Standard Test Method for
Diesel Fuel Diluent in Used Diesel Engine Oils by Gas Chromatography[1]

1. Scope

1.1 This test method covers the use of gas chromatography to determine the amount of diesel fuel in used engine lubricating oil. This test is limited to SAE 30 oil. The diesel fuel diluent is analyzed at concentrations up to 12 mass %.

NOTE 1—This test method may be applicable to higher viscosity grade oils. However, such oils were not included in the program used to develop the precision statement.

1.2 This test method is limited to gas chromatographs equipped with flame ionization detectors and temperature programmable ovens.

NOTE 2—The use of other detectors and instrumentation has been reported. However, the precision statement applies only when the instrumentation specified is employed.

1.3 There is some overlap of the boiling ranges of diesel fuel and SAE 30 engine oils. Moreover, the boiling range of SAE 30 oils from various sources can vary appreciably. As a result, the calibration can be altered by as much as 2 %, in terms of fuel dilution. When testing unknown or mixed brands of used engine oil, it should be realized that the precision of the method may be poorer than the precision obtained when calibrating with a new oil representative of the used oil being tested.

1.4 The values stated in SI units are to be regarded as the standard. The values stated in inch-pound units are for information only.

1.5 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory requirements prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D4626 Practice for Calculation of Gas Chromatographic Response Factors

E260 Practice for Packed Column Gas Chromatography

E355 Practice for Gas Chromatography Terms and Relationships

E594 Practice for Testing Flame Ionization Detectors Used in Gas or Supercritical Fluid Chromatography

E1510 Practice for Installing Fused Silica Open Tubular Capillary Columns in Gas Chromatographs

3. Terminology

3.1 *Definitions:*

3.1.1 For definition of gas chromatography terms, refer to Practice E355.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *fuel dilution, n*—the amount, expressed as a percentage, of diesel fuel found in engine lubricating oil.

3.2.1.1 *Discussion*—Fuel dilution may be the result of engine wear or improper performance.

3.2.2 *fuel diluent, n*—in used oil analysis, is the unburned fuel components that enter the engine crankcase causing dilution of the oil.

3.2.2.1 *Discussion*—In this method, the fuel diluent components being determined are from diesel fuel.

3.3 *Abbreviations:*

3.3.1 A common abbreviation of hydrocarbon compounds is to designate the number of carbon atoms in the compound. A prefix is used to indicate the carbon chain form, while a subscripted suffix denotes the number of carbon atoms.

For example: normal decane n–C_{10}
iso-tetradecane i–C_{14}

---

*A Summary of Changes section appears at the end of this standard

4. Summary of Test Method

4.1 Gas chromatographic techniques are used for analyzing the samples, by adding a known percentage of *n*-decane as an internal standard, in order to determine the mass percent of diesel fuel in the lubricating oil. A calibration curve is previously constructed which plots the diesel fuel to *n*-decane response ratio versus the mass percent of diesel fuel in lubricating oil mixtures containing a constant amount of internal standard. The mass percent of diesel fuel in the samples is determined by interpolation from the calibration curve.

5. Significance and Use

5.1 Some fuel dilution of the engine oil may take place during normal operation. However, excessive fuel dilution is of concern in terms of possible performance problems. This method provides a means to determine the magnitude of the fuel dilution, providing the user with the ability to predict performance and to take appropriate action.

6. Apparatus

6.1 *Gas Chromatograph*—Any gas chromatograph can be used that has the following performance characteristics:

6.1.1 *Detector*—A flame ionization detector is used. The detector must have sufficient sensitivity to detect 1.0 % decane with a peak height of at least 10 % of full scale on the data acquisition device under the conditions prescribed in this method. Follow the directions given in Practice E594. When operating at this sensitivity level, detector stability must be such that a baseline drift of not more than 1 % full scale per hour is obtained. The detector must be capable of operating continuously at a temperature equivalent to the maximum column temperature employed. Connection of the column to the detector must be such that no temperature zones exist below the column temperature (cold spots). Although thermal conductivity detectors have been used for this test, their use is not recommended.

NOTE 3—It is not desirable to operate a thermal conductivity detector at a temperature 5 to 10°C higher than the maximum column temperature employed. Operation at higher temperature generally contributes to higher noise levels and greater drift, and can also shorten the useful life of the detector.

6.1.2 *Column Temperature Programmer*—The chromatograph must be capable of linear programmed temperature operation over a range sufficient to establish a retention time of at least 1 min for the initial peak(s) and to elute the entire sample before reaching the upper end of the temperature program. The programming rate must be sufficiently reproducible to obtain retention time repeatability of 0.1 min (6 s) for each component in the calibration mixture described in 7.8.

6.1.3 *Sample Inlet System*—The sample inlet system must be capable of operating continuously at a temperature equivalent to the maximum column temperature employed, or provide an on-column inlet with some means of programming the inlet, including the point of sample introduction, up to the maximum temperature required. Connection of the column to the sample inlet system must be such that no temperature zones below the column temperature (cold spots) exist.

6.2 *Data Acquisition System:*

6.2.1 *Integrator*—Means must be provided for determining the accumulated area under the chromatogram. This can be done by means of an electronic integrator or computer based chromatography data system. The integrator/computer system must have chromatographic software for measuring the retention times and areas of eluting peaks (peak detection mode). The electronic range of the integrator/computer (for example, 1 V, 10 V) must be within the linear range of the detector/electrometer system used. It is desirable that the system be capable of subtracting each area slice of a blank run from the corresponding area slice of a sample run.

NOTE 4—Best precision and automatic operation can be achieved with an electronic integration system using a computer for data acquisition and control of the gas chromatograph.

NOTE 5—Some gas chromatographs have an algorithm built into their operating software that allows a mathematical model of the baseline profile to be stored in memory. This profile is automatically subtracted from the detector signal on subsequent sample analyses to compensate for any baseline offset. Some integration systems also store and automatically subtract a blank analysis from subsequent analytical determinations.

6.3 *Column*—Any column and conditions may be used, provided that under the conditions of the test, the separations occur in order of increasing boiling points. Moreover, the column must meet the performance requirements described in 8.2.1. The column resolution, *R*, shall be at least 3 and not more than 8. Since a stable baseline is an essential requirement of this method, compensation is required for column bleed, septum bleed, detector temperature control, constancy of carrier gas flow and instrument drift.

6.4 *Flow Controllers*—The gas chromatograph must be equipped with mass flow controllers capable of maintaining carrier gas flow constant to ± 1 % over the full operating temperature range of the column. The inlet pressure of the carrier gas supplied to the gas chromatograph must be sufficiently high to compensate for the increase in column back-pressure as the column temperature is raised. An inlet pressure of 550 kPa (80 psig) has been found to be satisfactory with the columns described in Table 1.

6.5 *Sample Introduction Devices:*

6.5.1 *Micro Syringe*—A micro syringe, usually 10 µL, is used for sample introduction.

6.5.2 Automatic sampling devices that reproducibly inject the same volume are highly recommended. The sample introduction devices should operate in a synchronous manner with the gas chromatograph.

6.6 *Vial,* 15-mL, screw cap.

7. Reagents and Materials

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on