How the Latest Advancements in ICP-OES Instrumentation and Software Address the Requirements of Demanding Methods

Poster Note 43307

Sanja Asendorf, Matthew Cassap, Elena Chernetsova
Thermo Fisher Scientific, Bremen, Germany

Overview

Purpose: To demonstrate the capability of the Thermo Scientific™ iCAP™ 7000 Plus Series ICP-OES with Thermo™ Scientific Qtegra™ Intelligent Scientific Data Solution™ (ISDS) software for the analysis of petrochemical samples.

Methods: Samples were simply diluted in white spirit and analyzed directly by radial Inductively Coupled Plasma – Optical Emission Spectrometry (ICP-OES) in conjunction with a Sprint sample introduction system. These results were compared to standard Speed method of analysis (peristaltic pump introduction).

Results: All of the samples analyzed by both methods had similar results demonstrating the suitability for this type of analysis.

Introduction

Analysis of trace elements by ICP-OES is a well established technique and is a requirement of many standard methods and protocols. One example of a standard method for petrochemical samples, is the American Society for Testing and Materials (ASTM) method for the analysis of new and used lubrication oils: ASTM D5185 “Determination of Additive Elements, Wear Metals, and Contaminants in Used Lubrication Oils and Determination of Selected Elements in Base Oils” by ICP-OES. This method is typically used as a tool for regular testing of used oils sampled from cars, train fleets, or even large construction or mining machines.

Wear metals are elements such as Fe, Cu and Ni, and their presence may indicate wear of metallic engine components. Other elements may provide evidence of contamination from foreign matter, for example Si entering the engine via a faulty filter. Additives, which are typically Ca, P or Zn based compounds, are added artificially to the oil to improve lubricating properties. Monitoring the depletion of these elements may therefore help in identifying optimum conditions and maintenance scheduling.
This work describes how coupling an innovative sample introduction system to powerful instrumentation, enables analysis time to be reduced significantly while maintaining the analytical requirement of the industry. In turn, this allows rapid decisions to be taken and imminent mechanical failure to be identified.

Methods

Sample Preparation

For all elements, except S, standards at 50, 100, 250 and 500 mg/kg were prepared, plus standards at 1000, 2500 and 5000 mg/kg for Ba, Ca, Mg, P and Zn. Separate standards were also prepared at 2500 and 5000 mg/kg for S. Y was used as an internal standard and added to the white spirit to obtain a 20 mg/L Y solution used for all further dilutions. All samples and standards were therefore diluted 1:10 (w/v) with the latter.

Thermo SCIENTIFIC

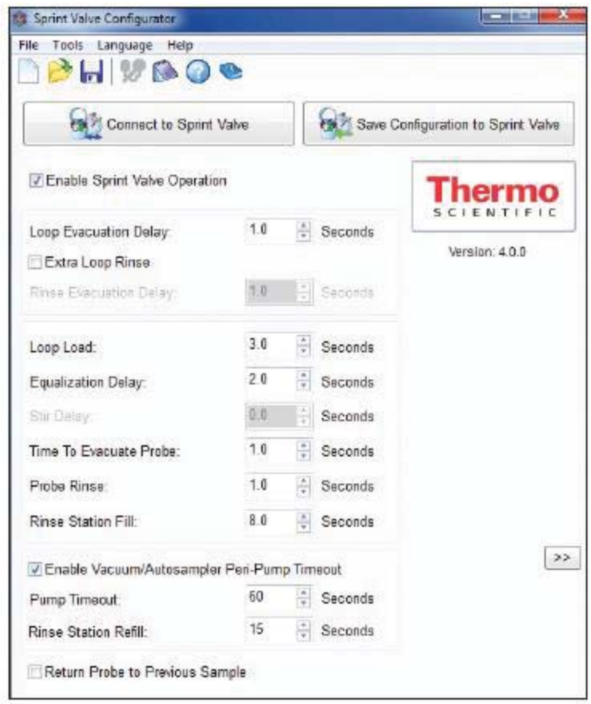
Test method

The Thermo Scientific™ iCAP™ 7600 ICP-OES Radial was chosen for the analysis. The Sprint valve of the iCAP 7600 ICP-OES was used in conjunction with the Sprint analysis mode to enable extremely fast analysis times. The standard organic sample introduction kit was also fitted to the instrument. The kit comprises a V-groove nebulizer, a baffled spray chamber and a 1mm center tube. All parameters used for the analysis including settings of the Sprint valve are listed in Table 1 and Figure 1. When using the sampling valve, flush and rinse behaviors are driven by the valve settings. There is no need therefore for faster flush pump speed or wash time to be set as with traditional methods. The flush pump rate is identical to the analysis pump speed (no pump stabilization time required) and the wash time in the method is set to zero.

TABLE 1. Parameters used for the analysis

Instrument parameters	
Inlet pump tubing	Solvent Flex (1.016 mm)
Outlet pump tubing	Solvent Flex (1.524 mm)
Loop size	1.02 mL
Pump speed	40 rpm
Uptake time	12 sec
Nebulizer	V-groove
Spray chamber	Baffled cyclonic
Centre tube	1 mm
Torch	EMT
Plasma parameters	
RF power	1350 W
Nebulizer gas flow	0.4 L/min
Auxiliary gas flow	1.5 L/min
Coolant gas flow	14 L/min
Radial viewing height	12 mm
Analysis parameters	
Analysis mode	Sprint
Exposure time	1 sec
Repeats	2

FIGURE 1. Sprint valve settings



Analysis and Results

Two typical oil samples (Oil A and Oil B) were analyzed following the Sprint method described previously. The results are shown in Table 2. They were compared to the concentrations obtained for the same oils analyzed with a traditional method. The Speed analysis was performed using the instrument peristaltic pump in a conventional way (no Sprint valve). Speed analysis mode was selected with five seconds integration time and two replicates. Analytical wavelengths were optimized for the method and may be different than defined for the Sprint analysis.

TABLE 2. Results of the analysis for the Sprint and Speed methods

Element	Oil A		Oil B	
	Sprint	*Speed*	*Sprint*	*Speed*
Ag	< 0.5	< 0.1	< 0.5	< 0.1
Al	2.1	1.9	8.9	9.6
B	< 2	0.8	< 2	1.3
Ba	< 1	0.2	3.0	3.2
Ca	2740	2830	17330	17750
Cd	< 0.2	< 0.05	< 0.2	< 0.05
Cr	0.9	0.8	0.8	0.7
Cu	1.3	1.6	1.7	1.6
Fe	20.6	20.6	30.6	29.3
Mg	248	246	41	41
Mn	0.6	0.6	5.0	5.3
Mo	4.3	4.4	1.1	0.8
Na	7.0	7.0	87	84
Ni	< 0.5	0.3	67	70
P	1040	1060	360	369
Pb	< 2	1.0	< 2	< 0.5
S	8180	7670	15010	14030
Si	4.2	4.3	20.0	18.9
Sn	< 5	< 1	< 5	< 1
Ti	< 1	< 0.2	1.5	0.6
V	< 1	< 0.2	62	66
Zn	1220	1240	419	419

Conclusion

The Thermo Scientific iCAP 7600 ICP-OES Radial was used successfully for high throughput lubricating oil analysis and the data demonstrated:

- The intelligent design of the sample introduction system with an integrated sampling valve enabled analysis time of less than 30 seconds per sample .
- Analytical performance was also demonstrated, providing accuracy, precision and stability for hundreds of samples, reducing the number of QC, re-calibrations and samples to be re-analyzed, and reducing cost of analysis per sample overall.
- The iCAP 7600 ICP-OES instrument offers superior analysis capacity for laboratories seeking optimum productivity.

www.thermofisher.com

Africa +43 1 333 50 34 0
Australia +61 3 9757 4300
Austria +43 810 282 206
Belgium +32 53 73 42 41
Canada +1 800 530 8447
China 800 810 5118 (free call domestic) 400 650 5118
Denmark +45 70 23 62 60
Europe-Other +43 1 333 50 34 0
Finland +358 10 3292 200
France +33 1 60 92 48 00
Germany +49 6103 408 1014
India +91 22 6742 9494
Italy +39 02 950 591
Japan +81 45 453 9100
Korea +82 2 3420 8600
Latin America +1 561 688 8700
Middle East +43 1 333 50 34 0
Netherlands +31 76 579 55 55
New Zealand +64 9 980 6700
Norway +46 8 556 468 00
Russia/CIS +43 1 333 50 34 0
Singapore +65 6289 1190
Spain +34 914 845 965
Sweden +46 8 556 468 00
Switzerland +41 61 716 77 00
UK +44 1442 233555
USA +1 800 532 4752

Thermo SCIENTIFIC
A Thermo Fisher Scientific Brand

PN43307-EN 0216S