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Standard Guide for Measuring the Adhesion of Crude Oils and Fuel Oils[1]

This standard is issued under the fixed designation F3633; 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 guide summarizes a method to measure the adhesion to a stainless-steel needle as means to compare the relative adhesion of the target oil.

1.2 This guide covers general procedures for measuring the adhesion of oils to stainless steel and does not cover all possible procedures that may be applicable to this topic.

1.3 The accuracy of this guide depends very much on the representative nature of the oil sample used. Certain oils can have different properties depending on their chemical contents at the time a sample is taken.

1.4 *Units*—The values stated in SI units are to be regarded as the 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 establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.6 *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. Summary of Guide

2.1 A standard stainless-steel needle is dipped into oil for 30 s then raised and allowed to drain for 30 min. The weight of oil adhered to the needle over the area of the needle immersed is taken as the adhesion per unit/area.[2-4]

2.1.1 An exception is for light oils and fuels that do not have enough adhesion to yield a significant result.

3. Significance and Use

3.1 A standard procedure is necessary to measure the adhesive properties of oil to enable comparison between oils.

3.2 This procedure uses standardized equipment and test procedures.

3.3 This procedure should be performed at the stages of weathering corresponding to the spill conditions of interest.

4. Interferences and Sources of Error

4.1 Large proportions of inorganic substances in the oil sample, such as water and sediment that are produced with the oil at source, can change the adhesion values. Water and sediment are typically removed during crude petroleum processing; however, if the oil sample is sourced upstream of this step, then it is necessary to decant the water before adhesion measurement to obtain an accurate value.

4.2 Interferences can be caused by contaminants, particularly residual oil or surfactants on labware, and other sample-handling supplies and apparatus that lead to nonrepresentative oil samples. All glassware and the measurement needle shall be thoroughly cleaned. The cleaning process includes thorough rinsing with dichloromethane to remove all traces of oil, wiping with a soft, non-linting cloth, followed by a final rinse with acetone. Once cleaned, precautions shall be taken to minimize contact of the labware and measurement needle with contaminants to prevent interferences.

4.3 Temperature is a factor in the adhesion value, so it is important that the oil and the equipment are at 20 °C or the selected test temperature before starting the adhesion measurement.

4.4 The handling of the samples is important. Contaminants that are introduced onto the vessel or handling equipment will affect the adhesion determination.

4.5 Oil sources, especially crude oil sources, vary much with production time and conditions. Oil samples shall be

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[2] "Summary methods for the analysis of physical properties, chemical composition, and behavior of petroleum products," *Emergencies Science and Technology Section (ESTS) Environment and Climate Change Canada*, 2019.

[3] Jokuty, P., Whiticar, S., McRoberts, K., and Mullin, J., "Oil Adhesion Testing—Recent Results," in *Proceedings of the Nineteenth AMOP Technical Seminar*, Environment Canada, Ottawa, ON, 1996, pp. 9-27.

[4] Jokuty, P., Fieldhouse, B. G., Fingas, M. F., Whiticar, S. J., and Latour, J., "Bitumen Adhesion as a Function of Time," *Proceedings of the Twenty-first AMOP Technical Seminar*, Environment Canada, Ottawa, ON, 1998, pp. 27-32.

treated as unique and are not necessarily representative of the source. Depending on the actual conditions under which this oil was sampled, different values for properties are measured.

5. Handling and Storing Oil

5.1 *Crude or Raw Oil Sample Storage and Preparation*—The bulk oil as received is mechanically mixed until fully homogenized before obtaining a working sample. Working samples are transferred to high-density polyethylene or glass bottles with leak-resistant screw closures with minimized head space and stored in a temperature-controlled room at 5 °C to reduce the potential for weathering. The sample is then shaken mechanically before use and brought to 20 °C before adhesion measurement.

6. Materials and Equipment

6.1 *Overview*—A penetrometer needle is slung from a balance. A sample of oil is raised to a specific level on the needle and allowed to remain there for 30 s. The oil sample is lowered and the oil allowed to drain from the needle for 30 min. The weight of the oil retained is divided by the surface area of the needle immersed to yield a mass/unit surface area.

6.2 *Equipment*

6.2.1 *Test Surface and Penetrometer Needle*—A stainless-steel penetrometer needle is used as the test surface; see Fig. 1. The needle is best marked to indicate the maximum depth of immersion. The marking can be taken as the point where the thin needle enters the wider brass support.

6.2.2 The holder and hook are comprised of a pin vise to grasp the needle and eye hook on the other end to couple to the balance hook.

6.2.3 A microbalance capable of measuring up to 40 g with an accuracy of ±0.1 mg is required. The balance requires a bottom hook to measure below the balance.

6.2.4 *Windshield and Stand*—A box of approximately 45 by 22 by 32 cm (length, width, and height) is built to act as a stand for the balance. The front is transparent to enable sight of the operation. A door on the front is necessary to manipulate the sample. In Fig. 2, the front of this windshield and stand is shown.

6.2.5 *Laboratory Jack*—A 15 cm laboratory jack is used to raise and lower the oil test beaker.

6.2.6 *Beaker*—A 100 mL beaker or another stable, transparent container of sufficient depth is used to contain the oil for the measurement.

7. Measuring with the Apparatus

7.1 *Checking the Temperature*—The apparatus and oil should be at 20 °C before proceeding.

7.2 *Taring the Apparatus*—Hang a clean, dry penetration needle under the balance within the plexiglass stand. Close the stand cover. Let the hanging needle become stable with no movement and tare the balance.

7.3 *Introducing the Sample*—Place the 100 mL beaker or container containing the oil sample on top of the jack. The depth of the oil sample should be sufficient to immerse the needle fully. Position the container so that it is under the needle.

7.4 *Immersing the Needle*—Raise the container using the jack until the needle is fully submerged to the mark. Take care to immerse only the stainless-steel needle and not the brass support. If the oil meniscus touches the brass support, oil can be trapped at the top of the needle and increase the apparent adhesion of the oil.

7.5 *Measurement*—The needle is allowed to rest in the oil for 30 s after which the beaker is lowered. The needle is left to hang undisturbed for 30 min with the plexiglass shroud closed. After the 30 min have passed, the mass is recorded. If a drop of oil is retained at the point of the needle, this should be removed by touching a glass rod or something similar to draw off the droplet without disturbing the needle surface.

7.6 *Viscous Oils*—If the oil sample is relatively viscous, be careful when raising the oil sample to immerse the needle. If

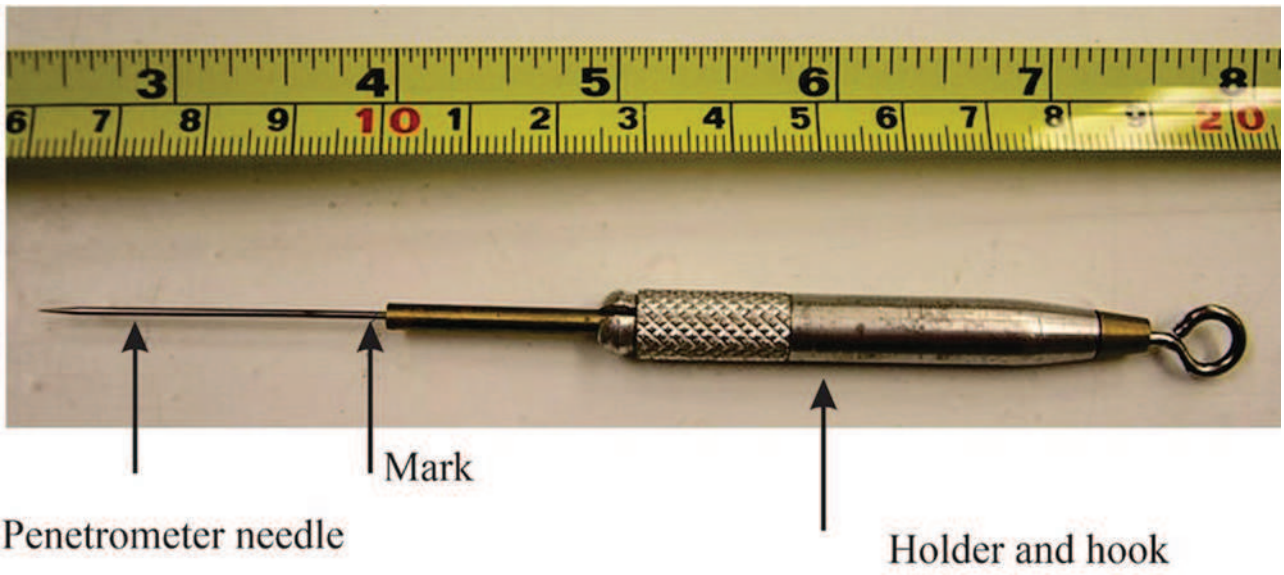

FIG. 1 Photograph of Penetrometer Needle and Holder

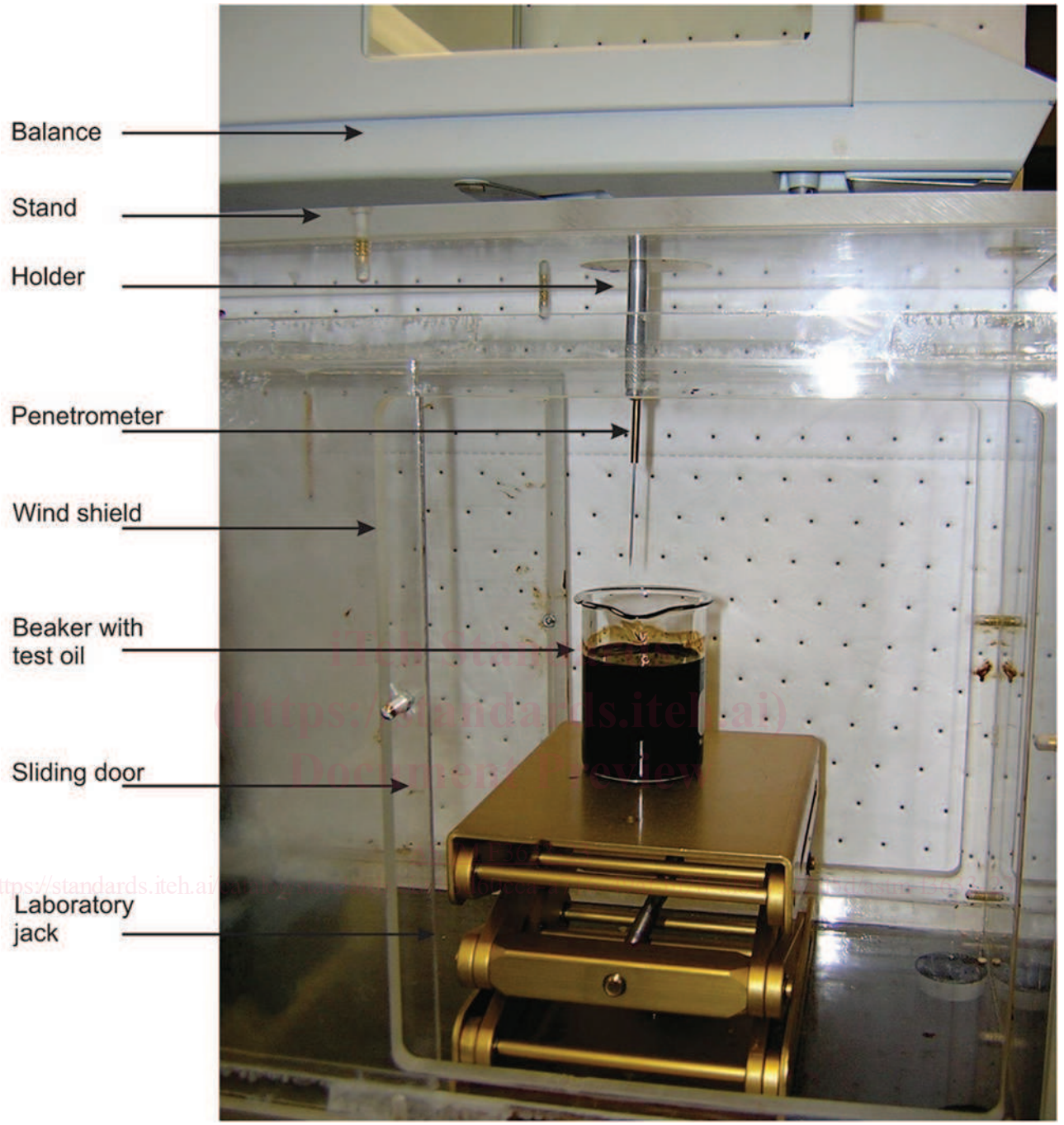

FIG. 2 Stand Showing Various Components of the Adhesion Measurement Devic

the oil is sufficiently viscous, then the needle may be lifted and fall off the hook attached to the balance. Raising the needle should be done very slowly to allow penetration of the needle into the viscous oil.

7.7 *Cleaning*—Clean the needle thoroughly with dichloromethane, wipe with a soft, non-linting cloth, followed by a final rinse with acetone, and allow it to dry completely before reuse.

7.8 *Repetition*—The measurement is repeated three times and the mean average reported. The oil sample should be sealed following needle dipping to limit evaporation of the sample between measurements. If the standard deviation is more than 5 %, the measurement should be repeated on a new oil sample and the needle thoroughly cleaned.

8. Calculation and Reporting

8.1 The weight of the oil retained is divided by the area of the needle. In Table 1, the area calculations for two different needles are shown.

8.2 The adhesion of a reference oil is measured and compared with previous results to monitor consistency as a means of quality control. A nonvolatile commercial product such as a