



**Alex Stewart International**

**12<sup>th</sup> INDIA INTERNATIONAL GOLD  
CONVENTION, 2015 . GOA**

**Sampling and Analysis of Dore & Gold Bullion**

**Andrew Smith  
Precious Metals Business Executive  
Alex Stewart International**





# Alex Stewart **International**

## **Brief Introduction to Alex Stewart International Corporation (ASIC)**

Alex Stewart International Corporation (ASIC) has provided inspection and analytical services to the metals and minerals industry for 37 years and has a regional network of companies spanning over 40 countries. Our organization provides fast, flexible and comprehensive inspection and analytical laboratory services with our experienced inspectors regularly attending at the major global precious refineries.

We have a full metals and minerals analytical facility in our UK laboratory which is internationally recognised for both party and umpire analysis, providing either classical wet chemistry and fire assaying or modern instrument methodology i.e. AA, ICP-MS/OES and XRF.

Our operations in India are managed through Stewart Surveyors and Assayers Pvt .Ltd , who are the official and sole representatives in India for the ASIC Group. Their offices are headquartered in Mumbai with a further 10 offices strategically located in the major ports and cities in India.

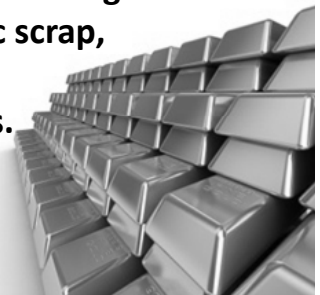
We also have a well-established gold and silver analytical laboratory in Dubai, located in the Gold & Diamond Park.

Our company specialises in the sampling and analysis of an extensive range of precious metal bearing materials including gold & Dore bullion, jewellery, residues, scrap ,catalysts, resins, electronic scrap, PGM gauzes also bullion vault inspections, inventories and audits.

We are also accredited COMEX, LBMA , LPPM, DMCC and IPMI members.

Further information is also available on our website :

[www.alexstewartinternational.com](http://www.alexstewartinternational.com)





# Alex Stewart **International**

## SECTION 1

### Introduction - Sampling Dore & Gold Bullion

Our organization has been witnessing the processing and sampling of Dore and bullion shipments at global precious metals refineries for many years.

The sampling and melting procedures adopted at the various refineries show a surprising and varied choice of procedures which in general can be broken down according to the following main criteria :

- The refiners melting and sampling facilities.
- The Refiners preferred procedures.
- Input material.
- Contractual terms for melting and sampling i.e. maximum lot size or monetary value of a melting lot.
- End usage.

The following is an example of what we consider to be the most commonly practised sampling procedures.





# Alex Stewart **International**

## SAMPLING OF DORE BULLION

As mentioned, there are several different sampling methods utilised for the sampling of Dore & gold Bullion throughout the world, with the various refineries each applying their own preferred method.

Our representatives have experienced numerous methods; therefore we have given our additional comments on the advantages and disadvantages of each method.

We list below the most commonly applied methods of drawing samples from Dore / gold Bullion:

1. Drilling, without melting.
2. Drilling after melting.
3. Sawing.
4. Capillary tube sampling ,(also referred to as vacuum tube or pin sampling).
5. Button (dip) sampling.
6. Shot (grain) sampling

A brief description of each method is as follows:

[www.alexstewartinternational.com](http://www.alexstewartinternational.com)





# Alex Stewart **International**

## 1. DRILLING WITHOUT MELTING

- Weigh bars/ingots.
- Clean surfaces prior to drilling.
- Drill bars in template manner.
- Combine drillings pro-rata for each bar according to the nett weight.
- Mill and screen drillings to produce coarse, medium and fines portions for analysis, (this is optional as some refiners will mix the drillings to form one sample).
- A refinement on this procedure is to re-melt the drillings to produce a single bar which is then drilled again to produce final samples.





# Alex Stewart **International**

## 2. DRILLING AFTER MELTING

- Melt entire lot, remove slag.
- Pour into ingot moulds, cool, then clean and weigh the bars/ingots.
- Drill resultant bars /ingots using the template method.
- Mill and screen drillings to produce coarse, medium and fines portions for analysis; (this is optional as some refiners will mix the drillings to form one sample).
- Analyse the drillings from each bar/ingot separately or prepare a composite sample representing the total melt.
- Again, a refinement of this procedure is to re-melt the drillings to produce a single bar which is then drilled again to produce final samples





# Alex Stewart **International**

## 3. SAWING

- Melt the entire lot.
- Cast into bars/ingots.
- Clean and remove any surface slag
- Weigh bars/ingots.
- Sample bars/ingots using a mechanical reciprocating saw.
- Blend sawings pro-rata according to the after melting nett weight of the bars.
- Alternatively, a percentage of the cast bars will be selected and drawn for sawing.
- Uniform fine sawings are produced for analysis.





# Alex Stewart **International**

## 4. VACUUM TUBE/CAPILLARY TUBE (PIN) SAMPLES

- Melt the entire lot, remove surface slag.
- Insert a sealed glass capillary tube into the molten metal.
- Contact with the molten metal breaks the top of the capillary and the metal is drawn into the tube by vacuum.
- Alternatively, some refiners may draw pin samples from the top (start), middle & bottom stages of pouring the melt .The assay results from the 3 stages of drawing samples are then averaged. Either the individual stages are assayed or a composite sample is prepared, however this will relate to the total after melting weight of each melt and not to the weight of the individual stages of casting i.e. top, middle & bottom.
- The capillary tube produces a thin pin sample which is then either cut into the requisite number of samples or the process is repeated to produce further separately drawn pin samples.





# Alex Stewart **International**

## 5. BUTTON/DIP SAMPLE/XRF PICTURE SAMPLES

- Melt the entire lot, remove slag.
- Draw button/dip /XRF picture samples after stirring.
- Pour into moulds, (normally several individual dip samples are drawn).
- Either use the entire button as a sample or roll and cut or drill to give final sample portions.
- Samples from XRF picture mould samples are normally cut into small sectional pieces for analysis.
- Alternatively, some refiners may draw dip samples from the top (start), middle & bottom stages of pouring the melt .The assay results from the 3 stages of drawing samples are then averaged. Either the individual stages are assayed or a composite sample is prepared, however again this will relate to the total after melting weight of each melt and not to the weight of the individual stages of casting i.e. top, middle & bottom.
- All dip samples should be thoroughly cleaned e.g. by a wire brushing or similar method to remove any surface slag prior to analysis.





# Alex Stewart **International**

## 6. SHOT/GRAIN SAMPLING

- Melt the entire lot.
- A sample is drawn off into either a graphite ladle or pre-heated small crucible (at a similar temperature to the melt).
- A sample is poured into a tank (vat) of water and the molten metal flow agitated by stirring.

Alternatively, some refiners may draw shot/grain samples from the top (start), middle & bottom stages of pouring the melt. The assay results from the 3 stages of drawing samples are then averaged, alternatively the individual stages may be assayed or a composite sample prepared, however this will relate to the total after melting weight of each melt and not to the individual stages of casting i.e. top, middle & bottom.

Alternatively, the molten metal can be poured onto a wooden board suspended in water and hand paddled to assist in the formation of grain.

The granulated metal ("shot") is then collected in a basket.

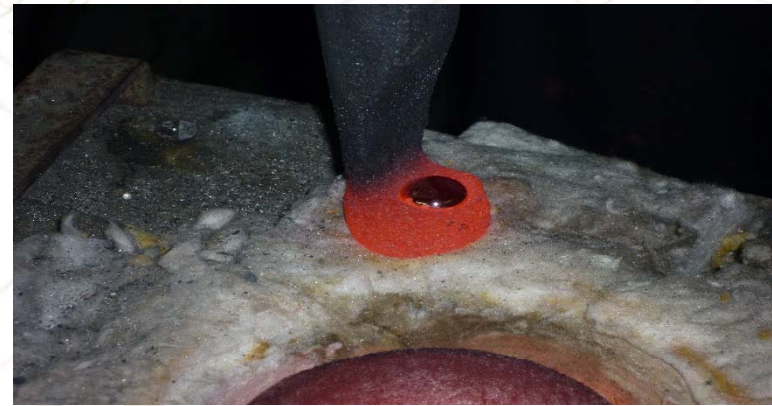
The water is then drained off and the grain is oven dried prior to weighing, mixing and drawing final samples.

There are also more sophisticated and advanced methods of grain sampling systems which produce a very fine; un-oxidised uniformly sized grain, suitable for analysis.





# Alex Stewart International



[www.alexstewartinternational.com](http://www.alexstewartinternational.com)



# Alex Stewart **International**

## SECTION 2

### Our comments on Sampling Methods

- 1 **Method #1 (DRILLING WITHOUT MELTING)**. Drilling all the bars is time-consuming and labour intensive. It also produces more than one fraction for analysis. As the material has not been melted, the ingots may not be homogeneous throughout.  
The ingots may have slaggy inclusions or slag on the surfaces which may be over or under compensated during the drilling process.  
Free slag or iron/magnetic areas on the bars cannot be accurately represented by this method.  
Rough-cast bars with uneven surfaces may cause problems in drilling.
- 2 **Method #2 (DRILLING AFTER MELTING)** is also time-consuming and labour intensive. It also produces more than one fraction for analysis.
- 3 **Methods #1, 2 (DRILLING ) & 3 (SAWING)**, must be carefully controlled in order to avoid contamination by the drill or saw.





# Alex Stewart **International**

## SECTION 2 Cont'd

- 4 **Method #3 (SAWING)**: gives fine, uniform material from a good cross-section of the ingot which is easily mixed to obtain samples for analysis.
  
- 5 **Method #4 (VACUUM TUBE/CAPILLARY TUBE (PIN) SAMPLES)**  
Produces instant samples, requiring less preparation time before analysis. Requires some degree of expertise and experience to ensure a full sample is produced as this method may be unsuitable for some types of Dore especially those with a complex matrix and certain impurities.  
The slag, especially from certain types of Dore with self-generating slag may prevent a continuous metal flow through the glass capillary tube, resulting in a discontinuous or heterogeneous pin sample which may be in the form of several broken pieces.
  
- 6 **Method #5 (BUTTON/DIP SAMPLE/XRF PICTURE SAMPLES)** is probably the most widely accepted method of drawing samples in the molten state, however care should be taken to ensure segregation has not taken place within the button/dip samples.

Refiners will generally cut the samples in half to inspect for any visual internal segregation





# Alex Stewart International

- 7 The final comments on the procedure is method #6 (SHOT/GRAIN)**
- **The base metal present in 'Dore' bullion will cause discolouration in grain also dip samples due to the effects of surface oxidation, sometimes(in the form of a thin film).**
  - **Prior to drawing samples and casting into grain, the slag should be removed from the molten metal surface.**
  - **Manual graining i.e. using a wooden paddle will produce an irregular sized grain, although a consistent metal flow and sideways 'rocking' of the paddle during pouring will produce a reasonably consistent grain. However, agglomerated grain is a common occurrence.**
  - **Uniform fine particle grain can only be produced with the installation of a graining system, whereupon the molten metal flow is bombarded under high-pressure water jets.**
  - **In our opinion, based on our experience attending at the major refiners Dore bullion does not consistently lend itself to the graining procedure.**
  - **The problems related in graining metal is emphasized by the presence of a number of metals contained in the alloy in particular iron/magnetics which can produce segregated grain/shot.**
  - **We would therefore not recommend graining as the best method of sampling Dore. Our preferences would be for button/dip /XRF picture samples and vacuum tube sampling, (only for certain tried and tested Dore shipments), also the sawing of re-melted ingots.**

[www.alexstewartinternational.com](http://www.alexstewartinternational.com)





# Alex Stewart **International**

## SECTION 3

### **3) Dore – Example For Drilling Bars (pre-melt)**

- 1. If possible, document processes with photographs**
- 2. Weigh bars**
- 3. Comment on cleanliness / appearance of bars – e.g. slaggy surfaces, striations in the material, slag inclusions ,porous surfaces or cavities, magnetics, roughness/smoothness etc**
- 4 Clean surfaces of the bars by wire brushing, if any loose/flaking slag is noted, retain and weigh this. Re-comment on appearance of surface after cleaning if different**
- 5 Apply drill template to the bar (see below image)**  
**Measure in approximately 10mm from the corners for the two corner drilling points**  
**Draw line from remaining corners to find centre-point at intersection**

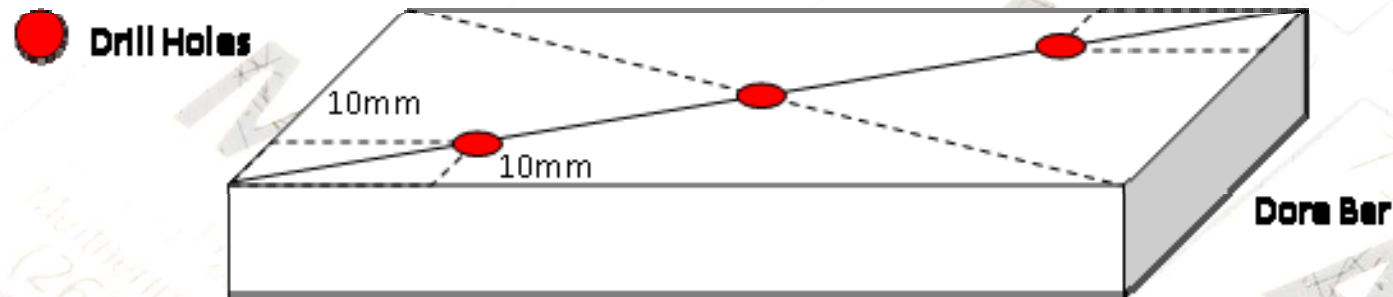




# Alex Stewart International

## SECTION 3 Cont'd

### EXAMPLE OF DRILLING TEMPLATE



6. Select approximately a 5mm drill bit, (standard alloy) and ensure this is thoroughly clean.
7. Drill the bars at the points marked, ensuring the drill bit passes through the entire depth of the bar, ensure no surface other than the bar is penetrated by the drill. If the depth of the bars precludes drilling the entire depth then the bars should be drilled to half the depth on the top surface and drilling repeated on the underside of the bar therefore ensuring that the entire bar depth has been sampled.



# Alex Stewart **International**

## SECTION 3 Cont'd

8. Collect all drill shavings from the three points on each bar and mix together. If milling equipment is available then the drillings can be lightly milled to produce a more uniform sample which can then be screened and weighed and coarse and fine proportionate samples prepared.
  - a) Ensure to pick free shavings that have not broken away freely from the bar
  - b) Ensure to remove any shavings still attached to the drill-bit
  - c) Ensure to keep separate samples from different bars
9. Weigh the bulk sample and bar (separately) to confirm all the sample has been collected
10. Reduce 'bulk' sample to 10g per bar; package, seal and label as required.
11. Repeat process as required for each bar



# Alex Stewart International



[www.alexstewartinternational.com](http://www.alexstewartinternational.com)



# Alex Stewart **International**

## SECTION 4

### 4) Dore – Example for sawing bars (pre-melt)

1. If possible, document processes with photographs
2. Weigh bars
3. Comment on cleanliness / appearance of bars – e.g. slaggy surfaces, striations in the material, slag inclusions, porous surfaces or cavities, magnetics, roughness/smoothness etc
4. Clamp the Bar with a side / long edge face-up and tighten sufficiently
5. Clean Surfaces of bars
6. Re-comment on Appearance of Bars

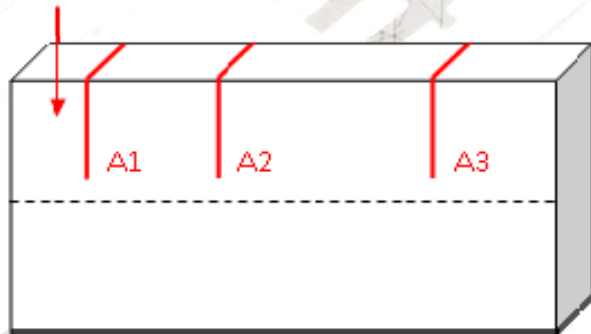




# Alex Stewart International

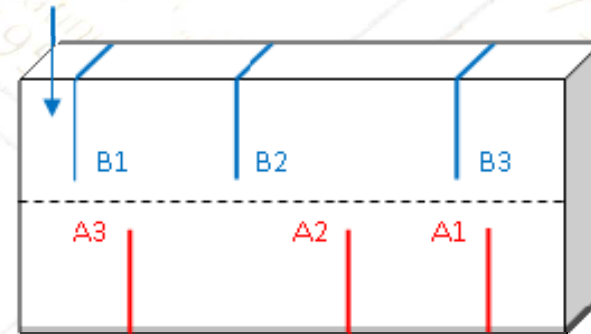
7. Make three incisions using the mechanical saw blade from the outside towards the middle of the bar, rotate the bar & re-clang
  - a) Ensure the cuts reach the 'centre' line of the bar
  - b) The incisions should not be uniform, and should be staggered (different distances apart) to ensure that the bar is not cut into segments
  - c) Ensure to keep separate samples from different bars

Saw cuts A1-3 into half the depth of the bar



Rotate bar & clamp/hold.

Saw cuts B1-3 into half the depth of the bar



8. Weigh the bulk sample and bar (separately) to confirm all sample has been collected
9. Reduce 'bulk' sample to approximately 10g per bar; package, seal and label as required.
10. Repeat as required

[www.alexstewartinternational.com](http://www.alexstewartinternational.com)





# Alex Stewart **International**

## SECTION 5

### Analysis

The accuracy and homogeneity of the sample produced from melting has a crucial bearing on the correct evaluation of a consignment.

To put this in perspective a sample submitted for analysis may weigh for example only 0.1 to 1 gramme which may represent a lot for example of 100 Kgs or more

### SAMPLE PREPARATION PRIOR TO ANALYSIS

As can be appreciated from the various procedures previously mentioned, the samples for analysis submitted to the laboratories will be in one of several forms which require different means of preparation before the chemists can proceed with the analysis.

This may involve drilling, stamping pressing, rolling, sawing or cutting to produce a suitable sample for analysis.

It is essential that all equipment is thoroughly clean before commencing this preparation to avoid possible contamination.

The accuracy of balances of course is of paramount importance as samples are often weighed to an accuracy of up to 7 decimal places /gm.





# Alex Stewart International



[www.alexstewartinternational.com](http://www.alexstewartinternational.com)



# Alex Stewart International



[www.alexstewartinternational.com](http://www.alexstewartinternational.com)



# Alex Stewart **International**

## SECTION 5

### Analysis (cont'd)

#### ANALYSIS

The generally accepted international standard assaying procedure for Dore and gold bullion analysis is by the corrected fire assaying method.

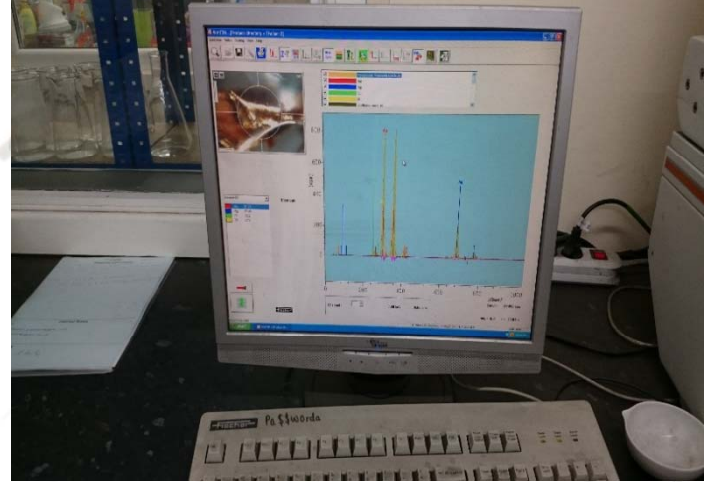
Higher grades of gold bullion i.e. + 999 to 9999 are generally assayed using the ICP Method.

We will be pleased to advise further details on assaying procedures etc.





# Alex Stewart International





# Alex Stewart **International**

## SECTION 6

### Summary

**Applying the correct melting and sampling procedure is a crucial factor in achieving both commercial and technical agreement between buyer and seller.**

**Poor/inaccurate sampling leads to delays in settlement and possible further delays if umpire assaying is required.**

**Poor/inaccurate evaluation of a shipment will also result in final stock balance differences. If basic care and good housekeeping, together with the general guidelines mentioned in this presentation are adopted during these operations then this will considerably reduce the possibility of errors.**

**We would like to thank the organizers for the inviting our company to give this presentation.**





# Alex Stewart **International**



[www.alexstewartinternational.com](http://www.alexstewartinternational.com)