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February 15, 1945

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## Report of the 200 Area Technical Group For Period February 8 to February 15, 1945

### General

In connection with the investigation into the causes and elimination of the high waste losses being encountered in the $LaF_3$ product precipitation, a method of producing $LaF_3$ in a granular, crystalline form has been developed. This crystalline $LaF_3$ resembles $BiPO_4$ in its mechanical and handling properties, centrifuging easily and can be readily slurried. Laboratory tests show that the $LaF_3$ crystals carry well under process conditions, and can be metathesized to a granular $La(OH)_3$. These findings, if corroborated in the plant would offer the most attractive method for improving yields and time cycles in the crossover and metathesis step yet considered.

### Plant Performance

Processing of the T-5-02-B series started on 2-8-45 at a product concentration equivalent to 10% of maximum. The average performance on the runs made to date are as follows:

| Step | Waste Loss % | Material Balance % | B Decontamination Factor |
|---|---|---|---|
| Extraction | 0.7 | 99.7 | 8.5 |
| First Cycle B.P. | 1.3 | | |
| First Cycle P. | 0.3 | 94.7 | 7000 |
| Second Cycle B.P. | 0.3 | | |
| Second Cycle P. | 0.3 | 98.0 | $3x10^5$ |
| Cross Over Bi B.P. | 0.2 | | |
| Cross Over La B.P. | 0.7 | | |

The excellent material balances currently being obtained in Building 221 are noteworthy.

### Building 221

The background activity in most of the process vessels as recorded on the Beckman cell meters appears to be leveling off at about 1-3% of the value recorded when the vessels contain process solution. In the case of the 5-4 and 13-4 tanks (extraction and first by-product solution tanks) however, a progressive increase in the background activity is taking place and now is 55% and 5.3% respectively of that when the vessels are full. In the case of 5-4, the increase is probably associated with insoluble residues from the metal solution, while in 13-4, the accumulation of insoluble scavengers is probably responsible.

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3-1635

As reported previously, the activity on the 221 Building fan casings increased to approximately 120 mr./hr. at the end of the January series and then decreased to about 40 mr./hr. during the shutdown that followed. No increase was observed during the dissolver operation of the current series but an increase was immediately apparent when the first extraction was made on 2-7-45. The level at the present time is 100 mr./hr., the increase to this level has been step-wise and to date it has been impossible to correlate the rate with any operations performed in Building 221.

Analysis of the activity deposited on the fan casing has shown that only a small fraction is due to iodine, the remainder being due to general fission product contamination. This suggests that the source of the contamination is from spray entrained into the air by the centrifuges. Confirming this is the fact that appreciable amounts of product were also found associated with the fan casing activity. The ratio of gamma activity to product represented a "decontamination factor" of about 30 suggesting that the activity is coming from all the operating centrifuges in the canyon. Product has also been found on the samples taken from the walls and top of sections 8 and 13. Considerable Pu was also found in the Section 13 sample, as might be expected.

Considerable attention has been given to the performance of the samplers in both 221 and 224 Buildings. Observation of the circulation through the samplers using Lucite models has revealed several short comings of the present design. Most serious is the fact that the actual circulation in the cup of the sample is extremely sluggish even when through-put rate is high, hence flushing of the cup is difficult. It has been found necessary to start and stop the sampler several times during the 10 minute circulation period in order to increase turbulence and assist in flushing out the cup. The size of the capillary air leak has been found to be quite critical. In addition, it has been found desirable to have a vacuum gage on each sampler to insure that circulation is being obtained.

## Building 224

### A Recycling of Combined Isolation Supernatants

A production test (T-1-8) of the plan of recycling combined isolation supernatants from several charges, instead of singly, has recently been completed. The 231 recycles of T-6-1-B-1 (second half), B-2, B-3 and T-4-12-B-6-C and the F-10-P from T-6-1-XC, containing approximately 5 lbs. of Ia were combined. This master recycle was returned to D-1, where $HNO_3$, water, $Na_2Cr_2O_7$, and $KMnO_4$ had been previously added to the usual D-1-0 solution weight and composition. After oxidation, a single $LaF_3$ precipitation was carried out, the slurry centrifuged, the cake washed by displacement with 6% $HNO_3$ and skimmed to a 30-gal. heel. The effluent was then carried through the usual reduction and $LaF_3$ product precipitation in Cell E and $KOH-K_2CO_3$ metathesis in Cell F.

A high E-3-WS waste loss (7%) was obtained on this run (T-6-1-BX-1) but it is not felt that it was connected with this recycling procedure. This E-3-WS was reworked with a full extra charge of Ia(2 shots of 1.25 lbs. each) and carried on through metathesis as a separate charge (T-6-1-BX-1-A). The over-all data for this run are given in Table below, showing a loss in either E-2 or Cell F on BX-1 which was recovered in BX-1-A.

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3-1635

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Combined Recycling Run and Subsequent Rework

| Run No. | Product Yields (%) |  |  |  |  |  | Product Losses (%) |  |  |  | Final |  |  |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
|  | F-3-RC | E-1-O | E-3-CS | S-1-X | F-1-PS | F-10-P | E-4-BP | R-3-MB | F-7-WS | F-9-MS | Yield | Loss | E.E. |
| T-5-1-RR-1 | 100 | 97.6 | 89.3 | -- | 73.8 | 70.4 | 1.2 | 7.8 | 0.7 | 0.3 |  |  |  |
| T-5-1-RR-1A | -- | -- | -- | 7.5 | 26.2 | 24.4 | -- | 0.7 | 0.1 | 0.1 |  |  |  |
| Overall | 100 | 97.6 | 89.3 | -- | 100.0 | 94.8 | 1.2 | 0.7 | 0.8 | 0.4 | 94.8 | 3.1 | 97.9 |

## B F-3-WS Waste Losses

The program outlined last week for following all X operations on the next active run was carried out during T-5-02-B-1. Samples were taken for analysis during reduction, digestion, and centrifugation.

During the first February production run (02-B-1) in 224-T some 40 progress samples were taken covering the reduction, product precipitation, and the two centrifugation steps. In all cases, the samples were prepared for counting within an hour after the samples were drawn so as to eliminate as much possible any changes which might occur upon standing. The data obtained show about 72% efficiency in product removal in the first plant centrifugation and about 63% for the second (a second shot of $LaF_3$ precipitated prior to second centrifugation), thus leaving about 7.2% of the product in the final effluent (based on S-1-X assay). The results obtained on centrifugation in the laboratory show marked differences in the properties of the $LaF_3$ before and after plant centrifugation. Laboratory centrifugation of the original $LaF_3$ slurry removed 99% of the product but only about 85% of the product in the effluent from the first centrifugation in the plant was removed by a comparable laboratory centrifugation. However, after the second shot of lanthanum had been added, the efficiency of laboratory centrifugation was about 98%, but dropped to about 90% on the effluent from the second plant centrifugation step.

Although it appears that the high F-3-WS product losses are caused by the leak-through of fines through the centrifuge, (the results of chemical runs made in 224-U, although not clear cut, confirm the view,) it is not known at the moment where these fines are being generated - a) poor flocculation of the $LaF_3$ during precipitation and digestion in the precipitator, b) peptization during the jetting operation, or c) micronizing during the centrifugation. An attempt is to be made to separate these variables.

The immediate program is as follows: a) Two runs to be carried through three $LaF_3$ strikes and centrifugations with the same total La split into three equal shots, b) Two runs to be carried through the entire cross-over with "distilled water" obtained by condensing process steam in R-1, since deionized water was used at Clinton in comparison with the present untreated water which contains 25 mg./l.Ca.

## C High Pressure Water Jet for Removal of Lanthanum Hydroxide Cake from F-2 Centrifuge

Initial trials of the high pressure water spray in the lower half of F-2 centrifuge in 224-U building indicated that the lanthanum hydroxide cake was readily removed when operating with 300 lbs. water pressure. A second jet will be supplied to the high pressure spray header which will permit jetting into the two lower shelves in the centrifuge basket. It is expected that this modification will be completed by Feb. 13, 1945. [redacted]

3-1635

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### D Distilled Water Supply for 224 Building

For test purposes, a supply of distilled water was requested for use in the next few process runs in 224-T Building. Use of the Cell 3 precipitator for condensation of steam from the spargers was suggested and it was found that about 1700 lbs. of steam/hour could be condensed without difficulty. Provision is being made for suitable piping modifications for jetting the condensate from D-3 to the operating gallery where a hose connected to the piping can be used to feed water to the weigh tank.

## 231 Building

### A Operation

Equipment clean-up and routine maintenance was underway during the week. Arrangement of the Cell 3 equipment was agreed upon and installation has been started. Operating procedures for the February series have been prepared.

### B Nitrite Method for Destruction of Excess Hydrogen Peroxide

On the basis of laboratory tests employing simulated peroxide supernatant solutions, it has been established that the quantity of 20% $NaNO_2$ solution required to react with the $H_2O_2$ present in the supernatant is 80% (±1%) of that calculated from the equation: $H_2O_2 + NaNO_2 \longrightarrow H_2O + NaNO_3$. This has also been confirmed on large scale tests conducted in Cell 2. On this basis, a simplified operational procedure has been drawn up which does not involve spot tests to determine the end-point of peroxide destruction. This procedure will be tried in Cell 1.

### C Study of the Pre-Reduction Step

Preliminary data on the use of 1% $H_2O_2$ by weight at 50°C as a function of time indicate that the reduction of Pu (VI) to Pu (IV) is substantially complete in 1.5 - 2.0 hours, but possibly incomplete in the 1 hour heating period now in use in the plant. The data are not as yet conclusive, and further tests are in progress. A sample of F-10-P solution from run T-4-12-P-6-0 is being used in these studies.

### D Possible Precipitation of $Pu(SO_4)_2$ During Concentration (Distillation Step)

Tests described in last week's report have been continued. Addition of $H_2SO_4$ (to make solution 0.25 M) to a nitric acid solution of product (168g. Pu(IV)/l.- 10N $HNO_3$) showed no precipitation after standing for 4 days, but a small amount of precipitate was observed at 12 days. This precipitate is dark green whereas product sulfate is brick red. This solution will be allowed to stand a while longer before identification of the precipitate is attempted.

### E Equipment for Drying the Final Product Solution

The temporary drying equipment for the final product solution is being modified to permit safer and more convenient operation when drying the next several lots of product solution. In the main, the modifications include the substitution of a stainless steel condenser for the first glass condenser in the previous drying train, and modification of the condensate receiver to permit sampling of the condensate without disconnecting the receiver.

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Design of the permanent drying equipment to be set up in Cell 5 is progressing. Sketches are complete covering the design of a plug to be installed in the sample can after filling in the cell, and which will remain in position thereafter during drying and shipping. A general overall arrangement of the drying equipment, and method for connecting the sample can to the drying equipment, and removal therefrom has been reviewed.

## Process Chemistry

### Crystalline Lanthanum Fluoride

A very promising lead has been developed from the study of methods for the improvement of centrifugation characteristics of $LaF_3$ in the development of a procedure for the preparation of crystalline "$LaF_3$" of comparatively large particle size, which settles rapidly to a low bulk. The ratio of lanthanum to fluorine in the precipitate has not been determined but the compound contains no other cation than lanthanum and behaves chemically in a manner similar to the familiar gelatinous $LaF_3$.

The procedure for precipitation of the crystalline "$LaF_3$" involves preparation of an insoluble but well defined crystalline salt of lanthanum that does not contain fluorine which is added as the solid (in slurry form) to the product solution 1M in $HNO_3$ and 0.5M in HF and the solid is changed to "$LaF_3$" but the external form of the original solid is retained. More specifically, the lanthanum compound on which most of the work has been done is potassium lanthanum sulfate which is prepared from solutions of lanthanum ammonium nitrate and $K_2SO_4$ at about 75°C. If the concentration of $K_2SO_4$ is held at about 0.5M virtually no lanthanum is present in the supernatant solution. The double sulfate is a white granular salt, and the form of the crystals is determined by the relative amounts of $La_2(SO_4)_3$, $K_2SO_4$ and $H_2O$ in the crystals which in turn may be varied by adjusting the $K_2SO_4$ concentration during the precipitation. The double salt is readily soluble in 1M $HNO_3$ and somewhat less so in water alone. However, if the salt is added to a solution 1M in $HNO_3$ and 0.5M in HF none of the salt appears to dissolve and the crystals retain their structure upon conversion during the subsequent digestion period.

Product carrying of 98-99% has been obtained using "$LaF_3$" of this type after a normal digestion period of one hour at room temperature. This test was at product concentration equivalent to 250 grams of product per ton of metal.

The crystalline $LaF_3$ metathesizes readily to a $La(OH)_3$ of essentially the same particle size and the resulting hydroxide is readily soluble in $HNO_3$.

By the use of this type of "$LaF_3$" in the by-product precipitation step, gamma decontamination factors of 100 have been obtained on Ba-La tracer activity. In these tests and in the experiments on product carrying the lanthanum salt was added in one shot in an amount equivalent to 100 mg. of La/liter.

Experimental work is continuing with attention being given to adapting the procedure to plant equipment. Complete bulk reduction cycle trial runs are also being made in the laboratory. It is, of course, recognized that tests will have to be made on a plant scale before the merits of the procedure can be determined.

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3-1635

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## Improved Decontamination in One Cycle

By operating at low acidity in the by-product precipitation step and using Cb-Zr-BaSO$_4$ scavengers, gamma decontamination factors of over $10^5$ were obtained for the cycle and over $10^6$ for the cycle plus the extraction step. These runs were made starting with metal solution spiked with plant dissolver solution from the 30g/ton material to provide the mixed fission activity. Product concentration was at tracer levels. Measured product loss in the wastes was about 5% for the cycle exclusive of the extraction step.

## Process for Recovery of Product from a BiPO$_4$ By-Product Precipitate Containing Substantial Amounts of BiPO$_4$ in the $\beta$ Form

If the less soluble beta form of BiPO$_4$ were formed in a by-product precipitation step considerably more HNO$_3$ than is now used would be required to dissolve the precipitate. If such a solution were to be reworked by present process procedures, process volumes in excess of tank capacities might be involved. A recovery process is therefore being worked out which is based on the use of twice the normal amount of HNO$_3$ for dissolving the by-product BiPO$_4$ (which would be adequate for complete dissolution of the BiPO$_4$ if it were all in the $\beta$ form) followed by removal of a by-product precipitate at approximately 2N HNO$_3$ and reduction of acidity to 1N by neutralization prior to the product precipitation step. One run was made using NaOH for neutralization and another involved the use of (NH$_4$)$_2$HPO$_4$. Satisfactory product recovery (tracer levels) and decontamination factors (Cb-Zr tracer) were obtained. The product precipitate, however, was somewhat difficult to dissolve when NaOH was used for the neutralization. Runs are now being repeated at product concentrations representing 100% loss at a product level of 30 grams per ton of metal.

## Factors Influencing Evolution of Iodine in the Metal Dissolution Step

Particular attention has been given to the evaluation of various analytical procedures for determination of iodine in the metal and scrubber solutions in order to obtain a satisfactory and reliable procedure. This has been one of the major difficulties in this study.

Samples of metal solution (4-7-M) from runs T-3-OZ-B1, -B2, and B3 have been analyzed for iodine by the best method available and the amount of iodine found was about 10% of the total calculated amount that should be present in the metal at the time the assay was made. This means that residual iodine remains in the solution after the metal dissolution step which may be evolved in subsequent process steps.

## Plant Fission Product Studies

On the basis of analytical data obtained by the Analytical Section on analyses of plant solutions before and after the cross-over cycle for fission product elements, the following conclusions can be drawn:

1) The beta activity before and after the cross-over is associated predominantly with the rare earth fraction.

2) A relatively high and hitherto undetected percentage of Ru is present before and after the cross-over.

3) The low levels of activity in samples precludes any reasonable accuracy for analysis of gamma emitters and variations of gamma decontamination in different runs cannot be proven to be real.

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3-1635

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Conversion of $Pu(OH)_4$ to a Difficultly Soluble Form on Heating in Metathesis Solutions

In studies which are being carried out to determine how and when product is oxidized during metathesis and the subsequent dissolution step, it has been found that $Pu(OH)_4$ after digestion at 80°C with either 15% KOH or 15% KOH-10% $K_2CO_3$ solution is relatively difficult to dissolve in nitric acid and that a relatively large proportion of the product in solution is in the abnormal or polymerized form. These tests have been made on straight $Pu(OH)_4$ and tests are in progress to determine behavior after digestion when $Pu(OH)_4$ is coprecipitated with $La(OH)_3$ and also when hydroxides are formed by the metathesis of the mixed fluoride.

Carrying of $SO_4^=$ by Product Peroxide

When product peroxide is precipitated from sulfate-containing solutions, sulfate is carried by the precipitate. Data available here and from other Sites indicate that the ratio of Pu to sulfate is somewhat variable and may depend on precipitation and washing conditions. As reported last week, a Pu to $SO_4^=$ ratio of 3 to 1 was found in the final composite solution from plant runs 12-P-5 through 01-P-3. In laboratory precipitations made under approximate 231 Building precipitation conditions, sulfate carrying was as follows:

| $PuO_4$ Wash | $Pu/SO_4$ Mole Ratio |
|---|---|
| Twice with 1N $HNO_3$ | 5.8 |
| Twice with $H_2O$ | 4.8 |

On the basis of these tests, it appears that some sulfate was recovered by washing with 1N $HNO_3$.

The sulfate content was also determined on a product solution on hand which had been prepared from $PuO_4$ precipitated from a solution 1N in $HNO_3$ and 0.2N in $H_2SO_4$ containing 34 mg. Pu/ml. The $Pu/SO_4$ ratio was 6.7.

Some time has been spent on the development of a satisfactory analytical procedure for the determination of sulfate in product solutions.

Radiation from Pu

Comparisons of the harder electromagnetic radiations from Site X and H.E.W. (January production composite) Pu show that all components have, within experimental error, the same energy values and relative intensities. The results are tabulated below:

| Component | Source | Half Value Thickness (gm Pb/cm²) | Energy Mev | Extrapolated Specific Counting Rate, 3rd Shelf c/m/mg. |
|---|---|---|---|---|
| 1 | X | 3.38 | 0.43 | 1.67 |
| | HEW | 3.34 | 0.42 | 1.67 |
| 2 | X | 0.42 | 0.17 | 1.76 |
| | HEW | 0.44 | 0.17 | 1.57 |
| 3 | X | 0.13 | 0.1 or 0.06 | 3.0 |
| | HEW | 0.12 | 0.1 or 0.06 | 4.3 |

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3-1635

The above results indicate that both samples were completely decontaminated and that no harder radiations due to other causes, such as other heavy isotopes, are appearing in significant quantities in the H.E.W. product produced at the power level of the material processed in January. The specific counting rate of purified Pu, measured on a standard mica window GM tube with a standard Al-Pb-Al "sandwich" is 1.02 c/m/mg for the second shelf, and 0.64 c/m/mg for the third shelf.

## Apparatus and Equipment

Operation and performance tests have been made on the nitrogen chamber alpha counter and reliable and reproducible results were obtained for counting rates up to 200,000/minute. The counting rate was independent of sample position over a region of ≥5 cm. diameter on the sample holder. Coincidence loss, due primarily to the Offner scaler was about 2% at 150,000 c/m. The usefulness of this counter was demonstrated in the counting of an alpha sample having too much beta activity for the air alpha counter and which was too large to fit in the Simpson counter.

Standards ($U_3O_8$ in ceresin wax) were prepared for the liquid-sample gamma ray measuring apparatus. Tests on the three instruments (3706, 222-T, 231) were made to check stability and performance and to find and have faults corrected.

Work is in progress in assembling and setting up apparatus for the low absorption counter.

The quartz micro balance has been carefully recalibrated by weighing of small lengths of fine platinum wire individually and in various combinations, and checked against total weight obtained by weighings on the Ainsworth micro balance.

## Semi Works

### Building 221-T Head-End

Operations in the Head-End were resumed on a three-shift basis on February 12, 1945, to check the performance of the so called fluosilicate process recently recommended by Clinton. Five runs are contemplated. Material for these runs was obtained 1) from 35 slugs not charged to the February plant series and 2) by dissolving about 250 lbs. of metal from the accumulated heel in the plant dissolver. Thus, no product usable by the plant during the February series is being held up. After the Head End runs an additional 25% of a standard plant batch will be available for recovery in the plant.

One dissolver run, HS-3, was conducted in which the standard coating removal operation was used and the metal was dissolved by the three-batch procedure. The quantities of reagents in the metal dissolving were reduced by a ratio of 35 to 43, since only 35 slugs were used instead of the standard number of 43 slugs. No difficulties were encountered in these dissolving operations. The pertinent data covering this run is given in the following Table.

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**Table I**

|  | Time Heating To Boil | Time At Boil | Weight Of Metal Dissolved | Weight of UNH |
|---|---|---|---|---|
| MS-3 - 1st Batch | 1 hr. | 4 3/4 hrs. | 93.8 lbs. | 198 lbs. |
| MS-3 - 2nd Batch | 3/4 hr. | 4 3/4 hrs. | 93.8 lbs. | 198 lbs. |
| MS-3 - 3rd Batch | 1/2 hr. | 8 hrs. | 86.4 lbs. | 182 lbs. |
| Total |  |  | 274 lbs. | 578 lbs. |

Since 35 slugs contain 295 lbs. metal, approximately 21 lbs. of metal remained in the dissolver.

Prior to MS-3 run, current plant metal solution obtained from the dissolver heel containing 680 lbs. UNH was transferred from plant to the Head-End, stored, and blended with the metal dissolved in the Head End.

## Building 221

### Chemical Runs

A chemical run was made through the $LaF_3$ by-product and product steps of the bulk reduction process in cells E and F in order to determine how well the equipment operates under these conditions. The only apparent difficulty experienced was in removing the $LaF_3$ cake from the centrifuge bowls. The $LaF_3$ cake could not be removed by the plant procedure of spraying water into the bowl, but it was removed readily by plugging the centrifuge in a manner similar to that used in dissolving $BiPO_4$ cakes.

### Bismuth Phosphate Metathesis

After completion of the bismuth metathesis runs previously reported, all vessels were inspected. It was found that a sizeable quantity of $BiPO_4$ precipitate had built up in the precipitators, thus indicating the necessity of adding the dissolving acid to the precipitators and jetting it to the bowl rather than spraying the acid directly into the bowl as had been done in the previous runs.

A series of four simulated extractions were made and the cake metathesized and dissolved with the objective of combining two extractions into one run for decontamination.

It was found that in all cases essentially 75% of the total Bi is dissolved in the first acid portion, and that the fourth acid portion contains very little Bi. It was also found that the final solution contains 10% to 20% of the initial phosphate present. This may come from any one or all of the following sources:

1) Residual free $H_3PO_4$ left in the precipitator.
2) Incomplete metathesis.
3) Failure to wash out all the $K_3PO_4$ resulting from metathesis.

The results indicate that 3) is unlikely since the fourth wash contained no phosphate. In no case could undissolved precipitate be detected in the bowl after the dissolving procedure was completed.

Following this series, inactive "X" metal was dissolved and extracted under normal conditions, for the purpose of checking the metathesis procedure with metal solution. The first dissolver run produced metal solution of abnormally high acidity (1N at 30% UNH) due to condensate from the dissolver ex-

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3-1633

honst jet running back into the vessel. This was overcome temporarily in the second run by operating the condenser hot; and a solution of normal acidity was produced (0.25N at 20% XNH). The extraction precipitates from these two runs were metathesized, using conditions similar to those employed in the chemical runs except that each was given a one-hour metathesis and four washes. See results below:

| Run | Acid Portion | % Theoretical Bi in Sol'n. | % Theoretical $PO_4$ in Sol'n. |
|---|---|---|---|
| EM-14 | 1st | 69.0 | -- |
| EM-14 | 2nd | 23.4 | -- |
| EM-14 | 3rd | 4.5 | -- |
| EM-14 | 4th | 1.3 | -- |
| EM-14 | Composite | 65.3 | 16.0 |
| EM-15 | 1st | 44.0 | -- |
| EM-15 | 2nd | 40.8 | -- |
| EM-15 | 3rd | 24.1 | -- |
| EM-15 | 4th | 5.6 | -- |
| EM-15 | Composite | 89.0 | 17.8 |

Acidity of starting solution for EM-14: 1.0N

Acidity of starting solution for EM-15: 0.25N

The results of the first run (EM-14) should be discounted because the high acid content of the metal solution caused approximately 25% Bi loss in the metal waste. It should be noted that in the normal run (EM-15) the metathesized cake was not as readily soluble in acid as in the straight chemical runs. It is probable that longer contact time of the acid and cake is indicated in this step.

The phosphate content of the final acid solution is essentially the same as that obtained in the chemical runs, namely 10% to 20% of that combined as $BiPO_4$.

When the KOH liquor from metathesis was skimmed into the metal waste, local precipitation occurred. In the case of the high acid waste the precipitate redissolved quite readily with agitation. In the waste of normal acidity, the small residual precipitate did not redissolve completely, but broke up into a finely divided suspension which caused no jetting difficulties. No undissolved precipitates from either run could be detected in the bowl after the dissolving operation was completed.

L.Squires, Chief Supervisor
Sep.Eng. - Technical Department

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