Designation: ~~D4419 – 90 (Reapproved 2010)~~ D4419 – 90 (Reapproved 2015)

Standard Test Method for Measurement of Transition Temperatures of Petroleum Waxes by Differential Scanning Calorimetry (DSC)[1]

This standard is issued under the fixed designation D4419; 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 transition temperatures of petroleum waxes, including microcrystalline waxes, by differential scanning calorimetry (DSC). These transitions may occur as a solid-solid transition or as a solid-liquid transition.

1.2 The normal operating temperature range extends from ~~15°C~~15 °C to ~~150°C~~150 °C (Note 1).

1.3 The values stated in SI units are to be regarded as the standard.

1.4 *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. Referenced Documents

2.1 *ASTM Standards:*[2]

D87 Test Method for Melting Point of Petroleum Wax (Cooling Curve)
D1160 Test Method for Distillation of Petroleum Products at Reduced Pressure
D3418 Test Method for Transition Temperatures and Enthalpies of Fusion and Crystallization of Polymers by Differential Scanning Calorimetry
E472 Practice for Reporting Thermoanalytical Data (Withdrawn 1995)[3]
E473 Terminology Relating to Thermal Analysis and Rheology
E474 Method for Evaluation of Temperature Scale for Differential Thermal Analysis (Withdrawn 1986)[3]

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *Differential Scanning Calorimetry (DSC)*—A technique in which the difference in energy inputs into a substance and a reference material is measured as a function of temperature, while the substance and a reference material are subjected to a controlled temperature program. The record is the DSC curve. Two modes, power-compensation DSC and heat-flux DSC, can be distinguished depending on the method of measurement used. For additional background information refer to Practice E472, Terminology E473, and Test Method E474.

4. Summary of Test Method

4.1 Separate samples of petroleum wax and a reference material or blank (empty sample container) are heated at a controlled rate in an inert atmosphere. A sensor continuously monitors the difference in heat flow to the two samples. The DSC curve is a record of this difference versus temperature. A transition in the wax involves the absorption of energy relative to the reference, resulting in an endothermic peak in the DSC curve. While the transition occurs over the temperature range spanned by the base of the peak, the temperature associated with the peak apex is designated the nominal transition temperature (Note 1).

NOTE 1—Test Method D87 also monitors energy transfer between wax and a standard environment. The highest temperature DSC transition may differ from the melting point because the two methods approach the solid/liquid phase transition from different directions.

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[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum ~~Products~~ Products, Liquid Fuels, and Lubricantsand is the direct responsibility of Subcommittee D02.10.0A on Physical/Chemical Properties.

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[2] 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.

[3] The last approved version of this historical standard is referenced on www.astm.org.

5. Significance and Use

5.1 DSC in a convenient and rapid method for determining the temperature limits within which a wax undergoes during transitions. The highest temperature transition is a solid-liquid transition associated with complete melting; it can guide the choice of wax storage and application temperatures. The solid-solid temperature transition is related to the properties of the solid, that is, hardness and blocking temperature.

NOTE 2—For a relatively narrow cut petroleum wax, the lowest transition will be a solid-solid transition. A narrow cut wax is one obtained by deoiling a single petroleum distillate with a maximum range of ~~120°F~~120 °F between its 5 % and 95 % vol in accordance with Test Method D1160 boiling points (converted to ~~760 torr).~~ 760 torr). The DSC method cannot differentiate between solid-liquid and solid-solid transitions. Such information must be predetermined by other techniques. In the case of blends, the lower temperature transition may be envelopes of both solid-liquid and solid-solid transitions.

5.2 Since petroleum wax is a mixture of hydrocarbons with different molecular weights, its transitions occur over a temperature range. This range is one factor that influences the width, expressed in °C, of the DSC peaks. The highest temperature transition is a first-order transition. If, for a series of waxes, there is supporting evidence that the highest temperature transition of each wax is the major first-order transition, its relative width should correlate with the relative width of the wax's molecular weight distribution.

6. Interferences

6.1 The test specimen must be homogeneous and representative. The small sample size ~~(10 mg)~~ (10 mg) makes these requirements particularly important.

6.2 Intimate thermal contact, sample-to-pan and pan-to-sensor, is essential to obtain accurate and reproducible results.

6.3 The heating rate must be the specified ~~10~~10 °C/min ± ~~1°C/min.~~1 °C/min. Faster or slower rates will produce a different transition temperature and transition peak width.

7. Apparatus

7.1 *Differential Scanning Calorimeter,* operating in either power compensation or heat flux mode, capable of heating at ~~10~~10 °C/min ± ~~1°C/min~~1 °C/min from ~~15°C~~15 °C to ~~150°C.~~150 °C. Controlled cooling capability is preferred but not essential. The calorimeter must be able to record automatically the differential signal (WE or WT) versus temperature with a temperature repeatability of ~~±0.5°C.~~±0.5 °C. If the differential record is versus time, the calorimeter must have the capability to make a simultaneous record of temperature versus time.

7.2 *Sample Pans,* of aluminum or other metal of high thermal conductivity, excluding copper and its alloys.

7.3 *Reference Material*—Glass beads, alumina powder, silicon carbide, or any material known to be unaffected by repeated heating and cooling and free from interfering transitions. The specific heat capacity of the reference should be as close as possible to that of the test material.

7.4 *Recorder,* capable of recording heat flow versus temperature.

8. Reagent

8.1 *Nitrogen,* or other dry inert gas supply for flushing the sample compartment.

9. Calibration

9.1 Using the instrument manufacturer's recommended procedure, calibrate the instrument's temperature scale over the temperature range of interest with appropriate standards. These include, but are not limited to:

	Melting Point	
Standard 99 % Purity Min.	°C	K
Phenoxybenzene **(1)**[4]	26.9	300.0
p-Nitrotoluene **(2)**	51.5	324.8
Naphthalene **(3)**	80.3	353.6
Benzoic Acid[A]	122.4	395.7
Adipic Acid **(4)**	153.0	426.3
Indium Metal **(1)**	156.6	429.9

[A] See Test Method D3418. 99.98 % purity available from U.S. Bureau of Standards as SRM 350.

9.2 The specimen weight and test procedure should be those specified in Section 10~~10.~~, except that the precycle (11.3) is omitted.

[4] The boldface numbers in parentheses refer to the list of references at the end of this test method.