Application Note

RADIAN ASAP for Quick and Simple QC Screening for Additives in Lubricant Oils

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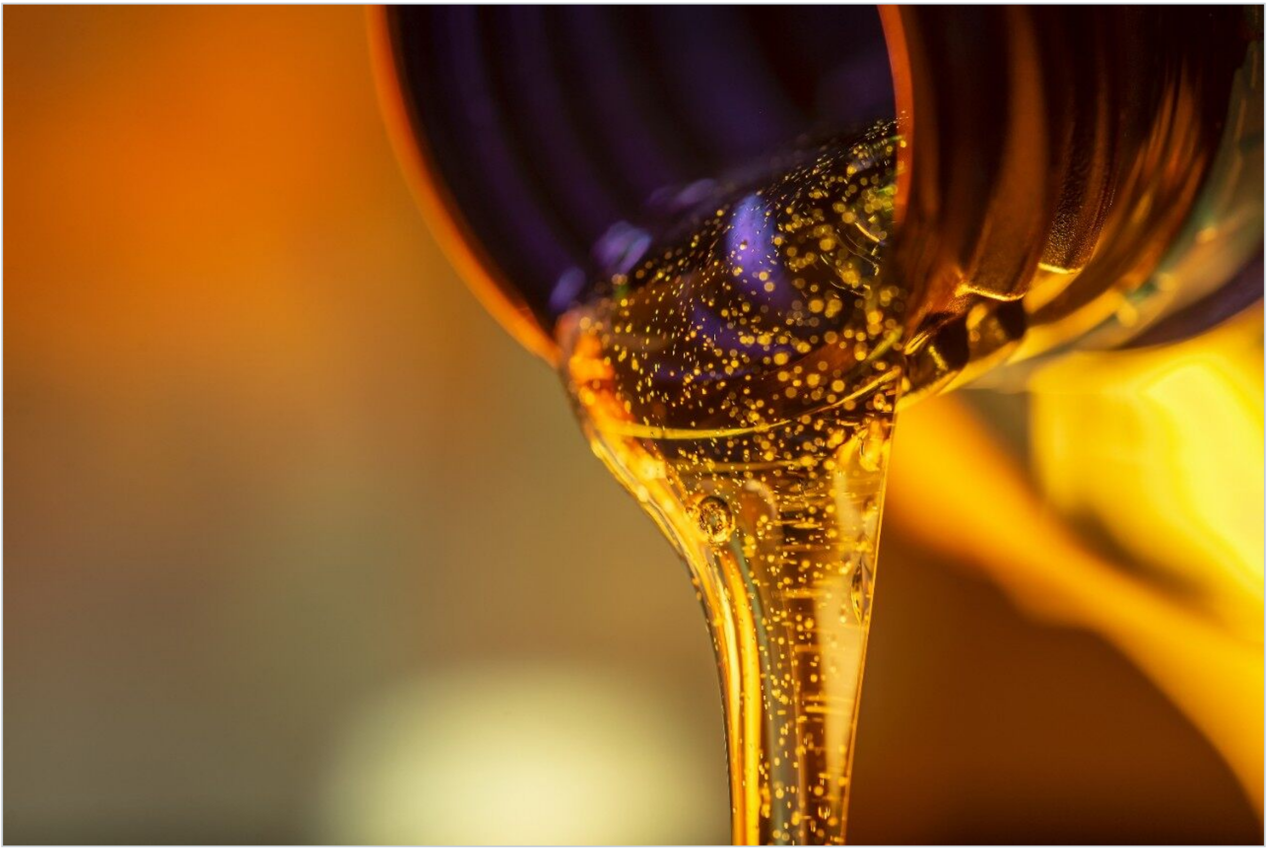

This is an Application Brief and does not contain a detailed Experimental section.

Abstract

The analysis and monitoring of lubricant oil formulations plays an important role in lubricant oil manufacturing and in understanding the performance of lubricant oils over time. The RADIAN ASAP System was used for the rapid analysis of lubricant oil, with minimal sample preparation and the deployment of a thermal gradient to help deconvolute the complex sample. Both the base oil itself and key compounds in the additive package were easily identified using this approach. The quick and simple technique could be deployed by analytical scientists or lubricant technicians to readily observe the components of their lubricant oils, facilitating fast decision making.

Benefits

- Quick and simple to use, with an easy "dip and analyze" approach
- Limited sample preparation required
- Temperature ramping functionality enables thermal deconvolution of complex samples
- Data available within minutes to enable accelerated business decisions
- Small instrument footprint, without the need for a separation system, maximizes the deployment of laboratory real estate

Introduction

Lubricant oils are widely used in many industrial applications, including process machinery and vehicle engines – in fact, any scenario where surfaces come into contact and wear could potentially pose a problem. To ensure the correct performance for a specific application, lubricant oils are formulated from a base oil and an additives package that is specifically created to confer particular performance characteristics to the lubricant oil.

Currently, numerous different tests and methodologies exist for lubricant oil additive analysis, but these can have various limitations. Some focus only on specific elements, for example zinc in zinc dialkyl dithiophosphates (ZDDP) using ICP-AES,[1,2] while others are non-specific, for example FTIR that detects the presence of specific functional groups but cannot identify the exact molecules of interest.[3] In this work, we illustrate the benefits of using fast, direct analysis with mass detection to discover and monitor lubricant oil additives.

Results and Discussion

A commercially available, domestic car engine lubricant oil was analyzed in triplicate using the RADIAN ASAP System. The oil was dissolved in 9:1 toluene:methanol with 0.1% formic acid to make a solution of 1 mg/mL. Prior to analysis, 1 mL of the solution was pipetted into an ACQUITY vial to produce a solution that occupies a known volume and depth.

A new glass sampling rod (capillary) was used for each analysis. Using the RADIAN ASAP System, each glass capillary was cleaned using a rapid, automated “bake-out” procedure that exposes the tip of the capillary to a stream of high-temperature gas to burn off any residual contamination transferred from the packaging of the glass capillary. A simple “dip and swirl” technique was used to sample the solution for 15 seconds for each analysis. Pipetting 1 mL into the ACQUITY vial, and sampling for identical amounts of time, ensured that the exposure of the glass capillary to the analyte was reproducible throughout the experiment.

The data were acquired using an automated ballistic temperature ramp from 100 °C to 600 °C over two minutes, which was pre-programmed in MassLynx instrument control software, as shown in Figure 1. The use of a temperature ramp enabled a degree of deconvolution of the complex sample, since different components appear as their different boiling points are reached, while ensuring the rapid evolution of information-rich data.

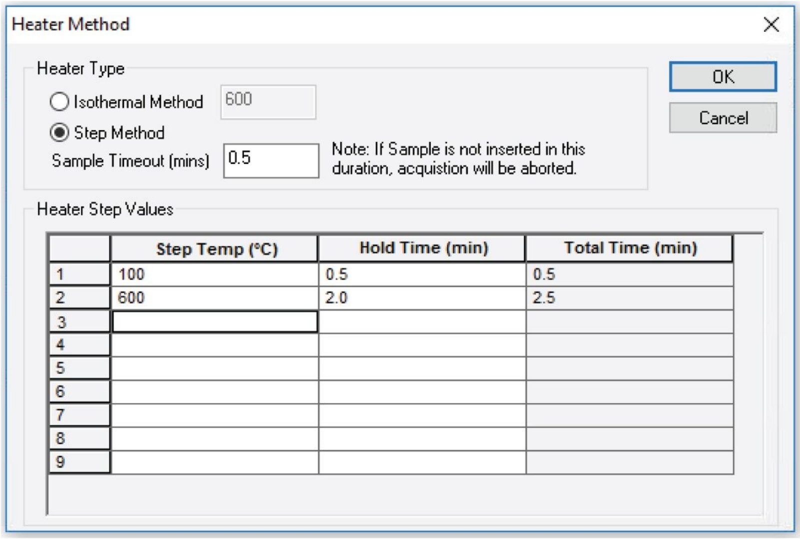

Figure 1. The pre-programmed temperature ramp used for the analysis of domestic car engine lubricant oil.

Figure 2 shows the thermal profile generated as different ions are produced with an overlay of the applied temperature ramp, and Figure 3 shows the mass spectra produced at different points in the thermal profile. Due to the ionization approach used by the RADIAN ASAP, both components of the lubricant oil additive package and the lubricant base oil itself can be detected. We can see that smaller, lower mass species, including species related to the solvents, evolve at lower temperatures immediately on initial insertion of the capillary and larger, higher mass species are observed as the temperature increases.

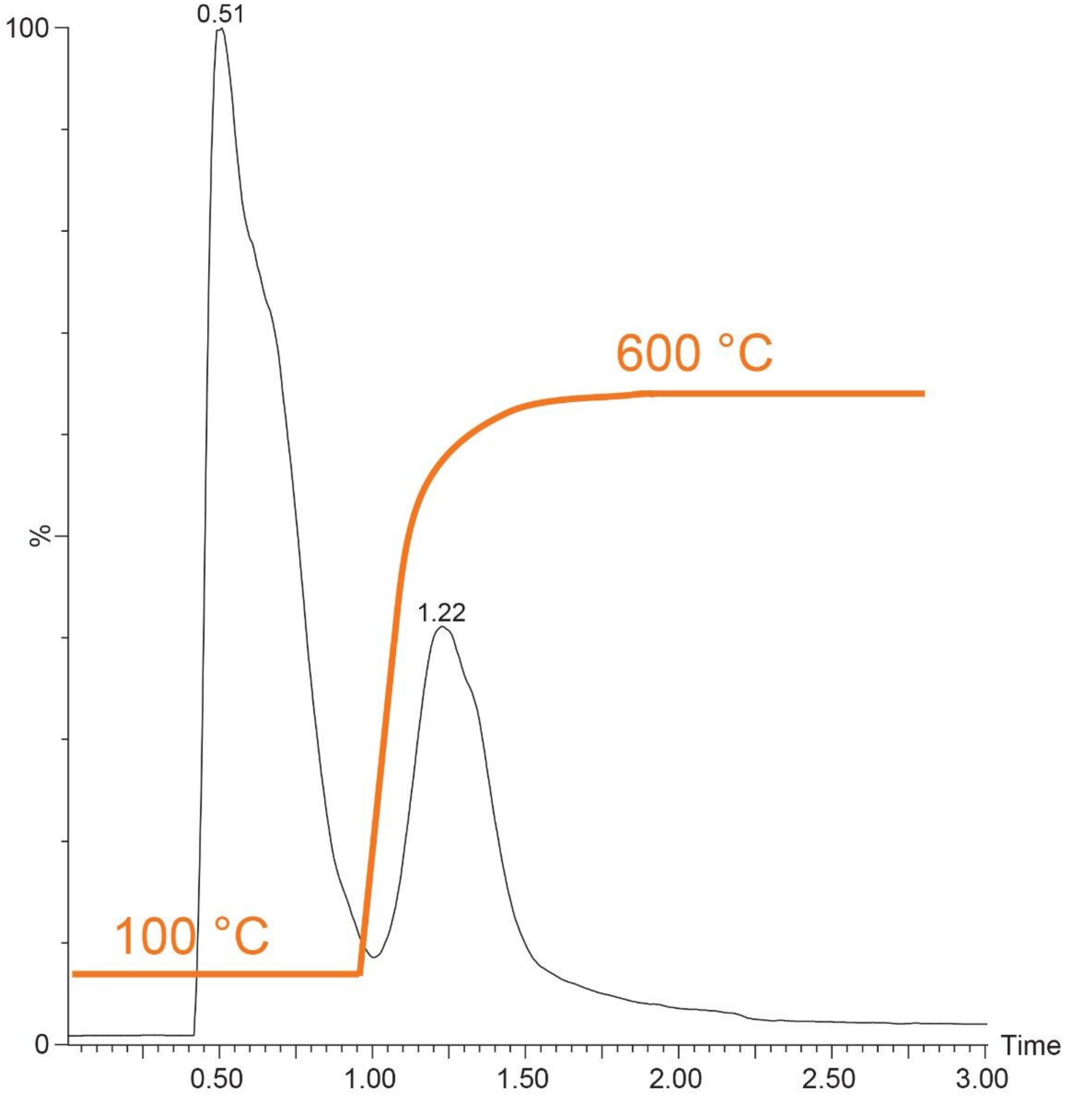

Figure 2. The thermal desorption profile (black line) resulting from the temperature ramp, with an overlay showing the temperature gradient profile (orange line).

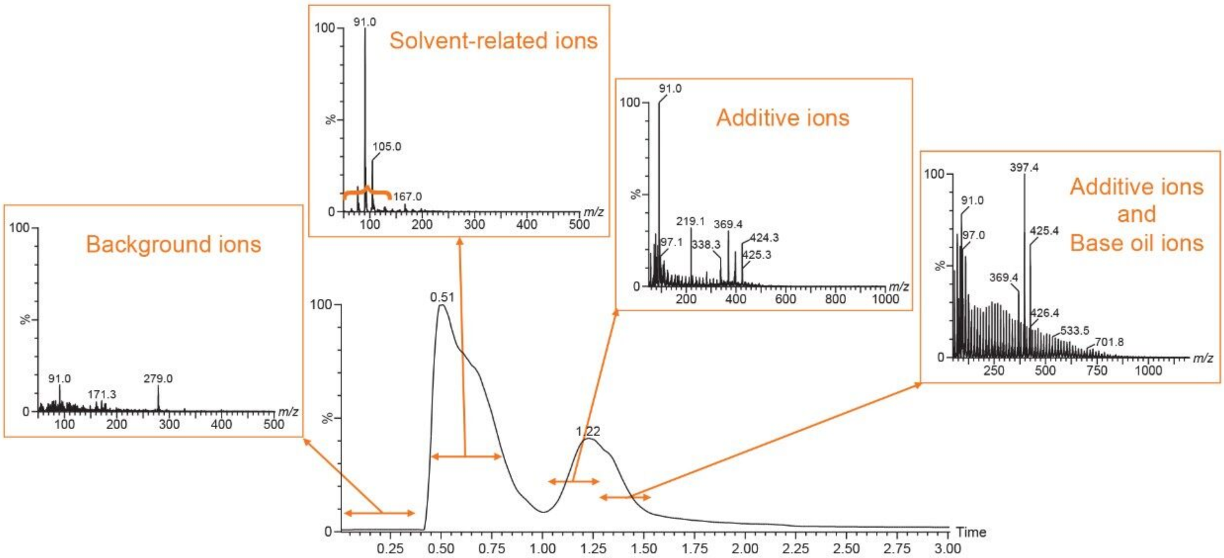

Figure 3. Spectra showing the ions evolved at different stages during the temperature ramp.

Figure 4 shows magnified regions of the mass spectrum that correspond to examples of additive species that have been detected in a sample of commercial car engine lubricant oil, with associated proposed chemical structures. These ions can subsequently be monitored to ensure the consistent presence of essential additive components.

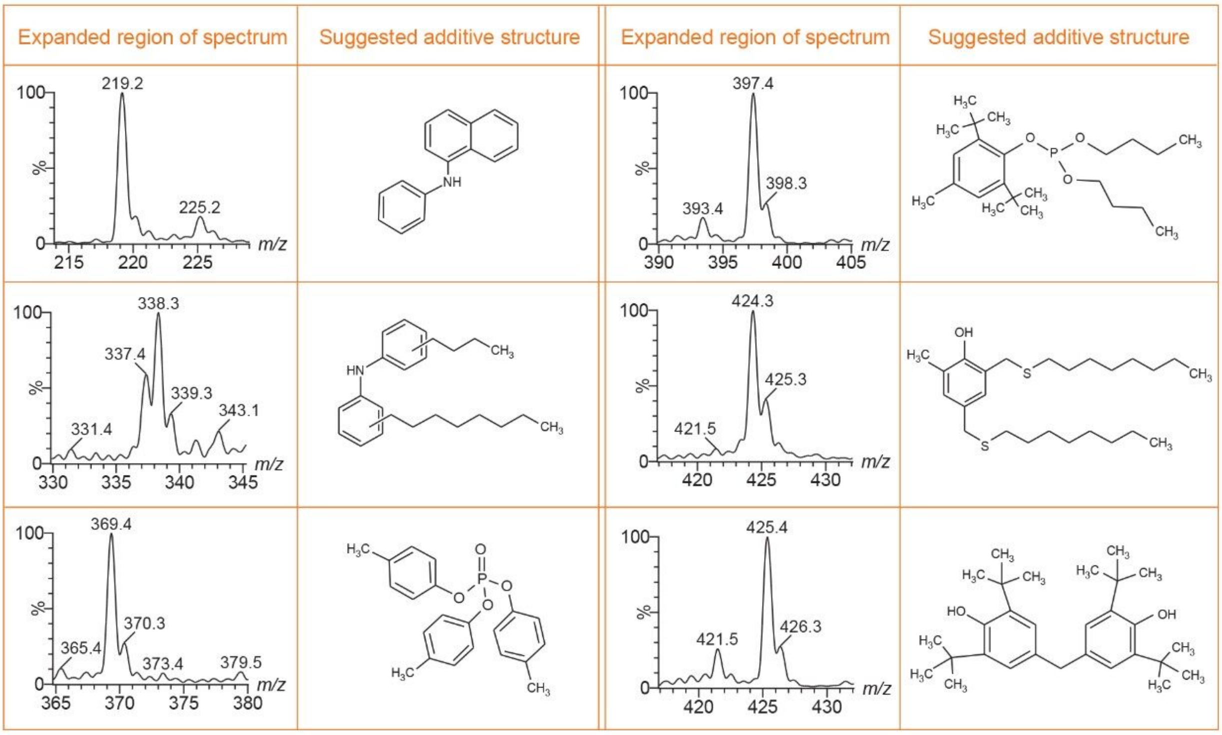

Figure 4. Selected expanded regions of the spectra shown in Figure 3 with associated suggested additive structures.

A series of ion clusters, which is typically produced by the base oil, is shown in Figure 5, illustrating that this approach is particularly useful for monitoring either the base oil or the additives package, or both. A single technique is used, rather than multiple different instruments, to build a full picture of the lubricant oil sample, which streamlines the analytical workflow.

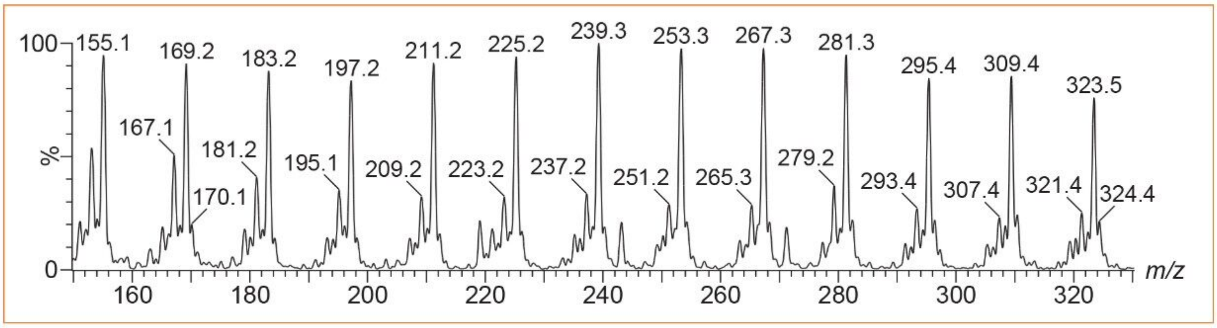

Figure 5. Expanded region of the spectrum produced at higher temperature showing a series of ion clusters related to the base oil.

Conclusion

The RADIAN ASAP System was shown to be a valuable tool for monitoring the components of lubricant oil. Both lubricant oil additives and the lubricant base oil were readily observed using a very simple and quick analysis protocol. The system incorporates useful features, such as automated cleaning of the glass sampling capillary via a bake-out button and programmable temperature ramping to aid deconvolution of complex samples. Additionally, the system offers the power of mass spectral selectivity and sensitivity in a small instrument footprint. The rapid evolution of information-rich data provides a platform on which key business decisions can be made.

References

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