



## D3524 Standard test method for diesel fuel diluent in used diesel engine oils by gas chromatography

**KEY WORDS: ASTM, D3524, Diesel, Lubricating oils, engine oils.**

### INTRODUCTION

The ASTM D3524 describes the method to determine the amount of diesel in used engine lubricating oil using a gas chromatograph (GC) in combination with a flame ionization detector (FID).

While using the engine some fuel dilution of lubricating oils takes place during normal operation, this may be the result of engine wear or improper engine performance. Fuel dilution in engine oil is caused by unburned fuel components entering the engine crankcase causing the dilution. When excessive fuel dilution occurs the lubricating oil performance will be degraded. The products viscosity will decrease and this can cause performance problems in the engine. This method can analyse the diesel fuel diluent up to 12 mass%, keep in mind that this test is limited to SAE 30 oil.

This application is applicable on the SCION Instruments 4X6 and the new 8X00 GC-platform, Figure 1 shows the new SCION Instruments 8X00 GC platform.



**Figure 1. SCION Instruments 8300 and 8500 GC platform equipped with the 8400PRO autosampler.**

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## EXPERIMENTAL

This analysis can be implemented on the 8300-GC and the 8500-GC platform. The analysis was performed on the Scion 8500-GC analyser equipped with a robust FID detector using a narrow bore ceramic jet, and a 100 positions 8400PRO autosampler.

Diesel fuel contamination in lubricating oil is usually kept under 3.4 mass%. Excessive amount of diesel will dilute the oil and decreases its viscosity. The change in viscosity can cause performance problems in the engine, that is why it is important to monitor these levels and periodically change engine oil.

The ASTM D3524 is a perfect and simple method for the determination of diesel in lubricating oil. In this method a known percentage of n-Decane as an internal standard is added in order to determine the mass percentage of diesel in the oil.

## RESULTS

All the results were calculated according to the described method in ASTM D3524.

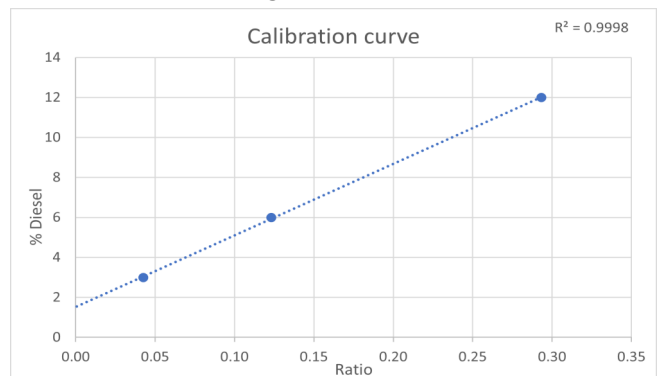
The calibration curve prepared with a concentration of 3 to 12 mass% showed a correlation ( $R^2$ ) greater than 0.9998, which is an excellent result (Figure 3).

**Table 1. Analytical conditions**

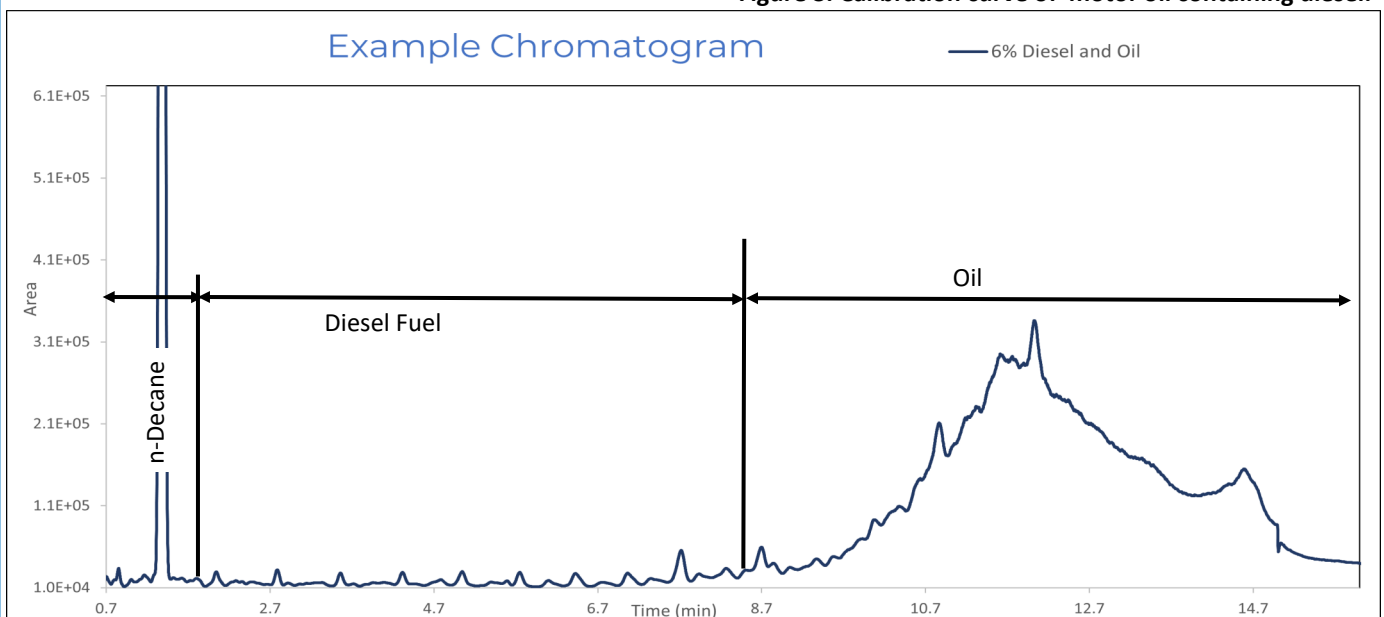
<b>Injector</b>	COC: 255 °C
<b>Column</b>	Simdist
<b>Oven Program</b>	70°C (1.0 min), 16°C/min to 325°C
<b>Carrier</b>	Helium, 30 ml/min
<b>Detector</b>	FID with ceramic jet, 350°C
<b>inj. Volume</b>	0.2 µl
<b>Autosampler</b>	8400PRO
<b>Software</b>	Compass CDS

The repeatability RSD% of the method was determined with ten consecutive injection with motor oil containing 6 mass% diesel. The analysis showed a repeatability of 0.83% which is an excellent result.

Figure 2 shows an example chromatogram of motor oil containing 6 mass% diesel.



**Figure 3. Calibration curve of motor oil containing diesel.**



**Figure 2: Example chromatogram of motor oil containing 6% diesel.**

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As a check a quality control (QC) sample of 3.5 mass% diesel in motor oil was made. After analysis 3.4 mass% was found.

Two unknown oil samples were collected from a local garage and measured. The first sample had a diesel concentration of 7.0 mass% and the second one 7.6 mass%.

### CONCLUSION

The Scion 8X00-GC analyser equipped with a split/spitless injector, Scion Instruments column and FID is capable of performing ASTM D3524 in a way that complies to the method.

The equipment of the 8X00-GC analyser is pre determined, for ordering information or customisation, please contact your local sales representative.

Although the 4X6-GC series is not shown in this application note it is possible to perform this analysis on the SCION Instruments 4X6 GC series.

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