Multi-Elemental Analysis of Edible Oils by ICP-OES

Reliable quality control for edible oil production with the Agilent 5800 VDV ICP-OES

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Introduction

Edible oils are integral to both domestic cooking and large-scale food processing, supplying essential fatty acids and enhancing flavor profiles. These oils are extracted from oilseeds—such as canola and soybean—and from oil crops such as palm. According to the latest OECD-FAO Agricultural Outlook report, global demand for these oils will remain strong over the next decade.[1]

To enhance product shelf life, taste, and aesthetic appearance, the oils undergo a series of refining and treatment procedures. Since each of these stages is a potential source of contamination, analytical testing is undertaken to identify any substances that have been added unintentionally.

Standards published by international organizations, such as the Codex Alimentarius General Standard for Contaminants and Toxins in Food and Feed, specify maximum levels for a few inorganic contaminants in edible oils.[2] Regional regulations and internal quality-control (QC) thresholds further define elemental impurity limits that manufacturers must meet. As a result, elemental testing is conducted at key stages of oil production, including receipt of the "undegummed" raw material, degummed crude oil, during and after refining, and in the final product. Robust analytical methods are required to accommodate the matrix challenges at each stage and to ensure product quality, consistency, and safety.

To support global harmonization of analytical methods, the American Oil Chemists' Society (AOCS) publishes a comprehensive collection of standardized protocols. In this study, we adapted the standard procedure described in AOCS Official Method Ca 17a-18: Determination of Trace Elements in Oil by Inductively Coupled Plasma Optical Emission Spectroscopy (22-Element Method).[3]

Analyzing carbon-rich oilseed matrices by ICP-OES poses several challenges. For example, the high-organic load can destabilize the plasma, suppress analyte signals, and deposit carbon on sample introduction components. To address these issues, the method requires robust instrumentation and optimized workflows. Agilent ICP-OES instruments have previously been used for the analysis of soybean and palm oils.[4,5] The Agilent 5800 Vertical Dual View (VDV) ICP-OES with Agilent ICP Expert Pro software was used in this study to perform multi-element analysis on canola oil samples representing the following four key stages of the refining process:

- Undegummed
- Crude
- Refined and bleached (RB)
- Refined, bleached, and deodorized (RBD)

Given the diverse composition of the four samples, smart tools within the ICP Expert Pro software were used to optimize method development, analytical performance, and maintenance schedules. The tools included IntelliQuant Screening, Fitted Background Correction (FBC) and Fast Automated Curve-fitting Technique (FACT) background correction techniques, and Early Maintenance Feedback (EMF).

Experimental

Instrumentation

All measurements were performed using an Agilent 5800 VDV ICP-OES with an Agilent SPS 4 autosampler and cover kit. The instrument is designed for speed, robustness, and stability. It features a vertical plasma optimized for the analysis of samples with a high solids content or organic matrix. It also uses a solid-state RF (SSRF) system operating at 27 MHz and a high speed (1 MHz) charge-coupled device (CCD) detector. The SSRF ensures that the plasma remains stable over long runs and the VistaChip III detector enables fast warmup, rapid analysis times, and high sensitivity.

An Easy-fit torch with a tapered 1.4 mm id quartz injector was used alongside the standard sample introduction kit, comprising a double-pass glass cyclonic spray chamber and a SeaSpray U-series concentric glass conical nebulizer. The fully demountable torch enables quick access to the quartz glassware for easy cleaning in a muffle furnace, while the torch's loading mechanism ensures automatic alignment of the plasma. The torch also includes a replaceable, extended high-purity quartz outer tube for organic applications. This torch configuration is recommended for routine radial view analysis of semivolatile organic solvents such as kerosene and xylene or samples that are prepared in these types of solvent. A mixed gas of oxygen in argon was injected through the optional gas inlet of the instrument to reduce carbon buildup on the sample introduction system and further increase plasma stability.

To reduce carryover between samples without increasing analysis time, the SPS 4 autosampler was enabled to perform bubble injection rinse. This feature introduces air bubbles at the start of the rinse cycle by repeatedly dipping the probe into the rinse port. Table 1 lists the instrument and method parameters.

Table 1. Agilent 5800 VDV ICP-OES instrument and method parameters.

Parameter	Setting	
RF Power (kW)	1.4	
Read Time (s)	10	
Plasma Gas Flow (L/min)	12.0	
Auxiliary Gas Flow (L/min)	1.20	
Nebulizer Flow (L/min)	0.7	
Viewing Mode	Radial	
Viewing Height (mm)	6	
Stabilization Time (s)	12	
Rinse Time (s)	30	
Bubble Injection Rinse	Enabled	
Replicates	3	
O_2/Ar (%)	40	
Peristaltic Pump Tubing	Sample	SolvaFlex, black/black
	Waste	SolvaFlex, blue/blue
	Internal Standard	SolvaFlex, green/orange*

** For on-line addition of the IS. However, gravimetric addition of the IS to the diluent is recommended for oil samples.*

Standard and sample preparation

Four calibration standards ranging from 1 to 25 mg/kg were prepared using Agilent standards in Agilent 75 cSt hydrocarbon oil. These standards included 500 µg/g A21+K and a single element arsenic (As) at 1000 µg/g. To achieve consistent viscosity, additional 75 cSt base mineral oil was added when needed, to give a total oil concentration of 20% w/w. The solutions were then diluted with Agilent "A-Solv" —an odorless kerosene—to achieve a 1:5 weight-by-weight (w/w) hydrocarbon-oil-to-A-Solv ratio. This same 1:5 w/w mixture was used as the calibration blank. The organic matrix can add unwanted background interferences that can interfere with some analytes in this study. Thus, the blank solution served as the matrix blank and interferent in the Fast Automated Curve-fitting Technique (FACT) model.

Three Continuous Calibration Verification (CCV) solutions were prepared to check the accuracy and robustness of the calibration throughout the analysis. The CCVs were prepared in an identical manner to the calibration standards, but with different stock standards. CCV solutions 1 and 2 comprised 2.5 and 12.5 mg/kg solutions of A21+K and As, respectively, while CCV 3 comprised a 100 mg/kg A21+K solution. CCV 3 was used to verify the linearity of the phosphorus (P) calibration.

The four commercially available canola oil samples were diluted five times (w/w) in A-Solv. This matrix matching ensured consistent viscosity of solutions across the analytical sequence.

To evaluate the accuracy of the method, a spike recovery test was performed on two aliquots of the RB canola oil sample at 0.5 and 50 mg/kg for all elements except As, which was spiked at 1 and 50 mg/kg. The multi-element spiking was done using Agilent 900 µg/g A21+K, another multi-element standard in 75 cSt hydrocarbon oil, and the 1000 µg/g single element standard for As.

Method development

Calibration

In this study, P was measured in the undegummed and crude canola samples at 100 mg/kg, which is equivalent to 500 mg/kg in the samples once the 5x dilution factor has been applied. This exceeds the upper calibration point of 25 mg/kg. To confirm linearity for all elements, including P up to 100 mg/kg, the three CCVs were analyzed in triplicate over three consecutive days. All elements recovered within ±10% of their expected concentration, validating the calibration.

The correlation coefficients listed in Table 2 were all above 0.9996, further confirming the linearity of the calibration curves for all elements. A representative calibration curve for Cr 283.563 nm up to 25 mg/kg is shown in Figure 1.

Background correction

The ICP Expert software includes various background correction techniques to address variable background signals from different sample matrices. The default FBC technique automatically models the background signal under the analyte peak, enabling accurate correction of both simple and complex background structures. FBC requires no input from the user.[6] For example, the P 213.618 nm line is interfered by Cu 213.598 nm. FBC can accurately model both peaks and the background structure, ensuring accurate correction of the P 213.618 nm peak (Figure 2).

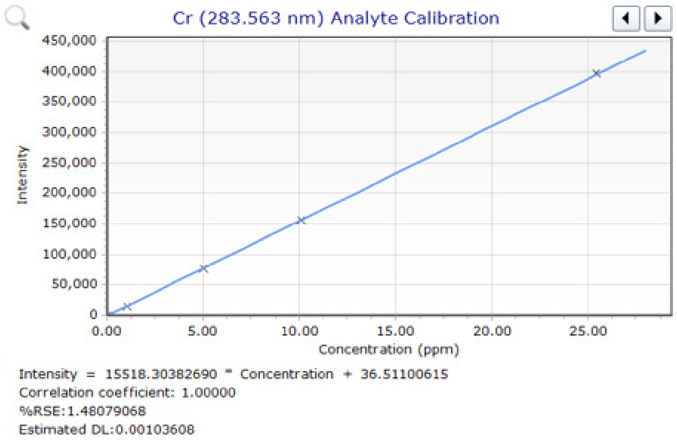
For more intense or overlapping background structures that FBC cannot fully correct, FACT is available.[7] FACT is effective for correcting background or elemental interferences that partially overlap the analyte peak, allowing for clearer separation of the analyte from the background and interferent.

As shown in Table 2, FBC was used for most of the analyte wavelengths in this study while FACT was used to provide definitive correction against any matrix-related interferences on the remaining elements. The FACT models used in this application included the matrix blank and the analyte only. A separate elemental interferent solution was not required as any spectral overlaps were due to the organic matrix rather than other elements. FACT correction was required for K 766.491 nm due to the intense matrix interference at 766.274 nm (Figure 3).

Applying the most appropriate background correction technique enables both lower detection limits and enhanced result accuracy, ensuring reliable quantification even in complex matrices. Co 228.615 (15 mg/kg) was used as the internal standard, minimizing possible fluctuations during the analysis.

Table 2. Element, wavelength, background correction selection, and correlation coefficient. Co 228.615 was used as the internal standard.

Element, Wavelength (nm)	Background Correction	Correlation Coefficient
Ag 328.068	Fitted	1.00000
Al 308.215	Fitted	0.99999
As 188.980	FACT	0.99997
B 249.772	Fitted	0.99981
Ba 614.171	Fitted	1.00000
Ca 396.847	Fitted	0.99993
Cd 214.439	Fitted	0.99996
Cr 283.563	Fitted	1.00000
Cu 327.395	Fitted	1.00000
Fe 259.940	FACT	0.99999
K 766.491	FACT	1.00000
Mg 280.270	Fitted	0.99997
Mn 257.610	Fitted	0.99999
Mo 284.824	Fitted	0.99997
Na 589.592	FACT	0.99999
Ni 231.604	FACT	0.99999
P 213.618	Fitted	1.00000
Pb 220.353	FACT	0.99998
Si 251.611	Fitted	0.99999
Sn 283.998	FACT	0.99999
Ti 334.941	Fitted	0.99999
V 309.310	Fitted	0.99987
Zn 206.200	Fitted	0.99999



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Intensity = 15518.30382690 * Concentration + 36.51100615
Correlation coefficient: 1.00000
%RSE:1.48079068
Estimated DL:0.00103608
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Figure 1. Cr 283.563 nm calibration curve with a correlation coefficient of 1.0000.

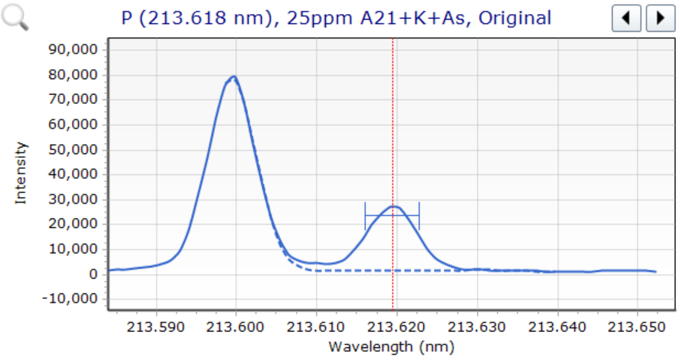


Figure 2. FBC used to correct background structures and a Cu interference on P 213.618 nm in a 25 mg/kg calibration solution. The dashed blue line represents the FBC model used for interference correction.

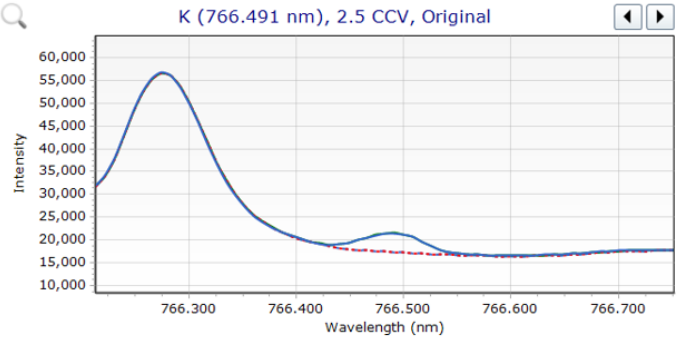


Figure 3. FACT model used to correct matrix-based interferences on K 766.491 nm in a 2.5 mg/kg CCV (the corrected signal is represented by the solid blue line). The signal from the CCV matrix-based interferences (red dashed line) overlaps the green analyte line. The blank is represented by the light-blue dashed line.

IntelliQuant Screening

The powerful IntelliQuant Screening semiquantitative analysis tool acquires full-spectrum measurements for each sample within seconds and calculates semiquantitative data based on premeasured calibrations.[8] For greater data accuracy in this application, an IntelliQuant calibration was prepared using two matrix-matched standards.

For method development before a full analysis, an IntelliQuant Screening was used to acquire semiquantitative data for the four canola oil samples. As shown in Figure 4, the periodic table 'heatmap' view of the results for the crude canola oil provided a helpful at-a-glance snapshot of the sample's contents, identifying high concentrations of K, Mg, Ca, and P. A pie graph view of the data (Figure 5) shows the relative concentrations of elements in the crude canola oil, allowing for quick comparison of the sample's elemental composition.

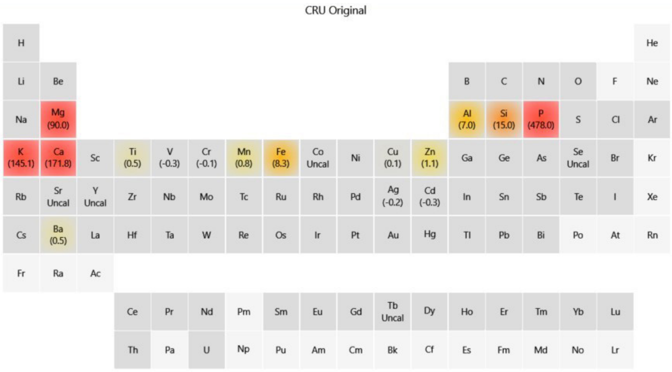


Figure 4. IntelliQuant Screening periodic table heatmap, showing approximate elemental concentrations (ppm) in the crude canola oil sample.

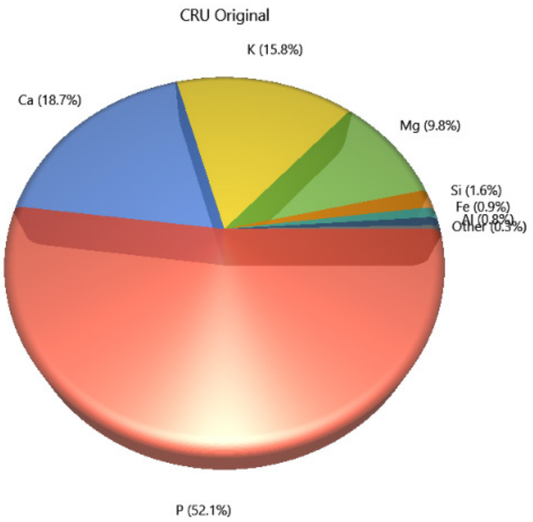
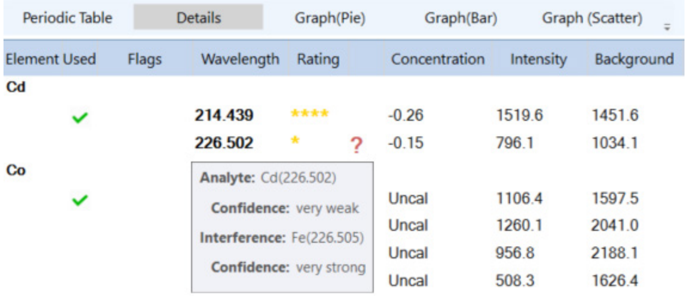


Figure 5. IntelliQuant Screening pie graph, showing relative concentrations (%) of elements in the crude canola oil sample.

IntelliQuant Screening also provided recommendations for wavelengths using a star-rating system. In samples like undegummed and crude canola oils that contain a relatively high concentration of Fe, IntelliQuant recommended the use of the Cd 214.439 nm line, as indicated by the green tick and five-star rating (Figure 6). Clicking on the red question mark next to the 226.502 nm line informs the analyst of a likely interference from Fe 226.505, as shown in the 'details' tab in Figure 6. This wavelength-selection feature ensures accurate, reliable quantitative results without wasting time on measuring interfered analyte lines.



Element	Used	Flags	Wavelength	Rating		Concentration	Intensity	Background
Cd								
	✓		**214.439**	★★★★		-0.26	1519.6	1451.6
			226.502	★	?	-0.15	796.1	1034.1
Co								
	✓					Uncal	1106.4	1597.5
						Uncal	1260.1	2041.0
						Uncal	956.8	2188.1
						Uncal	508.3	1626.4

Analyte: Cd(226.502)
Confidence: very weak
Interference: Fe(226.505)
Confidence: very strong

Figure 6. IntelliQuant Screening star-rating system, showing the best analyte wavelengths to use for the quantitative method. Hovering over the '?' reveals why a wavelength has not been recommended, as demonstrated by the Cd 226.502 nm line which is subject to a Fe interference.

Results and discussion

Spike recovery test

The RB canola oil was spiked at two levels: low (0.52 mg/kg for most elements, 1.09 mg/kg for As) and high (49.1 mg/kg for most elements, 45.0 mg/kg for As). The average spike recoveries from triplicate analyses, each conducted three times, for the 23 elements were all within 100 ±10% (Table 3). The results demonstrate the robustness of the 5800 ICP-OES system's ability to handle the organic matrix and provide accurate results.

Table 3. Recoveries of low- and high-level spikes of the RB canola oil with the Agilent 5800 VDV ICP-OES.

Element and Wavelength (nm)	Unadjusted RB Oil Sample	RB Oil Low Spike	RB Oil High Spike	Low Spike Recovery (%)	High Spike Recovery (%)
	Measured Concentration (mg/kg)				
Ag 328.068	<MDL	0.55	48	104	98
Al 308.215	0.01	0.55	49	102	99
As 188.980	0.02	1.1	46	100	101
B 249.772	0.02	0.56	49	101	100
Ba 614.171	0.01	0.55	48	102	98
Ca 396.847	0.03	0.59	46	104	93
Cd 214.439	<MDL	0.56	48	106	98
Cr 283.563	0.004	0.56	48	105	98
Cu 327.395	<MDL	0.55	48	104	98
Fe 259.940	0.01	0.57	48	106	99
K 766.491	0.05	0.59	48	101	98
Mg 279.553	0.01	0.58	45	106	91
Mn 257.610	0.005	0.57	48	106	97
Mo 284.824	0.01	0.55	49	102	99
Na 589.592	<MDL	0.55	48	103	98
Ni 231.604	<MDL	0.57	49	108	99
P 213.618	0.04	0.59	51	104	103
Pb 220.353	0.03	0.57	48	101	98
Si 251.611	0.08	0.59	48	96	98
Sn 283.998	<MDL	0.55	48	104	99
Ti 334.941	0.003	0.54	48	102	98
V 309.310	0.003	0.54	50	102	101
Zn 206.200	<MDL	0.55	49	104	100

Note: All unadjusted RB samples are above MDL unless noted. They are rounded to one significant figure.

MDLs and quantitative results

Method detection limits (MDLs) were determined by running 10 repeat analyses of a 1:5 oil-to-A-Solv blank (Table 4). The MDLs were calculated as three times the standard deviation of the concentration readings multiplied by the dilution factor (5x), averaged from running the worksheet three times on non-consecutive days. All MDLs were below or close to the estimated limits of detection (LODs) of the few elements in AOCS Ca 17a-18 and the regulatory maximum levels specified in CODEX 193-1995 for As and Pb at 0.1 and 0.08 mg/kg, respectively.[2,3]

Long-term stability

A 9.5-hour long-term stability test was conducted by analyzing a 5 mg/kg CCV after every 10 measurements of an oil sample (Figure 7). All measurements were within 100 ±10% of the expected concentrations, with %RSDs below 2.5% over the entire run.

To further assess instrument stability, a 30-minute stability test was conducted twice daily over five consecutive days. Each session began with a 10-minute warmup, followed by a calibration and the analysis of ten spiked crude oil samples. From each run, one representative element was selected, and its measured concentration was plotted over the five days (Figure 8). With all elements showing a %RSD below 3% and normalized concentrations within ±2.5%, the data confirms the stability and precision of the 5800 for stop-and-start analysis following a short warmup.

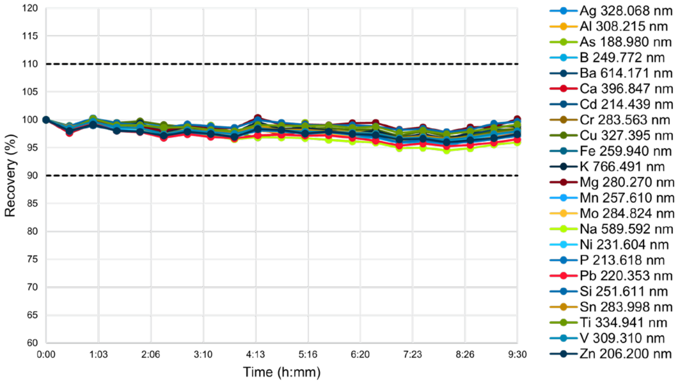


Figure 7. Long-term stability, showing normalized concentration (relative to the first reading) over 9.5 hours.

Table 4. Dilution-corrected MDLs and quantitative data for the four canola oil sample concentrations obtained using the Agilent 5800 VDV ICP-OES.

Element and Wavelength (nm)	MDL (mg/kg)	Estimated LOD in AOCS Method (mg/kg)	Concentration with Dilution Factor Applied (mg/kg)			
			Undegummed	Crude	RB	RBD
Ag 328.068	0.005		<MDL	<MDL	<MDL	<MDL
Al 308.215	0.063		5.38	5.93	0.071	<MDL
As 188.980	0.031		<MDL	<MDL	0.12	<MDL
B 249.772	0.008		0.04	0.03	0.10	0.02
Ba 614.171	0.001		0.16	0.20	0.16	<MDL
Ca 396.847	0.002	0.05	115.36	138.93	0.16	0.31
Cd 214.439	0.003	0.2	<MDL	<MDL	<MDL	<MDL
Cr 283.563	0.004		0.03	0.02	0.02	<MDL
Cu 327.395	0.010	0.05	0.017	0.20	<MDL	<MDL
Fe 259.940	0.009	0.05	5.35	7.03	0.06	0.03
K 766.491	0.201		104.96	125.95	0.23	<MDL
Mg 280.270	0.001	0.05	69.43	77.89	0.07	0.11
Mn 257.610	0.001		0.71	0.95	0.02	<MDL
Mo 284.824	0.017		<MDL	<MDL	0.03	<MDL
Na 589.592	0.021	0.1	0.82	0.56	0.056	0.056
Ni 231.604	0.021	0.05	<MDL	0.03	<MDL	<MDL
P 213.618	0.071	0.05	422.58	494.07	0.18	0.63
Pb 220.353	0.060	0.5	<MDL	<MDL	0.17	<MDL
Si 251.611	0.029	0.1	14.02	13.48	0.40	0.17
Sn 283.998	0.053		0.06	<MDL	<MDL	<MDL
Ti 334.941	0.001		0.34	0.30	0.02	<MDL
V 309.310	0.004		0.11	0.12	0.01	<MDL
Zn 206.200	0.006		0.90	1.27	<MDL	<MDL

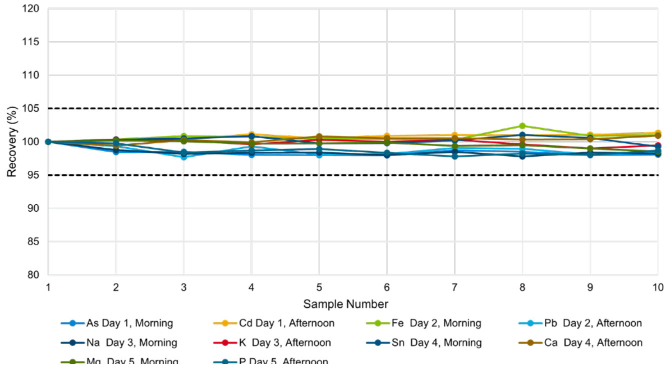
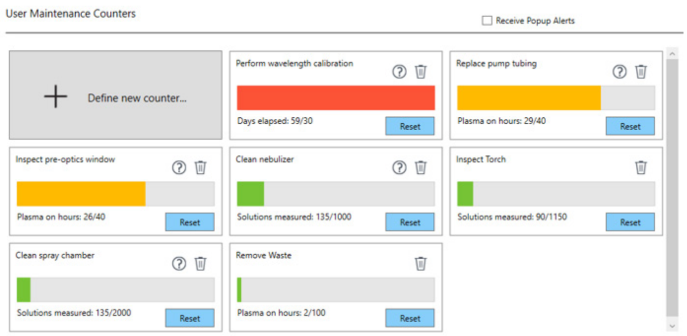


Figure 8. Stability study over five days, showing a selection of elements from morning and afternoon testing.

Early Maintenance Feedback

Using a series of counters and sensors, the EMF function alerts the operator when maintenance is required, ensuring the 5800 ICP-OES maintains optimal performance during use. The analysis of organic-based samples often causes carbon buildup in the sample introduction system, which can reduce the accuracy and precision of the quantitative results. Operating the 5800 ICP-OES under optimized conditions enabled 1150 samples to be analyzed before the torch required cleaning. This information supports setting the user-defined solution counter for “Inspect torch” to a higher threshold than anticipated, reducing unnecessary interventions and saving analyst time. The EMF’s traffic light color-coding system indicates maintenance status and highlights tasks that are required to be performed immediately, simplifying its use (Figure 9).



Perform wavelength calibration: Days elapsed: 59/30

Replace pump tubing: Plasma on hours: 29/40

Inspect pre-optics window: Plasma on hours: 26/40

Clean nebulizer: Solutions measured: 135/1000

Inspect Torch: Solutions measured: 90/1150

Clean spray chamber: Solutions measured: 135/2000

Remove Waste: Plasma on hours: 2/100

Figure 9. Maintenance counters for EMF, ensuring the Agilent 5800 VDV ICP-OES remains in optimal condition during use.

Conclusion

The Agilent 5800 VDV ICP-OES powered by Agilent ICP Expert Pro software simultaneously quantified 23 elements in four types of canola oils. The undegummed, crude, refined and bleached (RB), and refined, bleached, and deodorized (RBD) samples were diluted in A-Solv before analysis.

For the analysis of the diluted oil samples, the 5800 was configured with the standard sample introduction system and Easy-fit organic torch. A gas mix of oxygen in argon was used to reduce carbon buildup.

To assist the analyst during method development and with instrument maintenance while ensuring data quality throughout the analytical sequence, the following software tools were used:

- IntelliQuant Screening: This powerful routine streamlined method development by recommending optimal calibration ranges and interference-free wavelengths for all analytes, ensuring accurate analysis of analytes in the edible oil samples.
- FBC and FACT background correction: These techniques successfully corrected for background structures and spectral interferences arising from the various canola oil sample matrices, ensuring low detection limits and data accuracy.
- Early Maintenance Feedback: Extensive sensors and counters alert the analyst when maintenance is required based on actual use, maintaining instrument performance and reducing downtime during unnecessary maintenance. The torch did not require cleaning for 1150 samples, due to the robust design of the instrument, sample introduction system, and parameter optimization.

All MDLs were below or close to the estimated limits of detection specified in AOCS Ca 17a-18, confirming the instrument's sensitivity for the quantification of trace elements in edible oils. The accuracy of the 5800 ICP-OES method was confirmed by recoveries of all analytes spiked at high and low levels in the RB oil, with results within 100 ±10%. Excellent long-term stability was demonstrated during the analysis of oil samples over 9.5 hours. Recoveries of the 5 mg/kg QC sample were within ±10% of the expected values, with RSDs ≤2.5% for all elements over the entire run. The 5800 also remained stable during a stop-and-start analysis following short warm up times, showing the reliability of the SSRF system.

The study has shown that manufacturers and importers of edible oils can confidently rely on the 5800 VDV ICP-OES method for trace element quality control throughout the production process, in accordance with established official methods.

References

1. OECD-FAO: OECD-FAO Agricultural outlook 2025-2034 (accessed November 2025)
2. CODEX 193-1995: fao.org/fao-who-codexalimentarius/sh-proxy/en/?lnk=1&url=https%253A%252F%252Fworkspace.fao.org%252Fsites%252Fcodex%252FStandards%252FCXS%2B193-1995%252FCXS_193e.pdf (accessed November 2025)
3. AOCS Ca 17a-18: Determination of Trace Elements in Oil by Inductively Coupled Plasma Optical Emission Spectroscopy (22-Element Method) (accessed November 2025)
4. Kapadnis, G.; Ghosh, B.; Jain, V.; Kar, P.; Vig, N.; Balakrishnan, K. Determination of Dimethyl-polysiloxanes (DMPS) in Edible Fats and Oils by ICP-OES, Agilent publication, 5994-4440EN
5. Shuping, L. Elemental Analysis of Palm Oil using ICP-OES, Agilent publication, 5994-1952EN
6. Fitted Background Correction (FBC) —fast, accurate and fully automated background correction, Agilent publication, 5991-4836EN
7. Real-time Spectral Correction of Complex Samples using FACT Spectral Deconvolution Software, Agilent publication, 5991-4837EN
8. Agilent IntelliQuant Screening: Smarter and quicker semiquantitative ICP-OES analysis, Agilent publication, 5994-1518EN

Products used in this application

Agilent products

SolvaFlex black-black tubing

SolvaFlex blue-blue tubing

SolvaFlex green-orange tubing

Easy-fit fully demountable radial view torch with a 1.4 mm id quartz injector for semi-volatile organics

SeaSpray U-series concentric glass nebulizer

Glass, double-pass spray chamber

500 µg/g A21+K wear metal standard in 75 cSt hydrocarbon oil

900 µg/g A21+K wear metal standard in 75 cSt hydrocarbon oil

1000 µg/g arsenic (As) single element standard in 75 cSt hydrocarbon oil

5000 µg/g cobalt (Co) single element standard in 75 cSt hydrocarbon oil

75 cSt hydrocarbon oil

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