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AMERICAN SOCIETY FOR TESTING AND MATERIALS
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Standard Test Method for Gasoline Diluent in Used Gasoline Engine Oils by Gas Chromatography¹

This standard is issued under the fixed designation D 3525; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method utilizes gas chromatography to determine the amount of dilution in used gasoline fuel engine oils.

1.2 This test method is limited to gas chromatographs equipped with flame ionization detector and programmable oven.

NOTE 1—The use of other detectors and instrumentation has been reported. However, the precision statement applies only when the instrumentation specified is employed.

1.3 The applicability of this method to gelled used engine oils has not been adequately investigated to ensure compliance with the indicated repeatability and reproducibility. Gelled oils are defined as oils that develop structure on standing, but that return to their original fluidity with light agitation.

1.4 The values stated in SI units are to be regarded as the standard. Inch-pound units are provided 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 limitations prior to use.*

2. Terminology

2.1 Definitions of Terms Specific to This Standard:

2.1.1 fuel dilution—the amount, expressed as a percentage, of engine fuel found in engine lubricating oil. This may be the result of engine wear or improper performance.

2.2 Abbreviations:

2.2.1 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.

Example:

normal decane	$n\text{-C}_{10}$
iso-tetradecane	$i\text{-C}_{14}$

¹ This test method is under the jurisdiction of ASTM Committee D-2 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.04.04 on Chromatographic Methods.

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3. Summary of Test Method

3.1 Gas chromatographic techniques are used with the sample, containing a known percentage of n -tetradecane as an internal standard, to determine the weight percent of the sample boiling below the boiling point of the internal standard.

4. Significance and Use

4.1 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.

5. Apparatus

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

5.1.1 *Detector*—Only a flame detector can be used in this method. The detector must have sufficient sensitivity to detect 1.0 % n -tetradecane with a peak height of at least 40 % of full scale on the recorder under the conditions prescribed in this method, and without loss of resolution. The detector must be capable of operating continuously at a temperature equivalent to the maximum column temperature employed, and it must be connected to the column so as to avoid any cold spots. When operating at this sensitivity level, detector stability must be such that the baseline drift of not more than 1 % per hour is obtained.

5.1.2 *Column Temperature Programmer*—The chromatograph must be capable of temperature program operation over a range sufficient to establish a retention time of 0.25 min (15 s) for the initial peak and to elute the internal standard totally. For determination of fuel dilution the reproducibility of the programming rate is not significant, although a retention time repeatability of 0.3 min (18 s) should be available.

5.1.3 *Sample Inlet System*—The sample inlet system must be capable of operating continuously at a temperature equivalent to the maximum column temperature employed. The sample inlet system must be connected to the chromatograph column so as to avoid any cold spot.

5.2 *Recorder*—A recording potentiometer with a full-scale response time of 1 s or less can be used.

5.3 *Column*—Any column and conditions may be used, provided, under the conditions of the test, separations are in