भारतीय मानक ब्यूरो
BUREAU OF INDIAN STANDARDS

Doc. No. : PRTD/AR/**PF:03**	Issue No. : 2	Issue Date 30 Sept. 2020	**Report of Action Research**

1.	**Action Research Project No.** *(as assigned by PRTD)*	AR/0013
2.	**Title of the Action Research Project**	Recovery of Silver from Nitric Acid (parting acid)
3.	**Name & Designation of Officer**	Muninarayana R, Sc. D, SROL
4.	**Employee No.**	065307
5.	**Deptt./BO/RO & Place of Posting**	SROL
6.	**Date of Approval of the Project**	08 May 2020
7.	**Objective of the Project**	1. To establish % recovery of Silver from parting acid by different methods. 2. To establish the best method that could yield the highest purity of Silver 3. To establish cost incurred in recovery by different methods.
8.	**Report of Action Research Activities**	Report attached as Annexure-1
9.	**Conclusion & Recommendations**	Mentioned in the attached report please.
10.	**Any other relevant information**	Nil

Note: Suggestions from HMD & PRTD have been included in this final report. Therefore, my earlier report dated 16/11/2020 may please be ignored.

Muninarayana R
Date 12/02/2021
12 Feb 2021

HSROL 12/02/2021
Sc.G & DDGL
DDG(PRT)

Annexure-1

a) Table of Contents

a) **Introduction (Action Research Project Proposal to be clearly mentioned)**

During testing of Gold for its purity as per IS 1418, Silver is added around 2.3-3 times of fine Gold content in the sample. Therefore, around 83 gms of Silver is added for testing 100 samples (in 10 batches) of Gold of fineness 916 along with 4 proof Assay samples in each batch.

The referral Assay lab of SROL is testing around 350-400 samples/month and the quantity of silver added to test these may samples would be 290-330g/month. The added silver along with the silver already present in the samples is removed in parting process in Nitric acid.

The silver present in the nitric acid may be extracted which otherwise goes as waste during neutralization and disposal of nitric acid.

Besides generating revenue recovery of Silver from parting acid reduces environmental pollution.

The aim of the project is to adopt a suitable procedure for extraction of silver from Nitric acid. In this project, 3 methods have been employed to recover Silver from Nitric acid (parting acid).

b) Review of Literature (Background research/Literature Survey/any other means etc)

i) Journal of Chemical Education: Recovery of Silver from Laboratory Wastes
ii) Separation and purification Technology : Solvent Extraction of Silver from Nitric Acid solutions
iii) A Simple procedure for recovery of Silver from Silver Chloride using Thiourea, published in Anais Assoc.Bras.Quim.,48(I),43-45,1999
iv) Recovery of Silver from Silver Chloride Residues, Dept . Of Chemistry, University of Northern Iowa
v) 911 Metallurgist : How to Recover Silver from Nitrate Solutions
vi) Gold Refining Forum.com

c) Methods & Materials, Data, Details of Field Visits for studies & research etc.

Method- 1

Initially, Silver Nitrate contained in Nitric Acid converted to Silver Chloride solid by the slow and batch wise addition of Sodium Chloride, allowed to stand for 2 to 3 days to precipitate completely and the Nitric Acid layer is decanted to other container, neutralized the acid cautiously with dilute Sodium Hydroxide till effervescence off (pH 7.0) and discarded.

The Silver Chloride precipitate formed was washed thoroughly with distilled water. To the solution of Silver Chloride in distilled water, 28-30% Ammonia was added slowly while stirring until the dissolution was complete. Then, 0.94 M Ascorbic acid was added until no more silver was formed. The silver formed was allowed to settle and ammonical solution was decanted and saved for reuse. The silver was filtered off in a Buchner funnel and washed three times with distilled water.

The silver was allowed to dry in oven at 110 ^{0}C for 2 hrs. The dried mass is ransferred to graphite crucible, placed in Muffle Furnace at 1050 ^{o}C (for 30 min). When melting is complete, the metal is removed from the crucible. The advantage of using this method is use of inexpensive reagents and equipments; recovered silver is pure with all the effluents being biodegradable. The silver yielded was tested at NABL accredited Laboratory and the results obtained was 999.8 ppt for fineness which was free from Gold and platinum Group metals.

Flow Chart

Silver Nitrate (contained in Nitric Acid)

↓ ← Sodium Chloride

Silver Chloride allowed to Settle for 2-3 days, Nitric acid layer is decanted

↓

Washing Silver Chloride with distilled water

↓

Addition of Ammonia till dissolution of Silver Chloride is complete

↓ ← Addition of 0.94M Ascorbic acid till formation of Silver stops

Silver is allowed to settle

↓

Filtration

↓

Drying in Oven (110 ^{0}C for 2 hrs)

↓

Melting in Muffle at around 1050 ^{0}C for 30 Min

↓

Pure Silver

Method- 2

Sodium Chloride is added to the parting acid containing Silver to precipitate out Silver Chloride. The precipitate is washed several times with water and dilutes nitric acid to remove the impurities such as copper, nickel, lead etc if present. Again it is washed with distilled water.

Transfer the precipitate to beaker using water and cover it with Dilute nitric acid and then add thiourea solution (100g/L) or thiourea crystals agitating manually until precipitate is dissolved.

Add dilute Sod. Hydroxide until the Solution becomes slightly alkaline. Immediately black silver is precipitated. After a few minutes filter the precipitate and wash with water, dry in oven at around 110 ^{0}C for 2 hrs and finally calcinate the silver sulfide in Graphite Crucible at 1050^{0}C for 30 Min. After cooling metallic silver is removed. The silver yielded was tested under XRF and the result obtained was 980ppt for fineness.

Flow Chart

Parting acid containing Silver

↓ ← Sodium Chloride

Washing with Water, dilute Nitric acid & Water

↓

Transfer the Precipitate to beaker and cover it with Dilute Nitric Acid

↓

Add thiourea solution (100g/L) or thiourea crystals agitating manually until precipitate is dissolved.

↓

Add dilute Sod. Hydroxide until the Solution becomes slightly alkaline

↓

Black silver Sulfide is precipitated

↓

Filter the precipitate

↓

Wash with Water

↓

Drying in oven at 110 ^{0}C for 2 hrs

↓

Melting at 1050 ^{0}C for 30 min

↓

Silver

Method-3

Sodium Chloride is added to the parting acid containing Silver to precipitate out Silver Chloride. To the precipitate Glucose is added and thorough mixing is done. To the mix caustic is added till evolution of Gas stops. The precipitate is taken onto the muslin cloth and washing of the precipitate is done with hot water.

The precipitate is dried in oven at 110 ^{0}C for 2 hrs and heated at 1050 ^{0}C for 30 min to get Silver. The silver yielded was tested at NABL accredited Laboratory and the results obtained was 999.8 ppt for fineness which was free from Gold and platinum Group metals.

Flow Chart

Parting acid containing Silver ← Sodium Chloride

↓

Silver Chloride allowed to Settle for 48 hrs, Nitric acid layer is decanted

↓

Add Glucose & Caustic to the precipitate of Silver chloride

↓

Washing the precipitate with hot water and filtering in muslin cloth

↓

Drying in oven at 110 ^{0}C for 2 hrs

↓

Melting at 1050 ^{0}C for 30 min

↓

Silver

d) Results &Analysis

Method	Amount of silver added during testing of 100 Gold samples	Amount of silver recovered from parting acid	% Recovery	Purity of recovered Silver as reported by NABL Accredited Laboratory	Cost incurred for recovery (includes Manpower) in Rs.
Method 1	83.184g	78.3g	94.12	999.8 ppt	2300
Method 2	78.29g	83.51g	106.6	980 ppt *	1500
Method 3	81.73g	78.10	95.55	999.8ppt	1500

* result obtained under XRF . Since the purity obtained was less, the sample was not tested at NABL Accredited Laboratory

e) **Summary and Conclusions**

(i) Purity Analysis :

- 999.8 ppt fineness Silver was obtained by method 1 and method 3.
- 980 ppt fineness Silver was obtained by method 2 as per XRF analysis

(ii) Recovery Analysis:

- % recovery by method 1 is 94.12
- % recovery by method 2 is 106.6 which could be due to presence of impurities such as Mg, Lead etc as evident from XRF Analysis
- % recovery is 95.55 by method 3.

(iii) Suitability of Method:

- Method 1 and 3 are suitable for recovery of Silver as both yielded silver of fineness 999.8 which was free from Gold and platinum Group metals.
- Method 2 is not suitable as purity of recovered Silver was lesser.

(iv) Cost-benefit analysis:

Cost efficiency: (Cost of silver recovered/Cost incurred for recovery) x 100

Approximate cost of Silver of 999 purity as on date (20/10/2020) = Rs. 62/gm

Method -1: Cost efficiency = 211%

Method-3: Cost efficiency = 322.8%

For further analysis, details were sought from A & H Centres on the method of extraction of silver employed by them, if any, along with the purity & % of recovery of Silver. 6 responses have been received.

2 A& H centres are following the method 3 specified above for recovery of silver from parting acid. 2 centres are recovering it by using copper rods (which is consumed during the process)

2 centres are not doing recovery by themselves.

f) Recommendations

- The RALs of BIS can adopt the method 1 or Method 3 to recover Silver from Nitric acid for eco friendly disposal of Nitric acid used in parting process of Gold testing. A&H centers may also be suggested to adopt method 1 or 3 to recover Silver from Parting acid
- The recovered silver may be further refined and reused for testing of Gold after testing it for fineness, presence of Gold and Platinum Group elements at a NABL Accredited Laboratory or certified silver may be purchased on buy back basis for recovered silver
- This ARP may be helpful in formulation of a standard on recovery of silver from parting acid.

g) Details of the BIS support availed with justification, bills/vouchers, etc., as relevant

The available equipments and consumables at SROL have been used (2 additional consumables have been purchased at a total cost of Rs. 1484)

2 samples of recovered silver have been tested at NABL accredited laboratory at a total cost of Rs.9440/-

1 TA assisted in the project.

DECLARATION OF ORIGINAL WORK

I, Muninarayana R, Scientist-D, Employee No.065307 hereby declare that the Action Research Project titled

"Recovery of Silver from Nitric Acid (parting acid)" is the original research work done by me. I have not copied from any other Action Research Project or any other work of similar nature and topic done by any person/institution/body either published or yet to be published. Data and information from other sources, used if any, have been with prior permission, wherever required and is duly acknowledged appropriately in the project report submitted by me.

This declaration is made on Friday of 12th Feb 2021

12/02/2021

Sign. of Officer with Date