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Simultaneous Sulfur and Hydrocarbon Chromatograms Using Gas Chromatography with Pulsed Flame Photometric Detection

Application Note 10780997

Keywords
Diesel Fuel
Fuel Oil
Hydrocarbons
Gasoline
Kerosene
PFPD
Sulfur

The pulsed flame photometric detector (PFPD) is excellent for the determination of sulfur compounds in various hydrocarbon matrices. It is a very sensitive and selective detector for the detection of sulfur in a complex matrix. The pulsed flame photometric detector uses a flame that is repetitively ignited and extinguished instead of a continuously burning flame. The use of a pulsed flame instead of a continuous flame adds the time dimension to flame photometric analysis as well as wavelength selectivity. Compared to a standard flame photometric detector (FPD), quenching is reduced in the PFPD. The reduced quenching allows a lower split ratio to be used during sample injection. This improves the sensitivity of the PFPD compared to a standard FPD. Figure 1 shows the emission profiles of phosphorus, sulfur, and hydrocarbon. By integrating the curve from 10 milliseconds to 24 milliseconds, it is possible to determine sulfur with no hydrocarbon contribution. Phosphorus interference is eliminated by using the proper filter.

Most detectors can only process one channel of information. The OI Analytical Model 5380 PFPD uses digital electronics including a digital signal processor to analyze the waveforms generated by the detector. As a result, the detector controller can generate two independent analog signals. The controller design allows two time slices of the waveform to be analyzed simultaneously and two corresponding analog signals to be generated for the data system.

Not only can a sulfur peak be generated that is free from hydrocarbon interference, but a hydrocarbon peak with no sulfur interferences can also

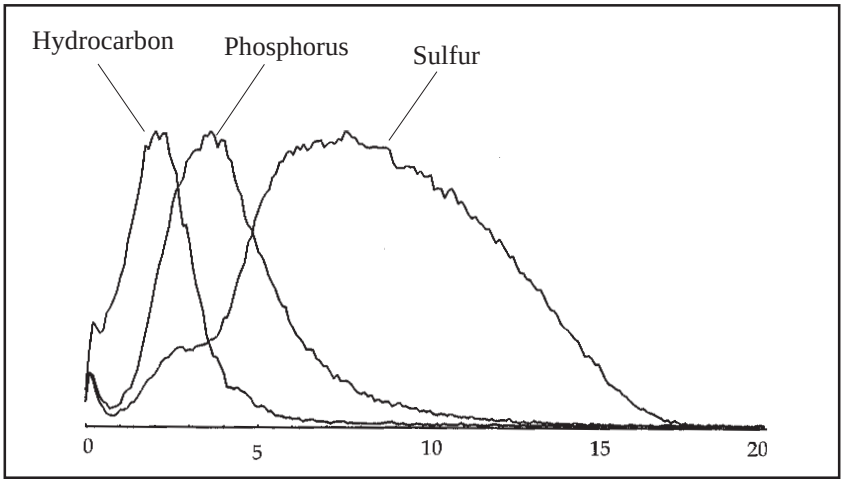

Figure 1. Emission in the PFPD

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be generated. Using the dual channel capability of the OI Analytical Model 5380 Detector Controller, an interference-free sulfur trace may be generated at the same time a sulfur-free hydrocarbon FID-like trace is generated. The detection limit for a hydrocarbon is approximately 10 ng. The hydrocarbon mode is not as sensitive as a true flame-ionization detector, but by using the dual channel capability of the controller, a sulfur chromatogram and a hydrocarbon chromatogram may be generated with one injection into one column going into one detector.

Examples of the dual channel operation of the PFPD follow. Figure 2 is the sulfur and hydrocarbon chromatograms of a sample of regular unleaded gasoline. Figure 3 is the sulfur and hydrocarbon chromatograms of a sample of diesel. Note that the distribution of the hydrocarbons and the sulfur compounds both indicate heavier compounds than the gasoline as would be expected from the volatility. Figure 4 is the chromatogram of a high sulfur kerosene, and Figure 5 is the chromatogram of a low sulfur kerosene. The hydrocarbons and sulfur compounds are approximately the same volatility range as the diesel fuel. the hydrocarbon chromatograms are similar, but the sulfur chromatograms show a dramatic difference in the kerosenes. Figure 6 is the sulfur and hydrocarbon chromatograms of a fuel oil. The sulfur and hydrocarbon chromatograms indicate the compounds are the heaviest of the samples analyzed.

These examples illustrate the potential applications of the dual channel mode of the PFPD for hydrocarbon fuel analysis.

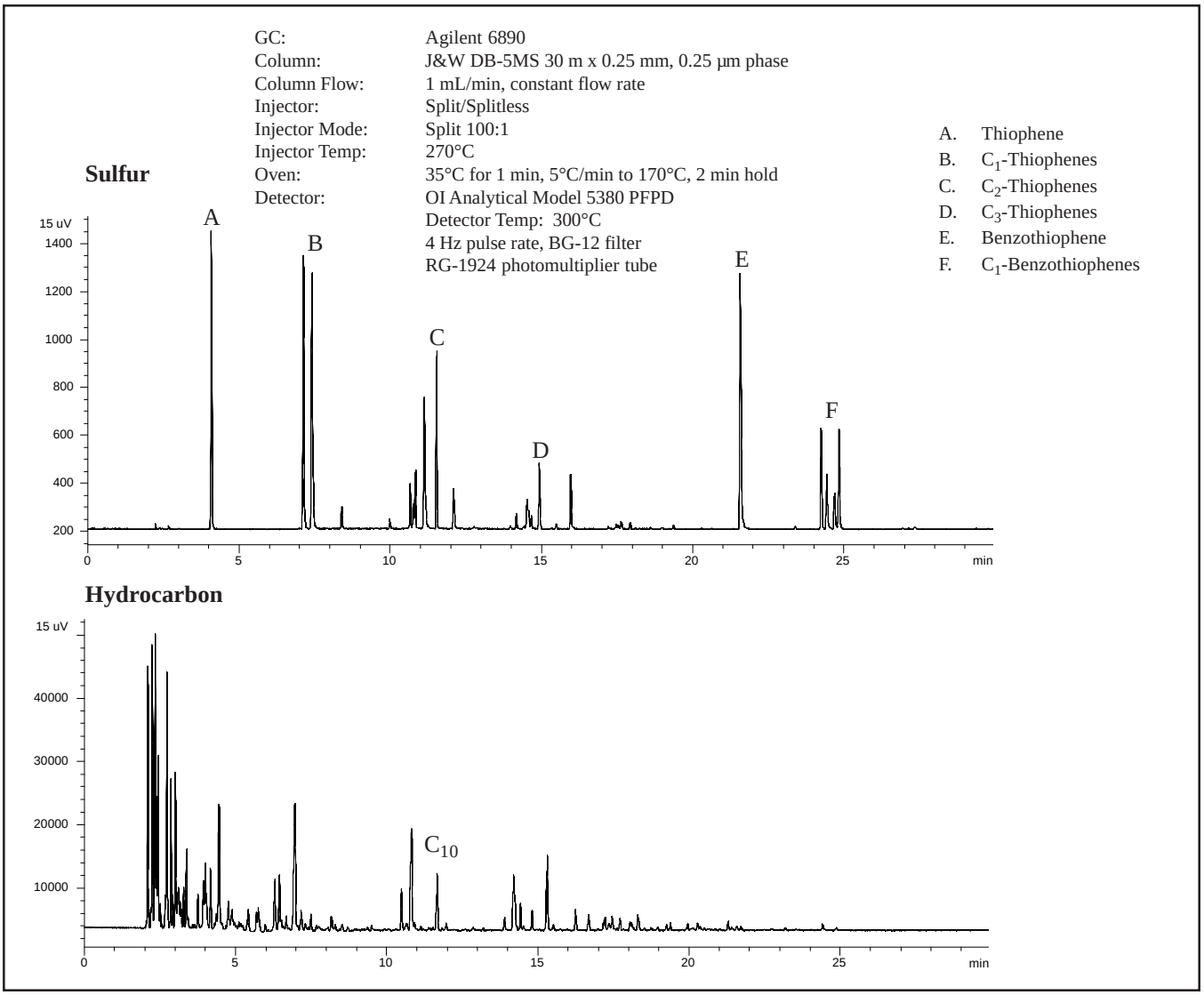


Figure 2. Regular Unleaded Gasoline, Sulfur and Hydrocarbon Chromatogram

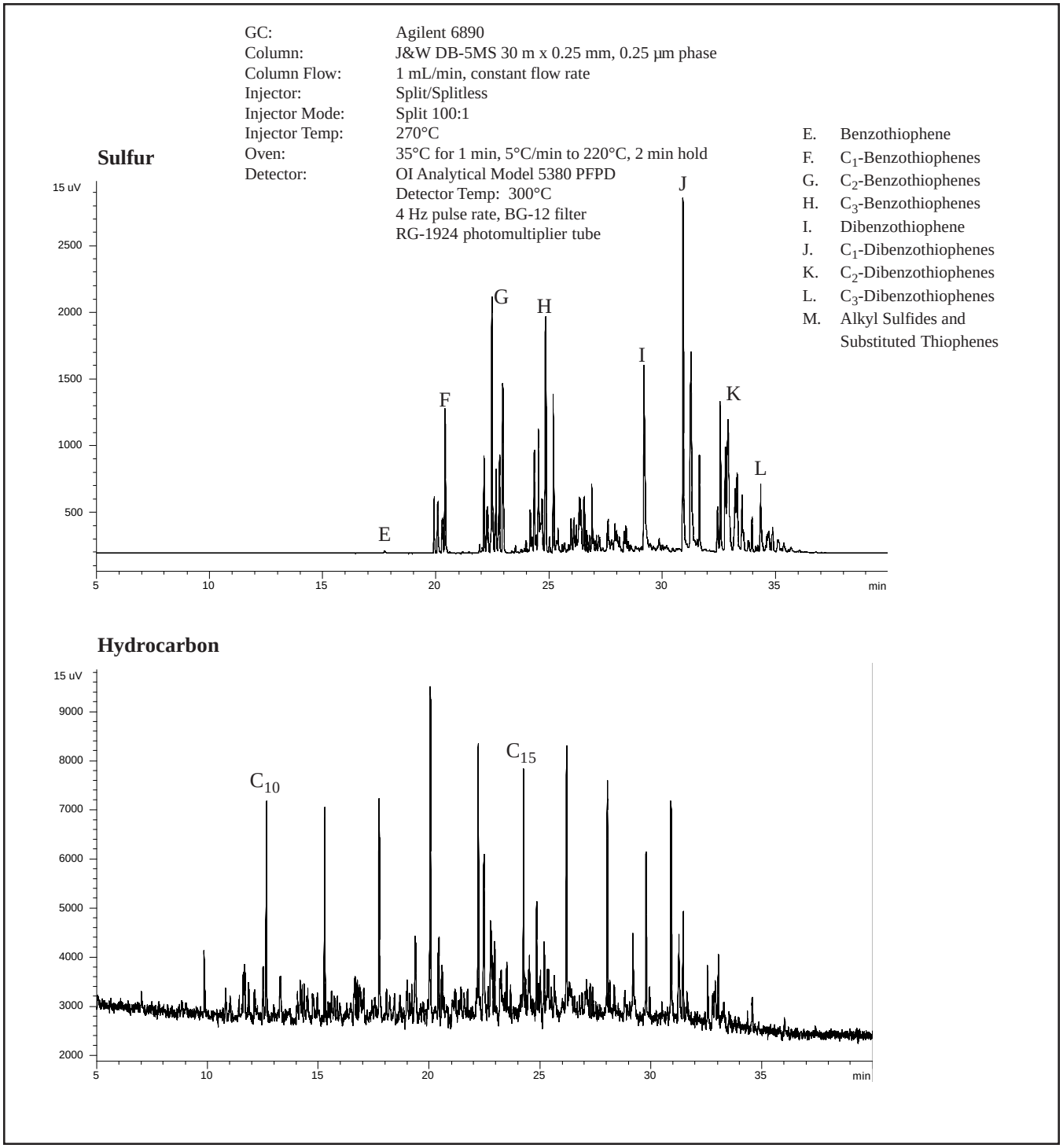


Figure 3. Diesel Fuel, Sulfur and Hydrocarbon Chromatogram

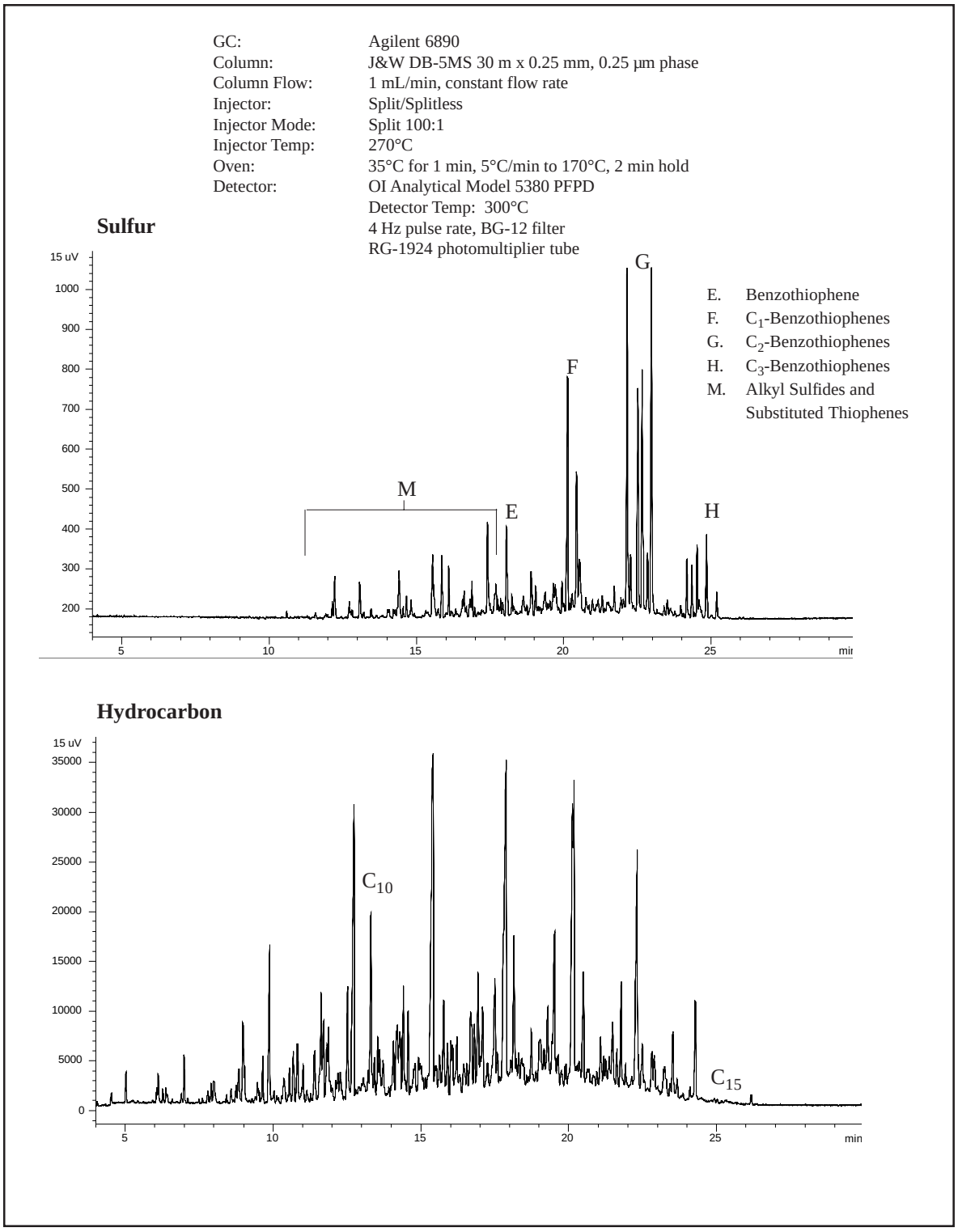


Figure 4. High Sulfur Kerosene, Sulfur and Hydrocarbon Chromatogram

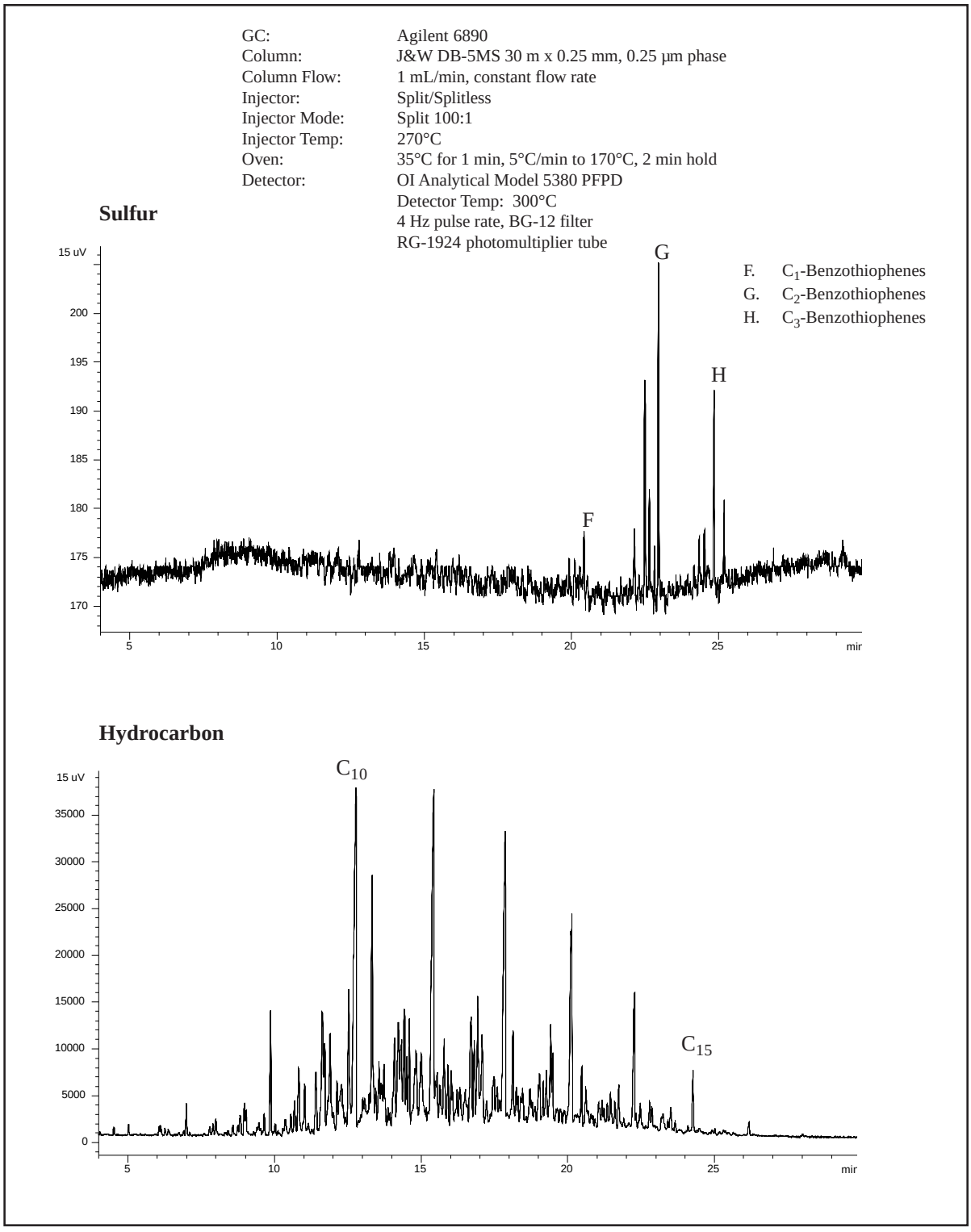


Figure 5. Low Sulfur Kerosene, Sulfur and Hydrocarbon Chromatogram

GC:	Agilent 6890
Column:	J&W DB-5MS 30 m x 0.25 mm, 0.25 µm phase
Column Flow:	1 mL/min, constant flow rate
Injector:	Split/Splitless
Injector Mode:	Split 100:1
Injector Temp:	270°C
Oven:	35°C for 1 min, 5°C/min to 260°C, 2 min hold
Detector:	OI Analytical Model 5380 PFPD
	Detector Temp: 300°C
	4 Hz pulse rate, BG-12 filter
	RG-1924 photomultiplier tube

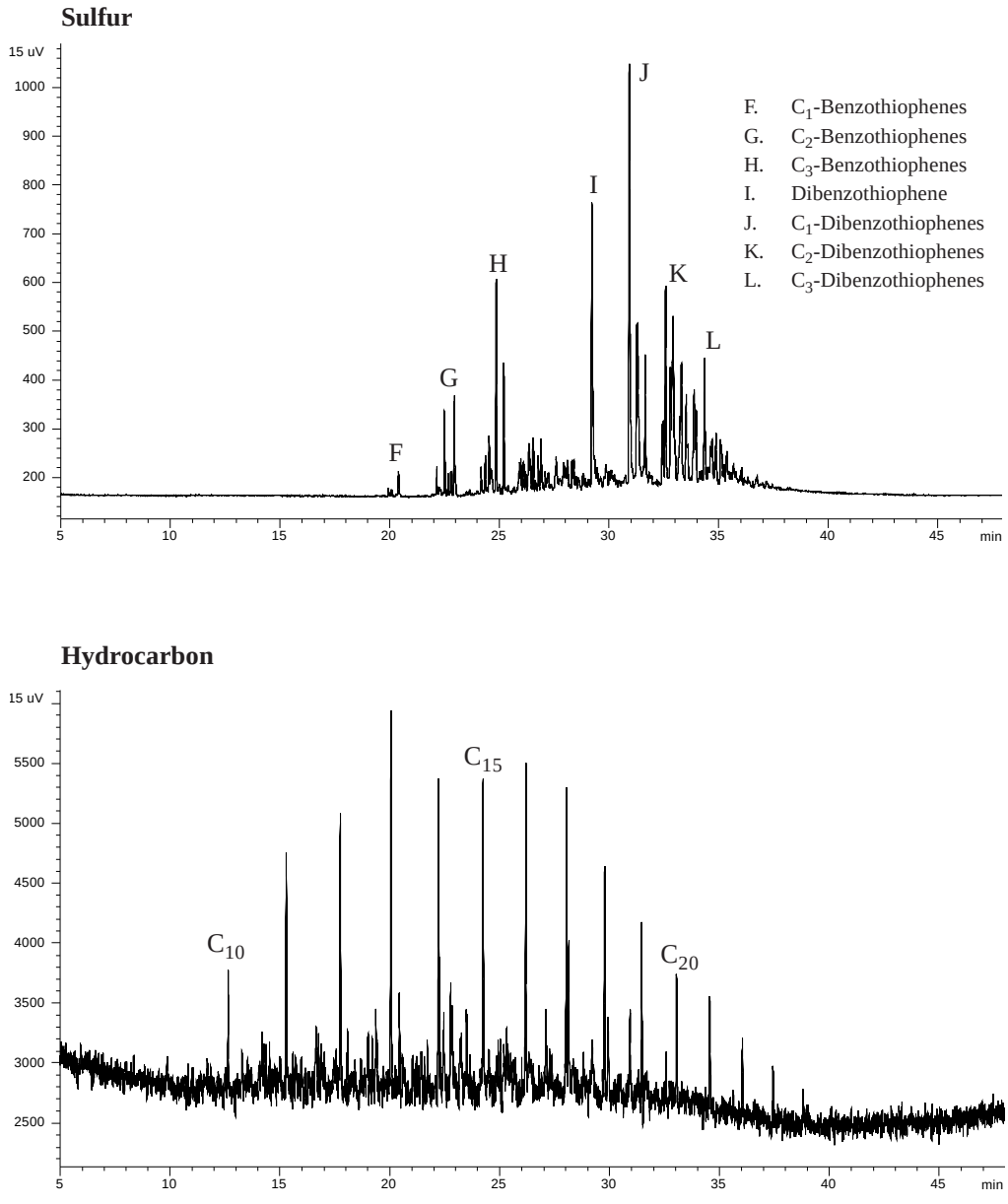
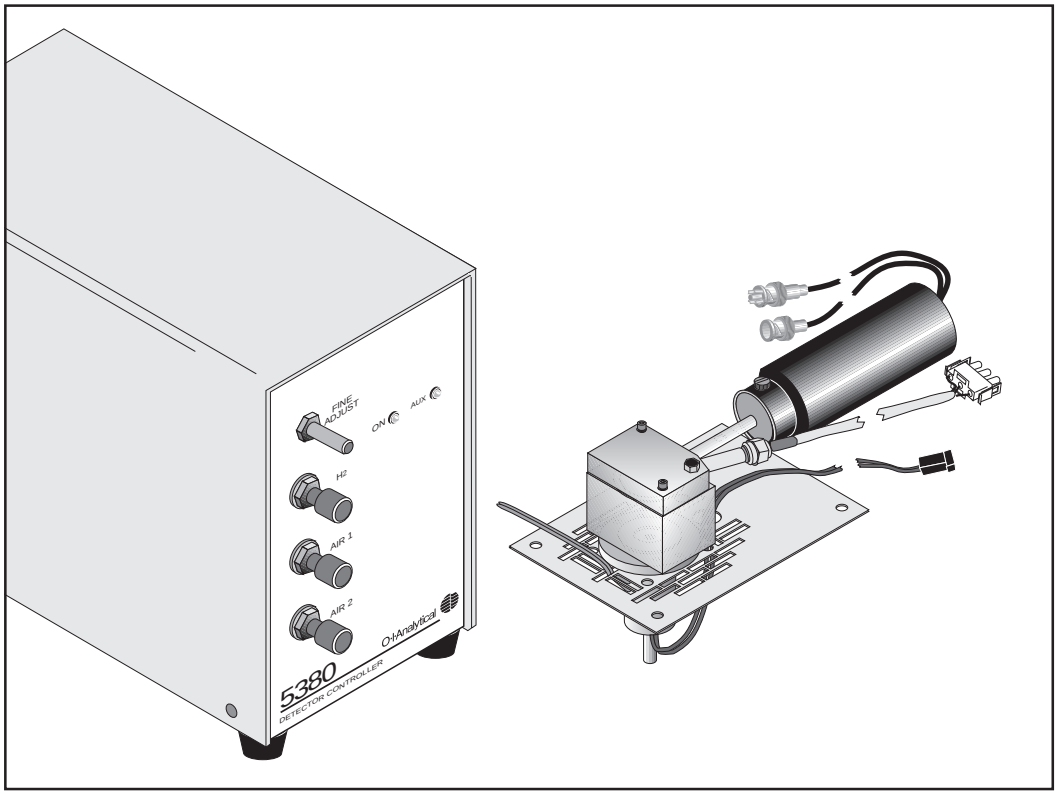


Figure 6. Fuel Oil, Sulfur and Hydrocarbon Chromatogram



OI Analytical Model 5380 Pulsed Flame Photometric Detector (PFPD)

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