# Benefits of running organic matrices using the Agilent 5100 ICP-OES—fast, robust, high performance analysis

## Technical Overview

### 5100 ICP-OES

## Introduction

Many laboratories are required to analyze metals present in organic solvents and chemicals. Typical applications include the analysis of used and unused lubricating oils and base oils for additives elements and wear metals [1], or metals in organic matrices such as crude petroleum, asphalts, gas oils, diesel and jet fuels and their blending components, pyrolysis oils, and fatty acid derivatives [2]. Inductively coupled plasma optical emission spectrometry (ICP-OES) is one of the most widely used techniques for monitoring metals in organic based products due to its inherent robustness and ease-of-use. However, the analysis isn't without its challenges. The maintenance of a stable and robust plasma is difficult to achieve when introducing organics to the ICP, particularly volatile ones, and the carbon content of organics gives rise to interferences due to the formation of C-based molecular bands.

Agilent Technologies

The Agilent 5100 Synchronous Vertical Dual View (SVDV) ICP-OES with solid state RF generator and flexible background correction offers a new approach for the high performance analysis of organics. Corrosion-resistant materials and internal positive pressure keep corrosive vapors out, ensuring reliability even in the harshest lab environments.

## Sample introduction kits

Agilent has a range of sample introduction systems for the analysis of organics, with selection depending on the volatility of the samples. A convenient ICP-OES application kit for common organic solvents like kerosene, xylene and white spirits features a 1.4 mm id Easy-fit torch, solvent resistant tubing, and a double-pass glass cyclonic spray chamber. The kit that is designed for the analysis of volatile organics includes elastomer pump tubes, a concentric nebulizer, and a demountable Easy-fit torch with 0.8 mm id injector.

## Robust and reliable plasma

### Solid state RF system and multiple MFCs

A key consideration when running organics on an ICP-based instrument is whether the combination of radio frequency (RF) generator and torch system is robust enough to sustain the plasma throughout the analysis. The 5100 ICP-OES features a solid state RF (SSRF) system that operates at 27 MHz, over a wide power-range of 700 W to 1500 W in a dual view configuration. This is in contrast to other dual view ICP-OES systems, where the RF power is limited to 1350 W. As the Agilent RF system is able to rapidly adjust to changes in plasma conditions, the 5100 ICP-OES can handle a wide range of organic samples, from volatile organics such as methanol, gasoline or naphthalene to semi-volatile organics such as biodiesel or wear metals in oils. Long term stability of the emission signal is achieved through the accurate control of all plasma gas flows in the torch using mass flow controllers (MFCs) and the use of thermostat-controlled optics. In fact, plasma conditions similar to those used for aqueous samples can be used for organics, without the need for increased plasma gas flows. To demonstrate this capability, a 5 ppm multi-element organometallic standard (Conostan S-21) in kerosene was run as a sample for 4 hours, with a rinse step included between each sample. The %RSD for all elements was found to be less than 1% over the 4 hour period (Figure 1), demonstrating excellent stability over the analytical period.

### Vertically oriented plug-and-play torch

Agilent's 5100 ICP-OES features a vertical torch in all three available configurations of the instrument (SVDV, Vertical Dual View and Radial View), and the SVDV-configured model can be operated in axial, radial, vertical dual view and synchronous vertical dual view modes for maximum flexibility [3].

A vertically oriented torch is the standard configuration for analyzing organic matrices. However, vertically oriented torches are usually viewed radially leading to a trade off in sensitivity. This is not the case for the 5100 ICP-OES which delivers the detection limits that are typical of an axially viewed plasma instrument. Furthermore, the vertical plasma prevents carbon from building up on the torch and fewer solvent droplets from accumulating in the injector tube, leading to less maintenance and torch cleaning, and fewer replacement torches.

Agilent's plug-and-play torch is a quick, simple and effective torch loader mechanism that automatically aligns the vertical torch and connects all gases for fast start up and reproducible performance (Figure 2). Once the torch has been loaded, there is no need for further adjustments of the torch and no optical alignment of the axial viewing position is required. This greatly reduces instrument-to-instrument variability, leading to more consistent and reproducible results.

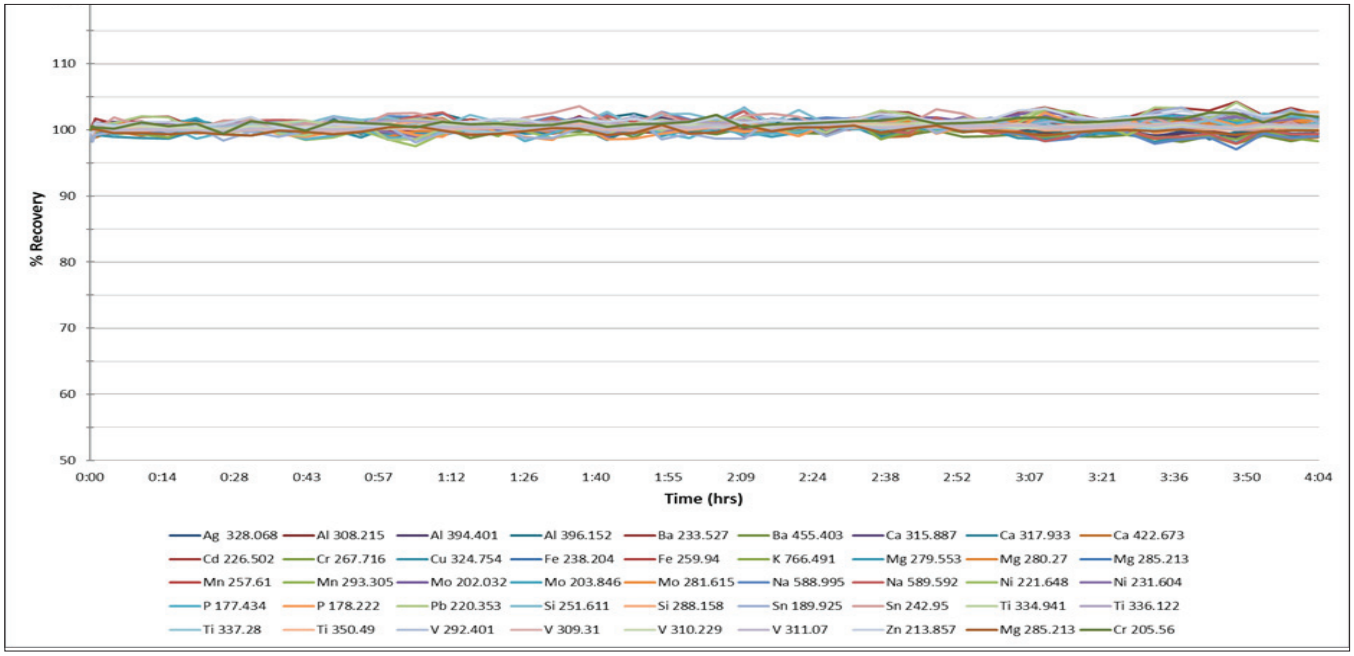


**Figure 1.** Long-term stability plot for all wavelengths of a 5 ppm multi-element organometallic standard in kerosene.


**Figure 2.** Sequence of 3-easy steps to load the torch into the instrument for fast start up and reproducible performance.

## Analytical performance

### Linear dynamic range (LDR)

Linear calibrations with a correlation coefficient greater than 0.999 were obtained for all wavelengths studied in an oil sample diluted in kerosene. Figure 3 shows a calibration curve for Ca 422.673 up to 50 ppm with a correlation coefficient greater than 0.9999 and less than 2% calibration error on each calibration point. Excellent linearity for all wavelengths demonstrates the capability of 27 MHz SSRF system to maintain a stable plasma when analyzing organic samples, ensuring that concentrations beyond the highest calibration standard can be measured with confidence in samples such as additives or wear-metals in oil. The expansive LDR also allows the number of calibration standards to be reduced, which means more time can be spent analyzing samples, with less time spent on calibration.

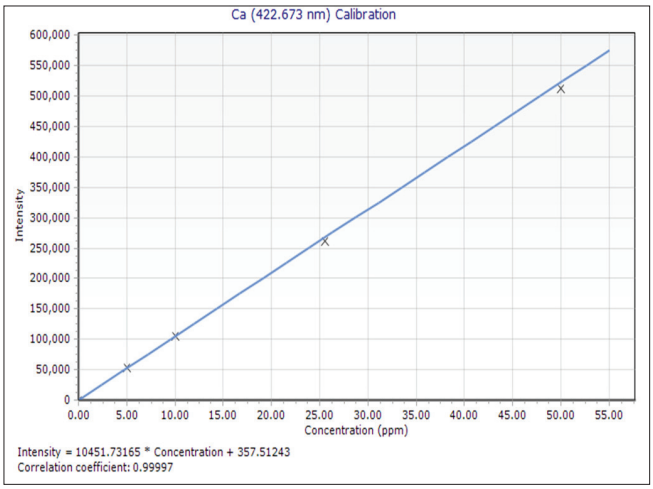


**Figure 3.** Calibration curve for Ca (422.673 nm) in oil diluted in kerosene.

## FACT background correction

The complex background structure that arises during the analysis of organics can create analytical challenges, particularly where low detection limits are required. Using Agilent's Fast Automated Curve-Fitting Technique (FACT) background correction software algorithm to model the background, a far more accurate measurement of the analyte signal can be achieved compared to other background correction techniques [4]. FACT models are easily created based on the spectrum of a blank and analyte. As can be seen in Table 2, for the determination of sodium and potassium in biodiesel dissolved in kerosene, FACT can lower the quantitative limit by an order of magnitude.

**Table 1.** Comparison of Fitted Background Correction (FBC) and FACT background correction techniques on the typical MDL of K and Na in biodiesel dissolved in kerosene.

| | MDL (ppm) | | | |
|---|---|---|---|---|
| | **FBC K (766.491)** | **FACT K (766.491)** | **FBC Na (588.995)** | **FACT Na (588.995)** |
| **Axial** | 0.069 | 0.010 | 0.048 | 0.009 |
| **Radial** | 0.052 | 0.035 | 0.087 | 0.002 |
| **SVDV** | 0.044 | 0.036 | 0.021 | 0.006 |

As demonstrated in Figure 4, FACT removes complex background structures providing accurate determination of low level sodium in used oils.

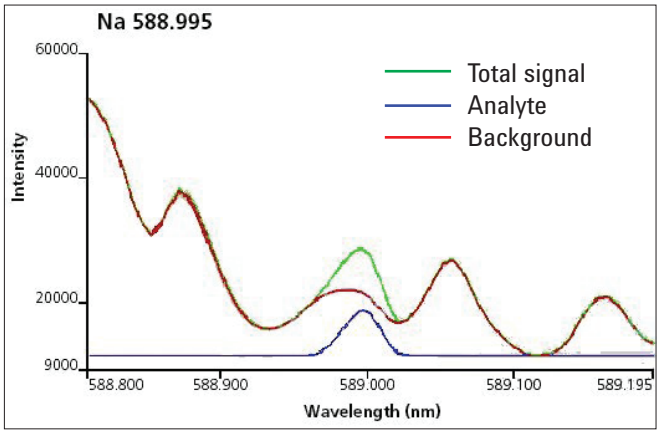


**Figure 4.** FACT background correction on Na (588.995 nm) in a used oil sample.

### Rapid speed of analysis and low argon consumption

The 5100 ICP-OES uses a high speed VistaChip II CCD detector with continuous wavelength coverage that is ideally suited to applications requiring high throughput, high sensitivity, and a wide dynamic range. Productivity can be further increased by fitting the 5100 ICP-OES with an Agilent SVS 2+ Switching Valve System that is positioned between the spray chamber and the peristaltic pump of the spectrometer. The SVS 2+ rinses the sample introduction system while the next sample is being introduced, reducing sample uptake, stabilization times, and rinse delays. The increase in sample throughput means that less argon is consumed during an analysis leading to a reduced cost per analysis, without any compromise in analytical performance.

In a recent study using the Agilent 5100 ICP-OES for the determination of metals in oil samples per the ASTM D5185 method for lubricating oils for wear metals and additives, the 5100 ICP-OES was fitted with an SVS 2+ and an Agilent SPS 3 Autosampler for automated sample introduction [5]. A sample analysis cycle time of 30 seconds per sample and low total gas consumption of 9.5 L argon per sample was achieved. For a high throughput lab, the fast sample analysis time equates to 120 samples per hour or 960 samples for an 8 hr day.

## Summary

By combining a series of innovative hardware and software features, the Agilent 5100 ICP-OES is ideally suited to the determination of trace metals in organic samples. The advantages are:

- **Robust and sensitive.** The vertical torch orientation provides a high level of robustness allowing analysts to measure semi- and volatile organic solvents with the sensitivity associated with an axially viewed plasma.
- **Reliable, maintenance free plasma.** The instrument's solid state RF system creates a plasma that is suitable for the stable analysis of organics over long analysis times.
- **Improve detection limits using FACT.** Spectral matrix effects caused by the carbon present in organic samples can be modelled and removed using FACT background correction within the ICP Expert software.
- **Save time and reduce costs.** For labs looking to maximize the productivity of their ICP-OES analyses, the 5100 ICP-OES with its high speed VistaChip II CCD detector fitted with an innovative SVS 2+ switching valve delivers exceptional sample throughput and the lowest gas consumption per sample of any ICP-OES.

## References

1. ASTM D5185-13, Standard Test Method for Multielement Determination of Used and Unused Lubricating Oils and Base Oils by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES)

2. ASTM UOP389-14, Trace Metals in Organics by ICP-OES, ASTM International, West Conshohocken, PA, (2014), www.astm.org

3. Real-time spectral correction of complex samples using FACT spectral deconvolution software, Agilent publication 5991-4837EN, (2014)

4. Benefits of a vertically oriented torch— fast, accurate results, even for your toughest samples, Agilent publication 5991-4854EN, (2014)

5. Improved productivity for the determination of metals in oil samples with ASTM Method D5185, using the Agilent 5100 Radial View (RV) ICP-OES, Agilent publication 5991-5271EN, (2014)

**www.agilent.com**



Published February 20, 2015
Publication number: 5991-5498EN

**Agilent Technologies**