Designation: D5442 – 93 (Reapproved 2013)

Standard Test Method for Analysis of Petroleum Waxes by Gas Chromatography[1]

This standard is issued under the fixed designation D5442; 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 covers the quantitative determination of the carbon number distribution of petroleum waxes in the range from n-C_{17} through n-C_{44} by gas chromatography using internal standardization. In addition, the content of normal and non-normal hydrocarbons for each carbon number is also determined. Material with a carbon number above n -C_{44} is determined by its difference from 100 mass % and reported as C_{45+}.

1.2 This test method is applicable to petroleum derived waxes, including blends of waxes. This test method is not applicable to oxygenated waxes, such as synthetic polyethylene glycols (for example, Carbowax[2]), or natural products such as beeswax or carnauba.

1.3 This test method is not directly applicable to waxes with oil content greater than 10 % as determined by Test Method D721.

1.4 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

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 consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[3]

D721 Test Method for Oil Content of Petroleum Waxes

D4307 Practice for Preparation of Liquid Blends for Use as Analytical Standards

D4626 Practice for Calculation of Gas Chromatographic Response Factors

E260 Practice for Packed Column Gas Chromatography

E355 Practice for Gas Chromatography Terms and Relationships

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *carbon number*—a number corresponding to the number of carbon atoms in a hydrocarbon.

3.1.2 *cool on-column injection*—a sample introduction technique in gas chromatography where the sample is injected inside the front portion of a partition column at a temperature at or below the boiling point of the most volatile component in the sample.

3.1.3 *low volume connector*—a metal or glass union designed to connect two lengths of capillary tubing. Usually designed so that the tubing ends are joined with a minimum of either dead volume or overlap between them.

3.1.4 *non(normal paraffin)hydrocarbon (NON)*— all other hydrocarbon types excluding those hydrocarbons with carbon atoms in a single length. Includes aromatics, naphthenes, and branched hydrocarbon types.

3.1.5 *normal paraffin*—a saturated hydrocarbon which has all carbon atoms bonded in a single length, without branching or hydrocarbon rings.

3.1.6 *wall coated open tube (WCOT)*—a term used to specify capillary columns in which the stationary phase is coated on the interior surface of the glass or fused silica tube. Stationary phase may be cross-linked or bonded after coating.

4. Summary of Test Method

4.1 Weighed quantities of the petroleum wax and an internal standard are completely dissolved in an appropriate solvent and introduced into a gas chromatographic column that separates the hydrocarbon components by increasing carbon number. The column temperature is linearly increased at a reproducible rate until the sample is completely eluted from the column.

4.2 The eluted components are detected by a flame ionization detector and recorded on a strip chart or computer system. The individual carbon numbers are identified by comparing the retention times obtained from a qualitative standard with the

[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.04.0H on Chromatographic Distribution Methods.

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[2] Carbowax is a registered trademark of Union Carbide Corp.

[3] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

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