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DEVELOPMENT OF A TEN STAGE MIXER SETTLER FOR U235 SOLUTIONS

PART I

by

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Sydney, November, 1958.

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Abstract

This report deals with the development of a 10 stage mixer settler. The unit is based on extraction conditions expected for processing Hifar fuel elements, on which information is available, but the equipment can be used for other fuel types.

Qualitative experimental work, which was necessary before the multistage unit could be designed with confidence, is reported for a single stage mixer settler.

A brief review of the criticality problem for processing U235 enriched fuel is given and actual dimensions are specified for the case of Hifar elements.

The proposed work with the 10 stage unit is given.

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1. INTRODUCTION

It may be assumed that liquid–liquid extraction will be the unit operation used for separating fissile or fertile material from fission products in fuels from Hifar and the H.T.G.C. reactor. It may also be assumed that mixer settlers are suitable contacting devices. In the case of Hifar fuel element processing, tri–butyl phosphate in kerosene will be the extractant for uranium.

As a basis for developing mixer settler equipment, the fuel element throughput for Hifar at full power has been accepted, as well as the feed, stripping, and organic flowrates necessary. With these conditions, it is possible to measure the efficiency of the mixer settlers and adequacy of the final design in relation to stirrer speeds, backmixing or bypassing.

This approach is necessary since the H.T.G.C. fuel element has not yet been selected or developed, nor is it possible to determine at present if it is necessary to process these elements at all. It is important to remember however, that the experience obtained for Hifar flow conditions on such variables as port sizes, pressure drops, emulsification, phase separation and stirrer shapes, will be applicable to other fuel systems.

This report deals with the development necessary to design a multistage mixer settler unit. A single stage is first designed and used experimentally with water and kerosene–TBP phases only, to determine the optimum values of the various design variables in order to build a multistage unit for full scale extraction runs.

2. SELECTION OF MIXER–SETTLER DIMENSIONS

(a) Solvent and Diluent Selection

A survey of relevant literature revealed that di–butyl phosphate is invariably the most important undesirable product of both chemical hydrolysis and radiation damage to T.B.P. (1) (2) (3). Other impurities present in the original T.B.P. may effect extraction to some extent, or yield undesirable products of radiolysis. For active work the purest T.B.P. available should be selected in order to eliminate this latter effect as far as possible. For the present work, commercial grade T.B.P. was used which was obtained from Thomas Denton and Co. Pty. Ltd., Sydney. The T.B.P. had a density of 0.976 g/ml. at 20°C with a refractive index of 1.4205 at 20°C.

The diluent is the primary source of offending products, (3) (4), on a long time performance, especially when in contact with nitric acid solutions. Products of radiolysis in the diluent are not removed by an alkaline wash, and hence accumulate. An extensive survey of available kerosene diluents was carried out, and a selection made on the basis of low aromatic content, low unsaturated content and a guarantee of reproduceability of the product. The first two properties ensure a minimum build–up of offending products of radiolysis, as the hydrocarbon selected should be low in unsaturated and aromatic content for maximum radiation and chemical stability, while the third is necessary for stable operation. The diluent selected was Vacuum solvent R100, Odourless Mineral Spirit obtained from Vacuum Oil Co. Pty. Ltd.

This kerosene diluent gave a boiling range of 179° – 202°C and a refractive index of 1.4195 at 20° C. The density was 0.754 g/ml. at 20°C and the bromine number 0.9. The analysis figures compare favourably with those quoted for T.B.P. and kerosene used by others for extraction (1) (5) (6) (7). Analysis figures for the aromatic content of the kerosene are as yet not available from A.C.S. group. Figures for monobutyl phosphate, di–butyl phosphate, free phosphoric acid and butanol are also not yet available.

(b) Availability of Elements for Reprocessing

At full power, Hifar operates at 10 M.W. A 20 percent burnup of the U235 fuel is proposed, which means that 500 grams of the 2.5 kg. charge will be used. As 1 gram of U235 produces about 1 M.W.D., this burnup represents the production of 500 M.W.D. This means that the complete charge of fuel elements will need to be renewed every 50 days.

The actual proposed changing cycle is shown below and this corresponds to a burn up of considerably less than 20 percent (8).

Run 12 days,	change 8 elements,	2 days	–	14 days
" 12 days,	" 8 "	2 "	–	14 "
" 12 days,	" 9 "	2 "	–	14 "
total	25 elements in			42 days

Assuming the reactor operates at 10 M.W. for 11 months of the year on this changing cycle, a maximum of 200 elements would be available for reprocessing each year.

(c) Reprocessing Cycle

It is considered that 3 elements per day is the maximum that could be handled in one dissolver (9). Treating 3 elements per day with the solvent extraction operating continuously, the processing time for 200 elements is $\frac{200}{3}$ days or approximately 10 weeks.

A longer processing period may be required depending on the dissolution step, but a large increase in processing time would mean smaller solution flow rates and smaller extraction equipment which would give fabrication and operation difficulties.

(d) Solution Concentrations and Flow Rates

(i) It is anticipated that an acid deficient solution will be produced in the dissolution stage, and some figures for the expected concentrations of the most important ions after one irradiation are (10)

Al : 2.5 M.
U (total) : 3.57 g/l.
NO_3 : –1.2 N.

In order to obtain these concentrations, it will be necessary to strip the elements of all mechanically removable aluminium, and it is assumed that this will be done (9).

The total bulk of solution at this concentration per element will be 25.2 litres, containing 90 grams of uranium and 1700 grams of aluminium. This results in a flow rate of:

$$25,200 \times \frac{3}{24 \times 60} = 52.5 \text{ cc/min. for the feed}$$

(ii) A 5% v/v solution of T.B.P. in kerosene has been selected as solvent. A total of 8 litres is required for each element. This results in a loading of about 11.2 grams per litre of uranium after extraction. This gives a solvent flow rate of:

$$8,000 \times \frac{3}{24 \times 60} = 16.6 \text{ cc/min.}$$

(iii) A flow–rate for the stripping solution of 6.25 cc/min. has been adopted.

(iv) Organic will enter the backwashing section at the rate of 16.6 cc/min., and the aqueous backwash flow–rate will be adjusted to $5000 \times \frac{3}{24 \times 60} = 10.4$ cc/min., 5 litres of aqueous backwash being required per element. This will result in a final uranium concentration of 18.0 grams per litre in the aqueous backwash.

(e) Mixer–Settler Design

Using the above flow rates, an estimate of the required mixer–settler size has been made by scale down from units in use at K.A.P.L. (11) (12) (13).

It is understood that solvent residence times of approximately 1 minute and 8 minutes are used for the mixer and settler respectively in the Dounreay processing plant for Hifar type fuel elements. The aqueous residence times are not known.

Residence times vary widely for different systems and can only be reliably determined by actual operation. Also, in both mixer and settler there is a residence time for both aqueous and organic phases depending on flowrate of each phase and the volume of either the mixer or settler occupied by the particular phase.

Residence times used here have been based on total flow of both phases, as a first approximation. There is very little information in the literature on typical residence times but some have been collected in Table I to give an indication of their order of magnitude.

Some settling tests using 5% T.B.P. and water slightly acid with nitric acid were carried out. These results are also shown in Table I. Residence times based on the total flow of 2 minutes and 8 minutes in the mixer and settler respectively were finally selected.

Total flow of both phases in the extraction section is:

52.5 (feed) + 16.6 (solvent) + 6.25 (strip) ≑ 75.5 cc/min.

Thus the approximate mixer size required is 150 cc. and the approximate settler size required is 600 cc.

Scale up and scale down of mixer–settler units can be effected simply by geometric similarity (13). This means that the dimensions of the scaled down unit may be obtained by multiplying those of an existing unit by the ratio of the cube roots of the required volumes.

The K.A.P.L. mixer–settler has the following dimensions:

Mixer: 3" x 3" x 2½" – capacity 370 cc.

Settler: 3" x 8" x 5" – capacity 1970 cc.

Thus the required mixer size reduction factor is $\sqrt[3]{\frac{150}{370}} \doteqdot 0.74$ which gives a new mixer size of 2.2" x 2.2" x 1.9".

Required settler size reduction factor is $\sqrt[3]{\frac{600}{1970}} \doteqdot 0.67$, which gives a new settler size of 2.05 x 5.5 x 3.4

Hence the required design of the mixer settler unit is:

Mixer: 2.5" x 2.5" x 2.0"

Settler: 2.5" x 6" x 3.5"

In actual fact, the mixer depth was made variable by use of a false bottom as this dimension is not liable to simple scale down as all other dimensions are (13).

3. EXPERIMENTAL WORK WITH THE SINGLE STAGE

A perspex single stage mixer–settler (see Fig. 1) was made up according to the dimensions determined above. All ports and baffles were made so that they were interchangeable, in order to determine optimum sizes.

Initially, tests were carried out in beakers to determine the best volume ratio of organic to aqueous from the point of view of settling characteristics. It was found that it was essential for the organic to be the continuous phase since with organic in aqueous dispersions, emulsions were formed which tended to be very stable and settled very slowly.

Tests were carried out over a range of organic to aqueous ratios. At high organic to aqueous ratios, fine aqueous drops in suspension tended to settle slowly, but as the ratio was decreased settling characteristics improved until at an organic to aqueous ratio of 2:1, best settling characteristics were obtained. Good disengagement could still be obtained at lower values provided the aqueous phase was always dispersed in the organic. This effect can be promoted by drawing the aqueous phase into the organic by means of a suitable impeller. As the ratio was further decreased, settling characteristics deteriorated and at a ratio of about 1:2 the organic phase could no longer be maintained as the continuous phase, and stable organic in aqueous emulsions tended to form. This result is confirmed by Treybal (14) who found that it is difficult to obtain dispersions containing more than approximately 75 percent by volume of dispersed liquid or less than 25 percent continuous phase. This is attributed to the fact that the void space in a closely packed bed of spheres is 26 percent by volume (14a). For ratios of liquids of 1:3 or less by volume, the liquid present in the minority will probably be the dispersed phase.

After the initial tests, the following variables were investigated on the single stage:

(a) Mixed phase port height
(b) Mixed phase baffle
(c) Mixed phase port size
(d) Mode of heavy phase entry to mixing compartment
(e) Effect of paddle–type impeller speed and position on pumping and mixing
(f) Diameter of paddle–type impeller blades
(g) Centrifugal–type impeller operation
(h) Effect of flow rate with both impeller types

In each case all variables other than the one being investigated were kept constant, and measurements of flow rates, interface level, length of settling zone, stirrer speed, temperature, heavy phase head required for flow and in some cases, organic to aqueous ratio in the mixer were taken.

(a) Mixed Phase Port Heights

As the mixed phase port height was varied, it was found that the head required for aqueous flow increased with port height, until a point was reached where backmixing began to occur and operation became unsatisfactory as there was a tendency towards emulsification and a much greater settling area was required. Severe emulsification occurred when the port was moved below the interface. Best operating conditions occurred when the port was between ¼" and ½" above the interface.

(b) Mixed Phase Baffle

After investigating several different baffle arrangements, it was found that a baffle, open at both sides, 1½" wide, not less than 1" high and central over the port was the most satisfactory, from the point of view of low pressure drop and even entry to the settler. This baffle was best positioned on the mixer side of the mixed phase port, between 1/8" and ¼" from the wall.

(c) Mixed Phase Port Size

The most interesting effect noted as a result of varying the mixed phase port size was that it caused a marked change in the organic to aqueous ratio in the mixer. Virtually all difference in heavy phase head required for flow can be attributed to the difference in organic to aqueous ratio noted in the mixer with the different port sizes. It appears that a ¼" diameter hole is quite large enough to take the flow without appreciable pressure drop. The organic to aqueous ratio as measured by sampling increased from 0.3 for the ¼" diameter hole to about 3 for 1" x ¼" slot. Mixing appeared to be uniform, and the dispersion finer in the case of ¼" diameter hole. A ½" x ¼" slot was then tried, and found to be very satisfactory, giving an organic to aqueous ratio very close to 2, which had been found previously to be the optimum for quick settling.

(d) Mode of Heavy Phase Entry to Mixing Compartment

The false bottom below the mixing section (used in the above experiments) was removed, and a series of baffle arrangements over the aqueous inlet tried, to determine whether this compartment was essential. None of these arrangements were satisfactory due to backmixing or unfavourable pressure drops which occurred. Mixing also appeared to be less satisfactory than with heavy phase entry below the impeller and there was no pumping action. It was therefore decided that the false bottom would be essential for proper operation.

Entry of the aqueous phase to the mixing compartment was by way of a hole in the false bottom, the actual aqueous inlet port from the previous settler being below the false bottom. A hole of ¾" diameter was found to be most suitable as smaller holes introduced too large a pressure drop and larger holes caused backmixing to take place.

(e) Effect of Paddle-Type Impeller Speed and Position

Best mixing conditions were obtained with the impeller about ½" above the hole from the false bottom, but at this height, no pumping action was obtained, and thus the speed of the impeller had no effect on the heavy phase head required for flow.

Some pumping was obtained when the impeller was ¼" above the base, but not enough for stable operation. With the impeller 1/8" above the base, there was a significant reduction in the required head for heavy phase flow, but the effect of speed variation was small in comparison. Higher speeds were necessary at this depth to obtain reasonable mixing.

A riser tube from the false bottom was next incorporated in an attempt to combine better mixing with good pumping. The top of the tube was ½" above the mixer base, and the impeller was set 1/8" above this. This resulted in good mixing and a reasonable pumping action, but it is questionable whether this type of impeller could produce the inherently stable system which is produced by the centrifugal-type impeller. The only method of controlling the interface with the paddle-type impeller would be to raise or lower it as required. Tests will have to be carried out on a bank of mixer-settlers before any true assessment of impeller characteristics can be made.

(f) Diameter of Paddle-type Impeller Blades

All initial tests were carried out using an impeller consisting of four ¼" wide blades on a ¼" diameter shaft, each blade being ½" high. Best mixing speed with this impeller was about 400 r.p.m. as measured by a stroboscope. An impeller with blades each 3/8" wide was then tried, and it was found that the best mixing speed for this impeller was about 290 r.p.m. Mixing appeared to be better, and the heavy phase head was slightly reduced. There was less tendency for a vortex to form in the mixing compartment, and entry of the mixed phase into the settling compartment was much more even. An impeller with still wider blades (½" wide) was also tried, but it was found to be unsatisfactory, as the slow speed required to prevent spillage produced poor mixing. It was decided on this evidence that the 3/8" wide paddle blades would be the best to use for future tests.

(g) Centrifugal-Type Impeller Operation

A centrifugal-type impeller similar to the K.A.P.L. design (12) was made up and tested. The impeller consisted of four curved vanes, ¼" high, contained between two circular discs, 1" in diameter. A hollow intake tube, ½" long extended below the bottom disc. Best operation was found to occur when the intake tube extended ¼" into the heavy phase below the false bottom. Mixing blades on top of the centrifugal assembly were found to be essential. Quite a significant reduction (0.1") in the head required for heavy phase flow was noted, and it is felt that this would make the operation of a bank of mixer-settlers much easier. Reasonable control of the pumping action can be exercised either by varying impeller speed or height. It was noticed that after any alteration of the system conditions was made, the interface rapidly settled to its new height and remained quite steady. It has not been possible to obtain such stability with the paddle-type impeller.

(h) Effect of Flow Rate on both Impeller Types

Quite large variations in organic flow rate were used during the tests carried out. These variations do not appear to have had any detectable effect on the interface level. Experiments were therefore carried out in which the aqueous flow rate was varied within the limits of half to double the anticipated flow rate for the mixer-settler bank, i.e. 25 cc/min. to 100 cc/min. The resultant change in interface level for both the paddle-type and centrifugal-type impellers was measured.

The variation in interface level which occurred in the case of the paddle-type impeller was found to be about twice as great as that for the centrifugal-type impeller, but in both cases the variation was reasonably small. This indicates that control over the small anticipated flow fluctuations will not be too difficult. Some difficulty may be experienced at a higher flow rate with the paddle-type impeller, as it was noted that the required head for heavy phase flow exceeded the static head at flow rates greater than 60 cc/min. As previously stated, it is felt that no final selection of impeller type can be made until a bank of mixer-settlers has been built and operated.

(i) Other Effects Noted

(i) The settling section has proved to be adequate to cope with any reasonable variation in flow rates. Only about 1/3 of the total settling area is required under optimum conditions and it has been found that flooding of this section does not occur until flow rates are at least three times the normal aqueous flow rate of 50 cc/min.

(ii) The free surface level in the mixing section is always lower than that in the settling section. Due to this, the relatively small baffle in front of the organic inlet port was quite effective in preventing backmixing through this port.

(j) A summary of the dimensions and location of the various mixer-settler features as determined above, and as incorporated in the 10 stage mixer-settler design is given below:

Mixer dimensions	2½'' x 2½'' x 2¾'' high
Settler dimensions	2½'' x 6'' x 4'' high
Mixed phase port dimensions	½'' x ¼'' high
Mixed phase port location	centre 2'' above settler base
Mixed phase baffle dimensions	1½'' x 1½'' high
Mixed phase baffle location	base ¼'' above mixer base
Organic phase port dimensions	1'' x ¼'' high
Organic phase port location	centre 3⅜'' above settler base
Organic phase baffle dimensions	2½'' x 1'' high
Organic phase baffle location	base 3'' above settler base
Aqueous phase port dimensions	1'' x ¼'' high
Aqueous phase port dimensions	base ¾'' above settler base
False bottom location	½'' below mixer base
Dimensions of hole in false bottom	¾'' diameter

4. CRITICALITY CONSIDERATIONS

(a) General Considerations

The design of the single stage mixer-settler unit having been finalised, it was decided to build a bank of ten such units to investigate operation under flowsheet conditions. Before this was done, it was felt that it was necessary to determine whether such a bank would fall within critically safe limits.

A survey of available literature on criticality considerations in solutions of U235 enriched uranium was carried out in order to determine whether the proposed mixer-settler design for the solvent extraction operation of the chemical processing plant is safe.

Any one of the following variables may effect the point at which criticality is reached in a solution, and hence may be used to exercise control of criticality (15) (16) (17) (18) (19) (20).

(i) Mass of Fissionable Material

If the mass of fissionable material in a single isolated vessel is kept below a certain value, criticality cannot be reached.

(ii) Elemental Constituents of a Solution

Atoms of elements other than the fissionable material may cause a variation in the conditions under which criticality is reached. These atoms may act either as moderators or as neutron poisons. For example, the main non-fissionable elements present in Hifar fuel reprocessing solutions are hydrogen, aluminium, carbon and nitrogen. Hydrogen acts as an extremely good moderator, whilst the effect of aluminium is negligible. Nitrogen, in the nitric acid solution is a neutron poison, as it absorbs neutrons. Atoms which act as moderators greatly decrease the minimum critical mass, while those acting as poisons increase this value.

(iii) Concentration

Some concentration (i.e. some value of the ratio of moderator to fissionable material) will give the minimum critical mass for any solution, while some other concentration will give the minimum critical volume. There is a concentration below which criticality is impossible.

(iv) U235 Enrichment

For solutions of any concentration of uranium, or of any volume, there is a value for the U235 enrichment of the uranium below which criticality cannot be attained.

(v) Poisons

Heavy neutron absorbers such as cadmium, either in solution or as plate surrounding the solution act as poisons. They increase the minimum critical mass and are more effective if dispersed homogeneously.

(vi) Geometric Considerations

The critical mass depends strongly on the surface area to volume ratio. When this ratio is a minimum for a particular shape, critical mass is a minimum. As the shape is elongated, greater masses are required for criticality. This leads to the concept of "infinite" cylinder diameter" and "infinite slab thickness." Below these values cylinders of any length or slabs of any area cannot be made critical.

(vii) Reflecting Materials

Some materials reflect back neutrons escaping from the active material. This causes a decrease in the minimum critical mass. Water is a serious hazard in this respect as it is an excellent reflector. For design purposes, it is normal to consider the active material completely surrounded by a water reflector.

(viii) Interaction

Individual sub–critical systems may become coupled as a result of interchange of neutrons. Hence the minimum critical mass per unit will decrease. Where the material between the units is water, they are effectively isolated by 12" of water.

(b) Application

Hifar fuel processing solutions will contain, on reaching the solvent extraction section, uranium (about 90% U235) aluminium and fission products as nitrates. The latter can be neglected from criticality considerations as they represent only a very small proportion of the material present. All the following figures are calculated as if the uranium were present as fluoride, not nitrate. This introduces an additional factor of safety as nitrogen is a neutron poison. (21) (22).

For design purposes, it is normal to consider the case where the solution is at optimum moderation, completely surrounded by a water reflector and contains uranium of 90% U235 assay, as this is the worst set of conditions possible (23).

Geometric limitation is considered the best criterion for safety, although quite a number of other parameters can be, and are used in order to achieve an "always safe" design. A number of these parameters and their minimum values for criticality are listed in Table II. Some commonly accepted design values are also listed. The values given for volume and mass apply to spherical configurations which results in the minimum critical value. Configurations other than spherical yield higher critical values. It is seen in table II that the safe design thickness for an infinite slab is 1.4 inches which is less than the proposed finite slab of 4 inches. Hence a method for determining critical slab heights for a finite slab was required.

Heavy absorbers such as boron steel and cadmium plate improve safety by absorbing neutrons.

Stainless steel containers up to a reasonable thickness increase the minimum critical slab thickness slightly. (23). If 1/16" stainless steel plate is used for the mixer–settler bank, this will increase the minimum critical mass.

Henry, Mallet and Newlon (23) give some experimental figures for safe dimensions for U235 solutions in stainless steel containers, and some safe figures used in the design of pipe–line layouts.

(i) A cylinder 12" diameter x 4.8" high in a water reflector was found to be safe.

(ii) A slab 20" x 20" x 5.5" high in air was found to be safe.

Removal of the reflector will increase cylinder diameter by 50%, slab height by 70% and volume by 135%.

For pipe configurations:

(iii) 90° equilateral L, T and + connections may be made with pipes whose diameter is reduced from the allowable infinite cylinder diameter of 5" by factors of 1.1, 1.2 and 1.3 respectively.

(iv) A 5" diameter pipe may be curved in a 2' radius providing neutron absorbing material equivalent to water is placed inside the curve.

(v) A 4" diameter pipe may "T" into an always safe (1.25" thick) slab.

(vi) Any number of 1" pipes may "T" into a 5" pipe provided these points are 15" apart.

(vii) Any group of pipes within a 5" circle or a 1.25" slab are geometrically safe and may be treated as a solid pipe or slab respectively, e.g., several small pipes within a 4" diameter circle may "T" into an always safe slab.

(c) Calculations for Finite Systems

Schuske and Morfitt (24) (17) give a simple empirical equation relating vertical height of a cylinder to its diameter for a water enclosed stainless steel vessel, using uranyl fluoride at 93% U235. A short account of the method used by them, and the results obtained is given below.

A series of curves are obtained by plotting critical height of a cylinder as a function of its diameter for various moderations (i.e., H : U235 ratios). See Figure 2. If H represents the cylinder height, and D the cylinder diameter, then the vertical asymptote (a) of any of these curves represents the diameter of the just critical infinite cylinder at the given moderation and the horizontal asymptote (b) represents the heights of the just critical infinite slab at a given moderation.

It was found that the following equation gave the best fit to these hyperbolic curves:–

$$(D - a)(H - b) = C$$

where "a" and "b" are the minimum critical diameter and minimum critical slab thickness respectively at a given moderation and C is a constant.

Values of "a", "b" and "C" for a series of H : U235 ratios have been calculated and appear in Table III.

By using the above constants in the formula, minimum critical height as a function of diameter and moderator can be calculated. Schuske and Morfitt further defined the constants "a" and "b" such that the value of "C" remained constant at 104 for all moderations, but since we are concerned only with the condition of optimum moderation, for design purposes it is better to take the values given at H:U235 ratio of 50, (22a).

Some values of minimum critical heights for various cylinder diameters are given in Table IV for conditions of optimum moderation, using the values of "a", "b" and "C" given above. These are compared with experimental values.

It can be seen that excellent agreement between experimental and calculated values was obtained.

Schuske and Morfitt have also developed an equation for the case of interaction between two active vessels (17). They found both experimentally and empirically that two cylinders can be considered to be isolated if separated by 12" of water or its equivalent.

(d) Application of Mixer–Settler Design

The above results have been applied to determine whether the ten stage mixer–settler proposed for reprocessing Hifar fuel elements is safe, and to determine what modifications may be necessary to ensure safety.

Overall dimensions of the proposed mixer–settler are 27" x 9" x 4" high.

Schuske and Morfitt's equation applies to cylinders, and thus, if design is such that the cylinder of equal base area and height, and hence volume, to the above rectangular slab is found to be safe when at optimum moderation and fully reflected with water, the slab will most certainly be safe.. This follows from the fact that critical mass is dependent on the surface area to volume ratio, the rectangular slab of equal volume and height to the cylinder having a greater surface area. The aqueous phase has been found to have a more efficient moderating effect than the organic phase (17a).

For the ten stage mixer–settler, the diameter of the cylinder of equal height and volume is:–

$$D = \sqrt{\frac{27 \times 9 \times 4}{\pi}} = 17.6'' = 44.6 \text{ cm.}$$

Substituting this in the formula, and using the constants at optimum moderation, the minimum height for criticality is found.

$$(D - a)\ (H - b) = C$$

$$(44.6 - 14.10)\ (H - 5.85 = 102.6$$

$$30.5\ (H - 5.85) = 102.6$$

$$H = \frac{102.6}{30.5} + 5.85$$

$$= 3.36 + 5.85 = 9.21 \text{ cm.}$$

$$= 3.63''$$

This means that if ten stages of the proposed size are used, the height should be controlled below this value. A suitable value for control could be 3.5" .

Alternatively, if a height of 4" is required, the area of the mixer–settlers must be reduced. The maximum allowable area is given by:–

$$(D - a)(H - b) = C$$

$$(D - 14.10)\ (10.16 - 5.85) = 102.6$$

$$(D - 14.10)\ 4.31 = 102.6$$

$$D = \frac{102.6}{4.31} + 14.10$$

$$= 23.8 + 14.10 = 37.9 \text{ cm.}$$

$$= \underline{14.9''}$$

From this, the maximum allowable area is:–

$$\text{Area} = \frac{\pi\ 14.9^2}{4} = 174 \text{ in}^2 .$$

There are two possible ways in which the area of the mixer–settler bank can be brought within this value.

(i) <u>By reducing the number of stages</u>

$$\text{Permissible number of stages} = \frac{174}{243} \cdot 10 = 7.1$$

i.e. 7 stages are allowable.

(ii) By reducing the settler length

$$\text{Permissible settler length} = \frac{174}{243} \cdot 9 - 3 = 6.4 - 3$$

$$= 3.4''$$

i.e. Settler must be reduced from 6" to 3.4" long.

It should be possible to carry out any of the three possible reductions in dimensions calculated above without seriously affecting the operation of the mixer–settlers.

Although the dimensions of the mixer–settler have been evaluated after an assessment of the best criticality data and calculation procedures available, it must be emphasised that a review of these calculations and concepts must be made before a mixer–settler bank could accept U235 solutions (25).

(e) Recommendations

For ease of operation, and so that as many stages as possible can be operated in the one bank, it would be more convenient if criticality could be controlled by reducing the height. A ten stage bank of mixer – settlers of overall dimensions 27" x 9" x 3.5" high would be the most suitable.

These recommendations were later checked and tentatively confirmed as satisfactory by the site Criticality Officer (25).

The depth of the settler from the organic overflow to the bottom of the settler was made 3 inches, i.e. the liquid depth is 3 inches. For reason of flexibility and to reduce any splashing tendency during trial operation it was decided to use 4 inch overall depth of the settler for the initial 10 stage unit.

Figure 3 is a sketch of a single–stage of the final ten stage unit, showing essential dimensions, Figure 4 is a view of the ten stage unit, and Figure 5 is a view of the final assembly ready for use which shows the panel made for individual stirrer control.

5. FURTHER WORK

It is intended that the following work be carried out:–

(a) T.B.P. in kerosene and water will be circulated and the suitability or otherwise of paddle stirrers as effective mixers and pumps will be determined.

(b) Pressure losses and interface stability will be measured with the T.B.P.–kerosene–water system.

(c) With a feed of 2.5 M.Al, –1.2N.NO_3, 3.57 g/l U into the 5th stage and with a strip solution of 1.0M.Al, 1.0N NO_3, the proposed 1st cycle extraction step will be checked (10).

(d) The solutions will be recirculated and the backwash step will be checked.

(e) Operation at above ambient temperatures may be attempted.

(f) Development of a probe device to indicate interface position during active operation will be attempted.

(g) Using the uranyl nitrate solutions, the efficiency of mixer–settler bank will be determined.

(h) Equilibrium diagrams for the various systems will be obtained, from samples taken during operation.

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APPENDIX

TABLE I

Residence Times for Various Systems

System	Residence Time (Based on total flow)		Reference
	Mixer	Settler	
5% T.B.P.– Kero. – HNO_3 – $Al(NO_3)_3$	–	1 min.	(26)
Hexone – water – acetic acid.	1.7 – 3.3 min.	6.3 – 12.6 min.	(27)
6% T.B.P. – Kero. – HNO_3 – $Al(NO_3)_3$	1.5 min.	From ½ min. to several hours, but for most cases 3 – 3.5 min. sufficient for settling.	(28)
Various systems in "Mini–mixer"	2 – 160 minutes total		(13)
Uranyl nitrate – T.B.P. and water – acetic acid–Hexone in "midi–mixer".	1 – 2 minutes total		(13)
20% T.B.P. – Kero. – HNO_3	¾ min.	5½ min.	(29)
5% T.B.P. – water – HNO_3	–	From ½ min. to many hours. For general case 3–4 min. satisfactory for settling.	This experimental work.

TABLE II

Minimum Critical Values and Design Values for "Always Safe" Design.

Parameter	Min.Crit. Value	Safety Factor	Design Value	Reference	Original Reference
Infinite cylinder diameter	5.5 ins.	1.1	5 ins.	(23)	American
	5.3"	–	5"	(21)	"
	5.5"	–	–	(24)	"
	5.5"	–	–	(22a)	"
	–	–	5"	(17)	"
	–	–	5"	(16)	"
	5.3"	–	–	(18a) *	" (30)
	–	–	5"	(18) *	" (19) *
	6.0"	–	–	(10)	British
	6.0"	–	–	(18) *	"
Infinite slab thick-ness	–	–	1.5 ins.	(23)	American
	–	–	1.5"	(21)	"
	2.25 ins.	–	–	(24)	"
	–	–	1.4"	(17)	"
	–	–	1.5"	(16)	"
	–	–	1.4"	(24a) *	"
	1.9"	–	–	(18a)	" (30)
	–	–	1.4"	(18) *	" (19) *
	1.9"	–	–	(10)	British
	1.9"	–	–	(18) * (18a) *	"
Volume (Sphere)	–	1.2	4.4 ltr	(23)	American
	6.3 ltr.	–	4.4 "	(21)	"
	6.3 "	–	4.4 "	(17)	"
	–	–	4.4 "	(16)	"
	6.3 ltr.	–	–	(24a) *	"
	6.3 "	–	–	(18)	" (19) *
	6.3 "	–	–	(18a) *	" (21)
	5.6 "	–	–	(18) * (18a) *	British
Concen-tration	–	1.9	5g/l	(23)	American
	–	–	5g/l	(21)	"
	–	–	5g/l	(17)	"
	–	–	5g/l	(16)	"
	11.6g/l	–	–	(24a) *	"
	11.6g/l	–	–	(18)	" (19) *
	11.0g/l	–	–	(18) *	British

/contd...

TABLE II (contd.)

Parameter	Min.Crit. Value	Safety Factor	Design Value	Reference	Original Reference
Mass (Sphere)	800g	2.3	350g	(23)	American
	800g	2.3	350g	(21)	,,
	800g	2.3	350g	(17)	,,
	–	–	350g	(16)	,,
	800g	–	–	(24a)	,,
	800g	–	–	(18) *	,, (19) *
	780g	–	–	(18) *	British

* Considerable confusion exists in some American and British literature as some figures quoted are minimum critical figures while others are design figures, the factor of safety not being indicated.

The above table tabulates all the available critical data for U235 solutions and indicates minimum values and design values. These have been interpreted from the literature data by the present authors.

TABLE III

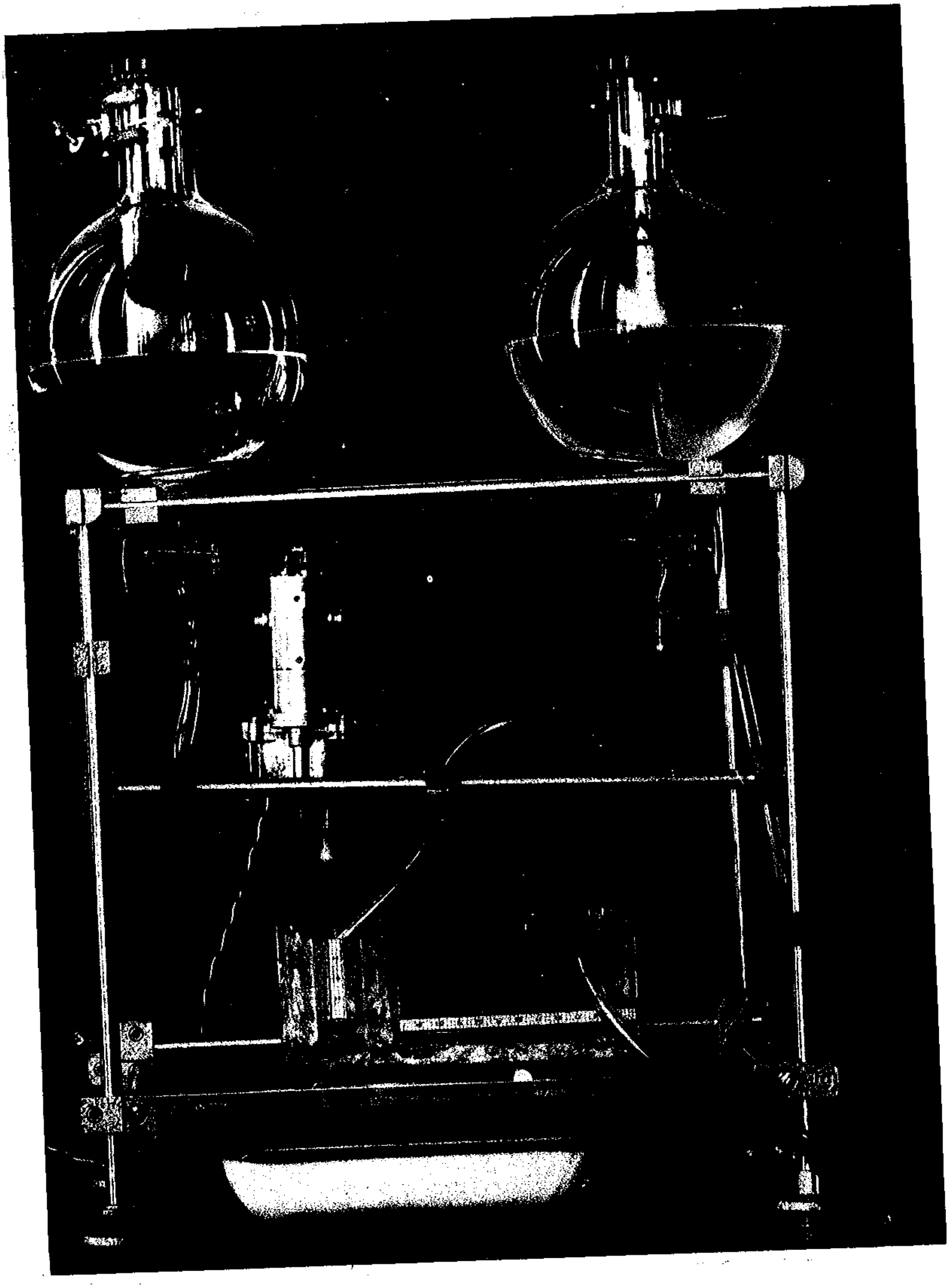
Variation of "a", "b" "C" with Atomic Ratio.

Atomic ratio, H:U235	Inf. slab height b. (cm)	Inf. cylinder diam. a. (cm)	Constant C
31.6	6.42	14.22	99.0
50	5.85	14.10	102.6
100	6.24	14.58	104.1
200	7.60	16.10	104.9
300	9.25	17.88	108.4
400	11.54	20.02	104.1

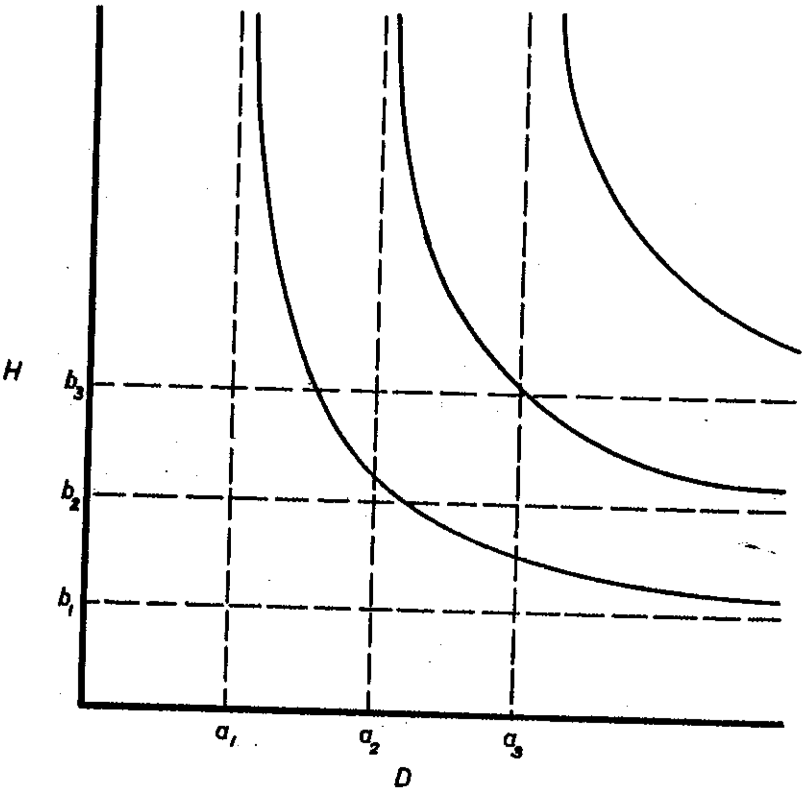
TABLE IV

Experimental and Calculated Values for Minimum Critical Height at Various Cylinder Diameters at Optimum Moderation

Cylinder dia. (inches)	Calculated Min. Crit. Ht. (cm)	Experimental Min.Crit.Ht. (cm)
15	10.1	10.1
12	12.6	12.1
10	15.0	14.9
9	17.8	17.6
8	22.2	22.3
7	32.4	33.7
6½	47.1	48.4



<u>FIGURE 1</u> : General Set-up with Single Stage Mixer-Settler.



CRITICAL CYLINDER HEIGHT versus CRITICAL CYLINDER DIAMETER AT VARIOUS MODERATIONS.

FIGURE 2.

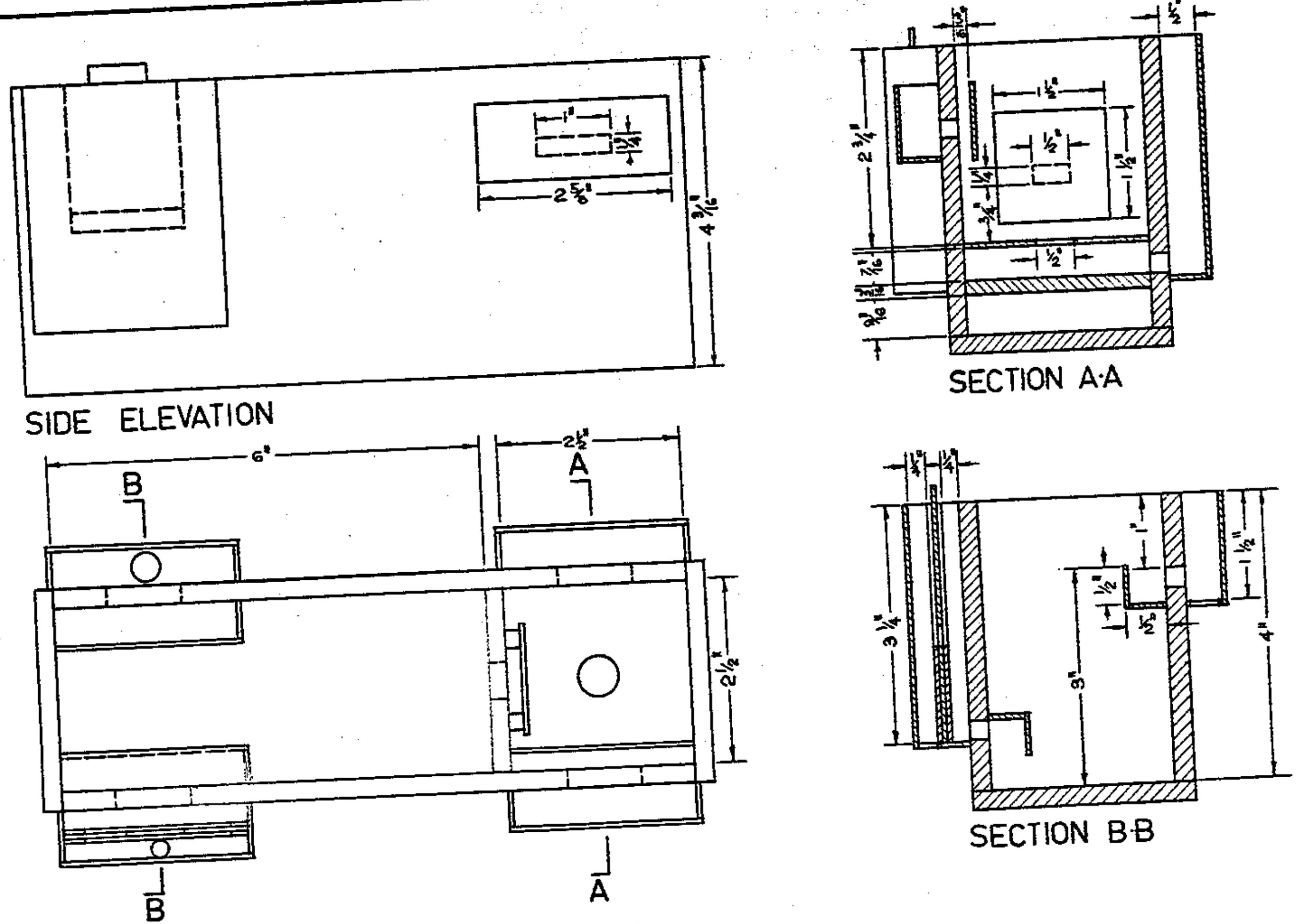


FIGURE 3.

ONE STAGE OF THE TEN STAGE UNIT.

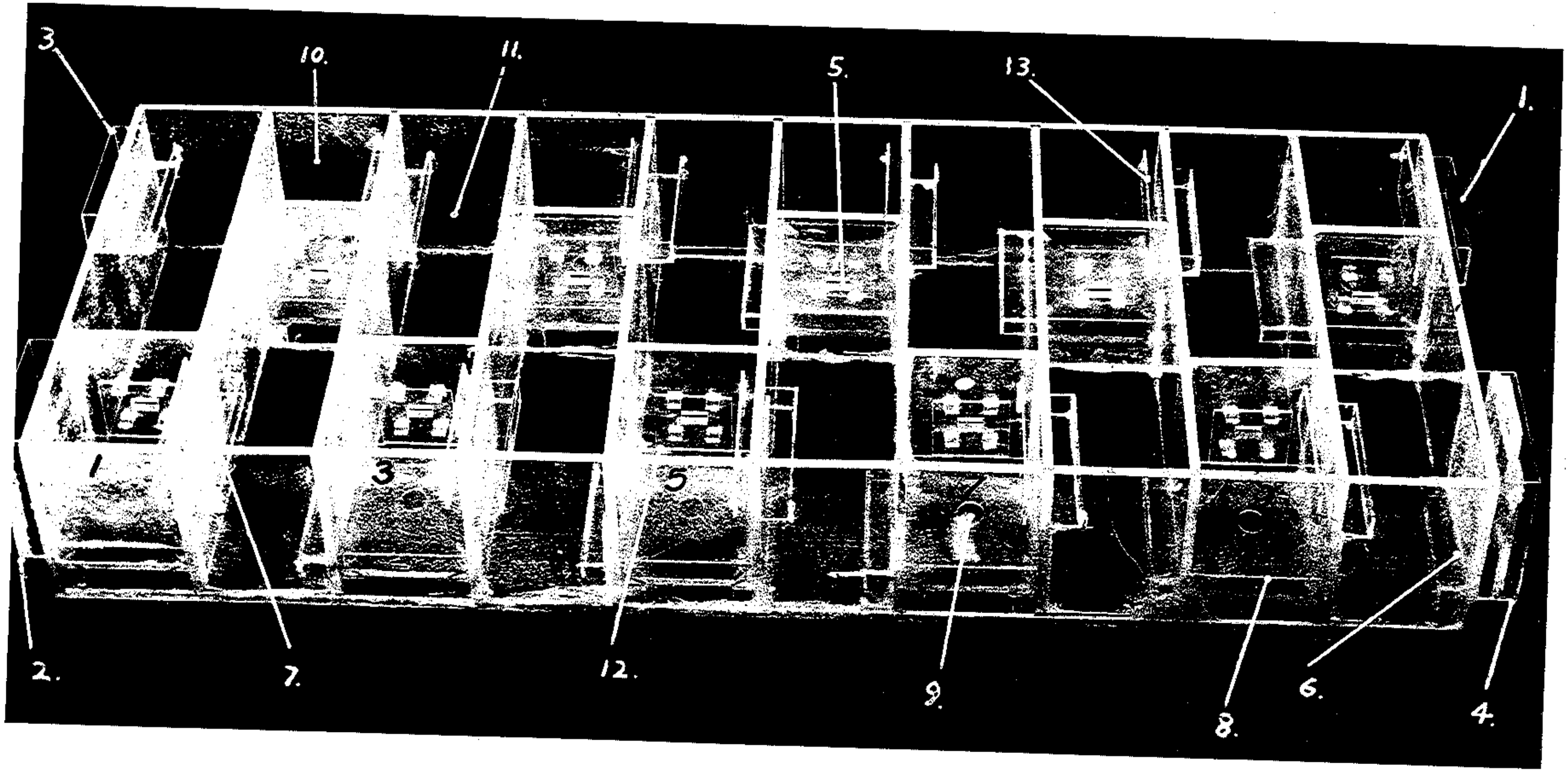


FIGURE 4 : 10 Stage Mixer-Settler.

1. Light phase feed box.
2. Heavy phase feed box.
3. Light phase overflow box.
4. Heavy phase overflow wier and box.
5. Mixed phase port.
6. Heavy phase port.
7. Light phase port.
8. False bottom.
9. Hole in false bottom.
10. Mixing compartment.
11. Settling compartment.
12. Mixed phase port baffle.
13. Light phase port baffle.

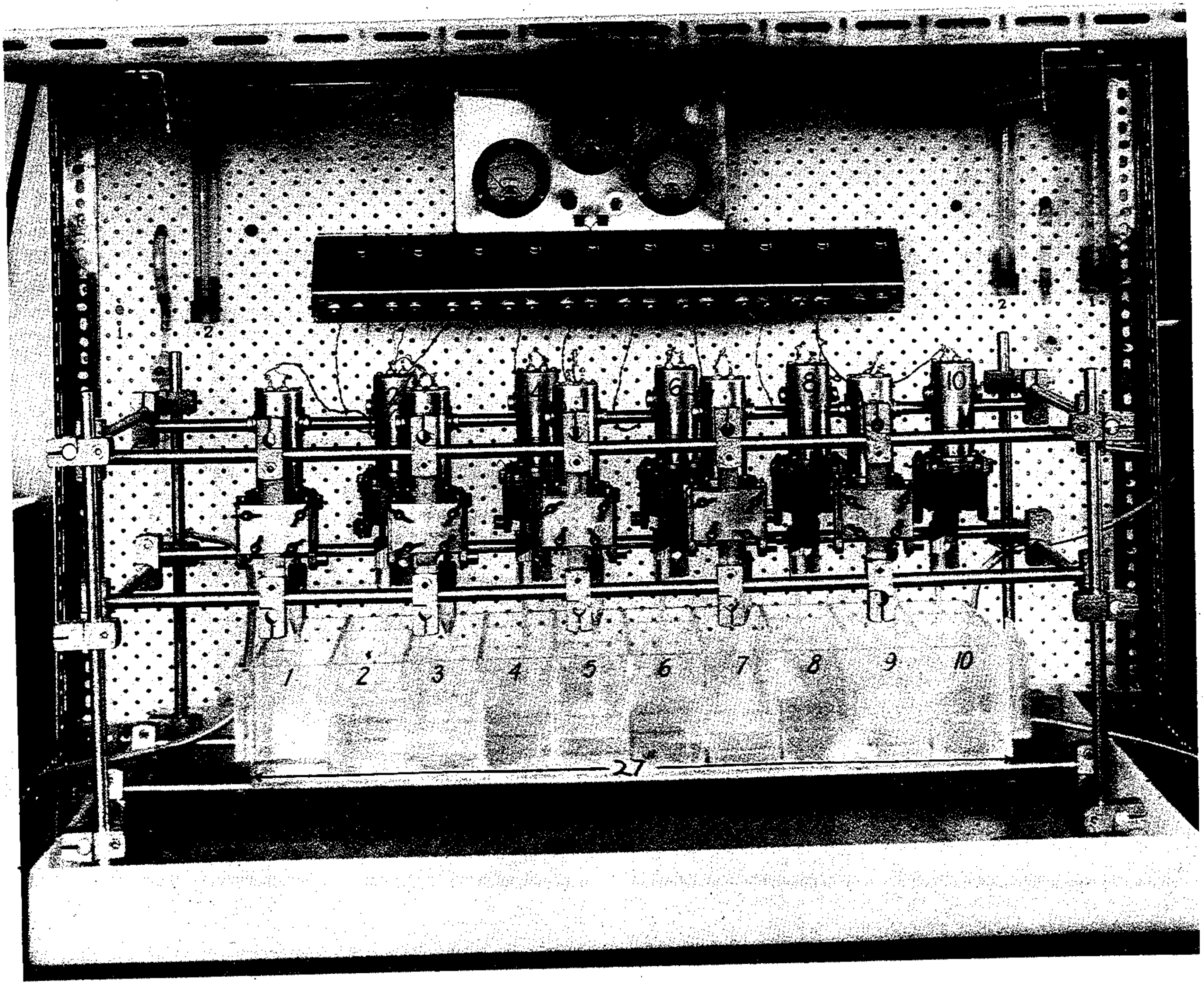


FIGURE 5 General View of Ten Stage Mixer-Settler Bank.