THE VACUUM STRIPPING OF CARBON DISULFIDE FROM AROMATIC ABSORPTION OIL

by

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ACKNOWLEDGEMENT

The writer is greatly indebted to Mr. S. C. Schwarz of Portland Gas and Coke Company for his helpful suggestions in pursuing this study and for his generosity in making available data and material of his company. Thanks are due to Professors Walton, Schulein, and Putnam for their helpful suggestions and criticisms.
THE VACUUM STRIPPING OF CARBON DISULFIDE FROM AROMATIC ABSORPTION OIL

The existence of sulphur compounds in gaseous fuels in small quantities presents a problem to the gas industry because of the necessity of removing these compounds to some extent before a gas can be used as fuel. The maximum sulphur content of such gases is controlled by the Public Utilities Commissions in the communities in which the gas is manufactured and distributed. In Portland, Oregon the maximum sulphur content allowable is set at 30 grains per 100 standard cubic feet of gas.

SULPHUR COMPOUNDS IN GAS

The sulphur compounds present in gas are placed in two classes, hydrogen sulfide and organic sulphur compounds. Hydrogen sulfide is the most abundant compound present. Of the many organic sulphur compounds that may be present the following are those most common:

<table>
<thead>
<tr>
<th>Compound</th>
<th>Formula</th>
</tr>
</thead>
<tbody>
<tr>
<td>Carbon Disulfide</td>
<td>CS₂</td>
</tr>
<tr>
<td>Mercaptans</td>
<td>R-SH</td>
</tr>
<tr>
<td>Thiophene</td>
<td>C₄H₄S</td>
</tr>
<tr>
<td>Carbon Oxysulfide</td>
<td>COS</td>
</tr>
</tbody>
</table>

Of these CS₂ is the most abundant, commonly occurring in gases in quantities up to 100 grains per CCF. CS₂
usually represents over 80% of the organic sulphur content and the remaining 20% consists of all the others, thiophene usually being most of that.

REMOVAL OF SULPHUR COMPOUNDS FROM GAS

In most cases H₂S is substantially completely removed from the gas. The processes used for its removal are well developed and no further mention will be made here concerning this compound. For a summary and short description of these processes the reader is referred to an article by Powell (5).

The methods for removal of the organic sulphur compounds from gas are not so well developed as for H₂S. A publication of the British Institution of Gas Engineers (4) presents a summary of this sulphur removal problem.

In most cases normal oil washing for the removal of light oils from manufactured gas removes about 35% of the sulphur content which is all that is necessary to keep the sulphur below the maximum allowable. However, for more complete removal an oil absorption process requiring a large amount of oil circulation is necessary, and this presents the problem of economically stripping such a large amount of absorption oil. Other processes that have met with some success but are not used on a
commercial scale are the active carbon adsorption process which removes 75% of sulphur content and a catalytic process described by Tupholme (9) where sulphur compounds are oxidized and reduced leaving the sulphur as SO₂ and H₂S.

Today the organic sulphur in gas is no major problem with most companies as its content in the purified gas can be of such a quantity that its removal is not necessary, or at least its removal from gas by normal oil washing as mentioned above is sufficient. As pointed out to the writer by Mr. S. C. Schwarz of Portland Gas and Coke Company, the trend in the gas industry seems toward less sulphur content; and, if at some time in the future, it becomes necessary to remove sulphur to quantities below 10 grains per CCF, then sulphur removal will become a problem.
CARBON DISULFIDE ABSORPTION AND STRIPPING

The subject chosen for this thesis study resulted from a discussion by the writer with Mr. Schwarz, and it represents but one small part of the general problem of sulphur removal from gas.

The most probable way for a gas company to remove sulphur compounds, if it became necessary, would be by oil absorption as such a system would probably already be in operation for light oil recovery. Therefore it was decided to study some phase of this type of operation only. Since CS₂ is usually the only organic sulphur compound present to any extent worthy of consideration, the subject was simplified by limiting it to that compound alone.

The operation of gas scrubbing with absorption oil to remove the CS₂ is very common and straightforward and no particular problem would exist here other than the circulation of about three times the absorption oil necessary for light oil recovery. As a result the subject was further limited to a study of the stripping of the rich absorption oil in order to remove the CS₂ to prepare the oil for recirculation. Since the CS₂ exists in the gas in such small quantities its recovery would be of no importance.
Even a study of the general subject of stripping would be too extensive for consideration here and as a result this thesis discusses only one method; a system proposed by Mr. Schwarz (7) as a solution to this general problem of sulphur removal should it ever be confronted by his company.

PROPOSED OPERATION BY PORTLAND GAS AND COKE COMPANY

A general discussion of the operation of the Portland Gas and Coke Company is here included as many ideas used in this study came from there.

Gas is made by oil cracking in regular gas generators. The gas is water scrubbed to remove tars and is passed through purifiers filled with iron oxide where substantially all of the $\text{H}_2\text{S}$ is removed. It is then passed through scrubbing towers where a heavy aromatic absorption oil called creosote is circulated and about 85% of the benzene and heavier aromatic compound content of the gas, amounting to 1% to 2%, is removed. About 35% to 50% of the 50 grains per CCF of $\text{CS}_2$ present in the gas is also removed. The rich absorption oil is steam stripped under vacuum to remove these light oils and $\text{CS}_2$ and then cooled for recirculation. The light oil fraction is further refined for separation of the aromatic compounds and the small amount of forerunnings of this
refining process containing the CS$_2$ as well as some light hydrocarbon are fed into a coke oven, along with the oil feed, where severe cracking takes place. Thus the sulphur content of the gas manufacturing system is not removed but probably converted to H$_2$S by the cracking process.

The removal of CS$_2$ from the gas to the extent of 90% could be accomplished in the same system but it would require the circulation of about 24 gallons of oil per thousand cubic feet of gas instead of 8 as used at present. The stripping of such a large amount of oil would be rather expensive in such a single system as it would have to be stripped to the extent of removing the benzene. Benzene is comparatively easy to absorb and hard to strip while CS$_2$ is the opposite, and each phase of the process would have to be operated on the basis of the compound most difficult to remove.

Figure 1 shows the absorption oil flow of the present system for benzene absorption and oil stripping and the system proposed by Schwarz for removal of CS$_2$ as well as benzene.

As shown in the diagram the proposed system would circulate the 8 gal/MCF oil used in the benzene absorber through the CS$_2$ absorber as well as 17 gal/MCF of oil from the CS$_2$ stripper. The total oil from the CS$_2$
absorber would be split, 8 gal/MCF going to the benzene stripper and then back to the benzene absorber, and the rest, 17 gal/MCF, passing through the CS₂ stripper. As a result of this the oil to the CS₂ absorber would total 24 gal/MCF and would be substantially saturated with benzene. The problem here is to economically strip the large amount of oil in the CS₂ stripper for the removal of CS₂ only. The benzene is still removed in the normal way by steam stripping.

In the system it is proposed by Schwarz to use a high vacuum and a slight amount of indirect heat. He states that the benzene present in the oil would act as a carrier and aid in removing the CS₂. The removal of some of the benzene here would not matter as the products of both strippers would be combined for further refining.

DETAILS OF STRIPPING SYSTEM

It was decided to compare this 3 component method of continuous stripping described above with a two component system in which no benzene is present and the CS₂ is removed from the absorption oil without the aid of a carrier.

It was necessary to determine what concentration of CS₂ would exist in the rich absorption oil. The only commercial operation for this type of sulphur removal
Fig. 1

Present System
- Lean Gas
- Rich Gas
- C₆H₆
- CS₂ and C₆H₆
- stripper

Proposed System
- Lean Gas
- RICH GAS
- C₆H₆ Absorber
- CS₂ Absorber
- C₆H₆ Stripper
- CS₂ Stripper
- 8 gal/MCF
- 17 gal/MCF
described in detail was found in the British Institution of Gas Engineers publication (4) and much of the data used to determine this concentration were obtained from there.

Assuming the CS$_2$ content of the gas to be 100 gr/CCF, in order to remove 95% of the CS$_2$, a column of 12 theoretical plates circulating 23.3 gal of oil per MCF would be necessary. The CS$_2$ content of the rich oil would be 0.15 mol percent and would have to be stripped 95% to 0.0075 mol percent CS$_2$ before recirculation. The calculations made to determine these figures are shown in Appendix I.

By using indirect heat and no outside agent such as steam for stripping as proposed the method becomes one of fractional distillation in which refluxing is necessary. Following the operation at Gasco again, since the overhead product from this still would be combined with the other light oil product from a benzene stripper and then further refined, it would be permissible to allow some of the absorption oil itself to distill over with the CS$_2$. Also since the difference in volatility between the CS$_2$ and the absorption oil is so great, by operating the stripping section of the distillation column only, that is, bringing the feed into the column on the top plate, the vapors distilled off in equilibrium
with the feed liquid would be comparatively rich in CS$_2$
content and small in volume.

Instead of using creosote for the absorption oil for this study it was decided to use the commercially pure Gasco product "Xylol," which is essentially xylene, C$_8$H$_{10}$. This compound would be much cleaner to handle and calculations could be simplified by considering it a single pure compound.

The study of this system becomes rather interesting because of its oddity - feed and reflux both entering on the same plate from which the distillate vapors are removed. Only theoretical calculations and laboratory experimental verification can prove its ability to work satisfactorily.
THEORETICAL CALCULATIONS

For theoretical consideration of any absorption or distillation system the equilibrium constant, \( K \), for the compound in question must be known. This constant expresses the ratio of concentration of a compound in the gas or vapor phase of the system to its concentration in the liquid phase as follows:

\[
K = \frac{Y}{X}
\]

where \( Y \) is the mol fraction of the solute in the gas and \( X \) is the mol fraction of the solute in the liquid. If Raoult's Law applies then this constant may be expressed as

\[
K = \frac{p}{P}
\]

where \( p \) is the partial pressure of the solute in the gas and \( P \) is the total pressure of the system. In this case the equilibrium constant can be calculated directly from vapor pressure data, but otherwise it must be determined experimentally.

The values of \( K \) given by the British (4) for \( \text{CS}_2 \) in oil check closely with the values obtained by Raoult's Law (see Appendix II). Therefore, all calculations made in this study use vapor pressure data and apply Raoult's Law.
Using the method described by Robinson and Gilliland (6, p. 137-150) for rectification of multi-component mixtures the proposed stripping problem was calculated for both the two component and the three component systems. The three component system was to contain 7% by weight benzene (9.3 mol %) as well as the CS\textsubscript{2} content of 0.15%. Operating conditions were to be 28" mercury vacuum total pressure, feed entering as saturated liquid, vapors to leave in equilibrium with feed, and concentrations of CS\textsubscript{2} in feed and stripped product as previously mentioned. The figure of 28" Hg vacuum was chosen for this study as it represents a practical value used in commercial operation for vacuum stripping.

After many trial calculations, the set of data as shown in Table 1 was chosen for comparison with experimental study. Shown in Appendix III are the calculations from which the data for Table 1 were obtained.

A calculation was made using no reflux ratio and as expected showed that a column of infinite theoretical plates would be necessary to accomplish the desired separation. Of all the reflux ratios tried, the value of 0.5 gave the best design as to size of stripping column.
### TABLE 1

**SUMMARY OF THEORETICAL CALCULATIONS**

<table>
<thead>
<tr>
<th>Component</th>
<th>2 Component System</th>
<th>3 Component System</th>
</tr>
</thead>
<tbody>
<tr>
<td>Feed Composition - mol %</td>
<td></td>
<td></td>
</tr>
<tr>
<td>CS₂</td>
<td>0.15</td>
<td>0.15</td>
</tr>
<tr>
<td>C₆H₆</td>
<td>-</td>
<td>9.30</td>
</tr>
<tr>
<td>C₈H₁₀</td>
<td>99.85</td>
<td>90.55</td>
</tr>
<tr>
<td>Vacuum - inches of mercury</td>
<td>28</td>
<td>28</td>
</tr>
<tr>
<td>Reflux Ratio</td>
<td>0.5</td>
<td>0.5</td>
</tr>
<tr>
<td>Bottoms Composition - mol %</td>
<td></td>
<td></td>
</tr>
<tr>
<td>CS₂</td>
<td>0.0075</td>
<td>0.0075</td>
</tr>
<tr>
<td>C₆H₆</td>
<td>-</td>
<td>7.0500</td>
</tr>
<tr>
<td>C₈H₁₀</td>
<td>99.9925</td>
<td>92.9425</td>
</tr>
<tr>
<td>Distillate Composition - mol %</td>
<td></td>
<td></td>
</tr>
<tr>
<td>CS₂</td>
<td>3.06</td>
<td>2.17</td>
</tr>
<tr>
<td>C₆H₆</td>
<td>-</td>
<td>41.43</td>
</tr>
<tr>
<td>C₈H₁₀</td>
<td>96.94</td>
<td>56.40</td>
</tr>
<tr>
<td>Number of Theoretical Plates</td>
<td>6</td>
<td>6</td>
</tr>
<tr>
<td>Top Temperature - °F</td>
<td>131.5</td>
<td>110</td>
</tr>
<tr>
<td>Bottom Temperature - °F</td>
<td>132.5</td>
<td>117</td>
</tr>
<tr>
<td>Ratio - Feed/Distillate</td>
<td>21.4</td>
<td>15.2</td>
</tr>
<tr>
<td>% Xylene in feed lost in distillate</td>
<td>4.5</td>
<td>4.1</td>
</tr>
<tr>
<td>Heat Requirement</td>
<td>Btu/100 mols feed</td>
<td>392,500</td>
</tr>
</tbody>
</table>
The data used for xylene in the calculations were the average values for ortho-, meta-, and para-xylene, assuming that the material used would be a mixture of these three forms and would act as one component rather than three separate ones of close physical properties. Appendix IV lists the data used for these calculations.
EXPERIMENTAL WORK

In order to make an experimental check on the theoretical calculations of the two systems described, a laboratory stripping still was constructed and operated. Distillations of the two component system, xylene with 0.15 mol percent carbon disulfide were first made using various reflux ratios, followed by similar distillations of the three component system, xylene with 0.15 percent carbon disulfide and 9.3 mol percent benzene.

ABSORPTION OIL

The absorption oil used for this work was commercially pure xylene, a product of the Portland Gas and Coke Company. Figure 2 shows the A.S.T.M. Distillation Curve of this oil (labeled "Gasco" xylene). Since the xylene used was actually a mixture of ortho-, meta-, and para-xylene, the distillation curve did not flatten out but increased constantly over the range of boiling points of the three forms, 138.4°C for the lowest and 142.7°C for the highest. The presence of some lighter compounds, probably a small amount of toluene, and some heavier aromatic polymers are shown by the shape of the lower and upper ends of the curve.

Also shown in Figure 2 is the curve resulting from the addition of benzene to the amount of 7 weight percent
FIG. 2
A. S. T. M. DISTILLATION CURVES FOR ABSORPTION OILS

"GASCO" XYLENE

93% XYLENE - 7% BENZENE
BY WEIGHT

TEMPERATURE - °C

PERCENT DISTILLED

160
150
140
130
120
110
0  25  50  75  110
(9.3 mol percent) which was the oil used for the three component distillation.

COLUMN DESIGN

Available for use as a laboratory stripping still was a pyrex glass tube four feet long by 60 mm inside diameter and 3/8" by 3/8" porcelain raschig ring packing material.

From data given by Robinson and Gilliland (6, p. 219-221) a laboratory column of this size and type of packing should have an H.E.T.P. (Height Equivalent to a Theoretical Plate) of between 4.3" and 6.8". For this column a figure of 6" H.E.T.P. was chosen.

For a column of 6 theoretical plates as calculated this required 36" of packing, leaving 6" of the 4-foot column open at the bottom for the reboiler section and 6" open at the top.

For the operation of this column a vapor velocity of 1.5 ft/sec was chosen. From calculations based on data in Sherwood (8, p. 138-144) and the figures obtained in the preliminary calculations, the liquid velocity was well below the flooding velocity of this type of column. Pressure drop through the column calculated as less than 0.1" Hg.

For good liquid distribution the tower diameter to
packing size ratio should be greater than 8 (8, p. 136). The ratio actually existing was 6.3 as nothing smaller than 3/8" packing was available at the time of the construction of this column.

COLUMN CONSTRUCTION

The column itself was constructed as follows:

The ends of the tube were sealed with rubber stoppers. In order to protect the rubber from direct contact with the oil, the stoppers were faced on the insides each with a layer of cork.

The bottom outlet tubing extended up into the tube about 4" above the stopper. A wire screen supported from the bottom stopper on brass legs was used as the support for the packing and it held the packing about 6" above the stopper, leaving the lower part open as the reboiler section.

The raschig rings were dumped loosely into the column to a packing height of 35" and an inch of 1/4" glass beads was laid on top to give better liquid distribution.

Five openings were supplied in the top stopper as follows: Liquid feed inlet, vapor outlet, vacuum gauge, thermometer, and reflux inlet. The liquid inlet tubing extended into the tube close to the top of the packing.
Sodium silicate solution was used as a cement to give a tight seal to the stoppers.

The column was then supported in an exactly vertical position and insulated with 1" thick magnesia pipe covering. Figure 3 shows the arrangement of the column and its outlets (the reflux inlet not shown) and photographs, Figure 5 and Figure 6, show the insulated column in place.

PRELIMINARY APPARATUS CONSTRUCTION

The apparatus as shown in the diagram, Figure 3, was the original arrangement used to attempt a laboratory distillation as a check on the calculations.

A 500 ml graduated dispensing burette, 1, was used as the feed storage. A laboratory condenser, 2, was used to preheat the feed by using hot water as heat source. The stopcock, 3, in the feed line was for control of the feed rate to the column, 6.

A standard bourdon tube dial vacuum gauge, 5, graduated from 0 to 30 inches of mercury vacuum was connected to the top of the column as well as a thermometer, 4, to record the temperature in the top of the column. A 50 ml burette not shown in the diagram was connected to the top of the column for feeding reflux. This burette was not shown as it was never actually used in the experiment.
The vapor line from the still passed through a cold water condenser, 12, and into a 250 ml graduated dispensing burette, 13, used as the distillate receiver. This receiver was also connected to the vacuum pump.

The bottoms line was connected by means of a 3-way stopcock, 9, to the bottoms receiving flask, 10, and the bottoms sampling flask, 11. These flasks were also connected to the vacuum pump.

The heat to the reboiler section of the still was furnished by a beaker, 7, to be filled with water and heated by means of a bunsen burner, 8.

A centrifugal vacuum pump, 14, used to maintain the system under the desired vacuum was driven by a 1/15 H.P. motor, 15.

All tubing used was of standard 10 mm diameter pyrex glass with tygon plastic tubing joints. For sealing the tubing into the equipment rubber stoppers were used with inside layers of cork where the oil would come in contact with the stoppers.
FIG 3 - PRELIMINARY ARRANGEMENT OF STRIPPING APPARATUS

NOMENCLATURE OF EQUIPMENT ON NEXT PAGE
<table>
<thead>
<tr>
<th>NUMBER</th>
<th>NAME OF EQUIPMENT</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>Feed Burette</td>
</tr>
<tr>
<td>2.</td>
<td>Feed Preheater</td>
</tr>
<tr>
<td>3.</td>
<td>Feed Rate Control Valve</td>
</tr>
<tr>
<td>4.</td>
<td>Top Temperature Thermometer</td>
</tr>
<tr>
<td>5.</td>
<td>Vacuum Gauge</td>
</tr>
<tr>
<td>6.</td>
<td>Stripping Column</td>
</tr>
<tr>
<td>7.</td>
<td>Bottoms Reboiler Heat Supply</td>
</tr>
<tr>
<td>8.</td>
<td>Bunsen Burner</td>
</tr>
<tr>
<td>9.</td>
<td>Bottoms Control Valve</td>
</tr>
<tr>
<td>10.</td>
<td>Bottoms Receiving Flask</td>
</tr>
<tr>
<td>11.</td>
<td>Bottoms Sampling Flask</td>
</tr>
<tr>
<td>12.</td>
<td>Overhead Condenser</td>
</tr>
<tr>
<td>13.</td>
<td>Distillate Receiver</td>
</tr>
<tr>
<td>14.</td>
<td>Vacuum Pump</td>
</tr>
<tr>
<td>15.</td>
<td>Vacuum Pump Motor Drive</td>
</tr>
</tbody>
</table>
OPERATION OF PRELIMINARY EQUIPMENT

As was expected, the original arrangement as described did not operate satisfactorily and as a result it has been tilted "preliminary." But in the attempt to operate this system its peculiarities were studied and as a result a final operable system was reconstructed.

The proposed operation was as follows:

Carbon disulfide was to be added to the xylene absorption oil to the amount of 0.15 mol percent and the mixture placed in the feed burette. A feed rate of between 100 cc/min and 150 cc/min (this rate would give a vapor velocity near the maximum of 1.5 ft/sec) was to be set by the feed control valve and as it was fed into the still under 28" Hg vacuum it could be heated in the feed preheater to the saturation temperature.

By siphoning action the bottoms would leave the still and enter either of the bottoms flasks. These flasks were connected directly to the vacuum pump in order to maintain the whole system under the same vacuum as the still itself and thus not hinder the siphoning action. The bottoms could be siphoned into the large collecting flask and at any time during the operation a sample of the bottoms could be collected in the sampling flask.
By maintaining the beaker surrounding the reboiler section of the still full of boiling water enough heat could be transferred into the still to keep the liquid boiling and thus maintain the necessary heat for distillation.

By circulating cold water through the overhead condenser the distillate could be collected in the receiver and its rate of flow determined.

It was proposed to furnish reflux liquid to the column from a burette at a desired rate to maintain a determined reflux ratio between total overhead product and refluxed liquid to the column. A mixture was to be made up corresponding to the theoretically calculated mixture of CS$_2$ and xylene in the distillate and all of the overhead then collected as distillate.

With the very first attempt at operation it was found that the pump would not hold a constant vacuum. As the operation proceeded the vacuum would continue to drop off due to the absorption in the pump oil of vapors high in CS$_2$ content that were not condensed. The vacuum pump operated against the vapor pressure of this oil and as vapors were absorbed the vapor pressure of the oil would rise and thus lower the vacuum possible to obtain.

In an attempt to correct this situation ice water was circulated through the condenser, and the distillate
and the bottoms receiving and sampling flasks were placed in ice baths. This improved but did not entirely correct the situation.

Below are listed the operating difficulties encountered which made necessary a revision of the system:

1. Inconvenience of the system using all the ice baths and ice circulation as mentioned.
2. No constant vacuum even with the ice baths and circulation.
3. Too high liquid level maintained in reboiler section of still.
4. Insufficient siphoning action of the bottoms product out of the column.
5. Formation of condensate in the lower U-bend of the suction line.
6. Insufficient capacity of the distillate receiver.
7. Inconvenient arrangement of the bottoms flasks for disconnecting equipment and removing liquid after a run.
8. Insufficient heating capacity of the feed preheater.
9. Inability to maintain constant flow from the reflux burette.
The cause of item 3 was due to the height of the bend in the bottoms tubing that was necessary in order to allow the reboiler section of the still to be submerged in the water bath (see Figure 3). Actual boiling of liquid took place up in the packed section of the column. The restriction to the flow of liquid by the 3-way stop-cock hindered the siphoning action mentioned in item 4. Other items are self explanatory.

It was found that additional thermometers were needed to determine the actual feed temperature of the liquid before entering the still and the temperature of the liquid in the reboiler section of the still.

REVISION OF APPARATUS

As a result of the difficulties that made the preliminary system impossible to operate satisfactorily, the apparatus was revised as shown in the diagram, Figure 4.

Between the feed preheater, 2, and the feed control valve, 4, an electrical heating element, 3, was wound around the tubing in order to furnish more heat and thus obtain a higher feed temperature than it was possible to reach with hot water alone. On the vacuum side of the feed control valve was installed a thermometer, 5, to indicate feed temperature before entering the column.

The revision in the reboiler section of the still
consisted of replacing the water bath with an electrical hot plate, 9, and a reflector, 11, thus supplying heat by radiation. This made it possible to install the thermometer, 10, to indicate bottoms temperature and allowed a lower liquid level to be maintained in the still by making it possible to decrease the height of the bend in the bottoms outlet line.

The replacement of the 3-way stopcock by two single stopcocks, 12, to each of the bottoms collecting flasks, 13 and 14, and a rearrangement of the connecting tubing eliminated the hindrance to the siphoning action and made handling of this equipment more convenient.

In order to eliminate the use of ice water baths and ice water circulation in the condenser and to maintain a constant vacuum, the vacuum pump and motor drive were replaced by a regular laboratory cold water aspirator, 20.

Other additions to the system necessary were an auxiliary condensate receiver, 17, to give greater capacity, a trap flask, 18, in the lower vacuum line to keep condensate from plugging the line and flowing into the sampling flask, 14, and a water trap, 19, in the line to the aspirator. Not shown in the diagram was a connection in order to hold the reflux liquid in the reflux burette under vacuum so that flow from the burette could be maintained by liquid head only.
**TABLE 3**

**NOMENCLATURE OF EQUIPMENT NUMBERED IN FIGURE 4**

<table>
<thead>
<tr>
<th>NUMBER</th>
<th>NAME OF EQUIPMENT</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>Feed Burette</td>
</tr>
<tr>
<td>2.</td>
<td>Feed Preheater</td>
</tr>
<tr>
<td>3.</td>
<td>Auxiliary Feed Preheating Coil</td>
</tr>
<tr>
<td>4.</td>
<td>Feed Rate Control Valve</td>
</tr>
<tr>
<td>5.</td>
<td>Feed Temperature Thermometer</td>
</tr>
<tr>
<td>6.</td>
<td>Top Temperature Thermometer</td>
</tr>
<tr>
<td>7.</td>
<td>Vacuum Gauge</td>
</tr>
<tr>
<td>8.</td>
<td>Stripping Column</td>
</tr>
<tr>
<td>9.</td>
<td>Bottoms Reboiler Heat Supply</td>
</tr>
<tr>
<td>10.</td>
<td>Bottoms Temperature Thermometer</td>
</tr>
<tr>
<td>11.</td>
<td>Radiation Shield for Heater</td>
</tr>
<tr>
<td>12.</td>
<td>Control Valves for Bottoms Flasks</td>
</tr>
<tr>
<td>13.</td>
<td>Bottoms Receiving Flask</td>
</tr>
<tr>
<td>14.</td>
<td>Bottoms Sampling Flask</td>
</tr>
<tr>
<td>15.</td>
<td>Overhead Condenser</td>
</tr>
<tr>
<td>16.</td>
<td>Distillate Receiver</td>
</tr>
<tr>
<td>17.</td>
<td>Auxiliary Distillate Receiver</td>
</tr>
<tr>
<td>18.</td>
<td>Suction Line Condensate Trap</td>
</tr>
<tr>
<td>19.</td>
<td>Aspirator Water Trap</td>
</tr>
<tr>
<td>20.</td>
<td>Water Aspirator</td>
</tr>
</tbody>
</table>
The photographs, Figures 5 and 6, show the final arrangement of the apparatus as described. The equipment is numbered on the photographs as in the diagram, Figure 4.

FINAL OPERATING PROCEDURE

It was decided to use a method of refluxing other than attempting to add a small amount of liquid to the column through the reflux burette at a constant and accurate rate. Since the feed was being introduced into the top plate of the column it was possible to accomplish the cooling effect of refluxing by introducing the liquid feed at a temperature below saturation instead of by the normal method of returning part of the distillate to the top of the column. It was believed that closer control of the feed temperature could be maintained than a reflux rate as originally proposed. Since the liquid feed temperature had to be controlled in either case, this method would also eliminate an additional variable. The method of converting the cooling effect of the liquid into a reflux ratio is given in the next section.

The operation of the equipment as revised could be maintained for a sufficient period of time with all conditions remaining constant for the system to reach equilibrium. A typical run would proceed as follows:
FIGURE 5
PHOTOGRAPH OF FINAL ARRANGEMENT OF STRIPPING APPARATUS
FIGURE 6
PHOTOGRAPH OF TOP SECTION
OF STRIPPING STILL
The system would reach a constant vacuum of near 28 inches of mercury and the column would heat up until distillate would start coming over in about 15 minutes. The feed rate would be set at about 100 to 125 cc/min and the distillation continued for about 10 minutes until conditions remained constant. Then for another 10-minute period the following data would be taken at one-minute intervals:

- Volume liquid fed
- Volume distillate collected
- Vacuum
- Temperature at top of still
- Temperature at bottom of still
- Feed temperature

Near the end of the run a sample of the bottoms product would be collected. At the end of the run the total volume of bottoms would be measured in order to determine a material balance over the system for the entire run. The feed mixture and the bottoms product would then be analyzed for CS₂ content.

**ANALYTICAL PROCEDURE**

An analytical procedure for the determination of CS₂ content of absorption oil which was simple and rapid yet accurate to the degree desired was worked out after
a study of various methods given in the literature.

By a method given by Altieri (1, p. 355) separation of the CS₂ from the oil was accomplished by its reaction with a 10% solution of KOH in ethyl alcohol. By addition of water to the oil-alcohol mixture the compound formed with the CS₂ and the alcohol would separate out into the water layer.

By the method used by the American Gas Institute (3) the water layer mixture was neutralized with acetic acid and titrated with 0.01 normal iodine solution to blue endpoint using starch as indicator.

EXPERIMENTAL RESULTS

In Appendix V are given the results of all the runs made with the distillation apparatus as described, followed by notes and comments on each run.

The first few runs made were merely trial and several of the runs following these were incomplete in data because of the use of an inaccurate thermometer in the feed line. The first run that was completely successful and could be used for comparison with theoretical calculations was #9. Five distillations of the two component system were successful using various reflux ratios followed by four successful distillations of the three component system. An abstract of the results of these
runs that were used as comparisons is given in Table 4.

With the exception of runs 3 and 5 all distillations were carried out with an attempt to keep all variables near the values of the theoretical calculations and constant from one run to the next.

The feed rates were held between 100 and 125 cc/min and no change was made in the heat input to the reboiler. It was found that unless this heat input was left constant at maximum there was danger of a sudden break in the distillation which would ruin the run. A convenient method to express the rates of flow for comparison was by the ratio of feed rate to overhead rate. Although the heat input to the reboiler was constant, these ratios show considerable variation as shown in Table 4.

The determination of the equivalent reflux ratio was made by the formula:

\[ R = \frac{(C).(F).\left(\frac{T_t - T_f}{D}\right)}{D} \]

Where:
- \( R \) = Reflux ratio
- \( F \) = Feed rate - cc/min
- \( T_t \) = Temperature of top of column - °F
- \( T_f \) = Temperature of feed - °F
- \( D \) = Overhead rate - cc/min
- \( C \) = Constant - 0.00263 for two component system
  - 0.00287 for three component system
### TABLE 4

**SUMMARY OF RESULTS OF EXPERIMENTAL DISTILLATIONS**

#### 2 Component System

<table>
<thead>
<tr>
<th>Run No.</th>
<th>Feed Rate (cc/min)</th>
<th>Ratio F/O</th>
<th>% CS$_2$ in Feed</th>
<th>% CS$_2$ Stripped</th>
<th>Reflux Ratio</th>
</tr>
</thead>
<tbody>
<tr>
<td>9</td>
<td>119</td>
<td>9.4</td>
<td>0.150</td>
<td>92.1</td>
<td>0.67</td>
</tr>
<tr>
<td>10</td>
<td>118</td>
<td>12.0</td>
<td>0.153</td>
<td>94.9</td>
<td>0.73</td>
</tr>
<tr>
<td>11</td>
<td>112</td>
<td>9.4</td>
<td>0.149</td>
<td>92.4</td>
<td>0.56</td>
</tr>
<tr>
<td>12</td>
<td>125</td>
<td>13.0</td>
<td>0.147</td>
<td>92.6</td>
<td>0.47</td>
</tr>
<tr>
<td>13</td>
<td>124</td>
<td>8.6</td>
<td>0.141</td>
<td>89.3</td>
<td>0.02</td>
</tr>
</tbody>
</table>

#### 3 Component System

<table>
<thead>
<tr>
<th>Run No.</th>
<th>Feed Rate (cc/min)</th>
<th>Ratio F/O</th>
<th>% CS$_2$ in Feed</th>
<th>% CS$_2$ Stripped</th>
<th>Reflux Ratio</th>
</tr>
</thead>
<tbody>
<tr>
<td>14</td>
<td>100</td>
<td>14.3</td>
<td>0.161</td>
<td>92.6</td>
<td>1.23</td>
</tr>
<tr>
<td>15</td>
<td>110</td>
<td>11.4</td>
<td>0.144</td>
<td>90.0</td>
<td>0.39</td>
</tr>
<tr>
<td>16</td>
<td>114</td>
<td>11.4</td>
<td>0.150</td>
<td>90.7</td>
<td>0.56</td>
</tr>
<tr>
<td>17</td>
<td>100</td>
<td>12.0</td>
<td>0.150</td>
<td>91.4</td>
<td>0.96</td>
</tr>
</tbody>
</table>
The derivation of this equation is given in Appendix VI.

The distillations were made with the vacuum system running full and only slight variations in its value were experienced. However, the three component system ran about one inch mercury less vacuum than the two component system.

On most runs overall material balances were made and each one checked closely on total input and output. On the result sheet these checks are noted by an OK. No balance of CS$_2$ in the system was possible as there was always some lost from the distillate as vapors. Therefore, there was no need to run a CS$_2$ determination on the distillate as this could easily be calculated.

Run #13 was an attempt to operate at zero reflux ratio. However, it was not quite possible to get the feed temperature up to the top temperature of the column and a slight reflux ratio resulted. Even with this small ratio the CS$_2$ removed was 89.3%.

With the exception of Run #9, all distillations show the decrease in CS$_2$ removal with decrease in reflux ratio which is in accordance with theory.

Run #3 was made solely for the purpose of checking the material balance of the system before the actual distillations were started. Run #5 was made in an attempt to limit the overhead product flow as much as possible.
As noted, even with four times the normal feed-overhead ratio 85% stripping was accomplished. At times other runs were started but not completed because of operating difficulties. Such runs were not recorded on the result sheet.
COMPARISON OF EXPERIMENTAL RESULTS WITH CALCULATIONS

Run #12 of the 2 component system and Run #16 of the 3 component system most closely approach the operating conditions used in the theoretical calculations. Table 5 shows this comparison.

**TABLE 5**
COMPARISON OF EXPERIMENTAL RESULTS WITH CALCULATIONS

<table>
<thead>
<tr>
<th></th>
<th>2 Component</th>
<th>3 Component</th>
</tr>
</thead>
<tbody>
<tr>
<td>CS$_2$ content of feed mol %</td>
<td>0.150</td>
<td>0.147</td>
</tr>
<tr>
<td>Vacuum - inches of Hg</td>
<td>28.0</td>
<td>28.6</td>
</tr>
<tr>
<td>Reflux Ratio</td>
<td>0.50</td>
<td>0.47</td>
</tr>
<tr>
<td>Ratio - Feed/Distillate</td>
<td>21.4</td>
<td>13.0</td>
</tr>
<tr>
<td>Top Temp. - °F</td>
<td>131.5</td>
<td>125.5</td>
</tr>
<tr>
<td>Bottom Temp. - °F</td>
<td>132.5</td>
<td>132</td>
</tr>
<tr>
<td>% CS$_2$ removed</td>
<td>95.0</td>
<td>92.6</td>
</tr>
</tbody>
</table>

The above table clearly indicates that the column did not act as 6 theoretical plates as designed for CS$_2$ removal did not reach 95% in either case, although the actual operating conditions approached those used in the calculations. A study of Table 4 shows that to reach the desired 95% stripping it was necessary to increase the reflux ratio to 0.73 in the 2 component system while with
the 3 component system it was not possible to reach 95% stripping with the highest reflux ratio experimentally used.

Calculations show that approximately the same size of column is necessary to accomplish the desired separation in either of the systems. However, as actually experienced the separation was less efficient to quite a degree with the 3 component system than with the 2 component system. Calculations show only a slight trend in this direction (indicate that theoretical plates for the 3 component system might be 6+ while for the 2 component system 6 even), but certainly not enough to explain this difference. Apparently the difference in operating variables between the two systems was enough to cause the column to act differently as a distillation unit.

The deviation of top temperatures of the column from calculated values is mainly due to the difference of the actual operating vacuum from the 28 inches of mercury value. The larger temperature differences from the top of the column to the bottom in the experimental work than calculated are due to the fact that the xylene did not act as a single compound but as a mixture as shown by its distillation curve, Figure 2.

With the experimental apparatus as set up it was not possible to obtain heat balance data for comparison with
that calculated.
CONCLUSIONS

The experimental data obtained compared with the theoretical calculations closely enough to prove either distillation system studied operable for the stripping of CS₂ from absorption oil.

The method of stripping as proposed by Schwarz (the 3 component system) would undoubtedly operate with less heat requirement than the simple 2 component system, but experimental results indicate it might actually require a larger stripping column.

If the operation on a commercial scale would be similar to that now used by Gasco, in that the overhead product of the stripping still would be combined with the light oil fraction from a benzene stripper and further refined, then loss of absorption oil in this system would not be important as it would later be recovered. It would then be possible to operate with the stripping section only of a distillation column and supply the reflux cooling by the method used in this study.

Before this system could be proved economically practical it would have to be compared with other systems of stripping possible, such as steam stripping, stripping with other condensable vapors, and stripping with non-condensable gases.
BIBLIOGRAPHY


7. Schwarz, S. C. Confidential report to Portland Gas and Coke co.


APPENDIX

ABSORBER DESIGN CALCULATIONS TO DETERMINE CS\textsubscript{2} CONCENTRATION IN ABSORPTION OIL

Design Data:

CS\textsubscript{2} to be absorbed from gas at 70°F and one atmosphere pressure.

Gas to contain 100 grains of CS\textsubscript{2} per 100 standard cubic feet of gas.

CS\textsubscript{2} content of scrubbed gas to be 5 gr./CCF

Oil used for absorption to be xylene, molecular weight = 106, sp. gr. = 0.86.

Equilibrium constant = 0.374 (See Appendix II).

Lean absorption oil to contain only 5\% of CS\textsubscript{2} present in rich absorption oil.

Design of this system by the British (4) uses slope of operating line = 1.3 times slope of equilibrium line.

Calculations

\[
Y_1 = \frac{(100)(359)(530)}{(7000)(76.1)(100)(492)} = 0.000725
\]

\[
Y_2 = (0.05)(0.000725) = 0.0000363
\]

\[
L/G = (0.374)(1.3) = 0.486
\]

Graphical solution as given by Sherwood (8, p.82-89) gave the following data:

\[
X_1 = 0.001500
\]

\[
X_2 = 0.000075
\]

Theoretical Plates required = 12
The system as designed seemed reasonable and, therefore, the values of $X_1$ and $X_2$ as found were used as basis for the stripping system studied.

NOMENCLATURE

$Y_1$ - concentration of CS$_2$ in rich gas - mols/mol

$Y_2$ - concentration of CS$_2$ in lean gas - mols/mol

$X_1$ - concentration of CS$_2$ in rich oil - mols/mol

$X_2$ - concentration of CS$_2$ in lean oil - mols/mol

$L/G$ - slope of operating line - ratio of mols of liquid to mols of gas
APPENDIX II
COMPARISON OF EXPERIMENTAL EQUILIBRIUM CONSTANTS FOR CS₂ WITH RAOULT'S LAW

From data given by the American Gas Association (2) the distribution coefficient, $K'$, expressed as $x/y$ where $x$ and $y$ are concentrations of CS₂ in terms of pounds per cubic foot of liquid and gas, respectively, at 73.5°F and one atmosphere pressure for CS₂ in kerosine and hydrocarbon gas is 263.

Assume kerosine of sp. gr. = 0.81, molecular weight = 190

Expressing this equilibrium constant as $K = Y/X$ in concentration terms of moles/mol:

$$K = \frac{(359)(535)(62.4)(0.81)}{(263)(492)(190)} = 0.395$$

Assuming Raoult's Law and using vapor pressure data as given in Appendix IV at this Temperature and pressure:

$$K_p = \frac{\text{partial pressure of CS}_2}{\text{total pressure}} = \frac{345}{760} = 0.454$$

Using the above method of calculation, comparison of the equilibrium constants given by the British (4) with Raoult's Law:

<table>
<thead>
<tr>
<th>Temp. °F</th>
<th>$K'$</th>
<th>$K$</th>
<th>$K_p$</th>
</tr>
</thead>
<tbody>
<tr>
<td>60</td>
<td>335</td>
<td>0.301</td>
<td>0.325</td>
</tr>
<tr>
<td>70</td>
<td>275</td>
<td>0.374</td>
<td>0.407</td>
</tr>
<tr>
<td>80</td>
<td>227</td>
<td>0.461</td>
<td>0.503</td>
</tr>
<tr>
<td>90</td>
<td>191</td>
<td>0.558</td>
<td>0.618</td>
</tr>
</tbody>
</table>
APPENDIX III

THEORETICAL CALCULATIONS OF STRIPPING SYSTEM

TWO COMPONENT SYSTEM

ASSUMPTIONS

Operation at 28" Hg vacuum = 49 mm Hg abs. press.
Reflux Ratio = 0.5
Overhead vapor to leave in equilibrium with feed
Feed enters as saturated liquid
Feed composition

| CS₂ | 0.15 mol % | 0.0306 |
| C₈H₁₀ | 99.85 mol % | 0.9694 |

Bottoms to contain 0.0075 mol % CS₂

CALCULATION OF DISTILLATE COMPOSITION

At 131.5°F

<table>
<thead>
<tr>
<th>Comp.</th>
<th>X</th>
<th>P</th>
<th>XP</th>
<th>Y</th>
</tr>
</thead>
<tbody>
<tr>
<td>CS₂</td>
<td>0.0015</td>
<td>1022</td>
<td>1.5</td>
<td>0.0306</td>
</tr>
<tr>
<td>C₈H₁₀</td>
<td>0.9985</td>
<td>47.5</td>
<td>48.9</td>
<td>1.0000</td>
</tr>
</tbody>
</table>

Distillate composition

3.06 mol % CS₂
96.94 mol % xylene

CALCULATION OF NUMBER OF THEORETICAL PLATES

Basis: 100 mols feed

CS₂ balance: 0.15 = 0.0306D + 0.000075W

W = 100 - D

D = 4.67 mols
\[ W = 95.33 \]

Reflux ratio, \( O/D = 0.5 \)

\[ O_n = (0.5)(4.67) = 2.34 \]

\[ V_n = O_n + D = 2.34 + 4.67 = 7.01 \]

\[ V_m = V_n \]

\[ O_m = O_n + F = 2.34 + 100 = 102.34 \]

Operating line for \( CS_2 \)

\[ Y_m = \left( 0 \right) \frac{(X_{m+1})}{W} \frac{V_m}{X_w} \]

\[ Y_m = 14.6X_{m+1} - 0.00102 \]

Plate-by-plate calculations starting at bottom at 132.7°F

<table>
<thead>
<tr>
<th>Comp.</th>
<th>( X_w )</th>
<th>P</th>
<th>XP</th>
<th>( Y_w )</th>
<th>( X_1 )</th>
</tr>
</thead>
<tbody>
<tr>
<td>CS(_2)</td>
<td>0.000075</td>
<td>1040</td>
<td>0.079</td>
<td>0.0016</td>
<td>0.00018</td>
</tr>
<tr>
<td>C(<em>8)H(</em>{10})</td>
<td>0.999925</td>
<td>49.0</td>
<td>49.0</td>
<td>0.9984</td>
<td>0.99982</td>
</tr>
</tbody>
</table>

continuing up the column to the 6th plate at 131.7°F

<table>
<thead>
<tr>
<th>Comp.</th>
<th>( X_5 )</th>
<th>P</th>
<th>XP</th>
<th>( Y_5 )</th>
<th>( X_6 )</th>
</tr>
</thead>
<tbody>
<tr>
<td>CS(_2)</td>
<td>0.00105</td>
<td>1023</td>
<td>1.085</td>
<td>0.0221</td>
<td>0.00158</td>
</tr>
<tr>
<td>C(<em>8)H(</em>{10})</td>
<td>0.99895</td>
<td>47.95</td>
<td>49.0</td>
<td>0.9779</td>
<td>0.99842</td>
</tr>
</tbody>
</table>

The \( CS_2 \) concentration of the 6th plate agrees closely with the feed concentration, therefore, a column of 6 theoretical plates can be assumed necessary to accomplish the desired separation.
THREE COMPONENT SYSTEM

ASSUMPTIONS

Operation conditions same as in two component system except for feed composition

Feed composition

<table>
<thead>
<tr>
<th>Comp.</th>
<th>X</th>
<th>P</th>
<th>XP</th>
<th>Y</th>
</tr>
</thead>
<tbody>
<tr>
<td>CS₂</td>
<td>0.0015</td>
<td>710</td>
<td>1.063</td>
<td>0.0217</td>
</tr>
<tr>
<td>C₆H₆</td>
<td>0.0930</td>
<td>218</td>
<td>20.27</td>
<td>0.4143</td>
</tr>
<tr>
<td>C₈H₁₀</td>
<td>0.9055</td>
<td>30.5</td>
<td>27.6</td>
<td>0.5640</td>
</tr>
</tbody>
</table>

Distillate composition

2.17 mol % CS₂
41.43 mol % benzene
56.40 mol % xylene

CALCULATION OF NUMBER OF THEORETICAL PLATES

Basis: 100 mols feed

By CS₂ balance - D = 6.58 mols

W = 93.42 mols

By benzene balance - bottoms composition -
0.0075 mol % CS₂
7.0500 mol % benzene
92.9425 mol % xylene

0/D = 0.5

0ₙ = 3.29 Vₙ = 9.87 Vₘ = Vₙ

0ₘ = 103.29
Operating line for CS$_2$

\[ Y_m = 10.48X_{m+1} - 0.00071 \]

Operating line for C$_6$H$_6$

\[ Y_m = 10.48X_{m+1} - 0.667 \]

Plate-by-plate calculations starting at bottom at 117°F

<table>
<thead>
<tr>
<th>Comp.</th>
<th>(X_m)</th>
<th>P</th>
<th>XP</th>
<th>(Y_m)</th>
<th>(X_1)</th>
</tr>
</thead>
<tbody>
<tr>
<td>CS$_2$</td>
<td>0.000075</td>
<td>790</td>
<td>00.06</td>
<td>0.0012</td>
<td>0.00018</td>
</tr>
<tr>
<td>C$_6$H$_6$</td>
<td>0.070550</td>
<td>245</td>
<td>17.27</td>
<td>0.3495</td>
<td>0.0969</td>
</tr>
<tr>
<td>C$<em>8$H$</em>{10}$</td>
<td>0.929</td>
<td>345</td>
<td>32.05</td>
<td>0.6492</td>
<td>0.9030</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>49.4</td>
<td>1.0000</td>
<td>1.0000</td>
</tr>
</tbody>
</table>

continuing up the column to the 6th plate at 109°F

<table>
<thead>
<tr>
<th>Comp.</th>
<th>X$_5$</th>
<th>P</th>
<th>XP</th>
<th>Y$_5$</th>
<th>X$_6$</th>
</tr>
</thead>
<tbody>
<tr>
<td>CS$_2$</td>
<td>0.00107</td>
<td>675</td>
<td>0.727</td>
<td>0.015</td>
<td>0.0015</td>
</tr>
<tr>
<td>C$_6$H$_6$</td>
<td>0.1072</td>
<td>207</td>
<td>22.2</td>
<td>0.457</td>
<td>0.1072</td>
</tr>
<tr>
<td>C$<em>8$H$</em>{10}$</td>
<td>0.8917</td>
<td>288</td>
<td>25.7</td>
<td>0.528</td>
<td>0.8913</td>
</tr>
<tr>
<td></td>
<td>48.6</td>
<td></td>
<td>1.00</td>
<td>1.00</td>
<td></td>
</tr>
</tbody>
</table>

The CS$_2$ concentration of the 6th plate agrees closely with the feed composition, therefore, a column of 6 theoretical plates can be assumed necessary to accomplish the desired separation.
SUMMARY OF HEAT REQUIREMENTS

Basis: 100 mols feed - reference temperature of 70°F

Two component system:

Heat content of overhead 134,000 Btu
Heat content of bottoms 258,500 Btu

Total 392,500 Btu

Three component system:

Heat content of overhead 164,100 Btu
Heat content of bottoms 185,500 Btu

Total 350,600 Btu

Physical data obtained from values given in Appendix IV

NOMENCLATURE

D = volume of distillate, mols
F = volume of feed, mols
O = volume of overflow down the column, mols
P = vapor pressure, mm Hg
V = volume of vapor passing up the column, mols
W = volume of bottoms product, mols
X = concentration of solute in liquid, mols/mol
Y = concentration of solute in vapor, mols/mol

Subscripts: n refers to any plate above the feed plate
m refers to any plate below the feed plate
w, 1, 5, & 6 refer to particular plates
APPENDIX IV
PHYSICAL DATA

VAPOE PRESSURE DATA

Figure 7 gives the vapor pressure curves of carbon disulfide, benzene, and xylene. The curve for xylene is the numerical average of the values for ortho-, meta-, and para-xylene.

SPECIFIC HEATS

<table>
<thead>
<tr>
<th>Component</th>
<th>$70^\circ F$</th>
<th>$110^\circ F$</th>
<th>$130^\circ F$</th>
</tr>
</thead>
<tbody>
<tr>
<td>CS$_2$</td>
<td>0.235</td>
<td>0.235</td>
<td>0.235</td>
</tr>
<tr>
<td>C$_6$H$_6$</td>
<td>0.405</td>
<td>0.425</td>
<td>0.440</td>
</tr>
<tr>
<td>C$<em>8$H$</em>{10}$(mixture)</td>
<td>0.395</td>
<td>0.410</td>
<td>0.420</td>
</tr>
</tbody>
</table>

LATENT HEATS OF VAPORIZATION

<table>
<thead>
<tr>
<th>Component</th>
<th>$T_v$ - $^\circ F$</th>
<th>$L_v$ - cal/gm</th>
</tr>
</thead>
<tbody>
<tr>
<td>CS$_2$</td>
<td>115</td>
<td>84.09</td>
</tr>
<tr>
<td>C$_6$H$_6$</td>
<td>176</td>
<td>94.17</td>
</tr>
<tr>
<td>C$<em>8$H$</em>{10}$(mixture)</td>
<td>282</td>
<td>81.71</td>
</tr>
</tbody>
</table>

The above data was taken from Chemical Engineers' and Chemistry and Physics handbooks.
FIG. 7
VAPOR PRESSURES

TEMPERATURE - °F

VAPOR PRESSURE - MM Hg

CARBON DISULFIDE

XYLENE

BENZENE
# APPENDIX V

## EXPERIMENTAL RESULTS

<table>
<thead>
<tr>
<th>Run No.</th>
<th>Date</th>
<th>Rate (g/min)</th>
<th>Ratio F/O</th>
<th>Vacuum °Hg</th>
<th>Bottom Temp.</th>
<th>Top Temp.</th>
<th>Feed Temp.</th>
<th>CS₂ content mol %</th>
<th>% CS₂ Reflux</th>
<th>Matl Balance</th>
</tr>
</thead>
<tbody>
<tr>
<td>2 component system</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1</td>
<td>5-1</td>
<td>136</td>
<td>11.0</td>
<td>12.4</td>
<td>28.3</td>
<td>132</td>
<td>123</td>
<td>0.150</td>
<td>0.0042</td>
<td>97.4</td>
</tr>
<tr>
<td>2</td>
<td>5-5</td>
<td>117</td>
<td>9.6</td>
<td>12.2</td>
<td>28.3</td>
<td>130</td>
<td>121</td>
<td>0.162</td>
<td>0.0070</td>
<td>95.7</td>
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<tr>
<td>3</td>
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<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>OK</td>
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<tr>
<td>4</td>
<td>5-8</td>
<td>100</td>
<td>6.8</td>
<td>14.7</td>
<td>28.6</td>
<td>126</td>
<td>117</td>
<td>0.143</td>
<td>0.0090</td>
<td>93.8</td>
</tr>
<tr>
<td>5</td>
<td>5-12</td>
<td>197</td>
<td>4.8</td>
<td>40.7</td>
<td>28.4</td>
<td>129</td>
<td>123</td>
<td>0.063</td>
<td>0.0130</td>
<td>85.0</td>
</tr>
<tr>
<td>6</td>
<td>5-13</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>28.8</td>
<td>129</td>
<td>116</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>7</td>
<td>5-15</td>
<td>118</td>
<td>10.0</td>
<td>11.8</td>
<td>28.8</td>
<td>128</td>
<td>119</td>
<td>0.149</td>
<td>0.0141</td>
<td>90.7</td>
</tr>
<tr>
<td>8</td>
<td>5-20</td>
<td>103</td>
<td>12.5</td>
<td>9.3</td>
<td>28.6</td>
<td>128</td>
<td>120</td>
<td>0.139</td>
<td>0.0190</td>
<td>86.4</td>
</tr>
<tr>
<td>9</td>
<td>5-20</td>
<td>119</td>
<td>12.7</td>
<td>9.6</td>
<td>28.7</td>
<td>129</td>
<td>121</td>
<td>0.150</td>
<td>0.0120</td>
<td>92.1</td>
</tr>
<tr>
<td>10</td>
<td>5-26</td>
<td>118</td>
<td>9.8</td>
<td>12.0</td>
<td>28.2</td>
<td>132</td>
<td>124</td>
<td>0.153</td>
<td>0.0080</td>
<td>94.9</td>
</tr>
<tr>
<td>11</td>
<td>5-26</td>
<td>112</td>
<td>12.0</td>
<td>9.4</td>
<td>28.2</td>
<td>132</td>
<td>125</td>
<td>0.149</td>
<td>0.0125</td>
<td>92.4</td>
</tr>
<tr>
<td>12</td>
<td>5-26</td>
<td>125</td>
<td>9.6</td>
<td>15.0</td>
<td>28.6</td>
<td>152</td>
<td>126</td>
<td>0.147</td>
<td>0.0110</td>
<td>92.6</td>
</tr>
<tr>
<td>13</td>
<td>5-30</td>
<td>123.5</td>
<td>14.5</td>
<td>8.7</td>
<td>28.1</td>
<td>134</td>
<td>130</td>
<td>0.141</td>
<td>0.0152</td>
<td>89.3</td>
</tr>
</tbody>
</table>

### 3 component system

<table>
<thead>
<tr>
<th>Run No.</th>
<th>Date</th>
<th>Rate (g/min)</th>
<th>Ratio F/O</th>
<th>Vacuum °Hg</th>
<th>Bottom Temp.</th>
<th>Top Temp.</th>
<th>Feed Temp.</th>
<th>CS₂ content mol %</th>
<th>% CS₂ Reflux</th>
<th>Matl Balance</th>
</tr>
</thead>
<tbody>
<tr>
<td>14</td>
<td>6-3</td>
<td>100</td>
<td>7.0</td>
<td>14.3</td>
<td>27.4</td>
<td>144</td>
<td>116</td>
<td>0.161</td>
<td>0.0125</td>
<td>92.6</td>
</tr>
<tr>
<td>15</td>
<td>6-4</td>
<td>110</td>
<td>9.7</td>
<td>11.4</td>
<td>27.4</td>
<td>144</td>
<td>122</td>
<td>0.144</td>
<td>0.0140</td>
<td>90.0</td>
</tr>
<tr>
<td>16</td>
<td>6-4</td>
<td>114</td>
<td>10.0</td>
<td>11.4</td>
<td>27.4</td>
<td>145</td>
<td>122</td>
<td>0.150</td>
<td>0.0140</td>
<td>90.7</td>
</tr>
<tr>
<td>17</td>
<td>6-6</td>
<td>100</td>
<td>8.3</td>
<td>12.0</td>
<td>27.6</td>
<td>144</td>
<td>120</td>
<td>0.150</td>
<td>0.0131</td>
<td>91.4</td>
</tr>
</tbody>
</table>

OK
<table>
<thead>
<tr>
<th>Run No.</th>
<th>Remarks</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>No feed temperature and thus no reflux ratio obtained. Accuracy of CS₂ determination doubtful.</td>
</tr>
<tr>
<td>2.</td>
<td>Same as #1</td>
</tr>
<tr>
<td>3.</td>
<td>This run was made as a check on the material balance of the system only</td>
</tr>
<tr>
<td>4.</td>
<td>An inaccurate thermometer gave erroneous feed temperature readings from this run on until #8.</td>
</tr>
<tr>
<td>5.</td>
<td>An experiment at high feed rate and high ratio F/O. Still no accurate or constant overhead rate.</td>
</tr>
<tr>
<td>7.</td>
<td>Discovery of the inaccurate thermometer in feed line.</td>
</tr>
<tr>
<td>8.</td>
<td>First run with complete data. Believed bottoms sample contaminated thus showing high CS₂ content.</td>
</tr>
<tr>
<td>10.</td>
<td>Good run.</td>
</tr>
<tr>
<td>11.</td>
<td>Good run.</td>
</tr>
<tr>
<td>13.</td>
<td>Attempt to operate with feed at saturation and thus zero reflux ratio.</td>
</tr>
<tr>
<td>14.</td>
<td>Good run. Start of 3 component system distillations</td>
</tr>
<tr>
<td>15.</td>
<td>Feed rate not very constant</td>
</tr>
<tr>
<td>17.</td>
<td>Good run.</td>
</tr>
</tbody>
</table>
APPENDIX VI

REFLUX CALCULATIONS

Cooling effect of feed entering less than saturated:

\[ H_f = (F)(S)(C_p)(T_t - T_f) \]

where:

- \( H_f \) = cooling effect, cal/min
- \( F \) = feed rate, cc/min
- \( S \) = specific gravity of feed
- \( C_p \) = specific heat, cal/gm/°C, \( = 0.41 \)
- \( T_t \) = temperature of top of column - °F
- \( T_f \) = temperature of feed - °F

\[ H_f = 0.228 F S (T_t - T_f) \]

Conversion of \( H_f \) into equivalent cc/min of overhead assuming same cooling effect by vaporizing overhead material:

\[ H_o = \frac{H_f}{(L_v)(s)} \]

where:

- \( H_o \) = Equivalent reflux of overhead, cc/min
- \( L_v \) = Heat of vaporization of overhead, cal/gm, \( = 86.6 \) for 2 component sys. \( = 79.3 \) for 3 component sys.
- \( S \) = Specific gravity of distillate (assume same as feed)

Reflux Ratio, \( R = \frac{H_o}{D} \)

\( D \) = overhead rate cc/min

Substituting:

\[ R = \frac{0.00263 F (T_t - T_f)}{D} \] for 2 component system

\[ R = \frac{0.00287 F (T_t - T_f)}{D} \] for 3 component system