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(PAJ)

**Determination of flash point —
Part 2: Rapid equilibrium closed cup
method**

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Contents

	Page
Introduction	1
1 Scope	1
2 Normative references	1
3 Term and definition	2
4 Principle	2
5 Reagents	3
6 Apparatus	3
7 Apparatus preparation	4
8 Sampling and preparation of samples	5
9 Sample handling	6
10 Test procedure	6
11 Calculation	8
12 Expression of results	9
13 Precision	9
14 Test report	10
Annex A (normative) Rapid equilibrium closed cup test apparatus	11
Annex B (normative) Thermometer specifications	15
Annex C (normative) Verification of apparatus	16
Annex D (informative) Test cup insert	19
Annex JA (informative) Classification of test methods	20
Annex JB (informative) Comparison table between JIS and corresponding International Standard	21

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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Attention is drawn to the possibility that some parts of this Standard may conflict with a patent right, application for a patent after opening to the public, utility model right or application for registration of utility model after opening to the public which have technical properties. The relevant Minister and the Japanese Industrial Standards Committee are not responsible for identifying the patent right, application for a patent after opening to the public, utility model right or application for registration of utility model after opening to the public which have the said technical properties.

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 2: Rapid equilibrium closed cup method

Introduction

This Japanese Industrial Standard has been prepared based on the third edition of **ISO 3679** *Determination of flash point—Rapid equilibrium closed cup method* published in 2004 with some modifications of the technical content in order to conform to the domestic actual situation.

The portions underlined with dots in this Standard are the matters modified from the International Standard. A list of modifications with the explanations is given in Annex JB.

The use of this Standard may involve hazardous materials, operations and equipment. This Standard 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** specifies a method for the determination of the closed cup flash point of petroleum, paints (including water-borne paints), varnishes, paint binders, adhesives, solvents, fatty acid methyl esters (hereafter referred to as “FAME”) and related products, having closed cup flash points within the range of $-30\text{ }^{\circ}\text{C}$ to $300\text{ }^{\circ}\text{C}$ by using the flash point test apparatus according to rapid equilibrium closed cup method.

NOTE 1 This standards group includes the test methods specified 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 3679:2004 *Determination of flash point—Rapid equilibrium closed cup method* (MOD)

In addition, a symbol (MOD) which 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 Standard. 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 5600-1-2 *Testing methods for paints—Part 1: General rules—Section 2: Sampling*

NOTE : The corresponding International Standard: **ISO 15528** *Paints and 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 3171 *Petroleum liquids—Automatic pipeline sampling*

3 Term and definition

For the purposes of this Standard, the following term and definition apply.

3.1 flash point

lowest temperature of the test portion, corrected to a barometric pressure of 101.3 kPa, at which application of a test flame 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

A test portion of specified volume is introduced into the test cup, which is maintained at the temperature of the estimated flash point of the material under test. After a specified time, a test flame is applied and the presence or absence of a flash is observed. Further tests, with fresh test portions at different temperatures, are carried out until

the flash point is determined to the sensitivity specified.

5 Reagents

5.1 Cleaning solvent

An appropriate solvent for the removal of traces of the previous test portion 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 effective for the removal of gum-type deposits.

5.2 Verification liquid

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

6 Apparatus

6.1 Flash point test apparatus

The rapid equilibrium closed cup test apparatus as specified in Annex A.

When using an automatic test apparatus, the result obtained shall be within the tolerance shown in the accuracy of this Standard. The dimensions and mechanism of the test cup and cover shall conform to the specifications of Annex A and the test procedure shall conform to clause 10. The automatic test apparatus shall be adjusted and handled entirely according to the manufacturer's instructions for use.

When a doubt arises in a result, the result obtained with the manual test apparatus shall be taken as a judging standard.

When measuring the flash point over the whole range of – 30 °C to 300 °C, more than one instrument having different measurement temperature should be required. Moreover, when measuring test portions that require cooling, use a test cup cooler or test apparatus for low temperature.

6.2 Syringes

6.2.1 2 ml syringe

A syringe adjusted to deliver $2.00 \text{ ml} \pm 0.05 \text{ ml}$ of test portion.

If required, 2 ml syringe equipped with a needle suitable for use with the apparatus at test temperatures up to and including 100 °C is applicable.

When testing FAME, a 2 ml syringe is used at all temperatures.

6.2.2 5 ml syringe

A syringe adjusted to deliver $4.00 \text{ ml} \pm 0.10 \text{ ml}$ of test portion.

If required, 5 ml syringe equipped with a needle suitable for use with the apparatus at test temperatures above 100°C is applicable.

When testing FAME, a 5 ml syringe is not used.

6.3 Barometer

The readable scale shall be accurate to 0.1 kPa .

Barometers precorrected 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 to $\pm 5^\circ\text{C}$.

If required, it shall be used for warming the sample. It is recommended that the oven is of explosion-protected design.

6.5 Cooling bath or freezer

It shall be capable of cooling the sample to 10°C below the expected flash point, and controlling the temperature to $\pm 5^\circ\text{C}$.

If a freezer is used, it shall be of explosion-protected design.

6.6 Draught shield

It is used with fitted at the back and on two sides of the instrument, if required to minimize draughts.

A shield, 350 mm high, 480 mm wide and 240 mm deep, is suitable.

6.7 Cup insert

It is shown in Annex D.

For samples that are difficult to remove, a thin metal cup insert can be used.

7 Apparatus preparation

7.1 General

Select the appropriate flash point apparatus (or thermometer) for the expected flash point temperature. Follow the manufacturer's instructions for the corrected set-up and operation of the flash point apparatus. The use of a cup insert for potentially adherent materials is described in Annex D.

7.2 Location of apparatus

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

Use of a draught shield is recommended when protection from draughts is not available.

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

7.3 Cleaning of the test cup and accessories

Clean the test cup, cover and its accessories with an appropriate solvent to remove traces of gum or residue from the previous test. A stream of clean dry air may be used to remove the last traces of solvent used. The filler orifice may be conveniently cleaned with a brush. Follow the manufacturer's instructions for the care and servicing of the instrument.

7.4 Apparatus verification

Verification of test apparatus shall be as follows.

- a) Verify the correct functioning of the apparatus at least once a year by testing CRM. 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 of the method at the time of certification of this CRM.

It is recommended that periodical verification checks are made using SWSs.

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

- 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.

If the test apparatus fails the verification test, the following items should be checked.

- 1) The cover makes a vapour-tight seal with the test cup.
- 2) The shutter provides a light-tight seal (Check for leakage of vapour from the shutter).
- 3) Adequate heat-transfer paste surround the thermometer bulb and immersed portion of the thermometer barrel which are in contact with a heating block.

8 Sampling and preparation of samples

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

8.2 Place sufficient sample volume for testing in a tightly sealed container made of material appropriate to the liquid being sampled.