GOLD ASSAY (FIRE ASSAY METHOD)

This Standard prescribed the cupellation or fire assay method for assaying of gold jewellery/ artefects covered in IS 1417.

GOLD ASSAY(FIRE ASSAY METHOD)

Terminology:

Fine Gold : -It is gold having fineness(part per 1000 ,represented as ‰) 999 and above without any negative tolerance and free from Pb, Hg, Bi, Se and platinum group metals.

Cupellation:-An oxidizing fusion of lead, gold ,silver and associated base metal in a cupel which absorbs the lead oxide along with base metals oxides leaving a lead of gold and silver (along with platinum group metals, if any) on the cupel.

Parting:-Separating of silver from gold by selectively dissolving the silver in acid , using nitric acid.

GOLD ASSAY(FIRE ASSAY METHOD)

- STEP 1:Melting Process

  Put the scrapped metal part of jewellery in charcoal crucible and covered with charcoal powder.
- Put the crucible in melting furnace for 3-5 minutes at the temperature of 1068-1070°C.
- Cool down the metal and make a strip using rolling mill.
- This process make alloy homogeneous in purity.


Graphite Crucible

GOLD ASSAY(FIRE ASSAY METHOD)

- STEP 2:weighing
- XRF the alloy(strip) and note down the percentage of metals present in the strip.
- Duplicate/Analysis Samples:-
- Transfer two samples of the alloy(strip) for each lots between 125 mg to 250 mg weight to the nearest ±0.01 mg.
- Eg 180 mg of two sample for a lot.

GOLD ASSAY(FIRE ASSAY METHOD)

- Pure gold /check gold /fine gold(999.9‰):
- Calculate weight of check gold according to the sample weight and percentage of gold present in the alloy(strip).
- Eg. If the percentage of gold in strip is 916.3(obtained in XRF)
- The check gold(in mg)= $\frac{sample\ weight\ X\ Purity\ of\ gold\ obtained\ in\ XRF}{100}$
-

$$= \frac{180X\ 91.7}{100}$$

$$= 165.06$$

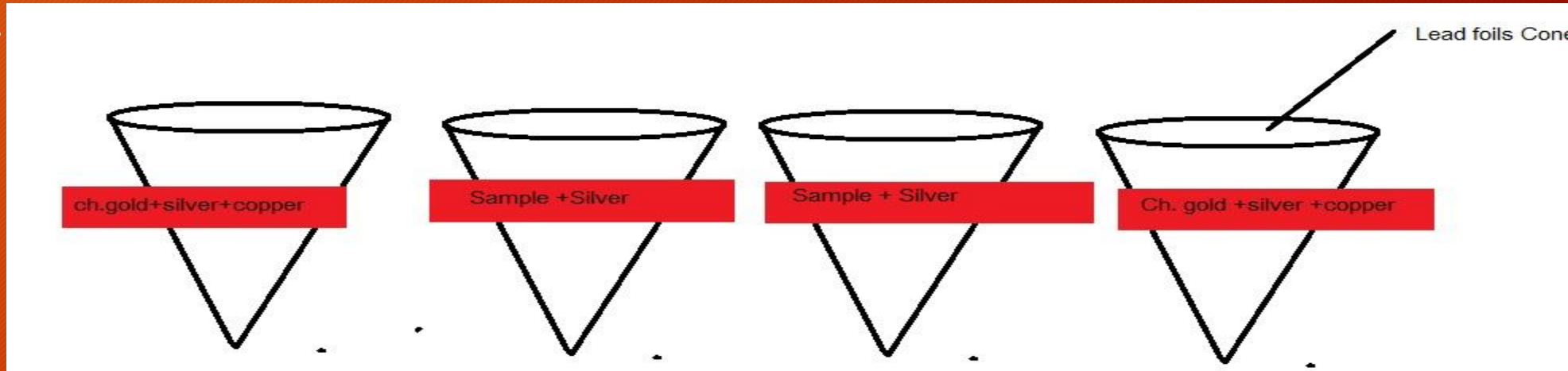
GOLD ASSAY(FIRE ASSAY METHOD)

- Copper :similarly find the weight of copper using sample weight and percentage of copper present in strip.
- Eg :if there is 6% of copper in XRF
- Copper (in mg)=$\frac{180X6}{100}$ = 7.2
- Silver: add 2.3 to 3.0 times wt. of pure gold/check gold.
- Silver (in mg)= 2.5X165.06=412.65.

GOLD ASSAY(FIRE ASSAY METHOD)

- Lead :4 gram (if the sample wt. is less than 200 mg)
- 6 gram(if the sample wt. is more than 200 mg up to 250 mg)
- Here the sample wt. is 180 mg so take 4 gm. of lead.

- Now make the open cone of the lead foil and the metal according to the figure.



GOLD ASSAY(FIRE ASSAY METHOD)

- Table for 4 sample and 2 check gold……

sample	Au	Ag	Cu	Pb
P1(ch. gold)	165.747mg	414.35mg	7.2mg	4gm
S1 (sample)	180.564mg	414.75mg	-	4gm
S2 (sample)	180.385mg	414.82mg	-	4gm
S3 (sample)	180.653mg	414.35mg	-	4mg
S4 (sample)	180.455mg	414.33mg	-	4gm
P2 (ch. gold)	165.726mg	414.12mg	7.2gm	4gm

GOLD ASSAY(FIRE ASSAY METHOD)

- Now make the ball of each cone putting metals inside as describe in the above figure, using balling pliers .

GOLD ASSAY(FIRE ASSAY METHOD)

- STEP 3: Cupellation
- Put all the balls in the pre-heated magnesia cupel in the
- furnace at 1100ºC,
- Put this cupel inside the cupellation furnace.
- Temperature -1050ºC-1100ºC
- Time : 20-30 minutes(Depending on the amount of lead used)
- Maintain oxidizing atmosphere

GOLD ASSAY(FIRE ASSAY METHOD)

- Fixed the temperature of furnace at 800ºC.
- Withdrawal the cuples while temperature come down approx 800ºC.
- After the oxidation is complete cool down the gold and silver button forms and clean it with brush.
- Flattered the button with hammer.
- Anneal the flattered button to red heat at 700ºC for 5 minutes using annealing tray on annealing furnace

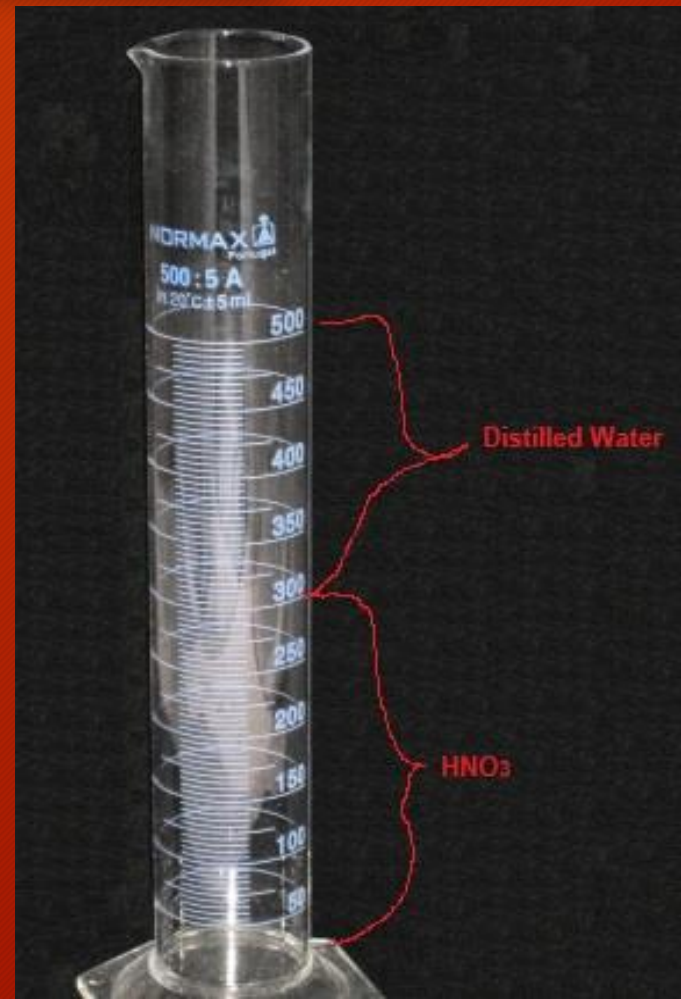
GOLD ASSAY(FIRE ASSAY METHOD)

- Roll them to form strip of 0.12 to 0.15 mm on rolling mill
- Anneal the rolled strip.
- At 700ºC for 5 minutes.
- Roll them to form cornet.
- Keep the rough surface outside without touching the surface of each other layer.

GOLD ASSAY(FIRE ASSAY METHOD)

- Parting process:
- Parting 1: Prepare Dilute nitric acid of 1.2 specific gravity
  (take 200ml of nitric acid+300ml distilled water).

Parting 2: Prepare Dilute nitric acid of 1.3 specific gravity
(take 300ml of nitric acid +200ml distilled water).



GOLD ASSAY(FIRE ASSAY METHOD)

- Now heat the hot plate at 92-95ºC(below 100ºC)
- Dip the parting tray with cornet in parting flask containing parting:1 chemical for 15 minutes.
- Silver dissolve in nitric acid .
- $3Ag(s)+4HNO3(aq) \longrightarrow 3AgNO3(l)+NO(g)+2H2O$
- Now wash the tray with hot distilled water.
- Again repeat the same process in parting :2 for 10 minutes.
- Wash 2 times with distilled water.
- Annealing of cornet:700ºC for 5 minutes.

GOLD ASSAY(FIRE ASSAY METHOD)

- Weight the cornet and note according to their sample number
- Calculations:

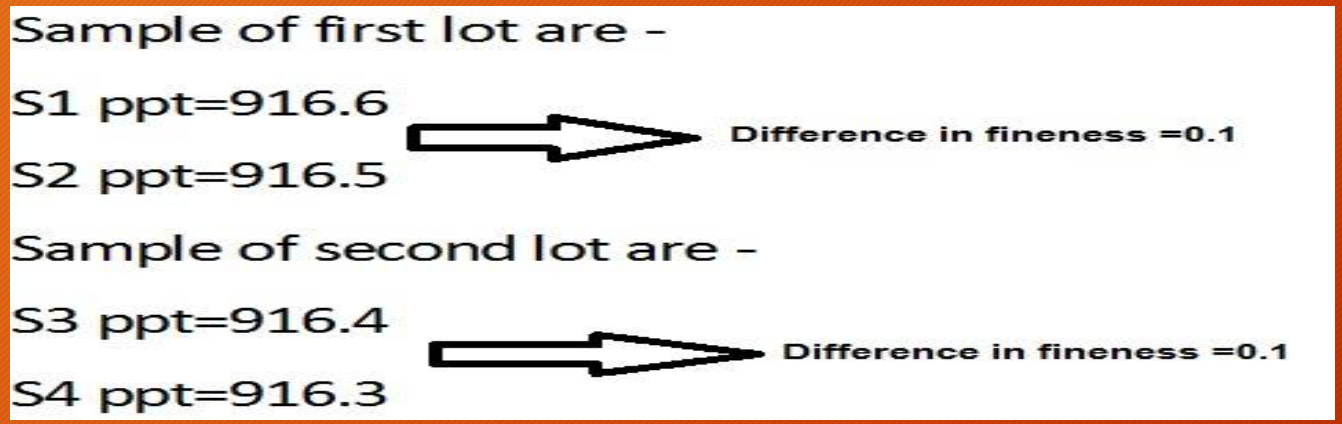
Sample No./Code no.	Initial wt. of sample(mg) m1	Wt. of gold cornet after assaying(mg) m2	Δ (mg) M1-m2=	Avg Δ (mg)
C1(check gold)	165.747	165.763	Δ1=-0.012	
S1	180.564	165.523 ←-S1'		(Δ1+Δ2)/2
S2	180.385	165.340 ←-S2'		
s3	180.563	165.485 ←- S3'		=-0.010
S4	180.455	165.368 ←-S4'		
C2(check gold)	165.726	165.806	Δ2=-0.008	

GOLD ASSAY(FIRE ASSAY METHOD)

- Fineness of Sample:
- sample ppt= $\frac{Final\ Cornet\ Wt. + avg\Delta}{Initial\ sample\ wt.} X1000$
- S1 ppt= [{165.523+(-0.010)}/180.564]X1000
- = 916.64≈916.6
- Similarly
- S2 ppt=916.5,
- S3 ppt=916.4,
- S4 ppt=916.3.

GOLD ASSAY(FIRE ASSAY METHOD)

- Result:



- If the difference in fineness is less than 0.5-→ result OK
- If the difference in fineness is greater than 0.5-→ Repeat the assay
- Now average fineness for first lot is (916.6+916.5)/2=916.55≈916.5
- Now average fineness for second lot is (916.4+916.3)/2=916.35≈916.3