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Standard Guide for Evaluation of Biodegradable Heat Transfer Fluids[1]

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1. Scope*

1.1 This guide[2] covers general information, without specific limits, for selecting standard test methods for evaluating heat transfer fluids for quality and aging. These test methods are considered particularly useful in characterizing biodegradable water-free heat transfer fluids in closed systems.

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

1.3 *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]

D86 Test Method for Distillation of Petroleum Products and Liquid Fuels at Atmospheric Pressure
D91 Test Method for Precipitation Number of Lubricating Oils
D92 Test Method for Flash and Fire Points by Cleveland Open Cup Tester
D93 Test Methods for Flash Point by Pensky-Martens Closed Cup Tester
D95 Test Method for Water in Petroleum Products and Bituminous Materials by Distillation
D97 Test Method for Pour Point of Petroleum Products
D189 Test Method for Conradson Carbon Residue of Petroleum Products
D445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and Calculation of Dynamic Viscosity)
D471 Test Method for Rubber Property—Effect of Liquids
D524 Test Method for Ramsbottom Carbon Residue of Petroleum Products
D664 Test Method for Acid Number of Petroleum Products by Potentiometric Titration
D893 Test Method for Insolubles in Used Lubricating Oils
D1160 Test Method for Distillation of Petroleum Products at Reduced Pressure
D1298 Test Method for Density, Relative Density, or API Gravity of Crude Petroleum and Liquid Petroleum Products by Hydrometer Method
D1500 Test Method for ASTM Color of Petroleum Products (ASTM Color Scale)
D2270 Practice for Calculating Viscosity Index from Kinematic Viscosity at 40 °C and 100 °C
D2717 Test Method for Thermal Conductivity of Liquids (Withdrawn 2018)[4]
D2766 Test Method for Specific Heat of Liquids and Solids (Withdrawn 2018)[4]
D2879 Test Method for Vapor Pressure-Temperature Relationship and Initial Decomposition Temperature of Liquids by Isoteniscope
D2887 Test Method for Boiling Range Distribution of Petroleum Fractions by Gas Chromatography
D4052 Test Method for Density, Relative Density, and API Gravity of Liquids by Digital Density Meter
D4175 Terminology Relating to Petroleum Products, Liquid Fuels, and Lubricants
D4530 Test Method for Determination of Carbon Residue (Micro Method)
D5864 Test Method for Determining Aerobic Aquatic Biodegradation of Lubricants or Their Components
D6384 Terminology Relating to Biodegradability and Ecotoxicity of Lubricants
D6743 Test Method for Thermal Stability of Organic Heat Transfer Fluids
D7042 Test Method for Dynamic Viscosity and Density of Liquids by Stabinger Viscometer (and the Calculation of

[1] This guide is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.L0.06 on Non-Lubricating Process Fluids.

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[2] The background for this standard was developed by a questionnaire circulated by ASTM-ASLE technical division L-VI-2 and reported in *Lubrication Engineering*, Vol 32, No. 8, August 1976, pp. 411–416.

[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.

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

Kinematic Viscosity)

D7044 Specification for Biodegradable Fire Resistant Hydraulic Fluids

E659 Test Method for Autoignition Temperature of Chemicals

G4 Guide for Conducting Corrosion Tests in Field Applications

2.2 *OECD Standards:*[5]

Test No. 203 : Fish, Acute Toxicity Test

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 For definitions of terms used in this guide, refer to Terminology D4175.

3.1.2 *fluid aging, n*—process of fluid degradation associated with the loss of intended performance of the fluid, which includes fluid composition changes, soot formation, and the deposit of materials on a surface (fouling).

3.1.3 *fluid quality, n*—describes the fluid's appropriateness for the intended application including factors necessary for safety and environmental awareness or compliance.

3.1.4 *heat transfer fluid, n*—fluid that remains essentially a liquid while transferring heat to or from an apparatus or process, although this guide does not preclude the evaluation of a heat transfer fluid that may be used in its vapor state. Heat transfer fluids may be hydrocarbon or petroleum based, such as polyglycols, esters, hydrogenated terphenyls, alkylated aromatics, diphenyl-oxide/biphenyl blends, and mixtures of di- and triaryl-ethers. Small percentages of functional components such as antioxidants, antiwear and anti-corrosion agents, TBN, acid scavengers, or dispersants, or a combination thereof, can be present.

4. Significance and Use

4.1 The significance of each test method depends upon the system in use and the purpose of the test method as listed under Section 5. Use the most recent editions of ASTM test methods.

5. Recommended Test Procedures

5.1 *Pumpability of the Fluid:*

5.1.1 *Flash Point, Closed Cup (Test Method D93)*—This test method detects low flash ends which are one cause of cavitation during pumping. In closed systems, especially when fluids are exposed to temperatures of 225 °C or higher, the formation of volatile hydrocarbons by breakdown of the oil may require venting through a pressure relief system to prevent dangerous pressure build-up.

5.1.2 *Pour Point (Test Method D97)*—The pour point can be used as an approximate guide to the minimum temperature for normal pumping and as a general indication of fluid type and low temperature properties. Should a heat transfer system be likely to be subjected to low temperatures when not in use, the system should be trace heated to warm the fluid above minimum pumping temperature before start-up.

5.1.3 *Viscosity (Test Method D445 or D7042)*—Fluid viscosity is important for determining Reynolds and Prandtl numbers for heat transfer systems, to estimate fluid turbulence, heat transfer coefficient, and heat flow. Generally, a fluid that is above approximately 200 cSt is difficult to pump. The pump and system design determine the viscosity limit required for pumping. The construction of a viscosity/temperature curve using determined viscosities can be used to estimate minimum pumping temperature.

5.1.4 *Specific Gravity (Test Method D1298 or D4052)*—Hydraulic shock during pumping has been predicted via the use of a combination of density and compressibility data.

5.1.5 *Water Content (Test Method D95)*—The water content of a fresh heat transfer fluid can be used to indicate how long the heat transfer system shall be dried out during commissioning, while raising the bulk oil temperature through the 100 °C plus region, with venting, before the system can be safely used at higher temperatures. The expansion tank should be full during the operations to ensure that moisture is safely vented in the lowest pressure part of the systems. Positive nitrogen pressure on the heat exchange systems minimizes entry of air or moisture. Heat transfer systems operating at temperatures of 120 °C or greater shall, for reasons of safety, be dry, because destructive high pressures are generated when water enters the high temperature sections of the system. Heating the oil before it is placed in service also removes most of the dissolved air in the oil. If not removed, the air can cause pump cavitation. The air can also accumulate in stagnant parts of the system at high pressure and could cause an explosion.

5.1.6 *Vapor Pressure (Test Method D2879)*—Vapor pressure, which normally increases with increasing operating temperature, is an important design parameter. Organic heat transfer fluids exhibiting high vapor pressures should be used only in systems with sufficient structural integrity. Operation of vapor phase systems requires knowledge of the equilibrium vapor pressure.

5.2 *Safety in Use:*

5.2.1 *Autoignition Temperature (Test Method E659)*—This test relates to the autoignition temperature of a bulk fluid. Hydrocarbon fluids absorbed on porous inert surfaces can ignite at temperatures more than 50 °C lower than indicated by Test Method E659. An open flame ignites leaking hydrocarbon fluids exposed on a porous surface at any temperature.

5.2.2 *Flash Point (Test Methods D92 and D93)*—Some heat transfer fluids are volatile and present a fire hazard at slightly elevated temperatures, or even below 25 °C.

5.2.3 *Biodegradation (Test Method D5864)*[6]—An environmental concern, and toxicity (like acute fish toxicity, see OECD Test No. 203) is more of an immediate concern for personnel that may come into contact with the heat transfer fluid as well as plant and animal life that may come into contact with effluent that migrates to streams, rivers, or reservoirs. Safety in use information is included a Material Safety Data

[5] Organisation for Economic Co-operation and Development (OECD), 2, rue André Pascal, 75775 Paris Cedex 16, France.

[6] As heat transfer fluids are mainly used in closed and sealed circuits, this guide is limited to biodegradation issues. An extension to "environmentally friendly" or "ecoevaluated" heat transfer fluids including aquatic toxicities may be an object of future revision.

Sheet (MSDS). See Terminology D6384 for terminology relating to biodegradability and ecotoxicity. A basis for biodegradable classification for hydraulic fluids is found in Table 4 of Specification D7044 and while no specific biodegradability classifications are available for heat transfer fluids, the environmental persistence of heat transfer fluids should be evaluated.

5.3 *Effect on Equipment:*

5.3.1 *Effect on Rubber or Elastomeric Seals (Test Method D471)*—Most seals in heat exchange equipment are made of steel or other metal. If rubber seals are present, it is desirable to maintain rubber swelling in the range of 1 % to 5 % to prevent leakage because of poor seal contact. Seals may degrade in some fluids. As an oil deteriorates in service, additional tests may be required to ensure that seals remain compatible with the altered oil. The temperature ranges of the tests should correspond to temperatures to which seals will be exposed in service.

5.3.2 *Corrosion (Guide G4)*—These tests concern selection of materials of construction with fluids usable for heat transfer systems. Guide G4 uses test metal specimens fixed within the stream of test fluid under use. The specimens and conditions for test shall be specified for each system.

5.4 *Efficiency:*

5.4.1 *Thermal Conductivity (Test Method D2717) and Specific Heat (Test Method D2766)*—These thermal conductivity and specific heat tests are difficult to carry out, facilities for performing them are few, and the precision data is yet to be established. Values can be estimated for design use from the general chemical composition. Differences contribute to efficiency to a lesser degree than values such as viscosity, moisture contamination, and other measurable values in 5.1 and 5.5 of this guide. The values for thermal conductivity and specific heat may be available from the fluid supplier.[7]

5.5 *Service Life:*

5.5.1 *Thermal Stability, Laboratory Tests*[8]—Thermal stability is here defined as the resistance of a hydrocarbon liquid to permanent changes in properties that make it a less efficient heat transfer fluid. These changes may be related to alterations in the liquid's properties, such as viscosity, or to the tendency to foul heat exchanger surfaces with insulating deposits. Normally, testing should be done in the absence of air and moisture to stimulate "tight" systems. The test may be useful for assessing the remaining service life of a used fluid, or it may be used to compare the expected service life of competitive new heat transfer fluids.

5.5.2 The following test methods can be used to determine the change in values between new and used fluids, or between a fluid before and after subjection to a laboratory thermal stability test. These test methods have been found especially useful for determining the end of a fluid's service life when an identical fluid has been monitored with the same tests throughout its service life. These test methods can also detect leakage of foreign material into the heat transfer fluid.

5.5.2.1 *Precipitation Number (Test Method D91) and Insolubles (Test Method D893)*—These test methods determine the extent to which insolubles that may contribute to fouling of metal surfaces are increasing.

5.5.2.2 *Flash Point (Test Methods D92 and D93)*—A lowering of flash point is indicative of thermal cracking to produce lower molecular weight hydrocarbons. A rapid increase may indicate that fluid is being exposed to excessive temperatures.

5.5.2.3 *Carbon Residue (Test Methods D189, D524, and D4530)*—An increase of carbon residue during service provides an indication of the fluid's tendency to form carbonaceous deposits. These deposits, which may impair heat transfer, are caused by precipitation of high molecular weight materials produced by thermal cracking of the fluid.

5.5.2.4 *Viscosity (Test Method D445 or D7042)*—An increase in viscosity may reduce the fluid's ability to transfer heat (see 5.1.3). Cracking of hydrocarbons in high temperature service in closed systems often causes a decrease in viscosity. Thus a change in viscosity taken by itself is insufficient to judge the performance of a fluid in service.

5.5.2.5 *Distillation (Test Methods D86, D1160, and D2887)*—Distillation can show directly the percentage of a fluid that has cracked into lower boiling products or has been converted into higher boiling products. Distillation data can serve as the sole criterion for changing a heat transfer fluid.

5.5.2.6 *Neutralization Number (Test Method D664)*—A marked increase in neutralization number is a warning of oxidation in the system, which may be the result of leaks. In high-temperature service (200 °C), organic acids may decompose, and the use of infrared analyses may serve as a more reliable method for detection of oxidation.

5.5.2.7 *Color (Test Method D1500)*—In itself, color is not important, but may be the initial indication of chemical changes in the heat transfer system.

5.5.2.8 *Viscosity Index (Practice D2270)*—The viscosity index of a fluid may change during service. Generally, the viscosity of a heat transfer fluid is not measured at the operating temperature (see 5.5.2.4). If the viscosity index of new and used fluids are known, the viscosities at operating temperature can be estimated and compared.

5.5.2.9 *Water Content (Test Method D95)*—Small amounts of water present in heat transfer systems may cause corrosion, high pressures, or pump cavitation.

6. Keywords

6.1 biodegradable; characterization; heat transfer fluid; heat transfer oil; heat transfer system

[7] Useful estimates may be obtained from sources such as the "Technical Data Book, Petroleum Refining," American Petroleum Institute, 1220 L St., N.W, Washington, DC 20005-4070.

[8] Test Method D6743 measures thermal stability of organic heat transfer fluids.