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OMRE COOLANT PURIFICATION

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A DIVISION OF NORTH AMERICAN AVIATION, INC.

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OMRE COOLANT PURIFICATION

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ABSTRACT

The operation of a small batch vacuum distillation system built as an integral part of the Organic Moderated Reactor Experiment (OMRE) has demonstrated satisfactory separation of high boiling decomposition products from the organic coolant. Information gained in the operation of the batch system and data obtained in the related development program have been used to design a continuous flash distillation system presently being constructed at the OMRE.

I. INTRODUCTION

A singularly peculiar aspect of the organic reactor concept is the use of a moderator and coolant which undergoes radiation damage to the extent that decomposition products must be removed and an equivalent amount of fresh material added to the system. At first glance this may appear to be a serious disadvantage. However, the experience gained in operating the Organic Moderated Reactor Experiment (OMRE) and the results of related research and development programs verify that the replenishment operation does not impair the concept either technically or economically. In this report purification covers that phase of the replenishment operation in which the damaged material is removed from the reactor system.

A word or two, in general, concerning the degradation process is in order. The principal degradation process is a polymerizing reaction resulting in the formation of an organic mixture having a volatility considerably lower than the initial coolant. To a lesser extent, some products are formed which have volatilities higher than the fresh coolant; these are called low boilers. Another group of decomposition products have volatilities in the same range as the fresh coolant constituents and are retained as a part of the reclaimed coolant. A fourth group, accounting for approximately 1-3% of the decomposition products, are light hydrocarbon gases and hydrogen. In the case of the OMRE these gases are removed by a continuous nitrogen purge system; more advanced organic reactor designs provide a degasifier system. Purification as covered in this report deals with the largest of these four groups of decomposition products, namely the polymerized material of lower volatility than the initial coolant. This material is generally referred to as high boiler or HB.

Undoubtedly, there are a number of ways in which the high boiler fraction might be removed from the coolant. To date, Atomics International has only given active consideration to the vacuum distillation method. Since there is considerable difference in the volatilities of the high boiler and the undamaged coolant, vacuum distillation lends itself so well as a separation technique that there is little likelihood that any other method will prove more advantageous.

With the decision to construct and operate an organic moderated reactor experiment, several supporting research and development programs were set up. One of these was a distillation study to parallel and support the OMRE Purification System throughout its design, construction and operation phases. During OMRE design this project aided in establishing the Purification System flowsheet and proved the operability of special equipment. Later, as the OMRE was put into operation, it provided useful supplementary information. More recently, it has provided data for modification of the OMRE Purification System, changing the system from a batch operation to a continuous process. In addition, the project has initiated studies of high boiler fractionation.

II. THE OMRE PURIFICATION SYSTEM

When the design of the OMRE was initiated, there were practically no data available upon which to base the design of the purification system. However, it was concluded that in all probability the separation would be very similar to that required for the separation of commercial terphenyls from pyrolytic tars. The commercial process employs a single-plate distillation carried out at a pressure of 30 to 50 mm Hg. In October 1955, using a sample of irradiated eutectic mixture of diphenyl and ortho-terphenyl which was produced in the recently completed MTR in-pile experiment NAA-18, the first distillation of the irradiated material was performed. This laboratory glassware test substantiated the similarity to the commercial process.

A. DESCRIPTION OF THE OMRE PURIFICATION SYSTEM

The original OMRE purification system was a small, simple, batch-type distillation process. It has undergone considerable evolution until it is now a combination flash and batch system.

The essential components of the system as it exists today, include the still, a distillate hold tank, a condensing system, a vacuum system with vapor traps, and supply tanks. Figure 1 shows a schematic flow diagram and Figure 2 is a photograph of the system.

The still supply tank, as the name implies, feeds material into the distillation system. It is a 300-gallon vessel which receives the irradiated coolant from the reactor and feeds into the still. A 1/4-inch globe valve in the line between these two vessels is used to control the still feed rate. The vacuum still is a 200-gallon vertically-mounted tank provided with four thermo-siphon legs spaced radially 90 degrees apart. These legs are 4-foot lengths of 1 1/2-inch pipe extending down from the bottom of the still tank body. They are connected to a common centrally located cold leg. The four thermo-siphon legs are heated by electrical clam shell heaters. The still body is heated by electrical induction circuits characteristic of the OMRE pre-heat system. The choice of thermo-siphon heating for the still was prompted by the need to preclude the possibility of unsteady boiling usually encountered in boiling at low pressure in pot type vessels.

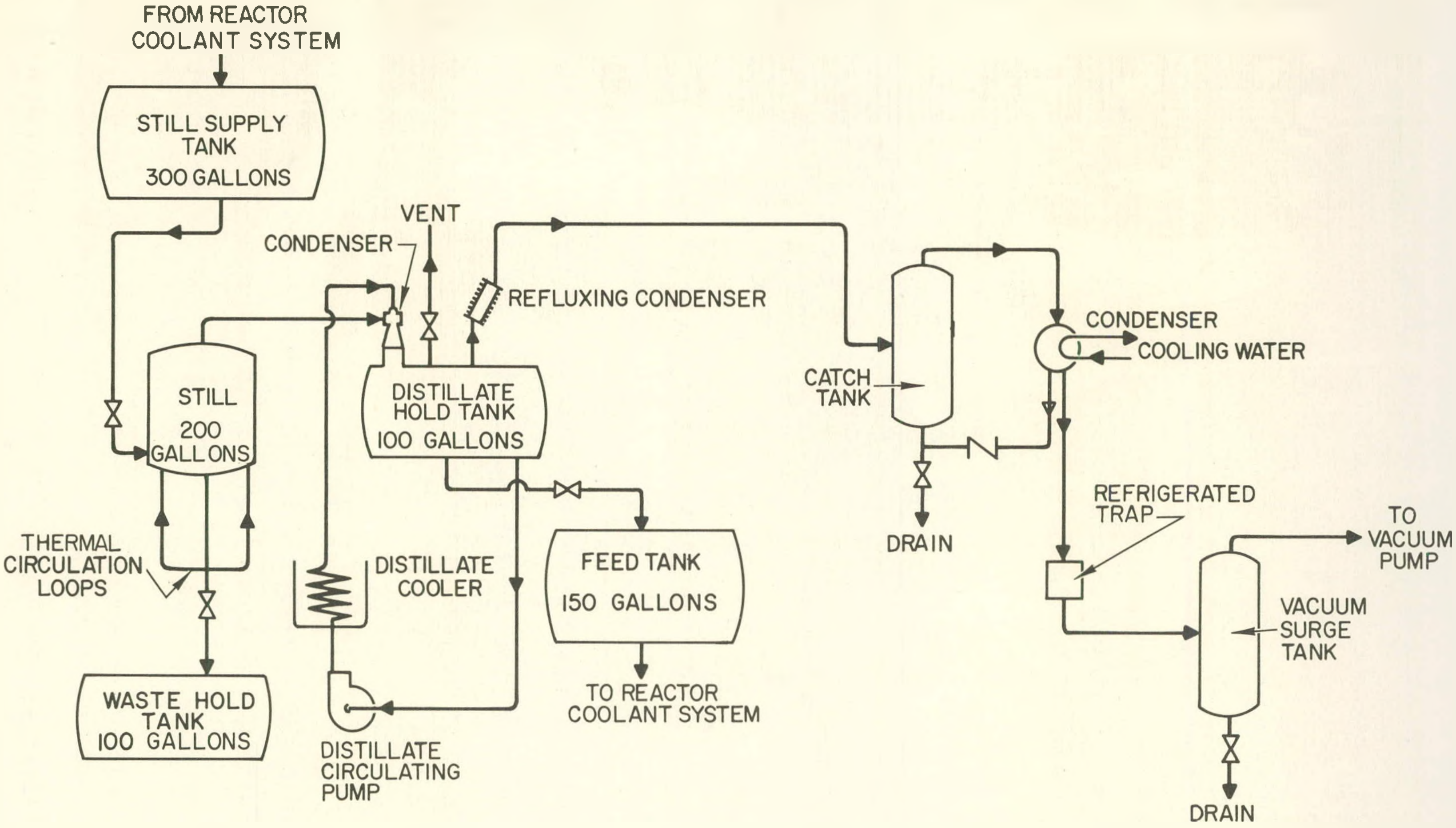


Figure 1. Schematic Flow Diagram of the OMRE Purification System

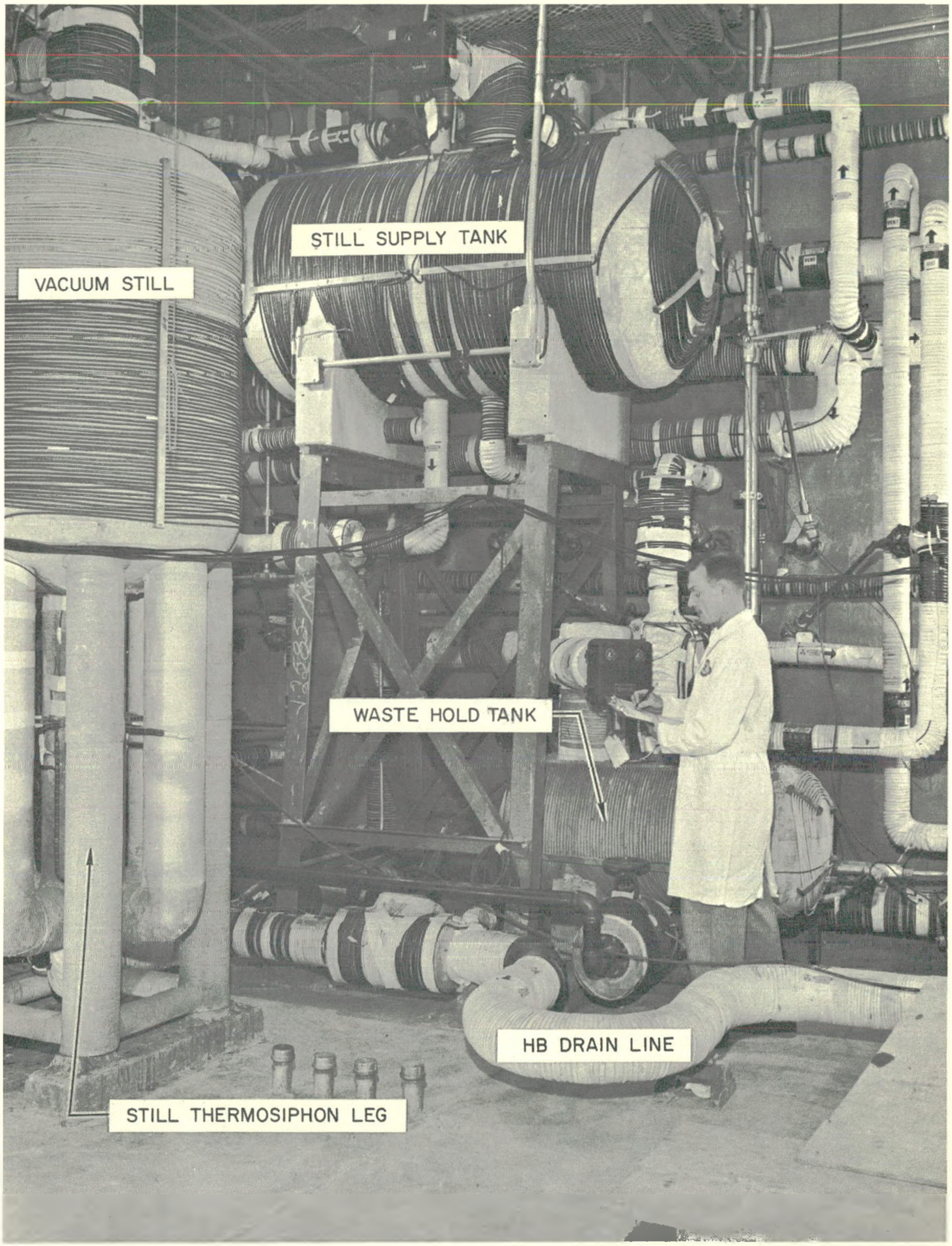


Figure 2. Photograph of the OMRE Purification System

The vapors are directed from the still through a 4-inch overhead line to the condenser which is mounted directly above the distillate hold tank. The condenser is a direct contact type in which the vapors are quenched with a spray of cooled distillate. This type of condenser was selected to prevent freezing of any distillate fractions in the condenser. Cooled distillate is supplied to the unit by a canned rotor pump, located 16 feet below the distillate hold tank. The distillate is pumped from the distillate hold tank, through the condenser and back into the hold tank. Heat is rejected from the system through a cooler located between the distillate recirculating pump and the condenser. The cooler consists of a coil immersed in a tank of water with the organic material flowing in the coil.

The distillate hold tank is a horizontally-mounted 100-gallon vessel. This tank provides storage for the recirculating cooled distillate. During the course of a run, the quantity of material in the condenser circuit accumulates. At the completion of the run the distillate is routed to the feed tank which feeds the reactor coolant system. A heel of 15 gallons is retained as condenser quenching fluid for the next run.

Vacuum is maintained by means of a mechanical vacuum pump. There are several traps in the line from the distillate hold tank to the vacuum pump to prevent the vacuum pump oil from becoming fouled by condensed organic vapors. The first trap is essentially a refluxing condenser followed by a knock-out or catch tank, then by a water cooled condenser, next by a refrigerated trap reducing the temperature of the vapors to -5°F, and finally by a vacuum surge tank (See Fiqure 1).

HB remaining in the still after the run is discharged to the waste hold tank (the system is first vented to atmospheric pressure). From the waste hold tank the HB is loaded into 55 gallon drums.

B. INFORMATION LEARNED FROM OMRE PURIFICATION SYSTEM OPERATION

The separation of the high boiler fraction from the undamaged portion of the coolant is relatively simple operation - much simpler in fact than the early organic reactor concepts envisioned. OMRE purification experience has shown that a simple batch distillation performs an entirely adequate separation. Also, operation of the OMRE Purification system has pointed up the need for a much higher processing capacity than was originally thought necessary and the need for special means of handling low boilers. Happily, the purification system can perform one function for which it was not intended originally -- this is to decontaminate the coolant.

1. Adequate Separation with a Single Stage System

The one technical aspect that results in the purification being a relatively simple operation is a very favorable vapor-liquid equilibrium. This is illustrated by the equilibrium diagram for Santowax OM and HB shown in Figure 3. This plot is based on the assumption that the irradiated coolant behaves as a binary mixture of Santowax OM and HB and that HB has a molecular weight of 460. The curve was developed using Raoult's and Dalton's laws. HB volatility data were calculated from experimental Santowax R - HB equilibrium data using the same two laws.[1] To illustrate how favorable the diagram is, consider a 30% liquid coolant mixture at its boiling point under a pressure of 20 mm Hg. This particular composition corresponds to a Santowax OM mole fraction of 0.83. The overhead product would have a mole fraction composition of 0.999 and the underflow composition from the stage below would be 0.05 mole fraction.

The above illustration is for a continuous single stage system and does not represent the OMRE system exactly. As originally intended, the OMRE purification system was to operate as a simple batch differential distillation process (the composition of the pot contents and the composition of the vapors are undergoing continuous change). Simple batch differential distillation follows the Rayleigh equation which is

$$\ln \frac{W}{W_o} = \int_{x_o}^{x} \frac{dx}{y-x}$$

where W refers to the moles of material in the still and x and y are the mole fractions in the liquid and vapor phases respectively. The subscript "o" refers to conditions at the beginning of a run. Solution of the equation generally requires a graphical integration since x-y data are usually available in graphical form only. For the Santowax OM - HB system, the solution has been obtained.

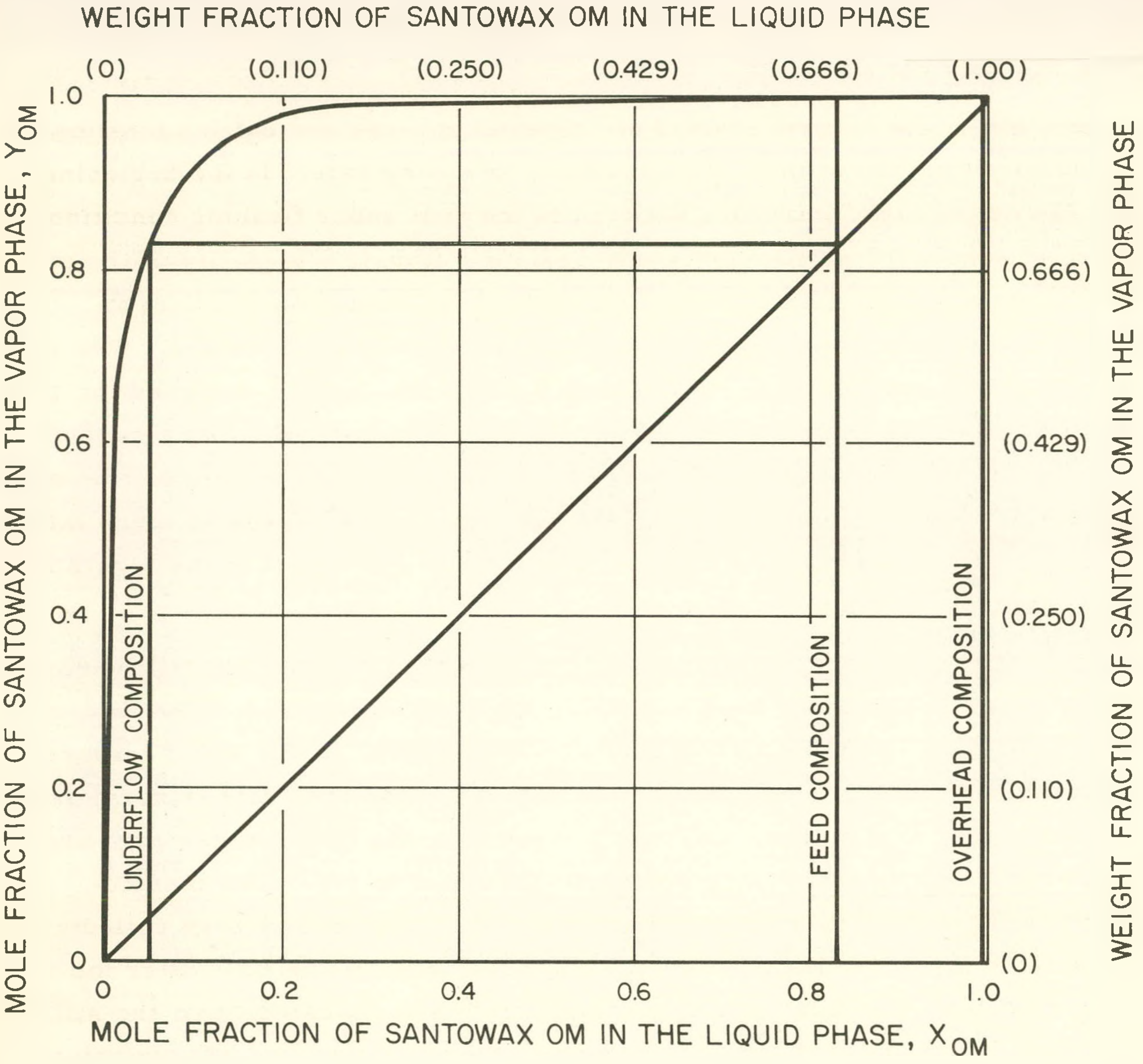


Figure 3. Equilibrium Diagram of Santowax OM and HB at a Pressure of 20 mm Hg

For easier interpretation, results are presented in terms of weight percent rather than mole fraction and are shown in Figure 4. Agreement with actual OMRE still cut-off temperature data is very good; the final still bottoms composition usually contains approximately 5% Santowax OM after 70% of the initial charge has been distilled.

Adoption of the current method of combination flash and batch operation, was motivated by the need to increase the processing rate. In the beginning, there was no intent to introduce the feed to the still under flashing conditions since it was thought that flashing would seriously impair the quality of the product. However, in an effort to improve methods of operation to effect an increase in capacity, flashing was tried with extremely good results. The product quality was not impaired even though the still was never designed for such operation and the flashing rate could only be coarsely controlled by a manual valve with the only knowledge of the feed rate indicated by the rate of rise of the liquid levels in the still and the distillate hold tank. The reason so much latitude in operation was possible can now definitely be attributed to the favorable vapor-liquid equilibrium.

In the current method of operation, the coolant is fed into the still under flashing conditions with the feed rate being manually controlled. The limiting rate is determined by the capacity of the vacuum pump. If the still pressure rises much above 20 mm Hg, the rate is reduced. The feed rate is normally about 100 lb/hr. The amount flashed, a function of the feed temperature and the still temperature, may vary somewhat from run to run. The thermosiphon heating system is actuated when sufficient material has been collected in the still to cover the legs, which usually occurs about one hour after the start of the run. The flashing portion of the run is terminated when the still supply tank is emptied. From this point on, the operation is a simple batch differential distillation. The run is terminated when a 510°F overhead temperature is reached and maintained for 30 minutes.

2. Lack of Sufficient Processing Capacity

One of the principal reasons for selecting a batch design was to give flexibility in processing rate. The capacity of a batch system is simply a function of the size of the batch and the number of batches processed in a given

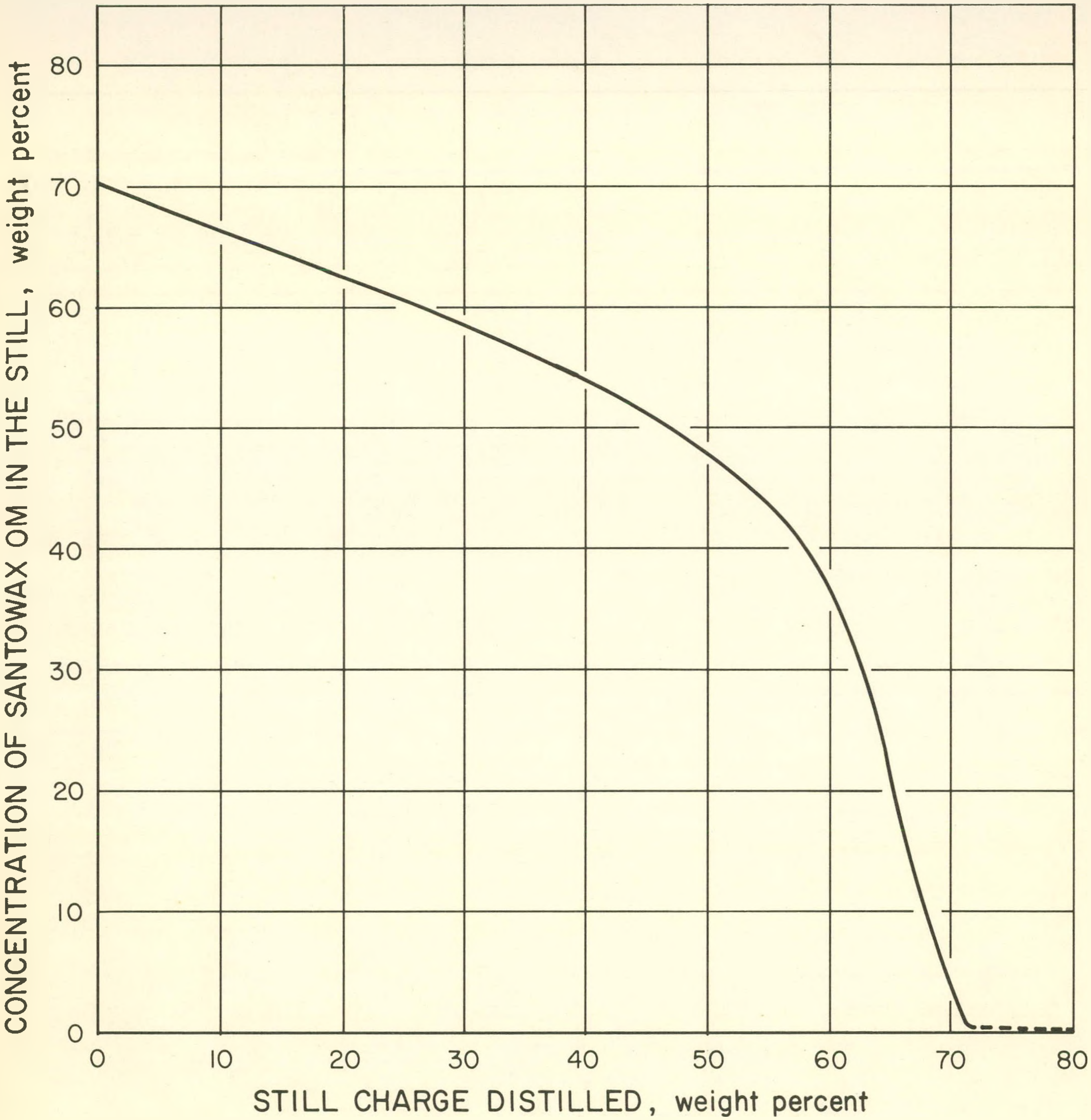


Figure 4. Calculated Still Pot Composition During a Simple Batch Distillation of Irradiated Santowax OM Determined from the Rayleigh Equation

period of time. Originally it was anticipated that the purification system would need to process 586 lb/batch of 30% coolant and only one batch per day. If the through-put had to be increased, the number of batches processed per day would be increased. Nevertheless, the OMRE purification system capacity proved to be inadequate, particularily when the reactor was operated at an HB concentration of 10%. Radiation decomposition data for the particular conditions and coolant mixture to be used in the OMRE were lacking at the time design was in progress; G_p was estimated to be 0.03 molecules per 100 ev of energy absorbed in the coolant and percent energy absorbed was estimated to be 6%. Both of these estimated values proved to be low. For OMRE conditions and 30% HB coolant, G_p is 0.08 and the percent energy absorbed is 7.8%.[2] To cope with the need for increased processing rate, several modifications were made in equipment in addition to adopting a flashing procedure.

The capacity of the thermo-siphon heating system is 12 1/2 kilowatts. This is the capacity-determining factor when the system is operated as a true simple-batch system. With the introduction of the flashing mode of operation, reactor heat is utilized since the coolant is not allowed to drop below its flash temperature. By adopting the flashing procedure, the thermo-siphon heating system was no longer the rate limiting factor. Instead, the heat rejecting system or the still cooler became limiting. As mentioned in the previous section, the still cooler is a coil immersed in a tank of water. Originally, it was intended that the water be maintained at its boiling point since this would serve as a simple temperature regulating technique. With the need to increase the processing rate, the coil was increased in size and the temperature of the water was reduced below its boiling point to increase the heat transfer rate.

Another aspect which tended to reduce the capacity of the system was the time required to let the system down to atmospheric pressure before the distillate hold tank could be discharged to the feed tank. The system was modified to allow transfer under vacuum conditions. This was accomplished by utilizing the distillate recirculating pump, which has a discharge pressure above atmospheric, to pump a portion of recirculating coolant into the feed tank.

The vacuum pump also tended to limit the capacity of the system. The original vacuum pump was a 13 cfm displacement unit. Although adequate for the original design conditions, it proved to be undersized when the flashing method of operation was adopted. To overcome this difficulty, a 47 cfm displacement pump was installed.

With the combination of all these equipment modifications and the change to a flashing process for the initial part of the run, the capacity of the system was increased to meet reactor demand. Being a batch and non-automatic system, it requires constant attention and is wasteful of manpower. To improve this aspect of the OMRE, a continuous system is being provided. The improved system is discussed in detail later in this report.

3. Trapping of Volatile Components

The trapping system, to prevent the hot polyphenyl materials from reaching the vacuum pump while preventing these high freezing point materials from plugging the vacuum lines, has been a constant problem. Originally there was one vapor trap for removal of vapors from the gas stream drawn from the distillate hold tank by the vacuum pump. This trap consisted of an inclined helical finned pipe with proper temperature control to enhance refluxing of liquid material at the entrance portion of the trap and to freeze the remaining material at the exit portion. Periodically, the exit portion would become plugged, requiring the trap to be isolated from the vacuum pump and heated to permit the frozen deposit to melt and flow back into the distillate hold tank. Although a mock-up of the trap proved satisfactory in laboratory tests and the actual unit operated satisfactorily during the shakedown tests, it proved inadequate in actual operation because of the presence of the low boiler fraction. The presence of a low boiler fraction was undetected until initiation of distillation of irradiated coolant on an extensive scale; all early laboratory work indicated that the low boiler fraction was either non-existent or of insignificant quantity.

It was found the presence of low boilers tended to keep the material condensed in the trap in the liquid state thus making the freezing point problem less important than originally estimated. The low boiler fraction was analyzed to contain 12.7% benzene, 15.6% toluene, 18.0% ethyl benzene, with the remaining 53.7% containing xylene, trimethyl benzene, n-propyl, butyl benzene, indane and possible other similar compounds. The mixture of low boilers and diphenyl was continually drawn over into the vacuum pump, fouling the vacuum pump oil.

After many modifications, the trapping system has been made to operate satisfactorily. The system is now a train consisting of the original helical finned trap, a knock-out or catch tank, a small water cooled heat exchanger and finally a refrigerated trap (See Figure 1).

4. Distillation as a Decontamination Method

Operation of the distillation system has shown it to be an effective means of removing metallic inorganic materials from the coolant. Non-metals are not so effectively removed. Recent data[2] on OMRE still decontamination factors are listed in Table I. Most of the radioactivity in the coolant has been found to be the result of activation of impurities introduced with the fresh coolant or introduced into the coolant from rust, welding slag or metal particulates remaining in the pipes and vessels since the construction of the system. During the early period of reactor operation, efforts to remove these impurities by filtration proved ineffective because of mechanical difficulties in operating filters at the temperature and pressure conditions of the reactor. In these circumstances, the still proved to be of great value in removing these impurities. If circumstances are such that the entire coolant charge can be processed before reintroduction into the primary reactor circuit, removal is very effective. When conditions require that each batch of distillate be immediately returned to the reactor system, the concentration of non-volatile radioactive impurities approaches zero asymptotically as the still operates for an infinite period of time. Thus, the effective decontamination factor for the entire reactor coolant charge is relatively small since actual practice requires termination of such a mode of operation after a few weeks. Obviously, there is a simultaneous reduction of HB concentration, a reduction generally not desired. On one occasion at the OMRE, this method of operation was used over a three-week period to achieve a decontamination factor of 2 1/2 fold for the entire coolant charge.

TABLE I

OMRE STILL DECONTAMINATION FACTORS

Isotope	D. F.*	Isotope	D. F.*
Na^{24}	>25	P^{32}	22
Cu^{64}	>25	S^{35}	3
As^{76}	16	Fe^{59}	400
Mn^{54}	>32	Se^{75}	2

$$* \text{ Decontamination Factor} = \frac{\text{Still Feed Activity in disintegrations/minute/gram}}{\text{Still Product Activity disintegrations/minute/gram}}$$

III. THE LABORATORY DEVELOPMENT PROGRAM

The supporting development program has performed work in three areas. A limited amount of work was done in direct support of OMRE purification system operation. Also completed, were some experimental investigations for recovering the more volatile fraction of HB. The third and main effort provided considerable information for the modified or continuous OMRE purification system design.

A. LIMITING STILL POT TEMPERATURES

To provide OMRE operation with knowledge of the latitude permitted in still pot temperature, particularly with regard to the thermo-siphon leg temperature, testing was conducted to determine the safe maximum temperature. These tests were designed to simulate OMRE still operation as closely as possible. The apparatus, shown in Figure 5, consisted of a series of HB samples contained in capped, 5 1/2-inch long, 2-inch (diam) black iron pipe nipples, arranged in a radial pattern. Each unit was connected to a common pipe header which also served as the condenser. Melted HB was poured into each unit and and allowed to freeze before connecting the nipple to the header. Each nipple was independently heated with clam-shell heaters and the temperature manually controlled by small variable transformers. Temperatures were taken by thermocouples attached to the outside surfaces simulating the indication method for OMRE thermo-siphon leg temperature. A vacuum was maintained similar to still operation; 20 mm Hg. Temperatures were maintained for four hours, the time required to process one batch of coolant. Since the primary concern was ability to discharge the HB from the OMRE still, coking was arbitrarily assumed to have occurred if a particular test would not pour clean at a temperature of 350°F. Units maintained at 800°F poured clean, ones at 825°F did not.

B. RECOVERY OF THE MORE VOLATILE FRACTIONS OF HB

One of the principal experimental efforts of the supporting development program has been the investigation related to recovering a portion of the HB. If the simple batch distillation is carried to the maximum temperature just under the threshold temperature at which thermal decomposition becomes extremely rapid, 56% of the HB is distilled as light-brown distillate.

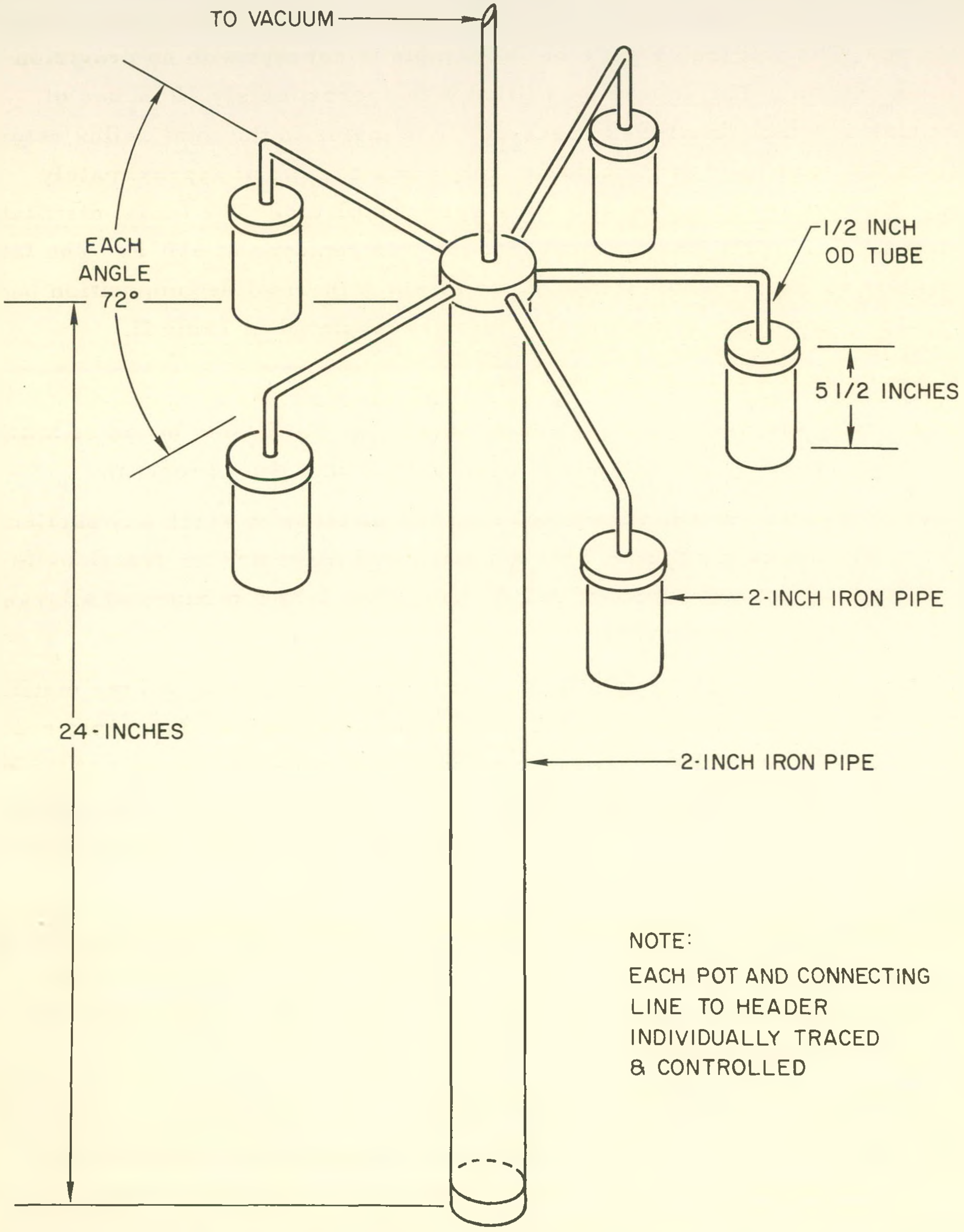


Figure 5. Still Pot Coking Test Apparatus

For further investigation, a high-temperature vacuum distillation column was set up. The unit (See Figure 6) was simple in concept with no provision for reflux control. The column was filled with approximately 20 inches of Podbielniak 1/4-inch "Heli-Pak" packing. A sampler in the total reflux return line from the condenser to the column collected a sample of approximately 200 cc. Eventually two successful runs were completed. The initial distillation temperature was 680°F and the final fraction was removed at 810°F. The latter temperature is the threshold temperature at which thermal decomposition became very rapid. Significant data from this test are tabulated in Table II.

From these tests it was learned that:

1. After the first fractions are distilled off, distillation becomes much easier to perform due to a lessened freezing point problem.
2. There are no significant temperature plateaus at which any particular fraction is distilled. This indicates that there are no fractions in any appreciable amount in HB, but rather it is a mixture of a large number of compounds.
3. Based on freezing point and volatility considerations, all the distilled fractions could be returned to the reactor system as favorable reactor coolant materials. Thus, if desired, the molecular weight of the HB fraction in the coolant may be reduced while maintaining the HB concentration at a desired level by operating the still at a higher cut-off temperature and processing at a faster rate.

The results of the HB distillation experiment showed the need to repeat the work on a more refined basis using more elaborate equipment, and a high-temperature high-efficiency distillation apparatus has been constructed. Operation of the unit commenced in October, 1959.

C. LABORATORY FLASHING EXPERIMENT

With the decision to modify the OMRE purification system making it a continuous flashing process, there was a need for experimental evidence proving the proposed flow scheme to be adequate. Several experiments were carried out. One of these was a bench scale flashing experiment to prove there were no major defects in the proposed flashing equipment design. Possibility of an

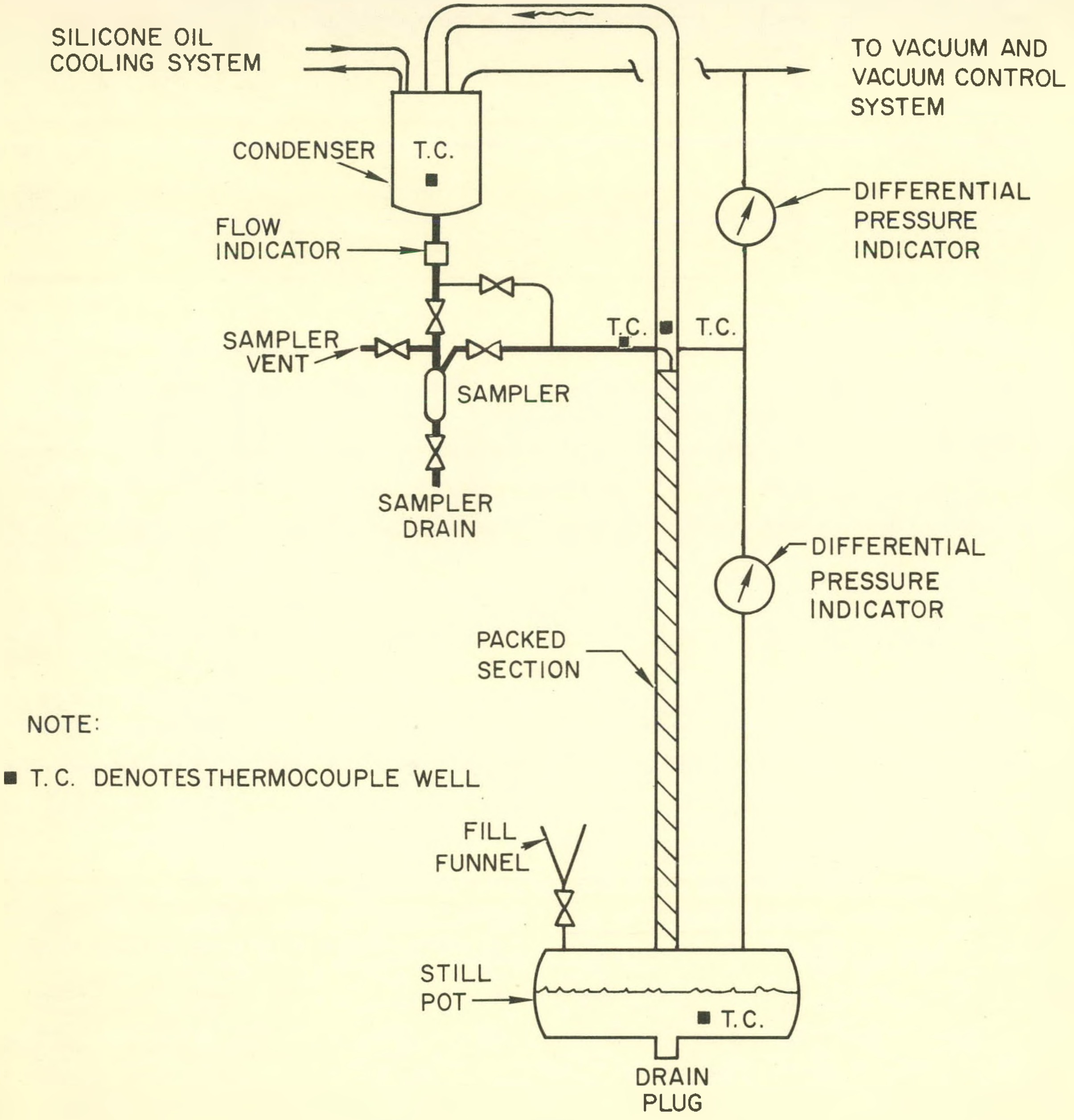


Figure 6. Schematic Diagram of the Experimental Distillation Apparatus for Recovery of the More Volatile Fractions of HB

TABLE II

HB FRACTIONS OBTAINED BY HIGH-TEMPERATURE DISTILLATION UNDER A PRESSURE OF 20 mm Hg AND TOTAL REFLUX

Still Pot Temperature (°F)	Overhead Temperature (°F)	Melting Range (°F)	Color	C/H Ratio	Molecular Weight
680	464	218-240	Lt. grey	1.19	238
708	513	228-286	Lt. tan	-	-
715	532	226-278	Lt. brn.	1.27	269
730	571	237-298	Brown	-	-
740	607	197-318	Brown	-	-
750	624	159-294	Brown	-	-
755	633	147-158	Drk. brn.	1.28	348
765	662	111-125	Brown	-	-
780	610	120-138	Brown	-	-
805	692	116-147	Brown	1.27	397

entrainment problem was of primary interest. The apparatus was not an exact prototype of the proposed OMRE design, but the experiment was conducted to simulate the anticipated flow, temperature and pressure conditions of the proposed OMRE flash chamber. A schematic diagram of the apparatus is shown in Figure 7. The flash chamber was of concentric cylindrical construction similar to the OMRE design. Unlike the OMRE system, the laboratory model was not equipped with a stripping column.

From the beginning of the experiment, the need for an adiabatic apparatus was evidenced; an aluminum jacket entirely surrounding the flash chamber fulfilled this need. The jacket was supplied with five independent heating circuits. Thermocouples were situated on both the chamber wall and the jacket in a paired arrangement. Sufficient heat was added to the jacket to make the temperature difference between the chamber wall and the jacket essentially zero. The apparatus was considered to be adiabatic when an essentially zero temperature drop could be maintained.

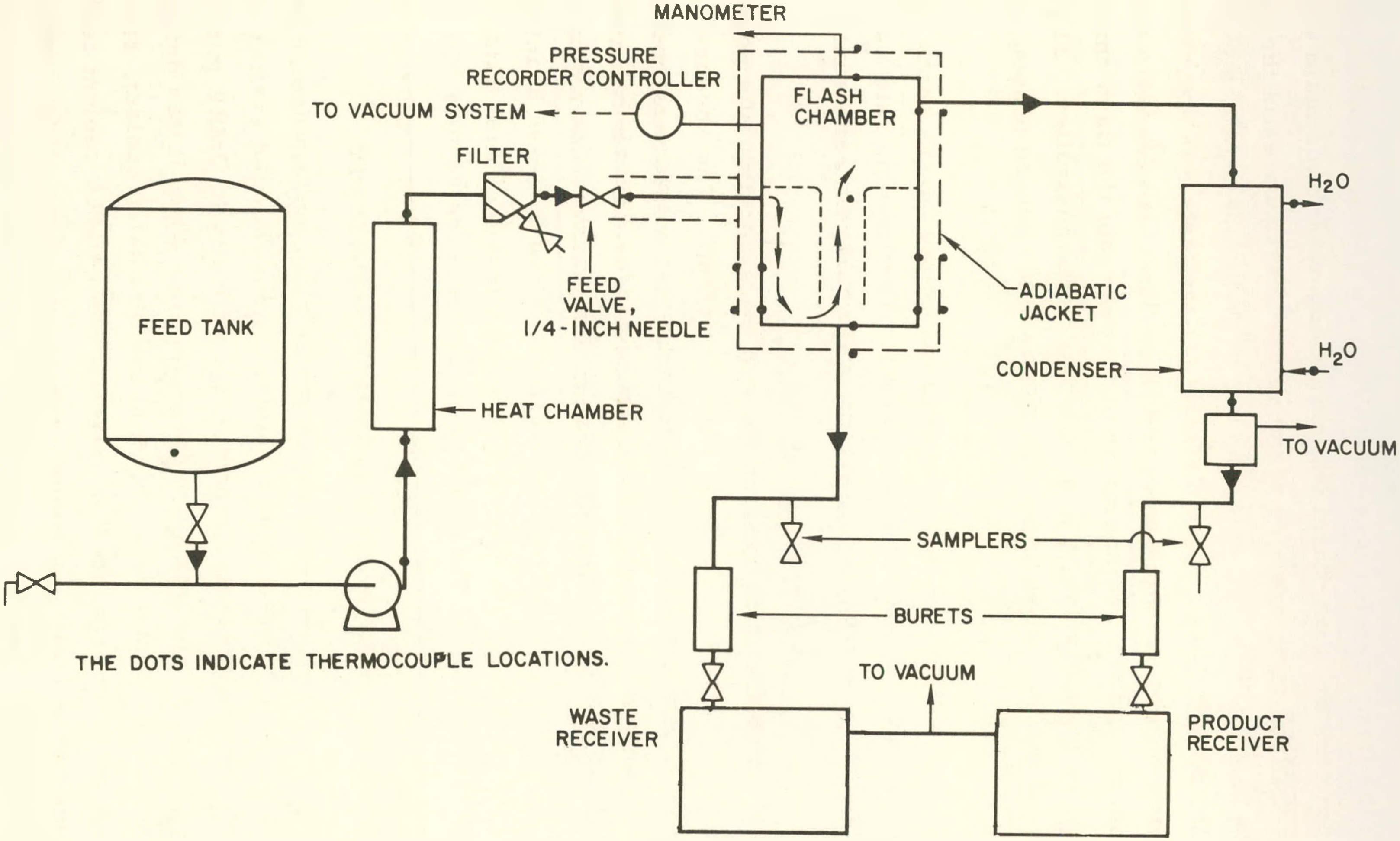


Figure 7. Schematic Diagram of the Bench Scale Flashing Experiment

Flow to the flash chamber was controlled by a 1/4-inch needle valve. A small high temperature centrifugal pump was employed to maintain a constant pressure on the high pressure side of the valve. Particulates in the feed material proved to be a problem requiring the use of an in-line strainer to prevent plugging of the needle valve. Feed material for the tests was a synthetic mixture approximating the composition of OMRE coolant. HB for the mixture was received from the OMRE. Temperature of the feed into the flash chamber was maintained at a constant value by a West temperature controller. The proper vacuum was maintained on the apparatus by use of a Foxboro pressure controller actuating a solenoid valve in the line to the vacuum pump.

Distillate and bottoms were collected in calibrated burets which in turn could discharge into two 4-liter vacuum flasks. The flashing rate was determined by the quantity of distillate collected in the buret in a given interval of time.

An important result of the experiment was the production of a good grade of distillate. The product was a clear amber material. The absence of black coloring matter in the distillate gave satisfaction that entrainment was negligible and the proposed design was not in serious error. Of a more quantitative nature, data were obtained giving the percent flashed at a given set of operating conditions. These data are presented in Figures 8 and 9. Also of interest is the drop in temperature as the material is being flashed. These data are presented in Figures 10 and 11. An effort to correlate these results with the amount of material which theoretically could be flashed proved unsuccessful.

D. INVESTIGATION OF LOW BOILER REMOVAL METHODS

Another facet of the continuous OMRE purification system design laboratory support was the means of handling low boilers. As mentioned earlier in this report, the low boilers presented considerable difficulty in OMRE purification operation due to carry-over into the vacuum system. When it was determined that the low boilers accounted for only 1% of the irradiated coolant, special distillation equipment to separate this material from OMRE coolant could not be justified. A method to treat continuously the vacuum pump oil for removal of the low boilers and diphenyl carried along with the low boilers was then developed.

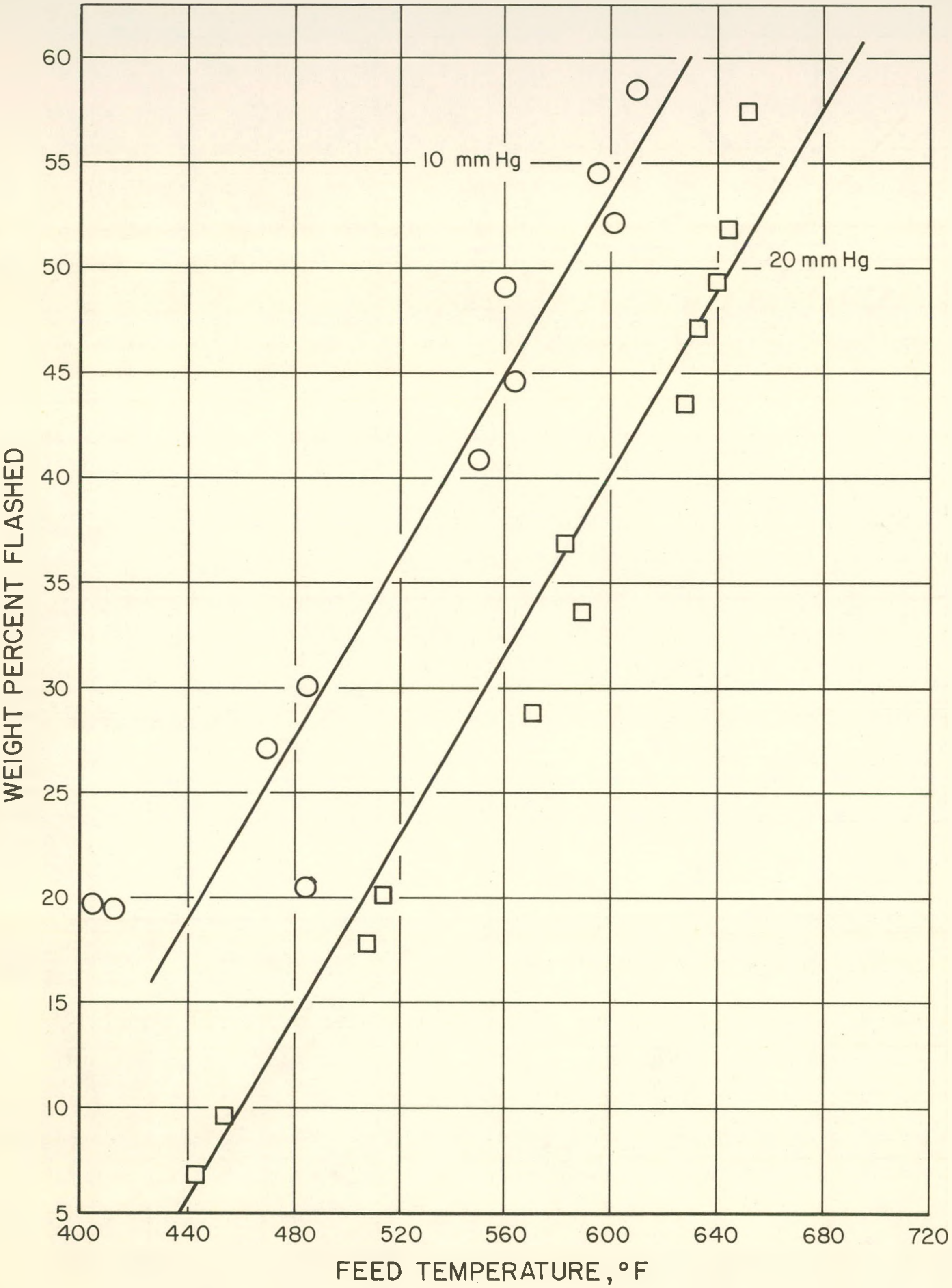


Figure 8. Percent of Material Flashed in Experimental Flashing Test of 30% HB Coolant

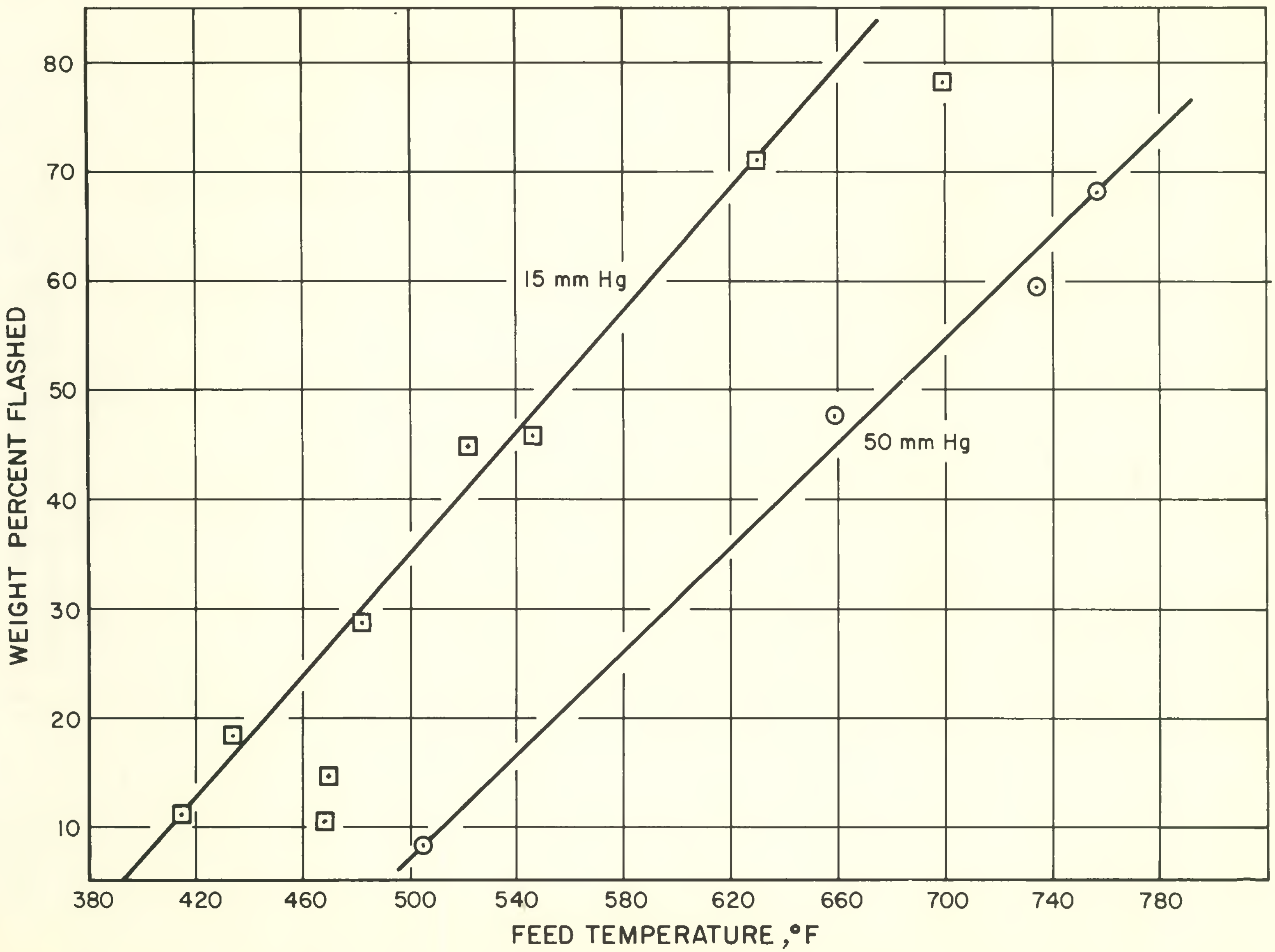


Figure 9. Percent of Material Flashed in Experimental Flashing Test of 10% HB Coolant

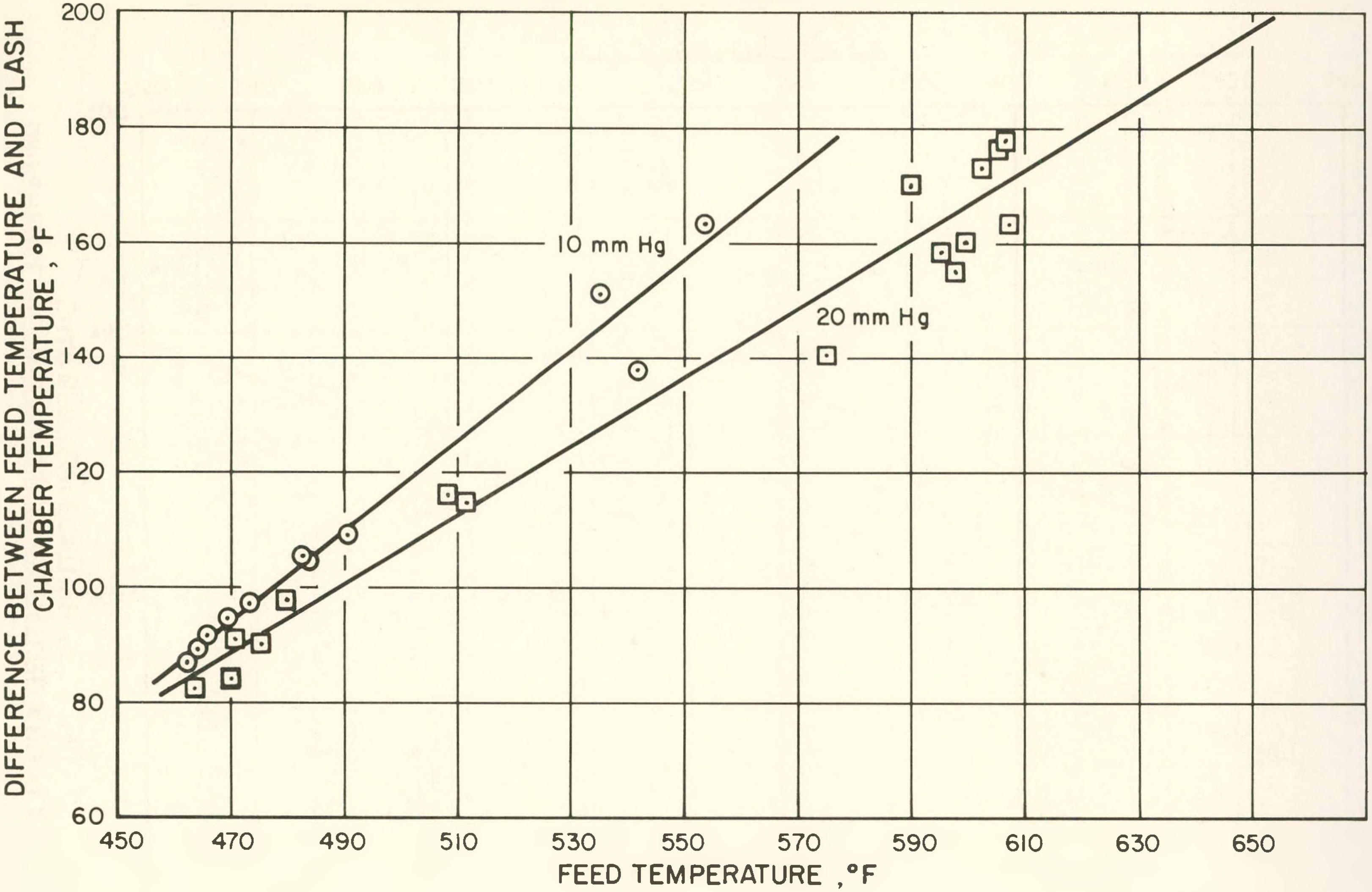


Figure 10. Difference Between Feed Temperature and Flash Chamber Temperature as a Function of Feed Temperature for 30% Coolant

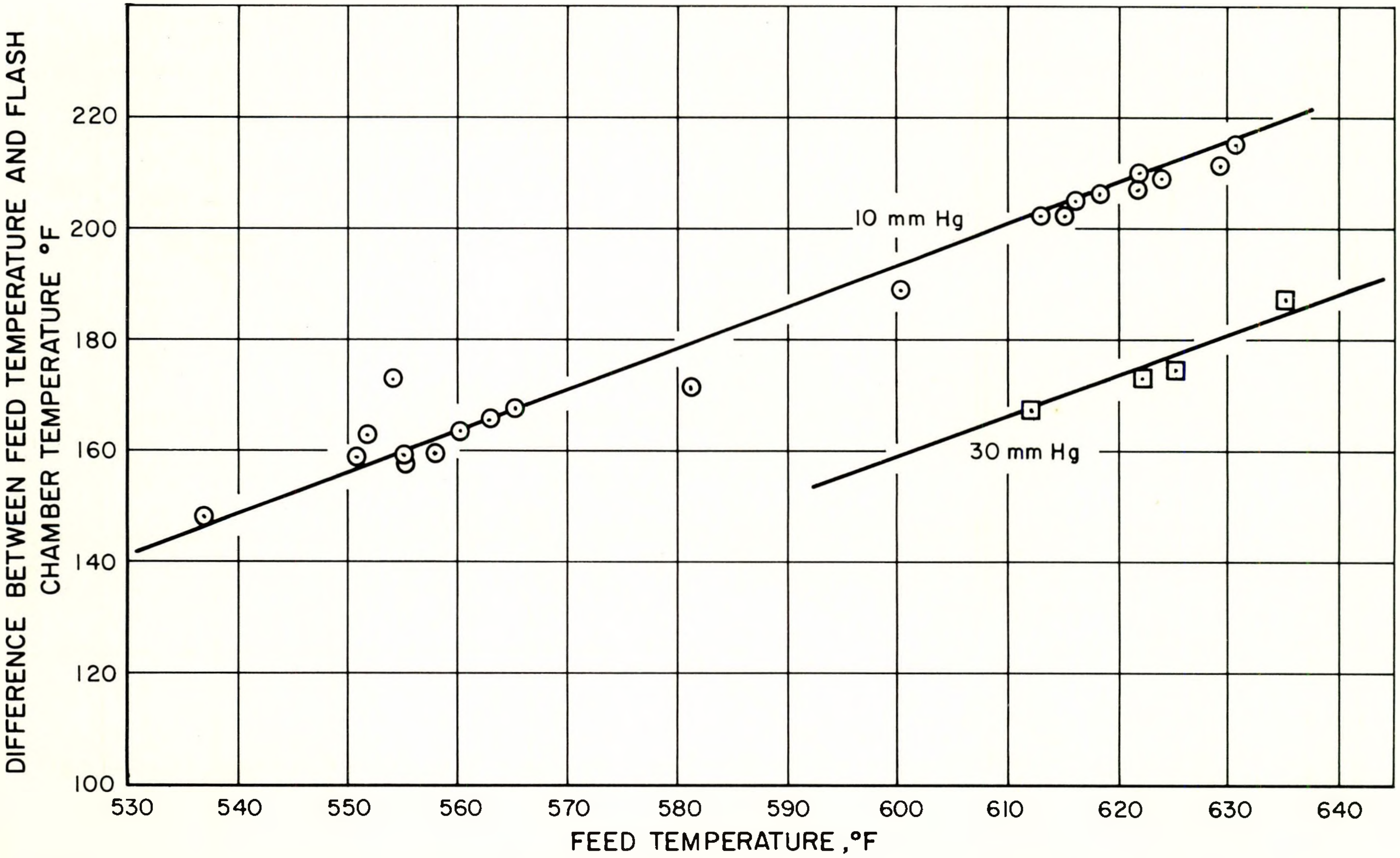


Figure 11. Difference Between Feed Temperature and Flash Chamber as a Function of Feed Temperature of 10% Coolant

Because of the large difference in volatility of the low boilers and vacuum pump oil, a flashing process adequately reclaims the vacuum pump oil. Several makers of commercial oil reclaiming units are available to fulfill this need. However, the removal of diphenyl is much more difficult. Diphenyl in vacuum pump oil is not detrimental from the standpoint of volatility but a phase separation occurs when the oil becomes saturated. This is particularly troublesome in intermittent operation where the temperature of the pump decreases causing the precipitation of considerable quantities of diphenyl on the internal moving parts of the pump. With heavy deposits, startup of the pump is impossible.

Standard Oil of California No. O. C. 15 turbine oil was employed at the OMRE in preference to a high grade vacuum oil, primarily for economic reasons. Therefore, our efforts were to remove diphenyl from turbine oil. Investigation of the solubility of diphenyl in turbine oil showed that a concentration up to 7% could be tolerated. Therefore, the effort was to develop a method to reduce the diphenyl concentration below the 7% value; adsorption, crystallization, and distillation methods were tried. Absorbing agents such as Fullers Earth, activated carbon and silica gel showed no removal tendency. Crystallization also showed little promise because of the high viscosity of the oil at the low temperatures required in the subsequent filtration step.

Distillation was tried with considerable success. Both single stage batch and flashing tests were conducted. The batch tests employed ordinary chemical glassware while the flashing test utilized the same apparatus used in the coolant flashing tests (See Figure 7). The results of the tests conducted at 20 mm Hg are given in Table III. The results show no particular advantage of either type of distillation process. However, the development of equipment for either process would be undesirably complicated. In effect, a small vacuum distillation system for oil recovery would be added to the larger coolant recovery distillation system.

TABLE III

SEPARATION OF A 10% DIPHENYL IN TURBINE OIL MIXTURE BY VACUUM DISTILLATION AT 20 mm Hg

Flash Test		Batch Test	
Temperature of Feed to Flash Chamber, (°F)	% Diphenyl Remaining in the Bottoms	Temperature of Pot (°F)	% Diphenyl Remaining in the Bottoms
360	8.4	355	8.0
375	7.1	375	7.3
400	6.6	392	6.2

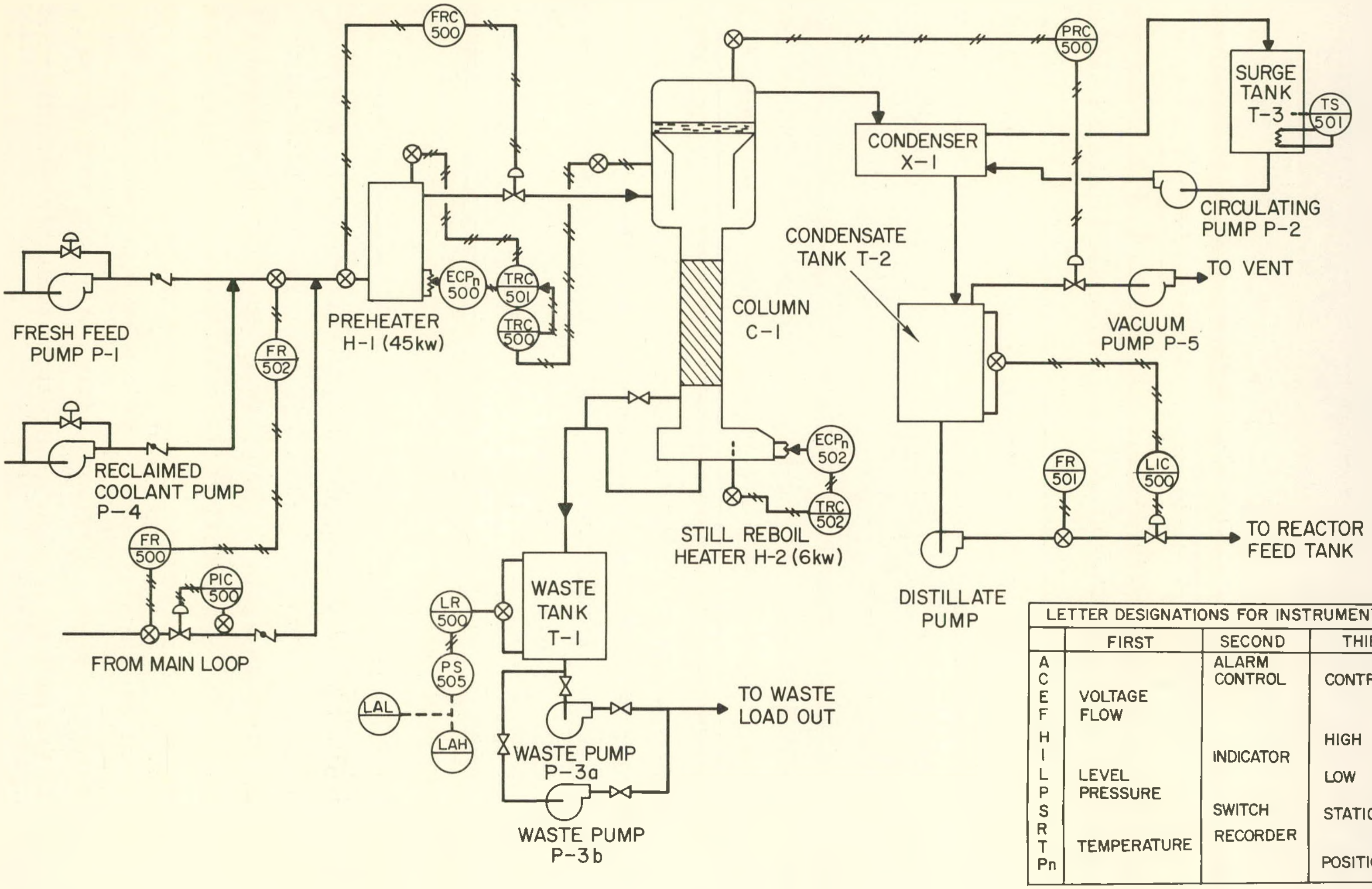
A search was made for vacuum equipment which could tolerate diphenyl and low boilers. Recently a pump claimed to tolerate a gas stream polluted with volatiles and other contaminants, was made available for the new OMRE Purification system. This pump is described in the next section of this report. If this new pump should prove inadequate, resumption of development work will be necessary.

IV. DESCRIPTION OF THE CONTINUOUS OMRE PURIFICATION SYSTEM

A major improvement being added to the OMRE is the new purification system to be placed in operation in November 1959. As already mentioned the new system is a continuous flash distillation process built adjacent to the present batch system. The design of the new system includes most of the improvements shown to be desirable as a result of two years of operation of the present system and related research and development work. Provisions have been made for processing three distinct feed materials at a varying over-all feed rate from 1/4 to 1 gallon per minute. The system is essentially automatic requiring little operator attention except for the waste discharge operation.

By reference to the flow diagram, Figure 12, it is seen that three streams feed into the system; these are (1) irradiated coolant from the main reactor loop, (2) fresh coolant added to replace the HB discharged from the system, and (3) reclaimed coolant resulting from seal leakage, various purging operations, or material drained as a result of numerous maintenance operations. Either fresh coolant or reclaimed coolant is pressurized and pumped into the column feed line by means of Viking gear pumps P-1 and P-4 respectively. The flow rate from either pump is controlled by the setting on a pressure control valve in a by-pass line around either pump. An interlock is provided so pumps P-1 and P-4 cannot be operated simultaneously, insuring that proper pressure and flow control of the incoming streams is possible. Coolant from the main loop is reduced from main loop pressure to 75 psi by PIC-500. The total flow rate is recorded and controlled by FRC-500. Since pumps P-1 and P-4 are essentially constant volume pumps, the difference between the output of either of these pumps and the total flow is supplied from the main loop. The latter stream, in effect, is controlled by FRC-500, the total flow controller, but the flow is recorded on instrument, FR-500. Flow recorder FR-502 records the flow from pump P-1 or P-4, whichever is operating. The sum of the flows indicated on the two pen instrument, FR-500/FR-502, should equal reading on FRC-500.

The streams from the main loop and the fresh or reclaimed coolant are combined and heated to the required feed temperature of approximately 700°F by a 45 kw heater, H-1. The feed temperature is controlled by TRC-500/501 which actuates a pneumatically controlled variable transformer, which in turn



LETTER DESIGNATIONS FOR INSTRUMENTS

	FIRST	SECOND	THIRD
A		ALARM	
C		CONTROL	CONTROLLER
E	VOLTAGE		
F	FLOW		
H			HIGH
I		INDICATOR	
L	LEVEL		LOW
P	PRESSURE		
S		SWITCH	STATION
R		RECORDER	
T	TEMPERATURE		
Pn			POSITIONER

Figure 12. Schematic Flow Diagram of the Continuous OMRE Purification System

actuates the largest of four electrical circuits to the heater. The other three circuits are turned on or off manually. The feed is reduced from 75 psig to 20-50 mm Hg, causing a large fraction of the volatile coolant to vaporize as the material enters the column. The vapor-liquid mixture enters the flash chamber section of the column tangentially. The flash section is of concentric cylinderical construction with the feed entering the annular section. In this section the major portion of the liquid phase is disengaged from the vaporized feed. After emerging at the bottom of the annulus, the vapors are diverted upward through the chimney or center section while the liquid descends into the packed section of the column. To further increase phase separation efficiency, a 4-inch thick section of wire mesh entrainment eliminator is placed at the top of the chimney.

Vapors from the top of the column pass through a 6-inch line to the shell side of a shell and tube condenser, X-1. Cooling for the condenser is provided by a pressurized ethylene glycol-water mixture. The mixture is circulated from a surge tank, T-3, through the condenser and returns to the surge tank. The tank, equipped with both heating and cooling coils, is controlled by TS-501 to maintain the desired temperature of the cooling system. Pressurization is provided for the surge tank in order to raise the boiling point of the mixture above the normal boiling point at atmospheric pressure. This feature is necessary to accomodate a high melting point coolant such as Santowax OMP.

Distillate from the condenser drains to the condensate tank, T-2, and is transferred by a canned rotor pump to the existing feed tank. The liquid level of the condensate tank is controlled by LIC-500 which actuates the control valve on the pump discharge. The distilled coolant is pumped from the feed tank to the main loop by the Lapp Pulsifeeder pump; its rate is controlled by a pneumatic stroke positioner.

Returning our attention again to the column, consider the second stream being discharged from this unit; the high boiler stream. The liquid phase descending into the packed section from the flash chamber has a high concentration of high boiler but does contain some coolant fractions which are desirable to retain in the reactor system, therefore, some stripping is necessary and this is accomplished in the packed section where approximately two theoretical stages are provided. Because the column has to accomodate varied feed conditions, the operating pressure has to be established at the proper value to preclude flooding. With a feed rate of 1 gallon per minute of 10% HB coolant, the proposed operating pressure is 50 mm Hg. The calculated percent of flooding at this condition is 90%. The column was actually designed to meet this condition. At a feed rate of 1/4 gallon per minute of 30% HB coolant, the proposed operating pressure will be reduced to 20 mm. The percent of flooding at this condition was estimated to be 16%. The latter condition is too low for good column efficiency, but nevertheless satisfactory since so few theoretical states are required.

The stripping section is packed with 5/8-inch 316-SS Pall Rings. Stainless Steel was selected as the material for the packing because of the eventuality that the packing might become encrusted with thermal decomposition products, requiring removal of the packing from the column and dissolution of the encrustation by an acid wash. The reboiler is a horizontally mounted section flanged to the bottom of the column. The reboil heat is supplied by a 6 kw electrical heater, H-2, controlled by a pneumatically operated voltage regulator, ECPn-502. The liquid level in the reboiler is maintained by a loop in the discharge line. The reboiler drains by gravity to a waste tank, T-1, mounted on the wall of the pump pit. Either one of two Viking gear pumps, P-3a or P-3b, located below the waste tank is used to pump the HB from the waste tank to the load out station. The waste load out operation is carried out whenever 40 to 45 gallons of waste are accumulated. A high- and low-level alarm on the waste tank notifies operating personnel when pumping should start and stop. This is the only non-automatic aspect of the entire system.

The vacuum system employs a mechanical vacuum pump. Of special note is the absence of traps or condensers for removal of volatile material before entry into the vacuum pump, P-5. The vacuum pump is capable of operating under these conditions since it is a sliding vane type, operating on a once-through oil system. The spent oil, probably never more than a quart an hour, is emitted with the exhaust. System pressure is maintained by controller PRC-500 with the sensing element located in the flash chamber. The controller actuates a control valve on the line from the condensate receiver to the vacuum pump.

Provisions are included in the system to allow for sampling of all major processes streams; column feed, overhead product, and bottoms product. Sampling will be a direct rather than a remote operation since it is expected that the radiation level of the process area will be low enough to permit access to the process equipment at all times. Pre-heating of all lines, vessels, pumps, and instrument sensing elements will be by electrical induction where such a method is applicable; for other instances resistance heaters are provided.

V. CONCLUSIONS AND RECOMMENDATIONS

OMRE purification system operation and related development work have demonstrated that vacuum flash distillation easily and adequately separates HB from the coolant. Proof that the separation can be performed continuously and automatically will be demonstrated in the forthcoming operation of the new system being installed at the OMRE.

There is an area of particular interest which deserves further investigation. By operation of a purification system to return selected more volatile fractions of HB to the reactor system and by increasing the processing rate it will be possible to decrease the molecular weight of the HB fraction of the coolant. This method of controlling the average molecular weight and other related properties needs further experimental investigation. Also, an economic study should be conducted to determine the proper balance of purification system operating expenses and fresh coolant makeup costs against desired coolant properties.

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