Designation: F3418 – 20

Standard Test Method for Measurement of Transition Temperatures of Slack Waxes used in Equine Sports Surfaces by Differential Scanning Calorimetry (DSC)[1]

This standard is issued under the fixed designation F3418; 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 The slack waxes typically used in equestrian surfaces comprise a blend of different waxes and oils containing a variety of hydrocarbons, chain lengths and structures.

1.2 The blend of wax and oil determines the mechanical properties of the surface material as well as the response of the wax to temperature. The combination of lower and higher carbon weight materials, oil content and hydrocarbon structures also control how the wax will change over time.

1.3 The differential scanning calorimetry (DSC) test is used to determine temperature transitions and melting range of wax samples. DSC can therefore demonstrate differences in heat flow rates between extracted wax samples. The wax samples are extracted from samples of the surface materials and used in a standard test based on Test Method D4419 (**1**).[2] This procedure involves thermal cycling of samples between –30 and 94 °C using a known control.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[3]

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

D4419 Test Method for Measurement of Transition Temperatures of Petroleum Waxes by Differential Scanning Calorimetry (DSC)

E472 Practice for Reporting Thermoanalytical Data (Withdrawn 1995)[4]

E473 Terminology Relating to Thermal Analysis and Rheology

E474 Method for Evaluation of Temperature Scale for Differential Thermal Analysis (Withdrawn 1986)[4]

3. Terminology

3.1 *Definitions:*

3.1.1 *differential scanning calorimetry (DSC), n*—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.

3.1.1.1 *Discussion*—There are two modes of operation: power-compensation DSC and heat-flux DSC. That can be distinguished depending on the method of measurement used. For additional background information refer to Practice E472, Terminology E473, and Method E474.

[1] This test method is under the jurisdiction of ASTM Committee F08 on Sports Equipment, Playing Surfaces, and Facilities and is the direct responsibility of Subcommittee F08.28 on Equestrian Surfaces.

Current edition approved Jan. 1, 2020. Published January 2020. DOI: 10.1520/F3418-20.

[2] The boldface numbers in parentheses refer to a list of references at the end of this standard.

[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.

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

4. Summary of Test Method

4.1 Separate samples of petroleum wax and a reference material or blank (a sample container filled with air) 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 changes in 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.

5. Significance and Use

5.1 DSC is 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 binders used in synthetic equine sports surfaces, provide information on the effect of operational track temperatures on binder melting, as well as giving indications of changes in the binder over time. The solid-solid temperature transition is related to the properties of the solid, that is, hardness and blocking temperature, although these slack wax-wax based binders typically contain oil contents greater that 20 % by mass and consequently are not in a hardened state unless subjected to very cold temperatures (well below –17 °C).

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 de-oiling a single petroleum distillate with a maximum range of 49 °CF between its 5 and 95 % vol in accordance with Test Method D1160 boiling points (converted to 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 degrees Celsius, 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 (10.0 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 10.0 ± 1.0°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 10.0 ± 1.0°C/min from –30.0°C to 150.0°C. Controlled cooling capability is preferred but not essential. The calorimeter must be able to automatically record the differential signal (WE or WT) versus temperature with a temperature repeatability of ±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. Reagents and Materials

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

9. Specimen Preparation

9.1 To ensure homogeneity, completely melt the entire sample by heating it to 10.0°C above the temperature at which the wax is completely liquid. Using a clean transfer pipet, transfer a few drops to the surface of a clean sheet of aluminum foil to form a thin wax film. Separate the wax from the foil and break it into pieces.

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

10. Calibration and Standardization

10.1 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 (2)	26.9	300.0
p-Nitrotoluene (3)	51.5	324.8
Naphthalene (4)	80.3	353.6
Benzoic Acid[A]	122.4	395.7
Adipic Acid (5)	153.0	426.3
Indium Metal (2)	156.6	429.9

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

11. Procedure

11.1 Weigh 10.0 ± 1.0 mg of the wax pieces into a sample pan and insert the pan in the calorimeter sample compartment. Record the mass.

NOTE 3—Intimate thermal contact, sample-to-pan and pan-to-sensor, is essential. Ensure that pan bottoms are flat and that sensor surfaces where pans rest are clean. If the equipment is available, it is advantageous to