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DISSOLUTION OF URANIUM-ALUMINUM ALLOY SLUGS

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CHEMICAL TECHNOLOGY DIVISION

UNIT OPERATIONS SECTION

DISSOLUTION OF URANIUM-ALUMINUM ALLOY SLUGS

D. L. Foster
E. O. Nurmi

JUL 7 1952

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1.0 Abstract

Procedures for nitric acid dissolution of cast or extruded uranium-aluminum alloy slugs were evaluated on a scale of 20 to 40 gram moles per batch.

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2.0 Introduction

Studies were made on the nitric acid dissolution of 7.5% uranium - 92.5% aluminum alloy slugs, 1.36" in diameter and 8" long. A procedure of dissolution was needed to provide the necessary operating and design data for the Idaho Chemical Plant, and the ultimate goal desired was a procedure that would meet as nearly as possible the following requirements:

(a) Complete dissolution,

(b) Short dissolving cycle (less than twenty hours),

(c) Final dissolving solution 1.6M in aluminum,

(d) Final dissolving solution 0.2M in acid deficient.

In order to arrive at such a procedure, the following variables were studied:

(1) Type of slug (method of fabrication),

(2) Effect of acid concentration upon dissolving rates,

(3) Quantity of acid required,

(4) Catalyst and/or chemical additives required other than nitric acid,

(5) Effect of type of condensers upon acid economy.

Two methods of dissolution were initially considered for this work, caustic dissolution and nitric acid dissolution. Since more work had previously been done on nitric acid dissolution and, therefore, would presumably require less development work, this method was selected as the one to be developed. The caustic method also presented other disadvantages in that in further processing there were more steps of a complicated nature, and the uranium losses were higher than with the nitric acid dissolution.

A satisfactory procedure for the dissolution of MTR assemblies in nitric acid had previously been developed. This procedure used 4.0 moles of nitric acid per mole of aluminum, with the acid being added over a three hour period, 0.1% of the assembly weight as mercuric nitrate was used as a catalyst, the dissolving temperature was 90-107°C, and the dissolving time was 14 hours. The MTR assemblies are also uranium-aluminum alloy; therefore, the dissolving procedures should be somewhat comparable, differing chiefly in the fact that the assemblies present a much larger surface area than do slugs.

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3.0 Summary

The dissolution of uranium-aluminum cast alloy in nitric acid with mercuric nitrate as a catalyst produced a final solution 1.2 molar in aluminum nitrate and 0.8 to 1.0 molar in nitric acid in ten hours. This final solution was higher in nitric acid content than was desirable. To reach the specified acidity would have required more than twenty hours, which would have required an excessive number of dissolvers for the Idaho Chemical Process Plant.

In this study, 93 to 100% of the dissolver charge was dissolved. Complete dissolution was desired to control the criticality hazard, but it was demonstrated in consecutive runs that there was no build up of uranium in the dissolver.

Three procedures were developed for cast slugs (Procedures 1, 2, and 3). These procedures were identical in:

(1) Run time (ten hours),

(2) Completeness of dissolution (93 to 100%),

(3) Amount of catalyst required (5% of weight of alloy as $Hg(NO_3)_2$.

These procedures differed only in acid requirements due to different condenser arrangement. Procedure 1 incorporated a downdraft condenser with all condensate refluxed to dissolver. In Procedure 2, a downdraft condenser was used, but condensate was drawn off. Procedure 3 used an updraft condenser.

It was found that extruded slugs dissolved much more readily than did cast slugs. With the extruded slugs, 100% dissolution was obtained in four hours and produced a final solution 0.2 to 0.2 basic in HNO_3 normality and 1.2 to 1.4 molar in aluminum.

In all cases, it was found that the optimum dissolver feed concentration was between 4 and 5 N HNO_3. The optimum mole ratio of HNO_3 to alloy was found to be 4.0 moles of HNO_3 per mole of alloy in Procedure 1 for cast slugs and in the procedure for extruded slugs. Procedures 2 and 3 for cast slugs required 4.35 moles of HNO_3 per mole of alloy.

Mercuric nitrate was necessary as a catalyst in the nitric acid dissolution of uranium-aluminum. It was found that 5% of the alloy weight as mercuric nitrate is optimum for dissolution of cast slugs and that 2% of the alloy weight is sufficient for extruded slugs. Other chemicals, such as potassium nitrate, sodium nitrate, manganese sulfate and sodium nitrite suggested for breaking passive films produced little or no beneficial effect.

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The condenser, when connected in the downdraft manner, recovered 0.2 to 0.3 moles of nitric acid per mole of alloy more than when connected as an updraft condenser. This increase in recovered acid is due to the fact that in a downdraft condenser the entire condenser surface was wetted wall, giving higher absorption surface and more time for oxidation of NO to NO_2 while still in contact with an absorbing surface. The updraft condenser uses co-current cooling and countercurrent absorption, with all of the water vapor condensed out in the lower portion of the condenser.

4.0 Extruded Versus Cast Slugs

In arriving at procedures which would fulfill, as nearly as possible, the requirements set forth as a goal, several problems arose. Of these, the ones of prime importance are:

1. Extruded versus cast slugs.
2. Initial nitric acid concentration.
3. Moles of acid per mole of alloy.
4. Chemical additives.
5. Condenser type, updraft versus downdraft.

The J-slugs loaded in the Hanford reactors have the following dimensions:

Length - 8" ± 0.01"

Diameter - 1.359"

Bonding thickness on sides - 0.02"

Bonding thickness on ends - 0.35"

Can thickness - 0.02"

The chemical compositions of the can, bonding agent, and slug proper are:

(a) Can - 2S aluminum

(b) Bonding agent - 11.2 - 11.5% silicon, 88.5 - 88.8% aluminum by weight

(c) Slug - 92.5% aluminum, 7.5% uranium by weight. The uranium is 93.5% U^{235}.

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Extruded Versus Cast Slugs (continued)

Slugs approximating the J-slug composition and dimensions were made by the Oak Ridge National Laboratory Metallurgy Division. Detailed methods of fabrication are reported in ORNL CF-50-12-23. Types of slugs used in the dissolving studies are outlined below. Figure 1 shows a cast unmachined slug, a cast machined slug and a cast machined and bonded slug as "A", "B", and "C", respectively. The extruded slugs were similar in appearance to the cast machined slugs.

(a) Extruded Slugs

Uranium-aluminum alloy 7.5% U^{238} and 92.5% aluminum by weight, extruded through a die to 1.36 inches diameter and cut to 8 inch lengths. Some of these slugs were dipped in aluminum-silicon (14-18 gms. Al Si/slug) bonding material prior to dissolving.

(b) Cast Slugs

Uranium-aluminum alloy, 7.5% U^{238} and 92.5% aluminum by weight, cast in molds to 1.5 inches diameter and cut to 8 inch length. Some of these slugs were then machined to 1.36 ± 0.004 inches diameter and then dipped in aluminum silicon bonding material prior to dissolving.

Extruded slugs dissolved at a much more rapid rate than did cast slugs. Cast slugs under optimum conditions required 10 hours for near complete dissolution, whereas extruded slugs were completely dissolved in four hours.

The initial work on the dissolving of uranium-aluminum alloy slugs was done using extruded slugs fabricated by the Metallurgy Division of Oak Ridge National Laboratory. It was found that the slugs could be completely dissolved in four hours using 4.2 to 4.25 N HNO_3, 4 moles HNO_3/mole alloy and 2% (by weight of slug) mercuric nitrate as a catalyst.

Because actual pile loadings were to be made with cast slugs, a number of cast slugs, fabricated by the Metallurgy Division, were obtained to investigate possible differences in dissolving characteristics. Nearly complete dissolution (98-100%) of the cast slugs was obtained in ten hours under optimum conditions after extensive testing of various procedures.

Figure 1 shows quite graphically the difference in dissolving characteristics of extruded slug as compared to cast slug. Slug remnants D and E are from extruded slug dissolvings and F and G are cast slug remains. Slug D was removed from the dissolver after ninety minutes dissolution using Procedure 4. Slug E was removed from the dissolver after three hours using Procedure 4. Slug F was removed from the dissolver after three hours dissolution time using Procedure 1. Slug G was removed after five hours dissolution time using Procedure 1.

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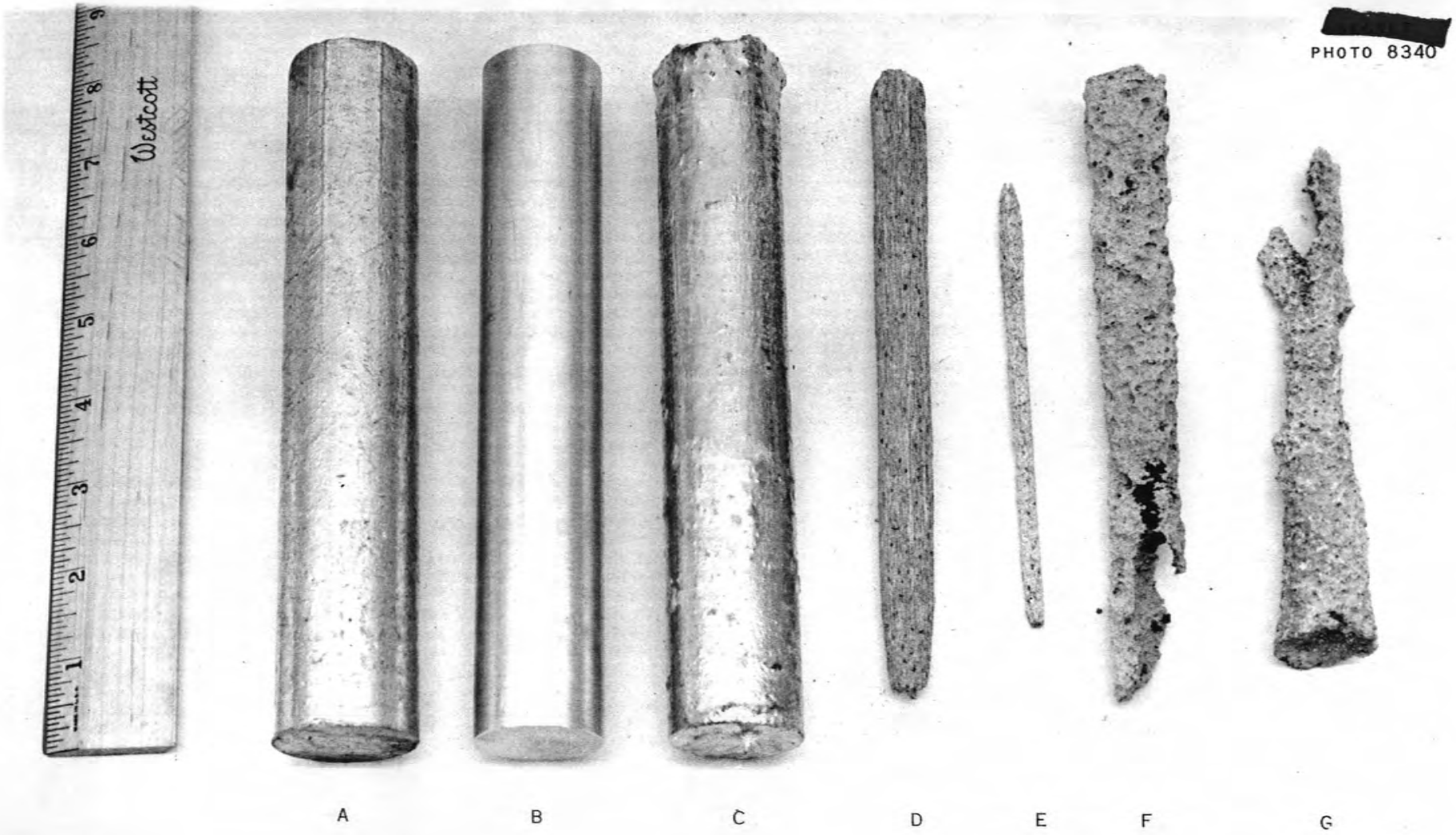

FIGURE 1
SLUG TYPES

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Figure 2

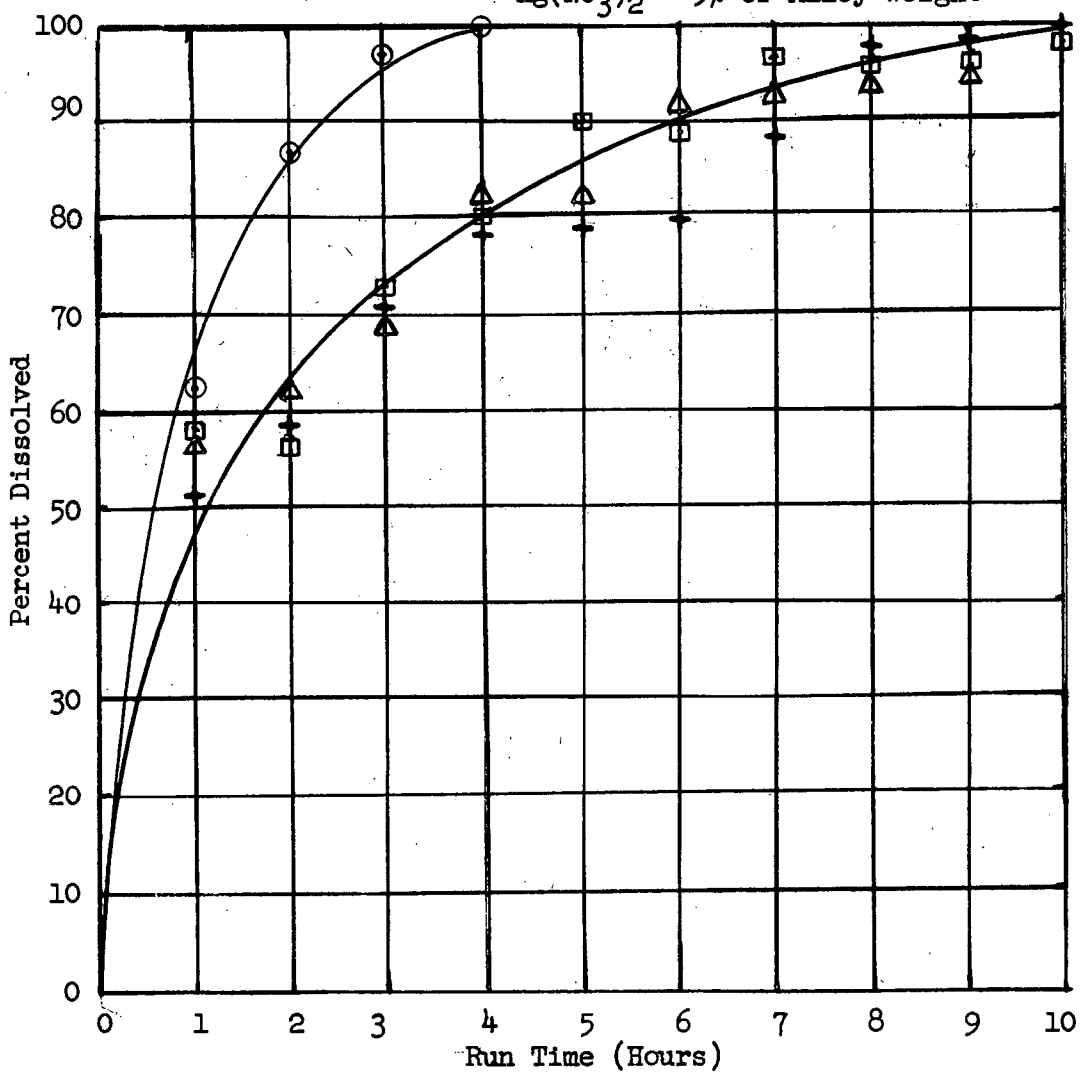
Dissolving Characteristics of Extruded Slugs

Vs. Cast Slugs

Legend:

Typical Extruded Run
Conditions: Temperature - 100-103°C, Dissolving Time - 4 hrs.
HNO_3 - 4.0 Moles per Mole - 4.25 N
$Hg(NO_3)_2$ - 2% of Alloy Weight

Average Cast Runs
Conditions: Temperature - 100-103°C, Dissolving Time - 10 hrs.
HNO_3 - 4.35 Moles per Mole - 5.0 N
$Hg(NO_3)_2$ - 5% of Alloy Weight



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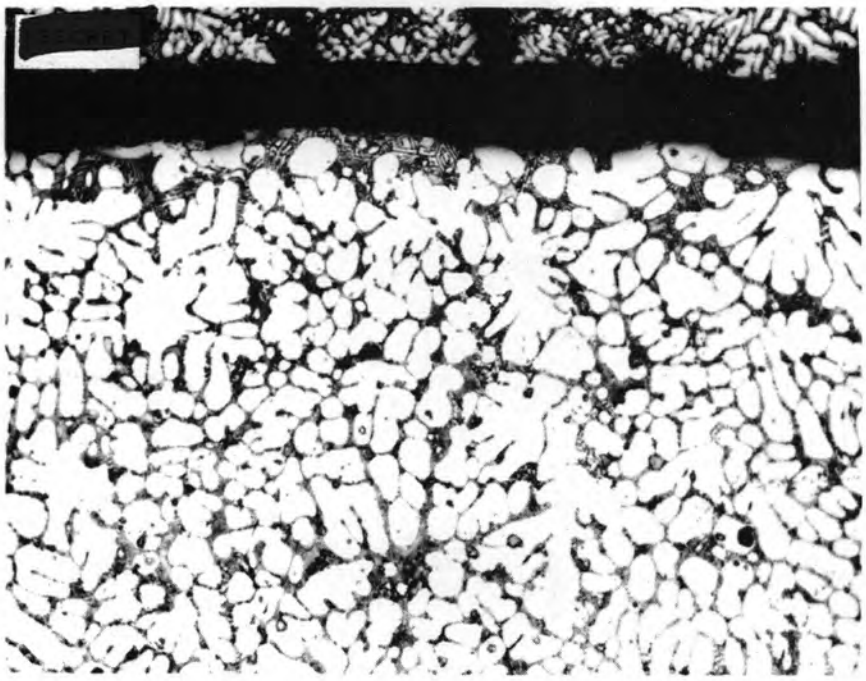
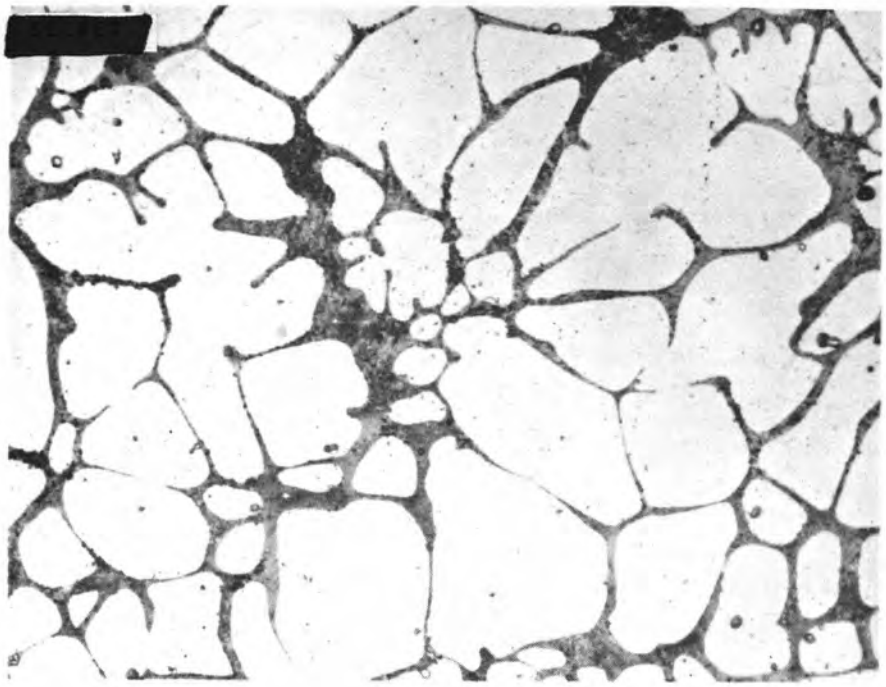


Y-2601 SURFACE OF SLUG 250X



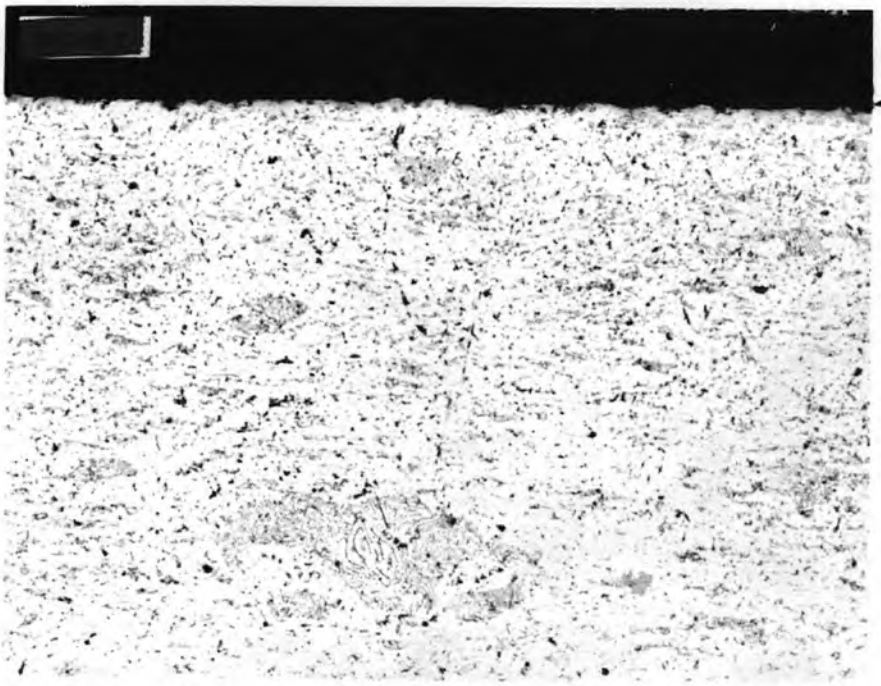
Y-2604 CENTER OF SLUG 250X

FIGURE 3
PHOTOMICROGRAPHS OF CAST SLUGS
(7.5% U-Al ALLOY)

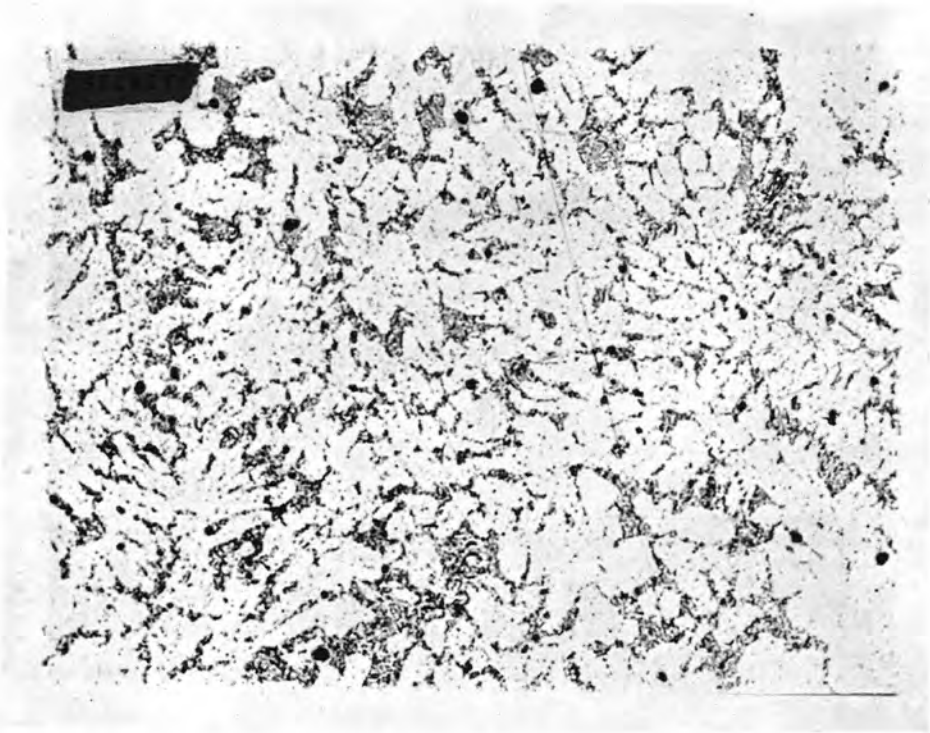
Table 1

Dissolving Characteristics of Extruded Slugs
Vs. Cast Slugs

Slug Type	% Hg.	Dissolving Time (Hours)	Moles HNO_3 / Mole Alloy	% Dissolved	Remarks
Extruded	2.0	4	4.0	99+	12 Runs Updraft Condenser
Cast	5.0	10	4.0	97.8	6 Runs 1 Slug Downdraft
				94.7	9 Runs 2 Slugs Downdraft
			4.35	99	7 Runs Updraft
				93	8 Runs Updraft - all Condensate Removed



Y-2603 SURFACE OF SLUG 250X



Y-2621 CENTER OF SLUG 250x

FIGURE 4
PHOTOMICROGRAPHS OF EXTRUDED SLUGS
(7.5% U-AL ALLOY)

Extruded Versus Cast Slugs (continued)

It is quite apparent from the photograph that extruded slugs dissolve much more uniformly than do cast slugs. In all cases, when partially dissolved cast slugs were inspected, it was observed that the center core of the slug had been preferentially attacked. Large pits were found in the outer surface as is plainly shown in Figure 1. All extruded slugs dissolved evenly over the entire exposed surface.

Comparisons of the dissolving characteristics of the two types of slugs under optimum dissolving conditions are shown in Figure 2 and Table 1. It is felt that the difference in the dissolving rates is due to the crystalline structure of the two types of slugs. An explanation of this difference has been presented*. From a metallographic study that was made, it was shown that the slugs consisted of particles of UAl_4 distributed in pure aluminum. In extruded slugs, this distribution is very uniform, whereas, in cast slugs, the particles form a network around the grain boundaries of pure aluminum. The differences in structure are shown in Figures 3 and 4. Since UAl_4 is dissolved more rapidly by acid than is pure aluminum, uniform distribution of this compound would favor the dissolving.

5.0 Initial Acid Concentration

It was found that 4 to 5 N acid is the best starting range. The first four runs were made adding the 70% HNO_3 continuously to the boiling water and mercuric nitrate catalyst in the dissolver. The acid was metered to the dissolver through a rotameter at rates from 15-35 ml/minute over a ten hour period. These runs did not prove to be successful due to continuously changing acid requirements for optimum dissolving rates. An excess or deficiency of nitric acid during the course of a dissolution may completely halt the reaction.

As it was difficult to estimate the progress of the dissolution at any time with any degree of accuracy, it was decided to add all of the acid in one batch and determine at what initial acid concentration the optimum dissolving cycle could be obtained. Work performed along this line proved that the best initial HNO_3 concentration for optimum dissolving was in the range of 4 to 5N as is shown in Table 2.

Data were reported in HW-18353** on the dissolving rates of uranium-aluminum alloy in known concentrations of $Al(NO_3)$ and normalities (Appendix B). Their dissolving rates at an aluminum nitrate concentration of 0.0 N (corresponding to an initial dissolving rate) are plotted in Figure

* Hamby, D. E. - Metallurgical Factors Affecting the Dissolving Rate of 7.5% U - 92% Al Alloy CF No. 50-12-23

** Hill, D. F. and Leitz, F. J. - Progress Report for June, 1950 - Chemical Research Section, HW-18353

Initial Acid Concentration (continued)

5 vs. HNO_3 normality. This curve shows an optimum initial dissolving rate at approximately 4.5 N. A hypothetical quantity "percent dissolved in X hours" can be calculated for a range of initial acid concentrations, assuming that 100% dissolution can be obtained in X hours at 4.5 N HNO_3, as follows:

percent dissolved in X hours at initial HNO_3 concentrations

$$\text{"a"} = \frac{\text{Dissolving rate at "a"}}{\text{Dissolving rate at 4.5 N}} \times 100$$

(Dissolving rates are obtained from Figure 5)

The dotted curve in Figure 6 is a plot of "percent dissolved in X hours" so calculated.

Plotting the results (of the writers) on extruded slugs for a six hour dissolving cycle at varying initial HNO_3 normalities gives the solid portion of Figure 5.

The optimum initial acid concentration is shown to be 4 to 5 normal by either approach.

It is evident from these data that acid concentrations above 5 N or below 4 normal have a deleterious effect on the rate of dissolving.

6.0 Moles of Nitric Acid Per Mole of Alloy

Optimum mole ratios of 4.0 moles HNO_3 per mole of alloy for procedures 1 and 4 (cast slug with a downdraft condenser with all reflux returned to the pot and extruded slugs with updraft condenser) and 4.35 moles HNO_3 per mole of alloy for procedures 2 and 3 (cast slugs with a downdraft condenser with all the reflux removed, and cast slugs with an updraft condenser) were found.

No experimental work was done outside the range of 4.0 to 4.35 moles acid per mole of alloy since these ratios used in conjunction with 4-5N HNO_3 result in higher final acid concentrations (ca, 0.5 - 1N) than those outlined in the specifications for a final dissolver solution (0.0N to 0.2N basic). Therefore any mole ratio greater than 4.35 moles per mole of alloy would only aggravate the situation and require more neutralizing solution and boildown time. If less than 4.0 moles of acid per mole of alloy were used the dissolving time would be greatly increased (from 10 hours to perhaps 20 hours) as the dissolution of the alloy in low acid concentrations (less than 1N HNO_3) and high aluminum concentrations (i.e. greater than 1 Molar Al.) is extremely slow as can be seen by referring to fig. 5.

[redacted]

Table 2

Effect of Initial H^+ Normality

Slug Type	Mols H^+ / Mol Al	% Hg	Initial H^+ (N)	Time hrs	% Dissolved	No. of runs
Cast	4.3	5.0	5.3	10	90	2
			5.0	10	99	7
Extruded	4.0	2.0	6.70	6	70	2
			5.68	6	93	8
			4.93	4	94	1
			4.21	5	100	3

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Figure 5

Initial Dissolving Rate vs H^+ Normality

From Data of Hill and Leitz

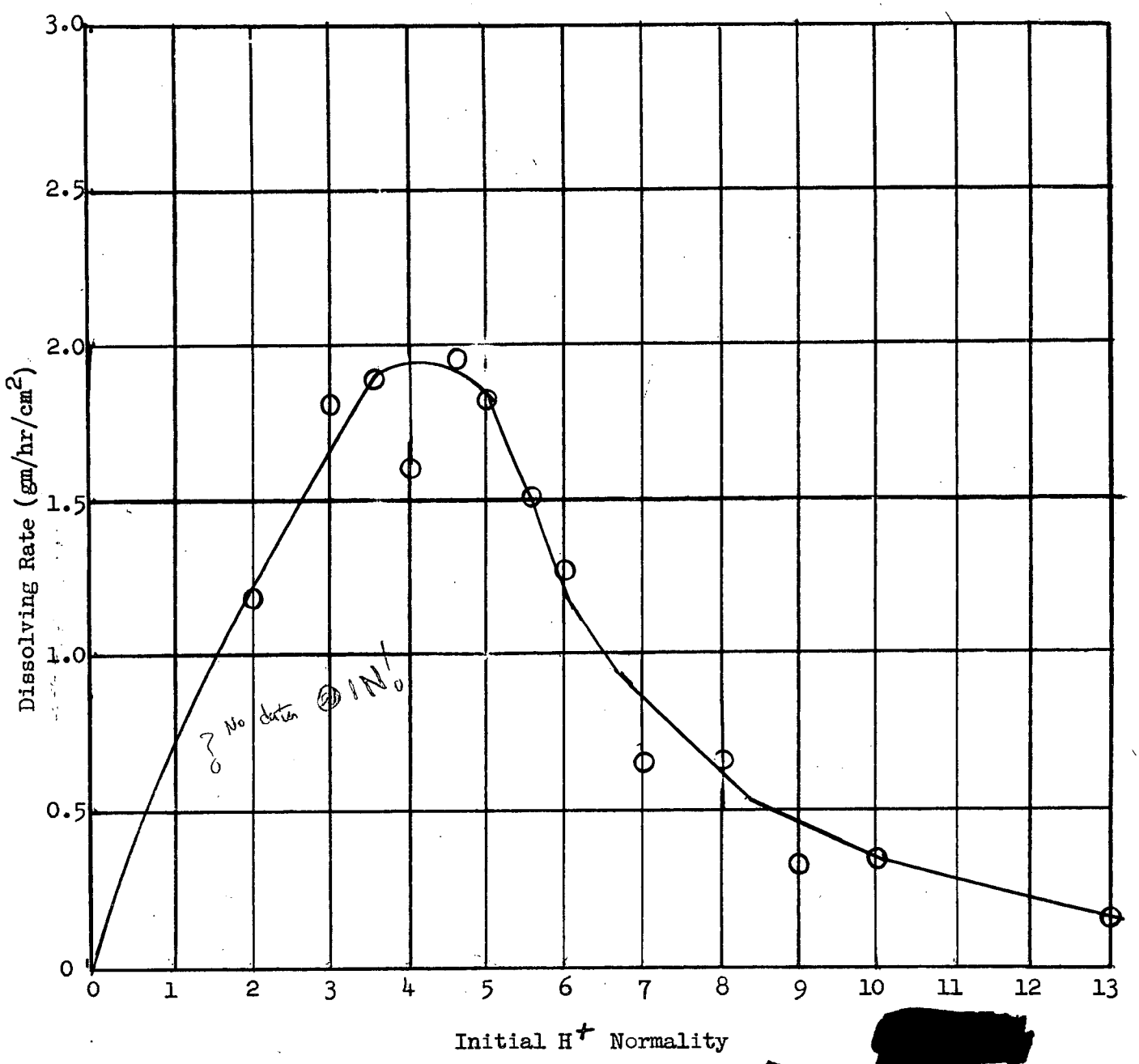

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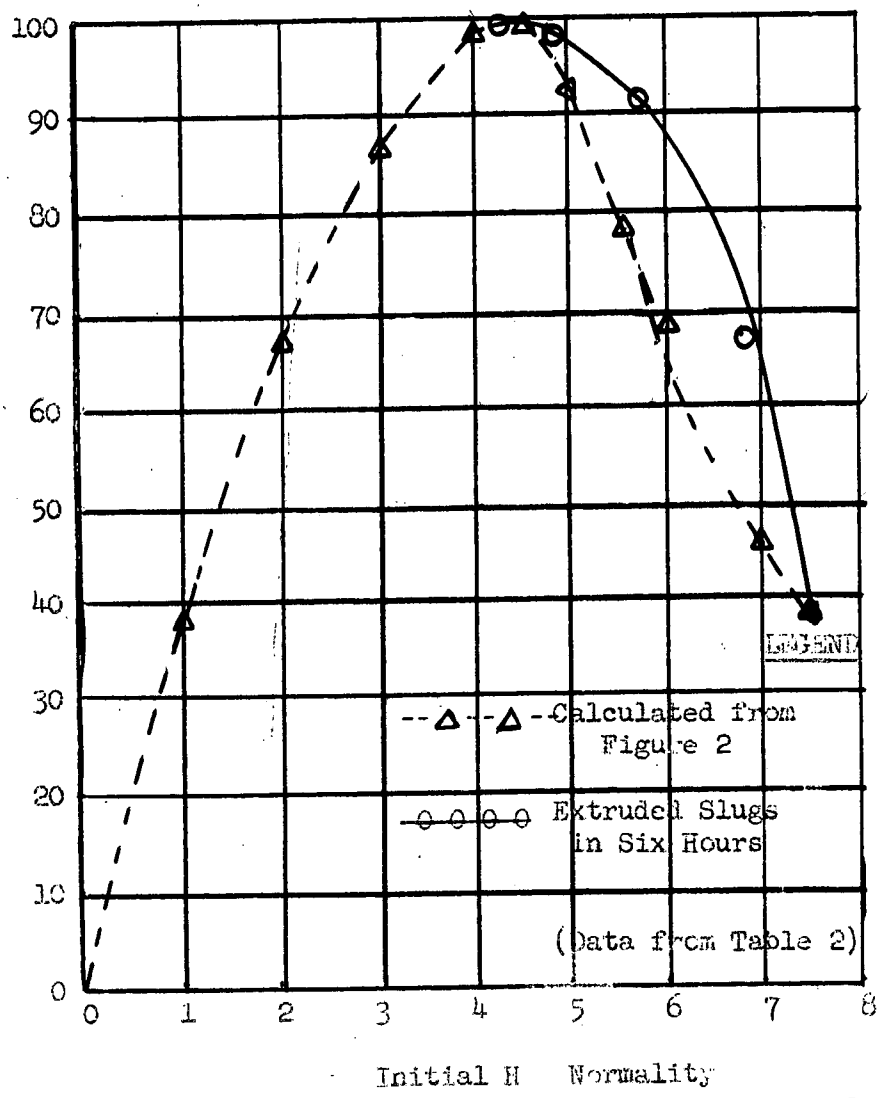
Figure 6

Percent Dissolution at Constant Time
vs. Initial H Normality



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6.0 Moles of Nitric Acid Per Mole of Alloy (continued)

Having established the initial nitric acid normality as 4-5N it was necessary to determine the minimum quantity of acid to be added in order to satisfy the conditions of complete dissolving, low final acid concentration, and short dissolving cycle. This quantity depends upon the type of condenser used. The effect of varying the mole ratio for an updraft condenser procedure 3 is shown in Table 3, also shown are the best ratios for other procedures. The percent of slug dissolved in 10 hours time varies from 70% at 4.0 moles per mole of alloy to 99% at 4.35 moles per mole of alloy using the updraft condenser. It was found that 4.35 moles of acid per mole of alloy using cast slugs was the best ratio for updraft type runs, or down draft runs in which all condensate is removed from the system. The best ratio for downdraft type runs using cast slugs with all the reflux returned to the pot or with extruded slugs and an updraft condenser was 4.0 moles of acid per mole of alloy. Because of the more favorable acid economy a downdraft condenser with all reflux returned to the pot was used in the final development studies.

7.0 Chemical Additives

In investigations on chemical additives necessary in dissolving runs, 5% of the alloy weight as mercuric nitrate was found to be the minimum concentration for complete dissolution. Other chemical additives had no beneficial effect.

Mercuric nitrate is necessary as a catalyst in dissolving the U-Al alloy slugs in nitric acid. In studies for determining optimum amounts of mercuric nitrate, over a range of from 2 to 57% it was found that 5% was the minimum quantity to carry the dissolving as nearly as possible to completion. A shorter dissolving time could be obtained with about 50% mercuric nitrate but since it was desired to keep the amount of chemical additives at a minimum to prevent further processing complications and to minimize cost, the 5% mercuric nitrate was considered optimum (see Table 4 and Figure 7).

Chemical Technology Division Report FLS-501* suggested that traces of other chemical compounds might have a beneficial effect upon the dissolving rate. Such additives as KNO_3, $NaNO_3$, $NaNO_2$ and $MnSO_4$ were tried with slight or no effect on dissolving rates. Reference to Table 5, shows slight improvement in dissolution rate using .008M $NaNO_3$, and no improvement with 0.1MKNO_3 when compared with a standard run using 2% Hg and 4.0 moles acid per mole of alloy. Comparing runs at 5% Hg and 4.0 acid to alloy mole ratio shows a detrimental effect using 0.1 M $NaNO_3$. Runs at 5-6% Hg and 4.35 acid to alloy mole ratio also show an adverse effect on dissolution using 0.09M $MnSO_4$, 0.02M $NaNO_2$ when compared to runs without these chemicals.

[redacted]

Table 3

Effect of Moles HNO_3
per Mole of Alloy

Conditions

$Hg(NO_3)_2$ - 5%
Initial H (N) - 4.75 - 5.0
Run Time - 10 Hours

Mols H^+ / Mol Alloy	% Dissolved	Remarks
4.0	70	Cast Slugs - Updraft Condenser - 1 run
4.2	82	Cast Slugs - Updraft Condenser - 1 run
4.35	99	Cast Slugs - Updraft Condenser - 7 runs
4.35	94	Cast Slugs - Downdraft Condenser All Reflux Removed - 6 runs
4.0	98	Cast Slugs - Downdraft Condenser Reflux Returned to Dissolver - 6 runs

[redacted]

[redacted]

Table 4

Effect of Mercuric Nitrate
Concentration Upon Dissolution

Conditions

Acid Ratio - 4.3-4.5 Moles per Mole
Initial H (N) - 4.25 - 5.0
Run Time - 7-8 hrs.
Cast Slugs

No. of Runs	% $Hg(NO_3)_2$	% Dissolved
1	2.0	73
2	4.6	84
7	5.0	99
2	6.0	83
2	10.0	70
1	35.0	95
5	46.0	98
1	57.0	99

[redacted]

Table 5

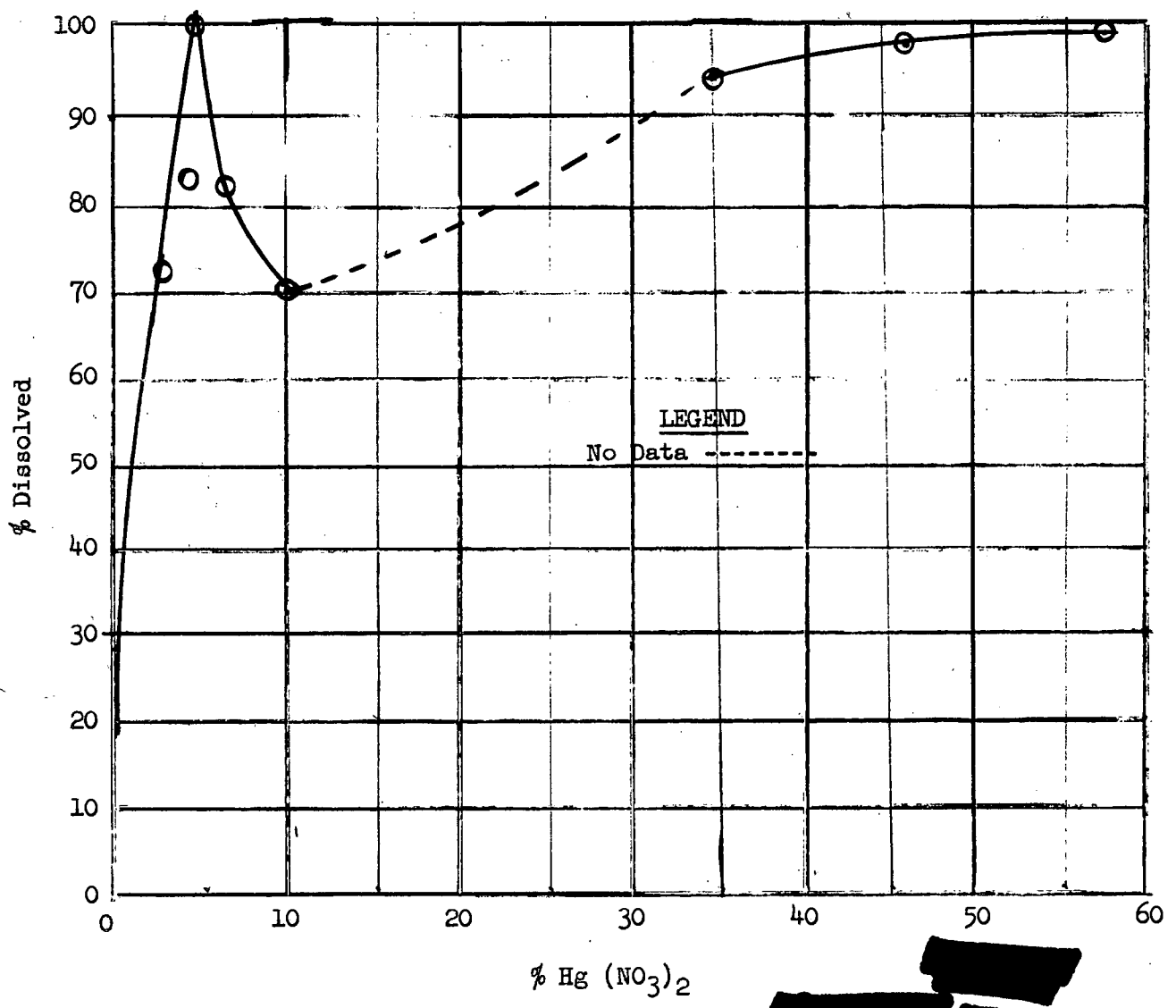
Effect of Chemical Additives Upon Dissolution

Moles HNO_3 / Mole Alloy	% Hg	Chemical Additives	No. of Runs	Avg % Dissolved	Beneficial effect
4.0	2.0	None	1	63	-
		0.1M KNO_3	1	68	None
		0.008M $NaNO_3$	2	73	Slight if any
	5.0	None	6	98	-
		0.1M $NaNO_3$	2	79.5	None
4.35		None	7	99	-
	6.0	0.09 M Mn SO_4	2	83	None
		0.02 M $NaNO_2$	1	88	None

[redacted]

Figure 7

Effect of Mercuric Nitrate
Upon Dissolution



[redacted]

[redacted]

8.0 Updraft vs. Downdraft Condenser

The studies conducted on condenser types showed savings of from 0.2 to 0.3 moles of HNO_3 per mole of alloy when a downdraft condenser is used instead of an updraft condenser.

The reflux condenser on the dissolver may be piped in two ways. The vapors and gases may be admitted to the bottom of the condenser and allowed to pass up and out of the condenser, (updraft) or the gases may enter the top of the condenser and pass downward and out of the condenser. (down draft). The liquid from the condenser in either case may be returned to the dissolver or may be discharged through a seal loop to another vessel.

The updraft or standard reflux condenser is usually used in most refluxing operations because of its simplicity of installation and maximum return of condensable material. The investigation of the downdraft condenser was dictated by the desire to absorb the maximum amount of nitrogen dioxide from the off gases and to return this as nitric acid to the dissolver. The major concern in removing the NO_2 was maximum acid economy and to reduce the volume of dissolver off gases to be handled in further processes to make the dissolvings "fumeless".

From the viewpoint of maximum acid economy, it would appear that a downdraft condenser would be most suitable. In this type of condenser, the dissolver off gases enter the top and the cooling water enters the bottom resulting in countercurrent cooling and co-current absorption. In this manner there is more efficient cooling, higher absorpiton area due to entire condenser surface being wetted wall, and more time for conversion of NO to NO_2 and absorption of NO_2. This method would then result in greater absorption of the nitrogen oxides thereby returning more acid to the pot so less would be required initially. This has been found to be true as can be seen by Table 6. Assuming that the actual amount of acid reacting with a mole of slug is constant in both downdraft and updraft runs, then if a greater amount of acid is recovered and returned by the downdraft condenser the apparent quantity of acid consumed of the initial charge would be less. This procedure would then allow less acid to be added initially and give the same final solution concentrations. One slug runs of this type indicate a saving on initial nitric acid of 0.2 to 0.3 moles acid per mole of slug.

As can be seen also from Table 6 the apparent acid consumption per mole of slug is greater for 2 slug runs than for 1 slug run. Evidently a smaller amount of acid per mole of slug is being returned by the condenser in the 2 slug runs. Probably the explanation of the decrease in returned acid is due to the fact that the gas velocity through the condenser has doubled cutting the residence time for oxidation of NO to NO_2 and absorption of NO_2 in half.

[redacted]

[redacted]

Table 6

Effect of Condenser Type Upon
HNO_3 Economy

Conditions

Initial H^+ (N) - 4.75 - 5.0 N
% Hg $(NO_3)_2$ - 5%
Run Time - 10 hours

Moles HNO_3 / Mol Alloy Charged	Final H (N)	Apparent mols HNO_3 used / Mol Alloy	% Dissolved	Condenser Type	No. of runs
4.35	0.96	3.51	99	Updraft	7
4.0	0.95	3.27	98	Downdraft 1 Slug	6
4.0	0.94	3.39	94	Downdraft 2 - Slug	9

[redacted]

9.0 Recommended Procedures

It was found that two of the procedure requirements could not be met, namely the final solution that was 1.6 M in aluminum and 0.2 M acid deficient. The following procedures meet as nearly as possible all of the requirements.

Procedure #1

Dissolving Time - 10 hours
Dissolver Temperature - 100 - 103°C
HNO_3 - 3.6 moles per mole of alloy diluted to 4.75N at the start.
0.4 moles per mole of alloy diluted to 4.0 N and added after 3 hours.
$Hg(NO_3)_2$ - 5% of alloy weight
Condenser - downdraft
Reflux - 10 to 20% of total volume per hour returned to dissolver (if total dissolver solution were 6 liters then reflux would be 600 to 1200 cc per hour or 10 to 20 cc per min.)
Oxygen - admitted to the system between dissolver and condenser.

By using this procedure, cast slug dissolvings should be 95-100% complete with a final solution that is 1.1 to 1.3 M in aluminum and an acid concentration of 0.8 to 1.0 N, (See Table 7).

Procedure #2

Dissolving Time - 10 hours
Dissolving Temperature - 100 to 103°C
HNO_3 - 4.35 Moles per Mole of alloy diluted to 4.75N and added at the start
$Hg(NO_3)_2$ - 5% of alloy weight
Condenser - downdraft
Reflux - 10 to 20% of total volume per hour drawn off and collected for use as feed make up. Dissolver volume maintained constant by hot water addition.
Oxygen - admitted to system between dissolver and condenser.

By using this procedure, cast slug dissolving should be 93-100% complete with a final solution that is 1.1 to 1.3 M in aluminum and an acid concentration of 0.5 to 0.8N.

Procedure #1 is recommended over procedure #2 because of simplicity of operation and less equipment required. Procedure #2 has an advantage in that the dissolution rate is independent of the reflux amount and concentration so long as the volume is maintained constant. For results of runs of this type see runs 149-161, Appendix "A".

[redacted]

Table 7

Results of Runs Using

Procedure #1

Conditions

HNO_3 Mole Ratio - 4.0
Initial N rmality - 4.75
Acid Addition - 3.6 Moles/Mole at start,
0.4 after 3rd hr.
Slug Type - Cast
% Hg $(NO_3)_2$ - 5%
Run Time - 10 Hours

Run No.	No. of Slugs	Final H (N)	Final Al (M)	% Dissolved
137	1	1.22	1.13	94
138		1.25	1.20	96
139		1.45	1.20	94
140		0.95	1.25	98
141		0.95	1.31	99
142		0.73	1.26	100
143		0.85	1.23	97
144	2	1.03	1.10	91
145		1.16	1.14	88
146		1.04	1.20	98
147		1.16	1.20	93
148	1	0.43	1.08	90

[redacted]

PROCEDURE I

IO HOURS

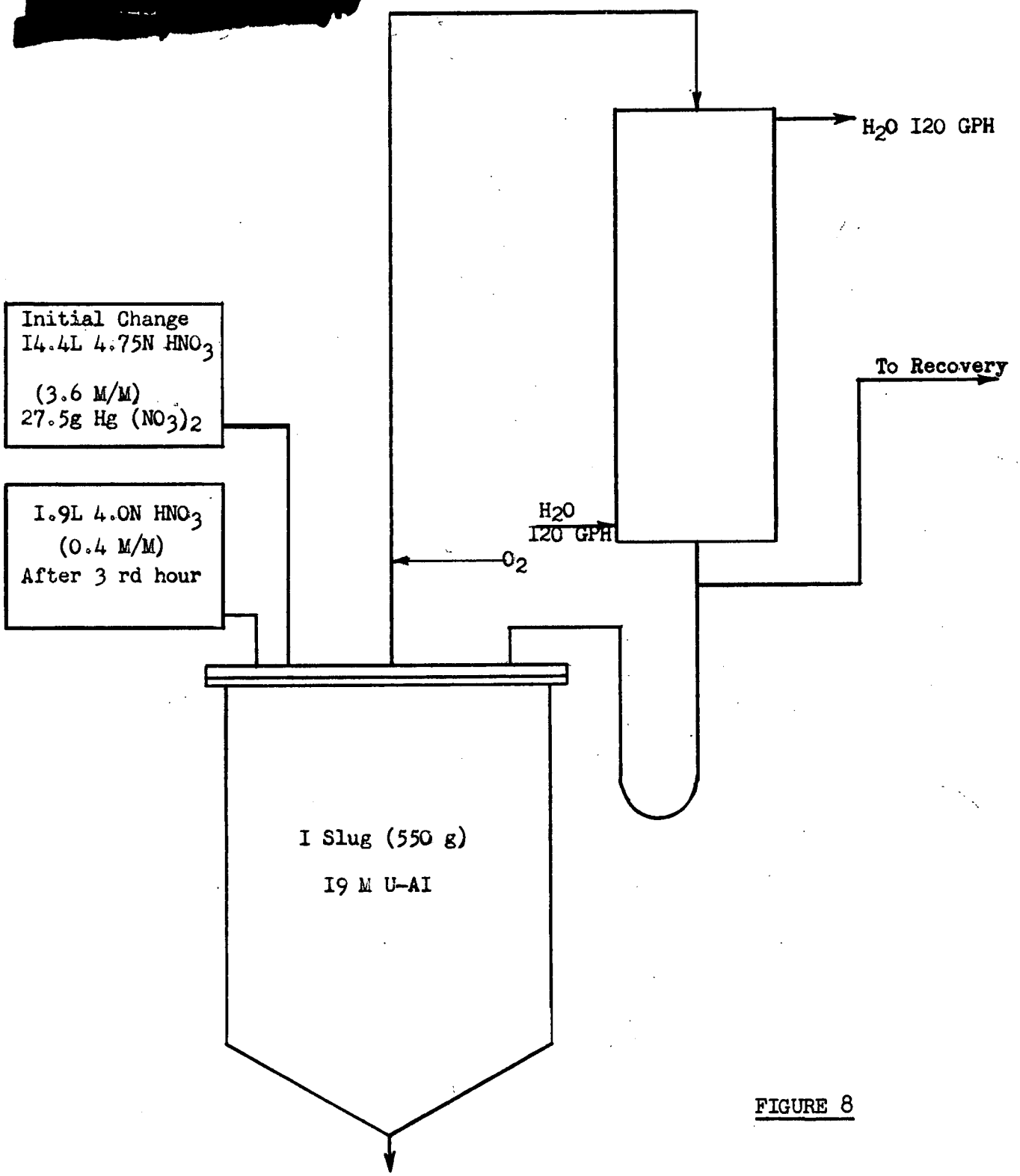


FIGURE 8

[redacted]

6/4/5I UNOP-32

Drawn by G.B.Hinsmore

[redacted]

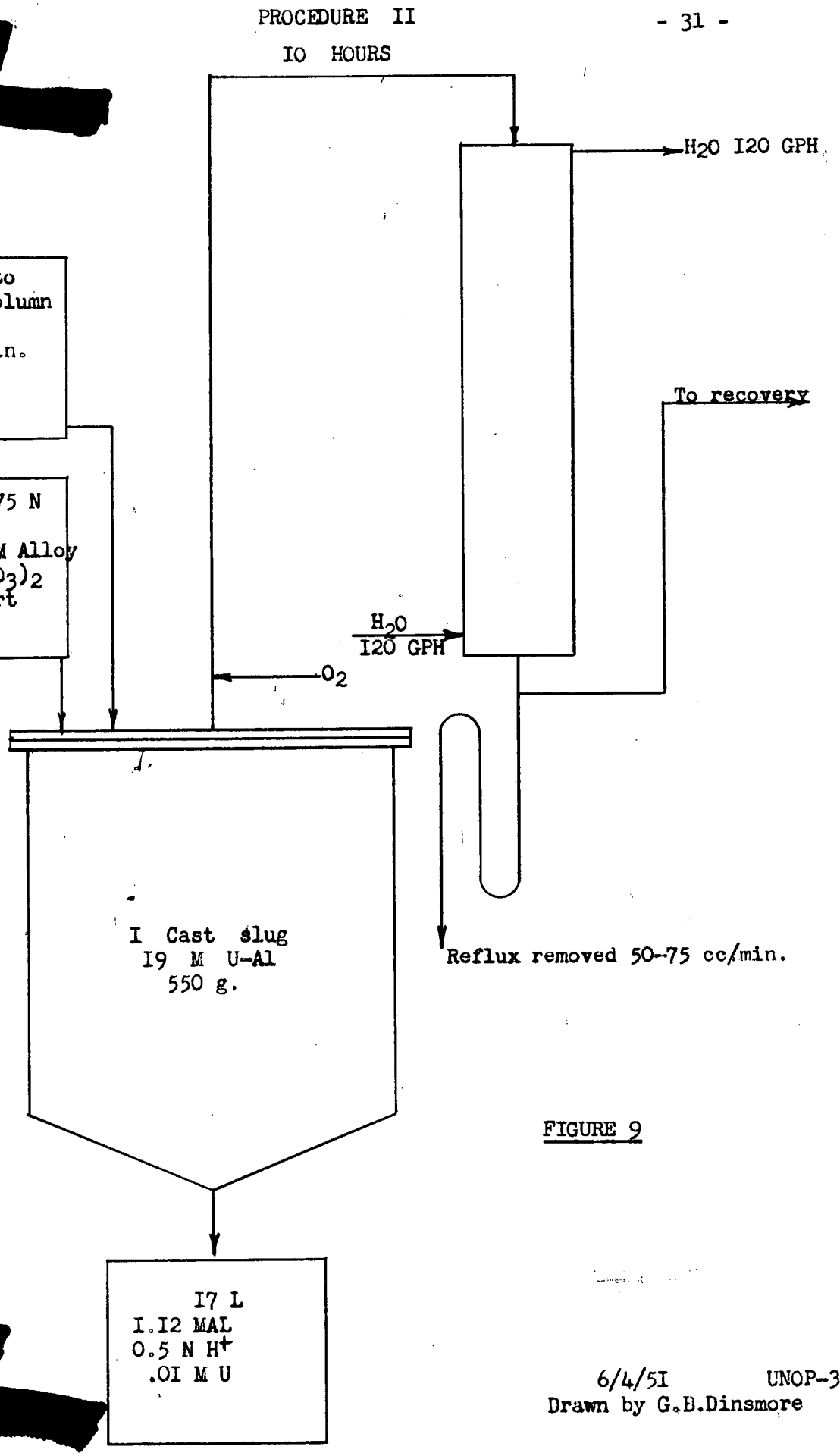


FIGURE 9

[redacted]

6/4/5I UNOP-33
Drawn by G.B.Dinsmore

9.0 Recommended Procedures (Continued)

Procedure #3

Dissolving Time - 10 hours

Dissolver Temperature - 100-103°C

HNO_3 - 4.35 Moles per mole diluted to 5N and added at the start

$Hg(NO_3)_2$ - 5% of alloy weight

Condenser - updraft

Reflux - 10 to 20% of total volume per hour returned to dissolver.

Oxygen - admitted to system between dissolver and condenser

By using this procedure cast slug dissolvings should be made 98 to 100% complete with a final dissolver solution that is 1.1 - 1.3M in aluminum and an acid concentration of 0.8 to 1.0N.

This procedure was most consistent in giving 100% dissolutions but is not recommended because of low acid economy. For results of runs of this type see runs 89-99, Appendix "A".

Procedure #4 (Extruded Slugs)

Dissolving Time - 4 hours

Dissolver Temperature - 100-103°C

HNO_3 - 4.0 moles per mole diluted to 4.25N and added at the start

$Hg(NO_3)_2$ - 2% of alloy weight

Condenser - Updraft

Reflux - 10 to 20% of total volume per hour returned to dissolver

Oxygen - None

By using this procedure, extruded slug dissolvings should be 98 to 100% complete with a final solution that is 1.1 to 1.3M in aluminum and an acid concentration that is 0.2N acid to 0.2N acid deficient. If extruded slugs could be used this procedure would be far more advantageous than the other three proposed because it meets practically all of the requirements set forth. The dissolving time is only 40% of that required for other procedures. The amount of catalyst required is less than half of what is required by other procedures and the final solution concentrations are more nearly in line with what is desired. For results of runs of this type see runs 10-23, Appendix "A".

[redacted]

PROCEDURE III
IO HOURS

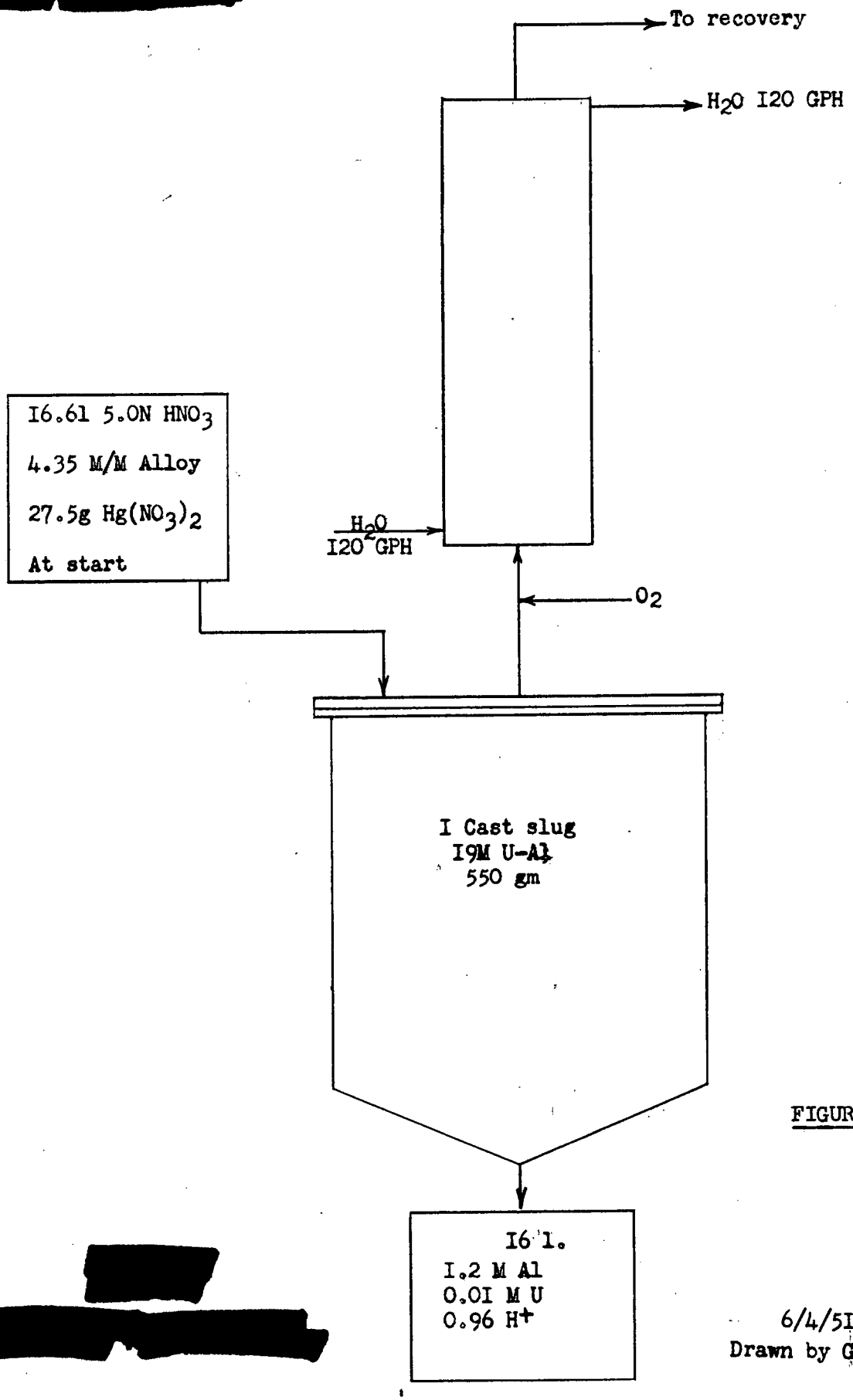


FIGURE 10

[redacted]

6/4/5I UNOP-34
Drawn by G.B.Dinsmore

[redacted]

PROCEDURE IV

4 HOURS

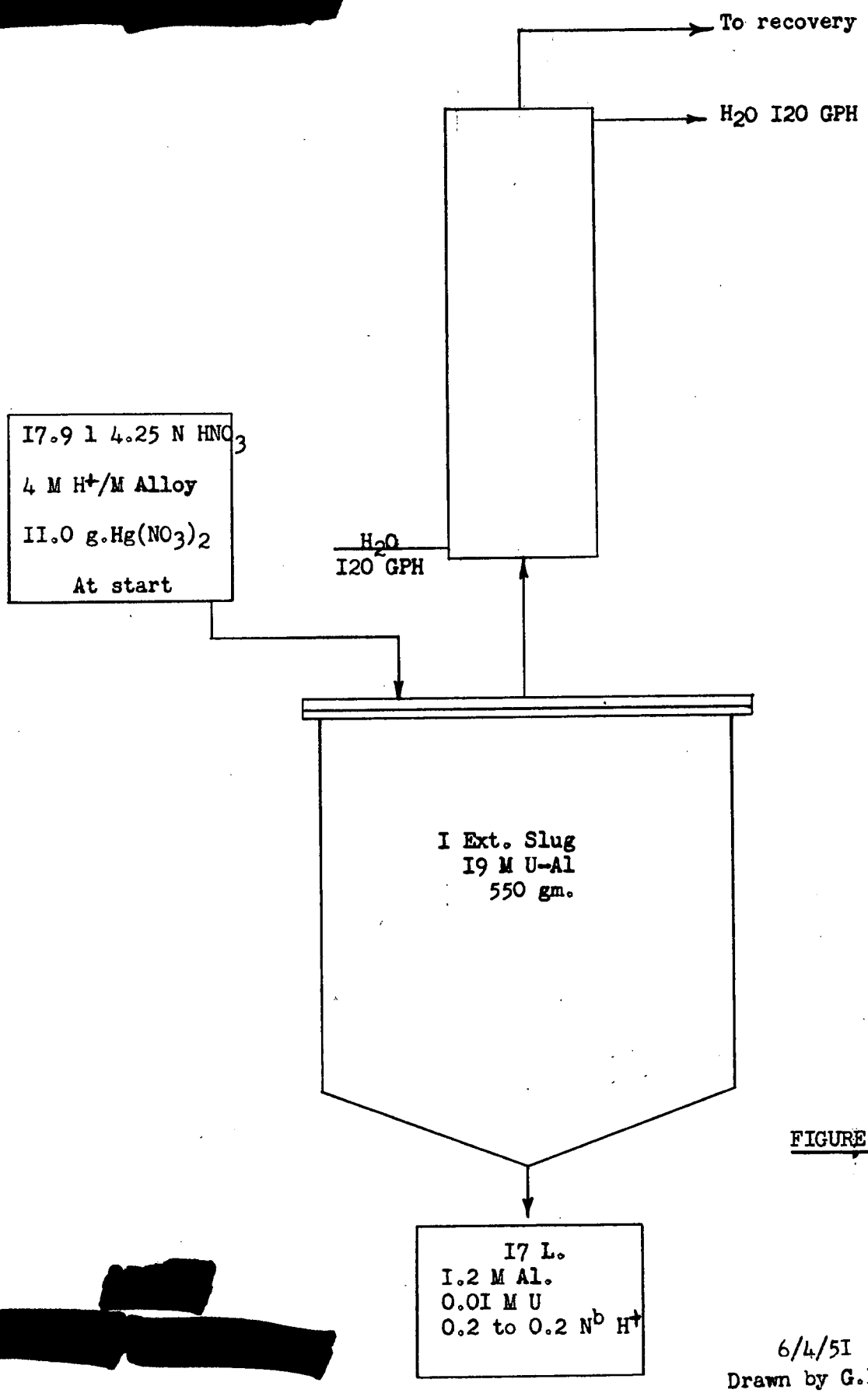


FIGURE 11

[redacted]

6/4/5I UNOP-35

Drawn by G.B.Dinsmore

10.0 Equipment

Three dissolvers were used in the J slug dissolving development work. The first was the "hot" dissolver located in Cell A, bank B of building 3503, the second or "cold" dissolver and the one in which the majority of the development work was performed was designed and built especially for these studies and was located in building 3503 on the 10' level.

The hot pilot plant dissolver in Cell I of the hot pilot plant (3019) was used to test procedures on an eight slug basis to determine possible scale up difficulties from one and two slug dissolvings.

"Hot" Dissolver

The dissolver, condenser and solution tanks in Cell A, bank B of building 3503 were used for the initial dissolving studies. The dissolver was a 65 gallon capacity, 18" diameter x 4'2" high jacketed vessel, made of 309 (Niobium stabilized) stainless steel, equipped with dip tubes for sp. gravity, liquid level manometers, thermocouple well, sampler, jet transfer lines, a 3" IPS vapor off gas line to the condenser, a two inch IPS spool piece, flanged, for slug loading, and a 1/2" IPS line for chemical addition through a funnel from the third level.

The condenser consisted of 3 coils of 1/2" OD, 16 gauge, 309 (Niobium stabilized) steel tubing, each coil having 35 turns, approximately 6.5 sq. ft. cooling surface with the coolant entering and leaving at the top. The shell was 7-3/8" diameter, constructed of 11 gauge type 309 (Niobium stabilized) stainless steel with a 3" IPS vapor inlet and a 2" IPS gas outlet. The non-condensibles passed through a scrubber and vacuum system to the atmosphere. A thirty gallon capacity stainless steel, type 347, tank equipped with an agitator was used as the dissolver solution retention tank before final disposal of the dissolver solutions. Figure 12 is a sketch of the dissolver setup.

Instrumentation

A six point recorder, Brown Electronik Potentiometer 0-200°C range, was used to indicate and record the temperatures of the dissolver, condenser water inlet and outlet, dissolver jacket outlet, off gas leaving condenser and the temperature in the retention tank.

A two pen, Taylor level meter, range 0-100 inches liquid level, specific gravity recorder indicator, was used to determine the liquid level and specific gravity in the dissolver throughout the run.

Water to the condenser was metered through a 0-200 GPH rotameter.

Steam to the dissolver jacket was controlled manually and operating pressures recorded periodically.

[redacted]

CELL A BANK B HOT DISSOLVER

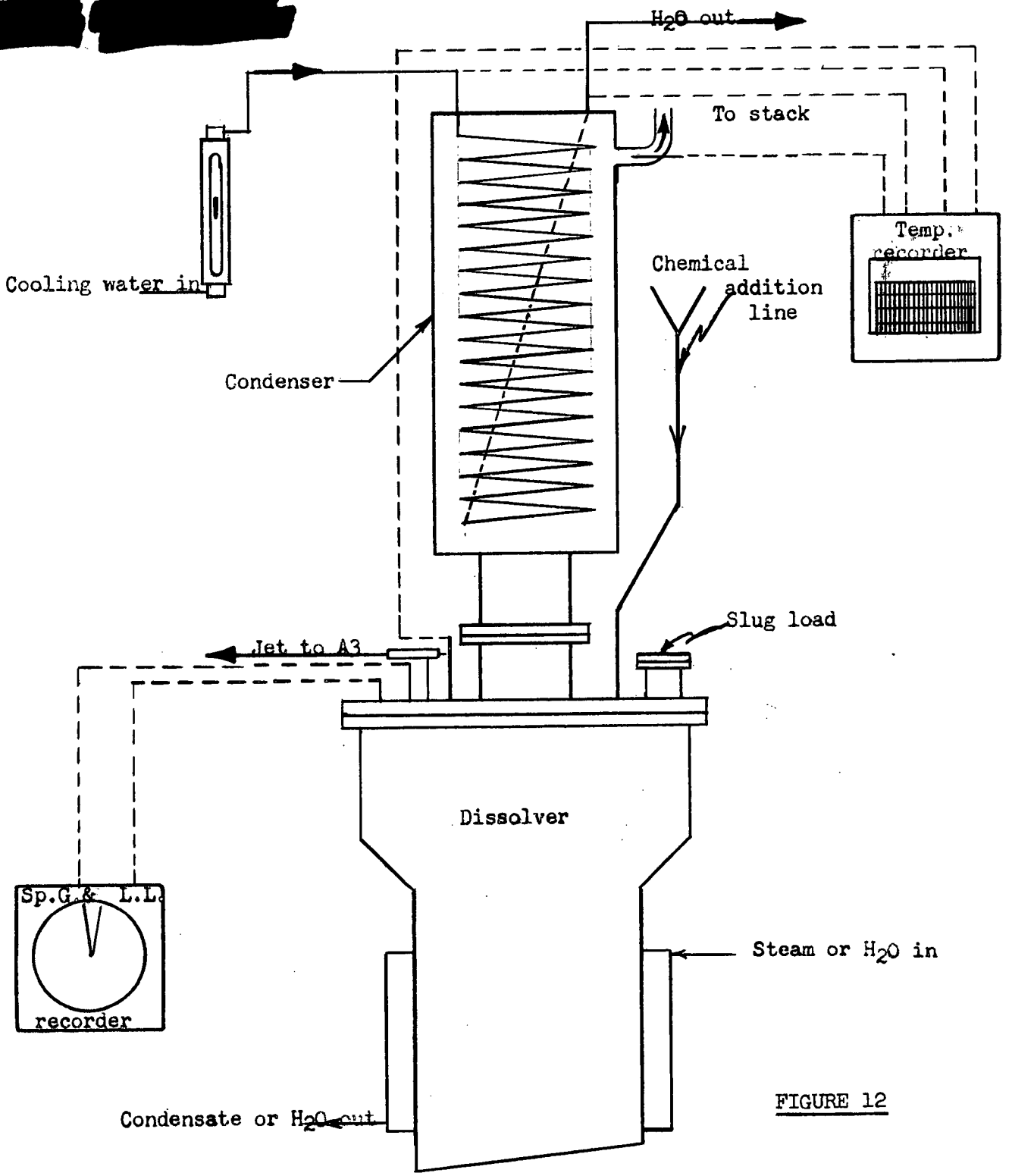


FIGURE 12

[redacted]

6/2/51 UNOP 3I
Drawn by G.B.Dinsmore

10.0 Equipment (Continued)

Cold Dissolver

As the dissolver in Cell A was oversize for the experimental work and rather difficult to obtain design information from, and it was needed for radioactive feed preparation for "hot" column runs a new dissolver was designed, constructed and erected on the 10' level north side of building 3503.

The design of the cold dissolver was dictated by four factors, (a) size to handle 1 to 2 slugs with the slugs always immersed in the solution, (b) ease of loading slugs and removing remnants after dissolution period, (c) provision for visual observation of the reaction and dissolver contents, (d) close control of the heating element in order to determine heat consumption and release by the reaction.

The new experimental dissolver was designed to operate with a maximum of two J slugs, and consisted of a conical bottom stainless steel tank (type 347) of 1/4" plate, 12 inches in diameter by 34" high with a 4" deep cone. It was heated by an internal coil of 3/8" pipe, 35 feet long. Dip legs were provided for Sp. gravity, liquid level monometers, and sampler and thermocouple well. A 3/4" IPS solution addition line with funnel was mounted on the top head. The dissolver was drained through a 1/2" IPS line at the bottom. Two sight glasses mounted on 2" IPS pipe were provided at the bottom. The slugs were added through a 3" IPS hand hole equipped with a sight glass on the top head. The off-gas and vapor line to the condenser was a 2" IPS line at the top of dissolver.

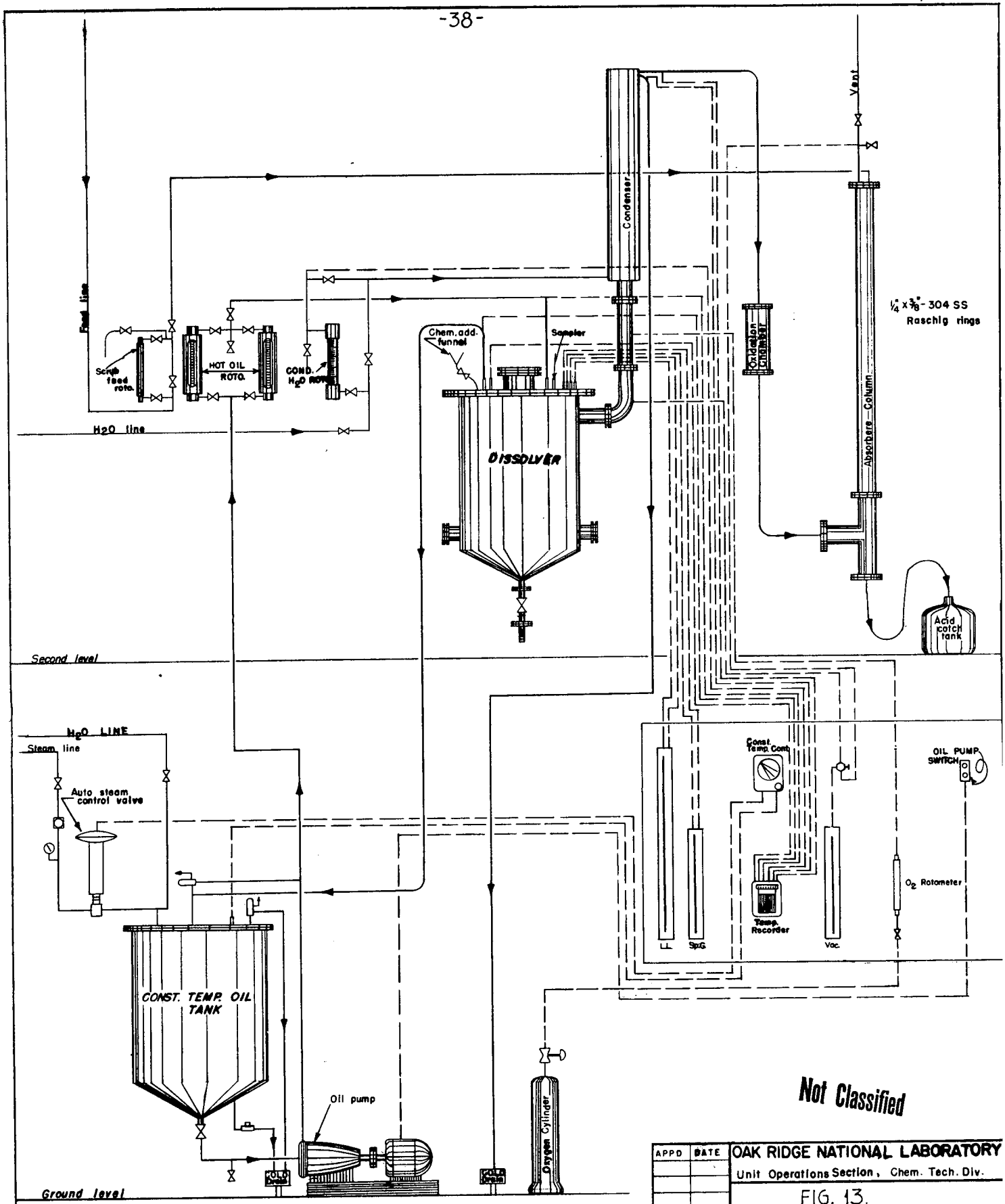
Condenser

The condenser consisted of four 1 inch stainless steel tubes six feet long inside a 5" stainless steel shell. The bottom vapor line was 2" IPS and the top vapor line 1" IPS. Vapors pass through the tubes with coolant on the outside. Approximately 6.5 square feet of cooling surface was available in the condenser. The condenser was operated with gas entering at bottom during the earlier runs than it was converted so that the gas entered at the top of the condenser for the rest of the development program. Figures 13 and 14 show the experimental dissolver setup with updraft and downdraft condenser respectively. Figures 15, 16, 17 and 18 are pictures of the experimental dissolver setup.

Two - four foot packed glass columns of 3" diameter pyrex glass were mounted in series for off-gas absorption. The packing used was 1/4" x 3/8" 304 stainless steel raschig rings. Vapor lines for the glass columns were 1/2" IPS 347 stainless steel pipe. The absorber water flowed counter-current to the gas flow.

The dissolver was heated by pumping hot di-butyl carbitol through the coil. The di-butyl carbitol was maintained at a constant temperature in a 60 gallon stainless steel tank heated by a steam coil. The temperature was controlled by an air actuated steam control valve operated by a Brown Electronik temperature recorder-controller.

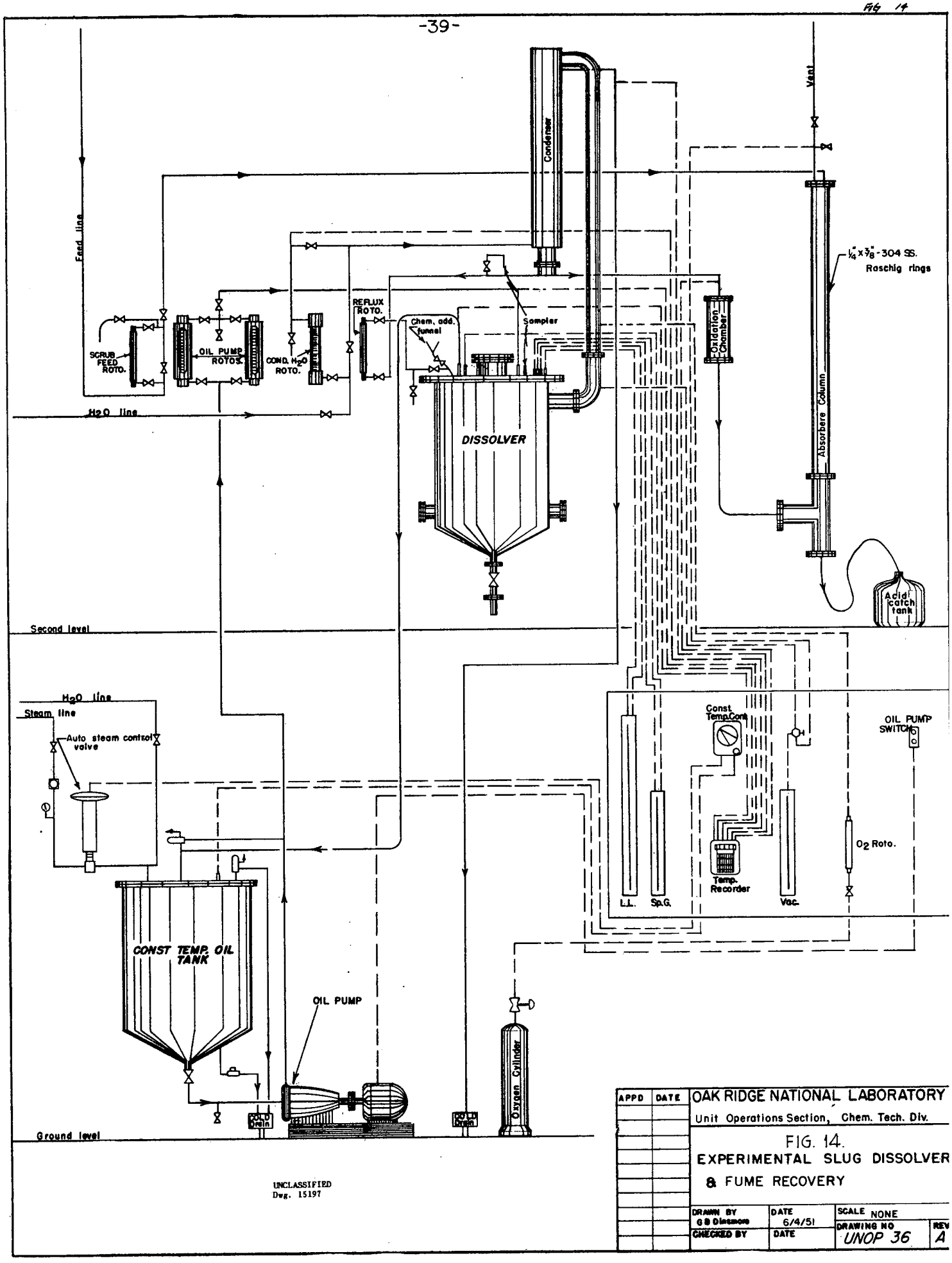
The pump used to circulate the oil was a 10 GPM Blackmer positive displacement pump equipped with 1/2 HP, 1745 RPM motor.



Not Classified

APPD	DATE	OAK RIDGE NATIONAL LABORATORY Unit Operations Section, Chem. Tech. Div.		
		FIG. 13. EXPERIMENTAL SLUG DISSOLVER & FUME RECOVERY		
		DRAWN BY G. B. Dinsmore	DATE 12/2/50	SCALE NONE
		CHECKED BY	DATE 12/5/50	DRAWING NO. UNOP 17

UNCLASSIFIED
Dwg. 15196



UNCLASSIFIED
Dwg. 15197

APPD	DATE	OAK RIDGE NATIONAL LABORATORY Unit Operations Section, Chem. Tech. Div.			
		FIG. 14. EXPERIMENTAL SLUG DISSOLVER & FUME RECOVERY			
		DRAWN BY G B Dismore	DATE 6/4/51	SCALE NONE	
		CHECKED BY	DATE	DRAWING NO UNOP 36	REV A

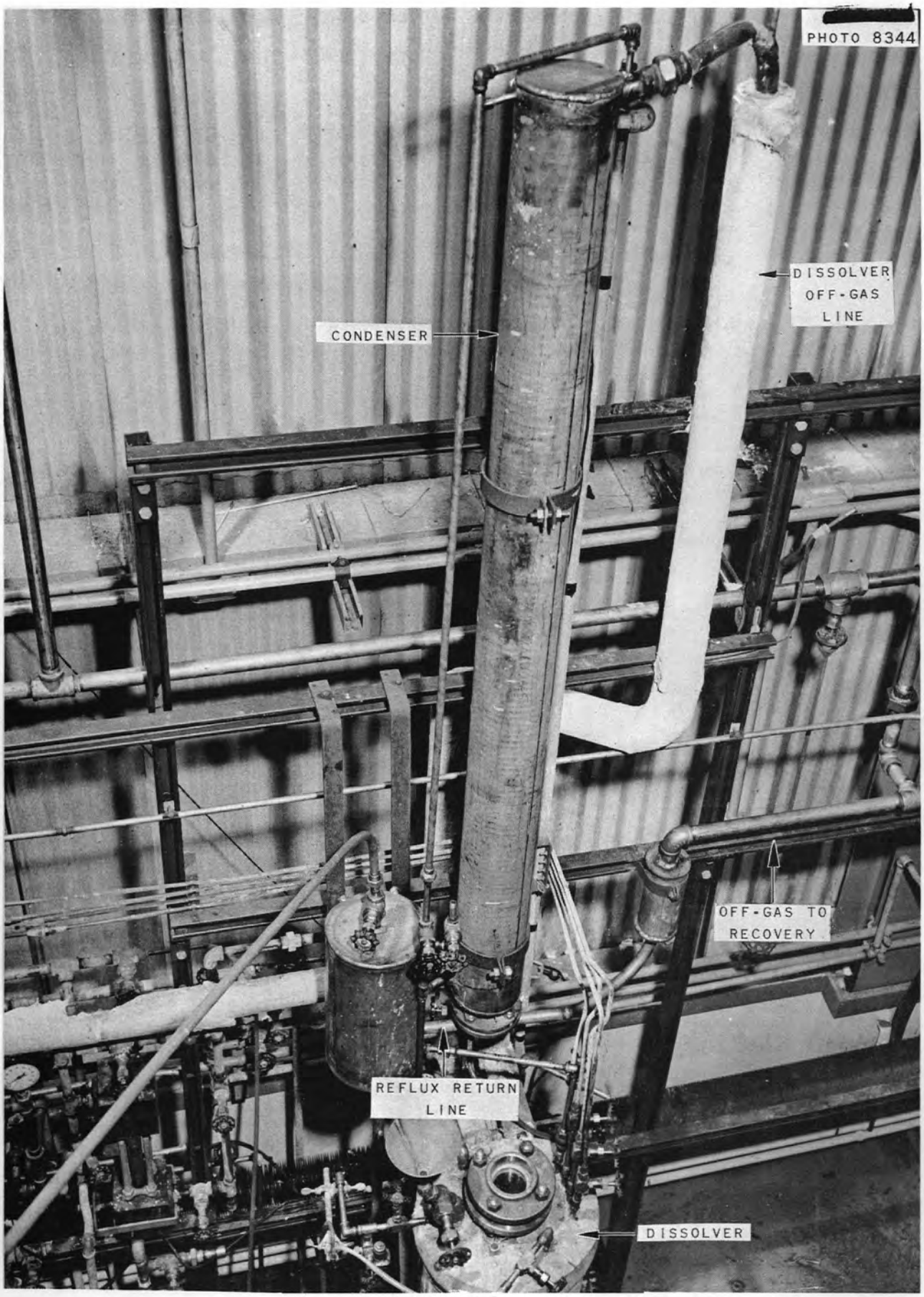


FIGURE 15
EXPERIMENTAL DISSOLVER DOWNDRAFT
CONDENSER SETUP

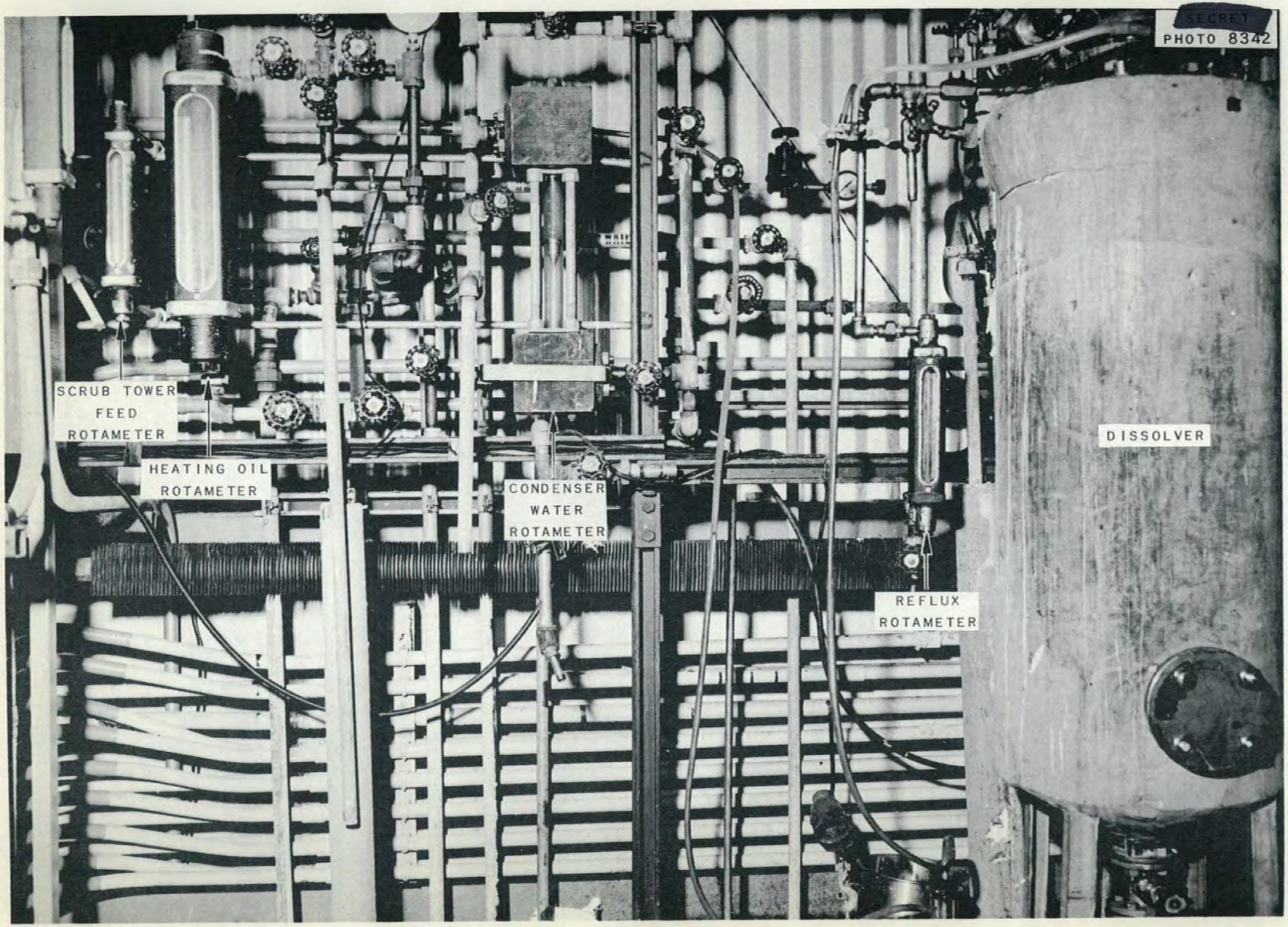


FIGURE 16

EXPERIMENTAL DISSOLVER AND FLOWMETERS

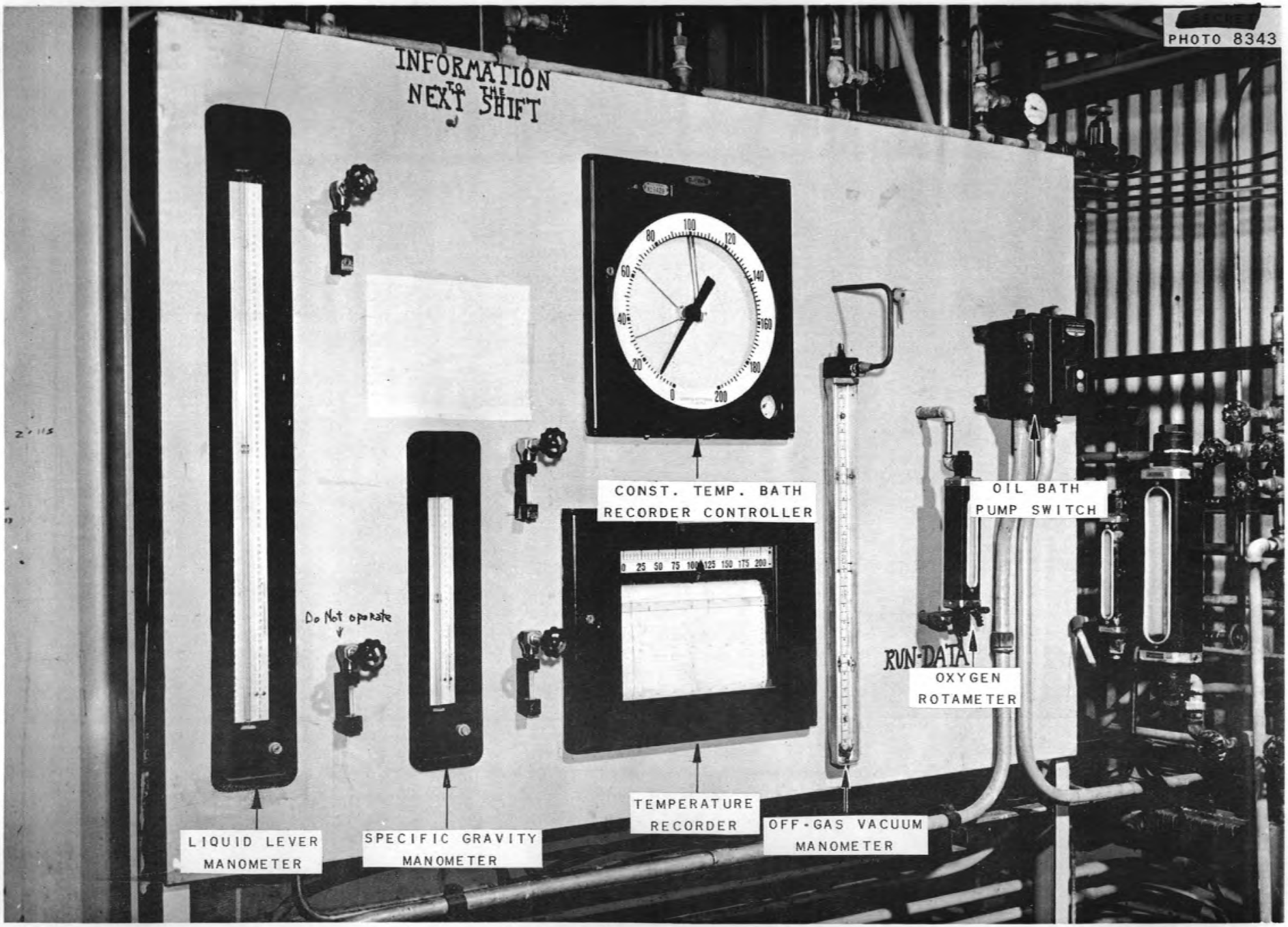


FIGURE 17
EXPERIMENTAL DISSOLVER INSTRUMENT PANEL

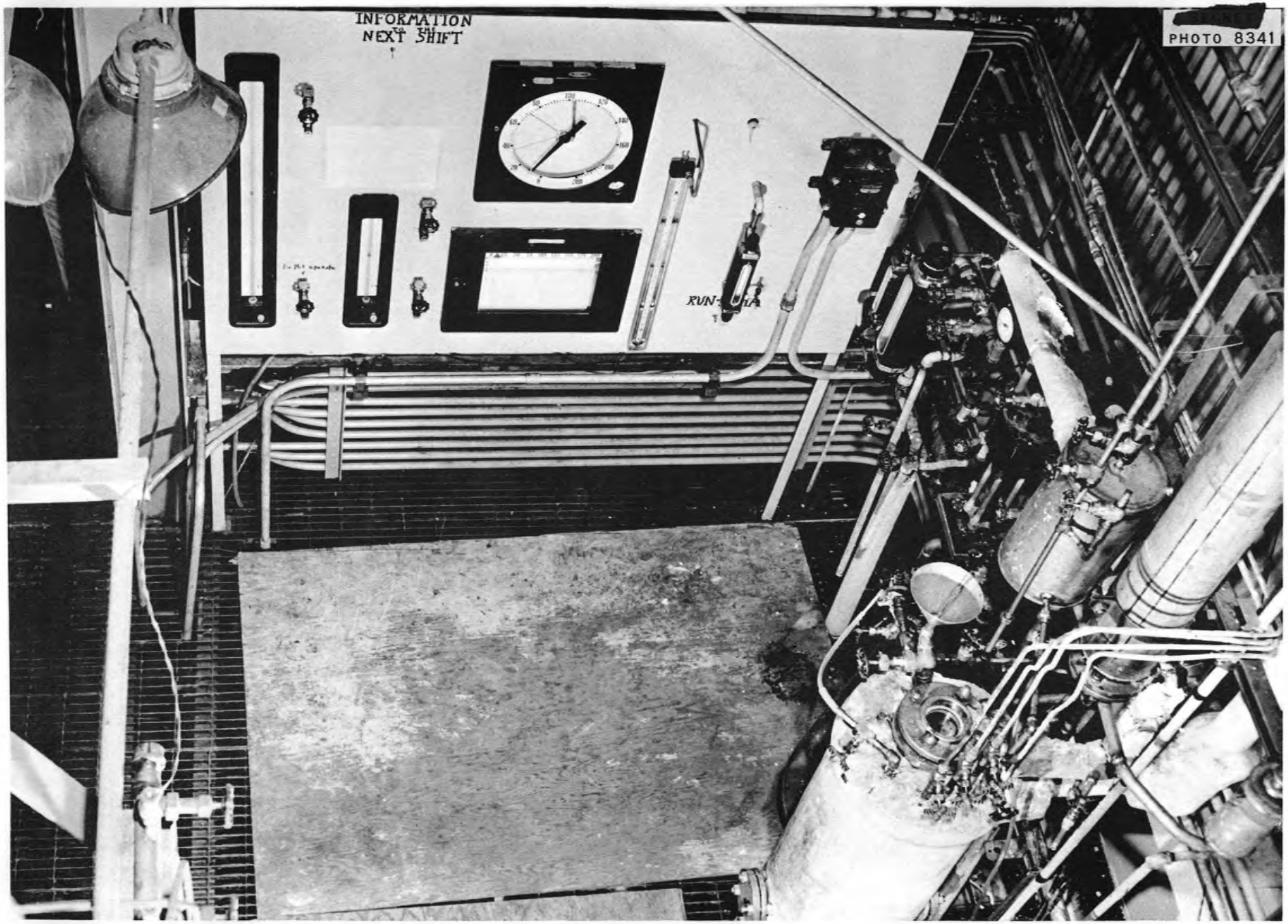


FIGURE 18
OVERALL DISSOLVER EQUIPMENT HOOKUP

Instrumentation:

The dissolver content liquid level and specific gravity were indicated by purge type manometers.

The six point Brown Electronik Potentiometer recorder recorded the temperature of the dissolver contents, the inlet and outlet temperature of the heating fluid, the inlet and outlet temperature of the condenser cooling water and the temperature of the dissolver off-gases entering the condenser.

The Brown Electronik recorder-controller connected to a thermocouple in the oil bath maintained the bath temperature constant by actuating an air operated steam control valve.

Rotameters were used to measure the flow of cooling water to the condenser, the flow of hot oil to the dissolver coil, and the flow of water to the absorber columns.

Samples from the dissolver were removed by use of house vacuum and ordinary laboratory vacuum flasks. The dissolver, oil bath, oil lines, and vapor off-gas line to the condenser were insulated with 85% magnesia insulation.

Operating Procedures:

Slug Loading and Unloading:

"HOT" Dissolver in Cell A, Bank B

Slugs were loaded through the slug chute from the top of the cell bank to the "Hot" dissolver for the first few runs. When it was found that analytical results of dissolver contents were not accurate enough to determine the extent of dissolving within ± 5% the "hot" dissolver was modified to permit the loading of slugs through a 2" IPS flanged nipple on the dissolver. The slugs were then placed in stainless steel wire baskets and lowered into the dissolver by wires attached to the baskets and the lower side of the closure flange. This permitted the withdrawal of the slug baskets for visual inspection and weighing of the undissolved portions of the slugs at the conclusion of a run. An Ohaus, 2Kg capacity laboratory platform balance was used to weight the individual slugs.

Two-Slug Experimental Cold Dissolver

Slugs were loaded to the experimental dissolver through the three inch diameter hand and peep hole. At the conclusion of a run the solution was drained by gravity to a drum and any undissolved portions of slugs were removed with a pair of 4 foot tongs.

Chemical Addition

Hot Dissolver in Cell A, Bank B

Chemical addition to the Cell A dissolver was through a funnel on the 20' level of Cell A. It was possible to continuously meter in the solutions through a rotameter or add them batchwise through the funnel. Upon completion of a dissolving the solution was transferred by a steam transfer jet to a neutralization and retention tank.

Cold Experimental Dissolver

Chemical addition to the cold dissolver was batchwise through a valved funnel and a batch feed tank located above the dissolver. The solution was drained by gravity from the dissolver to a catch drum located below the dissolver.

Operation

A typical operating procedure was to place the slugs in the dissolver, close the hand hole and then add the dissolving solution, usually 4-5 N HNO_3 and Hg $(NO_3)_2$.

The instruments were checked for operation and the water to condenser was turned on at the specified rate. Heat was then turned to the dissolver until the thermocouples indicated a temperature of 80-90°C in the dissolver then turned off. The temperature was allowed to rise to boiling (ca 101°C) due to heat of reaction and then the solution was kept at boiling with addition of heat as the reaction proceeded. Samples of the dissolver were taken hourly. A record of the liquid leve, and sp. gravity, in the dissolver, and temperatures, pressures, and flow rates were taken every 15 min. for the first hour and every half hour thereafter. At the end of the run the heat was turned off the dissolver emptied and any slug remnants that remained were weighed and recorded.

Sampling

"Hot" Cell A Dissolver

Samples of the dissolver solution in the "23" dissolver in Cell A Bank B were taken with an air jet recirculating type sampler of similar design to those used in the ORNL hot pilot plant.

Experimental 2 slug dissolver

The experimental dissolver was sampled by vacuum into a 15 ml. centrifuge tube. Four passes were usually taken to insure a representative sample. Sample taps were provided in the off gas lines to and from the condenser for taking gas samples. Condensate from the condenser was sampled by gravity through a stream sample tap in the reflux return line to the dissolver.

[redacted]

Hot Pilot Plant Dissolver

The hot pilot plant dissolver in Cell #1, Building 3019, used for the 8 slug (1/2 full scale) dissolving tests of procedure 3 was a 107 gallon flanged head vessel, 26" ID and 48" high. It was constructed of 3/8" plate sides, 3/4" plate sloping bottom, type 309 Niobium stabilized heat treated stainless steel. Heating or cooling of the dissolver was by means of a 30 inch high, bottom jacket and a 12 inch high, upper jacket constructed from type 347 stainless steel. A 3/4" crash plate to absorb the slug shock and a 17 inch diameter retainer basket for protection of service piping is contained in the vessel.

Services:

1. Solution transfer from the dissolver to a mix tank or a waste neutralizer was by means of two 3/4 Schutte-Koerting steam transfer jets.

2. Solution addition to the dissolver was by gravity flow from the gallery weigh tank.

3. Solution agitation was obtained from a combination steam and air sparger.

4. Solution samples were obtained by means of an air operated recirculating jet sampler in a shielded cubicle in the penthouse.

5. Vacuum supply was furnished by a 100 CFM Croll-Reynolds steam jet eductor discharging into the 1200 area stack.

6. Slug addition was by means of a 3" IPS curved pipe from the penthouse floor to the dissolver.

Instrumentation:

1. Automatic or manual temperature control was obtained by a Brown Circular Chart Temperature Recorder Controller operating three Masonneilan air operated diaphragm valves through two Taylor Transfer Panels. Solution temperature was recorded on an L and N strip chart Speedomax and a Brown TRC and indicated on a Brown Precision Indicator.

2. Vapor temperature was indicated on a Brown Precision Indicator from a thermocouple in the off-gas line immediately above the dissolver.

3. Liquid Level and specific gravity were recorded on a Hagan Ring Balance.

4. Tank vacuum was recorded and controlled by a Taylor Fulscope Controller operating a Masonneilan air operated diaphragm valve located in the off-gas line in the penthouse.

[redacted]

Condenser

The condenser, constructed of type 347 stainless steel, was mounted directly on the dissolver on a 6" IPS spool piece. It was 8' long, 11" O.D. with the lower 48" jacketed and packed with 1" x 1" x 1/16" type 347 stainless steel raschig rings. The top section had three cooling coils suspended from the top and made of 3/8" IPS 347 stainless steel pipe with a cooling surface of approximately 28 square feet. The vapor outlet from the condenser was 4" IPS pipe. Water was used as the coolant.

E. M. Shank provided much of the information on the hot pilot plant dissolver details of construction. A sketch of dissolver equipment in the Pilot Plant is shown in Figure 19.

HOT PILOT PLANT DISSOLVER

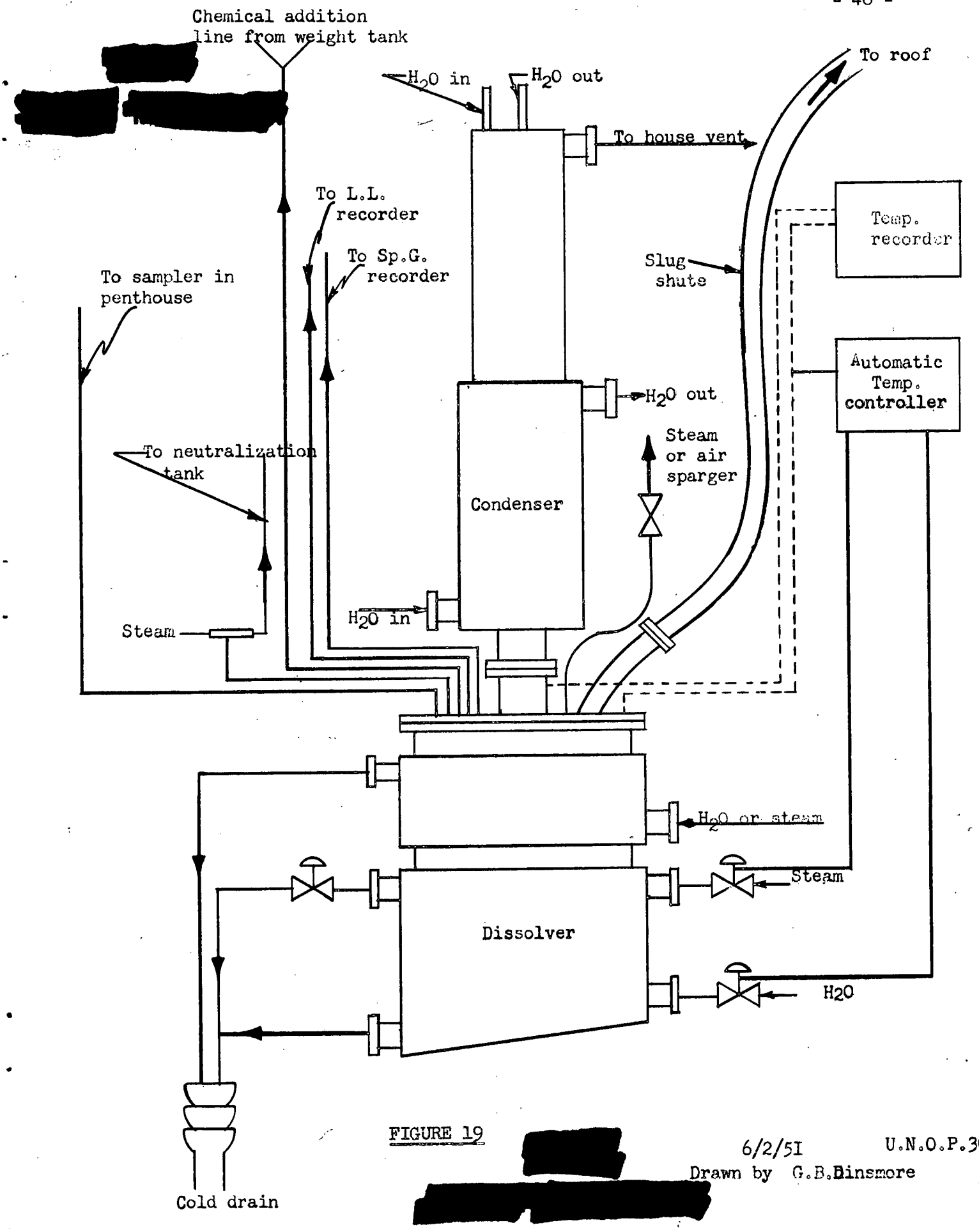


FIGURE 19

[redacted]

6/2/51 U.N.O.P.30

Drawn by G.B.Binsmore

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APPENDICES

Appendix "A" - Tabulated Runs

Appendix "B" - Data from HW 18353 Progress Report for June 1950 Chemical Research Section

Appendix "C" - Pilot Plant Runs

[redacted]

[redacted]

APPENDIX A

TABULATED RUN RESULTS

Run No.	Slug Type	No. of Slugs	Method of Acid Addition	Initial HNO_3 N	Final H^+ N	Moles HNO_3 / Mole Alloy	Hg^{++} %	Added Chemicals Chemical M	Coat Removal	Run Time Hours	Boil-down time Hours	Final Al Conc. M	Reflux Cond.	Oxygen to off-gas	% Dissolved	Remarks
1	Ext.	2	Cont 35 cc/min		2.02	4.75	2		none	7	none	0.81	Std.	none	84	1 hr digestion after all acid in
2	Ext.	2	Cont 35 cc/min		3.2	4.75	2		none	7	none	0.58	Std.	none	55	1 hr digestion after all acid in
3	Ext.	2	3 parts	1.1	1.57	4.35	2	$KMnO_4$ .003	none	9	none	0.90	Std.	none	85	1/5 HNO_3 1st hr, 1/5 HNO_3 2nd hr. 3/5 rest of run
4	Ext.	2	3 parts	1.1	1.63	4.35	2	$KMnO_4$ .003	none	12	none	0.80	Std.	none	69	Same as Run 3
5	Ext.	2	All at start	8.65	3.00	4.8	2	$MnSO_4$ .003	none	10	none	2.03	Std.	none	76	Soln all added at start, Boiled for 10 hrs.
6	Ext.	2	All at start	6.70	0.78	3.75	2	$MnSO_4$ .009	none	10	none	1.67	Std.	none	78	Same as Run 5
7	Ext.	2	All at start	6.70	0.70	3.75	4	$MnSO_4$ .009	none	10	none	1.79	Std.	none	85	Same as Run 5, Hg added in 4 parts
8	Ext.	2	All at start	4.21	0.66	4.6	4	$MnSO_4$ .009	none	10	none	1.17	Std.	none	100	Same as Run 5, Hg added in 4 parts
9	Ext.	2	All at start	4.21	0.30^B	4.0	3	$MnSO_4$ .009	none	8	none	1.13	Std.	none	100	Same as Run 5, Hg added in 3 parts
10	Ext.	2	All at start	4.21	0.06^B	4.0	2	H_2SO_4 .009	none	6	none	1.11	Std.	none	100	Same as Run 5, Hg added in 2 parts
11	Ext.	2	All at start	4.21	0.09^B	4.0	2		none	6	none	1.10	Std.	none	100	Same as Run 5, Hg added in 2 parts
12	Ext.	2	All at start	4.21	0.36^B	4.0	2		none	6	none	1.20	Std.	none	99	Same as Run 5, Hg added in 2 parts
13	Ext.	4	All at start	4.21	0.19	4.0	1.5		none	6		1.13	Std.	none	100	Same as Run 5, Hg added in 2 parts
14	Ext.	2	All at start	4.21	0.16^B	4.0	2		none	4	none	1.18	Std.	none	99	Same as Run 5, Hg added at start only
15	Ext.	2	All at start	5.68	0.20	4.0	2		none	6	none	1.41	Std.	none	93	Same as Run 5, Hg added at start only
16	Ext.	2	All at start	4.21	0.02^B	4.0	2		none	4	3	1.13	Std.	none	100	Same as Run 5, Hg added at start only
17	Ext.	2	All at start	4.21	0.02^B	4.0	2		none	4	1	1.25	Std.	none	99.5	Same as Run 5, Hg added at start only
18	Ext.	2	All at start	4.93	0.35	4.0	2		none	6		1.28	Std.	none	88	Same as Run 5, Hg added at start only
19	Ext.	2	All at start	4.93	0.02	4.0	2		none	4		1.30	Std.	none	99+	Same as Run 5, Hg added at start only
20	Ext.	2	All at start	4.93	0.23	4.0	2		none	4		1.33	Std. [redacted]	none	94	Same as Run 5, Hg added at start only

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APPENDIX A (Continued)

TABULATED RUN RESULTS

Run No.	Slug Type	No. of Slugs	Method of Acid Addition	Initial HNO_3 N	Final H^+ N	Moles HNO_3 Mole Alloy	Hg^{++} %	Added Chemicals Chemical M	Coat Removal	Run Time Hours	Boil-down time Hours	Final Al Conc. M	Reflux Cond.	Oxygen to off-gas	% Dissolved	Remarks
21	Ext.	2	All at start	4.21	0.08[B]	4.0	2		none	4		1.20	Std.	none	100	Same as Run 5, Hg added at start only, Bond agent added
22	Ext.	2	All at start	4.21	0.03[B]	4.0	2		none	4	1-1/2	1.21	Std.	none	100	Same as Run 5, Hg added at start only, Bond agent added
23	Ext.	2	All at start	4.21	0.49	4.0	1		none	4		1.07	Std.	none	88	Same as Run 5, Hg added at start only
24	Ext.	2	4 parts	4.45	2.58	4.0	2		none	4		1.49	Std.	none	66	HNO_3 added in 4 parts, Bond agent added
25	Ext.	2	All at start	4.21	0.08	4.0	2		none	4		1.25	Std.	none	95	Same as Run 14
26	Ext.	4	4 parts	4.45	2.50	4.0	2		none	3		1.40	Std.	none	69	HNO_3 added in 4 parts, Bond agent added
27	Ext. & Dip	4	All at start	4.21	1.05	4.0	2		none	4	2	0.99	Std.	none	85	Same as Run 14. Bond agent on slug
28	Ext. & Dip	2	All at start	4.21	.005	4.0	2		none	4	1-1/4	1.25	Std.	none	98	Same as Run 14, Bonding agent on slug
29	Ext. & Dip	2	All at start	4.21	.35	4.0	2		none	4	1-1/2	1.12	Std.	none	93	Same as Run 14, Bonding agent on slug
30	Cast & Dip	2	All at start	4.20	3.50	4.2	2		none	4		0.32	Std.	none	28	Same as Run 14, Bonding agent on slug, cast slugs
31	Cast & Dipped	2	All at start	4.20	2.15	4.2	2		none	6		0.61	Std.	none	52	Same as Run 30, Bonded slugs
32	Cast & Dipped	2	All at start	4.20	1.14	4.2	2		none	10		0.92	Std.	none	66	Same as Run 30, Bonded slugs
33	Cast & Dipped	2	In parts	3.18	2.65	4.1	2		none	6		0.45	Std.	none	38	Acid & $Hg(NO_3)_2$ added in parts
34	Cast & Dipped	2	In parts	4.43	2.33	4.1	2	H_2SO_4 .009	Yes	6		0.65	Std.	none	52	1 hr coat removal using dilute HNO_3, then bulk of HNO_3
35	Cast & Dipped	2	After coat Rem.	5.0	2.25	4.1	2		Yes	5		0.78	Std.	none	63	1 hr coat removal using dilute HNO_3, then bulk of HNO_3

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APPENDIX A (Continued)

TABULATED RUN RESULTS

Run No.	Slug Type	No. of Slugs	Method of Acid Addition	Initial HNO_3 N	Final H^+ N	Moles HNO_3 Mole Alloy	Hg^{++} %	Added Chemicals Chemical M	Coat Removal	Run Time Hours	Boil-down time Hours	Final Al Conc. M	Reflux Cond.	Oxygen to off-gas	% Dissolved	Remarks
36	Cast & Dipped	2	All at start	4.20	1.08	4.1	10		none	6		0.89	Std.	none	76	Determination or effect of increased $Hg(NO_3)_2$
37	Cast & Dipped	2	After Coat Rem.	4.85	1.8	4.1	10		yes	5		0.92	Std.	none	66	1 hr coat removal using dilute HNO_3, then bulk of HNO_3
38	Cast & Dipped	2	After Coat Rem.	4.85	1.2	4.1	6		yes	6		0.90	Std.	none	73	1 hr coat removal using dilute HNO_3, then bulk of HNO_3
39	Cast & Dipped	2	After Coat Rem.	4.85	0.34	4.1	6		yes	11		1.1	Std.	none	83	1 hr coat removal using dilute HNO_3, then bulk of HNO_3
40	Cast & Dipped	2	After Coat Rem.	5.3	0.70	4.3	6		yes	10.5		0.96	Std.	none	94.3	1 hr coat removal using dilute HNO_3, then bulk of HNO_3
41	Cast & Dipped	2	After Coat Rem.	5.3	0.92	4.3	6		yes	10.0		0.89	Std.	none	85	1 hr coat removal using dilute HNO_3, then bulk of HNO_3
42	Cast & Dipped	2	After Coat Rem.	5.3	0.97	4.3	6	H_2SO_4 .009	yes	11.0		0.72	Std.	none	81	1 hr coat removal using dilute HNO_3, then bulk of HNO_3
43	Cast & Dipped	2	In 2 parts	5.3	1.73	5.35	20		yes	9.0		0.835	Std.	none	94	1 hr coat removal, then 4.3 Moles HNO_3/Mole alloy added, 1.05 M HNO_3/M @ 4 hr
44	Cast & Dipped	2	All in @ start	5.0	0.15	4.0	46		none	9.0		1.3	Std.	none	100	Determination of effect of increased $Hg(NO_3)_2$
45	Cast & Dipped	2	All in @ start	5.0	0.72	4.0	46		none	5		1.1	Std.	none	88	Same as Run 44 only shorter time
46	Cast & Dipped	2	All in @ start	5.0	0.5	4.0	46		none	6		1.16	Std.	none	86	Same as run 44 only shorter time
47	Cast & Dipped	2	All in @ start	5.0	0.1	4.0	46		none	9.0		1.16	Std.	none	98	Same as Run 44
48	Cast & Dipped	2	All in @ start	5.0	0.65	4.5	46		none	9.		1.1	Std.	none	100	Same as Run 44 with additional HNO_3
49	Cast & Dipped	2	All in @ start	5.0	0.55	4.5	46		none	8	3	1.17	Std.	none	100	Same as Run 48 with 3 hour boildown
50	Cast & Dipped	2	All in @ start	5.0	0.88	4.5	46		none	6		1.07	Std.	none	94.6	Same as Run 48 only shorter time

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APPENDIX A (Continued)

TABULATED RUN RESULTS

Run No.	Slug Type	No. of Slugs	Method of Acid Addition	Initial HNO_3 N	Final H^+ N	Moles HNO_3 Mole Alloy	Hg^{++} %	Added Chemicals Chemical M	Coat Removal	Run Time Hours	Boil-down time Hours	Final Al Conc. M	Reflux Cond.	Oxygen to off gas	% Dissolved	Remarks
51	Cast & Dipped	2	All in @ start	5.0	0.52	4.5	46		none	8	3	1.08	Std.	none	98	Same as Run 49
52	Cast & Dipped	2	All in @ start	5.0	0.58	4.5	46		none	8		1.12	Std.	none	99+	Same as Run 49 with no boildown
53	Cast & Dipped	2	All in @ start	5.0	0.95	4.5	5		none	8		1.03	Std.	none	86	Same as Run 52 with reduced Hg $(NO_3)_2$
54	1 cast 1 Ext. 1 Dipped	2	All in @ start	5.0	1.40	4.5	5		none	6		0.89	Std.	none	84	Extruded slug 100% dissolved cast slug 70% dissolved
55	Cast & Dipped	2	All in @ start	5.0	1.25	4.5	5		none	8		0.97	Std.	none	90	Cast slugs heated to 600°C & chilled in CO_2, CCl_4
56	1 cast 1 Ext.	2	All in @ start	5.0	0.53	4.3	5		none	10		0.77	Std.	none	84	Extruded slug 100% dissolved Cast slug 70% dissolved
57	Cast & Dipped	2	All in @ start	5.0	0.58	4.5	5		none	8		1.11	Std.	none	99	Heated to 600°C, Quenched & punched on one end
58	Cast & Dipped	2	All in @ start	5.0	0.48	4.5	35		none	8		1.05	Std.	none	95	Same as Run 52 with Reduced Hg $(NO_3)_2$
59	Cast & Dipped	2	All in @ start	5.0	0.41	4.5	57		none	8		1.1	Std.	none	99	Same as Run 52 with increased Hg $(NO_3)_2$
60	1 cast 1 ext.	2	All in @ start	4.2	0.16	4.1	2		none	7		0.94	Std.	none	100	Cold-rolled slugs 35% increase in length
61	Cast	2	All in @ start	4.2	0.53	4.1	2		none	7		0.94	Std.	none	88	Cold-rolled slugs 35% increase in length
62	Ext.	2	All in @ start	4.2	0.23	4.1	2		none	7		0.94	Std.	none	100	Cold-rolled slugs 35% increase in length
63	Cast & Dipped	2	In parts	7.95	3.2	4.7	2		none	7.5		1.33	Std.	none	42	Acid in by parts - increased normality
64	Cast & Dipped	2	In parts		2.3	4.0	2		none	9.5		1.20	Std.	none	54	Acid in By parts
65	Cast & Dipped	2	All at Start	4.2	1.03	4.0	2	KNO_3 1	none	8		0.85	Std.	none	58	Observation of effect of KNO_3

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APPENDIX A (Continued)

TABULATED RUN RESULTS

Run No.	Slug Type	No. of slugs	Method of Acid Addition	Initial HNO_3 N	Final H^+ N	Moles HNO_3 / Mole Alloy	Hg^{++} %	Added Chemicals Chemical M	Coat Removal	Run Time Hours	Boil-down time Hours	Final Al Conc. M	Reflux Cond.	Oxygen to off-gas	% Dissolved	Remarks
66	Cast & Dipped	2	All at start	4.35	0.73	4.0	2	$NaNO_3$ .008	none	8		0.92	Std.	none	77	Observation of effect of Na NO_3
67	Cast & Dipped	2	All at start	4.35	1.02	4.0	2	$NaNO_3$ .008	none	8		0.75	Std.	none	69	Same as Run 66
68	Cast & Dipped	2	All at start	4.8	0.29	4.0	5	$NaNO_3$ .0106	yes	11		1.05	Std.	none	78	Same as Run 66 with increased HNO_3 & $NaNO_3$ Molarities
69	Cast & Dipped	2	All at start	5.0	0.58	4.0	5	$NaNO_3$ .01	yes	10		1.13	Std.	none	81	Same as Run 68 with increased HNO_3 N
70	Cast & Dipped	2	All at start	6.00	0.3	4.35		$NaNO_3$ .02	yes	11		1.38	Std.	none	88	Same as Run 68 with increased HNO_3 N increased $NaNO_3$
71	Cast & Dipped	4	By parts	4.1	0.5	4.1	7		none	12		0.97	Std.	none	93	Dissolving sol. added in 3 steps with each step jetted out before next is added
72	Cast & Dipped	4	By parts	4.1	1.5	4.1	5		none	8		0.77	Std.	none	70	Dissolving Sol. added in 4 steps with each step jetted out before next is added
73	Cast & Dipped	4	By parts	4.1	0.65	4.1	10		none	12		0.92	Std.	none	88	Dissolving sol. added in 4 steps with each step jetted out before next is added
74	Cast & Dipped	4	By parts	4.5	0.50	3.25	3		none	12		1.00	Std.	none	68	Dissolving sol. & slugs added in 3 steps-each solution jetted out before next is added
75	Cast & Dipped	4	By parts	4.3	0.95	4.0	7		none	12		1.1	Std.	none	82	Dissolving sol added in 3 steps-with each solution jetted before next is added
76	Cast & Dipped	2	All @ start	5.0	0.41	4.3	3.3	$La(NO_3)_3$ to H_2O .001 $K NO_3$ .002 $La(NO_3)$ to H_2O .001	none	12		1.15	Std.	none		Studies for optimum conditions
77	Cast & Dipped	2	All @ start	5.0	1.2	4.3	3.3	$K NO_3$ .002	none	12		1.5	Std.	none		Studies for optimum conditions
78	Cast	2	All at start	5	2.03	4.0	2		none	8		0.7	Std.	none	54	Studies for optimum conditions
79	Cast	2	All at start	5	1.25	4.3	2		none	8		0.9	Std.	none	73	Studies for optimum conditions
80	Cast	2	All at start	5	.5	4.3	5		none	8		1.02	Std.	none	91	[redacted] for optimum conditions

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APPENDIX A (Continued)

TABULATED RUN RESULTS

Run No.	Slug Type	No. of Slugs	Method of Acid Addition	Initial HNO_3 N	Final H^+ N	Moles HNO_3 Mole Alloy	Hg^{++} %	Added Chemicals Chemical M	Coat Removal	Run Time Hours	Boil-down time Hours	Final Al Conc. M	Reflux Cond.	Oxygen to off gas	% Dissolved	Remarks
81	Cast	2	All @ start	5.0	1.2	4.35	4.6		none	8	none	1.1	Std.	none	94	Studies for finding optimum mole ratio & % Hg
82	Cast	2	All @ start	5.0	1.6	4.35	4.6		none	10	none	1.07	Std.	none	89	Studies for finding optimum mole ratio & % Hg
83	Cast	2	All @ start	5.0	0.98	4.35	5.6		none	8	none	1.09	Std.	none	98	Studies for finding optimum mole ratio & % Hg
84	Cast	2	All @ start	5.0	1.74	4.0	5.6		none	8	none	1.01	Std.	none	78	Studies for finding optimum mole ratio & % Hg
85	Cast	2	All @ start	5.0	1.3	4.15	5.6		none	8	none	1.07	Std.	none	90	Studies for finding optimum mole ratio & % Hg
86	Ext Re-melted & recast	2	All @ start	5.0	2.35	4.35	5.0		none	8	none	0.80	Std.	none	70	Studies for finding optimum mole ratio & % Hg
87	Ext Re-melted & recast	2	All @ start	5.0	1.65	4.2	5.0		none	8	none	0.99	Std.	none	82	Studies for finding optimum mole ratio & % Hg
88	2 partly dissolved	2	All @ start	5.0	2.30	4.35	5.0		none	8	1.75	0.83	Std.	none	77	Studies for finding optimum mole ratio & % Hg
89	Cast	2	All @ start	5.0	0.95	4.35	5.0		none	10	5	1.11	Std.	Before Cond	100	Studies with O_2 added
90	Cast	2	All @ start	5.0	1.30	4.35	5.35		none	9	6.5	1.01	Std.	Before Cond	100	Studies with O_2 added
91	Cast	2	All @ start	5.0	1.53	4.35	5.0		none	8	2.5	1.07	Std.	Before Cond	100	Studies with O_2 added
92	Cast	2	All @ start	5.0	0.95	4.35	5.0		none	8	2	1.18	Std.	Before Cond	100	Studies with O_2 added
93	Cast	2	All @ start	5.0	1.13	4.35	4.0		none	9	3.25	1.13	Std.	Before Cond	100	Studies with O_2 added
94	Cast	1	All @ start	5.0	0.99	4.35	4.0		none	8	1.25	1.07	Std.	Before Cond	100	Studies with O_2 added
95	Cast	2	All @ start	5.0	1.00	4.35	4.0		none	10	none	1.21	Std.	Before Cond	100	Studies with O_2 added
96	Cast	2	All @ start	5.0	1.17	4.35	4.0		none	8	none	1.13	Std.	Before Cond	100	Studies with O_2 added
97	Cast	2	All @ start	5.0	1.55	4.35	4.0		none	8	none	1.08	Std.	Before Cond	98	Studies with O_2 added
98	Cast	2	All @ start	5.0	1.11	4.1	4.0		none	8	none	1.18	Std.	Before Cond	96	Studies with O_2 added
99	Cast	1	All @ start	4.25	1.13	4.0	2.0		none	9	none	0.95	Std.	Before Cond	92	Studies with O_2 added
100	Cast	2	All @ start	5.0	2.3	4.0	4.0		none	8	none	0.87	Downdraft	Before Cond	70	Effect of downdraft cond. and finding optimum conditions

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APPENDIX A (Continued)

TABULATED RUN RESULTS

Run No.	Slug Type	No. of Slugs	Method of Acid Addition	Initial HNO_3 N	Final H^+ N	Moles HNO_3 / Mole Alloy	Hg^{++} 5	Added Chemicals Chemical M	Coat Removal	Run Time Hours	Boil-down time Hours	Final Al Conc. M	Reflux Cond.	Oxygen to off gas	% Dissolved	Remarks
101	Cast	2	All @ start	5.0	3.23	4.35	4.0		none	8	none	0.62	Downdraft	Before Cond	60	Effect of downdraft cond. and finding optimum conditions
102	Cast	2	All @ start	5.0	2.43	4.35	5.0		none	8	none	0.80	Downdraft	Before Cond	68	Effect of downdraft cond. and finding optimum conditions
103	Cast	2	All @ start	5.0	2.70	4.35	5.0		none	10	none	0.96	Downdraft	Before Cond	66	Effect of downdraft cond. and finding optimum conditions
104	Cast	2	All @ start	5.0	2.35	4.35	8.0		none	10	none	0.89	Downdraft	Before Cond	81	Effect of downdraft cond. and finding optimum conditions
105	Cast	2	All @ start	4.35	2.10	3.5	5.0		none	10	none	0.84	Downdraft	Before Cond	63	Effect of downdraft cond. and finding optimum conditions
106	Cast & Bonded	2	All @ start	4.35	2.0	3.5	5.0		none	10	none	0.72	Downdraft	Before Cond	61	Effect of downdraft cond. and finding optimum conditions
107	1 cast 1 ext	2	All @ start	4.5	1.4	4.0	8.0		none	10	none	0.99	Downdraft	Before Cond	83	Effect of downdraft cond. and finding optimum conditions
108	Cast	2	At @ start	4.0	1.3	4.0	8.0		none	10	none	0.97	Downdraft	Before Cond	87	Effect of downdraft cond. and finding optimum conditions
109	Cast	1	At @ start	4.5	0.93	4.0	5.0			10	none	1.16	Downdraft	Before Cond	89	Reflux Removed - H_2O added to maintain volume
110	Cast Pieces	4	All @ start	3.5	2.2	3.3	5.0			8	none	0.60	Downdraft	Before Cond	53	
111	Cast	1	All @ start	3.5	1.08	3.3	5.0			8	none	0.78	Downdraft	Before Cond	64	
112	Cast	1	All @ start	5.0	0.55	3.5	5.0			8	none	1.17	Downdraft	Before Cond	90	
113	Cast	1	All @ start	4.5		4.0	5.0			8	none		Downdraft	Before Cond	100	Pure Aluminum slugs not alloy
114	Cast	1	All @ start	4.5		4.0	5.0			4	none		Downdraft	Before Cond	100	Pure Aluminum slugs not alloy
115	Cast	1	All @ start	4.5		4.0	5.0			3	none		Downdraft	Before Cond	100	Pure Aluminum slugs not alloy
116	Cast	1	All at start	5.0	1.98	4.0	5.0			4	none	1.15	Downdraft	Before Cond	80	Reflux to drain - H_2O added to maintain volume
117	Cast	1	All at start	4.5	0.49	4.0	5.0			8	none	1.4	Downdraft	Before Cond	78	Reflux to drain - H_2O added to maintain volume
118	Cast	1	All at start	4.5	0.40	4.0	5.0			6	none	1.48	Downdraft	Before Cond	80	Reflux to drain - H_2O added to maintain volume

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APPENDIX A (Continued)

TABULATED RUN RESULTS

Run No.	Slug Type	No. of Slugs	Method of Acid Addition	Initial HNO_3 N	Final H^+ N	Moles HNO_3 / Mole Alloy	$Hg(NO_3)_2$ Added %	Chemicals Added Chemical M	Coat Removal	Run Time Hours	Boil-down time Hours	Final Al Conc. M	Reflux Cond.	Oxygen to off gas	% Dissolved	Remarks
119	Cast	1	All at start	4.5	0.95	4.0	5.0			8	none	1.2	Downdraft	Before condenser	91	Reflux to drain for 1-1/2 hours 1 L H_2O added
120	Cast	1	All at start	4.5	0.79	4.0	5.0			8	none	1.13	Downdraft	Before condenser	92	Reflux to drain for 1-1/2 hours 1 L H_2O added
121	Cast	1	All at start	4.5	1.24	4.0	5.0			8	none	1.18	Downdraft	Before condenser	87	Reflux to drain for 1 hour
122	Cast	1	All at start	4.5	0.50	4.0	5.0			10	none	1.32	Downdraft	Before condenser	99	Reflux to drain for 1 hour
123	Cast	1	All at start	4.5	0.63	4.0	5.0			10	none	1.30	Downdraft	none	95	Reflux to drain for 1 hour
124	Cast	1	All at start	4.5	0.50	4.0	5.0			10	none	1.13	Downdraft	Before condenser	97	Reflux to drain for 1 hour H_2O added to maintain volume
125	Cast	1	All at start	4.5	0.75	4.0	5.0			8	none	1.21	Downdraft	Before condenser	99	Reflux to drain for 1 hour
126	Cast	1	All at start	4.5	0.70	4.0	5.0			10	none	1.20	Downdraft	Before condenser	94	Reflux to drain for 1 hour
127	Cast	1	All at start	4.5	0.85	4.0	5.0			10	none	1.23	Downdraft	Before condenser	83	Reflux to drain for 1 hour
128	Cast	1	All at start	4.5	1.22	4.0	5.0			9	none	1.08	Downdraft	Before condenser	90	Reflux to drain for 1 hour
129	Cast	1	All at start	4.5	0.46	4.0	5.0			10	none	1.16	Downdraft	Before condenser	100	Reflux to drain for 1 hour
130	Cast	1	All at start	4.5	1.22	4.0	5.0			10	none	1.09	Downdraft	Before condenser	100	Reflux collected for 1 hour added back after 3rd hour
131	Cast	1	All at start	4.5	1.36	4.0	5.0			10	none	1.08	Downdraft	Before condenser	83	Reflux collected for 1 hour added back after 3rd hour
132	Cast	1	All at start	4.5	0.89	4.0	5.0			10	none	1.21	Downdraft	Before condenser	98	Reflux collected for 1 hour added back after 3rd hour
133	Cast	1	All at start	4.5	0.75	4.0	5.0			10	none	1.21	Downdraft	Before condenser	97	Reflux collected for 1 hour added back after 3rd hour
134	Cast	1	All at start	4.5	0.90	4.0	5.0			10	none	1.21	Downdraft	Before condenser	100	Reflux collected for 1 hour added back after 3rd hour
135	Cast	1	All at start	4.5	0.89	4.0	5.0			10	none	1.21	Downdraft	Before condenser	96	Reflux collected for 1 hour added back after 3rd hour
136	Cast	1	All at start	4.5	1.19	4.0	5.0			10	none	1.16	Downdraft	Before condenser	93	Reflux collected for 1 hour added back after 3rd hour

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APPENDIX A (Continued)

TABULATED RUN RESULTS

Run No.	Slug Type	No. of Slugs	Method of Acid Addition	Initial HNO_3 N	Final H N	Moles HNO_3 / Mole Alloy	Hg^{++} %	Added Chemicals Chemical M	Run Time Hours	Boil-down time Hours	Final Al Conc. M	Reflux Cond.	Oxygen to off gas	% Dissolved	Remarks
137	Cast Bonded	1	Majority @ start Rest @ 3rd hour	4.75	1.22	4.0	5.0		10	none	1.13	Downdraft	Before Cond	94	Procedure 1 3.6 moles/mole HNO_3 at start 0.4 moles/mole @ 3rd hour
138	Cast Bonded	1	Majority @ start Rest @ 3rd hour	4.75	1.25	4.0	5.0		10	none	1.20	Downdraft	Before Cond	96	Procedure 1 3.6 moles/molt HNO_3 at start 0.4 moles/mole @ 3rd hour
139	Cast Bonded	1	Majority @ start Rest @ 3rd hour	4.75	1.45	4.0	5.0		10	none	1.20	Downdraft	Before Cond	94	Procedure 1 3.6 moles/mole HNO_3 at start 0.4 moles/mole @ 3rd hour
140	Cast Bonded	1	Majority @ start Rest @ 3rd hour	4.75	0.95	4.0	5.0		10	none	1.25	Downdraft	Before Cond	98	Procedure 1 3.6 moles/mole HNO_3 at start 0.4 moles/mole @ 3rd hour
141	Cast Bonded	1	Majority @ start Rest @ 3rd hour	4.75	0.95	4.0	5.0		10	none	1.31	Downdraft	Before Cond	99	Procedure 1 3.6 moles/mole HNO_3 at start 0.4 moles/mole @ 3rd hour
142	Cast Bonded	1	Majority @ start Rest @ 3rd hour	4.75	0.73	4.0	5.0		10	none	1.26	Downdraft	Before Cond	100	Procedure 1 3.6 moles/mole HNO_3 at start 0.4 moles/mole @ 3rd hour
143	Cast Bonded	1	Majority @ start Rest @ 3rd hour	4.75	0.85	4.0	5.0		10	none	1.23	Downdraft	Before Cond	97	Procedure 1 3.6 moles/mole HNO_3 at start 0.4 moles/mole @ 3rd hour
144	Cast Bonded	2	Majority @ start Rest @ 3rd hour	4.75	1.03	4.0	5.0		10	none	1.10	Downdraft	Before Cond	91	Procedure 1 3.6 moles/mole HNO_3 at start 0.4 moles/mole @ 3rd hour
145	Cast Bonded	2	Majority @ start Rest @ 3rd hour	4.75	1.16	4.0	5.0		10	none	1.14	Downdraft	Before Cond	88	Procedure 1 3.6 moles/mole HNO_3 at start 0.4 moles/mole @ 3rd hour
146	Cast Bonded	2	Majority @ start Rest @ 3rd hour	4.75	1.04	4.0	5.0		10	none	1.16	Downdraft	Before Cond	98	Procedure 1 3.6 moles/mole HNO_3 at start 0.4 moles/mole @ 3rd hour
147	Cast Bonded	2	Majority @ start Rest @ 3rd hour	4.75	1.16	4.0	5.0		10	none	1.20	Downdraft	Before Cond	93	Procedure 1 3.6 moles/mole HNO_3 at start 0.4 moles/mole @ 3rd hour
148	Cast Bonded	1	Majority @ start Rest @ 3rd hour	4.75	0.98	4.0	5.0		10	none	1.20	Downdraft	Before Cond	99	Procedure 1 3.6 moles/mole HNO_3 at start 0.4 moles/mole @ 3rd hour
149	Cast Bonded	2	All @ start	4.75	0.43	4.35	5.0		10	none	1.08	Downdraft	Before Cond	97	All reflux removed cold H_2O added to hold volume
150	Cast Bonded	2	All @ start	4.75	0.58	4.35	5.0		10	none	1.09	Downdraft	Before Cond	100	All reflux removed cold H_2O added to hold volume

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APPENDIX A (Continued)

TABULATED RUN RESULTS

Run No.	Slug Type	No. of Slugs	Method of Acid Addition	Initial HNO_3 N	Final H N	Moles HNO_3 / Mole Alloy	Hg %	Added Chemicals Chemical M	Run Time Hours	Boil-down time Hours	Final Al Conc. M	Reflux Cond.	Oxygen to off gas	% Dissolved	Remarks
151	Cast Bonded	2	All at start	4.75	0.35	4.0	5.0		10	none	1.00	Downdraft	Before Cond	90	All reflux removed cold H_2O added to hold volume
152	Cast Bonded	2	All @ start	4.75	1.10	4.0	5.0		10	none	1.18	Downdraft	Before Cond	92	All reflux removed cold H_2O added to hold volume
153	Cast Bonded	2	All @ start	4.75	0.31	4.35	5.0		10	none	1.21	Downdraft	Before Cond	88	All reflux removed cold H_2O added to hold volume
154	Cast Bonded	2	All @ start	4.75	0.68	4.35	5.0		10	none	1.01	Downdraft	Before Cond	91	All reflux removed cold H_2O added to hold volume
155	Cast Bonded	2	All @ start	4.75	0.43	4.35	5.0		10	none	1.22	Downdraft	Before Cond	93	All reflux removed cold H_2O added to hold volume
156	Cast Bonded	2	All @ start	4.75	0.60	4.4	5.0		10	none	1.01	Downdraft	Before Cond	93	All reflux removed cold H_2O added to hold volume
157	Cast Bonded	2	All @ start	4.75	0.4	4.35	5.0		10	none	1.08	Downdraft	Before Cond	97	Same as Run 149, Procedure 2 with hot H_2O addition
158	Cast Bonded	2	All @ start	4.75	0.47	4.35	5.0		10	none	1.12	Downdraft	Before Cond	98	Same as Run 149, Procedure 2 with hot H_2O addition
159	Cast Bonded	2	All @ start	4.75	0.45	4.35	5.0		10	none	1.06	Downdraft	Before Cond	92	Same as Run 149, Procedure 2 with hot H_2O addition
160	Cast Bonded	2	All @ start	4.75	0.17	4.35	5.0		10	none	1.06	Downdraft	Before Cond	97	Same as Run 149, Procedure 2 with hot H_2O addition
161	Cast Bonded	2	All @ start	4.75	0.44	4.5	5.0		10	none	0.99	Downdraft	Before Cond	95	Same as Run 149, Procedure 2 with hot H_2O addition
162	Cast Bonded	2	4 M/M @ start .35 M/M 3rd hr	4.75	0.70	4.35	5.0		10	none	1.00	Downdraft	Before Cond	86	Same as run 157 only acid added in parts
163	Cast Bonded	2	All @ start	4.75	0.55	4.35	5.0		10	none	1.06	Downdraft	Before Cond	90	Same as run 157
164	Cast Bonded	2	All @ start	4.75	0.15	4.35	5.0		10	none	1.0	Downdraft	Before Cond	86	Same as run 157 only partly dissolved slugs

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Appendix B

Data from HW 18353 Progress Report for June 1950 Chemical Research Section
Page 28 Table XIX

To: R. H. Beaton

By: O. F. Hill and F. J. Leitz

$Al(NO_3)_3$ M	HNO_3 M	Dissolution Rate Mg/hr./CM^2	$Al(NO_3)_3$ M	HNO_3 M	Dissolution Rate Mg/hr./CM^2
0	0.0	19	0.5	2.0	803
0	2.0	1160	0.5	2.5	813
0	3.0	1811	0.5	3.0	906
0	3.5	1872	0.5	3.25	1159
0	4.0	1573	0.5	3.5	1254
0	4.5	1990	0.5	3.75	997
0	5.0	1800	0.5	4.0	1241
0	5.5	1500	0.5	4.5	1975
0	6.0	1355	0.5	5.0	860
0	7.0	646			
0	8.0	642	1.0	0.5	343
0	9.0	310	1.0	1.0	487
0	9.5	345	1.0	1.5	547
0	13.3	133	1.0	1.75	681
			1.0	2.0	532
			1.0	2.25	702
			1.0	2.5	460
			1.0	3.0	342
			1.0	4.0	189

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Appendix C

Pilot Plant Runs

A series of 9 runs were made under the supervision of Mr. E. M. Shank, Pilot Plant Section, Chemical Technology Division, ORNL, in the hot pilot plant dissolving equipment for purposes of testing one of the recommended procedures at a scale of approximately 1/2 of a production type run. Since at that time the Pilot Plant was using an up-draft condenser, procedure 3 was chosen to be demonstrated as feasible at 1/2 production scale.

There was no means of visual inspection of slugs at the end of the dissolvings so the extent of dissolving was determined by dissolver solution analysis.

Based upon aluminum analysis these nine runs gave an average of 97% dissolved per 10 hour run as is shown in Table "C". With entrainment separators and a more efficient condenser reducing the solution loss from the dissolver, these dissolvings would probably reach 100% completion. Two clean-out runs were made to remove any slug residue from the dissolver. By analysis these runs picked up very little uranium or aluminum (450 gms) indicating near complete dissolvings in prior runs.

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TABLE "C"

PILOT PLANT RUNS

Run No.	No. of Slugs	Wt. of Slugs GMS	Wt. of Al	Mols H^+ / Mol Alloy	Initial H^+ (N)	Final H^+ (N)	Initial Vol (L)	Final Vol Liters	% Hg	Final Sol GMS. Al / L	GMS Al Dissolved	% of Al Dissolved	Total GMS of Aluminum Dissolved	Total Wt. of Aluminum Charged GMS	Total % of Al Dissolved	
IDOB-1	8	4485	4140	4.35	4.75	.03	142	127	5.0	29.7	3770	91.0	3770	4140	91.0	
IDOB-2	8	4500	4163	4.35	4.75	0.42	150	120	5.0	34.2	4088	98.1	7858	8303	94.5	
IDOB-3	8	4492	4155	4.35	4.75	0.04	142	94	5.0	40.05	3740	90.0	11598	12458	93.0	
IDOB-4	8	4498	4460	4.35	4.75	0.96	190	258	5.0	17.10	4413	107.2	16011	16618	96.8	Water added to keep up vol
IDOB-CO-1*	none					5.15		113	5.0	0.765	86.7		16098	16618	97.0	
IDOB-5	8	4500	4163	4.35	4.75	0.30	142	209	5.0	20.25	4235	101.7	20333	20781	97.8	Water added to keep up vol
IDOB-6	8	4519	4180	4.35	4.75	0.265	150	184	5.0	21.38	3925	93.9	24258	24961	97.2	Water added to keep up vol
IDOB-7	8	4490	4153	4.35	4.75	0.075	142	217	5.0	18.9	4090	98.3	28348	29114	97.5	Water added to keep up vol.
IDOB-8	8	4479	4143	4.35	4.75	.18	147	186	5.0	21.27	3958	95.5	32306	33257	97.2	Water added to keep up vol
IDOB-9	8	4499	4162	4.35	4.75	.0813	142	216	5.0	17.78	3983	95.7	36289	37419	97.2	Water added to keep up vol
IDOB-CO-2*	none							185	5.0	1.97	364		36653	37419	97.5	

*Clean out runs

[redacted]

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D. L. Foster

E. O. Nurmi

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