



Sheffield Analytical Services

Analytical Services

a division of Sheffield Assay Office

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Agenda

Introduction

Testing Au 999 ‰ and 999.9 ‰

Comparison of Test Methods

Testing Ag in Accordance with 15096: 2020

Preliminary Testing for Fine Pt

Conclusions



Introduction: SAS involvement with fine PM Testing

Sheffield Analytical Service commercial testing

Partnership with London Assay Office

Trial of the Pyx

Determination of fine Au and Silver for in-house Test Methods



Fine Gold Testing

ISO 15093:2020

Au 999‰

- Solid/Liquid ratio 10g/L
- Au reference 999,9‰
- Calibration standards 1000mg/kg (10mg/L)
- No calibration checks
- No acid blanks

Au 999,9‰

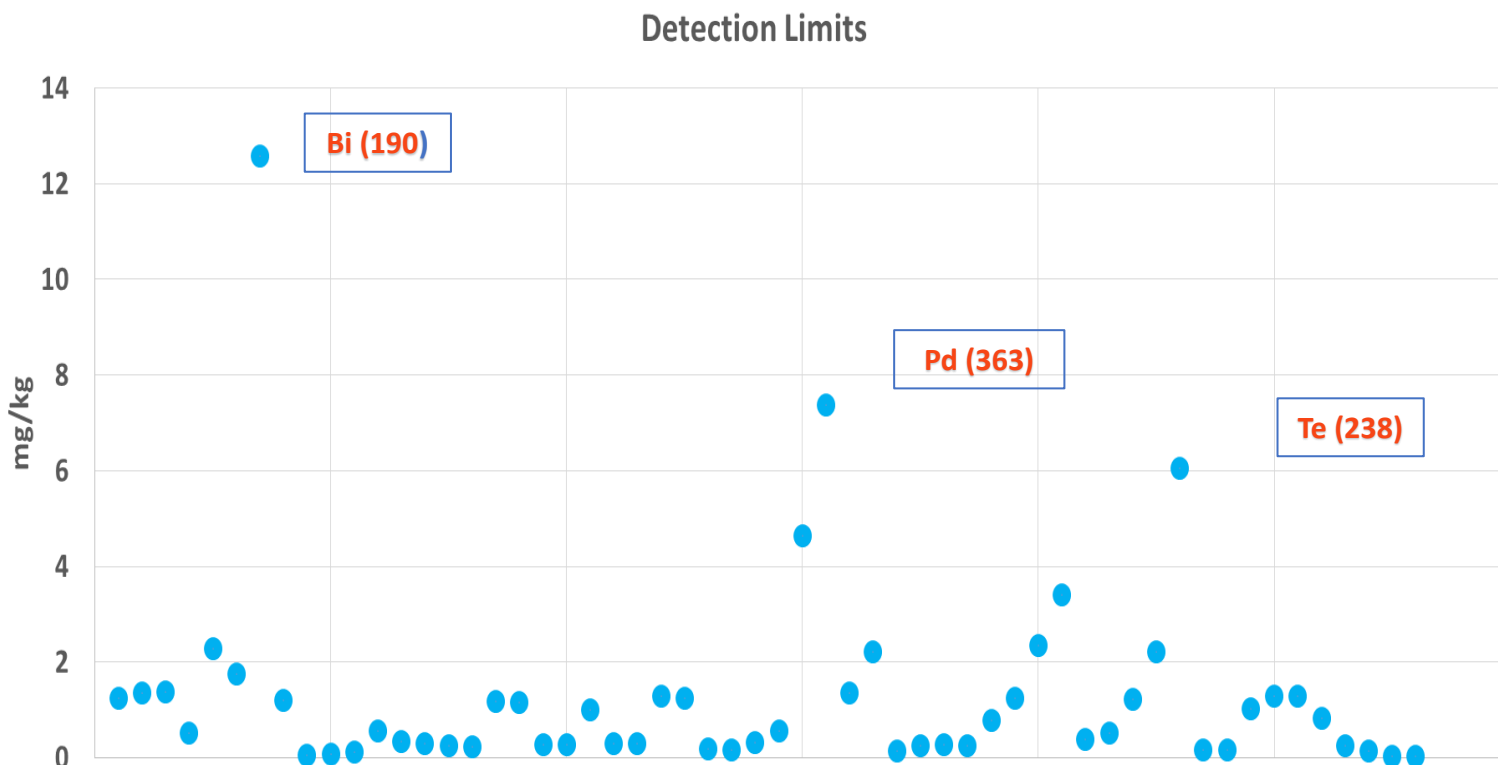
- Solid/Liquid ratio 40g/L
- Au reference 999,99‰
- Calibration Standard 100mg/kg (4mg/L)

In-house method A

Method A
999‰
& 999,9‰

- Solid/liquid ratio 10g/L
- Au reference 999,99‰
- Calibration standards (50mg/kg = 0.5mg/L)
- Calibration Checks 5mg/kg
- Acid blanks
- AQC Au 999‰

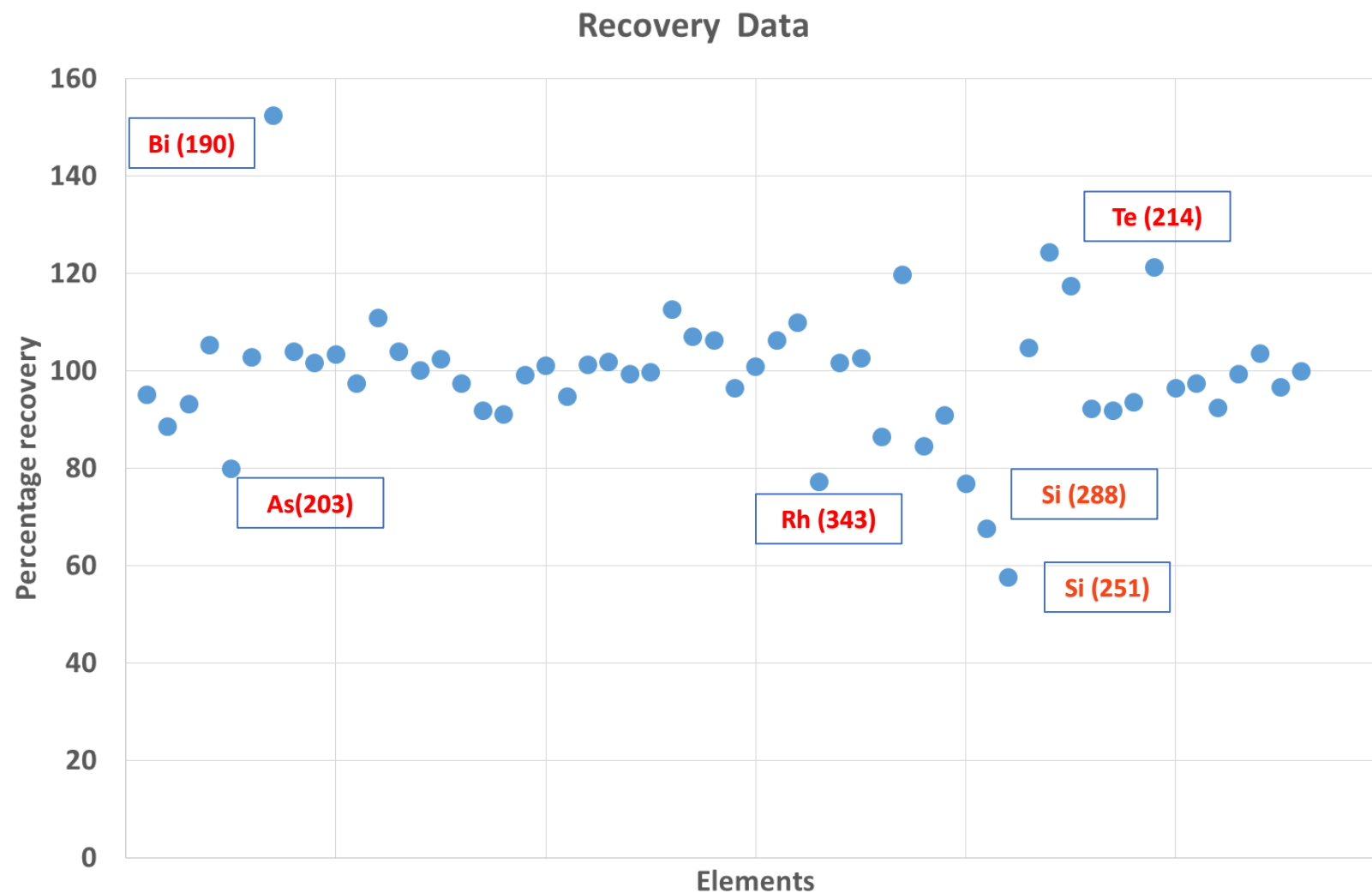
Limits of Detection for Fine Au using Method A



Note: Iridium cant be measured precisely using ICP-OES

Elements & Wavelengths		
Ag (328.068)	In (230.606)	Se (196.026)
Ag (338.289)	Mg (279.553)	Se (203.985)
Al (237.312)	Mg (280.270)	Si (251.611)
Al (396.152)	Mn (257.610)	Si (288.158)
As (188.980)	Mn (259.372)	Sn (189.925)
As (193.696)	Ni (216.555)	Te (214.282)
Bi (190.171)	Ni (230.299)	Te (238.579)
Bi (223.061)	Ni (231.604)	Ti (334.941)
Cd (214.439)	Pb (220.353)	Ti (336.122)
Cd (226.502)	Pd (340.450)	Tl (190.794)
Co (228.615)	Pd (363.469)	Tl (276.789)
Co (238.892)	Pt (214.424)	Tl (351.923)
Cr (205.560)	Pt (265.945)	W (207.912)
Cr (267.716)	Rh (343.488)	Zn (206.200)
Cu (324.754)	Rh (369.236)	Zn (213.857)
Cu (327.395)	Ru (245.657)	Zr (339.198)
Fe (238.204)	Ru (267.876)	Zr (343.823)
Fe (259.940)	Sb (206.834)	
Ga (294.363)	Sb (217.582)	
Ga (417.204)		

Recovery Data for Fine Au using Method A



Recovery data are calculated based on 6 repeats per elements with a content of 5mg/kg



ISO 15093:2020 in practise:

	Ag (328)	As (193)	Fe (259)	Pt (214)	Sb (206)	Te (214)	Sn (189)
2703Fine Au Blk-1	-12.0	-3.2	-2.1	-2.1	-4.0	-1.1	1.9
2703Fine Au Blk-2	-12.3	-3.5	-2.0	-1.9	-3.9	-0.9	2.3
2703Fine Au Blk-3	-12.3	-3.4	-2.2	-1.9	-3.9	-1.8	3.2
2703Fine Au Control-1-1	-7.8	-0.8	-1.5	-0.3	-1.4	-0.1	0.8
2703Fine Au Control-1-2	-7.0	-0.7	-1.5	-0.9	-0.9	-0.1	2.1
2703Fine Au Control-2-1	-3.0	4.2	-1.0	-0.2	4.0	5.5	6.6
2703Fine Au Control-2-2	-3.1	3.7	-1.1	0.1	3.3	5.0	6.7
Recovery Factors	1.25	0.88	0.96	0.97	0.90	0.97	0.95

High consumption
of Au 999,99‰

Contamination of Ag,
Fe, Pt, Sb, Te mainly in
fine Au used for
calibration and controls



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Testing of Fine Gold

In house Method B

- Solid/Liquid ratio 10g/L
- Calibration without Au with content from 5mg/kg to 50mg/kg
- Au 999,99‰ reference background
- Standard addition on samples; 3 trials per sample
- Internal standard Y (1000ppm) made from Y₂O₃
- AQC Au 999‰
- All samples are subject to clean stage prior to fine testing. This doesn't apply to reference material with 999,99‰ certification



Inter-laboratory Exercise for fine Au Using Different Methods

Elements	Royal Mint UK	SAO	SAO
	ISO 15093:2020	ISO 15093:2020	Method B
Ag	0.012	0.012	0.012
Ir	0.001	-	-
Pd	0	0.000	0.001
Pt	0.023	0.026	0.025
Rh	0	0.000	0.000
Ru	0	0.000	0.000
Sn	0.001	0.000	0.000
Al	0	0.000	0.000
Bi	0.001	0.001	0.001
Cd	0	0.000	0.000
Co	0	0.000	0.000
Cr	0	0.000	0.000
Cu	0.003	0.004	0.004
Fe	0.007	0.005	0.004
Mn	0	0.000	0.000
Ni	0	0.000	0.000
Pb	0	0.000	0.000

Elements	Royal Mint UK	SAO	SAO
	ISO 15093:2020	ISO 15093:2020	Method B
Pb	0	0.000	0.000
Zn	0.001	0.000	0.000
As	-	0.006	0.003
Ga	-	0.001	0.000
In	-	0.000	0.000
Mg	-	0.000	0.000
Sb	-	0.005	0.000
Se	-	0.000	0.001
Si	-	0.000	0.000
Te	-	0.000	0.000
Ti	-	0.000	0.000
Tl	-	0.002	0.002
W	-	0.001	0.001
Zr	-	0.000	0.000
Total exc Au	0.050	0.063	0.056
Au content	999.950	999.937	999.944

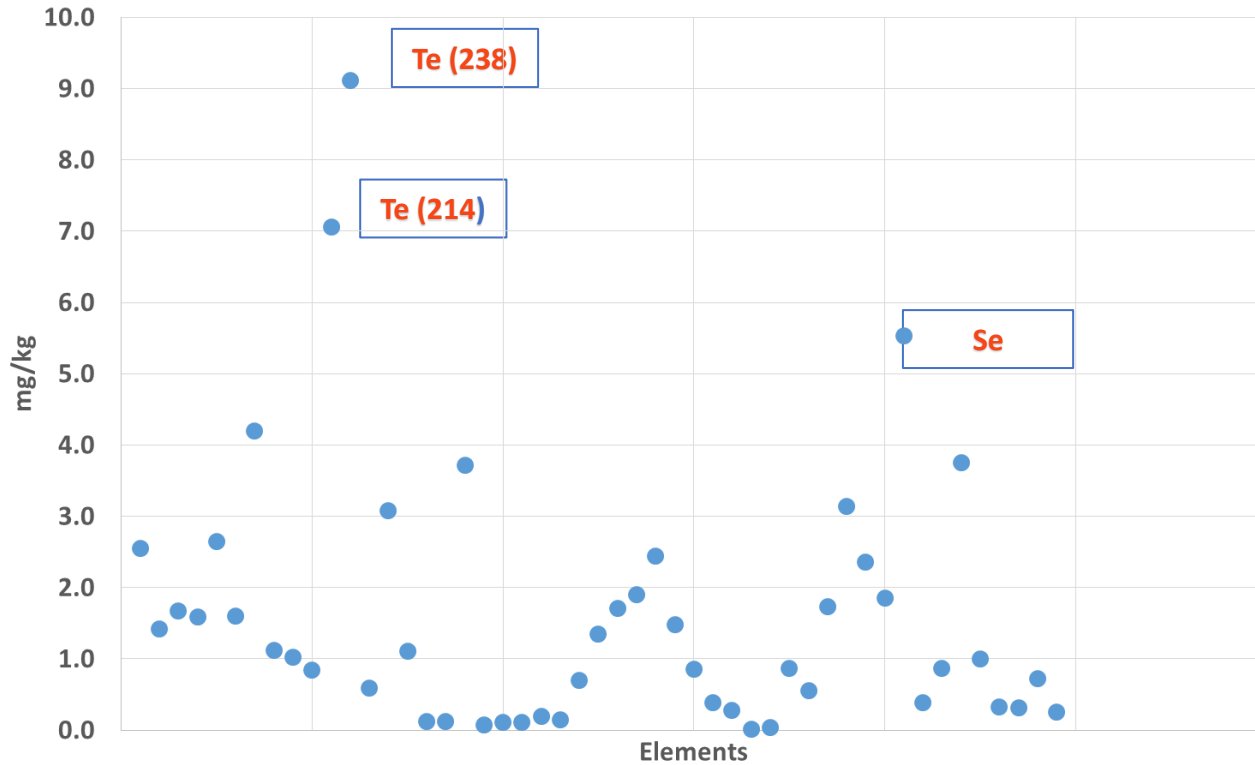
Fine Silver Testing In house Method based on ISO 15096:2020 Guidelines

**Method Ag
999‰ &
999,9‰**

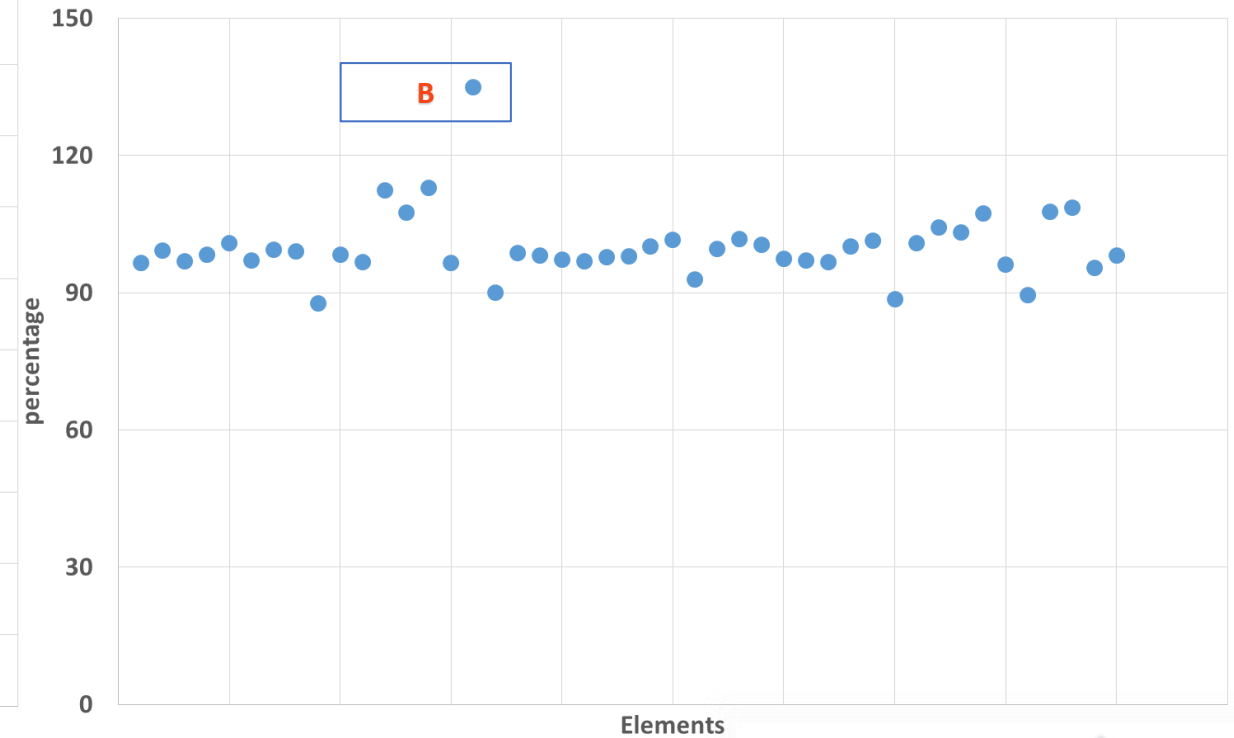
- Solid/liquid ratio 10g/L (ISO 15096 20-50g/L)
- Ag reference 999,99‰
- Sample are clean with IMS heat gently
- Two stages extraction;
 - Stage A: H₂O/HNO₃
 - Stage B: HCl/HNO₃
- Calibration standards for each extraction stage (100mg/kg) (ISO 15096 1000mg/kg)
- Control solutions 5mg/kg (ISO doesn't included checks or controls).
- Calibration and control solutions are unstable overtime, they are used within 12h
- Each stage solutions are measured using different programme and ICP-OES set up
- Samples are prepared in triplicate
- Acid blanks
- AQC Au 999‰

Limits of Detection for Ag in accordance with ISO 15096:2020

Detection Limits Fine Ag



Recovery Fine Ag



Recovery data are calculated based on 6 repeats per elements with a content of 5mg/kg





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Inter-laboratory Exercise for fine Ag Using ISO 15096:2020

Elements	Royal Mint UK	SAO
	ISO 15093:2020	ISO 15093:2020
Al	0.002	0.002
As	0.000	0.000
Bi	0.000	0.002
Cd	0.000	0.000
Co	0.000	0.000
Cr	0.000	0.000
Cu	0.048	0.050
Fe	0.008	0.002
Mg	0.000	0.002
Mn	0.000	0.000
Ni	0.000	0.000
Pb	0.016	0.008
Se	0.000	0.000
Zn	0.000	0.005
Au	0.002	0.002
Pd	0.050	0.056
Pt	0.000	0.000
B	-	0.000
Hg	-	0.002
In	-	0.000
P	-	0.000
Sb	-	0.000
Si	-	0.004
Sn	-	0.001
Ti	-	0.000
Total contamination	0.127	0.138
Ag result(ppt)	999.873	999.862

Testing Pt using ISO 15093:2020 guidelines

Method Pt 999‰

- Fine Pt was analysed using same test method than Au (Method A) in accordance with ISO 15093
- The acid blanks showed high negative values due to potential contamination of the reference materials used.
- Pt caused spectral interferences on the ICP-OES and false positive results and poor checked corrections were observed. However ICP-OES produced better results for Au, Ag, Pd, Ru, Rh etc.
- To understand the difficulty between potential contamination from reference material to match sample matrix, ICP-MS and ICP-OES 10,000mg/L was used along with reference materials

Conclusions

To analyse fine precious metals in challenging and require a structured guideline international standard. Use of PM reference materials is an important cost for commercial laboratories

Gold/Silver/ Platinum reference materials may have impurities at levels higher than stated in the certificates

Test method using calibration solution without matrix match , internal standard and standard addition shows good agreement with ISO 15093:2020. This test method reduces significantly the consumption of expensive Au reference materials

The testing of multiple fine silver samples using the ISO15096:2020 guidelines has revealed good method precision and no major issues . New test method is being tested currently.

Limited testing conducted for fine Pt in accordance with ISO 15096:2020 has showed significant spectral interference and the requirement to use ICP-MS



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Thank you very much

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