A sponsored Dedicated Dialogue

Petrochemical Analysis by ICP-OES



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nductively Coupled Plasma- Optical Emission Spectroscopy (ICP-OES) is used in the petrochemical industry for the analysis of trace elements in the extraction, refining process as well as in finished products and in-service chemicals such as lubrication fluids. To learn more about how ICP-OES is used in the petrochemical industry, the challenges and how to overcome them, LCGC talked with Matthew Cassap, ICP-OES Product Manager at Thermo Fisher Scientific.

LCGC: How commonly is ICP-OES used for samples analysis in the petrochemical and industrial industries?

Matt Cassap: For petrochemicals, it's frequently employed during the extraction and refining process of crude oils. It can be used to analyze drilling muds, to monitor water as a part of the extraction process, and for the final analysis of crude oil, focusing on elements that may cause issues during the actual refining process. Post-refining, it can be used to carry out QA and QC of finished products. ICP-OES is also used in chemical productions of paints, pigments, and solvents as a QA and QC tool, and commonly used to analyze additives in hydraulic fluid in-service lubrications to determine when preventative maintenance is needed.

LCGC: What is the advantage of using ICP-OES over another technique?

Matt Cassap: The advantages of ICP-OES are speed, sensitivity, and matrix tolerance. Compared to atomic absorption (AA) or microwave induced plasma spectroscopy (MIPS), ICP-OES is much faster and more sensitive. When analyzing used lubrication oils for wear metals, a typical analysis measures up to 25 elements. Since AA is a sequential technique, it takes a long time; in excess of 10 minutes per sample compared to two minutes or less for ICP-OES. The sensitivity of AA and MIPS is also insufficient to determine some of the trace elements in used oils compared to ICP-OES. Also, the spectrum produced by MIPS is very complex since it doesn't reach the same temperature as an ICP, resulting in interferences that can cause false positives. ICP-OES has the distinct advantage over ICP-MS of being able to analyze high dissolved solids matrices which are often a key requirement in the petrochemical industry.

LCGC: How many elements can be measured by an ICP-OES? Are some elements harder to accurately measure than others?

Matt Cassap: ICP-OES can analyze most of the elements in the periodic table with the exception of some of the noble gases, with good sensitivity and linearity. Typical Thermo Scientific[™] iCAP[™] 7000 Plus Series ICP-OES detection limits are below one ppb for most elements which is ideal for the petrochemical analyses. The extended wavelength range allows sensitive analysis of arsenic, sulfur and phosphorus, important to petrochemical applications. Some

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elements do have poor performance when analyzed by ICP-OES compared to other techniques, particularly chlorine. The reproducibility of chlorine data is poor because chlorine emits light in the deep UV region around 135 nanometers, and any water molecules or other molecules in the light path from the plasma to the optical system absorb all the light emitted from

the chlorine atoms. Consequently sensitivity just isn't sufficient enough for ICP chlorine analysis.

LCGC: How does an ICP-OES handle sample matrices containing volatile organics?

Matt Cassap: ICP-OES is very robust and capable of analyzing a wide range of organic samples. Crude oils or wear metals are anaThe advantages of ICP-OES are speed, sensitivity, and matrix tolerance. Compared to atomic absorption (AA) or microwave induced plasma spectroscopy (MIPS), ICP-OES is much faster and more sensitive.

lyzed in relatively non-volatile solvents like xylene. With volatile samples like naphtha, precautions are taken to prevent instability in the plasma that can result in non-reproducible results. The iCAP 7000 Plus Series ICP-OES can cool the sample immediately prior to plasma introduction, reducing the volatility, ensuring accurate and reproducible results. Alternative approaches include diluting samples like naphtha or gasoline in a non-volatile solvent like xylene. Dilution degrades detection limits however, so cooling the sample immediately before introducing it into the plasma is still the best approach.

LCGC: What are the typical sample throughput requirements for laboratories in these industries?

Matt Cassap: Sample throughput can vary greatly depending on the application. For a typical refinery or an extraction site, only a few samples per day are analyzed. For a laboratory analyzing used lubrication oil, or a service laboratory, it's common to analyze in excess of 1,000 samples per day, so speed and instrument stability are critical. The Thermo Scientific iCAP 7600 ICP-OES has a dedicated sample introduction system called a sprint valve that eliminates any washing uptake of the solution. This technique saves a minute and a half per analysis,

> critical time in high throughput laboratories.

LCGC: Can ICP-OES analyze aqueous and organic samples?

Matt Cassap: Yes, it can. Samples like drinking water, acid digestion, soils, foods, pharmaceuticals, and metals can all be analyzed by ICP-OES. Aqueous sample analysis is very common and can be easier than ana-

lyzing organic samples due to the absence of any heavy matrices. One potentially challenging area for aqueous analysis with relevance to the petrochemical industry is high salt samples such as brine. With these samples, it's critical to ensure that you have a specialized nebulizer and spray chamber to get a stable analysis.

LCGC: What types of samples are most commonly analyzed by ICP-0ES?

Matt Cassap: ICP-OES can be used to analyze most types of samples either directly or after preparation, e.g. acid digestion. The main application areas are environmental, food, metals, and petrochemicals. New application areas are also emerging, such as hydraulic factory gas extraction. ICP-OES is an established technique and well documented methods can be found for many types of samples for many new application areas.

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