Chloride Content of Rocky Flats Scrub Alloy Twelfth Campaign Solution Following Head End Treatment

by

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SAVANNAH RIVER PLANT

DPSP-88-272-85

August 8, 1988

TO: L. B. CURTIS, 221-F - D. F. CHOSTNER, 221-F

FROM: H. P. HOLCOMB, 772-F *HPH*CHLORIDE CONTENT OF ROCKY FLATS SCRUB ALLOY TWELFTH CAMPAIGN
SOLUTION FOLLOWING HEAD END TREATMENTINTRODUCTION AND SUMMARY

A single batch of dissolver solution from the twelfth Rocky Flats Scrub Alloy (RFSA) campaign has been analyzed for chloride content following head end treatment to reduce its concentration. Scrub alloy buttons were dissolved in Tank 6.4D during July. In subsequent head end processing, chloride was precipitated with mercurous ion added as the nitrate. The precipitate, Hg_2Cl_2 , was coremoved with the gelatin floc via centrifugation.

Duplicate samples from Tank 11.2, containing the head end product, produced excellent agreement between their density measurements and acid analyses, indicating them to be truly representative of the tank's contents. Duplicate aliquots from each of these solutions were analyzed using the turbidimetric chloride method developed in the Separations Technology Laboratory. These resulted in an average chloride value of 53 ppm ($\mu\text{g/mL}$) chloride for the head end product. Relative standard deviation of the measurement was ± 6 ppm ($n = 4$), a precision of $\pm 11\%$. Such a variance is normal at this low chloride level. Since initial chloride values prior to head end averaged 1365 ppm (0.0385M), as analyzed by Laboratories Department, a chloride DF of approximately 26 was obtained. Such a reduced chloride level (to less than 100 ppm) in the treated solution will permit further canyon processing with minimal corrosion.

EXPERIMENTALSamples Analyzed

A pair of samples from Tank 11.2, designated Laboratories #68145 and #68146, were taken on 8/3. Values for density and acidity

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between the samples showed excellent agreement. These samples were therefore considered to be representative. Duplicate aliquots from each sample were analyzed for chloride.

Each of the sample vials, containing approximately 12 mL, measured about 25 mr/hr at 5 cm directly over the capped pyramidal vial. Such a low level is a direct benefit of canyon flushing operations to reduce the very high residual activity from Purex operation. The lowered radiation levels are a necessity due to analytical operations requiring hands-on manipulations.

Samples were very clear in appearance and light tannish-brown in color. No solids were noted by Laboratories during their determinations nor were any noted by us.

Analysis of Chloride

The procedure¹ employed to determine the low values of chloride in the high alpha solutions from Tank 11.2 was also used for all previous RFSA campaigns, except for the first. A visual turbidity comparison was used for that campaign since our improved method had not been developed.

The newer analytical technique employs an instrumental measurement of turbidity formed when silver ion reacts with residual chloride in the sample aliquots. Sample pretreatment negated interferences by other solution components, principally Al^{+3} and Hg^{+2} . The latter, in solution, forms a very strong covalent bond with chloride. This compound ionizes only to a very small extent and, without pretreatment, can be the cause of low chloride results from usual analytical procedures.

Standards, prepared in a radioactively cold matrix closely simulating that of the RFSA solution and containing known quantities of chloride, were concurrently analyzed in like manner to provide a working curve of turbidity versus total chloride in solution.

Results

Individual values obtained for the chloride content of duplicate 1-mL aliquots taken from each Tank 11.2 sample solution are shown in Table 1. Also given is the average of the four results, reported as the analysis value, and the relative standard deviation for the average.

TABLE 1CHLORIDE CONTENT OF RFSA DISSOLVER SOLUTION FOLLOWING HEAD END

<u>Batch 7FHERF-1</u>	<u>Cl⁻ ppm (µg/mL)</u>
<u>#61845</u>	
Aliquot #1	60
Aliquot #2	51
<u>#61846</u>	
Aliquot #1	54
Aliquot #2	45
Average (n = 4)	53
Rel. Std. Dev.	6 (±11%)

DISCUSSION

Previous work² by R. S. Ondrejcin at SRL indicated that in order to protect downstream canyon equipment from chloride attack, the chloride content of RFSA solutions should be less than 100 ppm. This batch from the 12th campaign meets that criterion.

Based on Laboratories' value of 1365 ppm (0.0385M) chloride in the material prior to head end treatment, a Cl⁻ DF of 26 was obtained. Table 2 summarizes the chloride content of the solutions, before and after head end treatment, for those RFSA campaigns for which the turbidimetric technique was used to determine chloride following head end treatment. Pairs of numbers represent different batches of the same campaign processed through head end.

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TABLE 2CHLORIDE REMOVAL SUMMARY FOR RFSA CAMPAIGNS

<u>RFSA Campaign</u>	<u>Date Dissolved</u>	<u>ppm Cl⁻ Prior to Head End</u>	<u>ppm Cl⁻ After Head End</u>	<u>Chloride DF</u>
2nd	12/84-1/85	1600	75	21
3rd	3/85	1000	23	43
4th	9-10/85	1890-1235	75-45	25-27
5th	7/86	2930 (Avg)	62 (Avg)	47
6th	11/86	1525 (Avg)	74 (Avg)	21
7th	4-5/87	1030-850	39-24	26-35
8th	11-12/87	1240	26	48
9th	1-2/88	1350	37	36
10th	2/88	870	28-40	31-22
11th	5/88	1455	41	35
12th	7/88	1365	53	26

These data show that the DF obtained for chloride removal across head end for the 12th campaign is quite acceptable. The quantity of chloride present in the initial dissolver solution has increased to a level approximating those immediately prior to the 10th campaign. The latter had the second lowest chloride content so far. The 5th campaign, with a chloride level about twice that of the 11th or 12th, was successfully processed through head end. Therefore, with that buffer, concerns about increasing chloride should continue to be minimal.

REFERENCES

1. DPSPU-84-272-113, Determination of Low Chloride Values in Rocky Flats Scrub Alloy Solutions and Other Complex Matrices, H. P. Holcomb to J. T. Buckner, Jr., November 29, 1984.
2. Excerpt from talk, "Corrosion Study for Rocky Flats Scrub Alloy Program", by R. S. Ondrejcin, SRL, Dec. 2, 1983.

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