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**CESIUM-137 AS AN INDEX OF THE NUCLEAR
MATERIALS CONTENT OF COATING WASTE**

AUTHOR

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CESIUM-137 AS AN INDEX OF THE NUCLEAR MATERIALS
CONTENT OF COATING WASTE

by

R. A. Schneider

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Research and Engineering
Chemical Processing Department

October 11, 1961

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printed 8/20/94
3/23/78

HANFORD ATOMIC PRODUCTS OPERATION
RICHLAND, WASHINGTON

Work performed under Contract No. AT(45-1) - 1350 between
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CESIUM-137 AS AN INDEX OF THE NUCLEAR MATERIALS
CONTENT OF COATING WASTE

INTRODUCTION

For some time it has been feared that coating waste measurements for nuclear materials may be grossly understated. This fear has stemmed largely from the two-phase nature of the caustic coating solution in which uranium and plutonium are presumed to be present predominantly in the solid phase. It was suspected that the samples of coating waste were not representative of the tank in that they contained a much lower ratio of solids to liquid than did the tank. As a result of these misgivings, a program was undertaken to develop an independent and possibly an improved method of measuring the uranium and/or plutonium content of coating waste.

Since there is no chemical separation prior to coating removal, it was expected that the uranium and plutonium in coating waste would be accompanied by the same ratio of fission products as in dissolver solution. For the same reason, uranium and plutonium could be expected to be present in about the same ratio as in dissolver solution and that a measure of one would be a measure of the other. Based on the premise that the product materials are accompanied by a corresponding amount of fission products, a program was undertaken to evaluate the validity of using fission product concentration as an index of the product content. Since cesium-137 was long-lived, caustic soluble, and its production approximately a linear function of irradiation, it was chosen for evaluation. In addition to these features, several analytical methods for cesium-137 were available which held promise of yielding high precision in routine use.

For the cesium-137 content to be a valid measure of amount of product material discharged to underground storage via coating waste, the following points required experimental demonstration:

- 1) That the cesium concentration be nearly uniform throughout the total volume;
- 2) That the cesium does not migrate into the jacket and hence be present in coating waste without the corresponding amount of product material;
- 3) That there is no separation of cesium-137 from product materials prior to the coating waste sampling tank. It is possible that the solid phase, presumably containing the bulk of the product materials, could be preferentially left in the dissolver as a basic sludge after the transfer from the dissolver to the coating waste sampling tank. If this were the case, estimates by the cesium-137 method of the amount of product discharged to underground storage would be biased high.

SUMMARY AND CONCLUSIONS

An experimental program was carried out to evaluate the validity of using the cesium-137 content of coating waste as an indirect measure of the uranium and/or plutonium content. A small number of large volume samples and a large

number of normal (small volume) samples of Redox and Purex coating waste were used for the investigations. The distribution of cesium, uranium, and plutonium throughout the two-phase system was determined. A comparison of uranium by direct assay and uranium by cesium-137 was obtained for both Redox and Purex samples. A plant test was carried out to determine the extent of cesium migration and the extent to which product materials are transferred from the dissolver to underground storage in coating waste.

Typical coating waste samples were found to contain from 4-12% settled solids and clear liquid phases. The cesium concentration was found to be nearly uniform throughout the total volume, whereas uranium and plutonium were found to be present almost entirely in the solid phase. Cesium migration into the jacket was found to be below the significance level and for present purposes may be disregarded. In normal plant operations, it appears that most of the product materials in coating waste are transferred from the dissolver to underground storage. As yet, no metallic uranium fines or undissolved uranium oxide have been found in coating waste samples. Product materials in coating waste appear to originate largely from UNH solution left with the metal heel after acid dissolution. Little product material was found to be introduced in the jacket removal step or by the action of the coating removal solution on the metal heel. For Redox samples, uranium values by direct assay were found to agree with uranium values computed from the cesium-137 content within 40%. However, Purex direct uranium values were a factor of 4 lower than those computed from the cesium-137 values.

Sampling for cesium-137 was found to be markedly more consistent than for product materials. In general, the cesium-137 content was found to be a more sensitive measure of the relative product content of coating waste than the uranium content as measured by direct assay.

To date all the evidence indicates that the cesium-137 content of coating waste is a valid measure of the product content. However, more "back-up" data and a "routine use" evaluation are needed before the method can be considered for use as an official accountability method.

At the present the principal value of the method is as an estimate of the maximum possible product content of coating waste. Because of its high sensitivity, the method should also be valuable for detecting small changes in product content.

RECOMMENDATIONS

It is recommended that the cesium-137 method of measuring the product content of coating waste be evaluated on a "routine basis" for a period of three to six months by the Purex and Redox Plants. The program should include the sampling and analysis of individual batches in order to achieve a correlation between processing conditions and analytical results.

The analytical procedure is included as a separate enclosure. The method of relating cesium-137 content to product concentration is discussed on pages 10, 11, and 12.

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EXPERIMENTAL AND DISCUSSION

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Solid-Liquid Phase Distributions

Large volume samples (30-100 milliliters) of Purex and Redox coating waste were obtained and used for determining 1) the relative volumes of the solid and liquid phases, and 2) the distribution of cesium-137 and product materials throughout the two phases. The samples were investigated in the Process Chemistry cave. The volumes of the liquid and solid phases were measured visually by matching the phase level of the sample to the liquid level of a known volume of water contained in a vessel of the same dimensions as the sample container. The liquid phases of the samples were clear and water white. The solid phases were variegated in appearance, presumably precipitated sodium aluminate, mixed with brown or black particles and precipitated UNH. Following visual examination, the liquid phases were sampled and decanted off. The solid portions were then dissolved in nitric acid and sampled. Both phases of all samples were analyzed for cesium-137 and uranium. As a check on previous work, plutonium phase distribution was determined in one sample. The results are shown in Table I.

As is seen in Table I, uranium and plutonium are concentrated predominately in the solid phase, whereas the cesium concentration is nearly uniform throughout the two phases. Because of its even distribution, samples for cesium should be fairly representative of the slurry in the tank despite some sampling variations in the ratio of solids to liquid. In contrast, the product materials are concentrated in about 5-10% of the total volume. For these, sampling variations in the ratio of solids to liquids would be expected to have a marked effect. The between-sample data obtained to date for cesium-137 and uranium is shown in Table II. As shown by the data, the total variation (sampling plus analytical) for cesium appears to be largely analytical variation, whereas for uranium most of the variation appears to be due to sampling. It should be noted that sampling variation includes both the sampling of slurry in the tank and the sampling of the slurry again in the laboratory.

Tests for Uranium Metal Fines

Two experimental approaches were used to test for the presence of uranium metal fines or oxides in coating waste. These approaches are based on the assumption that the cesium-137 to uranium ratio would be the same in undissolved fines as in dissolver solution. In the first method the separated solid phases were dissolved in hot concentrated nitric acid and the cesium-137 to uranium ratio was measured. For the large volume samples, the average cesium-137 to uranium ratio of the dissolved solid phases was a factor of 10 lower than in dissolver solutions, demonstrating that only a small fraction of the uranium could be present as metal fines. In addition, part of the cesium found in the solid phase could be accounted for as mother liquor from the original liquid phase. For a more sensitive test for metal fines, the solid phase from a 100 milliliter sample was washed with 2.0 M NaOH. The wash removed the cesium-137 almost

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TABLE I

ANALYSES OF LARGE VOLUME SAMPLE OF COATING WASTE

<u>Sample</u>	<u>Volume</u> <u>% of total volume</u>	<u>U</u> <u>#/gal</u>	<u>Pu</u> <u>g/gal</u>	<u>Cs-137</u> <u>μc/gal</u>	<u>Volume</u> <u>% of total vol</u>	<u>U</u> <u>#/gal</u>	<u>Pu</u> <u>g/gal</u>	<u>Cs-137</u> <u>μc/gal</u>
1	3.6	0.336	-	$\sim 4.0 \times 10^4$	96.4	3.3×10^{-4}	-	3.0×10^4
2	11.9	0.126	-	$\sim 2.0 \times 10^4$	88.1	1.9×10^{-4}	-	3.1×10^4
3	10	0.049	1.4×10^{-2}	1.4×10^4	90	5.7×10^{-4}	1.5×10^{-5}	2.0×10^4
4	9	0.235	-	1.9×10^4	91	1.9×10^{-4}	-	3.4×10^4

Solid Phase After 2.0 M Caustic Wash

3	8	0.058	1.4×10^{-2}	$< 6 \times 10^2$
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TABLE II

BETWEEN SAMPLE VARIATION FOR URANIUM AND CESIUM-137
FOR DUPLICATE SAMPLES OF COATING WASTE

<u>Coating Waste</u> <u>Batch</u>	<u>Sample</u>	<u>U</u> <u>#/gal</u>	<u>Cs-137</u> <u>μc/gal</u>	<u>Range</u> ⁽¹⁾ <u>U, %</u>	<u>Range</u> ⁽¹⁾ <u>Cs-137, %</u>
1	1	8.2×10^{-2}	3.8×10^4	150	6
	2	2.0×10^{-2}	3.7×10^4		
	3	2.1×10^{-2}	3.6×10^4		
2	1	1.9×10^{-2}	6.2×10^3	123	3
	2	4.5×10^{-3}	6.0×10^3		
3	1	1.0×10^{-2}	3.6×10^4	37	3
	2	6.9×10^{-3}	3.7×10^4		
4	1	1.0×10^{-2}	3.2×10^4	57	6
	2	1.8×10^{-2}	3.4×10^4		
5	1	2.3×10^{-2}	3.3×10^4	30	6
	2	1.7×10^{-2}	3.1×10^4		

(1) Range of cesium-137 and uranium values for samples of the same batch expressed as per cent of the mean value. Analytical variation for cesium-137 is estimated to be ± 5 and for uranium $\pm 10\%$.

TABLE III

CESIUM MIGRATION-PRODUCT RETENTION TEST

<u>Process Conditions</u>	<u>Redox B-2 Dissolver</u>		
	<u>Solution volume</u> <u>gallons</u>	<u>Total U by</u> <u>Direct assay, lbs.</u>	<u>Total U by</u> <u>Cs-137, lbs.</u>
Before coating removal	823	27	19.7
After coating removal	964	21	23.8
After transfer to H-10	113	1.4	--

entirely while removing only traces of uranium. On the basis that uranium metal or oxide is not soluble in caustic, it was concluded that uranium metal fines or oxides were not present in measurable amounts.

Cesium Migration and Product Hold-up

An estimate of the extent of cesium migration into the jacket was made through a special plant test using the Redox B-2 dissolver. The metal heel was removed with nitric acid and the dissolver was charged with 16,400 pounds of metal. Large volume samples were then taken of the pre-coating removal solution and the after-coating removal solution. The cesium contributed by coating removal was determined by measuring the increase in the total cesium-137 in the dissolver. The amount of cesium-137 introduced by coating removal corresponded to the dissolution of about four pounds of uranium. This corresponds closely to the amount of uranium expected to be dissolved (0.02 - 0.04% of through-put) in coating removal.

From this fact and the small increase observed for total cesium-137, it was concluded that the cesium migration, if real, is small enough to disregard for present purposes. The test data is shown in Table III.

The correlation obtained between cesium-137 and uranium for a large number of coating waste samples also indicates that cesium-137 migration is not a major effect.

During the special test an estimate was also obtained of the amount of product materials which could be left in the dissolver as a basic sludge after a coating waste batch is transferred out. The coating waste solution in the special test described above was allowed to settle in the dissolver for 15 minutes and then transferred out without agitation to the sampling tank (H-10). In normal operations, the coating solution is kept agitated and jetted out with agitation to effect complete removal. However, in the special test a 15-minute settling period was used to obtain an estimate of the maximum amount which could be left. After jet-out, water was added to the dissolver, agitated, and a sample obtained. The results of this test showed that about 5% of the uranium remained in the dissolver as a basic sludge. The test data is shown in Table III. Since this small fraction was obtained using a special settling period, it was concluded that in normal operations the bulk of the product material is transferred out of the dissolver rather than remaining as a basic sludge.

From a recovery standpoint, it would be desirable to have most of the product remain in the dissolver as a basic sludge and be recovered during the succeeding acid dissolution. However, from a processing standpoint the silicon and other constituents of the sludge are not desirable.

Direct Uranium Assay Versus Cesium-137

Comparisons between uranium by direct assay and uranium computed from cesium-137 were obtained on both Redox and Purex samples. Redox samples were based on weekly composites of individual batches. The data is shown in Table IV for

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Redox samples and in Table V for Purex samples. As is shown by the data, direct uranium assay value for Redox samples agree within about 30-40% of the value computed from the cesium-137 content, whereas the uranium assay values for Purex are lower than those computed from the cesium-137 by a factor of about 4. As yet, there is no solid basis for explaining the good agreement found for Redox samples and the very poor agreement found for Purex samples. It is speculated that the difference observed is due to a sampling bias in that the Purex samples contain a smaller portion of uranium bearing solids than do the Redox samples.

Source of Product Materials

To date, all the evidence indicates that the product materials in coating waste originate mainly from the UNH material left with the metal heel after acid dissolution. Based on the cesium-137 values, Redox product losses via coating waste are about 0.16% of through-put. Of this, about 0.02 - 0.03% of through-put is contributed by the jacket removal step. Since there is some uranium introduced into the jacket in canning, this is not surprising. The action of caustic on the metal heel is also expected to contribute only a small fraction of the total loss. As evidence of this, a metal heel was left in hot coating removal solution for 48 hours and found to have a normal cesium-137 and uranium content.

Originally it was thought that the lowest uranium and cesium-137 values would be observed on the first coating waste batch following a dissolver clean-out. It has been found true that the lowest cesium-137 and uranium values have been observed on the first batch after heel removal. However, at the same time, some of the highest values for both uranium and cesium-137 have been found for the same conditions (See tables IV and V). This would indicate that heel removal allows the opportunity to flush the dissolver free of UNH solution prior to coating removal, but that in some cases flushing is not always complete.

Analytical

Uranium values on all samples were measured on acidified aliquots of coating waste using the hexone extraction - fluorimetric method, UF-1b. Cesium-137 assays initially were done using a cobalticyanide precipitation separation coupled with a gamma energy analysis and later by a tetraphenyl boron extraction separation which was also coupled with a gamma energy analysis. This latter method was adopted for routine use and most of the values shown in Tables IV and V were obtained by the tetraphenyl boron method.

The tetraphenyl boron method obtained from A. C. Leaf of the Hanford Laboratories operation was found to give a recovery of about 99% for cesium-137 in dissolver solution. Counting disks were essentially pure cesium with a ratio of about 9 to 1 of cesium-137 to cesium-134. On coating waste samples, the method gave about a 96% recovery.

Some carry-through of Zr, Nb-95 was occasionally noted on coating waste samples. For the worst case, this amounted to only about 10% of the cesium-137 activity.

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TABLE IV

REDOX COATING WASTE-URANIUM BY CESIUM-137 VERSUS URANIUM BY DIRECT ASSAY

<u>Serial No.</u>	<u>Cs-137 μc/gal</u>	<u>U by assay #/gal (1)</u>	<u>U by Cs-137 #/gal (2)</u>
6070-X	2.4×10^4	F ₁ 1.9×10^{-3}	1.6×10^{-2}
6141-X	2.5×10^4	1.4×10^{-2}	1.7×10^{-2}
6247-X	2.9×10^4	1.4×10^{-2}	1.9×10^{-2}
6507-X	3.3×10^4	2.3×10^{-2}	2.2×10^{-2}
6517-X	3.0×10^4	1.7×10^{-2}	2.0×10^{-2}
6567-X	3.1×10^4	1.7×10^{-2}	2.1×10^{-2}
6830-X	3.8×10^4	1.6×10^{-2}	2.9×10^{-2}
6907-X	3.1×10^4	1.2×10^{-2}	2.1×10^{-2}
6990-X	3.2×10^4	1.3×10^{-2}	2.1×10^{-2}
7014-X	3.4×10^4	1.3×10^{-2}	2.3×10^{-2}
7032-X	4.1×10^4	2.4×10^{-2}	2.7×10^{-2}
7178-X	4.2×10^4	2.7×10^{-2}	2.8×10^{-2}
7386-X	4.9×10^4	2.5×10^{-2}	3.3×10^{-2}
7482-X	5.2×10^4	2.1×10^{-2}	3.5×10^{-2}
7538-X	4.7×10^4	1.6×10^{-2}	3.1×10^{-2}
7707-X	4.2×10^4	1.8×10^{-2}	2.8×10^{-2}
7787-X	3.9×10^4	1.6×10^{-2}	2.6×10^{-2}
7914-X (5)	2.8×10^4	1.6×10^{-2}	1.8×10^{-2}
7929-X	5.0×10^4	2.4×10^{-2}	3.3×10^{-2}
7958-X	4.7×10^4	3.3×10^{-2}	3.1×10^{-2}
8027-X	4.9×10^4	1.9×10^{-2}	3.3×10^{-2}
8073-X	4.0×10^4	1.8×10^{-2}	2.7×10^{-2}
8199-X	4.0×10^4	2.0×10^{-2}	2.6×10^{-2}
8204-X	3.5×10^4	1.8×10^{-2}	2.3×10^{-2}
8224-X	3.5×10^4	2.9×10^{-3}	2.3×10^{-2}
8318-X	4.1×10^4	2.5×10^{-2}	2.8×10^{-2}
8379-X	4.1×10^4	F ₂ 7×10^{-3}	2.7×10^{-2}
8470-X	3.3×10^4	F ₃ 3.2×10^{-3}	2.2×10^{-2}
8548-X	2.7×10^4	F ₄ 4.7×10^{-3}	1.8×10^{-2}
8747-X	3.9×10^4	2.1×10^{-2}	2.6×10^{-2}
8888-X	4.2×10^4	2.9×10^{-2}	2.8×10^{-2}
8889-X (5)	3.78×10^4	8.2×10^{-2}	2.5×10^{-2}
8890-X (5)	3.66×10^4	2.0×10^{-2}	2.4×10^{-2}
8900-X	3.2×10^4	F ₅ 9.2×10^{-3}	2.1×10^{-2}
9066-X	4.8×10^4	1.1×10^{-2}	3.2×10^{-2}
9093-X	4.0×10^4	2.4×10^{-2}	2.7×10^{-2}
9240-X	3.9×10^4	1.3×10^{-2}	2.6×10^{-2}
9445-X	3.2×10^4	1.3×10^{-2}	2.1×10^{-2}
9497-X	3.4×10^4	1.8×10^{-2}	2.3×10^{-2}
9567-X	3.2×10^4	1.1×10^{-2}	2.1×10^{-2}
9650-X	3.9×10^4	1.0×10^{-2}	2.6×10^{-2}
9751-X	3.7×10^4	1.3×10^{-2}	2.5×10^{-2}
9798-X	3.8×10^4	F ₆ 7.4×10^{-3}	2.5×10^{-2}
9831-X	3.6×10^4	1.0×10^{-2}	2.4×10^{-2}
9945-X (5)	2.2×10^4	8.5×10^{-3}	1.4×10^{-2}
14-X	3.4×10^4	1.2×10^{-2}	2.3×10^{-2}
136-X	3.0×10^4	2.0×10^{-2}	2.0×10^{-2}
78-X	3.1×10^4	(3) 1.0×10^{-2}	2.1×10^{-2}
		$\bar{X}_1 = 1.7 \times 10^{-2}$	$\bar{X} = 2.5 \times 10^{-2}$
		$\bar{X}_2 (4) = 1.9 \times 10^{-2}$	

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Notes for Table IV:

- (1) Uranium values obtained by direct assay using method UF-1b on a 250ml $\rightarrow$ 5ml acid dilution of coating waste.
- (2) Uranium values computed from the cesium-137 assay of the 250ml $\rightarrow$ 5ml acid dilution used for the uranium assay. Calculations were based on a Cs-137/U ratio of 1.5×10^6 μ c of Cs-137 per pound uranium.
- (3) Average value of 48 determinations includes flyers $F_1 \rightarrow F_6$.
- (4) Average value excluding flyers $F_1 \rightarrow F_6$.
- (5) Samples of the first coating waste batch after a dissolver clean-out.

TABLE V

PUREX COATING WASTE - URANIUM BY CESIUM-137
VERSUS URANIUM BY DIRECT ASSAY

<u>Sample (1)</u>	<u>Cs-137 μc/gal</u>	<u>U by Assay⁽²⁾ #/gal</u>	<u>U by Cs-137⁽²⁾ #/gal</u>
July 19 Comp.	3.0×10^4	6.8×10^{-3}	2.4×10^{-2}
July 27 Comp.	3.5×10^4	8.5×10^{-3}	2.8×10^{-2}
August 2 Comp.	2.75×10^4	5.5×10^{-3}	2.2×10^{-2}
August 9 Comp.	3.1×10^4	5.2×10^{-3}	2.5×10^{-2}
August 22 Comp.	3.0×10^4	5.6×10^{-3}	2.4×10^{-2}
Sept. 1 Comp.	3.3×10^4	4.9×10^{-3}	2.6×10^{-2}
Sept. 9 Comp.	3.2×10^4	9.7×10^{-3}	2.6×10^{-2}
		$\bar{X} = 6.6 \times 10^{-3}$	$\bar{X} = 2.5 \times 10^{-2}$

Special Clean-out Samples

5603-A (3)	1.1×10^4	4.5×10^{-3}	8.8×10^{-3}
5616-A (3)	3.8×10^4	1.1×10^{-2}	3.1×10^{-2}
5951-A (3)	3.8×10^4	3.3×10^{-2}	3.1×10^{-2}

- (1) Weekly composite samples Purex coating waste.
- (2) Uranium values obtained by method UF-1b on the acidified coating waste. For composite samples the entire sample from 3-6 milliliters was acidified and assayed for uranium and cesium-137. Calculations were based on a cesium-137/U ratio of 1.25×10^6 μ c/lb U.
- (3) Individual samples representing the first coating waste batch after a dissolver clean-out.

The average ratio of Cs-137/U obtained for Redox dissolver solutions was 1.3×10^6 μc Cs-137/lb U. For the comparisons made in Table IV, this value was adjusted to 1.5×10^6 μc Cs-137/lb U to correspond to the average MWD/T and to allow for some product being left in the dissolver. A measured value of 1.13×10^6 μc Cs-137/lb U was obtained on the Purex July dissolver solution composite. This value was adjusted to 1.25×10^6 μc Cs-137/lb U to allow for some product being left in the dissolver. The measured values correspond to calculated values of 1.0×10^6 Cs-137/lb U for 600 MWD/T and 1.5×10^6 μc Cs-137/lb U for 900 MWD/T.

For the long term use of the method, it is suggested that the Cs-137 to Pu ratio of dissolver solution be used as the experimental basis for conversion of cesium-137 values to product concentration. Theory predicts the Cs-137 to Pu ratio to be approximately constant over the range of present exposures. As a consequence, further dissolver solution assays for Cs-137 should not be necessary once this ratio is solidly established. Cesium-137 concentration in coating waste may then be converted to plutonium concentration by use of the established constant and conversion to uranium concentration may be made from the plutonium concentration using the monthly measured Pu/U ratio.

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