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A HISTORY OF THE RADIOACTIVE BARIUM-LANTHANUM PROCESS AND PRODUCTION

W. E. THOMPSON, JR.

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A HISTORY OF THE RADIOACTIVE BARIUM-LANTHANUM PROCESS AND PRODUCTION

W. E. Thompson, Jr.

[redacted]

JUN 22 1949

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ABSTRACT

Large scale production of radioactive barium-lanthanum was requested by Los Alamos on April 8, 1944. The production process was developed and put into operation in building 706-C, whose facilities were converted to Ba-La production in five months, the first run being completed in September, 1944. In all, 9 shipments, totaling 3,852 curies were made using 706-C equipment. When it became apparent that Ba-La production was to continue on a long-term basis, building 706-D was constructed especially for Ba-La production. Construction of 706-D was completed and operations were started in May, 1945. A total of 31 shipments were made from 706-D from May, 1945 to March, 1949, delivering a total of 62,412 curies to Los Alamos.

The production operations in 706-C and -D have always been of an unpredictable nature. Many difficulties have been encountered and overcome with varying degrees of success. At present, the entire process is being reviewed by the Technical Division to determine what improvements can be made.

Future production of Ba-La is expected to be on a scale of 4 to 10 times larger than present operations. Investigations are under way to determine what new facilities must be provided.

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Introduction

In 1942, Chemistry Group IV under Dr. C. D. Coryell of the Metallurgical Project in Chicago, was engaged in "organizing chemical procedures and apparatus for a quantitative study of the individual fission products, their radiation characteristics and the chemistry of their separations". (1) Using uranium bombarded with neutrons resulting from the reaction, Be(d,n) carried out by Drs. Wahl and Seaborg at Berkeley, Dr. Coryell's group made rather extensive investigations into the barium-lanthanum fission product decay chain. (2) This work included fundamental research on the radiations emitted by the fission products and their daughters. (3).

With 30 grams of $UO_2(NO_3)_2 \cdot 6H_2O$ received from Berkeley Dr. Coryell, in July 1942, separated fission Ba^{140} as $BaCrO_4$ after ether extraction of U and precipitation of other groups and noted that the gamma activity was quite low. At the same time Dr. Sugarman separated $BaCl_2$ directly from uranyl nitrate using concentrated HCl and also failed to find high yields of the penetrating gamma activity. Mr. William Rubinson separated Ba^{140} by still another method with the same result: little of the expected gamma activity was found. (2) A growth of gamma activity was noted in all the separated Ba^{140} samples. Radiation studies and measurements led Dr. Coryell to make this statement in his Progress Report (CC-208) for July 25, 1942: "There is little doubt that much of the long-lived and penetrating gamma radiation from the fission products is to be ascribed to the 40 h La^{140} daughter of the 300 h Ba^{140}.....".

The following month, "because of the interest in the gamma radiation from the 40 h La^{140} growing from 300 h Ba^{140}, special attention was given to Ba, Sr, and La separations". (4) Using methods reported by the German chemists, Lieber (Naturwiss 27, 421, 1939) and Hahn and Strassmann (Naturwiss 27, 11, 1939), Dr. Coryell and his group were able to make effective separations of Ba, La and Sr. with good yields. These investigations continued for several months and on October 15, 1942 in the Metallurgical Project Monthly Report (CC-298) it was reported that, "It is seen that there is a negligible amount of penetrating radiation associated with the Ba^{140} decay". Thus it was established that the gamma radiation of the Ba-La chain was due to the 40 h La^{140}. In the same report it was noted that the La^{140} activity is maximum after 143 hours when it is 72% of the original Ba^{140} activity and also that the energy of the La^{140} gamma is about 1.7 Mev. (5).

Construction of the "Hot" Laboratory (706-C)

In autumn 1943 Dr. Coryell transferred to Clinton Laboratory as the Section Chief of a group which continued the fission product studies. This group was composed of many of the same individuals who were at Chicago in

1942 and their work in building 706-A with the Chemistry Division followed the general plan started at the Metallurgical Laboratory.

By the latter part of 1943 the need for additional facilities to continue the fission product studies was apparent and on October 21, 1943 Drs. C. D. Coryell and Henri A. Levy sent a memo to Dr. M. D. Whitaker requesting "that a new laboratory be erected which contains a number of concrete shielded hoods embodying devices for viewing and controlling chemical operations from the outside. Preliminary plans for the laboratory and accessory space are presented herewith. We consider it important to the Project that such facilities be made available soon, if possible, by January 1, 1944". (See Appendix A) (6)

This request was referred to Dr. A. H. Compton of the Metallurgical Project by Dr. Whitaker and on November 16, 1943 Dr. Compton sent the following memo to Major A. V. Peterson:

"1. Will you please authorize the construction at Clinton Laboratories of a Chemical Laboratory to be used as a "hot laboratory" in carrying out certain necessary processes in connection with the chemical and biological programs which require for their prosecution quantities of pure fission products.

"2. This specialized laboratory is necessary for the production of pure fission products. These are needed for both chemical and biological work involved in the Hanford program and in the limited studies which are to be carried on at General Groves' request on their possible use for offensive purposes. These needs can be listed in detail as follows:

"1) Fission products in pure form are required for following through the details of the decontamination processes proposed for Hanford.

"2) They are required also, in appreciable quantities, in order to identify those which may be formed in relatively low concentration, but which nevertheless lead to serious difficulties in the decontamination processes at Hanford concentrations.

"3) They are also needed so that the proposed processes may be carried through their complete cycle at the high radiation densities which will prevail at Hanford concentrations and not only at particular stages where tests can be made with external radiation sources as at present.

"4) They are also necessary for physiological tests to determine:

"a) The magnitude of hazards to operating personnel.

"b) Public health hazards including the effect of radioactivity in water, and

"c) Their potential value as an offensive weapon.

"3. The facilities required for this work are a suitable structure to house two banks of four properly shielded concrete cells together with the appropriate and necessary auxiliary facilities. These latter include:

"1. Four small laboratories for semi-hot operations

"2. Chemical purification laboratory

"3. General laboratory

"4. Counting room

"5. Storeroom facilities

"6. Glass blowing shop

"7. Small machine shop

"8. Mens change room, showers and toilets

"9. Womens change room, showers and toilets

"10. Utility room

"11. One office

"This building should have a total area of approximately 5,000 square feet of floor space.

"4. We would appreciate receiving your authorization for this construction on this unit be started immediately for it to serve its purpose in our program".

This request was approved and on December 11, 1943, ground was broken for the construction of building 706-C. Design of the building was underway at the same time and on December 20, the following teletype was dispatched from Clinton Laboratories:

DANIELS HAUPT HAWTHORNE

DESIGN DIVISION WILMINGTON
CONSTRUCTION IN ACCORDANCE WITH CL 706C-1 SHEET 1
IS HEREBY APPROVED. S. W. PRATT (8)

Construction started immediately and progressed rapidly with men on the job 20 hours a day.

Building 706-C was completed and turned over to the Chemistry Division operations group on March 3, 1944. (9)

Background of the Barium-Lanthanum Project

In December 1943, during his visit to Clinton Laboratories, Dr. J. R. Oppenheimer indicated that Los Alamos would be interested in having Clinton Laboratory prepare 100-curie quantities of some strong gamma emitter and inquired into the feasibility of doing this. Dr. Coryell suggested that La^{140} with a 1.7 Mev gamma might be suitable. He pointed out that Ba^{140} could be rather easily prepared and that past experience had shown that its chemistry is clean and relatively simple, its fission yield is high, and the 12-day half-life of Ba^{140} is long enough to make it suitable for shipment from Clinton Laboratories to Los Alamos. Dr. Oppenheimer agreed that La^{140} would be the most suitable of the known fission products and left without making or accepting any commitments. (10)

The question of Ba-La production was reopened on March 16, 1944, by Dr. A. H. Compton at the request of Dr. Oppenheimer. The tentative request from Oppenheimer to Compton came through General Groves for "about one curie of a mixture of radiolanthanum and radiobarium 'not necessarily separated from other active materials' at as early a date as possible "and" 100 curies of radiolanthanum together with its parent radiobarium with a delivery date about June 1st". (See Appendix A) (11)

The problem was given immediate consideration and some preliminary tests on the separation of barium from uranyl nitrate with $pbSO_4$ carrier were begun. (12) On March 28, 1944, Drs. C. D. Coryell and H. A. Levy made a feasibility report to Dr. R. L. Doan, stating that "this request can be filled by early summer with raw materials currently available, and with the present plant facilities if certain requirements of equipment and manpower .....can be met". (See appendix A) (13)

Dr. Compton made the official request for production of Ba-La on April 8, 1944, in a letter to Dr. M. D. Whitaker: "I have received a message from Oppenheimer in which he confirms his earlier request which I recently passed on to you for radiobarium and lanthanum. It appears that the promise of usefulness of this material has increased considerably since his first request.

"He would like to have the first delivery of about 100 curies sometime between July 15 and August 1st". (14) (See Appendix A)

The progress report of Dr. Coryell's Chemistry Section for April 15, 1944, states: "Radiochemical tests have shown a sequence of operations (involving precipitation of $PbSO_4$ from 45% UNH, solution of $PbSO_4$ in NH_4Ac, precipitation of $Pb(NO_3)_2$ with fuming HNO_3, solution of $Pb(NO_3)_2$ in water and precipitation of $BaCl_2$ added as a carrier with concentrated HCl) to be satisfactory for the extraction, concentration and decontamination of fission barium. Liter scale runs using stainless steel containers and approximating operating conditions and technique expected in the full-scale preparation showed fairly satisfactory radiochemical yields (about 60%) and operating characteristics". (15)

The equipment which had already been installed in 706-C for general fission product work was adapted for the 1 curie Ba-La preparation while the large scale, 100 curie equipment was being designed, built and installed. (16) By April 30, 1944, the large scale stainless steel equipment for the $PbSO_4$ extraction step was about two-thirds complete and the adaptation of the small scale equipment was even further along. Testing of the equipment already installed was in progress during May 1944, while liter-scale laboratory runs were being conducted simulating full-scale conditions and techniques. By means of these laboratory "dummy runs" the process had been perfected to give overall yields of better than 75%. (17)

The shipping container for 1 curie of La^{140} was fabricated early in May and on May 13 the first "hot" run was started with the dissolving of two slugs in the adapted fission product equipment. The final product, with 50 mg of inactive Ba carrier added, assayed 300 millicuries of Ba^{140} - about half what was expected. (18)

During this period of May-June 1944, process development through the liter-scale laboratory runs resulted in some changes in the general procedure which increased the overall Ba-La yield. The principal difficulties were encountered in the handling of small precipitates. Several methods of separating lead and barium from UNH using caustic precipitation and chromate precipitation were tried and abandoned for various reasons. Finally it was decided that the $PbSO_4$ precipitation was most satisfactory. By changing the method of adding H_2SO_4 to carry out the precipitation slowly and then digesting the precipitate, better yields were achieved. In order to partially remedy the troubles caused by handling small precipitates, it was decided to combine the $PbSO_4$ precipitates from three batches of UNH before dissolving them with ammonium acetate. The procedure called for loading into the dissolver 36 slugs, of which 12 slugs would be dissolved with HNO_3 for one batch. Before each new dissolving 12 new slugs would be loaded. Thus a minimum of 24-36 slugs would be present in the dissolver during the dissolving of each of the 3 batches of UNH. $BaSO_4$ would be precipitated with a $PbSO_4$ carrier by the addition of $Pb(NO_3)_2$ and H_2SO_4. These Ba, $PbSO_4$ precipitates would then be combined by the simple expedient of precipitating the second and third batches on top of the precipitate left from the earlier batches after the UNH was decanted.

The month of August was devoted to equipment testing and process development by making full scale tracer runs through the equipment. During this period it was found necessary to adapt the stainless steel precipitator tank to closed operation by installing a cover with a condenser and scrubbing tower on the off gas line. This was done to minimize contamination of the cell by radioactive spray.

Early in September 1944, testing of the equipment was completed and production operations were ready to be started (19).

706-C Equipment, Facilities and Process

The 706-C "Hot" laboratory was designed for the handling of 1-10 curie amounts of radioactivity. Because of the limitation of time and unusual circumstances, these facilities were adapted, without any major structural changes, to the production of greater than 100-curie amounts. This change in plans introduced two major problems: a) the handling of larger amounts of both active and inactive material than had been anticipated, and b) the shielding against radiation of an order of magnitude several times greater than the facilities were designed to accommodate.

The solution of the first of these problems - the handling of large amounts of materials - was relatively simple. Larger stainless steel equipment was installed for the phase of operations which involved large volumes.

The problem introduced by the additional radiation was never satisfactorily solved. The addition of sufficient shielding was not feasible from the standpoint of time and available space; so the arrangement adopted was to rotate the workers on the critical jobs, such as sampling, in such a way as to allow each operator to receive only a tolerance exposure in any day. There were overexposures to radiation in spite of this procedure, but none of a serious nature and the major handicap introduced was inconvenience, although the health hazard was certainly of prime importance (20).

The equipment for the Ba^{140} separation was installed in one of the two banks of cells in 706-C. The other bank of cells which had been used for the 1-curie preparation was returned to the normal fission product separation uses for which it was originally intended.

In the Ba^{140} operations cell stainless steel equipment was installed to handle the large volumes present through the extraction step. This equipment consisted of the following vessels and facilities:

1. Slug dissolver (65 gallon capacity)

2. Condenser for vapors from dissolver

3. Precipitator (with two decant lines)

4. Scrubbers for dissolver and precipitator off gas

5. Neutralizer tank

6. Underground waste storage tank outside the building

The two large stainless steel tanks, dissolver and precipitator, were shielded by piling lead brick around them to a height of $2\frac{1}{2}$ feet. The bricks were supported by frames fabricated from sheet steel. This arrangement supplied 4 inches of shielding on every side except the top, where it was believed that the distance from the solution to the top of the cell would provide sufficient protection (21).

The rest of the operation in the cell was carried out in glass equipment designed around a stang precipitator of 5-liter capacity. There were duplicate sets of glassware, one in Cell 1 and one in Cell 2, with crossover lines for transferring from one to the other in cases of emergency.

The initial procedure worked out for use was as follows:

1. Removal of aluminum jackets from 36 slugs

2. Dissolving uranium metal equivalent to 12 slugs using HNO_3 and transferring the solution to the precipitator

3. Coprecipitating Ba, $PbSO_4$ with H_2SO_4 and decanting UNH supernate

4. Loading 12 more slugs into the dissolver and removing aluminum jackets

5. Redissolving precipitate in UNH from a second dissolving equivalent to 12 slugs; then reprecipitating Ba and $PbSO_4$

6. Repeating steps 4 and 5 so that Ba from 36 slugs was separated from UNH

7. Separating and removing $PbSO_4$ by using hot ammonium acetate solution and decanting supernate

8. Metathesising $BaSO_4$ using fuming HNO_3

9. Dissolving $Ba(NO_3)_2$ with 6N HNO_3 and removing to storage

10. Performing steps 1-9 five times so that Ba is recovered from 180 slugs

11. Reducing solution volume by precipitating $Ba(NO_3)_2$ in batches with fuming HNO_3

12. Precipitating most of the residual lead as $PbCl_2$ using 1 N HCl and separating $BaCl_2$ filtrate

13. Precipitating $BaCl_2$ from concentrated HCl-ether mixture and removing remaining lead and impurities in the filtrate

14. Dissolving $BaCl_2$ and evaporating to dryness in the product shipping container

This process, as outlined, was used for the first three preparations. After the third preparation the ammonium acetate solution of lead sulfate was eliminated because of high losses. Metathesis of Ba, $PbSO_4$ with K_2CO_3 was substituted, followed by the separation and removal of $PbCrO_4$ (22).

706-C Hot Operations

A. H. Compton's letter of April 8, 1944, to M. D. Whitaker requested that the first 100 curie shipment be made between July 15 and August 1, 1944. However, it was not until the first of August that the glass apparatus for final preparation and purification of $BaCl_2$ was installed. At that time in a telephone conversation with Los Alamos, Dr. Coryell estimated that shipment could be made on August 15. Delay in getting final equipment alterations made and in testing the equipment held up operations until September 10, 1944, when the first run was started.

A considerable amount of the delay was due to the alteration of equipment to allow processing of the Ba-La with only two grams of dead Ba carrier instead of five grams as originally planned. A feeling of urgency seemed to dominate everyone associated with the Ba-La project - telephone calls to Los Alamos became almost daily routine with teletype messages filling in the gaps. There were many uncertainties which had to be clarified and problems which demanded immediate solutions. Coordination of the work at Clinton Laboratory with that at Los Alamos was quite difficult, although it was handled extremely well. For example, the product shipping cones were designed and fabricated at Los Alamos while the lead shielding to encase the cones was fabricated at Clinton Laboratories. Much coordinated effort was necessary to insure that the lead carrier would fit the cone in its final

design. Needless to say, the tension mounted as the deadline drew closer (23).

On September 10, 1944, the first large-scale preparation of Ba^{140} was started in 706-C with the loading and dissolving of 180 slugs from the pile. The operations progressed normally although somewhat behind schedule because of the difficulties in sampling. Fifteen dissolvings were made, with the fifteenth being held in reserve as a "stand-by" in case of operational difficulties.

Chemical analyses of the product solution gave the low result of 112 curies which led Dr. Coryell to note in his log that, "Crisis in Ba analyses of runs 1 to 4 is critical. Expected input from runs 1 to 4 about 210 curies. Analyses of 2 samples gave 112 and 122 curies respectively". These low analyses were found to be the result of faulty analytical methods and an alternative method gave results averaging 214 curies. During this period of crisis numerous key personnel stayed on the job as much as 3 and 4 days without going home. Short cat-naps and gallons of coffee served to keep them going until the run was over. Several individuals worked well over 100 hours this week; and in the weeks that followed long hours were the rule rather than the exception.

While the analytical difficulties were being solved, the stand-by dissolving was processed and combined with the rest of the run in the hope that it would pick up some activity left in the vessels. By this time the revised analytical results were completed, much to the relief of all concerned, and the final purification took place without further incident.

Radiation measurements on the product through the side of the carrier gave a value which was calculated to represent 300 curies. Subsequent measurements at Los Alamos indicated 314 ± 20% curies. The relief felt by the whole group at the successful culmination of the first preparation is shown by the final entry in Dr. Coryell's log, "Everybody was tired and happy and after the long letter * was sent (accompanying the product carrier to Los Alamos) Doan gave us a poem".
*See Appendix A.

"Ode to a Lost Nickel

"A nickel is a piece of dough
Whose value can't be questioned
Since Uncle Sam defines it so
You gotta take it, even though
You know darn well, as alloys go,
Intrinsically it's less than.

"Now this same nickel can become
A symbol strong and mighty
When harnessed to a proper bet
Involving jobs that must be met
By Coryell and his whole set
Of chemists, bright but flighty.

"We lost the bet - we're glad we lost
And so, we judge, is Oppie,
Who, wrapped in darkness and in doubt
Not knowing what 'twas all about
But sure that it would not get out
Had almost blown his topie

"So sing the praises of the coin
And sing also of chemists
Who break the rules and spoil the view
And yet when pressed with something new
They make a bet and then come through
With much more than the limits.

"R. L. Doan
September 18, 1948"

The first preparation of Ba^{140}, 314 curies, was transferred to the Contracting Officer for shipment, by truck, to Los Alamos on September 18, 1944 (24).

The second preparation was started on October 10, 1944, after complete re-installation of glass equipment in Cell 1. Operations proceeded smoothly except for minor equipment failures. Despite the ease of operation, there were rather high losses during processing and the final product assay was only 195 curies of Ba^{140}. The final product was held over about six hours before shipment to be studied. Electroscope measurements indicated this preparation to be weaker than the first one. An attempt was made to determine the energy of the gamma radiations by taking data with an elecroscope for plotting an absorption curve of the gamma radiation intensity. The beam coming from the unshielded source on the floor was studied from the roof of 706-C. At this distance, 30 feet, the intensity at 16 hours growth was 1100 mr/hr. (25). This preparation was shipped on October 17, 1944, accompanied by a letter of description (See Appendix A).

After the second preparation operations were "undertaken to recover Sr from the $BaCl_2$ mother liquor which contains about 8 curies of Sr and from 10 to 50 curies of Ba. This was introduced into the reactor vessel (precipitation filtration flask) but escaped through a hole due to a break which had not been previously detected. After thorough flushing, radiation levels in Cell 1 were 600 r/hr. After 10 days of tedious decontamination, radiation levels were lowered to about 1000 mr/hr at which time the broken

reactor vessel and the contaminated final product vessels were removed from the cell. This procedure followed by further scrubbing of several contaminated areas lowered the radiation to 300-800 mr/hr in the working area where replacements had to be made. The cell (#1) is now in good operating condition". This was written on October 30, 1944, in the Chemistry Division Progress Report (25).

The Cell 2 equipment was used for the third preparation which was shipped on November 23, 1944. The processing of 180 slugs gave an overall yield of approximately 50% with a material balance of 75%, the remaining 25% could not be accounted for. The final product assay was 280 curies. Operations were normal and satisfactory except that the mechanical agitator in the precipitator tank failed (27).

Several weeks of extensive decontamination reduced the radiation level to a point where repair work could be done and the agitator was removed and rebuilt. Other repairs were made and some modifications of equipment were adopted to rectify a number of weaknesses encountered earlier (28).

During the period it was decided to eliminate the NH_4Ac dissolution of the $PbSO_4$ precipitates and substitute a metathesis of $PbSO_4$ with K_2CO_3 followed by dissolution in weak HNO_3. It was belived that this would eliminate the losses due to incomplete dissolution of $BaSO_4$ in NH_4Ac. After the metathesis, the HNO_3 solution was transferred to the glass reactor and $Pb_3(OH_2)(CO_3)_2$ was precipitated to reduce volume. This precipitate was again dissolved in HNO_3 and $PbCrO_4$ was precipitated by the addition of $K_2Cr_2O_7$. From this point the standard $Ba(NO_3)_2$ precipitation with fuming HNO_3 and the final precipitation as $BaCl_2$ were carried out as usual (28).

It is interesting to note that in the period between the third and fourth preparations design, development and construction of building 706-D were started. This building was for the special purpose of making large-scale BaLa preparations.

During preparation # 4, a line broke while the $PbCrO_4$ precipitation was being made and resulted in the loss of two thirds of the Ba^{140}. The remaining one third was processed to completion and shipped on January 23, 1945. The final product assay was 160 curies. This break left Cell 2 so badly contaminated that it was not used again during the Ba runs.

At the end of the fifth preparation, in which 478 curies were shipped on January 31, 1945, the apparatus was "in unsatisfactory condition for further use at three critical points. The windshield wiper in the precipitator tank (cell 4) which partially failed during preparation # 4 is now completely out of service. Lack of this device makes it impossible to agitate small volumes of solution in the precipitator tank; this greatly

delays important steps in the process and is believed to lead to considerably higher losses in the metal waste fractions. It seems to us highly desirable to repair this mechanism before proceeding with another preparation. It is not known at this time whether or not this repair will require complete decontamination of Cell 4.

"The second breakdown is in the reagent or upper cow (magnetic channel selector) in Cell 2. This has been manipulated only with the greatest difficulty during the last two preparations and has gradually been getting worse. Its repair before using this apparatus again seems necessary.

"The third breakdown is the glass delivery tube leading from the Hastelloy valve to the evaporation outfit which was broken in raising the cone into position just prior to the evaporation" (29).

After more extensive decontamination, the necessary repairs and alterations were made. "In Cell 4, the mechanical agitator and several leaks in the solution and air lines were repaired. The glass equipment in Cell 1 was completely removed in order to effect adequate decontamination; the new equipment now in the process of installation contains a number of improvements - such as, the replacement of glass magnetic valves by "Hastelloy C" needle valves, the replacement of glass storage flasks with specially fabricated vessels of greater capacity, and the installation of additional shielding around the waste bottle. The new evaporation unit will be so arranged that the active sample will be shielded by four inches of lead, while the vessel which formerly held the final solution just prior to evaporation has been eliminated. These changes will greatly diminish radiation leakage during the final steps of the preparation. Cell 2, in which a large spill occurred during preparation 4, has not been entered. The equipment is not in an operable condition and it will require several weeks' work to decontaminate the cell and reconstruct the equipment before the cell can be used" (30).

The sixth preparation was started as soon as repairs were completed and was ready on March 26, 1945. Yields and material balance were poor for unknown reasons although the new equipment operated quite satisfactorily. The only difficulty encountered was contamination of the cells and ducts, because of a leak in the dissolver jacket. This run yielded 315 curies, which were shipped on March 26, 1945 (31).

On April 3, 1945, the seventh preparation of 560 c was completed without unusual difficulty. After this run 73 curies of Sr^{89-90} were successfully recovered from the HCl-ether waste.

After the seventh run a new stainless steel jacket was installed to replace the leaky dissolver jacket which had caused considerable contamination during the sixth and seventh preparations (31).

The eighth and ninth preparations took place in May 1945, and were by far the most successful of all the preparations to that date. Preparation number eight assayed 750 curies on May 16 and preparation number nine assayed 800 curies (Los Alamos assayed this shipment at 975 curies) on May 28. Both preparations were performed with ease; the equipment operated most satisfactorily and remained in good condition (32,33).

Building 706-D for the large scale preparation of Ba-La was completed and in operation by the end of May 1945; consequently, the ninth preparation was the last to be made in 706-C. The following table summarizes 706-C Ba-La operations.

Preparation & Date		Assay	Remarks
9-18-44	1	314 Curies	No operational difficulties. Analytical results gave trouble
10-10-44	2	195 C	High losses
11-23-44	3	280 C	Mechanical agitator in precipitator tank failed
1-23-45	4	160 C	Two-thirds of product lost when line broke during $PbCrO_4$ precipitation
1-31-45	5	478 C	Precipitator tank agitator failed, magnetic channel selector failed, glass tube for product transfer into evaporator broke
3-26-45	6	315 C	Yield poor. Equipment operated satisfactorily Dissolver jacket leaked
4- 3-45	7	560 C	No difficulty
5-16-45	8	750 C	Best run yet. No Difficulty
5-28-45	9	800 C	Operations normal. Last preparation in 706-C
		3852 Curies	

The 706-D Construction Period

Preparation of radioactive Ba^{140} on a 100-curie scale began in

September 1944, in building 706-C. The facilities used in this process were adapted from those designed and built for small-scale (1-10 curies) fission product separation. Many operational difficulties arose from the inadequacy of 706-C process equipment.

On October 19, 1944, after the completion of the second 706-C preparation, Dr. Harrison S. Brown gave consideration to future prospects and policy for radioactive barium-lanthanum production. In a memo to Dr. M. D. Whitaker at that time, Dr. Brown gave the following review of current status and future prospects for Ba^{140} production at Clinton Laboratories:

October 19, 1944

"To date two shipments have been sent - one of the order of 300 Y curies: the second unknown but probably about 200 Y curies. The first preparation succeeded beautifully. At its conclusion most of us were confident that our equipment would last for perhaps twenty weeks. The second preparation proceeded with much greater difficulty and there was considerable evidence that our equipment was deteriorating rather rapidly. It has been found that now certain lines are plugged and certain glass breakages have taken place. These can be repaired in time but it is certainly indicative of the problems that will confront us in increasing number as time goes on. We know our yields are much poorer than we had originally supposed. The analytical problems connected with the preparation are formidable. We are now in the position where we realize that no barium analytical result connected with the process can be completely trusted. I should like to emphasize at this point that none of this should cast any reflection on the individuals connected with designing and constructing the setup. They have done a magnificent job under the most trying of circumstances. We all appreciate the order of magnitude of the difficulties in such a preparation.

"It is apparent that continued production of lanthanum for an indefinite period of time will require:

1. New building facilities

2. New equipment facilities wherein glass is eliminated as much as possible

"In connection with the building facilities, there are three possibilities:

1. Carry out the preparation in the 205 Building

2. Build an entirely separate building for the purpose

3. Build an addition to the present 706-C Building.

It is the opinion of both Mr. Johnson and myself that, from the point of view of a long range program, construction of an annex to the 706-C Building would be the most preferable. As we see it, the following work must start now and continue until the new unit is completed:

"1. By some means or other keep the present production unit operating successfully so that Y will continue to have a supply of lanthanum

2. Train new men so thoroughly in the operations that they will be able to take over the routine operations on the new unit

3. These men under training can release men now on the job for developmental work on instruments, gadgets, and techniques that will be necessary for the new setup

4. It is obvious that a crew must be placed immediately on studying the analytical problems connected with the preparation

5. A large crew of chemists must immediately plunge into chemical research on both the existing process and on devising a modification of the existing process for use in the new operating unit

6. Mr. Leverett should have a group working very closely with our men. As we see it, he would be in charge of all necessary engineering design work".

In the following days of October, 1944, daily planning conferences were held and a decision was reached to request construction of Building 706-D, which would be an annex to 706-C (34). Accordingly, on November 3, 1944, Dr. M. D. Whitaker requested from the District Engineer of the Manhattan District authorization of the construction of Building 706-D. The description of the proposed building submitted to the District Engineer was as follows:

"1. A processing area 50 ft. by 85 ft. to include two adjoining process cells of overall dimensions 20 ft. by 60 ft. by 20 ft. high and necessary access openings, process lines and equipment, control panels, operating platforms and ventilation

2. A solution make-up room, 10 ft. by 20 ft. equipped with tanks, scales, pumps and chemical feed lines

3. A chemistry laboratory, 20 ft. by 21 ft. equipped with sinks, tables, hoods, services and special equipment

4. Two analytical laboratories each 9 ft. by 22 ft. for testing process samples. These will be of special construction

5. An area 30 ft. by 40 ft. for services in connection with the special hazards. Included are monitoring room, uniform dispensary, locker room, men's room, personnel equipment, decontamination room and health office

6. Process equipment decontamination room 10 ft. by 10 ft.

7. A monorail system for handling miscellaneous heavy equipment material and containers. This system will be connected to that existing in 706-C

8. Two offices each 11 ft. by 17 ft. and which will be easily accessible to the operating area

9. A conveying system for material from Building 105 to the top of the operating cells

10. Storage space for materials and equipment needed in this process will be made available by utilizing the area on top of the chemistry and analytical laboratories and offices. Loading platforms will be provided along the main East-West plant road

11. Additional transformers and relocation of present transformer bank will be necessary. Connections to relocated power lines and existing services". "The total estimated cost of the proposed work is $175,000". (See Appendix B)

It was requested that construction of Building 706-D be rushed to completion in four months. Design and development work were to be carried on at the same time construction was under way. It was hoped that in this way the completion date could be met.

The request for construction was approved by the District Engineer's Office and work began late in November, 1944. The program which had been set up by Dr. R. L. Doan in his memo of November 4, was as follows:

1. Design - The Design Group of the Technical Division was responsible for seeing that the finished design embodied the latest and best ideas of all the qualified groups working on the problem. This group was responsible for design and specifications of both the building and equipment

2. Specification of the basic chemistry and approval of the flowsheet - The Chemistry Division and Technical Division, Section I were to develop a modification of the 706-C process which would be most suitable for use in the new operating area

3. Consultation and advice when needed on small-scale remote control operations - Chemistry Division

4. Specification of location and type of health monitoring instruments - Health Physics Division

5. Experimental engineering on small-scale centrifuges, jets, remote control mechanisms, etc. - Technical Division, Section I

6. Assistance in preparation of 706-D operating procedure - 706-D Operations Group

7. Specifications of analytical methods and techniques - Chemistry Division (34).

Since construction and design work were proceeding simultaneously, daily Design Conferences were held in order to keep ahead of construction.

To illustrate the concerted effort which was applied to this job, over 120 drawings were made up and turned over to the construction group in the two months between November 15, 1944, and January 15, 1945; over 200 drawings were made in all.

Development and Research Groups were under the same pressure as the Design Group in that it was necessary for them to establish a process and equipment layout before the time arrived for the operating equipment and facilities to be installed. By February 21, 1945, the development of a suitable chemical process was virtually complete, with only the decision remaining as to which of several alternates to use. The proposed processes were as follows:*

Coating Removal

A. Dissolve the jackets in NaOH with $NaNO_3$ catalyst

B. Dissolve the jackets in HNO_3 with $Hg(NO_3)_2$ catalyst - (rejected - reaction unpredictable)

Metal Solution

Dissolve in HNO_3 at a temperature of 100-105°C

Sulphate Carrier Precipitation

A. Co-precipitate Ba with 100 gms of Pb in a 3.4 M H_2SO_4 solution

B. Co-precipitate Ba with Sr in place of Pb as a carrier - (rejected-separation of Sr later in the process too difficult)

C. Co-precipitate barium and uranyl oxalates - (rejected - barium not carried in precipitate)

D. Co-precipitate barium and uranyl selenate (rejected - UO_2SeO_4 too insoluble, required complete precipitation of UO_2 to bring down Ba)

E. Co-precipitate $Ba(NO_3)_2$ and $Pb(NO_3)_2$ with $Ca(NO_3)_2$ (rejected - incomplete precipitation)

F. Co-precipitation of Ba with LaF_3 using HF (rejected - introduced equipment problems and chemical hazards)

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* These processes are described in detail in the report, MonN-330 "The Development of a Chemical Process for the preparation of Ba^{140}". W. H. Baldwin and J. E. Savolainen, July 23, 1947

Lead Removal Before Metathesis

A. Extraction of $PbSO_4$ with ammonium acetate (rejected - requires a filter for adequate separation of Ba)

B. Extraction of $PbSO_4$ with ammonium tartrate (rejected - would not extract $PbSO_4$)

C. Extraction of $PbSO_4$ with NaOH (rejected - formed colloidal Ba)

D. KCN fusion at 300-400° to reduce Pb to metal, leaving BaS and $BaSO_4$ (rejected - not easy to do and BaS, $BaSO_4$ not easy to process)

E. Reduction of Pb to the metal with hydrogen (rejected - formed BaS which is too difficult to process)

F. High temperature conversion to Ba, $PbCl_2$ using CCl_4 (rejected-hazard of volatile fission products and corrosiveness of Cl_2 gas)

Since none of these methods were satisfactory, it was decided to postpone the lead separation until after metathesis.

Metathesis

A. Metathesis with 1M K_2CO_3

B. Metathesis with NA_2CO_3 (rejected - results not consistent)

C. Metathesis with fuming HNO_3 (rejected - too slow)

Lead Removal

A. Precipitate Pb as $PbCrO_4$ in 1N HNO_3

B. Electrolytic removal of PbO_2 from HNO_3 solution (alternate)

C. Electrolytic removal of Pb from perchloric acid solution (rejected - not safe for routine operations)

D. Precipitation of $PbHPO_4$ (rejected - pH too critical)

E. Precipitation of PbI_2 (rejected - some BaI_2 precipitation, PbI not completely precipitated)

F. Precipitation of $PbCl_2$ (rejected - same as above)

G. Precipitation of $PbBr_2$ (rejected - same as above)

Barium Isolation

A. Following $PbCrO_4$ precipitation, Ba is precipitated as the chromate in 1M NaOH. Centrifuge to separate $BaCrO_4$ precipitate

B. Following electrolysis, precipitate $BaCO_3$ for volume reduction and decant supernate

Final Product Concentration

A. Precipitation with fuming HNO_3 followed by dissolution in H_2O and evaporation to dryness

B. Precipitation of $BaCl_2$ with concentrated HCl. (rejected - too corrosive for stainless steel equipment) (35).

Thus, all processes were considered and weeded out until it was felt that a satisfactory process was derived. Two alternate procedures for lead removal were left in the process because it was not known whether equipment to carry out the more favorable process (electrolysis) would be available in time for the first production operations.

The equipment for carrying out this process was decided upon largely from previous experience in Buildings 205 and 706-C. However, there were some facilities which required considerable development work, such as:

1. Jets for solution transfer

   a) Should steam or air be used?

   b) What are the optimum operating conditions?

   c) What performance can be expected?

2. Pipettes for solution transfer

   a) What is the most advantageous design?

   b) What performance can be expected?

3. Centrifuges

   a) How can they be designed to keep maintenance to a minimum?

   b) How much product loss will be introduced by the use of centrifuges?

   c) Where are centrifuges necessary?

4. Final Evaporation

   a) How should the product container be heated?

   b) What air flow around the container can be allowed?

   c) How do the different methods of heating effect the distribution of product in the cone?

5. Samples

   a) How can representative samples best be taken?

These are a few of the major problems which confronted the various groups who were concerned with the construction of 706-D. Many other problems arose and were summarily settled by discussion in the daily Design Conferences. It is interesting to note that satisfactory solutions to the problems listed above were found - and were found in the short length of time allowed before the answer must be known to the construction engineers.

The ultimate result of the combined effort of design and development groups was embodied in the 706-D production facilities approved for construction. These facilities, it was believed, represented the most complete and operable designs which could be produced on the basis of knowledge at that time, with every reasonable arrangement for flexibility and future changes.

The 706-D Building is attached to the east end of the 706-C Building. The architectural lines of C-building were extended to join the taller structure which houses a cell block and attendant facilities. The process section has three stories and a monitor. In this section are included the solution make-up room, equipment decontamination room, operating cells, open operating area and storage areas (36).

Cells A and B form a concrete structure 20 feet high, measured from the first floor level, by 19 feet wide, exclusive of the entrance labyrinths, by 50 feet long. This cell block extends into the ground about 8 feet to give an inside height of 20 feet for Cell A and to allow for ventilation duct work under the cell floors. A secondary wall extends along the entire east side between the first and second floors to form passageways 3 feet wide to cell openings on either side of the partition separating the cells; concrete roofs at the second floor level complete the passageway. The other major appurtenance to the cell block is a ledge extending along the north side of the top for temporary storage of concrete slabs taken from the roof openings.

Besides the labyrinth openings, the cells are accessible through three large roof openings, two of which are equipment holes (5 feet sqaure) to allow tanks and equipment to be lowered into the cell, and one is three feet square for handling the shipping carrier in and out of Cell B. A fourth roof opening into Cell A, two feet square, holds the plug containing the slug loading funnel and pipe to the dissolver. The cell block also has 548 steel-lined holes stepped once from 6 inches diameter to 5 inches diameter; the 327 horizontal holes are mainly on 20-inch centers and the 221 roof holes are on 12-inch centers. Concrete plugs with curved pipes, shaft guides, etc. fill a large proportion of these holes; the remaining ones are filled with blank plugs. A single row of process pipes and conduit is cast in the walls above the horizontal plugged holes.

Cell A is 20 feet high by 10 feet wide and 25 feet long inside; these dimensions permit four-foot walls in the direction of Cell B and the labyrinth, two five-foot walls and five-foot thick roof. A six-inch wide concrete ledge fifteen feet above the floor extends most of the way around for supporting beams; inserts for mounting brackets, braces, etc. are cast in the ceiling and walls in the same pattern as the plug-holes. The floor slopes to a trench extending along the west side; this trench in turn slopes to a sump adjoining the partition. The floor, trench and sump are lined with lead; this covering extends 6 inches up the walls.

Cell B is much smaller than the other, being 15 feet high, 10 feet wide and 15 feet long. A six-inch ledge is located 10 feet above the floor. Inserts, floor covering and drainage are the same design as for Cell A. In the north-east corner the two cell walls and two 8-inch concrete walls, the latter beginning 42 inches above the floor, form a 3 by 4 foot flue leading to the roof opening, 3 feet square, through which the product carrier is lowered or raised. Inserts in the flue walls support guides for positioning the shipping carrier on a dolly. Stainless steel strips, sealed to the lead covering the floor, form a track for moving carrier and dolly back and forth in the cell.

The cells were equipped as follows:
Dissolver, A1 - for metal dissolving with HNO_3 capacity 165 gallons.

Condenser, A2 - for dissolver off gas. Reactor, A3 - for oxidizing NO fumes in the off gas to facilitate neutralization.
Caustic Scrubber, A4 - for neutralizing acid off-gases.
Neutralizer, A5 - for neutralizing acid metal waste; 450 gallon capacity.
Holdup Tank, A6 - for storage of neutralized metal waste until tank farm is ready to receive it, capacity 850 gallons.
Precipitator #1, A9 - for H_2SO_4 precipitation, settling, and decanting; 117 gallon capacity.
Catch Tank, A8 - for holding decantate from A9 pending analysis for Ba loss; capacity 130 gallons.
Precipitator Condenser, A10 - for precipitator off-gas.
Water Scrubber, A16 - for all vessel off gas except from A1 and A5.
Precipitators 2 and 3, B1 and B6 - for lead removal; capacity 11.6 gallons.
Precipitator Condensers. A12 and B18 - for B1 and B6 off gas, respectively.
Catch Tank, B3 - for holding up waste solution, capacity 58 liters.
Centrifuge Head Tank, B5 - capacity 1.0 gallon.
Centrifuge, B15 - 2200 rpm, 5-inch bowl.
Electrolytic Cell, B12 - for lead removal; capacity 6 liters.
Rotating Pipettes, B7 and B8 - for small volume solution transfer, capacity 8 liters.
Fixed pipette, B9 - for small volume transfer from B6 to B17, capacity 2.7 liters.
Storage Pot, B17 - for product hold-up for pipette transfers, capacity 2.8 liters.
Shipping Container Assembly, B19 - for evaporating product to dryness and placing it in the product carrier (36).

At the end of the four month period after the start of construction, at the time when it was hoped construction would have been completed, Building 706-D was essentially completed from an architectural standpoint. However, only about 15% of the process equipment had been installed at that date, March 20, 1945.

One month later the processing equipment was about 95% completed and installed. During this period the electrolytic cell arrived (about a month ahead of schedule) and was installed, giving facilities for carrying out either the chromate or electrolytic separation of lead. Since the electrolytic process was more favorable, it was decided that this method would be tried first.

On April 27, 1945, the Construction Group turned over the task of completing the building to Maintenance, since only a few minor installations, repairs and alterations remained. Cell A and the non-operating area had been turned over to Operations on April 2 and after inspection, testing and final approval, dummy runs were started. These runs indicated the need for some process changes, which were made. In the Technical Division Report for month Ending May 20, 1945 (CN - 2822) the following revised production process description was given:

"The process now consists of dissolving two hundred slugs in batches equivalent to fifty each in nitric acid, diluting the resulting solution to prevent precipitation or freezing of uranium salts, addition of lead and sulfuric acid to the solution to precipitate lead sulfate which carries the barium. The same lead is used over and over again, being added only to the first fifty-slug solution, and additional lead being added only to make up the solubility losses. The supernatant uranium solution is decanted from the precipitated lead sulfate. The precipitate is washed with sulfuric acid and water and the lead-barium sulfate is then converted to the carbonate with potassium carbonate solution. The cake is then dissolved in nitric acid and jetted into the electrolytic cell in Cell B, where it is electrolyzed for removal of the lead. The lead is plated out as lead oxide. After electrolysis, the practically lead-free solution is transferred to a precipitator and a barium carbonate precipitation is carried out. This completes removal of the lead and reduces the volume of the solution. The barium carbonate is then dissolved in strong nitric acid and fuming nitric acid is added to precipitate barium nitrate. The supernatant fuming nitric acid is decanted and the residual barium nitrate washed with additional fuming nitric acid, which is also decanted. This treatment removes iron. The residual barium nitrate is accompanied by one to two hundred cc of fuming nitric acid, which is evaporated to dryness, and the barium nitrate residue is then dissolved in water, transferred to another vessel for sampling, and passed to the final shipping cone where it is evaporated again to dryness".

The dummy runs which led to the adoption of the above process also showed up numerous equipment changes which were needed. The Technical Division Monthly Report (CN-2822) states that, "During this period almost every piece of equipment in the cell (B) has been rebuilt". A few of the major difficulties encountered are enumerated below:

1. The air heater for final evaporation was rebuilt four times to correct troubles caused by the fact that the steel housing around the heaters was so large that it was impossible to get the rated airflow across the heaters.

2. The fixed pipettes plugged repeatedly due to stainless steel cuttings from construction work and obstructions in the pipette lines caused by welding.

3. Dirt, scale and graphite-oil pipe thread sealer were found to be entering all Cell B tanks. To reduce this trouble stainless steel pipes were installed all the way back to the panelboards instead of just in process solution lines. Improved stainless strainers were installed in the steam lines.

4. Fuming nitric attacked the G-9 gaskets on all Cell B tanks, necessitating a change to Koroseal gaskets.

5. Rotating pipetts which originally lifted by a long screw, were found to have too much side to side movement in raising and lowering due to crookedness of the screw. Redesigned pipetts had a steel cable and reel for raising and lowering them.

6. It was found that the current necessary in the electrolytic cell was considerably greater than was anticipated, necessitating redesign of the electrical system to give a maximum of 50 amperes instead of 25.

7. The final product evaporation assembly required several days of adjustment to bring the various parts into proper alignment.

8. It was found that considerable steam condensation took place in the lines to the small Cell B jets, necessitating the installation of traps just ahead of the jets.

These changes in process and equipment were completed by the middle of May, 1945, and production runs were started at once.

The total cost for the construction and equipping of Building 706-D was $413,000 - as compared to the original estimate of $175,000. The manpower used on this project totalled about 1500 man months. Two thousand cubic yards of concrete were poured, a quarter of a million board feet of lumber were used; over five and a half miles of pipe and about a mile and a quarter of tubing were installed.

The time from the start of construction until the start of production operations was six months. It was possible to accomplish the job within half a year mainly because chemical development, equipment design and testing, and building construction were allowed to proceed in parallel fashion. The availability of panel board instruments used in the 200 Area and top priorities for materials were other large factors in completing the job in a few months (36).

706-D Production Runs

After numerous test runs using inactive slugs, a 200-slug "dress rehearsal" test run was started about May 15, 1945. Slug loading consisted of

199 inactive slugs and one "hot" slug. This run was processed through metathesis in Cell A with a yield of 93.4% to that point. Five tracer runs through Cell B gave an average yield of 76%.

The first production run was started on May 26, 1945, with the loading of 208 slugs into the A1 dissolver. Coating removal and the dissolving of four 50-slug batches were carried out with no difficulty. A total of 1,425 curies of Ba were dissolved. The lead and barium sulfate extraction was carried out on each batch separately, reusing the $PbSO_4$ carrier from the first batch. The supernatant UNH was decanted to a 0.7-inch heel (1000 ml) on all batches except batch C, when manometer troubles necessitated a "blind" decantation which was intentionally stopped early, leaving a 3½-gallon heel. The combined precipitates were given one dilute sulfuric acid and three water washes. Up to this point operations were satisfactory.

Two metatheses were made without difficulty. The first metathesis was decanted to B1 with jet A9-B1DD, which did not appear to operate properly, and the loss was 60 curies. The second metathesis was transferred with the A9-B1DC jet which gave an apparent loss of only 10 curies. An acid wash of tank A9 yielded 50 curies which were transferred directly to the B-12 electrolysis vessel. The metathesis solution in tank B1 was acidified with HNO_3 and transferred to B-12 where the electrolysis was carried out satisfactorily. After electrolysis, the lead content was zero and one-half gram of inactive Ba carrier was added. The volume reduction (evaporation) and fuming HNO_3 precipitations gave no trouble.

The heel of the last fuming HNO_3 decantation was evaporated to dryness and the $Ba(NO_3)_2$ product was taken up in water. The transfer of this solution to the product carrier through the B-17 vessel failed to work the first time, making it necessary to blow the solution back to B-6. A second attempt was successful, however, and the final product was evaporated to dryness in the product carrier without difficulty. Analysis of a product sample taken from B-17 just prior to final evaporation showed only 250 curies of Ba and about four times the tolerable amount of lead and iron. These results came as quite a shock to the Operations Group, since previous analytical results had indicated relatively low losses and complete lead removal. A satisfactory explanation of the losses or of the source of the contamination was never found.

Because of the low yield and the gross contamination, this product was not shipped. It was dissolved out of the product carrier with water and poured down a hot drain in the Semi-Works (37).

Following the failure of the first run in the final purification stages, the personnel of 706-C Building were requested to put their equipment in shape to receive the product from Run #2 and purify it, if this were found to be necessary. This was done and at the end of the second run, 706-D personnel were standing by to process the Ba through further pruification steps in their own equipment. This proved to be unnecessary, however,

and is of note only in that it shows an example of the extreme pressure which was being applied from all sides to speed up production of Ba^{140}. Also, this marks the last time that 706-C Building has been concerned in the production of Ba^{140}.

The second run was started on June 5, 1945, with the initial loading of 208 slugs; 624 slugs were loaded in all. A total of 2,063 curies of Ba were dissolved. No major operational difficulties were encountered in this run and the final product yield was 1,180 curies. The lead content of the product was above specifications again, but Los Alamos agreed to accept it.

During these first two runs, much difficulty with the A-16 off-gas blower system was experienced. After the completion of the second run the four-inch, black-iron off-gas line to the fan house was removed and replaced with stainless steel. A stainless steel fan was installed to replace the badly corroded one which had been in use. An ammonia system was installed to bleed ammonia into the off-gas line after the scrubber in order to insure complete neutralization of the off-gases.

The need for forced ventilation in the decontamination room became apparent during the first two runs. Consequently, an air and fume exhaust system was installed in the latter part of June.

The product from Run #2 was found by Los Alamos to be unstable and unsatisfactory for other reasons. Consequently, it was decided to change the product specifications from $Ba(NO_3)_2$ to $BaCl_2$. This change necessitated installation of additional equipment in Cell B to handle the conversion from $Ba(NO_3)_2$ to $BaCl_2$. Glass equipment was installed in Cell B for this purpose because of the known corrosiveness of HCl on stainless steel.

Both cells were decontaminated for equipment changes. In Cell A, a cyclone separator in the off-gas line, a valve in the A-16 scrubber drain line, and a new jet from the ventilating duct to the sump were installed. Duplicate sets of glass equipment, consisting of a Stang reactor and a pressure-vacuum transfer vessel in each set, were installed in Cell B with suitable valves and lines for their operation. One Stang reactor, B 21, was installed in place of tank B-17 which was removed; the other was installed just west of tank B1. Five Hastelloy C valves were used in conjunction with each apparatus to direct flow from the transfer vessels. These valves were operated by means of extension handles through the west wall, first floor, of Cell B. Optical instruments were installed so each setup could be viewed.

After these alterations were completed, the third run was started. The procedure for this run was varied slightly from that previously used in that the run was to be processed in three batches through the electrolysis step instead of being accumulated in the A9 precipitator as had been done in the first two runs. The run progressed smoothly up to the electrolysis step. At that point, when the first batch was being transferred from the

electrolysis cell to B6, the entire solution disappeared. After considerable investigation, it was determined that the solution had gone into the off-gas system. The new ventilating fan, which had been installed just prior to this run, was so powerful that it had sucked the solution out of the off-gas line during the process of transferring. Approximately 1,000 curies were lost in this way.

The other two batches were carried successfully to completion by keeping the off-gas valve closed during the B12-B6 transfer. During the evaporation of the last two batches which had been combined in B6, KNO_3 crystallized out of the concentrated solution, plugging the lines. This necessitated dilution to dissolve the KNO_3 and a fuming HNO_3 precipitation to remove KNO_3. The waste losses from this step were high - 400 curies.

The product solution transferred to the product carrier contained only about 800 curies, so the fuming HNO_3 waste was reprocessed to yield 300 curies which was added to the other 800 curies. The product contained only iron in amounts above the specifications and was shipped on July 23, 1945.

At the conclusion of this run five major repair jobs were necessary:

1. The elimination of jet discharge manifolds

2. The installation of a new B19 head heater

3. Repairs to the extension handles on valves to the glass equipment

4. Replacement of one glass transfer vessel which had broken

5. Shielding the exhaust blowers at the fan house

After extensive decontamination, these jobs were accomplished before August 6, 1945, when the fourth run started.

For the fourth run 864 slugs were loaded and processed through the volume reduction in B6 without incident. However, in transferring the solution from B6 to the glass reactor B21, the funnel to B21 plugged after only about 80 ml of solution had been added. The remaining solution was diverted to the funnel to B20 (a duplicate of B21) which also plugged after only 20 ml had been added. In order to recover the solution and continue operations, the solution in the funnels and transfer pipette were sucked up into a shielded glass vessel which was set up on top of the cell block and then discharged directly into the open top of B21 reactor through a Tygon tube lowered into the funnels and into B21 through an open roof plug. To decrease the swinging of the Tygon tube in the air sweep through the cell, a section of stainless steel pipe was fastened to its lower end. After the transfer of solu-

tion and B6 washes into B21, the stainless pipe attached to the Tygon tube struck and cracked the sintered glass disc in B21; so the solution was transferred to the duplicate vessel B20, where normal operations were resumed and completed without difficulty. The shipment consisted of 2100 curies.

Runs No. 5 and 6, each with about 2000 curies of product requested, were scheduled for completion on September 11 and September 16, respectively. Run 5, after electrolysis, analyzed 2000 curies. To compensate for anticipated losses in the final purification step, 400 curies of recovered waste from run #4 was added to run #5 in B6. The first fuming HNO_3 precipitation was made in B20 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 filtration had apparently stopped completely. Since the alternate glass equipment had been broken in the previous run, the run #5 product in B-20 was washed back into the Cell A precipitator, A9, where it was combined with an approximately equal amount of product from the series of extractions that had been intended for run #6. This combined solution was metathesized, electrolyzed for lead removal, and further purified in the stainless steel precipitator B6 by three fuming HNO_3 precipitations, with separation of the $Ba(NO_3)_2$ by settling and decanting. The final $Ba(NO_3)_2$ cake was dissolved in water and evaporated in the product shipping cone without difficulty. The final product assay was 2000 curies.

After run #5 a special preparation of 1,000 curies of La^{140} was prepared for Health Physics from the fuming HNO_3 waste.

At this time both cells were decontaminated for extensive repair work and alterations. In Cell A a new catch tank, A11, was installed to allow the recovery of high waste losses and to give more flexible operations. Other changes and repairs included the replacement of the cooling coils in the dissolver off-gas condenser, the installation of a larger A16 scrubber drain line, the introduction of condensate pots on the steam side of many jets in an effort to reduce dilution and the installation of an iodine collection pot on the A3-A4 line to collect fission iodine from the dissolver off-gas.

In Cell B practically all the black iron tank supports were so badly corroded as to require replacement. A stationary pipette, B2, was installed on tank B1 to make B1 a duplicate of B6. A new vessel, B17, of Hastelloy C was installed replacing the one removed after run #2.

Special lead cubicles, stainless steel lined, were built outside the west Cell B wall to hold the B20 and B21 glass equipment. The purpose of this installation was to enable the replacement or repair of glass equipment without a complete Cell B decontamination. Four cubicles were built to house 1) the B20 glassware, 2) the B21 glassware, 3) the Hastelloy C valves

for lines to and from both sets of glass equipment, and 4) a glas "crud" filter which would be used in filtering the solution during the transfer from Cell A to Cell B.

A movable funnel rack was installed to allow transfer from B1, B9, or B11 into either set of glass equipment or to B19. Much additional work in the form of repairs was carried out while the Cells A and B were accessible.

The work inside the cells and the construction and equipping of the external cubicles were completed in time for run #6 to be started on November 23, 1945. The first 15 slugs loaded for this run stuck in the slug chute to the dissolver on top of an Argonne slug (larger than normal "X" slugs) which apparently had gotten lodged there during dummy test runs just completed. By filling the dissolver almost full with 60% HNO_3 and heating it to 95° C. for five minutes, the slugs were dislodged. After removal of these slugs, operations proceeded normally until the fifth extraction in tank A9, when the mechanical A9 agitator froze. Maintenance succeeded in freeing the agitator shaft, but thereafter it ran noisily; so the decision was made to use air sparging instead of mechanical agitation for the rest of the run.

Unfortunately, the use of air sparging as a means of agitation led to unexpected operational difficulties. Very high losses (up to 1,000 curies) were experienced and the air on the third level became highly contaminated. The run was continued, however, recovering the high losses in the new tank, A11. An attempt was made to return the recovered material from A11 to A9. This was unsuccessful, as only 500 curies showed up after the transfer from A9 to Cell B.

A 40% K_2CO_3 rinse of A8 and A11 recovered at least some of the product. This was returned to A9 where it was re-extracted with $PbSO_4$. Meanwhile 200 more slugs were added to the dissolver to replace some of the losses.

Without other major difficulties the run was completed on December 7, 1945, and the final product assay was 1,200 curies.

The report from Los Alamos indicated that only 400 curies has been received by them. It was finally decided that an incomplete transfer from B17 (where the last sample was taken) to B19 must have been responsible for the loss.

After the completion of run #6, temporary repairs to the A9 agitator were made so run #7 could be started.

Run #7 was completed on December 20, 1945, with the shipment of 2,100 curies. To quote the weekly report for December 22, 1945, "No trouble was encountered during this run which was phenomenal. All scheduled times were met and the run took seven days" (38).

Run #8 was started on January 2, 1946, and completed on January 13, 1946, with only two unusual incidents. For some reason not clearly understood, about half the La^{140} was extracted by the HCl-ether instead of coming out in the fuming HNO_3 waste. At the time it was believed that the high gamma radiation from La^{140} caused polymerization of the ether, although later experience has indicated that this probably did not happen, and considerable difficulty was experienced in transferring the solution. Also, it was necessary to put the product through the final purification steps twice as Pb was well above (1500 mg) tolerance after the first treatment. Despite these operational difficulties 2,570 curies were shipped on January 13, 1946.

After this run, Cell A was decontaminated and permanent repairs to the A-9 agitator were made. Also, condensate pots were installed on all steam jet lines and steam spargers. The Cell A sump was chipped out and repoured. New lead sump lining was installed. A new line from the A-4 Scrubber to the northeast corner of 706-D Building was installed to replace a leaky line running under the concrete floor. It was decided that it would be easier to put in a new line than to dig up the floor to repair the old line.

This extensive maintenance work was completed the first week in March, 1946, and run #9 was started on March 4, 1946. This run was processed to completion with no difficulty and was felt to be the most successful to date. Two thousand curies were shipped.

Following run #9 four more dissolvings were made and carried through the regular process; the wastes from run #9 were added and a total of 1,275 curies was delivered to the Health Physics Division for special tests.

The lead cubicles and Cell B were decontaminated after this special run to allow the replacement of all Tygon lines. In Cell B Tygon lines from the movable funnel rack to the glassware were replaced with stainless steel pipe. In the cubicles new Tygon was installed.

The completion of run #10 on April 17, 1946, with the shipment of 1,800 curies marked what was felt to be the most successful run to that time.

In the processing of run #11 some difficulty was encountered with the freezing of UNH in tank A9 and with high waste losses. These problems were solved satisfactorily, however, and on May 15, 1946, 2,000 curies were shipped.

Run #12 was completed on June 17, 1946, without major difficulty and gave the best yield (73%) ever obtained. The product was the lightest in color ever produced and assayed 2,500 curies.

After the completion of run #12, Cell B was decontaminated for repairs. The B-12 electrolysis vessel was replaced with a new one; a solution addition line to B-17 was installed; a line from B-24 to an outside blister (J 1)

for HCL-ether waste removal was installed and other minor repairs were made.

Run #12 marked the first anniversary of the first successful 706-D run. At this point it seems fitting to review the first year's operation in brief form. The following table shows a brief resumé of production runs 1-12:

RUN NO.	CURIES SHIPPED	REMARKS
1	250	Not shipped - very low yield; gross contamination of product
2	1,230	$Ba(NO_3)_2$ product. First successful 706-D run
3	1,150	$BaCl_2$ product. New glassware used first time. 1,000 curies went up off-gas line
4	2,100	Funnels to glassware plugged - broke B21 filter disc
5	2,000	B20 disc plugged - unable to use glassware. $Ba(NO_3)_2$ shipped
6	1,500	Slugs stuck in chute. B21 to B19 transfer faulty. Showed only 400 curies at Los Alamos
7	2,100	Best run to date. No operational difficulties
8	2,570	Ether polymerized. Two HCl-ether extractions necessary to remove Pb.
9	2,040	Best run to date
10	1,800	No major difficulties - good run
11	2,000	Extraction Slurry froze - good run
12	2,500	Best run to date

During this year of operations 10,610 slugs were charged into the dissolver, 37,965 curies of Ba^{140} were dissolved, and 19,890 curies were delivered to Los Alamos (39).

The major equipment changes which took place in this year were summarized by W. A. Rodger in the report MonT-155 as follows:

DATE	CHANGE	REASON	RESULT
5-45	1) A16 ventilating fan replaced	Original black iron unit dissolved	Replacement also failed
	2) Slug carrier altered	Beams of activity 7R came out	Satisfactory
	3) Positive displacement blower installed in A16 system	Further attempt to repair item 1)	Also failed
6-45	4) Cyclone separator and NH_3 bleed installed in A-16 system	To cut down corrosive action in A-16 blower	Reasonably successful. Still some spray
	5) Filters on all air and steam boilers	Rust and scale getting through into tanks	Still some scale from black iron connections at PB's
	6) A16 off-gas system changed from black iron to stainless steel and permanent drains installed	To reduce corrosion failures and to make draining of condensate easier	No further trouble experienced
	7) Ventilation added to equipment decontamination room	Fumes made work in the room impossible at times	OK
	8) Glass equipment similar to 706-C's installed in Cell B in place of B 17 and next to B1	To allow preparation of $BaCl_2$ product	Produced $BaCl_2$ with difficulty Had little flexibility and was hard to repair
7-45	9) Cell B sump jets repiped to A6	To remove manifolded discharges	OK
8-45	10) Manifolded discharge lines in Cell B were removed	Solution backed up	OK
	11) B19 head heater replaced with steam and changed from stainless to Hastelloy	Electric heater failed and corrosion of stainless was noted	OK

DATE		CHANGE	REASON	RESULT
	12)	Sampler heads were replaced with new slip-on type	Original units stuck	OK
9-45 11-45	13)	A11, a spare catch tank backing up A8 was installed and piped up to A1,A5,A6,A8 and A9	To allow recovery of high waste losses and more flexible operation	Very satisfactor Has saved many hours of recover time
	14)	New coils put in A2	Old ones leaked	OK
9-45 11-45	15)	Iodine condensate collection pot installed in A3-A4 line	To allow collection of fission iodine	Highest potency collected: 80 mc/liter
	16)	A16 drain increased from 1" IPS to 2" IPS	To decrease scrubber flooding	OK
	17)	Steam pots were introduced on steam side of several jets	To decrease dilution and prevent backup	Higher steam pre sure required. Dilution less. N instance of back up. Wears out lead floor
	18)	Numerous Cell B vessel supports replaced	Black iron corroded during decontamination	Mere repair
	19)	Stationary pipette B2 installed on tank B1	To make B1 equivalent to B6	Never had to use yet
	20)	I3 replaced with jacketed equivalent	To allow better decontamination	OK
	21)	Suction lines to B9 and B11 increased from 0.105" ID tubing to 1/8" IPS	To reduce plugging	OK
	22)	Polystyrene under cover gasket removed - B12	Too hard to decontaminate	OK
	23)	Thermocouple well from B12 was removed and replaced with a piece of Pt wire	Invalidated upper 2000 cc of volume	OK

DATE		CHANGE	REASON	RESULT
	24)	New B17 of Hastelloy C, Tygon connections to glassware installed	To give less ionic contamination of product	OK except Ni; giv some trouble
	25)	Installed B24, a new Hastelloy C vessel	Auxiliary vessel	OK
	26)	Crud removal disc (removable) installed in external lead cubicle	To remove crud from solution on transfer to Cell B	No plugging trouble in Cell since installation
	27)	Screens put in golden horseshoe funnels	To prevent plugging	OK
	28)	Movable funnel rack to receive discharges of B2, B9 and B11 installed	To allow transfer to either set of glassware or B19	OK
	29)	Wherever possible black iron was replaced with stainless	Corroded	OK
9-45 11-45	30)	Off-gas gaskets removed	Collected activity	OK
	31)	Two sets of glassware of new design installed in lead cubicles external to Cell B. Valves all collected in a separate cubicle also external. Doors allow relatively easy access.	To allow replacement of glassware without decontaminating Cell B	Quite satisfacto Doors not big enough. Necessary to enter Cell B Occasionally to replace Tygon
1-46 3-46	32)	New bearings and a slip bearing at the tie ring were installed on the A9 agitator	Old bearings failed. Tie bearing was added for safety	OK
	33)	New Jet A9-A8DA (CL#1) was installed decanting to a 6" heel	To reduce losses and time cycle	Poorly. The jet installed was a lemon
	34)	Two sump jets were rerouted to W-11	To allow faster removal of cell water	OK

DATE	CHANGE	REASON	RESULT
	35) New off-gas line A4-205 installed	Leaks in old one could not be repaired	OK
	36) PB2 instrument lines replaced with stainless and all 3-line assemblies were made four	To reduce numerous instrument failures	OK
	37) Sampler doors were changed from sliding to swinging type	Sliding doors stuck and failed	OK
	38) Traps were installed in hot sink drains	Hot gases backed up	OK
	39) Tygon lines in Cell B were replaced with stainless and Hastelloy wherever possible	To reduce periodic maintenance in Cell B	Ni gives troubl in final produc
	40) Locker room facilities doubled.	Original too small	OK

These design changes coupled with normal improvements in operating techniques had put the process on what was felt to be a routine basis. Many problems which arose during the first year of operations seemed almost insurmountable; but eventually they were solved in a satisfactory manner and the run procedure which had been developed by June, 1946, has been used substantially unchanged ever since.

The thirteenth run was processed to completion on August 20, 1946. The final product analysis showed 2,900 curies; but radiation readings indicated only 2,000 curies. It was suspected that the 900-curie discrepancy might be due to incomplete transfer of the product solution from the sampling vessel to the shipping container. After the completion of this run, Cell A was decontaminated for the installation of an off-gas line from the A-16 scrubber to the 205 stack. Prior to this, the A-16 fan had discharged into the 30-foot fan house stack. Because of the amount of activity which was suspected of being carried out in the A-16 system, it was felt that the air contamination hazard would be reduced by letting the A-16 fan discharge into the 205 stack. Two underground 4-inch lines were installed between the fan house and the 205 Building, where they were joined in a "Y" and tapped into the off-gas duct leading to the stack. Only one 4-inch line was to be used in normal operations. The other was a spare - and suitable valves for changing from one line to the other were installed in valve pits at the fan house and at the southeast corner of 205 Building.

During the period of this shutdown much of the effort of the supervisory personnel was devoted to the preparation of an operating manual and to carrying out a training program. Also, during the period of the shutdown it was decided to decontaminate Cell B to make an investigation (at the request of Los Alamos) to determine the source of the Ni product contamination. Since Cell B was being decontaminated, it was decided to replace all Tygon lines in Cell B and the cubicles. All of this work was completed by the end of November, 1946, and the equipment was tested and ready for operation the first week in December, 1946. The results of the Ni contamination investigation were inconclusive.

The successful processing of Run #14 resulted in the preparation and shipment of 1,400 curies of Ba^{140}. Normal operations prevailed throughout this run; in fact, it was felt that this was the most efficient run that had been made to date. The product was shipped on December 10, 1946.

Run #14 was made by 706-D personnel without supervision from the Technical Division as a trial in preparation for the transfer of building 706-D to the Operations Division. After the successful completion of this run, the Operations Division took full charge of 706-D and the barium-lanthanum process.

Run #15 resulted in failure on January 28, 1947. This failure was attributed to several unfortunate operational difficulties: 1) the jets from A9 to Cell B were partially plugged. This caused large dilution of the product solution going to the electrolysis vessel and also made it impossible to rinse all the product out of A9; 2) low product analysis (850 curies) and high lead content of the B17 sample caused the decision to be made to reprocess the product and process an additional 300 slugs; 3) during the processing A9 mechanical agitator stuck; 4) during the second electrolysis a short circuit developed in B-12; 5) this, of course, resulted in very poor lead removal and the second B6 sample showed 1,667 curies and 19 grams of lead; 6) during the transfer from B6 back to B12 for further lead removal the crud filter plugged; 7) after the short in B12 was repaired and a third electrolysis was carried out, the final B6 sample showed only 750 curies, indicating a 1,000-curie loss in the crud filter. To quote the 706-D weekly report for January 29, 1947: "Hopes for product shipment were abandoned at this point".

Needless to say, after this run, Cell B was decontaminated for repairs. At the same time the A16 blower was also decontaminated for repairs, having broken down during run #15.

B12 was removed and rewired to eliminate the short circuit. The fan was repaired and reinstalled.

Run #15A was completed and shipped on March 4, 1947, without unusual difficulty. The final product assay was 1,900 curies.

Only 840 curies were shipped after run #16 on March 26, 1947. High decantation losses in A9 were responsible for the low yield.

The yield for run #17 was as good as that for run #16 was poor. On April 30, 1947, 2,478 curies were shipped after a trouble-free run.

After run #17, Cell A was decontaminated and tank A9 was removed for testing of the suction lines and jets. At the same time all necessary miscellaneous repairs in Cell A were made.

Run #18 was started on June 9, 1947, after the completion of the Cell A work and was successfully completed on June 18, 1947, with the shipment of 2,330 curies to Los Alamos. On June 20, 1947, a new concrete duct to replace the old metal cell ventilation duct was completed and put into use. This was expected to reduce considerably the radiation on the northeast side of the building.

No major difficulties were encountered in the processing of run #19 which was shipped on July 22, 1947. The final product assay was 1,800 curies.

The slugs dissolved for run #20 were found to have such a low Ba^{140} assay that extra slug loadings and dissolvings were necessary. On August 19, 1947, the final product, assaying 2,700 curies, was shipped after a successful run.

The transfer of run #21 from tank A 9 to Cell B was delayed by the failure of the jet to work. When the jet finally did work, the product transferred to Cell B assayed only 300 curies. An extensive search failed to locate the missing 2,500 curies (it seems likely, on the basis of later developments that the loss was due to holdup in a solution transfer line). More slugs were loaded and dissolved; all wastes were reclaimed and the second processing went very smoothly to completion. 2,600 curies were shipped on October 20. After run #21 a duplicate, spare A 16 fan was installed as a standby in case of failure of the other fan.

During the processing of run #22 an operating error occurred which caused a considerable product loss and necessitated the loading and processing of additional slugs. From this point on the progress of the run was generally unsatisfactory with no major difficulties encountered. Finally 1,100 curies were shipped on November 22, 1947. Almost immediately after the shipment was made, a check of the tanks (looking for lost curies) showed 1,300 curies still in tank A9. This was processed to completion with the addition of reclaimed wastes, and 1,320 curies were shipped on November 27, as run 22A. During this run it became apparent that the A9 agitator was broken; so after the run, Cell A was decontaminated for repairs. While Cell A was accessible, a new fixed pipette, A-13 was installed for the transfer from A 9 to Cell B.

Upon examination, the slinger ring of the A 9 agitator was found to have dropped 6 inches, cutting through three jet suction lines. This explained the great difficulty which had been encountered in transferring solutions from A 9 to Cell B. The jets were repaired and, as double insurance, a fixed pipette, A-13, was installed for transferring solution from tank A-9 to Cell B.

Run #23 was processed without difficulty except that a special processing was introduced to recover high waste losses from the last extraction decantation. This recovery was successful and on January 24, 1948, 2,750 curies were shipped to Los Alamos (their measurements indicated 3,690 curies). The high point of this run was the smooth and efficient operation of the new A-13 fixed pipette.

After this run, a dummy run using 12 Hanford slugs was made to determine the adaptability of the equipment to this type of slug. It was felt that Hanford slugs could be used with suitable procedure changes.

A faulty analysis of extraction wastes led to an attempt to recover high "losses" which resulted in the loss of a day's time and 200 more curies of run #24. After the run was transferred to Cell B, there was evidence of incomplete metathesis and the existence of $BaSO_4$ in the electrolysis vessel. This required that another metathesis be performed; so the product and wastes were collected in A-9 while more dissolvings were started to make up decay losses. The run was then processed to completion without further difficulty. Analytical results indicated a maximum of 2,750 curies shipped on March 13, 1948; however, radiation measurements indicated 3,575 curies. This discrepancy was unusual and could not be explained. Los Alamos reported that 2,920 curies were received.

Following this run, Cell B was decontaminated for the following repairs:

1. A new support for tanks B-1 and B-6
2. Repair of the product carrier dolly
3. Blanking off the B-6 overflow
4. Replacement of all Cell B tygon lines with stainless steel pipe
5. Repair of a leak in B-12 Pt liner
6. B-19 elevator overhaul and alignment

After the completion of Cell B repairs and alterations, dummy runs were made in Cell A to investigate further the feasibility of using Hanford slugs.

The processing of run #25 for the ARUU program of the AEC was completed with no unusual incident on July 17, 1948, with the delivery of 1,576 curies to the AEC. This product was delivered in three product carriers, as requested, containing 1,409 curies, 120 curies and 26 curies respectively.

Run #26 followed closely upon the completion of run #25 and was carried out with normal operation to its completion on July 23, 1948. 2,419 curies were shipped.

On September 3, 1948, run #27 was successfully completed with the shipment of 2,205 curies. No operational difficulties were encountered.

By this time a carrier had been built for transporting Hanford slugs to Oak Ridge. Plans were made to use Hanford slugs for the next run.

The Hanford slug run #28 was completed on November 21, 1948, with no operational difficulties. Only 1,380 curies were shipped, however, because of the unexpectedly low Ba^{140} content of the Hanford slugs. This low assay was apparently due to operational difficulties at Hanford which resulted in lower than normal pile flux (40).

In the latter part of 1948, it was determined that Building 706-D, during the time of a run, was a major contributor of air-borne radioactive particles. In an effort to reduce the number of particles escaping into the air through the off gas lines, air filter houses were installed on the cell ventilation duct, the A16-205 off-gas line and the A4-205 off-gas line. At the time of this writing the effectiveness of these filter installations has not been completely determined; but it is known that they substantially reduce the amount of radioactive material escaping into the air through the off gas and ventilation lines.

Hanford slugs were used again for run #29 which was shipped on January 16, 1949. High waste losses in the metathesis decantation and some product loss which is suspected to be due to product hold up on the B-21 reactor disc resulted in the low yield of 1,545 curies from this run. Again it was found that the Ba^{140} assay of the Hanford slugs was not as high as was expected.

For run #30 there were Hanford slugs and 113 Clinton slugs dissolved and extracted in three batches. When it was apparent from analyses of the dissolver samples that the Ba^{140} content of the Hanford slugs was again low, 161 additional Clinton slugs were added to the process.

Operations were normal through the metathesis step; however, at the time of the transfer to Cell B, it was noted that a complete transfer had not been obtained. At this point it was decided to split the run into two shipments. The first part was processed through the glassware with great difficulty and the resulting product was so small it was discarded.

The second portion of the run which failed to transfer to cell B was found as $BaSO_4$, indicating another incomplete metathesis. All tanks through which the product had passed were treated with metathesizing solution and the Ba^{140} collected in the extraction vessel in cell A.

Additional dissolvings consumed 311 more Clinton slugs which were processed with the Ba^{140} recovered from Cell B. Operations proceeded without major difficulties except that the glassware operation was the slowest on record. However, on February 27, 1949, the run was successfully completed with the shipment of 1,345 curies.

Run #31 got off to a poor start when the Hanford slugs stuck in the slug-loading chute. However, the slugs were discharged into the dissolver after a three-hour delay. The run proceeded to completion with very little operational difficulty. It was felt that this run demonstrated conclusively the feasibility of using Hanford slugs, since the product shipped on March 25, 1949, was the largest ever - 3,240 curies.

Following run #31 a major repair job on the cell ventilation ducts was started. The concrete duct-work in the walls and under the floor of the cells was so badly corroded that it was abandoned and a new tie-in was made to louvers installed in each labyrinth.

The following table summarizes the production runs 13-31:

RUN NO.	CURIES SHIPPED	REMARKS
13	2,000	Incomplete product transfer into shipping container, 900 curie loss
14	1,400	Normal Operations
15	- -	Complete Failure
15A	1,900	Normal Operations
16	840	High extraction losses
17	2,478	Normal Operations
18	2,300	Normal Operations
19	1,800	Normal Operations
20	2,700	Low Ba assay of slugs necessitated additional dissolvings
21	2,600	2,500 curies lost in transfer to

RUN NO.	CURIES SHIPPED	REMARKS
21 cont'd		Cell B. Additional dissolving to replace losses
22	1,100	Operating error caused high loss. Additional dissolvings necessary. Generally unsatisfactory operations.
22A	1,320	Recovered losses from run #22
23	2,750	High waste losses recovered successfully
24	2,750	Incomplete metathesis necessitated reprocessing. Additional dissolvings necessary.
25	1,576	Normal operations. Special AEC run.
26	2,419	Normal operations
27	2,205	Normal operations
28	1,380	Hanford slugs had low Ba^{140} assay. Normal operations
29	1,545	High losses in metathesis decantation and in the glassware. Also low Ba^{140} assay in Hanford slugs.
30	1,345	Incomplete metathesis, very slow glassware operations, high losses
31	3,240	Slugs stuck in loading chute. Operations normal after dissolvings started. Very successful run.

Current Status of Ba^{140} Production Operations

The most outstanding characteristic of the Ba^{140} production operations is the unpredictable nature of the process. Because of inherent uncertainties in the operating procedure, such as the decantation of wastes, it has been impossible to maintain a fixed production schedule or even to determine in advance exactly what materials will be required in the processing of a run. Erratic operation of the equipment has made it impossible to adhere strictly to a standard operating procedure; special waste recovery

steps or other emergency measures are needed so frequently that it has been impossible to leave the operation of equipment in the hands of Chemical Operators. A maximum of technical supervision has always been necessary; in fact, the glassware operations are usually carried out entirely by technical supervision.

Maintenance requirements have been great since the building was first put into operation. It is standard practice to make a "pre-run check" to determine what maintenance work is needed before the run is started. Despite these precautions, equipment failures are more nearly the rule than the exception. Fortunately, the flexibility of the equipment usually allows the run to be more or less satisfactorily completed despite equipment failures.

New cell ventilation ductwork is being installed inside the cells where the old duct has been eaten away by chemicals used in decontamination. Currently, no improvements of a permanent nature except those which are essential are being made. This policy results from the uncertainty of future plans for the 706-D Building as a permanent Ba^{140} production facility.

Future Prospects for Ba^{140} Production

Hanford slugs are to be used permanently as the starting material in 706-D as it has been demonstrated that this is feasible and much more economical.

Los Alamos has made a tentative request for eight 10,000-curie runs a year to be made starting in July, 1950. They have also indicated their interest in receiving 20,000-curie shipments at some later date.

The feasibility of Ba^{140} production at Hanford has been reviewed and the opinion was expressed in the feasibility report that such an undertaking could be accomplished only at the expense of work on the redox and other processes and at an estimated cost of four million dollars.

The present 706-D production program and facilities are being studied to determine the feasibility of continuing Ba^{140} production in this equipment until the permanent production facilities are ready. The Technical Division is to study the process and equipment now in use to determine what improvements can be made in the present process. This program is expected to last over a period of one year and will be concerned primarily with the following major problems:

1. Improvement of yields

    a) Primarily by improving the separation of solids from liquids

2. Reduction of contamination

   a) Covering all vessels to make an air tight system

3. Final product purification

   a) Choice of structural materials which will reduce contamination from vessels and lines themselves

The possibilities for future processing of Ba^{140} at Oak Ridge National Laboratory under four different plans are reviewed in the "Rala Process Study - Preliminary Report No. 1", Central Files number 49-4-38, by F. L. Culler, H. E. Goeller, and W. E. Unger. The four plans proposed are as follows:

"Plan I. Improvement of the existing processing facilities for the production of 2,200 plus curies under conditions of greater safety and reliability. Erection of new final product cubicles".

"Plan II. Alteration and expansion of the existing facilities to produce 10,000 curies under conditions of greater safety, reliability, and efficiency using existing cells and equipment. Erection of new final product cubicles".

"Plan III. Alteration to Cell A equipment and the erection of a new Cell B and final product cubicles".

"Plan IV. Erection of a new facility for processing irradiated 95% U^{235} slugs with recovery facilities for U^{235} and precautions for criticality.

"The first three plans are proposed for the interim production of Ba^{140}, the fourth, which will require rather extensive development, approaches a long range study for an entirely new plant. A fifth proposal is to develop a new Ba^{140} recovery process possibly utilizing ion exchange resins. Therefore:

"Plan V. Development of a new Ba^{140} process for use in a new processing facility at Hanford or the site of the new research reactor".

"Plan V can be carried out during the interim production period so that a new process could be incorporated in the design of new Ba^{140} facilities. It is possible that a new separations process for Ba^{140} can be developed using irradiated U^{235} as a feed material".

Summary of Ba^{140} Production Data

	Run #	Slugs Dissolved	Curies Dissolved	Curies Shipped	Yield %	Cell A Loss %	Cell B Loss %
1	5-28-45	208	1,007	250	25.	Losses not determined	
2	6- 5-45	624	2,185	1,230	54.	9.1	17.8
3	7-12-45	831	2,385	1,150	50.	8.6	40.
4	8- 6-45	1,144	2,840	2,100	67.	19.	8.
5	9- 4-45	835	4,356	2,000	46.	19.8	23.7
6	11-23-45	803	3,329	1,500	45.	31.2	34.2
7	12-14-45	924	2,760	2,100	70.7	14.9	3.6
8	1- 2-46	1,728	4,557	2,570	52.4	10.2	22.2
9	3- 3-46	894	3,556	2,040	57.5	19.	12.8
10	4- 7-46	886	3,665	1,800	49.1	29.7	5.4
11	5- 6-46	896	3,938	2,000	50.8	17.8	5.7
12	6-10-46	897	3,417	2,500	73.2	13.6	8.2
13	8-20-46	812	3,521	2,900	82.4	12.8	5.2
14	12- 9-46	564	2,540	1,423	56.0	25.1	8.7
15A	3- 4-47	1,383	2,783	1,925	69.2	18.0	6.7
16	4- 2-47	793	2,667	1,020	38.2	42.3	63.
17	4-29-47	892	3,535	2,478	74.1	15.4	8.5
18	6-18-47	912	3,452	2,300	66.8	28.4	4.7
19	7-21-47	905	3,087	1,800	58.3	22.9	5.6
20	8-19-47	1,047	3,390	2,700	79.7	22.4	4.
21	10-19-47	1,166	4,177	2,400	57.4	25.1	7.8
22	11-26-47	1,287	3,995	2,200	55.6	12.3	6.6
23	1-24-48	862	3,411	2,750	80.62	11.37	4.46
24	3-13-48	1,257	4,276	3,575	83.6	12.6	5.8
25	7-17-48	522	2,378	1,576	67.7	31.7	3.4
26	7-23-48	971	4,501	2,419	53.7	32.5	4.1
27	9- 3-48	851	3,087	2,196	71.1	18.6	7.2
28	11-21-48	*	2,619	1,380	52.7	26.1	7.7
29	1-16-49	*	3,061	1,545	50.5	24.4	9.7
30	2-27-49	(*	4,097	1,345	38.4	16.3	26.7
		(					
31	3-25-49	*	6,252	3,240	51.9	23.8	6.9
	Total		104,824	62,412			

* Hanford Slugs

Avg. Yield = 59.5%
Avg. Curies Shipped per Clinton Slug Dissolved = 2.2
Avg. Curies Shipped per Hanford Slug Dissolved = 48.0

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M. D. Whitaker

C. D. Coryell & H. A. Levy

The Hot Laboratory for the Separation of Fission Products.

1. Introduction

The need for very active preparations of individual fission product elements in connection with the program of Chemistry Section II on problems of importance to the whole project has become increasingly pressing. Some of these problems are discussed in the following section of this report. The projected preparations will involve a variety of chemical operations on materials of high radioactivity (1 to 10 C.); adequate protection of personnel will require the use of heavily shielded operation cells and necessitate the manipulation of apparatus by remote control.

Facilities for these operations which meet our requirements are not now available at the Clinton Laboratories and cannot readily be installed within any of the existing buildings. It is therefore requested that a new laboratory be erected which contains a number of concrete shielded hoods embodying devices for viewing and controlling chemical operations from the outside. Preliminary plans for the laboratory and accessory space are presented herewith. We consider it important to the Project that such facilities be made available soon, if possible by Jan. 1, 1944.

Other groups in the Research Division also have indicated pressing need for similar facilities. In particular, the Health Section under Drs. H. J. Curtis and W. E. Cohn is faced with problems entirely analogeous with our own in the preparation of highly active carrier-free solutions of fission products; and plans presented here have been developed in close collaboration with this group and include the requirements of both sections. The facilities proposed are thought to be adequate for similar operations needed by Drs. E. Shapiro, G. E. Boyd, and R. E. Stoughton.

2. Present Problems which Demand Operations on Hot Materials

There are mentioned briefly below some immediate problems facing Chemistry Section II which demand chemical operation on very active materials.

a) Decontamination Studies

Present studies of the decontamination of bismuth phosphate process precipitates are severely handicapped (1) by the lack of fission product solutions of sufficiently high activity, and (2) by the constantly recurring necessity for lengthy and difficult chemical analyses of mixtures for individual fission elements. The first of these difficulties will be overcome if materials and facilities are made available for the extraction of fission products from very active sources. The second difficulty may be greatly reduced by the preparation of very active tracer solutions of individual activities relatively free of corresponding inactive isotopes. Use of such tracer solutions will make possible, by means of direct counting, the determination of the chemical behavior during decontamination procedures of each fission element individually. As will be shown in a later section, the preparation of tracers of adequate strength will necessitate operations on exceedingly active materials.

b) Radiochemical standards for analytical procedures: development of new analytical methods.

Some of the present procedures for the determination of tracer quantities of radio-elements are subject to suspicion because, due to the lack of tracers in sufficient activity, it has not been possible to conduct test analyses upon controlled carrier-free standards. It is therefore of fundamental importance to the establishments of analytical methods that pure tracer solutions of the fission elements be prepared.

Developmental studies of new rapid radiochemical methods of analysis also will be greatly facilitates if the behavior of each fission element can be observed individually. Use of carrier-free tracer solutions will make possible this method of attack.

c) Detailed characterization of γ radiations.

Detailed studies of radiations from γ emitters require sources of high activity because of the relative insensitivity of measuring instruments to γ radiation and because of the necessity of working at small solid angle. Preparation of such sources will require the facilities of the new laboratory.

d) Identification of minor fission products.

The isolation and identification of radio-elements present in fission material in low concentration is often of great importance to the project. Thus in the decontamination of W material, in which an overall decontamination factor of 10^7 is demanded, a poorly decontaminating

element may be significant even if it contributes a fraction as low as 10^{-4} to the activity of the undecontaminated material. Laboratory studies of such minor fission products will require again highly active source materials; the operations involved in their isolation must be conducted in a special laboratory of the sort herewith proposed.

e) Special preparation for technological use.

Three special problems involving highly active preparations may be mentioned:

i. Pure γ emitters for (γ,n) reactions. Generation of mono-energetic neutrons by means of (γ,n) reactions is desirable for many special experiments. Isolation of strong sources of monochromatic γ radiation for this purpose may be carried out in the proposed laboratory.

ii. Preparations for use at site Y. Dr. E. Teller has indicated that workers at site Y may require highly active and highly pure preparations of individual fission species. Such preparations could be made in the proposed hot laboratory.

iii. β Sources for magnetic spectrographic studies. Studies of β-ray stectra by means of the magnetic spectrograph require pure sources of high activity; the isolation of suitable samples from fission material may require the use of the proposed facilities for hot operations.

3. Special Requirements for the Proposed Hot Laboratory

The requirements to be met by the proposed hot laboratory may conveniently be considered under two headings: (a) those arising from the need for operations on materials of very high activity and (b) those necessitated by the high chemical and radio-chemical purity of the preparations.

a) Requirements connected with high total activity.

It is expected that need will arise for the handling of total γ activities of the order of 10 C. As an illustration of the magnitude of radiation expected, we may consider the requirements for tracer solutions for decontamination studies. Suppose there is under investigation by means of a tracer preparation a chemical procedure which results in a factor of decontamination of 10^4. If the decontaminated sample is to give 5 x 10^3 c/m at a geometrical counting efficiency of 5%, this single experiment will require a quantity of tracer solution whose total activity is 10^9 dis/m; activity for 100 similar experiments would then total 10^{11} dis/m. If this activity is to be isolated in 25% chemical

yield from fission starting material in which the tracer element is present to the extent of 1%, a total starting activity of 4×10^{13} c/m, or nearly 20 C., is necessary. A considerable fraction of this original activity will necessarily consist of hard γ radiation.

Where decontamination must be studied by means of γ-radiations, as with the 35 d Cb^{93}, the activity requirements are automatically high because of the low measuring efficiency of γ radiation.

These considerations lead us to the following specifications for the proposed hot laboratory:

1. Shielding. Ten curies of 2 Mev γ-activity produces at a distance of one meter 90 roentgens per 8 hour day. Reduction of this exposure intensity to a safe level (under 0.1 r per day) thus requires an absorption factor of 1000. Thickness of common materials require to accomplish this reduction are the following:

Lead	13 cm.
Concrete	60 cm.
Water	150 cm.

The proposed laboratory will thus require 2 feet of concrete or 6 inches of lead shielding.

The raw material for chemical separations of fission products will be slugs taken from the pile. The activities associated with a slug operated normally are presented below.

One X slug exposed for 50 days at the center of a 1000 kw pile (40 kw/ton) omits the following number of curies of γ radiation at various periods after shutdown (see MUC-CDC-#76).

Time of cooling	γ curies/slug
0	160
15 min.	68
1 hr.	43
6 hr.	24
1 da.	15
5 da.	10
30 da.	3.7

The shielding proposed is thus adequate for operations on an intensely activated slug a few hours after removal from the pile.

ii. Remote control. The high level of activity of the material to be handled will require that many chemical operations be made completely by remote control. The operation cells of the proposed laboratory must therefore be equipped with suitable openings through which apparatus may be inspected and operations controlled from the outside.

iii. Disposal of waste. The operations are being planned on the liter scale, involving a few X slugs a week. Facilities should be provided for the disposal of 15 to 20 gallons per week of waste liquors with total γ activity up to 20 C. and β activity up to 40 C.

b) Requirements connected with the purity of preparations.

i. Chemical purity. Substantially all preparations projected for the hot laboratory must be protected from contamination by foreign materials to the same extent as are high-grade analytical reagents. This requirement necessitates the general use of glass apparatus. Use of metal engineering equipment designed for operation by remote control is ruled out by this condition, as is the use of all stainless steel vessels. For the same reason, waste liquors from engineering operations have been shown to be unsuitable as a source of fission products; our plans call for direct solution of active metal slugs from the pile and subsequent chemical operations in glass on the resulting solutions.

ii. Radiochemical purity. Tracer preparations must be highly pure with respect to other radio-elements; this condition results in low chemical yields and is one of the factors necessitating the handling of very high starting activities. The tracer solutions must also be relatively free of inactive carrier; at the very maximum, 1 to 10 mg. of material per millicurie may in some cases be tolerable, but for many preparations the requirements will be much more stringent (the specification of the Health Group in this respect are extremely stringent).

4. Proposed Procedure

Following is a brief description of the basic procedure insofar as it can be forseen at the present time.

a) Preliminary.

An active slug is to be removed from the exit canal of the pile. The aluminum jacket is to be removed mechanically if apparatus for this purpose is available. The slug is then to be placed in a platinum wire basket inside a shielded lead cart and transported to the hot laboratory.

b) Chemical.

i. Solution. The platinum basket containing the slug is placed in a glass dissolving vessel. If the aluminum jacket has not been removed previously, it is dissolved in dilute acid and the resulting solution discarded. The metal slug is then dissolved in nitric acid. The resulting nitrate solution is transferred to a special burette for volume measurement; it is then transferred to a shielded bottle for storage until needed for further experiment. Apparatus for the remote-control accomplishment of these operations has been designed and test models are being assembled.

ii. Extraction. For many purposes, removal of uranium from the nitrate solution by exhaustive extraction with ether is desirable. Apparatus for this operation, including devices for transferring solution from and to storage bottles and for concentration of the aqueous phase, has been designed and partially tested.

iii. Special Chemical Operations. Devices for carrying out by remote control many standard chemical procedures - precipitation, filtration, evaporation, distillation, etc. - are being designed.

iv. Adsorption Columns. Plans for carrying out separations by chromatographic adsorption and selective elution are under way.

5. Plans and Specifications.

Accompanying this report are preliminary plans and specifications for the proposed laboratory. Following are brief descriptions of some special features.

a) Shielded hoods.

The shielded hoods are to be built of reinforced concrete in banks of four cells each. Two banks are planned for immediate construction and space is provided for a third similar bank to be built at a future date. Each cell is 6' long by 4' deep (inside dimensions): one cell in each bank is 12' tall (to accommodate tall adsorption columsn or other extensive gravity transfer operations); the other three are 8' tall. All

walls and ceilings are 2' in thickness. A 2' x 2' passage furnishes access to each cell; closure is provided by lead doors 6" in thickness which are raised hydraulically from walls in the floor. Other special features of the structure are listed below.

i. Windows. The front and end walls of each cell are provided with tanks containing large glass windows which are to be filled with water or with zinc chloride solution; these tanks are embedded in the walls close to the top of the hood and are arranged so that escaping radiation is directed diagonally upward and away from the operators. They will provide general visibility of most of the interior of the cells. Radiation escaping through these windows will not be injurious if periods of observation are short.

Visibility of particular portions of the cells is provided by relatively small tubes with glass ends which are filled with a strongly absorbing solution such as aqueous lead perchlorate. For this purpose a number of sections of 6" pipe are to be embedded in the concrete walls. If any port is not needed for an extended time, the viewing tube may be removed and the port closed with lead plugs.

ii. Control Devices. A large number of sections of 1", 2", and 3" pipe are to be embedded in the walls and ceilings to accommodate rods, cables, tubes, wires, etc., which may be needed for control of apparatus. The roof of one cell is designed to contain a number of removable blocks in order to provide further access for control devices.

iii. Storage space for hot materials. Each cell contains at least one lead-covered channel below floor level in which hot materials may be stored.

iv. Transfer between cells. A number of sections of pipe are embedded in the interior walls; lines of glass or Saran tubing may be laid between cells by means of these openings to permit the intercellular transfer of fluid materials.

v. Interior supports for apparatus. Embedded in all interior walls, floors, and ceilings are a number of receptacles for 3/8" bolts; these will provide a flexible arrangement for support of apparatus.

vi. Waste Disposal. Each cell is provided, at the bottom of the storage channel, with a drain connected to the monitoring tanks.

vii. Ventilation. Each hood is to be provided with a fume vent of 1000 Cu. Ft. per Min. capacity. The fumes will not be radiologically dangerous.

b) Other features of the laboratory.

Surrounding the banks of shielded hoods there is proposed a laboratory containing the following auxiliary facilities:

i. Chemical benches for preparation and purification of reagents, and for "semi-hot" operations with portable shielding which cannot or need not be confined to the shielded hoods.

ii. Chemical fume hoods.

iii. A shielded counting room.

iv. A glass-blowing room.

v. A room for the wood and light metal work needed in the assembly and repair of apparatus.

November 16, 1943

Mr. Roger Williams
Post Office Box 1791
Wilmington, Delaware

Dear Roger:

Attached is a copy of a request for authorization for construction of a "hot laboratory" for chemical work at Clinton on which I should like to have your comments and, if you are in agreement with the recommendation, your approval in order that it may be submitted to Major Peterson for approval.

The memorandum is to a large extent self-explanatory and refers to the corresponding item in a memorandum that I sent to you through Mr. Squires two weeks ago. I should point out that in the meantime I have gone over the matter again very carefully, at one time having been very skeptical with regard to the reality of these needs. I myself have, however, been brought back to the position of feeling that the laboratory is really essential for the proper progress of our work for Hanford and our Health Division's program.

Our chemists see great difficulty in following the processes involved in the separation unless they have a supply of known materials whose chemical characteristics can be followed at the very low concentration levels that are characteristic of our decontamination work. This is the essential reason for needing supplies of pure radioactive materials. I have looked into the possibility of producing such materials by cyclotron methods. Though this can be done in specific cases it is very probable that we might in this way miss just those materials which may be causing trouble in the chemical procedure. The production of such artificial radioactive materials could, however, well be an adjunct to the process here recommended.

I also looked into the question of preparing these materials from the waste by-products from the separation canyon. The difficulty there is the large amount of impurities introduced by the chemical procedures involved. It will be much more economical of time and effort to take one or a few slugs and treat them separately for producing the individual materials involved.

At first I had thought that complete information with regard to the effects of intense radiations should be obtainable by using externally

supplied sources of radiation. It was pointed out to me, however, and I believe correctly, that such external radiation can be applied by x-ray tubes, cyclotrons,etc., only at certain phases of the chemical process, whereas at other phases such as during filtering, centrifuging and so on, we must rely upon radiation from the material itself if we are to be sure of what will happen.

The final consideration which convinced me of this need came from the physiologists. In order to make tests on the animals of the physiological effects of these radioactive materials, they need to know against what ones protection must be insured. This will involve preparation of sufficient concentrations to be of destructive physiological power. The investigation is needed with regard to accidental industrial exposure, protection of the neighboring public and also because of the limited work which we are asked to do with regard to the use of such materials in radioactive warfare.

I have persuaded Dr. Stone to abandon his request for the building at this time of a Xenon factory which would have been useful in testing animals against radioactive gas. The cost of the Xenon factory in money and time, seemed to me however prohibitive at this moment. If now we withdraw from him also the facility of obtaining known radioactive byproducts in sufficient quantity to do his legal experiments, he will literally have no effective method of studying the hazards involved in the use of these materials. While we must go ahead with the planning of Hanford with our present limited knowledge in this regard and thus take presumably excessive precautions with regard to safety, I believe the public would justly criticize us for negligence if we did not give the Health Division the support necessary to investigate and determine the actual magnitude of the hazards involved in the process which we are developing.

With regard to the magnitude of the plant to be built, it appears that two banks of four cells each will supply present needs with a reasonable degree of flexibility. It would appear to be probable that expansion of these facilities will be needed in the future but I do not see the need for providing for such expansion at this moment.

In the enclosed drawing I have circled in red pencil the portion of the proposed building which represents approximately what I have in mind in submitting this recommendation.

If, after going over this proposal, you doubt its validity, I would

Mr. Roger Williams November 16, 1943

be pleased if you will discuss it with me over the phone. If on the other hand it appears acceptable would you be agreeable to adding your signature to mine and transmitting the recommendation directly to Major Peterson? You might at the same time let Pardee know that it is being submitted so that the construction crew at Clinton can be prepared to receive authorization to go ahead.

Sincerely yours,

Arthur H. Compton

November 16, 1943

Major A. V. Peterson

Arthur H. Compton

Request for Authorization for the Construction of a "hot laboratory" for Chemical Work in Connection with Fission Products at Clinton Laboratories.

1. Will you please authorize the construction at Clinton Laboratories of a chemical laboratory to be used as a "hot laboratory" in carrying out certain necessary processes in connection with the chemical and biological programs which require for their prosecution quantities of pure fission products.

2. This specialized laboratory is necessary for the production of pure fission products. These are needed for both chemical and biological work involved in the Hanford program and in the limited studies which are to be carried on at General Groves' request on their possible use for offensive purposes. These needs can be listed in detail as follows:

1. Fission products in pure form are required for following through the details of the decontamination processes proposed for Hanford.

2. They are required also, in appreciable quantities, in order to identify those which may be formed in relatively low concentration, but which may nevertheless lead to serious difficulties in the decontamination processes at Hanford concentrations.

3. They are also needed so that the proposed processes may be carried through their complete cycle at the high radiation densities which will prevail at Hanford concentrations and not only at particular stages where tests can be made with external radiation sources as at present.

4. They are also necessary for physiological tests to determine:

   a. The magnitude of hazards to operating personnel.

   b. Public health hazards including the effect of radio activity in water, and

   c. Their potential value as an offensive weapon.

3. The facilities required for this work are a suitable structure to house two banks, each consisting of four properly shielded concrete cells together with the appropriate and necessary auxiliary facilities.

These latter include:

1. Four small laboratories for semi-hot operations.
2. Chemical purification laboratory.
3. General laboratory.
4. Counting room.
5. Storeroom facilities.
6. Glass blowing shop.
7. Small machine shop.
8. Mens' change room, showers and toilets.
9. Womens' change room, showers and toilets.
10. Utility room.
11. One office

This building should have a total area of approximately 5,000 square feet of floor space.

4. We would appreciate receiving your authorization for this construction as soon as is conventiently possible since it is important that construction on this unit be started immediately for it to serve its purpose in our progrem.

Sincerely yours,

Arthur H. Compton
Project Director

Roger Williams

November 23, 1943

To: A. H. Compton

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

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

The following is a summary of the factors entering into the construction of the Hot Laboratory (706-C). 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:

1. 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 10^7-fold decontamination at W.

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 γ 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 γ sources.

   e) Highly active β 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 8' and one is 4' x 6' x 12' in inner dimensions. 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 γ 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 706-A.

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 difficulty obtainable air conditioning equipment for the counter room and ether 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.

<u>Previous important communications on the laboratory</u> 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 CL 706-A - 23	C. D. Coryell & H. A. Levy to M.D. Whitaker (Clinton) The Hot Laboratory for the Separation of Fission Products	10/21/43
(4)	Plans CL-706C-1	Refer by date to Drawings of W. D. Webb and W. Taluc	10/30/43

March 16, 1944

To: M. D. Whitaker

From: Arthur H. Compton

In Re: Radio-lanthanum for Los Alamos

I have received a request from Mr. Oppenheimer via General Groves for:

(1) About 1 curie of a mixture of radio-lanthanum and radio-barium "not necessarily separated from other active materials" at as early a date as possible. This request is not one of greatest urgency but would nevertheless be appreciated by Mr. Oppenheimer.

(2) One hundred curies of radio-lanthanum together with its parent radio-barium with a delivery date about June 1st. "The active material would be in less than 10 grams of carrier and as pure as possible of other gamma activities." This request is one of major importance though as you will see it is not immediately urgent.

Will you advise me what can be done with regard to filling these requests together with an estimate as to the amount of work that it will involve? On the basis of your reply I shall let Mr. Oppenheimer know what he may expect in this regard.

March 28, 1944

R. L. Doan

C. D. Coryell & H. A. Levy

Conditions Required for Carrying Out The Lanthanum Preparation

Introduction

We have been requested to survey the possibilities for the preparation of a very hot source of high energy gamma radiation needed for operations early this summer at Site Y. In particular, a source in small volume of 100 curies of the 40h La^{140} (2.05 Mev γ) has been requested, to be preceded by a pilot run of 1 curie. The main job is a preparation on a radioactive scale never before handled, but the chemical and shielding problems are not insurmountable.

This request can be filled by early summer with raw materials currently available, and with the present plant facilities if certain requirements of equipment and manpower as discussed below can be met. We propose to isolate the 12.5d Ba^{140} from pile irradiated metal nearly at saturation with respect to this chain. The La^{140} will grow in the Ba sample in 5.5 days to a maximum activity 0.72 times that of the initial Ba activity, and eventually the La^{140} will decay with the 12.5d half-life of the Ba parent.

Starting Materials

The Ba^{140} saturation activity to be expected in the center of the pile in mid-May is 3.65 C/slug. (Estimated central power 75 watts per slug or 3 times the average; fission yield of the chain, 6%). If we have 40 days operation and 6 days cooling to the end of dissolving, the Ba activity in the dissolver will be 2.3 C/slug.

Method A. Single Batch

If 100 of these slugs are dissolved and processed in a single batch with chemical yield of 60%, 137 C of Ba^{140} will be obtained; this quantity will give the desired 100 C of La on growth. To carry out this dissolving operation in one batch will necessitate radiation reduction far in excess of that available with semiworks

shielding, and about 20 times that available at present in the 706-C banks. The volume of 20% UNH (10% U) solution obtained will be about 300 gallons. This volume of solution could not be handled in the 706-C cells.

Method B. Daily Installments

If 8 of these slugs from daily dumpings are dissolved and processed per day (with 60% chemical yield) an increment of 11 C of Ba will be collected each day, and the total Ba activity A available will be given essentially by the equation.

$$A = 11 \times 18 \left(1 - \exp(-n/18) \right)$$

where n is the number of days of continuous operation and 18d is the mean life of the Ba. The required amount (137 C) will be achieved in 1.14 mean lives or 21 days of operation. The maximum La activity per batch is 18.4 curies which can be handled in Building 706-C with minor auxiliary shielding. The volume of 20% UNH solution obtained per batch will be about 25 gallons. This volume can be handled in tanks in the 706-C cells.

Alternatives Involving Solution from the Plant Dissolver

The Ba content of normal material from the plant dissolver is much lower than that of the special material described above due to (1) shorter operating time, (2) much longer cooling time, and (3) lower average bombardment intensity. We estimate these factors to be 0.8/0.9, 0.25, and 2/3 respectively, so that solutions from the plant dissolver have only 15% as much Ba per unit volume as those from the proposed special dissolvings. Thus application of the installment procedure with plant solution would require processing 300 gal per day, a volume too large for Building 706-C. If the plant dissolver operated on younger material, undesirable uncertainties would be introduced in plant processing due to 93^{239} contamination.

It seems desirable to plan for the special dissolving in the 706-C building according to Method B, daily installments.

Proposed Chemical Process

To keep the amount of carrier within the limits imposed by Site Y it will be necessary to isolate the Ba using non-isotopic carrier such as Pb. $PbSO_4$ will be precipitated with Na_2SO_4 from 20% UNH solutions. Both Ba and Sr are carried well on $PbSO_4$; decontamination from other constituents is fairly good. The $PbSO_4$

will be filtered out and dissolved in NH_4Ac solution and the installments will be stored in a specially shielded container. The mother liquor will be neutralized and jetted to waste.

The carrier Ba will be added to the Pb solution and brought down as $BaSO_4$. It may be feasible to transfer the $BaSO_4$ directly to the final container. Otherwise the $BaSO_4$ will be metathesized to $BaCO_3$ by K_2CO_3 (or possibly directly to $Ba(NO_3)_2$ or $BaCl_2 \cdot H_2O$ by HNO_3 or HCl), dissolved in HCl, and $BaCl_2 \cdot H_2O$ brought down with concentrated HCl. This can be transferred in solution to the final container, where it will be precipitated as $BaSO_4$ and ignited to dryness.

Several alternative methods of separation and many variations of the method outlined are under consideration and test.

Shipping Problem

The container will be sealed and packed in Pb for shipment. The 100 C of 2 Mev γ rays will give 2.7×10^3 r/24h at 1 meter. We assume that this dosage will be tripled by secondary radiation produced in the shield, or that the radiation dosage at 1 ft. will be $\sim 10^5$ r/24h. Two inches of Pb effect a ten-fold reduction in dosage. One foot of Pb shielding will reduce the dosage to 0.1 r/24h at the surface.

The source will dissipate at maximum La growth about 0.4 watt of Ba β, 0.7 watt of La β, 0.2 watt of $\sim$ 0.3 Mev La soft γ, 1.2 watt of La hard γ, and possible $\sim$ 0.8 watt of Sr^{89}, Sr^{90}, and Y^{90}, β radiation.

Equipment Requirements

The preparation outlined above will require the design and construction of a dissolver which can handle 24 slugs (3 daily batches), and 30 gal. precipitation tanks for separation, waste neutralizing, and spares, together with steam jet ejectors, and agitation and filtration equipment. Provisions will have to be made for the disposal of off gases containing about 8 curies of 5.3d Xe^{133} per batch.

Manpower Requirements

The present permanent research staff of the hot laboratory group consists of Levy, Brady, Stang, Goldring, and Mehn, with some machine and glassblowing assistance. This group has the responsibility also of supervising and carrying out a large part of the other functions of the laboratory.

We consider that the Lanthanum Preparation will require the services of a chemical engineer full time for 1 month and part time until the assignment is completed because of the scale of operations and the special requirements for handling the materials until volume and mass reduction have been achieved. There will also be needed an operating crew of about 9 chemists (three shifts of two men each plus trouble shooting and supervision). This estimate will require the addition of seven new chemists to the group. These men should be brought in as soon as possible in order to be effective workers by the time isolation operations begin.

Additional Information which should be Requested from the Recipients

We would like as soon as possible answers in writing to the following questions:

1. How much γ radiation other than that of the 40h La (2.05 MEV and ~ 0.3 Mev) will be tolerated? What limit, for instance, will be set on contamination by γ radiation of intermediate energy, such as the 1.6 Mev radiations from the 77h Te-2.4h I pair or the 0.9 Mev of the 65d Zr-35d Cb pair?

2. How much β radiation other than that of the Ba-La pair will be tolerated? Will, for instance, an equal amount initially of the 55d and 31y Sr β radiations be acceptable? (The Sr-Ba separation would require more steps.)

3. What limitations are imposed on size and shape of the final container? What are the desirable specifications?

4. What are the exact specifications for the container for external handling?

5. Must the source be packed tightly in the container?

6. What are the limitations imposed on the shielding in mass, shape, and accessibility of the source?

7. What shipping arrangements will be made? Timing of the operations will depend on shipping arrangements, especially in the last steps.

8. Can a meeting be arranged between a Site Y and a Site X representative to cover the details of the above 7 questions?

9. Is the June 1 date subject to reconsideration after delivery dates on heavy equipment and arrival dates of new men have been set?

10. Will Site Y be of assistance in the procurement of difficultly obtainable equipment such as 25-12 stainless steel?

April 8, 1944

To: M. D. Whitaker

From: Arthur H. Compton

In re: Radioactive Sources for Y

I have received a message from Oppenheimer in which he confirms his earlier request which I recently passed on to you for radio barium and lanthanum. It appears that the promise of usefulness of this material has increased considerably since his first request.

He would like to have the first delivery of about 100 curies sometime between July 15 and August 1st. He would also like to have subsequent deliveries at three week intervals as suggested in Coryell's memorandum to Doan, although the precise timing is not important. For preliminary experimental work he wants a shipment of the order of one curie at an early date. It would be helpful if the material thus sent would be such as to give radiation similar to those of the 100 curie lots.

They are not concerned with any high requirement regarding freedom from other beta and gamma activities. It is my understanding that Y will fabricate the container for shipment and send it on to you. Oppenheimer will also arrange conferences between his representatives and your representatives for discussing procedures and the method of handling. May I suggest that you write Oppenheimer a letter to be sent through my office indicating with whom in your organization Oppenheimer should keep in contact on this matter.

Oppenheimer is interested also in the possibility of obtaining one curie source of the Zr-Cb pair and also of the 99 day 1.9 Mev gamma activity obtained by neutron capture in tantalum. He notes that he would like to be informed about the arrival of the curie sources but that they will prepare to use any of them effectively any time.

It is my understanding from General Groves that you and your men may communicate freely with Los Alamos regarding these matters by sending letters or teletypes through my office to Oppenheimer.

5/15/44

M. D. Whitaker

Harrison S. Brown

100 CURIES PRODUCTION JOB FOR Y

I would appreciate your checking into the following problems concerning our production job of the 100 curies of radio lanthanum for Y when you are in Chicago.

1. An improvement in the correspondence with Y on the problem is in order. A series of questions was sent to Mr. Doan on Monday, May 1. An answer was not received by me until Saturday, May 13.

2. We would like to design and fabricate the shielding for the shipment at Clinton.

3. Does Y intend to process chemically the material that we ship?

4. If they intend to process the material, will barium chloride as a precipitate be satisfactory?

5. Will aluminum interfere with their chemical processing? If not, a very simple container can be constructed that can be dissolved together with the barium chloride. This will alleviate the necessity of opening the container upon receipt.

6. In view of certain restrictions that are brought about by limitations in our experimental setup, we would prefer to design the immediate container at Clinton. Our present plans are that this container would be an aluminum cylinder 1.5 inches high and 1.25 inches in diameter with a force-fit top that cannot be removed.

7. How does Y intend to remove the container from the shielding? Do they prefer a wire arrangement or would a suction cup or electro-magnetic arrangement be satisfactory?

8. I would most strongly advise that Alvarez get together with me at Chicago well in advance of the shipping date for a final check to make sure that all experimental arrangements are satisfactory to both parties. I will not be available between May 27 and June 11.

Harrison S. Brown

INFORMATION FROM TELETYPE OF 5/12/44

By - Allison

To - Whitaker

TELETYPE ON 100 C AND 1 C JOB

Y is looking into the separation of lanthanum from barium. It would appear that the job is much easier if done there. Likewise, requirements on size and shape of the container would be considerably relaxed. A stainless steel bottle for use in shipping is being designed at Y. Attachments will be provided for remote handling. Also a lead case for protection will be constructed. Teletyping should be used in place of a Chicago conference for the present. It is hoped that a three foot string can be attached to the 1 curie job, for which the size and shape of the container is not important. No improvement is needed if the container is ten millimeters in diameter or less and spherical. The assumption is made that Clinton will provide the shield for the 1 C express shipment.

6 June 1944

Mr. M. D. Whitaker, Director,
Clinton Laboratories,
P. O. Box 1991,
Knoxville 11, Tennessee

Subject: Transmittal of Information.

Dear Sir:

A teletype has just been received from Mr. J. R. Oppenheimer stating that he will accept up to five grams of barium as chloride in first hundred curie shipments. He requests that mass be held to a minimum. Lanthanum should not exceed 10 milligrams. He will send a drawing of container as soon as available.

For the District Engineer:

Very truly yours,

EDGAR J. MURPHY,
Major, Corps of Engineers,
Assistant.

June 19, 1944

M. D. Whitaker

H. S. Brown

100 C. Lanthanum Production

The present situation exists in regard to our production schedule on the 100 C. Lanthanum job for Y. In the telephone conversation of July 3, 1944, between Coryell and Dodson, Y was asked to balance the receipt of 5 gms. of carrier on schedule or the receipt of about 2 gms. of carrier two weeks late. Dodson could not make up his mind on the phone, but the conversation indicated most strongly that 5 gms. was an absolute upper limit to what they could handle. After thinking over the situation, Y sent a teletype to the effect that they would accept 5 gms. This would indicate that timing is rather important as far as they are concerned.

By that time, however, flowsheet tests indicated that the yields to be expected in the existing process were such as to possibly run into the danger of running somewhat over 5 gms. of carrier. This, of course, would create a very grave situation. In view of this, it was decided to change over to a flowsheet which would give about 2 gms. of carrier associated with the barium, even if it should result in a two weeks delay, as predicted by Coryell in his telephone conversation of June 3, 1944. Needless to say, in spite of the flowsheet change, we are making every effort to keep within our original schedule of July 15 - August 1, 1944. However, I believe that Y should be informed of a possible two weeks delay and the reasons for that delay.

The large scale part of the extraction apparatus has been completed and the individual components have been tested. A series of dummy runs is to be made starting Tuesday, June 30, 1944. The small scale glass apparatus is now under construction and it is hoped that it will be complete and ready for dummy runs by Monday, July 5, 1944.

Harrison S. Brown

June 29, 1945

Mr. J. R. Oppenheimer
P. O. Box 1663
Santa Fe, New Mexico

Dear Mr. Oppenheimer:

CHANGES IN SPECIFICATIONS OF BARIUM LANTHANUM

I am attaching two copies of memorandum prepared by M. C. Leverett setting forth our understanding of the specifications which you want applied to the radio barium preparations which we send to you occasionally. The essential changes in your specifications are:

1. That the material supplied be chloride instead of nitrate.

2. That the iron, chromium and nickel content requirement be changed from 50 mg maximum of iron, chromium and nickel to iron 10 mg and chromium and nickel 5 mg each as maximum. A new purity requirement limiting strontium to 50 mg has also been added while the lead specification has been relaxed from 25 mg to 50 mg.

3. It is now formally requested that each shipment contains approximately 1000 curies instead of the 300 curie specification that was in effect at the time the building and equipment were designed.

Either change No. 1 or change No. 2 would necessitate the addition of another step in the process in new equipment to be added to the cell. It is believed that satisfactory plans are being carried out to put into effect these new specifications.

I want to request that all communications from your organization or any member thereof be sent in duplicate. I am assuming that you will also need duplicate copies of a lot of our communications so we will undertake to send you two copies each time.

Yours very truly,

M. D. Whitaker, Director

July 7, 1944

The District Engineer
U. S. Engineer Office
Manhattan District
P. O. Box E
Oak Ridge, Tenn.

Attention: Major E. J. Murphy

Dear Sir:

TRANSMITTAL OF INFORMATION TO Y

Will you please transmit in the usual way the following teletype to Mr. J. R. Oppenheimer, Attention: Mr. L. W. Alvarez:

"From present state of program in Ba-La preparations, August 1 seems earliest deadline we should try to meet. Delivery date August 15 less optimistic more probable

Will arrive Albuquerque 9:30 P.M , Saturday,July 8."

Very truly yours,

L. Helmholz

26 September 1944

Mr. M. D. Whitaker, Director,
Clinton Laboratories,
P. O. Box 1991,
Knoxville 11, Tennessee

Dear Sir:

The following information has been received from Mr. Oppenheimer by teletype this date for transmittal to you:

1. If it will facilitate your operations, delay of one week in next Ra-Ba shipment, which would enable shipment to leave Site X 16 October, would in this case fit in well with their operations. Earlier delivery is completely acceptable.

2. Chemical specifications should be identical with those of first shipment.

3. Their gamma measurement shows 314 curies Ra-Ba at time of your separation with estimated likely error of 20%.

4. Difficulties were encountered in milking Lanthanum. There were large amounts of brown sludge resembling ferric oxide. It is believed that this is due to radiation corrosion but this is only hypothesis. On this basis, would like to use liner. They are undertaking to furnish you a tantalum liner and will communicate with you as soon as they know the facts.

5. Material has proved very valuable to them and will continue to do so.

6. 300 curies will be very acceptable.

For the District Engineer:

Very truly yours,

EDGAR J. MURPHY
Major, Corps of Engineers,
Assistant.

SPECIFICATIONS FOR RALA SHIPMENTS

R. W. Dodson, 9-28-44

The desired properties of these shipments are:

(1) The sample should contain approximately 300 curies of Ba^{140}.

(2) The sample should be shipped as dry $BaCl_2$, the total amount of barium being one gram or less.

(3) Impurities in the sample should not exceed 5% by weight.

(4) The amount of lead in the sample should not exceed 25 milligrams.

(5) The sample is to be shipped in a container furnished by Project Y.

These specifications may be subject to change. It is realized that it is impractical to verify number (3) after the sample is evaporated in the shipping container; and number (3) therefore represents a hope rather than a specification subject to verification by the suppliers.

28 September 1944.

Mr. M. D. Whitaker, Director
Clinton Laboratories
P. O. Box 1991,
Knoxville 11, Tennessee

Dear Sir:

Mr. Oppenheimer has submitted by teletype the following information to you:

Reference is made to Memorandum from Compton to you, dated 8 April 1944.

In clarification of this point, would like to have deliveries of Ba-La at approximate three-week intervals.

This request should not affect one weeks delay in next shipment which has been discussed between Dodson and Coryell.

Deliveries should continue for at least four months.

Within the near future, they will be in better condition to make estimates concerning the ultimate program.

In view of experience on the first shipment, they would like very much to aim at 300 curies for each shipment.

These statements and summary of specifications on chemical procedures and method of shipment will be sent to you by mail within the next few days.

For the District Engineer:

Very truly yours,

EDGAR J. MURPHY,
Major, Corps of Engineers
Assistant.

September 29, 1944

Dr. M. D. Whitaker
P. O. Box E
Oak Ridge, Tennessee

Dear Dr. Whitaker:

We are sending you the enclosed two memoranda for your files. One of them gives a summary of specifications as to chemical form; the other gives a summary of schedule requirements. I think all the facts mentioned have been known to you in the past, but it may be a convenience to have them assembled in two brief statements. If there are matters not touched on which you would like clarified, please let us know.

I should again like to express my appreciation on behalf of Project Y for the work which has been done under your direction in providing us with RaLa. It is a vital service for our success.

Sincerely yours,

J. R. Oppenheimer

September 18, 1944

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 $BaCl_2$ 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 $BaCl_2$ 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 amall 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, 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 Helmholz 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 upper two are narrower, the top being approximately half way up the wall to the lowest pin. The orange iron stains do not appear greatly to exceed in intensity those noted in tests made here in collaboration with 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

October 17, 1944

Mr. J. R. Oppenheimer

Attention: Mr. R. W. Dodson

In Re: Description, Composition, and Packaging Of Special Source # 2

Dear Sirs:

We are transferring to Major E. J. Murphy for shipment special source # 2 described below.

Description

The source consists of $BaCl_2$ evaporated in the Ta-lined stainless steel cone furnished by you. Evaporation was carried out by dribbling in 60 ml of solution followed by two 25 ml washes.

The sample has a uniform light salmon color. (A similar tinge was noted previously in a dummy evaporation of $BaCl_2$ in the Ta liner.) The sample covers fairly uniformly the whole of the cone and encroaches somewhat, perhaps 1 cm, into the cylinder. The conditions for observing the distribution on the cylinder were very poor but there is no evidence for splatter. The large size of the area covered may derive from a slow evaporation rate, possibly due to poor heat conduction between cone and liner.

About 1 cm above the cone bottom we observe a very narrow darker ring and above this a slightly dark band about 5 mm wide where the precipitate seems to be a little thinner and the dark Ta shows through. In this same general region there is a very irregularly shaped dark blotch. This may be a place where the crust has flaked off from the darker Ta, or a place where the sample is thin due to impingement of the warm air stream, or it may be an impurity which has fallen in, perhaps partly coated by $BaCl_2$. (A piece of de Khotinsky cement was found on and removed from the stainless steel flange before the plug was inserted.) The blotting paper coating of the block and cart were spotted with fine pieces of rust. The cone itself was protected by a cover until time for filling, but it stood open in the cell for 6 hours; a very small amount of the rust and dirt may have gotten in.

Pale blue-grey luminescence was noticed by all observers after short dark-adaptation at a periscope fitted with a rubber eye cup. (Rather dramatic luminescence was observed in the precipitates and solutions from the HNO_3 washes transferred to the glass apparatus from the cell 4 storage bottles.)

Composition

The source contains less than 800 mg of the cation Ba as chloride, presumably in the anhydrous state. (This is the amount of carrier used; losses have not yet been evaluated.) The Ba was obtained by processing 180 select Clinton slugs of 40d irradiation and 3-5d cooling at the time of dissolving. The flow sheet on the engineering side was that transmitted to you in early September and variation A was used each time since the Ba assays of the NH_4Ac solutions were always small. The operations in the glass apparatus began with precipitations of (Pb,Ba) $(NO_3)_2$ from 5 liters accumulated HNO_3 wash, without addition to the Pb therein contained (1.0 gm/liter). $PbCl_2$ was removed twice from solutions 1 N in HCl and the first removal seemed fairly inefficient. After the volume reduction by a $Ba(NO_3)_2$ precipitation, $BaCl_2 \cdot H_2O$ was precipitated with concentrated HCl and ether and the precipitate was washed with alcohol-HCl followed by ether. Separation time for this source was 00:30 CWT, October 17th, 1944. This precipitate was dissolved in water and assayed photometrically for Pb by the dithizone micromethod with new glassware for dilution. The Pb values obtained were 32 and 55 mg in the total sample, which is within the stated requirements for the source.

We are in a state of utter confusion about the assay of the source although we anticipate considerable clarification in the next few days. The control analyses and the assays, made in another group by the standard radiochemical method, suggest that the value may be lower than that of our last preparation but may lie between 100 and 300 nominal curies (UX_2 equivalent).

Mr. R. R. Coveyou, of the Health-Physics Division, has made several measurements on the radiation level of the source in the inner assembly placed in the open air. This value, measured 11.6 hours after separation time, was 52 mr/hr at 1 meter from the center of the cylinder. This value is the average of four sets of readings taken at different angles each of which seemed precise but which spread over a range of about 15%. (We have also taken a radio autograph of this container with a 1 gm Ra source and find a number of flaws of small solid angle and several large ones; quantitative interpretation has not been made.)

The dose meter was Lauritsen electroscope calibrated against a 27 mg standard Ra source enclosed in a half mm Pt sheet. We estimate hastily from these data the assay 220 C (calibrating hard gamma radiation with a soft gamma standard), assuming that the effective shield thickness is 5.25" of Pb, that the half-thickness is 16 gm/cm^2, and that 1 C of a source emitting 2.0 MEV gamma gives a dosage of 9300 R/hr at 1 cm (Coveyou's very hasty assay of source # 1, based on similar measurements, was 300 C).

Packaging

The stainless steel cone is firmly fastened with tiny screws to the steel collar. This latter has been Ag plated on a Cu base. The same sort of gasket is used this time as last time. There is evidence for a small amount of beta contamination on the upper part of block 2.

Status of Apparatus & Process

During the course of this preparation we obtained fairly strong evidence that the over-all yield is approximately half of that we expected from laboratory and tracer runs. Approximately 60 C was picked up, for instance, from the precipitator by the first batch of UNH from dissolving the heels, and we feel that much can be done to improve yields although the interpretation depends strongly on the analytical picture.

At this early date we know of no failure within the cell 3, which contains the dissolver and waste neutralizer, although we are having more and more difficulty with sampling lines due to corrosion, failure of cement, and accumulation of very high contamination (dosages at the sampler - 300-5000 mr/hr).

In making a change today of the external filter on cell 4 (precipitator and storage bottles), a glass line was broken. This cell has not previously been decontaminated and may be the most difficult of the three that have been used. There is also a leak in one of the storage bottles and a failure in a sample line. We had already decided that a second filtration and storage assembly must be provided between the precipitator and the glass apparatus, but had hoped to install it external to the cell to avoid the necessity for extensive alteration within the cell.

Cell 1 containing the glass apparatus used a second time now presents several problems. It is impossible to withdraw samples from the waste bottles, and therefore to measure losses in fractions disposed of

in this vessel. The check valve in the magnetic valve manifold has frozen shut and it is therefore impossible to transfer any solution from any of the three transfer and storage flasks back to the reactor. This precludes for instance the possibility of making any by-product precipitate without repair or revision of the system. It also makes exceedingly difficult the removal of any waste solutions from the Ba transfer flask which leads to the final product vessel. No solution can be be removed from the Ba storage flask. Solutions can be removed from the Sr storage flask only via a line leading outside the shield.

The prospects of early operation of the operating unit are very poor because of the tedious nature of decontamination processes coupled with the limitations imposed by failure of equipment. It required 12 days to achieve decontamination of cell 1 to a level of 300-800 mr/hr and it involved the exposure of a crew of six men to tolerance dosage almost every working day. Critical adjustments made just before the cell had to be put into operation to meet the present commitment necessitated dosages of 300-400 mr for several of the skilled men. Further minor failures could have made the picture still darker under the pressure of immediate operation. The extent of repair to be done is very large.

As soon as the chemical-analytical situation clears somewhat or a better chemical assay in terms of our nominal (UX) unit is available we shall write you the details of our assay. We request that you give us the details of your assay method (Ra) and as many particulars as possible on the physical and chemical properties of the sources we have sent you.

Yours sincerely,

M. D. Whitaker, Director

Clinton Laboratories

By_____
Charles D. Coryell

Approved for Chemistry Division

By_____
Harrison S. Brown

October 21, 1944

The District Engineer
U. S. Engineer Office
Manhattan District
P. O. Box E
Oak Ridge, Tenn.

Attention: Major E. J. Murphy

Dear Sir:

TRANSMITTAL OF INFORMATION TO Y

Will you please transmit by teletype the following information to Mr. J. R. Oppenheimer at Site Y:

"We have 242 curies of unaccounted activity from the second preparation measured at separation time. We have located about 60 accumulated with 40 contained in spare dissolvings. We have not exhausted all possibilities of recoverable Ba but we probably will not understand the setup before Wednesday.

We succeeded in repairing the Cell 4 break temporarily, at least, with Tygon. Due to the stuck check valve, we cannot make a by-product precipitate in Cell 1. If worthwhile amounts of Ba are recoverable, would you accept these with a fair amount of Pb. We would like to keep the Cell 2 apparatus clean against worse threats.

Please wire exact details of your dissolving process in operations and composition of buffer and any limits you know in your purification by-product operation on Ba. Our experiments establish that Pb does not carry Ba but does carry about 30% La in a phosphate separation.

We hope to have a spare filter and storage system in parallel with present ones in Cell 4 before the next large run."

Very truly yours,

M. D. WHITAKER, DIRECTOR
CLINTON LABORATORIES

November 24, 1944

Mr. J. R. Oppenheimer

Attention: Mr. R. W. Dodson

In re: Description, Composition, and Packaging of Special Source #3

Dear Sirs:

We are transferring to Major E. J. Murphy for shipment special source # 3 described below:

Description

The preparation of the source followed the usual course. It is, however, spread over a much larger area in the container than has been the case previously. The conical portion is completely covered and a fairly uniform disposition of the solid extends five or more cm up the cylindrical walls. At a still higher level are crescents of salt from evaporation of solution droplets. It is quite probable that plug # 3 will be found to be badly contaminated on the inside.

In the conical portion the solid is quite uniform, but shows fine deposit rings. There are several dark areas near the junction of cone and cylinder where the solid has fallen off the wall. In addition there is one brown speckled area of a type never before observed here. The color is suggestive of rust, but the size and apparent mottled aspect are curious. This area may be connected with perturbations from the hot air stream.

Most of the observers thought that the color was very similar to that of preparation #2, but I felt that the sample was more nearly pure white. It might be interesting to compare this description with that given in my transmittal letter of October 17th.

Composition

The Ba was obtained by processing 180 Clinton slugs of 45d irradiation in the most favorable portion of the pile. Discharge for all material occurred November 12th. We hope that we can analyze the pile power data for physical evaluation of Ba production. The run of dissolver samples shows approximately 3.1 separation time curies (UX_2 equivalent) per slug.

All the analytical data have not been collected and critically examined. Their general reliability seems somewhat higher than before. The assay for Ba at the end of the first isolation step in glass (first (Pb,Ba) $(NO_3)_2$ precipitation) was 309 curies. At this point 800 mg of carrier Ba had been added. I estimate that further losses are about 10%. A more accurate chemical assay may be furnished later.

Following this step $PbCl_2$ was removed once to leave a total of 900 mg of Pb in solution (much more than expected). We courageously proceeded with the $BaCl_2$ precipitation and found the following assays: 100, 50, and 40 mg, the mean of the latter two probably being more significant.

Separation time was 14:30 CWT, November 23d, 1944.

Packaging

Immediately following transfer to the final product vessel there was found that 15 ml had leaked into the final vessel some 30 cm below. An effort was made to close this valve tightly. It was later found that almost all of the 60 ml charge leaked out to the cone during the period consigned to the evaporation of the first portion, and the transfer may have occurred in big glurps. This mishap accounts for the excessive distribution of the final sample.

We found on removing the shield from the cell that the radiation level at the side of block #2 was about 1.6 r/hr after 20 hrs. growth. The value at the top was approximately 10 r/hr. The men who removed the blotting paper covering got badly contaminated on hands and clothes. Subsequently we exchanged blocks #2 and #3 for clean ones (our spares). You will probably find that the exterior of the cone and the surfaces of the silvered steel collar are very badly contaminated.

The physical details of the assembly are as for the last run. The second Ta lined cone was used. We shall need more cones and collars very soon.

Status of the Process and Apparatus

In the last rehabilitation period we were unable to pin down the lost curies mentioned in the previous letter and in several phone calls. At the time of entrance to the glass apparatus the material balance of dissolver material was: 50% in the HNO_3 storage bottle, 22% in waste of

in NH_4Ac (which was not processed), and 28% unaccounted for. (The corresponding figures for preparation #2 were 46%, 10%, and 44% of 537 separation time curies.)

I am fairly certain that the lost material is contained in fission $BaSO_4$ transmitted by the glass filter as colloid, which precipitates rather rapidly in the storage bottles, presumably principally in the NH_4Ac storage. We have discharged the supernatant to waste and we are giving the bottle a preliminary 3 day leach with 50% K_2CO_3 solution. This reagent, which will probably dissolve the Pb and metathesize the Ba, will be discharged and the bottle treated with HNO_3. We might obtain in this manner as much as 50 C remaining from # 2 and 150 C from #3. It was felt unwise to hold the current sample until this could be processed because of apparatus and contamination difficulties mentioned below.

We are studying the possibilities of the metathesis of $PbSO_4$ with 2 charges of 5 liters of 1 M K_2CO_3, using the two batches of metal heel for raw material. A blank on the tank following run 5 gave only a small yield of lost Ba in that tank. It is hoped that we can remove sulfate by this carbonate crossover, and thus minimize troubles due to the colloidality of the $BaSO_4$ of the 65 mg of fission Ba encountered in a preparation.

The current view of the apparatus is dim. There is a leak in a dissolver bubbling line which probably should be repaired.

The windshield wiper agitator in the precipitator is broken and we also have no sampling line to this vessel. The cell 4 by-pass is installed but was not used because of difficulties in solution transfer. The line from the external glass filter still has a tygon repair which is probably a potential weak spot. Cell 4 has never successfully been decontaminated, in spite of some investment of time, with this goal in mind, at the conclusion of the last preparation.

At two different times during the last preparation the filter in the new reactor in cell 3 had to be unplugged, presumably because of the precipitation of nitrate in the pores. This seems to have been caused in part by a leak in the pressure system below the filter membrane, although operations are normal under vacuum cycles. We do not know where this leak occurs. 8 # of pressure sustains the liquid but does not produce bubbling agitation. Decontamination may also be difficult because of the splatter that occurred on the failure of the outlet magnetic valve of the final apparatus.

Operations went very haltingly throughout the preparation. Over nine days were required in contrast with six for the previous two preparations.

At two different times in the past three weeks we have been plagued by widespread building contamination, apparently caused by an unsuspected splatter so rapidly tracked around that the source could not be established. On the first occasion many smears of 3000 c/m were observed; on the second, Wednesday last, smears as high as 8000 we observed with maxima in the sampling area and in the sample manipulation room. There was a tremendous increase in contamination occasioned by the removal from the cell of the shield and hot container cover. The building has now been declared unsafe for operations until it is thoroughly cleaned.

Prior to this effort, all sample heads were replaced with heads of slightly improved design and sampling operations have been less hazardous than before. The exposure is still great; further efforts will therefore be made to improve the equipment and technique.

During the course of the operations readings above tolerance (several quite high and many repeats for a few of the men) were obtained in 17 cases. Judging by previous experience the high incidence may continue during cell decontamination and modification unless we are very careful.

Physical Studies

A Pb absorption curve for preparation #2 was taken on the roof of the building with an electroscope placed in the gamma searchlight beam from the open container 32' below. It indicated only that we had made all the mistakes in choice of conditions that could reasonably have been expected. The absorbers were brought up towards the electroscope from close at hand. Roof shine was tremendous, and that from the observer amounted to about 10%. Our efforts to measure semi-quantitatively the neutron output from Be and D_2O were also inadequate.

The chemists put their efforts for this preparation almost exclusively on the obtaining of a good absorption curve. The absorbers covered the range from 0 to 12" of Pb, building away from an ionization chamber connected to a FP54 amplifier. The ionization chamber was about 4' above the ground, and the sample was suspended about 20' higher up. Other measurements were taken at a greater height. The absorption curve obtained was very smooth. A preliminary analysis indicated the presence of a soft component of 4.1 g/cm^2 half-thickness,

a hard component of 15.2 g/cm^2, and a non-absorbable component (earth shine) amounting to 1.3% of the initial reading. The ratio of soft to hard component at approximately 26 hrs. growth seems to be 0.42.

We are grateful to Dodson et al. for the fine report on the physical measurements you have made, and I request further information as it develops. We shall send you more details about our own studies in the near future.

One of our 3' sweep (30°) periscopes with 3" barrel is being shipped in the same convoy with this preparation.

Yours sincerely,

M. D. WHITAKER, DIRECTOR

CLINTON LABORATORIES

By_____
Charles D. Coryell

1/25/45

M. D. Whitaker

C. D. Coryell and H. A. Levy

Description of Special Source #4.

Will you please transmit to Mr. J. R. Oppenheimer, Attention Mr. R. W. Dodson, the following information:

"The information covered in letters accompanying previous shipments is presented below:

Description: The solution was transferred through a Hastelloy C valve to the cone at a top rate of 1 cc/min. After flow sheet evaporation time 7 cc of liquid remained, so the cone was heated 1 hour more. The source is spread fairly uniformly over the conical surface and rises but little into the cylindrical portion. There are 7-8 rings to be seen by searchlight with rough color spread from the center of white, gray, very dark, and brownish-dark, with a higher area somewhat brown. 5-6 rings differing in self-luminosity can be seen. The light intensity produced by the nearly evaporated and dry sources is the greatest we have seen.

Composition: We changed considerably the chemical procedure. $PbSO_4$ from 3 batches is returned with 1 M K_2CO_3 and then dissolved by 3 M HNO_3. The solution from 5 such triples contained 57 g of Pb and 525 curies (calculated at separation time) of Ba. To this point of the preparation the chemical yield of Ba was 80% and the over-all balance was 95%.

The acid solution was brought into 2 parts to the reactor, and each precipitated after addition of 0.5 g of Ba as $Pb_3(OH)_2(CO_3)_2$. The precipitate from both was dissolved in 1.5 liters of 1 M HNO_3 and $PbCrO_4$ brought down by making 0.05 M in $K_2Cr_2O_7$. That portion of the filtrate which was stored in the Sr flask during $PbCrO_4$ dissolution was lost when a Gelva cement connection failed between the steel and glass portions of the sampling line. The remaining portion of the Ba, approximately one-third, was recovered. Its assay was 190 C. Purification was carried out by $BaCrO_4$ precipitation from carbonate solution. $Ba(NO_3)_2$ precipitation for volume reduction and the usual final precipitation of $BaCl_2 \cdot H_2O$. We have estimated that a chemical loss of 6-10% is reasonable for these three steps. The final solution of the $BaCl_2$ showed the presence of 140 C of Ba, 10 mg of Pb, and undetectable Cr (<5 mg).

The value of 160±20 nominal (UX equivalent) curies is a reasonable one for the preparation. The separation time is taken as 11:40 CWT on January 23, 1945. By mistake 0.2 cc of 1 M HNO_3 was added to the final solution before evaporation.

Packaging: Packaging was carried out in the standard procedure. Contamination of Block 2 or Block 3 was not found. Radiation levels at the side on Block 2 amount to 1000 mr/hr and at the top to 100 mr/hr 12 hours after separation time. We have news that two cones arrived today. We request any more Pb blocks available.

Operability of Equipment: Cell 3 seems to be O.K. The mechanicl agitation in the precipitation tank of Cell 4 is damaged but the equipment seems operable. The operation of the Cell 4 substitute is excellent except for activity around sample heads. Cell 2 is in excellent shape except for the leak in the Sr flask which we do not need to use. Cell 1 is also operable, but since no changes have been made since the conclusion of preparation 3, it seems less promising than does Cell 2. We plan to start preparation 5 on January 26th.

February 2, 1945

Mr. J. R. Oppenheimer

Attention: Mr. R. W. Dodson

In re: Description, Composition, and Packaging of Special Source #5

Dear Sirs:

We are transferring to Major E. J. Murphy for shipment special source #5 described below:

Description

Preparation of the source followed, except for some details, the procedure used for #4. We will send you later, if you wish, a complete flow-sheet.

The distribution of the material in the cone is somewhat different than in previous cases. There appears to be a crust of solid in the bottom of the conical portion. In color it is a rather dirty brown, definitely darker than any previous preparation we have seen. The reason for this color we have not yet discovered. There is some material in the cylindrical portion of the container, though this is a small fraction of the total. In spite of this, plug 3, when removed from the shield after it was initially put in place, did not appear to be badly contaminated.

Composition

The final analyses were made on the $BaCl_2$ solution just before evaporation. They disclosed the following information:

The Ba assay is 478 $\pm$ 40 curies at separation time (1600 CWT, 1/31/45). Positive analyses were obtained for both Pb and Cr but the sensitivity is such that no precise figures can be given. Pb is reported as less than 23 mg and Cr as less than 17.

The reliability of the Ba assay is believed to be as great as in any previous preparation. The material balance up to the next to last analysis was very good. Approximately 70-100 curies, however,

are unaccounted for between the next to last and the last analyses. The two waste fractions here involved (the filtrates from the $BaCrO_4$ and the $BaCl_2$ precipitates) have not as yet been assayed, and it is possible that the missing material was lost in one of these. This question will be answered as soon as these solutions can be analyzed.

Chemical Ba in the sample should amount to about 800 mg.

Packaging

The Ta cone which arrived here on January 26 was used. As in preparation 4, the final solution was discharged to the shipping container through a "Hastelloy C" valve. When the cone was raised into position prior to the start of the evaporation, the glass delivery tube leading from the Hastelloy valve to the evaporation outfit was broken. It was necessary to install by means of strings and tongs a substitute delivery line of glass and tygon. This was accomplished in the course of 3-4 hours and at the expense of considerable radiation exposure. Evaporation otherwise proceeded normally. At the end of the evaporation a second mishap occurred. When the cone was lowered, the glass hood which carries vapors away from the evaporation area, whose support had been weakened when the delivery tube broke, fell into the shipping container on top of the sample. This was removed with the aid of a special pair of tongs and fortunately was found to have carried not more than 1-2 curies of Ba with it.

The Pb block #2 appears not to be badly contaminated.

Status of Apparatus

The apparatus is in unsatisfactory condition for further use at three critical points. The windshield wiper in the precipitator tank (Cell 4) which partially failed during Preparation # 4 is now completely out of service. Lack of this device makes it impossible to agitate small volumes of solution in the precipitator tank; this greatly delays important steps in the process and is believed to lead to considerably higher losses in the metal waste fractions. It seems to us highly desirable to repair this mechanism before proceeding with another preparation. It is not known at this time whether or not this repair will require complete decontamination of Cell 4.

The second breakdown is in the reagent or upper cow in Cell 2. This has been manipulated only with the greatest of difficulty during the last two preparations and has gradually been getting worse. Its repair before using this apparatus again seems necessary.

The third breakdown is the previously mentioned breakage in the evaporation apparatus of Cell 2.

Repair of these last two items, both necessary before Cell 2 can again be used, will require complete decontamination of this cell. It was in this cell as you will recall that the major spill preparation 4 occurred. We have no very good idea, therefore, as to how difficult it will be to decontaminate the cell and its equipment to a level that will permit personnel to work inside.

The condition of Cell 1 is still the same as it was at the end of Preparation 3. It is operable with great difficulty, and furthermore has not the several improved features that we installed in Cell 2 during the last long shutdown. We feel that we can not risk another preparation in this cell without a thorough overhaul. We have a pretty good idea, however, as to the difficulties of its decontamination and would estimate that a minimum of 3-4 weeks will be required to effect clean up and reconditioning.

The prospects for Preparation #6, therefore, are rather dark, but we will keep you informed and do the best we can.

Yours sincerely,

M. D. WHITAKER, DIRECTOR

CLINTON LABORATORIES

By_____

Henri A. Levy

M. D. Whitaker

March 27, 1945

H. A. Levy

Shipment of Special Source # 6

Enclosed is a message, consisting substantially of the following, to be transmitted to J. R. Oppenheimer, Attention R. W. Dodson, in re shipment of special source #6:

"The 6th preparation assayed 315 $\pm$ 30 curies UX equivalent at final precipitation time, 11 A.M. on 26 March, 1945. The material balance and yield from the process were both poor for unknown reasons. Analyses for impurities by a polarographic method revealed 2 mg Pb, 0.8 mg of Cr (upper limits), and undetectable amounts of iron and nickel. 1 gm Ba as chloride was used and is well concentrated in the lower third of the conical part of the Pt lined cone. The color was pure white with very strong luminescence and no appearance of oil or other foreign matter. We now believe that the oily impurity reported by you in the last preparation arose from condensation of Dowtherm from the heating bath. Dibutyl phthalate was substituted in the present run.

"The seventh preparation is now in progress. The dissolver jacket contains a leak to the outside of considerable size. no interference with operations is yet evidenced but no guarantee for the future can be made. The equipment including the glass is otherwise in good condition."

DATE: 4-4-45

TO: J. R. Oppenheimer - R. W. Dodson - Y

FROM: H. A. Levy - Cl. Labs.

Preparation number seven assayed five hundred sixty plus or minus fifty U X equivalent curies at separation time four A M April third. Polorographic analysis indicated eight milligram of Pb. two milligrams of Crl and no Fe. The preparation contains about nine hundred milligrams of Ba as the Chloride. The material is believed to be well concentrated in the bottom of the Ta vessel with only small quantities in the upper part of the conical sections and none in the cylindrical portion; however, this cannot be stated with certainity because of poor visibility. Contamination of blocks two and three appears to be negligible.

May 18, 1945

M. D. Whitaker

H. A. Levy

Shipment of Special Source #8

Enclosed is a message, consisting substantially of the following, to be transmitted to J. R. Oppenheimer, Attention of Mr. G. Friedlander, in re shipment of special source #8:

"The eighth preparation was assayed at 750 ± 70 curies (UX equivalent) at the time of final precipitation which was 6:00 P.M. on May 16, 1945. Analysis for impurities was carried out spectrographically and indicated contamination with 23 ± 5 mg. of lead, less than 5 mg. each of iron and nickel, and less than ½ mg. of chromium. The plant showed also presence of small amounts of strontium, aluminum, and calcium. We examined the final product visually and found it to be pure white, strongly luminescent, and distributed fairly uniformly over the conical part of the shipping vessel. Only a small amount was in the lower part of the vertical portion. The cone was tantalum lined. All the apparatus is in good condition. We would appreciate hearing the results of your measurements at the time of maximum growth of the daughter."

May 31, 1945

M. D. Whitaker

H. A. Levy & C. D. Coryell

Shipment of Special Source # 9

Please transmit the following information to Mr. J. R. Oppenheimer, attention Mr. G. Friedlander:

"The assay of the final solution of Preparation #9 was 800 ± 10% curies (UX equivalent) at final separation time, 20:30 CWT May 28th. The next to the last analysis (lead chromate filtrate) corrected for subsequent losses gives indication that the true assay may be higher by 150 curies.

Analysis for impurities was carried out spectrographically with the results: Pb, 17 ± 5 mg; Fe, Ni, and Cr, less than 2.5 mg each; Sr, Ca, and Al were detected in small quantities. One gram of Ba was added as carrier.

The preparation seems to be salmon colored with brilliant luminescence. It is well concentrated in the lower half of the cone but a small amount seems to be present in the lower half of the vertical portion. The vessel is Ta lined.

There seems to have been extensive deterioration of the Alloxite filter in the steel precipitator in Cell 4. In other respects the apparatus seems to be in good shape.

We request that you relay us information on your assay of the Preparation".

M. D. Whitaker October 19, 1944

H. S. Brown

FUTURE PROSPECTS AND POLICY FOR LANTHANUM PRODUCTION AT CLINTON

For the last several weeks many of us in the Chemistry Division have been giving very serious thought to the future outlook of the production of La^{140} for use at Site Y. We have all realized that existing equipment probably has a finite life. For this reason, if Site Y continues to request lanthanum shipments past the first of the year, a completely new setup is definitely in order. I am sure that you concur with me on that conclusion.

To date two shipments have been sent--one of the order of 300 Y curies; the second unknown but probably about 200 Y curies. The first preparation succeeded beautifully. At its conclusion most of us were confident that our equipment would last for perhaps twenty weeks. The second preparation proceeded with much greater difficulty and there was considerable evidence that our equipment was deteriorating rather rapidly. It has been found that now certain lines are plugged and certain glass breakages have taken place. These can be repaired in time but it is certainly indicative of the problems that will confront us in increasing number as time goes on. We know that our yields are much poorer than we had originally supposed. The analytical problems connected with the preparation are formidable. We are now in the position where we realize that no barium analytical result connected with the process can be completely trusted. I should like to emphasize at this point that none of this should cast any reflection on the individuals connected with designing and constructing the setup. They have done a magnificent job under the most trying of circumstances. We all appreciate the order of magnitude of the difficulties in such a preparation.

It is quite apparent that continued production of lanthanum for an indefinite period of time will require

1. new building facilities

2. new equipment facilities wherein glass is eliminated as much as possible.

In connection with the building facilities, there are three possibilities:

1. Carry out the preparation in the 205 Building

2. Build an entirely separate building for the purpose

3. Build an addition to the present 706-C Building

The first possibility is apparently out because of timing considerations with respect to production at Hanford. When it comes to a choice between the second and the third alternatives, it is the opinion of both Mr. Johnson and myself that, from the point of view of a long-range program, construction of an annex to the 706-C Building would be the most preferable. It is appreciated that the job, once it is stabilized, will be essentially a production job; but we are unable to see where a separate building would offer any great advantage over an attached one in this respect. An attached building offers the very great advantage that when lanthanum production ceases all hot cell facilities will be close enough together to be considered a single research unit. Facilities can be incorporated into the new addition that experience has shown are sadly lacking in the existing building. The manpower considerations involved in putting the lanthanum job on a more permanent basis are rather formidable. As we see it, the following work must start now and continue until the new unit is completed.

1. By some means or other keep the present production unit operating successfully so that Y will continue to have a supply of lanthanum

2. Train new men so thoroughly in the operations that they will be able to take over the routine operations on the new unit

3. These men under training can release men now on the job for developmental work on instruments, gadgets, and techniques that will be necessary for the new setup

4. It is obvious that a crew must be placed immediately on studying the analytical problems connected with the preparation

5. A large crew of chemists must immediately plunge into chemical research on both the existing process and on devising a modification of the existing process for use in the new operating unit

6. Mr. Leverett should have a group working very closely with our men. As we see it, he would be in charge of all necessary engineering design work

On the basis of the above, I would like to make the following specific manpower recommendations. I have talked with Mr. Johnson by telephone and he agrees with me on all points.

1. About ten men should be pulled off of certain existing Hanford studies and put on the analytical and chemical research problems involved

M. D. Whitaker, October 19, 1944

2. A request should be placed at the earliest possible time for nine more S. E. D. men to replace certain members of our present operating crew that we would like to transfer to remote control developmental work noted above.

3. An immediate request should be placed with the Du Pont company asking that Clinton Laboratories be permitted to retain the services of Mr. D. S. Webster for a continuing period of at least three more months. Mr. Webster has more actual experience in the lanthanum production job than any of Mr. Leverett's men and, because of this, we believe that his services would be of very great value in the design and early operating stages. Very fast action is needed in this respect as Mr. Webster is scheduled for transfer on October 27. Such a request is not unreasonable for Mr. Webster is scheduled to go into the Plant Assistance Group at Hanford and, with existing Hanford schedules, his services will probably not be desperately needed for some time.

Mr. Johnson and myself will, of course, see to it that the necessary transfers of work are made within the Chemistry Division.

If you can find it possible to see to it that the other recommendations are carried out, I can't help but feel that a satisfactory operating unit will be in good working shape in a reasonable length of time.

Harrison S. Brown

HSB:MJS

November 2, 1944

Mr. A. H. Compton
P. O. Box 653
Richland, Washington

Dear Mr. Compton:

BARIUM-LANTHANUM PRODUCTION

This letter will summarize for you the present status of the Ba-La production for Site Y. As you know, we have been attempting to produce this material in 100-Curie lots in the present 706-C Building which was designed to handle activities up to 10 curies. The apparatus which has been placed in the 706-C Building consists of stainless steel apparatus at the beginning of the process and glass apparatus in the latter part of the process. With these facilities, we have turned out two shipments for Y which have been assayed by them and found to contain about 300 curies each. They now request that we deliver 300-curie batches instead of the 100-curie size originally asked for.

The production of the two batches of Ba-La which have been delivered resulted in failures in the glass part of the apparatus which has made it necessary to decontaminate the cell containing the glass apparatus to a point where it is possible for men to work inside the cell for a short time. This decontamination literally takes weeks. Two sets of glass apparatus were provided in adjacent cells as alternates to each other. Only one set has been used to date and we are reluctant to undertake the preparation of a shipment without having both sets of apparatus in usable condition.

The information we have from Y is that they want a minimum of about five or six shipments and probably will want the shipments to continue at three-week intervals indefinitely. We believe that serious difficulties will be encountered in supplying these five or six shipments with the present apparatus but that this probably can be done. We further feel that we should not assume any obligation to continue these shipments for a longer period without providing more dependable apparatus and more satisfactory shielding arrangements than we now have unless someone else is in a position to take the responsibility for unsatisfactory performance.

A project has been prepared along the lines that were recently discussed with you which covers the main features of a new building for doing this work more satisfactorily. A copy of this project is attached. You will see that the estimated cost of this new construction is $175,000, and the time of completion is described on the project as four months after

authorization. This four months is an estimate and we will not be sure of that until the process design work has proceeded somewhat further toward completion. This date is arrived at on the assumption that no other major job of this kind will be required of our Maintenance Department. At Colonel Nichols' request, I am planning to show this project to him in the next day or so.

I should like to point out that the new facilities under discussion will in no way help us in meeting the Site Y requirements for Ba-La for the next four months and may even tend to interfere with the production for the next four months becuase of division of effort between production and planning for future production. It should also be pointed out that we are not in ideal shape to design the new facilities because of the unsatisfactory state of the present process. About fifteen chemists have recently been assigned the job of studying this Ba-La production process in order to get the information needed for design of new equipment and to understand the unexpectedly poor results which have been obtained in the preparation of the first two batches.

We are considering the desirability of asking Leverett to take on the operation of this new building in his Technical Division. I am sure that this will not appeal to the chemists but it seems the most effective way to insure satisfactory performance of the equipment.

Very truly yours,

M. D. WHITAKER, DIRECTOR
CLINTON LABORATORIES

MDW/er
Enc.-1

November 3, 1944

The District Engineer
U. S. Engineer Office
Manhattan District
P. O. Box E
Oak Ridge, Tennessee

Dear Sir:

706-C BUILDING FOR
BARIUM-LANTHANUM PRODUCTION

I am enclosing a copy of Clinton Laboratories Project No. C-43 which covers the major design features of a building for continuation of the Ba-La production for Site Y. You will recall from our discussion yesterday that this building is to replace the part of 706-C which has been used to date in the production of this material. This 706-C building was designed to handle activities up to 10 curies and has been modified in part to allow us to handle, unsatisfactorily, the larger shipments which are now requested by Dr. Oppenheimer.

We have undertaken to supply some five or six shipments of about 100 curies each from the modified equipment in 706-C and have made two such shipments each containing 300 curies on the basis of the assay at Y. We hope that we will be able to finish the five or six shipments with the present apparatus but feel that the probability of continuing these shipments at three-week intervals becomes decreasingly less with each shipment. We are submitting this project to Major Murphy for approval within the next few days. We feel that it should be approved or disapproved on the basis of urgency of the demand for this material after approximately March 1, 1945.

You will note that the project as written specifies that the building will be ready for use four months after authorization is received. We do not have sufficient information as to apparatus procurement difficulties and process difficulties to enable us to make this a firm date. We do feel, however, that this is a reasonable estimate of the time necessary for completion of these facilities on the assumption that they will be provided by our Maintenance organization and that no other major tasks not presently contemplated will be injected into the picture during the construction period.

Very truly yours,

M. D. WHITAKER, DIRECTOR

MDW/er
Enc.-2

Project No. C-43

Date October 31, 1944

CONSTRUCT BUILDING AND INSTALL EQUIPMENT

FOR BY-PRODUCT PROCESSING

I PRESENT FACILITIES AND TO WHAT EXTENT THEY ARE INADEQUATE

Building 706-C was originally designed for research purposes. Recent demands for two batches of a particular by-product have necessitated a temporary laboratory scale set-up which has displaced a large portion of the existing research facilities of the building. This set-up of laboratory equipment is not suited for stabilized output of the desired by-product. The probability of successful preparations decreases with extended usage of present equipment; consequently, there is no assurance that failure and delay can be avoided in future deliveries.

In order to assure future output, construction of suitable production facilities is necessary.

II GENERAL DESCRIPTION OF THE PROJECT

It is proposed to construct a building, 95 ft. by 95 ft., adjacent to Building 706-C and to install laboratory and production facilities necessary to fulfill continuing requests for the desired by-product.

III DETAILED DESCRIPTION AND ESTIMATED COST

Construct a building east of Building 706-C in general accordance with the floor plan as shown on the attached print CL 706-D-1. Wooden construction similar to that of Building 706-C is to be used. The proposed building will consist of the following:

1. A processing area 50 ft. by 85 ft. to include two adjoining process cells of overall dimensions 20 ft. by 60 ft. by 20 ft. high and necessary access openings, process lines and equipment, control panels, operating platforms and ventilation.

2. A solution make-up room, 10 ft. by 20 ft., equipped with tanks, scales, pumps and chemical feed lines.

3. A chemistry laboratory, 20 ft. by 21 ft., equipped with sinks, tables, hoods, services and special equipment.

4. Two analytical laboratories each 9 ft. by 22 ft. for testing process samples. These will be of special construction.

5. An area 30 ft. by 40 ft. for services in connection with the special hazard. Included are monitoring room, uniform dispensary, locker room, men's room, personnel equipment, decontamination room and health office.

6. Process equipment decontamination room 10 ft. by 10 ft.

7. A monorail system for handling miscellaneous heavy equipment material and containers. This system will be connected to that existing in Bldg. 706-C.

8. Two offices each 11 ft. x 17 ft. and which will be easily accessible to the operating area.

9. A conveying system for material from Bldg. 105 to the top of the operating cells.

10. Storage space for materials and equipment needed in this process will be made available by utilizing the area on top of the chemistry and analytical laboratories and offices. Loading platforms will be provided along the main E.-W. plant road.

11. Additional transformers and relocation of present transformer bank will be necessary. Connections to relocated power lines and existing services.

The Total Estimated Cost of the proposed work is $175,000.

IV REMARKS

This project is classified as New Construction.

The necessity for construction outlined in this appropriation request is wholly concerned with the anticipated need of an associated project, and is not to be considered an expansion of the normal activities for which Building 706-C was designed.

The construction herein outlined will take priority over project construction now in progress.

Under the provisions of Contract W 7405 Eng. 39, the U. S. Government will supply the funds for all expenditures on this project.

December 11, 1944

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

THE RADIOACTIVE PURITY OF FUTURE BA-LA PREPARATIONS

On November 28, 1944, a telephone request was made that you express your desires as to the radioactive purity of the Ba-La shipments that are to be made after March of 1945. On December 2, 1944, a verbal answer was received stating that the total gamma disintegration rate of the impurities should not exceed one per cent of the Lanthanum five days after our final separation. This was confirmed by you in a teletype dated December 2, 1944. We were given to understand that this restriction should apply to all gammas, both soft and hard. Before we can commit ourselves to a restriction such as has been proposed, we believe that the matter should be discussed further in order that all parties concerned will be fully cognizant of the difficulties that will be brought about at Clinton Laboratories in order to achieve such purity and of the difficulties that would be brought about at Site Y should the stated purity level not be achieved.

From our point of view any relaxation of radioactive purity requirements will increase the simplicity of our operations because of the fewer number of operating steps that would be necessary. We believe that you appreciate that the fewer steps there are in our process the higher will be the yield and the smaller will be the chance of equipment breakdown. Likewise, a simplification of our flowsheet would increase the probability of our starting our new operations in a reasonable length of time. We do not know what basis you have used for arriving at the one per cent figure. We are particularly surprised that this should apply to both soft and hard gammas. If you will remember, at one time a verbal statement was made to the effect that your restrictions as to soft gamma impurities were not too important. We are assuming that barium is not considered an impurity and in view of the fact that barium emits rather significant numbers of both soft and hard gammas, your request is all the more puzzling. We realize, of course, that very little of the barium precipitates with your lanthanum phosphate, but on this basis alone we feel that your desires could be at least modified to include only those gamma emitters that would coprecipitate with lanthanum phosphate. It may be that all obnoxious fission products will tend to be carried by lanthanum phosphate, but in any event

this point should be investigated.

Little is known at the present time concerning the purity of the shipments that we are now making. Various intuitive estimates have been made ranging from one to five per cent of the total lanthanum gamma activity. So far as we are aware there should be little need for your requiring greater purity than we are now achieving. Could it be that your figure of one per cent was based on a verbal estimate of present impurities that I have you on November 28? I said at that time that I thought that our impurities were below one per cent. If this is true, we think that a more critical evaluation would be desirable. On the basis of the above considerations, we would like to propose the following:

1) An attempt should be made to evaluate the radioactive purity of the present shipments.

2) We should like you to consider qualifying your desires to only those impurities that will precipitate with lanthanum phosphate.

3) We should like you to reconsider your request from the point of view of taking into consideration the energies of the gamma emitting impurities. If soft gammas are detrimental to your experiments, are Bremmstrahlung of any significance?

We will see what we can do here towards an evaluation of item 1 and an evaluation of item 2 to the extent of determining what fission products will tend to precipitate with lanthanum phosphate.

We will be happy to cooperate with you in attempting to achieve whatever purity limits you feel you require; however, we know you appreciate our desire to ascertain the need and importance of any and all restrictions that are placed upon our operations.

Very truly yours,

M. D. WHITAKER, DIRECTOR
CLINTON LABORATORIES

By
Harrison S. Brown
Assistant Director
Chemistry Division

March 1, 1945

Mr. J. R. Oppenheimer
P.O. Box 1663
Santa Fe, New Mexico

Attention: R. W. Dodson

Gentlemen:

As you know, we are engaged in the formulation of a new process for the production of Ba^{140}-La^{140} using equipment that will be amenable to more secure performance than is being achieved at the present time. Some weeks ago we requested that you specify your requirements as to the chemical and radiochemical constitution of the shipments that will be made subsequent to the initiation of operations in our new equipment. Your stated specifications are listed below.

1) The amount of radioactive lanthanum present in each shipment should be approximately 300 "Clinton" curies five days after our final barium precipitation.

2) The total weight of barium in the sample must be less than one gram.

3) The lead content must be less than 25 milligrams.

4) The sum of the weights of chromium, iron and nickel must be less than 50 milligrams.

5) The material shipped should be in a chemical form suitable for dissolution, buffering, and lanthanum phosphate precipitation.

6) The tolerances for radioactive impurities are as follows, in terms of the gamma disintegration rate of the impurity expressed in per cent of the La^{140} disintegration rate five days after the final barium precipitation:

Species	Tolerance (% La gamma)
65 d Zr	1.5
17 h Zr	18
35 d Cb	1.9

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

March 1, 1945

Species	Tolerance (% La gamma)
42 d Ru	3.2
60 d Ru	1.3
17 h Pd	18
11 d Sn	8
2.5 d Sn	18
*250 d Sb	0.8
90 d Te	12
30 y Cs	1.4
28 d Cs	11
55 d Pr	1.3
16 d Ru	4.5

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

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

X_i' = observed percentage of a given species

X_i = Tolerance for the species as given in the above table

In view of the fact that basic research has now been completed on the new process and trial runs have been completed in the existing 706-C Building equipment, we would like to inform you of the present outlook in regard to your specifications.

A. Item 1) can probably be met. The Building has been designed for production at a 300-curie level.

B. Specifications 2, 3, and 4 can probably be met.

C. Concerning items 5, the material shipped will be barium nitrate or chloride.

D. Our chemical tests indicate that all tolerances listed under Item 6 can probably be achieved.

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* probably does not exist

For your information, the new process will run approximately as follows:

Step	
I	Carrying of barium from UNH with lead sulfate
II	Metathesis of lead sulfate to lead carbonate.
III	Dissolution of lead carbonate in nitric acid. Two alternatives have been developed for the subsequent steps, both of which appear to be satisfactory.
IV (a)	Precipitation of lead chromate. The barium remains in solution.
V (a)	Alkalinization of the solution causing precipitation of barium chromate. The residual lead remains in solution.
VI (a)	Dissolution of barium chromate in nitric acid using peroxide.
VII (a)	Precipitation of barium nitrate using fuming nitric acid.
VIII (a)	Dissolution of barium nitrate.
IX (a)	Evaporation of barium nitrate to dryness in the shipping container.

The alternative steps are:

IV (b)	Electrolysis of lead from the lead nitrate solution.
V (b)	Evaporation of the solution to 10% of the original volume.
VI (b)	Precipitation of barium nitrate using fuming nitric acid.
VII (b)	Dissolution of barium nitrate.
VIII (b)	Evaporation of barium nitrate to dryness in the shipping container.

Yours truly,

M. D. WHITAKER, DIRECTOR
CLINTON LABORATORIES

By _____
Harrison S. Brown
Assistant Director
Chemistry Division

May 10, 1945

The District Engineer
U. S. Engineer Office
Manhattan District
Oak Ridge, Tennessee

Attention: Major Edgar J. Murphy

Dear Sir:

PROJECT C-43 FOR BUILDING 706-D

In confirmation of the recent conversation between yourself, Mr. Whitaker, and the writer, we desire to inform you of some of the statistics of interest in connection with the completion of Building 706-D:

267 Drawings were completed by the Project Group for the design of the building and equipment.

The 8 Draftsmen worked a total of 976 hours beyond 48 hours per week during the work on this project, and the 13 Engineers worked 2,283 hours beyond 48 hours per week.

Amongst the construction group, there were worked 33,549 additional overtime hours.

Considering the lack of detailed information available at the start of this job, the unique nature of the equipment, and the extreme urgency for the completion of the work, it is our feeling that the men in the Chemistry and Technical Divisions and in the Project and Maintenance Departments have completed an outstanding accomplishment, which we believe could hardly have been done in less than a year at a considerably greater expenditure by any other organization.

Very truly yours,

M. D. WHITAKER, DIRECTOR
CLINTON LABORATORIES

J. P. Sinclair, Manager

JPS:av