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REPORT NO. KAPL-961

Chemistry

General Electric Company  
KNOLLS ATOMIC POWER LABORATORY  
Schenectady, New York

210, 11805

A MINIATURE MIXER SETTLER FOR CONTINUOUS COUNTERCURRENT  
SOLVENT EXTRACTION

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November 25, 1953

Operated for the  
United States Atomic Energy Commission  
by the  
General Electric Company  
Contract No. W-31-109 Eng-52

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ACKNOWLEDGEMENT

We gratefully acknowledge the special assistance and advice of B. V. Coplan, W. O. Haas, and E. L. Zebroski and of many others who contributed to this work.

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ABSTRACT

A unit for continuous multi-stage countercurrent solvent extraction, the Miniature Mixer Settler, is described. The over-all size of a 16 - stage unit is 3 inches X 14 inches X 8 inches. The total holdup per stage is about 8 cc and flow rates to the unit may be in the range 0.05 to 4 cc/min. The device requires little operator attention, and stage samples may be conveniently withdrawn. Bank efficiencies have been measured as a function of total flow rate to the bank using as test systems either aniline or phenol as solute distributing between 0.8 M NaClO<sub>4</sub> and iso-octane. For the 12 - stage bank tested, 11.4 theoretical stages were found at a total flow rate to the bank of 0.97 cc/min. The efficiency fell off to 71% at 3.4 cc/min and flooding occurred at 4.6 cc/min for the system tested. Efficiency is shown to have been limited by settling rather than mixing efficiency.

## A MINIATURE MIXER SETTLER FOR CONTINUOUS COUNTERCURRENT SOLVENT EXTRACTION

H. Ward Alter, James W. Coddling, and Alfred S. Jennings

### I. INTRODUCTION

The widespread use of continuous countercurrent solvent extraction owes much to the laboratory scale development of suitable flow sheets. In addition, solvent extraction is often the preferred method for the separation of reaction products from organicsyntheses, or of fine pharmaceuticals. In general, such problems have been solved in the laboratory by the use of batch countercurrent extraction techniques.<sup>1</sup> Equipment for batch countercurrent extraction varies over a wide range of complexity but in principle performs batchwise stirring, settling, and countercurrent phase transfer by some characteristic pattern. The outstanding advantages of such units are the ability to approach 100% stage efficiency and, by the taking of equilibrium stage samples, to obtain a reliable equilibrium curve for the systems under investigation. Batch countercurrent extraction may be, however, both laborious and time consuming and may require careful technique. Devices used may be fragile or may not be suitable for the handling of highly toxic or valuable substances. Dynamic flow patterns and residence times may not be applicable to large-scale continuous equipment.

The existing need for a compact, continuous countercurrent, multi-stage extraction unit to operate in the range of holdup and feed requirements of laboratory batch countercurrent equipment has not been adequately met in the past by small-scale continuous extraction devices. We propose to describe in this paper a unit which we believe fulfills this need, the Miniature Mixer Settler. The unit has been developed over a period of three years in connection with other work.

### II. DESCRIPTION

The Miniature Mixer Settler is similar in operating principle to the "Pump-Mix" Mixer Settler described by Coplan, Davidson, and Zebroski.<sup>2</sup> The contactor consists of a number of stages, each stage containing a mixing section and a settling section. Every mixing section has an impeller driven by a shaft that enters the mixing section at the top of the stage. The impeller is similar to a small centrifugal pump, liquid being drawn up through an axial tube and discharged through radial holes. Since the capacity of the pump is designed to be greater than the heavy phase flow, the mixed phase-heavy phase interface in the mixing section will fall below the impeller tip and mixed phases from the top of the mixing section will be recirculated through the impeller. The net result is a "make and break" effect between the interface and the impeller tip. In this way the level of the impeller tip in each stage uniquely determines the interface in the settling section of the previous stage. The only leveling device required is a weir to drain the heavy phase from the last stage.

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In "pump-mix" mixer settler operation, therefore, the heavy phase is pumped from stage to stage through the bank with the light phase flowing in the opposite direction by gravity. The light phase pressure drop from stage to stage is negligible and permits horizontal operation of the bank. A conventional mixer-settler countercurrent flow pattern is followed; light phase from the settling section of stage "n + 1" and heavy phase from the settling section of stage "n - 1" flow into the mixing section of stage "n". The mixed phases then flow through a port to the settling section of stage "n".

Good operation is characterized by steady interfaces in all settling sections and by "make and break" interface-impeller action in the mixing sections. Feed streams are introduced continuously at the ends of a bank and into any stage or stages in a bank, making possible extraction with reflux or center feed. Similarly, products and raffinates may be collected at both ends or removed continuously from any stage. The unit may be shut down and started up without disturbing equilibrium, and light- and heavy-phase stage samples may be taken from settling sections either during operation or upon shutdown. The entire bank may be conveniently immersed in a bath for controlled temperature operation. Operator attention during a run is ordinarily limited to the taking of end-stream or stage samples.

The over-all size of a 16 - stage unit with impellers is 3 inches X 14 inches X 8 inches. The total holdup per stage is about 8 cc. Flow rates to the unit are in the range of 0.05 to 4 cc/min. Depending on the system, impellers are operated at 1,500 to 3,000 rpm. As will be shown in the next section, bank efficiency is in excess of 85%.

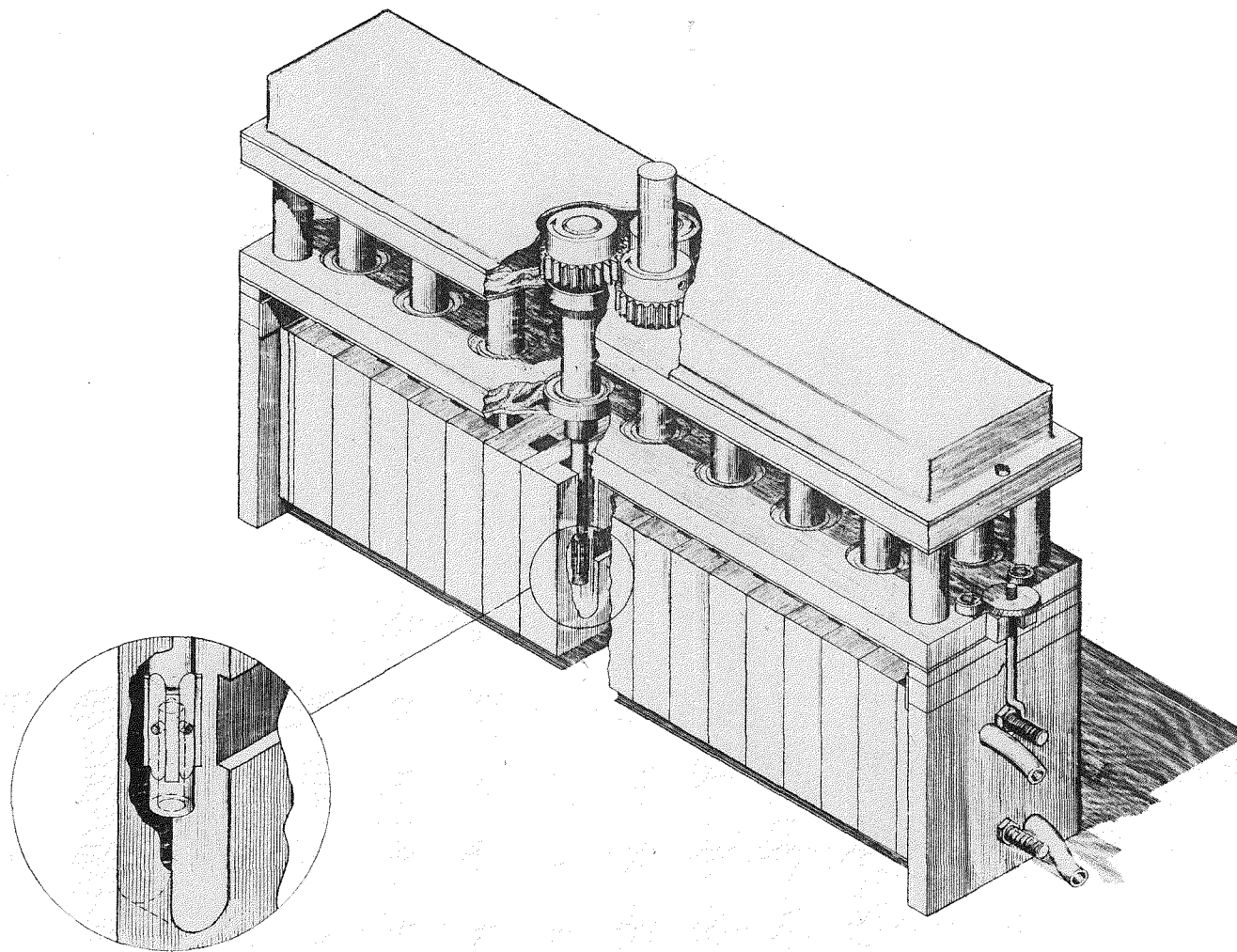
It was desired that the material of construction of the Miniature Mixer Settler be resistant to most solvents and aqueous solutions and be transparent. In the past, Fluoroethane has been used for this purpose but present units have been built from a plastic, "Homalite",\* which satisfactorily meets these specifications.

An isometric view of a complete unit is shown in Figure T-7A8017. Each stage in the unit, consisting of a 3/8-in. X 3/8-in. X 2 1/2-in. mixing section and a 3/8-in. X 1/4-in. X 2 1/2-in. settling section with associated ports, is milled from a 3/4-in. X 2-in. X 3-in. sheet of Homalite. Two types of stages are made, one being the mirror image of the other. These are placed together alternately with the front, milled face, of one stage touching the back face of its mirror image. (See Figure P-6A8772.) The ends are secured by metal end plates appropriately drilled. Two longitudinal rods, threaded at each end, go through all the stages and the end plates. Nuts on the ends provide the pressure necessary to hold the unit together and prevent leakage. Stage surfaces must be flat and free of tool marks.

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\*Homalite Corporation, Wilmington, Delaware.

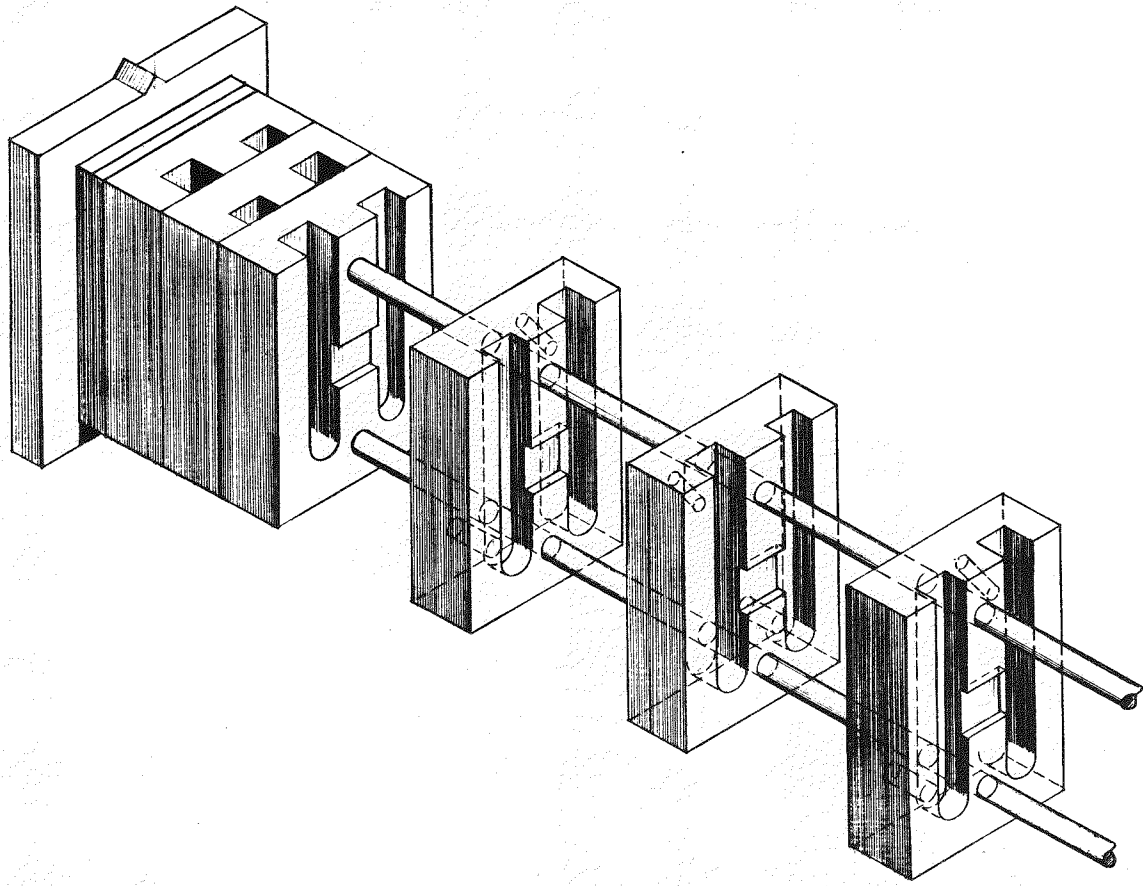
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ISOMETRIC VIEW OF 16-STAGE MINIATURE MIXER SETTLER  
SHOWING IMPELLER DETAIL

T-7A8017

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ISOMETRIC VIEW OF STAGE CONSTRUCTION AND ASSEMBLY

P-6A8772

In this way, units may be built up of as many stages as desired. We have not made greater than 20 - stage units but there is little doubt that longer units could be operated. If a great many stages are desirable multi-unit assemblies may be preferable.

The end plates also serve as supports for the impeller head. The impellers must be centrally located with respect to the mixing chambers and should be without appreciable wobble or whip. Each impeller shaft is supported by two bearings and is driven by a gear train at the top of the impeller head. The gear train is designed so that each impeller rotates in the same direction with respect to the port connecting mixing and settling sections. A General Electric "Thymotrol" drive has been found convenient for impeller speed control.

The accurate flow control demanded in many runs in the range of 0.05 to 4 cc/min was made possible by a feed system composed of 50-cc syringes driven by a rack and pinion arrangement. The pinion was turned at desired speeds by a suitably geared-down synchronous motor. A wide variety of flows and flow ratios could be obtained by varying pinion speed or size and by manifolding several syringes on one feed stream. Syringes were refilled manually at regular intervals. Flow ratios with the syringe drive were routinely good to 0.5%.

III. EXPERIMENTAL

In order to measure the efficiency of a solvent extraction unit, it is desirable to employ a liquid-liquid system which conforms as nearly as possible to the following ideal conditions:

1. The solute obeys the Nernst Distribution Law.
2. The liquid phases are immiscible.
3. Volume changes due to the mass transfer of solute are negligible.

The third condition implies the use of dilute solutions, which in turn requires that a sensitive analytical method must be available for the solute. In addition, it is convenient if the system exhibits a distribution coefficient close to one, a reasonable density difference between the phases, and a relatively low viscosity for each of the liquids used. Such a system is, of course, somewhat arbitrary and the efficiency so measured reflects the performance of the unit for that system only.

A system exhibiting these properties was found to be that of water-aniline-iso-octane. Aniline as the solute distributed between the aqueous and organic phases in a manner closely approaching ideality, with a distribution coefficient sufficiently close to one to allow a flow ratio (organic/aqueous) near unity. Iso-octane and water are not appreciably miscible and, furthermore, ultraviolet spectrophotometry provided a sensitive and accurate analytical method for the solute. The density difference between the two phases was 0.38 g/cc.

In order to investigate the efficiency of the unit at a flow ratio considerably different from one, phenol was substituted for aniline in an otherwise identical system. Here the distribution coefficient was near 0.1, and a flow ratio of about seven was employed. It was found that the addition of an inert organic-insoluble salt enhanced the settling properties of the system. Sodium perchlorate was chosen because its solutions have a high transparency in the ultraviolet region.

### Materials

Aniline (bp 183 to 186°C) and phenol were used as received from the Baker Chemical Company. The  $\text{NaClO}_4$  was recrystallized and prepared in a 4.0 M stock solution with de-ionized water of pH 6 to 7, which was used for all aqueous solutions. Care was taken that neither acidic nor basic contamination entered the system, since either would affect both the distribution coefficients and the analytical procedure. The iso-octane was of spectro-grade purity, from the Eastman Kodak Company.

### Analytical

The analyses were performed on a Cary Recording Spectrophotometer, Model 11MS, and a Beckman Quartz Spectrophotometer, Model DU. Known solutions of each solute were prepared and the spectra studied. The analytically important peaks and the corresponding extinction coefficients are listed in Table 1.

Table 1. Analytical Extinction Coefficients for Aniline and Phenol

<u>Solute</u>	<u>Solvent</u>	$\lambda(\text{\AA})$	<u>Molar Extinction Coefficient</u>
Aniline	iso-octane	2875	1830
	water	2800	1370
Phenol	iso-octane	2710	2060
	water	2700	1520

Both solutes obeyed Beer's Law within the range of analytical importance ( $10^{-4}$  to  $10^{-3}$  M). Furthermore, the presence of up to 1.0 M  $\text{NaClO}_4$  had a negligible effect on the spectrum of each solute, whereas somewhat less than this amount was found sufficient to promote rapid settling of the phases.

### Distribution Data

The theoretical equilibrium line for each solute was determined by batch distributions performed at room temperature by exhaustive equilibration of a volume of iso-octane to an aqueous phase of known solute concentration and 0.8 M  $\text{NaClO}_4$ . The results for aniline and phenol are shown in Tables 2 and 3 where  $C_o$  and  $C_a$  refer to the concentration of the solute in the organic and aqueous

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phases respectively, and  $E^{\circ}/a$  is the distribution coefficient ( $C_o/C_a$ ). For aniline an exhaustive batch extraction was also performed in order to determine whether "inextractable species" were present. The presence of only 0.4 M  $\text{NaClO}_4$  in the exhaustive extraction resulted in a somewhat lower, but still nearly constant, distribution coefficient. These extraction data are listed in Table 4.

The time required to reach equilibrium in a single contacting was found to be less than 15 seconds. Since this was considerably less than the holdup time per stage in the contacting unit for the flow rates used, rate effects were considered unimportant for this work. Because of the volatility of iso-octane, provision had to be made for inhibiting the evaporation of the solvent from the bank. A blotter, perforated for the impellers and placed across the top of the bank, was found satisfactory for this purpose. In addition, the organic end stream was collected in a container whose air vent was partially plugged with glass wool. As a result of these precautions, evaporation of the iso-octane was held to a tolerable minimum.

Table 2. Distribution of Aniline between Iso-octane and 0.8 M  $\text{NaClO}_4$

$C_o$ (M)	$C_a$ (M)	$E^{\circ}/a$
0.102	0.132	0.773
0.0685	0.088	0.778
0.0342	0.044	0.777
0.0205	0.0264	0.776
0.00684	0.0088	0.777
0.00337	0.0044	0.776

Average = 0.775

Table 3. Distribution of Phenol between Iso-octane and 0.8 M  $\text{NaClO}_4$

$C_o$ (M)	$C_a$ (M)	$E^{\circ}/a$
0.0171	0.132	0.129
0.0113	0.088	0.128
0.00568	0.044	0.129
0.00347	0.0264	0.131
0.00120	0.0088	0.136

Average = 0.130

Table 4. Distribution of Aniline between Iso-octane and 0.4 M NaClO<sub>4</sub> by Exhaustive Extraction

<u>Number of Extractions</u>	<u>C<sub>o</sub> (M)</u>	<u>C<sub>a</sub> (M)</u>	<u>E %/a</u>
1	0.0612	0.0808	0.757
2	0.0343	0.0449	0.764
3	0.0189	0.0253	0.747
4	0.0104	0.0141	0.738
5	0.00565	0.00763	0.740
6	0.00226	0.00308	0.734
7	0.000555	0.000757	0.733

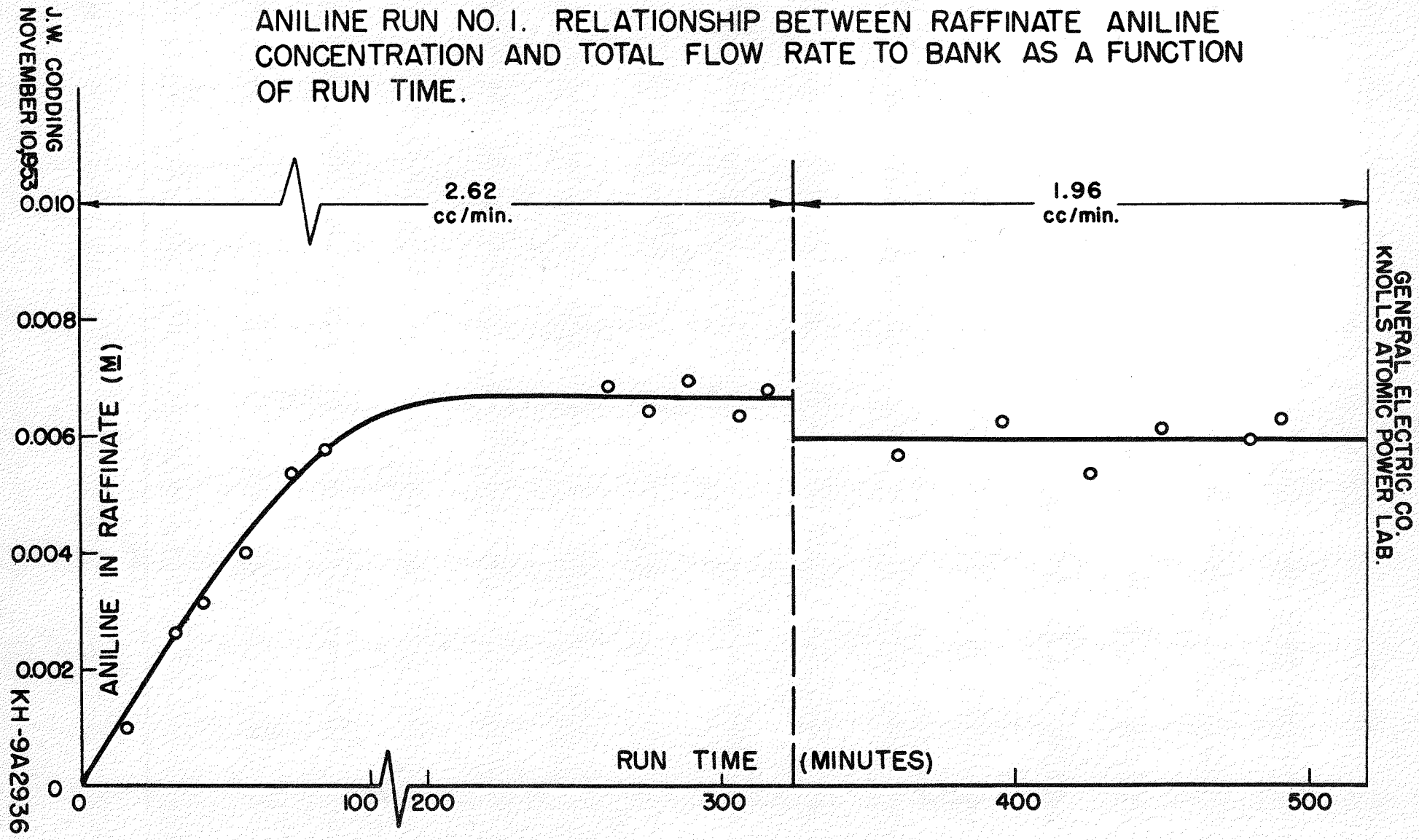
The extraction method used was to introduce a solute bearing aqueous stream at one end of a 12 - stage bank, and to extract this solute into the organic solvent entering at the opposite end. Since, for this system, the equilibrium and operating lines were linear, and the feed and extract concentrations and the flow ratios were known, the raffinate solute concentration (the "loss") became a direct measure of the efficiency of the unit. For a further study of bank operation, stage samples were withdrawn when equilibrium had been reached, as denoted by constant end-stream concentrations. Continuous volume measurements of the raffinate and extract streams provided a check on both the flow ratio and the total flow to the bank. The impellers were operated between 2,500 and 2,800 rpm for all runs.

#### Performance Data

In the first aniline run, the bank was operated at a total flow rate of 2.62 cc/min, with a flow ratio (organic/aqueous) of 1.40, until a constant aniline concentration in the raffinate (as shown in Figure KH-9A2936) indicated that dynamic equilibrium had been reached. This average "loss" value was then used to determine bank efficiency for this flow rate. A similar operation was carried out at a lower flow rate. (See Figure KH-9A2936.) When equilibrium had been established for the final throughput rate (1.96 cc/min total to the bank), the feed and solvent streams were turned off, the impellers stopped, and the impeller head removed. Organic and aqueous stage samples were withdrawn from the settling sections and analyzed for aniline. Figure KH-9A2937 presents the stage data in a conventional McCabe-Thiel plot, with average values of raffinate and extract concentrations also shown, as well as the theoretical equilibrium line from batch distribution measurements, and the operating line as calculated from material balance at the end of the run.

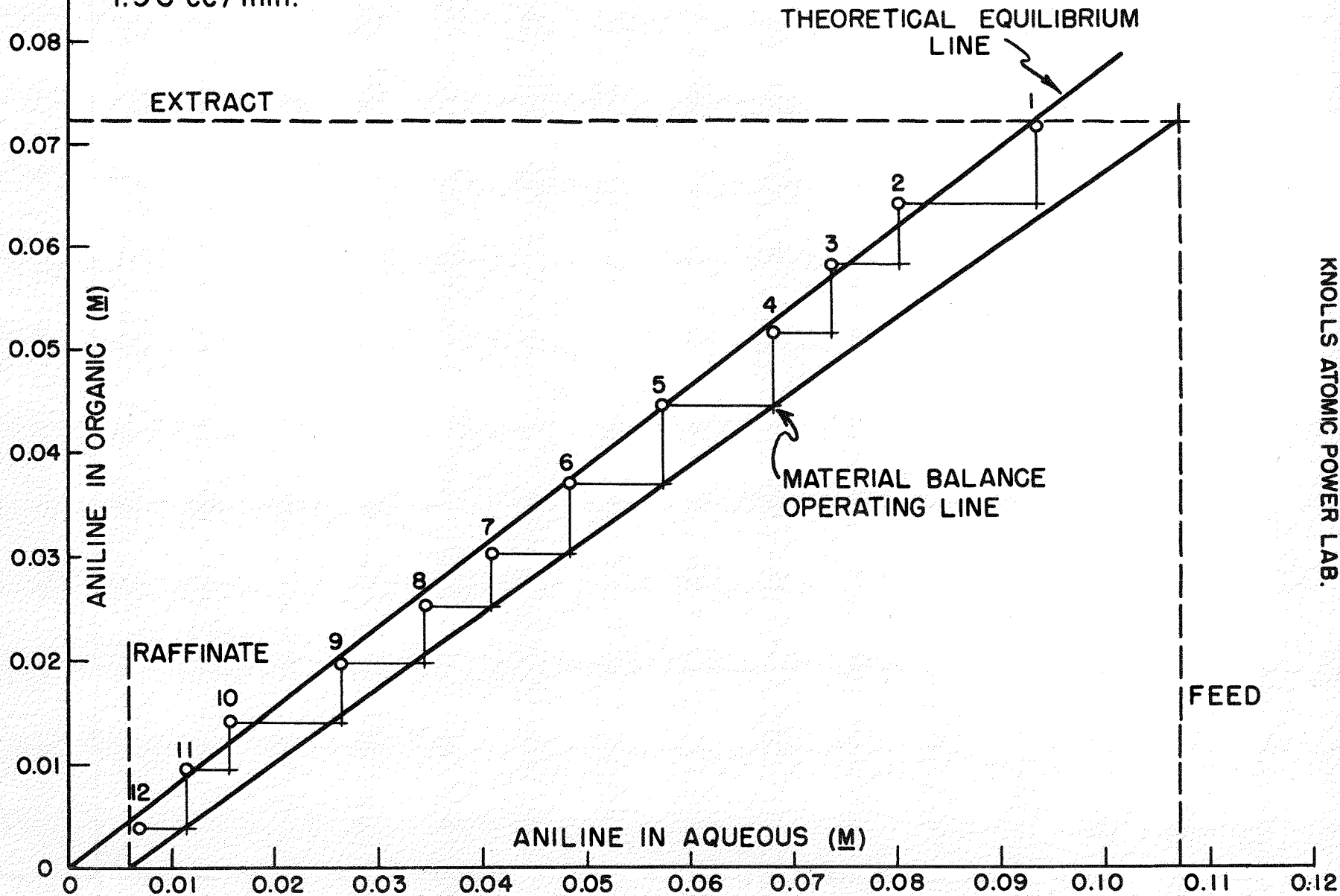
Figures KH-9A2938 and KH-9A2939 present similar data for the phenol run. A flow ratio of 6.85 was used, with the total flow set at 1.96 cc/min in order to compare bank operation between the two systems under similar throughput conditions.

ANILINE RUN NO. 1. RELATIONSHIP BETWEEN RAFFINATE ANILINE CONCENTRATION AND TOTAL FLOW RATE TO BANK AS A FUNCTION OF RUN TIME.



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ANILINE RUN NO. I. McCABE-THIEL PLOT FROM INDIVIDUAL STAGE SAMPLES.  
ORGANIC: AQUEOUS FLOW RATIO: 1.40. TOTAL FLOW RATE TO BANK  
1.96 cc/min.



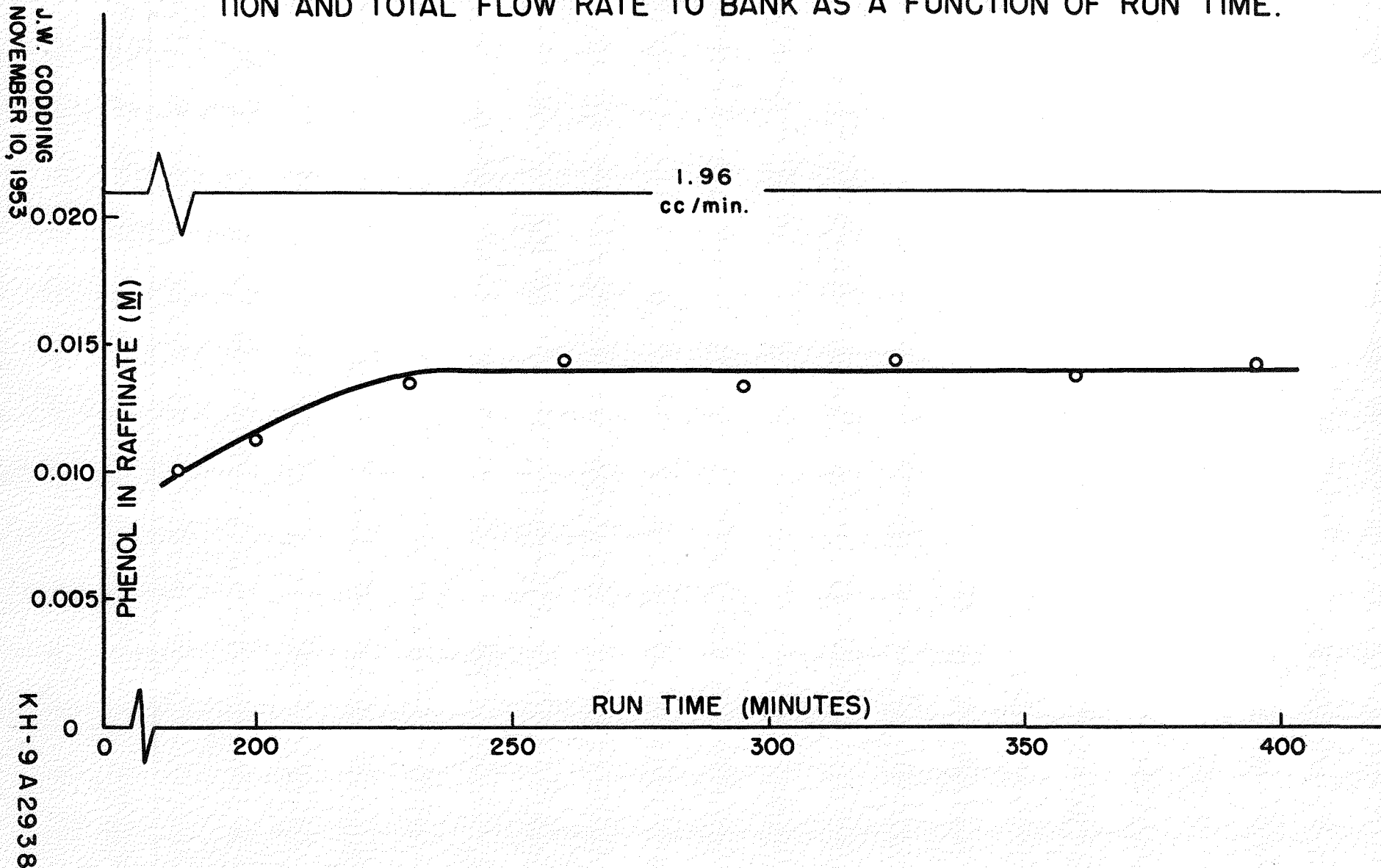
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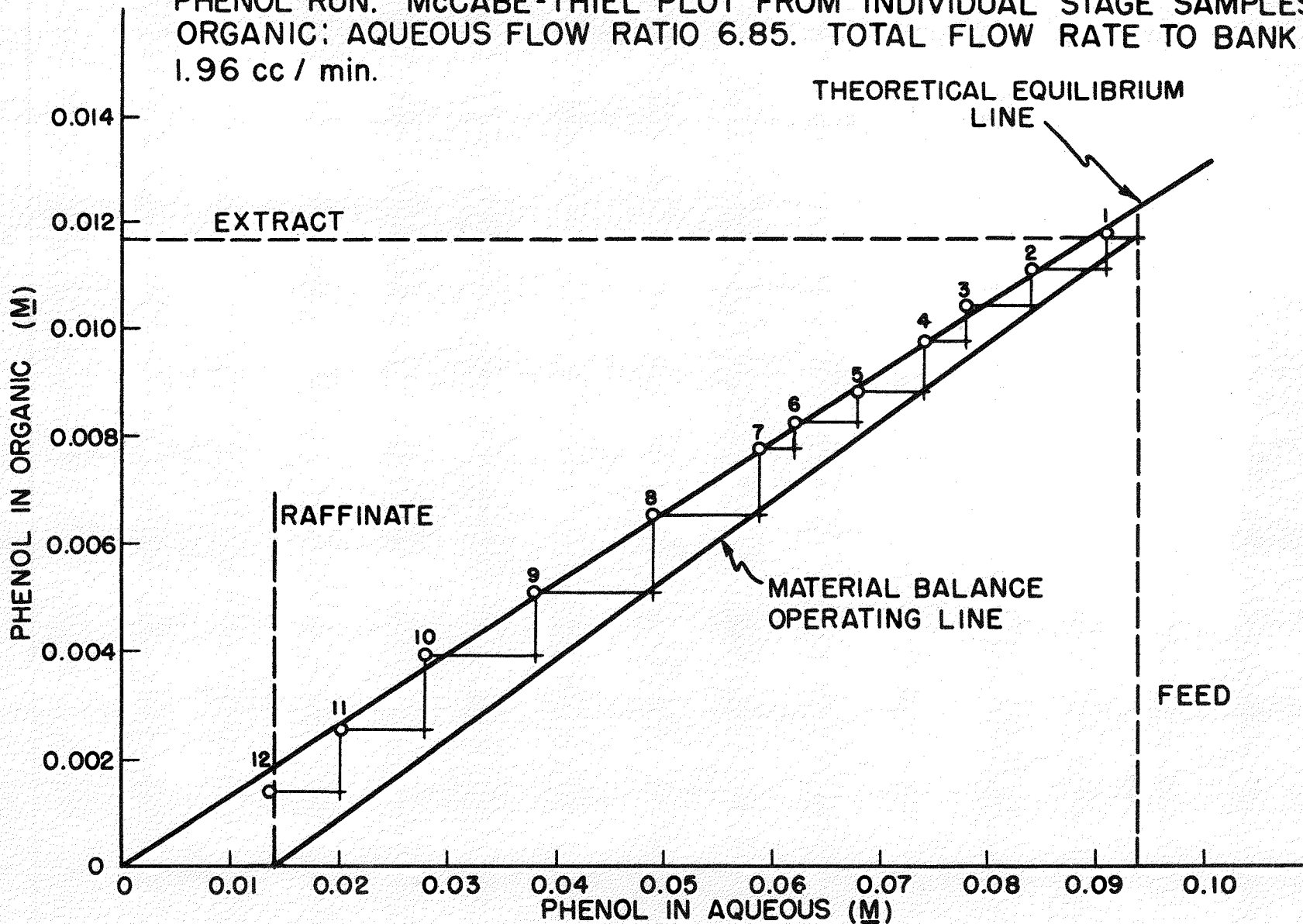
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PHENOL RUN. RELATIONSHIP BETWEEN RAFFINATE PHENOL CONCENTRATION AND TOTAL FLOW RATE TO BANK AS A FUNCTION OF RUN TIME.



PHENOL RUN. MCGABE-THIEL PLOT FROM INDIVIDUAL STAGE SAMPLES.  
ORGANIC: AQUEOUS FLOW RATIO 6.85. TOTAL FLOW RATE TO BANK  
1.96 cc / min.



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KH-9A2939

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A second aniline run was carried out to test the bank efficiency over a relatively wide range of total flow rates, up to the flow rate at which flooding occurred. Figure KH-9A2940 shows the variation in raffinate "loss" as the throughput rate was increased, with an average raffinate concentration indicated for each total flow rate.

#### IV. CALCULATIONS AND RESULTS

In the evaluation of performance of a continuous countercurrent multi-stage extractor, the number of equilibrium stages obtained in any system must be determined. For a mixer settler the bank efficiency is then the ratio of the number of equilibrium stages obtained to the number of actual stages in the bank. Barson and Beyer<sup>3</sup> have given an expression for solvent extraction calculations in special cases where pure solvent is used. The extraction expression they derive for the number of theoretical stages,  $N$ , is:

$$N = \frac{\log \left( \frac{1 - (x_1 - x_2)(a-m)}{mx_2} \right)}{\log \left( \frac{m}{a} \right)}$$

where  $a$  = slope of operating line =  $\frac{y_1}{x_1 - x_2}$

$m$  = slope of equilibrium line

$x_1$  = solute concentration in feed

$x_2$  = solute concentration in raffinate

$y_1$  = solute concentration in extract

Based on this equation, each set of equilibrium values of raffinate and extract concentrations was used to calculate the percent efficiency of the 12 - stage bank corresponding to that particular set of conditions. These results are summarized in Table 5, and presented in graphical form in Figure KH-9A2941. The efficiency is seen to be a function of total flow rate to the bank and is independent of flow ratio or solute within these limits.

Table 5. Run Results for the 12 - Stage Miniature Mixer Settler

Solute	Total Flow Rate to Bank (cc/m)	Flow Ratio (o/a)	Solute Concentration (M)			Theo- retical Stages	Per Cent Efficiency
			Feed	Extract	Raffinate		
Aniline	0.97	1.55	0.1025	0.0646	0.00235	11.4	95
Aniline	1.96	1.40	0.107	0.0723	0.00594	10.5	88
Phenol	1.96	6.85	0.0937	0.0117	0.0140	10.5	88
Aniline	2.20	1.55	0.1025	0.0642	0.00296	10.3	86
Aniline	2.62	1.40	0.107	0.0718	0.00667	9.6	80
Aniline	3.40	1.63	0.1025	0.0609	0.00329	8.5	71

The McCabe-Thiel diagrams (Figures KH-9A2937 and KH-9A2939) indicate that equilibrium was generally established in each stage of the bank for both runs shown, since the stage data fall on the theoretical equilibrium line in each case. Losses in bank efficiency were apparently due to poor settling and resultant back-mixing, as indicated by the position of some of the stage operating points above the operating line. In the second aniline run, where the total flow rate was increased to flooding, the bank became hydraulically inoperable at a flow rate of 4.60 cc/min, as evidenced by a lack of flow of the organic phase and resultant overflow of the stages involved. This stoppage of flow was due to blocking of the light phase ports by small aqueous droplets resulting from inadequate settling.

Such visual evidence of back mixing would tend to confirm the belief, based on the stage data and operating points, that the loss in theoretical stages was not due to inefficient mixing, but to poor settling at the higher throughput rates. Relatively simple stage modifications such as increasing the size of the settling section or the size of the light phase port should permit high efficiency operation at much higher flow rates if this is desirable.

The Miniature Mixer Settler has shown high efficiency and operability with systems other than those reported on in detail here. At moderate flow rates and with systems having reasonable settling properties, consistent performance of the unit at 85 to 95% efficiency may be expected.

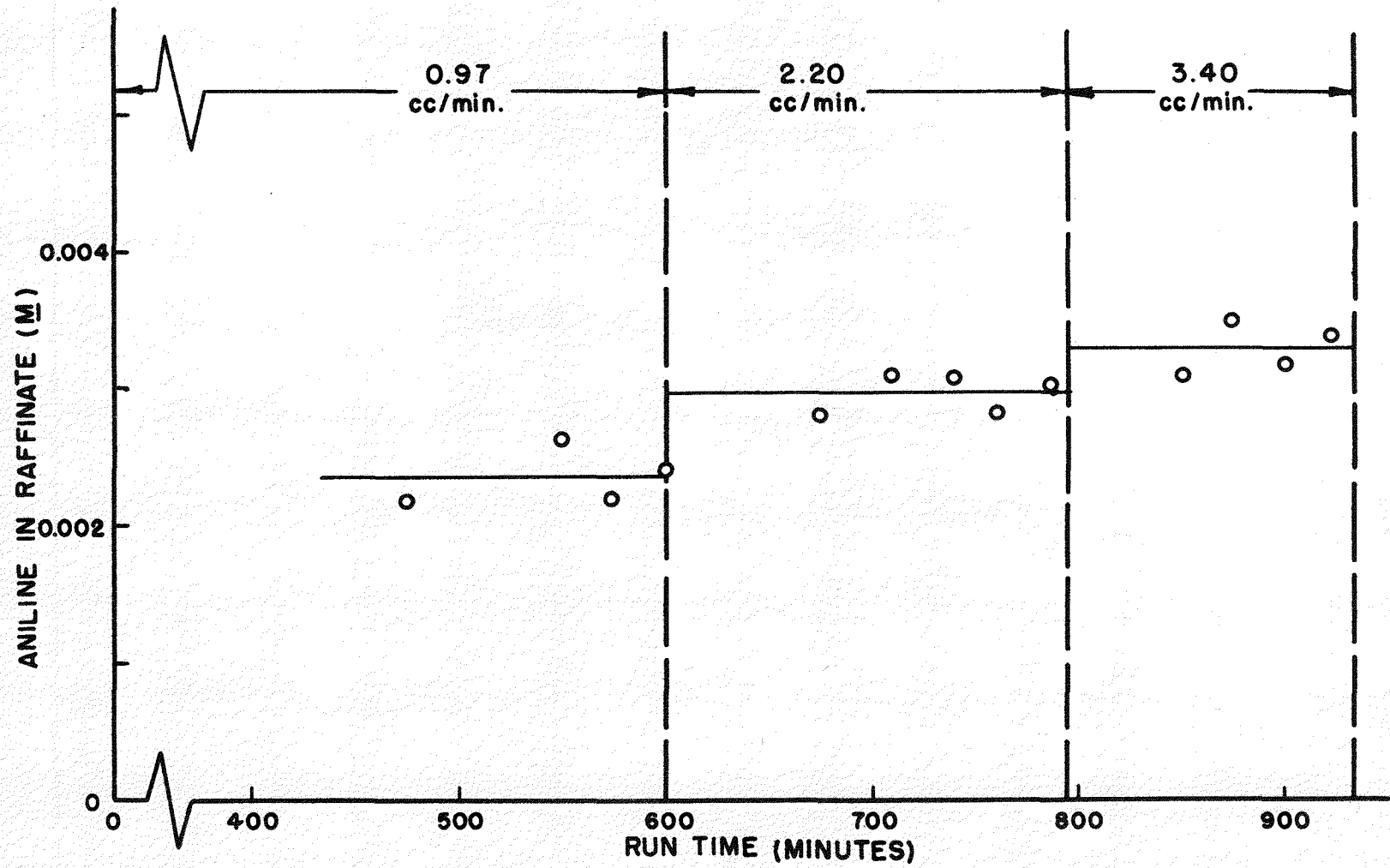
For those interested, complete production blueprints of the unit are available from this laboratory.

### ANILINE RUN NO. 2. RELATIONSHIP BETWEEN RAFFINATE ANILINE CONCENTRATION AND TOTAL FLOW RATE TO BANK AS A FUNCTION OF RUN TIME.

J. W. CODDING

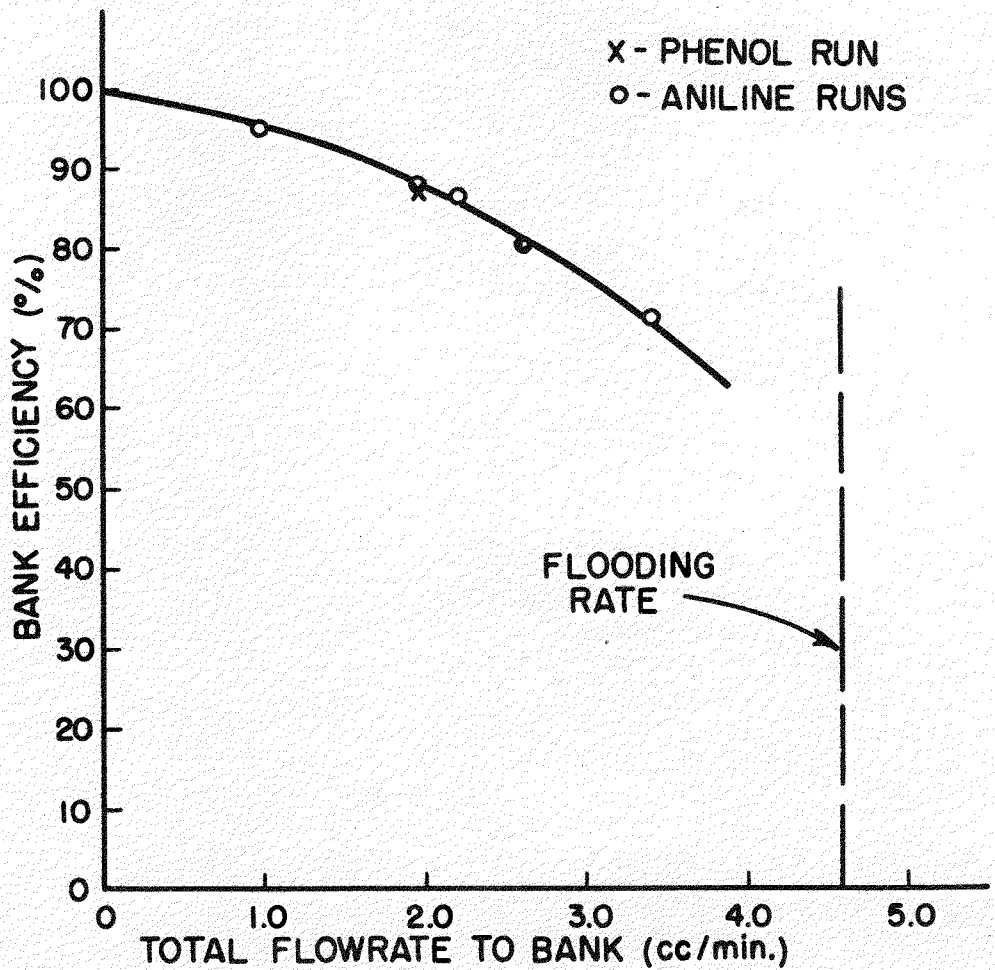
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PLOT OF BANK EFFICIENCY vs. TOTAL FLOW RATE TO BANK FOR ALL RUNS.



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NOVEMBER 10, 1953

KH-9A2941

GENERAL ELECTRIC CO.  
KNOLLS ATOMIC POWER LAB.

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