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ANALYTICAL PROCEDURE FOR:

DETERMINATION OF TUBALLOY IN SOLID SALVAGE RESIDUES

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ABSTRACT

Test results indicate that the hydrofluorination method for the analysis of tuballoy in solid salvage residues yields satisfactory results with a high degree of accuracy; while a modification of the procedure gives good results in approximately half the time.

Two other procedures for the determination of tuballoy in highly siliceous residues were used in these studies for comparing the results; however these are only briefly considered.

INTRODUCTION

While the determination of tuballoy in siliceous residues has been investigated by many workers, our attention was drawn to the possible use of hydrofluorination in tuballoy analyses by the report "Analysis For Tuballoy By Fluorination" by A. Milch[1]; in which hydrofluorination is followed by fluorination; however, after exhaustive tests on beta-residue samples, the procedure was found to be time consuming and inadequate for routine work.

After further investigation on Beta-residues, it was decided to continue using hydrofluorination but eliminating fluorination, and substituting instead a procedure which entailed subsequent reworking of the remaining residue. Since this procedure was found to work quite satisfactorily on all residue samples; five solid, salvage residue samples were obtained from Mr. D. P. Krause of Department 190 for the purpose of de-

termining the suitability of the hydrofluorination procedure, for possible adoption as a standard method in the Beta Analytical Department.

EXPERIMENTAL METHODS

HF Procedure

Determinations which were made by the complete hydrofluorination procedure (Monthly Progress Report, Department 195)[2] on solid, salvage samples submitted by Department 190 gave the following test results.

Lab. No.	Requisition No.	Material	% 1320
9335	910847	L. S. Solid	3.5
9336	910875	Carbon	48.0
9337	910786	Process liquid waste	1.67
9338	910792	Salvage Wash	1.68
9339	910884	Baffle strippings	0.63

Modified HF Procedure

Depending on the accuracy required and the time delegated to each sample, it is believed that the time per analysis could be tremendously reduced if the residue remaining after the first leach with 6N HNO_3 is discarded. This will eliminate fusions and subsequent reworking of the residue (steps 13-18 of procedure listed on page 5). The following results were obtained and the comparisons noted:

Lab. No.	% 1320 in Leach	% 1320 in Residue	Total 1320, %	% Error if Residue is discarded
9335	3.42	0.08	3.50	2.3
9336	47.6	0.40	48.0	0.80
9337	1.61	0.06	1.67	3.5
9338	1.65	0.03	1.68	1.8
9339	0.47	0.16	0.63	25.4

In the above procedure, five samples were hydrofluorinated at one time; however, there is no necessity to restrict the reactor size to hold

only five samples. The size of the reactor will depend primarily on the influx of samples per day. To insure validity of the results, all residues before they are discarded should be checked by the count method.

Two more procedures were used as a basis of comparison; however, these will be covered only lightly as most of the emphasis was placed on the hydrofluorination method.

Prolonged Leach Procedure

The procedure, which was followed in the analysis of residue sample No. 9335 is: The residue is treated with HCl and HNO_3 acid and after prolonged digestion, all the solids went into solution. The usual R_2O_3 precipitation and electrolytic purification, followed by precipitations with NH_4OH are made, and the results obtained are as follows:

Sample No.	Hydrofluorination Procedure	Prolonged Leach with HNO_3 and HCl
9335	3.50	4.67

The result obtained by the prolonged leach procedure is incorrect, due to the incomplete removal of impurities. The residue was discarded before the purity of the precipitate had been ascertained.

Leach Fusion Procedure

The procedure used on residue sample No. 9336 is: The residue is leached with HNO_3 and HCl for 1/2 hour, filtered and washed, the residue is ignited and fused with Na_2CO_3 and placed in solution with HCl. Both solutions are combined and dehydrated, digested with HCl, filtered and washed. The precipitate remaining is ignited, treated with HF and H_2SO_4, fused with pyro-sulfate, and both solutions combined. A double ammonia precipitation followed by a double ammonium carbonate separation is made

on the solutions, followed by electrolysis for complete removal of impurities. The tuballoy is precipitated with NH_4OH, filtered, charred, ignited and weighed as T_3O_8. Comparative results are as follows:

Sample No.	1. Complete HF Procedure	2. Modified HF Procedure	3. Leach Fusion Procedure
	% 1320	% 1320	% 1320
9336	48.0	47.6	48.2

RECOMMENDED PROCEDURE

The complete hydrofluorination procedure is as follows:

1. Weigh 2 gram of the dried sample in a nickel boat.
2. Place sample in monel reactor.
3. Bring temperature up to 650°C and turn on dry nitrogen.
4. Turn off nitrogen when temperature reaches 650°C.
5. Turn on HF.
6. Pass HF over sample for 3 hours holding the temperature at 650°C.
7. After 3 hours turn off HF.
8. Turn off rheostat.
9. Turn on nitrogen until system is free of HF.
10. Turn off nitrogen and remove sample when furnace is cool.
11. Brush sample carefully into a 250-ml beaker.
12. Leach for one-half hour with 6N HNO_3.
13. Filter and fuse remaining residue with carbonate combining both solutions. (The carbonate fusion may not place all the material in solution, nevertheless, every-

thing should be combined).

14. Evaporate solution and dehydrate residue with perchloric acid.

15. Filter and treat residue with HF, and make a pyrosulfate fusion.

16. Take residue up in HNO_3, filter, and the residue remaining is volatilized by the addition of ammonium iodide at 450°C.

17. Make a pyro-sulfate fusion on the final remaining material and take-up in HNO_3.

18. Combine all solutions.

19. Make 2 ammonia precipitations.

20. Electrolyze for complete removal of impurities.

21. Make solution up to volume and run polarographically or gravimetrically depending on the amount of tuballoy present.

CONCLUSION

The modified hydrofluorination procedure is recommended as being the best in so far as time consumption per sample and the accuracy attainable with the least amount of difficulty. If this procedure is used on a routine basis, a provision should be made for checking all discarded residues by the count method to insure complete validity of the results.

The complete hydrofluorination procedure will require more time per analysis, but a higher degree of accuracy will be obtained.

The use of either procedure No. 1 or No. 2 will depend primarily on the laboratories using the method.

ACKNOWLEDGEMENTS

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REFERENCES

1. A. E. Milch, USA

2. Monthly Progress Report, Department 195, C-3.100.24

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