theta} + Q_{loss}$$

a. Determine the total heat capacity ($\sum MC_p$)

MC_p of gamma attenuator = 6.178 Btu's/°F

MC_p of capsule basket = 0.572 Btu's/°F

MC_p of capsule: (from data sheet)

$$MC_{p_{CsCl}} = (6.67\#)(0.0433 \text{ Btu/\#}^\circ\text{F}) = 0.288 \text{ Btu's/}^\circ\text{F}$$

$$MC_{p_{316L}} = (10.05\#)(0.12 \text{ Btu/\#}^\circ\text{F}) = 1.206 \text{ Btu's/}^\circ\text{F}$$

MC_p of the water:

Liquid level (from data sheet) - 69 cm

Volume of water (from Figure 1) - 22.65 l

Temperature of water (from data sheet) - 75° F

ρ @ 75° F - 0.997 gm/ml

C_{p_w} @ 75° F - 0.999 Btu's/# °F

$$M = 49.74\#$$

$$MC_{p_w} = 49.69 \text{ Btu's/}^\circ\text{F}$$

$$\therefore \sum MC_p = \underline{\underline{57.93 \text{ Btu's/}^\circ\text{F}}}$$

b. Determine Rate of Temperature Rise
(from Data Sheet)

$$T_{b_1} = 24.7\% = 1.648 \text{ mV} = 90^\circ \text{ F}$$

$$T_{b_2} = 35.2\% = 1.893 \text{ mV} = 98.5^\circ \text{ F}$$

$$\Delta T_b = 98.5^\circ \text{ F} - 90^\circ \text{ F} = 8.5^\circ \text{ F}$$

$$\Delta \theta = 30 \text{ min} = 0.5 \text{ hrs.}$$

$$\Delta T_b / \Delta \theta = \underline{17^\circ \text{ F/HR}}$$

c. Determination Rate of Heat Loss

$$\bar{T}_b = \frac{90^\circ \text{ F} + 98.5^\circ \text{ F}}{2} = 94.25^\circ \text{ F}$$

$$T_a \text{ (from data sheet)} = 85^\circ \text{ F}$$

$$\Delta T_1 = \bar{T}_b - T_a = 94.25^\circ \text{ F} - 85^\circ \text{ F} = 9.25^\circ \text{ F}$$

(from Figure 2-1)

$$Q_{\text{loss}} \approx \underline{24 \text{ Btu's/HR}}$$

d. Calculate Power Output of Capsule Source

$$Q_c = (57.93 \frac{\text{Btu's}}{^\circ \text{F}}) (\frac{17^\circ \text{ F}}{\text{HR}}) + 24 \text{ Btu's/HR} = 1008.8 \text{ Btu's/HR}$$

$$Q_c = \underline{\underline{296 \text{ Watts} \pm 5\%}}$$

Note: If this technique were used for a SR capsule the error would be approximately $\pm 2.5\%$.

Part No. 2: To determine total curies in the capsule source

e. General Equation

1. for a single radioisotope:

$$[\text{KCi}]_I = \frac{Q_C / \eta}{Q_I} \bar{\pm} \%$$

2. for multiple radioisotopes:

$$[\text{KCi}]_T = \frac{Q_C / \eta}{\sum (Q_I \Psi_I)} \bar{\pm} \%$$

$$[\text{KCi}]_I = [\text{KCi}]_T \Psi_I \bar{\pm} \%$$

$Q_C \equiv$ measured power output of capsule source (watts)

$Q_I \equiv$ specific power of radioisotope (watts/KCi)

$\eta \equiv$ fraction of ionizing radiation absorbed

$\eta \equiv f(\text{Isotope})$

$\eta = 0.9775$ for Cs-137

$\eta = 1.0$ for Sr-90

$\Psi_I \equiv \frac{(\text{Ci/ml})_I}{(\text{Ci/ml})_T}$ from lab analysis of
liquid sample of feed batch

f. Compute The Total Curies in The Capsule Source

from (d): $Q_c = 296$ watts

assume pure cesium-137 in equilibrium with barium-137 from lab analysis; i.e., $\psi = 1.0$, $Q_I = 4.831$ watts/KCi, $\eta = 0.9775$

$$[KCi]_{Cs-137} = \underline{\underline{63 \pm 5\%}}$$

g. Compute The Purity of The Capsule Source

$$[WT]_I = \frac{[KCi]_I}{\mu_I} \pm \%$$

$$\text{Purity} = \frac{[WT]_I}{[WT]_T} \times 100 \pm \%$$

$\mu_I \equiv$ specific wt. of radioisotope (ci/gm) @ 100%

$[WT]_T \equiv$ total wt. from data sheet

$$\therefore [WT]_{Cs-137} = \frac{62.61 \text{ KCi}}{87 \frac{\text{ci}}{\text{gm}}} = 720 \text{ gms}$$

$$[WT]_{Cs-137} = 1.58 \text{ lbs} \pm 5\%$$

$$\text{Cs-137 Purity} = \frac{1.58 \text{ lbs}}{6.67 \text{ lbs}} \times 100 = \underline{\underline{24\% \pm 5\%}}$$

TYPICAL DATA SHEETSTEADY STATE TEMPERATURE MODE OF OPERATIONDIFFERENTIAL POWER MODE OF OPERATION (TWO REQUIRED)General Information

- *a. Date:
- *b. Capsule Number:
- c. Feed Batch Number:
- d. Principle Isotope: Strontium-90

Specific Information

- a. Capsule:
 - 1. Total weight 20.23#
 - 2. Weight of inner container with caps 5.21#
 - 3. Weight of outer container with caps 6.55#
 - 4. Weight of salt 8.47#
- b. Initial Water Level of the Bath
Without Capsule and Basket 69 cm
- c. Sparge Rate 0.5 l/min
- d. Corrected Watt Meter Reading (D.P.M. Only) _____
- *e. Set Point Temperature of CTB 90° F
- *f. Pressure Drop 24 psig
- *g. Steady State Temperature (% of chart) 41.5%
- h. CTB Heaters:
 - Time on 48 sec.
 - Time off 1 min. 20 sec.
- i. Time:
 - Initial 1:30 p.m.
 - Final 3:45 p.m.

*Information to be marked on both temperature records

SAMPLE CALCULATIONSSTEADY STATE TEMPERATURE MODE OF OPERATION

Part No. 1: To determine the power output of the capsule

a. Steady State Temperature Mode of Operation (S.S.T.M)

(from data sheet)

Temperature of CTB = 90° F
 $\Delta P = 24$ psig

Steady state temperature - 41.5% = 2.041 mV = 103.5° F

(from Figure 9)

@ 103.5° F, $Q_c = 3400$ Btu's/HR = 996 Watts $\pm$ 4%

b. Differential Power Mode of Operation (D.P.M)

(from part (d) of first data sheet)

$Q_{E_1} = 300$ Watts

(from part (d) of second data sheet)

$Q_{E_2} = 1300$ Watts

$\therefore Q_c = Q_{E_2} - Q_{E_1} = 1000$ Watts $\pm$ 4%

NOTES:

1. The first data sheet and the second data sheet for the D.P.M. will be identical except for part (d).
2. If this technique were used for a cesium capsule, the error would be approximately $\pm$ eight percent.

Part No. 2: Determine the total curies in the capsule source

- c. For the purpose of this example, assume the lab analysis indicate Sr-90 and Sr-89 isotopes.

$$\psi_{90} = .90$$

$$\psi_{89} = .10$$

$$[\text{KCi}]_T = \frac{Q_{c/\eta}}{\sum (Q_I \psi_I)}$$

$$\sum (Q_I \psi_I) = (6.75 \frac{\text{Watts}}{\text{KCi}})(.9) + (3.4 \frac{\text{Watts}}{\text{KCi}})(.1)$$

$$= 6.415 \text{ Watts/KCi}$$

$$\text{Total} = \frac{996 \text{ Watts}}{6.415 \text{ Watts/KCi}} = 155.29 \text{ KCi} \pm 4\%$$

$$\text{Sr-90} = (155 \text{ KCi}) (.9) = \underline{140 \text{ KCi}} \pm 4\%$$

d. Compute the purity of the capsule source

$$\begin{aligned} \text{WT. Sr-90} &= 140 \text{ KCi} / 142 \text{ ci/gm} = 984 \text{ gms} \\ &= 2.17 \text{ lbs.} \pm 4\% \end{aligned}$$

$$\begin{aligned} \text{Sr-90 Purity} &= 2.17 \text{ lbs.} / 8.47 \text{ lbs.} \times 100 \\ &= \underline{26\% \pm 4\%} \end{aligned}$$

APPENDIX B

FIGURES

VOLUME OF ER CAPSULE = 1.787 L
 VOLUME OF CS CAPSULE = 1.787 L

NOTES:

1) PERMISSIBLE LIQUID LEVEL
 OPERATING RANGE

2) LIQUID LEVEL RANGE AFTER
 CAPSULES ARE PLACED IN THE
 CALORIMETER BATH

FIG. 1

NOTE NO. 1

NOTE NO. 2

VOLUME VS. DEPTH

VOLUME
(LITERS)

DEPTH FROM BOTTOM (CM)

FIG. 2-2

NOTE: INSUFFICIENT DATA FOR
FOR HIGHER TEMPERATURE REGION TO
THEORETICAL RATE OF RISE

EXPERIMENT NO. 7
 3 COUPLES 1, 2, 3 6 FT. RECORD 87 °F / HR 3.00 °F / HR
 3 COUPLES 2 TRACE RECORDED
 3 COUPLES 4 EXIT AIR TEMP
 3 THEORETICAL 50.00 °F / HR

FIG. 8-1

FIG. 4

FIG. 5

FIG. 16

Figure 7-1

Figure 7-2

Figure 8-1

Figure 8-2

EXP. 5 27-30

90°F CTB 12 PSIG AP

90°F CTB 24 PSIG AP

POWER VS. STEADY STATE TEMPERATURE

110

100

90

80

70

STEADY STATE TEMPERATURE (°F)

Fig. 9

POWER (BTU/HR)

4000

3000

2000

1000

0

0

1000

2000

3000

4000

5000

6000

7000

8000

9000

VALVE LEGEND

- = GATE VALVE
- = BALL VALVE
- = NEEDLE VALVE
- = MANUAL CONTROL VALVE

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APPD FOR CONFORMANCE WITH DESIGN CRITERIA	DATE	U. S. ATOMIC ENERGY COMMISSION	
BY		RICHLAND OPERATIONS OFFICE	
FOR		Atlantic Richfield Hanford Company	
APPD		Richland, Washington 99352	
APPD J.C. Hall	5/1/72	E. F. D.	
ENGR J.L. HALL	5/1/72	CALORIMETER	
CPTG APPD		IN CELL "G"	
CHECKED		CLASSIFIED BY	DATE
DRAWN J.C. HALL	5/1/72	SLDS NO	225-E
SCALE		INDEX NO	7001
CLASSIFIED BY	DATE	DWG NO	H-2-36770
CLASSIFIED BY	DATE	SHEET NO	1
None		SHEETS	1

[illegible]

GENERAL NOTE
ALL ELECTRICAL CONNECTIONS
SPECIFIED ON THIS DWG. SHALL BE
IN ACCORDANCE WITH H-2-66477 &
H-2-66476

WEST WALL

SOUTH WALL

① ARRANGEMENT

H-2 67592	CELL ARMS-PLAN	G CELL
H-2 67593	CELL A-XG-ELE	G CELL
H-2 67046	FYP N CELL	N22 JOC
H-2 67045	N22 & DUAL	J-3 CELL
DATE REC. _____ DRAWING TITLE OR ORIGIN NO. _____		
REFERENCE DRAWINGS		
NEXT USED ON _____		

BY DATE	FOR	BY DATE	FOR	ADD	DESCRIPTION
APR 8 1968	CHAPMAN	APR 8 1968	CHAPMAN	BY DATE	
REVISIONS					
COMMENT PRT ISSUE NO.					DATE
DRAWING SPACE					DATE

APPRO. FOR DISSEMINATION NATL DEFENSE COUNCIL	DATE	U. S. ATOMIC ENERGY COMMISSION RICHLAND OPERATIONS OFFICE	
YES		Atlantic Richfield Hanford Company	
NO		Richland, Washington 99352	
APPRO. J. C. Mudd	7/2/50	CALORIMETER ARRANGEMENT G - CELL	
DISAP. J. C. Mudd	11/2/50		
DATE APPRO.			
REMARKS			
COPIES 1 - RAYFIELD 1 - L-10	7/2/50	CLASS. NO.	225-B
COPIES 1 - RAYFIELD 1 - L-10	7/2/50	SECURITY	8404
CLASS. NO.		SECURITY	
NONE		H-2-36926	-1-

APPENDIX C

DRAWINGS

UNCLASSIFIED

ARH-2619

APPENDIX D

DRAWING LIST

<u>DESCRIPTION</u>	<u>DRAWING NUMBER</u>
Calorimeter Assembly	H-2-36403
Calorimeter Details	H-2-36404 thru H-2-36406
Equipment Stands and Details	H-2-36407
Calorimeter Arrangement G-Cell	H-2-36926
Calorimeter Heat Exchanger	H-2-36927
Calorimeter Wiring Diagrams	H-2-36404 & H-2-36435
CTB Tank Modifications	H-2-36762
Tube Jumpers and Details	H-2-36763
Calorimeter E.F.D.	H-2-36770

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