

Analysis of Bovine Liver using the Agilent 5100 Synchronous Vertical Dual View (SVDV) ICP-OES

Application note

Food Testing and Agriculture

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Introduction

In order to protect public health and the environment, the testing of animal food products for a range of elements including nutrients, micro-nutrients and toxic elements is often subject to compliance, specified by national or international regulatory institutions. For example the Food and Agriculture Organization of the United Nations (FAO) and World Trade Organization (WTO) set up the Codex Alimentarius Commission to develop harmonized international food standards, guidelines and codes of practice, which often serve as a basis for national legislation. Food testing laboratories that are required to work within a regulatory framework must monitor a range of specified elements in a large number of samples on a daily basis.



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ICP-OES instrumentation is already commonly used in many food testing labs due to its reliability and ease-of-use, but the Agilent 5100 Synchronous Vertical Dual View (SVDV) ICP-OES takes the technique to a new level of operation and performance, particularly in terms of robustness, speed and reduced running costs.

Where most traditional dual view ICP-OES systems use a horizontal torch, the 5100 uses a more robust vertical torch orientation. This extends the torch lifetime, and also increases the matrix handling capability of the system. Uniquely, the 5100's Dichroic Spectral Combiner (DSC) technology combines axial and radial light from the vertical plasma, over the entire wavelength range, in a single reading. This ensures that speed is no longer compromised due to the need to measure radial and axial views sequentially. Sample throughput is also aided with the high speed VistaChip II CCD detector. With faster sample run times, the 5100 requires significantly less argon gas per sample, which can lead to significant savings for labs involved in high throughput analysis.

The Agilent 5100 uses ICP Expert software with software applets that include pre-set method templates for quick and easy method development. Method development is further simplified with the DSC technology which eliminates the need for the user to select the correct plasma viewing mode for each element. The operator is simply required to choose which elements and wavelengths are required, and the instrument performs the analysis in a single, synchronous measurement. For example, the nutrient elements such as sodium and potassium, that can be present in the sample at percent levels will be viewed radially while other elements, present at ppm or ppb levels, such as copper or zinc, will be viewed axially in the same reading to enable analysis of all elements over a wide concentration range. Only one reading of the sample is required, instead of the 2, 3 or even 4 readings that are required on conventional dual view instruments.

The 5100 includes a plug-and-play torch system that automatically aligns the torch and connects all gases for fast start up while ensuring reproducible loading of the torch from operator to operator. Minimizing instrument

to instrument variability within the sample introduction system (SIS) is important for labs that operate several instruments across multiple sites. These ease-of-use features of the 5100 reduce operator training time and greatly simplify method development and instrument operation.

This note describes the use of the Agilent 5100 SVDV ICP-OES for the trace elemental analysis of a bovine liver standard reference material (SRM).

Instrumentation

All measurements were performed using an Agilent 5100 SVDV ICP-OES with unique Dichroic Spectral Combiner (DSC) technology and vertical plasma that enables axial and radial view analysis at the same time. The sample introduction system consisted of a SeaSpray nebulizer, double-pass glass cyclonic spray chamber and a standard 5100 Dual View torch (demountable, quartz, 1.8 mm injector). The instrument uses a solid-state RF (SSRF) system operating at 27 MHz to deliver a robust plasma capable of excellent long term analytical stability.

The instrument operating conditions used are listed in Table 1 and the wavelengths and calibration parameters selected for the analysis are given in Table 2.

Table 1. The Agilent 5100 SVDV ICP-OES operating parameters used.

Parameters	Settings
Read time (s)	10
Replicates	3
Sample uptake delay (s)	20
Stabilization time (s)	10
Rinse time (s)	30
Fast pump (rpm)	80
RF power (kW)	1.20
Aux flow (L/min)	1.0
Plasma flow (L/min)	12.0
Nebulizer flow (L/min)	0.7
Viewing height (mm)	6

Experimental

Standard and sample preparation

To validate the method, the National Institute of Standards and Technology (NIST) Standard Reference Material (SRM) 1577 Bovine Liver was prepared for analysis. The SRM was digested using a Milestone Ethos microwave digestion system, with approximately 0.5 g of samples being accurately weighed into the digestion vessel. This was followed by the addition of 7 mL of concentrated HNO₃ and 1 mL of 30% H₂O₂. Samples were digested using the pre-loaded digestion methods, allowed to cool, then made up to 50 mL with DI water. The final acid concentration was approximately 12% v/v HNO₃.

A series of standards (1, 5, 10, 100, 250, 500 ppm) were prepared from multi-element standards in 1% HNO₃ and a multi-element spike of the bovine liver digest was prepared at 100 ppb.

Table 2. The wavelengths and calibration parameters selected for the analysis.

Element	Wavelength (nm)	Background Correction	Calibration Fit	Correlation Coefficient
K	766.491	Fitted	Linear	0.99966
Na	589.592	Fitted	Linear	0.99978
Fe	238.204	Fitted	Linear	0.99999
Cu	327.395	Fitted	Linear	1.00000
Zn	213.857	Fitted	Linear	1.00000
Mn	257.610	Fitted </td <td>Linear</td> <td>0.99998</td>	Linear	0.99998
Se	196.026	Fitted	Linear	1.00000
Pb	220.353	Fitted	Linear	0.99999
Cd	228.802	Fitted	Linear	1.00000
As	188.980	Fitted	Linear	1.00000
Ca	396.847	Fitted	Linear	0.99997
Co	238.892	Fitted	Linear	0.99999
Mg	279.078	Fitted	Linear	0.99968
Mo	202.032	Fitted	Linear	1.00000
Ag	328.068	Fitted	Linear	1.00000
Tl	190.794	Fitted	Linear	1.00000

Results and discussion

The linear dynamic range (LDR) for Na and K on the 5100 SVDV ICP-OES shows good linearity (Figures 1 and 2). Both elements can be calibrated up to 500 ppm with a correlation coefficient greater than 0.999 and less than 10% calibration error on each calibration point. This highlights the excellent LDR for Na and K in a single reading using the 5100 SVDV ICP-OES. On a conventional Dual View instrument, multiple readings of the sample would be required to achieve a comparable LDR.

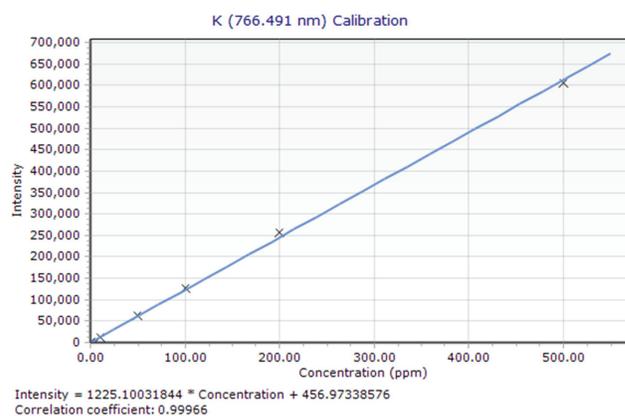


Figure 1. Calibration curve for K 766 line using the 5100 SVDV ICP-OES.

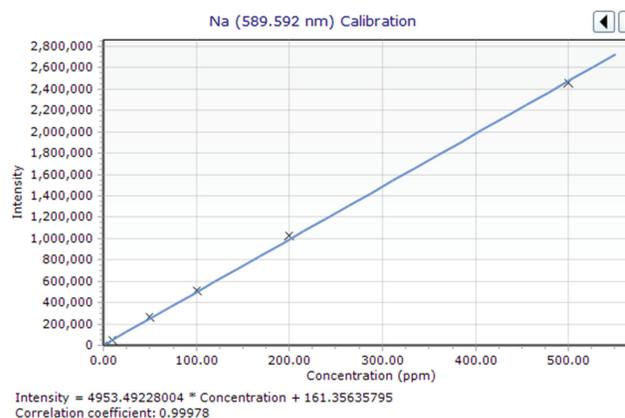


Figure 2. Calibration curve for Na 589 line using the 5100 SVDV ICP-OES.

All elements were determined in the Bovine Liver SRM digest using a single reading for major and trace elements. The results obtained with the 5100 SVDV ICP-OES for the certified values in the SRM are shown in Table 3, and Table 4 shows the results for the reference values in the SRM for those elements where certified values are not available. Good agreement with the certified and reference values was obtained, with the majority of results within 5% of the certified concentration.

The Method Detection Limits (MDL) were based on three sigma of ten replicate measurements of the blank solution.

Table 3. Results for NIST Bovine Liver 1577 SRM.

Element	MDL (mg/kg)	Measured values (mg/kg)	SD	Certified value (mg/kg)	SD	Recovery(%)
K 766	7.80	9832	5.2	9700	0.06	101
Na 589	9.08	2410	2.9	2430	0.013	99
Fe 238	0.17	258	1.9	270	20	96
Cu 327	0.16	203	1.1	193	10	105
Zn 213	0.33	131	0.56	130	10	101
Mn 257	0.008	9.8	0.01	10.3	1.0	96
Cd 228	0.13	0.26	0.02	0.27	0.04	96

Table 4. Results for NIST Bovine Liver 1577 SRM. Certified values are not available for the elements listed.

Element	MDL (mg/kg)	Measured values (mg/kg)	SD	Reference value (mg/kg)	Recovery(%)
Ca 396	6.0	126	0.16	123	103
Mg 279	0.83	603	2.4	605	100
Mo 202	0.18	3.4	0.05	3.2	106
Sr 407	0.01	0.142	0.002	0.140	102

For some elements present at trace levels, the levels in the SRM were below the limit of quantification. To further validate the method for these elements, the bovine liver digest was spiked with a multi-element standard at 100 ppb and the results are shown in Table 5. Excellent spike recoveries were achieved, with all elements showing 99% to 110% recovery.

Table 5. Results for Bovine Liver matrix spiked with 100 ppb multi-element standard.

Element	MDL (ppb)	Spike recovered values (ppb)	SD	Spike added (ppb)	Spike recovery (%)
Pb 220	4.8	109	0.003	100	109
Se 196	8.5	103	0.001	100	103
Co 238	2.0	110	0.002	100	110
Ag 328	2.1	107	0.001	100	107
As 188	12	99	0.004	100	99
Tl 190	7.7	103	0.001	100	103

Conclusions

The Agilent 5100 SVDV ICP-OES with DSC combines the sensitivity benefits of an axial plasma with the robust qualities of a radial plasma into a single platform so that all wavelengths can be detected in one measurement. This leads to greater precision, faster analysis times and reduced argon gas usage.

In this study, the 5100 SVDV ICP-OES was used to analyze a microwave-digested sample of a bovine liver SRM for a range of elements. An excellent linear dynamic range was demonstrated for Na and K, up to 500 ppm and good agreement with the certified and reference values was obtained.

The 5100 is ideally suited to meet the needs of food testing labs that require a high throughput, sensitive, multi-element technique, with a large linear dynamic range. Day-to-day operation and method development are simplified with a new, intuitive software interface and hardware features such as the plug-and-play torch that lead to excellent method repeatability between operators and from instrument to instrument.

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