

Atomic Absorption Screening Method Guide

Se in Blood Serum

Key Words

- Blood Serum
- Selenium
- Graphite Furnace
- Atomic Absorption
- Rapid Screening Method

Principle

The sample is diluted 1+4 with a special diluent containing 0.1 % m/v Triton X-100 and 1 % v/v nitric acid in deionised water and selenium is determined by Graphite Furnace Atomic Absorption Spectrometry using ELC cuvettes and employing 500 ppm Palladium as a modifier.

Reagents

Concentrated nitric acid (Spectrosol or equivalent)

Triton X-100 (High purity grade or equivalent)

Diluent mixture

Weigh 0.1 gram of Triton X-100 into a 100 mL flask, add about 50 mL deionised water and then transfer 1 mL of concentrated nitric acid into the flask. Dilute to volume with deionised water and mix well.

2 % Pd in 10 % v/v HNO₃ (SPEX CertiPrep or equivalent)

500 ppm Matrix modifier preparation

Transfer 0.5 mL of the Pd master solution to a 20 mL flask. Dilute to volume with deionised water and mix well.

Pooled serum blank standard (Example contained 0 µmol/L Se, certified Sero reference material)

Pooled serum high standard (Example contained 2.6 µmol/L Se, local reference material)

Working standards

Prepare working blank and top standard by dilution with deionised water, using the same procedure as for samples.

Sample Preparation

The serum samples were prepared in acid washed tubes immediately before analysis. 200 µL portions of serum were mixed with 800 µL of the special diluent containing 0.1 % m/v Triton X-100 and 1.0 % v/v nitric acid. Ensure that the solution is thoroughly mixed before analysis.

Instrument Parameters

Spectrometer | Serum (Se)

Measurement Mode: **Absorption** | Cook Book

Number of Resamples: **3**

Fast Resamples

Measurement Time: (s) **10.0**

Wavelength: (nm) **196.0**

Lamp Current: (%) **80**

Bandpass: (nm) **0.5**

Optimize Spectrometer Parameters

Signal: **Transient Height**

Transient Peak Measurement

Measure From (s): **0.00** To: **3.00**

High Resolution

Background Correction: **Zeeman**

Filer Rejection

Use Filer Rejection

Rejection Limit: (%) **95**

RSD Test

Use Test

If RSD greater than **1** %

AND signal greater than **1.1** Abs

Then **Flag and Continue**

Furnace | Serum (Se)

Cuvette: **Normal**

Injection Temperature: (°C) **0** | Programme Time (secs) **126.0**

Furnace Programme

	Temp (°C)	Time (s)	Ramp (°C/s)	Gas Type	Gas Flow	RD	RS	TC	HL
1	95	10.0	5	2 Inert	0.3 L/min				
2	95	10.0	1	2 Inert	0.3 L/min				
3	100	5.0	1	2 Inert	0.3 L/min				
4	110	10.0	1	2 Inert	0.3 L/min				
5	300	1.0	10	2 Inert	0.3 L/min				
6	1200	10.0	100	2 Inert	0.3 L/min				
7	1900	3.0	0	2 Inert	Off	<input checked="" type="checkbox"/>		<input checked="" type="checkbox"/>	
8	2600	2.0	0	2 Inert	0.3 L/min				<input checked="" type="checkbox"/>
9	20	5.0	0	2 Inert	0.3 L/min				

Clean Cuvette if sample greater than: **1** Abs

Calibration | Serum (Se)

Method: **Normal: Quadratic Least Squares Fit** | Use Stored Calibration

Concentration Units: **µmol/L**

Standards: **4** | Default Ratios

Standard Concentrations

Master Standard Conc: **10**

	1	2	3	4	5
0.260	0.520	1.520	2.360	3.000	6.000
0.000	0.000	0.000	0.000	0.000	0.000

Scaling Factor: **1**

Scaled Units: **µmol/L**

Calibration Checks

Acceptable Fit: **0.995**

Excess Curvature Limits

From: (%) **10**

To: (%) **10**

Bescale limit: **10** %

If any calibration checks fail: **Flag and Continue**

Sampling | Serum (Se)

FS95

Sample: **Sample Preparation: None**

Saggle Volume: (µL) **10.0**

Injection: **Intelligent Dilution Threshold (%) 100**

Working Volume: (µL) **20.0**

Standard Preparation: **Manual**

Standard Additions: **None**

Wash Autosampler if sample greater than: **1** Abs

Slow Solution Uptake | Automatic Spike

Slow Solution Injection | Spike Volume (µL): **20.0**

Sampling Delay | Waives: **1**

Matrix Modification

	Name	Volume (µL)	Order	Method
1		20.0	1	None
2		20.0	2	None
3		20.0	3	None
4		20.0	4	None
5		20.0	5	None
6		20.0	6	None

Volumes... | Reagent Details...

The use of slow injection into a warmed cuvette minimises the effect of sample viscosity and improves the drying performance.

Background correction using a Zeeman-based system is preferred due to the potential spectral interference from iron (present in any red cells carried over during separation).

Results

Sample	Serum (1)	Serum (2)
Selenium found ($\mu\text{mol/L}$)	1.02	0.52
Reference value ($\mu\text{mol/L}$)	1.1 +/- 10%	0.5 +/- 10%

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