Technical Notes
014

Exceeding Expectations

M stands for **M**etals

The concentration of metals is determined by a variety of elemental analysis techniques and is used for a wide range of petroleum products. To learn more about this line of testing, let's apply the **AmSpec** approach.

A = Application

The periodic table breaks down the elements into various categories that are determined by properties. If you look at the figure below, you will see that there are various metal groups. Any element within these metal groups can be determined by **Atomic Absorption** or **Inductively Coupled Plasma**. Anything outside of these groups (halogens, noble gases, etc.) cannot be analyzed by these methods and would need different instrumentation.

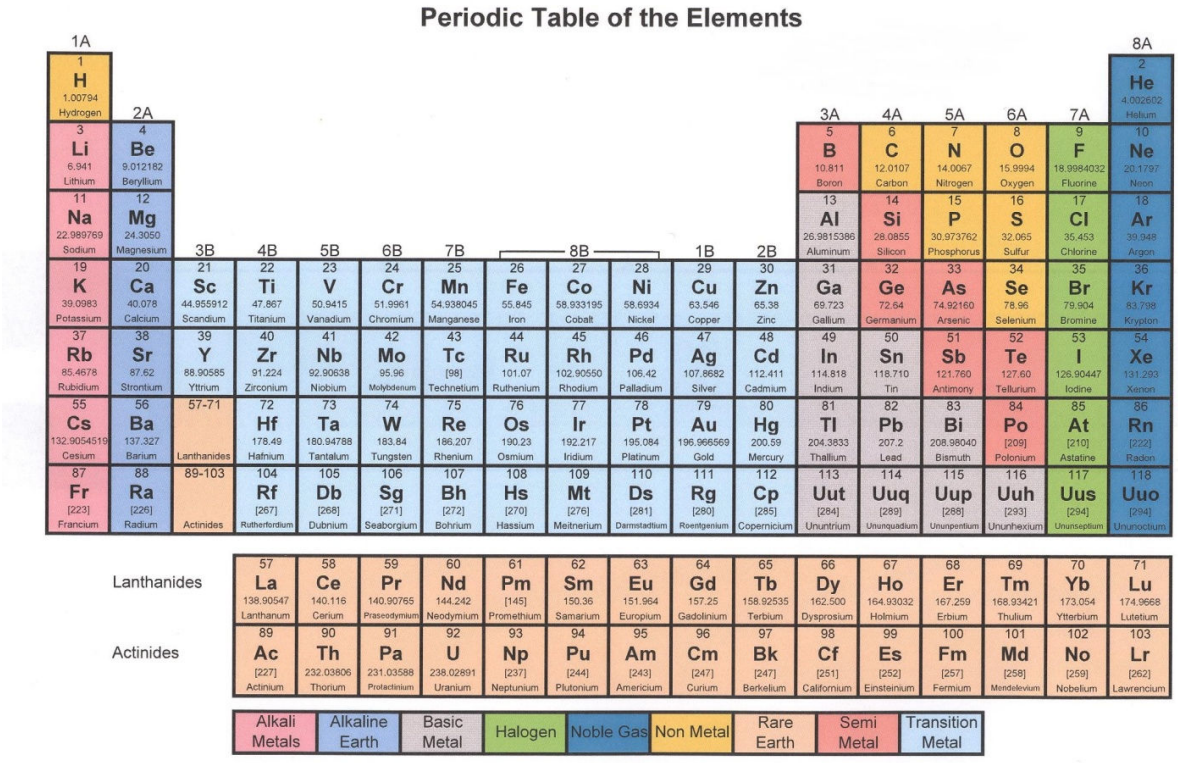

Figure 1 - This shows the classification of various elements.

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Atomic Absorption

Atomic Absorption (AA) is used for detecting metals in solution. The sample is fragmented into very small drops (atomized) and then fed into a flame. Isolated metal atoms interact with radiation that has been pre-set to certain wavelengths. This interaction is measured and interpreted. Atomic absorption exploits different radiation wavelengths absorbed by different atoms. The instrument is most reliable when a linear relationship exists between absorption-concentration. Atomizer/flame and monochromator instruments are key to making the AA device work. Relevant variables of AA include flame calibration and unique metal-based interactions. The AA can only run 1 metal at a time and the process is manual, meaning an operator must stand by during the entire process.

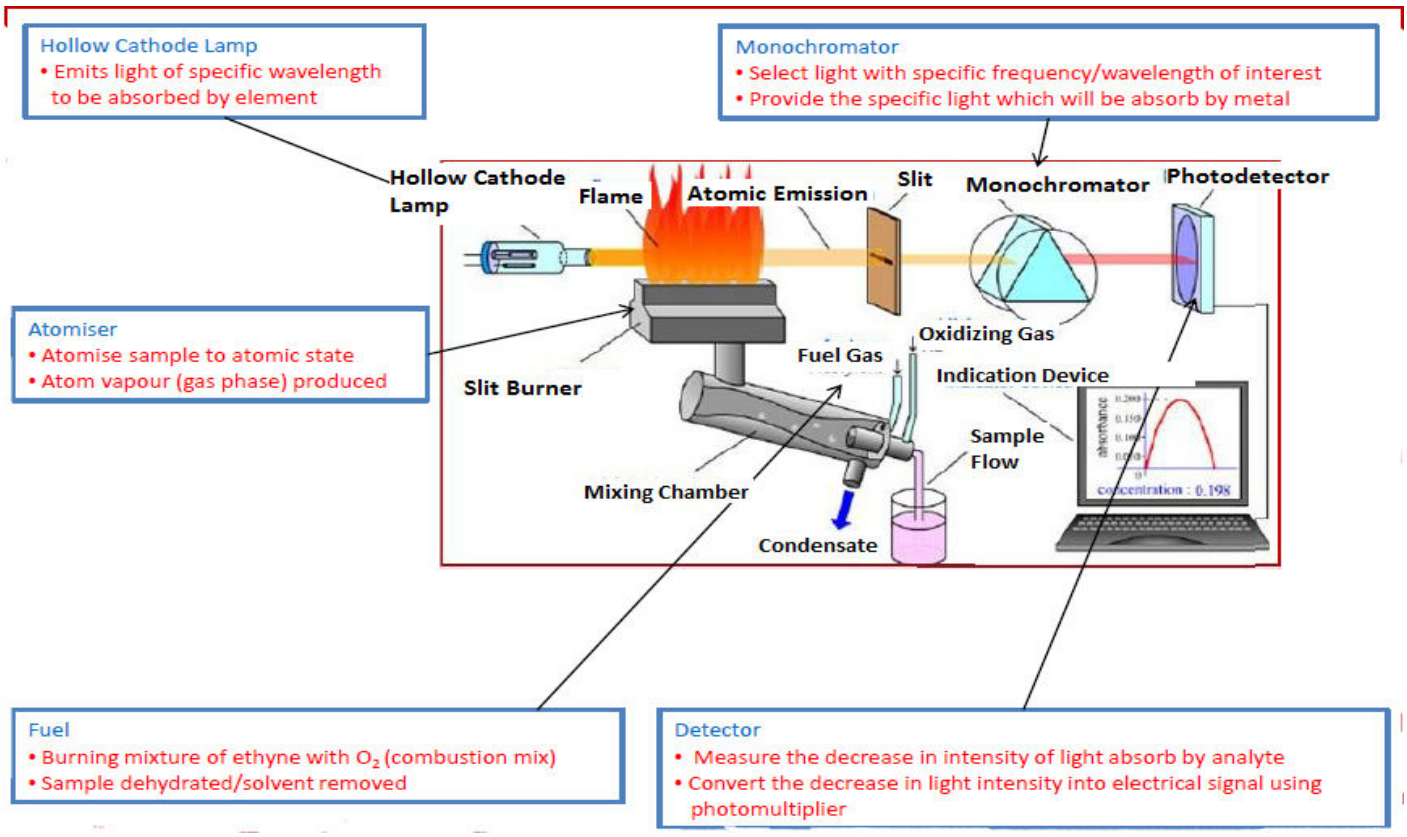

Figure 2 - Schematic of AA Instrumentation

Inductively Coupled Plasma

Inductively Coupled Plasma (ICP) Optical emission spectrometers determine analyte concentration via a quantitative measurement of the optical emission from excited atoms. When a material is heated sufficiently by the plasma, it will emit visible light in a discrete spectrum, characteristic of the elements in the material. Each element has its unique atomic emission spectrum (both visible light and x-rays). The ICP can run multiple metals at a time and is automatic, meaning the operator is free to do other testing while these metals are running.

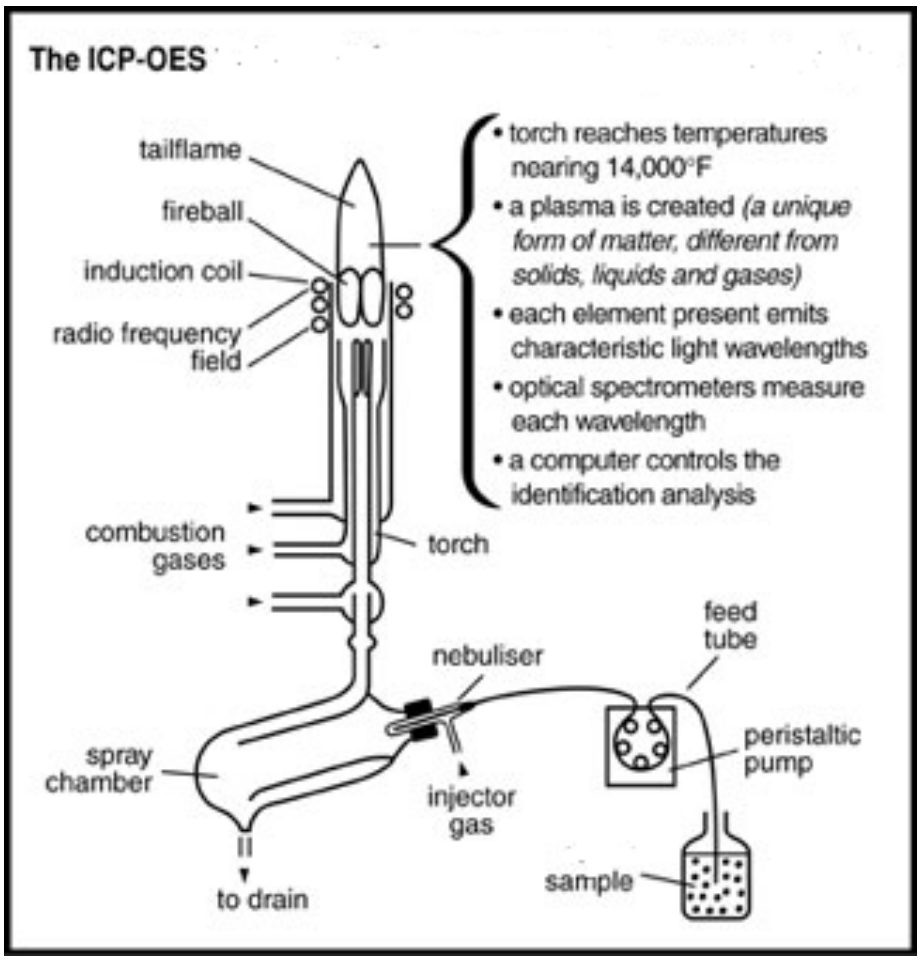

Figure 3 - Schematic Shows ICP Instrumentation

M = Methods

These are the most common methods that AmSpec uses to determine metal content:

D5078 - Determination of Nickel, Vanadium, and Iron in Crude Oils and Residual Fuels by Inductively Coupled Plasma (ICP) Atomic Emission Spectrometry

D5184 - Determination of Aluminum and Silicon in Fuel Oils by Ashing, Fusion, Inductively Coupled Plasma Atomic Emission Spectrometry, and Atomic Absorption Spectrometry

D5863 - Standard Test Methods for Determination of Nickel, Vanadium, Iron, and Sodium in Crude Oils and Residual Fuels by Flame Atomic Absorption Spectrometry

IP 470 – Determination of Aluminum, Silicon, Vanadium, Nickel, Iron, Calcium, Zinc, and Sodium in Residual Fuel Oil by Ashing, Fusion and Atomic Absorption Spectrometry

IP 501 – Determination of Aluminum, Silicon, Vanadium, Nickel, Iron, Sodium, Calcium, Zinc, and Phosphorus in Residual Fuel Oil by Ashing, Fusion and Inductively Coupled Plasma Emission Spectrometry

S = Scope

Method	Products	Scope
D5184	fuel oils	Aluminum - 5-150 mg/kg Silicon - 10-250 mg/kg
D5708	crude oil and residual fuel	None Available
D5863	crude oil and residual fuel	None Available
IP 470	residual fuels	Aluminum - 5 to 150 mg/kg Silicon – 10 to 250 mg/kg Vanadium – 1 to 400 mg/kg Nickel – 1 to 100 mg/kg Iron – 2 to 60 mg/kg Sodium – 1 to 100 mg/kg Calcium – 3 to 100 mg/kg Zinc – 1 to 70 mg/kg
IP 501	residual fuels	Aluminum – 5 to 150 mg/kg Silicon – 10 to 250 mg/kg Vanadium – 1 t0 400 mg/kg Nickel – 1 to 100 mg/kg Iron – 2 to 60 mg/kg Sodium - 1 to 100 mg/kg Calcium – 3 to 100 mg/kg Phosphorous – 1 to 60 mg/kg

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** *Please note below,* ***Turnaround Time*** *is defined as the actual length of time, on average, it takes to perform a particular method once the sample has arrived and logged in the lab, and prepared for testing. Please keep in mind that the ashing process is not part of the Turnaround Time.*

P = Procedure Notes

Method	Procedural Notes	Instrumentation	Turnaround Time
D5184 A	**Fusion Melt**	**ICP**	1 hour
D5184 B	**Fusion Melt**	**AA**	15 minutes per metal
D5708 A	**Organic Dilution**	**ICP**	1 hour
D5708 B	**Acid Digestion**	**ICP**	1 hour
D5863 A	**Acid Digestion**	**AA**	15 minutes per metal
D5863 B	**Organic Dilution**	**AA**	1 hour
IP 470	**Fusion Melt**	**AA**	15 minutes per metal
IP 501	**Fusion Melt**	**ICP**	1 hour

Acid Digestion – Sulfuric Acid is added to the sample and heated until dryness. The sample is then placed into a 525 ^{o}C oven until all the carbon has been removed. The timing of this procedure depends on the product. Lighter products such as crude oil will take a few hours, but heavy residual fuels will take over 8 hours. The sample is removed from the oven and the ash is digested with a nitric acid solution. Deionized water and diluted nitric acid are added to bring the solution to the approriate volume. The solution is now ready to be run by AA or ICP.

Fusion Melt – The sample is burned off by flame and then placed into a 525 ^{o}C oven where it remains until all the carbon has been removed. The timing of this procedure depends on the product. Lighter products such as crude oil will take a few hours, but heavy residual fuels will take over 8 hours. The sample is removed from the oven where flux (a mixture of 90% dilithium tetraborate and 10% lithium flouride) is added and placed into a 925 ^{o}C oven for 15 minutes. The fusion melt is dissolved with tartaric acid and diluted up to volume with deionized water. The solution is now ready to be run by AA or ICP.

Organic Dilution – Sample is diluted with an organic solvent such as kerosene or xylene. This solution is now ready to run by AA or ICP. This sample preparation is the quickest way to determine metals, but is not suitable for VGO and other products that have low metal concentrations.

E = Equivalents

ASTM	IP	ISO	DIN	JIS	AFNOR
D5184	377				
D5708					
D5863					
IP 470					
IP 501					

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C = Cause & Effect

Metals are a by-product of processing fuel. They are found in oils from wear, contamination, and additives. When fuels are combusted, metals present in the fuels can form low melting compounds that are corrosive to metal parts. Metals that are present at trace levels in petroleum can deactivate catalysts during processing. Thus, these test methods discussed above can be used to aid in determining the quality and value of the crude and residual oil.

For any questions about these methods, please contact Jennifer Nesci at JNesci@amspecllc.com

Also, please download the new & improved AmSpec Smart Phone app for a number of useful conversion tools and information.