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**Determination of flash point —
Part 3 : Pensky-Martens
closed cup method**

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Foreword

This translation has been made based on the original Japanese Industrial Standard established by the Minister of Economy, Trade and Industry, through deliberations at the Japanese Industrial Standards Committee according to the proposal of establishing a Japanese Industrial Standard from Petroleum Association of Japan (PAJ), with a draft being attached, based on the provision of Article 12 Clause 1 of the Industrial Standardization Law.

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JIS K 2265 consists of the following 4 parts, under the general title “*Determination of flash point*”:

Part 1 : Tag closed cup method

Part 2 : Rapid equilibrium closed cup method

Part 3 : Pensky-Martens closed cup method

Part 4 : Cleveland open cup method

Determination of flash point—Part 3: Pensky-Martens closed cup method

Introduction

This Japanese Industrial Standard has been prepared based on the third edition of **ISO 2719** *Determination of flash point—Pensky-Martens closed cup method* published in 2002 with some modifications of the technical content in order to conform to the domestic actual situation.

The portions given sidelines or dotted underlines in this part of **JIS K 2265** are the matters modified from the original International Standard. A list of modifications with the explanations is given in Annex JB.

The use of this part of **JIS K 2265** may involve hazardous materials, operations and equipment. This part of **JIS K 2265** does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this test method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

1 Scope

This part of **JIS K 2265** describes two procedures, A and B, using the Pensky-Martens closed cup tester, for determining the flash point of combustible liquids, liquids with suspended solids, liquids that tend to form a surface film under the test conditions and other liquids, whose flash point exceeds 40 °C.

Procedure A is used for the determination of the flash point of paints and varnishes that do not form a surface film, unused lubricating oils and other petroleum products not covered by Procedure B.

Procedure B is used for the determination of the flash point of residual fuel oils, cut-back bitumens, used lubricating oils, liquids that tend to form a surface film, liquids with suspensions of solids and highly viscous materials such as polymeric solutions and adhesives.

- a) Although technically kerosines with a flash point above 40 °C may be tested using this procedure, it is standard practice to test kerosines according to JIS K 2265-1. Similarly, unused lubricating oils are normally tested according to JIS K 2265-4.
- b) Residual fuel oils may be tested using Procedure A. However, when a doubt arises, it shall be tested using Procedure B.
- c) Used lubricating oils may be tested using Procedure A. However, the precision data for these materials is only valid for Procedure B.
- d) Water-borne paints may be tested using **JIS K 2265-2**. Liquids contaminated by traces of highly volatile materials may be tested using **JIS K 2265-2** or **ISO 1523**.

- e) Precision data is only valid for the flash point ranges given in clause 13.

NOTE 1 This Standards group includes the test methods shown in Annex JA.

NOTE 2 The International Standard corresponding to this part of JIS K 2265 and the symbol of degree of correspondence are as follows.

ISO 2719:2002 *Determination of flash point—Pensky-Martens closed cup method* (MOD)

In addition, a symbol (MOD) that denotes the degree of correspondence in the contents between the relevant International Standard and JIS shows modification according to ISO/IEC Guide 21.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this part of JIS K 2265.

The most recent editions of the standards (including amendments) indicated below shall be applied.

JIS B 7410 *Liquid-in-glass thermometers for testing of petroleum product*

JIS K 2251 *Crude petroleum and petroleum products—Sampling*

NOTE : The corresponding International Standard: ISO 3170 *Petroleum liquids—Manual sampling* (MOD)

JIS K 2265-1 *Determination of flash point—Part 1: Tag closed cup method*

JIS K 2265-2 *Determination of flash point—Part 2: Rapid equilibrium closed cup method*

NOTE : The corresponding International Standard: ISO 3679 *Determination of flash point—Rapid equilibrium closed cup method* (MOD)

JIS K 2265-4 *Determination of flash points—Part 4: Cleveland open cup method*

NOTE : The corresponding International Standard: ISO 2592 *Determination of flash and fire points—Cleveland open cup method* (MOD)

JIS K 5600-1-2 *Testing methods for paints—Part 1: General rules—Section 2: Sampling*

NOTE : The corresponding International Standard: ISO 15528 *Paints, varnishes and raw materials for paints and varnishes—Sampling* (IDT)

JIS K 5600-1-3 *Testing methods for paints—Part 1: General rule—Section 3: Examination and preparation of samples for testing*

NOTE : The corresponding International Standard: ISO 1513 *Paints and varnishes—Examination and preparation of samples for testing* (IDT)

JIS Q 0033 *Uses of certified reference materials*

NOTE : The corresponding International Standard: ISO Guide 33 *Uses of certified reference materials* (IDT)

JIS Q 0034 *General requirements for the competence of reference material producer*

NOTE : The corresponding International Standard: **ISO Guide 34** *General requirements for the competence of reference material producers* (IDT)

JIS Q 0035 *Certification of reference materials—General and statistical principles*

NOTE : The corresponding International Standard: **ISO Guide 35** *Reference materials—General and statistical principles for certification* (IDT)

JIS Z 8401 *Guide to the rounding of numbers*

JIS Z 8402-4 *Accuracy (trueness and precision) of measurement methods and results—Part 4: Basic methods for the determination of the trueness of a standard measurement method*

JIS Z 8402-6 *Accuracy (trueness and precision) of measurement methods and results—Part 6: Use in practice of accuracy values*

ISO 1523 *Determination of flash point—Closed cup equilibrium method*

ISO 3171 *Petroleum liquids—Automatic pipeline sampling*

3 Term and definition

For the purposes of this part of **JIS K 2265**, the following terms and definitions apply.

3.1 flash point

lowest temperature of the test portion, corrected to a barometric pressure of 101.3 kPa, at which the application of an ignition source causes the vapour of the test portion to ignite momentarily and the flame to propagate across the surface of the liquid under the specified conditions of test

4 Principle

The specified quantity of test portion is placed in the test cup of a Pensky-Martens apparatus and heated to give a constant temperature increase with continuous stirring. An ignition source is directed through an opening in the test cup lid at regular temperature intervals with simultaneous interruption of stirring. The lowest temperature at which the application of the ignition source causes the vapour of the test portion to ignite and propagate over the surface of the liquid is recorded as the flash point at the ambient barometric pressure. This temperature is corrected to standard atmospheric pressure using an equation.

5 Reagents

5.1 Cleaning solvent

The solvent shall be suitable to remove the traces of sample tested before from the test cup and cover.

NOTE : The choice of solvent will depend upon the previous material tested, and the tenacity of the residue. Low volatility aromatic (benzene-free) solvents may be used to remove traces of oil, and mixed solvents, such as toluene-acetone-methanol (TAM), may be efficacious for the removal of gum-type deposits.

5.2 Verification liquids

A series of certified reference materials (hereafter referred to as a “CRM”) or the secondary working standards (hereafter referred to as a “SWS”) as specified in Annex A.

6 Apparatus

6.1 Flash point apparatus

Pensky-Martens closed cup test apparatus as specified in Annex B.

If automated equipment is used, ensure that it has been established that the results obtained are within the precision of this part of **JIS K 2265** and that the test cup and cover assembly conform to the dimensional and mechanical requirements as specified in Annex B and the test procedure conform to clause 10. If automated testers are used, the user shall ensure that all of the manufacturer's instructions for adjusting and operating the instrument are followed.

Under certain circumstances, the use of electric ignition sources may give different results to those obtained when using a flame ignition source. In addition, electric ignition sources may give variable results.

In cases of dispute, unless explicitly agreed otherwise, the manual determination of the flash point using a flame ignition source shall be considered as the referee test.

6.2 Thermometers

Thermometer No. 30 (PMF) or 31 (PMF) as specified in JIS B 7410.

The initial choice of thermometer shall be based on the expected flash point.

In addition, when an electric type of temperature-measuring devices is used, it shall meet the requirements for accuracy and have the same response as the thermometers specified in.

6.3 Barometer

The scale shall be readable to 0.1 kPa digits.

Barometers pre-corrected to give sea-level readings, such as those used at weather stations and airports, shall not be used.

In addition, although there are some barometers whose reading is corrected to 0 °C automatically, this correction is not necessary.

6.4 Heating bath or oven

It shall be capable of controlling the temperature of sample to ± 5 °C as specified in 9.1.4.

It is used for warming the sample, if required. The oven shall be of safety design.

7 Apparatus preparation

7.1 Location of the apparatus

Support the apparatus on a level and steady surface in a draught-free position.

When draughts cannot be avoided, it is good practice to surround the apparatus with a shield of suitable size.

When testing samples that produce toxic vapours, the apparatus may be located in a draught chamber with an individual control of air flow, adjusted so that vapours can be withdrawn without causing air currents around the test cup during the test.

7.2 Cleaning the test cup and its accessories

Wash the test cup, cover and its accessories with an appropriate solvent to remove any traces of gum or residue remaining from a previous test. Dry the test cup using a stream of clean air to ensure complete removal of the solvent used. An apparatus shall be cleaned and maintained according to the manufacturer's instructions.

7.3 Apparatus assembly

Examine the test cup, the cover and other parts to ensure that they are free from signs of damage and deposits. Assemble the apparatus in accordance with Annex B.

7.4 Apparatus verification

Apparatus verification shall be as follows.

- a) Verify the corrected functioning of the apparatus at least once a year by testing a CRM in accordance with Procedure A or Procedure B. The result obtained shall be equal to or less than $R/\sqrt{2}$ from the certified value of the CRM, where R is the reproducibility at the time of certification of this CRM.

It is recommended that more frequent verification checks be made using SWS.

A recommended procedure for apparatus verification using CRMs or SWSs, and the production of SWSs, is given in Annex A.

- b) The numerical values obtained during the verification check shall not be used to provide a bias statement, nor shall they be used to make any correction to the flash points subsequently determined using this apparatus.

8 Sampling and preparation of samples

8.1 A sample for test shall be sampled and prepared according to the sampling of a primary sample and the preparation of a secondary sample as specified in **JIS K 2251**, or according to the methods which are specified in **JIS K 5600-1-2** or an equivalent standard. In the case of automatic sampling, the sampling and preparation may be in accordance with **ISO 3171**.

8.2 Place samples in tightly sealed containers, appropriate to the material being sampled. Its quality shall be sufficient for a test.

For safety purposes and prevention of loss of vapour, ensure that the sample container is only filled to between 85 % to 95 % of its capacity. However, any container made of plastics (such as polyethylene or polypropylene) shall not be used because the volatile substance in the sample may vaporize through the wall of the container.

In the case of performing two or more tests, the subsample amount should be chosen so that the conditions of 9.1.1 is applied.

8.3 Store the samples in conditions to minimize vapour loss and pressure build-up. Avoid storing the samples at temperatures in excess of 30 °C.

9 Sample handling

9.1 Petroleum products

9.1.1 Subsampling

Subsample at a temperature at least 28 °C below the expected flash point. If an aliquot of the original sample is to be stored prior to testing, ensure that the container is filled to at least 50 % of its capacity.

NOTE : If the sample amount is below 50 % of the capacity of a sample container, then a test result may be affected.

9.1.2 Sample containing undissolved water

If a sample contains undissolved water, remove undissolved water from an aliquot using the decantation method prior to mixing. The flash point results can be affected by the presence of water. For certain fuel oils and lubricants, it may not always be possible to remove undissolved water using the decantation method. In such cases, the water should be separated from the aliquot physically, prior to mixing, using suitable dehydrators such as calcium chloride, or filtering a sample through the filter paper or the absorbent cotton. If this is not possible, the material should be tested in accordance with JIS K 2265-2.

NOTE 1. When the residual fuel oil containing much water is tested, a sample may bubble or boil over from the test cup during heating.

NOTE 2. When the sample contains water-soluble combustible components, such as alcohol, if water is removed, then such combustible components may be removed together. Therefore, the reliable result cannot be obtained.

9.1.3 Samples that are liquid at ambient temperature

Mix samples that are liquid at ambient temperature by gentle manual shaking prior to the removal of the test portion, taking care to minimize the loss of volatile components.

9.1.4 Samples that are semi-solid or solid at ambient temperature

For samples that are semi-solid or solid at ambient temperature, heat the sample in its container in a heating bath or oven to a temperature of 30 °C ± 5 °C, or a higher temperature not exceeding 28 °C below the expected flash point, whichever is the higher, for 30 min. If after 30 min the sample is still not completely liquefied, extend the preheating for further 30 min periods as required. However, avoid overheating the sample, as this

could lead to the loss of volatile components. Then, agitate the sample gently.

9.2 Paints and varnishes

Prepare the samples according to JIS K 5600-1-3.

10 Procedure

10.1 Procedure A

Procedure A shall be as follows.

- a) Fill the test cup with the test portion to the level indicated by the filling mark. Place the lid on the test cup and put it in the heating chamber. Ensure that the locating or locking device is properly engaged and insert the thermometer into the lid. In the case of a gas test flame, light the test flame applicator and adjust to a diameter of 3 mm to 4 mm in order to fit the standard bead of the lid. In the case of the ignition source of electric type, switch on the alternative ignition source. Light the heater flame or switch on the electric heater and supply heat at such a rate that the temperature of the test portion as indicated by the thermometer increases at 5 °C/min to 6 °C/min, and maintain this heating rate throughout the test. Stir the test portion at a rate of 90 r/min to 120 r/min, stirring in a downward direction.
- b) When the test portion is expected to have a flash point of 110 °C or below, make the first application of the ignition source when the temperature of the test portion is $23\text{ °C} \pm 5\text{ °C}$ below the expected flash point, and thereafter at 1 °C temperature intervals. Cease stirring and apply the ignition source by operating the mechanism on the cover, which controls the shutter and ignition source, so that the source is lowered into the vapour space of the test cup in 0.5 s, left in its lowered position for 1 s, and quickly raised to its high position.
- c) When the test portion is expected to have a flash point of above 110 °C, make the first application of the ignition source when the temperature of the test portion is $23\text{ °C} \pm 5\text{ °C}$ below the expected flash point, and thereafter at temperatures which are a multiple of 2 °C. Cease stirring and apply the ignition source by operating the mechanism on the cover, which controls the shutter and ignition source, so that the source is lowered into the vapour space of the test cup in 0.5 s, left in its lowered position for 1 s, and quickly raised to its high position.
- d) When testing a material of unknown flash point, conduct a preliminary test at a suitable starting temperature. Make the first ignition-source application at 5 °C above the starting temperature and follow the procedure given in 10.1 b) or 10.1 c), as applicable.
- e) Record, as the observed flash point, the temperature of the test portion read on the thermometer at the time when the ignition-source application causes a distinct flash in the interior of the test cup. Do not confuse the true flash point with the bluish halo that sometimes surrounds the ignition source at application preceding the actual flash point.