Chloride Content of Dissolver Solution from Rocky Flats Alloy Thirteenth Campaign Following Head End Treatment

by

H. P. Holcomb

E. I. du Pont de Nemours and Company

Savannah River Site

Aiken, South Carolina 29808

INFORMATION ONLY

RECEIVED

OCT 22 1998

OSTI

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

DOE Contract No. _____

This paper was prepared in connection with work done under the above contract number with the U. S. Department of Energy. By acceptance of this paper, the publisher and/or recipient acknowledges the U. S. Government's right to retain a nonexclusive, royalty-free license in and to any copyright covering this paper, along with the right to reproduce and to authorize others to reproduce all or part of the copyrighted paper.

MASTER

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

INTER-OFFICE MEMORANDUM

SAVANNAH RIVER PLANT

Key Words

Rocky Flats
Scrub
Anode Heel
Alloy
Dissolver
Solution
Chloride

DPSP-88-272-104

September 20, 1988

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

FROM: H. P. HOLCOMB, 772-F *HPH*

CHLORIDE CONTENT OF DISSOLVER SOLUTION FROM ROCKY FLATS ALLOY THIRTEENTH CAMPAIGN FOLLOWING HEAD END TREATMENT

INTRODUCTION AND SUMMARY

F-Canyon continues to obtain good chloride removal from Rocky Flats Alloy solution during head end treatment. One single batch of solution from the thirteenth alloy campaign, dissolved earlier this month, has been successfully processed. This was the first campaign to combine Rocky Flats Scrub Alloy (RFSA) with Rocky Flats Anode Heel Alloy (RFAHA) in the same dissolution solution (TA-2-1183). 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 this batch, a set of duplicate samples was initially obtained from Tank 11.2. However, due to repeated acid and density measurements by Laboratories, the volume of sample remaining was insufficient to permit chloride analysis. Samples of the same solution, now transferred to Tank 13.3, were analyzed. During the transfer, a 2% dilution occurred, but this error is not significant due to the larger error in the chloride analysis.

For the single batch of RFSA-RFAHA material processed from the thirteenth campaign, the head end product contained 52 ppm ($\mu\text{g/mL}$) chloride, a DF of 22. Relative standard deviation of the measurement was ± 6 ppm ($n = 4$) for a precision of $\pm 12\%$.

EXPERIMENTAL

Samples Analyzed

A pair of samples from Tank 13.3, designated Laboratories #66879 and #66880, was taken on 9/19. Values for density and acidity

L. B. CURTIS - D. F. CHOSTNER

Page 2

September 20, 1988

between these two samples were in good agreement. So, the samples were considered to be representative of the tank's contents. Duplicate 1-mL aliquots from each sample were analyzed for chloride. Each of the sample vials measured about 30 mrad/hr (25 mr/hr) at 5 cm directly over the capped sample 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 clear in appearance and light tannish-brown in color. No solids were noted in the samples from Tank 13.3. However, in the initial samples from Tank 11.2, a small quantity of white solids was noted in the bottom of each vial. The solids appeared to be mercurous chloride.

Analysis of Chloride

The procedure¹ employed to determine the low values of chloride in the high alpha solutions from Tank 13.3 was also used for all previous RFSA campaigns, except 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 13.3 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.

September 20, 1988

TABLE ICHLORIDE CONTENT OF RF ALLOY DISSOLVER SOLUTION AFTER HEAD END

<u>Batch 9FDRFS-1-3</u>	<u>Cl⁻ ppm (µg/mL)</u>
<u>#66879</u>	
Aliquot #1	47
Aliquot #2	53
<u>#66880</u>	
Aliquot #1	60
Aliquot #2	50
Average (n = 4)	52
Rel. Std. Dev.	6 (±12%)

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 13th campaign meets that criterion.

Based on Laboratories' value of 1150 ppm (0.0325M) chloride in the material prior to head end treatment, a Cl⁻ DF of 22 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.

L. B. CURTIS - D. F. CHOSTNER

Page 4

September 20, 1988

TABLE 2CHLORIDE REMOVAL SUMMARY FOR RF ALLOY CAMPAIGNS

<u>Alloy 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
13th*	9/88	1150	52	22

*Combined RFSA-RFAHA Campaign

These data show that the DF obtained for chloride removal across head end for the 13th campaign is quite acceptable. However, the quantity of chloride present in the initial dissolver solution was reduced due to the inclusion of RFAHA, which has essentially no chloride associated with it. The chloride value obtained following head end was equivalent to that in the previous campaign. The DF was slightly less.

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.

HPH/h