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PROCEDURES AND RESULTS OF THE JANUARY 1964 HEAVY ISOTOPES PRODUCTION CAVE RUN

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UNIVERSITY OF CALIFORNIA

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PROCEDURES AND RESULTS OF THE

JANUARY 1964 HEAVY ISOTOPES PRODUCTION CAVE RUN

J. L. Green, J. T. Haley and B. B. Cunningham

SEP 1965

## I. INTRODUCTION

During the period from January 14 to July 15, processing and analytical work was carried out on the most recent phase of the UCRL (Berkeley) heavy isotope production program. The work was done in the water cave facilities which are operated under the direction of P. W. Howe and J. T. Haley. The processing chemistry was carried out under the direction of Dr. B. B. Cunningham and J. L. Green.

## II. SLUG DESCRIPTION AND IRRADIATION SCHEDULE

The slugs consisted of an outer aluminum jacket  $5/8$  inch O.D. and 4 inches long. An internal plug containing 2.5 gm  $\text{AmO}_2$  mixed with Al powder was pressed into an internal cavity  $3/8$  inch in diameter and  $3-1/4$  inches long. Two identical slugs, UCRL 29-1 and 29-2 were then placed in the L-45-center position in the Materials Testing Reactor and irradiated for approximately 5 years to an integrated flux of  $6 \times 10^{22}$ .

The average composition of each slug at the end of irradiation is shown in Table I.

Table I. Slug Composition.

<u>Actinides</u>	<u>Fission Products (mg)</u>	
Pu; few mg	Zr; 550	Y; 115
Am <sup>241</sup> ; ca. 30 mg	Mo; 305	Rh; 93
Cm <sup>244</sup> ; 215 mg	Ru; 230	Nd; 70
Bk <sup>249</sup> ; 6-7 $\mu\text{g}$	Ce; 170	Pm; 60
Cf <sup>252</sup> ; 33 $\mu\text{gm}$	Te; 155	
Es <sup>253</sup> ; 200 $\mu\text{g}$	Sr; 125	

The actinide compositions are actual while the fission product compositions are computed.

The results of mass analyses run on the Cm and Cf fractions are shown in Tables II and III, respectively.

Table II. Cm Mass Analysis (Cm VI-609).

$$\frac{245}{244} = 0.01259 \pm 0.00027$$

$$\frac{246}{244} = 0.1571 \pm 0.0017$$

$$\frac{247}{244} = 0.00443 \pm 0.00013$$

$$\frac{248}{244} = 0.00525 \pm 0.002$$

242 & 243 - interference from other elements.

Table III. Cf Mass Analysis (CfVI-605).

$$248/252 \leq 0.0017$$

$$249/252 \leq 0.0231 \pm 0.003$$

$$250/252 = 0.193_6 \pm 0.002_5$$

$$251/252 = 0.0560 \pm 0.0008$$

$$253/252 \leq 0.0025_8 \pm 0.0001$$

$$254/252 \leq 0.0001$$

Bk<sup>249</sup>, Cm<sup>248</sup> and Es<sup>253</sup> all present

### III. PROCESSING

A graphic description of the chemistry involved is included in the form of a simplified flow sheet. The general features of the flow sheet should be self-evident; however, a short discussion of the difficulties encountered and a brief rationale of some of the major blocks of the process are in order.

#### A. Dissolution

Due to the large amounts of Al present in the slugs (~50 gm each), it was decided to dissolve the total slug in NaOH - NaNO<sub>3</sub> solution. This technique would presumably have two major virtues; 1) the elimination of Al in the form of soluble aluminates and 2) the minimization of H<sub>2</sub> evolution which would have otherwise constituted an explosion hazard.

The dissolution was begun using this approach; however, it shortly became apparent that the cores were not readily soluble in the basic medium. It was, therefore, necessary to alter the initial flow sheet to accommodate the HCl dissolution of the cores. Because of the large amount of Al in these biscuits, it was necessary to go through subsequent hydroxide precipitations. In order to remove residual Al and the large amounts of KCl generated in the neutralizations, it was further necessary to carry out extensive washing procedures. These deviations from the initial procedure introduced considerable unexpected and admittedly undesirable extra handling of the activity. It is also possible that the acid-base conversions may be undesirable from the standpoint of Bk recovery due to the possible loss of material as polymeric species either as colloids or by sorption on glass vessels. It should be noted that berkelium, unlike curium and the transberkelium actinides is readily oxidized to the +4 state in alkaline solution.

One further point of importance, in this area, is the solid residue from dissolution. It has been noted, both in this run and in the past, that

a jet black solid material of unknown composition appeared as an insoluble residue after dissolution. This material was isolated and washed thoroughly and submitted for spectrographic analysis. The material was found to consist of Pd, Ru, Mo, and Al in the ratios 1 to 1 to 0.2 to 0.2 respectively. The Pd and Ru are presumably pure fission products present either as oxides or as metals (reduced by Al during dissolution). The Mo occurs as a fission product and also as inactive molybdenum compounds used as die lubricants during the preparation of the capsules.

#### B. Lithium Chloride Columns

A standard procedure used for the separation of the fission products and the bulk of the Cm from the transcurium elements is elution from Dowex-1 anion exchange resin using 10 M LiCl.

It has been noted repeatedly in the past and in the first two LiCl columns in this run, that the speed of elution of the various activities was much more rapid than expected and that banding of the activities, as evidenced by the formation of the characteristic glows of the rare earths and of Cm, occurred very late in the elution. This was noted particularly in the case of the second LiCl column which broke through very rapidly and yielded very poor separations. The reason for this was discovered to be the fact that the standard 12 mm x 14 cm columns used for the separation were very badly overloaded (~50-90%). This condition readily explains the peculiarities noted above. The fact that separations have even been achieved under these conditions speaks very highly of the efficiency of this separation.

Initially, there was some concern regarding the possible oxidation of Bk on the columns; therefore, the eluent solution was made 0.05 M in  $\text{NH}_3\text{OH}\cdot\text{HCl}$ . It is now felt that such additions are unnecessary. Crude tracer studies using Ce indicated that in soln's saturated in  $\text{H}_2\text{O}_2$  or  $\text{Cl}_2$ , the observed  $K_D$ 's were



appropriate to  $Ce^{+3}$ . It was feared that the presence of the ion exchange resin might shift the oxidation potential far enough to cause difficulty; however, as mentioned above, this does not appear to be the case. In acid solution, of course,  $H_2O_2$  stabilize  $Bk^{+3}$  and presumably the  $H_2O_2$  produced by the radiolysis of  $H_2O$  would act in this capacity in the column, assuming that the effective oxidation potentials are not greatly affected by the resin.

A white insoluble material appeared during feed preparations which on spectrographic analysis proved to be predominately Al. This material was separated out, washed and retained for further analysis. It has been suggested that this phase is a corundum like material resulting from the effects of intense radiation on Al in solution.

### C. Butyrate Columns

The intra-actinide separations were, as usual, carried out by elution from ammonium form Dowex-50 X 12 using 0.175 M  $\alpha$ -hydroxyisobutyric acid at pH = 4.9. This particular step caused a great deal of confusion, particularly with respect to Bk recovery. The Es-Cf separation was not especially good, but it was adequate, and the Cf-Cm separation was quite good; however, when the so-called Bk fraction was analyzed, it was found to contain only 10-15% of the anticipated yield. After a very extensive analytical program, it was shown that the remainder of the Bk was in the Cf peak and was temporarily lost on a Cf target prepared for accelerator bombardment. The cause of this difficulty is still not particularly obvious to the authors; although, it is known that "But" columns behave rather badly if the feed solutions are not very clean or if a significant amount of the resin is converted to the hydrogen form during loading. At any rate, it was at this point that it was realized that the Bk chemistry had gone disastrously astray.

#### D. Berkelium Recovery

The principal difficulty involved in processing Bk under these conditions, is that it is very difficult to make rapid radioassays.  $\text{Bk}^{249}$  is a very soft  $\beta$  emitter (115 keV) with a half-life of 290 days. It is virtually impossible to  $\beta$  count the material because of the tremendous number of  $\beta$  emitters present as potential contaminants.  $\text{Bk}^{249}$  does emit an  $\alpha$ -particle (5.48 MeV) but the branching ratio is so small ( $2 \times 10^{-5}$ ) that it is very difficult to separate the Bk  $\alpha$ 's from  $\alpha$ -emitting contaminants unless the sample is quite pure. The technique developed for use involved extensive  $\beta$  decontamination using LiCl columns and  $\alpha$  decontamination using the extraction of  $\text{Bk}^{+4}$  into HDEHP from an oxidizing medium. The Bk purified in this manner was then  $\alpha$  counted for an appropriate length of time to determine the growth rate of  $\text{Cf}^{249}$   $\alpha$ 's into the sample. This information allows the calculation of the amount of  $\text{Bk}^{249}$  on the plate. This method is relatively fool-proof; although very extensive  $\alpha$  decontamination is necessary to make the growth figures statistically significant.

This technique had not been developed at the time of the first "But" column; therefore, there was, at that time, no information whatever regarding the amount of Bk in the feed to that column. Nothing had been thrown away during previous processing except glassware, which had been carefully rinsed before it was discarded; therefore, it was possible to evaluate potential losses.

Each and every residue generated during processing was analyzed for Bk before disposal with the exception of ion exchange resin used after the Zr pickup column, glassware, and a few solid residues associated with the target preparations which, except for the glass, are probably not significant since a material balance can be made across their point of origin. The results of these analyses are presented both on the flow sheet and in Table IV. A

brief description of the analytical technique is shown in Fig. II. The significance of these results will be discussed later. Suffice it to say, at this point, that virtually all of the Bk which reached the initial LiCl column feed (the earliest available analytical point) was eventually recovered or accounted for.

#### E. Material Balance

Due to the large amount of analytical work carried out, it is possible to make overall material balances with considerable confidence. In order to report yields in terms of mass, it is necessary to choose a set of half-lives for the materials of interest. The data used for the calculation of this run are shown in Table V. These half-lives are the presently accepted values excepted in the case of Cf<sup>252</sup>. The most recent value for the Cf<sup>252</sup> half-life is  $2.46 \pm 0.004$  yr and the fission to  $\alpha$  ratio is  $31.3 \pm 0.2$ . To remain consistent through the run, 2.55 yrs was used in this work; although, decay corrections were made using the more accurate value.

Major errors are

1. Accuracy of the volume measurement on the sampled solution.
2. Dilution and micropipetting accuracy.
3. Counting statistics.

The magnitudes of the first two errors are not known to any accuracy. The limits shown in Table VI are 62% confidence counting limits except in the cases noted. Probably, a reasonable limit for overall accuracy should be set at 5%; although, the internal consistence of the numbers is better than this would indicate.

The case of the Bk assays, however, differs in that, in some cases, considerable chemistry was necessary in preparing satisfactory growth plates. These results, therefore, contain an additional uncertainty involving chemical

Table IV. Bk Assay Summary

	Bk on 1/14 ( $\mu\text{gm}$ )	Assay Result ( $\mu\text{gm}$ )	Date
Supers	0.20	0.16	4/7
KB 3*	7.5	7.0	3/12
Cinders	0	0	4/27
KB 21*	2.36	1.62	6/14
KB 25*	0.86	0.60	6/4
White Salts	0.04	0.03	6/8
KB 50*	10.7	10.1	3/12
Rare Earths	0.05	0.04	4/13
Zr	0	0	6/3
Amon. Resin	0	0	4/28
Assay 82	10.7	10.1	3/12
Assay 86	14.4 $\pm$ 1	13.0	3/26
Cone 32	1.45	1.36	3/12
CmI	0	0	3/23
Latimer Law	0.13	0.10	4/13
Combined Bk	10.1 $\pm$ 2%	8.05	4/16
CmII	0	0	3/23
Cf Tail Gatti	0	0	4/10

\* KB (Kitty bucket) - relatively large analytical samples taken from LiCl column feed solutions.

Table V. Half-Lives Used for Calculations

	$T_{1/2}$	c/min/ $\mu$ gm(50%)
Cm <sup>244</sup> $\alpha$	18 yr	$9.1 \times 10^7$
Cf <sup>252</sup> $\alpha$	2.55 yr*	$6.2 \times 10^8$
f	80 yr	$3.95 \times 10^7$
Bk <sup>249</sup> $\beta$	290 d	
Cf <sup>249</sup> $\alpha$	360 yrs	$4.4 \times 10^6$
Es <sup>253</sup> $\alpha$	20 d	$2.9 \times 10^{10}$

\* 2.64 yr used for decay corrections.

yield. The only portion of the procedure in which the yield has been checked is the extraction-scrub-strip cycle. Preliminary work using a Ce tracer indicated a yield of ~85% when six scrub strips were used. Losses this high are not consistent with  $K_D$  information available; therefore, the losses are probably mechanical. Mechanical losses in batch extractions are typically variable and difficult to reproduce; therefore, this figure should probably not be regarded as firm.

In the case of Cm<sup>244</sup>, the results indicate excellent internal consistency, very low losses and a total output of 429 mg Cm<sup>244</sup>. A yield of 405 mg of Es<sup>253</sup> was realized and no losses are apparent although the first analysis was fairly late in the process.

The Cf<sup>252</sup> results are self-consistent to within counting statistics. In addition to this, all residues were neutron counted prior to elimination; therefore, it is possible to state that Cf<sup>252</sup> losses up to the first target

Table VI. Material Balance Summary

	$Cm^{244}$ (mg)	$Bk^{249}$ ( $\mu$ gm)	$Cf^{252}$ ( $\mu$ gm)	$Es^{253}$ (ng)
LiClI Feed	210 $\pm$ 2	7.5	32.3 $\pm$ 0.5 <sup>c</sup>	-
LiClIII Feed	221 $\pm$ 2	3.3	33.7 $\pm$ 0.5 <sup>c</sup>	-
LiClVIII Feed	429 $\pm$ 4	10.7	71.8 $\pm$ 4	-
ButI Feed	4	14.4 (10.7) <sup>a</sup>	-	455
ButI Out	-	1.5	67	405
Other	-	8.6 <sup>b</sup>	-	-
Total Out	429 $\pm$ 4	10.5 $\pm$ 0.2	66 $\pm$ 0.7	405

- = unassayed

<sup>a</sup>Assay 86 = 14.4 It would appear that the result of assay 86 (14.4  $\mu$ gm) was too high. 10.7 checks other points in the material balance.  
Assay 82 = 10.7 See Bk summary.

<sup>b</sup>By difference

<sup>c</sup>By  $f/\alpha$  by PHA + Total  $\alpha$  on Low Geo. counter.

Note: limits listed are 62% confidence counting limits with the exception of Total Bk Out in which case the limit is overall confidence.

preparation were no more than 2-3  $\mu$ gm, i.e., less than 3-4%. The total output of  $Cf^{252}$  up to the first target preparation was 66  $\mu$ gm.

In the case of  $Bk^{249}$ , the internal consistency of the analyses through the process is surprisingly good. Since all residues were assayed, with the exception of glassware, it is possible to sum inputs and outputs over the entire process. The information is shown in Table VII.

Table VII. Bk Material Balance

<u>Analysis</u>	<u>Input</u>	<u>Output</u>
Hydroxide Sup'n	0.2	0.2
KB 3	7.5	
KB 21	2.4	
KB 25	0.9	
Salts and Rare Earths		0.1
Latimer Law		0.1
Combined Bk	<u>          </u>	<u>10.1</u>
	11.0	10.5

It is known that the output number is good to within only a few percent; therefore, this may be regarded as a minimum input figure. Because of the chemical yield problem, however, the sum of the inputs may be low by as much as ~15%. This leads to the conclusion that the input to the LiCl column based on the analytical results was between 10.3  $\mu\text{gm}$  and 12.5  $\mu\text{gm}$  and that the output was 10.3  $\mu\text{gm}$  Bk<sup>249</sup>.

## IV. DISCUSSION OF RESULTS

The results involving Cm, Cf, and Es appear straightforward and require no further discussion. The implications of Bk yields; however, deserve additional attention. Considerable confusion exists regarding the Bk-Cf ratio that should be expected from irradiations of the type involved here. This information is essential for the estimation of the chemical yield through a process and for the evaluation of difficulties involved in specific areas of a flow sheet. Data is locally available for several previous runs which allow somewhat more practical estimates than have been available in the past. In order to compare Bk/Cf ratios between runs it is desirable to present the data in terms of an index which is independent of a choice of a set of half-lives. Such an index is the ratio of the total growth rate of Cf<sup>249</sup> in the Bk in alpha counts per minute per day to the total fission count rate in the Cf<sup>252</sup> in fission counts per minute. This comparison is presented in Table VIII.

The first three entries are in reasonable agreement and indicate a growth/fission ratio of  $6-7 \times 10^{-5}$ . These data appear to be more or less independent of starting material and irradiation time; therefore, it will be assumed that they indicate the actual Bk content of the undissolved slugs. This being the case, one is forced to conclude that losses of ~15% occurred on slug 29-1 and almost 50% on slug 29-2. From the analytical data available for this run, it appears certain that such losses must have occurred prior to the first LiCl columns since material balances beyond this point are internally consistent to a maximum of 10-15%. Due to the fact that all head end residues with the exception of glassware were analyzed, it is necessary to conclude that either the loss involved the glassware or that one of the analyses failed. A positive choice between these alternatives is not possible at present.

Since there is no particular reason to doubt the assays, it is suggested that the head end procedure be altered. It is known that in basic solution Ce



Table VIII. Bk<sup>249</sup>-Cf<sup>252</sup> Ratio Estimation from Previous Runs

Date	Run	Starting Material	Irradiation Time	nvt	Growth-Fission
3/64	Livermore 3/64	-	-	-	$6.7 \times 10^{-5}$
5/59	Berkeley 5/59*	70mgCm <sup>244</sup> , 110mgPu <sup>242</sup>	14 mo	$1.4 \times 10^{21}$	$6.4 \times 10^{-5}$
/58	Berkeley /58*	192mgPu <sup>242</sup> , 85mgCm <sup>244</sup> 48mgAm <sup>243</sup>	~3 yr	~10 <sup>22</sup>	$7.5 \times 10^{-5}$
1/64	This run-overall		5 yr	$6 \times 10^{22}$	$3.9 \times 10^{-5}$
	Slug 29-1	2.5gmAm <sup>241</sup>			$5.2 \times 10^{-5}$
	Slug 29-2	2.5gmAm <sup>241</sup>			$2.7 \times 10^{-5}$

\*Several non-identical slugs processed.

and, therefore, Bk are easily oxidized to the  $+4$  ion. The  $+4$  hydroxides are highly susceptible to the formation of polymeric colloids, particularly under near neutral conditions. Many successful hydroxide carries were performed during later processing; therefore, it would appear that a direct hydroxide strike efficiently carries Bk. During the actual dissolution, however, the hydroxides are precipitated only around the slug and the supernatants are never efficiently "swept". It is also possible that the hydroxide concentration immediately adjacent to the dissolving slug could be considerably depressed. All things considered, it appears that peptization at this point followed by sorbtion on the many glass vessels which the supernatant contacted would explain all of the observations. This being the case, it is highly recommended that, in the future, the dissolution be carried out in acid and be followed by direct hydroxide strikes to remove aluminum.

In future processing involving slugs similar to those discussed here, it is recommended that the flow sheet shown in Fig. III be used.

It is further strongly suggested that core samples of the original slug be carefully analyzed and that Bk assays be run concurrent to the main processing stream.

# Simplified Flow Sheet

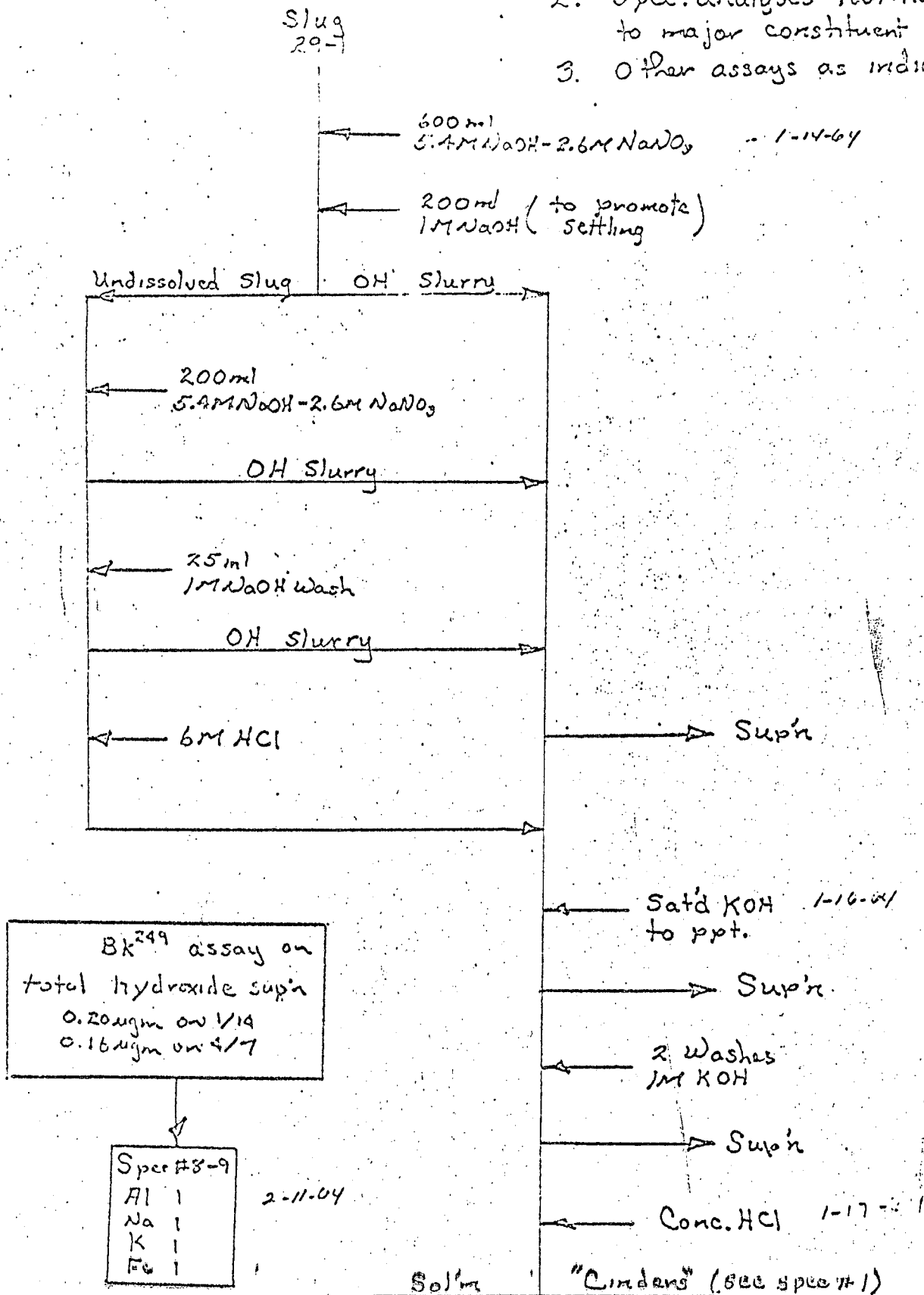
UCRL-16314

6

FIG. I

1/64 Cave Run

1. — BK<sup>249</sup> assay
2. Spec. analyses normalized to major constituent equal one
3. Other assays as indicated





Continued From  
Page 1

Sol'n

"Cinders"

sup'n

Multiple  
6M HCl  
Digestions

$Cp^{252} = 32.3 \mu\text{gm}$   
 $Cm^{244} = 210 \text{mg}$  1-18-64

BK<sup>241</sup> Assay  
on total "Cinders"  
0.9 gm

K B 3  
BK<sup>249</sup> assay  
7.5  $\mu\text{gm}$  on 1/14  
7.0  $\mu\text{gm}$  on 3/12

Spec # 1  
Ru 1  
Pd 1  
Mo 0.2  
Fl 0.2  
Ce <0.02

1-17-64

Convert to  
12 M LiCl  
0.1 M HCl

LiCl I  
12 mm X 14 cm  
1/18/64

10M LiCl  
0.1M HCl  
0.05M NH<sub>2</sub>OH

Rare Earths

Spec # 10  
Fl 0.2  
Ce 0.1  
Gd 0.01  
Y 0.06  
K 1

2-25-64

4M LiOH  
to ppt. 1-20-64

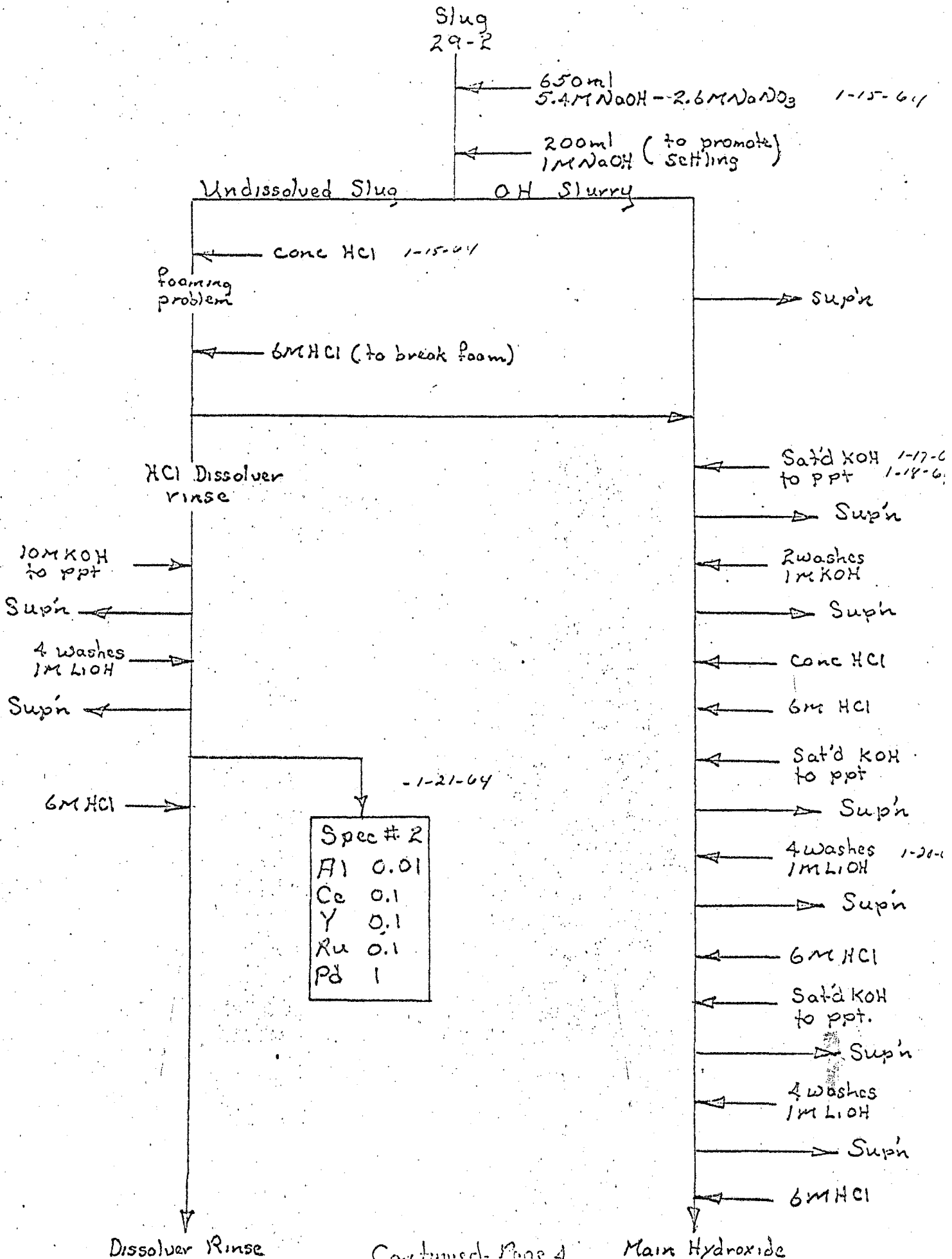
Sup'n

4 Washes  
1M LiOH

6M HCl  
to dissolve

Combine with output  
From LiCl II for  
feed preparation for  
LiCl III







Continued From  
Page 3

Dissolver Rinse

Main Hydroxide

Convert to  
12M LiCl  
0.1M HCl

-1-21-64

Multiple  
6M HCl  
Digestion

Cinders

sup'n

Convert to  
12M LiCl  
0.1M HCl

-1-21-64

Cinders

White Salts

White Salts

KB 19

KB 21  
BK<sup>249</sup> assay  
2.36  $\mu$ gm on 1/14  
1.62  $\mu$ gm on 6/14

BK<sup>249</sup> assay  
0.09  $\mu$ gm on 1/14  
0.03  $\mu$ gm on 6/18

KB 25  
BK<sup>249</sup> assay  
0.86  $\mu$ gm on 1/14  
0.64  $\mu$ gm on 6/14

Spec #3  
Fl 1  
Pd 0.01  
Ce < 0.002

-1-29-64

10M LiCl  
0.1M HCl  
0.05M NH<sub>2</sub>OH

LiCl II  
12mm x 15cm  
1/22/64  
No Separation

CP<sup>252</sup> = 33.7  $\mu$ gm  
Cm<sup>249</sup> = 221 mg

4M LiOH  
to ppt  
4 washes  
1M LiOH

Sup'n 1-24-64  
1-25-64

6M HCl  
to dissolve

To LiCl III

Cm + Trans Cm  
from LiCl I

Total output  
from LiCl II

Convert to  
12M LiCl  
0.1M HCl

$C_{252} = 71.8 \mu\text{g}$   
 $Cm^{244} = 429 \text{ mg}$

KB50  
BK<sup>244</sup> assay  
10.7  $\mu\text{g}$  on 1/14  
10.1  $\mu\text{g}$  on 3/12

10M LiCl  
0.1M HCl  
0.05M NH<sub>2</sub>OH

LiCl III  
28 mm X 1/2 cm  
1/27/64

Rare Earths

BK<sup>244</sup> assay  
0.05  $\mu\text{g}$  on 1/14  
0.04  $\mu\text{g}$  on 4/13

Fe<sup>+++</sup>  
KOH  
to ppt

1-31-64

Sup'n

Trans Cm  
in 6M HCl  
Strip

Spec #11  
Al 0.2  
Ce 1  
Gd 0.1  
Y 0.6  
2-25-64

Fe<sup>+++</sup>  
KOH  
to ppt  
Sup'n  
Wash  
6N HCl

Fe<sup>+++</sup> 1-28-64  
10M KOH  
to ppt  
Sup'n  
10M NH<sub>4</sub>OH - 0.1M KOH  
to ppt  
Sup'n  
6M HCl

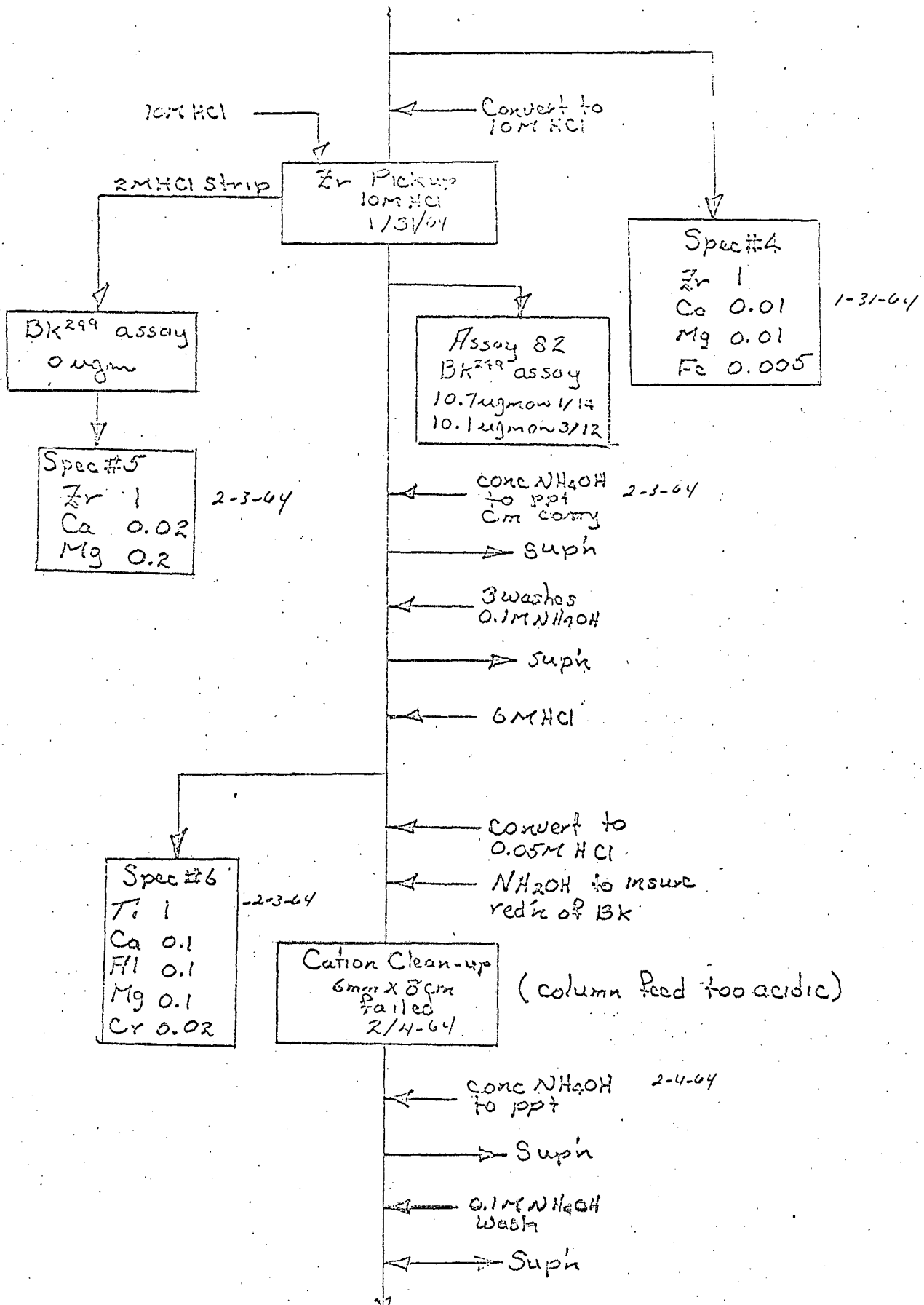
Convert to  
HNO<sub>3</sub>

Storage

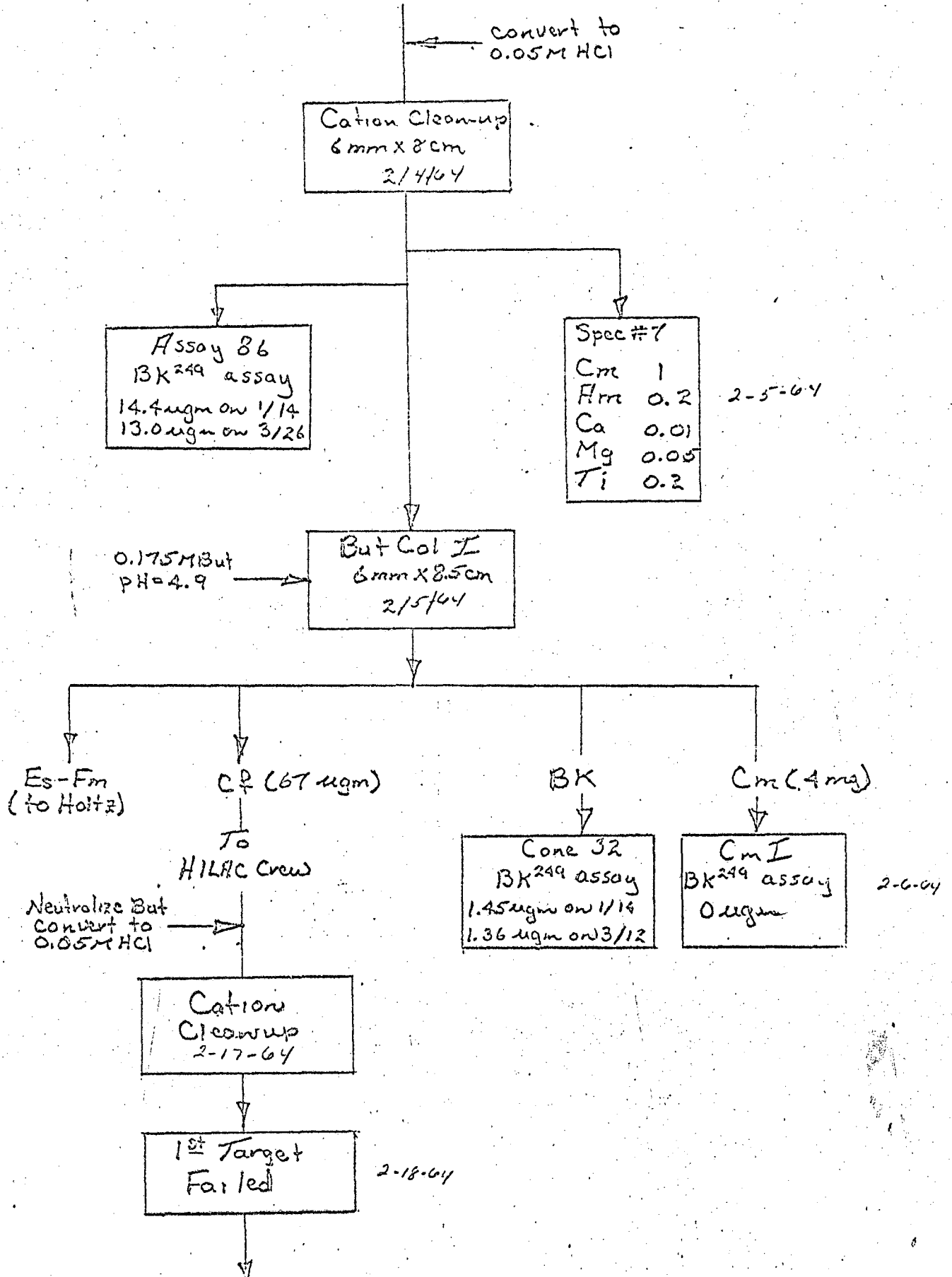
Fe Pickup I  
6M HCl  
1/29/64

Convert to  
8M HCl  
H<sub>2</sub>O<sub>2</sub>

Fe Pickup II  
8M HCl  
1/30/64







CP Fraction  
HILAC Crew

Fe Carry

LAW 2-19-64

HILAC LAW  
BK<sup>249</sup> assay  
0.13  $\mu$ g on 1/14  
0.10  $\mu$ g on 4/13

Fluor  
Fe Pick-up  
2/20/64

Convert to  
0.05M HCl

Cation  
Clean-up  
2/21/64

Target

Target  
To HILAC  
2-21-64

Sup'n

Bombard

Fe Carry

Dissolve

Scrounge  
3-3-64

Fe Pick-up  
2/20/64

Fe Carry

Fe Carry

But Col  
(bad separation)

Fe Pick-up  
3/2/64

Fe Pick-up  
2/2/64

Cation Clean-up  
3/1/64

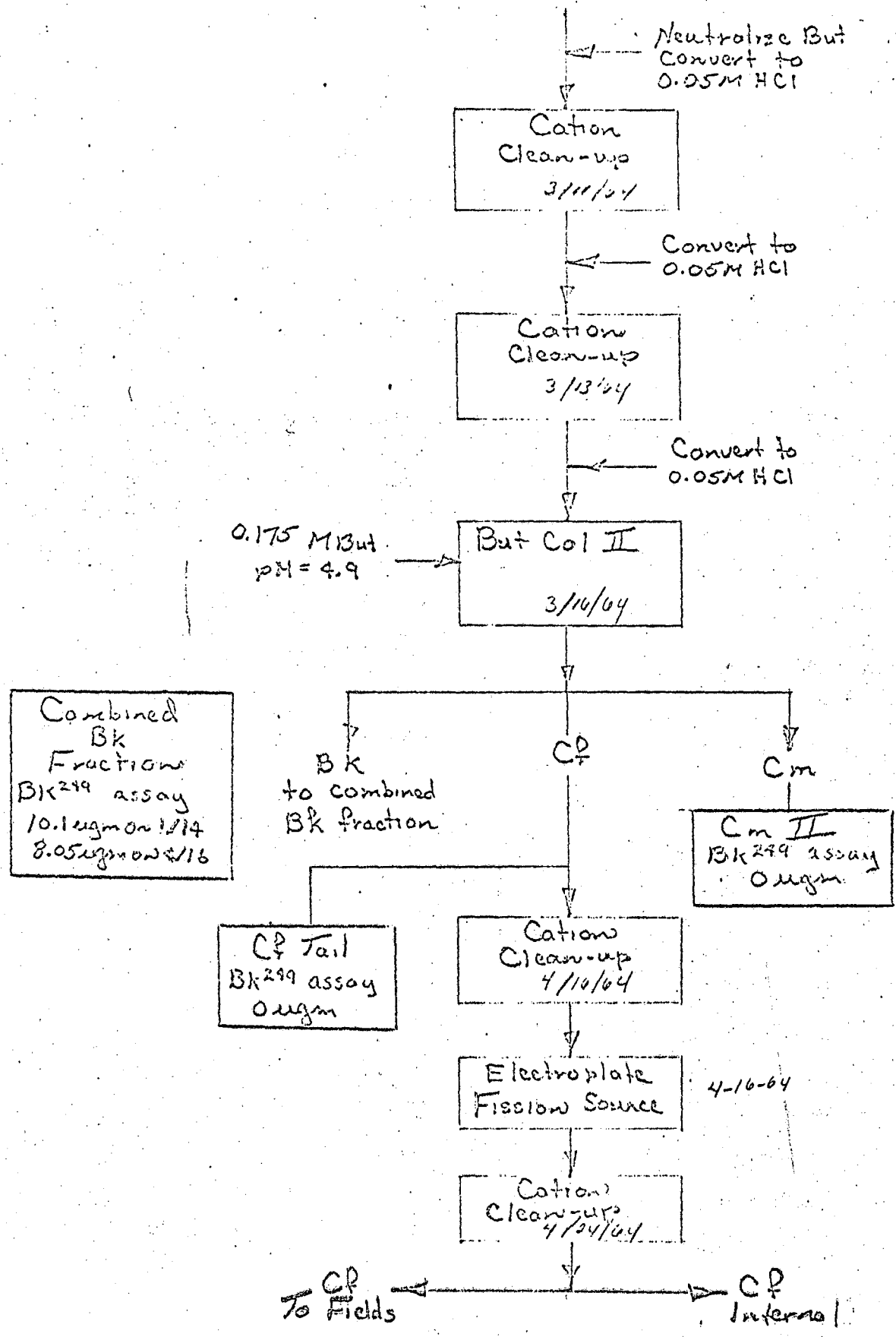
Es-Fm  
(To HILAC)

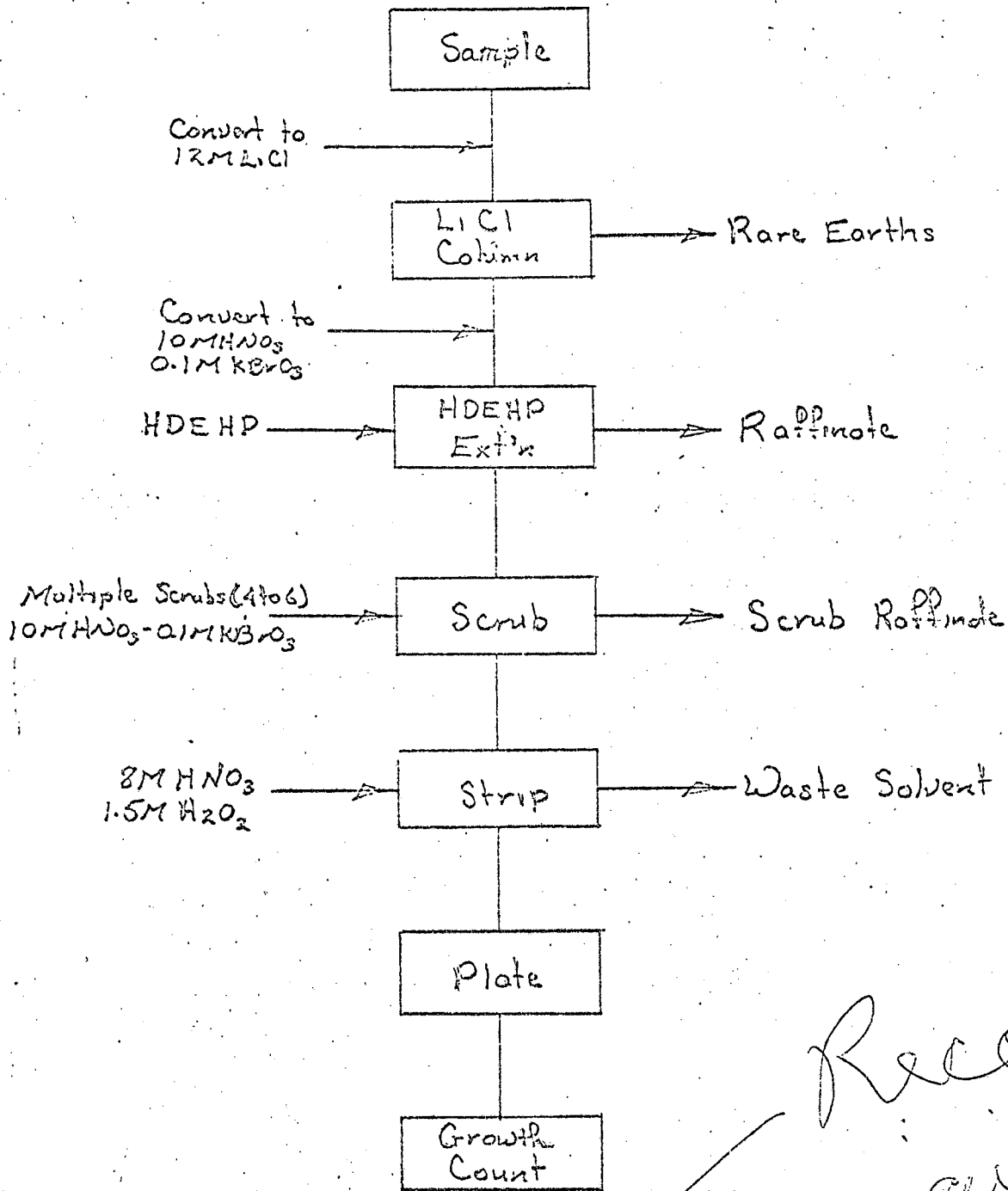
But Col  
3/1/64

Es-Fm  
(to Holt)

CP  
BK  
To combined  
BK Fraction

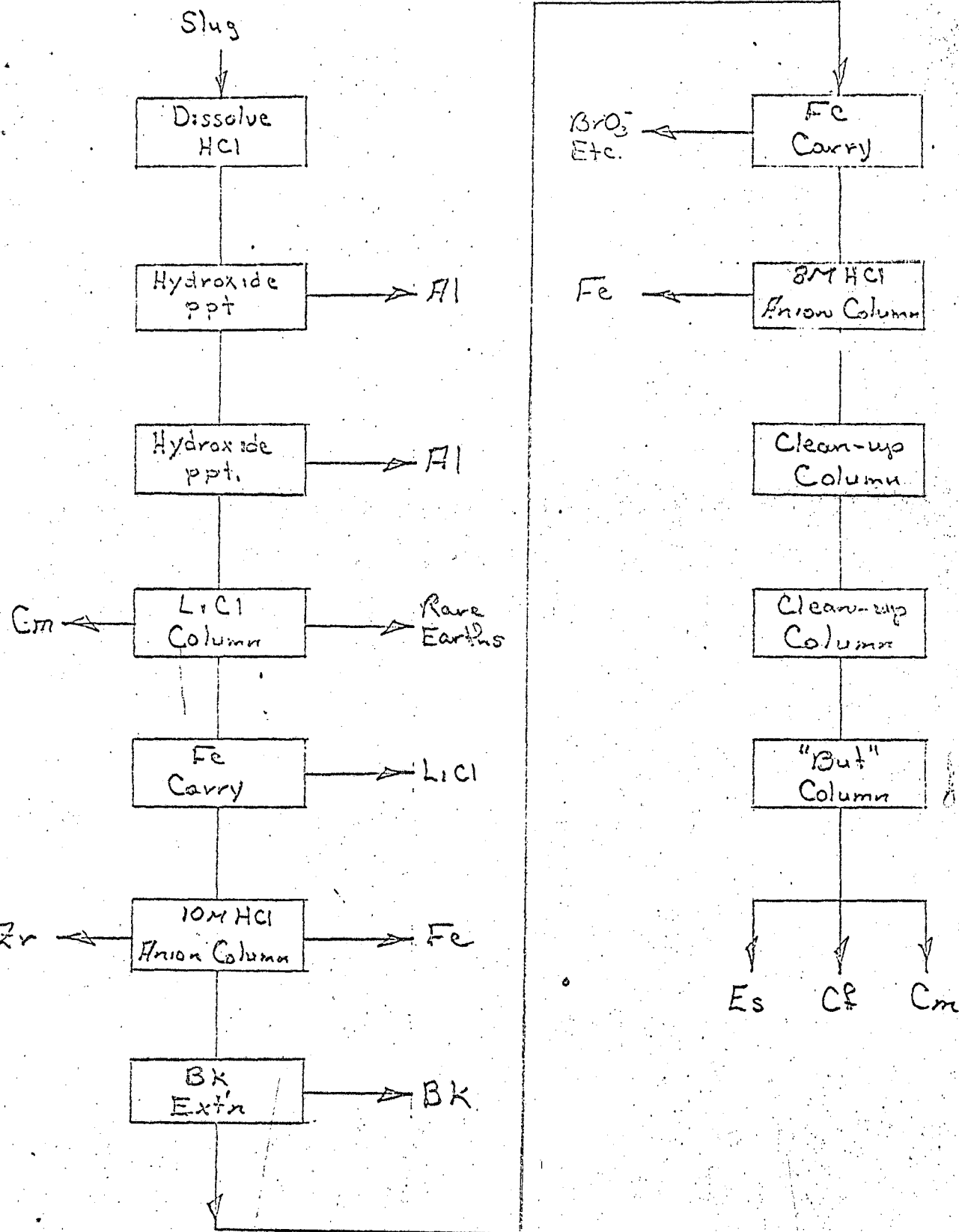
To Cave Crew





*Recopy original*

BK<sup>249</sup> Assay Procedure  
Figure II



Recommended Flow Sheet.

Figure III

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