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ORNL  
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Date January 18, 1950

Subject Discussion of Role Program with  
Hanford Personnel.

By L. P. Emler

To C. H. Rucker

Copy # 7A

*L. B. Emler*

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TO

Mr. C. N. Rucker

January 18, 1970

By Authority of

Mr. L. B. Emler

ATC Name  
AUG 27 1971 Title Date

ORNL NATIONAL LABORATORY  
CENTRAL FILES NUMBER  
50-1-86

Discussion of RaLa Program with Hanford Personnel

Messrs. W. M. Hartly and Robert L. Lance from the General Electric Company at Hanford accompanied by Mr. W. W. Love, Jr., of the Hanford AEC Office spent January 9, 10, and 11 at ORNL to review the current RaLa development program. They were extremely interested in all of the development phases of the new resin column process for the isolation of radioactive barium. There was some question in their minds, however, whether they could utilize the results of this development work because of their present commitment to have a 10,000-curie RaLa plant in operation by January 1, 1951. The major weakness in accepting the new column process was the lack of reproducible runs and a Los Alamos evaluation of the final product.

We raised the question with them as to what sort of reaction might be expected from the General Electric people if we recommended that their January 1, 1951, operation date be postponed until a number of successful column runs were completed at ORNL. The answer to this question, they believed, rested entirely with the Atomic Energy Commission.

During the course of our discussions it was suggested by the Hanford people that one individual at ORNL be appointed as the Hanford liaison man. This matter has been discussed with Messrs. F. L. Steahly and F. L. Caller and it was agreed that Mr. W. E. Unger will serve in this capacity. Essentially, this job will require that Mr. Unger keep Mr. W. M. Hartly of Hanford informed of the development program here at the Laboratory, while in turn Mr. Hartly will address his informative correspondence to Mr. Unger. I have asked that each of them send me copies of their correspondence so that I may maintain a complete file on the RaLa program.

ORIGINAL SIGNED BY  
L. B. EMLER

L. B. Emler

LBE:vp

1. C. N. Rucker
2. W. M. Hartly - Hanford
3. F. L. Steahly
4. F. L. Caller
5. E. J. Witkowski
6. W. E. Unger
7. L. B. Emler

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ORGANIZATION

CENTRAL FILES NUMBER

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Date January 16, 1950

Subject RaLa Meeting with Hanford Personnel  
at ORNL.

Copy # 10A 1  
W. K. Eister

By W. K. Eister

To F. L. Steahly

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GENERAL FILES NUMBER  
50-1-65

January 16, 1950

To: F. L. Steahly

From: W. K. Mister

This document consists of 2 pages.  
No. 10 of 10 copies, Series 7

Re: RaLa Meeting with Hanford Personnel at ORNL  
Hanford: W. M. Hardy-Technical Division-AEC, Robert L. Lance, Separations Department, William W. Low, Jro-AEC.

By Authority of

*ATC*

AUG 27 1971

Object: To determine (1) the status of the RaLa Program at Hanford Title Date  
(2) the purpose of their visit (3) the probable effect of the  
ORNL Development Program on the Hanford Program.

Hanford Program Status

The RaLa Program at Hanford is based on a directive from AEC (GEH-15783) which specifies that Hanford have a RaLa plant in operation by January 1, 1951 and estimates the cost at about \$3,000,000.

The important program dates are as follows:

- March 1950 - Final cost estimate
- August 1950 - Analytical laboratory in operation
- October 1950 - Plant construction complete and process development started in the plant equipment
- January 1951 - First production run

The sulfate precipitation equipment will be sized to make 10 kc runs. This would increase the barium content from 2 to 6 grams on this basis. The present 706-D process is to be used with only minor modifications.

Development work sponsored Hanford consists of an electrolysis study, and an analytical study - a corrosion study is anticipated.

They are considering the possibility of using longer irradiated metal and recovering the plutonium.

A development program (HW 13463) was submitted by Hanford to the AEC, but the AEC did not take action on it.

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Date January 16, 1950

Subject RaLa Meeting with Hanford Personnel  
at ORNL.

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W. K. Eister

By W. K. Eister

To F. L. Steahly

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To: F. L. Steahly

January 16, 1950

From: W. K. Bister

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Re: RaLa Meeting with Hanford Personnel at ORNL  
Hanford: W. M. Hardy-Technical Division-AEC, Robert L. Jones, Separations Department, William W. Low, JFC-AEC.

By Authority of

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AUG 27 1950

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They are considering the possibility of using longer irradiated metal and recovering the plutonium.

A development program (HW 13463) was submitted by Hanford to the AEC, but the AEC did not take action on it.

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(Authority for change in classification) (Date)

by C. S. Peters 3/29/94  
(Signature of Person making change) (Date)  
Verified by: J. S. Norman 3/29/94  
(Signature of person verifying change) (Date)  
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The equipment design is now being started, and in February the equipment now in the cells will be removed.

Purpose of Visit

To review the ORNL Development work.

They were very interested in our entire program. The major weakness from their point of view was the lack of reproducibility runs and consumer evaluation.

Effect of ORNL Program

Hanford is now committed to the 706-D process and only developments adaptable to that equipment maybe utilized.

In the event that the 10 kc run planned on the ion exchange column in 706-D is successful. Then the 706-D equipment might be used for RaLa Production. Equipment alteration costs of \$300,000 would be involved. The Hanford operation could then be dropped saving the commission about \$2,700,000.

- cc: REBlanco
- IRHiggins
- EJWithowski
- LBKalet
- JODavis
- FRBruce
- FLCuller
- EGReid
- WKEister

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To CF

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Date E. January 24, 1950

Subject Re: Metal Waste Disposal from the  
RaLa Process.

Copy # 5A  
L. B. Emlet

By E. C. Stewart

To E. J. Witkowski

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August 22, 1994.

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# INTER-COMPANY CORRESPONDENCE

OAK RIDGE NATIONAL LABORATORY

Post Office Box P  
OAK RIDGE, TENN.

(INSERT NAME) COMPANY CARBIDE AND CARBON CHEMICALS CORP. LOCATION

TO  
LOCATION

E. J. Witwoski  
Bldg. 706-D

DATE **January 24, 1970**

ATTENTION  
COPY TO

F. L. Culler  
F. R. Bruce  
J. O. Davis  
L. B. Balet  
D. G. Reid

ANSWERING LETTER DATE

SUBJECT

OAK RIDGE NATIONAL LABORATORY  
CENTRAL FILES NUMBER  
**50-1c-129**

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TO

By Authority of

**AUG 27 1971**

Name Title Date

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*[Signature]*

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The present flowchart for the metal waste disposal from the Bala process recommends the neutralization of the metal solution with soda ash and followed by the addition of tri sodium phosphate.

Operation of the proposed Metal Recovery Unit to be placed South of the Tank Farm Area would be enhanced by the elimination of the phosphate salt, but continuing the use of the soda ash. The neutralization of the metal solution with 50% NaOH would be preferred but the jetting characteristics of the diuranate formed are not understood to the point of recommending its use.

In view of the fact that no phosphate have been added to the Pilot Plant Metal Waste streams and no difficulties have been evident, it is recommended that the use of phosphates from 706-D metal wastes be eliminated.

*[Signature]*  
**E. C. Stewart**

ped

RECORDED  
INDEXED  
JAN 27 1970  
OAK RIDGE NATIONAL LABORATORY

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*Ra La*

Central Files No. 50-1-144

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ORNL  
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Date January 26, 1950

Subject RaLa Process Development

Copy # 1 CEL

By A. H. Holland, Jr.

To C. N. Rucker

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In Reply Refer To:  
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Oak Ridge, Tennessee  
January 26, 1950

ORNL Central Files No.  
50-1-144

Carbide and Carbon Chemicals Division  
Union Carbide and Carbon Corporation  
Post Office Box P  
Oak Ridge, Tennessee

Attention: Mr. C. N. Rucker, Director  
Oak Ridge National Laboratory

Subject: RALA PROCESS DEVELOPMENT

Gentlemen:

The following teletype message has been received from Los Alamos  
Scientific Laboratory, Los Alamos, New Mexico:

"Tentative agreement reached here on strontium toler-  
ance up to 200 curies per batch regardless of size.  
This cannot be confirmed without further discussion  
of details. Information follows by letter.

It is requested that this information be made available to  
Mr. W. E. Unger, Technical Division.

Sincerely yours,

/s/ Albert H. Holland Jr.  
Albert H. Holland, Jr., M.D.  
Director of Research and Medicine

CC: C. E. Center, K-25  
S. R. Sapirie

Shilling:mw

COPY typed 2-13-50 \_jv  
For: L. B. Emlet

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David C. Hamlin 3/19/95  
Technical Information Officer Date  
ORNL Site

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By Authority of  
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Name Title Date  
AUG 27 1995

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Jed Davis 3/8/95  
ADL signature Date  
Single review of CORP-declassified  
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Date: 2-1-50

This document consists of 2 pages. No. 1 of 11 copies. Series A.

To: RaLa Design File

From: F. L. Culler

Subject: Conference on RaLa Progress

ORNL NATIONAL LABORATORY  
CENTRAL FILES NUMBER  
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F. L. Steahly 3/21/50  
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| 2. J. A. Swartout | 8. L. B. Emlet     |
| 3. J. O. Davis    | 9. E. J. Witkowski |
| 4. F. R. Bruce    | 10. R. E. Blanco   |
| 5. W. K. Eister   | 11. I. R. Higgins  |
| 6. W. E. Unger    |                    |

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To: RaLa Design File  
From: F. L. Culler  
Subject: Conference On RaLa Progress

February 1, 1950  
Classification Cancelled

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By Authority Of \_\_\_\_\_  
By *JAV* Date AUG 30 1971

Present: Steahly, Swartout, Davis, Bruce, Eister, Unger, Culler, Emlet, Witkowski.

A meeting to discuss the results of the recent high level RaLa resin column run and to discuss future program was held on January 27, 1950. The following summarizes important points of the discussion:

1. Members of the Chemical Development Section expressed confidence in the resin column process for barium purification as a result of the two high level runs just concluded. Resin stability has been demonstrated at high level of radiation and the general operability of the acid-citrate and the one column Versene process. No additional runs will be required in the resin cubicle unless data collected during the high level runs reveal previously unknown problems. If this is the case, another run should be made with regular RaLa production material at as high an activity level as possible. Los Alamos, of course, must agree to this test.
2. Unger, Higgins, and Blanco will concentrate on the preparation of a report for RaLa in the coming two weeks. In addition to a major report, a supplementary letter to be transmitted to the AEC outlining results of RaLa process development and the construction program at ORNL.
3. Emlet said that at the present time no definite schedule had been established for Bldg. 706-D alterations. However, an attempt will be made to finish the design so that construction may start some time around June 1, 1950.
4. A construction request is necessary to provide money for the design of the RaLa facilities. The development budget for fiscal year 1949 has already been overrun. Emlet indicated that the Atomic Energy Commission is waiting for the construction request.
5. Plant design will proceed on the basis of the installation of a crud filter, of a process filter, the one column Versene process in which metathesis is still necessary. Handling and loading facilities for the new Los Alamos carrier will be designed and installed. Design work will now proceed with the utmost haste with less attention being given to assistance in the development program.

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*R. S. Rust* 1/19/95  
ADD signature Date

Floyd L. Culler

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[REDACTED]

February 16, 1950

To: F. L. Stearny

From: E. E. Bianco

Subject: Processes Used at Los Alamos for The Separation of Lanthanum and Barium;  
Specifications for Oak Ridge Hala Product.

Los Alamos Personnel Contacted:

- F. Hammond, J. A. Leary, D. Mueller,
- J. Lillenthal, W. Smith

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*[Signature]* 1/18/95  
 ADD signature Date

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- 6. M. [REDACTED]y
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Processes for the Separation

of Lanthanum from Barium

(1) Nitric Acid Process (preferred process)

The weight of barium plus strontium cannot exceed two grams because of the low solubility of barium and the fixed volume of the operating equipment. Therefore only green slugs may be used in the Sala process.

- (a) Dissolve the barium-lanthanum product in 25 ml water and add 50 ml of fuming nitric acid to precipitate barium nitrate.
- (b) Centrifuge using the shipping container as the centrifuge cone.
- (c) The supernate containing the lanthanum and such soluble impurities as iron, chromium, nickel, and lead is decanted to a second centrifuge cone. The volume is brought up to 200 ml with water and a small amount of hydrofluoric acid is added to precipitate lanthanum fluoride. Any nickel present also precipitates as the fluoride.
- (d) The precipitate is separated by centrifugation, washed with water, and centrifuged again. This is the final form of the product.

(2) Alkali Process

The weight of barium plus strontium may be as high as 6 gms. in this process so that either green or old slugs may be used.

- (a) Dissolve the barium-lanthanum product in water and hydrochloric acid at pH 2.
- (b) Add ammonium hydroxide until the pH is 8.0 to precipitate lanthanum hydroxide. The pH is determined with pH hydrion paper. Impurities such as iron, chromium, lead, and nickel also precipitate.

- (c) The lanthanum precipitate is separated from the solution containing barium by centrifugation.

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- (d) The precipitate is dissolved in acid and lanthanum re-precipitated by the addition of oxalic acid and a small amount of fluoride. Any nickel or lead present also precipitates. Iron and chromium are soluble.
- (e) The precipitate is separated by centrifugation, washed with water, and centrifuged again. This is the final form of the product.

Discussion:

At present the alkali process is used exclusively and all separations are performed by filtration. The present equipment is too small to handle the volumes required in the acid process. The acid process is preferred since approximately 17% of the barium and strontium is carried by the hydroxide precipitate in the alkali process. The nitric acid process will be used as soon as the new building is ready in the fall of 1950.

Difficulties encountered in present processing are:

- (1) Plugging of the first filter by organic material and/or hydroxides of impurities such as iron, chromium, nickel, lead, and silica.
- (2) After dissolution of the hydroxide cake and addition of fluoride, an acid insoluble substance, assumed to be barium and lanthanum silica fluoride, forms on the vessel walls.
- (3) Plugging of the second filter by nickel fluoride.

At present the first lanthanum milking containing the impurities is discarded. Subsequent milkings are then performed without difficulty.

If the product is dissolved in weak nitric acid, the solution turns alkaline within a few days as a result of the formation of radiation decomposition products. The pH of a sodium hydroxide or hydrochloric acid

solution remains constant. It has also been observed that the resistance of stainless steel to nitric acid is lowered by the presence of radiation decomposition products.

Oak Ridge RaLa Product Specifications

The stringency of the RaLa product specifications depends on which process is used at Los Alamos.

Old Equipment (filter)

New Equipment (Centrifuge)

Fall of 1950

Nitric Acid Process

Barium and Strontium	2 gm
Iron	500 mg
Nickel	10 "
Chromium	10 "
Lead	200 "
Sodium	10 gms
Radio Strontium	100 curies

Alkalie Process

Barium and Strontium	2 gm	6 gms
Iron	10* mg	10 mg
Nickel	10 "	10 "
Chromium	10 "	10 "
Lead	50 "	50 "
Sodium	1.5 "	10 gms
Radio Strontium	100 "	100 curies

\* Los Alamos estimates that the product contains approximately 50 mg at present.

The nitric acid process specifications are tentative and will be confirmed at a later date but the order of magnitude is correct.

The radio strontium specification is based on the known decontamination of barium obtained at present in the Los Alamos alkalie process and the fact that their lanthanum final product can contain as high as 100 millicuries of Sr

[REDACTED]

The sodium specification is based on solubility considerations so that 30 ppm of sodium can be tolerated in either process in the new equipment.

Nickel must be low in the nitric acid process since nickel fluoride is insoluble and will appear in the product.

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This document consists of 4 pages and 0 figures. No. 12 of 15 copies, Series A

Date February 21, 1950

Subject RaLa Program

ORNL NATIONAL LABORATORY  
CENTRAL FILES NUMBER  
50-2-135

By C. E. Larson

To A. H. Holland, Jr.

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- 4. C. E. Center
- 5-6. C. E. Larson
- 7. A. M. Weinberg
- 8. J. A. Swartout
- 9-11. F. L. Steahly
- 12. F. C. VonderLaan
- 13. L. B. Emlet
- 14. H. Stringfield, Jr.
- 15. D. W. Cardwell

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David R. Hawkins 2/3/95  
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OAK RIDGE, TENN.

February 21, 1950  
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U. S. Atomic Energy Commission  
Post Office Box E  
Oak Ridge, Tennessee

~~Or Changed To~~  
By Authority Of [Signature]  
By [Signature] Date AUG 30 1951

Attention: Dr. A. H. Holland, Jr.  
Director of Research and Medicine

Subject: RALA PROGRAM

Gentlemen:

The Oak Ridge National Laboratory RaLa development program has progressed to the point where design and construction of revised production facilities for Building 706-D are justified. Alteration of the plant for continued production of 2500 curie batches of Ba<sup>140</sup> utilizing the ORNL process was approved in a letter from R. W. Cook to C. E. Center, dated September 16, 1949, and a tentative authorization to spend \$122,400 was given. It now appears to be more reasonable to equip the 706-D RaLa area to accommodate the new ion exchange process for the production of 10,000 curie batches. The process has been successfully demonstrated up to the 3000 curie level of activity. Desirable portions of the present equipment will be integrated into the new facilities. The total cost of all changes and additions is now estimated to be \$287,400, including overhead. This figure includes \$165,400 for lead for shielding, \$22,400 for other materials and \$49,800 for labor. A change recommendation for this revision is being prepared for submission.

Information obtained from such a facility should be invaluable in the development of a process for RaLa production from the more desirable sources soon to be available such as the M.T.R.

It was proposed at one time that this laboratory use standard Hanford production slugs instead of "green" slugs so that plutonium could be recovered as a by product of the RaLa process. Conversations with consumer personnel have indicated the inadvisability of such a procedure since the product would contain three times as much cold barium and force the use of a decidedly less efficient milking process at their site.

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1-31-95  
Single copy of ORNL-2874-1  
document was submitted by DOE Office of  
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To: A. H. Holland, Jr.

-3-

February 21, 1950

The suggested major process improvements are:

1. Filtration of the lead-barium sulphate precipitate to replace settling and decantation, a source of high losses in the old process. A dissolver, solution purification filter, and a product filter have been demonstrated on a full plant scale at tracer activity level with losses less than 2%.

2. Separation and purification of the barium product from lead and other impurities has been demonstrated using an ion exchange column on full scale tracer level and on full scale, high activity runs. The resin process was successfully used in three high activity level runs using actual RaLa feed at 600, 1500 and 3000 curie levels, respectively. The resin column replaces all of the old RaLa process vessels except the dissolver and precipitator, thus eliminating hazardous and unreliable purification equipment.

3. On full scale tracer level runs, starting with jacketed uranium slugs and proceeding through barium elution from the ion exchange column, product recovery has been consistently greater than 95%\*.

The process to be used in the revised ORNL plant will consist of the following major steps:

- |   |                    |
|---|--------------------|
| 1. Slug jacket removal in caustic   | Existing equipment |
| 2. Uranium dissolution in nitric acid   | Existing equipment |
| 3. Removal of crud from the dissolver solution by filtration using filter aid | New equipment      |
| 4. Lead-barium sulphate precipitation   | Existing equipment |
| 5. Lead-barium precipitate filtration   | New equipment      |
| 6. Product dissolution and purification by resin adsorption and elution       | New equipment      |
| 7. Product evaporation and loading  | New equipment      |

\*The old process yields cover a range from 30 to 70%.

To: A. H. Holland, Jr.

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February 21, 1950

It is planned to issue the following technical reports on RaLa development within the next month:

- (1) Higgins, I. R., Blanco, R. E., Unger, W. E., Preliminary Demonstration of RaLa Ion Exchange Processes at the Kilo-curie Level, ORNL-623
- (2) Blanco, R. E., Laboratory Development of an Ion Exchange RaLa Process, ORNL-620
- (3) Higgins, I. R., Semi-Works Demonstration of a Precipitation and Ion Exchange RaLa Process, ORNL-621
- (4) Unger, W. E., Design Considerations in the RaLa Process, ORNL-622
- (5) Blanco, R. E., Laboratory Development of the Versene Ion Exchange RaLa Process, ORNL-625 (To be issued in six or eight weeks)
- (6) Goeller, H. E., Culler, F. L., Calculated Production of  $\text{Be}^{140}$  from M.T.R. 25 Fuel Assemblies, ORNL-626
- (7) Culler, F. L., Goeller, H. E., Unger, W. E., RaLa Study - Preliminary Report #1, ORNL Central Files #49-4-38 (Issued April, 1949)

Yours very truly,

OAK RIDGE NATIONAL LABORATORY

*C. E. Larson*  
C. E. Larson  
Director

FLSteahly-FLCuller/sjp

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CHANGE RECOMMENDATION

CENTRAL FILED NUMBER  
50-9-41

OAK RIDGE NATIONAL LABORATORY

DATE February 28, 1950

PROJECT NO. CR-110

Request for \$287,400

"This document consists of 2 pages  
No. 12 of 25 copies, Series A

TITLE: RaLa Production Plant

Issued by \_\_\_\_\_  
Chief Engineer  
Engineering Department

This document has been approved for release  
to the public by:

David R. Hamlin 1/30/95  
Technical Information Officer ORNL Site Date



Approved by \_\_\_\_\_  
Superintendent  
Eng. & Maint. Division

INV.

To be commenced  
upon authorization

" \_\_\_\_\_  
Supt. or Director of  
Using Division

To be ready for use  
September 1, 1950

" \_\_\_\_\_  
Director

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Authorized \_\_\_\_\_  
Atomic Energy Commission

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DATE February 28, 1950

PROJECT NO. CR-110

CLASSIFICATION CANCELLED

Led Davis / 1/30/95  
DD signature Date

Single rereview of CCRP-declassified documents was authorized by DOE Office of Declassification memo of August 22, 1984.

CHANGE RECOMMENDATION

"RaLa" PRODUCTION PLANT

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By Authority Of DOC

By CEB Date AUG 30 1950

JUSTIFICATION

The program for future RaLa production was outlined in a letter from A. H. Holland to C. N. Rucker, August 24, 1949. Pursuant to the Commission's desires, Oak Ridge National Laboratory undertook the necessary research and development to provide a better RaLa production process and plant.

We are confident that the design of the necessary plant has been stabilized to the extent that we are in a position to make a firm estimate of cost of the installation at this location. Therefore, following the directions of the letter of R. W. Cook to C. E. Center, September 16, 1949, we are submitting a construction request for this work. The Oak Ridge National Laboratory plant has produced the following major process improvements:

1. The settling and decantation of the lead barium sulphate precipitate has been replaced by filtration. A dissolver product filter to remove undissolved silicious crud and a sulphate-metathesis filter have been demonstrated. This eliminates the unit over which major losses have occurred in the past.
2. Lead separation and product purification have been accomplished using a resin exchange column. Product losses have been negligible and undesirable product contaminants have been removed to meet consumer specifications; and the radiation stability of the ion exchange resin has been demonstrated with three high-level runs using ca. 500 to 3000 curies of product.
3. On tracer-level runs, starting with jacketed slugs and processing through product elution, barium recovery has been consistently greater than 95%.

Development work was performed on equipment comparable in size to that required in the final plant. The resin column replaces all of the old RaLa process vessels except the dissolver and precipitator, thus eliminating the hazardous and unreliable purification equipment.

DESCRIPTION

The following additions to the Oak Ridge National Laboratory RaLa production facilities are now in progress:

1. Duplicate dissolver solution filters and radiation shields.
2. Duplicate precipitate filters and radiation shields.
3. Duplicate resin column purification equipment and radiation shields.
4. New product handling equipment for revised Lcs Alamos carrier.

Existing equipment will be used for:

1. Slug jacket removal in sodium hydroxide
2. Uranium dissolution in nitric acid
3. Lead-barium sulphate precipitation and metathesis.

It will be necessary to provide new equipment for:

1. Crud filtration of dissolver solution
2. Lead-barium sulphate filtration
3. Product purification by resin absorption and elution
4. Product evaporation and loading.

The installation will be made in existing laboratories in Building 706-D, where, due to the recent renovation of the off-gas system, adequate facilities are available to control and prevent radioactive gas release. However, because of limited space available in that location, considerable lead will be required for shielding.

#### COST

The estimated cost of this installation is ----- \$287,400.

Note: This estimated cost includes laboratory overhead as prescribed in OR-13, revised September 7, 1949.

#### REMARKS:

Although the work up to the present time has been classified as Research and Development, the above estimated expenditure of \$287,400, (whether the new plant is used as the production plant or as a standby for the installation at Hanford) can be considered as a capital addition to the Oak Ridge National Laboratory facilities.

The expenditure of \$122,400, for the installation of RaLa facilities at Oak Ridge National Laboratory was authorized by A.E.C. but was not included in the original budget for FY-1950, (Letter R. W. Cook to C. E. Center, September 16, 1949). The Capital cost of \$287,400, was, however, included in the revised mid-year budget for FY-1950.

The work will be performed by the Oak Ridge National Laboratory forces according to plans and specifications prepared by the Technical and the Engineering and Maintenance Divisions of the Oak Ridge National Laboratory.

It is requested that the necessary Directives be issued for the accomplishment of this work.

ORNL  
CENTRAL FILES NUMBER  
50-8-121

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ORNL  
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Date March 14, 1950

Subject Modified RaLa Plant at ORNL.

Copy # 2B  
G. E. Larson

By R. W. Cook

To W. J. Williams

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David R. Hamlin 1/30/95  
Technical Information Officer Date  
ORNL Site

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ORNL  
CENTRAL FILES NUMBER  
50-8-121

Walter J. Williams, Director, Division of  
Production, Washington  
R. W. Cook, Manager, Oak Ridge

March 14, 1950

MODIFIED Classification Cancelled  
ORNL

SYMBOL:

RM:JHR

Changed To

By Authority Of *JAC*

By *EEB*

Date **AUG 30 1971**

On March 8, 1950, Mr. N. J. Carothers of your office informed Dr. J. H. Roberson, ORO, that the ORNL Rala program should be reviewed in Washington, ORO, before a decision could be made on the Oak Ridge facilities. A summary of recent correspondence of the Rala production facilities program in ORO is included herein for your information, together with a statement of the problem, as we see it, and our recommendation.

Recent History of Rala Program.

August 24, 1949. In a letter to the Commission, ORNL stated as follows:

"The present Rala process contributes materially to the particulate and gaseous airborne activity at the Laboratory. This fact, plus the hazardous condition of the equipment and the inefficiency of the operation demands that immediate steps be taken to improve the Rala production procedure."

August 26, 1949. Memorandum, Walter J. Williams to R. W. Cook, extended the time of Rala production at Oak Ridge until January 1, 1951, at which time Hanford would take over production, presumably at the 10,000 curie level.

September 16, 1949. By letter to Carbide, and after discussion with Mr. Carothers, the Commission authorized the process development and design proposed in the ORNL letter of August 24, 1949, and committed itself to approve the proposed construction as improvement to the present plant.

September 26, 1949. Memorandum, R. W. Cook to W. J. Williams, advised your office of the above action.

February 21, 1950. In a letter to the Commission, the ORNL reviewed the results of the process and design studies, and recommends a different modification, which includes an ion exchange step. Cost of this modification is estimated to be \$287,400, and its completion is conservatively set as September 1, 1950.

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*Led Davis* 1/30/95  
Date

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Single rereview of CCRP-declassified documents was authorized by DGE Office of declassification memo of August 22, 1984.

March 14, 1950

The Problem as It Concerns ORO.

1. ORO has been instructed to maintain production until January 1, 1951.
2. The present facility and process are inefficient, unreliable, and hazardous, and the Commission is committed to modification thereof.
3. A modified facility, as now proposed by the ORNL, would be safe, efficient, and provide satisfactory production as required. Its operation would provide additional data for the design of new Rala plants. This includes the suggested installation at Arco, for which the ORNL has an informal commitment to develop a process.

Recommendation:

It is recommended that the proposed modification of the ORNL plant be approved. Factors supporting this recommendation are:

1. The chemical inefficiency and unreliability of the present process.
2. Radiological hazard of the present operation.
3. ORO program commitments to continue production.
4. Obtaining a Rala standby plant at low cost capable of 10,000 curie source production.
5. Applicability of Rala technology to be obtained by this operation, both at Hanford and Idaho.
6. Ability to relieve Hanford of Rala process operation if desired.

Factors also considered in arriving at this recommendation are:

1. The short useful operating time projected for the plant (four months minimum).
2. Hazard of shipping irradiated uranium from Hanford to Oak Ridge.
3. Hazard connected with high level radiochemical separations in Oak Ridge.
4. We are not in a position to assess the importance of the diverted plutonium, estimated to be 650 grams per year, for 8 Rala sources of 10,000 curies, using 100 day irradiated material. (Present

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Mr. Williams

- 3 -

March 14, 1950

irradiation is about 45 days, and Los Alamos indicates preference for this material because of its lower inert barium content.

As requested by Mr. Carothers, a copy of the recent ORNL letter and the ORNL Change Recommendation are attached for your information. The ORNL letter of August 24, 1949, was transmitted with our memorandum of September 26, 1949.

/s/ SRS  
R. W. Cook

Enclosures:

1. CR-110
2. CF 50-2-135, Cy 3A

CC: A. H. Holland, Jr.

Roberson:aw



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ORNL  
CENTRAL FILES NUMBER  
50-4-7

ORNL  
MASTER COPY

Date April 5, 1950

Subject Shipment of RaLa by Air.

Copy # 3A  
*Wm. Linger*

By L. B. Emler

INV.  
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To C. E. Larson

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Dr. C. E. Larson

By Authority Of

Date AUG 30 1950 April 5, 1950

Mr. L. B. Ealet

By [Signature]

ORNL

CENTRAL FILES NUMBER

Shipment of RaLa by Air

50-4-7

A meeting was called by Mr. Edward McDougall of the Los Alamos Atomic Energy Commission to discuss the feasibility of shipping the slugs for the RaLa process from Hanford to Oak Ridge by a C-54 aircraft, and also of transporting the RaLa from Oak Ridge to Los Alamos in the same fashion. The meeting was scheduled to be held at the Argonne National Laboratory on March 30 and 31, 1950. It was attended by the following persons:

John Robinson)  
Ray Armstrong)-Oak Ridge Atomic Energy Commission  
P. J. Selak )

Y. R. Holquist)-Hanford Atomic Energy Commission  
D. H. Kilgore )

Edward McDougall - Los Alamos Atomic Energy Commission

Clark Carr - President of Cargo, Inc. (This is the company which operates the airlift between Albuquerque and Los Alamos.)

Phil Hammond )-Los Alamos Scientific Laboratory  
Jim Lillenthal)

Bill Harty )- Hanford  
Clark Harrison)

L. B. Ealet)- Oak Ridge National Laboratory  
W. E. Unger)

It appears that Los Alamos has recently acquired a C-54 aircraft. They apparently are now attempting to find enough work for it in order to justify its procurement. Mr. McDougall was the person who raised the question of feasibility. In October, 1947, Oak Ridge National Laboratory suggested to the Atomic Energy Commission that the use of aircraft for the transportation of RaLa slugs from Hanford to Oak Ridge could be justified purely on an economical basis. However, the Washington Office of Production of the Atomic Energy Commission directed that any radioactive materials shipped by air be enclosed in a crash-proof carrier. Their definition of a crash-proof carrier was one which would stand a free drop from 5,000 feet. Personnel of the Laboratory did not believe that the advantage to be gained by air shipment would justify a large development program to supply such a container. As a result, all of our shipments of RaLa slugs from Hanford to Oak Ridge are made by rail express.

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Date 1/18/85

Single review of CCRP-declassified documents was authorized by DOE Office of Declassification memo of August 22, 1994.

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During the discussions at Argonne, it developed that the personnel representing the contractors of the three Atomic Energy Commission installations were not particularly concerned whether RaLa shipments were made by air, rail express, or truck. They felt that the mode of transportation was entirely an Atomic Energy Commission decision. The greatest advantages to be gained by air shipment were:

1. Less losses of product by decay and, consequently, requiring the loading of fewer slugs into the dissolver.
2. Less waste of lanthanum during the initial purification process at Los Alamos, since the product would be "milked" earlier and thus allow more equal-sized isolations.
3. The cost figures prepared by Mr. Clark Carr indicated that air shipments were more economical providing the Atomic Energy Commission did not require extraordinary security precautions.

This phase of our discussion was completed with the decision that Mr. McDougall would prepare a document to be submitted to the Washington Atomic Energy Commission Office requesting their approval of air shipments of RaLa and other radioactive materials. Messrs. John Robinson and Ray Armstrong also discussed the possibility of making product shipments from here to other sites with Mr. McDougall.

By far the most interesting and also profitable discussions were held with Messrs. Phil Hammond, Los Alamos, and Bill Harty, Hanford, regarding the current status of the RaLa Program at their respective installations. Mr. Hammond related that the change on the Weapons Program at Los Alamos may alter their requirements for 10,000-curie sources of RaLa by January 1, 1951. Unofficially, he estimated that these larger-sized sources may not be necessary until January, 1953. He emphasized, however, that no definite decision had been made at the present time and, therefore, it would be unwise for us to deviate from the official date of January 1, 1951. The immediate schedule for RaLa at Los Alamos was discussed in detail. It was decided that the next RaLa run would start at Oak Ridge National Laboratory on April 8, 1950. At the completion of this run Mr. Hammond anticipated a sixty-day shutdown of the Los Alamos facilities which would provide ample time to modify the piping arrangements in Cell A of the 706-D Building. Mr. Hammond agreed to inform us by phone as soon as this sixty-day shutdown appeared eminent.

Mr. W. M. Harty of Hanford reviewed in detail the status of the RaLa design and construction program at Hanford. He emphasized that they are presently proceeding under the original Atomic Energy Commission directive to have their new equipment operable by January 1, 1951. The cost estimate that they are presently working on amounts to \$2,968,000.

April 5, 1950

Some of the personnel at General Electric believe that both the completion date for the project, as well as the cost estimate, will be met; however, this will be confirmed in two or three months. At the present time approximately \$140,000 in work orders have been written, but Mr. Harty did not know how much of this had actually been spent. At the present time they have between 100 and 110 men working on the RaLa Program at Hanford. Forty of these are draftsmen and another forty are construction people. The remaining are engineers, chemists, etc. The process which they are planning to use is identical with the obsolete method now being used at Oak Ridge National Laboratory except that the decantation from A-9 will be replaced with centrifugation. In addition, all of their equipment will be adaptable to remote control maintenance and replacement. Mr. Harty indicated that there were rumors around Hanford to the effect that General Electric management was interested in being relieved of the responsibility of this project but that no action had been taken by the time he left. He estimated that if the project were stopped on the first of April, 1950, at least \$200,000 of the allocated costs would already have been spent.

Quite a bit of the information above is of this unofficial type of thing which Hanford is so adept in passing out. They make some statement or relate some detail and then qualify it by remarking that this is entirely unofficial or is only a rumor, etc. Under these circumstances, it is extremely difficult to properly evaluate the program; however, this information is passed along to keep you advised of what transpired during the Chicago meetings. If you wish, we will be happy to discuss this matter in more detail with you at your convenience.

ORIGINAL SIGNED BY

L. B. EMLET

L. B. Enlet

IBE:wp

1. C. E. Larson
2. F. L. Steahly
3. Wm. Unger
4. E. J. Witkowski
5. L. B. Enlet

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ORNL  
CENTRAL FILES NUMBER  
50-4-39

Date April 10, 1950  
Subject THE RALA PROCESS - SF SUPPLE-  
MENTAL MANUAL - Procedure #1  
By \_\_\_\_\_  
To \_\_\_\_\_

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CLASSIFICATION CANCELLED  
DATE JUN 23 1959  
*Edgar J. Murphy*  
CO-ORDINATING ORGANIZATION DIRECTOR  
OAK RIDGE NATIONAL LABORATORY  
AUTHORITY DELEGATED BY AEC 9-10-57 *EJM*

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*David R. Hammin* 1/30/95  
Technical Information Officer Date  
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- 4. C. E. Center
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- 6-7. H. L. Kilburn
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THE RALA PROCESS (NATURAL URANIUM AND PLUTONIUM)

The RaLa process is operated in Building 706-D for approximately one week in five weeks. The process consumes about seventy-six, four-inch Hanford slugs per run. Approximately thirty grams of plutonium are handled during each run. Plutonium and uranium are waste products of the RaLa process.

A. DESCRIPTION OF THE PROCESS

The slugs to be dissolved are loaded into the dissolver (A-1) where the aluminum coatings are partly removed by dissolution in hot NaOH-NaNO<sub>3</sub> solution. This first coating-removal solution and five subsequent dissolver washes are discharged into radiochemical waste. Another similar dissolution completes the coating removal, after which the solution and three subsequent dissolver washes are discharged to radiochemical waste. No sample is taken here. Next, sufficient reagents are added to dissolve one half of the uncoated slugs. The dissolving is allowed to progress until the specific gravity of the solution indicates that it is 70% UNH (uranium nitrate hexahydrate); then the solution is diluted with water to prevent freezing, sampled (LMA), and transferred to the precipitator (A-9).

In the precipitator, the extraction step is performed by coprecipitation of the product with PbSO<sub>4</sub>. The solution is digested and allowed to settle for eight hours after the necessary reagents have been added. The uranium-plutonium waste is then decanted to catch tank A-8, where it is sampled (8WMA); then it is transferred to resettling tank A-11, where it is allowed to settle again for several hours before decanting to catch tank A-6. The metal waste is sampled again at A-6 (6WMA) before it is neutralized in the neutralizer A-5 and discharged through A-6 to the Tank Farm metal storage.

The second half of the slugs by now have been sufficiently dissolved for dilution, sampling (LMB), and transfer of the solution to the precipitator through A-11 to pick up the heel from waste resettling. This transfer is followed by a ninety-pound water wash from the dissolver. A second extraction is performed on top of the cake from the first. After a long settling period, the metal waste is decanted to A-8, sampled (8WMB), resettled in A-11, dropped to A-6, resampled (6WMB), held for analytical results, neutralized, and discharged to the Tank Farm as in the case of the first batch.

Next, the cake in the precipitator is washed several times to remove all traces of U and Pu. The waste from each washing is decanted from A-9 to A-8 and held until all extraction washes are collected. Then this extraction wash waste is sampled (8WW) and transferred to A-11, where it is resettled until the end of the run.

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THE RALA PROCESS

A. DESCRIPTION OF THE PROCESS - (Continued)

The cake in A-9 is now converted to a carbonate by metathesis with  $K_2CO_3$ . After cooling and settling for several hours, the metathesis waste is decanted to A-8, sampled (8WC), and held until two washes of the cake are added to it. Then the entire metathesis waste is sampled (8WCW) and transferred to A-11 on top of the extraction wash waste to be held until the end of the run. The cake, if further processed by the remainder of the Rala process, has no bearing on S & F accountability because no U or Pu can be tolerated past the metathesis step.

Meanwhile, any undissolved slugs in the dissolver have been subjected to further dissolving. At the end of the run this solution is sampled (heels) and transferred from A-1 to A-6. The extraction and metathesis waste in A-11 is added to it; the whole is neutralized in A-5 and discharged through A-6 to the Tank Farm metal waste tanks.

B. SAMPLING

1. Blister Sample

The sampler consists of an air jet designed to circulate solution from the tank to be sampled through a glass sample cone and back to the tank. The entire assembly is surrounded by a lead "blister" to reduce the radiation hazard.

The air jet is turned on and the labeled glass sample cone, with a rubber gasket around the lip, is placed on the air jet. With the jet closed, the solution starts to circulate from the tank through the cone and back to the tank. The solution is allowed to circulate for a minimum of fifteen minutes.

The sample is then removed from the "blister" and placed in a lead carrier. It is taken in the carrier to the laboratory for analysis. A sample sheet showing date, volume of solution in the tank sampled, sample code number, etc., is turned over to the chemist who analyzes the solution.

2. Sample Types

A uranium analysis is regularly run on the slug dissolvings in order to determine the completeness of the dissolution by balance against the slug count. No analysis for uranium is run on the waste samples, and no analysis for plutonium is run on any of the samples. Below, in tabular form, is given information regarding the samples on which uranium is run. The plutonium figures in this table are estimated. A list of the other samples taken is also given.

ANALYSIS FOR BARIUM AND URANIUM REQUIRED  
ON THESE SAMPLES

SAMPLE CODE	VOLUME IN SAMPLE	VOLUME IN TANK	CONCENTRATION MG/ML		AMOUNT IN SAMPLE MGS.		AMOUNT IN BATCH GMS.		RATIO-SAMPLE TO BATCH	RADIATION R/HR @ 6"
			U	Pu	U	Pu	U	Pu		
LMA	1 ml	178 L	356	.081	356	.08	63,120	14.4	5-6 : 10 <sup>6</sup>	Approx. 5
LMB	1 ml	178 L	356	.081	356	.08	63,120	14.4	5-6 : 10 <sup>6</sup>	Approx. 5
Heels	1 ml	133 L	40.3	.0092	40.3	.0092	5,330	1.2	7-5 : 10 <sup>6</sup>	Approx. 3

THESE SAMPLES ARE NOT ANALYZED FOR URANIUM OR PLUTONIUM BUT CONTAIN THESE ELEMENTS

8WMA These samples are taken from Tank A-8 to determine the efficiency of the metal waste decant from Tank A-9 which follows the extraction. The volume is 1 - 2 ml and they read approximately 10 R/Hr at 6".

6WMA These samples are taken from Tank A-6 after the material represented by the 8WMA samples has been resettled to recover any product which may have been lost in the original decant. They are 1 - 2 ml in size and may read up to 10 R/Hr at 6".

8WV This sample is taken from Tank A-8 and is a sample of the water and acid washes which follow the batch B extraction. It is 1 - 2 ml in size and reads approximately 4 R/Hr at 6". This is the last sample in which uranium and plutonium may be tolerated.

THESE SAMPLES CONTAIN NO APPRECIABLE URANIUM OR PLUTONIUM

8WC This sample is taken from Tank A-8 and is a sample of the waste metathesis solutions from Tank A-9. It is 1 - 2 ml in size and reads approximately 2 R/Hr at 6".

8WCW This sample is taken from Tank A-8. It is a sample of the washes from the metathesis cake. It is 1 - 2 ml in size and reads approximately 4 R/Hr at 6".

6P This is the final product sample. It is taken from Tank B-6. It is approximately 1/2 ml in size and will read greater than 100 R at 6".

4. ~~SECRET~~

THE RALA PROCESS

B. SAMPLING - (Continued)

2. Sample Types

In general, the total uranium charged is obtainable as the sum of the three dissolver samples LMA, LMB, and the heel dissolving. While uranium analyses are not run on the following samples, the subsequent disposition of uranium can be represented as shown below: The 6WMA and 6WMB samples represent the uranium discharged as supernate from A-9 by the decantation of batches A and B, respectively. The 8WW sample represents the heel of uranium from the decant of batch B, which is left in A-8 plus the uranium which has been removed from the sulfate cake by washing in A-9 after the decant of batch B. Since heavy metals cannot be tolerated past this point, it is assumed that all the uranium has been removed from the sulfate cake at this time. The heel dissolving is neutralized and discharged to the metal waste system without further processing.

C. ANALYTICAL PROCEDURE

The following analytical method is used for the determination of uranium in RaLa dissolver samples:

1. Salicylate Method of Measurement

- a. Pipette sample of appropriate size (on RaLa dissolvers 1 ml. of 1:1000 dilution) into 25 ml. volumetric flask (1 ml. of 0.1/50 dilution on dissolver samples).
- b. Add 5 ml. buffer\* and 5 ml. 10% sodium salicylate. Dilute to about 23 ml. Adjust pH to about 9 (paper) with  $\text{NH}_4\text{OH}$  (shaking). Dilute to mark, shake well, and check pH (should remain 9). Run a blank containing all reagents.
- c. Read at 380 millimicrons, PC-6, filter with a Coleman Model 11 Spectrophotometer. Record transmittance. From the standard curve, read the number of micrograms in the sample.
- d. Calculation:

$$\text{mg. U/ml.} = \frac{\text{micrograms/sample}}{\text{Vol. (ml.)} \times 1000}$$

$$\text{In RaLa runs, mg/ml.} = \text{micrograms/sample}$$

$$\text{No. of Slugs} = \frac{\text{mg/ml} \times \text{known vol. (ml.)}}{1.18 \times 10^6}$$

\* 140 gms. of ammonium acetate in one liter of solution adjusted to pH 7.5 with ammonium hydroxide.

~~SECRET~~

## THE RALA PROCESS

### D. SOURCES AND LIMITS OF ERRORS

#### 1. Analytical Accuracy in Uranium Determinations

By the colorimetric method of uranium determination, described above and commonly employed in routine analyses for RaLa samples, the normal analytical error is  $\pm 5\%$  in samples containing more than 1 mg/ml uranium. The error can be as high as  $\pm 10\%$  in dilute samples (in the order of 0.1 mg/ml uranium). If special care is used, this error can be reduced to  $\pm 3\%$  for concentrated samples. The analytical error of the volumetric method of uranium determination can be as low as  $\pm 0.5\%$  if all nitrate ion is removed from the aliquot. Because nearly every sample at Oak Ridge National Laboratory contains considerable nitrate ion due to the nature of the processes, the elaborate procedure for nitrate removal would have to be employed on almost every uranium determination. The nitrate removal from hot samples becomes quite a tedious and time-consuming procedure because of the precautions imposed by the activity hazard. The procedure is not sufficiently developed for use on extremely hot samples.

The amounts of plutonium present in most samples are not sufficient to allow ionic-chemical procedures to be used for plutonium determination. The radiochemical procedures, based on counting the alpha activity, are accurate only to  $\pm 5\%$ . In dilute concentrations of plutonium (less than 1000 counts per milliliter) the error is as high as  $\pm 15\%$ .

#### 2. Volume Determination

In order to obtain a material balance, liquid volume measurement as well as analysis is necessary to determine the quantity of Source and Fissionable material present. Because of radioactivity, most volume measurements at Oak Ridge National Laboratory are made by remote control instruments which measure liquid level and specific gravity. The error introduced by each instrument is estimated by the Oak Ridge National Laboratory Instrument Department to be  $\pm 1\%$ . This means a total error of  $\pm 2\%$  for each volume determination by liquid level and specific gravity measurements.

Obtaining a representative sample is difficult at a number of points where samples are necessary for accountability. Undissolved solids, extremely large flows, and huge tanks not provided with adequate agitation make representative sampling almost impossible.

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Classification Cancelled

50-5-214

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FLS-340

356

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By Authority Of

Date AUG 27 1971

Chemical Technology Division

To: F. L. Steahly

Date: May 15, 1950

From: I. R. Higgins

Report Period: 2/10/50-5/10/50

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QUARTERLY REPORT

Title: RaLa Process - Unit Operations, Part I Ion Exchange Development

Work by: I. R. Higgins, W. E. Shockley, D. B. Masters

This document has been approved for release to the public by:



Secret Notebook no. CL-90, CL-288,

David Hamilton 7/9/95  
Technical Information Officer Date  
ORNL Site

SUMMARY

Development during this period has been concentrated on a "one column Versene" process, leading to a definitive decision to recommend including the metathesis step, and using 1.0 moles of Versene, feed pH 6.5 and a 3" x 24" Dowex 50 resin bed. These conditions were picked to allow plenty of safety factor for high barium yield and effective barium and strontium separation for handling a lead-barium sulfate cake which has been only about 99% metathesized. A total of 12 "one column Versene" runs have been completed. Sulfate precipitate is found in the process tanks when the sulfate content is too high or when the pH or Versene concentration is too low. High barium losses and poor strontium removal occur when the pH or Versene concentration is too high or the resin volume too small. Table I indicates, the very high degree of metathesis required to render all barium soluble in dilute nitric acid and, the importance of using a barium sulfate dissolving reagent, as Versene, in the process. Table II summarizes the significance data in the Versene process. Apparently pH 6.5 is the upper limit for the low barium loss. Runs are now in progress to determine the lower limit of pH.

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Jed Davis 3/8/95  
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Single rereview of CCRP-declassified documents was authorized by DOE Office of Declassification memo of August 22, 1994.

Resin Bed Pressure Drop: In order to determine if pressure was required on the ion exchange feed tank to overcome the resin bed resistance, pressure drop was measured on a 3" x 7" and 3" x 18" resin bed with and without vapor locking. Table III indicates that the bed resistance to flow at the rates planned, 0.5 ml/min/cm<sup>2</sup>, is very small even with vapor locking.

TABLE I

Completeness of Metathesis

Based on amount of barium precipitated from the low acid solution of the metathesized cake

Run	Grams Ba	% Ba Precipitated	% Metathesis of Total Sulfate Cake	
4	1	83	98.6	Sulfate Analysis
5	1	4	99.9	_____
8	1	9	99.8	_____
17	1	95	98.4	_____
24	1	20	99.7	_____
26	1	63	98.9	98.7
27	1	19	99.7	97.9
28	1	6	99.9	99.5
30	1	6	99.9	97.6
33	1	9	99.8	_____
45	2	2	99.9	100
46	2	3	99.9	_____
47	0.5	3	99.9	_____
49	2.0	<u>90</u>	<u>97.0</u>	_____
Average		30	99.5	

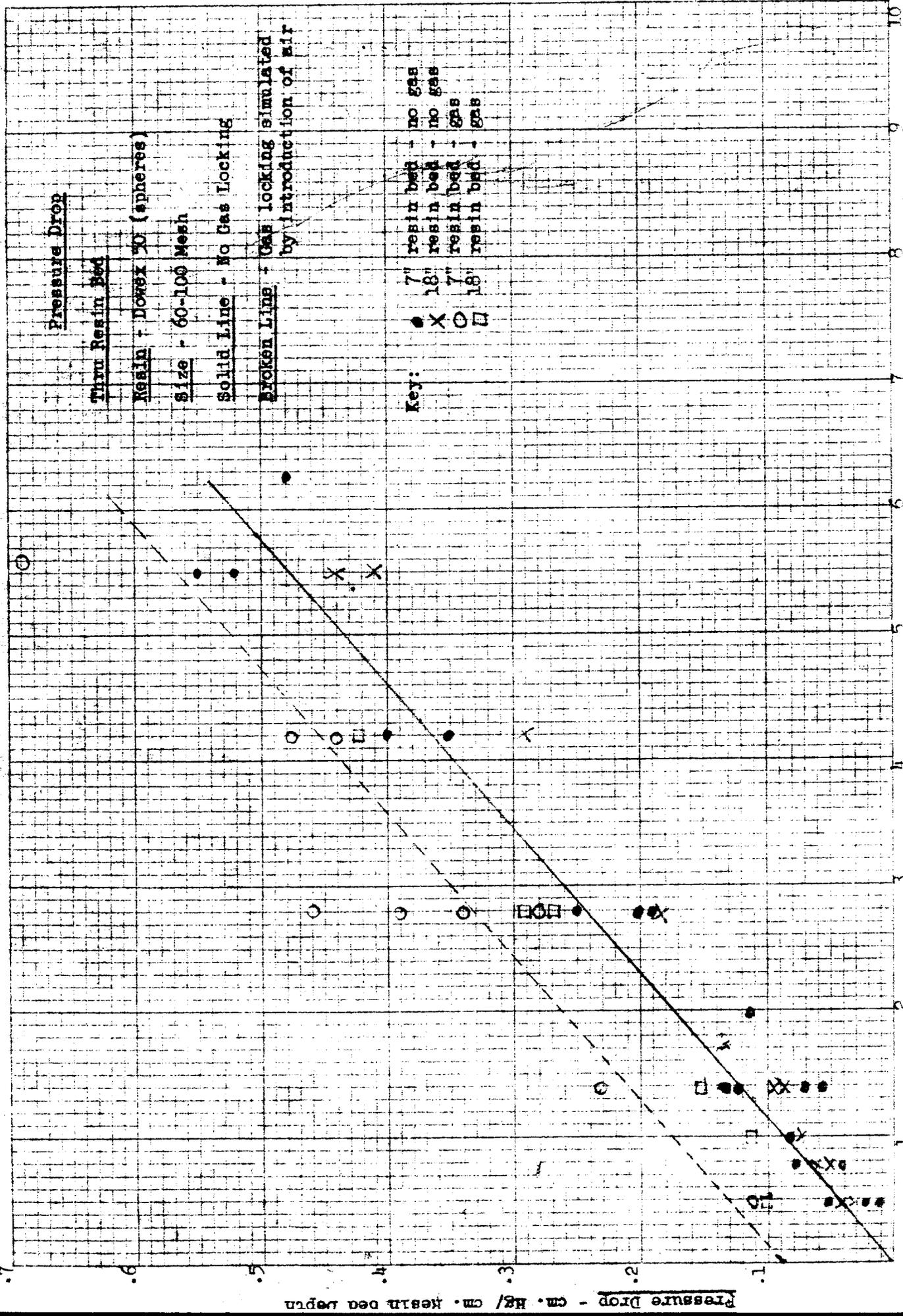
Note - % completion of metathesis calculated assuming BaSO<sub>4</sub> solubility in weakly acid metathesis solution equal to published solubility of BaSO<sub>4</sub> in water.

TABLE II  
 Critical Factors in the "One Column Versene" Process for Low Barium Losses and High Barium-Strontium Separation Process Steps

1. Solution of the lead-barium sulfate or carbonate precipitate in nitric acid or Versene.
2. Adjustment of pH and feeding to the Dowex 50 resin column.
3. Elution of any remaining strontium with Versene and pH 6.3.
4. Elution of the sodium with HCl.
5. Elution of the barium with 6 M HNO<sub>3</sub>

Run	Metathesis	Column Size	Feed		Percent Barium Losses				Percent Strontium in Product	
			Mole Versene	pH	Grams Ba and Sr	Settled in Tanks	Column Breakthru	Strontium Waste		Sodium Waste
706-D	Yes	3x7	0.65	6.0	1	52.2	0.6	0.5	0.1	0.66
36*	No	3x20	1.14	6.3	1	13.4	0.3	0.3	0.1	1.92
37*	No	3x20	1.14	6.4	1	54.2	0.3	0.1	0.1	0.30
38*	No	3x20	1.14	6.5	1	12.1	2.2	3.3	0.9	0.59
42	No	3x20	1.14	6.5	1	0.9	76.0	5.2	1.5	0.37
43	Yes	3x6	0.52	6.1	2	1.2	0.9	84.0	0.7	2.10
44	Yes	3x19	0.96	6.5	4	5.2	0.5	0.7	4.3	0.95
45	Yes	3x19	0.96	6.5	4	1.2	0.2	3.5	1.0	0.17
46	Yes	3x23	1.40	6.5	4	0.5	1.3	4.0	1.5	0.03
47	Yes	3x23	1.0	6.5	1	1.2	0.2	0.2	0.03	0.03
48	Yes	3x23	1.0	6.8	4	2.0	14.5	6.8	2.1	0.001
49	Yes	3x23	1.0	6.7	4	1.8	26.9	11.5	3.4	0.07

TABLE III



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50-5-215

JAD 356

By Authority Of

Date AUG 31 1971

FLS-341

Chemical Technology Division

To: By F. L. Steahly

Date: May 15, 1950

From: I. R. Higgins

Report Period: 2/10/50 - 5/10/50

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QUARTERLY REPORT

Title: RaLa Process - Unit Operations, Part II Auxiliary Equipment Development

Work by: I. R. Higgins, W. E. Shockley, D. B. Masters, H. O. Weeren, G. A. West, and C. D. Watson

Secret Notebook No. CL-80, CL-132, CL-82

INV. 64

1.0 Summary and Introduction

Various pieces of auxiliary equipment for the new RaLa change-over are being tested for operating characteristics and reliability before final installation.

- (1) Full scale crud and process filters with exact space arrangement to determine filtration rates, filter aid handling, and operating techniques.
- (2) Simulated product evaporation to determine quantitative transfer conditions to the shipping cone.
- (3) Surface evaporation from the shipping cone to determine the optimum operating conditions for rapid evaporation without splashing and,
- (4) Testing steam jets for use as a vacuum source. Preliminary indications are that all items can be used, some with modifications.

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Technical Information Officer Date  
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J. Giles Morgan 1-31-95  
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## 2.0 Experimental

### 2.1 Crud and Process Filtration

The Crud Filter: has been tested twice at full scale and the process filter once. 180 liters of 1.5 Sp. Gr. UNH was filtered through the 1.33 sq. ft. precoated star filter in 45 minutes using 5 g/l of 545 filter aid slurried in the crud containing UNH with a "Ruch" air agitator. The agitator formed a heavy mist when the liquid level was below one foot and an estimated 50 grams of the 9000 grams of filter aid was left in the center of the tank. The filter aid was completely backwashed with 60-80 l water.

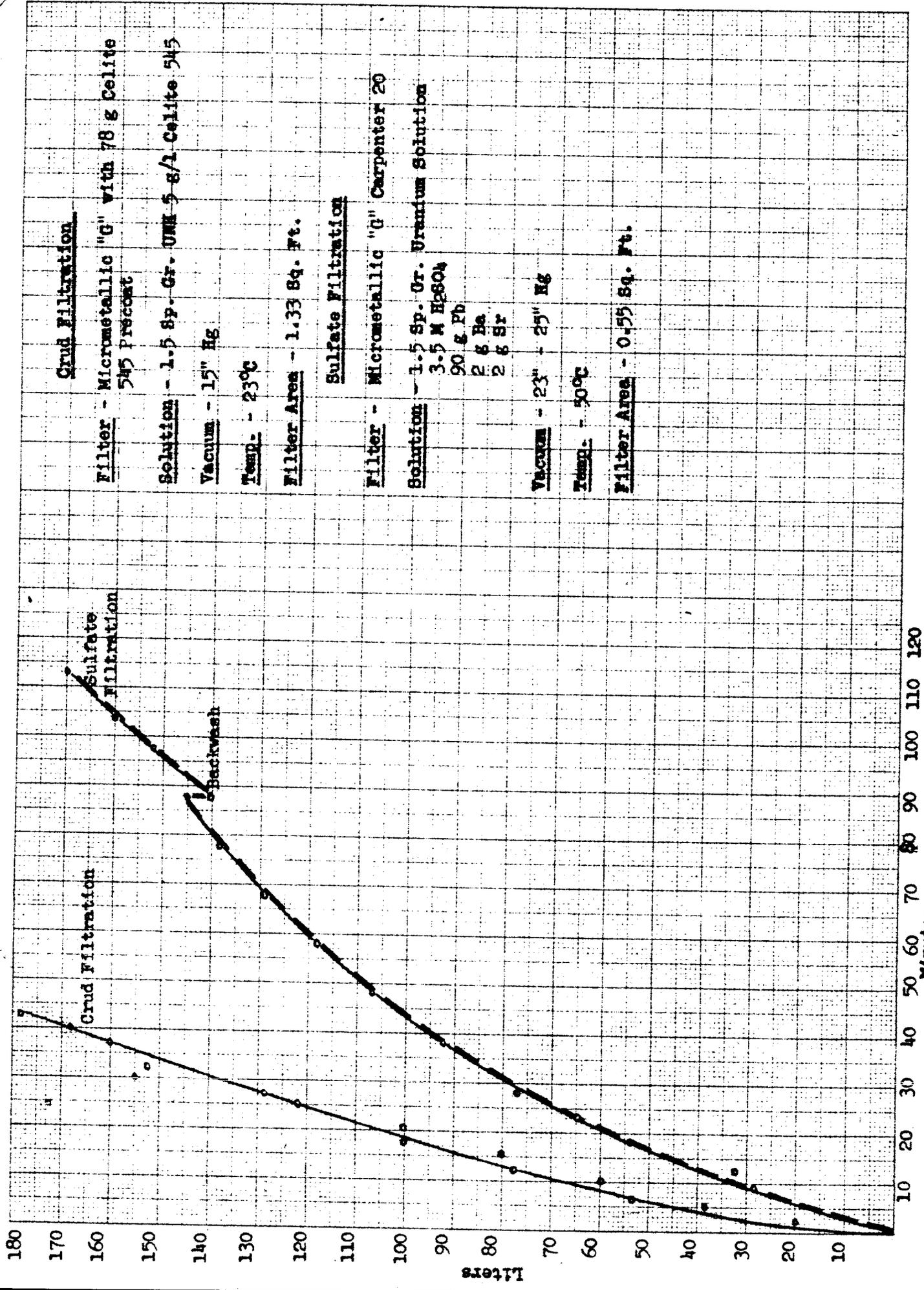
One Process Filter Run: has been made at full scale with the 0.55 sq. ft. Carpenter 20 "G" porosity filter using 90 g Pb, 2 g Ba, and 2 g Sr slurried in 180 liters of uranium solution at 3.5 M sulfuric acid, sp. gr. 1.5. The vacuum and transfer was made with a 3/4" S-K steam jet in 2 hours using 100# steam and maintaining 23"-25" vacuum. The filter was backwashed once during the run to unclog the filter. The total dilution from the steam jet was 57%. The precipitate was completely washed back to the precipitator with 10 l of water.

2.2 The Product Evaporator: was installed and no runs have yet been made to demonstrate quantitative product removal.

2.3 Shipping Cone: several tests have been made with total evaporation time from 4 to 9 hours. Ideal conditions of temperature and air flow have not been determined because in most runs agitation and splashing have occurred by the air flow leaking around the charging header and cone. The tightness of the cone fit determining whether splashing occurs or not. Evaporation without splashing was achieved at 0.05 # air/min. and 150°C in 7 hours with liquid temperature of 50°C under 20" Hg pressure.

2.4 Steam Jet Exhauster

The optimum operating conditions for the 3/4" S and K exhauster was found to be between 80 psig, spindle 1/2 way open - and 100 psig, spindle 1/4 open; with production of 27" Hg vacuum. For use as a vacuum source, a water-jacketed condenser is required to condense 1# of steam per minute mixed with 0.1# of air per minute. A water jet used as a contact condenser would discharge about 4 GPM to the "hot" drain, and would overload the drain system.



Crude Filtration

Filter - Micrometallic "G" with 78 g Celite 545 Precostat

Solution - 1.5 Sp. Gr. UHM 5 g/l Celite 545

Vacuum - 15" Hg

Temp. - 23°C

Filter Area - 1.33 Sq. Ft.

Sulfate Filtration

Filter - Micrometallic "G" Carpenter 20

Solution - 1.5 Sp. Gr. Uranium Solution  
3.5 M H<sub>2</sub>SO<sub>4</sub>  
90 g Pb  
2 g Ba  
2 g Sr

Vacuum - 23" - 25" Hg

Temp. - 50°C

Filter Area - 0.55 Sq. Ft.

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507-145

Date: July 28, 1950

This document contains  
2 pages. No 4  
of 6 copies. Series A.

To: F. L. Culler

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From: W. E. Unger

Subject: Specifications -- Rala Shipments

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F. L. Culler

July 28, 1950

W. E. Unger

Specifications -- Rala Shipments

As outlined in letter, Unger to Hammond, CF-50-7-27 dated July 8, 1950, a tentative Rala specification set forth in memorandum Blanco to Steahly, CF-50-2-93 dated February 16, 1950, was forwarded to Los Alamos for comment. Inasmuch as the tentative specification was based upon information from Los Alamos, and no adverse comment from Los Alamos was received, the present ion exchange process was developed to yield a product acceptable under those tentative specifications.

An earlier communication with Los Alamos (T. T. Hammond to Holland, NRS-97 October 1949, 1308 & GR-102) quoted a sodium specification at variance with that in the tentative specification on which the ion exchanger process had been based.

Letter Hammond to Unger CMR-10-120 dated July 18, 1950, states in part, "the information contained in the teletype still holds in that our preferred process (acid process) would have a sodium limit of 1.5 grams per shipment. This limit is based solely on solubility considerations."

"We have recently tested our processes in the presence of the impurities as listed and are prepared to tentatively approve these specifications with the exception of the correction in the sodium. In all cases we have not yet explored the upper limit of these impurities but feel that they can not safely be extended much farther. The sodium limit being based on solubility can not be extended at all."

The revised specifications tentatively approved by Los Alamos are as follows:

	<u>Acid</u>	<u>Alkali</u>
Ba and Sr	2 gm	5 gm
Fe	500 mg	10 mg
Ni	10 mg	10 mg
Cr	10 mg	10 mg
Pb	200 mg	50 mg
Na	1.5 gm	1.5 gm
Radio Sr	100 curies	100 curies

cc: FLStealy  
REBlanco  
ERHiggins  
EJWitkowski  
Rala File

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W. E. Unger

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David R. Herwin  
Technical Information Officer  
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FLS-433

From: W. E. Unger

Date: 8-14-50

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QUARTERLY REPORT

Classification Cancelled

Or Changed To

Title: 706-D Modification Project-Plant Design

By Authority Of

By \_\_\_\_\_ Date 8/14/50

Work by: G. B. Berry, L. Brewer, R. V. Foltz, J. E. Ruch, W. E. Unger,  
E. Wischhusen, R. E. Vaughan

Secret Notebook No.



SUMMARY

The 706-D Modification project will improve the efficiency and operability of the Rala plant by replacing the current decanting operation by filtration, and the present chemical precipitation purification by an ion exchange process. The alteration of Cell-A (present processing cell) piping; the addition of a dissolver solution clarifying filter cubicle and a process filter cubicle; and the construction of floor-pit cubicles to contain the ion exchange purification equipment, and a loading pit equipment to accommodate the new Los Alamos Product Carrier and shipping container, constitute the field construction scheduled.

The construction has proceeded smoothly as scheduled. Current cost estimates are within 15% of the initial estimates.

Schedule

The following production schedule was established by Los Alamos:

Last shipment before shutdown	June 28, 1950
First shipment after shutdown	Sept. 1, 1950
First shipment by new process	Nov. 1, 1950

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 David R. Herwin 1/30/45  
 T. E.

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Subsequent events have altered the consumers requirements. First, the new process equipment can accommodate only the new carrier, and the new carrier was designed by the consumer in anticipation of the completion by November of new processing facilities at Los Alamos, which have been delayed about 2 months. Further, Los Alamos requirements are apparently an expression of the demands of researchers whose needs cannot be anticipated exactly. In consequence, although it is planned here to be able to meet the above schedule, it is likely that the scheduled dates are a month to six weeks in advance of the actual deliveries required.

Construction

The field construction divided into three phases to minimize shut down time, has proceeded smoothly and rapidly in accordance with the following schedule, the uncompleted portions of which still appears valid;

Phase I	(Before decontamination of A-Cell July 1)	<u>Completion date</u>
	Cubicle floor excavations, Balcony filter support alterations, monorail footings	July 1, 1950
Phase II	(After decontamination of A-Cell and before startup, July 1--Sept. 1)	
	A-Cell process piping alterations and equipment additions, installation of crud and process filters and shielding	Sept. 1, 1950
Phase III	(After startup Sept. 1)	
	Loading Manipulator and shielding, Ion exchange cubicles and shielding, panel boards and instruments	Nov. 1, 1950

Phase II is about 80% complete. Filter units and shields have been fabricated and installation is in progress.

Phase III loading and resin pit liners are in the process of fabrication. The loading manipulator casting has been ordered. The two tantalum-lined process evaporator vessels have been placed on order.

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Design

Design has generally lagged behind schedule by one month but not to the detriment of construction.

The design effort has been divided roughly into three categories:

Process design--Process layout and process equipment

Special Mechanical design--Experimental design and special devices

Structural design--Hoist structures, excavation and concrete, massive shielding.

Approximately 60 drawings have been issued to date (see appendix), an estimated 20 will be issued to complete the project.

Development

Process development is essentially complete, culminating in the demonstration of the selected process schematically illustrated in appendix B.

Equipment development will continue in use-evaluation of proposed designs and test acceptance of process equipment. Plastic process valves, sampler units, replaceable valves and filter units, and other design features have originated in the solutions proposed to problems incurred in unit operations demonstration studies.

Cost

A cost estimate was made by the Plant Engineering Department August 1, 1950, as a check on the initial estimate made by the Technical Division January 19, 1950.

	<u>Cost Estimates, Including Overhead</u>		
	<u>Materials</u>	<u>Labor</u>	<u>Total</u>
January 19, 1950	187,800	99,600	287,000
August 1, 1950	137,000	184,000	321,000

Refinements in design explain the increased labor costs. The decrease in material costs are attributable to an economy in lead for shielding, a result of building the process cubicles under ground so that roof shielding only is required.

Appendix A

Drawings Issued to Date

D-7502	Monorail Framing
D-7503	Bldg. Alterations, Plan
D-7504	Monorail Entrance
D-7505	Pit Substructure
D-7506	Platform Alterations
D-7515	Crud Filter Cubicle Plan
D-7516	Crud and Process Filter Frames
D-7517	Crud Filter Plug
D-7518	Process Filter Cubicle Plan
D-7519	Concrete Plug
D-7625	Cone Manipulator Assy.
E-7626	Manipulator Sub-Assy. A
E-7627	Manipulator Sub-Assy. C
D-7628	Manipulator- Plug
D-7629	Manipulator - Detail No. 1
D-7630	Manipulator - Detail No. 2
E-7631	Manipulator - Detail No. 3
D-7632	Manipulator - Detail No. 4
B-7633	Manipulator Sub-Assy C No. 1
D-7634	Manipulator Sub-Assy C No. 2
D-7635	Manipulator Sub-Assy C No. 3
D-7727	Process Filter Cubicle Plug
D-7730	Crud Filter Hoist
D-7731	Process Filter Hoist
C-7775	Sampler Jet
E-7780	Sampler Valve
D-7637	Underground Piping
D-7736	Cubicles, Floor Plan
D-7735	Loading Cubicle, Shield
D-7734	Loading Cubicle, Sect. 3-3
D-7733	Pipe Channel Plugs No. 3
D-7729	Pipe Channel Plugs No. 2
D-7728	Cubicles Frame
D-7514	Pipe Channel Plug No. 1
D-7513	Cubicles Roof Plan
D-7512	Cubicles Sections
D-7511	Cubicles Sectional Plans
D-7510	Loading Cubicle Cover
D-7509	Loading Cubicle Sect 2-2
D-7508	Loading Cubicle Sect 1-1
D-7507	Loading Cubicle Plan
D7737	Pipe Channel Liner Plan

# ROUGH DRAFT

TD-1494	Schematic Flowsheet-Filter
TD-1528	Schematic Arr't. A-Cell
TD-1752	A-17 Tank
TD-1802	Pipe Schedule Filters
TD-1805	Jet and Valve Schedule Filters
TD-1754	Process Filters Piping Arr't.
TD-1803	Process Filters Blow Tank
TD-1761	Process Filter Details
TD-1800	Process Filter Clamp Assy.
TD-1855	Crud and Process Filter Valve Assy.
TD-1856	Crud and Process Rack Assy.
TD-1857	Crud and Process Valve and Gaskets
TD-1858	Crud and Process Filter Rack End
TD-1759	Crud Filters-Piping Arr't.
TD-1806	Crud Filter Details
TD-1807	Crud Filter Clamp Assy.
TD-1809	Crud Filter Clamp-Details
TD-1842	Tantalum Evaporator
TD-1843	Tantalum Evaporator Top Detail

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8-22-50

CARBIDE AND CARBIDE CHEMICALS CORPORATION

FOR OPERATOR'S USE ONLY

ANT X-10

# TELETYPE

CC NUMBER

COLLECT

PREPAID

Msg NR 837 Aug 502221002 GR 28

REF TT NR

TO: V. D. DONIHUE  
GENERAL ELECTRIC COMPANY  
HANFORD WORKS  
RICHLAND, WASHINGTON  
FROM: H. F. STRINGFIELD

This document consists of 1 page.  
No. 1 of 1 copies, Series A

DEPARTMENT: OPERATIONS

PHONE: 6387

MESSAGE: RE: OUR REQUEST FOR THREE HUNDRED OLD SLUGS. WILL YOU PLEASE GIVE US AN ESTIMATE OF PLUTONIUM CONTENT.

837

Rec'd by *cur* at *23/1425* z

Encoded by *cur* at *1436* z

Sent by *m.m.* at *1533* z

CENTRAL FILE NUMBER

50-8-82

**RESTRICTED DATA**

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If provisions of GM-74 Part II Par. 10d are followed, paraphrase not required. Handle as SECRET correspondence.

IF A DUPLICATE COPY OF THE MESSAGE AS TRANSMITTED IS DESIRED, CHECK HERE

APPROVED BY: *H. F. Stringfield*

TIME: \_\_\_\_\_ A.M. \_\_\_\_\_ P.M.

WCX-876 (Jan'48)

~~SECRET~~

ORNL  
CENTRAL FILES NUMBER

50-8-106

ORNL  
MASTER COPY

Date August 28, 1950

Subject Teletype re: Request for 300

Old Slugs.

Copy # 20

Central Files

By V. D. Donihee

To H. F. Stringfield

<sup>59</sup>  
<sup>FANI</sup>  
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to the public by:

David R. Hamlin 1/31/95  
Technical Information Officer Date  
ORNL Site

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By \_\_\_\_\_ Date \_\_\_\_\_

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By \_\_\_\_\_ Date 8/31/71

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50-8-106

THIS DOCUMENT CONSISTS OF 1 PAGES  
NO. 2 OF 2 SERIES C.

HM V D DONIHEE US AEC RICHLAND WASH  
TO US AEC OAK RDIGE TENN  
ATTN H F STRINGFIELD  
MSG NR 1439 AUGUST 30282134Z GR 37

~~RESTRICTED DATA~~  
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/CONTAINS AEC RESTRICTED DATA/

REF CLN STRINGFIELD EIGHT THREE SEVEN  
RE CLN YOUR REQUEST FOR THREE HUNDRED OLD SLUGS PD THE ESTIMATED  
FIGURE IS FIVE HUNDRED AND SIXTEEN GRAMS PAREN FIVE ONE SIX PAREN  
OF PRODUCT PD

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Ted Davis 1/30/95 Date  
ADD signature  
Single rereview of CCRP-declassified documents was authorized by DOE Office of Declassification memo of August 22, 1994.

Rec'd by cm at 1451Z  
Decoded by cm at 1455Z

~~SECRET~~

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5 7 1720

To: F. L. Steahly through F. L. Culler  
 From: Wm. E. Unger  
 Date: November 13, 1950

INV 62

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By Authority Of \_\_\_\_\_

By BMG Date SEP 1 1971

DISTRIBUTION

1. F. L. Steahly
2. F. L. Culler
3. W. E. Unger
4. F. B. Bruce
5. J. O. Davis *by receipt*
6. D. G. Reid
7. W. K. EISTEK

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PART NUMBER III

QUARTERLY REPORT

INV 64

TITLE: 706-D Modification Project Plant Design

WORK BY: G. B. Berry, D. Bottenfield, R. V. Foltz, J. E. Much  
W. E. Unger, E. Wischhusen, and R. H. Vaughan

This document has been approved for release to the public by:

*David C. Hamlin* 1/30/95  
 Technical Information Officer Date  
 ORNL Site

November 13, 1950

SUMMARY

The modification of 706-D building Kala facilities was planned in three phases to accommodate the production schedules required by the consumer. Subsequent schedule changes have allotted more time for testing equipment than was originally anticipated.

The installation of the crud and process filters (Phase I and II) is complete, but additional testing and process development is required to resolve filtration difficulties, attributed to silicic acid colloids, that developed during initial cold test runs.

The first full-scale production run is now scheduled by the consumer for January 1, 1951. Whether the new purification equipment (Phase III) will be available by that time, depends upon the delivery of essential components that has been adversely affected by the development of the international situation.

CONSTRUCTION

The 706-D modification project was divided into three phases to minimize the plant shutdown necessary and to accommodate the production schedule required by the consumer. Phases I and II required plant shutdown, and were to have been completed in time to permit an intermediate run by September 1, 1950. Phase III, new purification and loading equipment, was to have been ready to operate by November 1, 1950.

New developments changed the consumer's requirements, the intermediate run scheduled for September was cancelled, and the November start-up date was tentatively deferred until January 1, 1952.

Phase I and II are complete, phase III about 40% complete. The international situation has adversely affected deliveries of critical items essential to phase III, (purification equipment), and its completion before December 15, 1950 is unlikely.

TEST RUNS

Four complete cold runs have been attempted in the filtration equipment: two runs with Clinton slugs, and two with Hanford slugs. Filtration of the sulphated process solution proved troublesome and the difficulty was soon traced to silicic acid, from the 10 ppm silicon impurity in the uranium slugs and in the silicon-aluminum alloy bonding agent that survives the caustic jacket removal operation. The silicon chemistry is imperfectly understood, but apparently the silicon is converted to silicic acid during the metal solution with nitric acid. The silicic acid, though insoluble, is colloiddally dispersed and in this form passes through the crud filter. The precipitation of the product by sulphation of the metal solution, also causes agglomeration of the silicic acid colloid to form an infiltrable gel which progressively plugs the process filter.

November 13, 1950

Investigation is under way to develop remedial techniques. Selection of a better filter aid in the crud filters that will remove the silicic acid colloids, and determining the precipitation conditions that will inhibit agglomeration of what silica passes the crud filter will suffice to prevent further filtration difficulty. A design has been developed for a filter which will have about six times the filtration area if this should prove desirable.

Cold Hanford slugs, sufficient for four test runs, are on hand for such cold test runs as may be required. It is tentatively planned to make one cold Hanford slug run and one hot Clinton slug run during the month of December, and the full-scale hot Hanford slug run on January 1, 1951 or later as required by the consumer.

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*withdrawn*  
*12/24/95*

B-13

50-9-51

Date September 11, 1950

Subject ReLa Shipment - October, 1950.

ORNL  
MASTER COPY

Copy # 2A

By J. H. Roberson

C. E. Larson

To C. E. Larson

COPY  
Forwarded By  
G. E. LARSON

INT.

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INT. 64

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UNITED STATES  
ATOMIC ENERGY COMMISSION

In Reply Refer To:  
RMA:JS

51

Oak Ridge, Tennessee  
September 11, 1950

Carbide and Carbon Chemicals Division  
Union Carbide and Carbon Corporation  
Post Office Box P  
Oak Ridge, Tennessee

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By Authority Of \_\_\_\_\_

By ATG

Date AUG 31 1971

Attention: C. E. Larson, Director  
Oak Ridge National Laboratory

Subject: RALA SHIPMENT - OCTOBER, 1950

Gentlemen:

We have been advised by the Los Alamos Scientific Laboratory that they will require the next Rala source to be delivered to their site Monday, October 30, 1950, instead of October 1, 1950; hence it may be possible to extend the time needed for construction of the new facility.

Los Alamos has the responsibility for furnishing the new shipping container, but they will not be equipped to handle it in their extraction plant until about January 1951. Planning should be on the basis of using the old container until advice is received that Los Alamos is equipped to handle the new container.

Your cooperation in this matter is appreciated.

Yours very truly,

CLASSIFICATION CANCELLED

*J. Morgan 1-31-95*  
ADDITIONAL

Single review of CCFP-classified documents was authorized by DOE Office of Decommissioning (memo of August 22, 1991).

John H. Roberson  
Director of Research and Medicine

CC: C. E. Center, K-25  
R. W. Cook  
K. A. Dunbar

Shilling:mw

[REDACTED]

[REDACTED]

[REDACTED]

50-10-208 FLS-484

Cy. 2 of 5 copies Series FLS.

*File*

~~SECRET~~ *draft*

CF

October 31, 1950

J. O. Davis

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Wm. E. Unger

Or Changed To \_\_\_\_\_

RaLa Silica Problems

By Authority Of *Doc*

By *ECS* Date *AUG 31 1971*

1. The 706-D RaLa process entails separation of the product, barium sulphate, from the slug solution by filtration. Obtaining consistently satisfactory filtration rates has been a problem.

Dr. F. L. Steahly has requested that full advantage be taken of your experience and facilities to develop as speedily as possible a confirmation of the problems, and a solution. The following is an outline of information gained to date.

- 2. The operation consists of the following steps-
  - a. Slug jacket solution and removal in caustic
  - b. Slug solution in nitric acid
  - c. Crude filtration using filter aid
  - d. Product precipitation with sulphuric acid at 90°C
  - e. Filtration (process) of product without filter-aid
  - f. Metathesis of precipitate with potassium carbonate solution
  - g. Filtration (process) of the Metathesised precipitate

The process filtration of the sulphate precipitate proved difficult in early Semi-Works studies. Addition of the crude filtration step eliminated the difficulties.

A Semi-Works Mock-up of D-Bldg. equipment, full scale in every respect except the slug dissolver, demonstrated satisfactory operation consistently. The small dissolver, however, necessitated many small dissolvings, the dissolver solution being stored until a volume sufficient for a run had accumulated, (a week or so).

3. Five runs have been attempted in D-Building equipment. Nos. 1 & 5 were made with Hanford slugs, Nos. 2, 3, & 4 were made with CL slugs. Hanford slugs are cased beneath an aluminum - Silican alloy melt, which metallurgically bonds with the uranium slug and the aluminum can, providing the heat transfer necessary to carry away the energy generated in the slug during neutron bombardment. Apparatly, the adherent bonding flux residue that survives the caustic jacket removal step, contributes the only difference between the Hanford and Clinton slugs. (The respective silicon impurity contents of the uranium slugs should be checked, however).

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Single rereview of CRIP-declassified documents was authorized by DOE Office of Classification Management August 29, 1995

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*Dwight Johnson*  
Technical Information Officer  
ORNL Site

INV 52

October 31, 1950

The first cold run, with Hanford slugs, would not filter after sulphate precipitation. The sulphated solution was crud filtered, (removing the lead precipitate) and a new precipitation made by adding more lead to the sulphated solution. Filtration was satisfactory.

Filtration difficulties were blamed on silica. Apparently, the silica, as a poly-acid, was passing the crud filter, later agglomerating during the sulphate precipitation, to form aggregates of sufficient size to plug the process filter. Runs 2, 3 and 4, were aged slightly (about 2 - 3 hours) before crud filtering, and temperature was raised to 100° C and lowered to 30° C, to encourage the agglomeration of silica before crud filtration. After crud filtering the solution was protected from radical temperature rises, except during sulphation, to inhibit silica agglomeration. The three runs were successfully filtered.

The 5th run, made with Hanford slugs, following the procedure modification above, would not filter.

All metathesis filtrations have filtered satisfactorily, regardless of the history of the run;—further confirmation of the identify of the plugging agent as silica.

- (a. Higgins demonstrated that as little as 10 ppm Si would plug the process filter. Silica, as water-glass, was acidified with nitric acid, and the solution filtered thru a 6 inch diameter porosity to micro-metallic filter.)

4. The approach to a solution of the problem falls into three (3) categories:

- a. Silica agglomeration before crud filtration and inhibit agglomeration after crud filtration.

1. Temperature treatment: Prolonged boiling tends to accelerate agglomeration of unstable silicates. The effect of temperature changes before crud filtration should be evaluated. The dehydrating effect of sulphuric acid added during the sulphate precipitation, and the local heating from the heat of dilution of the sulphuric acid probably encourage silica agglomeration. The use of dilute sulphuric acid, or sodium sulphate for precipitation should be evaluated.

October 31, 1950

2. Acidity control: The Canadians (A.E.R.E C/R 548 April, 1950 - Butex Process, Fletcher & Johnson) report that slug dissolving, starting with 16 N nitric acid, and finishing not lower than 5 N, promotes the aggregation of silica into relatively coarse, filtrable particles. The nitrate ion tends to complex barium, however, and may result in intolerable product loss in the subsequent sulphate precipitation step.
  3. Precipitation or peptizing additions: The silica in the dissolver solution may be rendered filtrable by the formation of an insoluble silicate, perhaps by the addition of magnesium. Or, agglomeration after crud filtration may be inhibited by peptizing additives or agents that have a peptizing effect, such as tartaric acid.
- b. Use of compatible chemical processes.
1. The product precipitation might be carried out by a carbonate solution, the uranium remaining soluble as a carbonate complex. The silica is soluble in carbonate solutions. Unfortunately, the solubility of the uranium complex is not high, and the present equipment capacities may not accommodate batches of the required dilution.
  2. Precipitation of the product may be made before crud filtration, the product being dissolved from the filtering aid with versene, or with caustic and reprecipitated.
- c. Measures to remove silica.
1. The filter aid used is Sellite 545, Johns-Manville. It is prepared from diatomaceous earth by caustic treatment followed by calcination. The fused sodium silicates seal the interstices of individual particles and prevent the leaching out of soluble material that is objectionable to most consumers for its contribution to the color of the filtrate. The sodium silicate "flux" is acid soluble to a degree (some 10 to 20 ppm of silica per hour dissolve in our solution, it is estimated). This source of silica may be easily eliminated by the use of "unfluxed" filter aid.

TO: J. O. Davis

-1-

October 31, 1950

a. Measures to remove silica - continued

The commercial Sellite is imperfectly graded in size; the fines are not removed, and coarses are deliberately left in to improve the flow rates adversely affected by the fines. The fines are easily "cemented" by silica gels and sealed, the flow being diverted thru those channels having no fines and low silica removal efficiency. The grading of the filter aid is successfully accomplished in a Federal Classifier (K-25 has one in operation). The preferred range is 3 to 10 microns. Silica in neutral aqueous solution has reportedly been removed down to 0.1 ppm using calcified, unfluxed silica. A 25-pound sample of classified, unfluxed filter aid is being procured thru the offices of ~~L. Algren~~, A.E.C.

HA OHLGREN

2. Tap water, commercial nitric and sulphuric acids, all contain from 2 to 6 ppm of silica.
5. Hanford slugs for additional D-Bldg. test runs will be available about 6 November 1950, and selected remedial conditions can be put to the test beginning at that time.

It is important that the process be tested repeatedly with cold runs, and at least one tracer run, in time to begin a high level production run 28 December 1950.

WEU:dc

cc: FLS:teahly  
FLCuller  
EJWitkowski  
WEUnger

---

Wm. E. Unger

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C-5  
Pg. 2  
VLS-2.

TO: F. L. Culler

FROM: Wm. E. Unger

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ANI

SUBJECT: Sala Status Period November 8th to November 21, 1950.

DISTRIBUTION: FLCuller  
WEUnger (3)

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Technical Information Officer Date  
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Rela Status Period November 8, to 21, 1950

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DOCUMENTS WAS AUTHORIZED BY DODFC  
EXACT FILTRATION MEDIA OF AUGUST 1950

Silica Filtration Problem

UO section has found a factor of 20 difference in the flow rates of Carpenter 20 media and 347 media, both of G (4-10  $\mu$ ) porosity. The filters were small, were assembled by bead welding between pipe nipples, and admittedly were overheated badly during assembly. However, surface tension measurements gave substantially the same readings for both filters. The media will be compared again in filters assembled by gasketing, to obviate the effects of welding heat; and a use test will give the relative "pluggability" of the two media.

The Lab section has thoroughly reviewed all pertinent available data and have concluded that the problem will require more fundamental investigation than was originally anticipated. The filtration problem of plugging will be confirmed on 1:1000 scale, the factors influencing the plugging will be identified, and remedial techniques based on this and fundamental chemistry studies recommended. Conflicting observations of studies to date dictate such a thorough and systematic approach to the problem even at the expense of time. It now seems improbable, because of the complexity of the problem, that an answer can be expected in less than 8 week's time.

A filter of increased filtration surface will be fabricated, and installed in D-Building. This is the only expedient to ameliorate the silica filtration problem that is available at the present time.

D-Building Construction

The new carrier crane has been installed. The lead shielding around the floor pits is about 40% complete. Shop fabrication of the manipulator, formerly held up for delivery of the aluminum casting, is now proceeding smoothly. Flow-meter assembly, a discouraging problem, is now beginning to shape up and shows promise of successful operation. Ion chambers and indicating equipment is

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fabricated and ready for installation.

Deliveries of components materials have been firmed-up and all appear to be in line. Fabrication here of the stainless steel of the tantalum evaporator has improved delivery of the evaporator to December 15, 1950.

Crawford Priority

V. L. Looney, A.E.C., has forwarded to Washington, A.E.C. for final action a request for priority assistance for the Crawford Fitting Company's machine tool orders on Warner and Swassey. Looney has supported his request with statements of anticipated needs solicited from other A.E.C. installations and from Bechtel in connection with Arco-Chemical requirements. Looney is prodding the Washington-Office along and believes that the ball is rolling.

MTR Rala

There is a strong temptation in the Laboratory section to put Blanco on the silica job in jeopardy of the prosecution of the MTR Rala process development. We view this with alarm, because, although the silica problem is important and urgent, the MTR Rala development is scarcely less so.

Earl Shank is anticipating the need for corrosion information in the final selection of the MTR process, and is setting up test conditions for the metallurgy people.

Personnel

Following is a brief description of the present work load.

Foltz - Disconnect assembly - cubicle (TD-2001), A-Cell Channel Piping Detail (TD-1859), Channel Piping Loading Cubicle (TD-2002), Process Filter Type 2 (TD-1934).

Berry - Defected as of November 17, 1950.

Ruch - Air heater equipment for drying of final product. Assembly and organization of final design report.

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Vaughan - Panel Board Piping Detail. Field inspection and expediting.  
Procurement follow-up. Vacation November 27 to December 6, 1950.

Bottenfield - Drawing check. Investigation of vacuum transfer of lead  
shot. Organization of preliminary design report for MTR - Rala.

Wischhusen - Field inspection and equipment expediting. Loading cubicle  
valve and jet schedules. General Administrative Details.

Wm. E. Unger

WEU:rw

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*David Hammond 3/4/95*  
Technical Information Officer: \_\_\_\_\_ Date \_\_\_\_\_  
ORNL Site

**51-1-128**

F. L. Culler

January 2, 1951

W. E. Unger

Classification Cancelled or Changed  
TO \_\_\_\_\_

Rala Status 8 December to Date

By Authority of

*[Signature]*  
Name \_\_\_\_\_ Title \_\_\_\_\_ Date **AUG 31 1971**

706-D Modification Project.

Scheduled for completion 1 March 1951

Design 95% complete

Construction 70% complete

CLASSIFICATION CANCELLED  
*Ted Davis 3/8/95*  
SINCE RECLASSIFIED BY DGE ON 8/2/1971  
AUGUST 2 1971

Schedule:

21 December 1950, Dr. Phil Hammond, Los Alamos, via E. J. Witkowski, stated that the first regular sized batch will be required early in March, 1951; or, as a remote possibility, as early as 15 February (corresponds to starting the run as early as 1 February). This first batch is to be made in the new equipment and shipped in the new carrier. The new carrier is to be shipped to us at least two weeks before startup of 706-D building facilities. A 10 Kc batch will not be required before late in March.

Costs; Funds expended to date:

The following cost figures were obtained from Bob Martin, will be supplemented by later charges to be posted sometime next week.

	<u>Allotted</u>	<u>Expended as of:</u>	
		<u>30 Nov.</u>	<u>15 Dec.</u>
Structural, Shielding	\$159,000	\$ 55,000	\$ 85,000
Process Equipment	129,000	73,000	74,000
Special Equipment	33,000	36,000	37,000
Total	\$311,000	\$164,000	\$196,000
Balance as of 15 December:	\$311K - 196K	\$ 115,000	

TO: F. L. Culler  
From: W. E. Ungor

-2-

January 2, 1951

Silica Problem

The Laboratory Section investigation of the silica filtration problem has narrowed to three possible remedial measures:

(a) Apparently the silica causing the filtration difficulties is contributed predominantly by the bonding agent that survives the jacket removal step. It is proposed to re-institute the final acid wash to assist in undercutting and breaking loose residual insoluble uranium - aluminum silicates formed on the slug surface by the caustic jacket removal solution. Experiments are underway to determine the barium loss.

(b) The use of an anion exchanger to complement the filter aid in the crud filtration operation. The anion exchanger will selectively absorb sub-micron silica agglomerates, larger agglomerates are mechanically retained by the filter aid. Tests are underway to determine whether or not the anion exchanger absorbs barium.

(c) Fluoride ion combines with silica and inhibits its agglomeration. The fluoride ion can be added to the metal solution after crud filtration either as hydrofluoric acid or as sodium fluoride. Aluminum is then added to complex the excess fluoride ion and minimize corrosion. It is believed that the corrosion will be negligible, but investigation of the corrosion rate under process conditions is under way.

A modified process filter of about 3 square feet of filtration area is nearing completion and will be tested in a cold run in 706-B building the week of 8 January. Micro-Metallic Corporation will be willing to fabricate the units for about \$500 each, delivery 3 weeks from receipt of purchase order bearing a priority.

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TO: F. L. Culler  
FROM: W. E. Unger

January 2, 1951

**Construction Status:**

The present status of D-building construction is briefly outlined below:

<u>Status</u>	<u>Required Before Operation</u>
Manipulator installed	Install limit switch wiring Install charging heads, channel piping and disconnects. Install Air heater and piping. Conduct product transfer and drying tests.
Cover shell fabricated and poured with lead.	Use test, including covering and uncovering carrier, sealing and unsealing cone.
Pit lining completed	Install $\phi$ and $\gamma$ threshold chambers and electronic instrumentation. Install and test periscopes and pit lights.
Panel board erected	Install eluate tank system. Install pressure instruments and piping. Install electrical instruments and wiring. Install services piping.
Cubicle frames and shrouds fabricated. Some tanks and piping installed. One sampler unit installed Plug valves 90% completed, some under test.	Install Santalum evaporator, column assembly, complete piping, test for leaks and operability; Install in pits, connect instrument fittings, test instruments and connections; Conduct test runs. Column flushing and plug valve positioning to receive exacting check.

Every effort will be made to complete the project except for cold run testing by 1 February 1951. Overtime will be authorized liberally wherever the chances of making the deadline are enhanced by so doing.

WEU/de

W. E. Unger

51-1-141

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FLS

To: F. L. Steahly

January 18, 1951

From: E. B. Blasco  
I. N. Higgins

FLA-547

Subject: Proposed Schedule for Testing 706-D Re La Equipment

Classification Cancelled or Changed

TO  
By Authority of

AUG 31 1971

Available time: February March

- (1) Test and clean equipment - 2 weeks
- (2) Run I - Low Radiation Level - 1 week

Name	Title	Date
		3/4/95

Test of Ion Exchange Equipment

Synthetic ion exchange feed at 10 millicurie level. Must change recording resistors to  $10^5$ ,  $10^{10}$ , and  $10^{11}$ . Obtain complete product analysis for contaminants.

This document has been approved for release to the public by:

- (3) Run II - Moderate radiation level - 1 week

*David Robinson* 3/4/95  
 Technical Information Officer: Date  
 ORNL Site

Test of Correlation of Old and New

Equipment. Dissolve 35 cold W slugs and 1 hot I slug. Purpose: (a) To determine the efficiency of transfer from the old equipment to new, (b) to determine effect on ion exchange of impurities derived from actual operating equipment. Complete product analysis.

- (4) Run III - High Radiation Level Test - 1 week of complete process.

Dissolve 76 W slugs - 7000 curies. Purpose: Test complete process excluding final shipping cone evaporation. Product would be evaporated to 50 ml for use in Run IV. Determine capacity loss in resin during one process cycle.

- (5) Run IV. High Radiation Level Test - 1 week of Resin Stability.

Transfer product from Run III to head tank and make up synthetic feed. Adsorb feed on column and wash with water for 20 hours at normal flow rate to remove heat and gases. Determine resin capacity loss.

Purpose: To determine stability of resin.

Distribution:

- 1. FLS
- 2. FBlasco
- 3. JDeVie
- 4. FICuller
- 5. WInger
- 6. EWithowski
- 7. IHiggins
- 8. RBlasco

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*Ted Davis* 3/8/95  
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51-2-196

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TO: F. L. Culler

*Dwight Hamner* 2/2/95  
Technical Information Officer Date  
ORNL Site

FROM: Wm. E. Unger

SUBJECT: Status Report - 2 January 1950 and 22 February 1951

706-D RALA



2/26/51

The 200 (South) cubicle is complete except for two tantalum-lined fittings which were reportedly shipped by the fabricator yesterday.

The 300 (North) cubicle is about 30% complete, waiting on tantalum-lined evaporator, reportedly nearing completion.

Cold test runs will be begun in the 200 cubicle toward the end of next week.

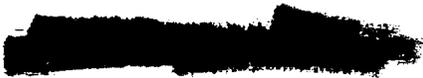
The charging pit with its manipulator, changing heads, and air heater are installed. The heater elements ordered with inconel sheaths, turned out to have 400-series stainless sheaths which corroded badly in less than 24 hours total operation. Inconel-sheathed replacement appears to be satisfactory. 500 ml simulated product solutions have been evaporated to dryness in about 9 hours. Splashing has been detected but is not excessive. Most of the latent heat of evaporation is probably supplied by conduction to the cone tip from the high temperature zone near the rim of the cone. The drying tests have been made with a chrome-plated brass dummy cone, which may conduct appreciably more heat than the platinum-lined stainless cone designed by Los Alamos. Los Alamos has promised to send a platinum-lined cone with the carrier.

The carrier, which Los Alamos calls a "pot", was shipped to Los Alamos by the fabricator 13 February, according to the AEC. We will probably receive the pot and cone, about 1 March. Final manipulator adjustments, simulated loading and drying tests would then be completed by 15 March.

The filtration problem is under control. A "type #2" filter with cylindrical labyrinth Media of 3 square feet area, was fabricated and tested. The Media of which it was made was visibly porous in areas; the surface tension test yielded only about 5 inches in contrast to the 18 inches to be expected of this porosity. As was expected, lead losses were high, corresponding to associated product losses of 5-10%. Filtration rates were high, better than 1 gallon per minute average and with only 30% decrease between initial and final rates. The U. O. Section attributed the failure of the Carpenter-20 flying saucer filters to a low sintering temperature of the Carpenter-20 which permitted the pores of the Media to fuse and close during welding. Two type #2 filters have been ordered from Micro-Metallic, and work orders were issued to build two 347 flying saucers. Ideally, one of each type would be installed and the two types compared, the final selection based upon resultant lead losses and filtration rates. Probably the 347 flying saucers will be used if the filtration rates prove satisfactory; otherwise the type 2 filters will be selected, regardless of the lead losses.

*Distribution:*  
1. F. L. Culler - [redacted]  
2. W. E. Unger - [redacted]  
3. J. E. [redacted] - [redacted]

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FLS 594



The lead shot transfer problem was given efficient attention by the Unit Operations Section. Shot transfer with a vacuum pump was tried and found feasible but had two outstanding disadvantages, the cost of the vacuum pump equipment and the ease with which the shot would "choke" (the formation of a column of shot greater than the vacuum pressure differential could support). This suggested a compressed air "pushing" action. An ingenious air jet was devised by the U. O. Section which lifts about 40 pounds of #4 lead shot per minute to a height of 10 feet. The jet is about 2 inches in diameter, will be the tip of a lance which will be used like a vacuum sweeper to unload the shot from the cubicle covers. An iron tank with a hopper bottom will be provided to receive the shot. The air exhaust from the tank will be laden with lead dust and may be a health hazard. This is being investigated.

Los Alamos has been called upon to produce a product of greater purity and size than heretofore. Dr. Phil Hammond of Los Alamos visited Oak Ridge, 2 February. The following schedule, he said "was being confirmed by letter thru the AEC":

Test run	1 April	1,500 + curies
Full scale test	14 May	30,000
Production	1 July	30,000
And every quarter thereafter		30,000

Los Alamos will not be able to tolerate more than 20% cerium by weight (Ba La Ce) which corresponds to a shipping time of about 2 days from LST, the last separation time of barium from its contaminants. This may require air shipment of the product which poses additional problems, loading equipment at Knoxville capable of removing the "pot", which now weighs about 8,000 pounds, from a truck and into the airplane, refrigerating equipment suitable for installation in the plane for cooling the pot, etc. Although the transportation of the product is clearly the AEC's responsibility, the AEC has no concrete plan and the job probably will ultimately be ours.

In the event that the shipping time must exceed 2 days, Los Alamos will merely discard the first milking, and this may prove most economical in the final analysis.

The 706-D Modification Project was initially planned for 2,200 curies, soon jumped to 10,000. The purification units are designed for a total life of 2 years, one year per cubicle, at a production rate of ten 10,000 curies-runs per year or 10<sup>7</sup> curies total life per cubicle.

Increasing the capacity of the runs to 30,000 curies involves employing both purification units in parallel, (sacrificing the production security of standby alternate equipment) and running each unit at 50% over design capacity. Fortunately the versatility of the equipment makes this physically possible, and the accumulated product of both cubicles can be shipped in one "pot", or the 15,000 curies can be shipped in each of two "pots". Los Alamos has had built only one carrier, however.



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The plastic valves, the plastic gaskets on the flow meter and column assembly, and the plastic insulation on the liquid level telemeter and monitor instrument wiring are probably the most vulnerable to radiation damage. Their expected life is based upon the best information available, but an error of a factor of 5 is not impossible and the life of the equipment may be greater than anticipated. Units may be replaced at a cost of about \$30,000 each, overhauled for about \$10,000 each.

The original Cell A equipment was designed for Clinton slugs, a great many of which were required; thus the dissolver has ample capacity for 300-400 Hanford slugs that will be required.

Carrier capacity for the shipment of slugs from Hanford is another matter. Four 39-slug carriers will be available, 2 iron carriers that were to have been retired from service because they are difficult to decontaminate, and two modified carriers to replace the iron ones, one built and one being fabricated (about 10% complete). It would not be practical to set up a bucket brigade cycle of slug shipment because of the nature of the dissolving step. (Jackets of all slugs are removed at once. Dissolving times vary from 4 hours for the first dissolving to 16 hours for the last.)

A new 148 slug-carrier has been designed. A minimum of shielding (9 inches of lead) will give a surface radiation of 50 MR per hour (cleared with HP and Hanford railroad people by E. J. Witkowski) but keep the total loaded weight below 6 tons. The thermal heat generated by the slugs, about 5,000 watts, is carried to a water jacket on the carrier by thermal siphon and dissipated to the atmosphere by free-convection. 100 3-inch fins will give the necessary surface area. It is probable that some boiling around the slugs will take place and the water jacket will probably operate at about 5 psi pressure. The design details will be forthcoming shortly in a separate report. The carriers will cost an estimated \$12,000 each. Originally two were planned, the Operations Division has since requested that 3 be built in the event that a lower barium content per slug would require the shipment of a greater number of slugs.

Hanford has reported that the 300-400 slugs will not upset pile operations at all, but cautioned us that a good two days might be required for slug selection and carrier loading.

Work orders for design, fabrication or procurement of the carriers have been issued and a separate sub-account has been set up under the 3900-788 Rala account to receive the charges.

MTR - RALA

No reportable progress.



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GENERAL



The group has from time to time run afoul of the procurement procedures, to the irritation of the Purchasing Department. We have been casual about observing some regulations, and have been ignorant of others. Last week we were the subject of a memorandum from Reams to Emlet Re: The Chromalox Heaters. We extended an apology to Reams and Overton of Purchasing, and Lee and Pike of Emlet's Staff. As soon as the heaters were found to be unsatisfactory we had contacted the Manufacturer's Representative in Atlanta. He was to "check further and advise us" which he did by letter, copy to the Purchasing Dept., informing us that replacements were being shipped at no charge. Purchasing received their copy of the letter before we did, and, concluding that we had made arrangements for procurement without consulting them, were rightfully indignant.

Costs of 15 - February 1951

Total \$241,582.



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Date January 8, 1951

Subject Ra La Process Filtration

This document consists of 5 pages.  
No. 1 of 15 copies.  
Series A.

To F. L. Steahly

From T. A. Arehart, R. E. Blanco,  
I. R. Higgins

**ORNL**  
**CENTRAL FILES NUMBER**

**51.1-12**

Before reading this document, please sign and date below:

\_\_\_\_\_  
\_\_\_\_\_  
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By Authority Of:

*AEC list 3/1/61*  
*R. M. E. H. C.*

For: H. I. Gray, Supervisor  
Laboratory Records Dept.  
**ORNL**

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*1/11/51*

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To: F. L. Steahly

-2-

Date: January 8, 1951

From: T. A. Arehart, R. E. Blanco,  
I. R. Higgins

Subject: Ra La Process Filtration

The purpose of this memorandum is to present Ra La processes changes which are recommended to alleviate the product filtration difficulties which have been encountered in the past.

#### 1.0 Recommended Process Changes

The results of Laboratory and Unit Operations studies indicate that the following modifications to the process should be made (on the basis of 76 Hanford slugs):

- (1) Substitution of a 3 ft<sup>2</sup>, "347" stainless steel process filter for the present 0.5 ft<sup>2</sup>, Carpenter "20" filter. This change has already been made.
- (2) Modification of the jacket removal step as follows:
  - (a) Use the present jacket removal procedure and,
  - (b) In addition, boil the slugs for 3 hours in sufficient 1.0M HNO<sub>3</sub> containing 0.20 gm/liter of Hg as mercuric nitrate to cover the slugs.
  - (c) Flush the dissolver very thoroughly with water before dissolving the charge.
- (3) Dilution of the dissolver solution to obtain a specific gravity, prior to crud filtration, of below 1.50.
- (4) The use of 800 grams of Celite 545 filter aid precoat on the crud filter and the employment of 800 grams of filter aid in the solution to be filtered.

Laboratory results indicate that past filtration difficulties are attributable to silica which may be rendered innocuous by a specific fluoride ion treatment. The fluoride procedure may be used in an emergency if filter

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plugging occurs, but is not felt necessary to employ it as a routine operation. The treatment consists of the following steps:

- (1) Back wash the filter with water.
- (2) Heat the solution to be filtered to the boiling point.
- (3) Add two pounds of 48% HF per 300 liters of solution. Agitate 1 minute.
- (4) Add 24 liters of 2M  $\text{Al}(\text{NO}_3)_3$  per 300 liters of solution.
- (5) Cool to 50°C and filter.

## 2.0 Discussion of Experimental Results

### 2.1 Filter Choice

Filters made of 347 sintered stainless steel are more desirable than those made of Carpenter 20 stainless steel since they are less subject to weld injury in fabrication and have less tendency to become plugged when filtering suspended materials such as colloidal graphite and silicon.

Despite very small weld leaks and a plate flaw, the labyrinth process filter successfully filtered freshly precipitated  $\text{PbSO}_4$ . This 3 ft<sup>2</sup>, 347 stainless steel "labyrinth" filter, was built at ORNL to replace the original 0.5 ft<sup>2</sup> Carpenter 20 filter. Since the flows may conceivably increase in size with use, it is recommended that this filter be used only for cold runs and that it be replaced by one free of flaws and capable of passing the air pressure porosity test before any hot work is attempted.

### 2.2 Jacket Removal

The present jacket removal procedure leaves a large amount of black, siliceous scale on the surface of the slugs. In addition, it is assumed that a quantity of scale settles on the bottom of the dissolver and is not removed when the solutions are drawn off. Laboratory runs on 0.26% of process scale showed that the presence of this siliceous matter in the dissolver solution was a direct cause of low process filtration rates. In each case, however, when

the siliceous material was completely eliminated the sulfate solution filtered in less than 1 hour using either a 347, or a Carpenter 20 stainless steel filter.

The siliceous scale was satisfactorily eliminated by boiling the slugs for 3 hours in 1.0M nitric acid containing 0.34 gm/liter of mercuric nitrate, after the completion of the present jacket removal steps. All suspended material was completely removed from the dissolver tank by vigorous flushing with water. The barium loss obtained in laboratory tests of this procedure was 0.02%.

It is concluded that every effort must be made to flush the silica scale from the dissolver tank. However, complete scale removal is not feasible since the scale will remain at the points of contact between slugs. In this case, it may be necessary to use either the anion resin or fluoride treatment since they have been shown to be adequate for elimination of reasonable amounts of silica.

### 2.3 Filter Aids and Agglomerators

Celite 545 is recommended over Dicalite 226-T because the former has a six fold faster filtration rate and, in addition, the latter contains acid soluble material which colors the filtrate a light yellow.

It is also recommended that the crud filter precoat be increased to approximately 1", because it was found that colloidal graphite penetrated a precoat of Celite 545 at least 1/8" and it is conceivable that colloidal silicon would penetrate at least this far.

The study of Aquadog (colloidal graphite) and Darco 6-60 (activated carbon) as silicon agglomerators was discontinued because silicon analyses are so poor that no conclusions could be reached. Visual inspection, however, indicated that Aquadog readily agglomerated and it is possible that the silicon was also agglomerated by it.

### 2.4 The Fluoride Treatment

A treatment for removing silica from uranium containing solutions has been patented in France. The procedure calls for adding concentrated HF at the rate of 1.56 g/l to the boiling and vigorously agitated solution.

When applied to the Ra La filtration problem, this method proved to be the most effective of any tried. However, because of fluoride corrosion, its application should be considered only as a last resort. Corrosion tests were run to evaluate this procedure. The solution containing 0.08M HF corroded stainless steel about 20 times faster than the 3.5M H<sub>2</sub>SO<sub>4</sub> alone yielding a corrosion rate of 200 mils per year.

In the proposed treatment aluminum is added to the solution to complex fluoride ion after the reaction with silica has occurred. With Al<sup>+3</sup> added to about twice (0.15M) the HF concentration, the fluoride corrosion was virtually stopped. The fluoride action on silica is effective, provided the Al<sup>+3</sup> is added at least one minute after HF addition. It is felt that the use of this treatment is entirely feasible in case filter plugging does occur.

#### 2.5 Use of Anion Exchangers for Silica Removal

Since strong base exchangers are recommended for silica removal, this approach was applied, with marked success, to the Ra La filtration problem. The amount of exchanger required has not been determined exactly but is approximately 10 liters per batch. In all runs employing Dowex A-1 resin, the filtration rates were much faster than with the regular Celite 545 treatment. Since the phenomenon is considered to be ion exchange rather than filtration, the factors of temperature, contact time, and bed depth were considered and found to be significant. Large particles of crud were also observed to be filtered out. Only a 0.1% loss of Ba was observed on the resin.

For application in the 706-D building equipment the star could be replaced by a flat, 700 x 60 mesh screen in the exchangeable crud filter unit. In use, 10 liters of the nitrate form of Dowex A-1 would be used to form a bed 8" thick x 8" diameter and the dissolver solution crud filtered through this at 50° - 60°C in a minimum of 1 hour.

This process is not recommended because it would require considerable revision of the existing equipment.

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1-4-51  
H/S/P

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OPERATED BY  
CARBIDE AND CARBON CHEMICALS DIVISION  
UNION CARBIDE AND CARBON CORPORATION  
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OAK RIDGE, TENNESSEE

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By Authority Of \_\_\_\_\_  
By *JOK* Date AUG 31 1971

DATE: January 19, 1951

SUBJECT: RaLa Schedules - Telephone Conversation  
with Dr. Phil Hammond, Los Alamos.

TO: F. L. Culler

FROM: W. E. Unger

ORNL  
CENTRAL FILES NUMBER  
51.1-54

COPY NO. 2A  
*F. L. Steakly*

CLASSIFICATION CANCELLED  
*W. E. Unger*  
ADD signature \_\_\_\_\_ Date 1/19/25  
Single rereview of CCRP-declassified  
documents was authorized by DOE Office of  
Declassification memo of August 22, 1994.

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LOCATION

TO F. L. Culler  
LOCATION Building No. 2067

DATE January 19, 1951

ANSWERING LETTER DATE

ATTENTION

COPY TO F. L. Steahly  
E. J. Witkowski  
F. R. Bruce  
Earl Shank  
Wm. E. Unger

SUBJECT RaLa Schedules -  
Telephone Conversation With  
Dr. Phil Hammond, Los Alamos

CLASSIFICATION CANCELLED  
*Wm. E. Unger*  
ADD signature Date 1/19/95

Single rereview of CCRP-declassified documents was authorized by DOE Office of Declassification memo of August 22, 1994.

Los Alamos has requested the first shipment 1 March 1951, of 1,600 to 10,000 curies. The run apparantly is to be experimental for Los Alamos, hence the indefiniteness of quantity. The run can be delayed until 15 March without inconveniencing Los Alamos, or, in the event that our new equipment is not ready by that time, Los Alamos will accept the run from the old equipment in the old carrier since Los Alamos "had planned to divide it up in the old facilities".

Beginning 1 April (tentatively) Los Alamos wants 30,000 curies in one shipment of one carrier, one shipment of two carriers, or two shipments of 15,000 curies each spaced as closely as operations will permit. Los Alamos is to deliver 11,000 curie batches of lanthanum, requiring a minimum of 15,000 curies of Ba-140 at LST. Approximately 130,000 curies total per year will be required (about 25% in excess of design capacity for D-building.) Carrier capacity for slugs is now limited to 2 carriers on hand, one nearing completion, and one not yet started in the shops. The carriers each have slug capacity equivalent to 3,500 curies of Ba. To produce 30,000 curies will require at least 8 carriers, or 4 carriers making a double trip by air, and the runs will involve about eight dissolvings. The ion exchange columns (duplicate equipment) will be pushing their maximum to hold 15,000 curies each.

This new schedule places additional emphasis on the MTR RaLa which should be designed by 2 assembly dissolvings or 50,000 curies nominal capacity.

Dr. Hammond plans on visiting Oak Ridge within the next two weeks to seek information on high temperature solubility of UNH.

*Wm. E. Unger*  
Wm. E. Unger  
Project Engineer

~~This document contains information which is exempt from public release under Executive Order 11652, February 22, 1950, and is to be controlled under the provisions of the Atomic Energy Act of 1954, and the Atomic Energy Regulations promulgated thereunder.~~

WmEU:ms

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77

ORNL  
CENTRAL FILE NUMBER  
51-2-9

DATE February 5, 1951

SUBJECT Slur Handling Information

BY H. A. Ohlgren

TO Mr. Wm. Ginkel, IDO, Idaho Falls, Idaho

*HT*  
*3.5*

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64

Distribution

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*211*

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This document has been approved for release to the public by:

*David R. Hammin* *11/30/95*  
Technical Information Officer Date  
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28-44  
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Bldg. 2067  
Oak Ridge National Laboratory  
Oak Ridge, Tennessee  
February 2, 1951

To: Mr. Wm. Ginkel  
Division of Technical Operations  
Idaho Operations Office, USAEC  
Idaho Falls, Idaho

From: H. A. Ohlgren

Subject: Slug Handling Information

Mr. B. P. Shepherd submitted a list of questions to me for which you have requested answers.

As you are aware, it is not easy to give direct answers to all your questions without dwelling in detail on the background and philosophies of the thinking underlying decisions made to date. In my opinion, it would be far better to obtain such background by spending time here in Oak Ridge with the groups of people who have been involved. I urge that you give consideration to this suggestion.

Some problems are still in a state of flux and complete resolution will not be obtained until the plant is in operation, but I shall endeavor to give answers to your questions as they are pictured now.

1. How are NP slug containers (SS liner fitting in cask) to be handled:
  - (a) From cask to storage? (b) From storage to process?

Answer

(a) The casks and buckets for shipment from Hanford are being designed by G.E. I understand that each unit will weight about eight (8) Tons. Each bucket will contain 18 irradiated slugs. Shipment is by truck. Each truck will carry two (2) casks. Upon arrival the casks will be unloaded from the truck by means of a 15 T. crane onto the decontamination bay located on the platform. Inspection and washing will be done at this point. If hot spots appear on the surface of the cask, provisions are made for washing with a solution containing a detergent. The cask is transferred to the unloading pit by means of the 15 T. crane. The depth of the unloading pit is about 28 ft. with about 24 ft. of water cover. As the cask is lowered the cover to the cask is removed. Provisions are made so that when the cask reaches the bottom of the pit it shifts position in a manner to engage the bucket to the monorail transfer hook. The bucket is transferred to the weight scale thence to the storage basin where it remains attached to the hook.

(b) On transfer to process, the buckets are transferred to the center compartment of the unloading pit by means of the monorail. It is expected that six chargers will be loaded on the day shift. The operation for each bucket consists of dumping 18 slugs on the floor of the center compartment. By means of tongs 15 of the 18 slugs will be placed in a vertical position in the charger. Consequently, five buckets from storage will provide the 90 slugs for six chargers. At this point an accurate count of slugs can be obtained. The chargers are removed successively from the pit and stored on the platform. A Ross carrier is contemplated for the transfer of the chargers from the SF Storage Building to the Process Building.

[REDACTED]

To: Wm. Ginkel, IDO - Slug Handling Information

2. What are the shielding requirements for 1(b)?

Answer - As presently planned, the thickness of lead shielding in chargers provided is: EHR and NP Material - 10 inches; MTR Assemblies - 12 inches.

3. How are the slugs to be fed to chute? Dissolver batch will be of different size than shipping batch.

Answer - The correct number of slugs per dissolver batch is accounted for in transfer from the storage bucket to the charger. The charger as it leaves the Storage Building will contain 15 slugs.

The mechanism of charging consists in mounting the charger properly over the charging chute. A portable unit is now being designed to remove the drawer from the charger. Upon opening the slug chute valve and removal of the drawer, the slugs will drop one by one into the dissolver. A slug counter (life of which is expected to be about six months) is contemplated.

4. Will facilities and equipment be available for handling (underwater or remote control) individuals slugs at storage point? Yes. At slug feed chute? Yes, by means of counter. Elsewhere? No, except by calculation from weighing buckets.

5. Are radiation level data available for slugs?

Answer - In general, this question cannot be readily answered until accurate information on burnup is known. At present it is assumed that burnup at Hanford will average about 15% of the 25 in the slug. The range may be from 8% to 22%.

(a) After removal from Pile? (b) After 60-day cooling period? (c) After additional 60 - 120 day cooling period?

Answer- See below (Figures approximate only)

NP Slugs

<u>Days Cooling</u>	<u>Watts/Slug</u>	<u>Curies/Slug</u>
0	$1.3 \times 10^5$	$2.6 \times 10^7$
50	$1.82 \times 10^2$	$3.6 \times 10^4$
100	$1.56 \times 10^2$	$3.1 \times 10^4$
150	$1.3 \times 10^2$	$2.6 \times 10^4$

MTR Assemblies

0	$1.3 \times 10^5$	$2.6 \times 10^7$
50	$1.7 \times 10^2$	$3 \times 10^4$
100	$8.3 \times 10^1$	$1.4 \times 10^4$
160	$5.3 \times 10^1$	$9 \times 10^3$

To: Wm Ginkel, IBO - Slug Handling Information

- 6. Are any difficulties in decontaminating anticipated (a) shipping casks?  
(b) Containers (liners) after use what decontamination procedure is planned at Arco?

Answer - (a) No. (b) No. Presuming you mean the facilities which are provided for decontamination of casks and buckets. For DFing such there is provided a portion of the loading dock for mild hot spots. If levels are too high, we can transfer the units to the Process Building wherein conventional means can be employed.

- 7. Do CFP design personnel approve proposed cask and liner design? Ohlgren has print of cask drawing.

Answer - Yes

---

H. A. Ohlgren

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 APR 11 1978

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CENTRAL FILES NUMBER

51-2-89

206  
Iodine  
reacts

"This document consists of 6 pages.  
No. 1 of 4 copies, Series A"

To: W. G. Stockdale  
From: F. L. Culler  
Subject: Iodine in Dissolver Solutions  
Date: February 16, 1951

Classification Cancelled  
Declassified To  
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INV. 64  
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AND

Please sign and date before reading:

W. G. Stockdale  
F. L. Culler

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Intra-Laboratory Correspondence  
OAK RIDGE NATIONAL LABORATORY

To: W. G. Stockdale  
From: F. L. Culler  
Subject: Iodine in Dissolver Solutions

Date: February 16, 1951

CLASSIFICATION CANCELLED

*Jed Davis* 1/30/95  
ADD signature Date

Single rereview of CCRP-declassified documents was authorized by DOE Office of Declassification memo of August 22, 1994.

The following are data on  $I_2$  in dissolver solutions extracted from Hanford Progress Reports that will be of interest to you:

HW-19503--Chemical Research Section, October 1950.

REMOVAL OF IODINE FROM DISSOLVER SOLUTION (C.H. Holm and C.R. McMullen)

A number of experiments were done during this period to determine the degree to which iodine, added in the zero oxidation state to a simulated dissolver solution, can be removed by sparging. The effect of variables such as temperature, spargant and spargant flow rate, acidity and UNH concentration on the rate of iodine removal was studied. To obtain iodine in the zero oxidation state for these experiments, inactive iodide and Oak Ridge  $I^{131}$  were combined, treated with excess  $NaHSO_3$  to insure complete reduction of the  $I^{131}$ , treated with excess  $NaNO_2$  to oxidize the iodide to iodine, extracted into  $CCl_4$  and stripped from the latter with a basic solution. This final basic solution was used to spike iodine into the solutions to be sparged. Table I gives the results of sparging under a variety of conditions.

As expected, iodine removal increased as the temperature increased. During Experiment 24 there was an initial surge of air through the apparatus such that the per cent of iodine remaining was lower than it would have been had the air flow been constant. Also, as expected, increased spargant flow rate resulted in increased iodine removal.

The use of an inert spargant such as nitrogen appears to have a slight advantage over oxygen-containing spargants. Thermodynamically at least, oxygen will oxidize iodine to iodate. Since iodate would not be readily sparged from a solution, such a reaction might well account for the differences observed. These studies continue to be hampered by the lack of adequate tracer scale analytical methods for the determination of the fraction of iodine in any given oxidation state when the iodine may be present in several oxidation states.

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Table I

EFFECT OF SEVERAL VARIABLES ON THE REMOVAL OF IODINE  
FROM DISSOLVER SOLUTION BY SPARGING

Sparging Time = 3 hours

Expt. No.	Temp. (°C.)	Spargant	Spargant Flow Rate cc/min/ml	M		Residual Iodine %
				UNH	HNO <sub>3</sub>	
25	20	Air	1.7(1)	1.5	0.3	23
23	50	Air	1.8	1.5	0.3	18
29	95	Air	1.7	1.5	0.3	13
24	95	Air	2.0	1.5	0.3	3.5
26	95	Air	0.35	1.5	0.3	27
29	95	Air	1.7	1.5	0.3	13
27	95	Air	5.0	1.5	0.3	2.9
30	95	O <sub>2</sub>	1.7	1.5	0.3	16
29	95	Air	1.7	1.5	0.3	13
28	95	N <sub>2</sub>	1.7	1.5	0.3	10
29	95	Air	1.7	1.5	0.3	13
31	95	Air	1.7	2.2	0.3	12
29	95	Air	1.7	1.5	0.3	13
32(2)	95	Air	1.7	1.5	0.3	26
41	95	Air	1.7	1.5	-0.3	16
39	95	Air	1.7	1.5	0.1	7
40	95	Air	1.7	1.5	0.3	3.5
38	95	Air	1.7	1.5	0.7	7.3
33	95	Air	1.7	1.5	1.0	8.3
42	95	Air	1.7	1.5	1.25	5.9
37(2)	95	Air	1.7	1.5	1.25	10.0
36	95	Air	1.7	1.5	1.5	27
35	95	Air	1.7	1.5	2.0	36.0
34(2)	95	Air	1.7	1.5	3.0	17.6

(1) 1.7 cc/min/ml corresponds to roughly 115 CFM on a plant scale.

(2) Metallic uranium was not present in Experiments 32, 34, and 37. It was present in all of the other experiments.

Concentration of uranium seems to have little effect on the ultimate removal of iodine, at least for the concentrations studied. However, the initial rate of removal was considerably greater for the higher UNH concentration.

Continued failure to remove all of the iodine even after many hours of sparging had led to the idea that, in the presence of metallic uranium some iodine is reduced to iodide by contact with the metal and not subsequently re-oxidized in the solution. This conjecture was not substantiated by the results of Experiments 29 and 32 where, under conditions otherwise comparable, the iodine was less completely removed in the absence of metallic uranium than in its presence.

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Iodine removal as a function of acidity appears to go through a broad maximum in the range of 0.1 M  $\text{HNO}_3$  and then decreases rather sharply with increasing acidity. The decrease at higher acidities may be due to some oxidation to iodate.

Although results have been given only for three hours of sparging, iodine removal as a function of time was determined for each of the experiments described. These data have been plotted in terms of the logarithm of the per cent iodine remaining versus time. Some general comments may be made regarding the curves obtained. Straight lines indicative of removal of a single component were obtained only in the case of high acidity, 1.5 to 3.0 M  $\text{HNO}_3$ . Resolution of the curves at the lower acidities indicated three components of increasing difficulty of removal by sparging. Half-lives of from one to five minutes for the first-removed component, 15 to 50 minutes for the second and >150 minutes for the third were obtained in the various experiments.

Removal of active iodine from simulated dissolver solution by co-precipitation on cuprous iodide has been further investigated as a function of initial iodide concentration, acidity and concentration of cupric ion. I131 and inactive iodide were added to the simulated dissolver solution. Excess  $\text{NaHSO}_3$  was added to insure that all of the I131 was present as iodide and to reduce iodine produced by the subsequent reaction with cupric ion. Following a 10 minute stirring period, cupric sulfate was added and the solution was stirred an additional 10 minutes before the resulting cuprous iodide was removed by centrifugation. Table II shows the results, in terms of per cent iodine removed, for a number of experiments involving the previously mentioned variables.

Table II

REMOVAL OF IODINE FROM DISSOLVER SOLUTION  
BY CO-PRECIPITATION ON CUPROUS IODIDE

Dissolver Solution: 1.5 M UNH,  $\text{HNO}_3$  as indicated  
0.133 M  $\text{NaHSO}_3$

<u>M</u> $\text{HNO}_3$	Iodide Initial <u>M</u>	<u>M</u> $\text{Cu}^{++}$	% I131 Removed
0.5	0.001	0.05	22
0.5	0.0015	0.05	72
0.5	0.0025	0.05	86
0.5	0.003	0.05	92
0.5	0.004	0.05	94
0.5	0.005	0.05	96(1)
0.5	0.006	0.05	96
0.5	0.005	0.0125	85
0.5	0.005	0.025	91
0.5	0.005	0.05	95
0.5	0.005	0.075	97
0.5	0.005	0.10	>99
0.5	0.005	0.05	92(1)
0.5	0.005	0.05	97
0.3	0.005	0.05	99
0.1	0.005	0.05	99
0.3	0.005	0.05	92(2)

- (1) These two values were obtained using simulated dissolver solutions prepared from UNH in the first case and  $UN_3$  in the second. The acidities may thus not be exactly comparable.
- (2) Iodine added as iodate.

Precipitation of 0.005 M iodide appears to be adequate to remove about 95% of the iodine. More complete removal may be obtained if more than 0.05 M cupric sulfate is added or if the acidity is reduced to less than 0.5 M  $HNO_3$ . Cupric salts other than the sulfate could be used where sulfate is objectionable in subsequent operations.

The last experiment noted in Table II indicates that the procedure used will give adequate removal of the iodine if present initially as iodate.

HW-19739---Chemical Research Section, November 1950.

REMOVAL OF IODINE FROM DISSOLVER SOLUTION (C. H. Holm)

Experience in the operation of the radioactive iodine production unit at Oak Ridge has shown that the presence of very small amounts of mercury in dissolver solution renders the iodine very difficult to remove by sparging. In view of the poor results obtained in recent attempts to remove iodine from simulated dissolver solution by sparging it was felt that an investigation of means of insuring that the active iodine remains unvolatilized until disposed of as waste was warranted.

Since mercury is not the only cation mentioned by Oak Ridge as interfering with the sparging of iodine, several cations known to form complexes with iodide were tried in a preliminary survey. Simulated dissolver solution (1.5 M UNH - 0.3 M  $HNO_3$  -  $10^{-5}$  M I) was made  $10^{-4}$  M in the cation under study. The solution was then air sparged at 45°C. at a flow rate corresponding to 575 CFM on a plant scale. Table VII shows the per cent of active iodine removed when  $Hg^{++}$ ,  $Cd^{++}$ ,  $Zn^{++}$ ,  $Pb^{++}$  or  $Bi^{+++}$  was present in the solution. The data presented show that, of these ions, only mercury has an appreciable retarding effect on the sparging of iodine.

Table VII

REMOVAL OF IODINE FROM SIMULATED DISSOLVER SOLUTION  
CONTAINING VARIOUS IODIDE-COMPLEXING CATIONS

Simulated Dissolver Solution: 1.5 M UNH - 0.3 M HNO<sub>3</sub> -  
10<sup>-5</sup> M I<sup>-</sup> traced with I<sup>131</sup> as I<sup>-</sup>

Concentration of complexing agent = 10<sup>-4</sup> M

Sparging temperature = 450 C.

<u>Complexing Ion</u>	<u>Time Sparged, Min.</u>	<u>Active Iodine Removed, %</u>
Hg <sup>++</sup>	60	0.06
Cd <sup>++</sup>	60	57.0
Zn <sup>++</sup>	30	61.3
Pb <sup>++</sup>	30	67.6
Bi <sup>+++</sup>	40	69.4
None-control	60	77.0

For any procedure involving retention of active iodine in solution to be successful in preventing contamination of the canyon air, it must insure that iodine is not volatilized in any succeeding plant operation. Although actual sparging is done only in storage cells (to insure proper mixing of successive dissolver cuts), all solution transfers are by steam jetting. These jetting operations may represent a certain amount of sparging. The degree to which iodine is removed from simulated dissolver solution containing 10<sup>-4</sup> M Hg (NO<sub>3</sub>)<sub>2</sub> by air sparging after each process step from dissolution to the bismuth phosphate extraction was studied. Table VIII shows the iodine removed at each step when the iodine was added to the initial dissolver solution as I<sup>-</sup>, I<sub>2</sub> or IO<sub>3</sub><sup>-</sup>. These results indicate that, even with sparging considerably more extreme than would be encountered under actual plant conditions, a negligible amount of iodine originally present as I<sup>-</sup> and small amounts originally present as I<sub>2</sub> and IO<sub>3</sub><sup>-</sup> are likely to escape from the solution between the conclusion of dissolving and the extraction step if mercuric ion is present.

Assuming that the escape of iodine from dissolver solution is small prior to the extraction step it is then desirable that the iodine not be carried on bismuth phosphate in order that it can be removed in the metal waste. To check this point, the extraction procedure was performed on each of the three solutions resulting from the studies reported in Table VIII. In the case where the iodine was added to the dissolver solution as I<sup>-</sup>, about 10 per cent of the iodine was carried by the bismuth phosphate; for iodine added initially as I<sub>2</sub>, two per cent and as IO<sub>3</sub><sup>-</sup>, three per cent was carried. Since some iodine does follow the bismuth phosphate it will be desirable to determine the behavior of the iodine during a bismuth phosphate by-product precipitation.

These experiments will be repeated omitting the mercury to determine how much of the iodine behavior noted may be ascribed to the presence of mercury. The results so far obtained are sufficiently promising to warrant investigating of the effects which the presence of mercury in the feed may have on the TBP process. The fate of iodine when complexed by mercury in dissolver solution prepared for Redox is also being considered.

Table VIII

EVOLUTION OF IODINE ON SPARGING PRE-EXTRACTION SOLUTIONS

Sparging rate = 8.5 cc/m/ml = 575 CFM plant scale

Hg  $\bar{M}$  =  $10^{-4}$

I  $\bar{M}$  =  $10^{-5}$  inert. Traced with I<sup>131</sup>

Tank	Composition of Solution	Sparging Time, Min.			% I <sup>131</sup> Removed			
		I <sup>-</sup>	I <sub>2</sub>	IO <sub>3</sub> <sup>-</sup>	I <sup>-</sup>	I <sub>2</sub>	IO <sub>3</sub> <sup>-</sup>	
Dissolver	1.52 M UNH 0.31 $\bar{M}$ HNO <sub>3</sub>	90	60	180		2.3	0.7	
Dissolver	Wash Water	30	30	30		0.0	0.1	
4-8	1.05 M UNH 0.215 $\bar{M}$ HNO <sub>3</sub>	30	30	60		0.2	0.3	
4-8	1.02 M UNH 0.209 $\bar{M}$ HNO <sub>3</sub> 0.99 $\bar{M}$ H <sub>2</sub> SO <sub>4</sub>	30	40	40		1.9	0.1	
6-3	0.6 M UNH 0.13 $\bar{M}$ HNO <sub>3</sub> 0.88 $\bar{M}$ H <sub>2</sub> SO <sub>4</sub>	30	30	30		0.9	0.04	
6-3	0.58 M UNH 0.126 $\bar{M}$ HNO <sub>3</sub> 0.56 $\bar{M}$ H <sub>2</sub> SO <sub>4</sub> 0.115 $\bar{M}$ NaNO <sub>2</sub>	30	30	30		1.7	0.5	
					Total:	0.5	7.0	1.7

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Date SEP 1 1971

TO: F. L. Culler

FROM: B. F. Bottenfield and Wm. E. Unger



3/1/51

SUBJECT: Increased Carrier Capacity for Hanford Slugs Destined for Rala Production.

ORNL has received 4" W slugs from Hanford in a carrier of satisfactory design, the "Phoenix" carrier, D-7781, which has a capacity of 19 8" slugs, or 38 4" slugs, corresponding roughly to about 3,000 curies of Barium 140. The Operations Division has two such carriers, permitting the production per run of about 6,000 curies, a third nearing completion, and a fourth scheduled for fabrication, representing a total slug capacity equivalent of about 12,000 curies. Recent revisions from Los Alamos have increased the run capacity to 30,000 curies. Rather than build 6 more Phoenix carriers to supply the necessary slug capacity, the design feasibility of a larger slug carrier has been studied.

INV. 65

It appears feasible to build two carriers each having a capacity of 74 8" and 148 4" Hanford slugs. The heat generated by the radio activity of the slugs will be dissipated from the shell of the carrier to the ambient air by free convection. Water circulation will conduct the heat from the slugs to the shell of the carrier.

Inasmuch as it will probably require some slight amount of boiling to induce the flow of water, it is assumed that the temperature out of the top of the basket will be 212° F. and the temperature at the bottom of the basket will be 203° F.

This document has been approved for release to the public by:

Heat to be dissipated:

$$Q = 33 \text{ watts/slug} \times 148 \text{ slugs} \times 3.41 \text{ BTU/hr/watt} = 16,700 \text{ BTU/hr.}$$

*Daniel R. Hamins* 3/9/95  
Technical Information Officer Date  
ORNL Site

Flow Calculation:

Assume average temp. across basket = 207° F.

Calculated equivalent diameter:  $D = 1.42 \times 10^{-2}$  ft./slug row.

Cross sectional area (calculated) =  $1.04 \times 10^{-3}$  sq. ft./slug row.

CLASSIFICATION CANCELLED  
*Ed. Borden* 3/8/95  
Date  
Single review of CCFR-declassified documents was authorized by DOE Office of classification memo of August 22, 1994

$$G = \frac{33 \text{ watts/slug} \times 4 \text{ slugs/row} \times 3.41 \text{ BTU/hr./watt}}{1.04 \times 10^{-3} \text{ ft.}^2 \times 1.0 \text{ BTU/\#} \cdot \text{F} \times 9 \cdot \text{F} \times 3600 \text{ sec./hr.}} = 13.36 \text{ \#/sec. ft.}^2$$

Pressure Drop Calculation

$$F = \frac{2fLG^2}{\rho^2 g D} \quad \& \quad f = \frac{16\mu}{DG}$$
$$F = \frac{32\mu LG}{\rho^2 g D^2}$$

where: F = head loss due to friction (ft.)

L = length of slug row (ft.)

$\rho$  = average fluid density (#/cu. ft.)

g = gravitational constant

D = Equivalent diameter (ft.)

$\mu$  = average fluid viscosity (#/ft. sec.)

$$F = \frac{(32)(1.970 \times 10^{-4})(1.9)(3.34)}{(60.0)^2(32.2)(1.42)^2 \times 10^{-4}} = 0.0171 \text{ ft.}$$

This is the force that would be required to induce the water to flow at speed adequate to cost the slugs. Less than 0.0001#/sec. of water evaporated at the top of the slug basket would produce this force.

Radiation from jacket and top surfaces:

$$q = 0.173 A \epsilon \left[ \frac{T_1^4}{100} - \frac{T_2^4}{100} \right]$$

where A = area in square ft. - 33.9 sq. ft.

T<sub>1</sub> = Temp. of radiating surface (°R) = 667

T<sub>2</sub> = Temp. of absorbing surface (°R) = 550

$\epsilon$  = emissivity of radiating surface = 0.3

$$q = (0.173)(33.9)(0.3) \left[ (6.67)^4 - (5.50)^4 \right] = 2330 \text{ BTU/hr.}$$

Convection from Top Surface

$$q = h A \Delta T = (1.0 \text{ BTU/hr.} \cdot \text{F sq. ft.})(5.90 \text{ sq. ft.})(212-90) = 720 \text{ BTU/hr}$$

To be removed by convection from finned jacket.

$$16,700 - 3050 = 13,650 \text{ BTU/hr.}$$

Calculation of number of fins required:

$$q = n l \sqrt{2 s k f_F} \tanh \sqrt{\frac{2 f_F L^2}{k s}} + f_u A_u$$

where

$f_e$  = effective heat transfer coefficient/ft.<sup>2</sup> carrier surface

$n$  = no. of fins/ft. carrier circumference

$l$  = length of fin (ft.) = 1 ft.

$s$  = thickness of fin (ft.)

$f_F$  = air film coefficient - fins to ambient air

$L$  = length of fin projection into air stream (ft.)

$k$  = thermal conductivity of fin material

$f_u$  = Air film coefficient - unfinned surface to ambient air

$A_u$  = unfinned area (sq. ft.)

Assume an area of one ft. in circumference x one ft. of fin length =  
1 sq. ft. of carrier surface

Assume  $s = 1/8" = 0.01029$  ft.

Assume  $f_F = f_u = 1.00$  BTU/hr. - sq. ft. - °F

Assume  $k = 9.0$  BTU/hr. - sq. ft. (°F/ft.)

Assume  $l = 3" = 0.25$  ft.

Required effective heat transfer coefficient ( $f_e$ ) may be determined from  
information already at hand:

$$q = f_e A \Delta T$$

$$\Delta T = (207-90)^\circ F = 117^\circ F.$$

$A$  = area of carrier surface without fins

$$q = 13,650 \text{ BTU/hr.}$$

$$f_e = \frac{q}{A \Delta T} = \frac{13,650}{(28.0)(117)} = 4.16 \text{ BTU/hr.} - ^\circ F - \text{sq. ft.}$$

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$$4.16 = \left[ n(1) \sqrt{(2)(0.01029)(9.0)(1)} \tanh \sqrt{\frac{(2)(1)(0.25)^2}{(9.0)(0.01029)}} \right] = (1) \left[ 1 - \frac{(1 \times n)}{(8 \times 12)} \right]$$

$$4.16 = 0.349n + 1 - 0.010 n$$

N = 9.3 fins/ft. of circumference = 81 fins

Use 100 to give us a 20% safety factor. This will give approximately 12 fins/ft., which is within reasonable spacing limits for finned surfaces.

Approximately eight inches of lead was used for shielding the Phoenix carrier.

It has been recommended by J. A. Lane of the Reactor Technology Division that nine inches of lead be used on the revised carrier. This is intended to give a maximum radiation at outer carrier surface of 50 MR. This tolerance has been accepted by the Operations Division, and, through Operations Division, Hanford and all parties involved in transportation of the carrier. The total weight of the filled carrier is calculated to be very close to 6 tons, which is the load limit of the 706-D Bldg. crane.

Distribution

- F. L. Culler
- E. J. Witkowski
- J. A. Lane
- W. E. Unger

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COPY NO. 6 -A

DATE: March 7, 1951

SUBJECT: REVISED LOS ALAMOS RALA REQUIREMENTS

TO: Dr. John H. Roberson, USAEC-ORO

FROM: C. E. Larson

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*for 3-8-51*

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March 7, 1951

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United States Atomic Energy Commission  
Oak Ridge Operations  
Oak Ridge, Tennessee

Attention: Dr. John H. Roberson

Subject: REVISED LOS ALAMOS RALA REQUIREMENTS

Gentlemen:

Reference is made to your letter dated February 15, 1951, on the above subject.

The alterations of the Oak Ridge National Laboratory RaLa Plant for the production of 10,000-curie shipments of RaLa are essentially completed. A 1,500-curie shipment can be produced in the old equipment by March 15, 1951, or through the new ion exchange system by April 1, 1951. (The latter alternative is preferred by both Los Alamos and ORNL, since it will permit testing of the new system.)

A recent tentative schedule proposed by Dr. R. P. Hammond of Los Alamos has indicated the need of 30,000-curie shipments. His proposed schedule was somewhat different from that shown in your request to ORNL for evaluation of the practicability of 30,000-curie RaLa runs, so his more recent schedule is included. It is as follows:

- 1,500 curies or more - April 1, 1951
- 30,000 curies or less - May 14, 1951
- 30,000 curies or less - Two or three more during Calendar Year 1951

Since the equipment was designed for 10,000 curies per shipment and no run has yet been made demonstrating capabilities of meeting or exceeding the present ORNL commitment of 10,000 curies, ORNL is in no position to guarantee the production of 30,000 curies per shipment without at the same time being assured of funds and time for major changes in process equipment, if any are required. However, while a definite commitment for 30,000 curies per shipment is not being made, it is the considered opinion of the Laboratory that it will be possible to produce 30,000-curie shipments with no appreciable costs due to alteration of the RaLa Production Plant and with a relatively small amount of money (\$37,500) for capital equipment.

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*Arvin S. Zuck* 1/18/95  
ADD signature Date

Single rereview of CCRP-declassified documents was authorized by DOE Office of Declassification memo of August 22, 1994.

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USAEC  
Dr. John H. Roberson

March 7, 1951

While it may be possible to have the shields ready for an attempt at production of 30,000-curie shipments by May 14, 1951, it is intended that the second run be made at the designed level of 10,000 curies. The attempt to reach the 30,000-curie level would be made only after a successful run at the designed level of the plant.

Assuming 30,000-curie shipments are possible, which is very probably right, these 30,000-curie shipments may be made as frequently thereafter as desired until the 200,000-curie combined life of the cubicles is expended. There are two cubicles replaceable at a cost of \$30,000 each, or can be overhauled for \$12,000 each.

Carrier Capacity

Hanford-irradiated slugs received by ORNL for barium production in the past were specially selected from the center of the pile and, at the time of discharge, contained an average of 139 curies per four-inch (1,800-gram) slug. During the transportation and processing time of ten days, decay reduced the available barium content to an average of eighty curies per slug. Approximately thirteen days' transportation, handling and processing time (the physical size of the metal solution equipment will require thirteen separate dissolvings and precipitations for thirty-kilocurie production), and the additional three days' decay will reduce the available barium content to sixty-eight curies per slug.

The chemical yield of the plant as modified is difficult to predict, but it is estimated that 85% to 95% yield will be obtained. The number of four-inch Hanford slugs required at 85% chemical yield and containing 140 curies of Ba<sup>140</sup> at time of discharge is:

<u>Chemical Yield</u>	<u>Available Curies/Slug After Thirteen Days' Decay</u>	<u>Number of Slugs Required for 30,000 Curies</u>
85%	57.8	520

Carriers available at present for the shipment of slugs from Hanford to ORNL are four 39-slug carriers, a combined carrier capacity of 156 slugs.

Three new 148-slug carriers are in the design stage and will be fabricated if required, at an estimated total cost of \$35,000. The total slug-carrier shipment capacity will then be:

Four 39-slug Carriers	156
Three 148-slug Carriers	<u>444</u>
Total....	600 Slugs

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USAEC

Dr. John H. Roberson

March 7, 1951

Ion Exchange (Purification) Cubicle Capacity

The ion-exchange purification cubicles, standby duplicates, are designed for a maximum of 10,000 curies each. Operated in parallel, a total of 20,000 curies can be produced. It is probable that the cubicles can be operated at 50% over the design capacity at the sacrifice of chemical yield or purity to make the total batch size 30,000 curies. The chemical yield is influenced by the radiation exposure, but the extent can be determined only from operating experience.

It is probable that no changes in the cubicle purification equipment will be necessary to produce 30,000-curie runs. It is estimated that the cubicles can survive a combined total of seven 30,000-curie runs. Any modifications aimed at extending the cubicle life beyond this figure will probably be quite costly. Additional panel board instrumentation necessary to permit the two cubicles to operate simultaneously will cost an estimated \$2,500.

Costs

The additional costs attributable to the increased production capacity of 30,000 curies per run are estimated to be:

Capital Costs

Three 148-slug Carriers	\$35,000
Additional Instrumentation	<u>2,500</u>
Total ...	\$37,500

Operating Costs

The estimated additional costs per run to produce 30,000 curies over the costs of producing 10,000 curies are itemized below:

Hanford Loading Costs	\$ 400
Transportation Costs	3,000
K-25 Loading and Hauling Costs	300
Operating Costs	1,000
Canal Handling Costs	200
Cubicle Overhaul Amortization	<u>2,400</u>
Total/Run...	\$ 7,300

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~~SECRET~~

~~SECRET~~

USAEC  
Dr. John H. Roberson

- 4 -

March 7, 1951

Source and Fissionable Materials Costs

A 30,000-curie shipment compared to a 10,000-curie shipment will necessitate the additional use of approximately 100 grams of plutonium and approximately 600 kilograms of uranium. Based on a slug cost of \$25.00 each, the additional slug costs are almost \$9,000 per shipment.

MTR - RaLa

The MTR-RaLa process is in the laboratory stage of development and experience gained in ten-to-thirty-kilocurie runs is expected to add very valuable information in making decisions regarding the choice of process to be used in the MTR facility.

Very truly yours,

OAK RIDGE NATIONAL LABORATORY

*C. E. Larson*

C. E. Larson  
Director

MERamsey-FLSteahly:wp:ab

Distribution:

- Copies 1-3. J. H. Roberson
- 4. C. E. Center
- 5. A. M. Weinberg
- 6. J. A. Swartout
- 7. F. L. Steahly
- 8. M. E. Ramsey
- 9. E. J. Witkowski
- 10. F. C. VonderLage
- 11-12. C. E. Larson

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X-426

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CARBIDE AND CARBON CHEMICALS DIVISION  
UNION CARBIDE AND CARBON CORPORATION  
DUC  
POST OFFICE BOX P  
OAK RIDGE, TENNESSEE

ANT

DATE: March 19, 1951

SUBJECT: Letter re: Future Ra La Slug Requirements.

TO: H. R. Freitas

FROM: H. F. Stringfield

ORNL  
RECORDS SECTION  
51-3-53

COPY NO. 3A

*H. F. Stringfield*

Publicly Releasable

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ORNL  
CENTRAL FILES NUMBER

51.8-55

CLASSIFICATION CANCELLED

Arvin S. Just 1/18/95  
ADD signature Date

POST OFFICE BOX P  
OAK RIDGE, TENN.

March 19, 1951

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United States Atomic Energy Commission  
Hanford Operations Office  
Post Office Box 550  
Richland, Washington

Classification Cancelled  
Or Changed To \_\_\_\_\_  
By Authority Of \_\_\_\_\_  
By \_\_\_\_\_ Date AUG 31 1971

Attention: H. R. Freitag

Gentlemen:

Reference is made to your teletype message Number 2298 dated March 8, 1951 wherein you request information on future Ra La slug requirements. As you perhaps know such requirements depend entirely upon Los Alamos' demand for extracted Ra La. Apparently, they are not in a position to forecast their requirements which in turn places us in a similar position in connection with scheduling slug deliveries.

Normally, Los Alamos directs its request to Mr. E. J. Witkowski, by telephone. We then request delivery of slugs for our process.

This arrangement has functioned very well in the past from our standpoint and we would like to request a continuation of this procedure.

In this connection we have requested Mr. W. J. Larkin, Oak Ridge, Atomic Energy Commission office, of Research and Medicine to officially confirm these arrangements by letter.

As the need for Ra La slugs arises we will give as much advanced notice as possible.

Very truly yours,

H. F. Stringfield  
H. F. Stringfield  
Accountability Representative

HFS:bb

cc: E. J. Witkowski  
H. F. Stringfield

This document contains information relating to the contents of this process. It is not to be distributed outside the applicable Federal laws.

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OAK RIDGE NATIONAL LABORATORY  
OPERATED BY  
CARBIDE AND CARBON CHEMICALS DIVISION  
UNION CARBIDE AND CARBON CORPORATION

MAIL  
POST OFFICE BOX P  
OAK RIDGE, TENNESSEE

19  
ANT

DATE: March 19, 1951

SUBJECT: Letter re: Future Ra La Slug Requirements.

TO: H. E. Freitag

FROM: E. F. Stringfield

ORNL  
GENERAL FILES GROUP  
51-3-53

COPY NO. 3A

*E. F. Stringfield*

This document has been approved for release  
to the public by:

*David R. Hemm* 1/30/95  
Technical Information Office: \_\_\_\_\_ Date  
ORNL Site

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POST OFFICE BOX P  
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March 19, 1951

United States Atomic Energy Commission  
Hanford Operations Office  
Post Office Box 550  
Richland, Washington

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By Authority Of \_\_\_\_\_  
Date AUG 31 1971

Attention: H. R. Freitag

Gentlemen:

Reference is made to your teletype message Number 2298 dated March 8, 1951 wherein you request information on future Ra La slug requirements. As you perhaps know such requirements depend entirely upon Los Alamos' demand for extracted Ra La. Apparently, they are not in a position to forecast their requirements which in turn places us in a similar position in connection with scheduling slug deliveries.

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In this connection we have requested Mr. W. J. Larkin, Oak Ridge, Atomic Energy Commission office, of Research and Medicine to officially confirm these arrangements by letter.

As the need for Ra La slugs arises we will give as much advanced notice as possible.

Very truly yours,  
*H. F. Stringfield*  
H. F. Stringfield  
Accountability Representative

HFS:bb

cc: E. J. Witkowski  
H. F. Stringfield

Classification changed to  
**UNCLASSIFIED**  
(Insert appropriate classification level and category)  
by authority of T. F. DAVIS 10-18-93  
(Authority for change in classification) (Date)  
by C. C. Peters 3/29/94  
(Signature of Person making change) (Date)  
Verified by: C. J. Norman 3/29/94  
(Signature of Person verifying change) (Date)

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OAK RIDGE, TENNESSEE

*Dist To Ph*

DATE: April 5, 1951

INV.  
64

ORNL  
CENTRAL FILES NUMBER  
51-4-24

SUBJECT: RALA PRODUCTION SCHEDULE FOR FISCAL YEAR 1952

COPY NO. 6A

TO: Dr. John H. Roberson

FROM: Dr. C. E. Larson

Classification Cancelled

~~Changed To~~

By Authority Of

By *H J S*

Date AUG 31 1971

Distribution:

- 1-3 J. H. Roberson
- 4 C. E. Center
- 5 E. J. Witkowski
- 6 F. L. Steahly
- 7 M. E. Ramsey
- 8-9 C. E. Larson



INV.  
62

INV.  
65

CLASSIFICATION CANCELLED  
*Allen S. Just* 1/18/95  
ADD signature Date

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No. 6 of 7 copies, Series A

**UCC**  
POST OFFICE BOX 1  
OAK RIDGE, TENN.

U. S. Atomic Energy Commission  
Oak Ridge Operations  
Oak Ridge, Tennessee

Attention: Dr. John H. Roberson

Subject: RALA PRODUCTION SCHEDULE FOR FISCAL YEAR 1952

Gentlemen:

A firm schedule for Rala product is needed since all Rala schedules that have been submitted in the past have always had very drastic revisions resulting in unnecessary costs and work schedule interruptions. The lack of definite schedules has also prevented the use of Rala slug carriers for transportation of W-slugs to Oak Ridge National Laboratory for the purpose of processing and other work. Efficient use of the men on the Rala Program has been hindered since the expectation of a Rala run in the immediate future has usually been present. The situation will be more acute in the coming year since Rala is not to be included in the fiscal year 1952 budget and the production schedule has a bearing on the problem of reassignment of the Rala personnel at the conclusion of production at ORNL.

In order to efficiently handle these problems it is considered necessary that a schedule be submitted to Oak Ridge National Laboratory by May 1, 1951, showing the desired curie content (based on curie size as shown in Report AECU-567, Absolute Beta Counting Using End Window Gieger-Mueller Counters, by L. R. Zumwalt, which indicates the curie size by absolute beta counting to be 1.45 times the value used in assigning curies for all Rala runs to date, i.e., one of the old curies equal 1.45 of the new curie) and shipping dates for all Rala runs to be made in Fiscal Year 1952.

Prompt consideration of this request for a Rala schedule will be appreciated.

MERamsey:wp

- 3. J. H. Roberson
- 4. C. E. Center
- 5. E. J. Witkowski
- 6. F. L. Steahly
- 7. M. E. Ramsey
- 8-9. C. E. Larson

Very truly yours,

OAK RIDGE NATIONAL LABORATORY

*C. E. Larson*  
C. E. Larson  
Director

~~SECRET~~

~~SECRET~~

51-4-211

FLS-627

To: FLSteahly

Date: April 6, 1951

From: REBlanco

Subject: Summary: ~~Cancelled~~ Run #1, April 5, 1951

This document consists of 2 pages and \_\_\_\_\_ forms. No. 1 of 5 copies, Series FLS

All analyses ~~or calculated to I-S-T.~~ or changed to By Authority Of By 2/2/51 SEP 22 1971 Precipitation Steps

INV. 64

	Ba (Curies)	% Total	Sr (Curies)	% Total
Dissolver Solution	248	100	118	100
Undissolved Ba	93	37.5	-	-
Metal Waste	2	0.8	-	-
Metathesis Waste	1	0.4	-	-
Metathesis Tank Rinse (A9)	3	1.2	-	-
Column Feed	224	90.3	70	59.3

Summary of Precipitation Steps

Total Loss	6	2.4		
Total Yield	224	90.3	70	59.3
Total Unaccounted for	18	7.25		

Ion Exchange  
(Percentages based on column feed)

Column Feed	224	100	70	100
Column Effluent Waste	2	0.89	25	35.7
Sr Waste (Versene)	0.014	0.06	5	7.1
Na Waste (HCl)	0.1	0.045	-	-
Product #1 (6 M HNO <sub>3</sub> )	204	91.07	0.146	0.21

Summary of Ion Exchange

Total Loss	2.114	0.94		
Total Yield	204	91.07	0.146	0.21
Total Unaccounted for	18	8.3		

Overall Summary Through Precipitation Steps and Ion Exchange

Total Loss	8.0	3.2	-	-
Total Yield	204	82.3	0.14	0.12
Total Unaccounted for	36	14.5	-	-

Total Solids	62 gm
Lead	124 mg
Fe	Not Reported
Ni	" "
Na	" "
Ce	0.0063 curies

This document has been approved for release to the public by:

David R. Hamlin 1/30/95  
Technical Information Officer Date  
ORNL Site

Nitric Acid Precipitation (80% HNO<sub>3</sub>)

Not enough carrier barium present to allow quantitative precipitation of Ba(NO<sub>3</sub>)<sub>2</sub>.  
(Percentages on overall basis)

Product After Precipitation	58	23.4	0.05	0.042
Total Solids	2.3 gm			
Pb	4.2 mg			
Ce	0.000 93 curies			

CLASSIFICATION CANCELLED  
Jed Davis 1/30/95  
ADD signature Date

DRAFT COPY

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FLSteahly

April 6, 1951

	<u>Ba</u> <u>(Curies)</u>	<u>%</u> <u>Total</u>	<u>Sr</u> <u>(Curies)</u>	<u>%</u> <u>Total</u>
--	------------------------------	--------------------------	------------------------------	--------------------------

Carrier barium (0.5 gm) was added to the waste nitric acid and the barium recovered by precipitation from 80% nitric acid.

<u>Recovered Product</u> (contaminated with Pb and Sr from waste tank) Pb                    309 mg	106	42.7	0.4	0.34
--	-----	------	-----	------

<u>Total Product Shipped</u> Pb                    313 mg	<del>164</del> <sup>152</sup>	<del>66.1</del> <sup>61.2</sup>	0.45	0.38
--	----------------------------------	------------------------------------	------	------

<u>Waste Tank Heel</u>	18.2	7.33	-	-
------------------------	------	------	---	---

Barium remaining in product tank after transfer of product to shipping cone.	12	4.83	-	-
--	----	------	---	---

<u>Overall Summary</u> Total barium accounted for	202.2 curies = 81.5%			
--	----------------------	--	--	--

REB:sjp

- Distribution:
1. FLSteahly ✓
  2. FRBruce
  3. IRHiggins
  4. WEUnger
  5. REBlanco

~~SECRET~~

C-5

ORNL  
CENTRAL FILES NUMBER

51-4-129

64  
LIV

B-137

"This document consists of 3 pages.  
No. 1 of 5 copies, Series 1A"

Date April 30, 1951

File \_\_\_\_\_

Subject Revision of Operating Procedure  
for 706-D Ra La Process

Those Eligible  
To Read The  
Attached

By R. E. Blanco

Copy # 1A

To E. J. Witkowski  
W. E. Unger

Before reading this document, sign and date below:

\_\_\_\_\_  
\_\_\_\_\_  
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~~By Authority Of~~  
~~By~~ J. J. [Signature]  
~~Date~~ SEP 1 1977

100  
NF

Distribution:

- 1. EJWitkowski ✓
- 2. WEUnger
- 3. IRHiggins
- 4. FRBruce
- 5. REBlanco

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[Signature] 1/18/95  
Date  
ADD signature

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~~SECRET~~

~~SECRET~~

To: E. J. Witkowski  
W. E. Unger

May 10, 1951

From: R. E. Blanco

Subject: Revision of Operating Procedure for 706-D RaLa Process

Make the following flowsheet changes:

A. Metathesis

Make three metatheses instead of two.

B. Ion Exchange Resin

Use C.P. Dowex 50 resin for all hot runs. (60-100 mesh)

C. Ion Exchange Flowrates

The maximum flow rate for all ion exchange solutions is 60 ml/min (see exceptions in parts D and F). The flows should be maintained as close to 60 ml/min as is practical.

D. Water Washes

- 1) Use 1 liter of water wash after the feed instead of 3.
- 2) Use 4 liters of water wash after the elution with 0.07M Versene pH 6.3 instead of 5. Release air from top of column after 3 liters of water wash.

The flow rates should be: 1 liter at 60 ml/min followed by 3 liters at 240 ml/min.

- 3) The flow rates on the water wash after the HCl elution should be 1 liter at 60 ml/min and 2 liters at 240 ml/min.

E. Sodium Elution

Use 12.0 liters of 1M HCl instead of 10.0 liters.

F. Product Elution

- 1) Use 5 liters of 6M HNO<sub>3</sub> at a maximum flow rate of 240 ml/min.
- 2) Follow with 7 liters of 6M HNO<sub>3</sub> at a maximum rate of 40 ml/min.

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*[Signature]* 1/18/95  
 ADD signature Date  
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~~SECRET~~

~~SECRET~~

To: E. J. Witkowski  
W. E. Unger

May 10, 1951

G. Product Evaporation

Use a maximum of 25 lbs of steam for the product evaporation.

H. Carrier Barium

Add 0.95 gm Ba(NO<sub>3</sub>)<sub>2</sub> (≈ 0.5 gm Ba) carrier to product tank before evaporation for all runs containing less than 10,000 curies.

REB:mp

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UNION CARBIDE AND CARBON CORPORATION

UCC  
POST OFFICE BOX P  
OAK RIDGE, TENNESSEE

DATE: 4/27/51

SUBJECT: Teletype re: RaLa Shipment

APR 27 1951

51-4-140

TO: E. J. Witkowski

FROM: R. P. Hammond, Los Alamos

COPY NO. 20

Central Files

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THIS DOCUMENT CONSISTS OF 1 PAGES  
NO. 2 OF 2 SERIES C.

ORNL  
CENTRAL FILES NUMBER

51-4-140

FM RP HAMMOND SCILAB LOS ALAMOS NMEX  
TO USAEC OAK RIDGE TENN  
NR S 150 272247Z GR52

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By Authority Of J. J. [unclear]

Date SEP 1 1971

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/CONTAINS AEC RESTRICTED DATA/

CONFIRMING ORAL AGREEMENT WITH E J WITKOWSKI  
OF ORNL THE NEXT RALA SHIPMENT IS REQUESTED FOR ARRIVAL AT LOS ALAMOS  
ON MAY TWENTY FIRST PD THE QUANTITY REQUESTED IS APPROXIMATELY FIVE  
THOUSAND CURIES PD TH IS REFERENCE NUMBER CMR TEN DASH ONE SIX ONE END  
FROM R P HAMMOND

END

CLASSIFICATION CANCELLED  
Carol S. Trust 11/8/95  
ADD signature Date  
Single rereview of CCRP-declassified documents was authorized by DOE Office of Declassification memo of August 22, 1994.

If provisions of GM-74 Part 10-10-1 are followed, none are required. Handle as ~~SECRET~~ correspondence.

27/2305Z

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Rec'd by [initials] at D305  
Decoded by [initials] at 28/1345

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51-5-272  
File-356

~~SECRET~~  
to the public by:

*David R. Brown*  
Technical Information Officer  
ORNL Site

2/3/95  
Date

FLS-680  
May 14, 1951

15  
NW

To: W. E. Unger  
From: R. O. Payne and E. W. Hinterleiter  
Subject: ORNL RaLa Process - Product Evaporator

INV.  
64

This document consists of 2 pages and 0 figures.  
No. 4 of 7 copies. Series FLS

Modification of equipment is suggested in order to improve operation of the final step evaporator in the Building 706-D RaLa Process. The existing procedure reduces a product stream of approximately 14 liters volume down to two liters in a steam jacketed tantalum evaporator. A one-quarter inch (outside diameter) tantalum dip tube for determining the liquid level extends to within 1/16 inch of the sloping bottom of the tank. As the salting point is reached, a precipitate (mostly  $\text{NaNO}_3$ ) forms and apparently plugs the dip tube, rendering further level measurement impossible.

In order to verify this action, a one-fifth scale synthetic solution was evaporated in a four liter glass beaker with a tantalum tube extending to within three-eighths inch of the flat bottom. Minimum air flow to the tube was provided ranging from an initial five to a final ten bubbles per minute and pressure drop from an initial 15 inches  $\text{H}_2\text{O}$  down to a final 1.5 inch. At the 400 ml mark, corresponding to the final process level, the probe was still functioning with about one-half of the final salt already crystallized. At this point, there was a 1/32 inch salt layer one-half inch long on the inside of the tube. Continued evaporation of the salt to dryness resulted in a large volume of cake (about 350 ml) which would correspond to nearly two liters in the full-scale process and sufficient to cover the bottom of the tube. Since it is the nature of the salt to creep up the walls (on glass and tantalum) and break off in large pieces, it is assumed that this action causes the dip tube to plug in the RaLa Process rather than crystal formation inside the air swept tube (the process air bubbling rate is considerably greater than that used in this test). The addition of 1.6 parts of  $\text{H}_2\text{O}$  per part of insoluble nitrate present permitted immediate functioning of the dip tube (at 23°C).

CLASSIFICATION CANCELLED

*J's Morgan* 1-31-95  
Date

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Classification Cancelled

Or Changed To

By Authority Of *Doc*

By *EPB*

Date SEP 1 1971

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Heating the solution to 80°C caused dissolution of all the crystals.

The Laboratory Section reports that reducing the residue to dryness introduces a potential explosion hazard from organic material extracted from the resin. Therefore, it is recommended that the end of the dip tube be raised to correspond with the final evaporator level. This height allows over one inch clearance of the tube with the tank bottom. When the pressure drop in the dip tube reaches zero during evaporation, the final level would be attained. A second dip tube at the present height could be used if desired. Relocation of the tubes to nearer the tank center also should assist in preventing plugging. Control of air rate to five - ten bubbles a minute and use of at least one-quarter inch inside diameter tubing would reduce the pressure drop in the system and improve accuracy.

EWH/rep

**Distribution:**

1. WEUnger
2. EWHinterleiter
3. FLSteehly
4. JCDavis
5. FEBruce
6. REBlanco
7. TAArchart

Intra-Laboratory Correspondence  
OAK RIDGE NATIONAL LABORATORY

- 1. FLSteahly ✓
- 2. FRBruce
- 3. IRHiggins
- 4. WEUnger
- 5. REBlanco

To: F. L. Steahly

Date: May 24, 1951

From: R. E. Blanco

INV.  
64

Subject: RaLa Run #44, May 14, 1951, Summary

This document consists of 2 pages  
No. 1 of 5 copies, Series FLS

All analyses calculated to the same time.

Precipitation Steps

	Gross (Curies)	Ba (Curies)	% Total	Sr (Curies)	Percent Total
Dissolver Solution	-	14,509	100	6,623	100
Dissolver Heel	-	329	0	-	-
Metal Waste	-	58	0.4	-	-
Metathesis Waste	-	378	2.6	-	-
Metathesis Tank Rinse (Ag)	-	337	2.3	-	-
Column Feed	17,434	13,888	95.7	2,245	33.9

Total Ba Yield 95.7%  
Total Ba Loss 4.9%

Ion Exchange Steps

Column Feed	17,434	13,888	95.7	2,245	33.9
Feed Tank Rinse	50	33	0.23	6	0.27
Effluent Waste	3,962	28	0.20	1,398	21.10
Sr Waste (Versene)	776	66	0.45	238	3.59
Na Waste (HCl)					

The eluate radiation monitor did not show any significant loss in the sodium waste. A valve broke during the product elution allowing the product to pass into the Sr and Na waste tanks. All wastes including dissolver heel were combined and returned to Tank A-9 for recovery by a sulfate precipitation and metathesis.

Classification Cancelled

~~By Changed To~~

Total column loss to this point was 0.88%

By Authority Of DOC

Total overall loss to this point was 5.78%

By REB

Date SEP 1 1971

This document has been approved for release to the public by:

1st Recovery Run  
Precipitation Steps

Total Ba curies in the system =	14,509+329=	14,838			
Metal Waste (SO <sub>4</sub> )	-	2,331	15.7	-	
Metathesis Waste	-	441	2.97	-	
Column Feed	7,211	4,828	32.5	478	
Metathesis Tank Rinse (Ag)		6,722	45.24	-	

Total Ba accounted for 14,322 96.4

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David R. Hamlin 2/2/71

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FLSteahly

May 24, 1951

<u>Gross</u> <u>(Curies)</u>	<u>Ba</u> <u>(Curies)</u>	<u>%</u> <u>Total</u>	<u>Dr</u> <u>(Curies)</u>	<u>Present</u> <u>Total</u>
---------------------------------	------------------------------	--------------------------	------------------------------	--------------------------------

These figures indicate that not more than 3.6% of the Ba remained on the resin column. The high figure for the A<sub>9</sub> rinse showed that metathesis was poor. As a result, the BaSO<sub>4</sub> was retained on the filter when the solution was transferred to the ion exchange feed tank.

Ion Exchange Steps

The ion exchange feed was initially pH 7.4 instead of the usual 11 and was yellow. When the pH was adjusted to 10 to ensure the complete dissolution of BaSO<sub>4</sub>, a yellow ppt. formed (U ?). The solution was adjusted to pH 6.3 and passed into the column.

(% based on column feed)

Column Feed	7,211	4,828	100	478	100
Feed Tank Rinse		432	8.9	15	-
Column Effluent	2,874	171	3.5	281	-
Sr Waste (Versene)	443	0.77	0.01	141	-
Na Waste (HCl)		89	1.84	4.3	0.89
Product		3,040	62.9	3.3	
Total Ba unaccounted for			22.8		

(Analytical accuracy poor due to the presence of precipitate in the feed).

Recovery #3

The wastes in A-9 were again precipitated with H<sub>2</sub>SO<sub>4</sub> and the precipitate metathesized.

	<u>Precipitation Steps</u>	
Metal Waste	-	283
Metathesis	-	116
A-9 Rinse	-	1,530
Column Feed	-	1,475
Total Barium		3,404

Total Ba unaccounted for (6,722 - 3,404) = 3,318

Ion Exchange Step

About twice the normal amount of acid was required to adjust the feed pH, indicating the presence of excess buffer. Was too much Versene Added?

(% based on Column Feed)

Column Feed	-	1,475	100	42	100
Feed Tank Rinse	-	23	1.56		
Effluent Waste	-	1,250	84.7	44	104
Sr Waste	50	45	3.05	0.89	2.1
Na Waste (HCl)	12	11.8	0.8	0.11	0.26
Combined product					
Recovery Runs #1 and 2	3,969	3,620	-	3.7	-
Fuming HNO <sub>3</sub> Waste	625	25	-	-	-
Product Tank Rinse	20	22	-	-	-

Product Analysis Pb - not detected

Final amount shipped - corrected to shipping time 3,204 curies

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OPERATED BY  
CARBIDE AND CARBON CHEMICALS DIVISION  
UNION CARBIDE AND CARBON CORPORATION  
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DATE: June 13, 1951.

This document consists of  
14 pages.  
No. 1 of 19  
Series A

SUBJECT: 706-D Modification Project  
First Full Level Production Run

COPY NO. 17

TO: F.L. Steahly

FROM: R.H. Vaughan

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TO: F.L. Steahly  
 FROM: R.H. Vaughan  
 SUBJECT: 706-D Modification Project  
 First Full Level Production Run

1.0 Summary.

73 W slugs were dissolved and processed thru the new equipment, crud filters, process filters, and ion-exchange purification cubicles. The mechanical failure of a distribution valve in the ion-exchange cubicle spilled the expected product yield, 12,000 curies, into the waste. The waste was subsequently reprocessed in stand-by equipment where 3000 curies was recovered as product and shipped 21 May 1951.

1.1 The slugs were charged to the dissolver on 13 May 1951, dissolved, processed, and the product shipped on 21 May 1951.

The slugs were dissolved in two batches and a heels dissolving, yielding the following amount of product.

CODE	C's Ba	C's Ba REFERRED TO LST	SLUGS APPROX.	C's/SLUG
1st METAL DISSOLVING	9066	8023	38.1	287
2nd METAL DISSOLVING	5443	4817	29.5	216.6
HEELS DISSOLVING	329	291	1.9	170
TOTALS	14,838	13,131	69.5	214 av.

All new equipment installed in conjunction with "A" cell operations functioned very satisfactorily. The crud filtration was fast - 2.8 gallons per minute average. The process filtration was also fast - 1.5 gallons per minute average. The losses thru these steps were very small, approximately 3%.

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Recovery run No.1 was carried thru up to the fuming nitric precipitation step. This product yield was retained in the product evaporator until recovery run No. 2 was completed up to the same point. The product yields were then combined in the product evaporator and processed to final completion as one run. The fuming nitric acid precipitation operation was carried out very satisfactorily. The transfer to the shipping cone was made easily and with only a small heel loss. The cone manipulator worked very well. The A-16 vacuum did not furnish enough air sweep thru the charging head for evaporation so the emergency jet from the charging heads to A-16 was utilized for this operation. This evaporation to dryness required 12 hours. 3157 curies of product was shipped to Los Alamos, 21 May 1951.

The following tabulations give results of all runs. The results of initial run are shown up to the point where plastic valve failed. All values given are referred to LST of 1320, May 20, 1951.

<u>PRECIPITATION</u>	<u>INITIAL RUN</u>	
FEED: DISSOLVER SOLUTION		12,840 curies
LOSSES: METAL WASTE - 1st EXTRACTION	23 curies	
METAL WASTE - 2nd EXTRACTION	28 "	
MERAPRESIS	334 "	
A-9 RINSE	296 "	
	683 curies	683 curies
YIELD: 94.6% BASED ON LOSSES		12,157 curies

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RESIN CUBICLE

FEED: METATHESIS FROM A-9 (analysed in feed tank)		12,157 curies
LOSSES: FEED EFFLUENT	25 curies	
FEED RINSE	29 "	
VERSENE WASTE	53 "	
HCL WASTE	88 "	
	<hr/>	
	195 curies	195 curies

\*YIELD: 98.3% BASED ON LOSSES

11,962 curies

\*At this point the mechanical failure of PV-23, a distribution valve, occurred and the product was lost to the waste. No confirmation of this yield could be made but it is reasonable to assume that such a yield could have been expected.

RECOVERY RUN NO.1

PRECIPITATION

FEED:	(1) RETURN FROM NO.200 RESIN CUBICLE	11,962	*1	
	(2) INITIAL METATHESIS LOSSES	334		
	(3) A-9 RINSE - INITIAL	298		
	(4) DISSOLVER HEELS	291		
		<hr/>		
		12,885 curies		12,885 curies
LOSSES:	METAL WASTE EXTRACTION	1216		
	HEELS	844		
	A-9 RINSE	5950	*2	
		<hr/>		
		8010 curies		8010 curies

YIELD: 37.9 % BASED ON LOSSES

4875 curies

- \*1 Assumed that all product stripped from the column.
- \*2 A-9 rinse showed a ppt - incomplete metathesis.

RESIN CUBICLE

FEED: METATHESIS FROM RECOVERY RUN NO.1  
(analysed in feed tank)

3869 curies

LOSSES: FEED EFFLUENT	151 curies	
FEED RINSE	382 "	
VERSENE WASTE	1 "	
HCL WASTE	77 "	
	<hr/>	
	611 curies	611 curies

YIELD: 84% BASED ON LOSSES  
69.5% BASED ON YIELD (product sample)

3258 curies  
2640 curies

RECOVERY RUN NO.2

PRECIPITATION

FEED: A-9 RINSE FROM RECOVERY RUN NO.1

5950 curies

LOSSES: METAL WASTE EXTRACTION	250 curies	
METATHESIS	116 "	
A-9 RINSE	1354 "	
	<hr/>	
	1720 curies	1720 curies

YIELD: 71% BASED ON LOSSES

4230 curies

RESIN CUBICLE

FEED: METATHESIS FROM RUN NO.2 (analysed in feed tank)		1305 curies
LOSSES: FEED EFFLUENT	1114 curies	
FEED RINSE	20 "	
VERSENE WASTE	41 "	
HCl WASTE	11 "	
	<hr/>	
	1186 curies	1186 curies
YIELD: 9.1% BASED ON LOSSES		119 curies
39.4% BASED ON YIELD *3		514 curies

\*3 Product yields from recovery run No.1 and No.2 combined in product tank.  
The value given here is obtained by difference between yield for run No.1  
and combination of run No.1 and No. 2.

FUMING NITRIC ACID PRECIPITATION

FEED: PRODUCT YIELD FROM RUN NO.1 and NO.2	3204 curies
LOSS: FUMING NITRIC WASTE	25 curies
YIELD: 98% BASED ON LOSSES	3179 curies
97% BASED ON YIELD	3157 curies

~~SECRET~~

## 2.0 Valve failure - other operational difficulties.

The valve which failed was PV-23, a 5-port 4-way distribution valve. The valve is constructed of a stainless steel hull, a fluorothene body, and a teflon plug. The plug is spring loaded from the bottom which provides the pressure to seat the tapered plug in the body. The rotation of the valve plug is obtained thru a worm and gear mechanism mounted at the top of the valve. This mechanism is actuated by a flexible cable from the valve position indicator handle mounted on the panel board. Fig.1, gives a sectional view of the construction and assembly of this valve.

The exact nature of the failure has not been determined. The first indication that the valve was not satisfactory was derived from the fact that the liquid level in the main waste rose and with no apparent reason. A check of the position indicator showed that the flow should have been directed to the product evaporator. The first thought was that the position indicator was out of adjustment, however, a quick test revealed that the flow from the column was directed to the waste tank no matter what position was indicated. From this test it was concluded that the valve plug could not be made to rotate.

Failure of the valve plug to rotate could be attributed to a number of things; namely, the gear pin had sheared, the cable had parted, or the gears had stripped. The pin which fastens the driving gear to the gear shaft may have sheared, but this appears unlikely since all these pins are of hardened tool steel.

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That the cable had parted or broken loose from its end fittings is rather remote since all the cable assemblies were tested at 35 inch-lbs of torque prior to final assembly with the valve. Also an attempt to pull the cable from within the casing were of no avail. If the cable had parted it should be easily removed from the casing since it turns freely within the casing during operation. The fact that the casing has a number of sharp bends in it between the valve and the position indicator handle on the panel board would not interfere with the removal of the cable by itself or a portion of the cable; but, one would find it difficult to remove the cable with the end fitting attached because this fitting is too long and rigid to permit it to pass around too sharp a bend in the casing. If the gears had stripped a grinding or grating sensation would be felt in turning the position indicator handle; but instead, the indicator handle continued to turn as would normally be expected.

During the period when this valve - and all the plastic valves for that matter - were being designed and constructed, speed was of great importance and no long term rigid and thorough test program could be justified. Bearing in mind that the valve had been used numerous times prior to the time of failure, it is difficult to pin-point the exact cause of failure. When the cubicle has been decontaminated, a closer inspection of the valve and its related parts will be made to determine the following: How the valve failed, why it failed, and what should be done to reduce the chance for future failure. A memo to cover the above will be issued at that time.



Other operating difficulties arose and although they did not affect the process efficiency they are undesirable and should be eliminated.

A solution back-up occurred in the process filter cubicle and valve 33 between the process blow tank and process filters got hot. This valve is located outside the filter cubicle.

The sparger lines to the feed tanks in the resin cubicles got "hot" from a solution back-up.

At various places radiation was detected along the openings between the filter plugs and plug holes in both the crud and process filter cubicle.

Valve 208, a plastic diaphragm valve which regulated the flow from the eluate tank to the column, leaked air into the eluate stream. This leakage of air is undesirable since there is a possibility of "vapor-locking" the column.

The area adjacent to the funnel valves was high in background - approximately 5R/HR.

Radiation was detected along the space between the resin cubicle top and the cubicle pit.

During the evaporation to dryness of the product the loading cubicle and carrier became inexplicably contaminated.

This briefly covers the during-run operational difficulties. Two after-run difficulties arose and require special mention.

[REDACTED]

After the product was shipped attempts to wash out the heels loss in the product evaporator and recover this in a spare cone for isotope shipments were made. In this operation the cone manipulator failed. No carrier was available in which to place the cone in order to disengage it from the manipulator head. A pair of tongs was used to clamp the cone and the head rotated to disengage. The head however could not be made to rotate. Since the head is acuated by a flexible shaft from a dial knob on the control panel, it was assumed the cable had parted. Thin strips of lead were wrapped around the head and the movable shield rotated to cover the charging heads, this reduced the background to allow enough time for repairs. Close inspection revealed that the failure occurred where the drive shaft was brazed to a brass driving gear. The gear and shaft were removed and in brazing together again an attempt was made to fill the cable socket with brazing material in order to give the maximum joint strength. The manipulator now works very well. The alignment of the manipulator with the product cone carrier was not disturbed.

During decontamination another plug valve, PV-21, failed. This failure was that the shaft had parted approximately two to three inches from the position indicator handle on the panel board. A small portion of the cable left in the end fitting was sweated out and the cable resoldered to the end fitting. The cable casing was reduced in length to correspond to the new cable length. The valve has been used numerous times since repairing and works very satisfactorily. No explanation could be offered as to why the cable parted. The condition of the valve will be given along with the memo covering PV-23, the distribution valve which failed.

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3.0 Plans are to decontaminate the No.200 resin cubicle, remove it from the pit, determine the exact nature of valve FV-23 failure, and make the necessary repairs to place the original equipment in an operable condition. Efforts will be made to reduce the radiation hazards along the cracks around the filter cubicles and the resin cubicle. All minor operational difficulties will be eliminated.

3.1 Several ideas have been advanced concerning the future plans.

Changes in the process in which the hydrochloric acid concentration has been reduced from 6N to 1N would tend toward the use of stainless steel type 316, since this particular type of stainless has shown very good corrosion resistance properties at higher concentrations of HCl than is now used. With the cooperation of the laboratory group a series of lab scale runs will be made using a type 316 stainless column to determine the feasibility of using the material thru-out the cubicle.

If such proves feasible then the use of a metal plug in the present plug valves could be justified. This offers a definite advantage over the use of a plastic plug - as now practised, in that only one substance would be subject to flow under the sealing pressure. The body would tend to resolve itself around the plug, offering good sealing characteristics, and leaving the plug free to turn with a minimum of torque.

In the case the use of stainless is not feasible the plug for the plug valve could be made from Hastelloy "C" material which is known to have excellent corrosion properties to HCl concentrations above the present flowsheet conditions. The only objection to the use of Hastelloy "C" is the time delay in procurement.

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It is possible to eliminate altogether the use of HCl in the process. The HCl is used to remove the excess sodium ion from the column which otherwise would be removed with the product. This contamination of the product is undesirable. However, a revision of the product evaporator or a purchase of a new one with revisions incorporated would make it possible to remove the sodium from the barium in the tank by fuming nitric acid precipitation step. The sodium ion concentration is such that a crystallization of sodium nitrate is experienced in the evaporation step proceeding the fuming nitric precipitation.

By raising the dip-legs they would not become plugged during this procedure and process control could still be maintained thru the liquid level indicator as is now practiced. Revision of the present product evaporator is impossible since this tank cannot be dismantled. Purchase of a new tank incorporating new dip-legs would require a long time (three to four months). On this basis this procedure is not practical.

The plug valves could be eliminated and replaced with enough standard globe and gate valves to give desired process flow requirements. This procedure would require major process changes and more flexible cables.

The use of the now existant HCl waste tank as a catch tank and subsequent jetting of each solution to its required tank is possible. This tank is made of Hastelloy "C" material and contamination of product with undesirables such as iron, chromium, and others is not too serious. However, cross-contamination of eluate solutions is possible.

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A spray wash preceeding the product removal step does not offer ultimate in safeguarding against this anticipated cross-contamination. Also this requires major equipment changes. However, such an equipment change does away with many remote valves and utilizes jets which seemingly are rather trouble-free. It would be well to mention here that attempts were made to titrate the column in No.200 cubicle to determine adequacy of the resin for this radiation level. No conclusive results were obtained and laboratory did not offer a definite statement regarding this point. This can only be answered by the successful completion of a run of the same radiation magnitude. In view of this stand, operations desires only to place the equipment in a satisfactory operable condition and this plan will be followed.

The engineering department is studying the use of metal plugs in the present valves; actually the repair of the present valves will be a replacement with new ones. The valves now in use are expected to be too hot to work on. A new radiation monitor will be ordered as well as a flow element for the induction flowmeter. In decontaminating it is highly probable that these instruments will be left inoperable.

Decontamination of the No.200 resin cubicle is well underway and is proceeding very satisfactorily.

Design and procurement of necessary material for repairs are in progress. Los Alamos tentatively sets 15 July 1951, as next desired shipment date. This date can be met.

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This document consists of 11 pages.

No. 2 of 12 copies.

Series A.

INV. 64

MEMORANDUM

TO: F.L. CULLER

FROM: W.E. UNGER

SUBJECT: DISCUSSION OF 706-D MODIFICATION PROJECT AND POSSIBLE EQUIPMENT DESIGN CHANGES

DATE: JUNE 18, 1951.

ORNL  
CENTRAL FILES NUMBER  
51-6-57

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MEMORANDUM

TO: F.L. Culler

FROM: W.E. Unger

SUBJECT: Discussion of 706-D Modification Project and  
Possible Equipment Design Changes

INTRODUCTION

This memorandum summarizes results of the first high level Rala run in Building 706-D from a design standpoint. An effort has been made to include all critical comments and to analyze equipment and general design problems.

The first run has indicated that some equipment changes should be made. Three possible schemes have been outlined. Please review these suggestions both for details contained in each scheme and for the scheme itself. We must decide on what changes are to be made in the 706-D equipment very soon since a run is now scheduled for the latter part of July, 1951.

The design section will appreciate any and all destructive and constructive criticism particularly constructive since some definite alterations must be made.

Cost estimates are only guesses and should not be used for appropriation requests.

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1. Run #44, the high level break-in run, flopped because of the mechanical failure of the distribution plug cock, valve PV-23. The cubicle is to be returned to operating condition by one of three alternatives (corresponding to attached sketches A, B, and C).

(A) Replace or repair valve PV-23 and any other equipment damaged during operation or decontamination procedures.

(B) Replace all plug cock valves with manifolds and remotely-controlled commercial Mason-Neilan hydraulic valves. Replace the column deaerator assembly with one of stainless, or glass and stainless, with a large resin passage. Install valve in resin flush line to W-12.

(C) Replace column assembly as in B, install resin flush valve as in B. Employ HCl tank as Column Catch tank - install jets from HCl tank for distribution of column effluent accumulated in HCl waste tank.

## 2. Decontamination Damage

Decontamination of the #200 Cubicle (preparatory to the inspection of the defective valve) has not been readily accomplished. Steaming was never employed, probably for fear of forcing contaminated gases from the cubicle pit. The cubicle has been flushed, inside and out, with water, versene, and nitric acid. The cubicle was withdrawn from the pit and submerged in nitric acid. The shroud cleaned up well, and has been returned to D-Building. The frame assembly now located in the burial ground, reads about 10-20 R. The tantalum evaporator filter and the waste tank are apparent hot-spots.

All valve drive assemblies contained acid-vulnerable parts. The acid inundation has probably ruined the valve drives (consisting of worm and pinion on the plug valves, and planetary transmissions on the gate and diaphragm valves), the flexible shaft assemblies, the ion-chamber monitor and the induction flowmeter assembly.

## 3. Desirable Changes

The Operations Division has itemized certain equipment inadequacies, some of which can be remedied.

Operations considers the lack of off-gas vacuum gauges and specific gravity manometers a serious inconvenience.

The manometer lines are all small (1/4 inch tubing), the combined effect of the air purge pressure drop and the surface tension pressure reflected back to the manometer is annoying.

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The torsional deflection in the flexible shaft valve controls and the back lash in the drive gearing require that the lost motion in positioning the plug valves be always taken up in a clockwise direction to insure registration between the plug and body ports of the valves. This feature (relatively common in all gearing not specially spring-loaded to absorb back lash), combined with the lack of confidence inspired by the plug valve failure in run #44, would make any plan to obviate the use of plug valves sure of acceptance.

The process flow lines have been plugged with foreign matter during cold-runs. Operations feels that a plug during a hot run is a serious probability. The need for pressurized feed is persistently ascribed to the pressure drop on the process lines (1/4 inch tubing) may be replaced with 3/8 inch tubing if the plug valves are eliminated, and the 1/8 inch I.D. flowrator canula may be enlarged to 1/4 inch I.D. at the sacrifice only of sensitivity.

The column assembly design has three disturbing features:

- (a) The resin passage from the deaerator thru the plastic valve and into the column is uncertain; and unless tedious techniques are employed there is danger of flowing the resin slurry into the deaerator and thence to the W-12 waste line. A valve must be installed in the W-12 line to remedy this.
- (b) Gas accumulated in the column displaces the liquid level which recedes below the resin level. If the passage between the column and the deaerator is left open (feasible if the W-12 line is valved) the gas would accumulate in the deaerator which has sufficient volume (4.9 liters) to accommodate all the gases introduced normally into the column system.
- (c) There is at present no way of determining the liquid level nor the resin level in the column. There seems to be no practical means of accomplishing this.

The volume of the feed tank (21 liters) and the HCl waste tank (18.4 liters) are too small for the volumes of process solutions they are required to handle.

The feed tank and tantalum evaporator manometers do not read to the bottom of the tanks. The tantalum evaporator can be altered only by the fabricator.

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Repair Scheme A

Return cubicle to original condition. No design changes.

1. Install Moore differential transmitter between Feed tank and cell air (connected to pipe fitting tapped into cubicle floor plate) for off-gas vacuum indication.
2. Determine nature of PV-23 failure and correct. Dismount and inspect stems of all valves. Replace flexible shafts through-out.
3. Replace Monitor.
4. Replace Flowrator.

COST

Flowmeter replacement	\$ 500.00
PV Valve inspection	500.00
PV-23 replacement	900.00
Flexible shaft replacement (8)	200.00
Ion Chamber replacement	500.00
Moore Transmitter	400.00
Shielding additions	500.00
Miscellaneous	600.00
	-----
Total	\$ 4100.00
20% Contingency	800.00
	-----
	\$ 4900.00

Repair Scheme B

Increases volume of feed and HCl tank, replaces plug valves with remotely controlled hydraulic valves, defer column air-displacement by employing deaerator as a gas accumulator.

1. Remove corner angle and remove and replace feed tank with one of 30 liter capacity, 3/8 inch manometer lines ( and additional specific gravity line). Drill floor plate to admit new manometer lines. Install two new Moore differential transmitters, one between short leg and specific gravity lines, one between short leg and cell air.
2. Remove column assembly and replace with integral stainless column deaerator.

Repair Scheme B, cont'd

3. Remove all plastic valves and replace with seven hydraulic-controlled valves and one flexible shaft-controlled gate valve.
4. Replace all affected process piping with 3/8 inch tubing.
5. Replace flexible shafts (3).
6. Inspect and repair valve transmissions (3).
7. Replace ion chamber monitor.
8. Replace flowrator.

COST

Feed Tank	\$ 700.00
Moore Transmitters	800.00
Column Assembly	600.00
Hydraulic Valves (7)	1400.00
Piping costs	1700.00
Flexible shafts	75.00
Valve transmissions	300.00
Ion Chamber replacement	500.00
Flowrator replacement	500.00
Shielding additions	500.00
Miscellaneous	600.00
	<hr/>
Total	\$ 7675.00
20% Contingency	1600.00
	<hr/>
	\$ 9275.00

Repair Scheme C

This utilizes the column arrangement of B, but uses a catch pot and jet system for distribution of the column effluent.

1. Same as B. Install new feed tank, specific gravity indicator, and off-gas vacuum indicator.
2. Same as B. Install integral column assembly.
3. Remove all plastic valves and install one flexible shaft-controlled gate valve and globe valve (resin flush and feed throttle, respectively).

Repair Scheme C, cont'd

4. Same as B. Replace process piping with 3/8 inch tubing.
5. Same as B. Replace flexible shafts (4).
6. Same as B. Valve transmissions.
7. Same as B. Replace Ion Chamber.
8. Same as B. Replace Flowrator.
9. Replace HCl waste tank with larger tank having necessary Hastelloy "C" jet dip lines.
10. Install two new jets on HCl waste tank.
11. Install tantalum jet discharge nozzle in tantalum off-gas neck.

COST

Feed Tank	\$ 700.00
Moore Transmitters	800.00
Column assembly	600.00
Piping costs	1700.00
Flexible shafts	100.00
Valve transmissions	450.00
Ion Chamber	500.00
Flowrator	500.00
Shielding additions	500.00
HCl Waste Tank	900.00
Jets (2)	200.00
Miscellaneous	1200.00
	<hr/>
Total	\$ 8150.00
20% Contingency	1600.00
	<hr/>
	\$ 9750.00

Recommendation

Laboratory tests (R.E.Blanco) are underway to determine the feasibility of using stainless steel for the column assembly. Preliminary results indicate that less than 300 mg of iron, 40 mg of chromium, and 10 mg of nickel will be contributed to the product stream. These quantities can be accommodated by the fuming nitric precipitation. It is recommended that the glass column be replaced with an integral stainless column deaerator assembly.

The ion exchange process has not been conclusively demonstrated at high radiation levels. It may be desirable to keep the investment in equipment to a minimum until the ion-exchange process has been successfully used in a series of high level runs. Scheme A represents the minimum cost.

Scheme B, presents the most easily operable equipment, but the relatively vulnerable valves are regarded with less confidence than simpler but less easily operable jet distribution system.

Scheme C, affords the most reliable equipment by reducing the number of movable parts. It will permit some cross-contamination of successive elutriants but this will probably not be serious.

*W.E. Unger*  
W.E. Unger

WEU:hh

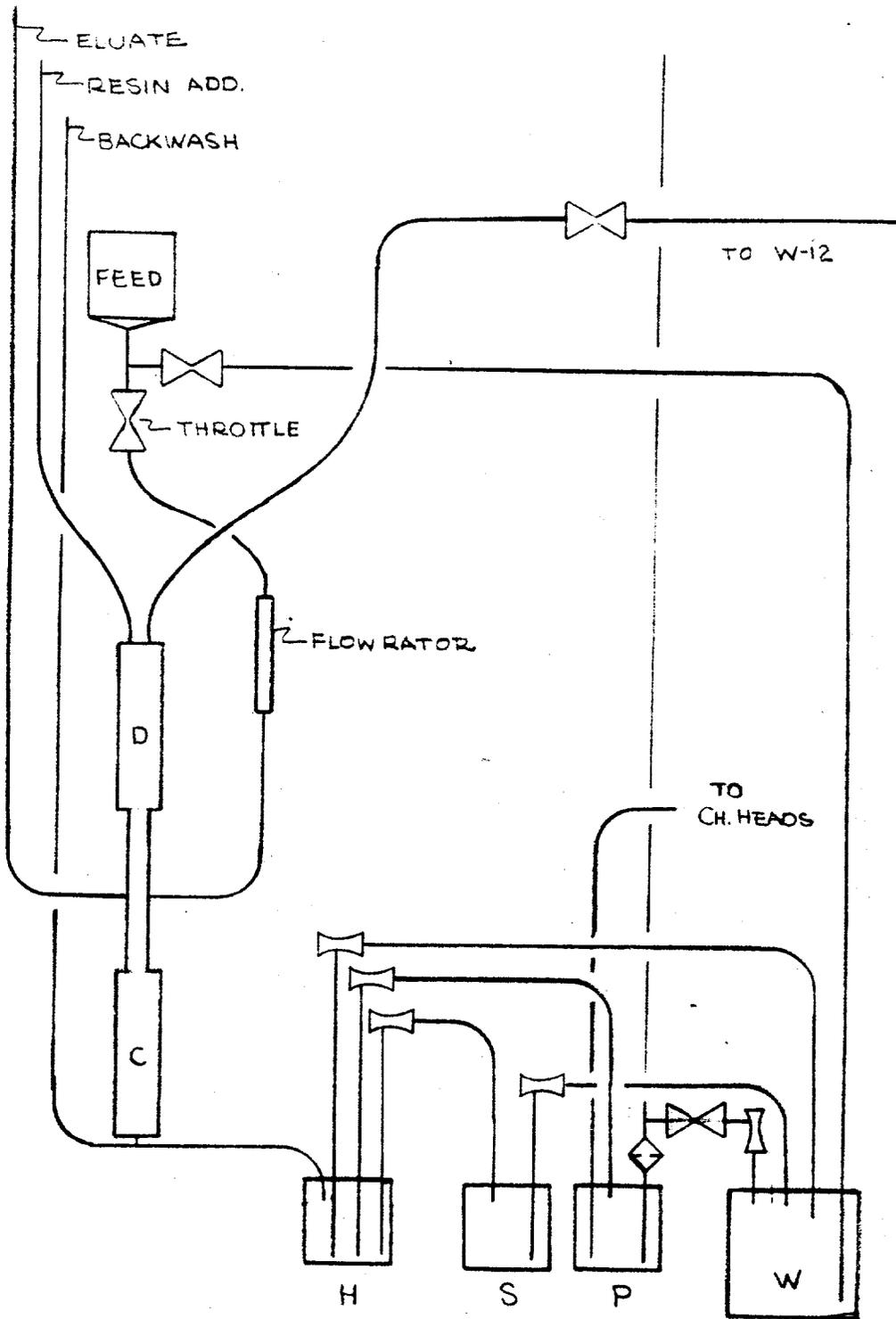
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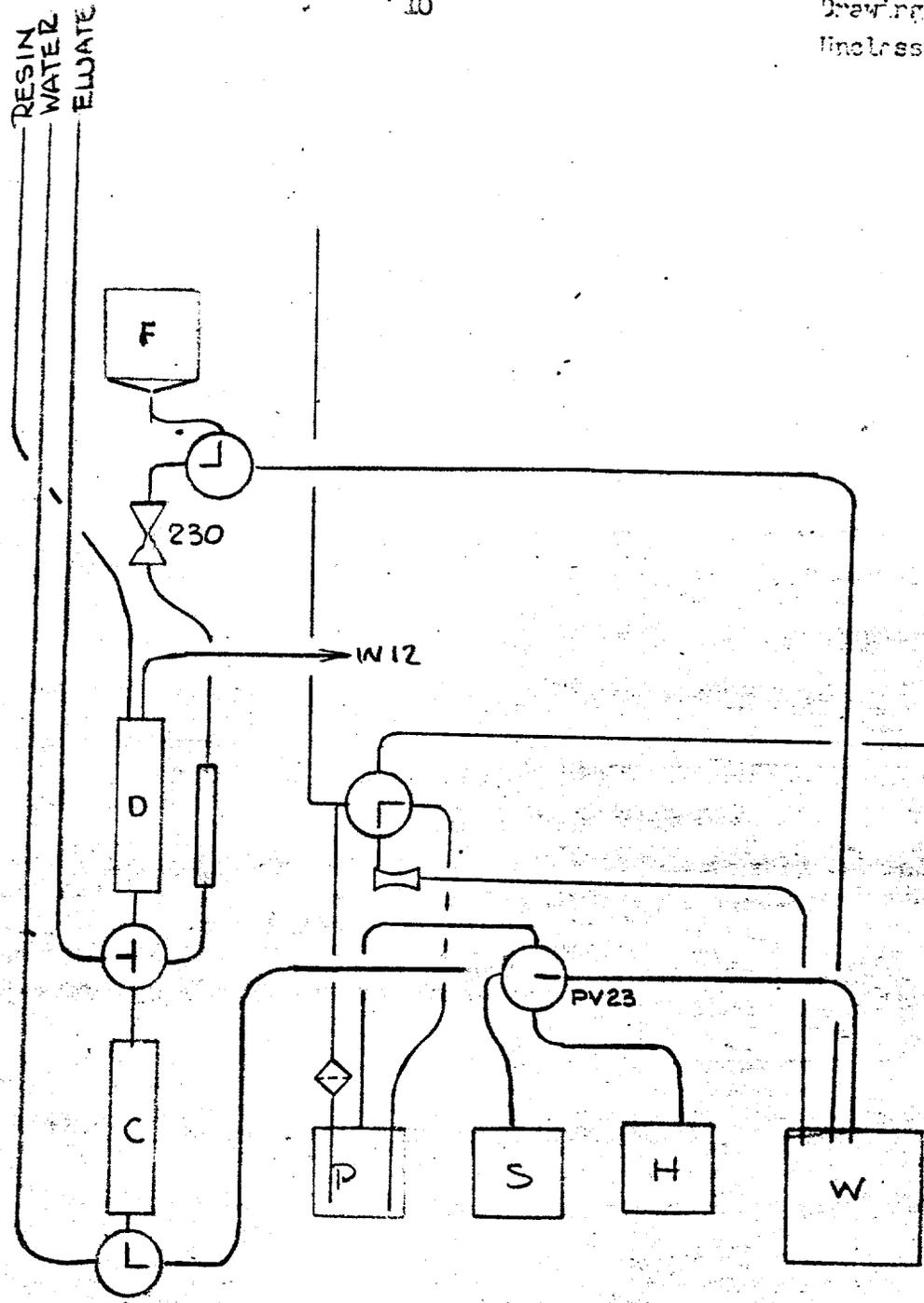
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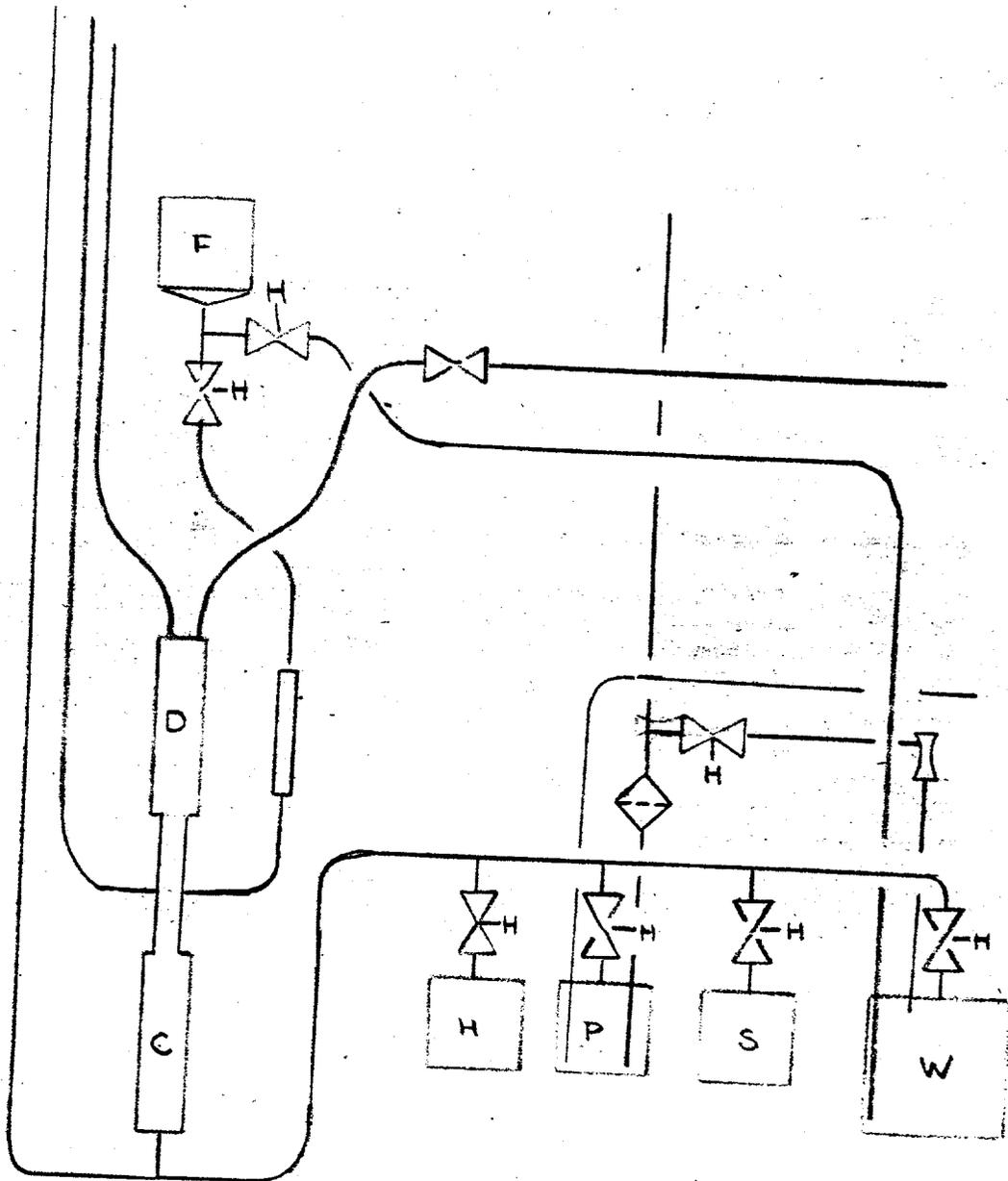
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5/26/51



A- As Is  
3/26/51



B-HYDRAULIC VALVES  
5/26/51

51-8-155

79  
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This document consists of  
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DATE: 10 August 1951

TITLE: MEB-B (MEB-Bola) Design

TO: F.L. STEADLY

FROM: W.E. UNGER

Classification Cancelled

By Authority Of \_\_\_\_\_

By \_\_\_\_\_ Date **SEP 01 1971**

PART NUMBER 2

QUARTERLY REPORT

WORK BY: G.B. BRIDG, B.F. BOTTENFIELD, P.E. BROWN, R.V. FOLTZ, E.M. SHANK,  
W.E. UNGER AND R.E. VAUGHAN

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~~\_\_\_\_\_~~  
~~\_\_\_\_\_~~

1.0 Summary:

The MTR-Sala process will be demonstrated in a full-scale pilot plant erected as a mock-up of the intended Arco plant.

The design of the pilot plant is still in the conceptual stage, approximately 10% complete.

2.0 Process Changes

The MTR-Sala flowsheet has changed; the assemblies will be dissolved in caustic as before, but the metal sludge will be dissolved in nitric acid instead of sulphuric and the barium then precipitated from the nitric solution by the addition of sulphuric acid.

3.0 Design Capacity

Los Alamos Schedule

Los Alamos has stated that the minimum acceptable shipments size is 30,000 curies measured at the time of the last chemical separation of barium from its decay products, lanthanum and cerium, and a maximum shipment time of 48 hours from the last separation time to arrival at Los Alamos. A longer delay will make the first lanthanum milking valueless because of the cerium growth. Los Alamos will accept larger shipments, apparently up to the shielding capacity of the carrier (about 100,000 curies).

The one-assembly batch appears to be optimum capacity for the separation equipment and ion-exchange isolation column. The 30,000 curies will be produced by accumulating the product of two successive runs, and making a single final chemical separation upon the combined product of the two runs. This will permit the processing of assemblies irradiated 12- days, yielding a product of higher active-to-inert barium ratio favored by Los Alamos.

Duplicate Philosophy

Los Alamos expects to schedule about 10 Sala shipments per year. The schedule may be delayed by as much as two months (by equipment failures, perhaps) without seriously affecting the program at Los Alamos. Most equipment failures can be repaired within that time, so that it would appear that duplicate processing equipment can be justified only on the basis of the doubled capacity.

[REDACTED]

[REDACTED]

This information is restricted data as [REDACTED]

[REDACTED]

This information is restricted data as [REDACTED]

Design Capacity, cont'd.

Storage Capacity

Criticality considerations limit the volume of metal-bearing wastes that can be stored in the equipment cells. It will be necessary to concentrate the metal wastes by evaporation in order to store the wastes of the eight assemblies to be processed in any four-month period as required by Los Alamos' production schedule.

4.0 Pilot Plant

The MTR pilot plant will be a full scale mock-up of the intended production plant at Arco. The pilot plant will yield precise operation information and demonstrate fully the process and equipment under simulated operating conditions.

Design Status - Pilot Plant

- Dissolver design is 50% complete.
- Conceptual Equipment Layout, 40% complete.
- Circle flowsheets, 90% complete.
- Equipment flowsheets, 90% complete.
- Conceptual assembly charging machine 10% complete.
- Conceptual product handling facilities, 10% complete.
- Service piping typicals, 70% complete.
- Preliminary cost estimate, 80% complete.

Essentially no work has begun on samplers, piping, shipping cone, charging heads, panel board nor chemical make-up areas.

\_\_\_\_\_  
Ma. E. Unger

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INV.  
64

ORNL  
CENTRAL FILES NUMBER  
51-9-39

DATE: September 11, 1951

SUBJECT: Teletype: RaLa Shipment

TO: E. J. Witkowski

FROM: Freitag

COPY NO. 1-C

E. J. Witkowski

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NO. 1 OF 2 SERIES C:

FM FREITAG US AEC RICHLAND WASHINGTON  
TO US AEC OAK RIDGE TENNESSEE  
ATTN E J WITKOWSKI

MSG NBR 3119      51 SEPTEMBER 11 1759Z      GR 45

PLEASE ADVISE AVERAGE CURIES OF RALA SHIPMENT MADE AUGUST EIGHT PD  
THIS INFORMATION DESIRED FOR COMPARISON WITH OUR CALCULATED FIGURES  
WHICH WERE REVISED TO INDICATE ABOUT TWO FIVE ZERO CURIES PER SLUG  
ON DELIVERY AT OAK RIDGE PD END REF ~~DEK~~ ODSF DEK

CCC LAST LINE READS REF ODSF DEK

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64

**DATE:** September 14, 1951

INV  
77

**SUBJECT:** Teletype: Slug Shipment

ORNL  
CENTRAL FILES NUMBER  
51.9-52

**TO:** H. R. Freitag

**COPY NO.** 1-A

**FROM:** E. J. Witkowski

Teletype Room

INV  
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contents

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ORNL  
CENTRAL FILES NUMBER  
51-9-52

September 14, 1951

To: US-AEC  
Richland, Washington

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Attention: H. R. Freitag

MSG # 508

Sept 511417002

GR 32

ANALYSES INDICATE THAT SLUGS SHIPPED ON AUGUST 8 CONTAINED 253 CURIES  
PER SLUG AT 6:00 A.M. ON AUGUST 11. END REF WITKOWSKI-508

*E. J. Witkowski*

E. J. Witkowski

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*Edmund* 11/8/95  
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Post Office Box P  
Oak Ridge, Tennessee

"This document consists of 8 pages.  
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DATE: October 2, 1951

SUBJECT: FUTURE RALA DEVELOPMENT AND PRODUCTION  
PROGRAM STUDY

TO: N. H. Woodruff

FROM: C. E. Larson

ORNL  
CENTRAL FILES INDEX  
51.10-193

COPY NO. 14-A

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October 2, 1951

U. S. Atomic Energy Commission  
Post Office Box E  
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Single rereview of CCRP-declassified documents was authorized by DOE Office of Declassification memo of August 22, 1994.

Attention: Dr. N. H. Woodruff, Director  
Division of Research and Medicine

Subject: FUTURE RALA DEVELOPMENT AND PRODUCTION PROGRAM STUDY

Gentlemen:

It has been requested in a letter from N. H. Woodruff, Atomic Energy Commission, to C. E. Larson, Oak Ridge National Laboratory, entitled "Future RaLa Requirements", (RMA:WBA:WJL), dated September 18, 1951, that Oak Ridge National Laboratory review the present status of the RaLa development and production program, and estimate the cost and time schedules that would be expected for the following program alternatives:

- Program #1 - Continued RaLa production at ORNL using Hanford or Savannah slugs.
- Program #2 - Pilot planting an MTR-RaLa process at ORNL with a subsequent production plant at Idaho.
- Program #3 - Pilot planting an MTR-RaLa process at Idaho with its subsequent conversion to a production plant at Idaho.

In addition it has been requested to determine the cost which will be incurred by April, 1952, on the present MTR-RaLa development program.

Program #1 is now in operation; however, the production goal of 30,000 curies per batch has not been reached. The first full level production run is expected in October, 1951. The time schedules for Programs #2 and #3 reflect the minimum time required for the completion of the development and plant construction (see Table I).

The program cost of RaLa production at ORNL (Program #1) will be \$501,000 for FY 1952 and \$444,000 per year for FY 1953 and succeeding years (see Table II). Since the present equipment was designed for only 10,000 curie batches,

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~~SECRET~~

Dr. N. H. Woodruff

- 3 -

October 2, 1951

it will probably be necessary to install additional purification facilities to assure the production of 30,000 curie batches in the ORNL RaLa plant. This will cost about \$400,000; \$100,000 for further chemical and equipment development and \$300,000 for the new equipment installation. The need for this additional expenditure will be dependent on the results of the next RaLa production run in the present ORNL ion exchange facility.

When it is possible to replace the Hanford slugs with Savannah slugs, the yearly program cost of the ORNL plant may be reduced from \$444,000 to \$372,000 as a result of the decrease in transportation cost.

If the RaLa production extends through a five year period, the total costs of Programs #2 and #3 will be \$4,169,000 and \$4,132,000, respectively, as compared to \$2,689,000 for Program #1 (see Table V). The costs of Programs #2 and #3 include the cost of Program #1 through the middle of FY 1954. This is necessary to provide continued production until the scheduled start-up date of a new plant provided by either Program #2 or #3. The costs of Programs #2 and #3 exclusive of the interim cost of Program #1 are \$2,625,000 and \$2,627,000, respectively, (see Tables III and IV).

Until recently, the RaLa development program at ORNL was essentially Program #2. If this program had continued, 15 man-years and \$800,000 would have been expended by April, 1952. However, the program is now being limited to chemical, unit operations and preliminary plant design study phases. This, and the preparation of final reports, will involve approximately eight man-years at a cost of \$215,000 in FY 1952.

All estimates have been made on the basis of 12 production runs per year containing approximately 30,000 curies per run. Reducing the number of production runs per year will probably not significantly reduce the yearly program cost. This is because of the usual experience of increased maintenance costs for a chemical plant not in continuous operation, and the requirement that the RaLa production be available on a two weeks' notice.

The cost data presented in this letter were based on the best information available at this time. It is believed that the cost estimates for the three programs are reliable for comparison purposes. However, following a definite program decision, further evaluation of the selected program will have to be made prior to the submission of a firm program proposal.

Very truly yours,

OAK RIDGE NATIONAL LABORATORY

*C. E. Larson*  
C. E. Larson, Director

FLSteahly-WKEister-CWSchersten/sjp/bb

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Table I

RaLa Production Program

Time Schedule\*

Program #1:	Plant now in operation
Program #2:	Pilot Plant (ORNL) Start-up July, 1952 Complete Jan., 1953 Plant Start-up (Idaho) Jan., 1954
Program #3:	Pilot Plant (Idaho) Start-up Nov., 1952 Complete July, 1953 Plant Start-up (Idaho) Nov., 1953

\*This time schedule assumes that the program decision is made early in November, 1951.

~~SECRET~~

Dr. N. H. Woodruff

- 5 -

October 2, 1951

Table II  
RaLa Program #1  
Cost Distribution

(Basis - 12 runs per year with production now available)

	FY 1952	FY 1953	FY 1954	FY 1955	FY 1956	Total
Development	\$ 87,000 <sup>(a)</sup>	\$ 25,000				\$ 112,000
Plant Modification	84,000 <sup>(b)</sup>	300,000 <sup>(c)</sup>				384,000
Operation and Maintenance	261,000 <sup>(b)</sup>	288,000 <sup>(b)</sup>	\$288,000	\$288,000	\$288,000	1,413,000
Feed: Hanford Cost	48,000	48,000	48,000	48,000	48,000	240,000
Transportation	96,000	96,000	96,000	96,000	96,000	480,000
Prod: Transportation	<u>12,000</u>	<u>12,000</u>	<u>12,000</u>	<u>12,000</u>	<u>12,000</u>	<u>60,000</u>
Yearly Total	\$588,000	\$769,000	\$444,000	\$444,000	\$444,000	
Total Program and Capital Cost						\$2,689,000

(a) \$12,000 now included in ORNL budget.

(b) Cost now included in ORNL budget.

(c) Capital cost; all other costs are program costs.

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Table III

RaLa Program #2

Cost Distribution

(Basis - 12 runs per year with production starting January, 1954)

	FY 1952	FY 1953	FY 1954	FY 1955	FY 1956	Total
Development	\$295,000	\$120,000				\$ 415,000
Pilot Pilot Facility	164,000	300,000				464,000
Pilot Plant Operation	-	213,000				213,000
Plant Design	88,000 <sup>(a)</sup>	218,000 <sup>(a)</sup>	\$ 15,000 <sup>(a)</sup>			321,000 <sup>(a)</sup>
Pre-operation		68,000	30,000			98,000
Idaho Plant		334,000 <sup>(a)</sup>	200,000 <sup>(a)</sup>			534,000 <sup>(a)</sup>
Plant Operation and Maintenance			144,000	\$ 288,000	\$ 288,000	720,000
Product Transportation			6,000	12,000	12,000	30,000
Yearly Total	<u>\$547,000</u>	<u>\$1,253,000</u>	<u>\$395,000</u>	<u>\$300,000</u>	<u>\$300,000</u>	
Sub-total						\$2,795,000
Total Program and Capital Costs						<u>-170,000<sup>(b)</sup></u> \$2,625,000

(a) Capital costs; all other costs are program costs

(b) Equipment transferred from the Idaho plant would reduce the pilot plant cost by \$50,000. Other pilot plant equipment remaining at ORNL would be capitalized at \$120,000. These cost transfers would probably occur in FY 1953.

~~SECRET~~

Dr. N. H. Woodruff

- 7 -

October 2, 1951

Table IV

RaLa Program #3

Cost Distribution

(Basis - 12 runs per year with production starting November, 1953)

	FY 1952	FY 1953	FY 1954	FY 1955	FY 1956	Total
Development	\$200,000	\$145,000				\$345,000
Plant Design	200,000 <sup>(a)</sup>	140,000 <sup>(a)</sup>				340,000 <sup>(a)</sup>
Idaho Plant	200,000 <sup>(a)</sup>	334,000 <sup>(a)</sup>				534,000 <sup>(a)</sup>
Pre-operation		78,000				78,000
Pilot Plant Operation		300,000				300,000
Plant Modification:						
Design		30,000 <sup>(a)</sup>	\$ 35,000 <sup>(a)</sup>			65,000 <sup>(a)</sup>
Construction			135,000 <sup>(a)</sup>			135,000 <sup>(a)</sup>
Pre-operation			30,000			30,000
Plant Operation and Maintenance			192,000	\$288,000	\$288,000	768,000
Product Transportation			8,000	12,000	12,000	32,000
Yearly Total	<u>\$600,000</u>	<u>\$1,027,000</u>	<u>\$400,000</u>	<u>\$300,000</u>	<u>\$300,000</u>	
Total Program and Capital Cost						<u>\$2,627,000</u>

(a) Capital cost; all other costs are program costs

~~SECRET~~

Dr. N. H. Woodruff

October 2, 1951

Table V

ReLa Production

Cost Distribution

(Basis - 12 runs per year)

	FY 1952	FY 1953	FY 1954	FY 1955	FY 1956	Total
Program #1						
Program Cost	\$588,000	\$469,000	\$444,000	\$444,000	\$444,000	\$2,389,000
Capital Cost		300,000				300,000
Yearly Total	<u>\$588,000</u>	<u>\$769,000</u>	<u>\$444,000</u>	<u>\$444,000</u>	<u>\$444,000</u>	
Total Program and Capital Cost						<u>\$2,689,000</u>
Program #2 (a)						
Program Cost	1,047,000	1,170,000	367,000	300,000	300,000	3,184,000
Capital Cost	88,000	682,000	215,000	-	-	985,000
Yearly Total	<u>1,135,000</u>	<u>1,852,000</u>	<u>582,000</u>	<u>300,000</u>	<u>300,000</u>	
Total Program and Capital Cost						<u>\$4,169,000</u>
Program #3 (a)						
Program Cost	788,000	1,092,000	378,000	300,000	300,000	2,858,000
Capital Cost	400,000	704,000	170,000	-	-	1,274,000
Yearly Total	<u>1,188,000</u>	<u>1,796,000</u>	<u>548,000</u>	<u>300,000</u>	<u>300,000</u>	
Total Program and Capital Cost						<u>\$4,132,000</u>

(a) Cost of Program #1 continues until plant start-up of Programs #2 and #3.

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51.11-188

This document consists of  
4 pages. Copy 17 of

17 copies. Series A.

DATE: November 29, 1951

SUBJECT: 706-D Building Process Modification Project  
Work Progress and Cost Analysis Summaries

TO: F.L. Culler

FROM: R.H. Vaughan

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Name Title SEP 1 1971 Date

To: F.L. Culler

From: R.H. Vaughan

Subject: 706-D Building Process Modification Work Progress and Cost  
Analysis Summaries.

The summaries as presented herein will not only describe the manner in which the final construction and installations were made but will also show a break-down of the overall program costs - development, design, construction and installation.

The construction and installation program concerning the new and revised equipment for the project was accomplished in three phases. This was done in order to minimize the plant downtime and also to be in line with the procurement and delivery dates on purchased and fabricated items. Briefly, these three phases are described as follows: (1) "A"-cell modifications which consisted of decontamination, installation of the filter vacuum systems-vacuum pots, jets, condensers, etc. and piping alterations necessary to make the proper tie-ins to the crud filter, process filter, and ion-exchange equipment; (2) Building alterations which consisted of footings, paddings, and structural work necessary for the crud filter, process filter, and the new 6-ton product carrier monorail, and excavation, forms, and concrete work for the pits, which were to receive the ion-exchange purification cubicles; (3) Fabrication, installation, testing, and break-in of all process and mechanical equipment. This included all ion-exchange purification equipment, instrumentation, samplers, product handling facilities, and product drying facilities.

The cost summary as shown herein not only embraces the "CR"-110 which was issued to cover the new construction and process changes but also includes those development costs which were accumulated through the Laboratory and Unit Operations Sections.

Specific reference is made to the Laboratory development of the Ion-Exchange Process for Purification of Radio Barium \* and the Unit Operations demonstration of Filtration and Centrifugation as applied to the isolation of Radio Barium.\*\*

\* Higgins, I.R., Blanco, R.E., Unger, Wm. E., - Preliminary Demonstration of RaLa Ion-Exchange Processes at the Kilocurie Level - ORNL-623

\*\* Higgins, I.R., - Semi-Works Demonstration of a Precipitation and Ion-Exchange Process - ORNL-621.

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*Dwight R. Humm* 3/9/95  
Technical Information Officer Date  
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TABLE NO. I

I. CONSTRUCTION BREAKDOWN (CR-110)	MATERIAL	LABOR	OVERHEAD	TOTAL
A. Design & Engineering	- - - -	\$31,650.00	\$31,650.00	\$ 63,300.00
B. Construction & Installation	\$79,485.00	64,722.00	64,758.00	208,965.00
TOTAL I.	\$79,485.00	\$96,372.00	\$96,408.00	\$272,265.00
II. DEVELOPMENT BREAKDOWN				
A. Design	\$ 3,000.00	\$49,000.00	\$ 62,000.00	\$114,000.00
B. Laboratory	20,000.00	47,000.00	60,000.00	127,000.00
C. Unit Operations	36,000.00	80,000.00	102,000.00	218,000.00
D. Break-in, Decontamination, etc	- - - -	6,800.00	6,800.00	13,600.00
TOTAL II. DEVELOPMENT	\$59,000.00	\$182,800.00	\$230,800.00	\$472,600.00
III. PROJECT TOTALS	\$138,485.00	\$279,172.00	\$327,208.00	\$744,865.00

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	<u>LABOR</u>	<u>MATERIAL</u>
I. DEVELOPMENT		
A. LABORATORY - CHEM. TECH.	\$ 47,000	\$ 2
B. UNIT OPERATIONS - CHEM. TECH.	80,000	3
C. DESIGN - GENERAL		
1. Chem. Tech.	\$ 24,700	\$ 1,000
2. Engineer	17,300	1,000
3. Instrument	7,000	1,000
	<hr/>	<hr/>
Sub-Total "C"	49,000	
SUB-TOTAL NO. I		\$176,000
II. CAPITAL DESIGN-PURCHASED		
A. ENGINEERING DEPT.		
1. Bldg. Alteration & Shielding	\$ 2,900	
2. Process Equipment	16,000	
3. Mechanical Equipment	10,750	
	<hr/>	
Sub-Total "A"	29,650	
B. INSTRUMENT DEPT.		
1. Instrument Engr. & Design	\$ 2,000	
	<hr/>	
Sub-Total "B"	2,000	
SUB-TOTAL II.		31,650
III. DECONTAMINATION		
A. OPERATIONS	\$ 2,000	
	<hr/>	
SUB-TOTAL III.		2,000
IV. CONSTRUCTION & INSTALLATION		
A. Bldg. Alteration & Shielding	\$ 24,239	3,664
B. Process Equipment	24,744	65,622
C. Mechanical Equipment	15,739	10,199
	<hr/>	<hr/>
SUB-TOTAL IV.		64,722
V. BREAK-IN		
A. CHEMICAL TECH	\$ 1,200	
B. OPERATIONS	3,600	
	<hr/>	
SUB-TOTAL V.		4,800
SUB-TOTAL V.		<hr/> <hr/>
VI. SUMMARY TOTALS		\$279,172

OVERHEAD

TOTAL

	\$ 60,000		\$127,000
	102,000		218,000
\$ 31,300		\$ 57,000	
21,700		40,000	
9,000		17,000	
<u>62,000</u>		<u>114,000</u>	
\$ 59,000	\$224,000		\$459,000
\$ 2,900		\$ 5,800	
16,000		32,000	
10,750		21,500	
<u>29,650</u>		<u>59,300</u>	
2,000		4,000	
<u>2,000</u>		<u>4,000</u>	
	31,650		63,300
2,000		4,000	
<u>2,000</u>		<u>4,000</u>	
	2,000		4,000
24,239		52,142	
24,780		115,146	
15,739		41,677	
<u>79,485</u>	64,758	<u>208,965</u>	
1,200		2,400	
3,600		7,200	
<u>4,800</u>		<u>9,600</u>	
<u><u>\$138,485</u></u>	<u><u>\$327,208</u></u>	<u><u>\$744,865</u></u>	

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OAK RIDGE NATIONAL LABORATORY  
Operated By  
CARBIDE AND CARBON CHEMICALS COMPANY



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OAK RIDGE, TENNESSEE

**ORNL**  
CENTRAL FILES NUMBER  
**53-1-72**

DATE: January 8, 1953  
SUBJECT: Teletype: RaLa Slugs  
TO: G. S. Penn  
FROM: E. J. Witkowski

COPY NO. 1-B

E. J. Witkowski

CLASSIFICATION CANCELLED  
DATE DEC 3 1958  
*Edgar J. Murphy*  
CO-ORDINATING ORGANIZATION DIRECTOR  
OAK RIDGE NATIONAL LABORATORY  
AUTHORITY DELEGATED BY AEC 9-10-57  
*EJM*

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ORNL  
CENTRAL FILES NUMBER  
53-1-72

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E J WITKOWSKI  
TO US AEC RICHLAND WASH  
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79  
ANT

**DATE:** December 6, 1951

**SUBJECT:** Teletype: Request for Forty Thousand Curies

**TO:** E. J. Witkowski

**FROM:** Freitag

**ORNL**  
**GENERAL FILES NUMBER**  
51.12-30

**COPY NO.** 1-C

E. J. Witkowski

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Arvin J. Zurst 1/18/95  
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Single rereview of CCRP-declassified  
documents was authorized by DOE Office of  
Declassification memo of August 22, 1994.

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FM FREITAG US AEC RICHLAND WASH

TO OAK RIDGE NATIONAL LAB., OAK RIDGE TENN

ATTN E J WITKOWSKI

MSG NR 3450

DECEMBER 51062047Z

GR 46 By

Classification Cancelled  
On Changed To  
By Authority Of  
Date

W J S

1 1971

REURPHONE WITH KILGORE THIS OFFICE DECEMBER FIVE CMA WE WILL PLAN  
TO DELIVER APPROXIMATELY FORTY THOUSAND CURIES ABOUT JANUARY SIX PD  
ACTUAL DATE CANNOT BE PREDICTED AT THIS TIME DUE TO INDEFINITE SCHEDU-  
LING OF SHUTDOWN PD WILL ADVISE FURTHER ASAP PD END REF ODSF CLN DEK

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FROM: Wm. E. Unger  
TO: F.L. Steahly

INV.  
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*D. R. Hammi 2/3/95*  
Technical Information Officer      Date  
ORNL Site

PART NUMBER VI  
QUARTERLY REPORT

INV.

TITLE: 706-D Modification Project  
MTR-B RaLa

WORK BY: T. . . rehart, G.B. Berry, B.F. Bottenfield, R.V. Foltz, E.M. Shank,  
.E. Unger and R.H. Vaughan.

DISTRIBUTION:

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*J. Morgan 1-31-95*  
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Date

SEP 1 1971

1.0 706-D Modification Project

Three runs of increasing activity levels have been made in the modified RaLa equipment. The filtration equipment has functioned about as designed; the purification equipment fell short of expectations and has been altered to correct the features that operating experience proved undesirable.

1.1 Alterations

The Versene Process adopted for the 706-D barium purification by ion-exchange exhibited an unexpected vulnerability to high levels of radiation, and in the last run a high percentage of the versene-complexed barium feed precipitated before reaching the column. The Laboratory Section has recommended a process modification that will employ a sodium acetate-complexed barium feed (far more stable toward radiation), followed by a versene elution, (the versene will be subjected to radiation for so short a time that its degradation will be negligible).

One of the two ion-exchange purification cubicles has been decontaminated and rebuilt to remedy certain features of the original design that were found to require correction:

- (a) The resin passage from the deaerator to the column was uncertain; and tedious techniques were necessary in resin addition to avoid flowing the resin slurry to the W-12 waste line. A valve was installed in the W-12 waste line to remedy this.
- (b) Gas accumulated in the column displaced the column liquid level resulting in "vapor locking" the resin bed. The feed line between the column and the feed tank was enlarged to 3/4-inch pipe to allow the gas accumulated in the column to be displaced upward into the feed tank.
- (c) All glass equipment and special plastic valves have been replaced with stainless equipment (made possible by lower concentrations of process reagents).
- (d) The induction flow meter element in the column feed line was removed altogether; process modifications now permit flow rates in excess of what can be obtained by the limited head available, making the flow-meter unnecessary.
- (e) Solution addition funnels were raised to a height convenient to the operators.

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### 1.1 Alterations - cont'd.

- (f) All process flowlines (except the product transfer and sample lines), formerly of tantalum, were replaced with 316 stainless 3/8-inch tubing to minimize the possibility of plugging.
- (g) The feed tank volume was increased from 18 to 27 liters.

### 1.2 Test Runs

The altered purification cubicle was completely tested by the Operations Division to insure against leaks, and then subjected to two cold test runs. Both runs were disappointing; the last run yielded the following analyses:

Feed	3.85 grams		
Losses			
Feed effluent	0.038 grams		
NaOH effluent	0.042		
Versene effluent	1.663		
HCl effluent	0.363		
Waste nitric	0.110		
Feed rinse	0.136	2.38 gms	62%
		<hr/>	
Product		1.35 gms	35%
		<hr/>	
Balance	3.85	3.73 gms	97%

The excessively high elution losses could be caused only by high pH of the versene solution. (The run was made with the versene pH high, but not enough so to have explained losses of this magnitude). It is probable that residues from the preceding caustic elution lodged in the piping manifolds, then mixed with the versene elution to effectively raise the versene pH, solubilizing the barium and enhancing the subsequent elution losses.

In the next test run a line wash will proceed the versene elution.

### 1.3 Schedule

The Operations Division reports that the November run has been tentatively rescheduled for December 20th.

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#### 1.4 Tantalum Evaporator

The Operations Division has requested that a spare tantalum product evaporator be designed and procured for them. The new evaporator has been designed (SK-101) to omit those features of the earlier evaporators that operating experience has proved unnecessary, and to add other features that will substantially increase its adaptability to equipment and process changes.

The new evaporator will differ from its prototype in the following respects:

1. There will be only one jacket.
2. The cover will be removable to afford access to the interior.
3. The dip-lines will be supported by the removable cover and will be adjustable as to depth of immersion (cover adapters of the Crawford Swagelok design will be used).
4. The coniform bottom will be reinforced with a perforated nickel cone.
5. The evaporator will not have a permanently attached filter.

The capacity and general proportions of the evaporator remain substantially the same.

The procurement of the new evaporator has been initiated by the Purchasing Department (Reqn. No. B-50537).

#### 2.0 MTR-B (Arco RaLa)

##### 2.1 General

The requirement of RaLa for Los Alamos research projects has grown from about 400 curies in 1945 to anticipated shipments of 30,000 curies. The steady trend toward RaLa shipments of increased size and the attendant quantities of irradiated uranium and plutonium that must be diverted from production channels to supply barium  $140$  in such quantities has emphasized the advantages of the MTR fuel assemblies as a rich source of barium. RaLa production at Arco from the MTR assemblies would be incidental to the uranium recovery process, and the barium extracted would be virtually a by-product. Accordingly, the Commission authorized the development of a suitable process and the design and erection at Idaho Falls of an MTR-RaLa facility.

It was proposed to carry out the process development at ORNL in a full-scale pilot plant that would demonstrate exhaustively the process and equipment. The final Idaho RaLa plant would then have been based upon sound operating experience at full-scale. Or, as an alternate, the process demonstration and pilot plant development could be carried out at Arco in a plant designed and erected for the purpose; and, after completion of the development, the plant would be converted to production.

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## 2.2 Justification of MTR-Pilot Plant at ORNL

The following considerations that favor the pilot plant development at ORNL, are briefly described:

### 1. Coordinated Process-Chemical Demonstration

The isolation of intensely radiative fission products is difficult from an operations viewpoint. The solution volumes are small and difficult to handle, the instrumentation and process control must be delicate, the processing time is limited (the product half-life is 12.8 days and the decay product, cerium, is limited by product purity specifications), and finally, the handling of process solutions requires the proper protection of operating personnel from the radiation hazards involved.

### 2. Equipment Demonstration

Process instrumentation and equipment are special and require special design because of radiation damage to the usual materials of construction and because of the perfection of performance required. The design of a production plant without an intermediate scale demonstration entails a risk that is not good practice for such high levels of radiation.

### 3. Confusion Factor at Idaho Falls.

Certain supplementary services are essential to process development; these are available at ORNL. Especially important are the following:

- a. Trained development personnel.
- b. Trained analytical personnel and analytical facilities.
- c. The operating techniques and knowhow of personnel connected with the current RaLa production facilities.

A development program at Idaho would coincide with the construction and start-up of the Chemical Processing Plant, and the consequent confusion and concerted demand on the limited facilities at IDO could seriously delay one or both programs.

### 4. Production Jeopardy at Idaho

The production of RaLa in untried equipment, with all the unpredictable difficulties to be encountered, could result in an interference with the proper operation of the 25 production plant. At ORNL the development can be carried out without threat of jeopardy to a major production facility.

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Justification of the MTR-Pilot Plant at ORNL - cont'd.

5. Cost

The cost of laboratory process development, pilot plant, design, construction, and operation at ORNL, and the design and erection of a production plant at IDO is estimated to be \$1,739,000.00.

The cost of laboratory development at ORNL, design and erection of a plant at IDO, operation of the plant for process development, and conversion of the plant to a production facility is estimated to be \$1,682,000.00.

The estimated cost differential is smaller than the accuracy of the estimate. It can be concluded, therefore, that economy is not materially a factor.

6. Research Value of Pilot Plant

The value of the pilot plant if erected at ORNL, will continue on in value as a research tool for the future development of processes involving filtration and ion-exchange at high levels of radivity.

2.3 Cost of RaLa Production

A degree of uncertainty has always been attached to the required quantities and shipment schedules imposed by the consumer, Los Alamos, and this, coupled with the scrutiny that is to be expected of the justifications for a project of this size, occasioned an overall survey of the RaLa program.

The ORNL contribution to the survey was of necessity limited to a comparison of the estimated relative costs of RaLa production at Idaho Falls from MTR fuel assemblies and at ORNL from Hanford slugs, and Aiken slugs.

The cost of RaLa production based upon ten 30,000 curie runs per year, and including transportation of feed (slugs), disposal of wastes, etc., are estimated to be as follows:

	ORNL		IDO	
	W-slugs	A-slugs	MTR	Ass'y
New expenditures required (Research, Design, Construction)	\$500,000	\$500,000	\$1,600,000	
Operating expenses, yearly				
Direct	305,000	245,000	185,000	
Direct + 10 year amortization	355,000	295,000	345,000	
Direct + 5 year amortization	405,000	345,000	505,000	

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Cost of RaLa Production - cont'd.

The low operating expenses at IDO make RaLa production there economically attractive on a long-range basis (more than ten years). If the RaLa demand is expected to be of relatively limited duration, then the continued production at ORNL from Hanford slugs, and later from Aiken slugs is more economical.

#### 2.4 Status of MTR-RaLa Design

The design of the MTR-RaLa pilot plant, intended for erection at ORNL, has been directed at providing a full scale model of the anticipated production facility to be erected later at IDO, in order to insure the most operable final plant and process.

Process and equipment flowsheets, instrument typicals, and some items of process equipment design are essentially complete; special equipment (certain process equipment, samplers, assembly charging machine, product manipulator) are in the conceptional stage. Further work was sharply suspended pending a crystallization of the consumer requirements and the effect that it might have on the Commission's decisions as to the mode of RaLa production.

MTR-RaLa design effort is currently limited to the assembly of pertinent information into an interim design report. The report is approximately 50% complete.

  
\_\_\_\_\_  
Wm. E. Unger

WEU:hh

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Low-lead plus  
RUN 44

6-7-51

at 1-6-55  
FRB 2-1-55

FLS-730

51-6-206

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TO: W.K. Eister  
FROM: W.E. Unger  
DATE: June 8, 1951.

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PART NUMBER V

QUARTERLY REPORT

TITLE: 706-D MODIFICATION PROJECT

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- 1 -

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The 706-D Modification project covered the development of a process, and the development, design, and installation of equipment to improve the yield and quality of the 706-D Bala plant. The project included the addition of a dissolver solution clarifying filter and a process filter to replace the former separation by decantation, the installation of duplicate ion-exchange purification equipment housed in floor pits, and the installation of product charging facilities to accommodate the product carrier adopted by Los Alamos.

### COSTS

The charge account (3370-36) was closed as of 26 May 1951. The costs as of that date had totaled \_\_\_\_\_ against an estimate (Unger to Stringfield, 19 Jan. 1950) of \$331,900.00, of which \$287,400.00, was budgeted (Schersten to Stringfield 28 March 1950) as project funds, the balance being chargeable against GEML operating funds. The cost increases occasioned by additional design complexities, equipment delivery delays, and competition with other urgent and high priority projects were more than off-set by savings realized from the use of reclaimed shielding lead made available by the Operations Division personnel.

### BREAK IN RUNS

The equipment was tested through a series of cold runs, one 200 curie run, and one full-scale 15000 curie run.

### 200 CURIE RUN

Approximately 60 Clinton pile slugs were charged 6 April 1951. The dissolver solution clarification filtration was slow (about 3 hours, a defective weld in the filter suction line was discovered and has since been repaired) but the process filtration was fast.

A backup from the filter to the blow-down line valve indicated the need for shielding around the valve. Hot feed solution or vapors backed up the cubicle feed tank sparger line, indicating the need for a constant purge flow of air thru the sparger line.

The ion-exchange run proceeded satisfactorily until the final nitric acid precipitation step. Apparently, the gram barium content was insufficient to exceed the solubility, and the product passed thru the filter to the waste. The waste was returned and reprecipitated satisfactorily with 500 milligrams of cold barium as a carrier.

The final evaporation in the shipping cone was slow, because of the poor heat liner of the shipping cone. This has been confirmed by Los Alamos and the liners for replacement cones are being assembled by brazing.

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~~SECRET~~ DRAFT

FULL-LEVEL RUN #44

Approximately 77, 1800 gram Hanford slugs were charged 13 May 1951. The dissolver solution clarification filtration and product filtration were fast (2.8 gpm and 1.5 gpm respectively). Hot solution back-up into the filter flow line and the ion-exchange feed tank sparger proved troublesome despite the precautions exercised by the operators. The blow line valve will be surrounded with additional shielding and the feed tank sparger line control valve will be moved close to the cubicle shielding.

The run progressed essentially without incident and with splendidly low losses until the product was eluted from the ion-exchange column. A plastic plug valve failed (a failure of the mechanical drive someplace between the valve plug and the panel board), in position to conduct the column effluent to the waste tank only. The product, with the accumulated wastes, was returned to "A" cell and reprocessed (with extremely high losses) through the spare ion-exchange equipment with a yield of about 20%.

The yields in curies from the two runs were as follows:

	LOW LEVEL		FULL LEVEL	
Dissolver Feed	248		14509	
Sulphate Precipitation	224	90.3%	13888	96.5%
Ion Exchange Column	204	82.3%	3620	24.9%
Fuming Nitric	164	66.2%	3595	24.7%

INSTRUMENTATION

Liquid Level Telemeter has worked well, about as predicted by the inventors. It is considered a useful check against the manometer but it characteristically is neither as dependable nor as accurate as the manometer. The telemeter accuracy is influenced by the relative conductivity of the solution (it behaves erratically on clean tap water, for instance), its reliability is reduced to that of its relatively vulnerable electronic circuits, and its life is limited to the radiation damage tolerable by the germanium diode element which for technical reasons must be located in proximity to the tank unit.

Induction flowmeter has proved to be a very sensitive indication of flow rate. It is apparently influenced but very little by the conductivity of the stream (the conductivity of the stream must be larger compared only to the insulating medium of the camula element, in this case, glass), and not at all by the density or viscosity of the stream. It was suspected that the deposition of foreign matter in the camula, tended to form an orifice with correspondingly higher flow velocities and indicated flow rate than was actually the case. This should be minimized, even at the sacrifice of sensitivity, by using as large a diameter camula as possible. The process unit appeared to be rugged and reliable, and its life limited only by the gaskets, and the electrical insulation on the magnet windings and electrical leads to the panel board.

~~SECRET~~ DRAFT

Instrumentation cont'd

Moore differential transmitters have worked well; however, the bellows elements do take a slight permanent set with each substantial pressure change with a corresponding annoying shift in the zero point. The panel gauges exhibited the tendency of gauges in general to display "friction-lag", making their readings appreciably less sensitive than the manometers with which they were compared.

SHIELDING

The bulk shielding is ample. Some "shine" will require that the space between the cubicle plug and the adjacent channel wall will have to be plugged with lead sheet. All other radiation was contributed by the back-up of process solutions into unshielded lines.

EQUIPMENT DESIGN

The operating difficulties that occurred during test runs and that are attributable to design features of the equipment are listed below:

Small lines

Minimum-sized process lines were selected because of the relatively low flowrates required, the expense of the tantalum tubing required by the product purity specifications. The lines have been plugged by welding scale, flakes sluffed from the teflon plug valves, and other unidentifiable foreign matter, even though the tanks and equipment were assembled with care and copiously washed.

The manometer lines were also of minimum-size, which increases the air purge pressure drop. Larger lines would have minimized the aggravation of the influence of the purge rate on the manometer reading.

Pressure Feed

It is always advisable to avoid using a process vessel as a vacuum or pressure pot, and some operational difficulties were expected if this proved necessary.

The feed tank was located in the top of the cubicle, and the receiver at the bottom to obtain as great a gravity head as possible. Provisions for augmenting the feed head by pressurizing the feed tank were made if the pressure drop across the column required it. The change from a low pressure to a higher pressure induces the back-up of solution into all unpurged lines that dip below the liquid level.

All gravity feed would have required cubicle space considerably in excess of what was available.

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COLUMN SYSTEM

The column must be capable of being charged with resin, added thru a funnel from outside the shielding; of being discharged by back flushing to a hot waste system; of accommodating process flow from inside the cubicle and eluate (cold) from without the cubicle. A valving arrangement was required but product purity specifications precluded the use of standard commercial valves, and special valves were designed and fabricated of poly-fluoro plastics. The physical characteristics of the plastics limited the valve port diameters to sizes too small to pass unslurried resin. As a result, operators must observe certain techniques in charging the resin slurries, adding to the complexity of operation.

The nature of the ion-exchange resin requires that the resin be always submerged. Any gases introduced into the system (by the radiation-induced disassociation of water, by the release of air dissolved in the elutriants, or blown in to the column at the end of a pressurized feed) accumulates in the column and gradually displaces the liquid level in the column which eventually recedes below the level of the ion exchange resin. There is no operational indication of when this has occurred, and operators are forced to interrupt the process periodically, change change valve settings, and allow any gases that may have accumulated to be displaced upward into the denatorator. Although with careful operation this is probably not necessary, the column is "bled" at least once in each run. This has increased the complexity of operation.

The column system should be provided with a means for determining the resin level at the beginning of a run, and the column liquid level continuously.

The specially-designed plastic plug valves are highly desirable from a process standpoint in that they minimize cross-contamination and the time required for valve settings. The plugs are driven by a worm and pinion, which in turn is driven by a flexible shaft extending from the valve to the panel board control handle and valve position indicator. The length of the flexible shaft and the torque is required to transmit combine to produce considerable (ca.15°) torsional deflection, referred to as "whip" or "back lash." The effect of the whip on the valve positioning is small because it is reduced by the worm and pinion drive by a ratio of 40:1, but the "springiness" of feel is disturbing to the operators who would have preferred a solid shaft drive.

The plastic valves, being dimensionally unstable, tend to flow over long periods of time to relieve concentrated stresses. The resulting deformation adversely affects the seal of the plug and the ease with which it is operated.

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### TANK DESIGN

The process tanks should have been equipped with inter-transfer jets for emergency use, with spray heads for rapid tank wash-downs, and with specific gravity manometer probes. The lines were held to minimum because of the limitations.

The tantalum evaporator manometer will read down to 500 ml volume. This is frustrating to the operators who must dissolve the final product in a maximum of 300 ml of water.

### SAMPLERS

The samplers operate fairly satisfactorily. The principal complaint is the special technique and "feel" that is necessary in positioning the sample receptacle under the sample valve. The samplers are mechanical contrivances and depend for their successful operation upon the nicety of construction. The slight differences in fabrication impart to the samplers unlike characteristics that is annoying to the operators who strongly prefer rugged simple equipment that functions very positively.

### FINAL EVAPORATION

The final evaporation that takes the product to dryness in the platinum-lined shipping cone was expected to take place in about six hours. All equipment development and installation tests were made with a brass dummy in lieu of the actual shipping cone which was not then available. The lower heat transfer across the platinum lining so decreased the evaporation rate that 12 hours were required to take the product to dryness. Future cones will be joined to the liners by silver brazing.

The charging heads are designed to avoid turbulence of the hot air stream to minimize the loss of product by "dusting". The flow of hot air is induced by the vacuum on the cone so that any leakage between the cone and the charging head would be a leakage into the off-gas system, not out into the loading cubicle. Despite this design precaution the cubicle and carrier became perceptibly contaminated. Both were easily decontaminated with a water wash.

### CONCLUSION

The mechanical failure of the process valve resulted in a non-standard run from which no positive conclusions can be drawn. All indications are that the ion-exchange process chemically accomplishes its purpose. The failure of the last run is attributable solely to equipment failure. Such features of the equipment as have evoked something less than favorable enthusiasm from the operators were clearly what at the time the choice was made appeared to be the optimum design.

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52-1-115

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DATE: January 21, 1952

SUBJECT: RaLa

BY: J. H. Pannell

TO: T. W. Clapper

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Oak Ridge, Tenn. January 21, 1952

Idaho Falls

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J. H. Pannell

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J. H. Pannell 1-31-95  
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The investigation of a precipitation process for the separation of barium has been finished and the results are being assembled in the form of a memorandum to Frank Steahly (at the suggestion of F. R. Bruce). In general terms the process is simple, rapid and small in scale; it has the disadvantage of involving two evaporations of mixed HCl-HNO<sub>3</sub> and two precipitations from concentrated HCl. As you will receive the detailed results in the above-mentioned memorandum, they will not be discussed here.

ORNL RaLa Production Run No. 46 was completed this week with apparently high success. That is to say, no large percentages of the barium were found in any of the waste solutions and one may infer a barium recovery of some 90%. Mechanical difficulties arose to plague the operators from time to time. As versene is still used to dissolve the residue of the metathesis cake which is not dissolved by a solution of sodium acetate, it is necessary to adjust the pH of this solution and run it through the ion exchange column without delay because of the susceptibility of versene to radiation decomposition. Unfortunately, more than two hours was consumed in attempting to sample this solution, probably because of misalignment of the sample cone. Eventually some of this solution contaminated the carriers and the floor around the port where

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readings in excess of 10r/hr became general. However, once a sample was obtained, there was no difficulty in getting the correct pH and the ion exchange operation worked well. The final evaporation was somewhat prolonged for during the first few hours no apparent evaporation took place. A total of about seven hours was required for this step.

An ionization measurement of the product was much lower than would be expected for the roughly 25,000 curies recovered so tests are now being made to determine if any unknown losses occurred. One possibility is that some barium still remains in the metathesis cake; another is that some was blown out with the hot air during the final evaporation. The desirability of having a barium assay on the ion exchange product effluent seems obvious. A number of improvements can also be made with a view to reducing the overall time required to separate the barium and ship it. Thus, a saving of two days, which is not inconceivable, is equivalent to 10% more product; therefore, a rapid process with a 5-10% loss may be preferable to a slower one with negligible losses.

As a consequence of the last RaLa run we are investigating the possibility of dissolving the partly metathesized  $BaCO_3-SO_4$  in  $HNO_3$  in order to (a) reduce the present repetitious metatheses to perhaps one, and (b) eliminate versene and its concomitant precise pH adjustment entirely. The chances of success with  $HNO_3$  appear to be good; they would be almost certain if the barium were not coprecipitated with 25 times its weight of lead.

The strontium-rich column effluent from the RaLa run is being returned to the column for further purification and use by the Isotopes Division. Long-lived  $Sr^{90}$  has widespread application as a gamma-free beta-emitter for use in thickness gauge

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DATE: February 18, 1952  
SUBJECT: Revision of the ORNL-RaLa Process Flowsheet  
TO: E. J. Witkowski  
FROM: R. E. Blanco

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## REVISION OF THE CHNL-BaLa PROCESS FLOWHEET

### I. Product Solubility

About 60% of the barium product from BaLa Run #46 was not soluble in water after one fuming nitric acid precipitation but was completely soluble after two precipitations. Three possible explanations for the formation of an insoluble salt from  $\text{Ba}(\text{NO}_3)_2$  are: (1) Radiation decomposition ( $\sim 9.56$  beta watt hrs./gm  $\text{Ba}(\text{NO}_3)_2$ ); (2) thermal decomposition (86.6 calories/gm  $\text{Ba}(\text{NO}_3)_2$ ); and (3) presence of sulfate or other resin decomposition products.

Laboratory experiments showed, however, that baking a  $\text{Ba}(\text{NO}_3)_2$  precipitate for 10 minutes at  $300^\circ\text{C}$  did not form an insoluble precipitate either in the presence or absence of peroxide. Irradiation of solid  $\text{Ba}(\text{NO}_3)_2$  for a week in the 3000 curie  $\text{Co}^{60}$  source ( $\sim 0.14$  watt hr./gm  $\text{Ba}(\text{NO}_3)_2$ ) did not form an insoluble compound. Experiments by Ghormley in which  $\text{Ba}(\text{NO}_3)_2$  was irradiated to 25 watt hrs./gm of  $\text{Ba}(\text{NO}_3)_2$  in a Van de Graaff beam or for eight days at the center of the X-10 pile, also showed no formation of insoluble material, although 20.0 and 3.0% of the nitrates were converted to nitrites in the two experiments respectively.

It was concluded that the barium was insoluble due to the presence of sulfate, although it is impossible to definitely state that there were no insoluble oxides present.

Only 80% of the barium is precipitated as water soluble  $\text{Ba}(\text{NO}_3)_2$

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Page 3

when the mole ratio of  $\text{SO}_4^{2-}/\text{Ba}^{++}$  is  $\sim 1.0$  using the present procedure for precipitation from fuming nitric acid. The following procedure is therefore recommended for the ORNL-Bala fuming nitric acid precipitation and the transfer of the product to the shipping cone. Experiments showed that in one precipitation this method will give a quantitative precipitation of  $\text{Ba}(\text{NO}_3)_2$  for a mole ratio of 1.0 and that two precipitations will eliminate any amount of sulfate.

Fuming Nitric Acid and Product Transfer Procedure:

- (a) Evaporate the column product eluate (17.0 L of 9.0M  $\text{HNO}_3$ ) to 8.0 L, cool to 20-25°C and add 9.0 L of 90-92%  $\text{HNO}_3$ , cool to 20-25°C, agitate 15 minutes and filter.
- (b) Wash the tank and precipitate with 2.0 L of 85% nitric acid.
- (c) Dissolve the precipitate in 300 ml of water and reflux this solution at the boiling point for 30 minutes to wash the tank walls by condensation as indicated in the regular flowsheet. Cool, and transfer the solution to the shipping cone. Analyses of dummy run product solutions indicated that 0.3 to 0.45 moles of nitric acid remain in the product tank as a filtration residue. The 300 ml product solution should therefore be 1.0 to 1.5 M in  $\text{HNO}_3$  and should dissolve a maximum of 4.5 gm of Ba as  $\text{Ba}(\text{NO}_3)_2$  at 15°C or 5.5 gm at 30°C for 1.0 M  $\text{HNO}_3$ ; or 3.3 gm at 15°C or 3.6 gm at 30°C for 1.5 M acid. A total of 5.0-6.0 gm of barium are expected as the product mass from a 30,000-40,000 curie run.
- (d) Dissolve any remaining barium in 100 ml of 0.1 M  $\text{HNO}_3$  by refluxing at the boiling point for 5 minutes, cool, and transfer the solution to

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Page 4

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(e) Repeat Step (d). The solubility of Ba as  $\text{Ba}(\text{NO}_3)_2$  in 0.1M  $\text{HNO}_3$  is approximately 4.5 gm/100 ml at 20°C.

(f) Add 17.0 L of 9.0 M  $\text{HNO}_3$  to the product tank and reflux at the boiling point for 15 minutes to dissolve any residual barium. Sample the solution and analyze for barium. If a significant amount is present, repeat the procedure outlined in Steps (a) through (e). If the amount to be recovered is less than 4,000 curies, 0.95 gm of  $\text{Ba}(\text{NO}_3)_2$  (~0.5 gm Ba), carrier should be added to the solution prior to its evaporation.

(g) The final product tank wash should be 17.0 L of 9.0 M  $\text{HNO}_3$  to insure an accurate material balance.

## II. Metathesis

The analyses from Run #46 showed that a maximum of 36% of the barium was soluble in the acetate column feed. This indicates that the balance of the barium was precipitated as  $\text{Ba SO}_4$  and that the sulfate removal by metathesis was only 97% complete. Experiments have shown that a quantitative elimination of sulfate is obtained by the precipitation of  $\text{Ba}(\text{NO}_3)_2 - \text{Pb}(\text{NO}_3)_2$  from 13 M nitric acid from solutions where the metathesis was only 90% complete. It is recommended that the following procedure be substituted for the third metathesis step on the completion of successful tracer level trial runs. The advantages of this procedure are: (1) Elimination of the pH adjustment of the highly radioactive Versene solution. In this case the Versene solution can be adjusted to pH 6.2 and passed through the

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Page 5

"cold solution tank rather than through 1-9 and the column feed tank; (2) the resin column irradiation time will be reduced by two or three hours which should result in a lower concentration of resin decomposition products in the barium eluate.

Nitric Acid Metathesis Procedure:

(a) Dissolve the cake from the second carbonate metathesis in 150 L of 6.0 N  $\text{HNO}_3$  and reflux the solution at the boiling point for 15 minutes.

(b) Cool to  $25^\circ\text{C}$  and add 15.0 L of 90-92%  $\text{HNO}_3$ . Cool to  $25^\circ\text{C}$ , agitate 15 minutes, and filter.

(c) Wash the tank and precipitate with 5.0 L of 13.0 N  $\text{HNO}_3$ . The filter system should be sucked as dry as possible to reduce the acid hold up in the line and filter to a minimum. The presence of an excessive amount of hold up acid would render the solution of the precipitate unsuitable as a column feed in the subsequent step.

(d) Dissolve the precipitate in sodium acetate and water and proceed as in the regular flow sheet. It will not be necessary to add the 2.0 L of 1.0 N  $\text{HNO}_3$  previously used to dissolve the metathesis cake.

*R. E. Bianco*

R. E. Bianco

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BY: James H. Pannell

TO: T. W. Clapper

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Oak Ridge, Tenn. February 25, 1952

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RaLa

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It is now acknowledged, by the Operations Division, that RaLa run number 46 narrowly escaped being a fiasco. Low ion-chamber readings on the final product were disregarded, at the time they were made, and the shipment, assumed to be about 25,000 curies, was started on its way. Dr. N. E. Ramsey had some misgivings, however, and he started an investigation which resulted in the discovery that two-thirds of the product was still in the final evaporator where it had not been redissolved, and transferred to the cone. It was quickly recovered and the original shipment recalled.

The most probable explanation for the difficulty encountered in redissolving the  $Ba(NO_3)_2$  is that it consisted largely of  $BaSO_4$  resulting from decomposition of the Doven-50 resin. Such decomposition may be caused by radiation or attack by the 9N  $HNO_3$  used as a barium elutriant. I investigated the latter effect several months ago and provided data from which the amount of sulfate produced could be roughly predicted. Another member of the laboratory section has studied radiation decomposition of the resin and has shown that loss of capacity can be correlated with sulfate found in solution. He obtained a value of 10% loss in capacity per watt-hour of adsorbed radioactivity per gram of resin. This effect is larger than that due to attack by  $HNO_3$ , moreover, it is also harder to control for it is not possible to foretell exactly how long the resin will be subjected to irradiation. Removal by radiation decomposition of 0.7 per cent of the sulfonic groups releases an amount of sulfate equivalent to the barium and although some of this sulfate would undoubtedly be eluted before the barium, one can see what a potential nuisance this resin decomposition might become.

As a consequence of the unexpected difficulties encountered in Run #46 it has been necessary for Ray Blanco to introduce an additional one and perhaps two, fuming  $HNO_3$  separations. A double precipitation of the barium in the column effluent will be required to ensure the removal of sulfate there; a precipitation of barium nitrate after dissolution of the metathesis cake is now being considered in order to eliminate sulfate at that point also. In the laboratory I have shown that a 90% metathesis cake (one containing 10% of its original  $SO_4$ ) can be dissolved best in 15 liters of 6N  $HNO_3$ . Addition of 15 liters of 90%  $HNO_3$  precipitates 99+% of the barium as  $Ba(NO_3)_3$ .

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Pannell to Clapper  
Subject: RaLa

February 25, 1952  
Page 3

Additional separations mean a longer total processing time and greater losses of barium so it appears that the ion exchange process is, perhaps, becoming more involved than is justified. In this connection there have been favorable comments on the precipitation process that I investigated somewhat briefly, although it must be admitted that ion exchange is the best choice for "cold" solutions.

Continuous measurement of pH in the RaLa column feed solution would make the versene process a more easily controlled one. At present, samples are removed and taken to an adjacent laboratory for measurements so that even when the sampler is working properly, several persons receive a severe dose of radiation. It has been tacitly assumed that pH electrodes would not work in a solution containing as much as one curie per milliliter and that remote maintenance is not possible. In the absence of any evidence for these assumptions I am beginning tests to determine how nearly valid they are. The first tests are aimed at discovering the effect of radiation on the glass electrode, as this is the crux of the problem. I am optimistic about the chances of measuring pH continuously in "hot" solutions and believe that there is a need for so doing in several current operations.

It appeared wise actually to determine the distribution of  $\text{HNO}_3$  between hexone and 1.6 M  $\text{Al}(\text{NO}_3)_3$  but this has turned out to be more difficult than was anticipated because of the extreme inaccuracy of chemical analyses for hydrogen ion in this system. As a result, at low concentrations of  $\text{HNO}_3$  it is not possible to say what the D.C. is but that values between 1 and 3 are all equally likely. For aqueous  $\text{HNO}_3$  concentrations originally between 0.5N and 2N, a D.C. of about 2, in favor of the hexone, has been obtained. It is expected that fairly accurate values can be obtained at these, and higher,  $\text{HNO}_3$  concentrations, from which the data for low acid systems may be deduced by extrapolation.

Among other activities of this month; I have had the Mathematics Panel construct a nomograph for computation of MTR fission product radioactivities and am attending a course on reactor theory.

Additional weekly time sheets are needed beginning with the week ending February 16.

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J. H. Pannell

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FROM: R. E. Blasco

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May 2, 1952

## 1.0 Introduction

Recent development work\* indicated that the pH adjustment of the radivo Pala Versene feed solution could be avoided and a single "cold" Versene solution used for strontium elution if the carbonate metatheses were followed by a precipitation of  $\text{Ba}(\text{SO}_4)_2$  from 13.0 M nitric acid. The nitric acid precipitation will remove the last traces of sulfate from the system if the carbonate metathesis has been at least 90% complete. The amount of acid remaining as a heel with the precipitate was a critical point since this acid would be present in the ion exchange column feed and could increase the barium losses in the ion exchange process.

Experiments were therefore performed in the full scale production equipment to determine both the acid hold up in the filter system and the number of carbonate metatheses required to remove 90% of the sulfate. The results showed that the acid hold up would vary from 2.0 to 5.0 moles depending on the filtration rate which in turn is dependent on the amount of crud on the filter. A 200 curie run indicated that the barium loss would be high if the acid content of the feed was 5.0 moles and 17.0 L of 0.07 M, pH 6.3 sodium Versenate were used for the elution of strontium. The pilot plant runs also showed that two carbonate metatheses would not eliminate 90% of the sulfate from the system.

## 2.0 Laboratory Development

A series of runs were made on 1/3<sup>rd</sup> scale to determine the effect of the nitric acid content of the ion exchange feed solution and also determine the optimum conditions for strontium elution with Versene. It was shown that 11.0 L of 0.07 M sodium Versenate at pH 6.3 was sufficient for the strontium elution and that under these conditions the total barium loss for the ion exchange process would be 2.0 to 5.0% for feeds containing up to 10 moles of nitric acid.

## 3.0 Conclusions

It is recommended that the usual three carbonate metatheses be used prior to the nitric acid precipitation to ensure at least 90% removal of the sulfate.

The effect of the nitric acid concentration of the feed solution is not as great as was expected, probably due to the buffering action of the sodium acetate. Thus this process appears worthy of further trial in the full scale equipment.

4.0 Summary of Operating Procedure for the Revised Process

4.1 Dissolution, extraction, and three carbonate metatheses as in regular flow sheet.

4.2 Nitric Acid Precipitation

4.21 Dissolve the metathesis cake in 15.0 L of 6.0 N HNO<sub>3</sub> and reflux the solution at the boiling point for 15 minutes.

4.22 Cool to 25°C. If the volume is less than 14.7 L, butt to 15.0 L with 6.0 N HNO<sub>3</sub>.

4.23 Add 15.0 L of 98-99% HNO<sub>3</sub>, cool to 25°C, agitate 15 minutes and filter.

4.24 Wash the tank and precipitate with 5.0 L of 13.0 N HNO<sub>3</sub> and suck filter system as dry as possible.

4.25 Dissolve precipitate in 2.0 L of water and 2.0 L of 2.5 N sodium acetate. Agitate 5 minutes and transfer to the ion exchange feed tank. Follow with 6 liters of water added through the slinger ring.

4.3 Ion Exchange

<u>Solution</u>	<u>Volume (Liters)</u>	<u>Flow Rate (Liters/min.)</u>
(a) Acetate feed	10.0	0.5-0.6
(b) Water (feed tank wash; sparge 1 min.)	2.0	0.5-0.6
The following solutions pass through the elution tank		
(c) Lead elution (1.5 N NaOH)	17.0	0.35
(d) Water	2.5	0.35
(e) Strontium elution	11.0	0.35
(f) Water	2.5	0.35
	7.5	0.50
(g) Sodium elution (1.0 N HCl)	15.0	0.44
(h) Water	4.0	0.44
(i) Product elution	6.0	0.35
	2.0	0.09

May 2, 1952

Note: The elution tank should be rinsed with water after steps (c), (e), and (g) to prevent contamination of succeeding solutions. The flushing should be performed as quickly as possible since there will be no liquid flowing through the column during this operation.

#### 4.4 Final Precipitation and Product Transfer

- 4.41 Evaporate the product solution to 6.0 L, cool to 20-25°C, and add 7.0 L of 50-92 HNO<sub>3</sub>.
- 4.42 Cool to 20-25°C, agitate 15 minutes, and filter. Wash with 2.0 L of 90% HNO<sub>3</sub>. Suck filter as dry as possible since excess acid will inhibit subsequent dissolution of the product.
- 4.43 Add 300 ml of water, reflux at the boiling point for 30 minutes, cool, and transfer to the shipping cans.
- 4.44 Add 100 ml of H<sub>2</sub>O, reflux for 5 minutes, and transfer to shipping cans.
- 4.45 Repeat 4.44.
- 4.46 Add 17.0 L of 9.0 HNO<sub>3</sub> to product tank and reflux at the boiling point for 15 minutes to dissolve any residual barium. Sample the solution and analyze for barium.
- 4.47 If a significant amount of barium remains, repeat steps 4.41 to 4.46. If the amount to be recovered is less than 4,000 curies, 0.95 gm of Ba(NO<sub>3</sub>)<sub>2</sub> carrier should be added to the solution prior to evaporation.

---

R. W. Blanco  
Laboratory Section  
Chemical Technology Division

REB:sp

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ORNL  
CENTRAL FILES NUMBER  
525-32

DATE: May 5, 1952

COPY NO. 8

SUBJECT: RaLa Meeting with Los Alamos Personnel  
at ORNL; Status of ORNL-RaLa

"This document consists of 3 pages.  
No. 8 of 8 copies, Series A"

TO: F. L. Steahly

FROM: R. E. Blanco

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Date SEP 01 1971

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To: T. E. Stealy

May 5, 1952

Page 2

Subject: RaLa Meeting with Los Alamos Personnel  
at ORNL; Status of ORNL-RaLa

A meeting was held in M. E. Ramsey's office on April 16, 1952, to discuss ORNL-RaLa. Those in attendance were Phil Hammond from Los Alamos, and M. E. Ramsey, E. J. Witkowski, Harris Blauer, W. D. Unger and R. E. Blanco from ORNL.

Carrier Cooling System

Phil Hammond stated that in their opinion the cooling system was necessary. At the time RaLa run #46 (23,000 curies) arrived at Los Alamos with the cooling system in operation, the carrier temperature was  $6^{\circ}\text{C}$  higher than room temperature. He also requested the use of a neoprene gasket in the future.

Size of Shipments

The size of the next few shipments was not critical and could vary from 20,000 to 30,000 curies.

Product Purity

The product from run #46 (23,000 curies) was treated at Los Alamos as follows:

(1) The product was dissolved in dilute nitric acid and poured into a clean container. A grey filter-Cel like material failed to dissolve.

(2) The  $\text{Ba}(\text{NO}_3)_2$  was then precipitated by the addition of 90%  $\text{HBr}_2$  and the solution centrifuged. The crud settled but soon rose again indicating a density close to 1.0.

(3) The supernate was decanted and discarded.

(4) After 2-3 days the precipitate was again dissolved and reprecipitated with 90%  $\text{HBr}_2$ . The supernate was decanted and the La precipitated by the addition of fluoride. The precipitate was recovered by centrifugation along with some of the unknown material and heated to  $400^{\circ}\text{C}$  in a subsequent processing step. The product was black and porous and was considered unsatisfactory.

(5) Another nitric acid precipitation on the remaining original product did not remove all of the impurity.

(6) Lanthanum was then added and a hydroxide precipitation made. On centrifugation the  $\text{La}(\text{OH})_3$  carried down all of the flocculent material.

(7) The supernate containing the barium was decanted and treated by the usual fuming nitric acid procedure. A satisfactory product was obtained in this case.

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*Handwritten:* 3/8/52  
L. E. Stealy

To: F. L. Steahly

May 5, 1952  
Page 3

Discussion:

It was assumed that the unknown material was organic because of the black color of the baked residue. The possibility of the presence of acetate, Versene, or resin radiation or chemical decomposition products was discussed. It was pointed out that acetate, Versene, and the resin chemical decomposition products are soluble in fuming nitric acid and are thus eliminated in the final ORNL step. The fate of radiation decomposition products is not known. A possibility was mentioned that the organic material could have come from the shipping cone gasket.

It was proposed that Los Alamos make the hydroxide precipitation regularly since they must discard the first milking anyway because of the large amount of Ce present. Phil Hammond agreed to this but pointed out that the AEC is considering air shipment in which case the first extract could be used if the product was processed within 48 hours of the L.S.T. at Oak Ridge.

It was also agreed that a single filtration of the final product at Oak Ridge, rather than a hydroxide precipitation, might eliminate the impurity since it is not soluble in water, dilute  $HNO_3$ , or fuming  $HNO_3$ . Los Alamos cannot do this since they do not have a filter and must resort to a hydroxide floc to carry the impurity down during centrifugation. Such a procedure would require the addition of several valves to the ORNL system in order to use the existing filter. It also assumes that the organic material is insoluble at the time of shipment, i.e., it is not soluble organic material which is subsequently rendered insoluble by the irradiation during shipment. It is interesting to note in this connection that no insoluble material was observed by the ORNL personnel in the final shipping cone prior to evaporation. The possibility of making a hydroxide precipitation at ORNL was discussed. It did not seem practical because it would require an extra tank which in all probability could not be fitted into the present equipment.

The other three products shipped to Los Alamos in 1951 were not evaluated for purity.

The alternatives to be considered are: (1) No further action should be taken at this time until the future of the RaLa program is more fully defined, the quality of additional shipments evaluated, or until air shipment is a reality; or (2) the equipment should be revised immediately while it is still relatively "cool" to determine the efficiency of a final filtration of the product.

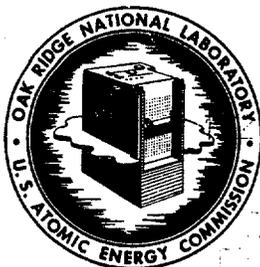
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F. E. Blanco

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**ORNL**  
CENTRAL FILES NUMBER  
52-7-16

RECEIVED - ORNL  
OFFICE OF DIRECTOR

DATE: July 1, 1952  
SUBJECT: RaLa Project Committee Meeting #1  
TO: M. E. Ramsey  
FROM: E. J. Witkowski

COPY NO. 2-A  
*C E Larson*

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By [Signature] Date SEP 1971

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[Signature] 1/19/95  
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		Noted
2	Larson, C. E.	<u>[Signature]</u>
3	H. E. SEAGREN	
1	Rueff, P. W.	1
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M. E. Ramsey

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A  
July 1, 1952

E. J. Witkowski

RaLa Project Committee Meeting #1

F. L. Steahly opened the meeting by explaining the purpose of the Committee organization and how the Committee will function. He reviewed the success with which other project Committees have operated within the Chemical Technology Division and thought that the RaLa Committee could operate with equal success by keeping everybody connected with the project informed of current developments and by a cooperative effort solve our mutual problems to the satisfaction of everyone concerned. The main points concerning the organization which were brought out are as follows:

1. The Committee will consist of five members. The Chemical Technology Division will be represented by R. E. Blanco of the Laboratory Section, T. A. Arhart of Unit Operations and W. E. Unger of the Design Section. The Operations Division will be represented by H. Blauer and E. J. Witkowski.
2. The Committee will meet at regular intervals to be sure that no problems are overlooked and that all problems are kept on a current basis.
3. The Directors of both the Operations and the Chemical Technology Division may attend the Committee meetings when they so desire or the situation warrants their presence.
4. There will be no permanent Committee Chairman. A Chairman for each meeting will be chosen by mutual agreement of the Committee members. The Chairman will usually be the member who has the most information to present to the Committee at that time. The Chairman will write the minutes for the meeting.

The rest of the meeting was devoted to a discussion of Los Alamos' current product appraisal. In a telephone conversation with E. J. Witkowski on June 30, R. P. Hammond indicated that there were some impurities in the last product which have created a great deal of difficulty at the end of the processing at Los Alamos. He believed that these were organic materials with possibly silica, boron or phosphorus. He said that if the batch we are now processing turns out no better, the entire project may be abandoned.

The group could not understand how any impurity which may have been sent with the product could remain with the product at the end of the Los Alamos process especially after Los Alamos had agreed to add the NaOH clean-up step. In an attempt to further purify the product in the run now in progress however, it was agreed that a second fuming nitric precipitation, plus a fuming nitric boiling step would be added prior to the transfer of the product to the shipping cone.

M. E. Ramsey

- 2 -

July 1, 1952

The possibility of revising the equipment in order to filter the product as it is transferred to the cone was discussed but it was agreed that it could not be accomplished in time to change the decision governing the future of the program.

It was suggested that a portion of the scrap from the Los Alamos product be brought to Oak Ridge for analysis since after several years of encountering similar difficulties, Los Alamos has failed to give us a specific appraisal of our product based on chemical analysis. It was also suggested that in the event the entire project were abandoned, one run should be made if for no other purpose than to analyze for product impurities in order to solve the mystery once and for all.

The possibility of the product becoming contaminated with gasket material in transit was also discussed. A suggestion was made to determine the efficiency of fuming  $\text{HNO}_3$  in removing gasket material as well as resin from the product.

Everyone agreed that we should make every effort to help Los Alamos out of the difficulty they are having with our product. The Committee agreed that representatives from Oak Ridge should visit Los Alamos when the next batch is processed to try to learn first hand what may be causing their troubles. The Committee felt that a first hand knowledge of their process may also suggest improvements which may be made in our own process.

*E. J. Witkowski*  
E. J. Witkowski

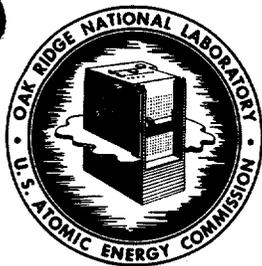
EJW:hg

cc: C. E. Larson  
F. L. Steahly  
W. E. Unger  
B. E. Blanco  
T. A. Arhart  
H. Blauer

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**ORNL**  
CENTRAL FILES NUMBER  
**52-7-17**

DATE: July 3, 1952  
SUBJECT: Teletype: Proposed Schedule for Next  
RaLa Discharge  
TO: G. F. Penn  
FROM: H. F. Stringfield

COPY NO. 1-A

AEC, Teletype Room



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ATT: G. F. PENN  
  
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FROM: H. F. Stringfield  
  
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W. J. Patten Date SEP 1971

*H. F. Stringfield*  
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MSG NR 389 July 5 2032000Z MESSAGE GR 92

IN REGARD TO OUR CONVERSATION OF JULY 2, WISH TO ADVISE THAT YOUR PROPOSED SCHEDULE FOR NEXT RA LA DISCHARGE IS SATISFACTORY, THAT IS, DISCHARGE ON JULY 23, 1952, SHIP ON JULY 24 ARRIVE AT OAK RIDGE ON JULY 28, 1952. WE DESIRE SAME AMOUNT AS SHIPPED ON HGE-ORL-138 NAMELY 54,000 CURIES.

ALSO WISH TO ADVISE THAT MILWAUKEE 1043 WAS SHIPPED ON JULY 3, 1952 LOADED WITH EMPTY CONTAINERS NUMBERED, PHOENIX 1, MONSTERS X-4377, X-36632, X-36614 AND TUSCOM 1.

End Ref Stringfield 389

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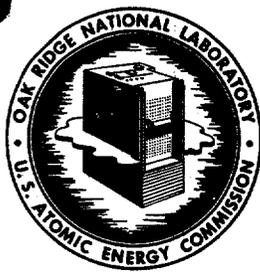
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**ORNL**  
CENTRAL FILES NUMBER  
52-10-111

DATE: October 13, 1952

COPY NO. 5-A

SUBJECT: FUTURE RALA REQUIREMENTS

TO: Mr. Kenneth Kasschau

FROM: Dr. C. E. Larson

Classification Cancelled  
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By Authority Of DOC  
By [Signature] Date SEP 2 1971

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- 1-3 Kenneth Kasschau
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- 11 Hezz Stringfield
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- 13 E. J. Witkowski
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OAK RIDGE, TENN.

October 13, 1952

U. S. Atomic Energy Commission  
Oak Ridge Operations  
Oak Ridge, Tennessee

CLASSIFICATION CANCELLED  
*Arvid J. Lusk* 4/15/95  
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Single rereview of CCRP-declassified documents was authorized by DOE Office of Declassification memo of August 22, 1994.

Attention: Mr. Kenneth Kasschau

Subject: FUTURE RALA REQUIREMENTS

- References:
1. Letter to NHWoodruff from CELarson, dated October 2, 1951; subj: Future RaLa Development and Production Program Study; ORNL CF #51-10-193.
  2. Letter to CELarson from NHWoodruff, dated September 18, 1951; subj: Future RaLa Requirements; ORO-10228.
  3. Letter to CELarson from NHWoodruff, dated May 11, 1951; subj: RaLa Production for Los Alamos; ORNL CF #51-5-85.
  4. Letter to JHRoberson from CELarson, dated April 5, 1951; subj: RaLa Production Schedule for FY 1952; ORNL CF #51-4-24.
  5. Letter to JHRoberson from CELarson, dated March 7, 1951; subj: Revised Los Alamos RaLa Requirements; ORNL CF #51-3-15.

Gentlemen: Concerning provisions for an adequate supply of RaLa...

Operation of the ORNL RaLa equipment has not been entirely satisfactory at the nominal 30,000-curie level. The two major reasons for our current production difficulties are the additional time required for the larger runs and the higher radiation exposure of personnel which evolve from the design level of 10,000 curies per batch versus operation at 20,000-30,000 curies per batch. Production of the larger-sized RaLa products necessitates the dissolution of the slugs in six batches, resulting in six separate extraction filtrations. These extraction filtrations generally get progressively slower with each successive filtration, requiring excessive processing time. Equipment failures have been relatively frequent, as no doubt would be anticipated for operation at present capacity and with equipment designed and built only to serve an interim period for production of RaLa until the permanent RaLa facility could be built at Arco or elsewhere. Presumably, the permanent facility would have been completed or near completion at this date, except for cancellation of the Arco development work until the firm decision was made on the future needs, if any, for RaLa.

**RESTRICTED DATA**

Future RaLa Requirements---  
CELarson to KKasschau

2.

October 13, 1952

This decision, according to recent information from Los Alamos, was to be made by the middle of September 1952; but current contacts with Los Alamos indicate that the decision as to the need for a continued supply of RaLa, if any, is still unsettled.

Since there is no alternate supply of RaLa, ORNL may now be expected to supply all RaLa needs for the next year from the interim RaLa equipment, regardless of the future course of the RaLa production program. Due to the difficulties of producing RaLa, it is our recommendation that if 30,000-curie RaLa runs are attempted, they be attempted only on a bimonthly basis. Smaller (10,000-curie) runs could be made on a monthly basis.

It is requested that the Laboratory be advised as to the nature of the RaLa demands that we will be expected to supply for the next two to three years. Definite information on RaLa requirements is necessary because it has been estimated that at least one year would lapse between the design and construction of the facility. Need for improved RaLa facilities for production of 30,000-curie runs at Oak Ridge National Laboratory was anticipated as early as October 1951, as shown in reference 1. This need has not changed; however, the time required for the design and building of a completely new RaLa production plant would be much greater than one year.

The long-range demand for RaLa--that is, on a five-to-ten-year basis--should be determined if possible, since ORNL studies have indicated that the long-range need is perhaps the major consideration in determination of the location of a permanent RaLa facility. If the long-range need is known, studies concerning provision for an adequate supply of RaLa will be clarified to the extent that fewer proposals will need to be evaluated.

Any information you may be able to obtain regarding the future needs for RaLa will be of value to the Laboratory.

CEL:MER:wp/le

Distribution:

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- 8-10 F. L. Steahly
- 11 Hezz Stringfield
- 12 M. E. Ramsey
- 13 E. J. Witkowski
- 14-15 C. E. Larson

Very truly yours,

OAK RIDGE NATIONAL LABORATORY

*C. E. Larson*  
C. E. Larson  
Director

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DATE 12/1/64

For The Atomic Energy Commission

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TO US AEC OAK RIDGE TENN

ATTN H F STRINGFIELD/E J WITKOWSKI, ORNL

MSG NR 4577

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Chief, Declassification Branch *OE*

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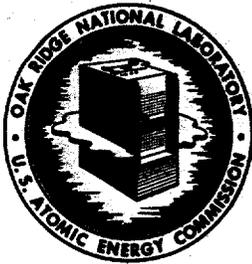
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**ORNL**  
CENTRAL FILES NUMBER  
52-11-145

DATE: **November 20, 1952**  
SUBJECT: **PLANNING FOR FUTURE RALA REQUIREMENTS**  
TO: **Mr. Kenneth Kasschau**  
FROM: **Dr. C. E. Larson**

COPY NO. **23**

Distribution

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POST OFFICE BOX P  
OAK RIDGE, TENN.

November 20, 1952

U. S. Atomic Energy Commission  
Oak Ridge Operations  
Oak Ridge, Tennessee

Attention: Mr. Kenneth Kasschau

Subject: PLANNING FOR FUTURE RALA REQUIREMENTS

Gentlemen:

This is in reply to your letter of November 19, 1952, on the subject, "Planning for Future RaLa Requirements," in which you state that it has now been decided that RaLa will be needed on a bi-monthly basis in 30,000-curie batches in the next five- to ten-year period. To meet this requirement, the RaLa program will, therefore, have to be divided into a temporary period involving production of RaLa in the present 3026 Building for a two- to three-year period, followed by a permanent facility which will provide 30,000-curie batches with an occasional combination of three of the smaller batches.

NE

The present RaLa facility consists of dissolving equipment designed for 500 curies and cubicles designed for 10,000 curies. This facility has been utilized to produce product of triple design capacity, 28,000 curies, but these runs were accompanied by process and equipment difficulties. Also, it should be noted that the present RaLa facility was to fill interim needs until a permanent facility was built. The present equipment has already met its expected interim commitments.

The present RaLa facility, must, of course, supply AEC requirements until a new facility can be built. It is intended that this be done by repair and only minimum alteration to the present equipment. Extensive alterations to existing equipment are considered economically impractical even though it is recognized that the supply of RaLa may be somewhat uncertain. Completion of a permanent RaLa facility at the earliest possible date, whether at ORNL or elsewhere, is strongly recommended by the Laboratory.

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FUTURE RALA REQUIREMENTS -  
CELarson to KKasschau

-2-

November 20, 1952

Since the size of the usual RaLa requirement will be 30,000 curies per run, rather than 100,000 curies, Oak Ridge National Laboratory may well be the most advantageous site for location of the permanent facility.

The Laboratory would recommend the construction of the new RaLa facility as an adjunct to the proposed multicurie fission product plant. Such a combination would provide the AEC with an opportunity to effect a saving in operations of the two facilities, in that personnel could be used interchangeably between the two plants. Secondly, the RaLa plant would provide a ready source of fission products of value to the Radioisotope Program and zirconium-niobium for radiological warfare evaluation.

The RaLa plant would utilize normal-size slugs from either Hanford or Savannah River reactors, which, of course, insures a continuously available supply of feed from production program reactors and avoids the complications encountered in the use of irradiated enriched U<sup>235</sup> as feed material. At a 30,000 curies-per-run level, ORNL would use a process similar to the precipitation process that was used for over forty RaLa runs, thereby permitting utilization of previous design and process experience gained over several years of RaLa production.

It is estimated that necessary unit operations and equipment development, design, and construction of the new facility would require from thirty to thirty-six months and would cost a total of approximately \$1,500,000.

In reference to a RaLa facility for the Idaho Chemical Plant, we would recommend a precipitation process similar to that proposed above for the Oak Ridge permanent facility. This recommendation is based on recent experience at ORNL and is in disagreement with the MTR RaLa proposal contained in our letter of October 2, 1951, to N. H. Woodruff, entitled "Future RaLa Development and Production Program Study" (ORNL CF 51-10-193).

Because of the differences in the feed materials proposed for Idaho and Oak Ridge, some chemical development would be necessary to make the proposed precipitation process applicable to MTR source material. The Laboratory would be willing to assist in this development program, but a reliable cost estimate would require a study of the joint effort of ORNL and Idaho personnel.

Very truly yours,  
OAK RIDGE NATIONAL LABORATORY  
*C. E. Larson*  
C. E. Larson  
Director

CEL:MER:FLS/vmw  
Attachment: 1 drawing

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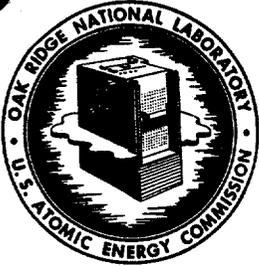
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**ORNL**  
CENTRAL FILES NUMBER  
**58-6-96**

COPY NO.

DATE: June 11, 1953  
SUBJECT: DISCUSSION OF ORNL RaLa  
PROCESSING PROBLEMS  
TO: F. L. Culler  
FROM: R. E. Blanco

"This document consists of 6 pages.  
No. 1 of 8 copies, Series A"

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DATE 10-19-60  
*Edgar J. Murphy*  
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F. L. Culler

DATE June 11, 1953

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E. J. Witkowski  
Central Files

ANSWERING LETTER DATE

SUBJECT Discussion of ORNL Ra La  
Processing Problems

CLASSIFICATION CANCELLED

*Robert J. Dunt* 1/18/95  
ADD signature Date

Single rereview of CCRP-declassified  
documents was authorized by DOE Office of  
Declassification memo of August 22, 1994.

1.0 INTRODUCTION

The purpose of this memo is to acquaint the interested parties with the current ORNL-RaLa processing problems. At a recent meeting several suggestions were made as to possible changes in the RaLa operating procedure which could increase the efficiency of the process. These changes are outlined below along with data from recent laboratory filtration studies and general background material. The background material was derived from a survey of the data from the last eight RaLa runs. It is expected that another meeting will be held shortly to discuss the problems and make definite recommendations for process changes.

2.0 GENERAL BACKGROUND MATERIAL

2.1 Filtration

RaLa run #52 was carried out during the week of May 25, 1953. The major product losses in this run were as follows: 40% as the result of filtration problems between the extraction tank, A-9, and the ion exchange feed tank; 6.8% in the fuming nitric acid waste; and an estimated 10 to 20% due to overflow of the product shipping cone.

The large loss during filtration has been attributed to the malfunction of a new large area filter. The filtration rate using this filter was excellent but it proved impossible to blow the precipitate back from the filter quantitatively. Only a portion of the filter is cleared in this

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manner and the remainder of the precipitated product is thus lost. A portion (18%) of the loss due to the use of this filter was the result of mechanical loss of fines through the filter. Previous runs had consistently shown losses of  $< 2\%$  through the small process filters. This variation in losses could be attributed to a difference in porosity of the two filters but is more likely due to the action of the siliceous crud present in this solution which acts as filter aid and provides a finer filtration media.

The installation of a large filter was made for two reasons; (1) a process change required the filtration of a much larger volume than originally specified, and (2) the filtration rate through the small filter had become progressively worse. This lower filtration rate appears to be the result of progressive plugging of the small filter or the failure to pull sufficient vacuum due to leaks in the system. Initial runs (#45 and #46) using the small filter had adequate filtration rates.

It was proposed that either the first large metathesis step or one of the small metathesis steps be eliminated to lower the filtration time and permit the use of the small filter. The large metathesis step was instituted as a combined metathesis and tank wash. In this case a carbonate metathesis is carried out and the final solution diluted with water until the tank is filled. The solution is then filtered. This procedure has proved to be an efficient method for insuring the removal of sulfate from the process lines and tank walls. A run (#46) performed prior to the use of this procedure showed that only 24% of the metathesis cake was soluble in the nitric acid-sodium acetate ion exchange feed solution but that the remainder was soluble in the subsequent Versene tank wash-feed solution. These figures can be compared with run #47, where a large tank wash was used. In this case 78% of the metathesis cake was soluble in the acetate feed. This illustrates the effectiveness of the large tank wash in removing sulfate. This is important, since Versene is comparatively much more unstable to radiation than acetate and thus its total radiation time should be minimized. Experience has shown (run #45) that if the curie content of Versene feed is as high as 21,000 curies and the total contact time is as high as 10 hours, that enough Versene will be

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decomposed to allow a rise in pH and subsequent precipitation of a portion of the product in the ion exchange feed tank. It should be noted that the Versene feeds in runs #44 and #46 contained 15,000 and 16,000 curies each and were successfully processed. In these cases the total contact time was low, ~2 hours. This data has been reviewed to point out the fact that it is advantageous to hold the curie content of the Versene feed to a minimum since unexpected equipment difficulties may cause a prolonged Versene exposure time. It is recommended that the third small metathesis be eliminated since the residual sulfate contamination appears to be derived from mechanical operations rather than from incomplete chemical metathesis.

### 2.2 Fuming Nitric Precipitation

The losses in the fuming nitric acid precipitation have ranged from 1.1 to 10%. It appears that this loss is largely mechanical by the passage of fines through the filter since a chemical loss should be fairly consistent under constant operating conditions. Los Alamos has recently stated that 3 grams of Ba carrier can be used. The use of this carrier should cut the loss in the fuming nitric acid precipitation by approximately one half.

### 2.3 Product Cone Overflow

This loss is the result of the difficulty in determining the exact volumes present in the final processing steps where the volumes are small. This loss has not occurred to any appreciable extent in previous runs.

## 3.0 LABORATORY FILTRATION DATA

A suggestion was made that diatomaceous filter aid be used to increase the efficiency of the small filter. Laboratory experiments were performed using Celite 545 to determine the effect of the two metatheses (boiling 4 M  $K_2CO_3$ ) and the alkaline (pH 11) sodium Versenate feed on the siliceous filter aid. The water filtration rates for a 1/2 inch cake were determined before and after the treatment outlined. The initial rate was 5.4 gal/in<sup>2</sup>/hr. The first carbonate filtrate was initially clear but turned slightly cloudy on standing overnight. The second carbonate filtrate and the Versene filtrate (after adjustment to pH 6.1) did not turn cloudy on standing. A

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few fines which came through the filter were observed, however. A portion of the Versene solution was passed through a RaLa scale ion exchange column and no appreciable drop-off in flow rate was observed. Analyses of the filtrate solutions for silica are not available as yet. The Celite cake showed a 7% weight loss. This is not considered significant in view of the amount of fines which passed through the filter. Judging from this data and the appearance of the filter aid, it appears feasible to use Celite 545 in the extraction and metathesis steps.

#### 4.0 PROPOSED OPERATING REVISIONS

The alternate revisions to be considered are listed below:

- (1) Use one large metathesis, where the tank is completely filled, and one small metathesis. Add filter aid (Celite 545 - 2 gm/in<sup>2</sup> of filter area) and 5 gm of Ba(NO<sub>3</sub>)<sub>2</sub> carrier to the first extraction solution.
- (2) Use two metatheses the first of which will be twice the volume of a normal small metathesis. This procedure may provide adequate washing of the process lines and prevent contamination of the cake with sulfate. In this case filter aid would not be required due to the smaller volumes. The barium carrier should not be added in this case since it would tend to increase the possibility of sulfate holdup. It would be added just prior to the boildown for the fuming nitric acid precipitation.
- (3) Two complete runs could be made of ~15,000 curies each using procedure (2) outlined above. The same resin would be used after regeneration with NaNO<sub>3</sub> and Na acetate. The first run should go completely through to the shipping cone to avoid the buildup of sulfate in the product tank. The extraction tank and process filter should be flushed and cleaned between the two runs. In this case 2 1/2 grams of Ba(NO<sub>3</sub>)<sub>2</sub> would be added as carrier prior to the fuming nitric acid precipitation steps.

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It appears that alternate #3 is the safest and gives the most assurance of a product shipment but it is obviously the most expensive and time consuming. A full batch using alternate (2) is not too attractive in view of the low filter rates observed on the small filter in the last run. Alternate (1) therefore appears to be the best choice although it runs the risk of the use of filter aid without a full scale pre-trial.

*R. E. Blanco*

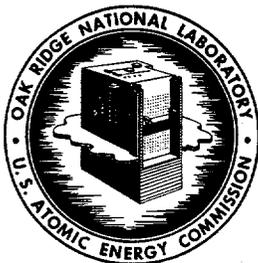
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POST OFFICE BOX P  
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ORNL  
CENTRAL FILES NUMBER  
58-6-80

DATE: June 11, 1953  
SUBJECT: Letter Re: Rala  
TO: G. F. Penn  
FROM: E. J. Witkowski

COPY NO. 2A

CLASSIFICATION CANCELLED  
DATE 10-19-60  
Edgar J. Murphy  
CO-ORDINATING ORGANIZATION DIRECTOR  
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June 11, 1953

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Series A

G. F. Penn  
U.S. Atomic Energy Commission  
Hanford Operations Office  
P. O. Box 530  
Richland, Washington

Dear Mr. Penn:

This letter is to confirm our telephone conversation of June 12.

It is understood that the next shipment of this slugs is scheduled to arrive on July 10, 1953. Also that the shipment will contain approximately twice the product we normally require, that is, 100,000 curies and no less than 90,000.

If there is any need for a change in schedule, we would prefer to receive it earlier. In that case we would appreciate being notified as far in advance as possible so that we may schedule our operations accordingly.

Very truly yours,

*E. J. Wilkowski*

E. J. Wilkowski, Superintendent  
Chemical Operations Department



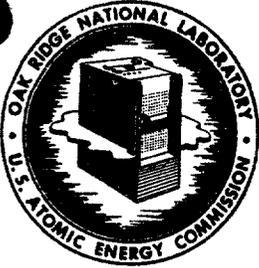
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**53.7.149**

DATE: July 23, 1953  
SUBJECT: HP HAZARDS - RALA  
TO: H. E. Goeller  
FROM: W. E. Unger

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*David R. Hammin* 2/3/95  
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OAK RIDGE NATIONAL LABORATORY

TO: H. E. Coeller

DATE: July 23, 1953

FROM: W. E. Unger

SUBJECT: HP HAZARDS - RALA

PRESENT: E. A. Charpie  
H. E. Coeller

E. G. Strunness  
W. E. Unger

ABC: Robert Myers  
R. P. Coekler

The background count of the OREL area has been steadily rising since permanent air-count records have been made. Many peaks have occurred and most have been explained, but the most consistent offender seems to be the Rala operation. The HP organizations are interested in reducing all contributions to air contamination and have resolved to begin with Rala, both because of the severity of the problem and the frequency with which the problem recurs. The recent indications that OREL will be asked to continue to supply Rala requirements for some years has emphasized HP's interest in the problem.

That the Rala operation is an offender is abundantly clear from the repeated coincidence of air-count "peaks" during Rala runs. By the time that the air-count monitors respond, so much time has elapsed that the particular step in the operation responsible for the air count is difficult to determine. HP technologists are further hampered by a lack of detailed information about the process, equipment, and operations.

HP regard their work as "detective" work and plan to cut down the time of response and confusion as to the source of the air count by locating recording monitor take-offs in the stack, tank farm vents, off-gas ducts, etc. They will require detailed familiarity with the operation to plan their work intelligently and will require a good deal of cooperation before, during, and after a run.

We suggested that this be instrumented by a committee with representatives from Operations, Chemical Technology Design, Shift Supervision, and Health Physics.

We briefly described the process and equipment, the probable potential sources of air contamination, and offered to go over drawings of D-Building equipment with HP representatives and supply them with any information and assistance at our disposal.

Strunness may take advantage of the offer. There has been over this many times before.

Bob Charpie was not keen on the "organization" of a committee, but I believe that the formal establishment of a committee saves a lot of confusion in the long run.

Wills E. Unger  
Process Design Section  
Chemical Technology Division

WJU:cmh

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/	C. E. WINTERS	
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CENTRAL FILES NUMBER  
53.10. 67

DATE: October 8, 1953  
SUBJECT: PROPOSAL FOR RAAI PRODUCTION  
TO: K. A. Kasschau  
FROM: C. E. Larson

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- 8. A. M. Weinberg
- 9. C. E. Winters ←
- 10. Hezz Stringfield, Jr.
- 11-14. F. L. Culler
- 15. E. J. Witkowski
- 16. M. E. Ramsey
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POST OFFICE BOX P  
OAK RIDGE, TENN.

October 8, 1953

United States Atomic Energy Commission  
Oak Ridge Operations  
Post Office Box E  
Oak Ridge, Tennessee

Attention: Mr. K. A. Kasschau, Director, Division of Research and Medicine

Subject: PROPOSAL FOR RALa PRODUCTION IN THE LABORATORY

Gentlemen:

The removal of construction funds for a new RaLa plant from the Idaho Operations Office FY 1954 budget extends the time that Oak Ridge National Laboratory will be required to continue operation of the existing interim RaLa unit. In view of this circumstance and in concern for the age and limited capacity of the existing plant, the Laboratory would like to repeat our proposal, made initially in December, 1952, to design and construct a permanent RaLa facility at Oak Ridge National Laboratory, at a total program cost of \$1,500,000 to be completed within 30 months from the time of program approval.

There are technical and economic advantages favoring the installation of the permanent RaLa plant in Oak Ridge rather than at the Idaho Chemical Plant. The process which would be used at ORNL would differ little from the process which has been employed for several years; a new and untried process must be used for feed from the MTR. Standard fuel slugs from either Hanford or Savannah River would be used as feed, thus assuring a reliable feed source from a number of production reactors rather than from a single experimental reactor. Plutonium and uranium slugs can be recovered in the Metal Recovery Plant.

It is our understanding that the estimated cost of development, design and construction for a RaLa plant at the Idaho Chemical Plant is \$2,700,000 (Report No. ACCO-14,203) as contrasted to our estimate of \$1,500,000. The potential saving of \$1,200,000 is particularly significant in view of Lewis L. Strauss's recent letter to S. R. Sapirie (dated August 14, 1953) stressing the importance of major program cost reductions.

Construction of the permanent facility at ORNL can be completed and operation started in 30 to 36 months, thus relieving the present inadequate plant at the earliest practicable date. The Laboratory would propose to schedule

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Mr. K. A. Kasschau -3-  
Proposal for RaLa Production

October 8, 1953

the design and construction of the facility so as to avoid interference with currently established programs.

Building a combined RaLa and Multicurie Fission Product Plant is a possibility if both of these facilities were to be located at ORNL. Advantages in lower initial cost and in the common use of experienced personnel for operation would accrue to each of the units. RaLa process waste would serve as an excellent source of kilocurie quantities of other fission products such as zirconium-niobium.

Regardless of the location of the permanent RaLa facility, it is recommended by the Laboratory that the RaLa plant be built at the earliest possible date. We would appreciate your early consideration and reply to this proposal.

Very truly yours,

OAK RIDGE NATIONAL LABORATORY



C. E. Larson  
Director

CEL-FLC:vmw

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X-426 (Revised 1-52)

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*Rala file*



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**ORNL**  
**CENTRAL FILES NUMBER**  
54-2-142

DATE: February 19, 1954

COPY NO. 1A

SUBJECT: Investigation of Rala  
Filtration Difficulties

TO: A. F. Rupp

FROM: E. J. Witkowski



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TO: A. F. Rupp ~~Or Changed To~~ DATE: February 19, 1954  
By Authority Of \_\_\_\_\_

FROM: E. J. Witkowski By WJW Date SEP 3 1971

SUBJECT: INVESTIGATION OF RALA FILTRATION DIFFICULTIES

CLASSIFICATION CANCELLED  
ADJ 1/18/95  
ADD signature Date  
Single rereview of CCRP-declassified documents was authorized by DOE Office of Declassification memo of August 22, 1994.

INTRODUCTION

One of the most troublesome parts of the Rala operation since the major equipment and process revisions were made almost three years ago, has been the frequent plugging of the process filters during the filtration of lead-barium sulfate precipitate out of UNH and the filtration of lead-barium carbonate slurry after matathesis. A decision was made to re-examine certain parts of the process after run No. 54 was completely lost in November, 1953, as a result of these difficulties. It became evident during this run that the process filters were being plugged by an incomplete removal of foreign material, probably silica, from the UNH in the preceding crud filtration step; also, that the fines in the Celite 545 filter aid were passing through the crud filter and interfering in the subsequent filtrations through the process filters. (For a complete report on run No. 54, see memorandum, E. J. Witkowski to A. F. Rupp, "Summary of Run No. 54 With Significant Analytical Results", C.F. No. 53-12-19).

The investigation was carried out in two parts. The first was to check the effectiveness of the coating removal procedure in removing the aluminum and the silicon alloy bonding material and the second part was to investigate the filter aid used in order to improve it or possibly entirely eliminate it from the process. The coating removal tests and filter aid classification were performed by Mr. P. B. Orr of the Operations Division. The microscopic studies and particle size determinations were made by Mr. T. E. Willmarth of the Analytical Chemistry Division.

COATING REMOVAL

The first test was an attempt to roughly duplicate, on a small scale, the coating and bonding removal procedure in use in order to visually ascertain the effectiveness of the procedure. The coating and bonding removal was performed on two 4" Hanford slugs in a stainless steel bucket heated with a gas burner.

The first step was the removal of aluminum by a solution of 10% NaOH and 20% NaNO<sub>3</sub> at a temperature of 100°-105°C for one hour. The second step was a

[REDACTED]

repetition of the first step to insure complete removal of the aluminum. It was apparent that the removal of the aluminum was complete after these two steps and that the second may have been superfluous. The black looking bonding material, however, was left on the uranium metal, practically untouched.

The two slugs were then treated with 44 lbs. of 6%  $\text{HNO}_3$  containing 6.4 gm. of  $\text{Hg}(\text{NO}_3)_2$  at a temperature of  $100^\circ\text{C}$  for three hours. This step, which was supposed to undercut the silicon alloy bonding, removed only a very small portion of the material which readily settled to bottom of the dissolver as a black flaky precipitate. The bulk of the bonding material remained on the slugs. The unre-moved bonding material would normally stay in the UNH as a precipitate after the uranium was dissolved and, as tests later showed, would plug the UNH crud filter unless filter aid were used.

The procedure for undercutting the bonding was revised and the slugs were heated at  $100^\circ\text{C}$  in a 20%  $\text{HNO}_3$  solution for 34 minutes. This procedure completely removed the black bonding material which had settled out in the bottom of the dissolver as a flaky precipitate. The surface of the slugs became bright and clean and showed little damage from the action of the acid. The solution contained only 4.94 mg/ml uranium; this uranium loss would be considered insignificant in the Rala process. The solution also contained 4.6 ppm of silicon.

In order to determine the extent of treatment with 20%  $\text{HNO}_3$  required, the slugs were again heated in 20%  $\text{HNO}_3$  for 15 minutes. There was no apparent precipitate in the solution and the solution contained 3.0 mg/ml uranium and 3.4 ppm silicon; the proportion of silicon to uranium was roughly the same in both cases.

An attempt was then made to find a suitable method for dissolving the fine pieces of bonding alloy desposited on the bottom of the dissolver to facilitate their removal. In order to get an adequate amount of this fine alloy deposit for test purposes, 10 ORNL slugs were dejacketted and the bonding removed using the same procedure as that used on the first two slugs.

The only solvents found which would dissolve the bonding material in a reasonable period of time were solutions containing free HF. Cold solutions of 5%  $\text{NH}_4\text{HF}_2$  and  $\text{NH}_4\text{F}$  dissolved the precipitate rapidly but reacted on the uranium slugs and formed another precipitate which was identified as  $\text{UF}_4$ . The formation of the  $\text{UF}_4$  precipitate plus the corrosive nature of the fluoride chemicals which would probably damage the equipment over a long period of time made their use unattractive in the Rala process.

Solutions of NaOH and KOH up to 50% concentrations with alternate treatments with  $\text{HNO}_3$  up to 70% concentrations showed no visible effects on the bonding alloy

even after prolonged periods of boiling in these solutions.

It was apparent that the removal of the bonding material from the dissolver would have to be accomplished by mechanical means; that is, vigorous washing. Since the bonding material is relatively heavy and not easily suspended by agitation with water a decision was made to try 50% NaOH.

To test the effectiveness of the 50% NaOH in suspending the bonding precipitate, eight more 4" Hanford slugs were de jacketted and bonding undercut with 20% HNO<sub>3</sub>. The dissolver was emptied except for the slugs and the bonding precipitate and 50% caustic was added, boiled and sparged. The caustic was then syphoned out and observed to carry a large portion of the undissolved precipitate.

#### FILTER AID TESTS

As mentioned in the introduction to this report, there was evidence that some filter aid passed through the UNH crud filter and contributed to the difficulties experienced in run No. 54. After the run was completed, the crud filters (Porosity G micrometallic stainless steel) were decontaminated, removed and tested. A water slurry of filter aid was filtered and the filtrate observed to be slightly cloudy. When the filtrate was boiled down, a white precipitate settled out which was identified under a microscope to be filter aid. The same test, made on two new replacement filters, gave identical results.

The literature put out by the Micro Metallic Corporation, the manufacturer of the filter media used, indicates that porosity "G" filters have a mean pore opening of 10 microns and that they can be expected to remove particles down to one-third the diameter of the mean pore opening. Since the microscopic examination of the Celite 545 filter aid revealed that 57% of the particles were under 4 microns in diameter and that many of the particles were less than 1 micron in diameter, it is not surprising that some of the filter aid sifted through the filter.

The penetration of the filter media by some small particles of filter aid might present no problem if it were possible to precoat the media and wash it with an adequate amount of water to eliminate the small particles. It might also work satisfactorily if it were possible to recirculate the UNH through the filter. Unfortunately, neither of these procedures is possible. The UNH must be added to the water from the precoating operation and it cannot be recirculated because of the nature of the equipment.

In order to roughly check the possibility of performing the UNH filtration without the use of filter aid and to attempt to improve the filter aid in the

event its use was necessary, three, two-slug batches of UNH were made by dissolving the slugs which had been thoroughly cleaned during the testing of the coating removal procedures. It was, of course, impossible to duplicate the exact operating conditions by doing the dissolving operations in a stainless steel bucket. The time required for the dissolving operation was several times that normally encountered in the regular Rala process and it was necessary to make additions of  $\text{HNO}_3$  while the dissolvings were in progress to compensate for the acid which evaporated.

It was found that (although the rates vary considerably) all batches could be filtered through a small laboratory type stainless steel porosity "G" filter if air was periodically blown back through the filter when the rate dropped below a practical level. This experiment could not be considered conclusive because of the variation in filtration rates between batches and because the crude equipment used made it impossible to approach the true operating conditions in the Rala process. The experiment served one purpose: it indicated that filtration without filter aid was possible and that it might be practical to try it in the Rala equipment.

To improve the grade of filter aid in the event its use was found necessary, a 3" diameter 6' long glass tube with a glass filter on the bottom and overflow on top was used to hydraulically classify the Celite 545 filter aid for the next Rala run. Using this equipment, enough filter aid was classified for the next complete run. An examination of the classified material under an electron microscope showed that the fines in the Celite could be reduced by this crude method from 57% to 34% under 4 microns in diameter (see photomicrographs). This material, although not completely satisfactory, would be an improvement over that used in all previous runs.

A small laboratory-type stainless steel filter was precoated with the classified filter aid and used to filter some of the UNH solution. The filtration was satisfactory with no noticeable decrease in rate. A second filtration of the filtrate was made without using filter aid, with no appreciable change in rate.

In addition to trying to improve the quality of the normally-used diatomaceous earth filter aid, a relatively new type of carbon filter aid, called "Nerofil", produced by Great Lakes Carbon Corporation was tested. The filtration rate through a precoat of this material was about 1/3 as rapid as that through diatomaceous earth, but the flow was steady. There is a possibility that this filter aid might work out more satisfactorily than the siliceous type because it would be inert to all the chemicals used in the Rala process.

[REDACTED]

[REDACTED]

[REDACTED]



Plate M 1479

Unclassified Filter Aid

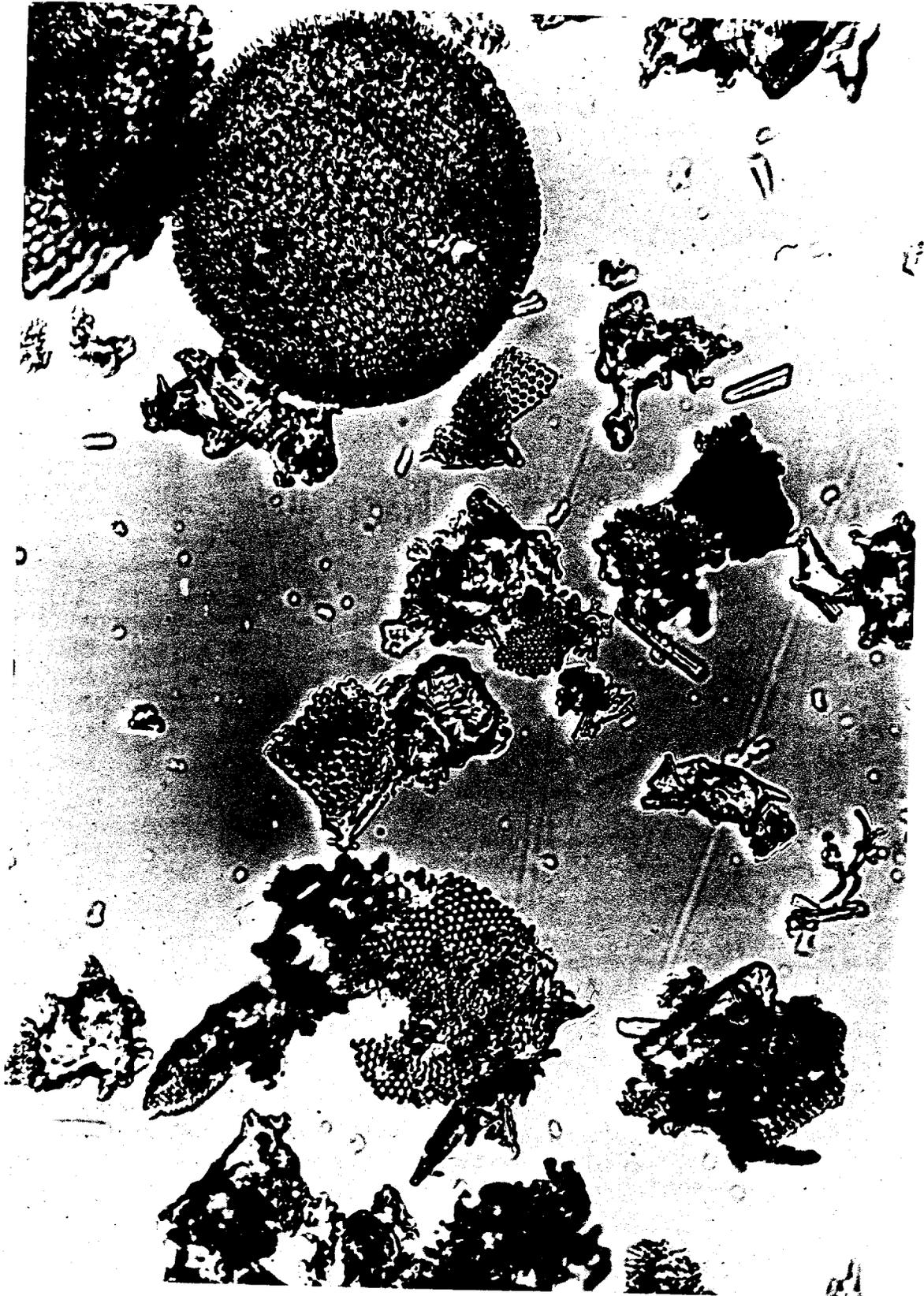


Plate M 1483

Classified Filter Aid

500 X

EXPERIENCE WITH AN EXPERIMENTAL AND A PRODUCTION RUN

Based on the results obtained in these tests it was decided to make a Rala run without using any filter aid in the filtration processes. An air line was connected to blow back the UNH crud filters to help clear them whenever they plugged. In the event it became necessary to use filter aid, the classified Celite material would be used.

To improve the chances for a successful filtration, with or without filter aid, the coating removal procedure was revised to include a 20% HNO<sub>3</sub> undercutting of the bonding material, the water washes were increased in volume and number, and the washing techniques were revised to flush out the precipitated bonding material.

A decision was also made to test the Nerofil filter aid on a full scale batch of UNH if some slugs were left over after the next Rala run.

The new slug coating and bonding removal and the UNH filtration procedures were tried on a full scale, first in a 138-curie experimental run and then in a 65,000-curie production run (No. 55).

The experimental run was made with four batches of UNH dissolved from a mixture of twelve 4" W, fifteen 8" W and 130 X bonded slugs. The UNH filtration of all four batches was successfully accomplished without using filter aid, at an average rate of 15 minutes per batch or 3 gal./min.; it was not necessary to stop the filtration to unplug the filters at any time. The sulfate extraction filtrations averaged approximately 40 minutes per batch or 2.25 gal./min. The two metatheses, with a combined volume of 30 gallons, filtered in 82 minutes or at an average rate of 0.37 gal./min. The filtration rates, as a whole, were the fastest and the most consistent ever experienced. At no time was it necessary to delay processing because of an excessive time required for filtration.

The production run which followed was made from 192 W slugs dissolved in 10 batches. It was the biggest and the most successful to date. The same new procedures were followed as in the test run. The results were comparable except that it was necessary to periodically speed up the UNH filtration by blowing back air through the filters. The first batch of UNH seriously plugged one of the filters but at no time was processing delayed by filtration difficulties. The plugged filter was readily cleaned after the run was over, by using 20% NaOH. No difficulties were experienced with the sulfate extractions and metatheses filtrations.

The Nerofil filter aid was tested in the filtration of a batch of UNH left over from the production run. Only 4.5 slugs of uranium were found to have

dissolved as compared to 19 in a normal batch. The UNH filtration rate was only 1 gal./min. as compared to 3 gal./min. average in the test run without filter aid but the rate was consistent and it was not necessary to blow back the filter. The rate was about what was expected. The metatheses and extraction filtrations were comparable to those in the experimental run and production run No. 55. The total activity contained in this batch was 872 curies. All analyzed waste losses were low but 158 curies (18%) could not be accounted for in the material balance. There is a remote possibility that the unaccounted for loss was a result of using the Nerofil filter aid.

CONCLUSION

Because of the success of the production run which can be at least partially attributed to the elimination of filter aid in the filtration of UNH, a decision was made to follow the same procedure in all future runs. In the event that filtration becomes difficult, Nerofil will be tried in place of Celite 545 previously used.

*E. J. Withowski*

EJW:hg

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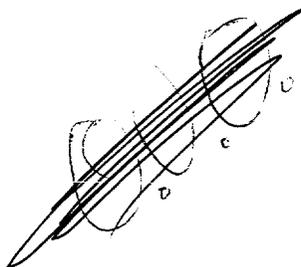
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OAK RIDGE, TENNESSEE

ORNL  
CENTRAL FILES NUMBER  
54-6-45

DATE: June 2, 1954  
SUBJECT: RALA PRODUCTION  
TO: K. A. Kasschau  
FROM: C. E. Larson



This document consists of 3 pages.  
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POST OFFICE BOX P  
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June 2, 1954

U. S. Atomic Energy Commission  
Post Office Box E  
Oak Ridge, Tennessee

~~Classification Canceled~~  
E. U. S.  
W. K. S.  
Date

Attention: Mr. Kenneth A. Kasschau

Subject: RALA PRODUCTION

Gentlemen:

Rala run No. 56, which was started on April 26, had to be discontinued because of the release of radioactive gases caused by an abnormally vigorous reaction in the dissolver. The Rala processing building was contaminated and the gases were carried northward by a light breeze contaminating buildings in that area with 22-hour  $P^{433}$  and 8-day  $P^{131}$ . This occurred at about 4:58 p.m. April 29; immediate decontamination and isolation procedures were applied in the affected areas during the night so that areas other than actual operating building were in fairly good shape by morning.

During the period of this emergency, personnel were evacuated from the affected areas, radiation surveys made, and all necessary safety precautions taken. No serious radiation exposures or internal dosage of radioactivity were sustained by personnel; a few radiation exposures higher than normal were sustained by personnel in the operating area resulting from emergency measures taken after the "fume-off" occurred, rather than from direct operating exposure. These exposures were largely to supervisors who directed and actually performed most of the emergency work.

In searching for the reason for this occurrence, the first question asked is whether there was an operational error. From the standpoint of standard operating procedures, this can be answered negatively, since the same operating procedure has been used for the past ten years. To answer more fully, one must review at least a part of Rala history. The original Rala plant was designed and built to produce 500 curies of Rala per batch on a relatively short-term basis. During the years, there has been a steady increase in demand: 1,000, 5,000, 10,000, and then 30,000 curies per run.

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J. B. Morgan 1-31-95  
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-2-

June 2, 1954

Indeed, in the previous successful run, over 65,000 curies were shipped in two batches. At the 30,000 curie level, this represents an increase in production of over 60 times the designed capacity of the plant! It is true that the finishing section of the plant was rebuilt in 1950 to increase the capacity to 10,000 curies (immediately increased to 30,000 curies), but the primary dissolving and precipitation sections of the plant have never been changed.

As a consequence of inadequate equipment capacity, it is necessary to dissolve the metal for these big runs in batches instead of all at one time, as should be done. In addition to this, time and corrosion have taken their toll, and the thermocouples in the dissolver have long since been gone. Repairs are not possible because the cell cannot be entered, and decontamination would be extremely difficult because of the disintegrating, heavily contaminated concrete walls and corroded, leaking equipment.

The recent "fume-off" occurred at the beginning of the addition of 60% HNO<sub>3</sub> for the fourth dissolving of the second portion of the run. It is believed that the residual uranium metal was heated to an abnormally high temperature by absorption of its radiation during the waiting period (28 hours) between the third and fourth dissolvings. The warm uranium, the surface previously etched by the other dissolvings, reacted vigorously with the nitric acid, and gases were given off in too great a volume for the off-gas system to handle.

It is not unusual for various kinds of mishaps to occur during Rala runs, for the equipment is old and the design is that of the early days of the Project. Back-ups into exterior lines and contamination from sampling and product removal are frequent. The recent spreading of contamination was unusual in that gaseous radioactivity was released containing short-lived iodine which is absorbed on dust, almost any kind of surface, grease, oil, paint, etc. Therefore, it was not possible to limit the spread of contamination to the operating building itself.

It is fortunate that virtually all of the contaminating activity was short-lived radioiodine, since the rapid decay helped during decontamination and radioiodine is not as toxic as general fission product mixtures. The buildings and grounds over which the gas passed were contaminated to give radiation readings ranging from 0.5 to 20 mr/hr. and averaging roughly 5 to 6 mr/hr. As a comparison, the much more widespread contamination with neptunium and general fission products from the Nevada bomb test fall-out in March 1953 gave radiation readings of about 2 mr/hr. This comparison is offered not to minimize the results of the recent Rala "fume-off," but rather to bring it into its true perspective.

It is not necessary to cite the many statements made to the AEC by the Laboratory, urging that a new Rala facility be built, either at ORNL or elsewhere, for the last letter to Carbide and Carbon Chemicals Company from the

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June 2, 1954

AEC, March 14, 1954, "Review of Proposed Rala Production Plant for ORNL," recognizes this fact as indicated in that letter.

"The AEC is aware of the difficulties experienced by ORNL in current production of sources required by Los Alamos Scientific Laboratory. You may be assured that every effort will be made to hasten the date at which your antiquated production plant can be retired."

We request that only those Rala runs most urgently required by Los Alamos be made in the future and that increased emphasis be placed upon the early start-up of Rala production at Arco.

Very truly yours,

OAK RIDGE NATIONAL LABORATORY

C. E. Larson  
Director

CEL:AFR:bg

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