

A STUDY OF THE PERFORMANCE OF A TEN-GALLON EXPERIMENTAL  
LUMMUS RECTIFYING STILL

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## I. INTRODUCTION.

Considerable has been written on the principles of rectification and the theories of distillation, yet there is a lack of information concerning the operation of commercial or semi-commercial types of rectifying stills. It was the object of this investigation to supply data with respect to the performance of an experimental rectifying still in the chemical engineering laboratories.

In this investigation two questions were considered; first, what quality of distillate may be expected with various mixtures of alcohol and water charged into the still?; second, what effect will varying the rate of distillation have on the quality of the distillate when the same concentration is used for each charge? It is believed that the data obtained concerning these problems will be helpful in securing the most advantageous operation of the still considered.

## II. THEORY OF THE RECTIFYING STILL.

That two miscible liquids of different boiling points may be separated when a mixture of the two is distilled, depends on the fact that the composition of the vapors arising from the liquid differs from that in the liquid from which they are evolved. Thus when a mixture of ethyl alcohol and water is distilled, the vapors contain a larger proportion of alcohol than the liquid which is being boiled. By making successive vaporizations and condensations it is possible finally, to get a vapor which becomes increasingly richer in alcohol until a constant boiling point mixture is reached. Since the vapors arising from a constant boiling point mixture have the same composition as the liquid, further separation of the two constituents by distillation is impossible.

Separation by distillation may be carried on by a series of vaporizations and condensations using a simple still, or the process may be performed

in one distillation by using a rectifying still. A rectifying still consists of the following principal parts: the kettle, the rectifying column, the dephlegmator, and the condenser. The rectifying column consists of a series of boiling plates placed one above another supplied with intakes for the reception of vapor from the plate below and with pipes conducting the overflow liquid to the plate below, and it is here that the successive condensations and vaporizations are carried on.

The operation of a rectifying still is essentially as follows; Vapors containing let us say X per cent of alcohol rise from the kettle through the empty column to the dephlegmator where they are condensed. The condensate is returned to the top plate of the column whence it flows toward the kettle, filling the boiling plates as it descends. Assuming the composition of the liquid in the kettle to remain constant, vapor containing X per cent of alcohol is now coming in contact with liquid containing the same per cent of alcohol. But such a condition cannot last because the equilibrium liquid for vapor containing X per cent of alcohol contains a smaller proportion of alcohol, and the vapor in equilibrium with a liquid containing X per cent of alcohol must contain a larger proportion of alcohol. Consequently a heat interchange takes place during which some of the water in the vapor is condensed and some of the alcohol in the liquid is vaporized. This results in a new liquid somewhat weaker in alcohol and a new vapor somewhat richer in alcohol. This process is repeated in each boiling plate of the column, and the vapors leaving the top of the column to enter the dephlegmator contain only a small proportion of water.

The dephlegmator is a partial condenser maintained at a temperature which will allow only the most volatile vapors to pass through it into the coil condenser. In the partial condensation which occurs in the dephlegmator, a larger proportion of the water is condensed than of the alcohol. The residual vapor rich in alcohol then enters the coil condenser and appears as distillate.

The condensate from the dephlegmator is returned to the column and flows toward the kettle. On the way down it loses most of its volatile constituent and has it replaced by the heavy constituent of the ascending vapors.

### III. CONSTRUCTION OF THE STILL.

The rectifying still operated in this problem was a Lummus Experimental Still. It consisted of a ten-gallon kettle, a rectifying column twelve chambers high and eight inches in diameter, a water tube dephlegmator, and a coil condenser.

### IV. OPERATION.

The liquid distilled was denatured alcohol as furnished by Store A of the University of Wisconsin; its specific gravity measured by the Westphal balance at 15 Centigrade was .819. The kettle was heated by a steam jacket. All distillations were started with the rectifying column empty, and no vapors were allowed to pass into the coil condenser until successive readings of the temperature at the top of the column showed but small variation. The water supply to the dephlegmator was then decreased, and part of the vapors were allowed to pass on to the coil condenser.

To study the relation between the specific gravity of the charge and that of the distillate, runs were made in which the concentration of the charge was increased from .982 specific gravity to .885.

In investigating the effect of the rate of distillation on the specific gravity of the distillate, denatured alcohol-water mixture of .937 specific gravity was used. Runs were made varying the rate of distillation from about six liters an hour to about thirty-five liters an hour.

### V. CONCLUSIONS.

As a basis of conclusion it was arbitrarily decided that alcohol of .82 specific gravity be considered as a desirable final product of distillation. Quality of the distillate was determined by specific gravity because

the alcohol contained a denaturant which made expression in per cent alcohol impracticable.

The curves on Plate I show that no alcohol of .82 specific gravity was obtained in the runs operating with a charge of higher specific gravity than .952. These curves also show that relatively little advantage is gained in changing the gravity of the charge from .937 to .904 as compared with the change in gravity from .952 to .937. By increasing the concentration of the alcohol from .952 specific gravity to .937, there were obtained for each one hundredth decrease in specific gravity, 2.07 liters more of .82 specific gravity distillate. By increasing the concentration of the alcohol from .937 specific gravity to .904, there were obtained for each one hundredth decrease in the specific gravity of the charge, 1.03 liters more of the .82 specific gravity alcohol.

The curves on Plate II show that varying the rate of distillation has little effect on the quantity of .82 specific gravity distillate obtained from a charge of .937 specific gravity. Six and one half liters of .82 distillate were obtained with the rate of distillation thirty-five liters an hour. Seven and three tenths liters were obtained in distilling at the rate of six liters an hour. Although the slower rate of distillation yielded an increase in product of about twelve and one half per cent, this advantage is greatly outweighed by the six hundred per cent increase in operating time. Furthermore, Run 11 made at the rate of thirty-five liters an hour produced more .82 specific gravity distillate than did Run 12 operating at the slower rate of twenty three liters an hour. The same condition holds as regards Runs 9 and 10. The abnormally low yield of Run 12 is probably due to the fact that the vapors were allowed to enter the coil condenser when the boiling point at the top of the column was  $76.5^{\circ}$  Centigrade. In other runs the temperature at this stage was about  $75^{\circ}$  Centigrade.

PLATE I

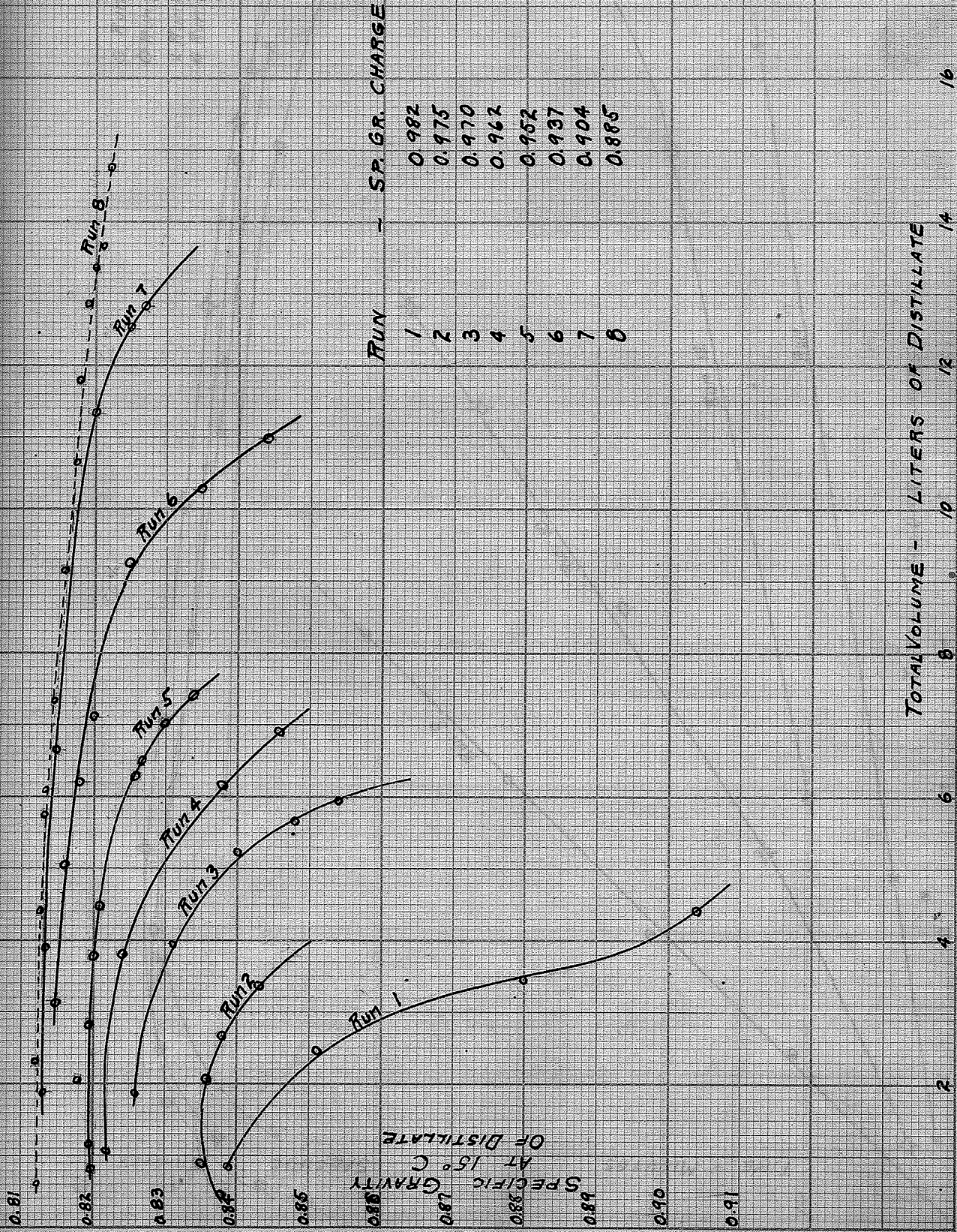
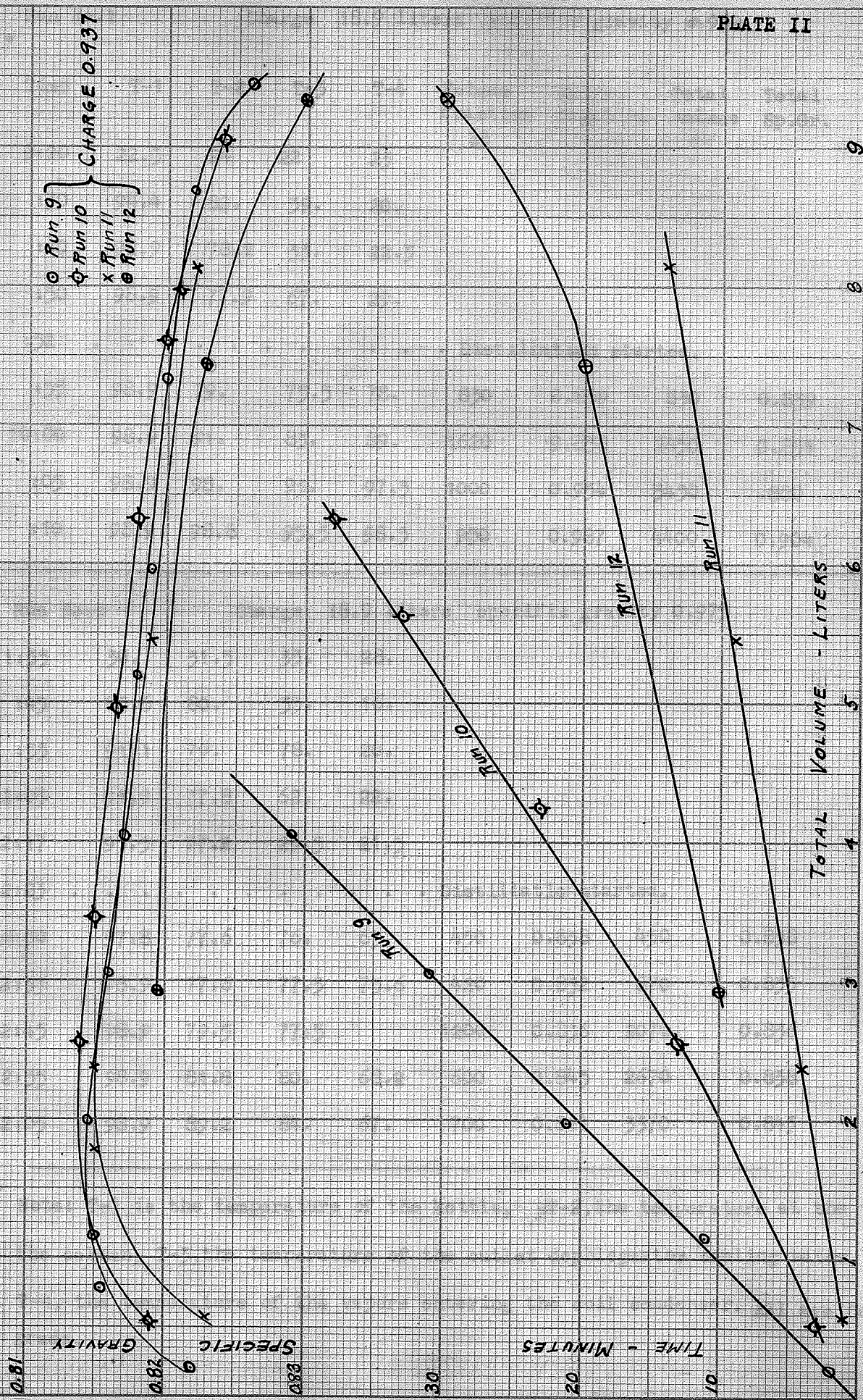


PLATE II



Run No.1 Charge 18.9 liters ,specific gravity 0.982.

Time	T-1	T-2	T-3	T-4	Volume fraction	Sp.Gr. fraction	Total volume cc	Total Sp.Gr.
9:20	22.5	24	22	23				
:30	94.4	82.	39.	20.				
:40	98.9	78.2	33.	22.3				
:50	98.9	77.9	67.	29.				
:52	. . .	. . .	. . .	. . .	. Distillation started.			
:55	98.9	79.	75.5	78.	830	0.839	830	0.839
10:00	98.9	91.	83.	89.	1620	0.858	2450	0.851
:05	98.9	98.	93.	97.5	1000	0.954	3450	.880
:10	98.9	98.8	95.5	98.5	950	0.987	4400	0.904

Run No.2 Charge 18.9 liters specific gravity 0.975

1:35	30.	31.5	33.	28.				
:45	85.6	80.	35.	16.				
:55	91.1	79.	78.	20.				
2:05	93.9	77.8	62.	22.				
2:17	95.5	77.2	29.5	21.5				
2:23	. . .	. . .	. . .	. . .	. Distillation started.			
2:30	97.8	77.6	76.	65.	450	0.838	450	0.838
2:35	98.9	77.6	77.3	75.6	420	0.832	870	0.835
2:45	98.9	79.5	77.5		1200	0.836	2070	0.836
2:55	98.9	81.8	80.	68.2	600	0.845	2670	0.838
2:05	98.9	89.2	84.	67.	700	0.861	3370	0.843

\* Note: T-1 is the temperature of the kettle, T-2, the temperature at the top of the column, T-3, the temperature of the outlet dephlegmator cooling water, T-4, the temperature of the vapors entering the coil condenser. All are Centi-grade.

Run No.3	Charge 18.9 liters				specific gravity 0.970			
Time	T-1	T-2	T-3	T-4	Volume fraction	Sp.Gr. fraction	Total volume	Total Sp.Gr.
9:55	22.5	24.	10.	19.5				
10:05	87.5	79.3	22	18.				
:10	93.3	78.3	34	19				
:20	98.9	77.8	30.5	20.				
:30	96.7	78.2	58.5	21				
:40	98.9	77.8	73.5	74.5				
							Distillation started.	
:50	98.9	78.2	76.5	77.6	1875	0.826	1875	0.826
11:00	98.9	81.	78.1	80.	2080	0.833	3955	0.831
:10	98.9	92.	88.5	91.5	1260	0.871	5215	0.840
:20	98.9	96.2	92.	95.2	425	0.935	5640	0.848
:30	98.9	97.3	93.5	96.6	300	0.959	5940	0.854

Run No.4	Charge 18.9 Liters				specific gravity 0.962			
1:15	27.	39.5	18.5	21.2				
:25	83.	80.	32.	17.5				
:35	95.6	77.8	27.	19.8				
:45	98.9	79.3	27.5	20.5				
:55	97.9	77.	26.	21.5				
2:05	98.9	76.5	25.	22.				
:15	98.9	76.3	73.	51.5				
2:20	.	.	.	.			Distillation started	
2:25	98.9	76.9	75.5	76.2	1080	0.822	1080	0.822
:35	98.9	78.3	76.5	77.8	2725	0.825	3805	0.824
:45	98.9	90.	85.	88.	2350	0.856	6155	0.838
:55	98.9	96.5	92.5	96.	750	0.927	6905	0.846

Run No.5

Charge 18.9 liters specific gravity 0.952

Time	T-1	T-2	T-3	T-4	Volume fraction	Sp.Gr. fraction	Total volume	Total Sp.Gr.
8:32	26.7	31.	19.	24.				
8:45	82.2	75.	12.	38.				
8:55	87.8	77.5	40.	26.3				
9:00	90.6	77.	49.	24.5				
9:10	93.3	76.5	21.	23.				
9:20	95.5	76.	12	21.5				
9:30	96.1	76.	38.5	21.5				
9:40	97.8	76	61.8	22.				
9:50	98.	76.	74.8	69.				
								Distillation started.
10:00	98.3	76.3	76.	75.5	800	0.820	800	0.820
10:10	98.6	76.3	76.	75.6	310	0.820	1110	0.820
10:20	98.6	76.8	76.6	76.	930	0.817	2040	0.818
10:30	98.6	77.2	77.	76.3	775	0.820	2815	0.819
10:40	98.6	77.5	77.4	77.	975	0.821	3790	0.820
10:50	98.6	78.	77.8	77.1	700	0.825	4490	0.821
11:00	98.6	83.	82.	80.5	1800	0.838	6290	0.826
11:07	.	.	.	.	180	0.856	6470	0.8265
11:10	98.6	87.2	85.5	82.				
11:20	98.6	95.	94.	88.5	550	0.880	7020	0.830
11:30	98.6	97.2	96.	95.	260	0.924	7280	0.834

Run No. 6

Charge 18.9 liters specific gravity 0.936

Time	T-1	T-2	T-3	T-4	Volume fraction	Sp.Gr. fraction	Total volume	Total Sp.Gr.
9:00	21.1	17.5	11.5	17.5				
:10	79.8	79.3	33.5	15.				
:20	82.2	78.	30.	22.5				
:30	97.2	77.	47.8	31.5				
:40	97.2	76.5	37.5	39.5				
:52	97.2	76.	36.5	40.				
10:00	97.2	76.2	64.2	52.5				
10:00	97.2	75.8	67.8	60.				
:14	.	.	.	.	.	.	.	Distillation started.
:20	97.8	76.	73.6	75.4				
:30	98.9	77.3	75.8	76.8	3125	0.815	3125	0.815
:40	98.9	77.9	76.3	77.3	1900	0.8195	5025	0.816
:50	98.9	78.2	76.8	77.2	1175	0.824	6200	0.818
11:00	98.9	79.6	76.5	78.	900	0.834	7100	0.820
:10	98.9	89.6	82.	85.	2150	0.841	9250	0.825
:20	98.9	97.9	93.5	97.	1025	0.918	10275	0.835
:30	98.9	99.	95.	98.5	700	0.982	10975	0.844

Run No.7. Charge 18.9 liters specific gravity 0.904

Time	T-1	T-2	T-3	T-4	Volume fraction	Sp.Gr. fraction	Total volume	SpGr Total
1:41	22.5	23.	13.5	19.8				
:45	79.	74.	46.5	41.				
:55	83.5	77.	35.5	48.				
2:00	86.	77.	39.5	45.5				
:10	87.5	75.8	40.	52.				
:20	91.	75.2	40.5	52.6				
:30	91.	75.	41.	52.				
:33	.	.	.	.	.	.	.	.
								Distillation started
:40	91.5	76.	73.	75.	1900	0.813	1900	0.813
:50	96.7	76.8	75.	76.	2000	0.813	3900	0.813
3:00	98.9	78.	75.8	77.4	3780	0.818	7680	0.815
:17	98.9	90.8	84.	87.	3650	0.830	11330	0.820
:20	98.9	93.8	87.5	90.	1200	0.870	12530	0.825
:30	98.9	96.3	91.	92.	300	0.924	12830	0.827

Run No.8

Charge 18.9 liters' specific gravity 0.885

Time	T-1	T-2	T-3	T-4	Volume fraction	Sp.Gr. fraction	Total volume	Total Sp.Gr.
9:05	22.5	25.5	14.4	22.				
:15	79.0	77.2	37.5	34.8				
:25	81.0	75.2	40.5	52.0				
:35	83.0	75.0	42.5	52.5				
:45	85.0	74.3	39.0	47.5				
:55	86.5	74.8	37.5	51.				
10:00	85.0	74.0	63.0	66.0	Distillation started.			
:05	86.0	74.8	68.0	73.0	600	0.812	600	0.812
:10	88.5	75.8	73.8	75.0	1700	0.812	2300	0.812
:15	91.0	76.8	75.0	76.0	2100	0.814	4400	0.813
:20	97.2	77.0	75.2	76.5	1350	0.815	5750	0.8135
:25	.	.	.	.	160	0.817	5910	0.814
:30	98.9	77.0	75.5	76.5	150	0.816	6060	0.814
:35	98.9	77.5	75.0	77.0	1300	0.818	7360	0.815
:40	98.9	77.8	76.0	77.2	1800	0.820	9160	0.816
:45	98.9	78.0	75.0	77.8	1500	0.824	10660	0.8175
:50	98.9	79.0	76.0	78.5	1250	0.830	11910	0.818
:55	98.9	81.5	76.5	80.0	970	0.841	12880	0.819
11:00	98.9	84.5	79.0	81.0	475	0.853	13355	0.820
:05	98.9	88.0	81.5	82.2	290	0.860	13645	0.821

Run No.9		Charge 18.9 liters specific gravity 0.937						
Time	T-1	T-2	T-3	T-4	Volume fraction	Sp.Gr. fraction	Total volume	Total Sp.Gr.
2:10	22.0	25.0	22.0	24.5				
2:20	79.0	78.0	44.0	26.8				
:30	88.0	78.8	30.0	27.0				
:40	88.0	76.1	52.0	38.8				
:50	94.4	75.0	51.0	40.8				
3:00	93.3	74.5	52.0	34.0				
:09	.	.	.	.	.	.	Distillation started.	
:10	93.9	74.4	70.0	73.0				
:11	.	.	.	.	205	0.822	205	0.822
:15	.	.	.	.	575	0.8135	780	0.8155
:20	94.4	75.4	73.0	74.6	360	0.813	1140	0.815
:25	.	.	.	.	500	0.8145	1640	0.8147
:30	96.7	76.0	73.5	75.2	330	0.815	1970	0.8145
:35	.	.	.	.	475	0.817	2445	0.815
:41	97.8	76.6	74.8	76.0	600	0.8175	3045	0.816
:45	.	.	.	.	500	0.819	3545	0.816
:50	98.3	77.0	75.0	76.3	500	0.820	4045	0.817
:55	.	.	.	.	520	0.820	4565	0.8175
4:00	98.9	77.3	75.5	76.8	630	0.823	5195	0.818
:05	.	.	.	.	350	0.823	5545	0.8185
:10	98.9	77.6	76.0	77.0	410	0.824	5955	0.819
:15	.	.	.	.	690	0.825	6645	0.8195
:20	98.9	78.2	76.2	77.4	680	0.829	7325	0.820
:25	.	.	.	.	500	0.832	7825	0.821
:30	98.9	81.6	78.2	79.2	430	0.8375	8255	0.8215
:35	.	.	.	.	425	0.848	8680	0.822

## Run No.9 - continued-

Time	T-1	T-2	T-3	T-4	Volume fraction	Sp.Gr. fraction	Total volume	Total Sp.Gr.
4:40	98.9	86.6	81.5	82.0	450	0.858	9130	0.825
:45	.	.	.	.	310	0.869	9440	0.826
:50	98.9	97.8	93.5	97.0	475	0.935	9915	0.830
:55	98.9	98.3	94.0	98.0	520	0.980	10435	0.837

## Run No.10

Charge 18.9 liters specific gravity 0.937

8:48	22.0	23.0	17.0	20.0				
9:00	83.0	77.0	37.0	40.0				
:10	93.3	75.8	36.0	40.0				
:20	96.7	75.1	32.5	40.0				
:30	97.2	75.0	33.0	36.3				
:36	97.8	74.8	71.5	73.0				
:37	.	.	.	.	.	.	Distillation started.	
:38	.	.	.	.	190	0.823	190	0.823
:40	.	.	.	.	330	0.817	520	0.819
:42	98.3	75.5	74.0	74.8				
:50	98.9	76.6	75.0	76.0	2025	0.813	2545	0.814
:55	.	.	.	.	900	0.8165	3445	0.815
10:00	98.9	77.3	75.8	76.8	850	0.819	4295	0.816
:05	98.9	.	.	.	650	0.820	4945	0.8165
:10	98.9	77.8	76.3	77.2	675	0.822	5620	0.817
:15	.	.	.	.	700	0.826	6320	0.818
:20	98.9	78.5	76.8	78.0	700	0.830	7020	0.819
:25	.	.	.	.	580	0.836	7600	0.820
:30	98.9	80.0	77.0	78.8	375	0.840	7975	0.821
:35	.	.	.	.	280	0.845	8255	0.822
:40	98.9	87.3	80.0	82.8	775	0.852	9030	0.824

## Run No.10 - continued-

Time	T-1	T-2	T-3	T-4	Volume fraction	Sp.Gr. fraction	Total volume	Total Sp.Gr.
10:45	.	.	.	.	425.	0.873	9455	0.827
:50	98.9	96.2	91.0	93.8	300	0.914	9755	0.830
11:00	98.9	98.2	94.0	97.0	390	0.960	10145	0.834

## Run No.11. Charge 18.9 liters specific gravity .937

1:25	23.5	33.0	18.0	30.0				
1:35	82.0	78.3	45.0	31.4				
:45	90.0	76.2	45.8	41.				
:55	96.7	75.3	37.5	39.0				
2:05	97.8	75.0	35.0	38.0				
:11	98.9	74.9	60.0	69.5				
							Distillation started.	
:12	.	.	.	.	575.	0.823	575	0.823
:14	.	.	.	.	1200	0.813	1775	0.815
:15	98.9	76.6	72.5	76.0	610	0.815	2385	0.815
:20	.	.	.	.	3070	0.820	5455	0.819
:25	98.9	83.0	77.0	81.8	2680	0.837	8135	0.822
:30	.	.	.	.	1500	0.881	9635	9.833
:35.9	98.9	98.1	93.5	97.8	825	0.965	10460	0.844
:45	98.9	98.9	95.0	98.5	1150	0.990	11610	0.858

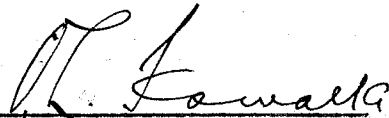
Run No. 11

Charge 18.9 liters specific gravity 0.937

Time	T-1	T-2	T-3	T-4	Volume fraction	Sp.Gr. Fraction	Total Volume	Total Sp.Gr.	
3:40	28.0	41.5	14.0	28.5					
:50	83.0	78.3	35.2	32.4					
4:00	97.8	78.6	24.0	25.6					
:10	91.5	76.5	71.0	74.3					
						Distillation started.			
:20	97.8	77.2	75.6	76.6	2925	0.8195	2925	0.8195	
:30	98.9	79.3	77.0	78.8	4509	0.8245	7434	0.823	
:40	98.9	92.2	88.5	90.0	1900	0.858	9334	90.830	
:51	98.9	97.3	92.6	96.3	725	0.944	10059	0.834	

APPROVAL

The foregoing thesis is hereby approved as a creditable study of an engineering subject, carried out and presented in a manner sufficiently satisfactory to warrant its acceptance as a prerequisite to the degree for which it has been submitted. It is to be understood that by this approval the undersigned does not necessarily endorse or approve any statement made, opinions expressed, or conclusions drawn therein, but approves the thesis only for the purpose for which it is submitted.



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Name



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Title

June 18, 1920.