Application Brief

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ASTM D7593—Analysis of Diesel for In-Service Motor Oils

Performance equivalence between Agilent Intuvo and 7890

Introduction

Diesel fuel contamination in lubricating oils is an important indicator of impending engine failure or required maintenance. Diesel engine manufacturers and service facilities optimize their maintenance schedules by routinely monitoring the diesel content of in-service motor oils. To avoid catastrophic engine failure, they need a fast and robust analytical method for this measurement. ASTM method D7593 uses capillary gas chromatography (GC) to quickly quantify diesel fuel found in these oils[1]. A recent Agilent Application Note describes operation and performance for the Agilent 7890 Series GC when measuring diesel contamination of in-service motor oil[2].

This Application Brief demonstrates equivalent analysis performance of the Intuvo 9000 GC and the 7890 GC when running this method.

Sample preparation

This method does not require any sample preparation. The calibration standards and the samples are injected neat. A backflush timing standard must be prepared containing 10 mg/mL eisosane (C_{20}) in carbon disulfide.

Results and discussion

In this method, undiluted in-service motor oil is directly injected into the GC inlet. The GC column separates any diesel fuel from the motor oil. To improve analysis speed and extend column life, the heavy motor oil hydrocarbons are backflushed from the column to the inlet split vent after the diesel fuel has eluted. Before running any samples, the backflush time is measured for each system by injecting the timing standard. Eicosane serves as a boundary marker between the diesel fuel and the motor oil. Figure 1 shows a comparison of the backflush time measurement for Intuvo and the 7890 series GC. Despite having very different flowpaths and backflush devices, both GCs were found to have nearly identical backflush times.

The Intuvo was calibrated using commercially prepared standards obtained from LGC, LGC Standards USA, 276 Abby Road, Manchester, NH 03103, USA. Three standard mixtures contained 2 % (w/w), 5 % (w/w), and 10 % (w/w) aged diesel fuel in a 75 centistokes (cSt) base oil. Additionally, a base oil sample containing no diesel was used as a 0 % standard blank. The correlation calibration coefficient (R^2) from a linear regression of calibration data was 1.000, exceeding the method requirement of 0.998. The R^2 for the 7890 GC calibration was 0.999[2].

Instrumentation

Intuvo configuration for ASTM D7593	
Syringe	Autosampler Syringe 0.5 µL (p/n G4513-80229)
Inlet	Split/splitless
Inlet liner	Ultra-Inert with glass wool (p/n 5190-2295)
Intuvo flowpath	Guard Chip (p/n G4587-60565) Inlet Flow Chip (p/n G4581-60031) D1 Post column backflush (p/n G4588-60302)
Analytical column	DB-1UI, 15 m × 0.25 mm, 0.25 µm (p/n 122-0112UI-INT)
Detector	Flame ionization (FID)

Intuvo operating conditions for ASTM D7593	
Injection size	0.1 µL
Inlet	350 °C, split flow: 100 mL/min
Guard Chip	Track oven mode
Bus	Default mode
Analytical column	Helium at 47.6 psig (3.5 mL/min) for 1.49 minutes Backflush: 2 psig at 1.49 minutes until end of run
Intuvo post column backflush	PSD 1: Helium at 27 psig for 1.49 minutes PSD 1: Backflush: at 1.49 minutes, 80 psig until end of run PSD 1: Vent flow: 5 mL/min
Column oven	225 °C
Detector	350 °C

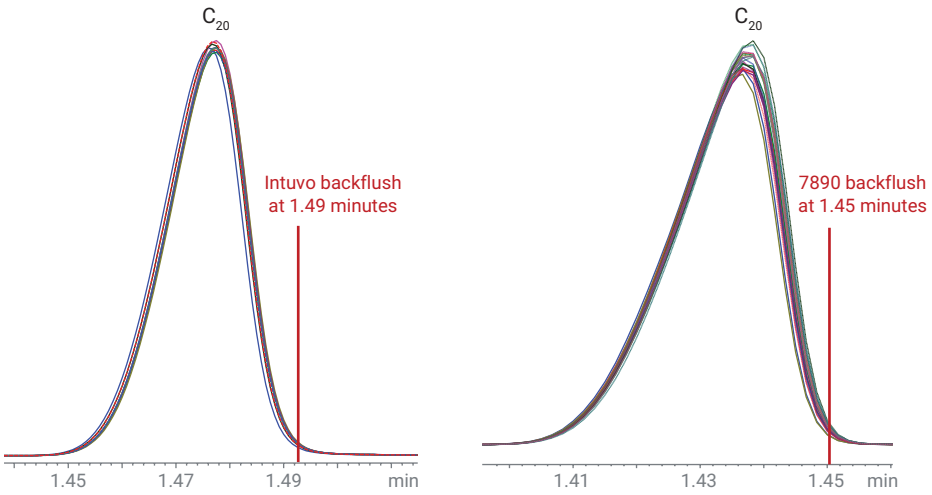

Figure 1. Backflush time setting on Intuvo (left) compared to 7890 (right).

After calibration, five in-service motor oils were each analyzed 10 times to measure the percent diesel content. Figure 2 shows chromatograms for these five samples.

The average diesel contents and measurement precisions were calculated for each sample, and the results were compared to those obtained with the 7890. Figure 3 shows this comparison.

Each bar represents a single sample's result on each GC. The average results for 10 injections and relative standard deviations (RSDs) are shown above the bars.

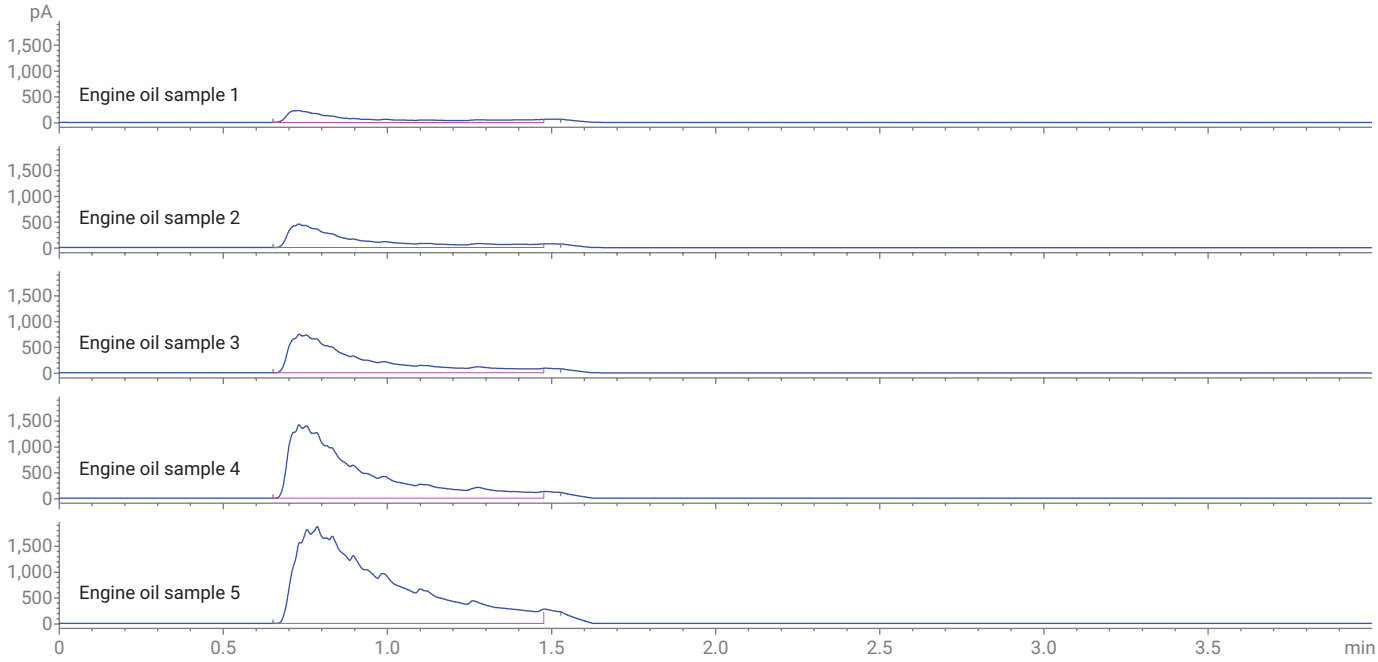

Figure 2. Diesel fuel contamination found in five in-service motor oils samples.

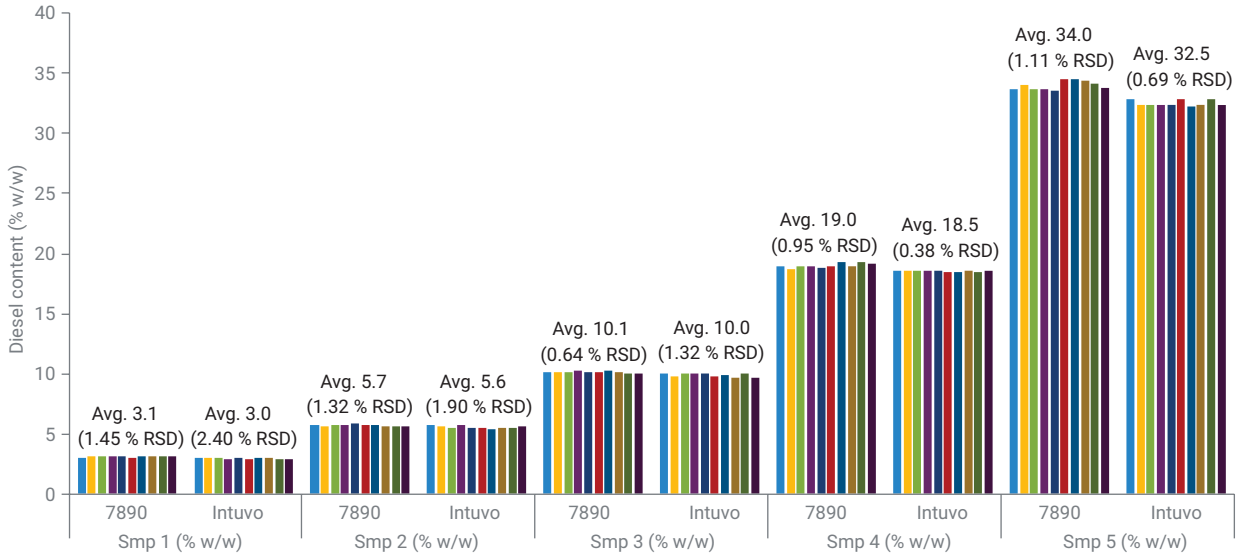

Figure 3. Comparison of 7890 and Intuvo for the analysis of diesel fuel contamination for in-service motor oil.

Conclusions

The Intuvo gas chromatograph was shown to be an excellent platform for measuring diesel fuel contamination for in-service motor oils with ASTM method D7593. A key to obtaining quality results and system robustness was the use of the blackflush technique for removing heavy sample matrix between runs. Intuvo smart flowpath components simplifies both the backflush hardware setup as well as routine operation. Intuvo was also shown to provide nearly identical system performance and analysis results when compared to the 7890 Series GC. No changes in the method or re-optimization was required for Intuvo to deliver the same outstanding performance as the 7890.

References

1. ASTM D7593-14, Standard Test Method for Determination of Fuel Dilution for In-Service Engine Oils by Gas Chromatography, ASTM International, West Conshohocken, PA, **2014**, www.astm.org.
2. Beard, K.; McCurry, J. Gas Chromatographic Analysis of Diesel Fuel Dilution for In-Service Motor Oil Using ASTM Method D7593, *Agilent Technologies Application Note*, publication number 5991-9278EN, **2018**.

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Printed in the USA, April 17, 2018
5991-9279EN

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