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REMOVAL OF LIGHT ENDS FROM COKE-OVEN LIGHT OILS

by

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SUBMITTED IN PARTIAL FULFILLMENT OF THE REQUIREMENTS FOR THE DEGREE OF

BACHELOR OF SCIENCE

from the

MASSACHUSETTS INSTITUTE OF TECHNOLOGY

1944

Signature of Authors

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Department of Chemical Engineering, February 28, 1944

Signature of Professor in

Charge of Research

Signature of Head of

Department

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Signature redacted

149 Highland Avenue Buffalo, New York February 21, 1944

Professor W. K. Lewis Department of Chemical Engineering Massachusetts Institute of Technology Cambridge, Massachusetts

Dear Sir:

Submitted herewith is our thesis on the subject "Removal of Light Ends from Coke-Oven Light Oils" in partial fulfillment of the requirements of the degree of Bachelor of Science in Chemical Engineering Practice.

Respectfully yours,

Signature redacted

Gardner E. Alden, '44 X-B

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Thornton Stearns, '44 X-B

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I SUMMARY

In the recovery of benzene, toluene and xylene from the coke-oven gases, the light oil which contains these components as well as others is run through a forerunnings column which removes the low-boiling constituents such as H_2S , CS_2 , etc. These light ends are returned to the gas main and every effort is made to separate benzene from these constituents. An investigation has been made to determine conditions necessary for efficient operation of the forerunnings column. It was found that the column is operating as efficiently as possible at the present time.

Temperatures, pressures, and flow rates of the forerunnings column were measured directly. The flow meters were calibrated. Analyses of the various streams entering and leaving the tower were determined by use of a laboratory rectification column of five theoretical plates.

It was found that the feed ranged in composition from 1.2 to 1.4 per cent by volume light ends, while the overhead product (forerunnings) varied from 25 to 50 per cent light ends. The bottoms contained a negligible amount. The amount of benzene lost in the forerunnings amounted to 50 to 60 pounds per hour. It was concluded that a hold-up in the forerunnings column existed.

It is recommended that the feed be introduced into the column on a lower plate in order to reduce the amount of benzene in the forerunnings. If this is not advisable it is recommended that the forerunnings be accumulated and re-run.

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II INTRODUCTION

In the recovery of benzene, toluene and xylene from the gases evolved in the carbonization of coal, a "light oil" containing these components as well as others is obtained by absorption in and subsequent steam stripping from a light straw oil. Following this operation the low-boiling constituents (H₂S, CS₂, etc.) are removed by rectification in a forerunnings column.

The forerunnings column (see Figure No. 1) is a 36plate bubble-cap tower. There are 22 caps per plate and the plates are spaced one foot ten inches apart. The feed is introduced continuously on the twenty-second plate from the bottom at a rate of about 700 gallons per hour. The bottoms pass through a cooler and are sent to storage. The forerunnings are withdrawn at a rate of about 12 gallons per hour. Forerunnings throughout this report are defined as the mixture of light ends and benzene withdrawn from the system as overhead product. The light ends are compounds whose boiling points are below that of benzene (80 °C.). The reflux rate into the top of the tower is about 700 gallons per hour.

Under present conditions the forerunnings are returned to the coke-oven gas main and burned as fuel. Although every effort is made to eliminate benzene from these return gases, there are some indications that this condition does not always exist. The investigation was made of the forerunnings column to determine the conditions necessary for efficient operation.

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III PROCEDURE

To determine the operating conditions of the column the following data were taken and called a run:

Temperatures:

Vapor at top of column Intermediate plate (26th) Cooled bottoms Condensate Water from condenser Feed Forerunnings (overhead product) Bottoms Pressures:

Pressure in steam chest (reboiler)

Pressure at base of column

Flow Rates:

Feed

Reflux

Forerunnings

The flow rate of the feed is indicated on a chart and was found accurate within five per cent by tank measurements. The flow rate of the forerunnings were read from a rotameter which was checked by a volumetric displacement calibration. The reflux rate is measured by an orifice flow meter. For calibration, the orifice was cleaned and a mercury manometer was connected across the pressure taps. Knowing the constants of the orifice, the flow was calculated and found to becaccurate within ten per cent.

Analyses of Streams Entering and Leaving Forerunnings Column

Feed:

The feed was analysed by a batch rectification in a five-theoretical-plate column (laboratory, see Figure No. 2). It was found that a two stage rectification was necessary. Several rectifications were made of the feed. The distillates from these operations were accumulated and re-run. The temperatures were taken up to 79 °C. which was the arbitrary limit set for the removal of the light ends. The boiling point of benzene is 80 °C.

Forerunnings:

The forerunnings were analysed in a single-stage batch rectification using the laboratory column. Bottoms:

The bottoms were analysed by the same method as the forerunnings. At no time was there any fraction found which boiled below 79 °C. Therefore it was assumed that the bottoms were free of light ends.

The curves from these analyses can be seen in Figure Nos. 3-7. Using the flow rates and analyses a material balance of light ends around the forerunnings column was calculated. Overall material balances around the laboratory column were also calculated.

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<u>Calculation of the Theoretical Number of Plates in</u> the Laboratory Column

A known mixture of benzene and toluene was added to the still pot of the laboratory column (approximately 0.1 mol per cent benzene). The column was set into operation with total reflux. The temperature of the vapor at the top of the column and the temperature of the mixture in the still pot were measured. Using composition-temperature equilibrium data,⁽¹⁾ the compositions of the vapor and bottoms were determined. These terminal compositions were plotted on an x-y diagram. The operating line is a 45° line on an x-y diagram (for total reflux), and the number of theoretical plates in the column were determined by the McGabe-Thiele method (see Figure No. 8).

Viscosity Determination

The viscosity of the forerunnings was determined by a standard Ostwald viscometer for use in the calibration of the reflux flow meter. The temperatures, time required to pass the reference points and the density of the product were recorded. The density was measured with a Westphal balance. The viscosity is calculated according to the (2) formula:

$$\frac{\mu_1}{\mu_2} = \frac{P_1 t_1}{P_2 t_2}$$

where μ_2 , t_2 , and ρ_2 are the viscosity, time, and density of the standard (water).

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Size S

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X = MOL FRACTION BENZENE IN LIQUID " " VAPOR

DATA FROM PAGE 1377 , TABLE 7 PERRY'S CHEMICAL ENGINEERING HANDBOOK - 2ND EDITION

AT STEADY ST	TE - TOTAL REFLUX	MIT STATION
STILL POT CON	APOSITION, X = 0.09	BETHLEHEM STEEL CO.
VAPOR "	" , Y = 0.94	FIGURE NO REPORT NO.
		6 F18-1843
		2-19-44 7.5.

IV RESULTS

- The column is operating as efficiently as possible under the present conditions of feed compositions, feed plate location, condenser temperatures, etc.
- Fifty to sixty pounds per hour of benzene are being put into the gasemain.
- 3. The feed to the column is from 1.2 to 1.4 volume per cent light ends*.
- 4. The bottoms from the column are free from light ends*.
- 5. The forerunnings from the column are from 24 to 48 volume per cent light ends* and about two per cent non-condensables. The remainder is benzene with a small percentage of higher boiling constituents.
- 6. A material balance of light ends around the forerunnings column does not close.

*See Figure Nos. 3-7.

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V DISCUSSION OF RESULTS

Present Operating Conditions

Since the ratio of the reflux rate to the vapor rate is very nearly one, the forerunnings column can be assumed to be operating with maximum rectification. The empirical relationship which states that the superficial vapor velocity in feet per second must be less than the distance in feet between plates is satisfied for vapor rates (when vapor is condensed) up to 1240 gallons per hour.

Benzene Recovery

No economic balance has been made of the benzene plant in order to determine whether or not the recovery of the benzene in the forerunnings is feasible. The amount lost is between one to two per cent of the benzene fed to the column. It has been assumed that the benzene should be recovered.

Addition of Plates to Column

If all the benzene were to be removed from the forerunnings, an equivalent of the five theoretical plates of the laboratory column would be needed. Assuming an average plate efficiency of thirty per cent, although in actual operation the efficiency would be less, it can be seen that if the number of plates above the feed were doubled (about 15 more plates) the vapor temperature at the top of the column would be about 35 to 40 $^{\circ}$ C. Under the present operating conditions the vapor temperature is about 68 to 74 $^{\circ}$ C.

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Location of Feed Plate

The stripping section appears to be operating more effectively then the enriching section of the column, since the analyses showed that no light ends were present in the bottoms. With the data at hand it is impossible to say how many plates are necessary to strip the light ends from the bottoms, but two or three times the present amount of light ends could probably be present in the bottoms and still be negligible. Therefore, there is a good possibility that if the feed were put in on the fifteenth plate the stripping section still would reduce the concentration of light ends to a negligible amount. It is certain that the benzene losses would be cut down if the feed plate were lowered. To what extent either of these factors would take place is impossible to predict. Actual operation of the column under these conditions is the only way to determine the effect of shifting the feed plate.

Re-running Forerunnings

If no physical changes in the column can be made, i.e. changing position of feed, in order to recover benzene the forerunnings should be accumulated until a batch large enough to re-run is collected. Operating the column with a reflux ratio of approximately one, it is certain that no benzene would be withdrawn in the overhead product. However, this re-run would require additional steam and time and may not be economically feasible.

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Analysis of Feed

The analysis of the feed was carried out in runs one and three in a single stage batch rectification. There were reasons to believe that these values for the per cent light ends would be high. Therefore, the two stage rectification was attempted. It was found that about 80 per cent of the first stage distillate was benzene. Since the percentages of light ends in the feed were small, it was not reasonable to expect a five theoretical plate column to separate all the fractions completely. Moreover, from the shape of the curve it can be seen that another rectification is needed to get good fractionation.

Analysis of the Bottoms

The bottoms from the forerunnings column are free from light ends by the analyses. It must be agreed that the rectification of the bottoms in the five theoretical plate laboratory column would not be very good. As a rough approximation, it would take three or four stages of rectification to get results comparable to a complete rectification. This procedure would be of doubtful precision since it is an accumulative process, and the losses by handling are quite appreciable.

Analysis of the Forerunnings

The analysis of the forerunnings appears to be an excellent fractionation. From the general trend of the curve it can be seen that the light ends are made of two principal fractions, one boiling about 40 $^{\circ}$ C. and the other about 45 $^{\circ}$ C.

Volume and Weight Percentages

It is observed that Figures 3 through 6 are plotted as temperature versus volume per cent, and that Figure 7 is plotted as temperaure versus weight per cent. In the analysis of the feed where low percentages of light ends are encountered the difference between volume per cent and weight per cent is only one part in one hundred. The difference between volume per cent and weight per cent in the analysis of the product amounts to ten parts per hundred. Weight per cent gives the higher value. <u>Comparison of Laboratory and Forerunnings Column</u>

When analysing the feed, the initial temperature of the vapor at the top of the column when operating at total reflux is about 20 °C. below that temperature in the forerunnings column. At any instant, a batch operation is equivalent to a continuous operation if the still pot composition for batch and the composition of a given plate for continuous are the same. From this it can be seen that the initial rectification at total reflux of the batch analysis of the feed is comparable to the section of the tower above the feed plate. The laboratory column gives a better rectification than the 14 plates in the forerunnings column. Thus it can be seen that at best the average plate efficiency of the forerunnings column is about 30 per cent. Material Balances of Light Ends Around Forerunnings Column

From a material balance around the column for runs four and five, it is evident that there is an increase in light ends in the system at rates of 50 and 60 pounds per hour respectively. In run number five, the tower had been running four hours during which time no forerunnings were withdrawn.

An inspection of the system would leave two explanations for the non-closure of the material balance. One, the light ends escape from the gas vent, and two, the light ends are being accumulated in the system. An attempt was made to absorb the escaping gases in activated charcoal, but not all the gases were absorbed. The weight of absorbed gases amounted to twelve ounces in fifteen minutes or three pounds per hour. This would account for only a small fraction of the 50 or 60 pounds per hour. Therefore, the only logical explanation for the descrepancy of the material balance is a hold-up in the system. The conditions of the tower justify this explanation, since the vapor temperature was dropping slowly. In runs one through four the only explanation is the loss of light ends through the vent since the temperatures had been constant over a period of several hours. Probably the best explanation is a combination of the two effects.

If the tower were run long enough, the system would approach steady state and become saturated with light ends. If the system became saturated either the product and/or the bottoms would have to contain abnormally large amounts of light ends. No such conditions were met.

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VI CONCLUSIONS

- The tower is operating as efficiently as possible under the present conditions of feed composition, feed plate location, condenser temperatures, etc.
- The plate efficiency of the forerunnings column is about thirty per cent.
- 3. The benzene in the forerunnings which is being put into the gas main could be all or partially recovered:
 - (a) By putting the feed into the tower at a lower plate.
 - (b) By accumulating and then re-running the forerunnings.
 - (c) Installing approximately 15 more plates above the feed.
- 4. There is a hold-up of light ends in the forerunnings column.

VII RECOMMENDATIONS

- 1. The location of the feed be changed to the fifteenth plate if possible.
- 2. If changing the location of the feed does not reduce the benzene losses, it is recommended that the forerunnings be accumulated and re-run, although this will require additional steam and time and may not be economically feasible.

VIII APPENDIX

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A DETAILS OF PROCEDURE

The laboratory rectification column (Figure No. 2) consisted of a 1000 cc. still pot, a column of about threequarters of an inch in diameter and 45 inches high, a cleverly conceived section to measure the vapor temperature and provide a means for reflux, and a condenser. All of the joints were ground to fit and were designed to be leak proof. The column was packed with one-quarter inch copper rings.

The reflux section of the tower was constructed so that the vapors were led past the thermometer before going to the condenser. The condensate fell back into the reflux section and was returned to the top of the packed section through a capillary tube. The product valve was located so that the condensate could be withdrawn continuously or totally refluxed.

The packed section of the tower was enclosed in a glass tube about an inch and one-half in diameter around which was wrapped a heating element. The purpose of the jacket and heating element was to cut down the heat losses from the column. If too much heat escaped from the column all the vapor was condensed before it reached the top. If, on the other hand, the heating element was so hot that there was a heat input to the column, the packed section acted as a reboiler and the efficiency of the column was greatly reduced.

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The product was led through a product cooler into a graduate. The lead was below the surface of the liquid in the graduate to cut down losses by evaporation. The receiver was placed on scales which weighed the amount of product. The entire arrangement, scales and graduate, could be raised or lowered in order to keep only the tip of the product line below the surface to obtain accurate weight measurements.

The non-condensable gases were led from the top of the condenser into an absorption system which consisted of three bubbler flasks in series and a condenser. The flasks were filled with benzene which would absorb any gases which were not condensed.

With the system described above it was possible to close overall material balances around the column to within one per cent. Prior to the time when the product was led below the liquid in the receiver, losses due to evaporation amounted to roughly 50 cc. in 750.

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B. TABULATION OF DATA

Forerunnings column

	0.7		Run Num	ber	h	F
Te	mperatures-°C.	1	5	2	4	5
	Top of Column	70	66	66	73	71
	22 nd Plate	86	84	85	85	85
	Cooled Bottoms	56	54	57	57.5	53
	Distillate	38	33	37	32	36
	Water from Cond.	14	14	15	12	12
	Feed	4.5	4.5	5.5	5.5	5.0
	Product	27	27	30	24	24.5
	Bottoms	84.5	84.5	84.5	85	84.5
P	ressures-lbs./in ² Gage					
	Base of Column	8.75	8.0	8.45	8.1	8.75
	Steam Chest	6.4	5.6	5.9	5.6	6.3
Fl	ow Measurements-Gallon	s/hour				
	Feed	710	710	720	725	710
	Reflux*	740	740	800	690	810
	Product*	11.8	7.3	12.6	13.1	0
An	alyses-Vol.% Light End	8				
	Feed	607 oct oct	000 ao am		1.39	1.19
	Product	48.5	48.5	36.5	24.8	26.8
	Bottoms	0	0	0	0	0
* X	Corrected value Weight per cent			Feed ³ Produ	c let ^x	1.20 28.8

B. TABULATION OF DATA

Laboratory Column

		Samp	le 1	Distil	late	Resi	due	Amt.	
Run	A CONTRACTOR OF	Vol.	Wt.	Vol.	Wt.	Vol.	Wt.	Absrbd	· B.P.
1	Product*	500		242	an an an		<u> </u>	8	
l	Feed	750		55					
1	Bottoms	750	gga ann ann	0	0				
2	Product*	750		360		00 or 00			56.8
2	Feed								107 co 100
2	Bottoms	750	att en en	0	0		Cit (20)		aa aa aa
3	Product*	500	469			240	212		58.8
	Tol. added	200	173						
	Lt. Ends			176	185				
	Benzene			198	172				
	BzTol.			33	28				
3	Feed	750		79	880 min na				83.5
3	Bottoms	750		0					aa an an
4	Product* a	7 50	674.4	183	177.7	560	477.4	15.1	
	Product* b	750	671.4	185	179.0	547	465.9	14.6	
4	Feed a	750	673.8	30	25.2		642.1	-1.0	86.4
	Feed b	750	670.5	70	63:5	888 400 400	603.1	-0.4	87.4
	Feed c	750	667.4	50	47.8		614.5	0.1	86.6
	Feed e	420	375.9	38	33.0	382	340.9	-0.6	em 400 000
	Feed (redist.)	226	199.7	37	34.1	190	160.8	0.2	
4	Bottoms a	750	672.1	0	0		an 100 ma	000 1007 1000	87.8
	* Overheed	Prod	uct or	Forer	unning	3			

B. TABULATION OF DATA

Laboratory Column (cont.)

Ru	n	Samp	le	Disti:	llate	Resi	due	Amt.	
		Vol.	Wt.	Vol.	Wt.	Vol.	Wt.	Absrbd	. B.P.
4	Bottoms b	<u>cc.</u> 750	gms.	<u>cc.</u> 0	gms.	<u> </u>	gms.	_ <u>gms</u>	88.6
	Bottoms c	750		0					88.8
5	Product*	800	720.0	207	202.5	560	489.2	16.2	
5	Feed a	800	707.1	44	38.5	750	664.4	-0.7	
	Feed b	800	709.7	34	30.0	766	677.8	0.7	
	Feed c	800	708.5	37.4	32.7		675.9	-1.2	
	Feed d	800	705.8	40.5	35.4	763	669.2	-0.4	
	Feed (redist.)		131.2		34.1	80 80 80	97.2		
5	Bottoms								
	* Orrenheed	Ducie		Demomun	antoga				

* Overhead Product or Forerunnings.

Determination of Number of Theoretical Plates in Laboratory Rectification Column

> Sample into distilling flask: 42.5 cc. of benzene 457 cc. of toluene Total reflux. Temperature at top: 83°C. Temperature at bottom: 106 °C.

Viscosity Determination for the Calibration of Reflux Flowmeter

	Temperature °C.	Time secs.	Density gms./cc.
	25.8	81.0	
Water	25.5	81.4	
	25.4	81.4	
	20.0	62.0	
Product	25.5	58.5	0.885
	24.4	59.5	

Ostwald viscometer - 5 cc. sample

The density was measured with a Westphal balance.

C. SAMPLE CALCULATIONS

Calibration of Reflux flowmeter (Orifice)

Orifice diameter - 0.762 in. Inside pipe diam. - 1.610 ins.

$$W = CYA \sqrt{\frac{2g_1 r^3 \Delta H}{1 - r^3}}$$

w -- Weight rate of discharge, 1bs./sec.

C -- Coefficient of discharge (no dimensions)

Y -- Expansion factor; for liquids Y = 1 (no dimensions)

A -- Cross-sectional area of discharge opening, sq. ft.

g1 - Acceleration due to gravity, ft/sec.2

A -- Density at upstream temperature and pressure, lbs./cu.ft.

ΔH -- Orifice pressure differential, feet of fluid of upstream density

S -- Ratio of orifice diameter to inside diameter of pipe Calculation of C:

$$Re = \frac{4w}{\pi D M}$$

 μ -- Viscosity of fluid at orifice, 0.000672 x centipoises D -- Orifice diameter, feet

w -- Weight rate of flow, lbs./sec.

To determine viscosity (Ostwald method)

$$\frac{\mu_i}{\mu_2} = \frac{\rho_i t_1}{\rho_2 t_2}$$

P -- Density of forerunnings at temp.T.

t₁ - Time to flow in voscometer at temp. T., secs.

M, -- Viscosity of forerunnings at temp. T., centipoises

P, -- Density of water at temp. T.

to - Time for water to flow in voscometer, secs.

 μ_{z--} Viscosity of water at temp. T., centipoises

$$\mathcal{M}_{i} = \frac{(0.885)(58.5)}{(1.00)(81.4)} (0.883) = 0.561 \text{ centi-poises}$$

For the Reynolds number (Re) equation:

Lowest flow rate, q = 410 gals./hr.

$$w = \frac{(410)(8.345)(0.885)}{3600} = 0.840 \text{ lbs./sec.}$$

$$Re = \frac{(4)(0.840)}{(3.142)(0.762/12)(0.000672)(0.561)} = 44,700$$

From a curve of Re versus φ , C can be determined (Fig. 12, page 852, Perry.)

C=0.61 and since this is the lowest flow rate, C equals 0.61 for the entire calibration range.

Calculation of A

A:
$$\pi/4(D^2/144)$$
 = sq. ft. of discharge opening
A. = $\frac{(0.785)(0.762)^2}{144}$ = 0.00316 sq. ft.

Calculation of AH

Manometer fluid: forerunnings over mercury

$$\Delta H = \frac{(13.60 - 0.885)(h)}{(12)(0.885)}$$
; h = manometer reading in inches

△H = (1.20)h

Calculation of β

$$Q = D_2/D_1 = \frac{0.762}{1.610} = 0.473$$

In order for w to read in gallons/hour directly,

multiply orifice equation by:

$$\frac{3600}{(8.345)(0.885)} = 488$$
w' = (488)CYA $\sqrt{\frac{2g_1 R^2 \Delta H}{1 - R^4}} = gals./hr.$
= (488)(0.61)(1)(0.00316) $\sqrt{\frac{(2)(32.2)(0.885x62.4)^2(1.2)(h)}{1 - 0.473^4}}$
= (0.942) $\sqrt{248,000}\sqrt{h}$
= (0.942)(498) \sqrt{h} = 469 \sqrt{h}
h. Meter read. Calc. Flow Per cent difference

n ins.	gals./hr.	gals./hr.	Per cent dille	rence	
1.1	410	492	(82/492)100	16.1	
1.5	530	574	(44/574)100	7.7	
9.1	1240	1410	(170/1410)100	12.1	

Calibration of Rotameter Measuring Product (Forerunnings):

(sec./cc.)(3600/3785)	11	gallons/hour
(sec./cc.)(0.952)	=	19

Meter Reading gals./hr.	Time secs.	Volume ccs.	Calculated Figals./hr.	low	
10	63	567	(567/63)(0.952)		8.57
9.9	107	948	(948/107)(0.952)	H	8.43
12.5	83	948	(948/83)(0.952)	=	10.9
15.0	69	948	(948/69)(0.952)		13.1
4.95	111	567	(567/111)(0.952)		4.87

Meter Reading gals./hr.	Calculated Rate gals./hr.	Difference gals./hr.	Per Cent Difference %	
10	8.57	1.43	14	
9.9	8.43	1.53	18	
12.5	10.9	1.61	13	
15.0	13.1	1.9	13	
4.95	4.87	0.08	16	

Average difference in reading from actual: 15%

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D LOCATION OF ORIGINAL DATA

Calibration of flow meters	82	6,7,9,10
Data on forerunnings column	82	4 - 8
Viscosity determination	82	11
Liquor analysis:		
Run 1	74	112 - 114
Run 2	74	115 - 117
Run 3	74	114,115,117-119
Run 4	74	120 - 141
Run 5	74	142 - 157
Calibration of laboratory	74	119

rectification column

E LITERATURE REFERENCES

- (1) Perry, J. H., Editor, "Chemical Engineering Handbook", 2nd edition, McGraw-Hill Book Co., New York, N. Y., page 1377, (1941).
- Glasstone, S., "Text Book of Physical Chemistry",
 D. Van Nostrand Co., Inc., New York, N. Y., page 490,
 (1943).