ABSORPTION COEFFICIENTS IN

LIGHT OIL SCRUBBERS.

by

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Professor G. W. Swett Secretary of the Faculty Massachusetts Institute of Technology Cambridge, Mass.

Dear Sir:

In partial fulfillment of the requirements of the degree of Master of Science, we respectfully submit this thesis entitled '' Absorption Coefficients in Light Oil Scrubbers. ''

Yours very truly,

Edward C. Forbes

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I SUMMARY

To obtain absorption coefficients for the light oil scrubbers at the Bethlehem Steel Company, Lackawanna, New York, it is necessary to know the equilibrium relationship of the vapor pressure of the various components of light oil over light oil-straw oil mixtures. The present investigators have determined the equilibrium pressures of benzene over benzene straw-oil mixtures at a temperature of 26° . The first method employed, was the analysis of the benzene-air mixture, existing in equilibrium over benzene-straw oil mixtures, by the methods of gas anal-In spite of numerous precautions, check results vsis. were impossible to obtain by this method. The second method consisted in measuring the increase in vapor pressure when a bulb containing benzene was broken in, and mixed with, straw oil contained in a closed vessel. This vessel was connected with a water manometer in which the level could be adjusted so that the total gas volume of the vessel, holding the straw-oil, plus the attendant tubing, remains The vapor pressure of benzene was measured over constant. a concentration range of 0 - 0.125 mol fraction of benzene and within this range was found to bear a linear relationship in mol fraction of benzene in the liquid phase. At the temperature employed ($26^{\circ}C$) and within the abovementioned concentration ranges the vapor pressures averaged 80.5 per cent of those calculated by Raoult's Law.

A number of runs were made on the plant light-oil scrubbers to determine their absorption coefficients. One test, performed on the north-east rich tower, gave a value for Kg a of 0.11 # mols of benzene/hr;/cu.ft;/(atm.) at an oil rate of 520 and a gas rate of 212 #/hr./sq.ft. However, this result is not believed to be reliable due to the difficulty of determining small differences in the concentration of benzene in the outlet and inlet gas streams. A Kg a for toluene of 0.3 # mols toluene/hr.)/cu.ft.(atm.) has been calculated from the same run but is thought to be more inaccurate than the value for benzene.

Two runs have been made on the south-east lean tower and the values of K_g a obtained were 0.320 and 0.336 # mols of benzene/(hr.)/cu.ft.)(atm.) at an oil rate of 557 and an average gas rate of 210 (214; 206 respectively) #/hr/sq. ft.) These values are lower than reported by former investigators (5)(12) but are so because, until now, Raoult's Law was presumed to hold and secondly the active surface of the wooden tower packing has decreased with time due to the deposition of foreign material. However, a recalculation of the values obtained by the present investigators assuming Raoult's Law to hold, gives a reasonable check with the values obtained by a former group (12) on the rich tower.

In the fractionation of certain samples, difficulty was experienced in determining the amounts of toluene and xylene.present. This was thought to be due to the relatively

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large "hold-up", of the present fractionating column and it was suggested that in the future a thinner or even shorter column be used.

II INTRODUCTION

The abundance of natural coal deposits promotes every kind of industry for human welfare, either as a source of heat or power. Yet in certain instances, the human civilization depends not only upon the heat or power supplied by coal, but also upon the reducing characteristics of coke.

Historically, coke was manufactured by the beehive oven process, but as the demand of coke for metallurgical purposes increased as time went along, large scale production required a thoroughly scientific investigation of the entire coking process. It was found that, during coking with air excluded, the volatile matter in the coal was, more or less, pyrolyzed and gasified to form the oven gas, leaving the fixed carbon in the coke. For each ton of coke produced, about thirteen thousand cubic feet of gas was evolved and since each cubic foot of this gas has a heating value of approximately 500 BTU., this gas could be very well utilized as a valuable fuel. Evidently the rejection of the exit gas in the beehive oven process encurred a great loss of potential energy and thus proved very uneconomical.

Further investigation proved that coke oven gas contained many valuable constituents such as ammonia, phenol, naphthalene, benzene, toluene, xylene and tar. For industries producing either gas or coke as their main product,

the recovery of the valuable by-products in the coke oven gas proved a very profitable practice, as all of these materials are employed in the chemical industries.

The evolution of the coking process from the uneconomical beehive to the most modern by-product oven showed remarkable progress with respect to efficiencies of yield of coke from coal and also the large recovery of by-products.

Coke, with outstanding characteristics for metallurgical purposes, is very extensively used. For large scale production of iron and steel, the daily consumption of coke in the blast furnace and tremendous volumes of gas needed for steel manufacturing and treatment is great enough to warrant that steel plants manufacture their own coke and coke-oven gas by establishing coking systems on location.

The Bethlehem Steel Company, Lackawanna, New York, maintains a daily production of four thousand tons of pig iron and consumes about six thousand tons of coke in blast furnace operation. Several million cubic feet of coke oven gas are also used in the period of a day, for numerous purposes throughout the plant. These large demands of coke and coke oven gas are supplied by the coke ovens located in the same plant.

For the coking of coal, there are four batteries of by-product ovens each of which has a capacity of twelve tons of coal. Three of these, consisting of fifty-seven oven per battery, are of the Koppers type and are heated by



producers gas which is produced from the coke produced in these ovens. The other battery, consisting of sixty ovens, is of Semet-Solvay type and is heated by coke oven gas generated in the latter ovens.

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After the introduction of coal, the red hot oven is sealed and the coal is then heated for approximately nine hours after which it is discharged. The gas emitted from the top of the oven during the coking process is led through a hydraulic main where most of the tar is precipitated, and a major part of the ammonia is dissolved. The gas from the hydraulic main is then washed countercurrently with water in a tower, for further removal of tar and ammonia. The washings from the tower and the liquids from the hydraulic main are mixed in settling tanks where tar separates from the liquor. After settling, part of the clear liquid is recirculated to the hydraulic main, the remainder is passed through a trombone cooler and then conducted to the ammonia distillation tower, and the phenol recovery column. Ammonia from the distillation tower and coke oven gas which still contains small amount of ammonia are both passed through sulphuric acid saturators. The ammonia reacts almost quantitatively with the acid to form ammonium sulphate, the resulting crystals settle in tanks, are then passed through centrifuges and finally dried in a rotary drier. Daily production of ammonium sulphate amounts to fifty-five tons.

The coke-oven gas, now freed from ammonia, phenol, and most of the tar, passes countercurrently through a water



spray tower in which most of the naphthalene is separated out and flows with water into a settling tank where it is skimmed off as crude product.

The gas from the spray tower flows to the light oil recovery system. This system consists of two sets of scrubbers arranged in parallel. Each set has two scrubbers in series, one for rich oil, and the other for lean oil. Each scrubber,15.5 feet in diameter and 123 feet in height, with a packed volume of 15,000 cubic feet, is packed with wood slats of $1/4^{"} \times 5-3/4^{"}$ set on edge, geparated by $1/2^{"}$ spacers (Figure II).

Gas, from the main, divides into two streams and flows (Figure I) from the bottom to the top of the two parallelly arranged rich scrubbers, while the partially benzolized straw oil which has been mixed after coming from the lean towers, is pumped to the top of the rich scrubbers. The gas from the top of the rich scrubbers, and still containing an appreciable amount of light oil, is further scrubbed countercurrently with stripped strawoil in the lean scrubbers.

After passing through the scrubbers, the coke oven gas flows to a booster house from where it is distributed to various locations in the steel and coke plant.

The straw oil from the bottom of the rich scrubbers, is stripped with steam and the vapor mixture passes through a fractionating column in which benzene, toluene and xylene, and steam are separated from each other. The denuded straw-

oil is cooled in trombone coolers and then recirculated to the lean scrubbers.

The daily production is approximately 11,500 gallons of benzene, 3300 gallons of toluene, and 855 gallons of xylene.

Since the production of light oil in the operation of the by-product coke oven plant $is_{\Lambda}^{\sigma^{\prime}}$ large scale and the knowledge of capacity coefficients in the light oil scrubbers is valuable for design and operating purposes, the object of this investigation was to study the operating conditions of the scrubbers and to determine the absorption coefficients.

In order to determine the coefficient of absorption accurately it is proposed to determine rate of oil flow and to analyze the concentration of light oil in the inlet and outlet of both oil and gas streams by the most accurate methods at hand. With the necessary data on temperature of both streams and pressure of the gas stream and a laboratory determination of the equilibrium of light oil in straw oil it is possible to calculate the coefficients of absorption.

It is not the intention of the authors to go extensively into the theory of absorption since there are many excellent textbooks on this subject,(6(8) (11) but a short explanation of the use of the term, absorption coefficient or Kga, would be advisable.

In the transfer of material between a gaseous and a liquid phase, it is found that the following equation represents the conditions during diffusion.

N_a = mols per unit time per unit area= $k_g(p_g - p_i)$ = $k_1(C_i - C_l)$

where $k_g = gas$ film coefficient

k₁ = liquid film coefficient

- pg = partial pressure of the solute in the main gas phase
- p_i = partial pressure of the solute at the interface
 between gas and liquid

Furthermore it is known that k_g is a function of temperature, total pressure, mean pressure of inert, as well as the various factors which determine the value of the diffusivity, and of the effective gas film thickness. Similarly, k_l is a function of solute concentration, diffusivity in the liquid and the effective liquid film thickness.

It is, naturally, very difficult to measure interface conditions but it is found, in the case of very soluble gases, that p_i is very nearly equal to p_e , the equilibrium value corresponding to C_1 . Likewise in the case of relatively insoluble gases Ci equals C_e , the equilibrium value corresponding to p_g . In both these cases, the film coefficients are now overall in character and can be written as K_g and K_l . For intermediate cases, the resistance of each film must be considered. However, it is convenient to employ a single overall coefficient instead of the two film coefficients k_g and k_l and we have our choice of two overall coefficients as defined by the equation 12

 $N_a = K_g (p_g - p_e) = K_l (C_e - C_l)$ K_g is the overall coefficient in terms of pressures, K_l the overall in terms of concentrations. One important consideration should be noted, and it is, that $(p_g - p_e)$ remains proportional to $(C_e - C_l)$ over the employed range of pressures and concentrations in order that K_g and K_l will remain constant. In other words, Henry's law (C = Hp) should apply to the system.

We have now defined an overall coefficient K_g to be the rate of interphase diffusion as mols per unit time per unit area per unit of driving force in terms of pressure. However, in the case of packed towers the interfacial area is practically impossible to measure, consequently, we introduce a new variable, a, defined as the interfacial area per unit of volume. Because both a and K_g depend to a very large extent on the nature of the packing, they are combined as a product which is then employed as an overall capacity coeffidecient on a volume basis. In the present investigation, the units employed for K_g a are pound mols per hour per cubic foot per atmosphere. The values for K_g a were calculated knowing the total pound mols transferred in the tower over a given period of time (total transfer obtained from a material balance), the packed volume of the tower, and the use of the proper driving force for diffusion (in this case, the logarithmic mean of the overall driving forces in terms of pressures at the top and bottom of the tower.)

IN REVIEW OF PREVIOUS WORK

A number of investigations on the operating conditions, of the old light-oil scrubbing tower from 1922 until 1930 and the new towers after 1930, have been made at the Buffalo Station, School of Chemical Engineering Practice.

Generally, a material balance around a light oil scrubbing tower was obtained by measuring the oil rate, and analyzing the light oil concentrations in the inlet gas stream, and the inlet and outlet oil streams.

The methods employed were as follows:-

1. Measurement of oil rate.

By closing the outlet value at the bottom of the scrubber and measuring with a stopwatch, the time required for the oil to rise a definite height, the oil rate can be calculated by knowing the inside diameter of the scrubber.

2. Measurement of gas rate.

a. In the early tests the gas rate was not measured but was obtained from plant data.

b. In later tests the gas rate was obtained by two methods:

(1) By a pitot tube traverse

(2) By calculations on the operation of the gas compressor.

<u>3. Determination of light-oil concentration in the</u> gas.

For the determination of the concentration of light oil it was necessary to first, remove light oil from the gas; second, to record the volume of light oil free gas; third, to strip off light oil from the absorbing reagent, and finally, to fractionally distill the light oil into its components.

To perform these operations the following methods were used.

a. To remove light oil from gas.

1. By means of activated charcoal.(3)(9)(10)

The dried gas was passed through a column packed with activated charcoal which acted as an absorbing reagent for the light oil. It was claimed that by using activated charcoal for gas, rich in light oil, very inconsistent results were secured.

2. By absorption of light oil in ether.

The dried gas was bubbled through ether in a bottle which was immersed in some kind of freezing agent to keep the temperature at -5° C. The gas from the bottle was led through a condenser coil cooled with a dry-ice gasoline mixture to a temperature not higher than -60° C., and then through a fractionating column cooled to the same temperature. By keeping the temperature low, not only the light oil was completely removed from the coke-oven gas but the loss of ether was negligibly small. It was claimed that this method was very satisfactory for the tests. The volume of coke-oven gas was measured by a wet gas meter in series with the absorber equipment.

b. Stripping of light oil from activated charcoal.(3)(9)(10)

The light oil absorbed by the activated charcoal was stripped by means of steam. The mixture of light oil vapor and steam was condensed and water separated. The light oil portion was then distilled in a fractionating volumn and the constituents were collected at their respective boiling temperatures.

c. Distillation of light oil.

When ether was used as the absorption reagent there was no necessity of stripping as in the case of the activated charcoal. The ether solution was run directly into a flask and fractionally distilled.

<u>4. Determination of the concentration of light oil</u> <u>in the oil streams.</u>

a. Sampling of oil in both streams.

Equal amounts of oil were taken from time to time during the run and mixed to obtain a composite sample.

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b. Stripping of light oil from the benzolized straw oil.

In the early tests, an oil sample of definite volume was stripped in a flask by means of steam which was generated in another flask. The oil was first warmed and then steam was passed into the flask. During distillation the oil was heated with an open flame to prevent any condensation of vapor before it reached the condenser.

It was claimed in later tests that by using steam under 5-10 lb. gauge pressure for both heating in closed coils, and stripping as live steam, a quicker and much more complete stripping could be expected.

c. Fractional distillation of light oil.

After the vapor was condensed and water was separated the light oil portion was fractionally distilled as mentioned above.

5. Determination of equilibrium of light oil between the vapor and liquid phases.

For the calculation of absorption coefficients in previous tests, it was assumed that Raoult's Law held for cases of gas and liquid phases in equilibrium. Since this assumption was doubtful, several experimental methods were employed to determine the vapor pressures of benzene in equilibrium with benzene-straw oil mixtures. a. Davis method.

Two flasks were connected to a manometer and freshly stripped oil was introduced into one of the flasks. Into the flask containing oil, a sealed glass bulb containing a definite weight of benzene was placed. Both flasks were opened to the air so that the manometer levels were the same and then the stoppers were closed. After the bulb was broken by some mechanical means, the vapor pressure of benzene was indicated by the difference of height in the manometer.

b. The combustion method. (i)(iz)

Vapor of a definite volume and containing benzene in equilibrium with liquid was dried by passing through calcium chloride tubes and then mixed with dry oxygen. The mixture was then burned in a combustion tube. The products of combustion were then collected and weighed in suitable absorption towers as used in quantitative analysis.

c. Gas analysis method. (2)

A certain volume of oxygen was measured in the burette and stored in the caustic pipette. A certain volume of vapor in equilibrium with liquid was next measured in the burette. The vapor and oxygen were mixed and burned in the combustion pipette. Carbon dioxide was absorbed by caustic. Gas free, from carbon dioxide, was again measured in the burette. The concentration of benzene in the vapor was calculated from the volume of contraction.

IV PROCEDURE

To obtain equilibrium data for benzene vapor pressure over benzene-straw oil mixtures, several procedures were followed. The first method tried, was to analyse samples of the gas in equilibrium with mixtures of benzene and straw oil. This was accomplished by the use of a measuring burette, a caustic absorption pipette, and a slow combustion pipette. In all cases the gas volumes were not measured volumetrically, but gravimetrically by displacement of the confining fluid which was either water or mercury. A measured volume of air was first taken into the burette and then passed into the slow combustion pipette. A sample of the equilibrium gas-mixture was next measured into the burette and a slow combustion performed. The products of combustion were then absorbed in the caustic pipette and the total contraction determined.

The second method employed to obtain equilibrium data was as follows. A glass bottle resting in a constant temperature bath was partially filled with a known amount of straw oil. Onto the flask was placed a sealed glass bulb containing a known amount of benzene. The system was closed completely by a shellacked rubber cork containing a glass tube leading to a water-filled manometer., The glass bulb was then broken, the bottle and its contents shaken, and the increase in vapor pressure noted on the manometer. The latter instrument was fitted with a leveling



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bottle so that the level of the liquid, in the manometer leg connected to the flask, was always at the same height. By this means the volume of the bottle plus the connected tubing, remained constant. By changing the amounts of benzene used, a number of runs were made at various benzene concentrations.

To obtain absorption coefficients for the operation of the plant scrubbers, the following procedure was followed. An absorption train as shown in Figure TVwas fitted to the sampling pipe on the downtake from the tower being investigated. A similar train was also fitted to the downtake from the preceding tower in the series. In this manner samples could be obtained of the entering and leaving gas streams from the tower. Gas from the tower was then allowed to flow through each train at the rate of approximately 20 cu.ft. per hour and sampling was continued until approximately 200 cu.ft. of gas had passed, the exact amount being read from the wet gas meter. During this period, grab samples of oil were taken separately from the entering and leaving streams. These were then mixed together to give two composite samples. At frequent intervals, the valve on the exit oil line from the scrubber was closed and the time required for the oil in the sight glass, to rise a given height was noted. Temperatures of the oil streams were also obtained from time to time by the use of thermometers and the gas stream temperatures were read from



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: ۲ برین plant installations. At the termination of the run, the bottle containing the light oil and ether was disconnected, metallic sodium was added to the contents and, after remaining several hours, the contents were fractionated in an apparatus as shown in Figure $\overline{\sqrt{1}}$ The straw oil samples were stripped in an apparatus shown in The water and light oil mixture, obtained Figure *▼* ٠ from the stripping operation, was then placed in a separatory funnel, the light oil being run into a bottle where it was dried with calcium chloride. The light oil was then fractionated in a column as shown in Figure VI The actual details of procedure and the precautions which had to be observed, may be found in the Appendix under "Details of Procedure".

From the above measurements, it was possible to calculate the concentration of light oil in the inlet and outlet gas and oil streams; and also the oil rate in the plant tower. It was then possible to calculate the mols of benzene absorbed in the tower for a given period of time. Since we also knew the packed volume of the tower as well as the vapor-liquid equilibria, Kga could be calculated. From the preceding data, a material balance around the tower would yield the gas rate, a knowledge of which would serve as a check on the results because it was known that the gas rate was relatively constant for the three runs.

All data was reported on a pound mol basis and it was not necessary to know the average molecular weight of the coke oven gas although this latter figure was found to 12.5 from analysis and plant records. From the data of previous investigators, (\mathcal{S}) it was considered that the absorption of light oil in the apparatus shown in Fig.IV was quantitative. 24a.





X RESULTS

1. The method of obtaining vapor pressure equilibrium data of benzene and straw oil mixtures by the use of gas analysis has proven unsatisfactory.

2. Vapor pressure equilibrium data obtained by the method of breaking a glass bulb, containing benzene, in a constant volume vessel, containing straw oil, and noting the increase in vapor pressure, is believed to be satisfactory. Data obtained by this method show that the vapor pressure of benzene over benzene-straw oil mixtures averages 80.5 per cent of that calculated from Raoult's Law at a temperature of $26 \circ C$. Unfortunately, time did not allow blank runs to be more that benzene indicates for which the found to be satisfield. The Kg afor benzene calculated from a run on

5. The Agaior benzene calculated from a run on the north-east rich tower was found to be 0.11 # mols of benzene per hour per cu.ft. per atmosphere at a gas rate of 212 and a liquor rate of 520 lbs. per hr. per sq. ft. Due to inaccuracy in the analyses of gas samples, this result is not believed to be reliable.

4. The absorption coefficients resulting from two runs on the south-east lean towers were 0.320 and 0.338 # mols of benzene per hour per cubic foot per atmosphere at an oil rate of 520 lbs. per hour per square foot and an average gas rate of 212 lb. per hour per square foot. These two results are believed to be reliable.

N. Summarized Results

1. Calibration of wet gasmeter

Table XVIII					
Run	Gasmeter A	Gasmeter B			
	Actual vol.	Actual vol.			
	Recorded Vol.	Recorded vol.			
At the rate of 5 c	u. ft. per hour.				
1	0.9930	0.9900			
2	0.9810	0.9789			
3	0.9839	0.9904			
Average	0.9863	0.9864			
At the rate of 20 cu. ft. per hour					
1.	0.9863	0.9664			
2	0.9838	0.9912			
3	0.9908	0.9877			
4	0.9791	0.9827			
Average	0.9850	0.9872			

2. Equilibrium of benzol between liquid and vapor phases

Vapor phase(Rault's Law) Liquid Phase Run 1 Mol Mol Mol Mol fraction fraction Ratio ratio 1 0.0578 0.05470.00770 0.00763 $\widetilde{X}_{1,p}$ 0.01326 2 0.1056 0.0956 0.013430.00932 3 0.0718 0.0672 0.00940 0.01750 0.01718 0.1422 0.1247 4

Tab le XIX

Table XIX continued					
Run	Vapor pha	Vapor phase(experimental)		Experimental	
	Mol Ratio	Mol fraction	kau From mol ratio	lt's Law From mol fraction	
1	0.00662	0.00598	78.2	78.4	
2	0.01088	0.01075	81.0	81.2	
3	0.00729	0.00723	77 ; 7	77.7	
4	0.01500	0.01475	85.7	86.0	

3. Specific gravity of straw oil

Table XX

Oil	True vol. of pipette c.c.	Weight of oil gm	Specific gravity
Stripped Oil	25.13	22.03	0.877
Inlet Oil	25.13	22.083	0.878
Outlet Oil	25.13	22.1027	0.880

4. Summarized Data for Plant Runs.

Table XXI

Run	I	II	III
Mol fraction of benzol in inlet rich gas	0.00927	0.00700	0.00585
Mol ratio of benzol in inlet gas	0.00939	0.00703	0.005875
Mol fraction of toluol in inlet gas	0.001253	0.000056	0.0000541
Mol ratio of toluol in inlet gas	0.001268	0.0000594	0.0000544
Mol fraction of benzol in outlet gas	0.00756	0.00303	0.002306
Mol ratio of benzol in in outletslean gas	0.00757	0.00304	0.002310
Mol fraction of tolutl in outlet gas	0.000918	0.0000845	0.0000398

		Table XXI	continued	
	Run	I	II	III
•	Mol ratio of toluol in outlet gas	0.000928	0.0000847	0.0000400
	Mol fr _a ction of benzol in inlet oil	0.03224	niñil	. iil
V	Mol ratio of benzol in inlet oil	0.0334	nil	nil
,	Mol fraction of toluol in inlet oil	0.00138	nil	nil
	Mol ratio of toluol in inlet oil	0.00143	nil	nil
· .	Mol fraction of benzol in outlet oil	0.0486	0.0361	0.0314
V	Mol ratio of benzol in outlet oil	0.0517	0.0374	0.03242
	Mol fraction of toluol in outlet oil	0.01294	0.0000948	0.0000611
	Mol ratio of toluol in outlet oil	0.01388	0.0000984	0.0000631
	Oil rate lb mols per hour	323 2	346	345
	Inert gas rate from benzol balance lb mo per hour	ols 3,230	3,250	3142 ¹
	Kga lb mol benzol per cu. ft., per hour, per atmosphere	0.11	0.320	Q•338
	K _g a lb mol toluol per cu. ft., per hour per atmosphere	0.33		
	Kga lb mol of benzol per cu. ft. per hour per atm. assuming Rault's Law holds	0.175	0.418	0.373
	N. T. U.		1.54	4.67
	H. T. U.		52. 5	48.0 [/]

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Ta	ble XXI -	- continued	
Run	I	II	III
Efficiency of benzol recovery in tower%	19.4	56.8	60.7
Lost of benzol assum- ing the mol ratio of benzol in the inlet gas of the first run is good for the rest	4	7 D 4	24 6
two runs %		32.4	24.6

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FIGURE VIII -











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IGURE XX



IGURE IAX

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FIGURE XVII



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VI DISCUSSION OF RESULTS

Method of Gas Analysis for Equilibrium Data

The exact reason why it was impossible to obtain check results of vapor pressure data from the method of gas analysis, is not clear. One possible source of error was thought to result from the method of introduction of the gas sample into the gas measuring burette. This was accomplished by raising a leveling bottle, holding water, and connected to the bottle containing the benzene-straw oil mixtures. The resultant increase in pressure was used to force the gas, above the straw oil surface, into the gas measuring-burette. At first, the pressure of the gas was not measured and variations may have occurred during several analyses. However, by introducing a manometer in parallel with the line leading from the straw-oil vessel to the gas burette and keeping a constant pressure during several analyses, no improvement was noted in the agreement of results.

The possibility was considered that the use of water as the confining fluid in the gas burette might cause error. The solubility of benzene in water is very low (6) but with the very weak benzene-air mixtures employed, any possibility of diffusion of bezene into or out of the confining fluid had to be obviated. Consequently, mercury was employed as the confining fluid, both in combustion and gas measuring burettes. Still no improvement was noted in the ability to check results.

Methods of manipulation could not be blamed for non-concordance of results, because several operators carried out the analyses with the greatest of care and differing lengths of time were employed for both the slow combustion and absorption of carbon dioxide.

The sole remaining possibility was that the combustion of the weak benzene-air mixtures was very irregular. For the concentrations used, there was at all times excess air for the combustion and no errors could have resulted from this cause. The filament temperature was varied from a dull red to a white heat and it was endeavored to pass every small portion of the gas sample past this filament several times. However, no agreement of results was obtained by this method and the authors can offer no further explanations.

Method of Vapor Pressure Measurement for Equilibrium Data

The method of breaking a bulb containing benzene proved satisfactory for obtaining vapor pressure equilibrium data. However, certain conditions must be strictly observed if this method is to be successful. In the first place, the containing vessel and tube leading to the mercury manometer should be kept at a constant temperature. The system behaves not only as a means of measuring increased vapor pressure due to the addition of benzene to the straw oil but also as a gas thermometer. This latter property must be strictly controlled and to ensure this condition a very sensitive thermometer should be employed in conjunction with a well-constructed, constant-temperature bath. The authors experienced some difficulty in maintaining constant temperature conditions with the present bath and a somewhat improved set-up would be advantageous. Room temperature conditions should also be controlled. The vent connected to the pressure vessel and shown in should always be opened, to obtain atmospheric Figure m pressure, before breaking the bulb. Certain conditions with respect to the bulb had to be met. Since the initial pressure in the straw oil vessel was atmospheric, it was necessary that the same total pressure should exist over the benzene in the bulb. The manner of filling the bulbs was such, that care had to be exercised to ensure the benzene was cooled to the temperature of the thermostat before sealing the end of the bulb. Bulbs made and sealed on one day should therefore not be used on a second day when atmospheric pressure is much different. An error that appeared in several of our runs resulted from insufficient shaking after the breaking of the benzene-containing bulb. In these cases, benzene tends to remain in the top portion of the liquid and inordinately high vapor pressures are It was assumed that in the missing of the benzene shown. and straw oil that no net expansion or contraction took place. The method of breaking the glass bulbs, by shaking

the container, proved rather tedious and it is suggested that some other means of accomplishing this end, be devised.

By observing the above rather numerous conditions, as closely as possible, a number of runs were made on benzene-straw oil mixtures of varying concentrations at 26.6°C. The results of these runs are shown in Figures VII-IX and agree fairly well with a linear relationship over the employed range of concentrations. The straight line on Figures \overline{vs} , \overline{N} , drawn through the experimentally determined points, represents equilibrium vapor pressures of benzene over benzene-straw oil mixtures as being \$1.0 percent of those calculated from Raoult's Law. On first thought, it would seem that the vapor pressure of benzene over these mixtures should obey Raoult's Law more closely but when we consider the dissimilarity in both molecular structure and weight between benzene and the components which make up the straw oil, our results are not illogical.

It should be further noted that we assumed the deviation from Raoult's Law, for the temperature at which our plant runs were made, was the same as at the temperature which was employed for our equilibrium runs. This seems logical because the temperatures were only slightly different and we have no reason to suppose that the change in the deviation from Raoult's Law is large with respect to temperature changes.

Plant Scrubber Runs

The run on the north-east rich tower gave us a value of K_{ges}^{α} of 0.11 # mols of benzene per hour per cubic feet per atmosphere at a gas rate of 212 and oil rate of 520 #/hr./ft². This value seems inordinately low compared to the figure obtained from the lean tower of .320 and .338 # mols benzene/hr.+ft². - atm. It is very probable that all the xylenes and a large part of the toluene will be absorbed in the rich tower due to the relatively lower volatility of these substances and the consequently larger driving force. On the other hand, the straw-oil and gas rates are such that, if the conditions in the rich tower are depicted graphically, the operating line and benzene equilibrium line are close to each other which means a low driving force for diffusion. Consequently there is only a small amount of benzene absorption in the tower. As a result of this, samples of the entering and exit gas streams, although they may show easily measurable quantities of benzene, may show only a very small difference in the amounts of benzene contained in the two streams. Since these differences are the quantities used directly for the calculation of K_{gas} , it is easily seen that the inaccuracy in Kgas for the north-east rich tower may be large. For example, with a benzene difference of maybe 10 mls. in the two samples and an unavoidable fractionation error of approximately 0.5 m.l., the inaccuracy in this measurement is apparent.

The possibility exists that greater accuracy may be obtained by recovering the benzene from much larger gas and liquid samples. The objection to this procedure rests in the great length of time required to obtain such large gas samples.

An additional factor which would reduce the benzene absorption in the rich tower, is the higher oil temperatures encountered. This would also tend to reduce the driving force for diffusion of benzene from gas into the liquid.

The physical condition of the wood cribbing in the rich tower will greatly affect absorption coefficients. It is known that these towers accumulate, in time, a considerable amount of tar, naphthalene and other foreign materials; the direct result, of which, is to reduce to a considerable extent the active surface area employed for absorption. This would, in turn, yield a lower value for the calculated K_{ga} . The length of time which had elapsed since the last cleaning of the tower was not known but was probably much longer than a year. It was known that the lean south-east tower had been cleaned about a year previously and the rich tower had been cleaned some time previous to that date.

Lean Tower Results

The results obtained from two runs made on the south-east lean tower checked fairly well. The values obtained for the Kg a's were 0.320 and 0.338 # mols benzene/hr./ft]³(atm) at an average gas rate of 210 #/hr./ft² and oil rate of 557 #/hr./ft². As mentioned before, the accuracy with which we could determine values for Kg a depended to a large extent on the magnitude of the difference in the amounts of benzene collected from the inlet and outlet gas samples. Fortunately, in the case of the lean tower this difference was fairly large, about 53 m/. m.l. in contradistinction to 21 m.l. in the case of These easily measurable differences the rich tower. allowed us to calculate our Kg a with a fair degree of accuracy, and we believe that the values obtained approach closely the true figure for the present operation of the south-east scrubber at the Bethlehem Steel Company. Data given in Sherwood (8) indicate rather higher values of Kg a than obtained by the present investigators; about .6 # mols/ (hr)/cu.ft.) (atm) at a gas rate of 240 #/hr./ft). and a liquid rate of 680 $\#/hr./ft^2$) We believe there are several reasons for this. The previous investigators (5) assumed that the vapor pressure of benzene in benzene-straw oil mixtures obeyed Raoult's Law. This assumption would, consequently, lead to smaller values for the log A_P term, for comparable conditions of operation, and, resulting from

this, larger values for Kg a were obtained in this report. On the other hand no data has been obtained on the individual film coefficients and the resulting effect of varying It follows that the value of Kga liquor and gas rates. may be considerably changed by variations in the gas or It is very likely that the gas-film is liquor rate. controlling and we should expect the gas rate to have the major effect on the value of Kg a and the liquor rate to play only a minor part. To check our results for both the effects of the deviation from Raoult's Law and different gas rate, our average Kga has been recalculated assuming Raoult's Law to hold and the resulting Kg a has been assumed to vary with the 0.8 power of G(# gas/hr./ft².) By doing so, we obtain a value of Kg a equal to .36 compared to .6 # mols/hr)/ft²)./atm) at the same gas rate as employed by (5)It would appear that there • must be some further reason why the Kga as obtained by the present investigators are lower than those formerly obtained.

A third reason for a lower value of Kg a in the present report is the aforementioned effect of the clogging of the wood packing by foreign material. The values for Kg a, obtained by (s), were done shortly after the tower had been cleaned; our values were obtained after the tower had been in operation for one year.

KG as for toluene and xylene

In the present investigation it was impossible to obtain absorption coefficients for toluene and xylene. The mechanical difficulties encountered in obtaining even satisfactory amounts of benzene in the gas samples, practically precluded obtaining measurable quantities of toluene and xylene. However, the design of a somewhat larger ether bubbler and condenser coil equipment should not prove difficult, thereby allowing an increased gas flow resulting in the collection of sufficient amounts of toluene and xylene in a reasonable period of time. A Kga of 0.3 # mol of toluene/hr//ft.³Hatml was calculated from rather meager data obtained from the run on the rich tower.

It should be noted that the use of the oil rate has been employed in obtaining the material balance around the tower. The greater $\frac{accuracy}{use}$ with which the oil rate could be measured led to its adoption for calculation purposes.

Sample Treatment

In the collection of light oil from the gas samples, one very important point was noted. Previous investigators (2) had noted that the occurrence of ethanol in the ether used for absorption of light oil was not uncommon. Since the boiling points of ethanol and benzene are so close, 75.3°C. and 50.1°C. respectively, grave errors would be introduced into the fractionation analysis for benzene if unknown amounts of ethanol were present. To guard against this possibility, all the ether used for absorption purposes was not only redistilled but also kept over calcium chloride, metallic sodium and clean iron nails.

On the temperature + volume graphs obtained from the fractionation of the light oil-ether mixtures, the amount of pure substance present was taken as that volume which distilled in the range of approximately $\pm 5^{\circ}$ C. of the true boiling point for the given material. Reference to Figures $\overline{x} - x\overline{x}$ will show that this was a logical assumption. This procedure was very satisfactory for benzene but, in the case of toluene and xylene, the amounts of these two substances present were so small that a reasonably flat portion in the distillation curve was very difficult to obtain. As mentioned before, the only means of securing accuracy in the measurement of the amounts of toluene and xylene would be the absorption of light oil from larger gas samples or the employment of a somewhat thinner, or possibly shorter, fractionation column in which the "hold-up" would be considerably reduced. The use of 1/4" copper wire spirals proved to be successful as packing material in the fractionation column and by the addition of naphthalene to the distillation flask practically all of the light oil fractions could be distilled off. Heat loss, however, was considerable from the column and it was necessary to lag very thoroughly towards the end of the distillation in order to prevent total condensation in the lower portions of the column.

During the stripping of the straw oil samples, several precautions had to be observed. To ensure that the last traces of light oil were removed from the straw oil, stripping was continued until the temperature in the vapor phase had risen to 220° F. and napthalene started to separate out in the condenser. The light oil-water mixture was then placed in separatory funnels, salt added, and sufficient time given for the system to completely separate into two phases. Particular care had to be taken that small light oil bubbles did not remain in the water fraction.

Molecular Weight of Straw Oil

In all the calculations of the present test, the average molecular weight of straw oil has been taken as 304. This agrees with the values obtained by a previous group (/2) and it does not seem probable that appreciable change in the molecular weight will have taken place since the previous determination.

Note on Kg a Obtained by a Previous Group

In addition to the K_g a of 0.6 # mols of benzene/ (hr.)/cu.ft.)/atm.) calculated by Lukes (5); a value of 0.358 #

mols benzene/hr.)(cu.ft.)(atm.) has been obtained by Walker(12) for the rich tower. This latter figure is rather close to that obtained for the lean tower by the present investigators and if the incorrect assumption is made that Raoult's Law holds, as Walker did, our average value would be 0.39 # mols of benzene/hr.)(cu.ft.)(atm.) In view of the sufficiently close agreement in these absorption coefficients determined independently and at different times, it is felt that the capacity coefficients determined by the authors are representative for the present operating conditions at the Bethlehem Steel Company, Lackawann, New York.

A recalculation of Walker's data using the correct vapor-liquid equilibrium data gives a value of 0.292 lb.mols benzene/hr.-cu.ft.-atm. for $K_{g}a$.

VII. CONCLUSIONS

I. The method of gas analysis is unsatisfactory for obtaining equilibrium vapor pressure data over benzenestraw oil mixtures for concentrations up to 0.125 mol fraction of benzene.

<u>II.</u> The method of direct vapor pressure measurement is satisfactory for obtaining equilibrium vapor pressure data over benzene straw oil mixtures for concentrations up to 0.125 mol fraction of benzene.

<u>III.</u> A value of 0.11 # mols benzene/(hr.)(cu.ft.)(atm.) as the absorption coefficient for the north-east rich tower is not believed to be reliable.

<u>IV</u>. Values of K_g a of 0.320 and 0.338 # mols benzene/ hr./ft.³ (atm.) at an oil rate of 557 and an average gas rate of 210 (214; 206) #/(hr.) (sq.ft.) are believed to be accurate for the south-east lean tower.

 \underline{V} . The capacity of the present absorption train is too small to obtain a sufficient light oil sample in a reasonable time.

<u>VI.</u> The present fractionating column is too large for dilute samples.

VIII. RECOMMENDATIONS

<u>I.</u> That an improved apparatus be constructed for the determination of vapor pressure equilibria by the bulb method. Particular care should be paid to temperature control and means of breaking the bulb.

<u>II.</u> That vapor pressure equilibria be obtained at different temperatures in an effort to determine the deviation from Raoult's Law at different temperatures.

<u>III.</u> That values of K_g a be obtained from the west towers in an effort to determine the effect of time after cleaning on the absorption coefficient.

<u>IV.</u> That the effect on K_g a of varying the gas rate be determined, if acceptable to plant authorities.

V. That a thinner and maybe shorter fractionating column be used for the light oil distillation.

<u>VI.</u> That a larger capacity absorption train be constructed or the two smaller ones be run in parallel.

APPENDIX

Expansion of Procedures

1. Determination of the benzol equilibrium curve by means of gas analysis.

a. Preparation of samples.

Straw oil from the stripper in the benzol recovery plant was stripped again in the laboratory in order to remove the last trace of light oil. About 1,000 grams of this oil was weighed accurately by difference, and poured into a three liters glass bottle.

Benzol, approximately equal to the amount which was necessary to make the desired mol fraction in this sample, was weighed accurately by difference in a corked Erlenmyer flask, and emptied into the bottle.

The bottle was immediately corked tightly with a stopper through which wase inserted a thermometer, and two bent glass tubings of different length. To the short glass tubing was connected a petce of rubber tubing, partially clamped. The long glass tubing, extending almost to the bottom of the bottle, was connected to a large leveling bottle, filled with water, by a long rubber tubing, clamped in the middle.

The leveling bottle was raised and the clamp opened until the long gla ss tubing was completely filled with water. Then both clamps were tightly closed. The bottle was well shaken and immersed, up to its neck, in a constant temperature water bath. When the temperature of the gas in the bottle was about the same as that of the water bath, the short glass tubing was opened to air for a short time in order to bring the pressure in the bottle to atmospheric.

I. Sample collected over water

The regular Fisher's gas analysis set was used except that the bottom of the burette was connected to the leveling bottle by a three way stopcock. By turning the stopcock to one position, water in the leveling bottle was allowed to come in contact with the water in the burette, turning to the other position water in the burette was allowed to be drained out to a receiver.

A gas sample was drawn into the burette by displacing the gas in the bottle with water, and displacing the water in the burette with the gas. The water removed from the burette through the three way stopcock was collected in a weighed beaker. The water was accurately weighed and its temperature measured. The pressure of gas in the burette was brought to atmospheric by opening the stopcock at the top to air for a few seconds.

Since at low mol fractions of benzol in the gas resulted, from the low mol fraction of benzol in the liquid phase, according to Rault's Law, there was more than enough oxygen present in the gas for perfect combustion, no fresh air or pure oxygen was added. The gas was run slowly into the combustion pipette, passing over the glowing filaments. This was repeated for ten to twelve times in order to secure complete combustion. Then the gas was bubbled through the caustic pipette several times to absorb all the carbon dioxide. The residual gas was brought to atmospheric pressure in the usual manner. The stopcock at the top of the burette was opened,
and the three way stopcock was turned to such a position that water in the burette was drained slowly into a weighed beaker. The three way stopcock was closed when the level of water reacha field the mark on the lower neck of the burette. The water was weighed and its temperature measured.

The volumes of the gas sample and the residual gas were calculated from the densities and weights of water observed. Having previously calibrated the volume between the zero mark and the mark on the lower neck of the burette, the decrease in volume was calculated.

Since perfect combustion requires, for each volume of benzol, seven and one half volumes of oxygen, a contraction of eight and one half volumes is betained. Thus the mol fraction of benzol in the gas was calculated from the volume of contraction.

II. Sample collected over mercury -

All the operations of gas analysis were the same as in "I" except that mercury was used for water in the burette and combustion pipette, allowing only a very small volume of water to cover the surface of mercury in the burette and pipette. Since mercury is 13.6 times heavier than water the weight of mercury, displaced by gas, was obstained by dividing the mercury into several 200 grams portions and weighing accurately on an analytical balance.

2. Determination of the benzol equilibrium curve by measuring the

partial vapor pressure directly.

e. in permiten of Nervel surpre-

a. Preparation of benzol sample

A clean, dry, long stem, thin walled glass bulb was accurately weighed, then warmed gently over a small flame. While it was still hot the opening of the stem was immersed in pure benzol. On cooling, benzol was sucked in due to the contraction of theair left in the bulb. When enough benzol had been collected, the bulb was removed, bringing the opening to the top. It was then cooled in air. After it was cooled sufficiently the opening was sealed by a narrow flame. The bulb was then weighed.

b. Preparation of manometer.

A U-tube was prepared with a peace of galss tubing sealed at the bottom of the U. To one arm of the U-tube was connected a prace of straight glass tubing, to the other a 90° glass elbow with a mark made one inch below the bent . The end of the glass tubing at the bottom of the U-tube was connected through a stopcock to a leveling bottle filled with slightly colored water.

c. The equilibrium bottle

The equilibrium bottle used was a wide mouth glass bottle of 1.8 liters capacity stoppered with rubber. The stopper carried glass elbows, tenth degree Centigrade thermometer and a hook from which was hanging a petce of string. Both sides of the stopper were coated with shellac to prevent leakage, and the absorption of benzol by rubber. After the stopper was inserted the thermometer would be two inches above the surface of the cil.

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d. Constant temperature bath

The water bath consisted of two galvanized iron buckets placed one inside the other. In bewween the two buckets, layers of cotton and glass wool were inserted as insulation, The inner bucket was filled with water.

e. Operation --- figure III

About 700 grams of restripped oil was weighed accurately by difference and poured into the partially wetted equilibrium bottle. The benzol bulb was hung on the string by means of Scotch tape. The stopper was carefully inserted and painted further with shellac. After the shellac was dried the botthe was immersed in the constant temperature bath up to its neck. One tube was connected to the bended arm of the manometer while the other was partially clamped. When the temperature of the air in the bottle became the same with that of the bath, the level of the two arms was adjusted to the mark of the bended arm, the stopcock closed and the glass tubing on the bottle was tightly wlamped. The benzol was b broken by shaking the bottle gently. The shaking was continued for a few times more to ensure thorough mixing. Since the equilibrium bottle was air tight, the addition of benzol vapor in the gas phase would cause an increase in pressure, and consequently the levels of the two arms of the manometer would be ultimately changed. When the temperature again became the same with that of the bath and the change of the manometer level ceased, the level of the bended arm was brought exactly back to its original mark by raising the leveling bottle and opening the stopcock. After this was ad mater. the secondariance encoded and the bid protoco in herea

adjusted exactly, the stopcock was closed, and the difference in level of these two arms was read accurately to 0,025 inch.
3. Determination of the specific gravity of oil

Water from a 25 c.c. pipette was accurately weighed and its temperature measured. The pipette was thoroughly dried.

From the density and weight of water and the weight of starw oil, the specific gravity of the oil at that temperature was calculated.

4. Material balance around the light oil scrubbing tower.

a. Preparation of ether for the removal of light oil from the gas

Ether of technical purity was dried over fused calcium chloride and decanted into a distilling falsk. By connecting the flask to the apparatus as shown in figure VI, the distillation and rectification of ether were accomplished. Pure ether was collected between the temperature of 345 °C. Pure ether thus preparaed was stored in a corked bottle with a few peices of sliced metallic sodium. A bent glass tubing with a very small opening at the top was inserted through the cork. The small opening allowed the release of any hydrogen generated by the action of water and sodium, but prevented the escape of ether.

b. Sampling and removal of light oil from gas.

Coke oven gas samples were taken from the inlet and outlet gas streams of the tower. A manometer connected through a tee in the sampling line measured the total pre-

ssure of the gas as shown in Figure IV. The temperature of the inlet gas was taken from the a utomatic recording chart. The rate of sampling was controlled by a valve in the line. The gas sample was led first through a trap to remove entrained droplets of oil water, and tar, then dried by passing through four calcium chloride drying bottles arranged in series. This dried gas was allowed to bubble through the ether in the extraction bottle, from which it passed through a condenser cooled in a dry ice-gasoline mixture, the temperature was kept constantly below -60°C and checked frequently with a copper -constanta, thermocouple and millivoltmeter. The last section of the condenser consisted of a column packed with copper rings which removed the last trace of light oil, and almost all of the ether left in the gas. Condensate flwoing down this column wahsed all the condensed light oil into the bottle through a pipe, one end of which was connected to the bottom of the column and the other was sealed by the ether in the extraction bottle. Finally the remaining inert gas was passed through a wet gas meter to measure its volume and then into open air. A thermometer and a manometer were connected to the wet gas meter to measure the temperature and pressure.of the exit gas.

c. Measuring the oil rate.

By temporarily shutting off the value of the outlet oil stream at the bottom of the tower, anf measuring, by means of a stop watch, the time required for the oil to raise a certain height as observed in the sight glass, it was possible to secure accurately the rate of oil flow in terms of inches height

per minute. With the cross section of the bottom of the tower known from the design data of the plant, the volume rate of flow could be calculated.

d. Oil temperature and sampling.

Samples of the inlet oil was taken every half an hour from the line in the pump house, and of the outlet oil from the value at the bottom of the tower. The temperatures of both streams were measured during sampling.

e. Stripping of oil samples. --- Figure V

From a definite volume or weight of oil sample the light oil was stripped in the apparatus as shown in Figure V. In stripping the oil was heated to about 100 °F by passing steam of 10 1b gauge through the heating coils. Then live steam was passed into the oil. Vapor generated from the oil by the steam was first passed through a packed column for the separation of the high boiling constituents, and then through the condenser. The condensate was collected in a receiver which was immersed in an ice water bath to prevent the vaporization of low boiling constituents of the light oil. The stripping was continued until the condenser was fully coated with a layer of naphthalene. The condensate was then separated in a separating funnel and the water layer washed with saturated brine to recover as much of the dissolved light oil as possible. The light oil was then dried for a few hours over calcium chloride.

f. Rectification of light oil

The light oil samples recovered from the gas or liquid streams (see sections b and e) were rectified separately in the apparatus as shown in Figure VI. Light oil sample was introduced into the distilling flask to which 100 grams of pure naphthalene was also added. The flask was then connected to the bottom of the rectification cloumn. During rectification, vapor from the liquid was first passed a one meter long column packed with copper rings. The vapor from the top of the column was condensed in a reflux condenser. Part of the condensate was drawn off as distillate while the rest was returned to the top of the column as reflux in order to promote the rectification.

To prevent heat losses from the column it was enclosed in a glass cylinder with an air space between. When distilling at temperatures of about 100 °C, it was necessary to provide additional heat by means of a resistance wire wrapped around the cylinder. At higher temperatures it was even necessary to wrap the glass shell with layers of cotton c loth.

During the rectification the temperature of the vapor at the top of the column, and the volume of the distillate drawn off, from the beginning of the run up to that temperature were recorded for each change of temperature. These data were used to prepare a plot ot temperature versus volume of distillate drawn off. From this plot the volume of benzol in the sample was taken as that cut distilled over between 70-95 $^{\circ}$ C, and the volume of toluol as that cut distilled over between 95 - 125 $^{\circ}$ C.

Summarized Data and Results

&. Summarized Data

1. Calibration of wet gasmeter

a. At the rate of 5 cu. ft. per hour.

		Table	<u> </u>	
Run	Weight of water lb.	Temp. of water ^o F	Cu. ft. of air read from meter	Pressure of air "H ₂ 0
	. •	Wet g	asmeter A	
1	41.532	74	0.6730	0.08
2	42.532	72	0.6975	0.08
3	42.798	70	0.6996	0.10
		Wet g	asmeter B	
1	42,406	71	0.6890	0.20
2	42,192	77	0.6925	0.20
3	40.532	79	0.6590	0.20
	b. At the r	ate of 20	cu. ft. per hour.	
		Wet g	asmeter A	
1	42.156	74	0.6878	1.44
2	42.312	75	0.6922	1.40
3	42.312	72	0.6870	1.40
4	41.562	73	0.6830	1.40
		Wet g	asmeter B	
1	40,968	74	0.6667	1.50
2	40.500	75	0.6576	1.40
3	41.594	73	0.6776	1.40
4	41.344	72	0.6768	1.50

2. Determination of the equilibrium of benzol between the vapor

and liquid phases by measuring the increase in pressure of the vapor phase.

	Ta ble	II		
Run	1	2	3	4
Weight of stra w oil	499.4 gm.	499.4	497.9	496.9
Mols of address straw oil Table block baser	1.643	1.643	1. 64	1.632
Weight of benzol	7.4005 gm	13.5193	9.1850	18.1214
Mols of benzol	0•089 8	0.1732	0.118	0.2324
Mol ratio of benzol to straw oil	0.0547	0.105	0.072	0.1426
Increase of pressure of vapor "H ₂ 0	2.34	4.40	2.96	6.08
l'emp. of vapor C	25.9	26.0	26.0	26.0
Atmospheric pressure m.m.	754.0	754.0	757.0	757.0
Temp. of water ^o F	83	81	82	82

3. Determination of the specific gravity of straw oil

		Table	III		
Run	Apparent vol. of pipette	Weight of water gm.	Temp. of water ^o F	Weight of stripped oil gm.	Temp. of stripped oil o _F
1	25 c.c.	25.040	80	22.0335	81
2	25 c.c.	25.060	81	22.0275	81
	Weight of inlet oil gm.	Temp. of inlet oil oF	Weight of outlet of gm.	of Temp of oil outlet o ^o F	il
1	22.0884	80	22.1060	08 O	
2	22.0786	80	22.099	4 80	

4. Run I, material balance around the Northwest rich tower a super

Table IV		
	Inlet	Outlet
Vol. of gas recorded cu. ft.	138.4	145.54
Ave. temp. of gas through gasmeter ^o F	47.5	47.5
Ave. pressure of gas thru. gasmeter "H ₂ 0	0.85	0.50
Ave. temp. of gas thru. tower ^o r	20.0	
Ave. pressure of gas thru. tower "H ₂ 0	15.5	
Atmospheric pressure m.m.	756.3	756.3
Ave temp of oil ^O C	28.5	30.6
Ave. rate of oil " oil per minute	1.942	
Vol. of oil used for stripping of light oil	16,000 c.c.	10,000 c.c.

4 .	Distillatio	n of li	ght oil	from	inlet	straw oi	1
. V	folume of	Temp o	C	Volume	of	Temp.	(
L	0 c.c.	65		11ST11 110	C.C.	79.	3
	1	70		126		80.	5
	2	77		134		82	
	4	78		139		102	
	12	79		141		110	
	15	79		143		122	
	21	79.3	5	145		127	
	34	79.5)	146		131	
	39	79.5	5	148		136	
	45	79.5	;	150	•5	139	
	57	79.5	5	153	•5	144	
	76	79.5	5				
	101	79 . 5	5				
	Atmospheric	pressu	ire	750	m.m.		
	Volume of 1	enzol		137	c.c.		
	Volume of t	coluol		7	.0.0.		
		<u>1</u>	a ble	VI			

Table

V

4.

Distillation of light oil from outlet straw oil

Volume of distillate	Temp. ^o C	Volume of distillate	Temp ^o C
0 c.c.	36	5 c.c.	77.
1	44	9	77
2	49	11	79
3	52	18	79.5
4	62	25	79.8

76

٥C

		Table	V1Continu	ed
4 .	Volume of distillate	Temp °C	Volume of distillate	Temp ^O C
	30 c.c.	79.8	135 c.c.	94
	40	79.8	138	98
	45	79.9	143.5	104
	50	80	150	109
	71	80	154.5	109.5
	81	80	157	110
	97	80.5	163.5	110.5
	106	80.5	168	111
	116	81	173.5	113.5
	123	. 82	175	117
	130	84	177	120
	132	86	179	126
	134	88	180	128
	Atmospheri	c pressure.	749 m.m.	
	Volume of	Benzol	132 c.c.	
	Volume of	Toluol	42 c.c.	
		(De 1-1 -	7777	

Table VII

Distillation of light oil from ether extract of inlet rich gas

Volume of distillate	Temp ^o C	Volume of a distillate	Temp °C
0 c.c.	35.5	15 c.c.	64
2	36.5	18	76
5	36.5	22	78
8	38	25	79
11	48	32	79.4
13	55	42	79 .7

Yolume of dis- tillate c.c. Temp °C Volume of dis- tillate c.c. Temp. 67 79.7 149 99 78 79.8 151 103 85.2 79.8 153.2 106 106 79.8 155 108 112 80.2 161 109. 125 80.5 162 110 130 81 165 110 135 82 169 112 138 84 171 126 142 87 175 133 144 87 176 139 147 93 177.2 147 148 96 141 c.c. Volume of benzol 141 c.c. 23 c.c.			Table	VIIcontinued	
67 79.7 149 99 78 79.8 151 103 85.2 79.8 153.2 106 106 79.8 155 108 112 80.2 161 $109.$ 125 80.5 162 110 130 81 165 110 135 82 169 112 138 84 171 126 142 87 175 133 144 87 176 139 147 93 177.2 147 148 96 Atmospheric pressure 750.6 m.m.Volume of benzol 141 c.c.Volume of toluol 23 c.c.	9 .	Volume of dis- tillate c.c.	Temp ^o C	Volume of dis- tillate c.c.	Temp. °C
78 79.8 151 103 85.2 79.8 153.2 106 106 79.8 155 108 112 80.2 161 109. 125 80.5 162 110 130 81 165 110 130 81 165 110 135 82 169 112 138 84 171 126 142 87 175 133 144 87 176 139 147 93 177.2 147 148 96 Volume of benzol 141 c.c. Volume of toluol 23 c.c.		67	79.7	149	99
85.279.8153.210610679.815510811280.2161109.12580.5162110130811651101358216911213884171126142871751331448717613914793177.214714896141c.c.Volume of benzol141c.c.		78	79.8	151	103
10679.8 155 108 112 80.2 161 $109.$ 125 80.5 162 110 130 81 165 110 135 82 169 112 138 84 171 126 142 87 175 133 144 87 176 139 147 93 177.2 147 148 96 $141 c.c.$ Volume of benzol $141 c.c.$		85.2	79.8	153.2	106
112 80.2 161 $109.$ 125 80.5 162 110 130 81 165 110 135 82 169 112 138 84 171 126 142 87 175 133 144 87 176 139 147 93 177.2 147 148 96 $Volume of benzol141 c.c.Volume of tolucl23 c.c.$		106	79.8	155	108
125 80.5 162 110 130 81 165 110 135 82 169 112 138 84 171 126 142 87 175 133 144 87 176 139 147 93 177.2 147 148 96		112	80.2	161	109.5
130 81 165 110 135 82 169 112 138 84 171 126 142 87 175 133 144 87 176 139 147 93 177.2 147 148 96		125	80.5	162	110
135 82 169 112 138 84 171 126 142 87 175 133 144 87 176 139 147 93 177.2 147 148 96		130	81	165	110
138 84 171 126 142 87 175 133 144 87 176 139 147 93 177.2 147 148 96		135	82	169	112
142 87 175 133 144 87 176 139 147 93 177.2 147 148 96		138	84	171	126
144 87 176 139 147 93 177.2 147 148 96		142	87	175	133
147 93 177.2 147 148 96		144	87	176	139
14896Atmospheric pressure750.6 m.m.Volume of benzol141 c.c.Volume of toluol23 c.c.		147	93	177.2	147
Atmospheric pressure750.6 m.m.Volume of benzol141 c.c.Volume of toluol23 c.c.		148	96		
Volume of benzol141 c.c.Volume of toluol23 c.c.		Atmospheric pres	sure	750.6 m.m.	
Volume of toluol 23 c.c.		Volume of benzol		141 c.c.	
		Volume of toluol		23 c.c.	
Table VIII			Table	VIII	

Distillation of light oil from ether extract of outlet lean gas

Volume of dis- tillate c.c.	Temp. °C	Volume of dis- tillate c.c.	Temp. °C
ο	35	69	79.8
2	58	85	79.8
3	65	92	79.8
4	66	110	79.8
5	70	118	79.8

		Table VII	I continued			
	Volume of dis- tillate c.c.	Temp. ^o C	Volume of dis- tillate c.c.	Temp. ^o C		
	7	76	119	81		
	9	77.5	122	88		
	11	77.8	124	94		
	14	77.8	126	99		
	15	78	128	104		
	16	78.5	131	108		
	17	79	134	109		
	20	79.5	136	110		
	. 23	79.5	138	110		
	27	79.8	140	110		
	35	79.8	142	112		
	38	79.8	143	170		
	52	79.8				
	Atmospheric pres	ssure	757 m.m.			
	Volume of benzol		120 c.c.			
	Volume of toluo	L	18 c.c.			
5.	Run 2, material	balance aro	und the n orthues	t-lean-tower		

Table	\mathbf{IX}
-------	---------------

			Inlet	Outlet
Vol.	of gas re	ecorded cu. ft	126.58	137.33
Ave.	temp. of gasmeter	gas through $o_{\rm F}$	40	40
Ave.	pressure gasmeter	of gas thru. "H ₂ 0	0.4	1.5
Ave.	temp. of tower	gas through	17.5	
Ave.	pressure tower	of gas thru. " H_{20}^{20}	15.5	

	Table IX	continued	
	· · ·	Inlet	Outlet
Atmos	spheric pressure m.m.	760	760
Ave.	temp. of oil ^o C	39.7	30.3
Ave.	oil rate " oil per minute	2.088	au an an an
Vo ₫.	of oil used for light oil stripping	16,000 c.c.	10,000 c.c.
	Table X		
Dist:	illation of light oil from i	nlet straw oil	

. Volume tillat	e of dis-1. Atoles te c.c.	Temp. °C
	0	35
	2	49
	3	120
	5	130
	7.5	139
Atmospheric pressure	m.m.	756.3
Volume of benzol		nil.
Volume of toluol		nil.
	_	

Table XI

5.

5.

Distillation of light oil from outlet straw oil

Volume of dis- tillate c.c.	Temp. ^o C	Volume of dis- tillate c.c.	Temp. °C
ο	3 5	84	79.7
4	44	92	80
6	61	99	82.5
8.5	76.5	102.5	93
12.5	⁷⁷ / 79	104.5	115

		Table	XI continued	
.تى	Volume of dis- tillate c.c.	Temp. ^o C	Volume of dis- tillate c.c.	Temp. ^O C
ti na si Atta	20	79.5	107.5	132
	36.5	79 .7	108	138
	50	79.7	111	143
	70	79 •7		
	Atmospheric prea	ssure m.m.	756.3	
	Volume of benzo	1	96 c.c.	
	Volume of toluo	L	3 c.c.	
		Table	XII	
5.	Distillation of	light oil :	from ether extract	of inlet rich gas
	Volume of dis- tillate c.c.	Temp. ^o C	Volume of dis- tillate c.c.	Temp. ^o C .
	Ø	35	73	79.7
	1	45	82	79.7
	4	59	86.5	79 .7
	8	74	101.5	79.7
	14	78.5	104.5	80.5
	21.5	79	105.5	91
	34.5	79.5	106	130
	60	79,5		
	Atmospheric pre	ssure m.m.	759.7	
	Volume of benzo	1	99 c.c.	-
	Volume of toluo	1	l c.c.	

.ک	Distillation of	light oil f	rom ether extract o	f outlet lean gas
	Volume of dis- tillate c.c.	Temp. °C	Volume of dis- tillate c.c.	Temp. ^O C
	0	35	28	79.5
. •	1	59	32	79.5
	4	65	35.5	79.5
	9.5	77	50.5	79.5
	14	79	51.5	110
	18	79.3	53	120
	22	79.5	53.25	130
	Atmospheric pres	sure m.m.	754.7	
	Volume of benzol		46.5 c.c.	
	Volume of toluol		1.5 c.c.	
6.	. Run 3, material	balance ar	ound Northwest lean	tower
		Table X	IV	۰.
			Inlet	Outlet
	Vol. of gas reco	rded cu. ft	. 137.59	189.9
	Ave. temp. of ga gasmeter o	s through F	39	39.7
	Ave. pressure of gasmeter "	gas thru. H ₂ 0	0.83	0.4
	Ave. temp. of ga tower	s through C	23.3	
	Ave. pressure of tower "	ga s thru. H ₂ 0	15.5	
	Atmospheric pres	sure m.m.	754.7	754.7
	Ave. oil temp o	C	37.8	28.3
	Ave. oil rate " minute	oil per	2.085	
	Weight of oil us ping light	ed for stri oil	p-	20 lb

Table XIII

,			
Distillation og	light oil f	rom outlet straw o	oil
Volume of dis- tillate c.c.	Temp. oc	Volume of dis- tillate c.c.	Temp. oC
0	65	68	79.2
2.5	71	75	79.5
6	76.5	80	79.5
10	78.5	81	80.5
21	79	84.5	81
30	79	87	90
42.5	79	89	112
47	79.2	90	127
55	79.2	92	136
60	79.2	93.5	147
Atmospheric pressure m.m.		746	
Volume of benzol		86 c.c.	
Volume of toluo	1	2 c.c.	

XV

Table

6

Table XVI

6 Distillation of light oil from ether extract of inlet rich gas

Volume of dis- tillate c.c.	Temp. °C	Volume of dis- tillate c.c.	Temp. oc
0	35	40	79
1.5	50.5	60	79
4.5	63	75	79
10	68	82.5	79
13.5	73.5	91.5	79
19	78	98	80
25.5	79	99.5	87

Table XVI c	ontinued
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Volume of dis- tillate c.c.	Temp. oC	Volume of dis- tillate c.c.	Temp. °C
30	79	101	145
Atmospheric pre	ssure m.m.	746	
Volume of benzo	1	90 c.c.	
Volume of toluo	1	l c.c.	

Table XVII

Distillation of light oil from ether extract of outlet lean gas

Volume of dis- tillate c.c.	Temp. ^o C	Volume of dis- tillate c.c.	Temp. ^O C
0	35	45.5	79.5
1.5	59	52	80.5
10.5	69	53	84
16.5	77.5	55	129
26	79	57	140
Atmospheric pressure m.m.		756.3	
Volume of benzol		48.5 c.c.	
Volume of toluo	1	1.0 c.c.	

	-	-		•
Vapor pressure m.m.	Temperature °C	T	1/T	
10	-12.5	260.5	0.00384	
50	12.2	285.2	0.00351	
100	21.8	294.8	0.00339	
200	43	316	0.003164	
300	53	326	0.00307	
400	61	334	0.00300	
500	67•6	340.6	0.00294	
600	73.2	346.2	0.00289	
700	78	351	0.00285	
760	80.1	352.1	0.00283	
Vapor pressure of	pure toluol at di	fferent tem	peratures ()
10	5	278	0.00360	
50	36.3	309.3	0.00324	
100	51.8	324.8	0.00308	
200	69.8	342.8	0.00293	•
300	81.1	354.1	0.002824	
400	89 . 7	36 2. 7	0.002752	
500	96 •7	369.7	0.002710	
600	102.5	375.5	0.002663	
700	107.8	380.8	0.002620	
760	110.6	383.6	0.002610	

Vapor pressure of pure benzol at different temperature (\cdots)

Sample Calculations

1. Calibration of wet gasmeters

2.

Weight of water in 1b 62.3 x density of HoO at t ^O C	= /	Actual Vol. displaced
41.531 62.3 x 0.9975	=	0.6675 cu. ft.
Actual vol. displaced Vol. recorded by meter	=	Correction factor
0.6675	=	0.9930
Equilibrium of benzol between	liqu	uid and vapor phase
Mols of benzol Molsofroil de corol		Mol ratio of benzol to oil.
0.0898	=	0.0578 Mol ratio of benzol to oil

Mols of benzol			=	Mol fraction of ber	n-				
Mols	of	oil	+	mols	of	benzol	_	zol to oil	
			0	.0898			-	0 0547 mol fract. 0	f
			1	.7328			- ,	benzol to oil	Ţ

Vap. pres. of pure benzol x mol fract. in liq. phase = partial vap pres. of benzol in vap. phase 106 x 0.0547 = 5.8 m.m. Hg

Partial pres. of benzol Atmospheric pressure	Π	Mol ratio of benzol in vapor
5.8		0.00770 mol metic of
754	_	benzol in vapor.

Prat. pres. of benzol
Atm. pres. + prat. pres. of benzol = Mol lfaction of benzol
$\frac{5.8}{759.8} = 0.00763 \text{ mol fraction of benzol.}$
$\frac{\text{Inches of } H_20/0.537}{\text{Atm. pressure}} = \text{Mol ratio of benzol ex-} \\ \text{perimentally detn.}$
$\frac{2.43/0.537}{754} = 0.00602 \text{ mol ratio of} \\ \text{benzol from expt.}$
Inches of $H_00/0.537$ = Mol fract. of benzol Atm. pres. + inches of $H_20/0.537$ from expt.
$\frac{2.43/0.537}{754 + 2.43/0.537} = 0.005875 \text{ mol fract. of} benzol from expt.}$
Mol fraction from expt = Percentage Mol fraction from Rault's Law
Mol ratio from expt. = Percentage Mol ratio from Rault's Law
$\frac{0.005975}{0.007630} \times 100 = 78.4\%$
$\frac{0.00602}{0.00770} \times 100 = 78.2\%$
Specific gravity of oil

Wt. of oil x density of H_2O at t^{OC} = specific gravity Weight of water

 $\frac{22.0305 \times 0.9965}{25.043} = 0.877$

з.

4.	Mate	erial balance around the northeost lean tower
		Run II.
	a .	$126.58 \times 0.986 \times \frac{290.5}{277.5} \times \frac{754.5}{760.75} \times \frac{775.75}{760.75} \times \frac{760.75}{788.9}$
	H	127.3 cu. ft. inert gas in inlet rich gas sample corrected at tower condition saturated.
	b.	$\frac{99 \times 0.872}{78 \times 454} \times 359 \times \frac{290.5}{273} \times \frac{760}{788.9}$
	=	0.896 c u. ft. of benzol in inlet rich gas sam- ple at tower condition.
	с.	$\frac{1 \times 0.862}{92 \times 454} \times 359 \times \frac{290.5}{273} \times \frac{760}{788.9}$
	=	0.00757 cu. ft. of toluol in inlet rich gas sam- ple at tower condition.
	d.	$\frac{0.896}{127.3} = 0.00703$ Mol ratio of benzol
	e.	$\frac{0.896}{127.3 + 0.896 + 0.00757} = 0.0070 \text{ Mol fraction of benzol}$
	f.	$\frac{0.00757}{127.3} = 0.0000594 \text{ Mol ratio of toluol}$
	g•	$\frac{0.00757}{127.3 + 0.896 + 0.00757} = 0.000056 \text{ Mol fraction}$ of toluol
	h.	$137.33 \times 0.986 \times \frac{290.5}{277.5} \times \frac{756.5}{762.8} \times \frac{777.8}{762.8} \times \frac{762.8}{788.9}$
	=	138.4 cu. ft. of inertigas in outlet lean gas sam- ple corrected at tower condition saturated.
	i.	$\frac{46.5 \times 0.872}{78 \times 454} \times 359 \times \frac{290.5}{273} \times \frac{760}{788.9}$
	·	0.421 cu. ft. of benzol in outlet lean gas sam- ple at tower condition.
	j.	$\frac{1.5 \times 0.862}{92 \times 454} \times 359 \times \frac{290.5}{273} \times \frac{760}{788.9}$
	=	0.61172 cu. ft. of toluol in outlet gas sample

at tower condition.

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	k.	$\begin{array}{rcl} 0.421 \\ 138.4 \end{array} = 0.00304 \text{ Mol ratio of} \\ benzol \end{array}$
	1.	$\frac{0.421}{138.4 + 0.421 + 0.01172} = 0.00303 \text{ Mol} \text{ fraction} $ of benzol
	m.	$\frac{0.01172}{138.4} = 0.0000847 \text{ Mol ratio of toluol}$
	n.	$\frac{0.01172}{138.4 + 0.421 + 0.01172} = 0.0000845 \text{ Mol frac-tion of toluol.}$
	0.	$\frac{2.088 \times 184 \times 62.3 \times 0.877 \times 60}{12 \times 304}$
	=	346 lb mol of straw oil sprayed into the tower per hour.
.*	p.	$\frac{10,000 \times 0.88 - (96 \times 0.872 - 3 \times 0.862)}{304 \times 454}$
	=	sample 0.0831 lb mol of straw oil in outlet rich oil
	q.	$\frac{96 \times 0.872}{78 \times 454} = 0.00236 \text{ lb mol of benzol in out-}$ let rich oil smaple.
	r.	$\frac{3 \times 0.862}{92 \times 454} = 0.0000621 \text{ lb mol of toluol in}$ outlet rich oil sample.
	S •	$\frac{0.00236}{0.0631} = 0.0374 \text{ Mol ratio of benzol}$
	t.	$\frac{0.00236}{0.0631 + 0.00236 + 0.0000621}$
	. =	0.0361 Mol fraction of benzol
	u .	$\frac{0.0000621}{0.0631} = Mol ratio of toluol$
	V .	$\frac{0.0000621}{0.0631 + 0.00236 + 0.0000621}$
	=	0.0000948 Mol fraction of toluol
*:	w.	$\frac{346 \times (0.0374 - 00)}{(0.00703 - 0.00304)} = 3,250 \text{ lb mols of}$ inert gas per hour.

·.

at 30.3 °C vapor pressure of pure benzol = 122 m.m.
$\frac{122 \times 0.0361 \times 0.81}{788.9} = 0.00449 \text{ mol fraction of benzol} $ in vapor phase in equi- librium with liquid. $\frac{346 \times 0.0374 \times 788.9}{15.230 \times 0.00276 \times 760} = 0.320 \text{ Lb Mol/Hr/(cu-ft)(Atm.)}.$
Assume Rault's holds
$\frac{122 \times 0.0361}{788.9} = 0.00557$ Mol fraction of benzol in vapor phase in equilibrium with liquid phase.
$\frac{346 \times 0.0374 \times 788.9}{15,230 \times \underbrace{-0.00303 - (0.00698 - 0.00557)}_{0.00303} \times 760}_{0.00303}$
0.418 lb mol/hr.cu.ft.Atm.
ht. x Kga x P = 80.5x .32 x 188 x 1.03 = 1.54N.T.U. G 3250
$\frac{80.5}{1.87} = 52.5 \text{ ft. H.T.U.}$
$\frac{0.00703 - 6.00304}{0.00703} \times 100 = 56.8 \text{ scrubbing ef-} \\ \text{ficiency of} \\ \text{benzol.}$
0.00304 0.00939 x 100 = 32.4 percentage loss of ben- zol in the outlet gas from lean tower assuming the mol ratio of benzol in inlet gas of the rich tower holds.

D. LITERATURE CITATIONS

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E. Location of Original Data				
Subject	Buffalo Station Data Book No.	Page		
Calibration of wet gasmeter	44	4 - 7		
Determination of Benzol equili- brium curve				
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a. Plant run	44	84		
b. Distillation of light oil i rich gas sample	in 33	138		
c. Distillation of light oil i lean gas sample	.n 33	137		
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