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Analytical testing of trace elements in refinery products using a robust ICP-MS approach

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Goal

The objective of this application note is to establish an analytical method that enables analytical testing of typical samples relevant to the petrochemical industry (including starting materials, such as crude oil, and high purity finished products, such as naphtha, with different boiling point ranges) using inductively coupled plasma mass spectrometry (ICP-MS).

Introduction

Control of impurities in the petrochemical industry is an essential step to ensure the quality of the crude, intermediates, and final products. Typical impurities such as metals (e.g., nickel, vanadium, mercury, or lead) are known as catalyst poisons that can affect the products even at very low levels. Premature failures of catalysts can lead to interruptions in the production cycle and high costs. Other elements, such as arsenic or sulfur, are liberated as toxic emissions when fuels are combusted, and hence their concentration is ever more regulated in common products. Sulfur in particular is a common impurity in crude oil and must be removed from most transportation fuels to meet ever more stringent air quality requirements.

In many laboratories, the analysis of crude, intermediates, and final products for trace elements is accomplished using inductively coupled plasma optical emission spectrometry (ICP-OES) or ICP-MS. ICP-OES has the advantage of high tolerance to different sample types, and especially its higher tolerance to organic solvents. ICP-MS is less commonly used, although it offers detection limits improved by several orders of magnitude.

The different viscosities of petroleum and many of its derivatives make it difficult to analyze these samples directly by ICP-OES or ICP-MS. As a consequence, the aspiration of organic liquids into the plasma normally requires additional equipment or sample pretreatment to maintain plasma stability, reduce carbon build up

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and minimize background interferences. Analysis of combustible organic samples like naphtha, vacuum gas oil (VGO), or aviation turbine fuel (ATF) using ICP-MS is more challenging compared to conventional aqueous samples due to the higher carbon content, which increases the potential for unexpected and intermittent extinguishing of plasma as well as carbon deposition in the interface region. The problems are mainly related to the fact that carbon from these substrates is often not completely ionized in plasma due to its high ionization potential (11.3 eV), leading to unionized carbon deposition on the interface.

Carbon-based interference on key analytes, such as chromium or vanadium, causes additional concerns but can typically be overcome using kinetic energy discrimination (KED) on single quadrupole instruments. Alternatively, reactive gases such as hydrogen have been proven to remove intense argon-based interferences. The use of triple quadrupole ICP-MS systems can further improve the detection of some elements challenged by severe interferences, such as sulfur.

The main objective of this application note is to establish a method enabling the analysis of a variety of typical samples relevant to the petrochemical industry, including starting materials such as crude oil up to high purity finished products such as naphtha with different boiling ranges, using ICP-MS. Optimized detection limits using dedicated modes of analysis were determined, and key analytes benefiting from the use of triple quadrupole ICP-MS were identified.

Experimental

Chemicals

- Fisher Scientific™, Xylene Certified AR for analysis (P/N X/0250/PB17)

- CONOSTAN™ Standard S-21 + K, 100 ppm (P/N 150-021-052)

- CONOSTAN oil base standards of Hg, As, Sb, Se, S, Y, and In SPEX CertiPrep™ Co Standard

The NIST™ standard reference material (SRM) of Fuel oil, NIST 1634c Trace elements in fuel oil (Gaithersburg, MD, USA) has been used to demonstrate accuracy and precision of the analytical method.

Sample preparation

A major challenge when analyzing different types of petrochemical samples is the difference in vapor pressure and viscosity, ultimately leading to the need for a dedicated configuration of the sample introduction system. Whereas samples with high viscosity, such as crude oil or fuel oil, require heating of the sample introduction system components to remain fluid, samples with a lower boiling point, such as naphtha, need cooling of the spraychamber to avoid overloading the plasma and sudden extinction. To homogenize the properties of all samples (e.g., viscosity) and with the objective to accommodate different sample types in a single batch, samples were prepared differently in each case.

Fuel oil/crude oil sample preparation: The oil sample was initially heated in a water bath to 40–60 °C and mixed thoroughly to ensure the homogeneity. About 1 g of homogenized oil sample was then weighed accurately in a 50 mL volumetric flask followed by dilution of ~300-fold using xylene as diluent employing further serial dilutions.

Heavy naphtha: Heavy naphtha was aspirated directly into the ICP-MS after addition of the required amount of internal standards stock solution.

Light naphtha: Light naphtha was diluted 10-fold using xylene followed by addition of the required amount of internal standards stock solution before aspiration into the ICP-MS.

For the quality control (QC) standard, an independently prepared solution containing 10 µg·L⁻¹ of all analytes in xylene was used to verify the validity of the calibration curve and was analyzed every 10 samples throughout the measurement of a larger batch.

Table 1. Concentration of elements in calibration standard solutions (µg·L⁻¹, except for sulfur given in mg·L⁻¹)

Analytes	LL-1	LL-2	LL-3	LL-4	LL-5	LL-6
B, Na, Mg, Al, K, Ca, Ti, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, As, Se, Mo, Ag, Cd, Sn, Sb, Ba, Hg, Pb	0.1	1	5	10	25	50
S	5	10	50	100		

To analyze various elements in a single consolidated method, six different calibration standards were prepared using a serially diluted stock standard solution of 1 mg·kg⁻¹ (w/w), prepared using oil-based single and multi-element standard solutions containing all analytes of interest and using xylene as a diluent. To cover the concentration range expected for the different samples, the calibration standards were prepared to cover a wide range of concentrations, ranging from 0.1 to 50 µg·L⁻¹ for common trace impurities. Similarly, separate standard solutions were prepared for sulfur in a concentration range of 5-100 mg·L⁻¹.

To improve accuracy and compensate for potentially occurring drift during a longer sequence, internal standardization has been applied. Yttrium and indium were spiked at a concentration of 20 µg·L⁻¹ to each blank, standard, or sample, and used as internal standards in the experiment. Although the selection of the internal standard should ideally match the mass range and ionization potential of the analytes in the best possible manner, a suitable correction is nevertheless achieved also for analytes of considerably lower (such as boron, sodium, magnesium, and aluminum) and higher mass (such as mercury and lead).

Instrumentation

A Thermo Scientific™ iCAP™ RQ ICP-MS equipped with 250 mL·min⁻¹ additional mass flow controller has been used in the study. The system was operated in conjunction with a Teledyne CETAC™ ASX-560 autosampler (Teledyne CETAC Technologies, Omaha, NE, USA). The sample introduction system was configured for the analysis of organic samples using the components summarized in Table 2. To tackle the challenge of carbon deposition, a constant flow of pure oxygen was introduced into the spraychamber elbow using an additional mass flow controller.

Pure helium and hydrogen were introduced to the collision/reaction cell (CRC) using dedicated mass flow controllers to enable operation with KED and hydrogen reaction mode in a single method, analyzing 26 various elements in single sample aspiration. Whereas the use of an inert gas such as helium collision with KED is an effective and proven approach to remove polyatomic interferences, use of reaction mode using a reactive gas such as hydrogen has shown effective removal of highly intense polyatomic interferences, mainly argon and carbon-based polyatomic interferences. Other reactive gases, such as oxygen and ammonia, can be used for interference removal as well but require a triple quadrupole ICP-MS instrument in order to pre-filter the ion beam and avoid the formation of additional interferences with other elements contained in the sample. Some elements that benefit significantly from the use of hydrogen in this matrix are listed in Table 2.

Table 2. Polyatomic interferences on key analytes benefitting from the use of hydrogen as a reactive gas

Isotope	Interference
$^{24}Mg^+$	$^{12}C_2^+$
$^{40}Ca^+$	$^{40}Ar^+$
$^{56}Fe^+$	$^{40}Ar^1H^+$
$^{80}Se^+$	$^{40}Ar_2^+$

All the above listed elements can be detected with improved detection limits through the use of a more abundant isotope, as the intense polyatomic interferences are removed in a more efficient manner when using hydrogen.

The addition of oxygen in plasma, on other hand, leads to a significant increase in the conversion of analyte ions (typically $[M]^+$) into oxide ions ($[MO]^+$ in normal conditions), which leads to more interferences. The instrument conditions and parameters were optimized by tuning to reduce oxide formation below 5% by increasing the distance of the torch relative to the interface. In combination with the use of KED for interferences removal, the observation of oxide interferences has been reduced considerably, which is demonstrated through exceptionally low blank equivalent concentrations (BEC) for many elements. Table 3 gives an overview about the optimized conditions used for analysis.

Table 3. System parameters for iCAP RQ ICP-MS and iCAP TQ ICP-MS

Instrument configuration	iCAP RQ ICP-MS	iCAP TQ ICP-MS
Nebulizer	PFA Microflow nebulizer (100 µL·min⁻¹)	
Interface cones	Pt – tipped sample and skimmer	
Skimmer cone insert	High matrix	
Spraychamber	Cyclonic quartz	
Injector	Quartz, 1.0 mm i.d.	
Auxiliary flow (L·min⁻¹)	0.8	
Cool gas flow (L·min⁻¹)	14	
Nebulizer flow (L·min⁻¹)	0.62	
Oxygen additional gas	20%	
RF power (W)	1550	
Sampling depth (mm)	6	
Number of replicates	3	
Spraychamber temp. (°C)	-5	
CRC gas 1 (Flow rate in mL·min⁻¹)	Helium, 4.8	Helium, 4.8
CRC gas 2 (Flow rate in mL·min⁻¹)	Hydrogen, 9.0	Hydrogen, 9.0
CRC gas 3 (Flow rate in mL·min⁻¹)		Oxygen, 0.35
CRC gas 4 (Flow rate in mL·min⁻¹)		Ammonia, 0.40
Number of sweeps	20	
Dwell time (s)	0.05	
CETAC ASX-560 Autosampler parameters		
Uptake time (s)	60	
Wash time (s)	60	

To reduce the impact of high vapor pressure of organic solvents in plasma, and to enable the analysis of various sample types in a single sequence, the spraychamber was cooled to -5 °C using the standard Peltier cooling system.

Though the iCAP RQ ICP-MS is fulfilling current requirements in terms of sensitivity and specificity, the use of a triple quadrupole system, such as the Thermo Scientific™ iCAP™ TQ ICP-MS, can offer a significant improvement in interference removal, and subsequently detection limits for some elements while maintaining all the analytical advantages of a single quadrupole system. Therefore, an iCAP TQ ICP-MS was used to evaluate the performance using the same configuration of the sample introduction system.

Table 4. Correlation coefficients (R^2) and instrument detection limits (IDLs) determined for all target analytes

Element	m/z	CRC gas	R^2	IDL (µg·L⁻¹)	BEC (µg·L⁻¹)
Boron (B)	11	H_2	>0.999	0.189	1.637
Sodium (Na)	23	H_2	0.999	0.098	2.265
Magnesium (Mg)	24	H_2	0.999	0.314	0.971
Aluminum (Al)	27	He	>0.999	0.122	0.523
Potassium (K)	39	He	0.999	0.059	1.815
Calcium (Ca)	40	H_2	0.999	0.158	3.639
Titanium (Ti)	48	He	>0.999	0.019	0.216
Vanadium (v)	51	He	>0.999	0.006	0.191
Chromium (Cr)	52	H_2	>0.999	0.018	0.174
Manganese (Mn)	55	He	0.999	0.01	0.015
Iron (Fe)	56	H_2	>0.999	0.016	0.245
Iron (Fe)	57	He	0.999	0.029	0.083
Cobalt (Co)	59	He	>0.999	0.002	0.012
Nickel (Ni)	60	He	>0.999	0.009	0.112
Copper (Cu)	63	He	0.999	0.034	0.825
Zinc (Zn)	66	He	>0.999	0.265	3.531
Arsenic (As)	75	He	>0.999	0.005	0.012
Selenium (Se)	80	H_2	>0.999	0.021	0.059
Molybdenum (Mo)	98	He	>0.999	0.001	0.003
Silver (Ag)	107	He	0.999	0.009	0.015
Cadmium (Cd)	111	He	0.999	0.029	0.123
Tin (Sn)	118	He	0.999	0.008	0.1
Antimony (Sb)	121	He	>0.999	0.001	0.003
Barium (Ba)	137	He	>0.999	0.024	1.660
Mercury (Hg)	202	He	0.999	0.006	0.053
Lead (Pb)	208	He	0.999	0.001	0.011

Data processing and software

The data acquisition and processing has been done using Thermo Scientific™ Qtegra™ Intelligent Scientific Data Solution™ Software. Qtegra ISDS Software contains a variety of functionalities supporting the comprehensive and accurate analysis of trace elements, such as easy setup of methods using different conditions and a full feature set for implementing automated quality control.

Results and discussion

Considering the wide range of expected concentrations of various elements in different refinery products, the calibration standards were prepared to cover the concentration range between 0.1 µg·L^{-1} to 50 µg·L^{-1} using six individual standards. The expected concentration of sulfur in the fuel oil CRM is around 2%, hence, the calibration standards for sulfur were prepared in the concentration range of 5 mg·L^{-1} to 50 mg·L^{-1}. Analytical data obtained from the linearity study is presented below in Table 4. Although ICP-MS is capable of analyzing sulfur at considerably lower levels (as will be demonstrated in this note), the SRM analyzed here required the analysis of sulfur at much higher concentrations.

As can be seen from Table 4, improved limits of detection can be achieved using a combination of two different collision gases and modes of interference elimination, namely helium and KED, as well as hydrogen and reactive conditions. However, addition of a second collision gas to the method will require a fill/flush cycle in the CRC, which leads to an increase in the method runtime of about 10 s. For high-throughput methods, it is often beneficial to analyze all elements using a single set of conditions, such as KED alone. Even though hydrogen allows for an improvement mainly for such interferences based on intense argon polyatomic, these can also be achieved using KED. In some cases, e.g., selenium, the use of hydrogen can allow the use of a different and more abundant isotope, leading to a higher sensitivity. In other cases, such as arsenic, the main interferences are more effectively removed using helium and KED. Table 5 contains a direct comparison between the detection limits and BECs between KED and hydrogen for some of the elements.

Table 5. Direct comparison of instrumental detection limits and BECs achieved for various elements using He KED and hydrogen reaction mode

Analyte	He KED		Hydrogen	
	LOD	BEC	LOD	BEC
Calcium (^{40}Ca)	24.6	209.6	0.158	3.639
Magnesium (^{24}Mg)	10.8	61.1	0.314	0.17
Chromium (^{52}Cr)	0.042	0.299	0.018	0.174
Iron (^{56}Fe)	0.029	0.083	0.016	0.245
Arsenic (^{75}As)	0.005	0.012	0.031	0.112
Selenium (^{80}Se)	0.623	5.15	0.021	0.059

Accuracy and precision

To test the accuracy and precision of the proposed method for the different sample types investigated in this study, a series of experiments have been performed. The accuracy was determined using a standard reference material (SRM) NIST 1634c, certified for the concentration of trace elements in fuel oil, which has been analyzed in triplicate. In addition to this, heavy naphtha with boiling points ranging from 160 to 180 °C was spiked with all elements of interest at a concentration level of 5 µg·L^{-1} and was used to verify the method accuracy in the same sample sequence as the fuel oil.

The results obtained from these experiments are detailed in Table 6 and Table 7. All values reported are an average of individual measurements of three independent sample preparations.

Table 6. Comparison of SRM values and experimental concentrations values obtained for NIST 1634

Analyte	Certified value (mg/kg)	Obtained value (mg/kg)	% Accuracy	% RSD
Nickel (Ni)	17.54	17.95	102.3	3.2
Vanadium (V)	28.19	29.12	103.2	2.8
Cobalt (Co)	0.1510	0.148	98.6	4.6
Arsenic (As)	0.1426	0.150	105.1	3.8
Selenium (Se)	0.1020	0.107	104.9	4.2

Analyte	Certified value (%)	Obtained value (%)	% Accuracy	% RSD
Sulfur (S)	2	2.1	105	3.4

Table 7. Results of spike recovery testing – % accuracy of various elements in naphtha sample spiked at a level of 5 µg·L⁻¹

Element	m/z	CRC gas	% Recovery	% RSD
Magnesium (Mg)	24	H₂	88.0	1.0
Aluminum (Al)	27	He	94.8	1.6
Potassium (K)	39	He	90.5	5.2
Calcium (Ca)	40	H₂	96.7	4.2
Titanium (Ti)	48	He	112..8	2.6
Vanadium (V)	51	He	93.9	2.0
Manganese (Mn)	55	He	105.5	1.7
Cobalt (Co)	59	He	100.7	2.8
Nickel (Ni)	60	He	91.6	3.4
Copper (Cu)	63	He	98.0	1.0
Zinc (Zn)	66	He	93.4	0.6
Arsenic (As)	75	He	101.8	1.8
Selenium (Se)	80	H₂	93.4	2.0
Molybdenum (Mo)	98	He	111.7	3.5
Silver (Ag)	107	He	92.2	0.9
Tin (Sn)	118	He	98.4	0.3
Antimony (Sb)	121	He	94.3	0.4
Barium (Ba)	137	He	97.6	0.6
Mercury (Hg)	202	He	110.4	1.5
Lead (Pb)	208	He	96.7	1.2

Stability study

One of the main challenges in the analysis of crude, intermediates, and final products in the petrochemical industry is the variability of the physicochemical properties of these samples, which often requires dedicated instrument configurations and methods for each of the sample types. To assess the stability of the system (including sample introduction system, plasma generation, and interference removal), a large sample batch was set up. The batch contained a mixed sequence of petroleum products, including crude oil, light naphtha, and heavy naphtha samples, and was analyzed continuously over a period of 12 hours.

In accordance with the sample preparation section above, the oil samples and light naphtha were diluted using xylene to reduce the viscosity effect, whereas heavy naphtha was run undiluted. Different dilution factors ranging from 100-fold to 500-fold were used for oil sample preparation to check for potential matrix effects affecting the sample introduction system and the plasma response.

For the entire batch, the response of the internal standards was monitored and indicated that the instrument performed consistently. Figure 1 shows the signal of both internal standards used in either of the two analysis modes (KED and H₂, respectively). In all cases, the internal standards read back in the range between 90 and 110% of their initial values, indicating that the proposed method is suitable for longer batch analysis containing a variety of sample types. Please note that the samples run in the fourth hour of the batch correspond to light naphtha. Although a slight increase in the response is observed while analyzing this matrix, the internal standard does not exceed 120% as a critical limit and immediately returns to 100% when switching to the next sample type.

Additionally, QC standards were analyzed every 10 samples to assess the instrument drift and the overall accuracy of analysis over the entire analytical run. Figure 2 shows a comparison of % accuracy of the expected value for the QC checks analyzed immediately after calibration (Initial) and the last QC check (Final) at the end of the analysis after 12 hours. The relative standard deviation of all the results obtained in all QC checks in the batch did not exceed 5%.
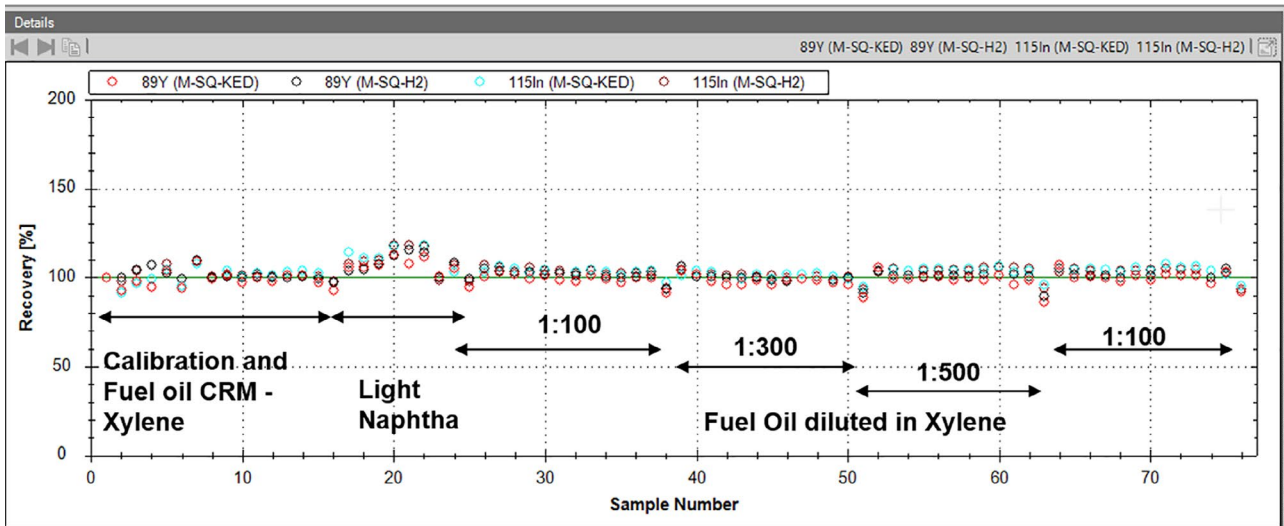
Figure 1. Response of the internal standards during a larger sample batch containing all sample types investigated in this study
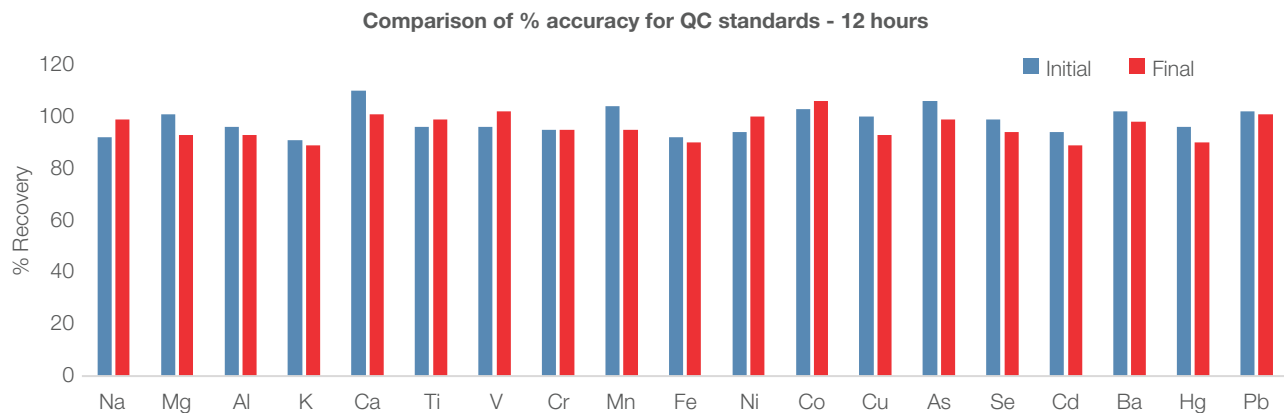

Figure 2. Comparison of % accuracy (as analyte recovery) for QC standards – Initial and final (after 12 hours)

The two QC checks returned excellent recovery values between 90 and 110% demonstrating that the data generated throughout the 12-hour continuous acquisition is accurate with the required degree of signal stability.

Some elements, such as sulfur, are biased by significant interferences neither removable through KED using helium nor hydrogen. For sulfur, and especially its main isotope ^{32}S, the use of oxygen (O_2) is a promising alternative, removing the interference caused through the formation of $^{16}O_2^+$ much more effectively and hence significantly improving detection limits. Figure 3 shows how a triple quadrupole ICP-MS can help to improve the analysis of sulfur despite the intense interference of $^{16}O_2^+$ through a mass shift reaction, leading to the formation of $^{32}S^{16}O^+$.
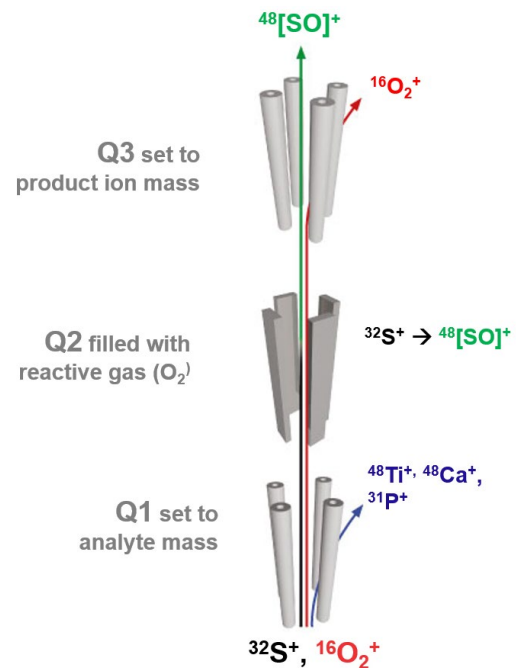

Figure 3. Interference elimination on sulfur using the iCAP TQ ICP-MS

Other interferences, affecting for example vanadium or chromium, can be removed efficiently by using ammonia (NH_3). However, the use of these gases on a single quadrupole ICP-MS instrument would generate new interferences, as all ions of all elements in the sample can enter the cell and can potentially react with such reactive gases. In addition, other elements may form ions with the same mass to charge ratio as the product ion of a mass shift reaction (e.g. $^{48}Ca^+$ or $^{48}Ti^+$ in case of $^{32}S^{16}O^+$), which may lead to an additional bias in the results. The solution is to use a triple quadrupole ICP-MS system, such as the iCAP TQ ICP-MS. Here, an additional quadrupole mass filter is situated axially in front of the CRC and allows a pre-filtering of the ion beam. In this case, only the analyte and its interference can enter the CRC, while all ions of lower and higher mass are eliminated and cannot create new interference downstream the mass spectrometer.

Table 8 compares the detection limits obtained previously using optimized settings for KED and H_2 for elements where an improvement was observed when using optimized settings on a triple quadrupole ICP-MS. Since for many elements, the same mode of analysis would be applied, an improvement is not observed in all cases. For example, when determining elements such as sodium, cobalt, nickel, copper, or zinc, KED mode still provides the best results. For other elements though, such as sulfur, arsenic and selenium, the detection limits can be significantly improved. For sulfur especially, the improved interference removal leads to a significant improvement of the achievable detection limits down to the low µg·L^{-1} range in this case. Noticeably, this IDL can be further improved below the value determined in this study (e.g., sub µg·L^{-1}). The reason is that the xylene used for preparation of the calibration standards contained a considerable amount of sulfur impurity (confirmed by the BEC of approximately 1.1 mg·L^{-1}), which increasesd the observed background signal and, as a consequence, contributed to an increased detection limit as compared to the true detection capability of the instrument. Most elements, such as vanadium, chromium, iron, or molybdenum, show comparable performance in terms of detection limits on either system, but only a triple quadrupole ICP-MS allows for full interference regardless of the interference, so that unexpected bias or failures can be effectively avoided.

Table 8. Instrument detection limits (IDL) comparison between single and triple quad ICP-MS

Element	m/z	Single quadrupole		Triple quadrupole	
		IDL (µg·L^{-1}) with helium (KED)	IDL (µg·L^{-1}) with hydrogen	IDL (µg·L^{-1})	Analysis mode (gas, Q1 analyte | Q3 analyte)
Magnesium (Mg)	24	10.8	0.314	0.085	H_2, ^{24}Mg | ^{24}Mg
Sulfur (S)	32	3315	122	13	O_2, ^{32}S | ^{32}S.^{16}O
Calcium (Ca)	44	N/A	2.729	0.176	H_2, ^{44}Ca | ^{44}Ca
Vanadium (V)	51	0.006	N/A	0.001	NH_3, ^{51}V | ^{51}V
Chromium (Cr)	52	0.042	0.018	0.002	NH_3, ^{52}Cr | ^{52}Cr
Manganese (Mn)	55	0.01	N/A	0.005	H_2, ^{55}Mn | ^{55}Mn
Iron (Fe)	56	N/A	0.016	0.01	NH_3, ^{56}Fe | ^{56}Fe
Arsenic (As)	75	0.012	0.031	0.004	O_2, ^{75}As | ^{75}As.^{16}O
Selenium (Se)	80	0.623	0.021	0.011	O_2, ^{80}Se | ^{80}Se.^{16}O
Molybdenum (Mo)	98	0.001	N/A	0.004	O_2, ^{98}Mo | ^{98}Mo.^{16}O
Silver (Ag)	107	0.009	N/A	0.002	O_2, ^{107}Ag | ^{107}Ag
Cadmium (Cd)	111	0.029	N/A	0.01	O_2, ^{111}Cd | ^{111}Cd
Barium (Ba)	137	0.024	N/A	0.012	O_2, ^{138}Ba | ^{138}Ba.^{16}O
Lead (Pb)	208	0.001	N/A	0.0005	No gas, ^{208}Pb | ^{208}Pb

N/A – Data not available

Conclusions

The results obtained from the experiments described in this application note demonstrate the following:

- The proposed method and instrument setup for elemental analysis of different petroleum products using the ICAP RQ ICP-MS and iCAP TQ ICP-MS represents a viable solution for laboratories performing oil and refinery product testing.

- The combination of sample introduction system together with a uniquely robust plasma generation system and a powerful CRC allows for sensitive analysis and comprehensive interference removal for all elements commonly analyzed in the industry. A common sample preparation method encompassing simple dilution of any products with either higher viscosity or lower boiling point in xylene offers complete flexibility for the analysis of a variety of based petroleum sample matrices.

- Whereas the use of KED and hydrogen is effective for removal of common polyatomic interferences in such challenging matrices, the use of other reactive gases such as oxygen or ammonia in combination with a triple quadrupole ICP-MS allows for an extension of the analytical capabilities. This is especially true for elements difficult to analyze previously, such as sulfur, but it can also provide improved detection limits for other elements, such as vanadium, chromium, manganese, iron, and arsenic, among others.

- A multi-mode analysis can be performed during a single aspiration of a sample, including different gases as well as combinations of single and triple quadrupole settings, allowing to cover a wide range of analytes irrespective of potential spectral interferences. The added time for switching cell gases is often negligible for the overall analysis time per sample.

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