Simultaneous determination of elements in Veterinary Samples using the Optimass.

Introduction
A contract veterinary laboratory submitted various sample to be analysed for nutritional and toxic elements. The samples were as diverse as soil, serum, blood, liver and thyroid samples.
Speed of analysis and accuracy of sample results were the two main criteria that were required.

Samples
Soil A, Liver B and C, Dried Thyroid D.
Blood samples E, F and G were used as standards for samples H, I and J.
Serum samples K and L were used as standards for samples M, N and O.

Soil A, Liver B and C, Dried Thyroid D were all prepared by the customer. The matrix composition is unknown for sample D.

Blood samples E, F and G were used as standards for samples H, I and J. Due to the low volumes, all of the blood samples submitted for analysis, these were diluted 10 times in 1%HNO_3 and 0.05%TritonX-100. Sample centrifuge before analyses.

Serum samples K and L were used as standards for samples M, N and O. Due to the low volumes of the serum samples submitted for analysis; these were diluted 10 times in 1%HNO_3 and 0.05%TritonX-100.

Instrumentation
The samples were analysed with an Optimass via direct Aspiration.
A MicroMist nebuliser was used to run Soil A, Liver B and C, Dried Thyroid D samples and a Burgener Peek Mira Mist nebuliser was used to run the Blood and Serum samples.

Two sets of Instrument Settings were used for the samples:
1. For High concentration elements, one second data acquisition and low extraction instrument settings was used.
2. For Low concentration elements, five seconds data acquisition and high extraction instrument settings was used.

One set of Instrument Setting and five seconds data acquisition was used to run the Blood and Serum samples.
For all settings 2 samples could be analysed per minute for all elements and isotopes in the periodic table.

Results
Table 1 shows the results for the soil sample. Note that excellent correlation was obtained between the expected Results and actual results. Also the excellent detection limits and calibration linearity.

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Soil Sample	Element	Expected Value (ppm)	Result (ppm)	Detection Limit (ppb)	Calibration Range (ppm)	Calibration Linearity R^2
	Pb	128.25 **135.00** 141.75	**135.26**	**0.006**	**1.0 – 50**	**0.9991**
	Cu	3.14 **3.30** 3.47	**3.06**	**0.06**	**1.0 – 50**	**0.9954**
	Zn	180.50 **190.00** 199.50	**197.87**	**0.45**	**0.1- 50**	**0.9970**
	Cd	1.34 **1.41** 1.48	**1.46**	**0.01**	**0.1- 50**	**0.9945**
	Fe	**NA**	**2112.0**	**0.04**	**1.0 – 50**	**0.9964**
	Mn	460.75 **485** 509.25	**486.98**	**0.03**	**0.1 – 50**	**0.9927**
	Se	0.029 **0.03** 0.032	**0.002**	**0.06**	**0.001 – 0.1**	**0.9990**
	Mo	0.076 **0.08** 0.084	**0.003**	**0.02**	**0.001 – 0.1**	**0.9998**
	As	9.785 **10.3** 10.815	**10.03**	**0.14**	**1.0 - 50**	**0.9957**

Table 1: Soil sample results.
NA – not available

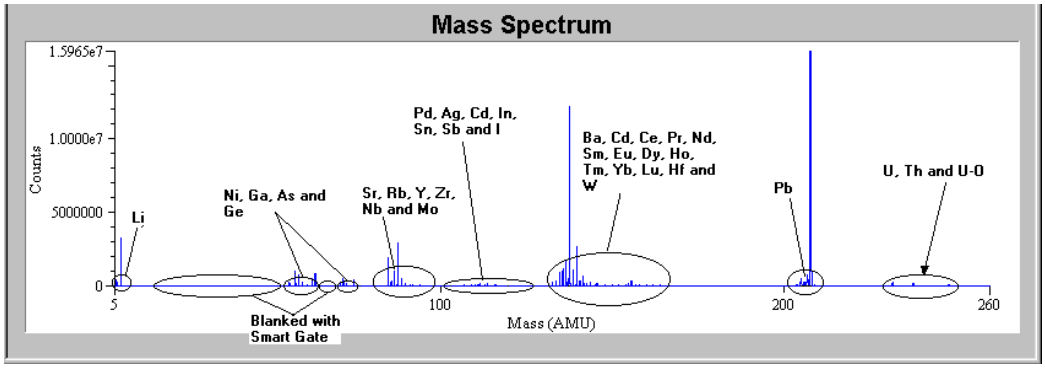

Figure 1: Soil sample mass spectrum shown with AMU 10 to 58.5 and 63.5 to 70.5 blanketed using Smartgate. Five second data acquisition time was used.

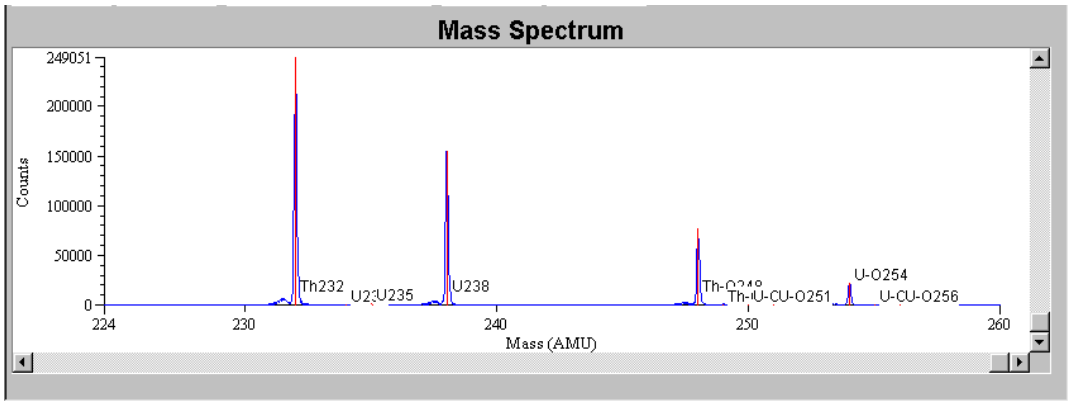

Figure 2: Soil sample mass spectrum showing AMU 230 to 260. Th_{232}, U_{234}, U_{235}, U_{238} and the Th-Oxides and U-Oxides isotopes can be seen.

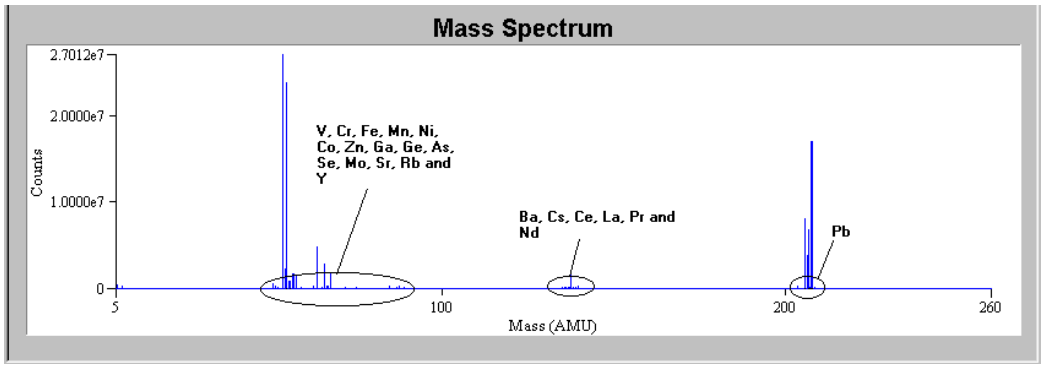

Figure 3: Soil sample mass spectrum with 10x dilution. AMU 10 to 50 is blanketed using Smartgate. One second data acquisition was used.

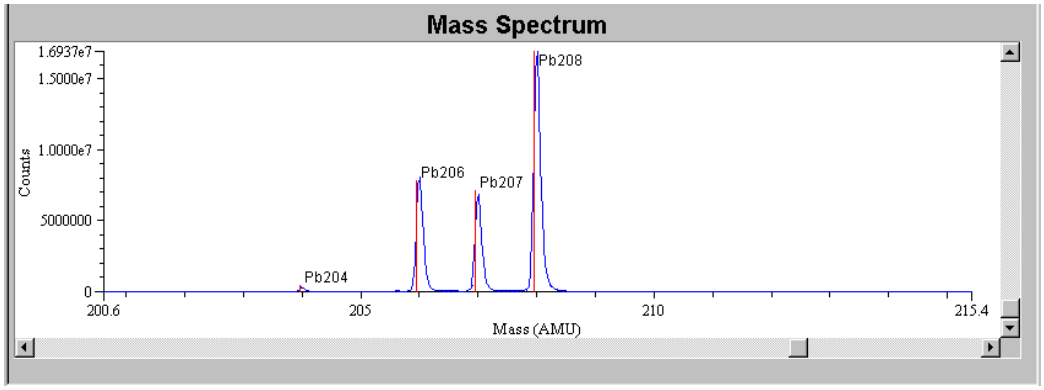

Figure 4: Soil sample mass spectrum showing the various Pb isotopes. The high resolution of the Optimass allows nearby peaks to be completely resolved.

There are two methods available for determining whether there are any interferences in any particular mass.

1) By comparing peak heights with natural abundant sensitivity (red lines) the user can quickly identify whether a peak has an interference. Any interferences would cause peaks not to follow natural abundance sensitivity lines. For the soil sample no interferences for the Lead isotopes, Pb_{204}, Pb_{206}, Pb_{207} or Pb_{208} are present.

2) As the Optimass is simultaneous, values for each isotope can be generated. If there are any interferences the isotope with the interference will give a vastly different value compared to the similar results given by the isotopes with no interferences.

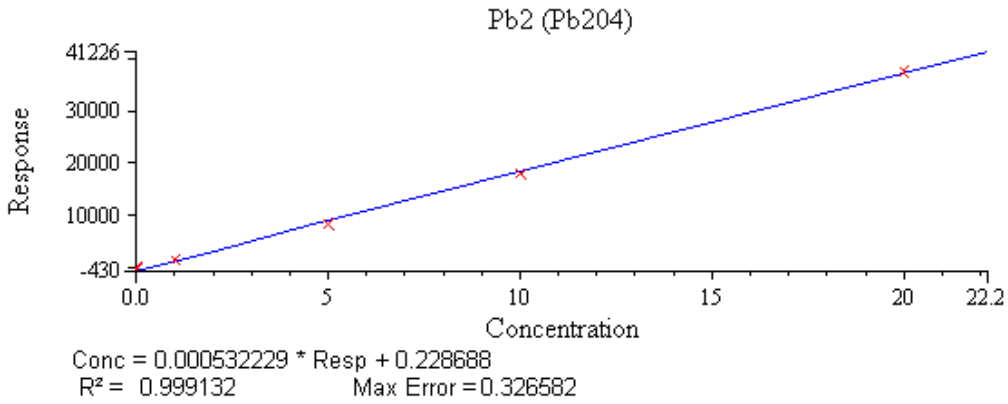

Figure 5: Pb_{204} calibration graph for soil sample.

Liver Sample B	Element	Expected Value (ppm)	Result (ppm)	Detection Limit (ppb)	Calibration Range (ppb)	Calibration Linearity R^2
	Cu	7.60 **8.00** 8.40	**6.56**	**0.06**	**100 – 1000**	**0.9967**
	Pb	**NA**	**0.026**	**0.45**	**5.0 – 1000**	**0.9998**
	Zn	6.06 **6.40** 6.72	**8.68**	**0.01**	**100 – 50,000**	**0.9958**
	Cd	**NA**	**5.71**	**0.04**	**5.0 – 1000**	**0.9997**
	Fe	8.74 **9.20** 9.66	**10.81**	**0.03**	**5.0 – 1000**	**0.9913**
	Mn	0.50 **0.53** 0.56	**0.46**	**0.06**	**1.0 – 100**	**0.9995**
	Se	0.029 **0.03** 0.032	**0.032**	**0.02**	**1.0 – 100**	**0.9996**
	As	0.009 **0.01** 0.011	**0.009**	**0.14**	**1.0 - 100**	**0.9997**

Table 2: Liver sample B results.
NA - Not available

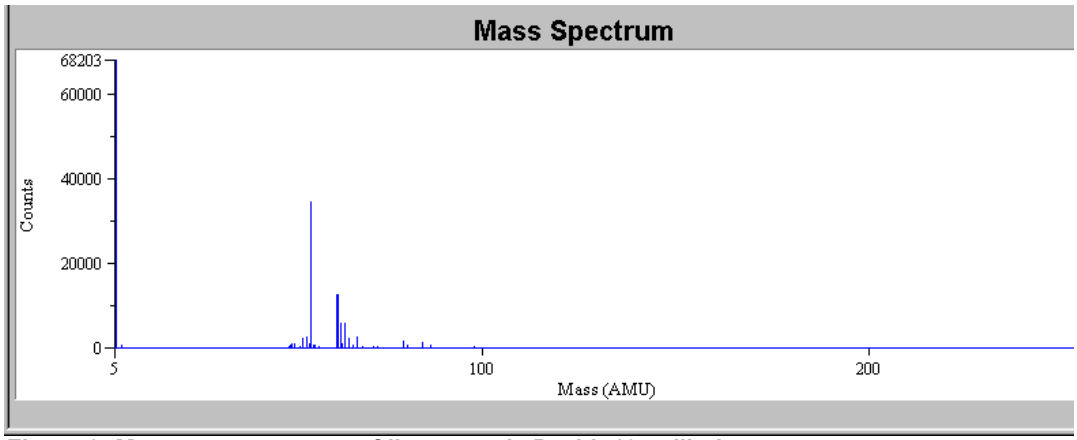

Figure 6: Mass spectrum scan of liver sample B with 10 x dilution.

Liver Sample C	Element	Expected value (ppm)	Result (ppm)	Detection Limit (ppb)	Calibration Range (ppm)	Calibration Linearity R^2
	Cu	12.54 **13.20** 13.86	**13.68**	**0.06**	**1.0 – 20**	**0.9913**
	Pb	**NA**	**0.23**	**0.45**	**0.001 – 0.1**	**0.9999**
	Zn	12.83 **13.50** 14.18	**13.28**	**0.01**	**0.005 - 1**	**0.9953**
	Cd	**NA**	**0.017**	**0.04**	**0.005 - 1**	**0.9999**
	Mn	0.38 **0.40** 0.42	**0.41**	**0.06**	**0.001 – 0.1**	**0.9988**
	Se	**NA**	**0.56**	**0.02**	**0.001 – 0.1**	**0.9989**
	As	**NA**	**0.43**	**0.14**	**0.001 – 0.1**	**0.9997**

Table 3: Liver sample C results.
NA: - Not Available

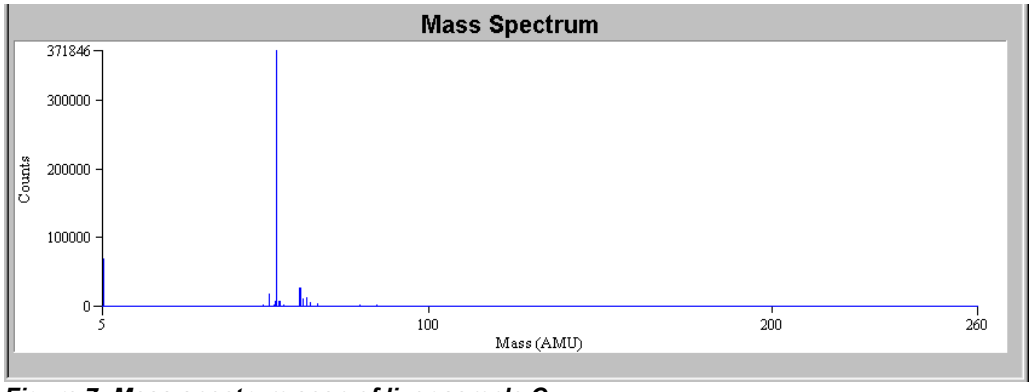

Figure 7: Mass spectrum scan of liver sample C.

Commercial reconstituted whole blood sample	Element	Expected Value (ppm)	Result (ppm)	Calibration Range (ppm)	Calibration Linearity R^2
	Pb	0.252 **0.24** 0.228	**0.244**	**0.030-0.640**	**0.9994**
	Cd		**0.016**	**0.000- 0.010**	**1.0000**
	Mn		**0.032**	**0.000-0.020**	**0.8791**
	Mo		**0.005**	**0.000-0.010**	**1.0000**

Table 4: Commercial reconstituted whole blood sample results.

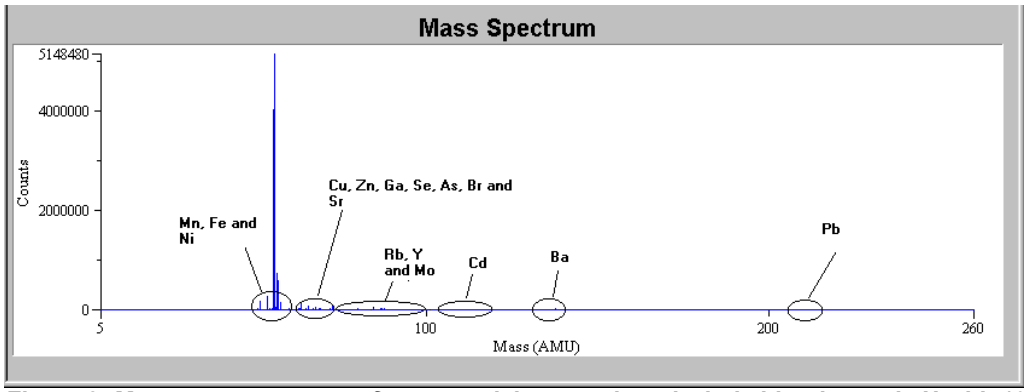

Figure 8: Mass spectrum scan of commercial reconstituted whole blood sample H with 10 x dilution.

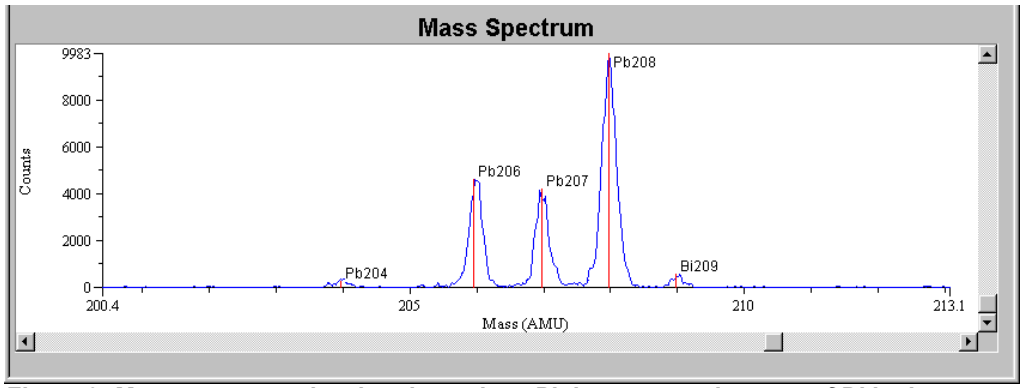

Figure 9: Mass spectrum showing the various Pb isotopes and a trace of Bi in the scan as per figure 9. The high resolution of the Optimass allows nearby peaks to be completely resolved.

Bovine Whole Blood	Element	Expected Value (ppm)	Result (ppm)	Calibration Range (ppm)	Calibration Linearity R^2
	Pb	NA	0.730	0.030-0.640	0.9994
	Cd	NA	<0.001	0.000- 0.010	1.0000
	Mn	NA	0.007	0.000-0.020	0.8791
	Mo	NA	0.005	0.000-0.010	1.0000

Table 5: Bovine whole blood sample with 10 x dilution results.

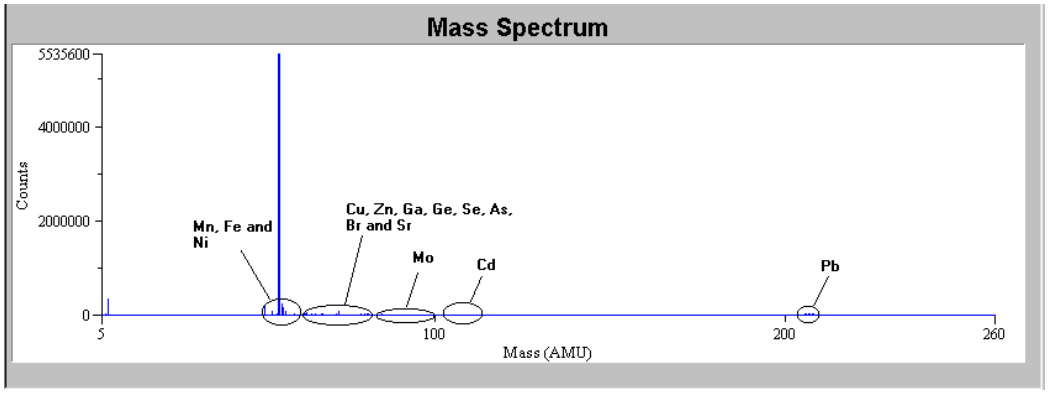

Figure 10: Mass Spectrum scan of Bovine whole blood sample with 10x dilution.

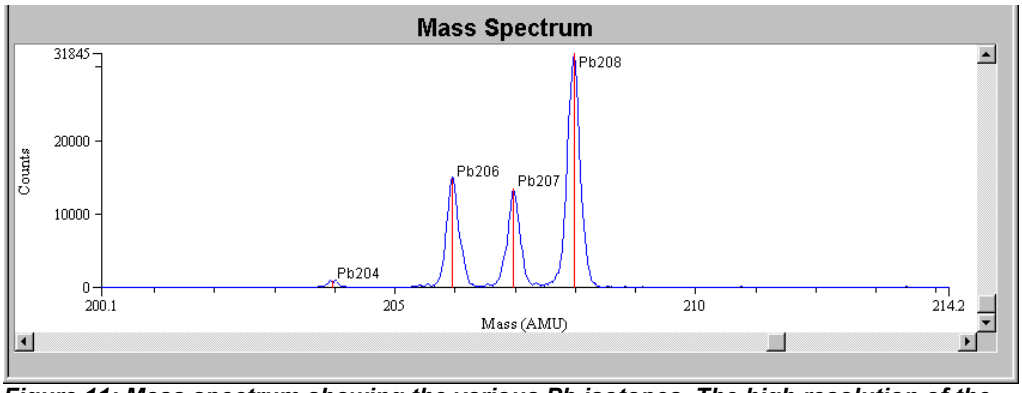

Figure 11: Mass spectrum showing the various Pb isotopes. The high resolution of the Optimass allows nearby peaks to be completely resolved.

Swan whole blood sample	Element	Expected Value (ppm)	Result (ppm)	Calibration Range (ppm)	Calibration Linearity R^2
	Pb	NA	1.66	0.030-0.640	0.9994
	Cd	NA	<0.001	0.000- 0.010	1.0000
	Mn	NA	0.007	0.000-0.020	0.8791
	Mo	NA	0.020	0.000-0.010	1.0000

Table 6: Swan whole blood with 10x dilution results.
NA – Not available

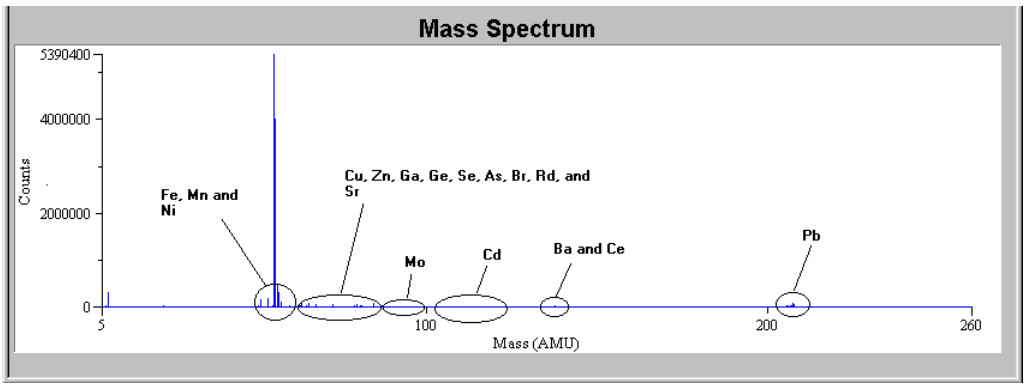

Figure 12: Mass Spectrum scan of Swan whole blood sample with 10 x dilution.

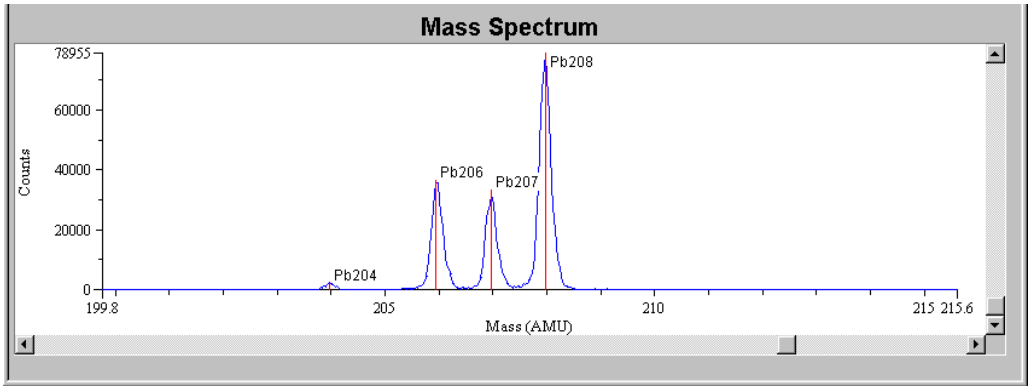

Figure 13: Mass spectrum showing the various Pb isotopes. The high resolution of the Optimass allows nearby peaks to be completely resolved.

Bovine serum	Element	Expected Value (ppm)	Result (ppm)	Calibration Range (ppm)	Calibration Linearity R^2
Sample M	Cu	1.00 **0.95** 0.90	0.791	0.000-0.176	1.0000
	Fe	NA	0.687	0.000-0.127	1.0000
	Zn	NA	0.812	0.000-0.113	1.0000
Sample N	Cu	0.63 **0.60** 0.57	0.600	0.000-0.176	1.0000
	Fe	NA	1.020	0.000-0.127	1.0000
	Zn	NA	0.815	0.000-0.113	1.0000
Sample O	Cu	0.82 **0.78** 0.74	0.780	0.000-0.176	1.0000
	Fe	NA	1.060	0.000-0.127	1.0000
	Zn	NA	0.635	0.000-0.113	1.0000

Table 7: Bovine serum sample M, N and O with 10x dilution sample results.
NA – Not Available

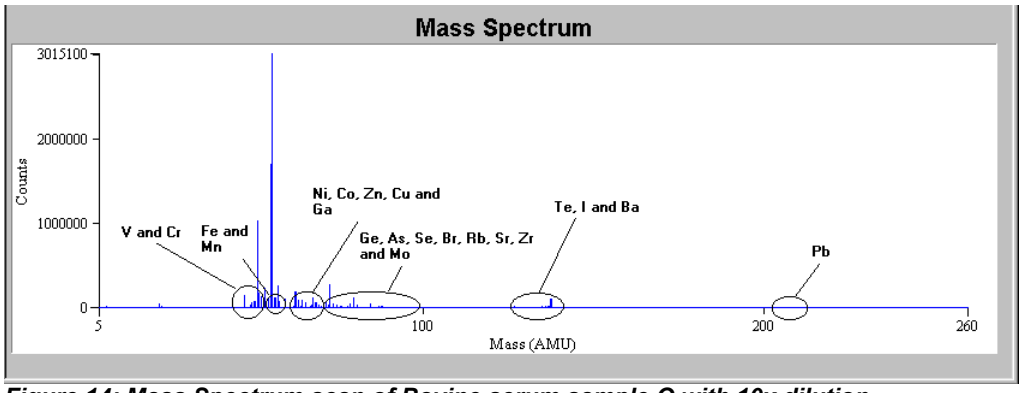

Figure 14: Mass Spectrum scan of Bovine serum sample O with 10x dilution.

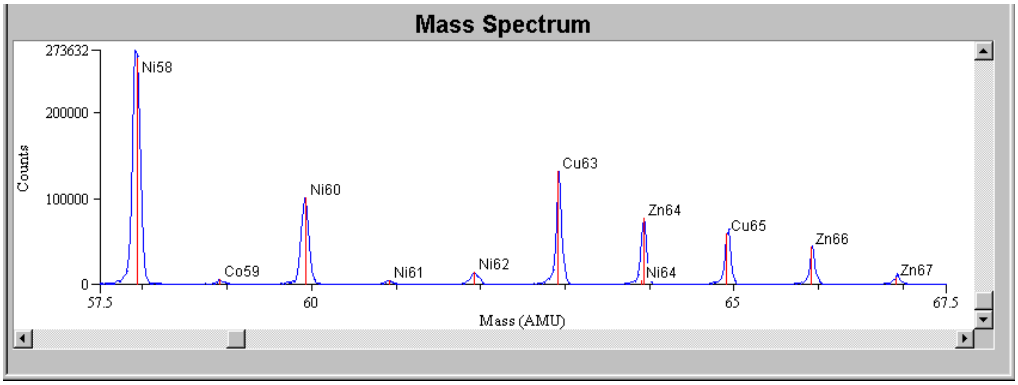

Figure 15: Mass spectrum showing various peaks from mass 57.5 to mass 67.5. The high resolution of the Optimass allows nearby peaks to be completely resolved.

Thyroid Sample

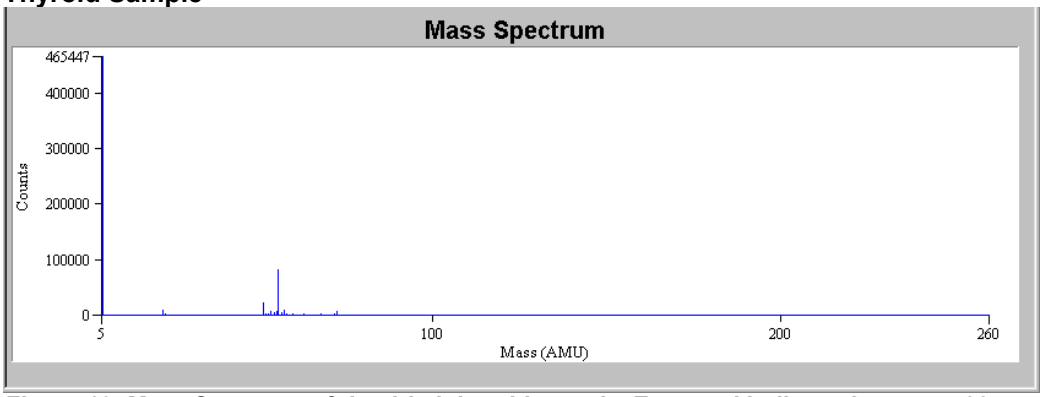

Figure 16: Mass Spectrum of the dried thyroid sample. Expected Iodine value was 7.22 ppm but no peak at mass 127 was obtained indicating no iodine.

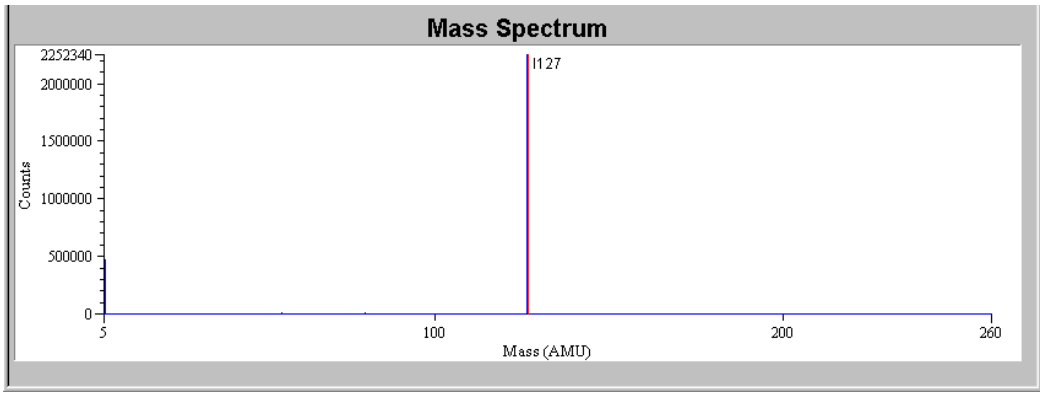

Figure 17: Mass spectrum of a 5 ppm I standard in same matrix as thyroid showing iodine can be detected.

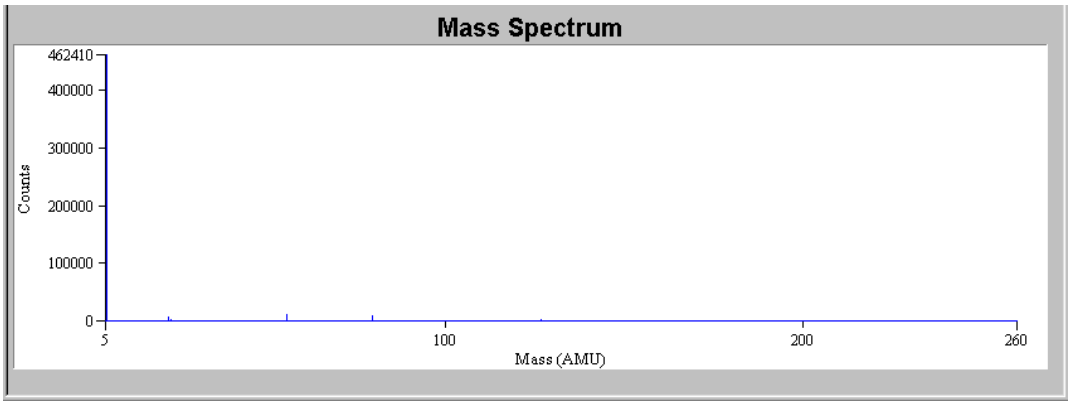

Figure 18: Mass scan of Blank 18 Meg DI water in same matrix as thyroid sample to prove that I peak shown in figure 22 is not an interference from the sample matrix.

The conclusion for the Thyroid sample was although it was claimed that the sample had 7.22 ppm Iodine, no Iodine was in fact present. It was later found that the iodine had been adsorbed by the plastic in the plastic vial the sample was sent and stored in.

DISCUSSION

Full Mass Spectrum for all of the samples was performed to confirm the elements of interest. Sets of calibration standards were prepared for high and low concentrations. Appropriate sample dilution was made to suit the calibration range.

Note: Only 9 mL of each of the samples A, B, C and D was submitted. For method development purposes 50 to 100 mL of these samples would normally be required.
Note: Only 1.5 mL of each of the samples E to O was submitted. For method development purposes 25 – 50 mL of these would normally be required.
The simultaneous nature of the Optimass allows for speedy method development to be performed even with small sample volumes.

The Optimass 8000 was optimized using Lyphochek Whole Blood Control sample. This in-house standard was spiked with 10ppb SQ standard. The blood and serum samples were run successfully with Burgener Peek Mira Mist nebuliser without any blockages in the nebuliser.

CONCLUSION

The samples analysis for Soil A, Liver B and C, Dried Thyroid D has been described in this application note.
Certified blood and serum samples submitted were used as calibration standards to determine the unknown sample concentrations.
The Optimass was able to not only obtain the results but exceed the customer's expectations. The speed of analysis enabled complete method development and analysis on sample volumes as low as 1.5 mL.
The simultaneous nature of the Optimass allows rapid screening for all masses for any samples and in the case of the Thyroid sample proved conclusively and rapidly that no Iodine was present in the sample.

The versatility and speed of analysis of the Optimass make it an indispensable technique for the analysis of biological samples and samples with complex matrices, especially with very small volume samples. The high resolution of the Optimass enables closely located peaks to be resolved and enable these to be quantitatively measured.

With the ability to analyse two samples per minute for all elements and isotopes in the periodic table, the Optimass is an indispensable instrument for any laboratory.

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