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# PROPOSED CRIB WASTE DISPOSAL PROCESS

January 26, 1950

At present the process which we are contemplating is carried out by adjusting the pH of the waste solution to 4.5-4.8 with nitric acid, passing it through iron filings, adding potassium ferrocyanide to the solution and adjusting the pH of the solution to 7.0-7.5. The activity removed is on the iron filings (about 2/3rds) and in the ferrocyanide precipitate (about 1/3rd).

An alternate method is the addition of potassium ferrocyanide to the solution at a pH of 4.5 and then passing the solution through iron filings, followed by neutralization. Here the ferrocyanide precipitate is formed during passage through the iron. In this case the stream itself washes the precipitate through the iron. The distribution of the activity is the same as in the previously described method. The only difference in these two methods is the order of treatment of the waste solution.

With the freshest crib waste which we have, which contains ruthenium, antimony, tellurium, cesium, zirconium, columbium and some of the rare earths, we have been able to reduce the beta activity from 2000-2400 c/min./ml. to 150-250 c/min./ml. by one or the other of the combinations of treatment with iron filings, potassium ferrocyanide, and neutralization. With either method the reduction in volume was approximately 99.8%.

A mixture of neutralized first and second cycle wastes was passed through iron filings at a pH of 4.5-4.8; potassium ferrocyanide was added to the solution and the solution was neutralized. After passage through this process twice the beta activity was reduced from 125,000 c/min./ml. to 5500 c/min./ml. The majority of this 5500 counts was due to zirconium and columbium. When this supernatant was treated with tannic acid, the remaining activity was reduced to approximately 300 c/min./ml.

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## APPLICATION OF THE ABOVE PROCESS

We believe that the above process can be applied to any aqueous wastes containing fission products. We believe that this will remove the majority of the fission products, particularly when the solution has been neutralized. In cases where a specific element or two gives unusual difficulty, it may be removed by an additional treatment, such as the possible removal of the last amounts of zirconium and columbium with tannic acid.

The first cycle supernatant, with which we have been working, contains no appreciable amount of plutonium so that we have no figures on its possible removal by our process, although it is our belief that an appreciable removal will be obtained.

## PROPOSED FIELDS OF ENDEAVOR IN WASTE PROCESSING

We feel that we can attack the following waste problems:

1. Bismuth phosphate wastes

    a. Crib wastes
    b. First decontamination cycle wastes
    c. Wastes from metal recovery

2. Redox wastes

    a. Aqueous wastes
    b. Hexone wastes

We believe that within the next two months we will be ready to start pilot plant work on the crib wastes on which we are now working (neutralized second decontamination cycle waste) and believe that the same general approach and process can be applied to the other crib wastes, samples of which we hope to obtain in the near future.

Laboratory work is now being carried out on the neutralized first cycle wastes and it is believed that we will be ready for pilot plant work on this waste late in 1950 or early in 1951.

It should be remembered that the general principles of cleaning up the wastes from the bismuth phosphate process can be applied to the redox wastes. The presence of hexone in some of the redox wastes should merely necessitate another step, in which the radioactive material would be transferred to the aqueous phase. The presence of the aluminum nitrate in some of these wastes may cause problems, but we do not feel that they are insurmountable.

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BISMUTH PHOSPHATE PROCESS

The bismuth phosphate process for the separation of plutonium from uranium and fission products is carried out in four principal steps after the uranium slugs have been decanned and dissolved.

1. Most of the uranium (99+%) and approximately 97% of the fission products are removed in the first waste solution, known as the metal waste. This is stored in tanks. A process has been developed at Oak Ridge for the separation of the uranium from this waste. No method has been developed as yet for treatment of the wastes from the metal waste recovery.

2. In the first decontamination cycle approximately 3% of the fission products, 2% of the plutonium and a small amount of uranium are removed. This solution is known as the first decontamination cycle waste. This waste is neutralized and stored in tanks. The decan solution (the aluminum jacket is removed with caustic) is used to neutralize, in part, this waste. Plans have been made to construct an evaporator to concentrate this first cycle waste. The plans are merely on paper and no money for an evaporator has been allocated as yet and will not be allocated before the next fiscal year.

3. The first decontamination cycle is repeated and resulting waste is known as the second decontamination cycle waste. This waste is composed of small amounts of fission products and a very small amount of plutonium. This waste is neutralized and the supernatant sent to crib, the sludge being stored in tanks.

4. The final step is the separation of the bismuth (added as phosphate in previous steps) from the plutonium by means of lanthanum fluoride carrier. The waste from this step contains only plutonium and this in very minute amounts due to the fact that the solution is recycled until the plutonium is practically all recovered. The waste from this step is sent to crib.

The wastes which at present go to crib, including "3" and "4" above, are as follows:

| Waste (see code) | Volume | Beta | | Plutonium | |
|---|---|---|---|---|---|
| | $10^6$ l/yr. | c/yr. | μc/l | gr/yr. | μg/l. |
| 2nd cycle neutralized | 5.0 | 71 | 14 | 8.8 | 1.8 |
| *5-6 | 22.7 | 2690 | 51 | 83 | 3.9 |
| 224 | 6.4 | < 67 | <8.9 | 2.8 | 0.4 |
| *231 | 7.9 | - | - | 34.5 | 4.4 |
| 222 | 9.6 | 23 | 2.4 | 5.9 | 0.6 |
| 234-5 | 9.4 | - | - | 15.9 | 1.7 |
| | 61.0 | | | | |

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CODE

5-6 leakage from canyons and solvent from off-gas scrubber in canyon - low salt content - neutralized and discharged to crib

224 cell washings, recycle waste, etc. - high in solids content

231 low in salt content due to recycling most of solutions

222 laboratory waste - not neutralized

234-5 this is not a bismuth phosphate process waste

* Because of the low salt content, it will probably be profitable to handle these separately from the others.

REDOX PROCESS

The principal advantage of the redox process over the bismuth phosphate process is that in the redox process the raw material (uranium) is separated from the plutonium and fission products so that it can be reused.

The redox process is a solvent extraction process using hexone (methyl isobutyl ketone). After the slugs are decanned and dissolved, the aqueous solution of uranium, plutonium and fission products is passed through an extraction process (Step 1A), counter-current to hexone. In this step a major separation is made of uranium and $Pu^{+4}$ from fission products, the latter being in the aqueous phase.

In the second extraction, (Step 1B) the plutonium is reduced from +4 to +3 and the major separation of plutonium from uranium is made, the plutonium being the aqueous phase.

In the third extraction (Step 1C) the uranium is transferred from the hexone to the aqueous phase.

The $Pu^{+3}$ from the second extraction step is oxidized to $Pu^{+4}$ and fed to the fourth extraction (Step IIA) where the final separation from the fission products is made, the $Pu^{+4}$ going into the hexone phase.

In the fifth extraction (Step IIB) the plutonium is transferred to the aqueous phase.

The above is described in five steps, each step being a separate solvent extraction. It is possible that more than one extraction column will be necessary for each separate step.

Aluminum nitrate is used for the salting out agent in the redox process.

The wastes from the Redox Process are divided into the aqueous and hexone wastes.

The aqueous wastes are the decan solution; the IAW waste, containing the majority of the fission products and a small amount of plutonium; and the IIAW waste, containing a small amount of fission products and plutonium.

The hexone wastes are the ICW waste, containing a small amount of fission products and uranium; and the IIBW waste, containing a small amount of plutonium and a minute amount of fission products.

The five extraction steps are designated as IA, IB, IC, IIA, and IIB, and since W designates waste, it can be seen from which extraction step the above waste came.

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F. C. Mead, Jr.

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