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RADIOASSAY OF PLUTONIUM

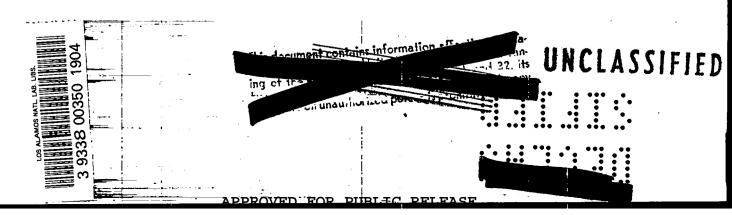
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ABSTRACT

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The development of the present radioassay method is given and the present procedure is described. Because of the use of improper values of the specific alpha activity, the reported radioassays on essentially pure plutonium solutions have been in error by as much as 6% on the average. However, when these radioassays are corrected for the isotopic composition of the plutonium, their probable error is found to be 1.7%. Radioassays on solutions simulating the P-1 and P-3 cuts usually submitted from the "B" purification process¹) and on a basic solution simulating those that are usually disposed of in the waste storage tanks indicated that probable errors of about 14.7%, 2.4%, and 19.1%, respectively, could be expected on these solutions. Production data, indicating the use made of the radioassay method by various groups, are given.



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RADIOASSAY OF PLUTONIUM

INTRODUC ?ION

UNCLASSIFIED Plutonium is an extremely expensive and hazardous meterial these facts, coupled with the desirability of knowing how much plutonium is where on a purely operational basis, made desirable a rapid micro method of estimating plutonium concentration in various solutions. Plutonium's property of decomposing by alpha particle emission at the rate of ~ 140,000 disintegrations per minute per microgram seemed to be the most likely one on which to base such a method of estimation. A method based on this property should be rapid, since the plutonium should not have to be separated from other constituents of the solutions. Also, because of the magnitude of the specific activity, the operations would have to be done almost by necessity on a micro basis. Consequently, the procedure for estimating plutonium concentration in various solutions by determining the alpha activity of small aliquots was developed, and the name radioassay was given to the process. The procedure consists of taking aliquots (which are usually diluted and re-aliquoted) of the solution to be assayed and evaporating them onto microscope cover glasses which are placed in a chamber of a linear amplifier and counted.

Practically the entire development of the radioassay method has been centered about the facilitating of the procedure and the minimizing of the errors connected with it. The errors that are encountered are: the error introduced by the statistical fluctuation of the rate of alpha particle emission by a small amount of plutonium; the self-absorption of alphas by the plutonium and the absorption of alphas by the other constituents of the sample; the error in the coincidence correction to be applied to an observed number of counts per minute due to the occurrence of two or more .#



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theoretically countable disintegrations in a time interval too small for the linear amplifier to respond to each alpha produced; counting errors introduced by the imperfect functioning of the linear amplifiers; the error in the specific alpha activity of the plutonium; and the errors introduced by the aliquoting - errors in pipet and flask calibrations and in the actual performance of the aliquoting operations, and errors introduced by contaminated glassware and reagents. The treatment of these errors is discussed in the next several sections.

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DEVELOPMENT OF THE METHOD

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Dilution: As stated in the introduction, safety, economy, and convenience considerations made desirable a micro method for the estimation of plutonium, and, by the very nature of the property on which it depends, the radioassay of plutonium is a micro method. Because of the magnitude of its specific activity, the amount of plutonium actually alpha-counted in a radioassay rarely exceeds half a microgram, more frequently being of the order of a fiftieth of a microgram.

Originally, the problem of putting approximately this amount of plutonium into the linear amplifiers from solutions of various concentrations was handled by using pipets varying in size from about $10 \rightarrow to 200 \rightarrow 0$. The procedure at this time consisted of mounting an aliquot of the sample directly onto a small piece of sheet platinum, which was heated to a lightorange color in a Fischer burner to remove as many of the other constituents of the solution as were volatile at this temperature. Occasionally it was found that the plutonium concentration was too great for even the smallest aliquots to be counted in the linear amplifiers; also, on occasions, large amounts of salts were left on the platinum counting slips even after the ignitions. Therefore, it became evident that the initial aliquots must in many cases be diluted, aliquots of the dilute solution then being evaporated onto the platinum slips for counting. -6-

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At about this time the job of decontaminating the platinum slips was becoming overburdening because of the increasing output of radioassays. Consequently, it was desirable to switch from platinum to glass slips, which could be discarded after one usage. The use of glass would prohibit the use of an ignition to remove fairly volatile salts from the slip before the counting took place, and so the amount of salts present on the slip would be a more constant problem. Also, the use of glass would undoubtedly lower the specific activity (in recorded counts per minute per microgram) because of the difference between the scattering powers of platinum and glass. These considerations made necessary some research to investigate the specific activity on glass slips and to determine the magnitude of the absorption of alphas by salts on the slip. There was not time to get enough data to be more than merely indicative of the effect of salts. Up to approximately 300 micrograms of salts on the counting slip, the plot of the absorption of the alphas closely followed the curve

$$A = 1.5\gamma^{1/2},$$

where A is the percentage absorption, and γ is the amount of salts in micrograms on the slip.

A systematic plan for diluting the solutions to be assayed was needed as a time-saving device, if nothing else. The plan should take into account the statistical nature of the counting and the absorption error introduced by other salts on the slip. Also, a method for determining the compatibility of the counting results on the two aliquots taken for each assay was desirable, and so the following was done. The upper limit of the compatible deviation of the two results was set as being equal to the sum of twice the standard deviation of the counting results and the percentage absorption of



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alphas as found in equation 1. A ten-minute counting interval was assumed, and so, expressing the maximum percentage compatible deviation as D, the equation

$$D = 1.5 \sqrt{\gamma} + 2 \cdot 100 / \sqrt{10r}$$
 (2)

was obtained, where r is the counting rate. Defining the dilution factor, f, as the size of the dilution made divided by the size of the aliquot mounted on the counting slip, denoting the salt concentration of the sample by s, the plutonium concentration by p, and the specific activity by a, and making the appropriate substitutions, 2 becomes

$$D = 1.5 s f + 0.241 p f . (3)$$

Minimizing D with respect to f, one finds that the dilution factor yielding the smallest maximum compatible deviation is given by

$$f = 6.2 \ s^{1/2} p^{1/2} \qquad (4)$$

Obviously, it is desirable that the deviation between the two aliquots counted in a radioassay should be as small as possible, and so the dilutions are made according to $\underline{4}$ on the basis of the estimated concentrations of the plutonium and other salts in the sample. In order to have a systematic method of making the desired dilutions, it was decided to use 30 λ pipets throughout and to vary the flask size appropriately. The accompanying dilution chart, Fig. 1, on which the means of obtaining the desired dilution is diagrammatically shown, was made and used. A limitation on the dilutions was imposed, however. When the amount of salts on the counting slip exceeded 100 γ , they tended to become detached from the slip and to fall off in the linear amplifier chambers, thereby contaminating the latter. Consequently, regardless of wirst the chart ...

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indicates, a sample is always diluted in such a manner that there are less than 100γ of salts on the slip. In connection with using the dilution chart, the practive of never estimating the total self concentration as being less than twice the plutonium concentration has been established. Fig. 2 shows the absorption error allowed as a function of the salts on the counting slip. To reduce the time required for the calculation of the rosults of the assays, charts were made showing the dilution factors obtained for the various combinations of pipets and flasks.

Pipets, counting slips, and flasks: In the beginning when no flasks were used, the pipets ranged in size from about 10 to 200 λ . They were made by drawing out ordinary glass tubing and marking the capillary at the desired height with a diamond pointed stylus. The pipets were filled and discharged by inserting into the large end a cork attached to thin rubber tubing which was held in the mouth, the necessary changes in air pressure being made by the resolutory system. The pipets were calibrated in the manner that is still in use by filling them with mercury and weighing them on an Ainsworth BCT (analytical chainomatic) balance. The tips of these pipets were not fire polished due to the thinness of the capillary walls.

At the time that the dilution system was established, it was decided to use pipets all the same size and for safety reasons to use a syringe instead of the mouth to fill and empty them. Misoo pipets were tried but discarded, because they would not reach far enough into the volumetric flasks. Then it was decided to draw~30A pipets out of commercially made blanks as shown in Fig. 3. The thicker walls of the capillary permitted fire polishing the tip and increased the life expectancy of the pipet. The

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The fire-polished tip was more resistant to chipping, and, furthermore, one could tell at a glance if the tip had been chipped. The means of operating the pipet was provided by inserting into its large end a cork, through which was an end of a piece of fine rubber tubing which was fastened to a B-D Tuberculin Syringe by means of a Vim Hypodermic Needle that was blunted to prevent cutting the rubber tubing. The outside of the tip end of the pipet was coated with Uniwax so that the aqueous solutions used would not cling there.

In use the pipet and its syringe are held in one hand and the container from which the pipet is filled is held in the other. A hand lens mounted on a ring stand is used by some operators to aid them in placing the meniscus at the mark. The pipet is discharged into a flask for dilution (or onto a counting slip) and is rinsed out by removing the cork from the pipet, putting three or four drops of $5N \ \text{RNO}_3$ into it, replacing the cork, and blowing the acid through the pipet into the same flask. In between uses the pipet is cleaned and dried by drawing through it in a vacuum type pipet washer concentrated nitric acid, water, apetone, and air.

The counting slips originally were small sheets of platinum about an inch square. On these the aliquots to be counted were mounted and ignited. In between usages the platinum slips were decontaminated. They were first rinsed in water, then immersed in hot concentrated sulfuric acid and then in hot concentrated nitric acid. This treatment did not always decontaminate the slips enough to permit their reuse, and so potassium pyrosulfate fusions were tried with not much more success. A switch from platinum to glass (with a corresponding modification of procedure as described above) seemed to be in order, and so, after some trials and discussion, number 2 microscope cover

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glasses were adopted for counting slips. Prior to their use the cover glasses are cleaned in hot nitric acid, distilled water and acctone, and are dried on sheets of Kleenex. Then they are put on a piece of Kleenex in Regent Cigarette boxes, where they are kept, when not in use, until after they have been counted.

When the dilution system was established, it was decided to make use of 1-, 2-, 5-, 10-, 25-, 50-, and 100- ml volumetric flasks. No calibrated flasks under 25 ml could be obtained, and so the 1-, 2-, 5-, and 10- ml flasks were calibrated by the radioassay group before being used. Up until very recently the flasks were calibrated by filling them with water from a calibrated drain-out pipet. Recently, mercury has been employed in the calibration. A volumetric of the size in question was calibrated by filling it with water and weighing it on an Ainsworth BCT balance. Then the volumetric was filled with mercury and the mercury was poured into the other flasks. Occasionally it was poured back into the water calibrated flask to make certain that its volume had not changed through spilling or thermal expansion.

After each assay the volumetrics were given to the recovery group for emptying. Their cleaning was done by the radioassay group. At first, they were given a water rinse, allowed to stand for half an hour with cleaning solution, given two rinses with cleaning solution, then several rinses with tap water, and were finally rinsed out with distilled water. To verify the decontamination of the flashs, several drops of the distilled water that remained in each flask when its contents were poured out were drained from it onto a microscope cover glass, which, when dry, was counted for two one minute intervals. If more than eight counts were recorded during either minute on a 1-, 2-, 5-, or 10- ml flask, the flask was recleaned. Fifteen counts per

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minute were allowed for the larger sized flasks. The cleaning solution rinses were thought to be not as effective as desirable due to the rather large number of volumetrics that had to be recleaned. Consequently, various other procedures were tried until the present method was adopted. Now, after the plutonium solutions are poured out by the recovery group, the flasks are rinsed with tap water and then allowed to stand for twelve hours filled with concentrated nitric acid, which is emptied into glass carboys for subsequent use as a reagent by the recovery group. After the nitric acid bath, the flasks are rinsed out with water and allowed to stand for twelve hours filled with a saturated sodium citrate solution. The sodium citrate solution is poured down the sink, and the flasks are rinsed with distilled water and checked for contamination as described above.

Counting and the specific alpha activity: The alcha counters have always been air chamber and nitrogen chamber linear amplifiers. At first the air chamber amplifiers were extremely sensitive to sound and vibrations and the utmost caution was required to use them satisfactorily. Considerable improvement has been made in their design, however, and they may now be used with no more than a nominal amount of cars. Coincidence curves have been made to correct for the effect of two or more alphas: being discharged into the chamber in a time interval too short for the amplifier to resolve the individual pulses. The practice of limiting samples for the air chamber amplifiers to those of 3,000 counts per minute or less was established; the nitrogen chamber amplifiers are probably not too reliable at more than 100,000 counts per minute, but, in practice, this figure is not even approached. Counting slips with more than 400 counts per minute are counted for two four minute intervals. Counting slips with less than 400 counts per minute are counted for two eight minute intervals to obtain smaller counting errors. For each assay (two counting slips) a -12-

determination of the background (observed counting rate with nothing in the chamber) is made and a standard is counted to make certain that the linear amplifier is functioning properly. The standards are aliquots of a plutonium solution that have been dried on platinum slips, which have been evaluated by being counted for long periods of time on various amplifiers. A special building was constructed for the counters, as it was found that there were too many interferences to their satisfactory performance in the laboratory building. The details of the coincidence correction curves and of the amplifiers will be given in a fortheoming report by S. R. Chadwick.

When platinum counting slips were used, a specific activity of 71,000 counts per minute per microgram, determined at the Metallurgical Laboratory in Chicago, was used. When glass slips began to be employed, the specific activity was lowered to 63,500 counts per minute per microgram on the basic of the counting rates observed when the same size aliquote of Pu were counted on a couple of platinum and on a couple of glass slips. The impression was gained over a period of time that the specific activity was about 1% low, and so some experiments were performed.²⁾ the results of which caused the adoption of the value 69,400 counts per minute ner microgram. At about this time the Hanford oile went into operation and began to produce plutonium with increasing amounts of isotopes 238 and 240 with a consequent increase in the specific activity. However, until very recently no correction has been made for the isotopic composition of the plutonium and the value 69,400 mas consequently been in error by as much as 6% . At the present time the value 73,000 counts per minute per microgram has been adopted as the basic value of the specific activity, and Fig. 6 has been prepared so that those who so desire may correct the radioassay results according to the amount of

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irradiation their sample has received.

PROCEDURE

The sample to be assayed is submitted to the radioassay group in a small stoppered vial or in a small (-4, ml) test tube, which is supplied by the radioassay group for the purpose, along with a request sheet, Fig. 4, on which the blanks for estimated total solids concentration, estimated product concentration, sample number, date, and name of the person submitting the sample are filled in. The blanks for volume of sample and description of sample may be filled in at the submitter's discretion.

Making use of Fig. 1 the amount of dilution is determined and indicated on the request sheet. Two aliquots, taken with different pipets, are each discharged into volumetric flacks. The aliquots are diluted to the marks with 5N HNO3, unless some other dilutant is specified by the submitter, and an aliquot of each solution is placed on a counting slip. (If the estimated concentrations of the sample are appropriate, the first aliquot is mounted directly onto a counting slip. Also, it is sometimes necessary to redilute the second aliquots and to mount the third ones on the counting slips.) Before the aliquots are out on the slips, the latter are dusted off with soft brushes and are labeled with the assay number using ordinary fountain pen ink. The numbered sides of the slips are put down on hot plates kept at about 30° C. Then the aliquots are dried on the slips, and the slips are placed back in the Regent Cigarette boxes, which are labeled with the assay number. The volumes of the pipets used are indicated on the request sheet.

The counting slips are subsequently counted and the appropriate blanks on the request sheet are filled in. The calculations are checked on a calculating machine, and, making use of the chart showing the dilution factors -14-

for various combinations of pipets and flasks and of Fig. 2, the remainder of the blanks on the request sheet are filled in. The product concentration, estimated error, deviation, and other blanks of Fig. 5, the report sheet, are filled in and given to the submitter of the assay. If the results of the assay meet his approval, the request sheet is filed and the sample is sent to the recovery group. If the results do not meet his approval, the assay is repeated. The usual criterion for the acceptability of a radioassay is that the observed deviation be not appreciably greater than the sum of the counting error and the absorption error. Regardless of how small these errors may be, a deviation of 2% or less is considered acceptable. After each assay, the counting slips are disposed of in a storage bottle for that purpose, and the Kleenex in the Regent Cigarette boxes is changed.

ACCULACY

Essentially pure plutonium salt solutions: An investigation of forty radioassays (see Table I) run on essentially pure plutonium salt samples, which had received a known amount of irrediation and which had been assayed by the chemical titration method, indicated that the radioassays averaged 0.76% lower than the chemical the ohemical titrations, with a probable error of a radioassay of 1.7%. A preliminary investigation into the cause of this discrepancy indicates that the radioassay group's pipets may be miscalibrated about 0.3% too high on the average. If the pipets are this high on the average, on this particular set of assays the average radioassay would be 0.06% lower than the chemical titrations and the probable error of a radioassay would be 1.6%. Solutions with other constituents: Solutions simulating the P-1 and P-3 assays submitted by the "B" purification process operators and a basic solution

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simulating those that are disposed of in the waste storage tanks were made in such a manner that the alpha counting rate would be known if there were no other salts present. This experiment did not take the specific activity into account, but in order to express the plutonium concentration in grams per liter the value $69_{0}400 \text{ c/m/\gamma}$ was used. The P-1 samples were made by putting 650 g Ca(NO₃)₂ 4H 0, 1.66 g NaNO₃, 6.8 g NaBrO₃, 8.4 g H₂SO₄, 6.6 g HNO₃ into enough water to make a liter of solution. The P-1A assays contained about 0.01 g/1 of Pu. The P-3 samples were 1.25M ENO₃, 0.107M HT, 0.035M HI₃, and 0.078M H₂C₂O₄. The P-3A samples contained 0.1 g/1 Pu and the P-3B samples were about 0.02 g/1 in Pu. The basic, C, solutions were made by putting 170 g MaNO₃, 58 g MaC1, 40 g NaOd, 40 g Na₂Al₂O₄ and ~90 g K₂SO₄ into enough water to make a liter of solution. The CA solutions were about 0.001 g/1 in plutonium and the CB solutions contained about 0.0002 g/1 of plutonium.

These solutions were submitted to the radioassay group as regular assays. Five operators did two assays each on each of the six solutions submitted. The plutonium concentration was estimated as being 0.02 g/l for the P-1A and P-1B solutions, 0.05 g/l for the P-3A and P-3B solutions, and 0.0005 g/lfor the CA and CB solutions, and the solutions were diluted on the basis of these figures with the exception that the dilution of the C solutions was directly from Fig. 1 with no limitation placed on the amount of salts that would be on the counting slips. The criterion for the acceptability of these assays was that the observed deviation be less than the sum of the counting error and the absorption error. Of the 60 assays submitted, 13 were not acceptable (although one was not receated and was not included in the data). Of the twelve



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that were repeated, three had to be rerepeated. The results of the acceptable assays in terms of the percentage of the amount of plutonium put into them is shown in Table II. On the basis of these results, the probable error of a radioassay on a P-1 type solution appears to be $14_07\%$, on a P-3 type, 2.4%, and on a C type, 19.1%.

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Table I

39H155.615872.000156.6-0.61JH151.415771.920153.10.312H119.215471.950153.2-7.713T150.111971.375153.0-1.714H152.615171.900153.7-0.7153155.011971.375154.20.516H154.311671.825153.90.317T171.413971.700152.51.4188151.711071.730172.90.6201151.421.372.930150.30.7354150.920172.750136.30.6354150.920172.757117.11.5358115.420072.700116.2-0.536H119.619372.757117.11.537H119.019472.550155.9-3.689H150.119372.650116.01.6914119.318972.900146.01.6914119.621372.970151.6-5.2138H151.620972.800116.52.6138H151.620972.800116.42.2138H150.119372.650118.40.9139H119.621372.900116.52.6138H151.620972.850118.50.6	Lot number	Chemical Titration	Parts per million 49 in U slug	Specific Activity	Radioassay	% difference between titration and radicassay
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1041 147+0 202 147+2 1+3		-				
	1042	147.0	616	75,700	347+2	1.3

Table II	

Results of radioassays in terms of percentage of known Pu concentration

Operator	P-1A	P-18	P-3A	P-3B	CA	CB
A	103.6%	131.7%	101.0%	99.8%	91.0%	68.3%
	9902 %	108 ₀ 9 %	101.4%	99.2%	8305%	
В	101 .8 %	106.6 %	102.9%	100.6%	89.1%	15509%
	95.4%	122.2 %	96.3%	104.6%	84.4%	93.5 %
C	106.5%	122.4%	101.4%	102.7%	86.0%	117.0%
	102.8%	115.0%	100-3%	103.1%	87.4%	137°6 %
D	103.6%	118 . 4%	96.7%	102.9 %	94-4%	74.3%
	98.6 %	132.9%	100°7%	102 .1 %	102.6 %	56.0 <i>°1</i> 0
E	113.8%	141.5%	9404 %	98.9%	94.6%	159.2 <i>%</i>
	113.6%	149.5 %	9400%	99-1%	97•7 %	117.4%

The P-1's were diluted in 25 ml volumetrics which resulted in counting slips with approximately 20 γ of salts and an absorption error of 4.6%. Counting rate on P-1A's:~125 c/m; counting error:~4.6%. Counting rate on P-1B's:~25 c/m; counting error:~10.0%.

The P-3's were diluted in 5 ml volumetries which resulted in counting slips with approximately 1.9 γ of salts and an absorption error of 2.0%. Counting rate on P-3A's:~1,300 c/m; counting error:~2.0%. Counting rate on P-3B's:~260 c/m; counting error:~3.4%.

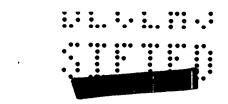


Table II (Continued)

-19-

The C's were diluted in 2 ml volumetrics which resulted in counting slips with approximately 175 γ of salts and an absorption error of 19.6%. Counting rate on CA's:~30 c/m; counting error:~10.0%. Counting rate on CB's:~7 c/m; counting error:~20%.



-20-

PRODUCTION

Since February, 1944, the radioassay group has done over 3500 assays. The following table shows the number of assays done for the purification and recovery sections, for the analytical group, and for others during the fiscal year October, 1944, to September, 1945.

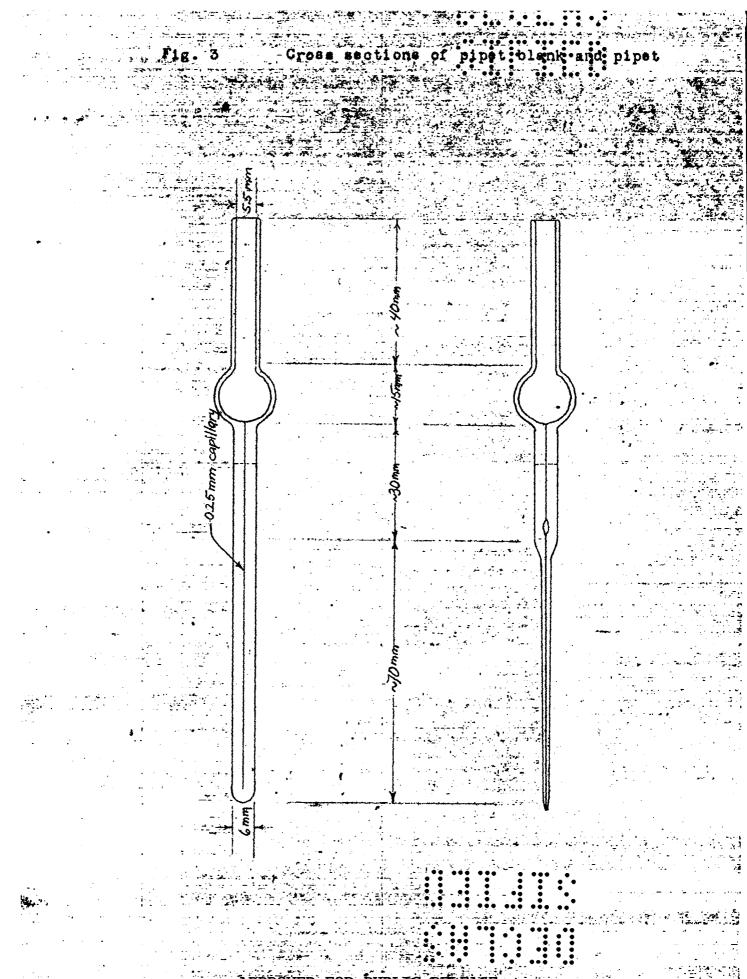
Month	Puri fi - cation	Recovery	Analytical	Miscel- lancous	Monthly totals
0ct., 1944	53	111	. 19	8	191
Nov.	55	83	15	1	154
Dec o	1114	86	22	23	175
Jan., 1945	46	131	10	26	213
Feb.	116	148	20	50	334
Maro	118	120	21	71	330
Apro	58	65	9 8	1 /4	235
Мау	157	101	18	12	288
June	282	62	20	3	367
July	209	46	39	35	<u>329</u>
Augo	280	39	6	25	350
Sept-	0	17	2	78	97
Totals	1,418	1,009	290	346	3,063
			بين المارية المتحدثة الأولية المراجع ا الم		

Of the 3,063 assays done during this period 424 had to be repeated.

lution chart SE SE Fig. × Sold and the second sec Ē× R.O S X 5 8 APPROVED E × FOR 00 30, PUBLIC Έ× ŜĮ. Ś RELEASE 0 105 10-3. 1.0# 10-2 10^{3} 102 0 10 10

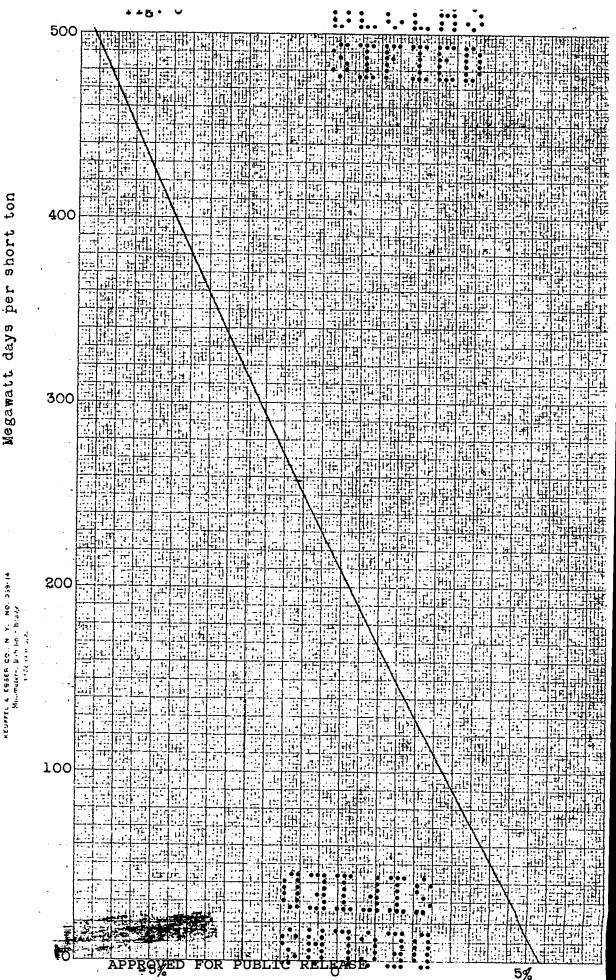
Absorption error <u>م</u>لم -----Fig 2 ontError 60 Thutth 50 APPROVED لل المراجر المرجبة أخب ملج 11:11 TIT 11 11111 -因 +++ - fr 1 - , 11111 111111 **THHH** i tilitta 17-11-11.5 TTTT: TTTTIS! 10 & salts on plate

FOR PUBLIC RELEAS



SST. TOTAL SOL			SAMPLE # DATE		
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PRODUCT CONC.	g./1.		
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DEV.	10	DATE	
VOL. OF SOL'N	ml .	NAME	
DESCRIPTION			
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