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PROGRESS OF LIQUID WASTE DISPOSAL STUDIES

Report written by:

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Progress of Liquid Waste Disposal Studies

Introduction:

Methods of liquid waste disposal now in use at AEC installations were developed to minimize the dangers involved during the formative years of atomic research. Organizations concerned with the research and development program have been acutely aware of the short-comings of waste disposal methods. Until recently, however, other major problems have limited the time available for studies on more satisfactory methods.

The Committee on Liquid Waste Disposal recently presented an over-all program for exploring all known or possible methods of liquid waste disposal at AEC installations. It was recommended that each installation study the aspects related to their problem. The immediate local problem consists of developing a method or process to reduce the concentration of plutonium in waste liquids far enough below the tolerance level to permit discharge without creating surface or sub-surface contamination.

In April, 1948, a cooperative research program was initiated involving the following agencies who formulated the program now in progress:

United States Public Health Service

University of California

A.E.C., Office of Santa Fe Operations

Laboratory studies were started in October, 1948, on the possibilities of utilizing biological processes (activated

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sludges) for concentrating and removing plutonium from liquid wastes.

Summary of Progress:

1. The tolerance limit of plutonium in liquid wastes has been described as 0.01 microgram per liter. By the use of activated sludges it is hoped to remove plutonium from the liquid so the resulting effluent will contain less than 0.001 microgram per liter. Results of radio-analyses in this report are stated in alpha counts per minute in which one microgram of plutonium is equivalent to about 70,000 alpha counts per minute or 140,000 disintegrations per minute. The "spikes" referred to in this paper consist of plutonium dissolved in acid solution.

2. A series of two exploratory experiments were performed in October and November 1948, to determine if activated sludge would effect any removal of plutonium from liquids. In the first experiment seven liters of mixed liquor was spiked with 553,000 counts (7.9 micrograms) of the material. After an aeration period of 20 hours analyses of the supernatant showed a reduction in plutonium content of approximately 95%. In the second experiment five liters of settled raw sewage spiked with 97,800 counts per minute per liter (total of 489,000 counts per minute), were added to three liters of activated sludge. At the end of 2 hours and 23 hours aeration periods the supernatants were examined for plutonium. This process was repeated daily for 5 days until the total addition of plutonium was 2,445,000

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counts per minute (about 35 micrograms). These analyses substantiated the results of the first exploratory experiment, giving 96% removal after 2 hours aeration and 99% after 23 hours.

3. The exploratory investigations showed that the procedure described by Langham (MDDC-1555 USAEC) used for the determination of plutonium in human urine was not directly applicable to the determination of plutonium in activated sludge effluents. A subsequent study of analytical methods resulted in a modification of the above procedure for application to activated sludge effluents. Forty-three replicate samples were analyzed using the modification. A statistical analysis of the data indicated about 85% recovery of plutonium in the samples with a probable error of plus or minus ~~3.31~~ 3.58 per cent. Appendix A of this report contains the modified procedure with supporting statistical data.

4. A second experiment was run using the modified analytical procedure. This experiment consisted of spiking 5 liters of sewage with about 500,000 counts per minute of plutonium and adding the spiked sewage to 3 liters of activated sludge. Sanitary characteristics of spiked sewage averaged as follows:

Biochemical Oxygen Demand(BOD)	205 ppm
Suspended Solids	135 ppm
pH	8.0

Samples of supernatant were analyzed after 3 hours and 23 hours aeration. This process continued for 11 cycles until the total

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amount of plutonium added was 5,719,000 counts per minute. In some cases the 3 hour sample was missed. During holidays and week-ends sewage feed and plutonium were not added. During the experiment there was no attempt made to control any factors pertinent to activated sludges except aeration period. Results of radio-analyses and partial sanitary determinations show the following pattern:

a. Efficiency of plutonium removal increased after the 6th spike. After the 9th spike the mixture was aerated for 48 hours showing a further increase in removal efficiency. Aeration continued for 72 hours after the 10th spike which again showed higher removal efficiency.

No. of spikes	Counts per minute per liter remaining in filtered supernatant 3 hrs.aeration	23 hrs.aeration	48 hrs.aeration	72 hrs.aeration
1	2100	435		
2	2400	530		
3	5800	2150		
4		2000		
5		3300		
6		6000		
7	10800	5600		
8	10600	4950		
9			3000	
10			2250	1740
11	6300	2100		

See Fig. 1.

b. Results of analyses of unfiltered supernatant samples showed the effect of suspended solids carry-over. These analyses, made on the 3 hour

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samples, showed from 5 ppm to 10 ppm suspended solids. The concentration of plutonium remaining in the unfiltered effluent varied from 2% to 28% higher than in the filtered effluent.

c. pH showed no definite pattern on removal efficiency although this factor should be investigated.

d. Concentration of solids in the mixed liquor varied from 1500 ppm to 2500 ppm with an average of 2180 ppm.

e. Longer aeration periods with no supplementary food indicated higher removal efficiencies.

f. Average BOD removal in the supernatant was as follows:

3 hour samples 73%

23 hour samples 88%

It is indicative from this experiment that careful and complete sanitary analyses should be carried on concurrently with the radio-analytical work. Control should be exercised over the variable sanitary factors in an effort to determine the type and characteristics of sludge, quality and quantity of food, volume of air, pH, etc., best suited for removal of plutonium from liquid wastes. Part of this data can be obtained from laboratory bottle experiments and part from pilot plant operation.

5. The previous experiments indicated the need for at least two stages of biological treatment. An experiment is in

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progress using spikes of 12,000 counts per minute per liter as the anticipated concentration of plutonium in the second stage. Partial data show 95% removal or about 600 counts per minute per liter of plutonium remaining in the 24 hour unfiltered supernatant.

Future Plans:

1. During the week of November 29th, Dr. C. C. Ruchhoft of U.S.P.H.S. and Mr. G. R. Scott of Black and Veatch, Consulting Engineers, conferred with personnel of the local project. An analysis of data available at that time indicated the following plan should be carried out for the immediate future:

a. Continue studies on the second stage with the 12,000 counts per minute per liter dosage.

b. Run a third set of experiments using spikes comparable to first, second and possibly third stages of treatment. Analyze the effluents after 6 hour and 24 hour periods of aeration including more complete sanitary analyses.

c. Initiate a series of experiments using actual plant wastes.

d. Study the chemical characteristics of plant wastes.

2. Design of a pilot plant was discussed with Mr. Scott. It was decided that the design should accomodate the entire Tech Area plant flow. In addition, plans were discussed for providing ample laboratory space to carry out all analytical work in connection with these studies, pilot plant control, and water treatment

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studies at the site of the pilot plant. March 1, 1949 was set as the desirable completion date for the pilot plant. The attached flow sheet for the pilot plant was prepared by Dr. Ruchhoft. (Appendix B).

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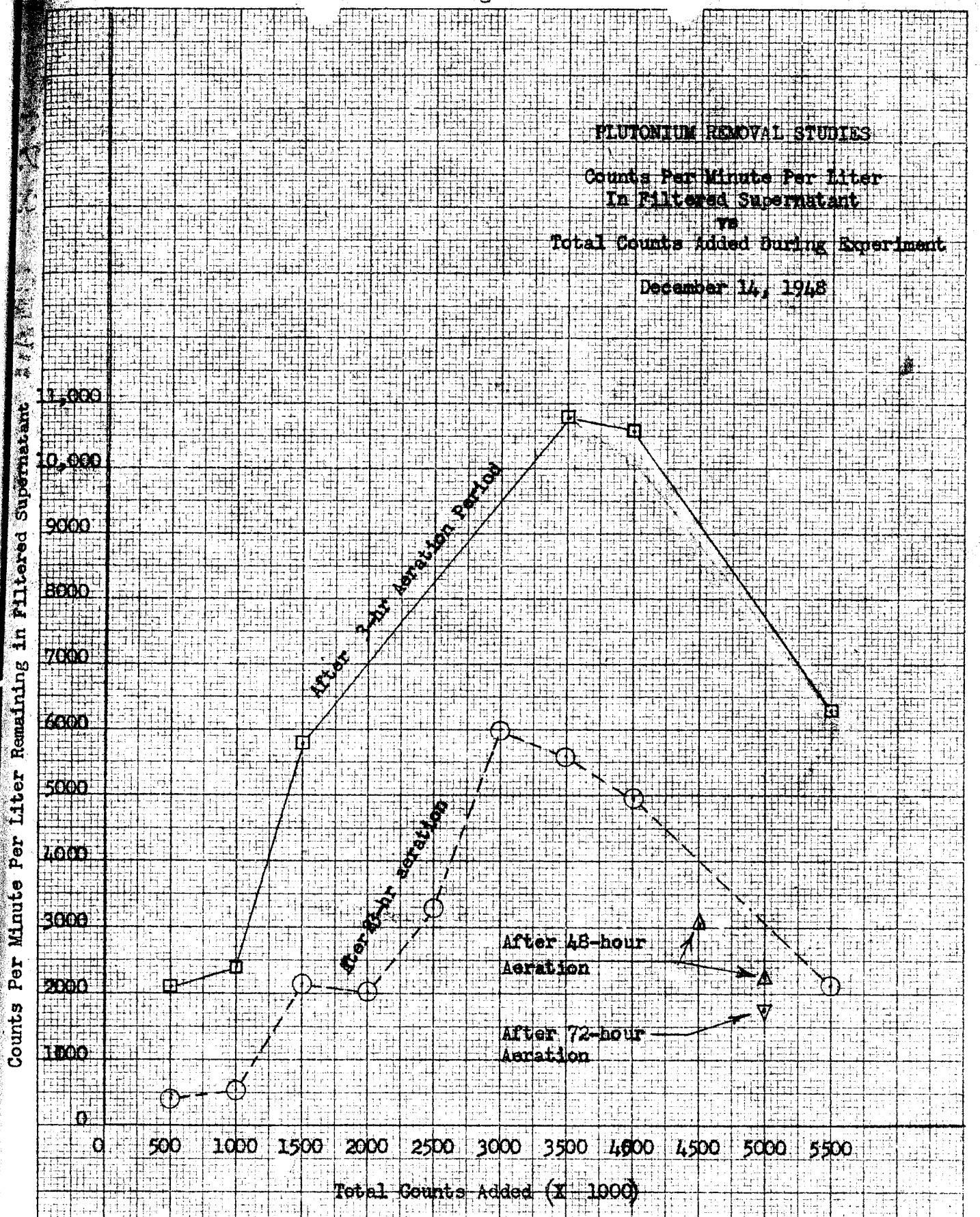
Figure 1



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Appendix A

Determination of Plutonium in Activated Sludge Effluent

The procedure to be described is the result of studies made to adapt the procedure described by Langham[1] to the determination of plutonium in activated sludge effluents. The procedure finally developed requires less work per determination, and should be less subject to interference by ordinary mineral constituents than the parent procedure. Also, the recovery of plutonium obtained appears to be slightly higher than that obtained by Langham and his co-workers.

Procedure for the Determination of Plutonium in Activated Sludge Effluent and Other Wastes:

Reagents:

Hydrochloric Acid, concentrated.

Hydrochloric Acid, 10%

Sulphuric Acid, 20%

Methyl Violet Indicator (saturated aqueous solution).

Ferric Chloride Solution, 1 mg of iron per ml

Cupferron - 6% aqueous solution, not over 5 days old and kept in refrigerator.

Chloroform - C.P.

Lanthanum Nitrate 0.41 g/100 cc in 0.1 normal hydrochloric acid.

Perchloric Acid - 70%, C.P.

Nitric Acid - concentrated C.P.

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Hydroxylamine Hydrochloride - 20% in distilled water.

Hydrofluoric Acid - concentrated.

Hydrofluoric Acid - 0.1 normal.

Ammonia - 50%, C.P.

Apparatus:

Infra-red lamps and appropriate supports and explosion shields.

Electrically heated oil bath, with variable transformer control.

Centrifuge.

Alpha counter.

Procedure:

Acidify a representative sample of a volume of 1 liter or less with a few ml. of concentrated hydrochloric acid. Concentrate the solution to a volume of 100 ml. or less by evaporation on an electric hot plate. If the sample chosen has a volume of 100 ml. or less, the addition of the hydrochloric acid and the concentration may be omitted. After the sample is reduced to a volume of approximately 100 ml., transfer quantitatively to a porcelain casserole (Coor's 3A is satisfactory). Rinse the flask used for concentration with several small portions of boiling 10% hydrochloric acid, and add the rinsings to the sample in the casserole. Add 1 ml. of 20% sulphuric acid solution and an additional ml. of the same solution for each 200 ml. of original sample volume. (These quantities have been found to work well in the case of activated sludge effluent.)

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If the sample does not contain substantial amounts of carbonaceous material, add 1 ml. of 10% glucose solution.

Using an infra-red lamp, evaporate the sample until dense fumes of sulfuric acid appear. If the sulfuric acid fumes do not appear, moisten the dry sample with 20% sulphuric acid solution and carry down to fumes. Continue to heat the sample under the lamp until the evolution of fumes is substantially complete and the sample no longer shows the presence of liquid sulphuric acid.

Transfer the casserole to a muffle furnace with an automatic temperature control. The muffle should be located in a hood. Heat the sample at 550°C until the carbon is completely burned off. (Usually, two hours is adequate.) After the carbon is burned off, remove the casserole from the muffle and allow it to cool. With a pipette, add 12-15 ml. of concentrated hydrochloric acid to the casserole, carefully moistening all the material on the sides of the casserole by allowing the hydrochloric acid to drip down the sides. Place the casserole under an infra-red lamp and evaporate the hydrochloric acid in the casserole almost to dryness. Wash the casserole four times with 10% hydrochloric acid, bringing the hydrochloric acid solution almost to a boil in the casserole swirling by hand over an open flame. Scrub the casserole thoroughly with a stirring rod and policeman after the first washing and leaching with the 10% hydrochloric acid. Combine the washings in a 50 ml. pyrex

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centrifuge tube.

Centrifuge for 15 minutes at 2000 rpm. Carefully decant the supernatant into a 125 ml. globe shaped separatory funnel. Add approximately 5 ml. of 10% hydrochloric acid to the residue in the tube, swirl gently to break up the precipitate. Avoid getting precipitate up above liquid level on the sides of the tube. Centrifuge the acid in the tube at 2000 rpm for 15 minutes and again decant the supernatant into the separatory funnel containing the liquid first taken off.

Add 1 drop of methyl violet. Add 50% ammonia until the liquid is a bright emerald green. The analyst should familiarize himself with the indicator colors at known pH values. The pH desired is 0.4 to 0.6. After the pH is adjusted, add 1 ml. of ferric chloride solution unless the sample already contains at least 1 mg. of iron. Add 2 ml. of cupferron solution and mix well.

Extract with 2.5 ml. of chloroform and collect the chloroform in a 50 ml. centrifuge tube. Separate the chloroform and add 0.5 ml. of cupferron solution and mix well. Extract with 2 ml. of chloroform. Add 0.5 ml. of cupferron, mix the cupferron and then extract with 2 ml. of chloroform. Add 0.5 ml. of cupferron. Make five serial extractions with 2 ml. of chloroform.

To tabulate this procedure:

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Extraction	ml. Cupferron Added before Extraction	ml. of Chloroform used
1	2	2.5
2	0.5	2
3	0.5	2
4	0.5	2
5	0	2
6	0	2
7	0	2
8	0	2

Place the combined extract in an oil bath at 65°C to evaporate the chloroform. (Caution: Heating above 65°C at this point will cause excessively rapid boiling with possible loss of material.) After the chloroform is nearly all gone, increase the temperature of the bath to 70° C and hold there for 15 minutes.

Remove the centrifuge tube from the bath and allow it to cool. Cautiously add 2 ml. of concentrated nitric acid and then 1 ml. of 70% perchloric acid. Place the centrifuge tube into a melted wax bath below 90°C. Slowly bring the temperature of the bath up to 190°C over a period of at least one hour. At the end of this period, the residue should be a pale straw color. If the residue is not a pale straw color, continue heating at 190°C until a pale straw colored residue is obtained.

Allow the digestate to cool. The cooled digestate should be colorless. Add 3 ml. of distilled water and 2 drops of hydroxylamine solution. Allow to stand for at least 2 hours. Add 1 ml. of concentrated hydrofluoric acid and 2 drops of lanthanum nitrate solution. Do not add more lanthanum nitrate because of subsequent plating difficulties. Mix thoroughly by swirling.

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Centrifuge for 15 minutes at 2000 rpm. Decant the supernatant and invert the tube on a paper towel to drain. Wash precipitate with 2 ml. of 0.1 normal hydrofluoric. Swirl the tube to break up the precipitate. Centrifuge at 2000 rpm for 15 minutes. Decant the supernatant and invert the tube on a paper towel to drain.

Transfer the precipitate in the tube quantitatively to a stainless steel plate. This is accomplished by slurrying the precipitate in about 0.1 ml. of distilled water and transferring it to the plate. Make a second, third, and fourth, slurrying with approximately 0.1 ml. of distilled water. Dry the slurry on the plate by placing it under an infra-red lamp. To avoid unduly rapid drying do not place the infra-lamp too close to the plate. Continue to apply heat for several minutes after the plate is dry.

Count the plate for a suitable period in a low background alpha counter. Due to incomplete (about 86%) recovery of plutonium it is necessary to multiply the observed results in counts per minute by 1.16 to obtain the count present. Report as counts per minute per liter.

Probable Error of Analysis for Effluent Samples of

100 - 1000 ml.

$$\% \text{ P.E.} = \sqrt{11.0 + \text{P.E.}_c^2}$$

where P.E_c is the per cent probable error of counting.

The procedure outlined has been studied with plutonium only present. The investigators have no knowledge of the

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efficiency of the method in separating plutonium from other alpha emitters.

Over a hundred determinations of plutonium with various procedures were attempted, using effluents to which a known "spike" was added, before an effective procedure was found. A number of different possible procedures were investigated in the course of the search for a satisfactory procedure. As is common practice, no presentation will be made of the data or procedures which did not prove to be of value.

The study of the procedure developed has been incomplete. The work was undertaken to develop a functional tool, and there has been no attempt to make a thorough evaluation of the factors and variables involved in the procedure presented.

Some further comments on the nature of the material analyzed are in order. The effluent from an activated sludge process will contain variable quantities of calcium, magnesium, sodium, potassium, chloride, sulfate, phosphate, silica, iron, aluminum, ammonia, nitrate, and organic matter. The organic matter may vary from whole micro-organisms to organic matter in true solution. Hence, it may be seen that activated sludge effluent is a complex material which offers a variety of materials which might interfere with a plutonium assay.

In Table 1 are given the results obtained using the procedure described on some spiked samples of effluent. In all cases, the spike added was the same (284.2 $\pm$ 1.6 counts). This

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spike was put into three different activated sludge effluents. Further, the amount of effluent to which the spike was added was varied from 100 to 1000 ml. in the case of a particular effluent.

Table 2 lists a group of summary values calculated from Table 1. Study of Tables 1 and 2 indicates that there was no significant variation in the recovery of plutonium within a given series, comparing one series of observations with another or the grouped values at a given sample size level. Fig. 3 gives an error distribution curve for the combined data. The distribution of variation, as shown by Fig. 1 is a normal error curve. Accordingly, statistical parameters calculated or derived from the data may be assumed to conform to the normal distribution pattern.

Dependency of Precision Upon the Number of Counts Observed

The precision of any procedure based on the counting of radio-active disintegration is contingent upon two sets of errors, the error of counting and the error of manipulation. While it may not be a completely valid assumption, it will be assumed that the manipulative error or loss and consequent recovery of plutonium is independent of the concentration of plutonium in the sample analyzed. Subject to this assumption, the probable error of analysis may be regarded as the resultant of the error of manipulation and that of counting. Since there obviously is no correlation between manipulative error and counting error, resolution may be obtained using the formula:

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$$\% \text{ P.E}_a = \sqrt{\% \text{ P.E.}_c^2 + \% \text{ P.E.}_m^2} \qquad (1)$$

where

$\% \text{ P.E}_a$ = Percent probable error of analysis

$\% \text{ P.E}_c$ = Percent probable error of counting

$\% \text{ P.E}_m$ = Percent probable error of manipulation

Table 2 shows that the probable error of analysis was found to be 3.58%. The counting period was ten minutes with the average number of counts observed 2452 over the ten minutes of counting. Starting with the formula: $\sigma = \sqrt{N}$ where σ is the standard deviation and N is the number of counts observed, it may be shown that:

$$\% \text{ P.E}_c = \frac{67.45}{\sqrt{C_m T}} \qquad (2)$$

where C_m is the observed counts per minute and T is the counting period in minutes.

The total number of counts observed consists of the counts caused by the sample examined and the background count. The statistical variation of the background count is a negligible part of the statistical variation at the activity levels where it is intended to apply this procedure. For a total of 50 or more counts per minute it is quite satisfactory to neglect the statistical variation of a background count of one count per minute or less after subtractive corrections have been made for the background count. Hence,

$$\% \text{ P.E}_c = \frac{67.45}{\sqrt{2452}} = 1.36 \%$$

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Using formula (1), the probable error of manipulation is found to be 3.31%.

Subject to the assumptions already made:

$$\% \, P.E_a = \sqrt{\% \, P.E_m^2 + \% \, P.E_c^2} \qquad (3)$$

$$= \sqrt{11 + \% \, P.E_c^2}$$

It is obvious that as the number of counts observed increases, that the error of ~~counting~~ analysis will approach 3.31%. Also, at low count levels, the assumptions made cease to be valid. Fig. 2 presents graphical solution of the formula (3) for several counting periods and may be used to estimate the analytical error of the procedure.

As already pointed out, this is admittedly an incomplete investigation of the factors involved in an analytical technique. There has been no evaluation of the effect of variable plutonium content in the total sample examined on the results obtained. The accuracy of the method has not been investigated. It is also possible that a few counts of plutonium may have been present in the effluents used, and this possibility has not been covered by blank determinations. Details of the procedure have not been individually evaluated, so that there may be some unnecessary steps involved. However, this work was undertaken solely to develop a satisfactory measuring procedure for a particular series of investigations, and not primarily as an analytical research study. Because errors considerably larger than those noted are acceptable for the purpose for which the

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method was intended, no further study of the method is contemplated at present.

Summary and Conclusions

1. An analytical procedure has been presented for the determinatio of plutonium in activated sludge effluents.

2. Statistical evaluation of the results obtained by the procedure outlined are presented.

3. Auxilliary formulae and graphs are given for the estimation of the errors involved in the use of the procedure outlined.

Bibliography

(1) Langham, Wright H., MDDC-1555, LADC-U.S.A.E.C., Technical Information Division, Oak Ridge Directed Operations, Oak Ridge, Tenn., "Determination of Plutonium in Human Urine".

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Table #1

Recovery of plutonium from activated sludge effluents spiked with 284.2 ± 1.6 counts per minute per sample. (10 minute counting period)

Series	ml. of Effluent Spiked 100	250	500	1000
A	244 c/m	250 c/m	249 c/m	Lost[C] c/m
A	248	247	256	234
A	239	237	240	232
A	265	Lost[C]	248	Lost[B]
B	255	240	250	244
B	238	230	259	254
B	230	228	260	247
B	238	255	251	Lost[B]
C	264	236	224	245
C	281	242	227	244
C	263	234	262	247
C	Lost[A]	258	211	236

Lost[A] - Lost due to Infra-red lamp which failed. (Fragment of lamp fell into sample causing mechanical loss.)

Lost[B] - Lost due to leaky extraction flask.

Lost[C] - Two samples were taken out of the data because they differed from the mean by more than 4 times the standard error. Of these samples, one was too high, one too low.

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Summary of Values Computed from Table #1.

Data Computed from	No. of observations	Mean	σ	σ_m	% P.E.
Series A	13	245.3c/m	9.1		
Series B	15	245.3	10.8		
Series C	15	244.9	17.8		
100 ml. Samples	11	251.4	15.5	4.7	
250 ml. Samples	11	240.9	9.6	2.9	
500 ml. Samples	12	244.8	15.5	4.5	
1000 ml. Samples	9	242.2	7.0	2.3	
All Samples Grouped.	43	245.2	13.0		3.58

To convert observed counts to reported counts multiply observed counts by 1.16.

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FIG. #2

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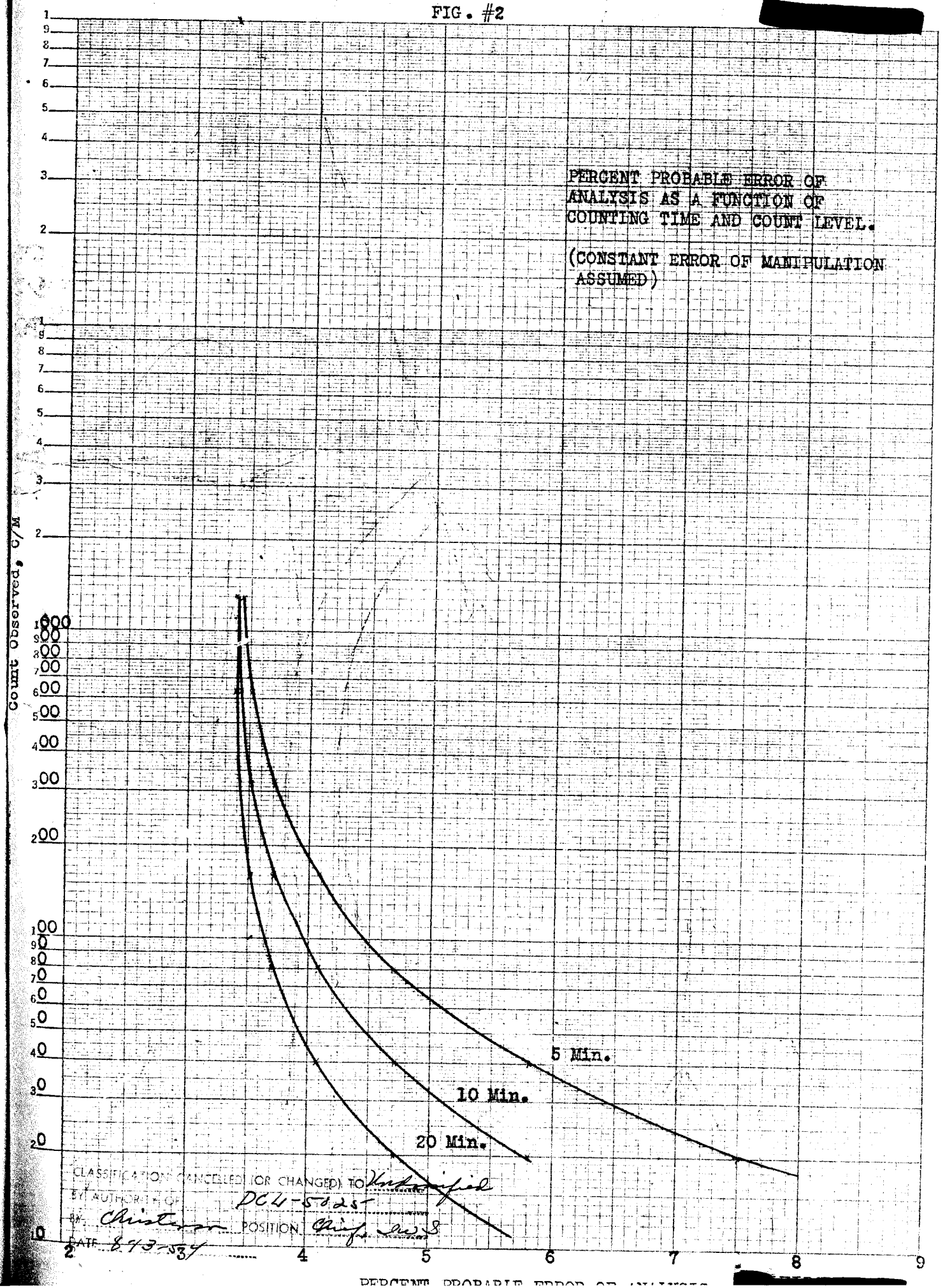
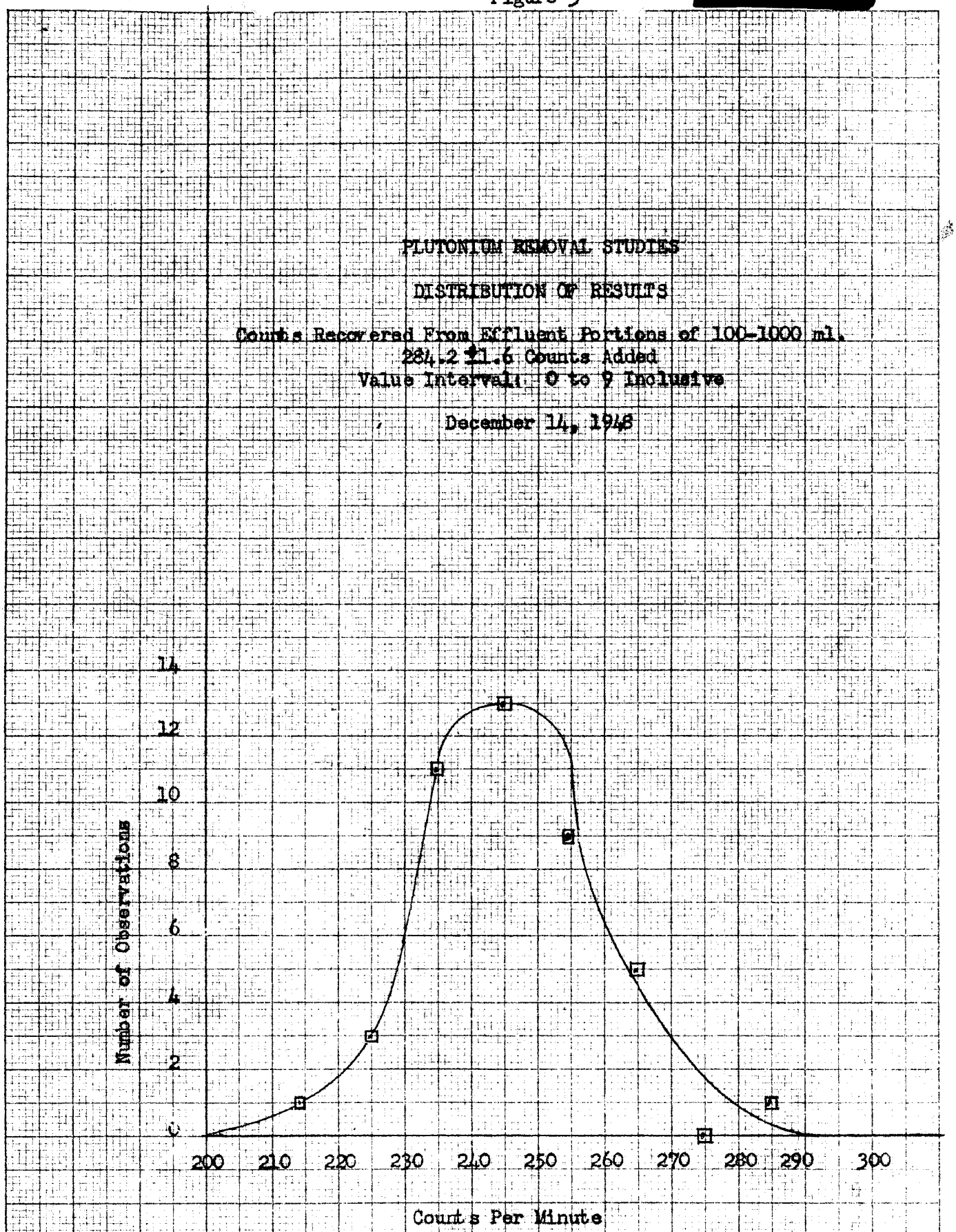


[redacted]

Figure 3 [redacted]



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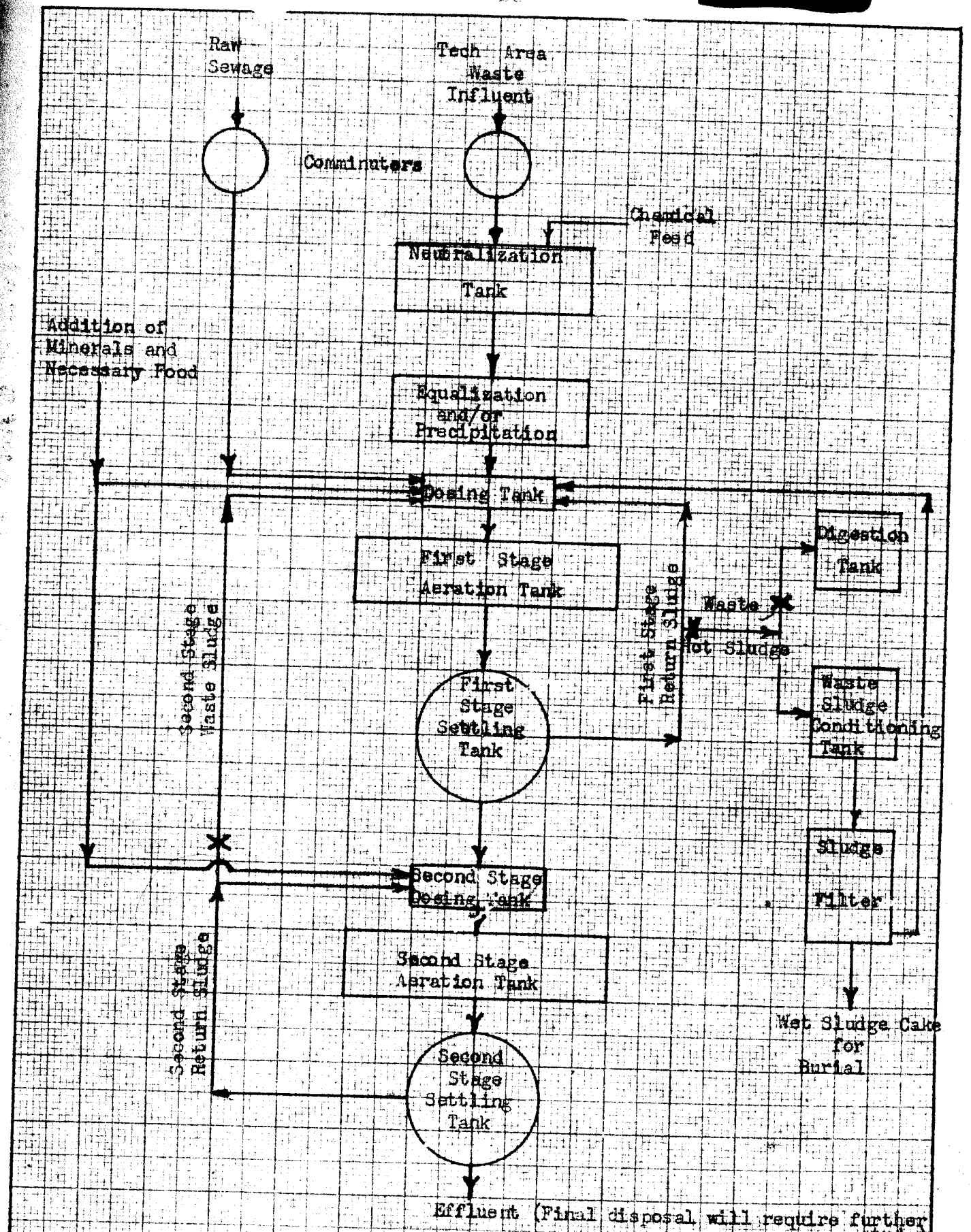

FLOW DIAGRAM
BIOLOGICAL PLUTONIUM REMOVAL PLANT
AEC Santa Fe Directed Operations-12/9/48

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APPENDIX-B

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