

November 8, 1943

Page 2

The following services were requested for the cells:

1. Drainage.
 - (a) Active process
 - (b) Non-active process
2. Water supply.
 - (a) Distilled
 - (b) Filtered
3. Electricity.
 - (a) A.C. 440 volt 3-phase
 - (b) A.C. 110 volt single phase
 - (c) D.C. 120 volt
 - (d) D.C. 12 volt
4. Compressed air 80 lbs.
5. Steam.
 - (a) 125#
 - (b) 40#
 - (c) 5#
6. Vacuum.

It was agreed that C. D. Coryell would secure data on the possibility of installing periscopes as a substitute for glass water chambers for observing the interior of the cells.

W. D. Webb

W. D. Webb

WDW/maw



A. H. Compton

C.D.Coryell, H.A.Levy, W.E.Cohn

The Hot Laboratory (Building 706C): Survey of Purpose and Construction Requirements

The following is a summary of the factors entering into the construction of the Hot Laboratory (706C). We cover briefly the unique character of the laboratory operations and the problems which can be attacked adequately only with materials produced in this type of laboratory. A plan embodying the minimum facilities required accompanies this memorandum. Features involving unnecessarily large expense have been eliminated, and an analysis of the requirements discloses nothing that should impede rapid completion of design and early completion of construction.

More detailed discussion of the needs and purposes of this type of laboratory is to be found in memoranda and plans listed at the end of this communication.

The purpose of the laboratory is to prepare pile products of high purity and specific activity from pile materials too radioactive (1 - 20 curies) for handling without heavy permanent shielding. The chemical operations must therefore be done by remote control. These operations include steps common to the laboratory rather than those common to the semi-works and separations plant in respect to:

- a) small size of equipment (chemical glassware),
- b) small amounts and high purity of materials,
- c) design for wide variety of unit operations, and
- d) high precision of control (including visual observation of all steps).

The materials prepared in this laboratory are urgently needed for the following work of the Clinton and Chicago programs pointing toward: W Operations.

I. Process Development Studies

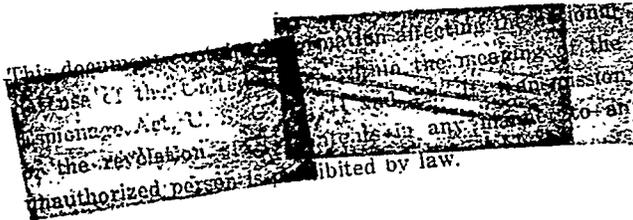
- a) Massive tracer preparations essential for extended decontamination studies on laboratory and semi-works scale.
- b) Characterization of hitherto unidentified fission products of very long life or of relatively low yield, important in achieving 10⁷-fold decontamination at W.

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

David R. Hamon 11/31/45
 Technical Information Officer
 ORNL Site

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DATE 1-5-51
 FOR THE ATOMIC ENERGY COMMISSION
Wilbur G. Strawn
 CHIEF DEPT.



- c) Attainment of W concentrations of the product and the fission products for process studies, particularly of the stability of operating conditions and materials towards intense radiations.
- d) Accumulation of stocks of pure fission material for the development of rapid analytical methods on process materials.

2. Study of Biological Hazards to determine:

- a) the toxicity of fission products taken into the bodies of animals and humans.
- b) The permissible pollution of public water supplies, fish breeding grounds, and agricultural areas by waste solutions from plant operation.
- c) The toxicity of radioactive dusts and gases resulting from plant operation, waste disposal and leakage, coating failure, explosion, or enemy action.
- d) The character of biological damage resulting from fission product poisoning.
(The requirements for this work are discussed in Memos 1 and 2.)

3. Fundamental Physical Studies

- a) Complete characterization of fission product & radiations.
- b) Study of other radioactive isotopes that can be produced by pile neutrons.
- c) The relation of biological damage to the chemical character of the radioactive source.
- d) Work involving strong monoenergetic & sources.
- e) Highly active & sources for spectrographic work.
(See also Memo 3.)

The minimum essential features of the laboratory are embodied in the accompanying plan and include:

1. Two banks of four interrelated shielded cells each. Three of the four cells are 4' x 6' x 18' and one 4' x 6' x 12'. Details of the cells have been described in a letter from Coryell and Levy to Whitaker, dated Oct. 21, 1943 (Memo 3). The cells have walls and ceilings of concrete two feet thick (adequate for 10 curies of 2 Mev & radiation in one spot). The walls are pierced for control observation, manipulation, and interconnection. A small passage with a lead door provides accessibility for installation of equipment. The size, shape, and facilities have been designed for specific chemical operations. All cells are ventilated by forced draft; one cell in each bank, designed for other work, is provided with cooling of intake air.

2. Four cubicles, shielded for semi-hot work (up to 1 C), in which operations are carried out after separation of specific elements from the main mass in the bank. These are essentially very small laboratories for one man, with 6" concrete partitions, where table shielding will be used.
3. Small counting room with 2' concrete walls and ceiling, and air conditioning to protect the counters.
4. Chemical laboratory for the preparation of highly purified reagents used in the bank separation processes, and for the preparation of samples for counting.
5. Work room for constructing the apparatus and remote control devices used in the cells. These are principally specially designed glass vessels and hand-tailored controls.
6. Facilities for operation and personnel: office, small utility room, change room, and lavatories.

The construction of the laboratory involves no new problems on the plantsite. The features embodied above are those of a normal chemical laboratory with the addition of large concrete hoods. General facilities need not be as elaborate as those in 706A.

Stainless steel is not required for the hot drains because the small volumes of active waste will be no more corrosive than ordinary chemical wastes. A small holding tank (~500 gal.) must be provided for monitoring, so that occasional hot discharge solutions can be sent to the main storage tanks.

Space for difficultly obtainable air conditioning equipment for the counter room and other cells is provided and installation may be made by Maintenance whenever the equipment arrives.

Much of the accessory equipment such as glassware, chemicals, and counting instruments is on hand, and the rest can be obtained through the Clinton Procurement Office. All remote control equipment and items such as periscopes will be designed and constructed through the Research Division in time for operation at the completion of the construction.

From the above considerations we see no reason why the laboratory building cannot be constructed in two months, as originally estimated by the Clinton Administration. Delay in completion beyond March 1 at the latest will defeat in large part the purpose of obtaining important information for W operations.

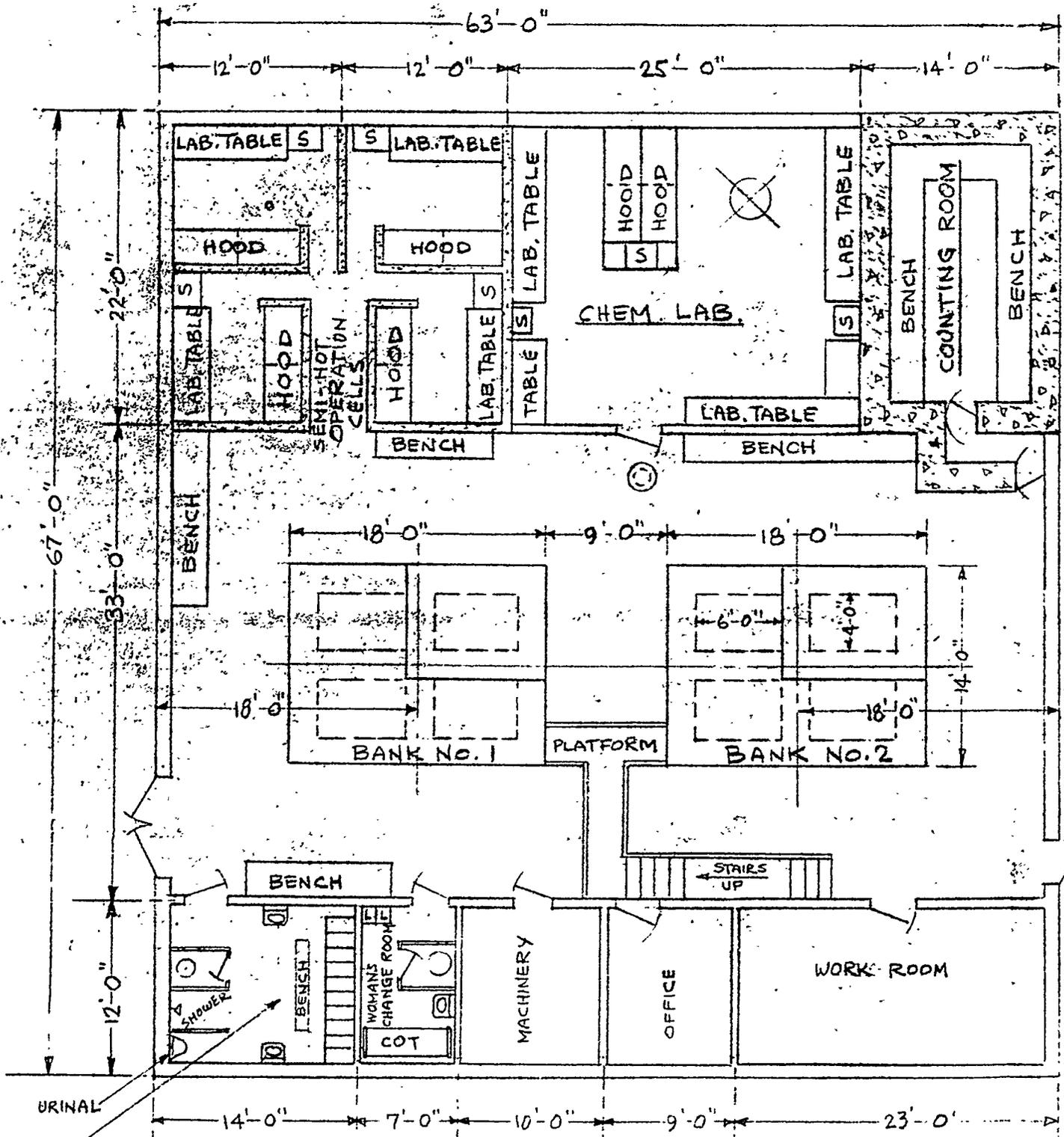


The personnel of the laboratory will be largely drawn from members of the Chemical and Biological Sections now involved on the same type of program with much weaker sources. The Hot Laboratory will increase tremendously the productivity of the effort and the scope of the investigations of the above groups. Other groups are completely dependent on these two Sections for the radioactive materials for their research programs.

Previous important communications on the laboratory are given below:

- | | | | | |
|-----------------------------|--------------|--|---------------------|---|
| (1) Memo | MUC-KSC-13 | W.E.Cohn to K.S.Cole (Chicago)
Extraction Facilities at Clinton | 9/7/43 |) |
| (2) Memo | | W.E.Cohn to H.J.Curtis (Clinton)
By-Product Separation Laboratory | 10/20/43 |) |
| (3) Memo & Preliminary Plan | | C.D.Coryell & E.A.Levy to M.D.
Whitaker (Clinton) The Hot Labor-
atory for the Separation of
Fission Products | 10/21/43 |) |
| | CL 706A - 23 | | | |
| (4) Plans | CL-706C-1 | Refer by date to Drawings of
W.D.Webb and W.Taluc | 10/30/43
et seq. | |



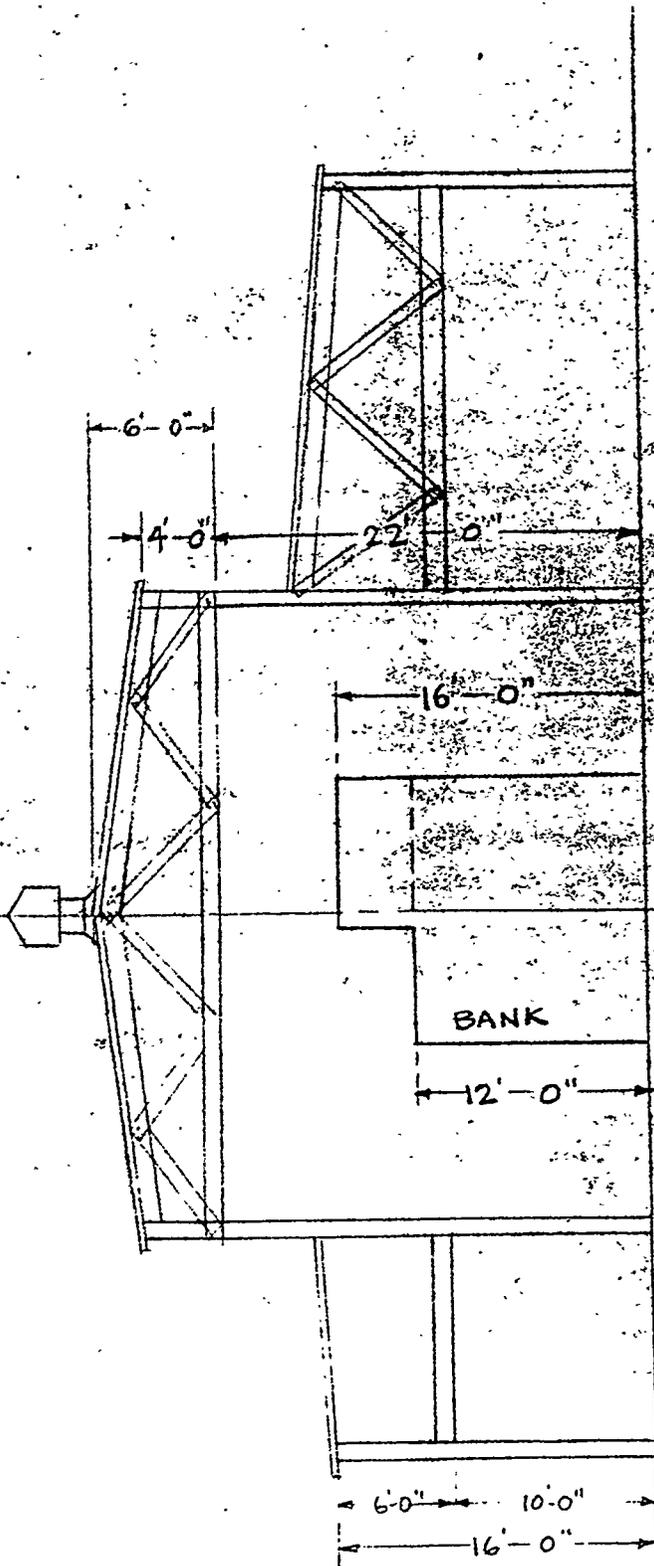


MENS CHANGE ROOM
10 DOUBLE-DECK LOCKERS

⊗ = OVERHEAD SERVICES WITH FLOOR DRAIN BELOW.

⊙ = SAFETY SHOWER WITH FLOOR DRAIN BELOW.

HOT LABORATORY

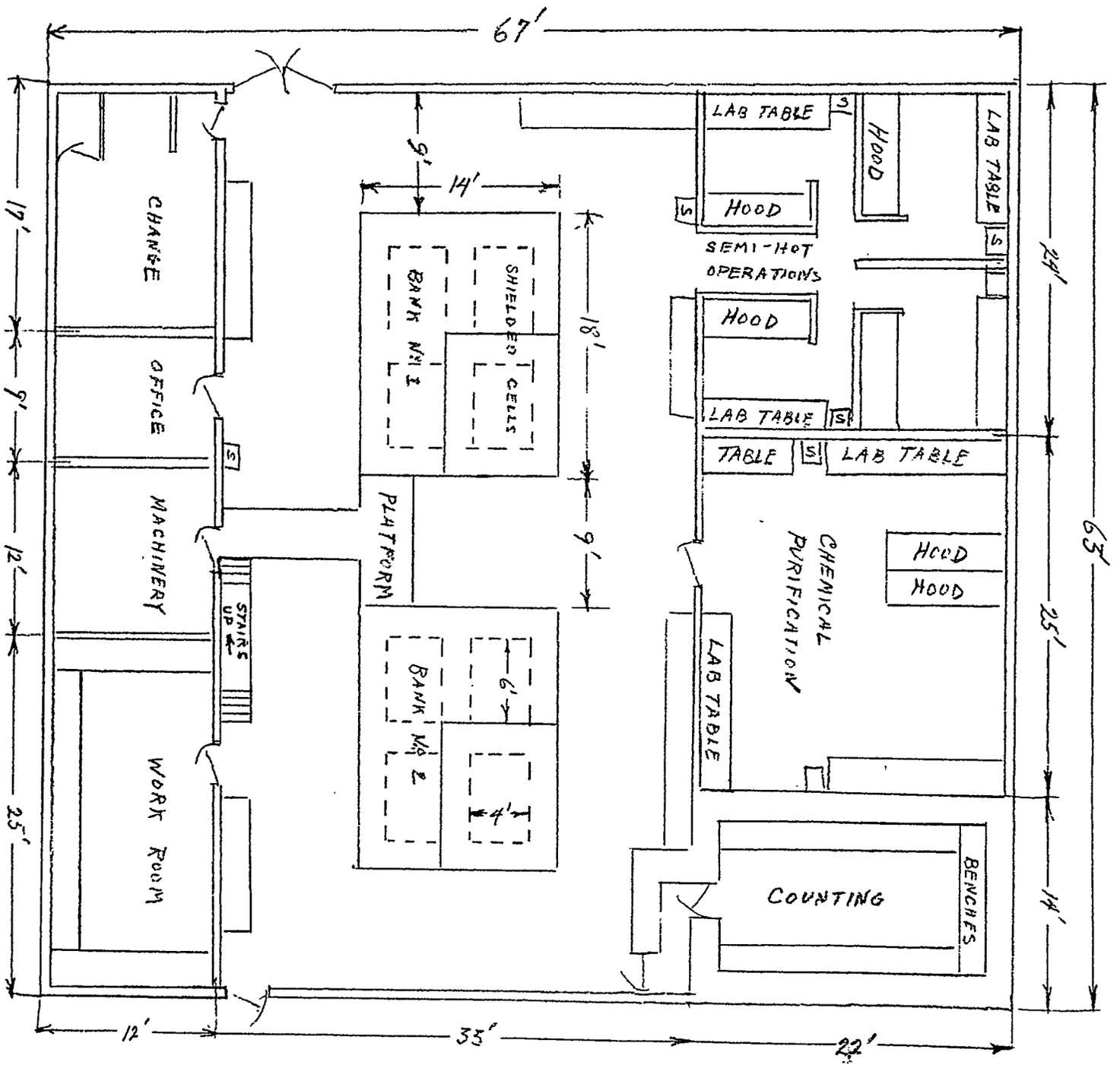


TYPICAL CROSS SECTION

CLINTON LABORATORIES

REV. BY DATE	REF. DRAW.	DATE 11/23/43
		SCALE 1" = 10'-0"
		DRAWN BY W.T.
		APPROVED
		PROJECT No. 9733
		CL-706-C-1

LABORATORY - BLDG. #706-C



HOT LABORATORY
 SCALE: 1" = 10' 0"
 & BANTS
 HAL
 11/12/43

DON'T SAY IT — WRITE IT

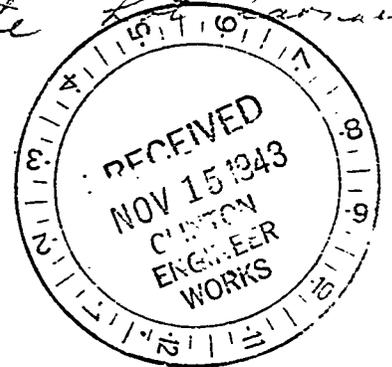
TO M. D. Whitaker

DATE Nov 15, 1943

FROM H. A. Lewis

In re: Hot Laboratory

Dr. Coryell asked me to furnish you with these two alternative plans for the Hot Laboratory.



ELEV. 805

36'-6"

E-31, 113
ELEV. 801

E 31, 145

*Omission of this section
is being considered.*

FLOOR LINE ELEVATION

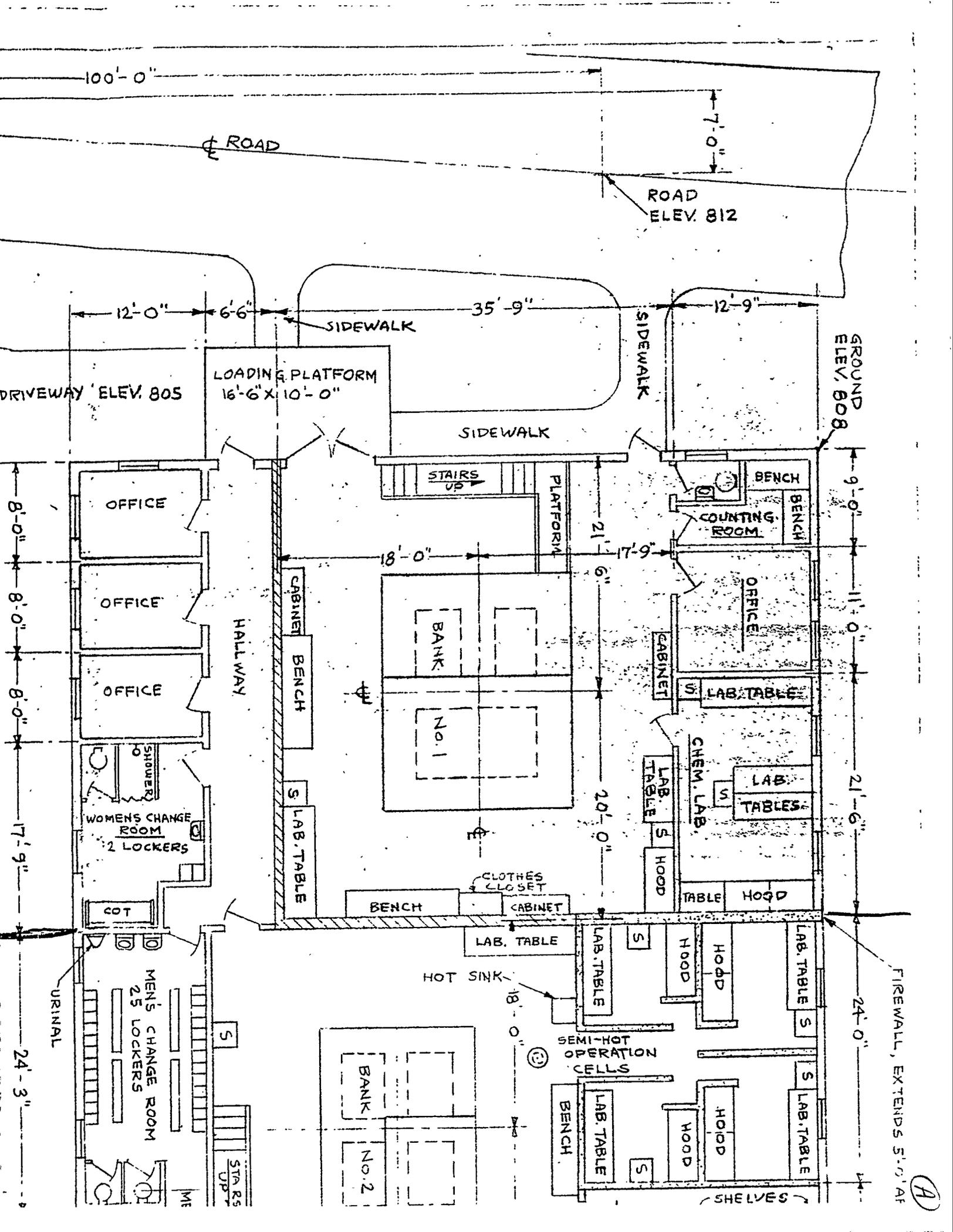


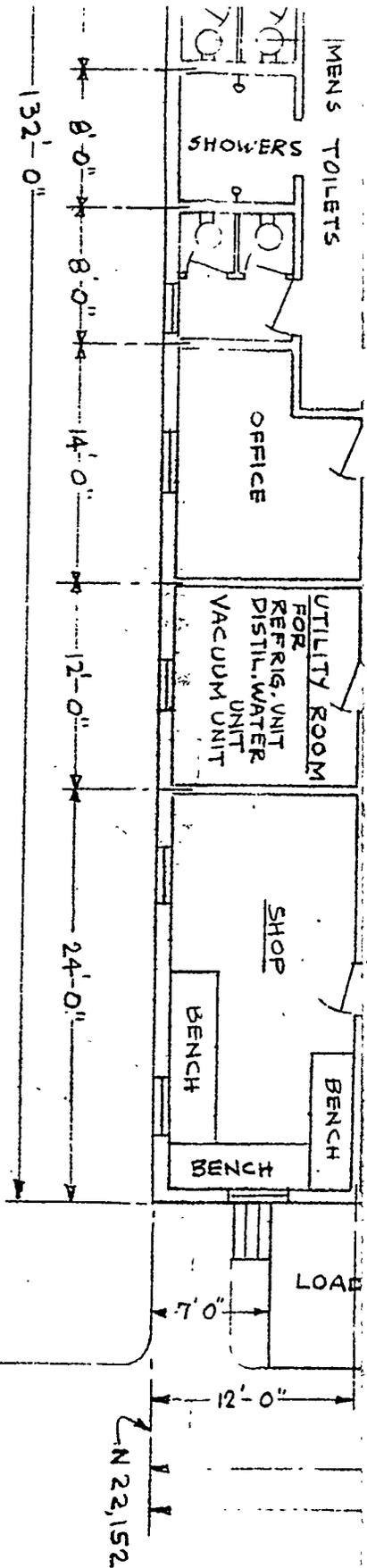
= OVERHEAD SERVICES WITH



= SAFETY SHOWER WITH FLC

ROAD





N 808

1TH FLOOR DRAIN BELOW.

FLOOR DRAIN BELOW.

PLAN - HOT LABORATORY - BLDG. #706-C

N 22,100
ELEV. 801

CLINTON LABORATORIES

REVISIONS		REV. BY DATE	REFERENCE DRAWINGS	DATE
1			W66038 W69037	11/13/43

W66038
W69037

DATE 11/13/43
SCALE 1" = 10'-0"

DRAWN BY W. TALUCI

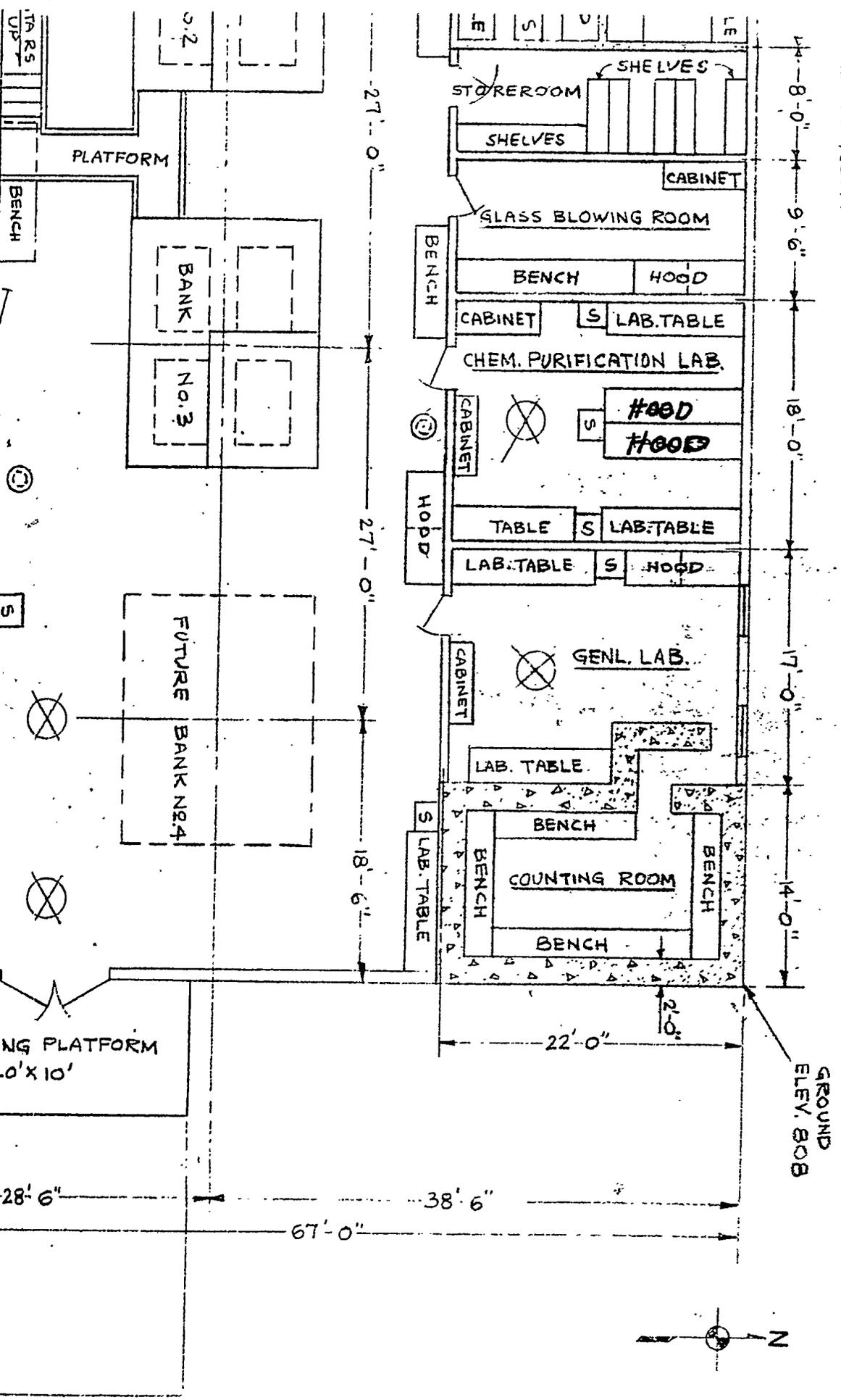
APPROVED

PROJ. No. 9733

CL-706-C-1

(A)

6' ABOVE ROOF





This document consists of 2
pages and 0 figures.
No. 2 of 9 copies, Series A

4/14/44

- 1. W. Johnson
- 2. R.L. Dean
- 3. H.D. Whitaker
- 4. Maj. Greagor
- 5. H.M. Parker
- 6. R.S. Stne
- 7. S.T. Cantril
- 8. Readers File
- 9. Central File

Warren Johnson

DECLASSIFICATION CANCELLED

S. T. Cantril

DATE OCT 15 1964

Medical.

706-C

The United States Commission

RSD

~~SECRET~~

Declassification Branch

COPY

The purpose of 706C is to provide safe working conditions for highly radioactive materials. I am concerned that the work in 706C be inaugurated and maintained at acceptable levels of safety. Thus far the "christening" of the building has not followed the original intent of the laboratory.

I have never felt that the Medical Department and Health-Physics Section should act in the role of policemen enforcing safety and precautions. We are responsible for making our independent surveys and checks for unsafe practices. The primary responsibility is placed on Supervision to be aware of the conditions under which work is proceeding and to be the first to detect and rectify those conditions when necessary. We are ready to cooperate in any way we can or are requested to do so.

My aim at this time is only to emphasize that 706C in my opinion can become a major hazard unless detailed care is taken in keeping conditions within safe limits. The condition of Room 703, 706A was ample evidence that a set of rules, even though made up by the men who are to do the work in that room, is not enough to insure safety. Practically all of the rules were apparently forgotten as soon as they were written. Surveys thus far made in 706C, copies of which are attached, indicate that far more care is needed than is being given to technique planning and housekeeping. It is not my intention to single out the men mentioned in these surveys. This group has received our recommendations and acted upon them accordingly. The surveys indicate that with the level of attention which has in the past been demonstrated by groups in 706A, who will be working in 706C, I have no optimism in feeling that concrete walls and remote control, lead bricks, etc. will keep the men away from radiation, their hands and gloves free from contamination, floors, benches, etc. free from contamination, unless renewed emphasis and fore-thought are placed on precautions. No end of concrete, lead, mopping, linoleum or other inanimate objects will function without a full appreciation on the part of the men that the limitations of protection are only as good as they wish to make them.

I frankly admit that thus far we have failed to get our point across with various men, and I think that Supervision can do so with more effect. I realize that the proposed program for 706C entails very high activity and that certain men who have been delegated to do this

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work are giving a great deal of attention to protection problems but I do feel that if work proceeds in 706G without some reorientation of the principles involved in radiation protection that we can expect activities which will either interfere with or completely stop the proposed program.

C
O
P
Y

STC/w

S. T. Cantril, M.D.

~~CONFIDENTIAL~~

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No. 2 of 5 copies. Series A.

~~SECRET~~

- 1. Levy
- 2. Curtis
- 3. Cohn
- 4. Central File
- 5. Reading File

April 20, 1944.

Henri A. Levy

Chemistry

W. E. Cohn

Biology

C
O
P
Y

In reply to your request for information on our plans for work in 706-C, the following estimates have been made. These are our maximum probable needs as visualized at this time, and should not be expected to hold too literally nor to limit the usual and routine upsets in plans indigenous to this type of work.

Our major purpose at Clinton, for which 706-C was originally conceived, is the preparation of large amounts (0.1-10 C) of carrier-free, impurity-free fission products and related activities, singly or in mixtures. The maximum needs for these may be estimated as follows:

- a. Mixed F.P.s (super-juice) 2 slugs per month (ca. 1.0 C each of long-lived material).
- b. Sr, Ru, Zr, Ba (in order of current demand): 2 slugs per month (ca. 0.005-0.2 C each).
- c. p32, p131, etc. (by neutron irradiation of S, Te, etc.): not over 1 preparation per month (0.01-1.0 C per run).

The facilities required for each of these are:

- a. Dissolver, extractor, ammonium nitrate remover, concentrator in cell banks. No semi-hot lab work.
- b. Dissolver, extractor, precipitation and filtration devices in cell banks; semi-hot lab space for final purifications.
- c. Semi-hot lab space only.

The time and manpower required per operation, as a guess, is:

- a. 1 week, 2 men, per slug.
- b. 2 weeks, 2 men, per slug.
- c. Very indefinite: probably 1/2 week, 1 man, per preparation.

This program, with its attendant analyses and research into new or improved methods (e.g., columns, electrochemical separations, etc.) will utilize all of our present manpower. Because of the over-lapping of parts of our programs with those of others, and the joint responsibility of all of us in maintenance,

100-50000-1

5-5-52

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4-20-44

construction and operation, it is not at all necessary that all parts of our particular preparations be carried out by members of this group. This will be required only where our particular needs deviate from those of the other groups concerned. The above manpower requirements, therefore, represent approximately what we visualize our ratio of manpower in: product out to be.

C
O
P
Y

WEC:mc

W. E. Cohn

~~SECRET~~

10-3088
10-3088
10-3088
10-3088

YH
SLUG #1First Hot Run, 5-13-44

Present: Brady, Goldring, Coryell, Levy; also Badger (for Health).

~ 12:15 P.M. Two hot slugs delivered to 706-C from canal.

5:45 P.M. Slug cart rolled in. Dolly rolled into cell with Coryell and Parker pulling lines, Goldring directing, Brady pushing, Levy on top of cell to remove bucket cover, etc., and Badger taking activity readings.

6:05 P.M. Slug placed in platinum basket.

6:10 Pt basket gripped and listed.

6:13 Coating remover cover removed at 6:13.

6:15 Cover put in proper place.

6:20 Basket in coat dissolver.

6:21 Tongs released.

6:23 Coat dissolver cover on. At this time activity at lower of top periscopes 4.5 mr/hr, at upper 6 mr/hr, at bottom of over-all viewer 100 mr/hr, just below bottom of viewer hole 12.5 mr/hr.

6:40 Dolly removed. With door open activity = ~ 60 mr/hr at edge of door and approx. 1 ft. above bottom of door. Six inches lower activity = 100 mr/hr.

6:30 Base (300 ml 6 N NaOH) added to coating dissolver. Reaction seemed fairly slow and even.

7:30 Reaction mixture was very dark colored and just a few bubbles were coming out.

7:40 Water run in to fill siphon and drain coating dissolver since bubbling had practically ceased.

7:42 More base added (300 ml 6 N NaOH).

7:48 Coat dissolver again drained.

7:52 Coating remover cap rinsed.

CLASSIFICATION CANCELLED

DATE OCT 17 1965

For The Atomic Energy Commission

H. R. Caswell
Chief, Declassification Branch

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

David R. Hamlin 3/10/95
Technical Information Officer Date
ORNL Site

~~SECRET~~

7:55 - Coating dissolver rinsed with four portions of 500 ml each
8:04 of water followed by another rinsing of 500 ml of 10% HNO₃
(2.3 M).

8:06 Rinsed again with 500 ml water.

8:08 Rinsed again with water through cap.

8:09 Cap removed.

8:10 Rinsed some more with an indeterminate amount of water.

Considerable difficulty was encountered in removing the basket from the coat dissolver. The basket was in the container leaning in such a position that the tongs could not reach the opening. It was necessary to insert a long rod through the viewer opening and hook around the tongs to help pull them into position.

9:15 The basket was hooked with one prong through a hole and the other resting against metal. After jiggling around a while it was decided to carry the basket in that position.

9:28 The basket was safely deposited in the dissolving flask.

9:30 The top was put on.

Air under sintered glass adjusted to 1.5 lb. With slug in dissolver flask activities were above viewer tank > 100 mr/hr, center of tank 80 mr/hr, hole below tank 70, 3 inches below hole 20 mr/hr, top periscope 5.5 mr/hr, next periscope 4, through window 5 - 5.5 mr/hr.

9:37 610 ml 70% HNO₃ added. Temp. 26°. Variac for heater set at 96.

9:41 Temp. = 42°.

9:42 Pressure = 1 1/4 lb., Temp. = 45°.

With door open, activity at edge of concrete and middle of door = 32 mr/hr. Near floor 6 inches away from concrete, activity = 80 mr/hr.

9:45 Variac shut off, temp. = 57°.

Activity just below bottom of door a few inches from concrete = 160 mr/hr.

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3

9:51 Temp. = 66°C.
9:47 Variac back on about 9:47.
9:53 Variac off when temp. reached 70° at 9:53.
9:55 Temp. = 76°C.
9:56 Temp. = 77°. Lots of fumes coming off.
10:01 Variac back on.
10:03 Temp. 76°.
10:06 Temp. 87°.
10:09 Temp. 96°, Variac shut off.
10:11 Temp. 100°.
10:13 Variac back on.
10:16 Variac off.
10:18 400 ml acid added. Temp. 85°, Variac back on at 96°.
10:20 Heater under sinter turned on to 2 amp.

Pressure = 1 3/8 lbs.
Variac to 85°

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4

Personnel: Nicholson, Levy. 5-14-44
10:27 A.M. 10:27 A.M.
Solution boiling quietly. Light fume discharge. Added 800 ml HNO₃. Temp. to 90°C. Air pressure 1.5 lbs.
10:29 A.M. Heavier fumes. Total volume 1750 cc.
12:31 P.M. Quiet boiling. Temp. 112°C.
1:10 Unchanged. Temp. 112°C.
1:10 - Background count on hand counter 152 x 64 + 11 in 47 min.
1:57 ~ 207 c/m.
2:15 Unchanged. Temp. 112°C.

5-15-44

Personnel: Goldring
Temp. 113°C. Vol. 1700 ml. Air Press. 1.5 lbs. Variac 70.
Added 400 ml H₂O. Vol. 2050 ml.
10:30 A.M. Sample taken.
Approx. 4 ml sample was taken. Direct reading radiation meter went off scale at 100 mr/hr at a distance of ca. 8 in.
11:30 Temp. 100°C. Var. 75. Added 150 ml HCOOH in ca 10 ml portions. By accident 20 ml were added once and the reaction became very violent.
11:40 The HCOOH was followed by about 10 ml of H₂O to clean out the line.
12:40 Soln clear, no NO₂ visible. Temp. 108°C, Variac 75.
1:05 P.M. Sample of 2 ml taken. Sample gave reading of 50 mr/hr at distance of one (1) foot. Sample gave 200 mr/hr at approx. 4 inches.

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5

GROSS BETA COUNT

G. Strickland

Mounted 0.10 ml of a 1:10,000 dilution.

Count = 7262 c/m

∴ Gross activity is 7.3×10^8 c/m/ml.

$$= \frac{7.26 \times 10^8 \text{ ml}}{0.0375 \times 60 \times 3.7 \times 10^7 \text{ mc.}}$$

= 8.7 mc/ml.

∴ Gross beta activity is approx. 17.4 curies (± 10%)

GROSS GAMMA COUNT

(E.L.N., L.S.G.,
E.K.H.)

Mounted 0.075 ml of a 1:100 dil. (1:1333).

Counting Efficiency: 1%

Geometry ~~(1:2860)~~ : 3.5%

(1:2860)
Count is 2450 c/m.

Gross count $2.45 \times 10^3 \times 1.33 \times 2.86 \times 10^6$

= 9.35×10^9 c/m/ml

$$\text{Activity } \frac{9.35 \times 10^9 \text{ c/m/ml}}{2.22 \times 10^9 \text{ c/m/ml}} = 4.2 \text{ mc/ml}$$

∴ Gross gamma activity is approx. 8.4 curies (± 10%).

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Dilution + liquid from storage bottle raised volume in buret to 2400 cm³ from 2000 cm³ approx.

NOTE

Need air inlet to vac line on buret to replace liquid drained from buret.

25 cm³ through cow into storage b to rinse lines. Cow to drain.

Rinsed flask 10 ml dist. H₂O out through aspirator tube.

450 cm³ conc. HNO₃. Variac on 85.

Flask and burette rinsed with sat. oxalic acid solution.

5-19-44

4:00 P.M. Ion chamber readings. Chamber through westernmost 2" hole.

Calibration: 10" Ω resistor - 4 volts = 170 mr/hr (?).

<u>Position of Chamber</u>	<u>Resistor</u>	<u>Volts</u>
1. Barely through ceiling	10" Ω	3.7
2. 6" out	"	10.0
3. Entire chamber out	"	23.0
4. Bottom 6' from floor (opposite condenser)	"	41.0 (37.5)
5. Bottom 5'2" from floor (opposite flask)	"	7100.
6. Bottom 3'6" from floor (opposite burette)	10 ⁸ Ω 10"	0.08 (zero off) 51.0 (47.0)
7. Bottom 1' from floor (opposite cow)	"	9.0
8. Bottom 4 1/2' from floor (opposite sinter)	10 ⁸	0.10 (zero off)

5-20-44

Chamber readings after various treatments

(5-22-44)

Same as before	After 2nd oxalic acid rinse	After NH ₄ HF ₂ rinse	After Pt basket removed
Ω Volts	Ω Volts	Ω Volts	Ω Volts

10" 4.9	10" 75	10" 22	10" 9	10" 4.0
10" 0.66				3.5
10" 0.62	10" 43	10" 23	10" 7.9	10" 1.8

~~SECRET~~

7

4:30 P.M. Hot 6 N HCl put in flask and burette - to stand over weekend.

5-22-44

More chamber readings.

After 2nd bifluoride rinse

4. Opposite flask	10"	2.0, 2.5
5. Opposite buret	10"	0.49

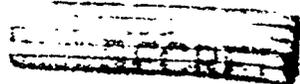
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334

CLASSIFICATION CANCELLED

DATE DEC 4 1963



REC. 37 0
No 207

For The Atomic Energy Commission

July 27, 1944

M. S. Smith

M. C. Leverett

H. A. Coryell
Chief, Declassification Branch

*Info: ...
...
7-27-44*

Emergency Project for Recovery of Product from 706C

This memorandum confirms our conversation of July 26 on the subject named.

At a conference with you attended by Mr. Coryell and Mr. McCullough, it was requested that the installation of a valve pit, tank, and auxiliaries be made at the west end of Building 706C for the purpose of holding the active waste material from this building. The reason for this installation is that it will make possible the recovery of several grams of product which might otherwise be run to waste. It is requested that this be carried on an "Emergency Project" basis. It was pointed out that the valve pit must be physically ready to use no later than Wednesday, August 2, 1944.

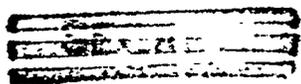
The valve pit will contain two valves both stainless steel and both capable of being operated from outside the pit. Presumably, extension handles will be provided on these valves which will run through shielding covering the pit to the surface. I would recommend the equivalent of not less than 3' of concrete shielding over the valves and lines in the valve pit. As requested by Mr. Coryell, an alarm system should be installed in connection with these valves so that it will be impossible to run waste into the line with both valves closed thus leading to backing up of hot wastes in the 706C Building. Some indicator also should be provided by means of which users of hot drains in the 706C Building will be automatically warned when the valve setting is such that waste is being diverted to the proposed storage tank. This is necessary because only the hot metal wastes from 706C should be run into this tank. The valve pit should be provided with a sump and provisions for draining or jetting out any liquid which may accumulate in the sump. It should also be so arranged so that it can be hosed down if necessary.

The tank should be equipped with the devices noted in our previous memorandum on this subject for measuring liquid level density, agitation, etc. It will be desirable to put the tank on an impervious footing and to equip it with a dry well in order to determine whether any leakage is occurring.

Other details of installation and construction can best be developed by personal contact. The tank should be ready for use in not later than three weeks from the present date.

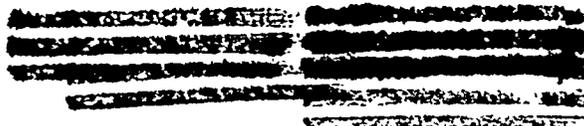
I hope you will feel free to call on us for any assistance in this matter. Mr. F. C. McCullough has been assigned the task of keeping in contact with progress on this job and of determining that it meets the needs of the 706C operators.

ap



M. C. Leverett

- 1 - Smith
- 2 - Schwertfeger
- 3 - Coryell
- 4 - Doan
- 5 - Leverett
- 6 - Reading File
- 7 - Central File



44-9-316



44-9-316
September 18, 1944

This document has been approved for release to the public by

David R. Hamm 9/18/45
Technical Information Officer Date
ORNL Site

Mr. J. R. Oppenheimer
Attention: Mr. R. W. Dodson

In re: Preparation, Composition, and Packaging
of Special Source

Dear Sirs:

We are transferring to Major E. J. Murphy for shipment the special source requested, containing 230 nominal curies as defined below.

The source consists of BaCl₂ in amount corresponding to less than 900 mg of cation (~750 mg) distributed in the lower part of the first stainless steel cone furnished by you. Analyses were made on three different microaliquots (5 λ from 50 ml) giving apparent values of 25, ~100, and 10 mg of Pb in the total sample. The first two values may be high due to Pb contamination of equipment; for the third new glassware was taken. The true value is thought to be between 10 and 25 mg.

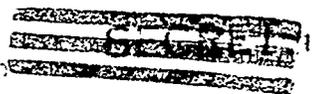
The radioactive Ba in this sample was obtained by processing 216 slugs which were exposed approximately 40 days in very favorable positions in the Clinton pile and cooled from 4-14 days (to the time of shipment). The last separation from La occurred at 1:09 A.M. C.W.T., September 18th. The chemical procedure is identical with that given you previously for the engineering scale operations and is different in only unimportant respects for the small scale operations (e.g. combinations of the batches in process to minimize use of Pb in Variation A). Variation B was not used. Two BaCl₂ separations were made.

No serious difficulty in the operation of any of the apparatus was encountered. Only the cell 1 glass apparatus was used. The only flaws noted in the cells were (a) a very small leakage which soon ceased at a Plicene seal between a glass and stainless steel line connecting cell 4 interim storage and cell 1 buret, (b) separation of a tygon cooling water line from spray trap condenser #1 preventing use of water cooling for this trap, (c) occasional sticking of the glass check valve below solenoid manifold, thereby slowing maximum filtration rate,

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DATE 9 18 1944
COMMISSION



and (d) plugging of sampler line to dissolver. Darkening of the glass in cell 1 was not serious; the most prominent occurrence was that in the lowest part of the reactor, the color being reddish-brown. The external glass filter of cell 4 was not changed during operations but rates were slow on the fifth batch of 36 slugs. We consider that the apparatus could be used again without modification.

We met with an unexpected problem in making Ba assay of solutions of tremendous specific activity (of the order one active atom in 300). After dilutions by factors of 10^7 - 10^8 , analyses were erratic and often quite low. It was found necessary to make all dilutions with 0.2 N HNO_3 to get reproducible and satisfactory results. The use of more acid together with Ba carrier at each step does not seem to raise the analytic values further.

Our assay is given in terms of a nominal curie, namely 2.2×10^{12} Ba beta counts per minute at 100% geometry (determined from UX_2 counting of thin samples). The Ba is precipitated three times as chloride, and counting extrapolated to separation time. We do not correct for the absorption inherent in approximately 30 mg of $BaCl_2 \cdot H_2O$ precipitate spread over an area of 2.5 cm^2 , plus that in 3.5 mg of cellophane, 4 mg of air, and 3 mg of mica, nor do we correct for scattering from the Al-lined California-style Pb housing (see section 3 of CC-529). This curie unit could be 20-30% larger than the theoretical curie, and our assays are low by the corresponding amount.

Due to the persistence of analytical error up to the last day more metal was processed than the minimum necessary to meet the request. The well established assay of our material at the beginning of the purification and concentration stages was 285 at nominal curie calculated for separation time. Returns have not yet come in on all analyses but we have good reason to believe that the assay of the material sent shows 230[±]30 nominal curie of Ba at 1:09 A.M. September 18th. Further data will be forwarded at a later date. We would appreciate any information you can give us with regard to the true activity of the Ba, and to correlation with hard gamma standards.

The stainless steel cone rests without gasket in the cone holder you furnished in June. This in turn rests in block 2 of the shield arrangement designed by Leverett providing 6" on the side and 4" on the bottom of shielding of Pb plus steel lining. The top is covered by block 3 which provides 9" of protection. This block has affixed to its shoulder by rubber cement a ring of 1/4" Koroseal which fits fairly well the cone and holder. The vent is not plugged. Detailed plans of the arrangement except for the gasket were given Helmholtz in June.

The sample was examined by periscope immediately after evaporation of the aqueous solution and appears to be fairly evenly distributed in three bands. The flat bottom is shiny. The lowest band is fairly broad and the two are narrower, the top being approximately half way up. The orange iron stains do not appear greatly increased in intensity those noted in tests made here. Collaboration with

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Dodson. The dribbling operation proceeded without untoward event and there is no evidence of appreciable splattering in the higher portion of the container. We are disappointed in the virtual absence of luminescent properties.

Hasty inspection for beta count indicated no appreciable surface contamination of the outside of blocks 2 and 3 assemblage. The side wall of block 2 gave at 7.5 hours after separation the high reading of 1800 mr/hr. The reading directly above the source outside the wooden shell was found to be 30 mr/hr. We request that you inform us what levels are observed at later times and at the time that block 2 is removed from the truck.

If it is needed we could send on loan one of our 3' scanning periscopes (design of G. S. Monk) with 30° angle of view with or without additional eyepiece of approximately 5-fold magnification.

Yours sincerely,

M. D. WHITAKER, DIRECTOR
CLINTON LABORATORIES

By Charles D. Coryell

Approved for Chemistry Division:

By Harrison S. Brown

CDC/b

Copies to:

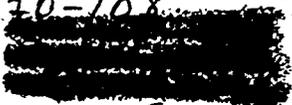
- 1,2. J.R. Oppenheimer *E.J. Murphy*
- 3,4,5. M.D. Whitaker
- 6. C.D. Coryell
- 7. H.S. Brown

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44-10-108



File

October 7, 1944

Jf



Mr. M. D. Whitaker:

RADIO LANTHANUM PRODUCTION

I would like to call to your attention the importance of an early decision with regard to the future production of radio lanthanum. As you know, the present equipment in Bldg. 706-C was designed for at most only a few preparations, whereas the prospect now seems to be that several more may be required. We do not know, of course, how many preparations can actually be made in the glass equipment now available, but owing to the temporary nature of the setup, each additional job increases the probability that breakdowns and delays will be encountered. It is hoped that it will be possible to make three to five more deliveries without major alterations of equipment or time schedules. However, any plans to go into 1945 with the present setup must be classed as highly speculative.

If the requirement for active lanthanum is going to continue beyond the next few months, it is essential to start now to make definite plans for such new construction and equipment installation as may be necessary. It does not seem very feasible to try to install the more permanent equipment in the 706-C Building for the following reasons: (1) The building was not designed to handle anything like the activities involved and consequently the shielding provided by the cell walls is entirely inadequate for the job under consideration; (2) The space inside the cells is too small for the proper arrangement of the necessary equipment; (3) As long as the lanthanum job continues to take up fifty percent of the facilities in the Hot Laboratory, many of the research functions for which the building was designed cannot be carried out.

Probably three months would be required to design and construct a building to house this job and to install the equipment. This means that if the facilities are to be ready by February 1, 1945, the design work must start in the very near future. Mr. Oppenheimer has indicated that he will advise us on January 1st whether additional deliveries are required. In view of the above considerations, this is not a satisfactory arrangement unless we are all prepared to accept the possibility of several weeks' or months' delay in deliveries when the present equipment can no longer be used.

CLASSIFICATION CANCELLED

DATE FEB 11 6 4

R. L. Doan

R. L. Doan

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

RLD/er

- cc: M. D. Whitaker
- Warren C. Johnson For The Atomic Energy Commission
- C. D. Corvell
- M. C. Leverett
- A. H. Compton
- Major Murphy
- R. L. Doan
- Reading File

Chief, Declassification Branch

Technical Information Officer 1/20/95

ORNL Site



Information affecting the national... the... region... to... by law.

CLINTON LABORATORIES

DATE October 27, 1944

TO W. C. Johnson

DEPARTMENT

FROM C. D. Coryell

DEPARTMENT

IN RE:

- 1. W.C. Johnson
- 2. H.S. Brown
- 3. G.E. Boyd
- 4. H.A. Levy
- 5. M.C. Leverett
- 6. C.D. Coryell
- 7. Central File
- 8. Reading File

CLASSIFICATION CANCELLED

DATE 3-2-51 FOR THE ATOMIC ENERGY COMMISSION

W.A. Strauss / H.C. DECLASSIFICATION BRANCH

As a result of discussions held yesterday with a view to optimum activation of the short and long term aspects of the barium job, five of us closely associated with it wish to make certain proposals to you with respect to the organization and coordination of the variegated groups necessarily involved. Those participating in the discussions primarily were G.E. Boyd, T.H. Davies, H.A. Levy, D.N. Hume, and C.D. Coryell.

As we see it there are at least a dozen different groups covering unit responsibility with respect to the successful maintenance of our present obligations in production and the successful accomplishment of our goal in planning, construction, and operation of the new 706-D unit scheduled to replace the present one. The organization problem is formidable since five divisions, Chemistry, Engineering Development, Analytical, Construction, and Health, are involved with complicated interrelationships within the parts. There is given in diagrammatic form with this memo an analysis due largely to Boyd. We of the present self-appointed provisional committee recommend that this committee be expanded to include the leaders of each of the units indicated in circles, and that this committee be the steering committee for work under Management-Top Supervision.

Besides the people indicated in the eleven circles, F.G. Rehm, D.N. Hume, T.H. Davies, G.E. Boyd, M.C. Leverett, D.H. Harris, H.S. Brown, and K.Z. Morgan, there are other people who also have considerable obligation and experience that might be appointed to the committee. This list would include yourself, McCullough, Ward, Brady, Bigler, Strickland, and probably others who come to mind.

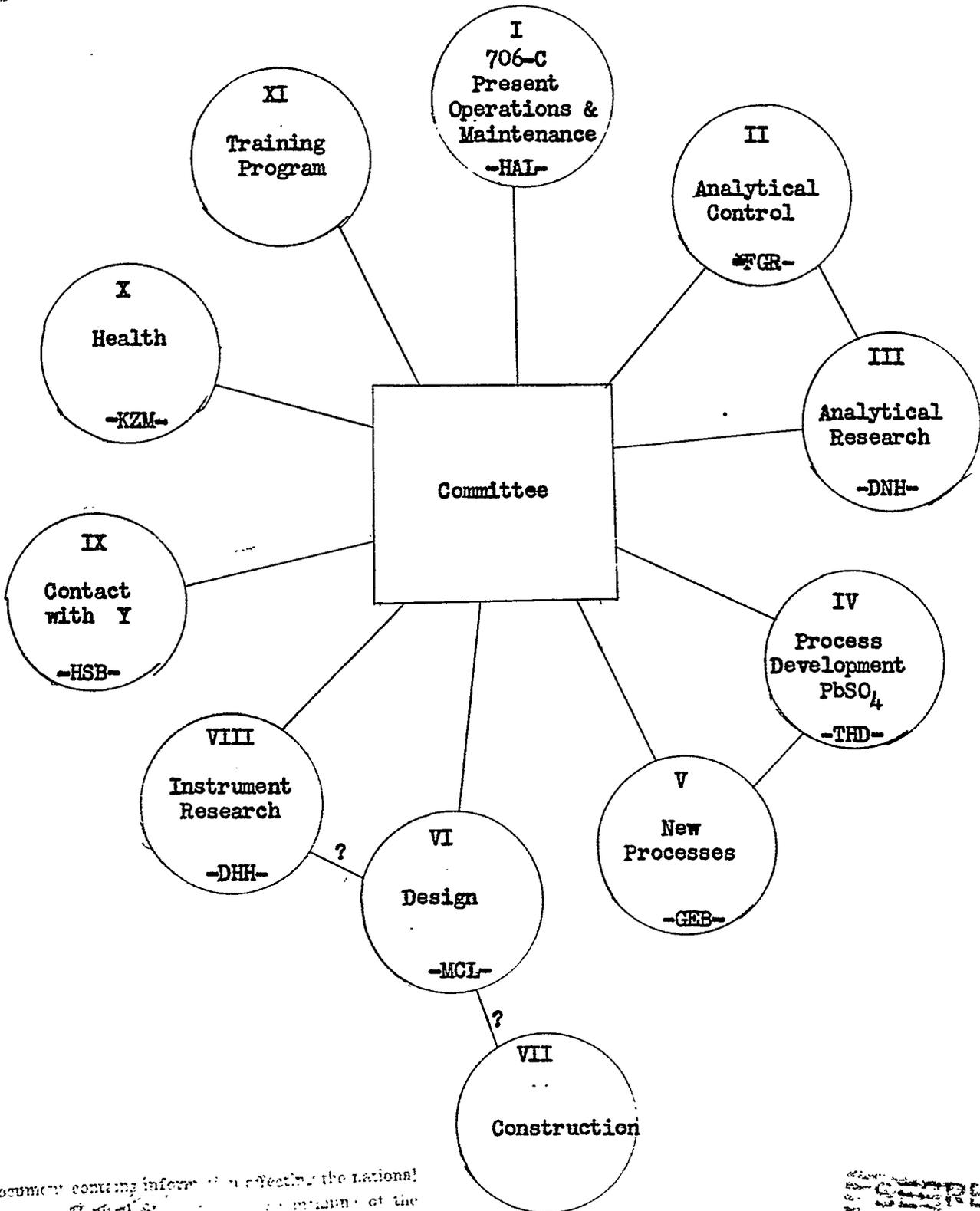
Certain other connections than the radial ones indicated in the figure may also be important such as a close bond between II and III, IV and V, and VI and VIII.

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C.D. Coryell

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10/28/44

- 1. K.Z.Morgan
- 2. J.E.Wirth
- 3. H.S.Brown
- 4. R.L.Doan
- 5. C.D.Coryell
- 6. W.E.Cohn
- 7. H.P.Gauvin
- 8. Central File
- 9. Reading File

K. Z. Morgan
H. P. Gauvin

SECOND HOT-RUN IN 706-C
10/9/44 to 10/17/44

The second Hot Run began on October 9, 1944. On this date 36 slugs were brought to 706-C from the canal in Building 105. These slugs were transferred in groups of 12. During these transfers a maximum reading of 150 mr/hr was obtained with a Lauritsen in contact with the bottom of the slug bucket. This reading was the largest of all readings obtained during the transfers that followed. After the 36 slugs were placed in the dissolver of Cell 3 a check of the outer walls of the cell indicated a maximum of 10/mr/hr at the slug inlet on top of the cell. Twenty-four slugs were brought down each day until October 13, 1944.

On October 10, a Victoreen survey of Cell 3 indicated a radiation level of 9 r/hr just over the shield in the cell. At the entrance to the cell, with the door open, the level was 80 mr/hr. The radiation level outside the cell was very low. A maximum of 15 mr/hr was obtained at the slug inlet on top of the cell.

A filter was removed from Cell 4 on October 11. This filter gave a reading of 10 r/hr at a distance of 3 feet. It was placed in a lead pot and brought to the storage area at the east of the building. During the removal of this filter the men worked for a few minutes in an area where the level was 4 r/hr. Some glassware fell on the floor in front of the cell and left spots which read 1.5 r/hr. These spots were washed down to 500 mr/hr. This area was posted until the radiation level fell to a safe value. On October 14, the level around the filter shield was again found to be 10 r/hr at 4 feet.

Fumes were found coming from the off-gas pipe of the storage tank at the west of the building. This pipe gave a reading of 200 mr/hr. at contact. Because of this, the area was posted.

A maximum of 300 mr/hr at 5 inches was observed during the sampling throughout the entire run. Film rings were worn whenever any danger of over-exposure was expected. These rings indicated total exposures of 230 to 630 mr over a period of three days. The higher readings were obtained by those working on the glass side. The highest reading obtained during sampling was 510 mr.

On October 17, the sample was removed from the cell and brought in the field west of the building. A maximum reading of 54 mr/hr was obtained at the distance of one meter from the pot. At contact with the pot the reading was 1.9 seconds for 50 divisions with the HP 13.

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DATE FEB 12 44
For The Atomic Energy Commission
H.P. Gauvin
Chief, Declassification Branch

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Before shipment the readings around the sample in the truck were as follows:

- At the wheels _____ less than 1 mr/hr
- At the sides of the container _____ less than 1 Mr/hr
- On top of the container _____ 25 mr/hr

The sample has been shipped and the process of cleaning is now in progress.

H. P. Gauvin

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44-10-107

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File _____

Those Eligible
To read the
Attached

Date 11-7-44
Subject HOT RUNS IN 706C

Copy # 3 *J. E. Wirth*

By J. E. Wirth
To M. D. Whitaker

Before reading the document, sign the date below

Name	Date	Name	Date
<i>J. E. Wirth</i>	<i>11/7/44</i>		

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

David R. Hamm 3/10/95
Technical Information Officer Date
ORNL Site

- 1. M. D. Whitaker
- 2. K. S. Stone
- 3. J. E. Wirth
- 4, 5 & 6. Central File
- 7. Readers File

This document consists of 6
 pages and 0 figures.
3 copies. Series A

11/7/44

M. D. Whitaker

J. E. Wirth

Medical

HOT RUNS IN 706C

~~SECRET~~ LIMITED

It is necessary to bring certain information to your attention regarding slight over-exposure (based on a tolerance of 100 mr/day) of personnel to radiation in the 706C Building during the recent hot runs. Analysis of the pocket meter records of the individuals concerned from 10/1/44 through 11/1/44 shows 11 persons with readings greater than 100 mr/day upon one or more occasions. (The readings being the lower one of two pocket meters for each day).

These records may be tabulated for convenience as follows:

Pocket Meter Readings > 100 mr/day from 10/1/44 - 11/1/44

<u>Number of Persons</u>	<u>Number of Days > 100 mr.</u>
3	1
2	2
3	3
1	4
1	5
1	10

Accumulated Weekly Readings

(By addition of daily pocket meter readings)

<u>400 to 500 mr.</u>		<u>700 to 800 mr.</u>	
<u>No. of persons</u>	<u>No. of times</u>	<u>No. of persons</u>	<u>No. of times</u>
2	1	3	1
1	2		
(None over 800 mr)			

Any Two Consecutive Weekly Periods

<u>800 to 1000 mr.</u>		<u>1000 to 1300 mr</u>	
<u>No. of persons</u>	<u>No. of times</u>	<u>No. of persons</u>	<u>No. of times</u>
2	1	2	1
(None over 1300 mr.)			

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Distribution Control
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 11/10/44

Pocket and badge meter readings show excellent correlation for weekly and two weekly periods and in many instances daily readings when the badges were read with sufficient frequency to give an adequate comparison. This may be noted when corresponding accumulated two week readings are tabulated for the seven individuals with the highest values.

Readings in mr. - Accumulated Total for Two Weeks

<u>Pocket Meter</u>	<u>Badge Meter</u>
405	355
675	575
715	580
975	885
985	850
1090	1095
1160	1255

Compact hand films were worn occasionally by persons whose hands were likely to get the greatest radiation exposure. The two highest daily values recorded were 530 mr and 510 mr on two different persons. The next highest reading was 220 in one day and this person also had a 120 mr reading and others less than 100 mr/day on alternate days for a total of 470 mr in 5 days. The only two others with high compact hand film readings are summarized as follows:

160 + 160 + 140 on alternate days for a total of 460 mr in 5 days
140 + 100 + (100 + 70) for a total of 410 mr in 3 days.

The basis for figures of tolerance to radiation exposure are in the main the following. In 1934 the International X-ray and Radium Protection Commission at the Fourth International Congress of Radiology, meeting in Zurich, put forward certain international recommendations for x-ray and radium protection.

The first of these reads as follows:

"1. The dangers of over-exposure to x-rays and radium can be avoided by the provision of adequate protection and suitable working conditions. It is the duty of those in charge of x-ray and radium departments to ensure such conditions for their personnel. The known effects to be guarded against are:

- (a) Injuries to the superficial tissues:
- (b) Derangements of internal organs and changes in the blood."

and for the first time gave the following on tolerance:

"The evidence available at present appears to suggest that under satisfactory working conditions a person in normal health can tolerate exposure to x-rays to an extent of about

0.2 international roentgen (r) per day. On the basis of continuous irradiation during a working day of seven hours, this figure corresponds to a dosage rate of 10^{-5} r per second. The protective values given in these recommendations are generally in harmony with this figure under average conditions. No similar tolerance dose is at present available in the case of radium gamma rays."

In 1937 the same International Commission meeting in Chicago put forward the following recommendations:

"1. The dangers of over-exposure to x-rays and radium can be avoided by the provision of adequate protection and suitable working conditions. It is the duty of those in charge of x-ray and radium departments to ensure such conditions for their personnel. The known effects to be guarded against are:

(a) Injuries to the superficial tissues;

(b) Changes in the blood and derangements of internal organs, particularly the generative organs."

Note change from 1934 rules by the addition of the above last 4 words.

"The evidence at present available appears to suggest that under satisfactory working conditions, a person in normal health can tolerate exposure to x-rays or radium gamma rays to an extent of about 0.2 international roentgen (r) per day, or 1 r per week. On the basis of continuous irradiation during a working day of seven hours, this figure corresponds to a tolerance dosage rate of 10^{-5} r per second. The protective values given in these recommendations are generally in harmony with this figure under average conditions."

Again note the addition of "or 1 r per week" to the 1934 rules. The 1937 recommendations also include gamma rays from radium. There have been no meetings of the International Commission since 1937.

An Advisory Committee on x-ray and Radium Protection was formed in the United States and through the medium of a handbook published by the National Bureau of Standards in 1931 (H-15) recommended a tolerance dose of 0.2 r/day. The next and, to my knowledge, last edition of the National Bureau of Standards Handbook on X-ray Protection (H-20) was published in 1936 and recommended 0.1 r/day as tolerance. In 1938 the National Bureau of Standards published a handbook (H-23 superceding H-18) on Radium Protection in which under recommendation 1.05 there is a note to the effect that "The tolerance gamma ray intensity derived from the chart is approximately 0.1 r per day". And again under storage recommendations in 3.04, "In any event, sufficient protection shall be provided to reduce the general body radiation to which a person may be exposed to 0.1 r per day for the person in question".

It may be seen therefore, that there are no standard rules and regulations but rather certain recommendations put out by the two bodies "The

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International X-ray and Radium Protection Commission of the International Congress of Radiology" and in the United States the "Advisory Committee on X-ray and Radium Protection." The latter group is composed of representatives from:

- (1) International Safety Committee and National Bureau of Standards for X-rays
- (2) Three Radiological Societies
- (3) X-ray equipment manufacturers,

and since July 1940 by a representative from

- (4) the National Cancer Institute of the United States Public Health Service.

These recommendations are subject to varying interpretations and at best represent in cautiously couched language the sum and average of many varied opinions. This is well described by Henshaw in a paper on "Biologic Significance of the Tolerance Dose in X-ray and Radium Protection" (Jr. N.C.I. June 1941)

Under the influence of the biologists and geneticists I even argued unsuccessfully as a member of the U. S. Advisory Committee on X-ray and Radium Protection, in its 1941 meeting, to lower the recommended tolerance level to 0.02 r/day and was appressed by the suggestion that the term "tolerance dose" be changed to "permissible dose". The latter term does not necessarily carry with it the impression that no effects would result as does the former term. This change in addition to others may appear in the next publication of the handbook.

Dr. Stone, in the second paragraph of a letter of May 15, 1943 to Mr. A.H. Compton, set a standard for the project by stating -

"Tolerance Dose. We have adopted the tolerance as set forth by the Bureau of Standards, namely 0.1 r/day. We have concluded that the intensity of radiation (the number of r per second) building up to the daily tolerance exposure is immaterial. The 0.1 r per day can be received in any manner whatever, provided it is not repeated within a 24 hour period."

Dr. Cantril adds his interpretation in the Project Handbook XII-B 1.3(b) second paragraph -

"The total duration factor can best be illustrated when we speak of the 'tolerance dose' by which we mean the amount of radiation to which a normal person can be exposed day in and day out without sustaining permanent damage. ..."

The recommendations of the two Committees referred to above do not mention "permanent damage". They do, however, give the impression that the 0.1 r per day may go on for life but do not definitely say it.

L. Taylor, the Chairman of the American Committee referred to above states in a paper in the J.A.M.A. in 1941:

"By the 'tolerance dose' is meant the amount of x-ray energy that a person can receive continuously or at repeated intervals without suffering any damage to the blood or reproductive organs."

Cantril gives a review of many allied phases of this subject in the Project Handbook and there is no need for repetition here.

There is need, however, to stress the points that (1) the tolerance dose is based not on definite facts and observed reactions following measured amounts of radiation over long periods of time but rather on clinical impressions or some partially observed reactions following amounts of radiation which can only be very grossly estimated in some cases and is an extremely nebulous amount in many (2) there is an attempt to allow a wide margin for safety to the individual based on continued exposure, but this does not consider genetics to any great extent.

The Safety Rules and Procedures Concerning Activity Hazards at Clinton Laboratories has set 0.1 r per day as tolerance for radiation exposure as is evidenced by the general rules in Section B-1 of the compiled group of Procedures and Rules adopted by the Central Safety Committee on 6/16/44. Rule 2 of the same Section states "No individual shall knowingly expose himself or cause others to be exposed to more than 0.1 r per day without prior written authorization of the Health Division.

I can condone such exposures as I report in the first part of this letter but I do not feel that I can give written authorization for them or their continuance for an indefinite time; nor does this letter constitute such authorization. I would have you bear in mind the following:

- (1) The exposures reported are believed, with fair assurance, to be the only ones occurring during these hot jobs. The personnel and supervision are cooperating with each other and with the Health-Physics Section to the fullest extent in getting accurate records as well as in doing everything possible for them to do under war exigencies, to allow no more exposure than is absolutely necessary to get the job done.
- (2) The exposures reported have a fair degree of accuracy and are not guesses.
- (3) It is not contemplated that they will be continued over any extended period.
- (4) I feel sure that no damage has been done to any of these individuals and that no ill effects will show up in them in the future from the exposures recorded above.

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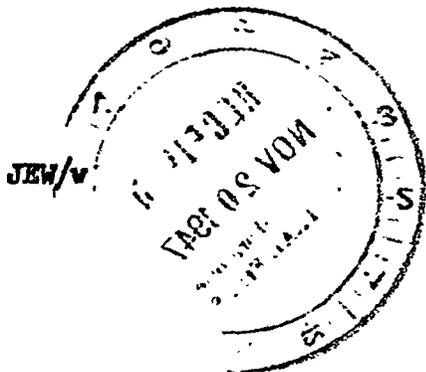
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(5) Supervision on the job has been acquainted with the exposures and plans in the future to continue to rotate personnel and to circumvent the hazard in all conceivable ways.

It seen impossible to handle such large quantities of active material with existing facilities with any fewer over-exposures than those given above.

Jewirth

John E. Wirth, M. D.



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 CLINTON LABORATORIES 8-9 R & C Files
 DATE 11-18-44

~~SECRET~~

TO C. D. Coryell DEPARTMENT
 FROM M. C. Leverett DEPARTMENT

IN RE: Basic Chemistry - Ia¹⁴⁰ Production

Classification Cancelled
 Or Changed To _____
 By Authority Of _____
 By [Signature] Date AUG 23 1971

This memorandum confirms our conversation of approximately ten days ago in which I pointed out to you that in order to complete construction of the 706-D building and to get its necessary equipment installed in 120 days, not more than 20 days from date of authorization could be permitted to elapse before the basic chemistry is specified. The project was authorized on November 11th, and it therefore appears that we shall need to know the basic chemistry by December 1st.

To assist you in making your decision as to a suitable chemistry I should like to state my opinion that simplicity, avoidance of steps which have not been performed a large number of times with the radiation shielding, and reliability of operation are the cardinal virtues in a production operation. These should be given considerably more weight than high yields or the desire to use a novel scheme of operation or preparation.

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

M. C. Leverett
 M. C. Leverett

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This document has been approved for release to the public by:
David R. Hamlin 1/20/95
 Technical Information Officer Date
 ORNL Site

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~~SECRET~~

210 This document consists of 1

1-6 Form a

CLINTON LABORATORIES

CLINTON LABORATORIES

GENERAL FILES NUMBER

44-11-341

DATE 11/24/44

- 1 & 2 M.D. Whitaker
- 3 & 4 E.J. Murphy
- 5 W.C. Johnson
- 6 C.D. Coryell

M. D. Whitaker

DEPARTMENT

FROM H. S. Brown

DEPARTMENT

IN RE:

SHIPMENT #3 OF Ba¹⁴⁰

XT

99
*ANI



The third preparation of Ba¹⁴⁰ has been completed and made ready for shipment. Enclosed is a complete report by C. D. Coryell on the preparation characteristics. Mr. Coryell's report should be transmitted to Mr. Oppenheimer - attention of R. W. Dodson.

Harrison S. Brown
Harrison S. Brown

Classification Cancelled

By Authority Of _____

By *Wayne*

Date AUG 23 1971

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David R. Hammin 2/3/95
Technical Information Officer Date
ORNL Site

CLASSIFICATION CANCELLED

J.S. Morgan 1-31-95
ADD signature Date

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²¹⁰ This document consists of _____ pages and _____ figures. No. _____ of _____ copies. Series A

CLINTON LABORATORIES

~~CONFIDENTIAL~~

DATE 11/29/44

- 1. J.E.Wirth
- 2. M.D.Whitaker
- 3. R.L.Doan
- 4. W.C.Johnson-Brown
- 5. C.D.Coryell
- 6. Reading File
- 7. Central File

TO J. E. Wirth DEPARTMENT

FROM C. D. Coryell DEPARTMENT

IN RE: 706-C OPERATIONS ^W₆₅

We who have been most closely associated with the production job in 706-C have been very distressed by the obviously poor conditions with regard to over exposure of personnel. I had high hopes that preparation #3 could be carried out as cleanly as was preparation #1. We shall not diminish, however, the effort we are expending to eliminate completely all exposure in excess of acceptable tolerance. I should like to point out that we are most pressed on the side of the glass operations. It is here that the very hottest sources are encountered without much extra Pb shielding; it is here that the hottest samples are withdrawn; it is here that the operations of final container removal must be carried out; and it is here that panel contamination can occur. We are operating with only six men able to handle the extremely complicated controls and it requires two shifts of three each so that rotation of men cannot be achieved. We have tried aggressively to obtain more men for the Building to be able to give training to more operators and to be able to have a more flexible manning schedule. Our efforts have been nearly unsuccessful and the manpower we have asked for is, in fact, partly committed to be trained in order to release men for 706-D operations. I think we should raise our sights and obtain more men but recall for two days from 706-D crews manpower needed for rotation on the hottest jobs.

I will appreciate any advice you can give us or any support on the difficult problems we have in continuing operations in an overcrowded building with equipment under designed for the very urgent goals set. I wish also to express whole heartedly my appreciation of your past interest and assistance and that of Dr. Morgan and the many members of his crew associated with this effort.

Classification Cancelled

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By Authority Of _____

By C. D. Coryell Date 11/29/44

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Ted Davis 3/8/95 Date
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This document has been approved for release to the public by:

David R. Hamm 3/9/95
 Technical Information Officer: Date
 ORNL Site

2 11 0 A
K. Z. Morgan

H. P. Gauvin

12/6/44
Health-Physics

Health-Physics

1. K. Z. Morgan
2. J. E. Wirth
3. R. S. Stone
4. H. S. Brown
5. R. L. Doan
6. C. D. Coryell
7. W. E. Cohn
8. H. P. Gauvin
10. Central File
9. W.H. Ray
11. Readers File

HOT RUN #3 in BUILDING 706-C.

The third "hot" run in Bldg. 706C began on 11/14/44. On this date 36 slugs were brought down from Bldg. 105. This process continued for 6 days. During these transfers a maximum reading of 160 mr/hr was obtained through the bottom of the slug bucket. Readings at the other five sides were all less than this.

During the entire run routine readings were taken at designated places around Bank 2 in order to have some immediate indication of the rise in radiation level. These readings indicated the level to be greatest at the air vents on top of the bank. Here, in contact with the vents, the level rose to approximately 300 mr/hr. However, no one remained here for any appreciable length of time. With the exception of the area near the filter disc of Cell 4, all other areas remained relatively low. The level at the face of Cell 1 rose only toward the end of the run. At this face the level rose to approximately 100 mr/hr. The men working here took all reasonable precautions to avoid over-exposure. However, some over-exposures were observed. These were indicated by the dose meters, pocket meters and film badges. The maximum reading as indicated by the dose meters was 220 mr for any 24 hour period.

During the sampling process, a maximum hand exposure of approximately 1800 mr (total for the run) was obtained. The maximum reading obtained from any one sample was greater than 1 r/hr. at about 6 inches. The general level between the sample heads was only 30 mr/hr.

On 11/18/44 the hot filter disc was removed from the shield at the entrance to Cell 4. This filter read 6 r/hr at 3 ft. It was placed in the "garden" overnight, unshielded. The following day this filter read 9.2 r/hr and was replaced in the shield. The dose meters indicated no over-exposures during this transfer.

Smear tests of the entire building were taken on 11/23/44. These smears indicated a rather high level of contamination. The high counts seemed to be concentrated in a definite path from the top of Bank 2 to semi-hot Cell 9. Cell 9 is where the hot samples were diluted.

It is suspected that the contamination was tracked from the cell to the remainder of the building. It was previously observed that the handling of the samples showed possibility of spills. The smears taken from the bench, floor and stools of Cell 9 all jammed the counter. Other smears throughout the building gave as high as 8000 counts/minute.

CLASSIFICATION CANCELLED

DATE 10-15-64

For The Atomic Energy Commission

H. P. Canale

Chief, Declassification Branch

By 11/29/44 the preparation of the sample was completed. Cell 1 was opened and a reading of 4 r/hr was obtained at the cell entrance. This same day the sample was removed from the cell. Gloves that had come in contact with the pot read 3 r/hr. These gloves were removed immediately. The total exposure was probably very low. The radiation level around the pot was 2 r/hr, above the pot much greater than 10 r/hr, perhaps as high as 30 r/hr. It was found that the pot was highly contaminated. The contaminated paper was removed from around the pot. It is believed that this and the moving of the pot from the cell caused the present state of contamination in the building. This contamination is by far the worst and most widespread ever encountered in the building.

It is believed that most over-exposures have occurred during the cleaning process after the hot-runs. At least the exposures have been more extended during these periods. Many cases of hair and shoe contamination have been observed during the cleaning of the cells. It is believed that this was picked-up from the soot on the cells' inner walls. It is suggested therefore that a building rule be set-up to require the wearing of rubbers and hats in the hot cells. The danger from contamination seems to predominate over that of direct exposure. This would lead one to believe that many of the dangers could be eliminated. Although care has been exercised in the cleaning of the cells it is believed that a little more thought devoted to the health aspects of this problem would prevent spreading of contamination.

In general, the cooperation given to the Health-Physics representative and the consideration given to health problems in this building have been very good.

HW/w

H. P. Gauvin





3 11 0 2 A

1/9/45

- 2. R. L.
- 3. R. S.
- 4. W. C.
- 5. H. S.
- 6. C. D.
- 7. W. E.
- 8. K. Z.
- 9. J. E.
- 10. Central
- 11. Readers

M. D. Whitaker

John L. Wirth

Medical



It was hoped that the experiences in 706C during the month of November 1944, which created conditions which were rather serious health hazards, would have been influential in bettering conditions in the future. The reference to the November experiences is particularly directed toward the conditions necessitating a change in the clothing program for 706C personnel, and the W-11 tank incident. Such, however, apparently is not the case and it becomes necessary to point out to you at this time that six serious spills occurred during the month of December 1944. It is believed that most of these could have been prevented by more care on the part of personnel.

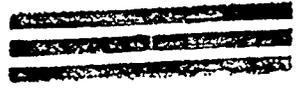
I would call your attention to two Health-Physics reports dated 12/11/44, covering the week of 1/2/44 and the week of 12/12/44, and two other reports dated 12/17/44 and 12/31/44, all by S. Block to K. Z. Morgan.

The six occurrences may be identified briefly as follows:

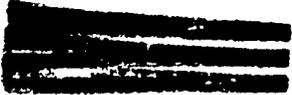
1. Water running from a Cell as a result of flushing flues on Bank 2 (12/4/44)
2. Stop-cock blown out of a Cell in Bank 1 (12/5/44)
3. A hot nitric acid spill in Cell 1, Bank 1, which leaked out on the floor (12/13/44)
4. Hot spill from Cell 4, Bank 2, when water which was being hosed into the Cell leaked out through a crevice in the Cell door (12/13/44)
5. UNH from the waste storage tank back-flowed, due to improper adjustment of a valve. (12/26/44)
6. A repetition of the same incident as in No. 5 (12/31/44)

It does appear that something could be done to prevent such occurrences. It is appreciated fully that both Mr. Coryell and Mr. Levy have been extremely cooperative with the Medical Division in an attempt to control contamination. It is apparent, however, that these two individuals

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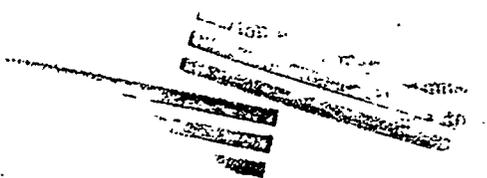
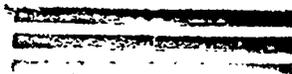
cannot be present constantly to prevent such occurrences. Responsibility and freedom of action for the individual worker is an excellent general principle, but in the face of repeated occurrences such as the above, it certainly appears that closer supervision, or other changes which you may be able to bring about are not only warranted, but necessary if work in the 706C Building is to continue without damage to personnel.



John E. Wirth

John E. Wirth, M. D.

JEW/w





CLINTON LABORATORIES

DATE 1/13/45

- 1. W. C. Johnson
- 2. R. L. Dean
- 3. J. E. Wirth
- 4. H. A. Levy
- 5. C. D. Coryell
- 6. W. E. Cohn
- 7. C. Files
- 8. R. Files

to W. C. Johnson

DEPARTMENT

FROM W. E. Cohn

DEPARTMENT

This document consists of 4 pages and 0 figures.
 No. 1 of 8 copies, Series A

IN RE: Recent Events in 706-C.

I have just read the letter of Wirth to Whitaker of 1/9/45 and feel that a certain amount of explanation and comment from me, as the head of one of the groups working in 706-C, is in order.

There are six incidents (accidents or hot spills are synonyms) listed in the above-mentioned letter. Of these, two occurred in locations used by Parker and Tompkins, and their co-workers, of my section. I will describe and explain these briefly before going on to more general items.

Item # 2: Stopcock blown out from an exposed "hot" line on the face of cell 2 in bank 1 (12/5/44). This is an apparatus failure caused by the piling up of several minor failures and emergencies, resulting in a totally new situation. It could have been prevented (what accident cannot?) if (1) such a compounding of emergencies could have been foreseen and (2) if there were sufficient time and absence of work pressure to permit the existence of an emergency to cause stoppage of work. Neither has been nor is yet the case in 706-C work. The steps leading up to item # 2 were as follows:

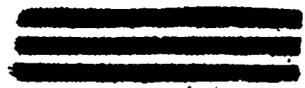
1. "Hot" UNH of wrong specifications supplied due to dissolver breakage in cell 1, bank 1 (not operated by us). Six days were lost in treating this solution and one apparatus failure resulted which was directly due to this faulty solution. In addition, we had already been delayed five days due to the closing of 706-C for decontamination.

2. Pressure to get material out for use. Since the dissolver was down, perhaps indefinitely (due to work pressure on Coryell's section), this represented the only source of badly needed material, and it could therefore not be jettisoned in favor of "right" material.

3. An apparatus failure, two feet from the UNH. Hot material could not be moved to shielded storage, and we dared not throw it out (see above). Conclusion: Devise a new way of extracting the solution.

4. In the emergency method devised, air pressure had to be used where normally not used. This caused "hot" ether (fluid, not vapor as reported) to push up against the stopcock.

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5. The ether was "hot" because the demands made on personnel by our emergency method prevented us from back-washing it to a safe level.

6. The stopcock "blew", allowing "hot" ether to run out on the face of the cell.

The ensuing events were as follows: the extraction was stopped, contaminated clothing (and hands, from replacing the stopcock) was removed or washed to a safe level, contaminated equipment was removed and washed or tagged, the spill was mopped up and measurements of surface activity were made. The face of the cell was washed, lowering the radiation to below 100 mr/hr. at six inches, and tagged. Then the extraction was finished, the hot solutions removed to other locations and processed, and we began the decontamination of apparatus to determine the nature of the internal failures.

Item # 3: Hot spill in cell 2 (not cell 1), bank 1, which leaked out of the cell and onto the floor (this was not nitric acid as stated but rather ammonium citrate being used as an apparatus wash); 12/13/44. This spill occurred directly underneath the spill reported above, re-contaminating the same area of wall with activity removed from the equipment. This spill occurred because the apparatus had been partly dismantled (due to first spill) and was initially confined to the interior of the cell. However, the fluid was partially discharged to the outside through an open hole near the inside floor.

Ensuing events: Wall and floor washed down immediately to radiation level of about 100-200 mr/hr. at 1" area blocked off. A work order was requested of the building manager to have the surface concrete chipped away since further washings proved ineffective. This work was not begun until about 1/4/45.

During part of the period that this area (wall and floor) was contaminated, the passageway could not be blocked off since a hotter area had been created in the only alternate passage. This undoubtedly led to some of the count-spreading which took place after the second spill.

In discussing these two spills, which are really part of one sequence of events, I wish to point out that there is little justification for the comments that have been made nor for the general alarm which has been sounded. Procrastination was not evident, as charged (Block to Morgan, 12/11/44), since manpower could not be released from the job in hand until the material was shipped out on 12/12 and 12/19; furthermore, all measures which research personnel could reasonably be expected to take were taken at once, and at no time was the situation out of hand or disregarded. In order to have effected immediate restoration of the pre-spill radiation level, two things would



have been necessary: (1) freedom from pressure to produce (this would also have eliminated the initiating accident); (2) research personnel would have been required to chip away the contaminated concrete. Neither of these was necessary or desirable.

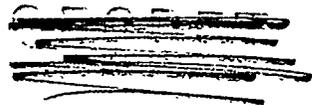
I wish also to refute any implication that the work being done when the accidents occurred was poorly planned or executed, that the apparatus was improperly designed, that the personnel involved was delinquent (before or after), that the accidents could have been prevented under the situations extant or by higher supervision, or that the accidents themselves were - or could have been - serious in consequence. I see no need at this time to argue these contentions, but I should like to outline the fundamental considerations upon which this view is taken:

1. 706-C is engaged in research work; it is not a routine production plant. Therefore, the unexpected must be expected, and accidents (spills, etc.) are not bona-fide evidence of incompetence, either high or low. Anyone can get into trouble; only the good man can get out of it, because he anticipates the unexpected.

2. All 706-C operations, to this date, have been conducted under pressure. To do our job, and get it done, means taking calculated risks. The "completely safe" 706-C device, apparatus or gadget has not been created, and may never be; there is not time, nor is there experience of a 706-C nature to guide us.

By "calculated risk" I do not mean that all possible precautions are not taken. On the contrary, the operations are as carefully planned as possible. With regard to the six events listed above only two had a reasonable probability of being avoided by forethought or higher supervision. All were "expected", in the sense that they were immediately detected properly handled and safely disposed of, with no damage to anyone save loss of time and manpower.

This last item in itself is as severe a punishment as one could wish, if punishment were in order. It is the most effective discipline that can be desired. No amount of rules or regulations, nor of immediate higher supervision, could have prevented the six spills (with two possible exceptions), nor will these prevent future spills of different natures. The knowledge that each individual responsible for a spill must clean it up is the best of all spurs to safer action. It is also to be recognized that all individuals are responsible for detecting spills within their spheres of action. Action upon these principles is our best and only hope of minimizing (but probably never eliminating) future spills.



There is much that could be done to make 706-C a safer place in which to work and to reduce both the number of accidents and their ensuing difficulties. 706-C was designed for a maximum of 80 C at a point, or 10 C of hard gamma (it is a one-slug building) and a maximum of 10 men; it is being used for 400 C of hard gamma production on a 200-slug basis, and is populated by over 20 men. Janitorial and laboratorian assistance is not in proportion to need in spite of efforts to procure it. The pressure to produce is antagonistic to the slowness and care with which we should prefer to operate on such super-hot material. I suggest that here is the proper place for action, now as at any other time.

Waldo E. Cohn

Waldo E. Cohn

WEC:dd

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cc: M. C. Leverett
F. R. Ward
E. D. Peterson
F. G. Rehm

W. A. Rodgers
W. P. Bigler
R. File
C. File

This document consists of 2
pages and 2 figures.
No. 2 of 9 A

February 9, 1945

TO: File

FROM: W. P. Bigler

PROPOSED 706-D SAMPLING SCHEDULE

This presents a tentative sampling set-up and notation resulting from a discussion with F. G. Rehm and W. A. Rodger. Future discussions and alterations will be formed using this schedule as a basis.

Samples will be taken with either a Standard plant type sampler (Std.) or with the T.Vallado design surface tension type (λ). In many cases solution can be had using the capillaries provided in the vessels. Emphasis will be placed not only on care and accuracy in sampling, but also that all samples required are taken and containers are marked so that there can be no confusion as to sampling data. Pertinent sampling data will be recorded in duplicate, one copy being retained by Operations and the carbon copy going to the Analytical Dept. Complete compilation of the run results can be made from these data sheets. The required samples are listed on the attached sheet.

W. P. Bigler

CLASSIFICATION CANCELLED

Jed Davis 3/8/95
ADD signature Date

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Classification Cancelled

~~of [redacted]~~
By Authority of DOC
By EES Date AUG 23 1971

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

David R. Hamon 3/9/95
Technical Information Officer
ORNL Site

Sample No.	Code	Vessel Sampled	Type Sampler	Vol. of Sample	Radiations	Vol. of Solution	Solution
1	1MA	A-1	Std.	>1cc	1.0R	27 gal.	Metal of 1st 150 slugs
2	9MA	A-9	"	"	"	49 "	" " " " "
3	8WMA	A-8	"	>2cc	"	80 "	Metal waste of 1st 150 slugs
4	1MAB	A-1	"	>1cc	"	27 "	Metal of 2nd 50 slugs
5	9MAB	A-9	"	"	"	49 "	Metal of 2nd 50 slugs plus Ba* of A
6	8WMB	A-8	"	>2cc	"	80 "	Metal waste of 2nd 50 slug
7	1MC	A-1	"	>1cc	"	27 "	Metal of 3rd 50 slugs
8	9MC	A-9	"	"	"	49 "	Metal of 3rd 50 slugs plus Ba* of A & B
9	8WMC	A-8	"	>2cc	"	80 "	Metal waste of 3rd 50 slug
10	1MD	A-1	"	>1cc	"	27 "	Metal of last 50 slugs
11	9MD	A-9	"	"	"	49 "	Metal of last 50 slugs plus Ba of A, B, & C
12	8WMD	A-8	"	>2cc	"	80 "	Metal waste of last 50 slug plus all washes
13	14WC1	A-14	"	>1cc	0.2R	17 l.	1st Metathesis waste
14	14WC2	A-14	"	"	"	"	2nd Metathesis waste
15	IP	B-1	λ	5 λ	0.3R	2.5 l.	1st prod. sample (Ba* in dil. HNO ₃)

At this point it should be possible to get a material balance. The next 2 samples taken depend upon the process used. (I or II)

I CHROMATE PROCESS

{16	IBP	B-1	λ	5 λ	0.1R		Dissolved PbCrO ₄
{17	3WCr	B-3	λ	5 λ	0.05R		Ba CrO supernate containing PbO ₂ =

II ELECTROLYSIS PROCESS

{16	6EPS	B-6	λ	5 λ	0.1R	3.0 l.	Electrolyzed prod. sol'n.
{17	12BP	B-12	λ	5 λ	0.01R	4.0 l.	Dissolved plated Pb.
18	3WIN	B-3	λ	5 λ	0.01R	18. l.	1st f. HNO ₃ waste
19	3W2N	B-3	λ	5 λ	0.01R	18 l.	2nd " " "
→	6P	B-6	λ	5 λ (1)*	1.0R	0.2 l.	Final product solution

* Several

1-2 M. C. Leverett
3 M. D. Peterson
4 W. A. Rodger

6 F. G. Rehm
7 Reading File
8 Central File

CLINTON LABORATORIES, Ward

CENTRAL FILES NUMBER

45-2-135

February 10, 1945

TO File

DEPARTMENT

FROM M. C. Leverett

DEPARTMENT

IN RE:

Purity of Ba-Ia Product

M.W.
ST

It is understood that the tolerances for various activities in the Ba-Ia product from 706-D are as follows:

The tolerances are given in terms of the gamma disintegration rate of the impurity expressed in percent of the Ia^{140} disintegration rate. All figures given refer to a time five days after final Ba separation.

Isotope	Half-Life in Days	Tolerance (%)
Zr ⁹³	65	1.5
Zr ⁹⁵	0.71	18.
Cb ⁹³	35	1.9
Ru ¹⁰³	42	3.2
Ru	~60	1.3
Pd ¹¹²	0.71	18.
Sn	11	8.0
Sn	2.5-3.3	18.
Sb	250	0.8
Te ¹²⁷	90	12.
Cs ¹³⁵	7,300-11,000	1.4
Ce ¹⁴¹	28	11.
Pr	55	1.3
Eu ¹⁵⁴	16	4.5

This document has been approved for release to the public by:
64
David L. Johnson 3/19/85
Special Agent in Charge
ORNDORF

CLASSIFICATION CANCELLED
ADD signature
Single review of CCRP-declassified documents was authorized by DOE Office of U.S. Classification memo of August 22, 1994.
Date 3/19/85

If more than one of the above impurities are present simultaneously, the following condition should be satisfied:

$$\sum \frac{X'_i}{X_i} \leq 1$$

Classification Cancelled

~~Or changed to~~

By Authority Of *DOC*

By *SEK* Date *AUG 23 1971*

Here X_i is the tolerance figure given in the third column of the above table for the impurity i when present alone; and X'_i is the disintegration rate of that impurity actually present, expressed in percent of the Ia^{140} disintegration rate.

For gamma-emitting impurities other than those listed above it is stipulated that their combined disintegration rate not exceed 20% of the Ia^{140} disintegration rate five days after final Ba separation. Any as yet unknown activities similar to those in the above table may have more stringent special tolerances, however.

M. C. Leverett
M. C. Leverett

2 984

Mark Lab. 706 D
C.F.
- 1 - 144

GENERAL ELECTRIC
CENTRAL FILE NUMBER
45-3-155

A-670

File ak

Date 3/12/45
Subject 706-B Design Notes - March 7th to 10th, Inclusive
By M. G. Everett
To File

Those Eligible
To Read the
Attached

Declass
fe

#2 C. S. Coyell

INV
9

Before reading this document, sign and date below

Name	Date
T. J. Gray	3/14
CDC	
TAD	Y. Adams
HAL	H. L. Henry 3/15/45
ELB	E. L. Brady 3/15/45
	L. Goldring 3/15/45

Name	Date

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

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



CLASSIFICATION CANCELLED

DATE 11/30/65

For The Atomic Energy Commission

March 12, 1945

To: File

From: M. C. Leverett

H. R. Canale
Chief, Declassification Branch *ac*

706-D Design Notes - March 7th to 10th, Inclusive

1. A determined effort is being made to have construction complete and dummy runs ready to start by April 15th. In an effort to eliminate unnecessary safety factors, installation of several vessels has been cancelled or scheduled on a tentative basis to come after the dummy runs.

2. The following vessels have been eliminated permanently:

A-13 #2 centrifuge (12" Bird) in Cell A

A-14 #2 centrifuge catch tank in Cell A

B-10 Cell B waste hold-up tank

B-13) Duplicate rotating pipettes for transfer of product solution from
B-17 (product solution sampling and storage vessel) to B-19 (evaporator)

These eliminations are possible because of the following reasons:

B-10 was thought to be required in Cell B for collection of wastes early in the design when Cell B operations were still indefinite and it appeared that acidic waste volumes might be large, and that it would be necessary to collect and hold them until a propitious time when A-5 would be available for neutralizing. More definite information on waste volumes became available about March 1st, and it then was decided that B-10 is not necessary.

B-13 and B-14 are found, on detailed analysis of transfers of liquids among B-6, B-12, B-17 and B-19, to be unnecessary if the equipment in Cell B is rearranged so that rotating pipettes B-7 and B-8 (duplicates) serve the electrolytic cell (B-12), the product solution storage vessel (B-17) and the final evaporator (B-19). This arrangement is feasible and B-13 and B-14 are therefore eliminated.

A-13 and A-14 would be used primarily in the event of failure of the $Pb(Ba)CO_3$ to settle properly after K_2CO_3 metathesis in A-9 precipitator. This metathesis has been carried out eight times in the semi-works on full plant scale, in stainless steel, in a full scale mock-up of that portion of A-9 in which the metathesis is carried out. UNH from inactive X slugs was used. In no case except one (where an operating error was made) did the product loss for the two-shot metathesis exceed 1.5%. The average, excluding the bloop, was 0.8%. Moreover, in four cases the metathesis supernate was centrifuged after settling, and its product content measured again. The average loss was reduced by only 0.2% of the product present.

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It is therefore concluded that

- a) Losses in metathesis without centrifugation are acceptably low;
 - b) This result is obtained consistently despite minor variations in conditions;
 - c) Centrifugation does not materially decrease the losses in this step.
3. The following vessels will not be installed for the dummy runs, but space for their installation will be kept, and they will be ready for installation by May 1st if at that time they are thought necessary on the basis of the dummy runs:

- A-7 #1 centrifuge in Cell A (12" Bird)
- A-11 #1 centrifuge head tank in Cell A
- B-2 #1 centrifuge in Cell B
- B-4 #1 centrifuge head pot in Cell B

The uncertainty about the need for these vessels is due to the following facts:

A-7, #1 centrifuge in Cell A, would be used

- a) For removal of dissolver crud suspended in the UNH solution;
- b) For removal of suspended $Pb(Ba)SO_4$ from the extraction supernate during decantation;
- c) Possibly, in the absence of A-13, for removal of suspended $PbCO_3$ from the metathesis supernate.

There is, a priori, some reason to suppose that the presence of suspended crud in the UNH would be troublesome, possibly by carrying of product or by plugging up jets. Actually neither of these troubles has materialized either in 706-C or in the semi-works runs. Use a) therefore appears unlikely. Suspended $Pb(Ba)SO_4$ in the extraction supernate has not been particularly troublesome in the semi-works runs. Eight runs using dissolver UNH, at one-fifteenth plant scale, in stainless steel equipment approximating a mock-up of A-9, have been made. In one of these an operating error led to a high loss. The remaining percentage losses were 10.7, 18.9, 6.6, 9.9, 3.6, 6.5, 7.6, for an average of 9.1%. A run on pure UNH gave 3.9% loss. A loss of 9% in this step would not be intolerable, but the lack of consistency in the results is disturbing. In ten cases the extraction supernate has been centrifuged after analysis and again analyzed. The average decrease in product loss was 1.1% per precipitation, or 4.4% total since there are four extraction precipitations performed during each complete preparation. It is not clear from these data whether the lack of uniformity in losses in the extraction step is due to failure of the $Pb(Ba)SO_4$ to settle or not - i.e., it is uncertain whether centrifuging the decantate would decrease the losses significantly. Moreover, it is

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uncertain what will result when the process is carried out on full scale. For these reasons it is judged unwise to make impossible the installation of A-7 and its head tank, A-11. Losses of product during the washing of the settled $Pb(Ba)SO_4$ have averaged 1.5% for the four runs for which complete data are available, running 0.7, 0.5, 0.3 and 4.5%. In all cases tested, centrifugation of the wash solution reduced the product loss to less than 0.1%, but the losses got by settling are usually so small that centrifugation to reduce them would not be justified. The final decision on A-7 (and A-11) will therefore rest on the decantation of the UNH from the $Pb(Ba)SO_4$.

Centrifuge B-2, and its head pot B-4, would be used

- a) For removal of suspended $PbCrO_4$ from the B-1 supernate after $PbCrO_4$ precipitation;
- b) As a spare for B-15 (#2 centrifuge, Cell B, used for $BaCrO_4$ centrifugation);
- c) Possibly, in the absence of A-13 and A-7, for removal of suspended $PbCO_3$ from the metathesis supernate.

There is as yet no significant semi-works experience on the $PbCrO_4$ precipitation and settling, but all indications from the laboratory are very favorable. Because, however, of the lack of data it is judged unwise to make impossible the installation of B-2 and B-4. Semi-works data will soon be available. Use of B-2 as a spare in case of failure of B-15 is at present given only minor emphasis. It is thought that if a centrifuge of the preferred type (over-driven) is installed as B-15, impending trouble can be either avoided by frequent inspection of external parts, or easily repaired in all except a few cases.

It is evident that it is improbable that both A-7 and B-2 will have to be installed. Therefore it is likely that considerable time will be saved by deferring installation of either until the need is demonstrated.

- 4. The electrolytic cell B-12 probably will not arrive on the plant in time to be installed before the dummy runs. It is requested that this vessel be installed at the first opportunity after its arrival, probably during the first decontamination. It is now thought that the electrolytic process will make a product containing less lead and in a shorter time than the chromate one. A higher specific activity also is possible.
- 5. All vessels and equipment shown on our equipment flowsheets but not specifically mentioned here are at present regarded as necessary to good operation, and are required for installation before dummy runs start.
- 6. It is understood that C. W. Moore and W. J. Feder will be authorized to make changes in the field from now on.

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3-12-45

7. Technical Division approval for such changes must come from Leverett, Ward, or Rodger, in that order. Technical Division field inspectors are not authorized to request changes; such requests must be made by Leverett, Ward or Rodger.
8. Occupancy of 706-D by Technical Division will be deferred as long as possible, but equipment clean-up, testing and calibration may require that Technical take over some areas before the entire installation is complete.
9. It was agreed that the last section of the mono-rail leading to 706-C could be omitted and put in Spare Parts. This will make it unnecessary to cut a hole in the dividing wall between 706-D and 706-C at present.
10. Leverett stated that the vessel off-gas collection system in both cells is necessary on the basis of 706-C experience and should not be omitted.
11. The lambda sampler is to be redesigned by Technical.
12. Leverett stated that B-15 (#2 centrifuge in Cell B) is an essential piece of equipment, even though laboratory work is being done to improve the settling of the BaCrO₄ precipitate.
13. It was agreed that J. T. Weills would be assigned to spend full time with H. S. Knotts starting March 13th or when available, until about April 1st.
14. Revised equipment flowsheets, jet lists, and Cell B arrangements will be submitted in accordance with the changes noted above.

M. C. Leverett
M. C. Leverett

- 1 H. S. Brown
- 2 C. D. Coryell
- 3 R. L. Doan
- 4-5 H. S. Knotts
- 6 E. J. Murphy
- 7-8 M. D. Peterson-
W. A. Rodger-
F. G. Rehm
- 9-10 M. C. Leverett
- 11 F. R. Ward

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45-43122

Date 4-11-45
Subject 706-D Design Notes, 3-27 through 4-7
To File
From M. C. Leverett

File Declarator
Copy # 2 C. D. Coryell

Before reading, please sign and date below:

Handwritten signatures and dates: M. C. Leverett 4/12/45, C. D. Coryell 4/11/45

Handwritten number 45-4-122 and a stamp with 'INV' and '64'.

- Distribution: 1 H. S. Brown, 2 C. D. Coryell, 3 R. L. Doan, 4-5 H. S. Knotts, 6 E. J. Murphy, 7-8 M. D. Peterson-W. A. Rodger-F. G. Rehm, 9 F. R. Ward, 10-11 M. C. Leverett



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

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~~SECRET~~

DATE 12/1/65

April 11, 1945

To: File

For The Atomic Energy Commission

From: M. C. Leverett

H. F. Carroll

Chief, Declassification Branch

706-D Design Notes. March 27th through April 7th

This memorandum is the concluding one of this series. Final modifications in 706-D will be taken care of in the means described in the memorandum on this subject dated April 7th. During the period preceding April 7th the following items of interest are noted:

1. A crack was found in the bottom of vessel B-1 during the polishing operations. Application of water pressure to the outside of the vessel did not produce leakage, from which it was concluded that no complete penetration of the weld had resulted. Probably only the inside weld was cracked. However, instructions were issued immediately to have made up a replacement for B-1 in the event that the cracked tank should fail.
2. The electrolytic cell, B-12, was shipped approximately one month ahead of schedule. This resulted in shifting emphasis from the chromate process to the electrolytic one inasmuch as it is considered to be somewhat better.
3. It was found that the proposed decantation rate out of B-6 after chromate precipitation was so fast that substantial quantities of the precipitate were carried by the decantate. To correct this condition one of the decantation lines was decreased in size from 1/8" IPS to .105" ID. This is the lower of the two decantation lines on this vessel.
4. It was found that barium chromate made according to flowsheet conditions does not centrifuge readily. A slower feed rate to the centrifuge may be necessary or it may be necessary to recycle the centrifugate from B-3 to B-5. Instructions to proceed with either of these changes are withheld until after further experimental work.
5. It was requested that the off-gas line from A-16 be relocated so as to go out the east side of the building at once rather than running outside the cell and through the principal operating areas. This change has been made.
6. A definite understanding was arrived at with regard to the false flooring on top of the cell block. This will cover only Cell B, extending, however, to the panelboards on the west, north and east sides.
7. The small periscope designed for viewing at right angles is to be redesigned to view at an angle 38° forward of the normal. This will permit a single instrument to be used to view B-12 and B-17. A replacement part for the periscope has arrived from Chicago.
8. The west end of Building 706-D was taken over on March 31st by the Technical Division.

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9. It was found that a leak existed between the wall of vessel A-1, the dissolver, and its jacket, the leak being in the weld between the vessel and the spacer ring. Several such leaks were found in both upper and lower rings. The small ones were repaired by peening and the larger ones were repaired by welding while spraying the inside of the vessel with water in order not to overheat the heat-treated parts.
10. It was discovered that Raschig rings had been put into several of the scrubbers and condensers without first removing the blue paint used to identify 25-12-S-Cb steel. A request that such paint be removed from all such rings except those in A-4 and A-16 was made. ||
11. An interference was discovered between the overflow baffle in A-8, the catch tank and the standard sampler assembly supposed to go into a nozzle directly over the overflow. Installation of the sampler assembly in a spare nozzle, nozzle J, was approved.
12. The cable length on the monorail tractor controls was increased so that the controls hang about two feet off the floor, and in the case of the upper monorail, about six feet from the hook.

M. C. Leverett
M. C. Leverett

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FILE NUMBER

45-4-143

A-670

4/12/45

Date 7060 Off-Gas Line

Subject _____

L. B. Milet

By J. P. Sinclair

To _____

File 71

Those Eligible
To Read the
Attached

*Declass
JK*

#2 H. A. Long

INV. 65

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Name _____ Date _____

H. A. Long *4/12/45*

HZ

ELB EJB *5/1/45*

MJG

MHH

SLW

REG R. Garber *4-13-45*

RJK *5/1/45*

Name _____ Date _____

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ANT

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David R. Hamlin
Technical Information Officer
ORNL Site

1/30/95
Date

April 12, 1945

Mr. J. P. Sinclair

- 1. J. P. Sinclair
- 2. H. A. Levy
- 3. L. B. Ealet
- 4. Reading File
- 5. Central File

L. B. Ealet

706 C Off-Gas Line

During the last run in the 706 C building the stainless steel off-gas line, between that building and the 200 Area stack, became highly contaminated. The actual dissolving of the material in 706 C apparently did not deposit any residual contamination in this line. The building up of activity seemed to start after all of the waste liquor was in the underground storage tank. The last dissolving batch was completed on Monday, April 2nd. No appreciable increase in activity in the stainless steel line was noted until Wednesday, April 4th. Even then the readings were comparatively low.

Thursday, April 5th the 706 C operations started to neutralize the waste liquor in the underground storage tank. The activity build up in the off-gas line was picked up by the #1 Integron located on top of the pile. The following readings indicated the maximum intensity recorded by this instrument for the periods shown:

Date	Shift	Readings
4-5	4-12	58 mr
4-6	12-8	51 mr
4-6	8-4	71 mr
4-6	4-12	48 mr
4-7	12-8	75 mr

Other readings obtained by the Health Physics Group proved without doubt that the radiation recorded by the Integron was coming from the stainless steel off-gas line. The riser in this line, located southeast of the 205 building, reached a maximum intensity of 1000 mr/hr. The steam ejector, located by the 200 Area stack, had a maximum measurement of greater than 1200 mr/hr.

706-C operation report that they neutralized approximately 1500 gallons of waste solution in a tank which has a maximum capacity of 1750 gallons. Apparently the freeboard was not sufficient to prevent the foam on the surface of the waste liquor from being sucked into the tank vent line and from there into the 200 Area stack.

Samples obtained from tanks W-1 and W-2 showed contamination of from 30,000 to 400,000 cts/cc/min. A sample of the material draining from the base of the 200 Area stack read over two million cts/cc/min.

The line between the oriface plate, located about twenty feet east of the jet and the 706 C building was successfully flushed with water. The intensity along the line between these points is now below 12 mr/hr. The jet, however, continues to read 300 to 400 mr/hr. Apparently the active material was baked in the line by the heat from the steam ejector. This portion of the line is being given an acid wash in an attempt to reduce the activity at this point.

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 APR 12 1945
 DIVISION OF CHEMISTRY
 U.S. DEPARTMENT OF ENERGY

Mr. J. P. Sinclair

-2-

April 12, 1945

In order to eliminate further contamination of this line during future runs in the 706 C building it was suggested to Mr. Levy that the waste liquor in the storage tank be neutralized and jetted to Well at least twice during the period of its accumulation, rather than waiting until the run is complete and neutralizing it all at one time. The suggested procedure will mean a several hour interruption of the dissolving operation but would also eliminate a definite hazard.

LEW:os


L. B. Enlet

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A-670

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4/26/45

Those Eligible
To Read the
Attached

Date _____ Weekly 706-D Report ~~XXX~~ for

Subject _____
Period 4/15/45 - 4/22/45

_____ J. R. Farmaker

By _____ E. Z. Morgan

To _____

3 R. S. Stone

64

Before reading this document, sign and date below

Name	Date
J. R. Farmaker	5/9/45

Name	Date

INV
64

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Daniel R. Hammin 1/31/95
Technical Information Officer Date
ORNL Site

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- 1. K.Z.Morgan
- 2. J.E.Wirth
- 3. R.S.Stone
- 4. W.A.Rodgers
- 5. M.D.Peterson
- 6. M.C.Leverett
- 7. W.H.kay
- 8. J.R.Farmakes
- 9. Central File
- 10. Readers File

consists of 2 figures.
 of 10 copies, Series A.

4/26/45

K. Z. Morgan

Health Physics

J. R. Farmakes

Health Physics

WEEKLY 706-D REPORT FOR
PERIOD 4/15/45 - 4/21/45

Daily checks of certain key points were continued to note any changes in activities of beams coming through 706-C wall. Marked decreases, as shown below, were observed.

	<u>4/16/45</u>	<u>4/17/45</u>
#1	15mr/hr to	3.8 mr/hr
#2	70mr/hr	17.5 mr/hr
#3	60mr/hr	11.5 mr/hr
#4	50 mr/hr	7.2 mr/hr

The writer was informed by R. A. Simons of 706-C that a shield had been installed. On 4/20/45 points 1, 3 and 4 increased about 50% and have remained at those levels. This was explained by a shifting of the shield preparatory to replacing it with another this week.

"Dummy" runs, D-1 and D-2, were started during the week. D-1 consisted of 50 low activity slugs processed through the first metathesis cake solution stage. Activity in Cell A was as follows: dissolver, 3.6 mr/hr at 2 inches; Waste solution tank, 8 mr/hr at 2 inches. The final cake solution (about 1 gallon) emitted 16 mr/hr at 2 inches thru a glass jug. D-2 involved 200 low activity slugs but a tracer solution was added and maximum activity in Cell A reached 60 mr/hr at 2 inches, this being at the dissolver. A 100 ml. sample gave 28 mr/hr at 4 inches. The cell is locked at all times and may be entered only with a supervisor's permission. Cell B has not been used in the process thus far.

The entire building and outside areas on the north and east sides were surveyed with a "walkie-talkie", after which "hot" spots were checked with a Lauritsen. Yellow trash can, placed in the center of the "sample garden" gave readings of 30-40 mr/hr at 3 inches. Apparently 706-C was responsible. Arrangements were made to have the can properly tagged and the contents disposed of. Readings directly above the opening in the concrete-block sample holder, east of 706-D were about 18 mr/hr. A danger tag was placed there.

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 For The Atomic Energy Commission

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Cell B showed no activity except for one small area on the floor which read 10 mr/hr at 8 inches; W.O. This was caused by a spill from a jug. Carbonate solution failed to remove contamination but the entire floor is to be covered with lead so no difficulties from this source are expected. The area north of the building has been covered with crushed rock and the slight surface activity has been further reduced.

All men working in 706-D take daily hand counts and no high values were obtained during the week. The building now has a probe and scaler in operation.

J. R. Farmakes

JRF/ah

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~~SECRET~~

April 26, 1945

1. W. C. Johnson
2. R. L. Doan
3. M. D. Whitaker
4. C. D. Coryell
H. A. Levy
5. W. E. Cohn
6. C. Files
7. R. Files

To: W. C. Johnson

From: W. E. Cohn

This document consists of 6
pages and 1 figures.
No. 1 of 7 copies, Series A

PROPOSED ADDITION TO 706-C

Recently I submitted to you a proposal to construct a somewhat larger semi-hot laboratory in the 706-C building to relieve the hazardous crowding to which Parker and his group are subjected in their high level fission product preparation work. That proposal was an emergency measure only; when I was asked if it would permit expansion of our activities, either as to amount of radioactivity handled per unit preparation or as to variety of preparations, I had to answer that it would not permit any expansion whatsoever.

It has been decided to expand my section (C-IV) by 50% as of July 1, since it is clear that both the variety and unit level of our active preparations are to increase. I have been instructed to state candidly what facilities are necessary in order that we may carry out our assignments and to correlate these with the facilities needed by related groups (Coryell - Levy) and to those of the Chemistry Division as a whole.

The attached plan for 706-C extension is the result of conferences between me and members of my section (particularly G. W. Parker and E. R. Tompkins both of whom have had a great deal of 706-C experience; G. W. Parker is in charge of our production group in that building and has worked continuously in the building for over a year), Coryell and Levy of C-II, and yourself. You will note that the additional space requested is about double that of the present 706-C building. This is not as shocking as it may sound at first. 706-C was designed without benefit of prior experience for about 10 men and not over 20 G of material and it is not to be used as a standard. Certainly it has exhibited many deficiencies which the proposed plan would alleviate. The 706-D building, designed to do one specific job which at present occupies half of 706-C, is also twice as big - showing that advantage has been taken of adverse 706-C experience in designing 706-D.

Our basic necessity is for semi-hot laboratory space; none which is suitable exists. Our second need is for ordinary laboratory space for our increased manpower, adjacent to the hot laboratory itself. A third objective is to increase the working and storage facilities of 706-C to proper size. Fourthly, we need to have our activities located in one group of laboratories for proper coordination. The proposed plan answers all of these needs.

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~~XXXXXXXXXX~~

W. C. Johnson

- 2 -

4/26/45

In the following pages I give a brief description of the facilities, their functions and the pertinent arguments relating to their size, location and special features.

I feel that this plan is a reasonably conservative statement of what C-IV needs to carry out its assignments and accommodate its projected manpower after July 1. Allowances are made for the needs of other groups of the Division for "hot-lab" facilities.


W. E. Cohn

WEC/dd

~~XXXXXXXXXX~~

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PURPOSE OF PLAN

1. To provide adequate and safe semi-hot facilities for Chemistry Section C-IV (Radio-isotope production section).

Present Status: 706-C semi-hot laboratories are completely inadequate and hazardous for the present level of C-IV's work. The proposed expansion of C-IV manpower is contingent upon more semi-hot space; continued operation even at present levels demands adequate facilities, at present non-existent.

Influence of 706-D Startup: Will render present semi-hot laboratories more useful to other chemistry groups; it will not solve C-IV's problem as these laboratories are not large enough nor of proper design for its work.

2. To provide adequate laboratory and office space for new research and process development groups to be set up in Section C-IV on July 1.

Present Status: One two-man laboratory, occupied by three men, and one office in 706-A; these are too far from 706-C and from each other to permit adequate coordination. Influx of at least four additional men on July 1 is not possible without additional laboratories. For proper coordination and efficient utilization of both brain and manpower, these laboratories and offices must be centralized in one unit.

Influence of 706-D Startup: Will release part of the one laboratory now in 706-C. This laboratory is not large enough for the three other groups of C-IV and is needed by other chemistry groups for their work. No office space will be released.

3. To provide adequate semi-hot, laboratory and work facilities for other chemistry groups dealing with high levels of activity.

Present Status: No space available except for Ba job. Work facilities grossly inadequate.

Influence of 706-D Startup: Will provide adequate space (except for work space) for the chemistry groups other than C-IV if C-IV is not competing for the same space. Work space will remain inadequate.

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4. To provide adequate storage space for both hot and cold materials in 706-C.

Present Status: Inadequate to the point of constituting definite safety and health hazards.

Influence of 706-D Startup: Nil.

REASONS FOR EXPANDING 706-C RATHER THAN 706-A

1. Semi-hot Facilities: Must be adjacent to and easily accessible from hot cells if hazardous and time-consuming transport problems are to be avoided; must be separated and shielded from regular laboratories; must be properly designed on the basis of past experience (no such facilities are extant).
2. Coordination of all activities of C-IV: Present arrangement of 706-A prohibits coordination in this location; obvious necessity of putting semi-hot laboratories next to and on same floor level as 706-C, and near 105 unit, makes 706-C the logical choice for such centralization; shortage of trained manpower indicates that maximum utilization of same will be achieved only by close contact of all members of section; work of all groups in the section must be closely correlated to prevent wasted effort.
3. Provision of adequate space for groups other than C-IV working with hot materials: Should be located adjacent to hot cells of 706-C and away from cold operating areas.
4. Work facilities (glassblowing, machines, etc.): These are principally geared to the construction of apparatus within the hot cells themselves and must be immediately accessible from this area.

Summary: Work related to and dependent upon the hot cells must, for efficiency and safety, be performed near these cells. Transport of activity must be minimized. Active areas must be segregated from non-active ones.

4/26/45

NECESSITY OF FACILITIES REQUESTED

1. Semi-hot Laboratories: This is the most pressing demand. The present laboratories are suitable for their original purpose: 1 man (in each of the four laboratories) not over 100 mC activity per laboratory, simple, routinized procedures. They are grossly and hazardously unsuited for the present and projected operations, which involve 5-10 men, up to 1000 mC activity, varied and varying procedures. Further, an unforeseen need has arisen for additional space within each laboratory for such things as H-P and counting instruments, hot balances, dish-washing facilities, hot and cold storage, etc. At the present level and constancy of operations, a certain amount of space must be provided so that these men who must remain in these laboratories continuously can protect themselves from the activity being processed. It has also been shown that the present laboratories are not sufficiently isolated from each other or from other areas.

The design submitted for the semi-hot block has the following features:

- a. Two large semi-hot laboratories, specifically designed and arranged for continuous semi-hot operations on the curie level. Each of these may be occupied by 3 men working on as many preparations. Benches are to be raised and shielded to give complete protection. These laboratories are for the production group of C-IV.
 - b. Two smaller semi-hot laboratories, one for intermittent use by the other three groups of C-IV for specific hot jobs, the other for use by other groups of the Chemistry Division on the same basis. Raised benches and adequate shielding are provided.
 - c. Service room (dish-washing, storage) and hot storage vaults, for general usage. No adequate hot storage facilities exist now and these are urgently needed.
 - d. Storage and machinery space on top of the concrete block, which will also have removable slabs for the entry of heavy shields, etc., are provided for.
2. Laboratories: After July 1, C-IV will have three groups of 2-3 men each besides the production groups of 5 men. These will be responsible for the development of 1) pile reactions, 2) chemical separation methods and 3) remote-control apparatus and techniques. Each will require a laboratory and access to an office; in addition, close contact with each other and with the production group is essential since their activities are largely in series and not in parallel with each other.

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4/26/45

(G-IV has at the present one laboratory far removed from the one office and from 706-C. This is highly unsatisfactory for the prosecution of the work in hand and is unworkable following section expansion).

The laboratories assigned in the sketch to groups II, III and IV are more or less conventional laboratories in size and design. They are located with reference to the overlapping and correlation of the work of each. Each laboratory has an adjoining office.

It has been shown in the past that one laboratory is needed as a service adjunct to the hot cells. Therefore, one additional laboratory must be provided for other groups who have work to do in 706-C (at the present, Levy's group is the only known example, hence the new laboratory and office in the old structure is labelled with his name).

3. Offices:

- a. G-IV Section Office (Cohn) - adjacent to working areas
- b. Levy's office - adjacent to laboratory
- c. Garber and H. P. Office - for building manager and health physicists

4. Work Facilities: The present shop and glassblowing room, necessarily located near the cells, are much too crowded for good work. In addition to enlarging these, certain space has been provided for other construction work, for drawing and designing, etc., all of which are not provided for at present.

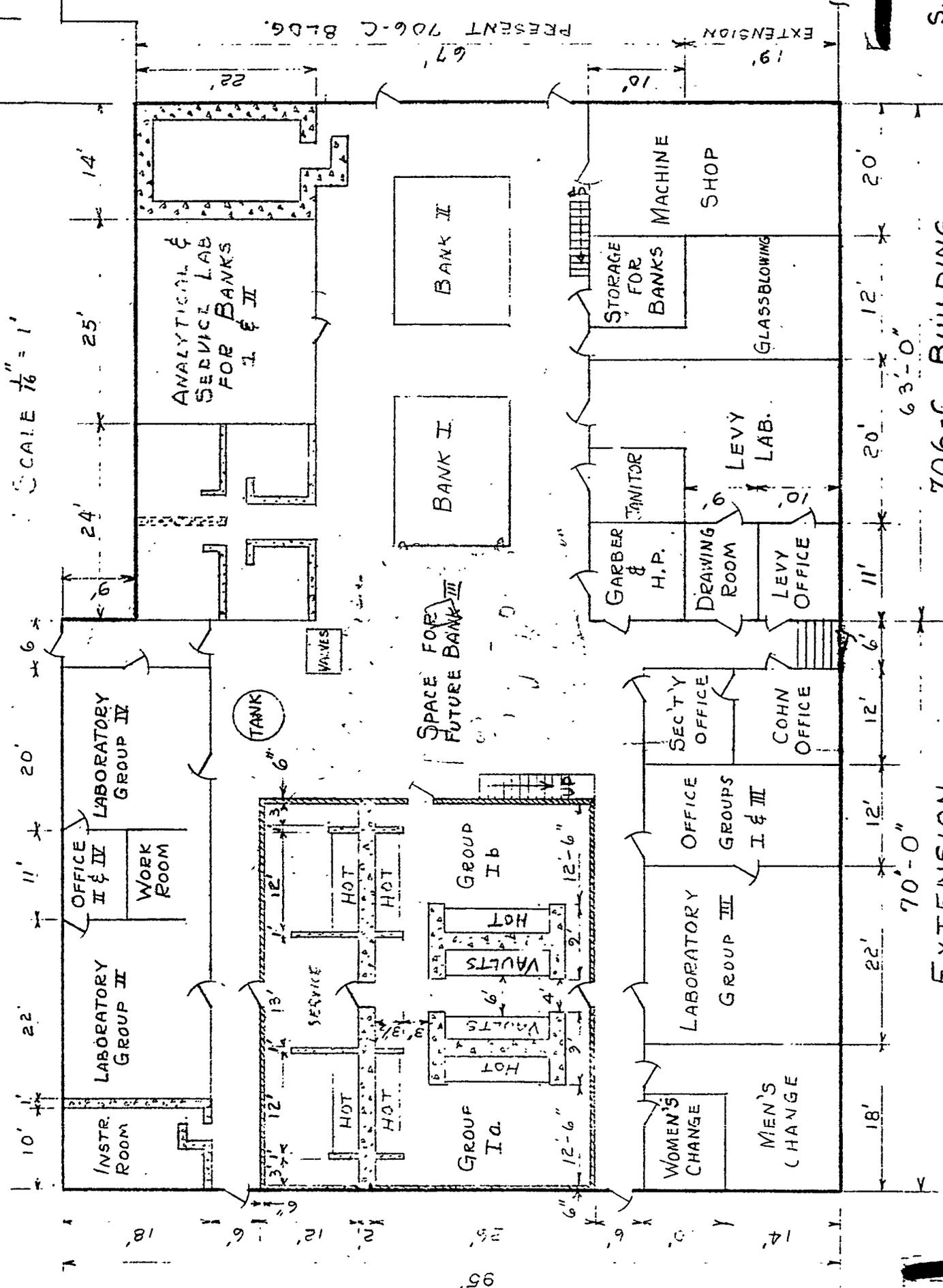
5. Change Rooms: These are required since the 706-D change room is not suitable either as to size or convenience, and does not have provisions for women occupants of 706-C.

6. Instrument Room: Required because of 706-D use of the present 706-C instrument room and to house other instruments (balances, colorimeters, etc.) which would overcrowd the other instrument room.

7. Storage and Janitorial Facilities: Badly needed in present structure.

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PROPOSED EXTENSION OF 706-C BUILDING



PRESENT 706-C BLDG. EXTENSION

S.W.
4-25-45

706-C BUILDING

EXTENSION

CONVENTIONAL LAB. AND OFFICE FURNISHINGS NOT SHOWN

45-5-247

A-670

File

Those Eligible
To Read the
Attached

Date May 22, 1945

Subject SAMPLES FROM 706-D

By Harrison S. Brown

To M. C. Leverett

#2
R. L. Doan

Before reading this document, sign and date below

Name	Date
	<u>MAY 22 1945</u>
<u>R. L. DOAN</u>	
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____

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

David R. Hamlin 11/30/95
 Technical Information Officer Date
 ORNL Site

2 7

a

5/22/45

- 2. R. L. Dean
- 3. W. C. Johnson
- 4. C. D. Coryell
- 5. J. M. Siegel
- 6. Reading File
- 7. Central File

M. C. Leverett

H. S. Brown

SAMPLES FROM 706-D

We in Chemistry would find it very helpful if suitable arrangements could be made for removing small samples of UNH solution from your dissolver in the 706-D Building. With the closing down of the 706-C engineering operations, 706-D will represent the only convenient source for really hot solutions.

We visualize fairly continuous need in the future for samples of the order of 5 cc in volume. If a reasonable arrangement can be decided upon, we would like to request, at the present time, five samples of 3-4 ml each of UNH solution from the first batch of active slugs dissolved in 706-D. This material is to be used by Mr. J. M. Siegel of Mr. Coryell's Section.

Your cooperation would be greatly appreciated.

H. S. Brown

CLASSIFICATION CANCELLED
 DATE 31 July 1952
 For The Atomic Energy Commission
 Chief, Classification Branch

JHK

~~CONFIDENTIAL~~
~~CONFIDENTIAL~~
~~CONFIDENTIAL~~
~~CONFIDENTIAL~~
~~CONFIDENTIAL~~



POST

1. M. C. Leverett
2. W. A. Rodger
3. W. A. Rodger
4. F. G. Rehm
5. M. D. Peterson
6. Central File
7. Reading File

17-5

TO: M. C. Leverett

FROM: M. D. Peterson

June 6, 1945

SPECIFIC GRAVITY OF H₂SO₄-HNO₃ MIXTURES

Sax has determined the specific gravities of H₂SO₄-HNO₃ mixtures in the neighborhood of the 65% H₂SO₄-2% HNO₃ solution used in the 706-D extraction process, at 25, 40 and 55°C., for use in the analysis of the mixed acid:

<u>% H₂SO₄</u>	<u>% HNO₃</u>	<u>Sp. gr. (60°/60°F. hydrometer) at</u>		
		<u>25°C</u>	<u>40°C</u>	<u>55°C</u>
55	2	1.458	1.445	1.436
	4	1.459	1.448	1.437
65	0	1.558	1.549	1.540
	2	1.559	1.546	1.537
75	2	1.659	1.647	1.638

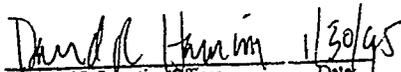
The specific gravities of the solution with 2% HNO₃ are plotted in the attached graph.

The change in specific gravity with HNO₃ concentration in the range 0 to 4% HNO₃ is entirely negligible for the proposed use - a 1% change in HNO₃ concentration causes less change in specific gravity than does a change in H₂SO₄ concentration of 0.2%, or in temperature of 3°C.

As of now discontinue the practice of having 65% sulfuric analyzed. Use the sp. gr. instead and add HNO₃ by weight.

M. D. PETERSON

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


 Technical Information Officer Date 1/30/45
 ORNL Site

DON'T SAY IT — WRITE IT

TO I. S. JOHNSON - I. H. BROWN

DATE 3/27/45

FROM I. I. BALDWIN

It is believed that the introduction of iron into 706-0 process solutions can be materially reduced by adding 2% H_2O_2 to the dilute H_2SO_4 used for the extraction precipitation. The 6% H_2O_2 coming into contact with hot stainless steel (slinger ring) may have caused excessive corrosion. Nitric acid (2%) has been found a satisfactory inhibitor even at 90° C.

DON'T SAY IT — WRITE IT

E. J. Johnson

TO W. A. Rodger

DATE May 16, 1945

M. D. Peterson

RE:

HNO₃ Concentration in Electrolysis

In our earlier electrolysis experiments it was shown that increasing HNO₃ concentration resulted in less reduction to metallic Pb and better dioxide plate, but we were limited to about 2.5N-HNO₃ by danger of precipitation of Pb(NO₃)₂ in the 1-1/4 and 2-1/2 liter electrolysis solutions then used. With the present 4-liter electrolysis solutions, such precipitation is not likely at 3M-HNO₃. Therefore, please use this increased HNO₃ concentration in all subsequent 706-D electrolyses. The semi-works has also made this change.

The literature on analysis of Pb by electrolysis to PbO₂ also indicates that 3N is the optimum HNO₃ concentration.

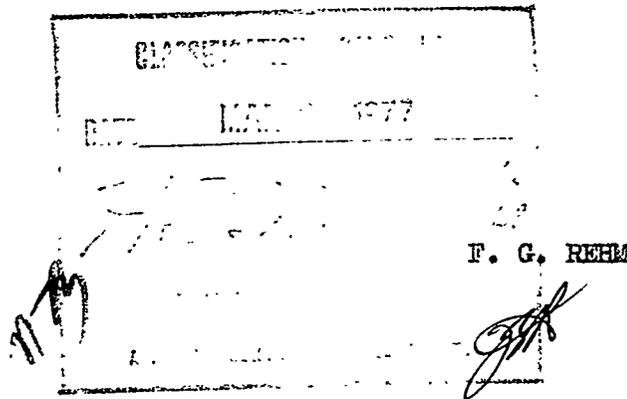
1. ~~M. C. Leverett~~
2. ~~M. D. Peterson~~
3. ~~R. Semmes~~
4. W. A. Rodger
5. ~~F. G. Rehm~~

March 16, 1945

PROPOSED 706-D CHEMICALS SPECIFICATIONS

Attached are proposed specifications for chemicals that will be used in 706-D operation and subsequent clean-ups. An estimation of the amounts of each necessary for ten runs is also shown.

The list is submitted for your comments relative to availability and standards of purity of chemicals suggested.



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PROPOSED 706-D CHEMICALS SPECIFICATIONS

1. BARIUM NITRATE ($\text{Ba}(\text{NO}_3)_2$) - Crystalline, C.P. grade

1 pound glass jars

Assay	99.00% Min.
Water insolubles	0.002% Max.
Chlorides (as Cl)	0.0005% Max.
Ca & Sr salts (as Cl)	0.05% Max.
Iron (as Fe)	0.0002% Max.
Heavy metals (as Pb)	0.0005% Max.

2. HYDROGEN PEROXIDE - (H_2O_2) 30% reagent grade

1 pound safety-cap bottles

Assay	30.0 ± 0.5% H_2O
Non-volatile matter	0.05% Max.
Chlorides (as Cl)	0.0002% Max.
Sulfates (as SO_4)	0.03% Max.
Phosphates (as PO_4)	0.009% Max.

3. POTASSIUM CARBONATE K_2CO_3 , Crystalline, reagent grade

25 pound drums

Assay	97.00% Min.
Chlorides (as Cl)	0.003% Max.
Water insolubles	0.01% Max.
Iron (as Fe)	0.0005% Max.
Heavy Metals (as Pb)	0.0005% Max.

4. LEAD NITRATE - $\text{Pb}(\text{NO}_3)_2$, Crystalline, C.P. grade

5 pound glass jars

Assay	99.00% Min.
Water insolubles	0.005% Max.
Chlorides (as Cl)	0.001% Max.
Iron (as Fe)	0.0001%

5. SODIUM BICHROMATE - ($\text{Na}_2\text{Cr}_2\text{O}_7$), fine crystalline, reagent grade.

5 pound glass jars

Assay	98.00% Min.
Water insolubles	0.005% Max.
Chlorides (as Cl)	0.005% Max.
Sulfates (as SO)	0.010% Max.
Calcium (as Ca)	0.005% Max.
Iron (as Fe)	0.0005% Max.

6. SODIUM CARBONATE (Na_2CO_3) crystalline, Technical grade

100 pound sacks

Assay	98.0% Min. (dry basis)
Water insolubles	0.05% Max.

7. SODIUM PHOSPHATE, TRIBASIC - ($\text{Na}_3\text{PO}_4 \cdot 12\text{H}_2\text{O}$), crystalline, Tech. Grade

100 pound sacks

Assay	99.0% (as $\text{Na}_3\text{PO}_4 \cdot 12\text{H}_2\text{O}$)
Water insolubles	0.010% Max.

8. SODIUM NITRITE - (NaNO_2), crystalline, reagent grade

5 pound glass jars

Assay	97.00% Min.
Water insolubles	0.005% Max.
Chlorides (as Cl)	0.001% Max.

9. SODIUM HYDROXIDE - NaOH, pellets, C.P. Grade

5 pound glass jars

Assay	97.00% Min.
Chlorides (as Cl)	0.001% Max.
Iron (as Fe)	0.002% Max.
Carbonates (as Na_2CO_3)	2.0% Max.
Heavy metals (as Hg)	0.005% Max.

10. SODIUM HYDROXIDE - NaOH, 50% liquid caustic, commercial grade

55 gallon steel drums

(no specifications)

11. NITRIC ACID, (HNO₃), C.P. reagent grade

7 pound screw-cap bottles, (or glass stoppered)

Assay (HNO ₃)	70.5 ± .5%	90 - 96%
Chlorides (as Cl)	0.0005% Max.	0.0005% Max.
Sulfates (as SO ₄)	0.0001% Max.	0.0001% Max.
Heavy metals (as Pb)	0.0005% Max.	0.0002% Max.
Iron (as Fe)	0.0001% Max.	0.0001% Max.
Nitric Oxides (as NO ₂)	---	---

12. NITRIC ACID (HNO₃), Commercial grade

12 gallon carbuoys

Assay (HNO ₃)	59 - 61%
Chlorides (as Cl)	0.05% Max.
Nitric oxides (as NO ₂)	0.05% Max.
Non-volatiles	0.02% Max.

13. SULFURIC ACID (H₂SO₄) - Technical grade

12 gallon carbuoys

Assay	91. % Min.
Sediment	0.005% Max.

QUANTITIES NEEDED FOR 10 FULL-SCALE RUNS

1. Barium Nitrate	1 pound
2. Hydrogen peroxide	3 pounds
3. Potassium carbonate	50 pounds
4. Lead nitrate	6 pounds
5. Sodium dichromate	7 pounds
6. Sodium carbonate	25,000 pounds
7. Sodium phosphate	20,000 pounds
8. Sodium nitrite	15 pounds
9. Sodium hydroxide, C.P.	15 pounds
10. Sodium hydroxide, liquid 50%	2,000 pounds
11. Nitric acid, CP 70%	22 pounds
" " , Fuming	39 pounds
12. Nitric acid, Technical - 60%	25,000 pounds
13. Sulfuric acid	9,200 pounds

DON'T SAY IT — WRITE IT

TO WAR
FROM M. D. PETERSON

DATE 3/3/45

SPECIFICATIONS FOR 706-D MATERIALS

Check specifications for all materials to be used in 706-D operation. The present specifications should be available from Semmes, or the Analytical Division files. Either O.K. these for each material, or make changes as required. Set up to check materials for whatever specifications may be critical.

TO:

1. E. D. Peters
2. A. A. Rodger
3. T. H. Baldwin
4. E. C. Johnson
5. F. G. Rehm

FROM: E. D. Peterson

March 7, 1945

Present 706-D MATERIAL SPECIFICATIONS

(from R. Semmes through MCL)

1. $\text{Na}_2\text{Cr}_2\text{O}_7$		98.0% min.
	Cl	0.05% max.
2. NaNO_2		97.0% min.
	Cl	0.01% max.
	insol.	0.1% max.
3. $\text{Ba}(\text{NO}_3)_2$		99.0% min.
	Cl	0.1% max.
	insol.	0.01% max.
4. H_3PO_4		70.0% min.
	Cl	0.01% max.
5. Na_3PO_4		"Commercial Grade"
6. HNO_3		60% min.
	NO	0.05% max.
	Cl	0.05% max.
	Non-vol.	0.02% max.
7. H_2SO_4		91.0% min.
	sediment	0.005% max.
		(battery acid grade)
8. Na_2CO_3		98.0% min. (dry basis)
	insol.	0.05% max.

706-D Operation:

Amt. of Raw Material Needed for 10 (Chromite) lbs.

1	H ₂ SO ₄ Tech, 95%	9,180.0 #	
2	HNO ₃ Tech, 60%	12,838.0 #	
3	C.P., 70%	21.6 #	
4	Ammonia, 95%	39.1 #	
5	NaOH Tech, 50%	1,203.0 #	
6	C.P., pellets	13.8 #	
7	Na ₂ CO ₃ Tech, solid	18,200.0 #	
8	Na ₂ PO ₄ · 12 H ₂ O Tech, solid	15,280.0 #	
9	Na ₂ NO ₂ C.P., Crys	12.7 #	
10	K ₂ CO ₃ " , "	50.0 #	
11	Na ₂ Cr ₂ O ₇ " , "	6.5 #	
12	Pb(NO ₃) ₂ " , "	5.7 #	
13	Ba(NO ₃) ₂ " , "		10 gms.
	<u>Total</u>	56,850.4 #	

WFB 3/13/45

January 30, 1945

L. D. Peterson

W. P. Bieker

SUMMARY OF REPORT ON L¹⁴⁰ PREPARATION IV
IN 706-~~6~~

This preparation produced the largest yield to date through the engineering side. Had not an apparatus failure on the glass side occurred, it probably would have been the largest source yet produced around 500 c. Due to this failure, approximately 200 c. were shipped. A subsequent run (v) was started to supplement run IV after a few days clean-up was affected.

The steps were as follows: Coating removal, dissolving, $\text{SO}_4 =$ precipitation, metathesis, K_2CO_3 volume reduction step, La separation with f. HNO_3 , Pb CrO_4 precipitation, 2nd f. HNO_3 , Ether-HCl separation, and evaporation. In general, the chemistry is very straight forward. They did find, however, that the reverse strike produced higher yields. Also, increasing the agitation and settling time and cooling slowly seems to indicate greater yields by cutting the loss in UNH supernate from 20% to 10% Ba.

The equipment as it stands does the job but requires excessive technique and care, is definitely hazardous from all standpoints, and doesn't give as good a yield as the chemistry of the process indicates. The transportation and discharging is adequate except for possible radiation in removing and replacing the slug-tube shielding plug. The dissolver is suitable except that the coating removal by acid is intrinsically violent. Solution transfer by jets could possibly be improved if there were strainers on the suction side. Agitation and off gas elimination is very poor in the precipitator. In either case, far from sufficient. The neutralizer needs adequate venting and perhaps scrubbing, agitation, a thermocouple, and some method of cooling before jetting.

The sintered glass filter outside Cell #4 is quite a millstone from the safety standpoint. The six storage tanks atop of Cell #4 is a necessary evil. They too are very hazardous and the operator of them is subject to over-exposure. The glass side is obviously fragile. Also, the control and operating devices on the glass side are highly complicated, offering many chances for mistakes and apparatus failures. The storage and handling of reagents is suitable except the HNO_3 storage tank, which is unsafe to charge and collects dirt and debris.

Finally, regarding safety and radiation, in defense of the present program, we may say that activity levels are too high, initial shield-

ing inadequate, and aisles too congested, mainly due to building design. However, the failure to adopt a good safety program permits hazards which could be avoided or decreased. The following are outstanding: Poor housekeeping, exposed sampling methods, no H.F. schedule enforcement, unsafe storing and shielding with bricks, flagrant omission of gloves, face guards and glasses, and occasional unnecessary over-exposures.

W. F. BIGLER

pages and 0 figures.
No. 1 of 6 copies, Series A

~~SECRET~~
CLINTON LABORATORIES

- 1. M. C. Leverett
- 2. W. A. Rodger
- 3. M. D. Peterson
- 4. R. K. Harris
- 5. R. B. Briggs

To M. C. Leverett

DATE 6/6/45

DEPARTMENT Technical, Director

FROM R. B. Briggs

DEPARTMENT Technical, Section II

IN RE: Discharge of Waste from 706-D Building

CLASSIFICATION CANCELLED

DATE 2/24/66

For The Atomic Energy Commission

H. R. Caswell
Chief, Declassification Branch

Due to the large amount of active waste discharged from 706-D Building during the first production run, certain difficulties have arisen in the operation of the 206 Area which will require the institution of some changes in procedure during future runs. Addition of calcium chloride to the waste tanks is giving little or no decontamination with some 706-D wastes, and the decontamination factor obtained in the Settling Basin is less than 4 at the present time. The present procedure of permitting W5 to overflow to W6 while jetting continuously from W6 is resulting in the discharge of some materials of relatively short half-lives, the activity of which could be reduced by proper hold-up in W5, and at times in the discharge of very active material from W6 which could be decontaminated in W5 if sufficient time were available.

Experience of the past two weeks has shown that 10,000 gal/day of material can be discharged from W6 without difficulty if the activity is no greater than 400,000 disintegrations/cc/min. Because some decontamination is achieved by hold-up in the tanks, and because the permissible activity of the outlet from the Settling Basin has not been exceeded at the given discharge rate, it is believed that an average of 10 to 15 curies per day can be received from 706-D operations. The total volume of liquid discharged should be kept below 10,000 gal/day and lower flows should be used, if possible, to permit longer hold-up times.

Since the main source of active material discharged to W11 and then to W5 has been process losses of 706-D product, it is requested that all process waste solutions be discharged to W9. This discharge should be direct, if possible, but when not possible, arrangements can be made for the 206 Area operators to handle the material thru W11. It is understood that the total volume of the product wastes is small (20% or less) compared to the volume of neutralized metal waste now discharged to W9, so there will be a small reduction in the number of runs which can be made without exceeding the capacity of the metal waste tanks.

In the 206 Area it is planned to revise the discharge procedure by jetting from W5 to W6 and from W6 to the Settling Basin to give better control over decontamination. The dip pipes on the jets from both tanks will be shortened so solids will not be discharged from either tank.

This document contains information affecting the national defense of the United States within the meaning of the Espionage Act, D. C. Code, Title 18, Section 793, and its transmission or the revelation of its contents in any manner to an unauthorized person is prohibited by law.

M. C. Leverett

W5 will be scavenged continuously and the supernatant will be jettied to W6 only on days when the liquid is near the overflow level, and when the amount of activity entering the tank is low. To make this procedure most effective it will be necessary for the 206 Area supervisor to have a daily forecast of the amount of activity which will be discharged to W11 during each shift. The forecast can be given to the area operator with the waste volume forecast on the 8-4 shift.

When the present situation has cleared, it may be possible to send more of the waste solutions to W5; thus, conserving the limited amount of space (87,000 gallons on 6/6/45) in W9. The procedures outlined above will be revised at that time.

R. B. B.

R. B. Briggs

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~~SECRET~~

A-670

File 45-6-107

6/12

Date _____

Those Eligible
To Read the
Attached

Subject 6/8/45

Report on Hot Run # 1, 706-D Bldg.

Copy # _____

By for Period 5/28/45 thru 6/2/45

To R. A. Simons

K. Z. Morgan

Before reading this document, sign and date below

59
ANN

Dec 10/0
23
12-3-6

Name	Date	Name	Date
<i>[Signature]</i>	<i>6/12/45</i>		

INV. 64

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

David R. Hamm 1/31/45
Technical Information Officer Date
ORNL Site

This document consists of 6 pages and 0 figures. No. 2 of 10 copies. Series A

~~SECRET~~

- 1. K.Z.Morgan
- 2. J.E.Wirth
- 3. R.S.Stone
- 4. M.C.Leverett
- 5. W.Rodger
- 6. M.D.Peterson
- 7. W.H.Ray
- 8. R.A.Simons
- 9. Central File
- 10. Readers File

6/8/45

K. Z. Morgan Health-Physics
 R. A. Simons Health-Physics

REPORT ON HOT RUN #1, 706-D BLDG.
FOR PERIOD 5/28/45 THRU 6/2/45

The first dissolving started on the morning of 5/28/45. At the time 200 slugs were in the dissolver and 50 slugs were dissolved at a time making a total of four batches. A typical survey of the slug bucket containing 26 slugs is as follows:

Front of bucket at 1'	200	mr/hr
Right side at contact	150	"
Left side at contact	100	"
Back at contact	85	"
Top at contact	25	"
Bottom at contact	150	"

It was found that in unloading the first load of slugs beams as great as 2 r/hr would come out of the cracks between the sliding lead plug and the walls of the carrier. The bucket was later redesigned so that the highest beam was 350 mr/hr next to the carrier. No trouble was encountered as far as discharging the slugs into the dissolver is concerned.

In regard to Health, principle trouble during the run came from the fan house, sampling, vent duct, and off gas lines, although three lines leading to panel board 3 became hot. These lines are pressure and density lines. Their highest activity was about 35 mr/hr at contact. The wall plugs were continually surveyed but no activity leaks were discovered. The highest activity through the cell walls was on the west side of Cell B second level during the electrolytic separation. The maximum activity at this point was about 10 mr/hr excluding the background from sampler B 1.

The fan house floor became contaminated (500 mr/hr soft beat at 4") because of a leak from the small fan housing. Hot condensate was collecting in the fan which sucks up fumes from all the cell tanks except the dissolver. The fan housing read >1 r/hr at contact and was replaced by another fan. A cyclone separator was also installed to catch the condensate in a trap so that it cannot be collected by the fan housing. At the end of the run the small fan off gas line had a maximum activity of 75 mr/hr at the fan house, 35 mr/hr before going into the fan house and 15 mr/hr where it comes out of Cell A.

7/24/46
 U.S. Atomic Energy Commission
 Health Physics Division

This document contains information affecting the national defense of the United States within the meaning of the Espionage Act, Title 18, U.S.C., Sections 793 and 794, and the transmission or the revelation of its contents in any manner to an unauthorized person is prohibited by law.

~~SECRET~~

~~SECRET~~

6/8/45

-2-

The 2' diameter cell vent duct at the end of the run read 80 mr/hr where it comes out of the cells and 120 in the fan house. This duct read between 80 and 120 mr/hr along its whole length. The emergency block iron off gas line which runs along the north side of the building read between 15 and 75 mr/hr at contact along its whole length. On the north side of the building inside the activity was 20 mr/hr due to this line. It has been suggested that the vent ducts and off gas lines area be fenced in and danger signs posted. Whether this will be done remains to be seen.

B-1, B-3 and B-6 samplers were reading above tolerance during the run. Highest readings at contact with the lead shield were:

B-1
B-3
B-6

75 mr/hr
50 "
80 "

Danger signs were posted around the samplers. These samplers were contaminated on the inside due to improper washing after the sample had been taken. Although no spills occurred at any of the samplers on the ground or 2nd levels the floor around B-1 sampler became contaminated up to 1300 c/m and around B-3 and B-6 samplers 1940 c/m. The floor around the other samplers on these levels were > 200 c/m. On the 3rd level the floor became highly contaminated around B-9 and B-17 probes. Smears were as high as 150 mr/hr. The floor now reads between 25 mr/hr and 100 mr/hr in this area in spite of several washings. Probe handles also became contaminated due to samplers touching the handling with hot gloves. Inside the shields of these probes the surface is highly contaminated (> 1000 mr/hr in B-17 probe). Most of the trouble encountered in sampling was due to lack of preparation before sampling and carelessness. The sampling technique has been improved however, and radiation doses as well as contamination should be greatly reduced on the next run. A great deal of the radiation received by samplers can be attributed to the over-curiosity of Health-Physics. It has been the custom in 706-C to take an electroscop reading on the samples before it is placed in shielded carrier, thus slowing up the transfer of hot samples from the sampler to the carrier and increasing the dose to personnel. This reading has now been eliminated so that an electroscop reading is taken only after the sample is shielded. The doses received by the samplers during the run as measured by film pack are as follows:

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~~SECRET~~

~~SECRET~~

6/8/45

-3-

<u>NAME OF SAMPLER</u>	<u>DATE</u>	<u>LOCATION</u>	<u>nr</u>
Levenson	5/28/45	R. wrist	70
		L. wrist	70
		Crouch	70
		Chest	70
		Cap	75
Pressly	5/28/45	R. hand	80
		L. hand	70
Newton	5/28/45	R. hand	70
		L. hand	80
Levenson	5/29/45	R. wrist	150
		L. wrist	150
		Cap	175
Levenson	5/30/45	R. wrist	110
		L. wrist	115
		Cap	125
Levenson	5/31/45	R. wrist	50
		L. wrist	30
		Cap	40
McLellan	5/31/45	R. wrist	120
Phillips	5/31/45	R. hand	150
		L. hand	170
		R. wrist	190
		L. wrist	150
		Cap	120
Bailey	5/31/45	R. hand	170
		L. hand	210
		R. wrist	150
		L. wrist	130
		Cap	110
Havorka	6/1/45	R. hand	30
		L. hand	130
		R. wrist	50
		L. wrist	50
		Cap	25
Dunlap	6/1/45	R. wrist	25
		L. wrist	30
		Cap	20
Weil	6/2/45	L. wrist	170
		R. wrist	190
		L. hand	210
		R. hand	210
		Cap	160

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Men working with the samples in the hot labs. received the following doses:

Name	Location	mr				
		5/28	5/29	5/30	5/31	6/1
Collins	R. hand	30	20			25
	L. hand	20	20			25
	Cap					30
	L. wrist					20
	R. wrist					20
Bernstein	R. hand	40				60
	L. hand	50				110
Rehm	R. hand	40		120		
	L. hand	30		130		
Reynolds	R. hand					90
	L. hand					85
Magnuson	R. hand	50				
	L. hand	40				

The monitron and integron chambers read as follows during the run. This tabulation does not account for momentary increase in activity due to the opening of 706-C filter disk or carrying hot samples and hot equipment by the chambers.

Location of chamber	Chamber	First part of run	Last part of run
Monitrons:			
Hot lab. #1	A	1 mr/hr	2 mr/hr
Cell A entrance	B	1 "	5 "
West side of cells	C	2 "	7 "
Above cells	D	1 "	6 "
Cell B entrance	E	2.5 "	5 "
Equipment decontamination room	F	7 "	17.5 "
Hot lab. #2	G	4 "	25 "
East side of cells	H	2 "	14 "

Integrans:

Ground floor SW corner of cell A	1	3 mr/8 hrs.	9 mr/8 hrs.
Panel board No. 1	2	15 "	25 "
2nd. level west side of cells	3	20 "	50 "
3rd level south end	4	2 "	4 "
3rd. level north end	5	10 "	40 "

Monitron C and integron No. 3 read high during the latter part of the run because of sampler B-1. Monitron D which is above the cells read high because of contamination around B-12 and B-19 samplers above

cell B. The increase in monitron D's reading cannot be attributed to contamination from the 706-C Bank 2 stack since 706-C was not running at this time. Monitron F at the equipment decontamination room read high because of the hot off gas fan which was being decontaminated in this room. Monitron G located in hot lab. #2 read high because of hot pipettes which were stored behind a brick shield near the chamber during the latter part of the run. Monitron H and Integron 2 read high because of samplers B3 and B6. Integron 5 read high because of contamination at B-19 polarograph column.

Precipitron air samples in 706-D counted about the same during the run showing that there was no appreciable increase in air contamination as the run progressed. Tolerance for alpha is 1.9 d/m per cu. ft. of air or 5×10^{-10} μ gms./cc of air or 3.1×10^{-11} μ curies per cc of air. Tolerance for Beta and Gamma in the air depends on the type of contamination. Anything over 1000 c/m beta and gamma in 706-D would be considered high by the author. Precipitron results are as follows:

<u>Date</u>	<u>Location</u>	<u>Time for run</u>	<u>α % of tolerance</u>	<u>B c/m</u>	<u>β & γ c/m per liter of air</u>
5/28/45	3' W. of A-1 sampler on ground floor	1 hr.	0%	85	8.7×10^{-3}
5/30/45	W. side of Cell A near Panel board 3	1 hr.	5 hr. decay 4%	93	9.5×10^{-3}
5/30/45	W. side of Panel Board 5 (cell B blocks removed)	2 hrs.	19 hrs. decay 1.4 %	102	12.6×10^{-3}
5/31/45	2nd level 3' from B-1 sampler	2 hrs.	21 hrs. decay 1.8 %	92	9.4×10^{-3}
6/1/45	2nd level E. side between B-3 and B-6 samplers	$\frac{1}{2}$ hr.	$3\frac{1}{2}$ hrs. decay 12.2 %	75	7.7×10^{-3}
6/2/45	E. side of fan house near contamination	$\frac{1}{2}$ hr.	-	536	54.9×10^{-3}

For a more accurate alpha count the sample should be allowed to decay at least 24 hours. The alpha percentages on 5/30/45 and 6/1/45 would probably have been much lower if the sample had been allowed to decay 24 hours. In the future this will be done except in special cases.

On 6/2/45 two precipitron samples were taken at guard tower #2 which is about 100 yds. E of the 706-D cell ventilation stack and cell tanks off gas stack located at the fan house. Results are as follows:

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Sample #1 on 6/2/45 between 9:05 A.M. and 9:35 A.M.

Alpha
β & γ

.5% of tolerance
39 c/m or 4×10^{-3} c/m per liter of air

Counts were taken at 8:50 A.M. on 6/4/45

Sample #2 on 6/2/45 between 9:36 A.M. and 2:51 P.M. Length of run was 5 1/4 hours.

Alpha
β & γ

.2% of tolerance
17 c/m or 1.7×10^{-3} c/m per liter of air.

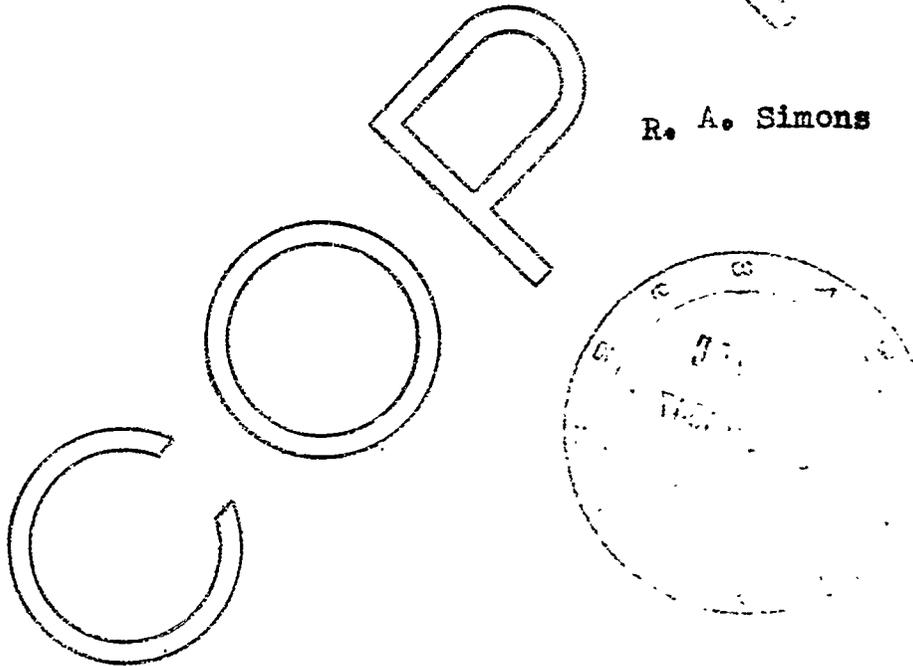
Counts were taken at 8:55 A.M. on 6/5/45.

General floor smears are less than 200 c/m as measured on a foot counter.

The final product is still in Cell B in the cene.

R. A. Simons

RAS/ah



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P. O. BOX 1663
SANTA FE, NEW MEXICO
June 21, 1945

CLASSIFICATION CANCELLED

DATE 2/24/66

For The Atomic Energy Commission

W. R. Canale
Chief, Declassification Branch *cc*

Mr. M. C. Leverett
c/o Mr. M. D. Whitaker
P.O. Box 1991
Knoxville, Tennessee

Dear Miles,

Your shipment #10 (preparation #2 from 706-D) arrived here June 18. The appearance of the sample was very much the same as when I saw it in your cell B before shipment. The material looked light brown and was well concentrated in the bottom of the cone, with no visible amounts higher than about one inch from the bottom.

On adding 0.01 N HNO₃ to the sample most of the light-colored material appeared to dissolve, but a very bulky-looking mass of dark brown material remained undissolved, even after 12 hours standing. After transferring the supernatant liquid to another cone-and-collar assembly, we measured the activity remaining in the shipping container and found it to be about 380 curies (radium equivalent). We were finally able to dissolve practically all the solid by prolonged treatment with 0.2 N HNO₃, and to combine the two solutions. The resulting solution is quite dark.

The only explanation of these observations that I can think of at the moment is as follows. The presence of undissolved impurities (rust flakes?) at the time samples for analyses were taken from vessel B-6, would not have shown up in your analytical results. However, such undissolved impurities may have been transferred to the shipping cone, where the barium nitrate would then have evaporated on top of such solid impurities. This would account for the low Fe analysis, for the relatively light color of the dry sample, and for the phenomena we observed on trying to dissolve the material.

I should like very much to know whether you consider this explanation reasonable or whether you have some other theory to explain all the observations. If my explanation is correct, this would of course constitute another, probably the strongest, argument for the addition of a filtration step at the end of the 706-D process, such as we discussed during my visit.

Due to the dissolving difficulties described above, we do not have as accurate an assay of the total activity as we normally would. Our best value for the total gamma-ray activity of La¹⁴⁰ at time of maximum growth (9:00 a.m., June 20) is 1250 ± 100 curies radium equivalent.

With my best regards to all the 706-D boys,

Sincerely yours,
Gerhart
Gerhart Friedlander

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David C. Hamrin 1/31/98
Technical Information Officer Date
ORNL Site

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w/d
1-4-64
J

A-670

File J

Those Eligible
To Read the
Attached

Date 6-13-45

Subject Contamination - Bldg. 706-D

Copy # 1

By M. C. Leverett

M. D. Peterson

file 1-5

To M. D. Peterson

INV. 65

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Name	Date
<u>M. D. Peterson</u>	<u>6/15/45</u>

Name	Date



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2 J. E. Wirth
3 W. A. Rodger
CLINTON LABORATORIES

4-5 M. C. Leverett
6 Reading File
7 Central File

DATE June 13, 1945
CLASSIFICATION CANCELLED
DATE 2/24/66
for The Atomic Energy Commission
Chief, Declassification Branch *ue*

TO M. D. Peterson DEPARTMENT
FROM M. C. Leverett DEPARTMENT

IN RE: Contamination - Building 706-D

I have the following suggestions to make regarding procedures for detecting and preventing the spread of contamination in Building 706-D:

1. An intensive course of personalized education of each man, to teach him personal neatness and cleanliness in handling potentially active substances and objects. For example, rubber gloves which have been used to handle a sample vial should not be used to open or close a sample blister door, since the gloves may have become contaminated. Use a piece of clean cheesecloth or paper instead, which can then be discarded.
2. Each day have a man armed with a supply of clean cheesecloth swabs wipe off each valve handle, door handle, desk top, stair rail, and other objects likely to be touched by hands; after each wiping have the swab tested for activity by a health-physicist or helper. If activity is found it should of course be cleaned up immediately.
3. Secure samples of the liquor drained from the cyclone separator on the off-gas line and also swabs from the inside of the main three foot ventilating duct, and have a fission analysis made on the activity contained, to identify the offending isotopes.
4. Change the point of addition of ammonia gas from the east side of Cell A in the four inch off-gas line to the flush-out line which connects into the collected off-gas line just before the gas is passed through the A-16 scrubber. It is possible that this may reduce the amount of activity carried through the scrubber. Care should be taken that the ammonia supplied is sufficient to maintain alkaline what spray is carried along by the gases leaving the A-16 scrubber.
5. Relocate the four inch off-gas line in such a way as to remove it from the vicinity of the building, and if possible to make it more easily shielded than in its present location.

*NH3 gas
→ A-16
off-gas*

Most of these items have been discussed among W. A. Rodger, J. T. Weills and me. This memorandum confirms these discussions. I should like to emphasize that although I feel that contamination is under control in Building 706-D, the general level is considerably too high and that every effort should be made to reduce it to an acceptably low amount. I think that the entire attention of the group may properly be focussed on this problem in the event that a period occurs in which no preparation is in process.

M. C. Leverett
M. C. Leverett

Surrey

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45-6-312

File _____

Date June 27, 1945

Copy # 15

Subject 706-D Operations Report

for Week Ending June 23, 1945

From M. D. Peterson

To M. C. Leverett

*W
1-6
g*

INV

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For The Atomic Energy Commission

J. R. Conwell
Chief, Declassification Branch

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706-D OPERATIONS
Report for Week Ending
June 23, 1945

Ba¹⁴⁰ Production (P.A. TX-I) (Group III, Rodger, Bigler)

Michener, Vallado, Witkowski, Collier, Croghan, Hovorka, Levenson, Weil, Weills, Bailey, Dunlap, Fairchild, McLellan, Newton, Ogle, G. E. Phillips

Cleanup

A general cleanup of the cells, equipment, and building has been in progress all week. All vessels were rinsed, then washed once with 60% HNO₃, twice with 20% HNO₃, twice with 25% NaOH and once with 20% HNO₃. Each wash involved two hours' sparging at 60 - 70°C., with samplers circulating, and with transfers of the solution through every jet line used during the run. On the last three washes, the tanks were filled to overflowing.

When this treatment was complete, both cells read greater than 1.5r/hr at the inside doorway. At this point the walls, floor, and all equipment in Cell B were thoroughly hosed down, which lowered the level at the door to 700 mr/hr.

The building has all been cleaned up, except for the following levels against the sample blisters:

<u>Location</u>	<u>Level, 6/23</u>
B3 blister	25 mr/hr
B6 blister	50 mr/hr
B1 blister	45 mr/hr

The fan house has been cleaned sufficiently to allow an 8-hour working period per day therein.

Mechanical

The four-inch black-iron off-gas line to the fan house has been completely removed, and is being replaced with a stainless one. The new stainless blower is being installed in the fan house. Underground drains are being put in from the fan housing and from the four-inch off-gas line to the waste tank, W-11.

The system for introduction of ammonia has been relocated to add ammonia ahead of the scrubber, A-16. ✓

The forced ventilation system in the equipment decontamination room is half completed.

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706-D Operations
Report for Week Ending 6/23/45

- 2 -

June 27, 1945

Program

It is hoped to have Cell B decontaminated for work therein by June 27. Glass equipment will then be installed to allow purification of the product by precipitation of $BaCl_2$ with HCl-ether solution, and shipping of $BaCl_2$ in place of $Ba(NO_3)_2$. Also, the jet discharge lines which manifold together will be separated to avoid repetition of the incident in which active solutions backed up into panelboard service lines.

Cell A will also be decontaminated as rapidly as possible, to allow installation of the cyclone separator in the cell.

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*Declays
FC*

45-6-346

A-670

File 1 E XT

Date 6/30/45

Those Eligible
To Read the
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Subject 706-D Production Run #1

By W. A. Rodger

#1
MDP

To M. D. Peterson

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Name	Date
<u>M. D. Peterson</u>	<u>7/6/45</u>

Name	Date

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cc: M. C. Leverett
R. L. Doan
Central File
Reading File
W. A. Rodger

TO: M. D. Peterson

FROM: W. A. Rodger

June 30, 1945

706-D Production Run #1

Dissolver Operation

On May 26, 208 slugs were loaded to the dissolver in 8 batches of 26 each. They had been pushed on May 25 and were calculated to have an average of 6.85 curies per slug active, and 2.64 mg/slug inactive barium. (Total 1425 curies and 550 mg) The operation was mechanically acceptable. No slugs stuck in the chute, but a defect in carrier design allowed high beams of radiation during dropping.

Aluminum jackets were removed from the slugs using NaOH-NaNO₃. This was the most violent reaction carried out in the dissolver on the run. It went without trouble.

Four batches of 50 slugs were dissolved. All went without trouble. The last batch was a little short, -more than 160 slugs apparently having been used up in the first three solutions.

Extraction

The four batches were extracted separately reusing the PbSO₄ carrier from the first in each of the subsequent extractions. All were decanted after three hours hot, and five hours cold settling. Decantations were followed down on the manometer and the jet turned off when the A9 liquid level was still above the decant slit in the suction line. On batch C, the oil manometer failed to function properly, so decantation was "blind" below 24". It was shut off early, leaving a 3-1/2 gallon heel. Cutting at 0.7" (1000 cc) was attempted on all four batches. This is believed to be unnecessarily low and will be increased on subsequent runs.

The combined cake from four extractions was given one dilute sulfuric acid and three water washes -- each three gallons. They

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For The Atomic Energy Commission

H. R. Conwell
Chief, Declassification Branch *cc*

M. D. Peterson

- 2 -

June 30, 1945

were settled 90 minutes and decanted to A8, using the same jet as during extraction decantation. This procedure was quite satisfactory.

Metathesis

Both metatheses followed flowsheet conditions. They were settled 90 minutes and decanted to B1. Jet A9-B1DD (1/8" IPS to 500 cc) was used on the first. It did not appear to operate properly and the loss was quite high (60 curies). Further, it is believed that the loss would have been much higher had the waste been acidified. On the second shot, A9-B1DC (3/8" IPS to 1000 cc) was used. This gave an apparent loss of only 10 curies, but a subsequent acid wash of the tank yielded 50 curies, leading to the above conclusion concerning acidification of these wastes. In the future all metathesis wastes will be sampled after acidification. Also, jet A9-B1DD will not be used if possible, and it will be replaced at the earliest opportunity.

The residual cake was dissolved in two acid treatments in A9 and jetted directly to B12. The final volume was 4 liters and acid normality 2.9.

Cell B Operation

Electrolysis was carried out at 15 amperes for three hours followed by 6 hours at 25 amps. One-half gram Ba carrier was added. After transfer to B6, the lead content was zero. Therefore, evaporation volume reduction was used. Seven hours were required to reduce the volume to 400 cc.

Two fuming nitric precipitations were made. Each was settled 2-1/2 hours and decanted to 250-300 cc. The first waste was sampled from the B3 sample blister. This will not be done again; a lambda sample will be taken. The waste is very hot--containing large quantities of Ia.

The heel of fuming nitric was evaporated to dryness. Five hours were allowed. This may not have been enough as the final solution was three normal in acid. The transfer equipment B6-B17 failed to work the first time and it was necessary to blow it back. About 35 cc in volume was picked up on this transfer. Also, about 100 mg of Fe and 260 mg Pb were found in the final product. These may have come in during the transfer. Their introduction is still unexplained.

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The final evaporation was carried out in five hours without difficulty. All the product-handling equipment worked satisfactorily.

Analyses

Waste losses, with the exception of the fourth extraction and the two metatheses were low. Unexplained losses, most of which occurred on transfer from Cell A to Cell B, were greater than 50%. Analysis of various tank washes during clean-up failed to locate the missing product. The suspicion is strong that either product analyses were low, or the starting solution analyses were high. Complete results appear in Table I.

Product Disposal

As the final product assayed only 250 curies and was highly contaminated, it was not shipped. The cone was removed from the cell in the normal carrier equipped with a special plug. It was taken to the semi-works where it was dissolved in water and jetted into a hot drain. No personnel over-exposure occurred and the cone was decontaminated sufficiently for reuse.

Contamination

Building contamination and personal exposure during and after this run were not satisfactory. High hand counts were experienced all during the run. Most of these were due to improper use or lack of use of gloves.

Sampling techniques left much to be desired and some over-exposures to hands occurred. Some of the sample blisters will need more shielding. Sample probes will always be hot and as they are in an operating area and so located that their use is awkward, great care will have to be exercised by all personnel to avoid over-exposure and spills.

Both the cell and vessel off-gas systems became hot. Contamination in the cell system may have been due to the failure part way through the run of the vessel ventilating blower. The cell fan gave readings as high as 1 r/hr at the end of the run but was readily cleaned up by hosing the housing out with water.

After the small fan had failed, it was removed for inspection in

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M. D. Peterson

4

June 30, 1945

the course of which a major spill of active condensate occurred. This caused a great deal of trouble with tracked activity before it could be brought under control.

An attempt was made to analyze the final solution in B17 using the polarograph. The attempt was not successful, and in the course of the experiment a considerable quantity (1 - 2 cc) of the final product solution was brought out through the shield. This not only made the B17 sampler hot (a condition which continues to date, as the shield is of black iron and cannot be properly decontaminated), but led to a major spill around B17 which took several days to completely clean up.

No member of the operating group received a general body over-exposure during or after the run. The troubles experienced, while very unpleasant, have instilled all personnel with a healthy respect for the difficulties involved in hot operations and it is believed that the contamination record will improve the next run.

Mechanical

As expected, Cell A equipment (with the exception of the A9 to B1 jet already mentioned) worked well. Likewise, little trouble was experienced with the mechanical operation of Cell B other than the B9 and B11 transfer pipettes which have already given trouble.

The vessel ventilating blower failed and it was necessary to complete the run on the stand-by tie in to the 205 stack.))

~~SECRET~~

TABLE I

Run #1 - Analytical Results

Fraction	Code	Curies Product*	Curies Lost	% of Total Product Dissolved
First Dissolving First Extraction Waste	1MA 8WMA	280	9	0.9
Second Dissolving Second Extraction Waste	1MB 8WMB	281	14	1.4
Third Dissolving Third Extraction Waste	1MC 8WMC	238	8	0.8
Fourth Dissolving Fourth Extraction Waste	1MD 8WMD	208	94	9.4
Cake Washes	8WW		4	0.4
First Metathesis Waste Second Metathesis Waste	1WCA 1WCB		56** 10**	5.6 1.0
Total Curies Dissolved	-	1007		
Product Sol'n before Electrolysis	12P	490		49
PbO ₂ Plate Solution	3WPb		-	-
Product after Electrolysis	6P	420		42
First Fuming Nitric Waste Second fuming Nitric Waste	3WFNA 3WFNB		16 3	1.6 0.3
Final Product Solution	17P	250		25
Tank Washes	-		50	5

*All curies calculated to 2400 5/31/45.

**These losses believed to be higher by a factor of at least 2.

TABLE II

SUMMATION OF LOSSES

	Curies	%
Total Pushed (calculated)	1450	100
Decay Loss (6 days)	410	28.3
Known Losses Cell A	195	13.5
Known Losses Cell B	69	4.8
Yield	250	17.3
Unaccounted Loss	526	36.1

Table III

Time Cycles

Operation	Time Required*
Charging 208 slugs	10 hours
Metal Solution	
Batch A	6 "
Batch B	6 "
Batch C	7 "
Batch D	12 "
Extractions	
Batch A	11 "
Batch B	11 "
Batch C	11 "
Batch D	11 "
Four Cake Washes	8½ "
Metathesis	
First	4 "
Second	4 "
Cake Solution	1½ "
Electrolysis	9
Evaporation Volume Reduction	7
Fuming Nitric Precipitations	
First	3½ "
Second	3½ "
Evaporation to Dryness	5 "
Solution	2 "
Evaporation	4 "

*Delays due to waiting for analyses not included.

Table IV

Extraction Decantations

Batch	Rate gal/min	Heel	Loss	
			Curies	% of Product Present
A	2.86	1000 cc	9	3.2
B	3.05	1250 cc	14	2.5
C	2.69	3.6 gal	8	1.0
D	3.02	1150 cc	94	9.4

Table V

1150 c at 9PM, 6/14 (last sign time)
 Pb 117 mg (colorimetric) 150 mg (spets.)
 U 3 (5 spets)
 Fe 10 "
 Ni 3 "
 Sr \approx 80 " [\approx 1/3 of Ba]
 Ba 230 mg (calc) none added.

45-7-265

[Handwritten signature]

A-670

File _____

Date 7/26/45

Those Eligible
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Subject 706-D Hot Run #3 - Health Report for
Period 7/12/45 thru 7/23/45

Copy # 2 *[Handwritten notes]*

By R. A. Simons

To K. Z. Morgan

[Handwritten notes: See w. 2, 4-15-0]

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<u><i>[Signature]</i></u>	<u>7/30/45</u>

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- 2. J.L. Wirth
- 3. R.S. Stone
- 4. M.C. Leverett
- 5. M.D. Peterson
- 6. W... Rodger
- 7. W.H. Ray
- 8. R... Simons
- 9. Central File
- 10. Readers File

K. L. Morgan

7/26/45
Health Physics

R. A. Simons

Health Physics

706-D HOT RUN #3 - HEALTH REPORT
FOR PERIOD 7/12/45 THRU 7/23/45

Twelve slug bucket loads, each carrying 26 slugs, were dropped into the dissolver in Cell A during 7/12 shift and swing. Dissolving started at about 8:00 PM on 7/12/45. A typical survey of the slug bucket was as follows:

Top	at contact	125 mr/hr
R. side	" "	200 "
L. side	" "	225 "
Front	" "	400 "
Back	" "	160 "
Bottom	" "	225 "

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The Atomic Energy Commission

Declassification Branch

Two series of 312 slugs each were to be dissolved, one after the other.

Before starting the run a complete walkie-talkie, zues, and smear survey was made on the floors of the building. 35 smears were taken at points which would be contaminated most. Results showed that all floors in the building were less than 150 c/m except in the equipment decontamination room and on the lead floor East of the Cell B probes on the 3rd level which was 500 c/m on smears. From this and previous surveys it was decided that rubbers would not be required except in the personnel decontamination room and the South hot lab., and, of course, the cells if anyone went into the cell corridors during the run. If the floor activity increased to a level greater than 200 c/m rubbers would be required. Two complete smear and walkie-talkie surveys and several partial surveys were made of the floors during the run and it was still found that it would not be necessary for personnel to wear rubbers except in the afore mentioned spots. This does not mean that the floors did not become contaminated during the run. In washing down B6 blister after sampling on 7/20/45, contamination leaked down to the floor, probably due to a leak in the blister, causing smears at the sample area to count 2000 c/m. The floor was immediately washed and brought down to 500 c/m directly under the blister and < 200 c/m at the sampling area. On 7/18/45 during the swing shift the South hot lab. became contaminated all over due to a small amount of hot solution from a B6 sample being sprayed on the floor while pipetting. This contamination was tracked on the ground level from the S. hot lab. to the East loading platform, the hand count room, personnel decontamination room, and the corridor between the hot

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labs. and 706-C Bldg. East wall. Smears in the South lab. were 5000 c/m. The other spots which were contaminated averaged about 2000 c/m. Areas were marked off and danger signs posted within an hour after the spill and within 2 hours the floors were being scrubbed. The men who received hot shoes from the spill washed their shoes down so that the hottest pair counted 3200 c/m. All personnel were probed. Within the next 24 hour period most of the hot areas had been brought down to less than 200 c/m. On 7/21/45 the floor again became contaminated under B6 blister after taking a sample and was cleaned up immediately. There was no trouble with floor contamination aside from the three aforementioned events. It is suggested that the floor in the S. and W. hot labs., the hand count room, and the West corridor, be painted even though smears are < 200 c/m, since a walkie-talkie picks up 2000 c/m from activity embedded in the floor at several spots in these areas.

During the run radiation hazards came namely from the fan house and sampling. On 7/9/45 the off gas blower at the S. E. corner of the fan house was 10 mr/hr and inside at the C. E. corner of the fan house the highest reading was 50 mr/hr. The 3' diam. cell ventilation duct was 27 mr/hr at the concrete base and 2' outside the fan house 13 mr/hr. At the end of the run on 7/23 a survey disclosed the following:

Against large vent duct inside the fan house	150 mr/hr
" " " " at concrete base East of 706-D	30 "
" " " " 4' outside fan house	135 "
At N. end of cat walk	45 "
At entrance to fan house	60 "
Background at S. E. corner of fan house	290 "
8' E. of small blower	75 "
Against large fan housing in fan house	175 "
Against off gas line in fan house	3.5 r/hr
2' from off gas blower	2 r/hr

On Monday, 7/16/45, at 1:00 PM the first series of slugs product solution was lost to W. tank at the tank farm by way of the cell tanks off gas scrubbers.

A survey the same afternoon after the mishap occurred showed that the activity at the fan house had increased by about a factor of four. The N. E. corner of 706-D which had previously been reading 20 mr/hr went up to 30 mr/hr outside the building and 15 mr/hr inside the building at the corner. However, 24 hours later the activity came down to a level slightly higher than what it was before the solution was lost to W. tank. The cell ventilation duct did not increase over 10 mr/hr during any part of the run which is far different than what happened during run #2 when the ventilation duct was reading 100 mr/hr at the concrete base East of 706-D building. The general background was 8 mr/hr at the East side of the building (inside) most of the run as compared to 50 mr/hr

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during the run #2. Also the background at the North inside of the building was about 6 mr/hr this run as compared to 30 mr/hr during run #2. This improvement comes from putting the emergency and cell tanks off gas lines under ground and shielding part of the cell tanks line with 2" of lead.

Outside of floor contamination at B6 sample blister most the trouble from sampling came when men would try to adjust the rubber stopper on B3 and B6 samplers and put the sample test tube on the sampling spout without the use of tongs against the advice of Health Physics and operations. Unfortunately the men did not wear film packs on their hands when this occurred since it was expected that the proper sampling technique as set up by operations which requires the use of tongs to adjust the stopper and test tube would be followed. It is understood that it would have been hard to do the job using tongs and for this reason the sampler spouts have been redesigned by operations in order to eliminate the necessity of inserting hands into the sample blister.

Doses received by the chemists and samplers on film packs during the run were as follows:

<u>Samplers</u>		<u>R. hand</u>	<u>L. hand</u>	<u>Forehead</u>	<u>wrist mr</u>
M. D. Peterson	7/22				
W. A. Rodger	7/21	0 mr	0 mr		
W. A. Rodger	7/22	0 "	0 "		
W. A. Rodger	7/22	30 "	15 "		
M. C. Leverett	7/21	15 "	15 "		
Chemists:					
B. F. Collins	7/18	100 "	45 "	25 mr	
B. F. Collins	7/21	40 "	55 "		
N. N. Bernstein	7/18			10 "	25 right 15 left
N. N. Bernstein	7/22			0 "	30 right 5 left
S. A. Reynolds	7/21				20 right 40 left
E. S. Pomerance (Spectograph work)	7/22	5 "	5 "		

Doses received while taking six capillary samples from B6 tank as shown by pocket meters on 7/23 are as follows: (all samples read greater than 11 r/hr 90% Beta at 2").

	<u>R. back of hand</u>	<u>L. back of hand</u>	<u>L. ankle</u>
W. A. Rodger	29 mr	23 mr	
L. K. Michner	27 mr		34 mr
E. Dunlap	19 mr	30 mr	

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Nine precipitron air samples were taken during the run. 1000 c/m β & γ is considered high by the author. Data is as follows:

<u>Date</u>	<u>Location</u>	<u>Time for run</u>	<u>β & γ c/m for 1/2 hr. run</u>	<u>β & γ c/m per liter of air</u>
7/13	2nd level by P.B. 1	38 mins.	102	1.1×10^{-2}
7/15	3rd level 1' from A1 funnel while dissolving	36 mins.	145	1.5×10^{-2}
7/17	3rd level 5' S.E. of slug chute 8' from Cell A funnels	1 hr.	84	8.6×10^{-3}
7/18	Fan house 4' N. of large fan	1 hr.	80	8.2×10^{-3}
7/18	3rd level midway bet. slug chute & cell A funnels	1 hr.	131	1.3×10^{-2}
7/19	3rd level 2' W. of cell A funnels	1 hr.	102	1.0×10^{-2}
7/19	Center of floor in S. hot lab.	1 hr.	93	9.5×10^{-3}
7/20	3rd level N. end 2' S. of #5 integron	1 hr.	14	1.4×10^{-3}
7/21	2nd level 2' N. of B1 blister near glass P.B. while product Sol. is in glass equip.	5 hrs. & 25 mins.	39	4.0×10^{-3}

Alpha counts on all nine samples were less than 5% of tolerance. Tolerance for alpha is 1.9 d/m per cu. ft. of air or 5×10^{-10} gms./cc of air or 3.1×10^{-11} curies per cc of air.

From the preceding data it can be seen that there was little activity in the air of the building during the run.

The core carried within an 8" shielded carrier was placed on the truck at 3:00 PM on Monday, 7/23/45. Readings were as follows:

2" from top of core carrier	120 mr/hr
Against 2 sides of carrier	10 "
	28 "
	55 "
	31 "
Truck cab	< 1 "
Left rear wheels of truck	1.1 "
Right rear wheel of truck	2.0 "

No contamination was left in the building by the carrier.

At about 9:30 PM Monday, 7/23/45, a back up of hot waste occurred in B1 to A9 jet pipe while jetting waste from B3 waste tank to A9

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7/26/45

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precipitator. B1 tank to A9 tank jet line is connected to a steam line which comes out of cell B on the West side, 2nd level and runs up to Panel Board 4A on the third level. B3 to A9 jet line is manifolded into B1 to A9 jet line before going into A9 tank. A steam pressure of about 75 psi forced most of the hot sol. into A9 but some of it was forced up the B1 to A9 jet line and into the steam line leading to P.B. 4A where the pipe was reading 25 r/hr at contact. On the 2nd level the general background on the West side of cell B was 800 mr/hr. Background at P. B. 5 on the East side of the 3rd level was 100 mr/hr. The Health Physics surveror, W. J. Joslin, received 140 mr and 160 mr on his clockhouse pocket meters for 7/23. There were two other men in the hot area at the time - H. Baily and F. A. Weil. Baily received 95 mr and >200 mr on his pocket meters and there is no record for Weil. The hot activity was discovered immediately, due to the monitrons ringing. This can be varified by the micromax charts. All personnel were immediately evacuated from the building and danger signs posted. The hot pipe was removed the next day and the building was brought down to its normal level of activity. Removal of the hot pipe will be reported on next week.

A similar increase in building background occurred at the end of hot run #2 when the cell B sump lines between P.B.3 and cell B on the ground floor became hot due to backups while jetting waste from B 3 waste tank to A5 neutralizer. Two jet lines from cell B sump, two jet lines from B3, and B1 precipitator to A5 jet line, all manifolded into a common line before going into A5 tank. Due to steam pressure some of the hot waste was sent into the sump jet lines and then into the steam lines leading to P.B.3. Between runs #2 and 3 the sump jet lines were run separately into A6 waste tank in order to eliminate this hazard at P.B.3. Further maintenance work involving the de-manifolding of certain jet lines in the cells will probably be done before the start of the next run. A fence will probably be installed around the fan house to decrease hazards in that area. Work between now and the next run will be concerned mainly with decontamination of Cell B so that maintenance men can get into the cell, and the maintenance work itself. A rough estimate by the author would be 2 weeks for decontamination and a week for maintenance work in cell B from 7/24/45.

ORIGINAL SIGNED BY AUTHOR

R. L. Simons

R.L.S/ah

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W.C.C. 11

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File 1-5

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INV. 65

Date 7/31/45

Subject 706D PRODUCTION RUN #2
(Shipment #10)

Copy # 1

By W. A. Rodger

MDP

To M. D. Peterson

Before reading this document, sign and date below

Name	Date
<u>M.D. Peterson</u>	<u>8/4/45</u>

Name	Date

INV. 65

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ORNL Site

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DATE 10/14/66
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W. A. Rodger
Chief, Declassification Branch

- 1. M. D. Peterson
- 2. M. C. Leverett
- 3. R. L. Doan
- 4. W. A. Rodger
- 5. Central File
- 6. Reading File

TO: M. D. Peterson
FROM: W. A. Rodger

This document consists of 13 pages and 0 figures. No. 1 of 6 copies, Series A

706D PRODUCTION RUN #2
(Shipment #10) (shipped June 16)

Dissolver Operation

The 624 slugs charged to the dissolver for Run #2 were added in three groups of 208. They were discharged from the pile on 6/4, 6/6, and 6/9 and were calculated to have an average of 6.6 Curies per slug active, and 2 mg per slug inactive, Barium at the time of discharge. (Total 4120 Curies and 1230 mg.) The charging operation was again mechanically acceptable and the defect noted on the first run, which allowed strong beams while dropping slugs, had been corrected. No slugs stuck in the chute.

The run was divided into three series, (2A, 2B, 2C) each consisting of 4 dissolvings and extractions followed by a metathesis, cake solution, and electrolysis. (The third series, 2C, consisted of only three batches, as all the slugs were dissolved by that time.) The second and third slug loadings were carried out following the third dissolving on runs 2A and 2B, respectively. All coating removal reactions and metal solutions went without difficulty. Metal analyses ran about 10% low, as only 500 slugs were accounted for analytically.

Extraction

Four extractions were made on 2A and 2B; three on 2C. The lead carrier was reused throughout each series. Batch 1A was settled 3 hours hot and only 4-1/2 hours cold in place of the usual 5. As this loss (32 Curies) was a little higher than normal, the extra half hour cold settling time was restored. All eleven decantations were stopped at 2 - 2.5" on the oil liquid-level manometer. The losses averaged 2% of the total per four-charge series, and ranged from 3.3% down to 0.5%. See Table 2 for complete extraction decantation results.

The extraction cakes from each of the three runs were given one acid (25% H₂SO₄) and 4 water washes, all of 3 gallon volume. On

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M. D. Peterson

7/31/45

the 2A and 2C washes, agitation was carried on for 10 minutes, followed by 80 minutes settling; on 2B, these times were respectively 5 and 75 minutes. All were decanted to AB using A9-ABDB, as during extraction. This washing procedure continues to be satisfactory.

The total waste losses involved were < 0.1%, 0.4%, and 0.4%. Hereafter, the shorter times will be used.

Metatheses

Chemical quantities on all three metatheses were increased 10% as insurance against higher losses. On 2A and 2B, the wastes were decanted to B1, using A9-B1DD, where they were acidified, sampled, and returned to the next extraction. For the metathesis of 2C, three carbonate treatments were used. All were decanted to AB using A9-ABDB. The 1.8% loss here was not recovered, as the waste was discarded. See Table 3 for metathesis conditions and losses.

All metathesis cake solutions were made in three shots. The final volumes after transfer to B12 were 3900, 4300, and 4100 cc.

Cell B Operation

All electrolyses were standard 3 hours at 15 amps. plus 6 hours at 25 amps. runs. No Ba carrier was added. The first two were transferred to B6 and the volume reduced to 4 liters by evaporation. The normal amount of carbonate was added for a carbonate volume reduction. As this failed to make the solution alkaline (the double batch requires double the usual amount of carbonate), all the product appeared in the waste. The solution was therefore returned to B6 and the volume again reduced to 4 liters by evaporation. Carbonate concentration was then brought up to 0.5 moles/liter. This caused crystallization of large quantities of KNO_3 , plugging all jet lines and bubbles, so it became necessary to add water to double the volume, heat to put the KNO_3 in solution, cool, and resettle the $BaCO_3$ precipitate. After settling for 5 hours, solution was decanted to a 375 cc heel, using B3-B6DA. A loss of 190 Curies (9%) was experienced.

This waste was transferred to B1 where attempts at recovery of the Ba were made. The centrifuge failed as the feed mechanism was plugged. This was not unexpected. Next, 155 cc of 6% $Pb(NO_3)_2$ were added, the slurry heated 1 hour and settled overnight. On decantation, a 75% recovery of the product was effected, but it was not considered worthwhile to add it to the final product, contamination was feared.

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The electrolyzed solution from series 2C was added to the heel from the 2A-2B volume reduction in B6 and the total volume reduced to 500 cc. by evaporation. Three fuming nitric treatments were made. A total loss of 172 Curies (8%) was taken on the three HNO₃ wastes. See Table 4 for conditions and losses on individual treatments.

The heel from the final fuming nitric step was evaporated to dryness in B6. As the B17 probe appeared to be covered with a white solid which might have been Pb(NO₃)₂, it was decided to sample in B6 instead of in B17. A total of 180 cc water was added to B6, and the solution was sampled and transferred to the final evaporator B19, via B11, B17 and B8. After evaporation was partially complete a 100 cc wash was carried through the same route from B6.

The final evaporation took 5-1/2 hours and went without difficulty. The product handling equipment worked satisfactorily, but one lifting lug on the cone adapter broke during transfer. It then became apparent that there were certain minor differences between cones made here and those fabricated by the customer. The customer's units will be used hereafter.

The final product had a slight brownish color. It was well down in the tip of the cone. Its analysis was as follows:

Product at IST, last separation time (9:00 P.M., 14 June)

1230 Curies

Pb	117 mg
Cr	3 mg
Ni	3 mg
Fe	10 mg
Ba	~ 230 mg
Sr	~ 80 mg

Analyses

While losses on carbonate volume reduction and fuming nitric treatments were high, they were within the expected limits. No unexpected high losses were found. See Table I for complete analytical data.

Equipment Rinse

A rinse of the final equipment indicated almost complete transfer of product to the shipping cone. On transferring this rinse

from B8 to B6, 85% of it disappeared. After some study, the trouble was diagnosed as a plugged funnel line, causing overflow into the rest wells. This conclusion was subsequently proved incorrect on Run 3, when the same thing happened to product solution. It was then discovered that the material was swept up into the B17 off-gas line through the B-17 - B6 overflow line which is tied into the B7 and B8 to B6 funnels.

Contamination

Run 2 followed Run 1 so closely that no time was available to make changes shown necessary by the first run.

The off-gas system became so hot that Panel Board I became as hot as 130 mr/hr. Sample blisters reached levels as high as 1 #/hr at point of nearest approach. Attempts to clean them out during the run resulted in readings on hand films of as high as 1000 mr/hr. Therefore, cleaning was deferred until the conclusion of the run.

Sampling during the run resulted in several overexposures of the hands (averaging 130 mr/hr) and some floor contamination was experienced around the third floor probes. This was expected, however, and was brought under control immediately after each sample was taken.

After the run had been completed, a waste being jetted into A5 from B1 backed up into the Cell B sump jets and out the steam lines into Panel Board 3. This raised the level of activity in that area to 3 to 5 r/hr, and caused almost the whole building to read over tolerance. It was necessary to evacuate all personnel for a while. The contaminated piping was removed and the level dropped to 1.5 mr/hr. The removal was accomplished without allowing activity to spread throughout the building.

Mechanical

The positive displacement blower was out of operation during part of the run, due to bad bearings. It was apparent in ^{both} batch Runs 1 and 2 that the whole off-gas system will have to be revamped.

The backup of activity into PB 3 was the direct result of manifolded jet discharge lines. Five lines from Cell B to A5 tie into the same discharge header. It is planned to run separate exit lines for each of the sump jets.

M. D. Peterson

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7/31/45

It was deemed inadvisable to use the B17 sample probe because it was covered with a white solid, probably $Pb(NO_3)_2$. This came from an attempted decontamination of the shield. As these shields are made of black iron and uncovered lead, decontamination is almost impossible. Critical shields must be replaced with stainless units; all will eventually be replaced as time permits.

Cell A equipment and other cell B equipment operated in a satisfactory manner.

TABLE I

Run 2 Analytical Results

Fraction	Code	Curies Product*	Curies Lost*	% of Total Product Dissolved
Series 2A:				
First Solution	IMA	176		
Extraction Waste	8WMA		24	1.1
Second Solution	IMB	163		
Extraction Waste	8WMB		7	0.3
Third Solution	IMC	178		
Extraction Waste	8WMC		24	1.1
Fourth Solution	IMD	229		
Extraction Waste	8WMD		23	1.1
Cake Washes	8WW		1	0.1
Miscellaneous Washes			2	
Total Curies Dissolved		746		
Product Solution before Electrolysis	12P	400		
Product Solution after Electrolysis	6P	425		
PbO ₂ Plate Waste	3WFPb		2	0.1
Series 2B:				
First Solution	IMA	184		
Extraction Waste	8WMA		15	0.7
Second Solution	IMB	173		
Extraction Waste	8WMB		4	0.2
Third Solution	IMC	180		
Extraction Waste	8WMC		16	0.7
Fourth Solution	IMD	233		
Extraction Waste	8WMD		6	0.3
Cake Washes	8WW		3	0.2
Miscellaneous Washes			1	
Total Curies Dissolved		770		
Product Solution before Electrolysis	12P	436		
Product Solution after Electrolysis	6P	1105		
PbO ₂ Plate Waste	3WFPb		26	1.2
Carbonate Vol. Red. Waste	3WC		189	8.7

TABLE I Run 2 Analytical Results (cont'd)

Fraction	Code	Curies Product*	Curies Lost*	% of Total Product Dissolved
Series 2C:				
First Solution Extraction Waste	IMA SWMA	266	18	0.8
Second Solution Extraction Waste	IMB SWMB	254	6	0.3
Third Solution Extraction Waste	IMC SWMC	149	7	0.3
Cake Wash Metathesis Waste	SWW SWCA		2 39	0.1 1.8
Total Curies Dissolved		669		
Total Dissolved 3 Series		2185		100
Product Sol'n. before Electrolysis	12P	551		
Product Sol'n. after Electrolysis	6P	1354		62
First final HNO ₃ Waste	3WFNA		91	4.2
Second final HNO ₃ Waste	3WFNB		55	2.5
Third final HNO ₃ Waste	3WFNC		26	1.2
Final Product Solution	17P	1180		54
Material Balance		1767		81

* all Curies based on 2400, June 15, 1945.

M. D. Peterson

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7/31/45

TABLE IA

Summation of Losses

Total Pushed (calculated)	<u>Curies</u> 4120	<u>%</u> 100
Decay Loss (10, 8, and 5 days each series respectively)	1400	34
Calculated Curies not found in dissolver solutions	540	13
Known losses, Cell A	200	5
Known losses, Cell B	390	9
Yield	1180	29
Unaccounted Loss	$\frac{410}{4120}$	$\frac{10}{100}$

TABLE II

Extraction Decantations

Batch	Rate of Decantation gal/min.	Heel cc	LOSS	
			Curies	% of Prod. Present at Time of Decantation
2A-A	2.8	2800	24	13.6
2A-B	2.35	2900	7	2.1
2A-C	2.5	2600	24	4.6
2A-D	2.4	2700	23	3.1
2B-A	2.4	2500	15	7.6
2B-B	2.45	2500	4	1.1
2B-C	2.84	3000	16	3.0
2B-D	2.4	2600	6	0.8
2C-A	2.7	3000	18	6.8
2C-B	2.7	2400	6	1.5
2C-C	3.1	2500	7	1.0

TABLE III
Metathesis Treatments

Treatment	Settling Time (Hrs.)	Jet	Heel (cc)	LOSS %	
				Curies	%
2A 1st	2	A9-BIDD	3000	58*	2.5
	2nd	A9-BIDD	2000	7*	0.3
2B 1st	2-1/2	A9-BIDD	3200	5*	0.2
	2nd	A9-BIDD	2000	1*	
2C 1st	2	A9-ASDB	3000	} 39	1.8
	2nd	A9-ASDB	3000		
	3rd	A9-ASDB	1400		

* These wastes were recycled into succeeding batches

TABLE IV
Fuming Nitric Treatments

Precipitation	Settling Time (hours)	Heel (cc)	Loss	
			Curies	%
1st	3	210	91	4.2
2nd	2-1/2	250	55	2.5
3rd	2-1/2	270	26	1.2

TABLE V

Time Cycles

Operation	Time Required (Hours)
Charging 208 slugs 2A 2B 2C	6-3/4 6 6
Coating Removal 2A 2B 2C	3-1/2 2 1-2/3
Metal Solution 2AA 2AB 2AC 2AD 2BA 2BB 2BC 2BD 2CA 2CB 2CC	7-1/4 6-1/2 7-3/4 5-1/4 6 6-1/2 9 5 5-1/2 7-1/2 14
Extraction 2AA 2AB 2AC 2AD 2BA 2BB 2BC 2BD 2CA 2CB 2CC	10 11-1/2 11-1/2 11 11 11 12 11 10-1/2 14 11

(continued)

Table V - Time Cycles (cont'd)

Operation	Time Required (Hours)
Extraction Cake Washes	
2A	8
2B	7
2C	8
Metatheses	
2A	10-1/2
2B	8-1/2
2C	13
Metathesis Cake Solutions	
2A	2
2B	1
2C	2
Electrolyses	9 hours each
Evaporation Volume Reductions	1-1/2 hours each
Fuming Nitric Precipitation in B6	
1st	5
2nd	3
3rd	3
Evaporation to Dryness in B6	5
Solution of Ba(NO ₃) ₂ in B6	2
Final Evaporation in B19	5-1/2

*DeCross
C-*

45-8-215

A-670

File 1-5 *XT*

Those Eligible
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Date 8/22/45

Subject 706-D PRODUCTION RUN #3

(Shipment #11)

By W. A. Rodger

To M. D. Peterson

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SM

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DATE 10/11/66

For The Atomic Energy Commission

706-D Production Run #3
(Shipment #11)

H. F. Carroll
TO
Chief, Declassification Branch

Mechanical Changes

Between Runs 2 and 3, both Cell A and B were decontaminated sufficiently to allow the following changes to be made:

1. Glass Equipment

After Run #2 the customer requested that $BaCl_2$ rather than $Ba(NO_3)_2$ be shipped. This required that we be able to run the HCl-ether process. To conserve time, glass rather than tantalum equipment was installed. Two Stang-type reactors were put into cell B. One, B21, was put in the place of B17 which was removed; the other, B20, was installed just west of B1. Five Hastelloy C valves were used in conjunction with each apparatus, to direct flow from the transfer vessels. These are operated by means of extension handles through the west wall, first floor, of cell B. Optical instruments were installed so that each glass set-up could be viewed.

2. Ventilating System

A stainless steel blower was installed for providing vacuum to the scrubber A16. The black iron line from A16 to the stack was replaced with stainless steel, and all of the line inside and proximate to the building was shielded with 2" of lead. Drains were cut into each of the four stages on the blower and provisions made for washing down the whole system and draining it directly to W11.

The cyclone separator which had been located at the fan house was placed inside cell A and its drain piped to the sump. ★

3. Cell B Sump Jets

The two cell B sump jets, which backed up following Run #2, were repiped individually to A6.

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4. Cell A Duct Jet

It was found that the cell A duct jet, a CI#2, was plugged. It was removed and replaced with a CI#1 jet.

Dissolver Operation

The 831 slugs charged to the dissolver for Run #3 were added as follows:

<u>Date Pushed and Charged to Dissolver</u>	<u># Loaded</u>
7/12	312
7/14	311
7/17	208

On 7/14 one slug stuck in the carrier. It was removed in the canal without trouble, placed in a different hole in the carrier, and discharged without further difficulty on the next load.

The slugs loaded were calculated to have an average of 7.8 Curies/slug active, and 0.9 mg/slug inactive Ba. (totals: 6500 Curies and 720 mg) at time of discharge.

The run was divided into three series, (3A, 3B, 3C) the first two each consisting of 4 dissolvings and extractions followed by meta-thesis, cake solution, and electrolysis. The third series was similar, except that it contained only 3 dissolving and extraction batches, as the slugs were essentially all gone at that time. The second and third slug loadings were carried out following the third dissolving of series 3A and 3B respectively. All coating removal reactions went without difficulty. During batches 3BA and 3BB, the liquid level and specific gravity instruments on the dissolver did not operate properly, leading to apparent volume readings some 10% high. The instruments were corrected and all subsequent batches went as expected. Probably because of these two high readings, the metal analyses were only 5% low (793 out of 831 slugs accounted for). About sixty slugs were dissolved in each batch, in place of the 50 in the previous runs.

Extraction

Four extractions were made on series 3A and 3B; three on 3C. The amounts of reagents were increased 20%, because of the increase in metal dissolved, and the lead carrier was re-used throughout each series, as usual. After batches 3AA and 3AB it became apparent that the extractor A9 was running very close to overflowing. Therefore,

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in all subsequent extractions the 120# of water usually added to dilute to flow sheet concentrations was omitted. This apparently did not cause increased waste losses. All other conditions were normal. The eleven decantations were cut at 1.5" to 3.5" on the oil liquid-level manometer. Jet A9-A8DB was used. Losses averaged 17 Curies per extraction, or 5.5% of the product from each individual dissolving. See Table 2 for complete extraction decantation results.

The extraction cakes from each of the three series were given the standard one acid, four water washes, all of 3 gallon volume. Each wash was agitated 5 min., settled 70 min., and decanted to a 1.8" to 2.5" heel on the oil manometer, using jet A9-A8DB. The waste loss in the combined washes were 2 Curies or less in each of the three series (see Table 1).

Metatheses

Reagent quantities on all metatheses were increased 20% over flowsheet to allow for the larger amount of Pb present. All were decanted to A8 using jet A9-A8DB and cutting at 0.7" to 2.0" heels. The wastes were acidified and sampled in A8. As all waste losses were low (none greater than 50 Curies) they were discarded rather than being recycled into the next extractor batch, as volume in the A9 extractor was at a premium. See Table 3 for metathesis conditions and losses.

The first metathesis cake solution was transferred into the electrolysis vessel in cell B in a single batch, without a subsequent wash. The other two were each followed with 2 washes. Final volumes in the electrolysis vessel were 3750, 4250 and 3950 cc, respectively.

Cell B Operation

All electrolyses were standard (3 hours at 15 amps. plus 6 hours at 25 amps.). In series 3A, on transferring the electrolyzed solution from the electrolysis cell B12 to the precipitator B6, the whole volume disappeared. After considerable study, it was determined that the solution had gone into the off-gas system. The off-gas header, the overflow from B17 to B6, and the two B7-B8 funnel lines to B6 all manifold together. The new ventilating fan is so powerful that if the off-gas valve is open at the time the B12 to B6 transfer is made, the solution is sucked almost quantitatively into the off-gas system. This accounts for the loss of part of the B6 rinse reported in Run 2, and probably is also the explanation for high unaccounted for losses on this transfer in both previous runs.

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The electrolyzed solutions from series 3B and 3C were combined in the precipitator B6 and evaporated to 500 cc for transfer to the glass purification vessel B21. However, both B9 and B11 transfer pipettes plugged with potassium nitrate that crystallized out on cooling, making it impossible to transfer to the glass equipment. Increasing the volume with water and/or heating the solution redissolved the KNO_3 and allowed the lines to clear, but it was found impossible to keep them open after the solution was cooled for transfer with a volume of less than 1 liter. It was therefore necessary to redissolve the KNO_3 by addition of water, and to remove the KNO_3 by making a fuming nitric precipitation in B6. The waste from this step contained almost 400 Curies, or 16.5% of the total product, but the dissolved $\text{Ba}(\text{NO}_3)_2$ was then readily transferred to the glass equipment.

One half gram carrier Ba was then added, and fuming HNO_3 and HCl-ether precipitations were made in the glassware.

The fuming HNO_3 and HCl-ether wastes from the purification in glass contained 1.4 and 7.1% of the product, respectively. Both filtration rates were unusually low - 4 hours required for the f. HNO_3 and 2 for the HCl-ether. There remained on the filter in the glass vessel probably a gram of white solid, insoluble in the HNO_3 and HCl-ether solutions used. This may be silica, originally in the slugs themselves (1 ppm SiO_2 in the slugs would account for the amount observed).

The final BaCl_2 product precipitate on the filter was apparently quite slowly soluble in water, as analysis indicated that the specified solution treatment dissolved only 150 Curies. The solution was therefore made 0.5 M in HCl and an additional four hours' contact was allowed. Analysis of the solution on the filter then showed from 800 to 1300 Curies. The lack of reproducibility in this analysis indicated that part of the product might be suspended rather than dissolved in the solution. The solution was therefore filtered and run into the spare glass vessel for re-analysis, which showed at least 900 Curies actually in solution, so it was transferred into the shipping cone and evaporated. The spare glass vessel could not be rinsed after this product solution transfer, because the extension handle on a valve broke off in the cell. Therefore, only 850 Curies was claimed as delivered into the cone.

The first rinse of the precipitator B6 contained about 300 Curies of Ba, so this solution was also given the standard f. HNO_3 and HCl-ether purification treatments in the glassware. The final solution from this treatment assayed 300 Curies Ba, and the contaminants were sufficiently low that only iron would be above the product specification if this was added to the product in the cone. The customer was consulted, and at his request this product solution was also added to the cone and evaporated.

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During evaporation of the 850 Curie product solution, the electric heater on the top of the evaporator burned out, so the head could not definitely be maintained above the dew point of the off-gas. This may have allowed some HCl to condense on the stainless steel head and to drip back into the cone, carrying iron from corrosion into the product.

The final product was quite brownish in color, and some was on the shoulder of the cone, although most of it appeared to be in the tip. Its analysis was as follows:

Ba	850 Curies at LST 22:00, 21 July plus 300 Curies at LST 05:00, 23 July
Pb	20-35 mg.
Fe	18 mg.
Cr	4-6 mg.
Ni	3-6 mg.
Sr	20-40 mg.
Ba (inactive)	< 850 mg.

Analyses

In Table I complete analytical data are presented. Percentages are all based on the last two series only. Table IA gives a summation of losses based on the last two series.

Contamination

As before, the vessel ventilating system became hot, the radiation in the fan house reaching several r/hr. The lead shielding on the small off-gas line prevented above tolerance readings in the adjacent working areas, however. Activity next to the cell ventilating duct remained at about 30 mr/hr throughout the run.

On 7/16/45, during solution of a batch of metal in the dissolver, the dissolver vacuum dropped to zero for about one minute, presumably due to a slug of condensate in the off-gas line to the 205 stack. At about this time the friskers at the restricted area gate and in 706-B went off simultaneously. A strong wind was blowing from the east, so it is likely that active gases were released from the dissolver into the cell and out the 706-D stack. The off-gas line was drained immediately, and no further trouble with the dissolver vacuum was experienced. {

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During the clean-up after the run, active waste being discharged from B3 to A9 backed out the steam line to the B1-A9 jet, necessitating evacuation of the building until that line could be removed. Readings were 30 r/hr close to the line. A similar incident occurred during the previous run, both due to jet discharge lines being manifolded into a common discharge header.

Slight personnel over-exposure occurred during sampling with the B3 blister. Additional shielding had been added inside the blisters, to cut down the general radiation therefrom, but this so decreased the room in the blister that the greater time now required for sampling resulted in over-exposure. This design will be changed.

Mechanical

During the first part of the run, the cell A sump overflowed several times. This was traced to seepage from a broken drain in the fan house. It was repaired.

As indicated in the previous section, more trouble was experienced with backing up of manifolded jets.

Three of the ten extension handles on the Hastelloy C valves for the glass equipment failed at the coupling.

Of the final product handling equipment, the electric head heater burned out and the elevator bound so badly as to be nearly inoperable.

Table I
Production Run 3
Summarized Analyses

(All analyses based on 24:00, 7/21/45)

Fraction	Code	Curies Product	Waste Losses (Curies)	% Total Product (2385 G.)
<u>1st Series:</u>				
First Dissolving Extraction Waste	3A-IMA 8WMA	278	5	
Second Dissolving Extraction Waste	3A-IMB 8WMB	277	8	
Third Dissolving Extraction Waste	3A-IMC 8WMC	237	58	
Fourth Dissolving Extraction Waste	3A-IMD 8WMD	235	14	
Cake Wash	8WW		2	
Metathesis Wastes	8WC		12.5	
All product to here lost into off-gas system				
<u>2nd Series:</u>				
Fifth Dissolving Extraction Waste	3B-IMA 8WMA	380	28	1.2
Sixth Dissolving Extraction Waste	3B-IMB 8WMB	415	5	0.2
Seventh Dissolving Extraction Waste	3B-IMC 8WMC	306	18	0.8
Eighth Dissolving Extraction Waste	3B-IMD 8WMD	263	17	0.7
Cake Wash	8WW		0.5	0.0
Metathesis Wastes	8WC		50	2.1
PbO ₂ Sol'n Waste	3WpB		33	1.4
Prod. in Sol'n after Electrolysis	6P	1284 (yield=94%)		54

Production Run 3 Analyses (continued)

Fraction	Code	Curies Product	Waste Losses (Curies)	% Total Prod (2385 C.)
<u>3rd Series:</u>				
Ninth Dissolving Extraction Waste	3C-IMA 8WMA	372	25	1.0
Tenth Dissolving Extraction Waste	3C-IMB 8WMB	337	11	0.5
Eleventh Dissolving Extraction Waste	3C-IMC 8WMC	312	19	0.8
Cake Wash	8WN		1	0.0
Metathesis Wastes	8WC		33	1.4
PbO ₂ Sol'n Waste	3WFPb		132	5.5
TOTAL CURIES DISSOLVED		2385		100
<u>Series 2 & 3 Combined:</u>				
Prod. Sol'n after Electrolysis	3B +C-6P	1361(?)		57(?)
First f. HNO ₃ Waste*	3WFNA		393	16.5
Second f. HNO ₃ Waste**	3WFNB		33	1.4
HCl-ether Waste	3WHCL		169	7.1
Product, first shot	20P	900		37.8
Product, second shot	21PA	300		12.6
		(Total yield = 50%)		
Rinse B6	C-1		140	5.9
Rinse B12-B8-B6	C-2		49	2.1
Rinse B12-B7-B6	C-3		8	0.3
Material Balance		2365		99.2

* In stainless precipitator B-6

** In glass vessel B-21

TABLE IA

Summation of Ba Losses, Series 3B and 3C

Fraction	Curies	%
Total dissolved (calculated as 510 slugs)	4,000	100
Decay loss (9, 7 and 4 days)	1,140	28.5
Calculated Curies not found in dissolver solutions	500	12.5
Known losses cell A	210	5.3
Known losses cell B	960	24.0
Yield	1,200	30.0
Unaccounted loss	10	0
<hr/>		
Total Curies found in dissolver solutions, calculated to 24:00, 7/21	2,385	100
Cell A waste losses	210	9
Cell B waste losses	960	40
Product yield	1,200	50
Material balance	2,370	90

Table II

Extraction Decantation

Batch	Rate of Decantation (gal/min)	Heel (cc)	Ba Loss	
			Curies*	% of Prod. Present at time of Decan'n
3AA	2.6	3700	5	1.7
3AB	2.4	2700	8	1.4
3AC	2.8	2900	58	7.3
3AD	3.1	2700	14	1.4
3BA	3.0	3500	28	7.5
3BB	2.3	3150	5	0.6
3BC	2.8	3700	18	1.7
3BD	3.1	2260	17	1.2
3CA	2.5	1200	25	6.8
3CB	2.6	2700	11	1.6
3CC	2.4	3000	2	0.2
Averages	2.7	2850	17	2.9

(*Calculated to 24:00, 7/21/45)

Table III

Metathesis Treatments

Treatment	Settling Time (hrs.)	Heel (cc)	Ba Loss	
			Curies*	% of Prod. Present
3A 1st	3½	3140	13	1.3
2nd	3	3300		
3B 1st	2½	2700	50	3.7
2nd	2½	1365		
3C 1st	2½	3400	33	3.2
2nd	2½	900		

(*Calculated to 24:00, 7/21/45)

Table IV

Time Cycles

Operations	Hours Required
Charging	
3A 312 slugs	8 1/4
3B 311 "	7 3/4
3C 208 "	4 1/2
Coating Removal	
3A	2
3B	1 1/2
3C	1 3/4
Metal Solution and Extraction	
	<u>Solution</u> <u>Extraction</u>
3AA	6 11
3AB	5 3/4 11
3AC	6 1/2 11 1/2
3AD	10 11
3BA	9 11
3BB	5 1/4 11
3BC	6 1/4 10 1/2
3BD	8 3/4 10 1/2
3CA	6 1/4 10 1/2
3CB	5 1/2 10 1/4
3CC	8 11
Extraction Cake Washes	
3A	7 3/4
3B	7
3C	7
Metatheses	
3A	11 1/4
3B	9 1/4
3C	9 1/4
Metathesis Cake Solution	
3A	2 3/4
3B	3/4
3C	3/4
Electrolyses, each	9

Table IV
(continued)

Operation	Hours Required
Transfer to glass, including a f. HNO_3 in B6	16
In B21	
f. HNO_3	4
HCL-ether	1
Final Evaporation	4
Load	2

W. A. RODGER

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Those Eligible
To Read the
Attached

Date 8/23/45

Subject 706-D PRODUCTION RUN #4

(Shipment #12)

Copy # 1

By W. A. Rodger

M. D. Peterson

To M. D. Peterson

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<i>M. D. Peterson</i>	<i>8/27/45</i>

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DATE 10/11/66

To: M. D. Peterson

For The Atomic Energy Commission

From: W. A. Rodger

H. F. Carroll
TC

Chief, Declassification Branch

706-D Production Run #4
(Shipment #12)

Mechanical Changes

Between runs 3 and 4 Cell B was decontaminated sufficiently to allow the following changes:

1. Glass Equipment

Both glass units were replaced.

2. Hastelloy Valve Extensions

More dependable connections were made between the valve stem and extension handle. Square-holed sockets were welded to each valve stem and the ends of the extension handles were squared to fit.

3. B19 Head Heaters

A new head heater was installed on the final evaporator. All parts which came in contact with process vapors are Hastelloy C and the head is heated with steam.

4. B10

B10, the Cell B waste hold-up tank, was installed, allowing elimination of all jet manifolds going back to Cell A.

5. Jet Manifolds

Of the remaining manifolded jet systems, all steam lines save one in each system were capped at the exterior cell wall.

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Dissolver Operation

The 1144 slugs changed to the dissolver for Run #4 were added as follows:

<u>Date Pushed and Charged</u>	<u>Slugs Loaded</u>
8/6	286
8/8	312
8/11	286
8/14	<u>260</u>
	1144

None of the 8/14 charging actually got into the run; all solutions made after 8/14 went into a "stand-by" series of extractions which was not needed. For purposes of calculation, the run has been considered to consist of the first 650 slugs charged to the dissolver. This includes all of the first two chargings plus 18% of the third. These slugs are calculated to have had an average of 7.5 Curies per slug active and 1 mg per slug inactive barium (Total 4900 Curies and 650 mg) at time of discharge.

The run was divided into two series. The first consisted of two pre-extractions of wastes from Run #3 and six 50-slug dissolvings and extractions. The series was followed by cake wash, metathesis, metathesis cake wash (to remove K_2CO_3 which has been troublesome on the past two runs), solution, and electrolysis. The second series, six more 50-slug dissolvings and extractions, was put through the same process and combined with the first in B6.

The second, third, and fourth chargings were made after batches 3AD, 3BA, and 3BF respectively. All coating removal reactions and metal solutions went without difficulty or incident.

Extraction

All twelve extractions were on the original 50 slug, 65% H_2SO_4 plus 100# dilution water, basis. The twelve decantations were cut at 3.8" to 4.2" on the oil liquid level manometer. Jet A9-A8DB was used throughout. Losses averaged 33 Curies per extraction or 14% of each individual dissolving. See Table 2 for complete extraction decantation results.

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On batch 4AA the waste loss was 88 Curies, well above normal. This was believed to be due to pick-up of residual activity from A8. However, the 4AB waste analysis showed 160 Curies lost so it was decided that the two pre-extractions may in some manner have used up too much lead. Therefore, 75 grams additional lead was added to 4AC. Subsequently, a resample of 4AB indicated this original analysis was in error, the loss being only 18 Curies. Again on 4AF a high (202 Curie) waste loss was recorded. As this was the last batch of the series, 15 grams of Pb were added to A8 and the slurry resettled and decanted to A5 where a normal loss appeared.

Losses on series 4B were a little high (average 36 Curies) but not enough to be alarming or to cause deviations from the standard procedure.

Both series were given standard cake washes. The usual low losses resulted.

Metatheses

Both metatheses were normal and losses were reasonable, although not accurately available on series 4A, as the metathesis liquors were mixed with the cake washes which converted any solid carbonate back to sulfate. Both metathesis cakes were washed twice with water to remove the potassium salt. The combined cake wash, metathesis liquor, and metathesis cake wash was put back into extraction 4BA without sampling. On series 4B the cake wash was discarded, and a fairly accurate analysis of metathesis liquors and washes was made. The loss for both was about 150 Curies. See Table 3 for metathesis conditions and losses.

The first metathesis cake was dissolved and transferred to the Cell B electrolysis vessel in a single batch of HNO_3 , the second in three. Final volumes in the electrolysis vessel were 3500 cc and 3950 cc, respectively.

Cell B Operation

Both electrolysis were standard (3 hours at 15 amps. plus 6 hours at 25 amps.). The electrolyzed solutions from the first series contained 150 mg Pb, but no Pb analysis was made on the second series. The two electrolyzed solutions were combined in the B6 precipitator, evaporated to 310 cc, and taken up in the stationary pipette B11 for

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transfer to the glass vessel B21. However, when the solution was discharged from the pipette into the funnel leading to B21, only about 80 cc passed through the funnel into the glass vessel, and the funnel then plugged. Solution from the pipette was then diverted into the funnel to the spare glass vessel B20. Here also the funnel plugged, after only about 20 cc had run into the glass vessel. In both cases, the transfer from the pipette was stopped before the funnel overflowed. To complete the operation, the solution in the funnels and pipette, and subsequent wash solutions, were sucked up into a shielded glass flask on top of the cell block and then discharged into the glass reaction vessel B21, through a plastic ("Tygon") tube lowered into the funnels and into B21 through an open roof plug. To decrease the swinging of the tube in the breeze of ventilating air in the cell, a section of stainless pipe was fastened to its lower end. After transfer of the first 150 cc wash of vessels B6 and 11 into B21, this metal pipe struck and cracked the sintered glass filter disc in B21, so the solution was transferred to the spare glass vessel, B20, where one-quarter of a gram of barium carrier was added, and a fuming nitric acid precipitation was made.

The second wash of B6 with 300 cc water picked up 1000 Curies of product, so this also was transferred into B20, and a second fuming nitric acid precipitation made. The third B6 rinse contained only 45 Curies, which was not recovered. The product in B20 was further purified with an HCl-ether precipitation, the $BaCl_2$ was dissolved, and the solution was analyzed, transferred to the final evaporation cone, evaporated to dryness, and the product shipped out, all without further difficulty.

The insoluble precipitate, presumably silica, was again noted in the glass equipment, but apparently in smaller amount than in the previous run. All filtrations through the sintered glass disc were rapid (greater than 20 cc/min.). The final $BaCl_2$ precipitate dissolved readily and completely in water.

Waste losses from the operations in the glassware were unusually low - each less than 35 Curies.

The final product was somewhat brown, but was the lightest colored material shipped from 706-D to date. Its analysis follows:

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Product at IST 21:00, 16 August - 2100 Curies

Fe	9-18	mg.
Cr	3-6	"
Ni	1-2	"
Pb	12-24	"
Sr	20-30	"
Ba (Inactive)	1000-1500	"

Analyses

Complete analytical data are presented in Table I. Table IA shows a summation of losses based on the first 650 slugs dissolved.

Radiation Exposure

In the Cell B maintenance and repair program, working times were calculated for a maximum radiation of 70 mr/day. Extra meters were worn by all persons entering the Cell, and where possible, these were checked at least once before the maximum working time had elapsed, to protect against overdoses from unrecognized beams or hot spots. With these precautions, in a total of approximately 250 exposures in the Cell B work, there were recorded two 2-fold overexposures, both due to violations of the rules set up, and six lesser overexposures.

In the transfer of product solution through the temporary glass vessel on top of Cell B, Witkowski received a large overexposure (8/16/45: 200 mr/day on pocket meter, 1260 mr/11 days on badge meter), and two others received less than 2-fold overexposures. During this hot transfer, the glass vessel was well shielded with lead on all sides but one, which was left open to allow for rapid manipulation, if necessary. This caused the South end of the building to be above tolerance for a time, but it was roped off and posted then, and returned to the usual background value when the transfer equipment was disposed of.

Contamination

In the Cell B work, two persons recorded high hand counts. In operating and disposing of the temporary transfer equipment above Cell B, several spots on the third floor were contaminated, and two persons received high hand counts. Numerous hot spots were found on valve handles, doors, railings, instruments, etc.

One of the 6P product samples from the B6 precipitator blister was taken without Health-Physics coverage, due to the Army's transfer without adequate notice of the HP man assigned to that shift. Apparently, the floor was contaminated during this operation, and activity was tracked over the second floor before it was discovered, several hours later.

All known contaminated spots have been cleaned up.

TABLE I

Run 4 Product Analyses

(all Curies are calculated to the originally scheduled shipping time, 24:00 Sat. Aug. 18)

Fraction	Code	Curies Product	Curies in Waste	% of Total Product Dissolved
<u>First Series (4A)</u>				
A9 Heel from Run #3 Extraction Waste	- -	40	1	
A1 Heel from Run #3 Extraction Waste	- -	40	7	
First Solution Extraction Waste	1MA 8WMA	207	88 ⁽¹⁾	3.1
Second Solution Extraction Waste	1MB 8WMB	245	18	0.6
Third Solution Extraction Waste	1MC 8WMC	246	5	0.2
Fourth Solution Extraction Waste	1MD 8WMD	175	2	0.1
Fifth Solution Extraction Waste	1ME 8WME	232	71	2.5
Sixth Solution Extraction Waste Waste after Reworking	1MF 8WMF 5WAF	212	202 ⁽²⁾ 3	0.1
Total Curies dissolved		1397		
PbO ₂ Solution Waste	3WPb		18	0.6
Product Solution after Electrolysis	6P	1105		

TABLE I
(Continued)

Fraction	Code	Curies Product	Curies in Waste	% of Total Prod. Diss.
<u>Second Series (4B)</u>				
First Dissolving Extraction Waste	1MA 8WMA	300	13	0.5
Second Dissolving Extraction Waste	1MB 8WMB	241	20	0.7
Third Dissolving Extraction Waste	1MC 8WMC	240	33	1.2
Fourth Dissolving Extraction Waste	1MD 8WMD	220	60	2.1
Fifth Dissolving Extraction Waste	1ME 8WME	200	50	1.8
Sixth Dissolving Extraction Waste	1MF 8WMF	243	33	1.2
Total Curies Dissolved (Series A & B)		2841		100
Series B Extr'n Cake Wash 8WV + Metathesis Wastes 8WC + Metatheses Cake Washes	8WV 8WC 8WCW		10 32 144(3)	5.1
PbO ₂ Solution Waste Product Sol'n after Electrolysis (Series A&B)	3WPb 6P	1915	-	67.5
B6 Rinse	-	1011		
First f. HNO ₃ waste	3WfNA		13	0.5
Second f. HNO ₃ waste	3WfNB		28	1.0
HCl Ether Waste	3WHCl		34	1.2
B6 final rinse	-		45	1.6
Final Product Solution	20P	1890		66.5
Material Balance	-	2576		90.5

- (1) This apparent loss is believed to be due to contamination in the sampling vessel A8.
- (2) Not lost. This waste was re-worked, as described in last week's report.
- (3) This waste was recycled into the third extraction series, which remains in Cell A.

TABLE IA

Summation of Ba Losses - Run #4

Fraction	Curies	%
Total Dissolved (calculated as 650 slugs)	4900	100
Decay Loss (12, 10, and 7 days)	2125	43.4
Known losses, Cell A	550	11.2
Known losses, Cell B	160	3.3
Yield	1890	38.6
Unaccounted for Loss	175	3.6
Total Curies found in dissolver solutions, calculated to 24:00, 8/18	2841	100
Cell A Waste Losses	550	19
Cell B Waste Losses	160	6
Product Yield	1890	67
Material balance	2600	92

TABLE II
Extraction Decantations

Batch	Rate of Decantation (gal/min.)	Heel (cc)	(a) Ba Loss	
			Curies	% of Prod. Present at Decantation
4AA	2.5	3620	(b) 88	30.5
4AB	2.8	3620	18	3.4
4AC	2.5	4230	5	0.6
4AD	3.1	3620	2	0.2
4AE	2.5	4150	71	6.0
4AF	2.5	4230	3	0.2
4BA	2.7	4150	13	4.3
4BB	2.3	4150	20	3.7
4BC	2.5	4230	33	4.2
4BD	2.4	4150	60	6.0
4BE	2.6	3600	50	4.2
4BF	2.5	2100	33	2.3
Average	2.6	3820	33 (26) ^c	5.5 (3.0) ^c

(a) calculated to 24:00 Saturday, 8/18
(b) believed to be picked up from contamination in A8
(c) Averages omitting the 4AA values

TABLE III

Metatheses

Treatment	Settling Time (Hours)	Heel (cc)	Ba Loss	
			Curies	%
<u>4A</u>				
1st metathesis	$2\frac{1}{4}$	3900	-	-
2nd metathesis	$2\frac{1}{2}$	2700	-	-
1st cake wash	$2\frac{1}{4}$	3900	-	-
2nd cake wash	$2\frac{1}{2}$	2700	-	-
<u>4B</u>				
1st metathesis	$2\frac{1}{4}$	2700	20	0.7
2nd metathesis	$2\frac{1}{2}$	3000		
1st cake wash	$2\frac{1}{4}$	2700	110	3.9
2nd cake wash	$2\frac{1}{2}$	800		

* calculated to 24:00 Saturday, 8/18

TABLE IV
Time Cycles

OPERATION	HOURS REQUIRED																										
Charging																											
286 slugs	9																										
312 slugs	7½																										
286 slugs	9-¾																										
Coating Removal																											
1st	2																										
2nd	2½																										
3rd	3½																										
Metal Solution and Extraction	<table border="0"> <tr> <td data-bbox="933 961 1071 997">Solution</td> <td data-bbox="1274 961 1437 997">Extraction</td> </tr> <tr> <td data-bbox="402 1024 462 1060">4AA</td> <td data-bbox="1315 1035 1421 1071">10-1/4</td> </tr> <tr> <td data-bbox="402 1060 462 1096">4AB</td> <td data-bbox="1315 1071 1421 1106">10-1/4</td> </tr> <tr> <td data-bbox="402 1096 462 1131">4AC</td> <td data-bbox="1315 1106 1485 1142">10 + 9-3/4</td> </tr> <tr> <td data-bbox="402 1131 462 1167">4AD</td> <td data-bbox="1315 1142 1356 1178">11</td> </tr> <tr> <td data-bbox="402 1167 462 1203">4AE</td> <td data-bbox="1315 1178 1421 1213">10-3/4</td> </tr> <tr> <td data-bbox="402 1203 462 1239">4AF</td> <td data-bbox="1315 1213 1421 1249">11-1/2</td> </tr> <tr> <td data-bbox="402 1239 462 1274">4BA</td> <td data-bbox="1315 1249 1421 1285">10-1/2</td> </tr> <tr> <td data-bbox="402 1274 462 1310">4BB</td> <td data-bbox="1315 1285 1421 1320">10-3/4</td> </tr> <tr> <td data-bbox="402 1310 462 1346">4BC</td> <td data-bbox="1315 1320 1421 1356">10-1/2</td> </tr> <tr> <td data-bbox="402 1346 462 1381">4BD</td> <td data-bbox="1315 1356 1356 1392">10</td> </tr> <tr> <td data-bbox="402 1381 462 1417">4BE</td> <td data-bbox="1315 1392 1421 1428">10-1/2</td> </tr> <tr> <td data-bbox="402 1417 462 1453">4BF</td> <td data-bbox="1315 1428 1421 1463">10-1/4</td> </tr> </table>	Solution	Extraction	4AA	10-1/4	4AB	10-1/4	4AC	10 + 9-3/4	4AD	11	4AE	10-3/4	4AF	11-1/2	4BA	10-1/2	4BB	10-3/4	4BC	10-1/2	4BD	10	4BE	10-1/2	4BF	10-1/4
Solution	Extraction																										
4AA	10-1/4																										
4AB	10-1/4																										
4AC	10 + 9-3/4																										
4AD	11																										
4AE	10-3/4																										
4AF	11-1/2																										
4BA	10-1/2																										
4BB	10-3/4																										
4BC	10-1/2																										
4BD	10																										
4BE	10-1/2																										
4BF	10-1/4																										
Extraction Cake Washes																											
4A	8½																										
4B	7¼																										
Metatheses																											
4A	8½																										
4B	9½																										
Cake Solution																											
4A	1/2																										
4B	¾																										

(continued)

TABLE IV (cont'd)

Time Cycles

OPERATION	HOURS REQUIRED
Electrolyses	9 each
Transfer to glassware	8
Transfer to B20	6
2nd final HNO ₃	2
HCl-ether	1-1/2
Solution and sampling	2
Evaporation	5
Loading	2

~~SECRET~~
10/11/45

1. K. Z. Morgan
2. J. E. Smith
3. R. E. Slattery
4. M. C. Berrett
5. M. D. Peterson
6. W. A. Rogers
7. J. E. Pruegel
8. R. A. Simons
9. R. E. Slattery
10. Central File
11. Readers File

K. Z. Morgan

Health Physics

R. E. Slattery

Health Physics

REPORT FOR BUILDING 706-D FOR
TWO WEEK PERIOD 9/24/45 TO 10/6/45

The two week period covered in the report was a period of decontamination of Cell B and of general building and equipment repair in 706-D.

No serious overdoses or accidents occurred during this period. The men worked at all times on a basis of 50 mr/day as a tolerance dose (a standard set by building operations) which allowed a considerable margin of safety; a great advantage in very "hot" work such as encountered in Cell B. The only men who incurred overdoses, were three operation supervisors (Kimshaw, Witkowski and Collier) who kept running in and out of the cell despite constant warnings from Health Physics (this too ceased before many days passed), and Health Physics men themselves, who, since the department was understaffed had to do more "hot" surveys than normal.

Initially the readings in Cell B were as follows (on the 27th of September after the removal of B-20 and B-21):

<u>Point</u>	<u>r/hr</u>
B-12	10.8
B- 6	5.0
B- 3	0.8
B- 9	17.6
B-11	>20.0 (instrument off scale)
B- 7	13.4
B- 8	>20.0 (instrument off scale)
B-19	8.0
Doorway	0.9
Center of cell	2.6

At the present writing (10/8/45) the general background in the cell is about 250 - 300 mr/hr and no tank remaining exceeds 1.5 r/hr. Certain hot spots have been covered with lead, and a lately discovered hot gas-ket (100 r/hr) between B-18 and B-6 is now being removed. The reason for the delay in its discovery is its general inaccessibility, being 15' off the ground in a vertiable maze of pipes and lines.

Decontamination has proceeded by the removal first, of all hot tanks, lines, glassware and pipes to lower the background as much as possible, and then washing with acid and a fire hose all hot spots and then the

~~SECRET~~

entire surface of the cell, which was in itself contributing to the background.

During the process of decontamination Health Physics constantly monitored all personnel entering the cell, fixing ridged time limits, and closely instructing all men involved. Some very difficult jobs were accomplished in this manner without overdoses.

The glass equipment in B-20 and B-21 was removed by first cutting the tygon lines connected to it and then smashing the glassware completely right out of its frame. Working times of from 5 to 15 seconds were allowed for the former job on the 25th and 26th of September in areas reading as high as 34 r/hr, but it was accomplished without incident or overdose. While smashing B-21 on the 26th L. Burris had some radioactivity contaminated nitric acid spill on him, but the immediate removal of his clothing and a subsequent shower removed all the material. K. Slattery and S. Rimshaw were also contaminated in removing Burris' clothing but a thorough shower also decontaminated them.

On the same date R. Slattery and G. Putnam of Health Physics removed and placed in the burial ground (by means of a truck) the can containing the hot material from the cell (21.5 r/hr on the outside of the can). The can had been stored on the S. E. platform but a sudden rainstorm necessitated its immediate removal for fear that water washing through the cans would contaminate the entire area. The job was accomplished without too much difficulty, but G. Putnam received 250 mr for two days and R. Slattery 115 mr for one day.

On September 29, 1945, the tank B-9 was removed from the cell by means of arc burners on the end of ten ft. extensions. It was rolled below the cell chimney by poles and transferred to a truck on the south platform by crane. While on the crane a survey was made indicating a general radiation of 45 r/hr, and 100 r/hr (at about 4") in the hottest spot. The discrepancy with previous noted surveys is due to the limited accessibility of the tank while in the cell. It became apparent that the cab of the truck was too warm to permit its disposal in the usual manner and it was transferred to the burial ground on the end of a 45' boom of a traveling crane. This was accomplished without incident, and a bulldozer covered the tank with earth. No more than 3' mr/hr came through the earth. It is of interest to note that the tank while reposing outside the building raised the radiation level in the 706-D change room from < 1 mr/hr to 5 mr/hr. (For a period not exceeding 1/2 hour).

Nothing else of unusual interest occurred during this period in connection with decontamination though considerable difficulty is being experienced in bringing the radiation level down to a point where repairs can be made in the cell with adequate time allowance. It has not yet reached that point despite spraying with everything from high pressure water to a mixture of nitric and hydrofloric acids followed by scrubbing of hot spots, and steaming. The practice of issuing pocket meters to every man entering the cells has been discontinued in most cases since an even

background permits the settling of time limits by means of instruments. This also allows Health Physics (which has had a limited personnel during this period) more time to monitor men adequately; in the long run is more satisfactory, since it is better to tell a man what to avoid and see that he avoids it, than to find out afterwards that he has overlapped into a hotter area.

The hot lead flooring on the third level (mentioned in previous reports) has been completely removed and disposed of on the advice of Health Physics, since it was all contaminated to a greater or lesser extent (25 to 70 mr/hr). The concrete floor which was below it still radiates about 40 mr/hr and runs as high as 1 r/hr in spots. Further decontamination and eventual resurfacing are in order.

A leak in the off gas duct where it enters the fan house roof spread rust colored stains (reading up to 300 mr/hr) over the roof and a portion of the N. side of the fan house, and on one spot in the fan house to the east of the blower. The press of work in the building proper has prevented rectification of these points, but since no one goes near the fan house and operations has been informed, Health Physics on the spot does not consider the matter immediately serious.

It has been noticed that back-ups in the hot sinks, from W-11 have been occurring, and one precipitron run recorded β & $\gamma = 10,422$ counts and $\alpha = 236$ counts. Operations has been informed and requested to see to the installation of adequate traps, through as yet nothing has been done. Similar difficulty occurred in 706-A two or three months ago but was rectified by the installation of traps between W-11 and the sinks in the main line.

In general the operations in this building have proceeded without hazard despite the difficult nature of the work. Operations in 706-D has set up an arbitrary standard whereby no man shall work in an area of >6 r/hr or receive more than 50 mr/hr per 24 hr. period. This last has been a direct result of the fact that for four months the men have received almost a complete tolerance dose and they feel that the end of the war has relieved the necessity for this practice.

R. E. Slattery

RES/ab

Handwritten scribbles

48-70

COMBOL-24 475

Statement of

H. G. ...

H. G. ...

... and state below

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Those ...

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Attached

Copy ...

JUN 68

Name

Date

Name

Date

Handwritten signature

JUN 68

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4. J. C. Kaskie
5. Central File

DATE 12-7-45

M. C. Leverett

DEPARTMENT

FROM J. C. Kaskie

DEPARTMENT

IN RE:

SHIPMENT #14

brunish - tan

BaCl₂ product had a slight dark appearance with a thin film on the wall up to 2" level. The bulk of the product was in the tip of the cone. It was shipped in a tantalum lined cone, collar, and carrier, with a "Koroseal" gasket.

Ba 1200 Curies analyzed at 0500 Dec. 6, 1945, last separation time.

Ba 300 Curies estimated due to belief of larger solution volume at time of sampling and due to a minute bubble in sampling tube.

1100 - 1060
1110
69
68
1175
1600
vs. 2000

Ba total 1500 Curies

Pb 42 mg
Fe 9 mg
Cr 6 mg
Ni 20 mg
Sr 6 mg
Ba 1130 mg

CLASSIFICATION CANCELLED

DATE 1/10/67

For The Atomic Energy Commission

H. F. Carroll

Chief, Declassification Branch

J. C. Kaskie

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[Redacted]

10-12

15-92
CENTRAL FILES NUMBER
75-21-162

DATE: AUGUST 29, 1945

SUBJECT: SHIPMENT # 15

Those desiring to read the attached

J. G. Sando

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H. G. Leverett

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Mr. Sando 10-12



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DATE 12-21-45

TO M. C. Leverett DEPARTMENT

FROM J. C. Kaskie DEPARTMENT

IN RE:

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SHIPMENT #15

BaCl₂ product had a slight dark appearance with a thin film on the wall up to 2" level. The bulk of the product was in the tip of the cone. It was shipped in a tantalum lined cone, collar, and carrier, with a "Koroseal" gasket.

Ba 2100 Curies analyzed at 0600
Dec. 20, 1945, last separation time.

Pb	18 mg
Fe	36 mg
Cr	2 mg
Ni	4 mg
Sr	25 mg
Ba	700 mg

J. C. Kaskie

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 DATE 1-11-67
 For The Atomic Energy Commission
H. P. Carroll
 Chief, Declassification Branch

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1204

CLINTON LABORATORIES

CENTRAL FILES NUMBER

SECRET 464-148



Date January 10, 1946

Subject 706-D Analytical Laboratory

Manual

File _____

Copy No. 3

To M. C. Leverett

From J. C. Kaskie

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M. C. Leverett 1-11-46

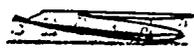


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21. Central File



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

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

706-D ANALYTICAL LABORATORY MANUAL

RECEIVING:

Every sample is delivered in a lead carrier to the S. Lab. by operations. Here the chemist will check the sample sheet which accompanies each sample for pertinent information such as HP reading, solution volume, and sample code. The lead carrier will be emptied and returned to the operator immediately after delivery of the sample. The sample tube is placed in a lead holder behind the upper barricade in the S. Lab. This is done to eliminate an accumulation of lead carriers in the Lab. The final step is to log the sample in the Lab. office.

SAMPLES

The 706-D sample code is patterned after that used in the 205 building. The first number refers to the vessel sampled. Letters following designate the type of sample, and a final A, B, C, or D refers to batch number in Cell A, or precipitation number in Cell B. Standard designations of:

- M Metal Solution
- W Waste
- P Product

are used.

CLASSIFICATION CANCELLED

DATE 1-11-67

For The Atomic Energy Commission

H. P. Canale
Chief, Declassification Branch

CELL A

- 1MA, 1MB, etc.
- 8WMA, 8WMB, etc.
- 5WMA, 5WMB, etc.
- 8AW
- 8WW
- 8WC
- 8WCW

- Dissolver Solution
- Extraction Waste
- Resettled Extraction Waste
- Acid Wash of Extraction Care
- Water " " " "
- Metathesis Waste
- Metathesis Waste and Water Wash

CELL B

- 6P
- B-6 Rinse
- 3WPB
- 3WFNA
- 3WEC1
- 17P
- B-17 Rinse

- Product After Electrolysis
- Rinse of B-6 Vessel
- PbO₂ Waste
- Fuming Nitric Waste
- HCl-Ether Waste
- Final Product
- B-17 Vessel Rinse

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ANALYSIS:

Under normal operating conditions each sample, except dissolver samples, is analysed only for Ba. Dissolver samples are analysed for "T" in addition to Ba. The final sample is run spectrographically to determine the amounts of contaminants. In some cases operations will notify the Lab. if special analyses are necessary.

DILUTIONS:

Activity of the sample will vary according to the type of sample and according to the phase or step in production. Dilutions are necessary to bring the number of counts per ml of solution down to a convenient counting rate. Convenient counting rates for our counters range between 1000 and 10,000 cts. per min., with the optimum at about 5000 cts. per min. The diluent used is 2N HNO₃. Normal dilutions of various samples are as follows:

LMA, LMB, etc.	0.1/50 x 1/100
8WMA, 8WMB, etc.	0.2/50 x 10/100
5WMA, 5WMB, etc.	0.2/50 x 10/100
8AW	0.1/50 x 1/100
8WV	0.1/100
8WC	0.1/50 x 1/100
8WCV	0.1/100 x 1/100
6P	0.01/100 x 1/1000
B-6 Rinse	0.1/100 x 1/100
3WPB	0.1/100 x 1/100
3WINA	0.1/100 x 1/100
3WEC1	0.1/100 x 1/100
17P	Capillary to /100 x 1/1000
B-17 Rinse	0.1/50 x 1/100

The above list of dilutions is not absolute, but is based on normal operating conditions aiming at a 2000 C. shipment. Dilutions in abnormal situations are based on number of Curies estimated and on solution volume. An approximate number to use in converting from Curies to counts is 1×10^{11} . An example follows:

Estimated -- 100 Curies
1 Liter

$$\frac{100 \times 1 \times 10^{11}}{1000} = 1 \times 10^{10} \text{ cts/min/ml}$$

That means that a dilution of 1×10^7 is necessary to bring the counts in the original solution down to a suitable counting rate.

DISPOSAL OF SAMPLE:

After the analyses on a sample have been run, recorded, and accepted, the sample tube must be removed from the open barrier to the closed vault. Record in the specified notebook the number of the vault and the position in which the sample is located. Also list in the record book the activity and the date upon which the analyses were run.

SUMMARY OF PROCESS:

Briefly, the latest process in use is to load 600 active slugs and to dissolve them in batches of 80 to 85. The first step is to remove the Al slug coats and send this solution to waste. Nitric acid is used to dissolve the slugs completely. Next a lead sulfate precipitation is made to extract the active Ba. Lead sulfate acts as a carrier for the barium sulfate. After all the slugs have been dissolved and extracted the sulfate are converted to carbonates in a metathesis step. Following this, the lead is removed by electrolyzing the solution to deposit PbO_2 on the anode. All other contaminants and fission products which may have followed through are removed by a fuming nitric acid and a hydrochloric acid -- ether separation. This final $BaCl_2$ solution is then evaporated to dryness for shipment.



<u>SAMPLE</u>	<u>TANK</u>	<u>CLASS</u>	<u>ANAL. FOR</u>	<u>REMARKS</u>
1MA	A-1	Prod.	Ba & "P"	Dissolver solution in HNO ₃
8WMA	A-8	Waste	Ba	PbSO ₄ Extraction Waste
5WMA	A-5	Waste	Ba	PbSO ₄ Resettled Extraction Waste
8AW	A-8	Waste	Ba	Acid Wash of Extraction Cake
8NW	A-8	Waste	Ba	H ₂ O Wash of A-8 after Extractions are Completed
8WC	A-8	Waste	Ba	Carbonate Waste From Metathesis
8WCW	A-8	Waste	Ba	H ₂ O Wash of A-9 Metathesis Cake and Metathesis Waste
6P	B-6	Prod.	Ba	Product Solution After Electrolysis
B6 Rinse	B-6	Prod.	Ba	Rinse of B-6 After Transfer of Product Solution
3WFB	B-3	Waste	Ba	Waste from Electrolysis - PbO ₂ Waste
3WFNA	B-3	Waste	Ba	Waste from Fuming HNO ₃ Separation if used
3WFEC1	B-3	Waste	Ba	Waste from HCl-Ether Separation
17P	B-17	Prod.	Ba	Final Product Solution Before Evaporation.
B17 Rinse	B-17	Prod.	Ba	Rinse of B17

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BARIUM DETERMINATION

Dilute the sample to the right magnitude. Mix thoroughly by vigorous agitation and by allowing the solution to stand at least 15 minutes. Take an appropriate aliquot - generally one ml.

Introduce the aliquot into a 50 ml centrifuge tube. Add 2 ml. of BaCl₂ carrier. Mix. Add 30 ml of 5 parts HCl - 1 part ether mixture. Place in an ice-water bath. Stir for one minute. Allow to stand until the precipitate begins to settle. Centrifuge for at least 2 minutes. Decant the supernatant liquor. Dissolve the cake in a minimum amount of water. Repeat the HCl ether precipitation 2 more times. Record the last precipitation or separation time.

Place a weighed filter paper disc on a semi-micro perforated funnel. Transfer the BaCl₂ centrifuge cake to the funnel by means of absolute alcohol acidified with HCl - 4% HCl by volume. Wash the precipitate 2 more times with the acidified alcohol and once with HCl-ether. Continue the suction for at least 30 secs. through the cake and paper until dry. Weigh the cake and paper to determine the yield.

Mount the filter paper and cake on the counting card. Determine the counts per minute under standard counting conditions.

Calculation of Curies at Separation Time

Volume of Material Sampled 1.80 x 10⁵ ml.

Volume analyzed 1 x $\frac{0.1}{50}$ x 1/100 = 1/50000 ml.

Yield = $\frac{\text{BaCl}_2 \text{ recovered}}{\text{BaCl}_2 \text{ used}} \times 100 = \frac{30.1}{33.6} \times 100 = 89.6\%$

C/M 3428, 3476 from counter

CCC 3512, 3563 corrected for coincidence

CCB 3485, 3536 corrected for background

CCS 3395, 3445 corrected for standard

CCY 3790, 3732 corrected for yield

CCY - average - 3761

C/M/V 3761 + 1/50000 = 1.88 x 10⁸

Counts per Curie (by definition) - 2.22 x 10¹²

Geometry 4.11%

Curies at Separation Time $\frac{1.88 \times 10^8 \times 1.80 \times 10^5}{2.22 \times 10^{12} \times 0.0411} = 3710$'s

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Calculation of Curies at Shipping Time

Determine the number of hours from separation time to shipping time. Read off from the "time vs % of product" curve the percentage of Curies existing at shipping time. Use this figure to calculate the Curies present at shipping time.

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"T" DETERMINATION

Pipette 1 ml from the 1st dilution of the metal solution sample (0.1/50) into a 25 ml volumetric flask. Add 5 ml of ammonium acetate beffer (140 g NH₄Ac, NH₄OH to bring pH to 7.5, dilute to 1000 ml), and 5 ml. of 10% sodium salicylate. Adjust pH to approximately 8 with ammonium hydroxide using litmus paper as indicator. Dilute to mark. Read in a Spectrophotometer at 380 m μ (PC-6 filter) against a blank of all reagents. Note absorption percentage. From graph read the amount of "T" in gamma.

Calculation

$$\text{Absorp.} = 41.0\% = 820 \gamma \text{ of "T"}$$

$$820 \gamma = 410 \text{ mg./ml.}$$

$$\text{Volume of material sampled} = 1.93 \times 10^5 \text{ ml.}$$

$$\frac{0.410 \text{ g/ml} \times 1.93 \times 10^5 \text{ ml.}}{1.10 \times 10^5 \text{ g/slug}} = 72.0 \text{ slugs}$$

Reagents:

1. Redistilled water is used in all solutions.
2. Extraction Dithizone: 30 mg. dithizone and 5 ml. absolute alcohol per liter of redistilled chloroform.
3. Standard Dithizone: 10 mg. dithizone and 5 ml. absolute alcohol per liter of redistilled chloroform. The solution is filtered through a dry paper, stored in a glass stoppered pyrex bottle and protected from light. Keep in the refrigerator.
4. Ammonium Citrate: 50 g. ammonium citrate dissolved in water, made alkaline to phenol red by adding ammonia and purified by shaking with small amounts of dithizone extraction solution until fresh portions remain green. Make to 100 ml. volume.
5. Potassium Cyanide: 10 g KCN per 100 ml. made by dilution of a solution of 50 g. of the salt per 100 ml. of water. Purify this concentrated solution in the same manner as #4.
6. Ammoniacal Cyanide Buffer: 2 g. KCN and 15 ml. of NH_4OH (sp. g. 0.9) per 100 ml. water. Purify this solutions as above.
7. Acid Buffer Solution: 9.1 ml. of concentrated HNO_3 diluted to approximately 500 ml. Add 2 ml. of 1% methyl orange. Add NH_4OH until color begins to change. Add 50 ml. of 0.1 M potassium acid phthalate. Add dropwise HNO_3 or NH_4OH until the color is a definite pink (pH 3.4). Dilute solution to 1 liter.

NOTE: All glassware must be lead free.

Procedure:

Introduce 15 ml. of ammonium citrate solution into a 125 ml. separatory funnel. Add the sample - generally not over 1/2 ml. and 1 drop of phenol red indicator. Make solution alkaline by adding ammonia. Add 3 ml. 10% potassium cyanide. Add 10 ml. of dithizone extraction solution. Shake vigorously for one minute. Drain the dithizone solution into a 125 separatory funnel containing 30 ml. of acid buffer solution. Introduce 5 ml. more of extraction dithizone into the original separatory funnel. (Shake* vigorously). (Combine extract in the acid buffer. To the combined extract, add dilute nitric dropwise until a definite pink color remains. Shake vigorously for 1 minute. Discard the dithizone. Wash the buffer with 5 ml. of pure chloroform. Discard all the wash. Add 10 ml. of standard dithizone by means of a pipette, 3 ml. of ammoniacal cyanide buffer (pipette) and 1 ml. of concentrated ammonia. (Shake* vigorously for 1 minute.) Filter the dithizone phase through a dry filter paper into a comparator tube and measure the absorption at 5100 A. Read from the standard absorption curve the amount of lead present. Check the instrument with pure chloroform which should give 100% transmission. Blanks and checks should be run on the curve frequently.

* Place separatory funnel in shaking machine and shake for 30 seconds.

NOTE: For 6P samples the one step method is adequate.

DIP MICROSAMPLERS

Description:

Microsamplers are a dip type sampler to obtain samples ranging in size from 10 lambda to 4 lambda. The samplers fill by capillary action once the tip has been immersed in the solutions to be sampled.

A glass sleeve to which has been fused a tip with a capillary bore of such a length that its capacity is about 5 lambda is held in a stainless steel sleeve. A diagram accompanies this paper.

Purpose:

Some solutions in the operation of 706-D contain activity of such an order of magnitude that a large sample are too hot to handle or would be robbing the total of a substantial amount of product. It was seen that a micro amount of sample was all that was necessary for control purposes. To fit the situation this sampler was devised.

Solutions sampled are the final product solution which carries all product in less than 100 cc of solution and the fuming nitric waste which is too hot to take more than a small amount.

Calibration:

The method of calibration is very simple - - an active solution is used having sufficient counts so that 5 lambda will count 5000-7000 Beta counts per minute.

The samplers are filled to capacity by capillary action. The tip is washed before emptying with only a drop or two of 2N HNO₃. By means of a rubber bulb, the contents of the sampler is blown into a watch glass.

The sampler is rinsed with 2N HNO₃ into the watch glass and the outside of the tip is rinsed. This is evaporated and counted.

Along with this a standard 5 lambda portion is evaporated and counted. By setting up a ratio of the counts contained in the known pipette to the unknown, it is possible to calculate the unknown, e.g.

Std.	5 lambda pipette contained	5000 cts/min.
Unknown	" " "	7500 "

$$\frac{5}{5000} :: \frac{X}{7500}$$

$$X = 7.5 \text{ Lambda}$$

Mounting:

The samplers are made in two different pieces, in different places and mounted in the Lab. The glass part is fabricated in the glass shop and the sleeve in maintenance shops. Calibration is done before mounting.

DeKhotinsky cement is used to hold the two pieces together. The glass is warmed in a Bunsen Flame and cement applied. Before inserting the glass into the steel sleeve, it is warmed. This is done to insure good flowing of the cement. One and one-half inches is the distance from end of sleeve to glass tip.

*Dechroo
C.*

CLINTON LABORATORIES

CENTRAL FILES NUMBER
46-1-189

B-137

X File _____

Date January 14, 1946

Those Eligible
To Read The
Attached

Subject Shipment #16

By J. C. Kaskie *NY 63*

Copy # 2 - Peterson

To M. C. Leverett

Before reading this document, sign and date below:

Name	Date
<i>Mr. Peterson</i>	<i>1/15/46</i>
<i>MT Kelly</i>	<i>1/15/46</i>

Name	Date

INT. 64

Publicly Releasable

This document has received the necessary patent and technical information reviews and can be distributed without limitation.

This document consists of 1 page and 1 copy Series No. 2 of the

1. M. G. Leverage
2. M. D. Paterson
3. W. A. Hedger
4. J. O. Kaskin
5. Central File

1-24-48

M. G. Leverage
J. O. Kaskin

~~SECRET~~

SHIPMENT #16

Sample had a better than usual appearance with a thin film on the wall up to 2" level. The bulk of the product was in the tip of the cone. It was shipped in a tantalum lined cone collar and carrier, with a "Koroal" gasket.

2570 Gms analyzed at 1830
January 18, 1948, last separation time.

4	mg	Cr
6	mg	Fe
7	mg	Hf
11	mg	Br
90	mg	Pb
1800	mg	Ba

J. O. Kaskin

~~Contains information affecting the national defense of the United States within the meaning of the Espionage Laws, its contents and transmission is prohibited by law.~~

~~SECRET~~

CLASSIFICATION CANCELLED

DATE 1/11/67

For The Atomic Energy Commission

H. P. Arnold
Chief, Declassification Branch

CENTRAL FILES NUMBER

46-1-438

A-670

*Done
11-17-69*

[Handwritten signature]

File _____

Those Eligible
To Read the
Attached :

Date January 29, 1946

Subject 706-D Production Run #5
(Shipment #13)

Copy # 2 - Leverett

By A. C. Vallado

To W. A. Rodger

Before reading this document, sign and date below

Name	Date
<u>ms</u>	<u>2-5-46</u>
_____	_____
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46-1-438

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

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

This document consists of 15 pages and 1 figures. No. 2 of 6 copies, Series A

1. W. A. Rodger
2. M. G. Leverett
3. M. D. Peterson
4. A. C. Vallado
5. Central File
6. Heading File

1-25-48

W. A. Rodger

A. C. Vallado

~~SECRET~~

708-D Production Run #5
(Shipment #13)

Mechanical Changes

At the end of Run #4, the transfer funnels into the duplicate glass vessels, B-20 and B-21 were both plugged and the sintered glass disk in B-21 was broken. Also one valve on B-20 was inoperative, the stem having sheared when the valve was in an open position.

The funnels were unplugged, the B-21 disk sealed with paraffin, and the line to the valve on B-20 was sealed off using a hemostat on the end of a pole. This work was accomplished without entering the cell. Although the equipment was in very bad shape it was ordained that we proceed with Run #5.

Dissolver Operations

The 1295 slugs charged to the dissolver for Run #5 were added as follows:

CLASSIFICATION CANCELLED.

Date pushed and charged

Slugs Loaded

DATE 11/7/66

for The Atomic Energy Commission

H. R. Conrad

Chief, Declassification Branch *CR*

8/14
9/4
9/5
9/7
9/8

260
260
260
258
257
1295



The slugs loaded on 8/14 remained in the dissolver between runs. While the contribution of active Barium from these was small, the inactive did not diminish and for the purposes of calculation all 1295 slugs are considered. They are calculated to have contained an average of 6.7 Curies per slug active and 1.2 mg per slug inactive Barium at time of discharge. (Total 8710 Curies and 1550 mg).

*170!
was discharged
8/15 - 8/21
see logbook*

It was planned to make two separate runs. Events occurring during the latter part of the first run (described later) make it impossible to complete that run, so both were combined into a single run.

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W. A. Rodger

2

1/25/46

Before starting either run the heel in the dissolver and the product remaining in A9 (originally called Run 4C) were processed through extraction and transferred to tank B1.

A total of 16 (average 61.3 slugs dissolved) metal solution and four coating removal reactions were carried out without difficulty or incident.

Extraction

name change

Eight extractions were made on series 5A (4C); nine on 5B; and 7 on 5C. The amounts of reagents were normal (2500 ml 6% $Pb(NO_3)_2$ for A batches, 500 ml for all others); (330 # 65% H_2SO_4 and 100# water); except when wastes were added to A9 prior to extraction. The twenty-four decantations were out at 2.5" to 4.5" on the oil liquid-level manometer. Jet A9-ASDB was used. Losses averaged 29 Curies per extraction, or 0.65% of the product from each individual dissolving. See Table II for complete extraction decantation results.

The extraction cakes from each of the three series were given the standard one acid, four water washes, all of 4 gallons volume. Each wash was agitated 5 minutes, settled 70 min. and decanted to a 2.2" to 5.4" heel on the oil liquid level manometer, using jet A9-ASDB. The waste loss in the combined washes were $3\frac{1}{2}$ Curies or less in each of the three series. (See Table I)

Metathesis

The three metathesis were normal and uniform reagent quantities were used. All were decanted to AS using jet A9-ASDB and cutting at 2.3" to 4.4" heels. The 5A waste was very low, but 5B and 5C were rather high (215 and 261 respectively). The metathesis waste from 4B was recycled into 5A-A extraction and the metathesis waste from 5B was recycled into 5C-A extraction. See Table III for metathesis conditions and losses.

The first metathesis solution was jetted to B1 without a cake wash. In B-1 it was treated with fuming HNO_3 to reduce the amount of potassium salts. Then transferred to B-12. The second and third metathesis were each followed with two washes. The cake solution of the third metathesis was transferred to B-12 by way of B-1, so as to recover the Curies lost in the 5A transfer. Final volumes in the electrolysis vessel were 4200, 4050, 4000 ml. respectively.

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1/25/46

SECRETCell B Operation

The first two electrolysis were standard (3 hours at 15 amps., plus 6 hours at 25 amps.). The last electrolysis was eleven hours (4 hours at 15 amps. and 7 hours at 25 amps.), to remove the large amount of lead present. No Barium carrier was added in all electrolysis.

After electrolysis and transfer to precipitator B-6, the product solution from the series of nine metal dissolving and extractions for Run #3 analyzed 2000 Curies (calculated to the estimated shipping time, 2400, Monday 9/10). To compensate for expected losses in the final purification step, 400 Curies of product, were added from the 600 stored in vessel B-1, remaining from Run #4. This spiked solution was evaporated from 4-5 L to 150 cc in B-6, diluted to 550 cc to dissolve all $Ba(NO_3)_2$, and transferred to the glass purification vessel B-20 through "Tygon" lines and the temporary glass suction flask on top of Cell B.

The first fuming HNO_3 precipitation was made in B-20 as usual, but the rate of filtration of the HNO_3 slurry through the sintered glass disc decreased within a few hours from about normal to nearly zero, so that only half had been filtered in 36 hours, by which time the rate was inappreciable. Since no alternate glass purification vessel was available, the Run #5 product in B-20 was washed back into the Cell A precipitator A-9, when it was combined with an approximately equal amount of product from the series of seven extractions that has been intended for Run #6. This combined solution was metathesized, electrolyzed for lead removal, purified in the stainless steel precipitator B-6 by three fuming HNO_3 precipitations, with the separation of the $Ba(NO_3)_2$ by settling, and the final $Ba(NO_3)_2$ cake was dissolved in water and evaporated in the final shipping cone.

The transfer of $Ba(NO_3)_2$ from the glass vessel B-20 to the precipitator A-9 was accomplished by puncturing the sintered glass disc and the bottom of the glass transfer vessel with a pointed stainless steel rod lowered through the sampling hole on a cord, allowing the HNO_3 solution to run through the stainless steel funnel under the glassware into transfer vessel B-22, from which it was jettied through B-10 to A-9. The transfer was followed with enough water washes to dissolve and rinse out the solid $Ba(NO_3)_2$ from the filter disc. This procedure was necessary, as there was no provision for transfer of solution above the filter disc in B-20, and the line between the B-20 and B-21 glass transfer vessels plugged, preventing sucking the solution below the filter disc into B-21, from which it could have been transferred normally.

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As in some earlier $Ba(NO_3)_2$ runs, lead contamination must have occurred in B-6, possibly during sampling, as the lead content therein after electrolysis was only 80 mg., which should have been decreased more than 4 fold by the three fuming HNO_3 treatments, according to previous experience.

The final evaporation to 5½ hours and went without difficulty. The final product solution was transferred to B-19 in two shots.

The $Ba(NO_3)_2$ after evaporation in the cone was only slightly darker in color than shipment #4, and was primarily in the tip of the cone, although a thin film extended about 2" up the walls to the original liquid level.

Analysis of product was as follows:

Product at LST, last separation time (1:30 A.M. , 17 Sept.)
2005 Curies

Pb	170 mg	Sr	11 mg
Fe	9-13 mg	Ba	600-1100 mg
Cr	6-11 mg		
Ni	4 mg		

To supply a high-intensity gamma ray source for experiments in the plant site by Health Physics, the La-containing fuming HNO_3 wastes from run #5 were evaporated in a special cone furnished by H.P.. With some difficulty this La source was delivered at about 1500 on 9/19/45. In order to make the B-9 transfer vessel work, it was necessary to dilute the La solution in B-6 to about 1 liter. As no more than 200 cc could be evaporated in the final cone at one time, the job was done in batches of uncertain size, as no means of measuring the volume added to the cone was available. It is estimated that about 1000 Curies were delivered.

Analyses

In Table I complete analytical data are presented. Table IA gives a summation of losses for the run based on the last two series.

Contamination and Radiation Exposure

The third floor over Cell B became contaminated during the operation and removal of the temporary glass transfer and "Tygon" lines above Cell B. However, no Health Physics personnel over-exposure reports were received. All hot operations were carried out in the presence of H.P. surveyors, and no general body or great specific over-exposures occurred, though some operations required very short exposures of hands in beams up to 40 r/hour.

1/26/46

During the evaporation of the La-containing fuming HNO_3 wastes in B-19, some solids in the solution caused bumping and contaminated not only the B-19 corner of the cell but the top of the plug as well. This gave rise to levels of radiation approximating 10 r/hour over the top of the carrier when it was removed. The carrier was removed and put on the truck without contaminating the building or over-exposing any personnel.

On the night of 9/16 - 9/17, the fan motor blew a fuse and was out of service one and possibly three hours. The warning signal was not in operation and some personnel were exposed to air well over tolerance. All were checked by medical; no member of the Operations group, but one person from Health-Physics was found to have picked up some counts in the lung, which, however has now disappeared. Steps have been taken to prevent a recurrence.

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1/26/46

H. A. Rodger

6

TABLE I
Curies Calculated to 24:00, 9-7-45

Fraction	Code	Curies Product	Curies in Waste
<u>First Series 4G</u>			
First dissolving Extraction Waste	1MA SWMA	105	8.9
Second dissolving Extraction Waste	1MB SWMB	107	5.8
Third dissolving Extraction Waste	1MC SWMC	95.4	6.0
Fourth dissolving Extraction Waste	1MD SWMD	92.7	4.8
Fifth dissolving Extraction Waste	1ME SWME	80.3	14.3
Sixth dissolving Extraction Waste	1MF SWMF	69.2	13.3
Seventh dissolving Extraction Waste	1MG SWMG	85.7	10.4
Eighth dissolving Extraction Waste	1MH SWMH	33.8	14.5
Extraction Cake Washes Metathesis Wastes	SWW SWC		0.7 12.0
Cake Soln. in A-9	9P	596	

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TABLE I
(Continued)

(All Curies are Calculated to the Shipping Time
24:00, 9-15-46)

	Code	Curies Product	Curies in Waste	% Total Product Dissolved
<u>First Series</u>	5A			
Product in Soln. From First Eight Dissolvings	9P	360		
First Fuming HNO ₃ Waste	5W1N		7.4	0.2
PbO ₂ Soln. Waste	5W2b		2.9	0.1
<u>Second Series</u>	5B			
First Dissolving Extraction Waste	1MA 8WMA	237	7.4	0.2
Second Dissolving Extraction Waste	1MB 8WMB	207	6.0	0.1
Third Dissolving Extraction Waste	1MC 8WMC	190	16.0	0.4
Fourth Dissolving Extraction Waste	1MD 8WMD	258	11.6	0.5
Fifth Dissolving Extraction Waste	1ME 8WME	222	97.8	2.3
Sixth Dissolving Extraction Waste	1MF 8WMF	211	45.2	1.0
Seventh Dissolving Extraction Waste	1MG 8WMG	188	30.1	0.7
Eighth Dissolving Extraction Waste	1MH 8WME	247	106	2.4
Ninth Dissolving Extraction Waste	1MI 8WMI	241	37.8	0.9
Cake Wash	8W		4.9	0.1

TABLE I
(Continued)

(All Curies are Calculated to the Shipping Time
24:00, 9-16-46)

Fraction	Code	Curies Product	Curies in Waste	% Total Product Dissolved
Metathesis Wastes	8WC		98.6	Recycled
Metathesis Wash	8WCW		215	Recycled
Sample Product After Electrolysis	6P-2	1688		
Water Rinse of B-6	6P rinse		73.5	1.7
PbO ₂ Solution waste	3WPb		10.7	0.2
<u>Third Series</u>	3C			
First Dissolving Extraction Waste	1MA 8WMA	257	8.6	0.2
Second Dissolving Extraction Waste	1MB 8WMB	262	0.4	-
Third Dissolving Extraction Waste	1MC 8WMC	284	98.2	2.3
Fourth Dissolving Extraction Waste	1MD 8WMD	276	20.2	0.7
A-8 Rinse	A8 Wash		45.9	1.0
Fifth Dissolving Extraction Waste	1ME 8WME	235	26.6	0.6
Sixth Dissolving Extraction Waste	1MF 8WMF	236	26.2	0.6
Seventh Dissolving Extraction Waste	1MG	148	54.2	1.2
Eighth Dissolving	1MH	36.7		
Cake Wash	8WF		4.8	0.1
Metathesis Waste	8WC		181	4.2

1/26/46

TABLE I
(Continued)

(All Curies are Calculated to the Shipping Time
24:00, 9-16-46)

Fraction	Code	Curies Product	Curies in Waste	% Total Product Dissolved
Metathesis Wash	8XCV		256	
Product in B-1	1P	2680		5.9
Product in Soln. After Electrolysis	6P	3070		
First Fuming HNO ₃ Waste	3WfNA		574)	
Second Fuming HNO ₃ Waste	3WfNAB		585)	
Third Fuming HNO ₃ Waste	3WfNABO		758)	
Final Product Sample	6PF	2008		17.7
B-6 Wash	6WfF		43.5	47.0
PbO ₂ Solution Waste	3WfPb		47.8	1.0
A-8 Rinse	8WfAY		50	1.0
Total Curies Dissolved		4356		100

~~W. A. Rodger~~

TABLE 1ASummation of Ba Losses - Run #5~~SECRET~~

Fraction	Curies	%
Total Dissolved (Calculated as 1295 slugs)	8,710	100
Decay Loss (34, 13, 12, 10, 9 Days)	4,550	52.2
Known Losses in Cell A	864	9.9
Known Losses in Cell B	1039	12.0
Yield	2005	23.0
Material Balance		97.1
Total Curies found in dissolver solution calculated to---2400. 4/16/45	4356	100
Cell A Waste losses	864	19.8
Cell B Waste losses	1039	23.7
Product Yield	2005	46.0
Material Balance	3908	89.5

~~SECRET~~

TABLE II
Extraction Decantations

Batch	Rate of Decantation (Gal/min)	Heel cc	Ba Loss	
			Curies	% of Product Present at Decantation
5AA	2.0	3310	8.9	8.5%
5AB	2.6	3310	5.8	2.7
5AC	2.5	3760	6.0	1.9
5AD	2.6	4370	4.8	1.1
5AE	3.4	4000	14.5	2.9
5AF	2.3	2370	13.3	2.3
5AG	2.7	4000	10.4	1.6
5AH	2.9	3760	14.5	2.1
5BA	2.4	4370	7.4	0.8
5BB	2.8	4370	6.0	0.6
5BC	2.8	4370	16.0	1.2
5BD	2.3	4370	11.6	0.7
5BE	2.6	4370	97.8	5.5
5BF	2.4	4640	45.2	2.3
5BG	2.5	4370	30.1	1.3
5BH	2.3	4480	106	4.3
5BI	2.7	2800	37.8	1.4
5CA	2.8	4370	8.8	0.3
5CB	3.3	4370	0.4	0.01
5CC	2.6	4370	98.2	2.8
5CD	3.1	4370	30.2	0.3
5CE	3.1	4550	26.6	0.4
5CF	2.8	4400	26.2	0.6
5CG	3.4	4370	54.2	1.2

TABLE III
METATHESIS

Treatment	Settling Time (Hours)	Heal co	Ba Loss	
			Curies	%
<u>5A</u>				
First Metathesis	2½	3650)	12.0	-
Second Metathesis	2½	6340)		
<u>5B</u>				
First Metathesis	2½	4750)	98.6	Recycled
Second Metathesis	2½	4900)		
First Metathesis Cake Wash	2½	4750)	215	Recycled
Second Metathesis Cake Wash	2½	1300)		
<u>5C</u>				
First Metathesis	2½	5760)	181	4.1
Second Metathesis	2½	3900)		
First Metathesis Cake Wash	2½	4320)	256	5.8
Second Metathesis Cake Wash	2½	5180)		

~~708-2-23-46~~

TABLE IV
Time Cycles

<u>Operation</u>		<u>Hours Required</u>	
<u>Charging</u>			
5AA	260 slugs	7	
5BA	260 "	6 $\frac{1}{2}$	
5BD	260 "	7 $\frac{1}{2}$	
5BH	258 "	5 $\frac{1}{2}$	
5CA	257 "	7	
<u>Coating Removal</u>			
First		2 $\frac{1}{2}$	
Second		2 $\frac{1}{2}$	
Third		3	
Fourth		2 $\frac{1}{2}$	
Fifth		2 $\frac{1}{2}$	
<u>Metal Solution and Extraction</u>			
		<u>Solution</u>	<u>Extraction</u>
<u>5A</u>	5AA	4	10 $\frac{1}{2}$
	5AB	3 $\frac{1}{2}$	10 $\frac{1}{2}$
	5AC	4	10 $\frac{1}{2}$
	5AD	4 $\frac{1}{2}$	10 $\frac{1}{2}$
	5AE	5	12 $\frac{1}{2}$
	5AF	5	10 $\frac{1}{2}$
	5AG	10 $\frac{1}{2}$	10 $\frac{1}{2}$
	5AH	12	13
<u>5B</u>	5BA	6	10 $\frac{1}{2}$
	5BB	5 $\frac{1}{2}$	10 $\frac{1}{2}$
	5BC	7	10 $\frac{1}{2}$
	5BD	5 $\frac{1}{2}$	10 $\frac{1}{2}$
	5BE	4 $\frac{1}{2}$	10 $\frac{1}{2}$
	5BF	6 $\frac{1}{2}$	10 $\frac{1}{2}$
	5BG	6 $\frac{1}{2}$	11 $\frac{1}{2}$
	5BH	5	10 $\frac{1}{2}$
	5BI	3 $\frac{1}{2}$	10 $\frac{1}{2}$

*actually
"4C"* →

TABLE IV
Time Cycles

<u>Operation</u>		<u>Hours Required</u>	
<u>Metal Solution and Extraction</u>		<u>Solution</u>	<u>Extraction</u>
<u>5C</u>	5CA	4	10½
	5CB	3	12
	5CC	4	10½
	5CD	4½	10½
	5CE	5	10½
	5CF	7	10½
	5CG	9½	12½
<u>Extraction Cake Washes</u>			
	5A		8½
	5B		8
	5C		6½
<u>Metathesis</u>			
	5A		8½
	5B		9½
	5C		8½
<u>Metathesis Cake Washes</u>			
	5A		Omitted
	5B		5½
	5C		5½
<u>Metathesis Cake Solution</u>			
	5A		-
	5B		3/4
	5C		7*
<u>Electrolyses</u>			
	5A		9
	5B		9
	5C		11

* A9 to B-12 through B-1.

TABLE IVTime Cycles

<u>Operation</u>	<u>Hours Required</u>
<u>Fuming HNO₃ Ppt. in B-6</u>	
First	4
Second	4 $\frac{1}{2}$
Third	4 $\frac{1}{2}$
<u>Volume Reduction in B-6</u>	
First	3
Second	2 $\frac{1}{2}$
<u>Solution and Sampling</u>	2
<u>Evaporation</u>	
First Shot	3 $\frac{1}{2}$
Second Shot	3
<u>Loading</u>	4

A. C. Vallado

CENTRAL FILES NUMBER

46-2-198



B-137

File 6

Date February 13, 1946

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To Read The
Attached

Subject 706-D Production Run #6

(Shipment #14)

By A. C. Vallado

Copy # 2 - Leverett

To W. A. Rodger

1/15/46

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This document consists of 12
pages
2 copies, Series A

1. W. A. Rodger
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W. A. Rodger Classification Cancelled 2-13-46

A. C. Vallado
By Authority Of [Redacted]
By [Signature] Date AUG 23 1971

706-D Production Run #6 CLASSIFICATION CANCELLED
(Shipment #14) Ted Davis 11/28/94
ADD signature Date

Mechanical Changes

Between runs #5 and #6, [Redacted] were decontaminated and a great deal of maintenance work was done, particularly in Cell B. For a complete account of this work see report written by W. P. Bigler on "Maintenance work done in 706-D for the period September 20 to November 20."

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

Dissolver Operations

The 803 slugs charged to the dissolver for Run #6 were added as follows:

<u>Date pushed and charged</u>	<u>Slugs loaded</u>
11/23	291
11/24	294
12/1	218
	<u>803</u>

The first 15 slugs dropped stuck in the delivery chute, apparently the last slug added on the previous dummy run had stuck causing the trouble. This was an Argonne egg (cylinder 2 1/2" x 2 1/4") which has one dimension slightly over 3". The dissolver was filled 3/4 full of 60% HNO₃ and heated to 95°C. Five minutes of this treatment broke the slug loose and cleared the chute. During this operation an air ejector, installed at the slug chute opening, discharged the off-gases outside the building.

The slugs loaded were calculated to have an average of 7.6 Curies/slug active, and 0.64 mg/slug inactive Ba (totals: 6064 Curies and 520 mg) at time of discharge.

The run was divided into two series. The first consisting of seven dissolvings and extractions followed by metathesis, recovery, cake solution and electrolysis. The second series was to make up for the very high losses incurred by the failure of the A9 agitator. It consisted of four dissolvings and extractions. The amounts of reagents used for the first seven dissolvings were increased 20% (510 # 60% HNO₃, 220# water) in order to dissolve 85 slugs/batch and thereby reduce losses due to decay. The next four dissolvings,

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2/13/46

the amounts of reagents were normal (420# 60% HNO_3 , 180# water), since the larger batches were causing A9 to overflow. The amounts of reagents used for the first two coating removals were changed (136# 60% HNO_3 , 306# 35% NaOH) to use 35% NaOH so as to prevent it from freezing in the outside tank M-11. The reagents for the third coating removal was based on 200 slugs (95# 60% HNO_3 , 216# 35% NaOH).

A total of eleven (average 77.6 slugs dissolved) metal solutions and three coating removal reactions were carried out.))

Extraction

Seven extractions were made on the first series and four on the second. The amounts of reagents were increased 20% (280# 90% H_2SO_4 , 1000 cc 20% $\text{Pb}(\text{NO}_3)_2$ for A batches, 300 cc 20% $\text{Pb}(\text{NO}_3)_2$ for all others), because of the increase in metal dissolved, and the lead carrier was re-used throughout each series, as usual. The dummy runs preceding this run showed that A9 overflowed with this increased batch size, so the 120# of water usually added to dilute to flow sheet concentrations was omitted and 90% H_2SO_4 used instead of 65% H_2SO_4 . After batches 6F and 6G, it became apparent that extractor A9 was running close to overflowing. Therefore, in all subsequent extractions the reagents were reduced in accordance with a smaller batch size.

On the fifth extraction, the A9 agitator froze. Maintenance succeeded in freeing it, but thereafter it ran noisily and drew twice the normal current. At this time it was not certain that the trouble was in the bearings and not inside the tank, so it was decided to use the air sparger instead of the agitator. The air sparging was not adequate for extraction. Very high losses (up to 1000 Curies) were experienced and the air on the third level became highly contaminated. The run was continued, however, recovering the high losses in A-11. An attempt was made to return the recovered material from A-11 to A9. This was unsuccessful as only 500 Curies were transferred to Cell B on the first attempt. A 40% K_2CO_3 rinse of A8 and A-11 recovered at least some of the product. This was returned to A9 where a PbSO_4 extraction was made upon it. In the second series the A9 agitator was operated manually by means of a crank and appeared to operate satisfactorily.

Eight of the eleven decantations were cut at 3.2" to 4.4" on the oil liquid level manometer. Jet A9-ABDB was used. However, on batches 6F, G and H, both A9-ABDB and A9-ABDA lines were plugged by the air sparging, and the metal waste solutions were transferred to A-11 by way of A8, settled and decanted to A-5. The losses on these batches were high (21, 60, and 87 respectively), especially on 6H, where the oil manometer failed to function properly, making the decantation "blind" below 24". Losses averaged 25 Curies per extraction or 8.7% of product from each individual dissolving. See Table II for complete extraction decantation results.

The extraction cakes from each series were given the standard one acid, four water washes, all of 4 gallon volume. The washes were agitated 5 minutes, settled 70 minutes (5 hours for the first series due to large volume) and decanted to a 3.1" to 8.5"* heel on the oil manometer using jet A9-ASDB. The waste loss in the combined washes was 25 Curies.

* Probably high due to leak in manometer line.

Metathesis

Reagent quantities in both metatheses were increased 14% over previous flowcharts, for the larger amount of Pb present. Both were decanted to A-8 using jet A9-ASDB and cutting at 2.2" to 2.5" heels. As the waste losses were low (17.0 and 25.0 Curies) they were discarded rather than being recycled into the next extractor batch, as the volume in the A9 extractor was at a premium. See Table III for metathesis conditions and losses.

Both metatheses cake solutions were transferred into the electrolysis vessel in Cell B in two batches followed by two washes via B26 and B27. Except for a valve sticking momentarily and one ball joint leaking slightly on the first transfer, the operation was successful. The crud filter system (B26 and B27) worked very well mechanically and was observed to remove fairly large quantities of suspended solids and floating oily material. Final volumes in the electrolysis vessel were 4100 and 4050 cc respectively.

Cell B Operation

The electrolyses time was increased (3 hours at 15 amps. plus 7 at 25 amps.) to remove the larger amounts of Pb present. 250 mgs of Barium carrier were added in the first electrolysis.

After the second electrolysis, the product was added to that from the first series in B6. It was then taken to dryness, dissolved in 2 liters of water and reconcentrated to 350 cc. On transferring to glassware, B11 was plugged, but was easily broken loose after which it worked without difficulty. In B21 a fuming nitric and an HCl-ether treatment were made. These went without difficulty.

The product was dissolved in 100 cc water and transferred to B17 for sampling. The product assay (1100 Curies) was somewhat lower than expected, but it is believed that two factors, poor samples (all were observed to have bubbles in the tubes), and failure to transfer all of the solution to B17, indicate that the actual value should have been 10-20% higher. A second solution, 150 cc, was put through the system picking up an additional 100 Curies. Subsequent rinses of B9, B11 and B21 were made to recover most of the missing product.

The customer's report indicated that only 400 Curies had been received in this shipment. No good explanation for this was available. The 1000 Curies found during rinsings were in all probability not transferred from B17 to B19. A possible explanation for this would be that one or more of the B21 valves had leaked. Subsequent tests have shown that they were not leaking when tested, however.

On subsequent runs the transfer from B17 to B19 has been made by way of B8 rather than by way of the glassware. It was also planned to make a physical measurement in this and all succeeding runs. Several runs should give an empirical check on the amount of product shipped.

The final evaporation on both solutions took $8\frac{1}{2}$ hours and went without difficulty. The dried product had a slightly dark appearance. The bulk of the solid was in the tip of the cone although there was a thin film on the wall up to a 2" level.

Analysis of product was as follows:

Active Barium 1200 Curies at 0500 Dec. 6, 1945

It is estimated that at least 300 more Curies were actually sampled as the volume in the sampling vessel was lower than expected and it was not possible to obtain a perfect sample for assay.

Contaminants

Pb	42 mg.
Fe	9 mg.
Cr	6 mg.
Ni	20 mg.
Sr	6 mg.
Ba	1130 mg.

Other Products

Following shipment of the preparation, a rinse of B21T and B17 was put in a cone and delivered to 706-C. It contained about 5 Curies of Ba.

The HCl-ether waste, containing 65 Curies of Ba and about 200 Curies of Sr (est.), was concentrated and given to 706-C.

Earlier in the run condensate from A3 was drawn off and delivered to 706-C. Due to poor technique of collection this solution was relatively low in Iodine.

Analyses

In Table I complete analytical data are presented. Table IA gives a summation of losses for the run.

Contamination and Radiation Exposure

From the standpoint of overexposures and contamination, this was by far the most successful run to date. No general body overexposures occurred during the entire run. The only building contamination occurred when Cell IV was cleaned up. After the transfer through Cell IV sufficient cleaning was done to allow the Cell door to be opened. The level at point of nearest approach was then only 2 r/hour. Some liquid, apparently held up in the gasket, ran out into the floor at that time, however, causing considerable floor activity which was readily cleaned up. The disk itself read about 60 r/hour at point of nearest approach. It was easily removed by use of tongs without overexposing any personnel. A small area near B6 sample blister was the only place in the building which had remained above tolerance for more than a few hours.

As mentioned earlier, high air activities (up to 5 times tolerance) were found on the third level during and after air sparging. It was believed that activity came out vents at the panel boards. All vents were closed and no further high readings were recorded.

One man, H. Bailey #2387, received 365 mr on a special ring film while taking a 6P sample. All other hot samples were taken without overexposure to hands.

No beams were found while the product was in any of the new lead cubicles, although back scattering through the concrete base amounted to about 1 r/hour at point of nearest approach.

TABLE I

(All Curies are calculated to 1200, 12/6/45)

Fraction	Code	Curies Product	Curies in Waste	% Total Product Dissolved
First Dissolving Extraction Waste	1MA 8WMA	388	15	0.5
Second Dissolving Extraction Waste	1MB 8WMB	311	6	0.2
Third Dissolving Extraction Waste	1MC 8WMC	296	13	0.4
Fourth Dissolving Extraction Waste	1MD 8WMD	337	5	0.2
Fifth Dissolving Extraction Waste	1ME 8WME	300	7	0.2
Sixth Dissolving Extraction Waste	1MF 8WMF	352	21	0.6
Seventh Dissolving Extraction Waste	1MG 8WMG	221	60	1.8
Eighth Dissolving Extraction Waste	1MH 8WMEH	342	81	2.4
Ninth Dissolving Extraction Waste	1MI 8WMI	301	10	0.3
Tenth Dissolving Extraction Waste	1MJ 8WMIJ	276	57	1.7
Eleventh Dissolving Extraction Waste	1MK 8WAK	205	4	0.1
Total Extraction Waste Loss	-		279	8.4
Metathesis and Cake Washes	-		263	7.9

(TABLE I
(Continued)

Fraction	Code	Curies Product	Curies in Waste	% Total Product Dissolved
Cell A Rinses	-		162	4.9
First PbO ₂ Solution Waste	3WPb ₁		6	0.2
Second PbO ₂ Solution Waste	3WPb ₂		17	0.5
Total Loss in B6	-		727	21.9
Total Product in B6	6P	2590		
Fuming Nitric Waste	3WIN		9	0.3
HCl-ether waste			65	1.9
Cell B Vessel Rinses			42	1.3
B9, B11, and Glassware Rinse			990	29.7
Total Cell B Losses			1106	33.2
Product Analyses Final Product Estimated Addition TOTAL	17P	1200 300 1500		45.0
Total Dissolved	0	3329		100
Material Balance				100

TABLE IA
SUMMATION OF Ba LOSSES - RUN #6

	Curies	%
Total Dissolved (calculated as 803 slugs)	6064	100.0
Decay Loss (14, 13, 6 Days)	2809	42.3
Known Losses in Cell A	704	10.6
Known Losses in Cell B	1129	17.1
Yield	1500	22.6
Material Balance		92.6
Total Curies Found in Dissolver Solutions Calculated to 12:00, 12/6/45	3329	100.0
Cell A Waste Losses	704	21.1
Cell B Waste Losses	1129	34.2
Product Yield	1500	45.0
Material Balance	3333	100.3

TABLE II
EXTRACTION DECONTINATION

Batch	Rate of Decantation Gal/min.	Heel cc	Ba Loss	
			Curies	% of Product Present at Decantation
6A	3.2	4150	15	3.9
6B	2.7	4150	6	0.9
6C	2.3	4260	15	1.3
6D	2.5	3840	5	0.4
6E	3.2	3840	7	0.4
6F	No Data		21	1.1
6G	No Data		60	2.7
6H	Manometer	Off	81	3.1
6I	3.2	3350	10	0.4
6J	2.2	4380	57	1.8
6K	2.4	4000	4	0.1

TABLE III

METATHESIS

Treatment	Settling Time (Hours)	Resl cc	Curies	% of Product Present at Decantation
<u>6G</u>				
First Metathesis	$2\frac{1}{2}$	3760)	17.4	0.8
Second Metathesis	$2\frac{1}{2}$	3900)		
First Metathesis	$2\frac{1}{2}$	5100)	16.0	0.5
Cake Wash)		
Second Metathesis	$2\frac{1}{2}$	1200)		
Cake Wash)		
<u>6K</u>				
First Metathesis	$2\frac{1}{2}$	3650)	25.9	0.8
Second Metathesis	$2\frac{1}{2}$	3480)		
First Metathesis	$2\frac{1}{2}$	5180)	262	7.9
Cake Wash)		
Second Metathesis	$2\frac{1}{2}$	500)		
Cake Wash)		

TABLE IV
TIME CYCLES

<u>Operation</u>			<u>Hours Required</u>
<u>Charging</u>			
6A	291	Slugs	16 $\frac{1}{2}$ *
6C	294	"	10
6H	218	"	8
<u>Coating Removal</u>			
First			2 $\frac{1}{2}$
Second			3
Third			2 $\frac{1}{2}$
<u>Metal Solution and Extraction</u>			
		<u>Solution</u>	<u>Extraction</u>
	6A	4 $\frac{1}{2}$	10 $\frac{1}{2}$
	6B	4 $\frac{1}{2}$	10
	6C	3 $\frac{1}{2}$	10 $\frac{1}{2}$
	6D	4	10 $\frac{1}{2}$
	6E	4	10 $\frac{1}{2}$
	6F	4 $\frac{1}{2}$	10 $\frac{1}{2}$
	6G	5	22 **
	6H	4 $\frac{1}{2}$	14 $\frac{1}{2}$ **
	6I	4 $\frac{1}{2}$	10
	6J	4 $\frac{1}{2}$	10 $\frac{1}{2}$
	6K	5 $\frac{1}{2}$	10
		11	10
<u>Extraction Cake Washes</u>			
	6G		12 $\frac{1}{2}$
	6K		6 $\frac{1}{2}$
<u>Metathesis</u>			
	6G		9 $\frac{1}{2}$
	6K		9
<u>Metathesis Cake Washes</u>			
	6G		5
	6K		5 $\frac{1}{2}$

* Slug chute plugged.
 ** Jets A9-ASDB and A9-ASDA plugged.
 Transferred to A8 to A11 and settled in A11 decanted to A5.

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B-137

Date February 25, 1946

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Subject 706-D Production Run #7

(Shipment #15)

By A. C. Vallado

Copy # 3 - Peterson

To W. A. Redger

Before reading this document, sign and date below:

Name	Date	Name	Date
<u>M. Killey</u>	<u>2/27/46</u>		
<u>Baldwin</u>	<u>2/28</u>		



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David R. Hamlin 1/31/95
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This document consists of 11 pages and 2 figures. No. 3 of 6 copies. Series A.

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2-20-46

W. A. Rodger

Classification Cancelled

A. C. Vallado

By Authority Of

By CAB Date AUG 22 1971

706-D Production Run #7
(Shipment #15)

Mechanical Changes

After run #6, Cell A was decontaminated to a point where about 10 minutes time was available in A9. Then it became known that run #7 must be started at once, so it was decided to go ahead with repairs to A9 on an emergency basis. The shell was removed without much difficulty and it was found that the lowest agitator bearing was badly worn, the agitator was wound around the liquid level line, two lines were bent, one decant line (to B-1) was missing its cap, and there was a bad leak in the liquid level line. No attempt was made to fix the bearing as this would have taken several weeks. A "tie ring" was installed on the spacer ring at the bottom of the tank to keep the vibration of the shaft to a minimum, a new liquid level line was installed, and the bent lines were straightened. No other repairs were undertaken at this time.

Dissolver Operations

The 924 slugs charged to the dissolver for run #7 were added as follows:

<u>Date pushed and charged</u>	<u>Slugs loaded</u>
12/14	310
12/15	364
12/18	250
	<u>924</u>

CLASSIFICATION CANCELLED

J. Morgan 12-30-99
ADD signature Date

Single rereview of ~~SECRET~~ classified the most successful run to date, none of the 12/18 documents was shown by DOE Office of into the run; all four solutions made after 12/18 (declassification) and in August 12, 1964 by "series of extractions which was not needed. This "Stand-by" was used as a starting heel for run #8. For purposes of calculation, the run had been considered to consist of the first 674 slugs charged to the dissolver. These slugs were calculated to have an average of 7.1 Curies per slug active and 0.75 mg per slug inactive barium (Total 4745 Curies and 507 mg) at time of discharge.

Due to the desire for a quick shipment by our customer, 85 slug batches were again used throughout the metal solutions, in order to speed up the run. Although it was known from run #6 that the A9 extraction tank would operate close to overflow. The run was a single series, consisting of two coating removals and seven metal solutions (average 86.3 slugs dissolved), all of which went without difficulty or incident.

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Extraction

All seven extractions were on the 85 slug, 90% H_2SO_4 , basis. The seven decantations were out at 3.4" to 3.0" on the oil liquid level manometer. Jet A9-ABDB was used throughout with the exception of 7A when jet A9-ABDB plugged and SWMA solution was transferred to All via A8 and decanted to A5. On batch 7E the waste loss was 205 Curies, well above normal. This was believed to be due to pick-up of residual activity from A8. The SWMA solution was transferred to All via A8 were 200 cc of 20% $Pb(NO_3)_2$ were added; solution was agitated, settled and decanted to A5. Losses averaged 34 Curies per extraction on 8.9% of product from each individual dissolving. See Table 2 for complete extraction decantation results.

The extraction cake was given a 25% H_2SO_4 wash (two shots of 40% each in 4-11 and jetted to 49 to recover Curies probably lost during transfers) and four water washes of 4 gallons each. The H_2SO_4 washes were combined in A9, agitated 5 minutes, settled one hour, and decanted to a 2.5" heel using jet A9-ABDB. The four water washes were agitated 5 minutes, settled 70 minutes and decanted to a 3.0" - 3.5" heel on the oil liquid level manometer using jet A9-ABDB. The waste loss in the combined washes was 1.5 Curies which was very low (see table I).

Metathesis

The metathesis was normal and uniform reagent quantities (similar to run #6) were used. Both metathesis shots were decanted to A8 using jet A9-ABDB and cutting at 2.0" to 2.4" heel on the oil liquid level manometer. The combined waste loss of the metathesis and metathesis cake wash was rather high (168 Curies), and was added to the first extraction of the next series, which will be the starting heel for run #8. See Table 3 for metathesis conditions and losses.

The metathesis cake solution was transferred to the electrolysis vessel (B12) in Cell B in a single batch followed by a water wash, via B26 (crud removal filter) and B27. No difficulty was encountered during this transfer and the system worked very well. The final volume in this electrolysis vessel was 450 cc.

Cell B Operation

The electrolysis time was increased (4 hours at 15 amps., plus 8 hours at 25 amps.) to remove the larger amounts of Pb present. No barium carrier was added.

After electrolysis the product was transferred to precipitator B6, evaporated to dryness, dissolved in 2 liters of water and reconcentrated to a 200 cc volume. The solution was then transferred to B21R by way of

[REDACTED]

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2/21/46

B11 without difficulty, where a fuming nitric precipitation was made. The fuming nitric filtrate was sucked over to B20T from B21T and then blown to B3, as it was believed that transferring directly from B21T might blow the precipitate off the disc. A barium chloride precipitation and subsequent washings were made in B21R and the HCl-ether wastes and washes were combined and sucked over to B20T from B21T for storage in the event of high losses. However, the combined losses were only 66 Curies, calculated to shipping time 0600 12/20/45, so the wastes were discarded.

The product solution was transferred from B17 to B19 by way of the moveable pipette B8 rather than by way of the glassware, as high losses were experienced in run #8 by the latter method. The final evaporation took 5½ hours and went without difficulty. The bulk of the solution was transferred to B19 in one shot followed by a 75 cc water rinse.

Final analysis of product was:

<u>Active Barium</u>	2100 Curies at 0600	Dec. 20, 1945
Pb	18 mg	
Fe	.36 mg	
Cr	2 mg	
Ni	4 mg	
Sr	25 mg	
Ba	700 mg	

The product had a slight dark appearance. The bulk of the product was in the tip of the cone, although there was a thin film up to 2" above the bottom. The customer's report indicated that 2300 Curies product were found.

Analyses

In Table I complete analytical data are presented. Table IA gives a summation of Barium losses for the run. Table IB gives direct radiation measurement which were made by moving the uncovered cone under the chimney and taking readings at the top of the cell.

Contamination and Radiation Exposure

No overexposures occurred during run #7. High air activity, which had been occurring during runs #6 and #7, was reduced by increasing the cell vacuums. This was accomplished by covering part of the intake openings.

During the repair of A9 eleven overexposures were reported; four were to maintenance personnel and were about 120 mr; one was to maintenance supervision (120 mr); one to Operations personnel (120 mr); and five to Operations supervision ranging from 100 mr to 360 mr. The 100 to 120 mr exposures were caused by numerous beams in the cell.

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W. A. Rodger

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2/21/46

On 12/15/45 during batch 7B dissolving, hot fumes backed up into the sinks in the Analytical Laboratories causing high air counts throughout the building. This situation was remedied by stopping the sinks. It appears that the trap recently installed in this line has not succeeded in correcting the condition and it will be necessary to install traps on each sink.

~~CONFIDENTIAL~~

TABLE I

(All Curies are Calculated to the Shipping Time
1200 12/21/45)

Fraction	Code	Curies Product	Curies in Waste	% Total Product Dissolver
First Dissolving Extraction Waste	1MA 8WMA	360	14.3	0.5
Second Dissolving Extraction Waste	1MB 8WMB	375	23.2	0.8
Third Dissolving Extraction Waste	1MC 8WMC	465	6.2	0.2
Fourth Dissolving Extraction Waste	1MD 8WMD	400	92.2	3.3
Fifth Dissolving Extraction Waste	1ME 8WME	377	80.9	2.9
Sixth Dissolving Extraction Waste	1MF 8WMF	439	19.9	0.7
Seventh Dissolving Extraction Waste	1MG 8WMG	345	8.8	0.3
Total Extraction Wastes			246	8.9
A8 Rinse	8AV		17.8	0.6
Cake Wash	8WV		1.5	0.05
Metathesis Waste	8WC		166	6.0
Metathesis Wash	8WVC		168	combined
Product from Run 6- Cell B	-	427		
A9 Rinse			11	0.4
Product Sample	6P	1864		
B6 Rinse	8VW		14.4	0.5

TABLE I
(Continued)

Fraction	Code	Curies Product	Curies in waste	% Total Product Dissolved
Final Sample of Product	17P	2100		
Fuming HNO ₃ Waste	SWIN		18	0.6
HCl-ether waste	SW-HCl		36	1.3
Glass Rinse	-		30	1.1
Total Curies Dissolved		2761		

2/22/46

TABLE IA

Summation of Ba Losses for Run #7

Fraction	Curies	%
Total Dissolved (calculated as 674 slugs)	4745	100
Decay Loss (7, 6 days)	1415	29.8
Known Losses in Cell A	282*	6.0
Known Losses in Cell B	99**	2.1
Yield	1952	41.5
Material Balance		79.2
Total Curies Found in Dissolver Solution calculated to 1200 12/21/45	2761	100
Cell A Waste Losses	282*	10.2
Cell B Waste Losses	99*	3.6
Product Yield	1952	70.7
Material Balance	2333	84.5
<p>* About 10% low due to transfers from A8 to A9. ** About 10% low due to no 3WPb sample.</p>		

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TABLE IB

Direct Radiation Measurements
Run #7

Curies Analyzed	2100	
Instrument	Time in Hours	R/Hour
25 R Meter	16	20.4
	26.5	28.2
G. E. Chamber	16	22.4
	17	23.5
	26	30.8
	27.5	31.45
100 R Meter	16	-
	26.5	25.8
250 R Meter	-	-

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TABLE II

Extraction Decantation

Batch	Rate of Decantation Gal/Min	Heel cc	Ba Loss	
			Curies	% of Product Present at Decantation
7A	No-Data	No Data	14.3	3.9
7B	3.2	3830	23.2	3.1
7C	No Data	4080	6.2	0.5
7D	No Data	3500	92.2	Recycled
7E	2.7	4080	80.9	Recycled
7F	2.5	4145	19.9	0.8
7G	2.6	4400	8.8	0.3

~~SECRET~~

TABLE III

Metathesis

Treatment	Settling Time (Hours)	Reel cc	Ba Loss	
			Curies	% of Product Present at Decantation
First Metathesis	$2\frac{1}{2}$	5400)	----- 166	6.0
Second Metathesis	$2\frac{1}{2}$	3760)		
First Metathesis Cake Wash	$2\frac{1}{2}$	4900)	----- 168	Combined
Second Metathesis Cake Wash	$2\frac{1}{2}$	3400)		

~~SECRET~~

TABLE IV

Time Cycles

<u>Operation</u>		<u>Hours Required</u>
<u>Charging</u>		
7A	310 Slugs	6
7C	364 Slugs	9 $\frac{1}{2}$
<u>Coating Removal</u>		
First		2
Second		2
<u>Metal Solution and Extraction</u>		
	<u>Solution</u>	<u>Extraction</u>
7A	3	9 $\frac{1}{2}$
7B	8 $\frac{1}{2}$	10 $\frac{1}{2}$
7C	3 $\frac{1}{2}$	10
7D	4 $\frac{1}{2}$	10 $\frac{1}{2}$
7E	5 $\frac{1}{2}$	10 $\frac{1}{2}$
7F	6	10
7G	6	10 $\frac{1}{2}$
<u>Extraction Cake Wash</u>		8 $\frac{1}{2}$
<u>Metathesis</u>		8 $\frac{1}{2}$
<u>Metathesis Cake Wash</u>		5 $\frac{1}{2}$
<u>Metathesis Cake Solution</u>		1 $\frac{1}{2}$
<u>Electrolysis</u>		12
<u>Volume Reduction in B6</u>		4 $\frac{1}{2}$
<u>Fuming Nitric Precipitation in B21</u>		2 $\frac{1}{2}$
<u>BaCl₂ Precipitation, Washing and Product solution in B21</u>		1 $\frac{1}{2}$
<u>Sampling</u>		1 $\frac{1}{2}$
<u>Product Evaporation</u>		5 $\frac{1}{2}$
<u>Loading</u>		2

This document consists of 13 pages and 8 figures. No. 3 of 6 copies. Series A.

1. W. A. Rodger
2. M. C. Leverett
3. M. D. Peterson
4. A. C. Vallado
5. Central File
6. Reading File

W. A. Rodger

2-28-46

A. C. Vallado

706-D Production Run #8 (Shipment #16) ~~Classification Cancelled~~

By Authority Of DC
By Sydney Peterson Date AUG 23 1974

Mechanical Changes

During the eleven day period between runs 7 and 8, no decontamination of cell equipment was attempted. It was believed that the temporary "tie ring" which was installed, before run #7, on the spacer ring at the bottom of A-9 tank would hold out for another run.

Dissolver Operations

The 1728 slugs charged to the dissolver for Run #8 were added as follows:

	<u>Date Pushed and Charged</u>	<u>Slugs Loaded</u>
CLASSIFICATION CANCELLED	12-18	250
<u>J. B. Morgan 1/3/95</u>	1-2	300
ADD signature Date	1-3	286
Single rereview of CCRP-declassified	1-5	286
documents was authorized by DOE Office of	1-7	286
Declassification memo of August 22, 1994.	1-8	320
		<u>1728</u>

The slugs loaded on 12/18 remained in Cell A between runs. While the contribution of active Barium from these was small, the inactive did not diminish and for the purpose of calculation all 1728 slugs are considered. They are calculated to have contained an average of 7.1 Curies per slug active and 1.0 mg per slug inactive Barium at time of discharge (Total 12,204 Curies and 1676 mg).

The run was divided into two series. The first consisting of twelve dissolvings and extractions (first four being the run #7 stand-by,) followed by metathesis, cake solution, electrolysis and volume reduction in B6. The second series consisted of five dissolvings and extractions. The amounts of reagents used for the coating removals (136# 60% HNO₃, 306# 35% NaOH) and for the metal solutions (510# 60% HNO₃, 220# water) were normal with the exception of batch 7J, where 200# 60% HNO₃ and 86# of water were used due to an excessive amount of UNH solution left over from the previous batch.

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~~CONFIDENTIAL~~



A total of seventeen (average 79.2 slugs dissolved) metal solutions and six coating removal reactions were carried out. Considerable difficulty was encountered in maintaining an operating vacuum in the dissolver. At first it was thought to be due to gases condensing in the off-gas system because of the cold weather, but it was later discovered that there was a leak in the line underneath the building. The off-gas system was drained many times to continue operations.

Extraction

Four extractions were made on the "stand-by" series from run #7; eight on the first series of run #8; and five on the second series. The amounts of reagents were normal for the 85 slug batches (280# of 90% H_2SO_4 , 1000 cc 20% $Pb(NO_3)_2$ for A batches, 300 cc 20% $Pb(NO_3)_2$ for all others), except when wastes were added to extractor A9 prior to the extraction step. The seventeen decantations were cut at 3.5" to 5.8" on the oil liquid level manometer using jet A9-ASDB. Losses averaged 24 Curies per extraction or 8.8% of the product from each individual dissolving. See Table II for complete extraction decantation results.

On batch 7I, the second extraction of the run #7 stand-by, an emergency cake wash was made in case additional product was required to butt up run #7 in Cell B. However, as previously described in the report for run #7, this was not needed.

The extraction cakes from each of the two series were given the standard one acid, four water washes, all of 4 gallon volume. The acid shots for the second series were added to A9 through A11, in order to recover product probably lost in that vessel during transfers. Each wash was agitated 5 minutes, settled 70 min. ($3\frac{1}{2}$ hours for the acid shots in the second series due to large volume) and decanted to 3.2" to 3.6" heel on the oil liquid level manometer using jet A9-ASDB. The waste loss in the combined washes was 3 Curies. (See Table I).

Metathesis

Both metatheses were normal and uniform reagent quantities (similar to run #7) for 85 slug batches were used. Both were decanted to AS using jet A9-ASDB and cutting at 2.1" to 2.6" on the oil liquid level manometer. The combined waste loss of the metathesis and metathesis cake wash was 65 Curies. See Table III for metathesis conditions and losses.

Both metathesis cake solutions were transferred to the electrolysis vessel in Cell B in two batches followed by two washes via B26 and B27. No difficulty was encountered during these transfers and the system worked satisfactorily. Final volumes in the electrolysis vessel were 4250 and 3825 cc respectively.

Cell B Operation

The electrolysis time for the first series was increased (4 hours at 15 amps. and 11 hours at 25 amps.) to remove the large amounts of Pb present. For the second series the time was standard. No Barium carrier was added.

After the second electrolysis, the product was added to that from the first series in B6. Due to the large amounts of Barium present the solution in B6 was diluted to 6.2 liters before sampling. It was then evaporated to dryness, dissolved in 2 liters of water and re-concentrated to 350 cc. The solution was then transferred to B21R (by way of B11) without difficulty, where a fuming nitric precipitation was made. As in the previous run, the fuming nitric filtrate was sucked over to B20T from B21T and then blown to B3, as it was believed that transferring directly from B21T might blow the precipitate off the disc. A barium chloride precipitation and subsequent washings were made in B21R and the HCl-ether waste and washes were combined and sucked over to B20T from B21T for storage in the event of high losses. The combined waste losses were very high.

The product was dissolved in 170 cc of water and transferred to B17, where it was discovered that the Pb was well above tolerance (1500 mg). Hence, it was necessary to transfer the product solution to B6 and run through a second purification in the glassware. On the first run through glassware, about half of the Laccame out in the HCl-ether waste rather than all in the fuming nitric waste. For some reason (probably the high gamma radiation present) the first HCl-ether waste began polymerizing immediately. This caused considerable difficulty with plugging of transfer lines and delayed the analyses of the HCl-ether waste losses which were very high (124 Curies for the first pass and 412 for the second) until after the run was shipped.

After the second pass through glassware the Pb analyzed 90 mg in B17, and the product was transferred to B19 in one shot. Some difficulty was encountered in raising the air temperatures to operating level. It appeared that the glass wool inserted in the line to filter the incoming air was causing the trouble, so with its removal, the run proceeded without difficulty. The actual heating time was $4\frac{1}{2}$ hours.

Final analysis of product was:

<u>Active Barium</u>	2570 Curies at 1830 1/12/46
Cr	4 mg.
Fe	4 mg.
Ni	7 mg.
Sr	11 mg.
Pb	90 mg.
Ba	1200 mg.

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The $BaCl_2$ appeared lighter than any that had been shipped to date. There was a thin film up to the 2" level; the bulk of the product was in the cone.

Analyses

In Table I complete analytical data are presented. Table IA gives a summation of Barium losses for the run. Table IB gives direct radiation measurements which were made by moving the uncovered cone under the chimney and taking readings at the top of the cell.

Contamination and Radiation Exposure

No overexposures occurred during run #8. Several high hand counts and numerous high shoe counts were experienced. Most of these were readily reduced within a few minutes. The off-gas system got quite hot and caused areas in the North and East of the building to run close to tolerance. A leak developed in the drain pan on A-1 blister. As this could not be repaired until the run was completed, the affected area was isolated.

It is believed that the occasional high air counts have been traced to the venting of jet A9-ASDB. A revised venting process, whereby a slight vacuum is allowed to form before venting, was used and seemed to have materially reduced the incidence of high counts.

Two very hot 6P samples were taken, both reading about 10-12 r/hour at one foot. Ten capillary type samples reading about 2 r/hour were also taken. On the second purification a single bucket type sample was taken of 17P. The volume of this sample was about 0.1 cc. It read ≥ 100 r/hour but essentially no γ as it was less than $1\frac{1}{2}$ hours old. No general overexposures were recorded on any of these operations. Two men, E. J. Witkowski (6P) and V. Hendrickson (17P - cap.) received 190 mr on special films worn on the fingers. All other film readings were below tolerance.

TABLE I

(All Curies are Calculated to 1200 12/21/45)

Fraction	Code	Curies Product	Curies in Waste	% Total Product Dissolver
<u>Starting Heel</u> <u>From Run #7</u>				
First Dissolving Extraction Waste	1MI 8WMI	568	70.2	4.6
Second Dissolving Extraction Waste	1MI 8WMI	423	6.8	0.4
Third Dissolving Extraction Waste	1MJ 8WMI	324	108	7.1
Fourth Dissolving Extraction Waste	1MK 8WMI	200	0	0
Total Dissolved Total Extraction Waste		1515	185	12.2

(All Curies are Calculated to 2400 1-13-46)

<u>Starting Heel</u> <u>From Run #7</u>				
Total Dissolved Total Extraction Waste		421	51.4	1.1
First Dissolving Extraction Waste	1MA 8WMA	355	6	0.1
Second Dissolving Extraction Waste	1MB 8WMB	325	48	1.1
Third Dissolving Extraction Waste	1MC 8WMC	275	18	0.4
Fourth Dissolving Extraction Waste	1MD 8WMD	433	7	0.2
Fifth Dissolving Extraction Waste	1ME 8WME	283	5	0.1

~~SECRET~~

TABLE I
(Continued)

(All Curies are Calculated to 2400 1-13-46)

Fraction	Code	Curies Product	Curies in Waste	% Total Product Dissolver
Sixth Dissolving Extraction Waste	1MF 8WMF	284	17	0.4
Seventh Dissolving Extraction Waste	1MG 8WMG	352	6	0.1
Eighth Dissolving Extraction Waste	1MH 8WHM	291	12	0.3
Ninth Dissolving Extraction Waste	1MI 8WMI	216	33	0.7
Tenth Dissolving Extraction Waste	1MJ 8WJM	334	67	1.5
Eleventh Dissolving Extraction Waste	1MK 8WKM	342	20	0.4
Twelfth Dissolving Extraction Waste	1ML 8WML	319	33	0.7
Thirteenth Dissolving Extraction Waste	1MM 8WMM	347	78	1.7
Total Dissolved Total Extraction Waste		4557	401	8.8
AS Rinse	8WA		391	8.6
Extraction Cake Wash	8WW		3	0.06
Metathesis Waste	8WC		51.2	Combined
Metathesis Cake Wash (Combined)	8WCW		65.2	1.4
Product in B6	6P	2780		
PbO ₂ Waste Solution	3WPb		28	0.6
First B6 Rinse	-		345	Recovered
Second B6 Rinse	-		50	1.1
First Fuming Nitric Waste	3WEN		20.7	0.4
Second Fuming Nitric Waste	3WEN		47.4	1.0

TABLE I
(Continued)(All Curies are Calculated to 2400 1-13-46)

Fraction	Code	Curies Product	Curies in Waste	% Total Product Dissolver
First HCl-ether Waste	-		124	2.7
Second HCl-ether Waste	-		412	9.1
First Final Product	17P	3140		
Second Final Product	17P	2390		
B21 Rinses			333	7.5

TABLE IA

Summation of Barium Losses - Run #8

Fraction	Curies	%
Total Dissolved (Calculated as 1728 Slugs)	12204	100
Decay Loss (28, 13, 12, 10, 8, 7, Days)	5735	47.0
Known Loss in Cell A	860	7.1
Known Loss in Cell B	1013	8.3
Product Yield	2390	19.6
Material Balance		82.0
Total Curies Found in Dissolver	4557	100
Solutions Calculated to 2400 1-13-46		
Cell A Waste Losses	860	18.8
Cell B Waste Losses	1013	22.2
Product Yield	2390	52.4
Material Balance	4263	93.4

TABLE IB

Direct Radiation Measurements for Run #8

Guries Analyzed	2570	
Instrument	Time in Hours	R/Hour
25 R Meter	16	21.1
	26.5	-
G. E. Chamber	16	24.85
	18	26.6
	23.5	31
	26	31.75
100 R Meter	16	19
	24	24
250 R Meter	24	30

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TABLE II

Extraction Decantation

Batch	Rate of Decantation Gal/min.	Heel cc	Ba Loss	
			Curies	% of Product Present at Decantation
7H	2.7	4000	70.2	12.2
7I	3.1	3950	6.8	0.7
7J	3.5	3760	108	8.2
7K	2.7	4240	0	0
8A	2.8	3760	6	0.8
8B	2.4	6130	48	4.3
8C	2.9	4150	18	1.3
8D	2.7	4400	7	0.4
8E	3.0	3480	5	0.2
8F	2.3	3950	17	0.7
8G	2.7	3550	6	0.2
8H	2.8	3930	12	0.4
8I	2.4	4130	33	1.0
8J	2.9	4180	67	1.9
8K	2.6	4090	20	0.5
8L	2.8	3480	33	0.8
8M	3.3	4230	78	1.7

TABLE III

Metathesis

Treatment	Settling Time (Hours)	Heel cc	Ba Loss	
			Curies	% of Product Present at Decantation
<u>8H</u>				
First Metathesis	2½	3300)	--- 62.2	Recovered
Second Metathesis	2½	3760)		
First Metathesis Cake Wash	2½	4900)	--- 14.4	
Second Metathesis Cake Wash	2½	1200)		
<u>8M</u>				
First Metathesis	2½	4000)	--- 51.2	Combined
Second Metathesis	2½	3650)		
First Metathesis Cake Wash	2½	4750)	--- 65.2	
Second Metathesis Cake Wash	2½	1000)		

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TABLE IV
Time Cycles

<u>Operation</u>		<u>Hours Required</u>
<u>Charging</u>		
7H	250 Slugs	5
8A	300 "	5½
8C	286 "	5½
8G	286 "	6
8J	286 "	4½
8M	320 "	6
<u>Coating Removal</u>		
First		4
Second		2
Third		3
Fourth		3½
Fifth		3½
Sixth		3½
<u>Metal Solution and Extraction</u>		
	<u>Solution</u>	<u>Extraction</u>
7H	5	10½
7I	7½	10½
7J	8½	10½
7K	10	10½
8A	5	10
8B	4½	11½
8C	2½	10½
8D	3½	10
8E	3½	10
8F	5	10½
8G	3½	10½
8H	3½	10½
8I	5	10½
8J	5	16
8K	3½	10½
8L	3½	11½
8M	3	13
<u>Extraction Cake Washes</u>		
7I*		9½
8H		7
8M		10½
<u>Metathesis</u>		
8H		9
8M		8½

TABLE IV
(Continued)

Time Cycles

<u>Operation</u>	<u>Hours Required</u>
<u>Metathesis Cake Washes</u>	
8H	5½
8M	5¾
<u>Metathesis Cake Solution</u>	
8H	1¼
8M	3½
<u>Electrolyses</u>	
8H	15
8M	10
<u>PbO₂ Plate Removal</u>	
8H	1½
8M	No Data
<u>Volume Reduction in B6</u>	
8H	1
8M	
First	4¼
Second	2½
<u>Fuming Nitric Precipitation in B21</u>	
First	3
Second	1¼
<u>BaCl₂ Precipitation and Washing Product Solution</u>	
First	2¾
Second	¾
<u>Sampling</u>	
First Run Through Glassware	2
Second Run Through Glassware	¾
<u>Final Product Evaporation</u>	
	7*
<u>Loading</u>	
	3½

* Actual heating time was 4¾ hours.

CENTRAL FILES NUMBER

46-3-187

5/2

XT

File 3-5

B-137

Date March 12, 1946

Those Eligible
To Read The
Attached

Subject Shipment #17 (RUN 9)

By J. C. Kaskie

Copy # 2 - Peterson

To M. C. Leverett

Before reading this document, sign and date below:

Name _____ Date _____

Name _____ Date _____

W. B. Biteram 3/24/46

M. T. Kelly 3/14/46

~~Classification Cancelled~~

By Authority Of PJC

By Signon Peterson

Date

AUG 24 1971

CLASSIFICATION CANCELLED

ADD signature

1/19/95
Date

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



Publicly Releasable

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5-12-48

CLASSIFICATION CANCELLED

M. G. Leverett
 T. C. Kaskie
 Approved _____ Date _____
 This document is classified "Secret" as authorized by DOE Office of Security Information memo of August 22, 1994.

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SHIPMENT #17

Ball had a livery than usual appearance with a thin film on the wall up to 2nd level. The bulk of the product was in the lip of the cone. It was shipped in a vanadium lined cone #18 and carrier #2 with a "torpedo" packet.

Sample 2000 grams analyzed at 0400
 March 11, 1948 last separation
 1144

NAME OF
 ROOM NO.
 CONC. NO.
 CONC. NO.
 CONC. NO.
 CONC. NO.
 CONC. NO.

Location: 2nd floor level of the chimney

GR. CHAMBER

Time From 1ST	g/hr
1st	18
2nd	17
3rd	11
4th	22

100% Victoreen Material

Time From 1ST	g/hr
1st	19
2nd	11

J. C. Kaskie

SECRET

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1. W. A. Rodger
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Classification Cancelled

4-10-46

W. A. Rodger

A. C. Vallado

E/Action [redacted]
By [Signature] Date 4-10-46

706-D Production Run #9
(Shipment #17)

Mechanical Changes

Immediately upon the completion of Run #8, the decontamination of Cell A was begun, and maintenance work started when the tolerance levels reached 50 to 100 mr/hr. Sampling blisters, Cell IV and the floor around the cubicles were decontaminated also. For a complete account of maintenance work done in the building between Runs #8 and #9, see the report written by E. J. Witkowski on 706-D Building and equipment repairs, alterations and checking during period 1-13-46 to 3-3-46.

Dissolver Operations

The 894 slugs charged to the dissolver for Run #9 were added as follows:

<u>Date Pushed and Charged</u>	<u>Slugs Loaded</u>	CLASSIFICATION CANCELLED
3-3	317	<u>Jed Davis 11/24/94</u> Date
3-5	307	ADD signature
3-6	270	Single rereview of CCRP-declassified documents was authorized by DOE Office of
	894	Declassification memo of August 22, 1994

The slugs loaded on 3-3, 3-5 and 63 of the ones loaded on 3-6 were used for Run #9. The remainder of the slugs loaded on 3-6 were used for the Health Physics run which followed Run #9. For the purpose of calculation all 894 slugs are considered. These were calculated to have an average of 7.8 Curies/slug active, and 0.63 mg/slug inactive Ba (Total: 6950 Curies and 565 mg) at time of discharge.

The run was made in a single series, consisting of eleven dissolvings and extractions followed by metathesis, cake solution, electrolysis and volume reduction in B6. The Health Physics run (referred to as second series) which followed run #9 consisted of four dissolvings and extractions followed by metathesis, cake solution, electrolysis and volume reduction in B1. This second series was primarily intended to act as a stand-by in case some unusual losses occurred during the main run. As this was not the case, it was delivered to Health Physics.

4/10/46

The amounts of reagents used for the coating removals were 136# 60% HNO₃, 682# of 20% NaOH. The 20% NaOH was used instead of the usual 35%, because the solution was stored in the outside tank M-11, where it could easily freeze. For the metal solutions, the reagents were decreased about 23% of previous runs (390# of 60% HNO₃ and 170# H₂O) in order to dissolve 65 slugs/batch instead of 85, thereby preventing extractor tank A9 from overflowing.

A total of fifteen (average 61.2 slugs dissolved) metal solutions and three coating removal reactions were carried out. Some difficulty was encountered in maintaining an operating vacuum in the dissolver during the coating removal reactions. It was discovered that the off-gas system to A-1 was sealed off by a combination of the higher solution level in the dissolver and the violent reaction. This will be rectified in the next run, by increasing the concentration of NaOH solution. All metal solutions were carried out without difficulty.

Extraction

The amounts of reagents for all fifteen extractions were decreased approximately 23% of previous flowsheet quantities for the smaller batch size (210# of 90% H₂SO₄, 670 cc of 20% Pb(NO₃)₂ for A batches and 120 cc of 20% Pb(NO₃)₂ for all others. The fifteen decantations were cut at 2.4" to 15.5" heels on the oil liquid level manometer using jet A9-ASDA with the exception of those prior to metathesis on which jet A9-ASDB was used. Larger heels were left in extractor A9, because the A9-ASDA suction line was set six inches off the bottom. Although extractor A9 did not overflow throughout the run, and larger heels were left behind, the extraction losses were more than during previous runs. The losses averaged 35 Curies per extraction or 11.2% of the product from each individual dissolving. A possible explanation for these increased losses could be the partial plugging of A9-ASDA jet as shown by the side range of decanting time (16 to 65 minutes). See Table II for complete extraction decantation results.

The extraction cakes from each of the two series given the standard one acid, four water washes, all of 4 gallons volume. Each wash was agitated 5 minutes, settled 70 minutes and decanted to 2.1" to 3.6" heel on the oil liquid level manometer using jet A9-ASDB. The waste loss for the combined washes was 5.5 Curies. (See Table I)

Metathesis

Both metathesis were normal and uniform reagent quantities were used. They were decanted to A8 using jet A9-ASDB and cutting at 2.2" to 4.5" heels on the oil liquid level manometer. The combined waste loss of the metathesis and metathesis cake wash of the first series was rather high (296 Curies). This loss was added to the first extraction batch of the second series. See Table III for metathesis conditions and losses.

Some difficulty was experienced in transferring the cake solution of the first series to the crud filter due to the plugging of the A9 to B26 jet. The solution was therefore transferred to B12, then sucked up by B7, discharged to B26, filtered through the crud filter to B27 and transferred to B12 in three shots. The cake solution of the second series was transferred directly to B12 without going through the crud filter, as the disc was partially plugged by material removed from the first series. Final volumes in the electrolysis vessel were 4000 and 4550 cc respectively.

Cell B Operation

The electrolysis time for the first series was increased one hour (3 hours at 15 amps. and 7 hours at 25 amps.) to insure a low Pb value. The second series was standard. No Barium carrier was added in either series.

Operation of the glassware was reasonably smooth. After electrolysis the product of the first series was transferred to precipitator B6, evaporated to dryness, dissolved in 2 liters of water and reconcentrated to a 300 cc volume. The solution was transferred to B21R by way of B11 without difficulty, where two fuming nitric precipitations were made followed by two barium chloride precipitations and subsequent washings. The combined losses were quite high (373 Curies or 10.5% of total product dissolved.) The disc in the reactor was more porous than any previous one. This allowed filtering rates 3-4 times faster than those previously experienced, but for some reason (probably due to fact that the drip tip was broken off) liquid held up in the disc to a great extent. Consequently when pressure was put under a dry disc, liquid would appear on top of the disc. This made it impossible to clean the transfer vessel as well as is desirable and probably accounted for the high iron contents in the product.

The product solution was transferred from B17 to B19 via the upper discharge funnel. B21 transfer vessel was rinsed to B17 with 40 cc of water and then transferred to B19 over the same route. The final evaporation took 4 hours and proceeded without difficulty.

Final analysis of run #9 product was:

<u>Active Barium</u>	2040 Curies at 0400 - 3-11-46 (LST)
Pb	23 mg
Fe	38 mg
Cr	4 mg
Ni	8 mg
Sr	1 mg
Ba	800 mg

The product was somewhat darker than usual, but had the usual distribution in the cone.

~~This document contains information of a confidential nature. Its disclosure to unauthorized persons is prohibited by law.~~

After electrolysis of the second series, the product was transferred to B1 with pipette B8, evaporated to dryness, dissolved in 2000 cc of water and reconcentrated to 350 cc. The bulk of the product was transferred to B19 via movable funnel B2 and evaporated to dryness. B1 was rinsed with 200 cc of water and transferred to B19 via the same route. The combined final evaporation time was 10½ hours.

The ECl-ether waste solution was evaporated in a Pt-lined cone and delivered to 706-C. This will hereafter be delivered as a liquid or not at all. The procedure was time consuming, difficult, and dangerous to equipment.

The overall Run #9 was the most successful to date; a full 2000 Curies were produced with a 57% yield; counting the Health Physics run essentially all the product was accounted for and from beginning to end it was never over 3 hours off schedule.

Analyses

In Table I complete analytical data are presented. Table IA gives a summation of Barium losses for the run. Table IB gives direct radiation measurements which were taken on the open cone from the top of the chimney. The G.E. chamber indicated 2000 Curies were present for shipping.

Contamination and Radiation Exposure

The building and off-gas systems remained cool throughout the run and there were no over-exposures. Only one incident (described in 206 Area Report for week ending March 9) marred the entire run.

During the early part of the run some increase in air counts occurred on the third level when jetting solution from A9-AB; this despite very careful venting procedures. The counts were generally below tolerance, however.

The first two days of the run, Iodine was collected from the trap between A3 and A4. As the metal which was being dissolved was quite young a large amount of material was available. The system was not properly trapped, however, and hot gases got into the vacuum system causing an increase in air counts. The experiment was discontinued until a more adequate set-up could be devised. Air counts returned to normal at once.

No working area in the building was over tolerance except that between B3 and B6 blisters. This area was just over. The cell off-gas system remained below 200 mr/hour throughout the run. Most working areas were below 25 mr/day.

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TABLE I

(All Curies are Calculated to 0400 - 3/11/46)

Fraction		Code	Curies Product	Curies in Waste	% Total Product Dissolved
<u>First Series</u>					
First First	Dissolving Extraction	1MA 8WMA	298	18	0.5
Second Second	Dissolving Extraction	1MB 8WMB	353	9	0.2
Third Third	Dissolving Extraction	1MC 8WMC	360	6	0.2
Fourth Fourth	Dissolving Extraction	1MD 8WMD	345	41	1.2
Fifth Fifth	Dissolving Extraction	1ME 8WME	341	14	0.4
Sixth Sixth	Dissolving Extraction	1MF 8WMF	310	27	0.8
Seventh Seventh	Dissolving Extraction	1MG 8WMG	280	62	1.7
Eighth Eighth	Dissolving Extraction	1MH 8WHM	288	65	1.8
Ninth Ninth	Dissolving Extraction	1MI 8WMI	350	63	1.8
Tenth Tenth	Dissolving Extraction	1MJ 8WMI	305	39	1.1
Eleventh Eleventh	Dissolving Extraction	1MK 8WMK	316	31	0.9
Total Dissolved			3556		100
Total Extraction Waste				375	10.6
<u>Second Series</u>					
First First	Dissolving Extraction	1ML 8WML	303	57	5.2

TABLE I
(Continued)

(All Curies are Calculated to 0400 - 3/11/46)

Fraction	Code	Curies Product	Curies in Waste	% Total Product Dissolved
Second Dissolving Second Extraction	1M2 SWM2	276	37	3.4
Third Dissolving Third Extraction	1M3 SWM3	277	23	2.1
Fourth Dissolving Fourth Extraction	1M0 SWM0	241	30	2.7
Total Dissolved Total Extraction Waste		1097	147	100 13.4
<u>First Series</u>				
Extraction Cake Wash	SWW		5	0.1
Metathesis and Metathesis Cake Wash	SWC) SWCW)		296	8.3
Total Cell A Loss			676	19.0
Electrolysis Loss	SWPd		32	0.9
Product in B5	6P	2380		
B-6 Rinse			52	1.4
Fuming Nitric Wastes	SWFN		21	0.6
HCl-ether Wastes			352	9.9
Total Cell B Loss			457	12.8
Final Product	17P	2040		
<u>Second Series</u>				
Extraction Cake Wash	SWW		0.5	0.04
Metathesis and Metathesis Cake Wash	SWC) SWCW)		191	17.4

TABLE I
(Continued)(All Curies are Calculated to 0400 - 3/11/46)

Fraction	Code	Curies Product	Curies in Waste	% Total Product Dissolved
Total Cell A Loss			338	30.8
Electrolysis Loss			29	2.6
Product in B1	1P	1275		

TABLE IA

Summation of Barium Losses - Run No. 9
(Based on LST 0400 - 5/11/46)

Fraction	Curies	%
Total Dissolved (Calculated as 687 Slugs)	5455	100
Decay Loss (8, 6, 5, Days)	1723	31.7
Known Loss in Cell A	676	12.4
Known Loss in Cell B	457	8.4
Product Yield	2040	37.4
Material Balance		89.9
Total Curies Found in Dissolver Solution Calculated to 0400 - 3/11/46	3556	100
Cell A Waste Losses	676	19.1
Cell B Waste Losses	457	12.8
Product Yield	2040	57.5
Material Balance	5173	89.4

TABLE IA

Summation of Barium Losses - Run No. 9
Including Product to Health Physics
(Based on LST - 0400 - 3/11/46)

Fraction	Curies	%
Total Dissolved (Calculated as 894 Slugs)	6950	100
Decay Loss (8, 6, 5, Days)	2083	30.0
Known Loss in Cell A	1014	14.6
Known Loss in Cell B	486	7.0
Product Yield	3315	47.7
Material Balance		99.3
Total Curies Found in Dissolver Solutions Calculated to 0400 - 3/11/46	4653	100
Cell A Waste Losses	1014	21.8
Cell B Waste Losses	486	10.5
Product Yield	3315	71.2
Material Balance	4815	103.5

TABLE IB

Direct Radiation Measurements For Run No. 9

Curies Analyzed	2000	
Instrument	Time in Hours	r/Hour
G.E. Chamber	16	16
	18	17 $\frac{1}{2}$
	26	21 $\frac{1}{2}$
	28	22 $\frac{1}{2}$
100 R Meter (1)	16	19
	26	21
100 R Meter (2)	26	20

TABLE II
Extraction Decantations

Batch	Rate of Decantation Gal/min	Heel Gals.	Ba Loss	
			Curies	% of Product Present at Decantation
<u>First Series</u>				
9A	2.0	4.75	18	0.6
9B	3.3	3.2	9	1.3
9C	1.3	3.2	6	0.6
9D	4.3	2.56	41	3.1
9E	4.5	3.0	14	0.8
9F	3.7	2.9	27	1.3
9G	3.4	3.2	62	2.7
9H	2.2	2.8	65	2.5
9I	4.3	3.84	63	2.1
9J	5.4	3.0	39	1.2
9K	2.5	0.72	31	0.9
<u>Second Series</u>				
9L	5.3	2.95	57	18.8
9M	3.6	3.57	37	6.4
9N	2.8	1.76	23	2.7
9O	2.9	0.56	30	2.7

TABLE III

Metathesis

Treatment	Settling Time (Hours)	Heal cc	Curies	% of Product Present at Decantation
<u>First Series</u>				
First Metathesis	$2\frac{1}{2}$	3480)	---- 94.5	2.7
Second Metathesis	$2\frac{1}{2}$	3480)		
First Metathesis Cake Wash	$2\frac{1}{2}$	4240)	---- 201.5	5.7
Second Metathesis Cake Wash	$2\frac{1}{2}$	1100)		
<u>Second Series</u>				
First Metathesis	$2\frac{1}{2}$	6800)	---- 133	12.1
Second Metathesis	$2\frac{1}{2}$	3650)		
First Metathesis Cake Wash	$2\frac{1}{2}$	4320)	---- 58	5.3
Second Metathesis Cake Wash	$2\frac{1}{2}$	2000)		

TABLE IV

Time Cycles

<u>Operation</u>	<u>Hours Required</u>	
<u>Charging</u>		
9A 317 Slugs	3 $\frac{1}{4}$	
9D 307 "	4 $\frac{1}{4}$	
9I 270 "	3 $\frac{1}{4}$	
<u>Coating Removal</u>		
First	3 $\frac{1}{4}$	
Second	2 $\frac{1}{4}$	
Third	2 $\frac{1}{4}$	
<u>Metal Solution and Extraction</u>		
<u>(First Series)</u>		
9A	2 $\frac{1}{4}$	11 $\frac{1}{4}$
9B	3 $\frac{1}{4}$	12 $\frac{1}{4}$
9C	2 $\frac{1}{4}$	10 $\frac{1}{4}$
9D	2 $\frac{1}{4}$	10 $\frac{1}{4}$
9E	3	10
9F	3 $\frac{1}{4}$	10
9G	3 $\frac{1}{4}$	10
9H	4	10
9I	3	10
9J	2 $\frac{1}{4}$	10 $\frac{1}{4}$
9K	2 $\frac{1}{4}$	10
<u>(Second Series)</u>		
9L	3 $\frac{1}{4}$	10
9M	4	10
9N	5	10 $\frac{1}{4}$
9O	7	11
<u>Extraction Cake Wash</u>		
First Series	7	
Second Series	7	
<u>Metathesis</u>		
First Series	9	
Second Series	8 $\frac{1}{4}$	

~~SECRET~~

W. A. Rodger

TABLE IV
(Continued)

4/10/46

Time Cycles

<u>Operation</u>	<u>Hours Required</u>
<u>Metathesis Cake Wash</u>	
First Series	5½
Second Series	5½
<u>Metathesis Cake Solution</u>	
First Series	2½*
Second Series	3½**
<u>Electrolysis</u>	
First Series	10
Second Series	9
<u>PbO₂ Plats Removal</u>	
First Series	4
Second Series	No Data
<u>Volume Reduction in B6</u>	
First Series	3½
<u>Volume Reduction in B1</u>	
Second Series	5½
<u>Tuning Nitric Precipitation in B21</u>	
First Series	2½
<u>BaCl₂ Precipitation, Washing, Product Solution</u>	
First Series	1½
<u>Sampling</u>	
First Series	½
Second Series	½
<u>Final Product Evaporation</u>	
First Series	4
Second Series	10½***

* Lines were plugged.

** By-passed crud filter.

*** Two shots.

TABLE IV
(Continued)

Time Cycles

<u>Operation</u>	<u>Hours Required</u>
<u>Direct Radiation Measurements</u>	
First Series	10
Second Series	10
<u>Loading</u>	
First Series	4
Second Series	6

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CLINTON LABORATORIES
CENTRAL FILES NUMBER
46-4-370

B-137

File JE XT

Date April 17, 1946

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Subject Shipment #18

By S. A. Reynolds

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4-17-44

M. G. Leverett

S. A. Reynolds

SHIPMENT #18

The barium chloride product had a tan color, lighter than most of the previous shipments. The bulk of the material was in the bottom of the can, but a thin and flakey crust of the white product covered the top of the can (see drawing #18) in a carrier with a heavy...

Net Weight at last separation (lb)
1.0000 (100.00%)

Classification Canceled

By Authority of

AUG 23 1971

TRANSITION RECORD

x/AT

19.0

24.5

The following remaining indicators 1000 Curium (within 2 1/2%) (The remaining 10 Curium - 1000 Curium were markings due to uncorrected activity of Curium - 1000 Curium)

S. A. Reynolds

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CLINTON LABS
CENTRAL FILE
46-5330

File J-E

B-137

Date May 17, 1946

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May 17, 1946

M. G. Laverett
W. A. Reynolds

- 1. M. G. Laverett
- 2. M. D. Petermann
- 3. W. A. Reynolds
- 4. J. C. Stangor
- 5. S. A. Reynolds
- 6. Central File

SHIPMENT #19

The barium chloride product was quite small in bulk, and of a darker than usual color. The material was apparently almost entirely in the bottom of the cone, for no film was visible on the wall. It was shipped in cone #4 (collar #4) in a carrier with a Korussal gasket.

Is¹³⁷ 3000 Curies, analysed radiochemically at D-40, 5-16-45, last separation time.

Spectrographic Analysis

Ca	11 mg
Fe	18 mg
Cu	18 mg
Al	7 mg
Sr	5.8 mg
La	5.4 mg

Classification canceled

By Authority of the Director
Date: 1/10/50

** Calculated as 500 mg by Operations, from pile data.

Radiation Readings

Time from I.S.T. (hours)	r/hr
16	ca. 21
22	ca. 27

The radiation reading indicates 2000 Curies (within ± 15%). The readings are somewhat in doubt because of failure of the Micromax recorder. Tester (of the Instrument Group) obtained the above readings from the V.T. Chamber itself.

S. A. Reynolds

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CLINTON LABORATORIES
CENTRAL FILES NUMBER

46-5-363

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Date May 20, 1946

Subject 706-D Production Run #10

(Shipment #10)

By A. C. Vallado

To W. A. Rodger

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W. A. Rodger 5/20/46



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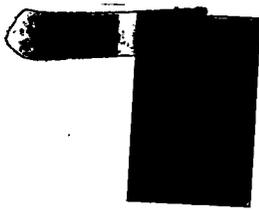
5-20-46

A. C. Vallado



[Handwritten signature]

706-D Production Run #10
(Shipment #18)



Mechanical Changes

As the glassware and other cell equipment was in fairly good condition, no cell decontamination or maintenance work was done between runs.

Dissolver Operations

The 886 slugs charged to the dissolver for Run #10 were added as follows:

<u>Date Pushed and Charged</u>	<u>Slugs Loaded</u>
4-7	365
4-8	324
4-11	197
	<u>886</u>

CLASSIFICATION CANCELLED.
Jed Davis 12/2/95
 ADD signature
 Single rereview of CCRP-declass documents was authorized by DOE Ofr
 Declassification memo of August 22, 1

The slugs loaded on 4-7 included 43 which were pushed on 3-22 and 3-24. For purpose of calculation 805 slugs are considered, since only 116 of the 4-11 loading were used for the run. These were calculated to have an average of 8.0 Curies/slug active, and 1.0 mg/slug inactive Barium. (Total: 6448 Curies and 800 mg) at time of discharge.

This run was very successful, made in a single series, consisting of twelve dissolvings and extractions followed by metathesis, cake solution, electrolysis and volume reduction in B6. The amounts of reagents used for the coating removals were uniform and the usual 35% NaOH was used with the return of warm weather. The amounts of reagents for the metal solution were uniform for the 65 slug batch.

A total of twelve (average 67.2 slugs dissolved) metal solutions and three coating removal reactions were carried out without difficulty.

Extraction

The quantities of reagents for all twelve extractions were uniform for 65 slug batches as in Run #9 (210# 90% H₂SO₄, 670 cc 20% Pb(NO₃)₂ for A batches and 120 cc of 20% Pb(NO₃)₂ for all others). All decantations were cut at 3.5" to 11.0" heels on the oil liquid level manometer using jet A9-ASDA with the exception of the last one on which jet A9-ASDB was used.

Losses averaged 36 Curies per extraction or 11.7% of the product from each individual dissolving. These losses are quite analogous to those found in the previous run, where it was believed that the higher losses were caused by a partial plugging of A9-ASDA jet as shown by the wide range of decanting time. However, for Run #10 this range was fairly uniform (17 to 25 minutes) and may be indicative of some faulty jet characteristic. See Table II for complete extraction decantation results.

The extraction cake was given the usual one acid, four water washes, all of four gallon volume. Each wash was agitated 5 minutes, settled 70 minutes and decanted to 2.2" to 3.2" heels on the oil liquid level manometer using jet A9-ASDB. The waste loss for the combined washes was 4.0 Curies. (See Table I).

Metathesis

The metatheses were normal and uniform reagent quantities were used. They were decanted to A9 using jet A9-ASDB and cutting at 2.2" to 2.5" heels on the oil liquid level manometer. The combined waste loss of the metathesis and metathesis cake wash was very high (655 Curies) and certainly the largest loss to date. It is thought that the usual 2½ hours settling may be insufficient, so this time will be increased to 4 hours for the next run. The 655 Curies were added to the extraction following the run and later discarded.

The bulk of the metathesis cake solution was transferred to the electrolysis vessel (B12) in Cell B in a single batch following by a water wash, via B26 and B27. No difficulty was encountered during this transfer and the system operated satisfactorily. The final volume in the electrolysis vessel was 4450 cc.

Cell B Operation

The electrolysis time was the same as the previous run (3 hours at 15 amps. and 7 hours at 25 amps.) to insure a low Pb value. No Barium carrier was added.

After electrolysis the product was transferred to precipitator B6, evaporated to dryness, dissolved in 2 liters of water and reconcentrated to 300 cc. The solution was transferred to B21R by way of B11 without difficulty, where 2 fuming nitric precipitations were made followed by 2 barium chloride precipitations and subsequent washings. The combined losses were normal (145 Curies or 3.9% of the total product dissolved).

The product solution was transferred from B17 to B19 via B8, the upper discharge funnel and moveable funnel. The final evaporation took 3½ hours and went without difficulty.

Final analysis of product was:

Active Barium 1800 Curies at 1200 - 4/15/46

Direct Radiation Reading - 1800 Curies \pm 15%

Pb	<13 mg	(not detectable)
Fe	3 mg	
Cr	2 mg	
Ni	13 mg	
Sr	3 mg	
Ba	600-1100 mg	(Spectrographic Analysis)

The product was tan colored, lighter and smaller in bulk than most previous shipments, and was distributed in the cone in the usual manner.

The HCl-ether waste was delivered to 706-C as a liquid in a special carrier designed for the purpose. This system is much more satisfactory than the evaporation previously used.

The run was the most successful to date. With the exception of some high air counts on the third level, no incident marred the run. The predicted schedules were maintained throughout.

Analyses

In Table I complete analytical data are presented. Table IA gives a summation of Barium losses for the run. Direct radiation measurements were made by moving the uncovered cone under the chimney and taking readings at the top of the cell.

Contamination and Radiation Exposure

No overexposures were reported.

During the entire run, the third level air counts were periodically high due to contaminated air blowing from the fan house stack with North and North East winds. These counts have been definitely traced to the 706-D stack. Increase in air contamination occur inside the building only during jettings, evaporations, and digestions which take place when there is no wind or wind directed from the stack toward the building. It is apparent that stack facilities are inadequate. Studies will soon be undertaken to determine remedial action.

TABLE I

(All Curies are Calculated to the Shipping Time - 1200 - 4/15/46)

Fraction	Code	Curies Product	Curies in Waste	% Total Product Dissolved
First Dissolving Extraction Waste	L1MA S1MA	255	15	0.4
Second Dissolving Extraction Waste	L1MB S1MB	254	28	0.7
Third Dissolving Extraction Waste	L1MC S1MC	284	10	0.3
Fourth Dissolving Extraction Waste	L1MD S1MD	374	10	0.3
Fifth Dissolving Extraction Waste	L1ME S1ME	395	71	1.9
Sixth Dissolving Extraction Waste	L1MF S1MF	299	27	0.7
Seventh Dissolving Extraction Waste	L1MG S1MG	290	17	0.5
Eighth Dissolving Extraction Waste	L1MH S1MH	255	16	0.4
Ninth Dissolving Extraction Waste	L1MI S1MI	262	22	0.6
Tenth Dissolving Extraction Waste	L1MJ S1MJ	334	35	1.0
Eleventh Dissolving Extraction Waste	L1MK S1MK	321	17	0.5
Twelfth Dissolving Extraction Waste	L1ML S1ML	342	161	4.1
Total Dissolved		3665		
Total Extraction Wastes			429	11.7
Cake Wash	S1WF		4	0.1

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TABLE I
(Continued)

Fraction	Code	Curies Product	Curies in Waste	% Total Product Dissolved
Metathesis Waste	8RC		459	12.5
Metathesis Wash	8RDF		196	5.4
Product in B-6	6P	2550		
Electrolysis Loss	3RFB		16	0.4
B-6 Rinse	6RF		35	1.0
Fuming-Nitric Wastes	3RIN		51	0.8
HCl-other Wastes	3W-HCl		114	3.1
Final Product in B-17	17F	1800		

TABLE IA

Summation of Ba Losses For Run #10
(Curies are Calculated to Shipping Time - 1200 - 4/15/46)

Fraction	Curies	%
Total Dissolved (Calculated as 805 Slugs)	6448	100
Decay Loss (26, 24, 10, 9, 6 Days)	2606	40.5
Known Losses in Cell A	1088	16.9
Known Losses in Cell B	196	3.1
Yield	1800	28.0
Material Balance		88.5
<hr/>		
Total Curies Found in Dissolved Solution Calculated to 1200 - 4/15/46	3665	100
Cell A Waste Loss	1088	29.7
Cell B Waste Loss	196	5.4
Product Yield	1800	49.1
Material Balance	3084	84.2

TABLE II

Extraction Decantation

Batch	Rate of Decantation Gal/min	Heel Gal.	Ba Loss	
			Curies	% of Product Present at Decantation
10 A	4.2	2.9	15	5.9
10 B	4.3	2.6	28	5.5
10 C	3.4	3.0	43	5.5
10 D	5.7	3.6	75	6.5
10 E	3.8	3.4	71	4.5
10 F	5.4	3.6	90	4.8
10 G	4.1	3.4	17	0.8
10 H	4.7	2.7	16	0.7
10 I	4.4	3.4	22	0.8
10 J	5.0	2.7	57	1.9
10 K	4.3	3.0	17	0.5
10 L	2.7	1.0	161	4.4

TABLE III

Metathesis

Treatment	Settling Time (Hours)	Reel cc	Ba Loss	
			Guries	% of Product Present at Decantation
First Metathesis	2½	3550))----- 459	12.5
Second Metathesis	2½	3480)		
First Metathesis Cake Wash	2½	4400))----- 196	5.5
Second Metathesis Cake Wash	2½	1300)		

TABLE IV
Time Cycles

<u>Operation</u>		<u>Hours Required</u>
<u>Charging</u>		
10 A	365 Slugs	5½
10 D	324 "	4½
10 J	197 "	2½
<u>Coating Removal</u>		
	First	2
	Second	1½
	Third	2
<u>Metal Solution and Extraction</u>	<u>Solution</u>	<u>Extraction</u>
10 A	A 3½	10
10 B	B 3½	10½
10 C	C 3	10
10 D	D 3½	10
10 E	E 4	10
10 F	F 3½	10
10 G	G 3½	10
10 H	H 3½	10
10 I	I 4½	10
10 J	J 5	10
10 K	K 4½	10
10 L	L 9	10
<u>Extraction Cake Wash</u>		7
<u>Metathesis</u>		6½
<u>Metathesis Cake Wash</u>		5½
<u>Cake Solution</u>		1
<u>Electrolysis</u>		10
<u>PbO₂ Removal</u>		3
<u>Volume Reduction in B6</u>		3½
<u>Fuming Nitric Precipitation in B21</u>		2½
<u>BaCl₂ Precipitation, Washing and Product Solution in B21</u>		2
<u>Sampling</u>		3
<u>Product Evaporation</u>		3½
<u>Loading</u>		2

KT

CLINTON LABORATORIES
CENTRAL FILES NUMBER
46-6-272

B-137

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Date June 19, 1946

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By S. A. Reynolds

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M. C. Leverett

S. A. Reynolds

- 1. M. C. Leverett
- 2. M. D. Peterson
- 3. W. A. Rodger
- 4. J. G. Stangby
- 5. S. A. Reynolds
- 6. Central File

Shipment #20

The barium chloride product was the lightest in color yet produced in 706-D. The amount of solid was larger than that in the last preparation (inactive Ba was over 50% greater), and a deposit was visible on the walls of the cone (#5) up to about two inches.

Ba* 2500 Curies, analysed radiochemically, corrected to 1100, 6-17-46, last separation time.

Spectrographic Analysis

Pb		57 mg.
Fe	less than	0.5 mg.
Cr	" "	0.5 mg.
Mn		17 mg.
Sr		0.5 mg.
Ba		850 mg.**

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 ADD signature _____ Date 11/2/95
 Single rereview of CCRP-declassified documents was authorized by DOE Office of Declassification memo of August 22, 1994.

**It has been calculated that about 200 mg. of inactive Ba is added in the water used in Cell A. Thus the Ba figure should be higher than that calculated from pile data.

Radiation Reading

The radiation reading ("skyshine") was 29.4r/hr. at 20 hours after L.S.T., indicating 2500 Curies.

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By JAB

Date AUG 24 1971

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10 g-u. p. 00352
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ADVISOR: GUYTON, JR.

1/6/65

TAB 17

Time Series

Operation _____ Court Required _____

Charges

1. 1st Degree Murder
2. 2nd Degree Murder
3. 3rd Degree Murder

Arrest

Arrested on _____ at _____

Investigation in B-1
Investigation in B-2

This document contains information
[Redacted]

This document consists of 11 pages and 0 figures. No. 5 of 6 copies, Series A

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W. A. Rodger

A. C. Vallado

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J. Morgan 1-10-95
ADD signature Date

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706-D Production Run #11
(Shipment #19)

Mechanical Changes

No cell decontamination or maintenance work was done between runs. A new crud filter disc was installed in Cell IV of the external cubicle, as the filtration rate was decreasing.

Dissolver Operations

The 896 slugs charged to the dissolver for Run #11 were added as follows:

Date Pushed and Charged

Slugs Loaded

5-5
5-6
5-9

321
324
261
896

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By *J. Morgan* Date

All 896 slugs charged to the dissolver have been considered for purposes of calculation. These slugs were calculated to have an average of 7.8 Curies per slug active and 0.58 mg per slug inactive barium (Total 7020 Curies and 516 mg) at time of discharge. The number of milligrams of inactive barium was lower than previous runs, because the slugs were not in the pile for such long periods.

It had been proposed to have a single series of twelve dissolvings and extractions followed by metathesis, cake solution, electrolysis and volume reduction in B-6 similar to Run #10. However, due to high losses in the extraction wastes it was necessary to make two more dissolvings and extractions. The amounts of reagents used for the coating removals and metal solutions were uniform and the latter consisted of 65 slug batches.

A total of fourteen (average 66.8 slugs dissolved) metal solutions and three coating removal reactions were carried out.

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7/30/46

After dilution of the metal solutions, attempts were made to boil out "hot" gases (which had been contaminating the air) before transferring to A9 via Al-A9 jet. However, after the freezing of extraction slurry of batch F, this boiling was discontinued.

Extraction

The quantities of reagents for all fourteen extractions were uniform for 65 slug batches as in Run #10 (210# 90% H_2SO_4 , 670 cc 20% $Pb(NO_3)_2$ for A batches and 120 cc of 20% $Pb(NO_3)_2$ for all others). All decantations were out at 2.9" to 11.3" heels on the oil liquid level manometer using jet A9-ASDA with the exception of batches L and M on which jet A9-ASDB was used. Losses averaged 53 Curies per extraction or 11.7% of the product from each individual dissolving. Several high waste losses were experienced during this run due to inefficient operation of both A9 decant jets. Most of the product, however, was recovered in subsequent resettling and decantations in A-11. The last extraction waste analyzed approximately 1000 Curies. These losses necessitated the use of the standby run which consisted of two batches of metal solution combined with rework waste. The procuring of the standby run delayed product shipment for two days. See Table II for complete extraction decantation results.

The run fell nine hours behind schedule due to the freezing of extraction slurry of batch F. It was apparently caused by a rise in the freezing point of solution after an attempt was made to boil out "hot" gases (which had been contaminating the air) from the dissolver solution prior to extraction.

The extraction cake of the first series was given the usual one acid, four water washes, all of four gallon volume. Each wash was agitated five minutes, settled 70 minutes and decanted to 2.2" to 3.6" heels on the oil liquid level manometer using jet A9-ASDB. The waste loss for the combined washes was 12 Curies. The extraction cake of the second series was given one acid and two water washes, then the batch M heel (8500 which was 394 Curies) was jetted from A-11 to A-9. Fifty pounds of water were added to A-11 and jetted to A-9, settled five hours and decanted to 2.1" to 3.2" heels on the oil liquid level manometer using jet A9-ASDB. The waste loss for the combined washes was 100 Curies (See Table I). The recovery cycle increased the usual cake washing time from 7 or 8 hours to 17.

Metathesis

In run #10 the combined waste loss of the metathesis and metathesis cake wash was very high and it was thought that the usual 2 1/2 hours settling time may have been insufficient. Therefore the settling time of the metathesis of the first rinse was increased to four hours. However, this loss was about 302 Curies, so it was thought that the increase in settling time did not have a direct effect on losses, and the usual 2 1/2 hours was

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7/30/46

applied to the metathesis of the second series. Uniform reagent quantities were used for both metatheses. They were decanted to A8 using jet A9-ABDB and cutting at 2.3" to 2.6" heels on the oil liquid level manometer. The combined waste loss of the metathesis and metathesis cake wash was 364 Curies, and for the second series it was 128 Curies. The 364 Curies were recovered and added to the extraction of batch A.

The metathesis cake solution of the first series was transferred to the electrolysis vessel (B12) in Cell B in two acid shots followed by a water rinse via B26 and B27. The metathesis cake solution of the second series was transferred to B12 via B26 and B27 in a single acid shot followed by an acid and water rinse. No difficulty was encountered during these transfers and the system worked satisfactorily. The final volumes in the electrolysis vessel were 4150 and 4120 cc respectively.

Cell B Operation

The time for both electrolyses was the same as the previous run (3 hours at 15 amps. and 7 hours at 25 amps.). No Barium carrier was added.

The product of the first electrolysis was transferred to precipitator B6, sampled and analyzed a mere 1323 Curies from 3455 Curies dissolved. A standby run of two batches and recovery of metathesis waste losses were used to bring the yield to the 2000 mark. The product of the first electrolysis in B6 was evaporated to dryness, dissolved in 2 liters of water and held for the standby run. The product of the standby run was combined with the product in B6, evaporated to dryness, dissolved in 2 liters of water and reconcentrated to 350 cc. The second 6P sample analyzed 2712 Curies from 3938 Curies dissolved and was more satisfactory. The product solution was transferred from B6 to B21R by way of B11 without difficulty, where two fuming nitric precipitations were made followed by two barium chloride precipitations and subsequent washings. The combined losses were normal (197 Curies or 5.0% of the total product dissolved).

The product solution was transferred from B17 to B19 via B8, the upper discharge funnel and moveable funnel. The final evaporation took 4 hours and went without difficulty.

Final analysis of the product was:

Active Barium 2000 Curies

Direct radiation reading 23.8 r/hr at 20 hours after LST indicated
2000 Curies (\pm 15%)

Pb	11 mg
Fe	18 mg
Cr	18 mg
Ni	7 mg
Zr	0.4 mg
Ba	540 mg (Spectrograph analysis)
	300 mg (Calculated)

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The product was comparatively small in bulk and of a color darker than that of several previous runs. The material appeared to be almost entirely in the bottom of the cone with practically no film on the wall.

The HCl-ether waste was delivered to 706-C in a liquid form.

Due to inefficient operation of decant jets, a portion of the product lost in the extraction wastes was in the form of a precipitate. This condition would make it difficult to obtain representative samples of the waste and may account for the low material balance of 74.4%.

Analyses

In Table I complete analytical data are presented. Table IA gives a summation of Barium losses for the run. Direct radiation measurements were made by moving the uncovered cone under the chimney and taking readings at the top of the cell.

Contamination and Radiation Exposure

No overexposures were reported.

The building remained free of contamination throughout the run. The air contamination remained negligible during the entire run due to favorable weather conditions. A series of air samples, taken from both off-gas lines, were analyzed in an attempt to determine the source of activity so that steps could be taken to prevent recurrences of building air contamination. The A-18 off-gas system was found to be the main source of activity, so a project was drawn up to tie the system to the 205 stack.))

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TABLE I
(Continued)

Fraction	Code	Curies Product	Curies in Waste	Total Product Dissolved
First Cake Wash	8WF ₁		12	0.5
Second Cake Wash	8WF ₂		100	2.5
First Metathesis Waste	8WC ₁		302	Recovered
Second Metathesis Waste	8WC ₂		35	0.9
First Metathesis Cake Wash	8WCF ₁		62	Recovered
Second Metathesis Cake Wash	8WCF ₂		93	2.4
Total Cell A Loss			702	17.8
Product in B-6	6P ₁ 6P ₂	1325 2712		
First Electrolysis Loss	5WPb ₁		No Sample	-
Second Electrolysis Loss	5WPb ₂		11	0.3
B-6 Rinse	6WF		17	0.4
Fuming Nitric Wastes	5WFN		66	1.7
HCl-other Wastes	5W-HCl		131	3.3
Total Cell B Loss			225	5.7
Final Product in B-17	17P	2000		

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TABLE IA

Summation of Ba Losses for Run #11
(Curies are Calculated to Shipping Time - 0740 - 5-16-46)

Fraction	Curies	%
Total Dissolved (Calculated as 896 Slugs)	7020	100.0
Decay Loss (12, 11, 10, 7 Days)	2876	40.9
Known Losses in Cell A	702	10.0
Known Losses in Cell B	225	3.2
Yield	2000	28.5
Material Balance		82.6
Total Curies Found in Dissolver Solution Calculated to 0740 - 5/16/46	3938	100.0
Cell A Waste Loss	702	17.9
Cell B Waste Loss	225	5.7
Product Yield	2000	50.8
Material Balance	2927	74.4

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TABLE II
Extraction Decantation

Batch	Rate of Decantation Gal/min	Resi Gal	Ba Loss	
			Curies	% of Product Present at Decantation
11 A	4.3	2.9	43	15.2
11 B	5.3	2.8	6	1.1
11 C	4.9	2.9	66	8.3
11 D	5.0	2.9	27	2.5
11 E	4.2	3.0	37	2.6
11 F	4.5	3.8	455	27.1
11 G	2.5	3.2	287	14.8
11 H	5.0	2.9	44	1.9
11 I	5.2	3.0	321	12.5
11 J	4.3	3.2	19	0.7
11 K	5.9	2.9	19	0.6
11 L	3.2	2890 cc	750	21.8
11 M	4.4	2.8	119	3.2
11 N	2.9	2.9	394	10.0

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TABLE III
Metathesis

Treatment	Settling Time (Hours)	Heel cc	Curies	% of Product Present at Decantation
<u>First Series</u>				
First Metathesis	4	3530)	----- 302	Recovered
Second Metathesis	4	3500)		
First Metathesis Cake Wash	4	5100)	----- 62	Recovered
Second Metathesis Cake Wash	4	1375)		
<u>Second Series</u>				
First Metathesis	2½	3640)	----- 35	0.9
Second Metathesis	2½	3550)		
First Metathesis Cake Wash	2½	4320)	----- 93	2.4
Second Metathesis Cake Wash	2½	1200)		

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TABLE IV
Time Cycles

<u>Operation</u>		<u>Hours Required</u>
<u>Charging</u>		
11A - 321 Slugs		3 $\frac{1}{2}$
11D - 324 "		5 $\frac{1}{2}$
11H - 251 "		5
<u>Coating Removal</u>		
First		2 $\frac{1}{2}$ 4 [?]
Second		3
Third		2 $\frac{1}{2}$
<u>Metal Solution and Extraction</u>		
	<u>Solution</u>	<u>Extraction</u>
11A	A 4	10
11B	B 5 $\frac{1}{2}$	10
11C	C 4 $\frac{1}{2}$	9 $\frac{1}{2}$
11D	D 4 $\frac{1}{2}$	10
11E	E 4	9
11F	F 3 $\frac{1}{2}$	21 $\frac{1}{2}$ *
11G	G 3 $\frac{1}{2}$	10 $\frac{1}{2}$
11H	H 3	10
11I	I 3	10
11J	J 4	10
11K	K 4 $\frac{1}{2}$	10
11L	L 16	10
11M	M 10 $\frac{1}{2}$	10
11N	N 9	10
<u>Extraction Cake Wash</u>		
First		7 $\frac{1}{2}$
Second		17 $\frac{1}{2}$ **
<u>Metathesis</u>		
First		12
Second		8 $\frac{1}{2}$
<u>Metathesis Cake Wash</u>		
First		8 $\frac{1}{2}$
Second		5 $\frac{1}{2}$

* Due to freezing of extraction slurry.
** Due to recovery of product in A-11.

TABLE IV
(Continued)

<u>Operation</u>	<u>Hours Required</u>
<u>Cake Solution</u>	
First	1 1/2
Second	1 1/2
<u>Electrolysis</u>	
First	10
Second	10
<u>PbO₂ Removal</u>	
First	2
Second	2 1/2
<u>Volume Reduction in B-6</u>	
First	2
Second	No Data
<u>Fuming Nitric Precipitation in B-21</u>	
	2
<u>BaCl₂ Precipitation, Washing and Product Solution in B-21</u>	
	4 1/2
<u>Sampling</u>	
	2
<u>Product Evaporation</u>	
	4
YE	3

A. C. Vallado

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B-137

Date August 8, 1946

Subject 706-D Production Run #12
(Shipment #20)

By A. C. Vallado

To W. A. Rodger

File _____

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This document consists of 10 pages and 0 figures. No. 4 of 6 copies, Series A

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A. C. Vallado

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8-8-45

By Authority Of _____
By [Signature] Date AUG 24 1971
706-D Production Run #12
(Shipment #20)

Mechanical Changes

At the end of Run #11 all Cell A vessels were cleaned with boiling nitric in an attempt to improve jet performance. All jets were then tested and found to be in good operating condition.

To facilitate some equipment replacements, the lead cubicles were decontaminated until radiation levels in working areas inside the cubicles were down to 500-1000 mr/hr. One set of glassware and defective Tygon tubing were replaced. The end of the B21-B17 Hastelloy O line, at the Tygon connection to cubicle exit, was found to be split. This connection was securely made and is expected to last until after Run #12, at which time Cell B will be decontaminated and the line removed and repaired.

Dissolver Operation

The 897 slugs charged to the dissolver for run #12 were added as follows:

<u>Date Pushed and Charged</u>	<u>Slugs Loaded</u>
6-9	295
6-10	276
6-12	328
	<u>897</u>

The slugs loaded on 6-9, 6-10 and 802 of the 6-12 loading were used for run #12. The remainder of the slugs loaded on 6-12 were used for a stand-by run which was later discarded. For the purpose of calculation only 771 slugs are considered. These were calculated to have an average of 6.9 Curies/slug active and 0.6 mg/slug inactive Ba (Total: 5300 Curies and 537 mg) at the time of discharge.

This run was the most successful to date, made in a single series, consisting of twelve dissolvings and extractions followed by metathesis, cake solution, electrolysis and volume reduction in B5. The amounts of reagents used for the coating removals (156# 60% HNO₃ and 306# 35% NaOH) and for the metal solutions (390# 60% HNO₃ and 170# H₂O) were normal for 65 slug batches.

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A total of twelve (average 64.5 slugs dissolved) metal solutions and three coating removal reactions were carried out with difficulty.

Extraction

The quantities of reagents for all twelve extractions were uniform for 65 slug batches (353# 65% H_2SO_4 , 670 cc 20% $Pb(NO_3)_2$ for A batches and 120 cc 20% $Pb(NO_3)_2$ for all others). 65% H_2SO_4 was used instead of the usual 90% as it would probably improve the settling rate in extractor A9. All decantations were cut at 2.6" to 11.0" heels on the oil liquid level manometer using jet A9-ASDA through batch F and A9-ASDB through batch L. Losses averaged 28 Curies per extraction or 9.6% of the product from each individual dissolving. These losses were somewhat lower than those experienced in Run #11 and can be attributed to the use of 65% H_2SO_4 and the more frequent use of A9-ASDB jet. During the first six decantations the A9-ASDA jet was used and the losses were high, necessitating an additional decantation to A-11. Then all metal solutions were transferred to A9 via A-11 instead of directly, to recover product. The A9-ASDB jet was used for the last six decantations and the losses tapered off. Apparently the jetting rate is directly proportional to the decantation losses, since the rate was 28-40 minutes for the B jet and only 20-24 minutes for the A jet. During the next run the A9-ASDB jet will be used for all decantations from A9. See Table II for complete extraction decantation results.

The extraction cake was given the usual one acid, four water washes, all of four gallon volume. Each wash was agitated 5 minutes, settled 70 minutes and decanted to 2.3" to 3.1" heels on the oil liquid level manometer using jet A9-ASDB. The waste loss for the combined washes was 7 Curies (See Table I).

Metathesis

The metatheses were normal and uniform reagent quantities were used. They were decanted to A8 using jet A9-ASDB and cutting at 2.0" to 2.2" heels on the oil liquid level manometer. The combined waste loss of the metathesis and metathesis cake wash was normal (130 Curies).

The metathesis cake solution was transferred to the electrolysis vessel (B12) in Cell B in three shots via B26 and B27. The first was 500 cc of 70% HNO_3 ; the second 320 cc 70% HNO_3 and 200 cc H_2O ; the last was a 500 cc water wash. No difficulty was encountered during this transfer and the system operated satisfactorily. The final volume in B12 was 4000 cc.

Cell B Operation

The electrolysis time was the same as Run #11 (3 hours at 15 amps. and 7 hours at 25 amps.). No Barium carrier was added.

8/8/46

After electrolysis the product was transferred to precipitator B6, evaporated to dryness, dissolved in 2 liters of water and reconcentrated to 310 cc. The product solution was transferred to B21R by way of B11 without difficulty, where two fuming nitric precipitations were made followed by two barium chloride precipitations and subsequent washings. The combined losses were normal (217 Curies or 6.5% of the total product dissolved.).

The product solution was transferred from B17 to B19 via B8, the upper discharge funnel and movable funnel. The final evaporation took 4 hours and went without difficulty.

Final Analysis of the Product was:

Active Barium 2500 Curies at LST 1100 - 6/17/46

Direction Radiation Reading 2500 Curies \pm 15%

Contaminants

Pb	57 mg
Fe less than	0.5 mg
Cr less than	0.5 mg
Ni	17 mg
Sr	0.8 mg
Ba	880 mg

The dried product appeared to be the lightest yet turned out in 706-D. It was large in bulk than some and had the usual distribution in the cone.

The run went without major difficulty and in most respects was the most successful to date. The yield (73%) and material balance (95%) were the best results ever recorded.

Approximately 20 Curies of Barium (from an equipment rinse), the Strontium (in the HCl-ether waste), and a small quantity of Iodine concentrate were delivered to 706-C.

Analyses

In Table I complete analytical data are presented. Table IA gives a summation of Barium losses for the run. Direct radiation measurements were made by moving the uncovered cone under the chimney and taking readings at the top of the cell.

Contamination and Radiation Exposure

From the standpoint of exposure the run was very satisfactory. No overexposures were reported. The building remained at or near background, and the only spill (in sampling 6P) was small and confined to the immediate B6 sample blister area.

W. A. Hodger

-4-

8/8/46

Building air contamination, as high as 2000 counts/min/half hour precipitron run were experienced every night between 10:00 PM and 7:00 AM, at which time atmospheric conditions were adverse.))

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TABLE I

(All Curies are calculated to the Shipping Time - 1100 - 6/17/46)

Fraction	Code	Curies Product	Curies in Waste	% Total Product Dissolved
First Dissolving Extraction Waste	1MA 6WMA	280	5	0.1
Second Dissolving Extraction Waste	1MB 6WMB	270	6	0.2
Third Dissolving Extraction Waste	1MC 6WMC	241	18	0.5
Fourth Dissolving Extraction Waste	1MD 6WMD	318	15	0.4
Fifth Dissolving Extraction Waste	1ME 6WME	274	12	0.4
Sixth Dissolving Extraction Waste	1MF 6WMF	309	15	0.4
Seventh Dissolving Extraction Waste	1MG 6WMG	260	13	0.4
Eighth Dissolving Extraction Waste	1MH 6WHM	243	14	0.4
Ninth Dissolving Extraction Waste	1MI 6WHI	341	28	0.8
Tenth Dissolving Extraction Waste	1MJ 6WJ	313	37	1.1
Eleventh Dissolving Extraction Waste	1MK 6WMK	296	17	0.5
Twelfth Dissolving Extraction Waste	1ML 6WML	270	150	4.4
Total Dissolved		3417		
Total Extraction Waste			330	9.6
Cake Wash	8WY		7	0.2

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TABLE I

Fraction	Code	Curies Product	Curies in Waste	% Total Product Dissolved
Metathesis Waste	8WC		67	2.5
Metathesis Wash	8WCW		45	1.3
Product in B-6	6P	3177		
Electrolysis Loss	3WPb		45	1.3
B-6 Rinse	6RW		20	0.6
Fuming-Nitric Wastes	3WLN		197	5.7
HCl-ether Waste	3W-HCl		80	0.6
Final Product in B-17	17P	2500		

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TABLE IA

Summation of Ba Losses For Run #12
(Curies are Calculated to LST - 1100 - 6/17/46)

Fraction	Curies	%
Total Dissolved (Calculated as 771 Slugs)	5300	100.0
Decay Loss (9, 8, 6 Days)	1860	35.1
Known Losses in Cell A	467	8.8
Known Losses in Cell B	280	5.3
Yield	2500	47.2
Material Balance		96.4
Total Curies Found in Dissolver Solution Calculated to 1100 - 6/17/46	3417	100.0
Cell A Waste Loss	467	13.7
Cell B Waste Loss	280	8.2
Product Yield	2500	73.3
Material Balance	3247	95.2

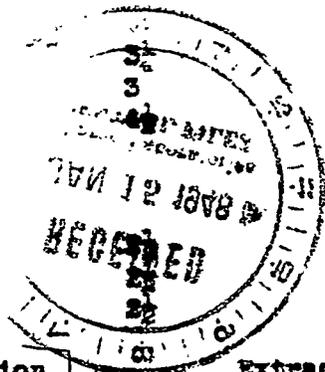
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TABLE IV

Time Cycles

Operation		Hours Required
<u>Charging</u>		
12 A	293 Slugs	
12 D	276 "	
12 I	323 "	
<u>Coating Removal</u>		
First		
Second		
Third		
<u>Metal Solution and Extraction</u>		
12 A		11
12 B		10
12 C		10
12 D		10
12 E		10
12 F		10
12 G		10 1/2
12 H		10 1/2
12 I		10
12 J		10 1/2
12 K		10
12 L		11
<u>Extraction Cake Wash</u>		6 1/2
<u>Metathesis</u>		9
<u>Metathesis Cake Wash</u>		5 1/2
<u>Cake Solution</u>		1 1/2
<u>Electrolysis</u>		10
<u>PbO₂ Removal</u>		4 1/2
<u>Volume Reduction in B-6</u>		2 1/2
<u>Fuming Nitric Precipitation in B-21</u>		1 1/2
<u>BaCl₂ Precipitation, Washing & Product Solution in B-21</u>		1
<u>Sampling</u>		
<u>Product Evaporation</u>		4
<u>Loading</u>		72



Solution	Extraction
A 5 1/4	11
B 6 1/2	10
C 6	10
d 4 1/2	10
e 4 1/2	10
f 5	10
g 5 1/2	10 1/2
H 7	10 1/2
I 5 1/2	10
J 4 1/2	10 1/2
K 5 1/4	10
L 4 1/2	11

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TABLE II
Extraction Decantation

Batch	Rate of Decantation Gal/min	Heel Gal.	EA LOSS	
			Curies	% of Product Present at Decantation
12 A	4.6	3.0	63	22.5
12 B	4.1	3.1	51	9.3
12 C	5.2	2.9	125	15.8
12 D	4.1	3.0	176	15.9
12 E	4.9	2.8	129	9.3
12 F	4.7	3.4	304	18.0
12 G	2.9	2.9	224	11.5
12 H	2.3	2.9	73	3.3
12 I	2.4	2.8	28	0.8
12 J	3.8	2.9	37	1.1
12 K	3.1	2.8	17	0.5
12 L	3.6	3340 cc	150	4.4

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TABLE III
Metathesis

Treatment	Settling Time (Hours)	Heel cc	Ba Loss	
			Curies	% of Product Present at Decantation
First Metathesis	2½	5480)	}----- 87	2.5
Second Metathesis	2½	3350)		
First Metathesis Cake Wash	2½	4520)	}----- 43	1.5
Second Metathesis Cake Wash	2½	1200)		

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CENTRAL FILES NUMBER

46-8721

12-576

August 21, 1946

Subject: Shipment #21

S. A. Reynolds

H. C. Leverett

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Shipment #21

The barium chloride product had about the same appearance as shipment #20. Its color was light tan. A film was noticeable on the wall of the cone (#16) up to about two inches.

Ba* 2900 Curies, analyzed radiochemically, corrected to 1145, 8-20-46, last separation time.

2000 Curies, by gamma radiation reading ("skyshine") (The sample for radiochemical analysis was taken in the B-17 vessel, and transfer and evaporation took place before the radiation reading. It is possible that some loss took place in these operations.)

Spectrographic Analysis

Pb	7 mg
Fe	<0.5 mg
Cr	1 mg
Ni	9 mg
Sr	0.2 mg
Ba	900 mg

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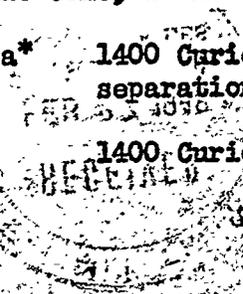
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SHIPMENT #22

The barium chloride product was quite light in color, lighter than most previous products. The solid was concentrated in the tip of the cone, with no noticeable film on the wall.

Ba* 1400 Curies, analyzed radiochemically, corrected to last separation time, 1500, 12-9-46.

1400 Curies, by gamma radiation reading ("skyshine").



Spectrographic Analysis

Pb	4 mg.
Fe	2 mg.
Cr	1
Ni	3
Sr	1
Ba	Lower than previously*

* Ba result not yet available, but will be reported soon.

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Date: December 25, 1946

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To M. C. Leverett

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Shipment #22

Supplementary Report

Spectrographic Analysis for Ba

Ba 300 mg.

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