Chloride Content of Dissolver Solution from Rocky Flats Scrub Alloy Ninth and Tenth Campaigns Following Head End Treatment

by

H. P. Holcomb

E. I. du Pont de Nemours and Company
Savannah River Site
Aiken, South Carolina 29808

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INTER-OFFICE MEMORANDUM

SAVANNAH RIVER PLANT

DPSPU-88-272-33

March 8, 1988

ACC #244004

Key Words

Rocky Flats
Scrub
Alloy
dissolver
solution
chloride

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

FROM: H. P. HOLCOMB, 772-F

HPT

INFORMATION ONLY

CHLORIDE CONTENT OF DISSOLVER SOLUTION FROM ROCKY FLATS SCRUB ALLOY NINTH AND TENTH CAMPAIGNS FOLLOWING HEAD END TREATMENT

INTRODUCTION AND SUMMARY

F-Canyon continues to obtain excellent chloride removal from Rocky Flats Scrub Alloy (RFSA) dissolver solution during head end treatment. One single batch of solution from the ninth RFSA campaign, dissolved in January and February of this year, and two batches from the tenth campaign, dissolved in February, have been successfully processed. Following dissolution in Tank 6.4D, chloride was precipitated with mercurous ion added as the nitrate. The precipitate, Hg_2Cl_2 , was concurrently removed with the gelatin floc via centrifugation.

For each of these three batches processed, a set of duplicate samples was obtained from Tank 11.2 or from Tank 13.3, containing the head end product. The samples were preanalyzed by Laboratories Department for density and acidity to ensure them to be representative of the tank's contents prior to chloride analysis by the Separations Technology Laboratory.

For the single batch of material processed from the ninth campaign, the head end product contained 37 ppm ($\mu\text{g/mL}$) chloride, a DF of 36. Relative standard deviation of the measurement was ± 5 ppm ($n = 4$) for a precision of $\pm 14\%$.

The dissolver solution from the tenth campaign contained significantly less chloride, only 870 ppm, than those previously. Due to volume, it was processed through head end in two batches. For the first batch, the head end product contained 28 ppm chloride with a relative standard deviation of ± 3 ppm, or $\pm 11\%$. The product from the second head end batch had 40 ppm chloride with a relative standard deviation of ± 3 ppm, or $\pm 8\%$. DFs were 31 and 22, respectively.

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EXPERIMENTAL

Samples Analyzed

For the single batch from the ninth campaign, a pair of samples, designated as Laboratories #31445 and 31446, from Tank 13.3, were analyzed. The chloride value for this solution, diluted somewhat from the actual head end product in Tank 11.2, was used by F-Canyon Sep Tech to backcalculate the chloride value of the solution in Tank 11.2.

For the first batch from the tenth campaign, samples #47241 and 47242 were analyzed; for the second batch, #47291 and 47293.

Agreement between acidity and density values between each pair was excellent. Therefore, the samples were considered to be representative of the tank's contents.

All samples were very clear in appearance and light tannish-brown in color. No solids were noted.

Analysis of Chloride

The procedure¹ employed to determine the low values of chloride in the high alpha solutions was also used for the 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 ions, principally Al^{+3} and Hg^{+2} . The latter, in solution, forms a very strong covalent bond with chloride. This compound ionized 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 product tank 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.

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TABLE 1CHLORIDE CONTENT OF RFSA DISSOLVER SOLUTIONS FOLLOWING HEAD END

<u>Run 1FDRFS1-3, Ninth Campaign</u>	<u>Cl⁻ ppm (µg/mL)</u>
<u>Tank 13.3, #31445</u>	
Aliquot #1	32
Aliquot #2	37
<u>Tank 13.3, #31446</u>	
Aliquot #1	37
Aliquot #2	27
Average (n = 4)	33
Rel. Std. Dev.	5 (±15%)
Average normalized to Tank 11.2 contents = <u>37 ppm.</u>	
<u>Run 2FDRF1-2 #1 - 1st Batch, 10th Campaign</u>	
<u>Tank 11.2, #47241</u>	
Aliquot #1	32
Aliquot #2	29
<u>Tank 11.2, #47242</u>	
Aliquot #1	27
Aliquot #2	24
Average (n = 4)	28
Rel. Std. Dev.	3 (±11%)
<u>Run 2FDRF1-2 #2 - 2nd Batch, 10th Campaign</u>	
<u>Tank 11.2, #47291</u>	
Aliquot #1	42
Aliquot #2	37
<u>Tank 11.2, #47293</u>	
Aliquot #1	37
Aliquot #2	42
Average (n = 4)	40
Rel. Std. Dev.	3 (±8%)

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DISCUSSION

Previous work² at SRL by R. S. Ondrejcin indicated that in order to protect downstream canyon equipment from chloride attack, the chloride content of RFSA solutions should be less than 100 ppm. All batches from these two RFSA campaigns meet this criterion.

For the ninth campaign, based on Laboratories' value of 1350 ppm (0.038M) chloride in the material in Tank 10.2 prior to head end treatment, a Cl⁻ DF of 37 in Tank 11.2 was obtained.

For the tenth campaign, based on Laboratories' value of 870 ppm (0.0245M) chloride in Tank 10.2 prior to head end, a Cl⁻ DF of 31 was obtained for the first batch, and 22 for the second batch.

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 the chloride following head end. Pairs of numbers represent different batches of the same campaign.

TABLE 2CHLORIDE REMOVAL SUMMARY FOR RFSA CAMPAIGNS

<u>RFSA Campaign</u>	<u>ppm Cl⁻ Prior To Head End</u>	<u>ppm Cl⁻ After Head End</u>	<u>Chloride DF</u>
2nd	1600	75	21
3rd	1000	23	43
4th	1890-1235	75-45	25-27
5th	2930 (Avg)	62 (Avg)	47
6th	1525 (Avg)	74 (Avg)	21
7th	1030-850	39-24	26-35
8th	1240	26	48
9th	1350	37	36
10th	870	28-40	31-22

These summary data show that the DFs obtained for chloride removal across head end for the ninth and tenth campaigns are about average, but quite acceptable. Also, consideration must be given that prior to head end treatment, the chloride content of the tenth campaign was the second lowest yet. Thus, Rocky Flats is doing a good job in removing surface salt from the alloy buttons shipped to SRP.

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