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DEVELOPMENT OF A TEN-STAGE MIXER-SETTLER FOR  
U235 SOLUTIONS

PART 2

by

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Abstract

Experimental work on a ten-stage mixer-settler is described. This includes the effects of impeller position and flow rate on its operation and the inactive extraction efficiency under conditions, such as are expected in the reprocessing of HIFAR fuel elements. Equilibrium data were determined for the systems used and a method for interface detection over a wide range of solution concentrations developed.

The mixer-settlers designed have been shown to be hydrodynamically practicable.

It was found that interface height is controlled satisfactorily by correct positioning of the impeller above the mixer base and that the pump mix type of impeller recommended elsewhere is not essential.



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## 1. INTRODUCTION

In order to gain experience of the behaviour of mixer-settlers as applied to nuclear fuel reprocessing in general, a ten-stage mixer-settler has been developed and operated on the basis of the fuel element throughput for HIFAR at full power.

In Part I, (Baillie and Cairns, 1958) the development necessary for the design of a multistage mixer-settler unit was reported. Optimum values of the design variables were determined and a review of the criticality problem was presented.

This report deals with the experimental work which was carried out using the ten-stage mixer-settler designed on the basis of the previous work.

The hydrodynamic properties of the mixer-settler unit have been investigated and a method for interface control has been found. Actual extraction runs under flowsheet conditions are reported and the mixer-settler efficiency determined. Probes for detecting the interface have also been developed.

## 2. PRELIMINARY TESTS ON THE TEN-STAGE MIXER-SETTLER

A critically safe ten-stage mixer-settler of the dimensions given by Baillie and Cairns (1958) was set up for operation. The organic phase was five per cent T.B.P. in kerosene and the aqueous phase water, slightly acid with nitric acid. The organic feed was to one end of the ten stage unit, and the aqueous feed to the other.

Initially, paddle type impellers were used, run at approximately 300 r.p.m., and  $\frac{1}{2}$  in. above the mixer base, but it was found that pumping action was stronger than had been anticipated. This caused the interface level to fall rapidly at normal flow rates, and it was necessary to raise the impellers to a position  $\frac{3}{4}$  in. above the base for the initial tests.

### 2.1 Effect of Flow Rate

With the impeller speed maintained between 280 r.p.m. and 300 r.p.m. and flowrates at the normal operating values for the extraction section (52.5 ml/min. for the aqueous phase, and 16.6 ml/min. for the organic phase) the value of the interface height in each stage when hydraulic equilibrium had been reached was measured. It was found that stable operation was reached after about two hours. Tests were then carried out to determine the effect of aqueous flow rate on the interface height. This was varied from 25 ml/min. to 85 ml/min. Interface height was found to increase slightly as the flow rate increased. The interface height of each stage could be adjusted individually, so long as the interface in the settler following the mixer in which the adjustment was made was below the mixed phase port. The average rise in interface height was less than 0.4 in. for each stage over the whole range of flows, or 0.1 in. for every 15 ml/min. increase in flow rate. It is expected that this will provide a reasonably stable system for the small fluctuations in flow rate.

### 2.2 Effect of Impeller Speed

To determine the effect of impeller speed on pumping action the speed was increased to 350 r.p.m. but no effect on interface level could be detected. Lowering the speed to 250 r.p.m. had no detectable effect on interface height either. Impeller speeds greater than 350 r.p.m. caused spillage and speeds less than 250 r.p.m. were not high enough to achieve good mixing.

### 2.3 Effect of Impeller Height

The height above the mixer base at which the impeller was set appeared to be the critical factor. Lowering the impeller  $\frac{1}{8}$  in. in the mixer, from  $\frac{3}{4}$  in. to  $\frac{5}{8}$  in. above the base, caused the interface of the preceding settler to fall about 0.4 in. at normal flow rates. The maximum flow before flooding was greatly increased. Lowering the impellers a further  $\frac{1}{8}$  in. to a position  $\frac{1}{2}$  in. above the base caused

pumping to become so effective that at normal flow rates, it was impossible to maintain an interface. A flow rate of 150 ml/min. (about three times the normal rate) was necessary to prevent the interface from falling out of the operable range.

## 2.4 Discussion

These investigations showed that the paddle type impellers were capable of exerting quite a considerable pumping action. Interface control over a reasonable range of flow rates was easily obtained by varying the depth to which the impeller was lowered. During preliminary tests on a single-stage mixer-settler it had been found that the best pumping action was obtained with the aqueous inlet port situated directly below the axis of the impeller. No pumping was obtained when the inlet port was at the outer wall of the mixer. This was confirmed by Williams et al. (1958), who used paddle type impellers in a mixer-settler unit with aqueous phase entry at the outer wall.

In their method for calculating interface heights, Williams et al. have assumed that the ratio of solvent to aqueous phase in the mixer is the same as the ratio of flow rates. In this work, the ratio of solvent to aqueous flow rates was 1:3.

It was found in our work that the ratio of solvent to aqueous phase in the mixing compartment was dependent on the size of the mixed phase port. By decreasing the size of port, any desired ratio of phases down to 0.3 could be obtained. This was achieved without any detectable change in the pressure drop through the system, being entirely due to recirculation of the solvent phase through the mixed phase port. Indications are that it is necessary to have a relatively large pressure drop through the mixed phase port to prevent recirculation.

In the mixer-settler used by Williams, the interface levels in the stages may increase as the distance from the overflow weir increases, due to density differences and the small unavoidable pressure drops through the transfer ports. No individual control is possible and adjustments can only be made by changing the height of the overflow weir.

It is considered that the mixer-settler developed is most suitable for application to nuclear fuel reprocessing, as it is possible to operate over a range of flow rates, to control interface heights individually at any desired level, and to select the solvent to aqueous ratio for the mixing compartment to give the best settling characteristics. Mixing characteristics, as determined by the impeller speed can be adjusted without affecting the interface height, which is controlled only by the height of the impeller above the mixer base.

## 3. FEED PREPARATION

### 3.1 Method of Preparation

In order to make up a feed solution for the extraction cycle, which was similar to that expected from the dissolution of HIFAR fuel elements, viz., 2.5M in aluminium, - 1.2N nitrate deficient and containing 3.57 g/l of uranium, it was necessary to prepare an acid deficient solution of aluminium nitrate.

Some aluminium sheet was obtained, the specification for which was close to that given in B.S.S. 1470 - 1.B. for reactor grade aluminium.

This specification is:-

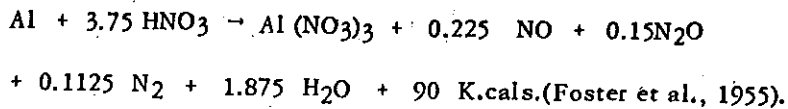
Al	:	99.5%
Si	:	0.15 - 0.30 %
Fe	:	0.20 - 0.35%
Cu	:	< 0.02%

The actual percentages of these elements in the sheet obtained were:-

Al	:	99.5%
Si	:	0.158%
Fe	:	0.371%
Cu	:	< 0.02%

Enough of this aluminium was dissolved in a calculated amount of nitric acid to give a solution of the required concentration and acid deficiency.

The equation for dissolution was assumed to be:-



Allowance was made for a 20% loss of nitric acid by boil off, and sufficiently less nitric acid was added to ensure that approximately the right acid deficiency was obtained.

A catalyst of 0.01 M mercuric nitrate was used to accelerate the reaction, which was carried out in a large stainless steel drum.

### 3.2 Discussion

Considerable difficulty was experienced in dissolving the silicon in the aluminium, but it was found that this could be done providing the solution was sufficiently nitrate deficient and refluxing was carried out for six to seven hours.

The solution obtained from the dissolution was not of the concentrations aimed at, and a considerable percentage of undissolved silica remained behind in the drum. Nevertheless, it was decided to use the nitrate deficient aluminium nitrate solution, which had been made up from the sheet for the initial extraction runs, bearing in mind that the behaviour of this solution may differ from that prepared from the actual fuel element aluminium. The solution was filtered, clarified, and the required amount of uranyl nitrate added to give a solution containing 3.57 g/l of uranium as expected from fuel element dissolution.

## 4. EQUILIBRIUM DATA

### 4.1 General

A literature search has been carried out to obtain information about the equilibrium curves for the systems to be used in the extraction and backwashing runs. A considerable amount of information is available on T.B.P. - diluent systems in equilibrium with aqueous solutions, but unfortunately very little of this information applies to systems of the type which will be encountered in the reprocessing of HIFAR fuel.

### 4.2 Relevant Equilibrium Data

Most of the figures which are available apply to 30% by volume solutions of T.B.P. in kerosene. This is the dilution used in the Purex process.

There is a small amount of data available for 5% T.B.P. in kerosene. Siddall, Parker and Prout (1957) give some figures for various aqueous systems in equilibrium with 5% T.B.P. The bulk of these data apply to a uranyl nitrate - nitric acid aqueous phase, such as in the backwashing section but some figures are given for systems in which aluminium nitrate is present.

Equilibrium diagrams for the extraction and backwashing sections of the American 25-T.B.P. process are given by Flanary et al. (1957). The fuel element treated is an enriched uranium-aluminium alloy encased in aluminium, similar to the HIFAR fuel element, but in this process 6% T.B.P. is used and the feed solution is acid, not acid deficient.

Further data concerning aqueous uranyl nitrate and uranyl nitrate - nitric acid systems in equilibrium with 4.5% T.B.P. as used in the 25-T.B.P. process are given by Andelin et al. (1956).

Codding (1958) has given a correlation from which, through the application of activity coefficients, the extraction coefficients for tracer uranyl nitrate between various solutions of basic aluminium nitrate and 5% T.B.P. can be determined. This correlation can be used to find the extraction coefficient for solutions of enriched uranium-aluminium alloy fuel elements in basic aluminium nitrate solutions, since the uranium constitutes only a small percentage of the ionic strength. Buck et al. (1958) have given equilibrium data for a system such as that proposed for reprocessing HIFAR fuel elements. These data are reproduced in Figure 4.

It was considered that, since the data available in the literature were so sparse, it would be desirable to determine the equilibrium curves for the systems used at the prevailing temperatures as the work was in progress. This was done by taking a double set of samples at the end of each active run and equilibrating one set from each stage of the mixer-settler. Points on the equilibrium curve were thus obtained in the range of uranium concentrations under which extraction was being carried out. The temperatures in the mixer-settler during the experimental runs were in the range 23°C to 24°C.

## 5. EXTRACTION AND BACKWASHING RUNS

### 5.1 Run No. 1 - Extraction

#### 5.1.1 Experimental Procedure

The feed solution, which was used in this run, did not correspond exactly in concentration to that with which it was proposed to check the extraction cycle. The aim was to determine the mixer-settler efficiency.

The aluminium concentration was 44.5 g/l (1.66M) compared with the projected 67.5 g/l, and it was present as aluminium nitrate of molarity 1.66M. 3.67 g/l of uranium in the form of uranyl nitrate was added, and this solution was fed into the fifth stage of the mixer-settler. The strip solution used in this run was 1.0M in aluminium nitrate, and was fed into the first stage. The composition of the entering streams is given in Table 2.

Initially, flow rates were set at 52.5 ml/min. for the aqueous phase and 16.6 ml/min. for the organic phase but as a result of interruption to the steady operation the flow rates were readjusted to a lower value three hours after start up. (40 ml/min. for the aqueous phase, and 14.0 ml/min. for the organic, the strip flow rate remaining at 6.75 ml/min.). This was done to prolong the run and to ensure that the operating line would fall below the equilibrium curve.

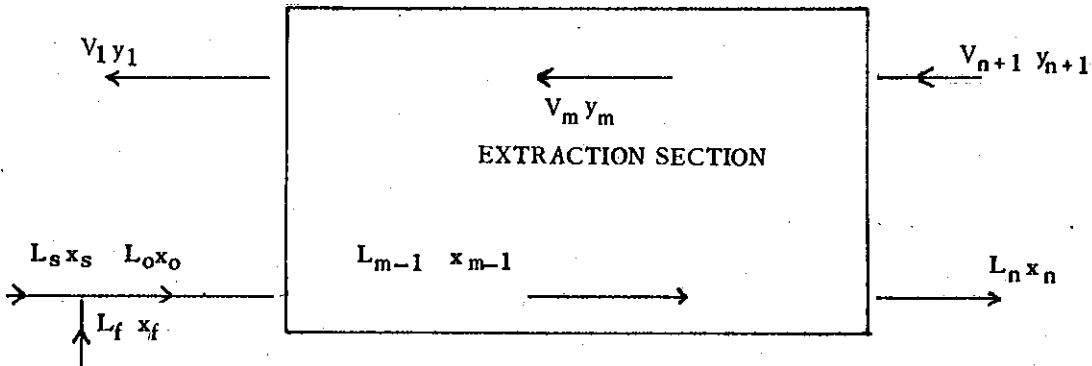
No further difficulties were encountered, and samples were taken at 5½, 7, 8½ and 10 hours from startup, an additional sample being taken at the end of the run for equilibration to establish the equilibrium curve for the system. Figure 5 shows the mixer-settler in operation during this run.

#### 5.1.2 Results

The uranium concentration in the samples taken from each of the ten mixer-settler units at the respective times from startup, and for the equilibrium samples, are given in Table 1.

#### 5.1.3 Calculation of Operating Line and Efficiencies

Firstly, the operating line for the extraction section was calculated as determined from the measured flow rates and concentrations.



- $V$  = flow rate of light phase (ml/min.)
- $L$  = flow rate of heavy phase (ml/min.)
- $y$  = concentration of uranium in light phase (g/l)
- $x$  = concentration of uranium in heavy phase (g/l)

From experimental work:

- $V_1 = 14.0$  ml/min.       $y_1 = 10.9$  g/l
- $L_s = 6.75$  ml/min.       $x_s = 0.925$  g/l
- $L_f = 40.0$  ml/min.       $x_f = 3.67$  g/l
- $L_o = 46.75$  ml/min.       $x_o = 3.28$  g/l
- $V_{n+1} = 14.0$  ml/min.       $y_{n+1} = 0$
- $L_n = 46.75$  ml/min.       $x_n = 0.0019$  g/l

The operating line has been plotted by taking the values of  $x_n$  and  $y_{n+1}$  as given above, and calculating values of  $y_m$  for various values  $x_{m-1}$ , assuming that the volume of each phase is exchanged.

The operating line is given by:

$$L_{m-1} x_{m-1} + V_{n+1} y_{n+1} = V_m y_m + L_n x_n$$

Since  $V_1 = V_2 = V_3$  etc.

and  $L_1 = L_2 = L_3$  etc.

$$V y_m = L(x_{m-1} - x_n)$$

The values of  $y_m$  for various values of  $x_{m-1}$  in the range of operation are listed in Table 3, and are plotted on Figure 1, forming the operating line. The equilibrium line, taken from the data on Table 1 is also plotted on Figure 1.

It can be seen that just less than five stages are theoretically required to obtain the extraction which was achieved. In practice, six stages were required, which indicated an overall extraction efficiency of approximately 80%.

Individual stage efficiencies have also been calculated on the basis of the Murphree efficiency. The fact that the experimental operating line did not fall exactly on the operating line based on the overall flow rates, complicated these calculations somewhat. The reason for this deviation was most likely that small changes in interface height, resulting in temporary local changes in flow rate, were occurring within the mixer-settler bank. Backmixing, or short-circuiting of one phase, although not observed, would have a similar effect.

Referring to Figure 2a, the Murphree extract stage efficiency is defined as the ratio of the number of moles of solute actually transferred to the number which would be transferred if the final extract were in equilibrium with the actual final raffinate, i.e.,

$$E_{m.e.} = \frac{y_1 - y_2}{y_1^* - y_2} \text{ etc.}$$

Similarly, the Murphree raffinate efficiency is given by:

$$E_{m.r.} = \frac{x_1 - x_0}{x_1^* - x_0}$$

Unfortunately, as stated above, the actual operating line did not correspond with the anticipated operating line, resulting in a further decrease in efficiency. To allow for this, two further efficiencies, based on the extract and raffinate streams, have been calculated.

These efficiencies were a modified Murphree efficiency, allowance being made for the deviation from the true operating line.

Referring to Figure 2b, the modified extract efficiency is given by:

$$E_{ce} = \frac{y_1 - y_2}{y_1^* - y_{1c}}$$

Similarly, the modified raffinate efficiency is given by:

$$E_{cr} = \frac{x_1 - x_0}{x_1^* - x_{0c}}$$

The four efficiencies mentioned above have been calculated for each stage and appear in Table 4. The efficiencies obtained are somewhat lower than anticipated but it is apparent that stable operation had not been reached when the run was terminated and that higher efficiencies would have been achieved had the run been prolonged. A further extraction run was therefore carried out.

## 5.2 Run No. 2 - Backwashing

The results of the backwashing run were most inconsistent. The mixer-settler bank was designed for much greater aqueous flow rates than are experienced in the backwashing section, and proved to be unsuitable for use in this cycle. At the very low flow rate of the aqueous backwash solution, it would take at least ten hours to displace all the aqueous phase in the system, and since it is usually necessary to have three or four throughputs before the final sample is taken, it is certain that equilibrium was not reached. Further work will have to be carried out before suitable extraction equipment can be recommended for this section.

## 5.3 Run No. 3 - Extraction

### 5.3.1 Experimental Procedure

This run was carried out over a total of nine hours during which time no interruptions to operation occurred. Samples were taken at four, seven, and nine hours after startup, as usual, a double sample being taken at the end of the run so that one set could be equilibrated to establish the equilibrium curve. The composition of the various entering streams is given in Table 5. The combination of the feed and strip streams resulted in a solution 2.18 M in aluminium, 4.65 M in nitrate ion and containing 3.43 g/l of uranium. The nitrate deficiency was -1.9 N. Flow rates and exit stream compositions are listed below. Flowmeter readings were checked by measuring the total inlet and outlet volumes in graduated cylinders.

### 5.3.2 Results

The uranium concentration in the samples taken from each of the ten mixer-settler units at the respective times from startup, and for the equilibrium samples, are given in Table 6.

### 5.3.3 Calculation of Operating Line and Efficiencies

Using the same symbols as for Run No. 1.

From experimental work:

$$V_1 = 15.3 \text{ ml/min. } y_1 = 13.4 \text{ g/l}$$

$$L_s = 6.75 \text{ ml/min. } x_s = 1.66 \text{ g/l}$$

$$L_f = 52.5 \text{ ml/min. } x_f = 3.66 \text{ g/l}$$

$$L_o = 59.25 \text{ ml/min. } x_o = 3.43 \text{ g/l}$$

$$V_{n+1} = 15.3 \text{ ml/min. } y_{n+1} = 0$$

$$L_n = 59.25 \text{ ml/min. } x_n = 0.00013 \text{ g/l}$$

The operating line is again fixed by the equation  $V_m y_m = L_n (x_{m-1} - x_n)$ .

Values of  $y_m$  for various values of  $x_{m-1}$  are listed in Table 7 and plotted on Figure 3, forming the operating line. The equilibrium line, from data in Table 6, is also plotted on Figure 3. In this run, only four actual stages were required to reduce the uranium value to the desired level. Steps have only been plotted down to the fourth extraction stage, which represents the achievement of the 99.99% recovery aimed at. Overall efficiency, that is the theoretical number of stages required divided by the actual number in this run, was approximately 98%.

Stage efficiencies based on the Murphree efficiency and modified Murphree efficiency, as described for Run 1, have also been calculated and are listed in Table 8.

#### 5.4 Discussion

The extraction efficiency for the ten-stage mixer-settler has been found to be in excess of 95% when operation is continued for at least nine hours. This is so for flow rates of the order for which the mixer-settler unit was designed. Some difficulty was experienced in expressing individual stage efficiencies as the points representing streams leaving the various stages did not always fall on the theoretical operating line. Wood and Williams (1958) have given a method for predicting uranium and nitric acid distribution in an extraction process assuming 95% efficiency, and that the operating and equilibrium lines are known. This method is of little use for interpreting results obtained in operation, but indicates the difficulty in estimating stage efficiencies. The Murphree efficiency gives the actual stage efficiency based on overall stream flow rates, but does not make allowance for the fact that these flow rates may deviate from the theoretical operating line. In an attempt to include such deviations in the individual stage efficiency, the modified stage efficiency, as previously described, has been developed. Efficiencies based on the raffinate stream are probably of greater importance, since the aim is to minimise uranium losses rather than to concentrate the uranium.

The Murphree raffinate efficiency gives the best indication of the mixing efficiency. In run No. 1 the average stage efficiency on this basis was 91% and in run No. 3, was 96.5%. The corrected raffinate efficiency, which allows for system fluctuations gives an average stage efficiency very close to the overall efficiency. In run No. 1,  $E_{c,r.}$  (av.) is 77%, compared with the overall efficiency of approximately 80%, and in run No. 3,  $E_{c,r.}$  (av.) is 100% compared with the overall efficiency of 98%. This indicates that the corrected raffinate efficiency gives the closest individual stage efficiency. Efficiency was poor at very low flow rates, such as in the backwashing and scrubbing sections. It is probable that backmixing and recirculation effects were greater than those due to new feed in these sections. This is to be expected, since the mixer-settler unit was designed on the basis of flow rates in the extraction section.

It is of interest to note that the theoretical operating line, when plotted on log-log paper, does not appear as a straight line, but tends asymptotically towards the concentration of uranium in the aqueous raffinate.

Some difficulty was experienced with uranium analysis at low values in the aqueous phase. The aluminium nitrate solution used in the feed makeup gave variable blank values, depending on its history, which rendered analyses below about 0.0002 g/l unreliable, and consequently analysis figures in this range have been rejected.

### 6. INTERFACE PROBES

The basis of the method used to detect the interface is the fact that the aqueous layer conducts electricity while the organic layer does not.

#### 6.1 Mark 1 Probe

In the first type of probe tested, a low voltage was applied through a series resistance across two probes, which extended to the base of the vessel in which the interface height was to be measured. Alternating current was used as a safeguard against sparking and to prevent polarization. The organic liquid used in the tests was a 5% solution of T.B.P. in kerosene, and the aqueous liquid was a solution of aluminium nitrate. The current flowing through the ammeter was found to be dependent only on the interface height over quite a range of concentration, (from 0.5M to 2.5M) and the ammeter could thus be calibrated in terms of interface height. The system was found to be quite reliable when applied to the extraction stages in the mixer-settlers, and also in the stripping section, where the concentration of electrolyte was quite large and did not vary greatly. Figure 6 is a photograph of the mixer-settler bank with the Mark 1 probes installed.

A set of ten such probes was next incorporated in the mixer-settler bank and tested during the back-washing cycle. In this case, it was found that there was a variation in current flow as the uranium concentration varied, due to the fact that the total concentration of electrolyte was low, and the change in concentration relatively great. The backwashing solution was only 0.01M in nitric acid, while the uranium concentration varied from 0.1M to practically zero. A more suitable method of interface detection was therefore sought.

### 6.2 Mark 2 Probe

This probe differed from the first type tested, in that it was not a continuously indicating probe. The range of possible heights for the interface was divided into a number of regions. The regions were:

- (A) A region of inoperability due to the interface being too low.
- (B) A region in which the interface was lower than desirable, but the mixer-settler was still operable.
- (C) A region of satisfactory operation.
- (D) A region in which the interface was higher than desirable, but the mixer-settler was still operable.
- (E) A region of inoperability, due to the interface being too high.

The probe was compounded of four separate contacts situated at the dividing values of the regions described above, each connected in parallel with the other through a resistance of 1000 ohms. A low A.C. voltage was applied across this probe and a second probe immersed in the aqueous phase. Over a range of electrolyte concentrations from about 2.5M to below 0.01M, it was found that the ammeter reading in the various regions was virtually unchanged. Calibration to show in which region the interface was situated was easily achieved. If necessary, the interface range could be divided into a greater number of regions.

Although this type of probe does not give such a complete picture as the Mark 1 type, it is certainly much more reliable. It is felt that in operation it would give a satisfactory method of interface control especially since the changes in interface height, which may occur, would be very slow. It would be possible to use the Mark 1 type probe in the stripping and extraction sections of the first cycle as the concentration of electrolyte is always high in these cycles.

## 7. CONCLUSIONS

A mixer-settler suitable for use in the extraction section of the first cycle of reprocessing HIFAR fuel elements has been developed, and the necessary experience for the ultimate design of an active mixer-settler bank for chemical processing work has been obtained. Baillie and Cairns (1958), Buck et al. (1958) and Siddall, Parker and Prout (1957) have stated that control of interface height is most easily achieved with the KAPL pump mix type of mixer. Experiments made to determine the effect of flow rate and impeller position on the interface level show that satisfactory control can be obtained by correct positioning of the impeller.

Impeller speed has no effect on pumping or interface control in the range used, but does affect the mixing efficiency. It is therefore possible to obtain the best mixing characteristics without affecting the interfaces.

Extraction efficiency was close to 100% under flowsheet conditions, and it was possible to reduce uranium losses to within the required 0.01%

It is proposed that when it is convenient, the decontamination efficiency should be checked also by using trace fission products. It is recommended that further work be done to determine whether a mixer-settler suitable for use at the lower flow rates experienced in the backwashing section can be developed.

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APPENDIX

TABLE 1

Uranium Concentration During Run No. 1

Unit	Time hr.	Uranium Concentration g/l	
		Aqueous Phase	Organic Phase
1	2	0.0027	0.0056
	5½	0.51	2.58
	7	1.51	7.91
	8½	2.18	9.41
	10	2.52	8.67
	Equilibrium	2.74	9.43
2	2	0.0023	0.22
	5½	0.19	5.86
	7	0.56	9.82
	8½	1.01	10.54
	10	1.17	10.51
	Equilibrium	2.20	9.55
3	2	0.0052	0.20
	5½	1.27	8.91
	7	2.46	10.78
	8½	3.15	11.05
	10	3.67	10.87
	Equilibrium	3.65	10.98
4	2	0.062	0.22
	5½	0.214	10.77
	7	0.302	11.39
	8½	0.626	10.74
	10	0.925	10.58
	Equilibrium	1.24	10.30
5	2	0.0070	0.041
	5½	0.271	11.45
	7	0.810	11.33
	8½	1.72	10.73
	10	1.84	10.91
	Equilibrium	1.56	10.99

(contd.)

(Table 1 continued)

Unit	Time hr.	Uranium Concentration g/l	
		Aqueous Phase	Organic Phase
6	2	0.0050	0.033
	5½	0.215	8.47 Extraction Section
	7	0.685	6.79
	8½	0.465	5.67
	10	0.498	6.05
	Equilibrium	0.397	6.09
7	2	0.0069	0.43
	5½	0.158	2.42 Extraction Section
	7	0.210	2.64
	8½	0.210	2.35
	10	0.170	2.11
	Equilibrium	0.119	2.10
8	2	0.0009	0.13
	5½	0.146	1.51 Extraction Section
	7	0.0471	0.77
	8½	0.0369	0.58
	10	0.0369	0.56
	Equilibrium	0.0238	0.56
9	2	0.00078	0.00038
	5½	0.00764	0.540 Extraction Section
	7	0.00764	0.230
	8½	0.00950	0.153
	10	0.00930	0.154
	Equilibrium	0.00660	0.132
10	2	0.00025	0.00016
	5½	0.00520	0.152 Extraction Section
	7	0.00150	0.0430
	8½	0.00228	0.0340
	10	0.00190	0.0340
	Equilibrium	0.00118	0.0270

**TABLE 2**

Composition of the Entering Streams for Run No. 1

Feed	Strip	Solvent
A solution of aluminium nitrate, 1.66 M and containing 3.67 g/l of uranium as uranyl nitrate	1 M. aluminium nitrate	5.13% T.B.P. by volume in odourless mineral spirit

**TABLE 3**

Values of Points on the Operating Line for Run No. 1

$x_m$ (g/l)	$y_{m-1}$ (g/l)
10	32.4
3.28	10.95
1	3.24
0.1	0.318
0.01	0.0263
0.005	0.0100
0.003	0.00350
0.0019	0

**TABLE 4**

Murphree and Modified Efficiencies for Run No. 1

Stage	$E_{me}$ (%)	$E_{mr}$ (%)	$E_{ce}$ (%)	$E_{cr}$ (%)
5	94	84	94	84
6	81.5	93	74	93
7	64.5	87	64	61
8	70.5	94	65.5	91
9	76.0	94	71.5	64
10	83	95	83	67.5

**TABLE 5**

Composition of Inlet and Outlet Streams for Run No. 3

Feed	Strip	Solvent
Nitrate deficient aluminium nitrate containing Al at molarity 2.36 and -2.0 N. nitrate deficient. 3.66 g/l nitrate present as uranyl	0.6M aluminium nitrate	4.83% T.B.P. by volume in odourless mineral spirit

**TABLE 6**

Uranium Concentrations During Run No. 3

Unit	Time hr.	Uranium Concentration g/l	
		Aqueous Phase	Organic Phase
1	4	0.138	5.90
	7	0.216	9.04
	9	1.37	9.18
	Equilibrium	0.396	9.70
2	4	0.011	9.04
	7	0.190	11.4
	9	1.09	12.4
	Equilibrium	0.566	12.4
3	4	0.009	11.0
	7	0.411	12.6
	9	2.72	12.7
	Equilibrium	1.72	13.1
4	4	2.17	13.2
	7	1.97	13.5
	9	1.66	13.8
	Equilibrium	0.28	14.0
5	4	1.47	14.4
	7	1.51	14.2
	9	1.28	13.4
	Equilibrium	2.0	14.0
6	4	0.011	3.52
	7	0.042	3.35
	9	0.124	3.58
	Equilibrium	0.086	3.40

(contd.)

(Table 6 continued)

Unit	Time hr.	Uranium Concentration g/l	
		Aqueous Phase	Organic Phase
7	4	0.0073	0.48
	7	0.0077	0.62
	9	—	0.53
	Equilibrium	0.0005	0.56
8	4	0.00031	0.022
	7	0.00038	0.022
	9	0.00041	0.026
	Equilibrium	0.00035	0.037

**TABLE 7**

Values of Points on the Operating Line for Run No. 3

$x_m(g/l)$	$y_{m-1}(g/l)$
10	39
3.43	13.3
1	3.9
0.1	0.39
0.01	0.039
0.001	0.00338
0.0005	0.00182
0.0002	0.000273
0.00013*	0

\* This was the lowest assay figure obtained, and has been taken as the raffinate concentration, although some doubt exists as to the accuracy of this figure, due to difficulties encountered in the uranium analyses at low values.

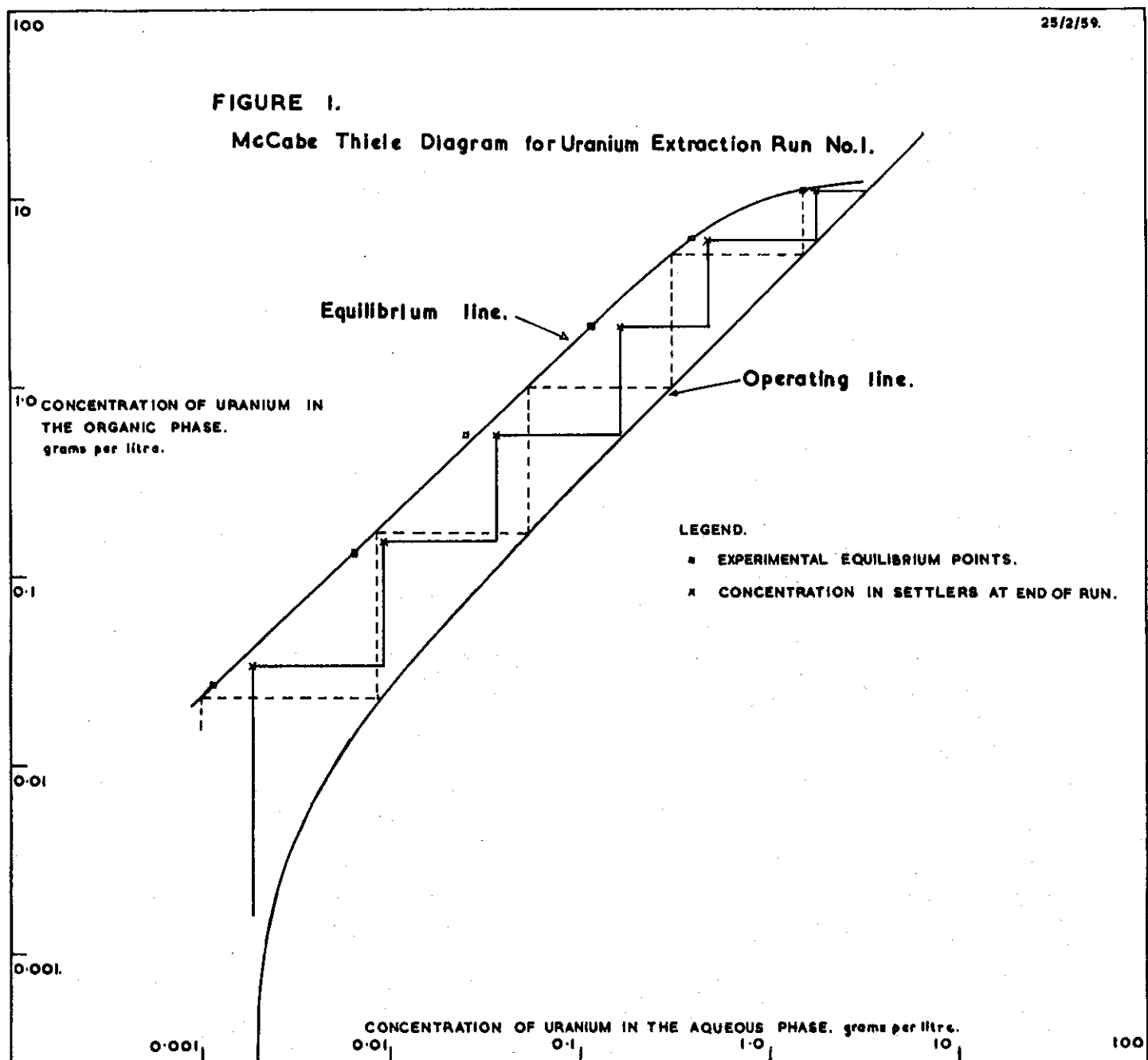
**TABLE 8**

Murphree and Modified Efficiencies for Run No. 3

Stage	$E_{me}(\%)$	$E_{mr}(\%)$	$E_{ce}(\%)$	$E_{cr}(\%)$
5	98	94.5	106.5	94.5
6	71.5	96.0	70	123
7	69	97.5	69	86
8	—	98	—	99



**FIGURE 1.**  
**McCabe Thiele Diagram for Uranium Extraction Run No.1.**



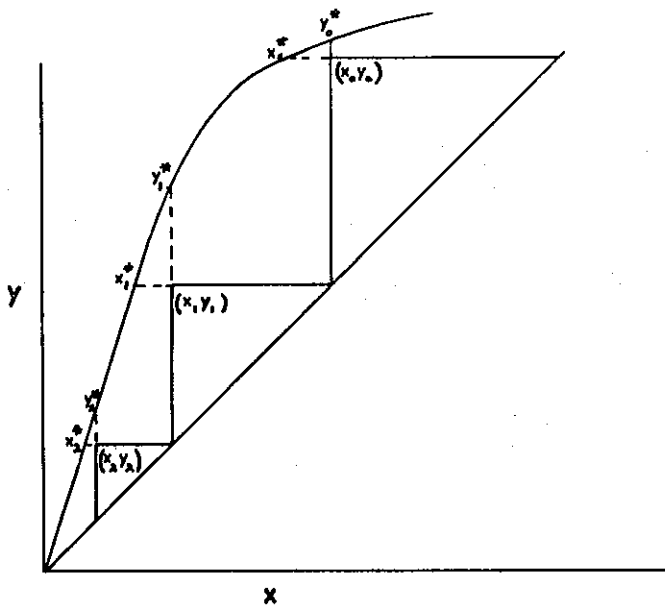


Figure 2a.

Basis of Murphree Stage Efficiency.

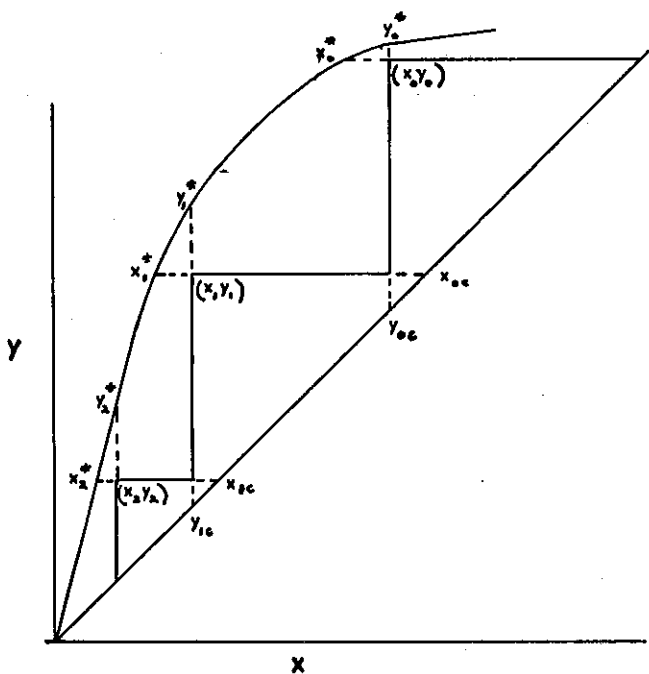


Figure 2b.

Basis of Modified Stage Efficiency.

**Legend.**

Symbols.

x — Concentration of Uranium in the Aqueous Phase.

y — Concentration of Uranium in the Organic Phase.

Subscripts.

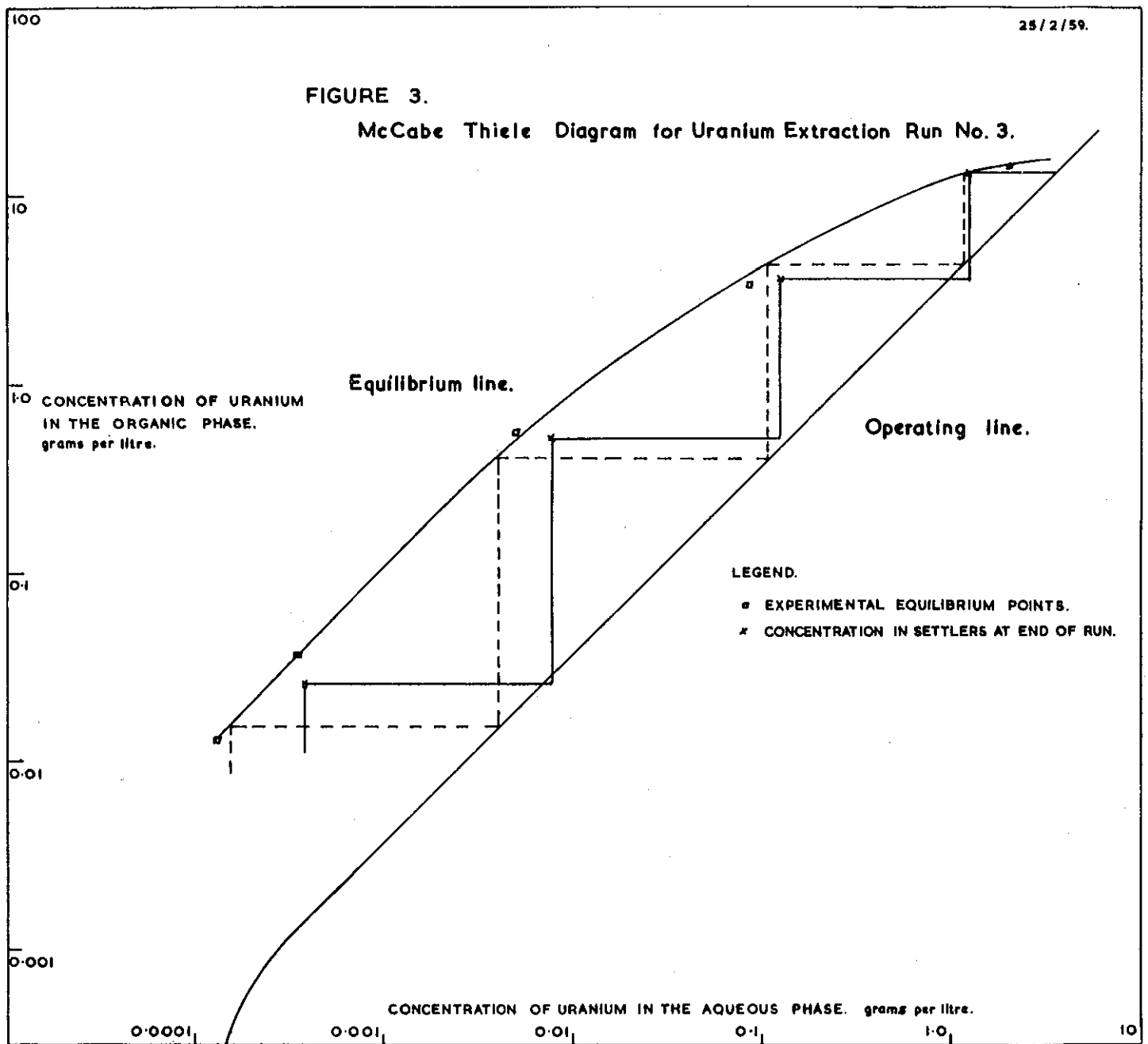
Numerical. — Stage Number.

c — Values for Modified Stage Efficiency.

Superscripts.

\* — Equilibrium Value.

FIGURE 3.  
McCabe Thiele Diagram for Uranium Extraction Run No. 3.



# EQUILIBRIUM DIAGRAMS FOR EXTRACTION AND BACKWASHING SECTIONS OF THE PROPOSED FIRST CYCLE.

FIGURE 4.

EXTRACTION EQUILIBRIUM LINE. BETWEEN 5% T.B.P. AND 2.34 M. ALUMINIUM AS A BASIC NITRATE 1.0 N. NITRATE DEFICIENT.

CONCENTRATION OF URANIUM IN THE ORGANIC PHASE. g/l.

BACKWASHING EQUILIBRIUM LINE BETWEEN 5% T.B.P. AND 0.01 N. NITRIC ACID.

CONCENTRATION OF URANIUM IN THE AQUEOUS PHASE. g/l.

0.001

0.01

0.1

1.0

10

100

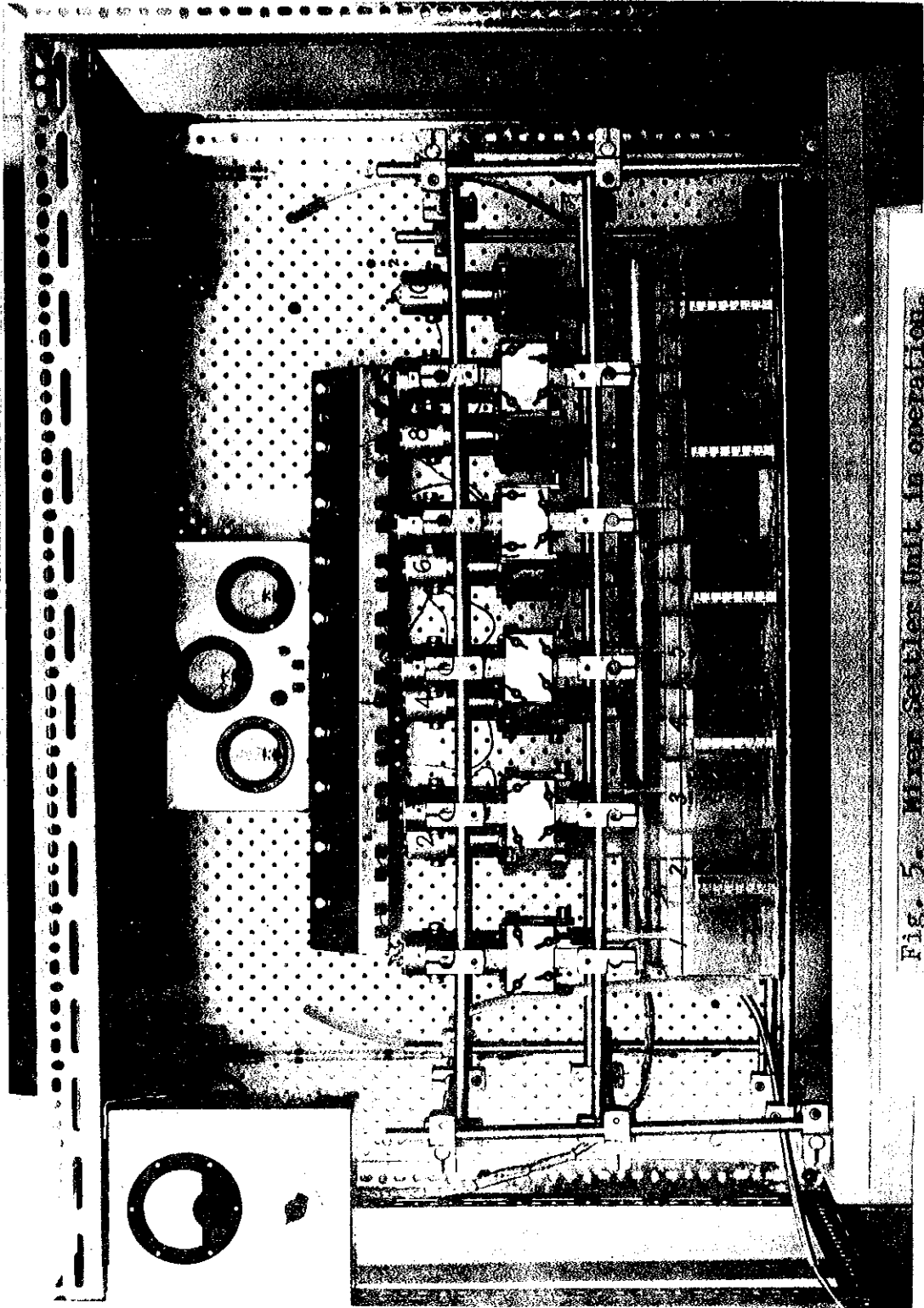


Fig. 5. Wren Series Unit in operation.

Fig. 6. Mixer-Settler Unit. SHOWS HOUSES IN POSITION.

