Essential oils — General guidance on the determination of flashpoint

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National foreword

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TECHNICAL REPORT

ISO/TR 11018

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Essential oils — General guidance on the determination of flashpoint

Huiles essentielles — Directives générales pour la détermination du point d'éclair



Foreword

(the International Organization for Standardization) worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the committee. represented on that International right to be organizations, governmental and non-governmental, in liaison with ISO, take part in the work. ISO collaborates closely with the also International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The main task of technical committees is to prepare International Standards, but in exceptional circumstances a technical committee may propose the publication of a Technical Report of one of the following types:

- type 1, when the required support cannot be obtained for the publication of an International Standard, despite repeated efforts;
- type 2, when the subject is still under technical development or where for any other reason there is the future but not immediate possibility of an agreement on an International Standard;
- type 3, when a technical committee has collected data of a different kind from that which is normally published as an International Standard ("state of the art", for example).

Technical Reports of types 1 and 2 are subject to review within three years of publication, to decide whether they can be transformed into International Standards. Technical Reports of type 3 do not necessarily have to be reviewed until the data they provide are considered to be no longer valid or useful.

ISO/TR 11018, which is a Technical Report of type 3, was prepared by Technical Committee ISO/TC 54, Essential oils.

INTRODUCTION

Carriers, insurance companies, stock managers, etc. need to know the flashpoint of combustible essential oils to provide the necessary conditions for meeting safety requirements for storage and transport.

It was therefore decided to add information on the flashpoint to each International Standard on the specifications of essential oils and to attempt to standardize a suitable test method for its determination.

A comparative study of the methods described in International Standards and other technical documents in similar fields such as paints, varnishes, petroleum and related products was then carried out (see annex A).

This study produced the conclusion that it was difficult to adopt a single method which could be applied to all essential oils, for the following reasons:

- there are a large number of essential oils and their chemical composition varies greatly;
- the recommended volume of sample for the use of certain apparatus (30 ml to 150 ml) is incompatible with the high price of certain essential oils;
- users cannot be forced to buy one type of equipment rather than another when there are several types in existence which respond perfectly well to the intended objectives;
- there exist already many references giving the flashpoint of certain essential oils with various types of apparatus.

In conclusion, it was felt to be impossible to standardize a single method, and it was therefore decided:

- simply to supply a guide for the user enabling selection of the most appropriate apparatus and method for the case under study;
- to add an annex to all essential oil standards to give the mean flashpoint for information, stating wherever possible the determination method.

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Essential oils - General guidance on the determination of flashpoint

1 SCOPE

This Technical Report provides a list of methods that can be used for the determination of flashpoint, together with information on their respective fields of application.

It is intended for use by test laboratories and organizations responsible for monitoring all safety conditions during the transport and/or storage of essential oils.

2 REFERENCES

NOTE - This list is not exhaustive.

International standards

ISO 1516:1981	Paints, varnishes, petroleum and related products - Flash/no flash test - Closed cup equilibrium method
ISO 1523:1983	Paints, varnishes, petroleum and related products - Determination of flashpoint - Closed cup equilibrium method (not equivalent to NF T 30-050)
ISO 2719:1988	Petroleum products and lubricants - Determination of flashpoint - Pensky-Martens closed cup method
ISO 3679:1983	Paints, varnishes, petroleum and related products - Determination of flashpoint- Rapid equilibrium method
ISO 3680:1983	Paints, varnishes, petroleum and related products - Flash/no flash test - Rapid equilibrium method

European standard

EN 57:1984 Petroleum products - Determination of flashpoint - Abel-Pensky closed tester

British standards

BS EN 22719:1994	Methods of test for petroleum and its products - Determination of flash point - Pensky-Martens closed cup method
BS 2000-170:1992	Methods of test for petroleum and its products - Part 170: Determination of flash point - Abel closed cup method
IP 34/85(87)	Flashpoint by Pensky-Martens closed tester 1
IP 113/53	Flashpoint (closed) of cutback bitumen 1
IP 170/81	Flashpoint by the Abel apparatus ¹

French standards

NF M 07-011 July 1988	Produits pétroliers - Combustibles liquides - Point d'éclair en vase clos au moyen de l'appareil Abel. (Not equivalent to ISO)
NF M 07-019 December 1988	Produits pétroliers - Détermination du point d'éclair en vase clos supérieur à 50 °C au moyen de l'appareil Pensky- Martens. (Not equivalent to ISO)
NF M 07-036 June 1989	Détermination du point d'éclair - Vase clos Abel-Pensky. (Identical to EN 57)
NF T 30-050 December 1983	Peintures et vernis - Détermination du point d'éclair en vase clos sous agitation. (Not equivalent to ISO 1523: 1983)
NF T 60-103 December 1968	Produits pétroliers - Point d'éclair en vase clos des lubrifiants et huiles combustibles. (Not equivalent to ISO)
NF T 66-009 March 1969	Point d'éclair en vase clos des bitumes fluidifiés et des bitumes fluxés au moyen de l'appareil Abel. (Not equivalent to ISO)

¹ Available from the Institute of Petroleum

German standards

DIN 51755: 1974 Prüfung von Mineralölen und anderen brennbaren Flüssigkeiten - Bestimmung des Flammpunktes im geschlossenen Tiegel nach Abel-Pensky (Testing of mineral oils and other combustible liquids - Determination of flash point by the closed tester according to Abel-Pensky)

DIN 51758: 1985 Prüfung von Mineralölen und anderen brennbaren Flüssigkeiten – Bestimmung des Flammpunktes im geschlossenen Tiegel nach Pensky-Martens

DIN 53213-1: 1978 Prüfung von Anstrichstoffen und ähnlichen lösungsmittelhaltigen Erzeugnissen - Flammpunktprüfung im geschlossenen Tiegel, Best-immung des Flammpunktes (Testing of paints, varnishes and similar products containing solvents - Flash point test using closed cup

Standards of the American Society for Testing and Materials (ASTM)

ASTM D 56-87	Flashpoint by Tag closed tester
ASTM D 93-85(87)	Flashpoint by Pensky-Martens closed tester
ASTM D 3278-82	Flashpoint point of liquids by Setaflash closed cup apparatus

3 DEFINITION

For the purposes of this Technical Report, the following definition applies.

3.1 flashpoint: Temperature, corrected for a pressure of 101,325 kPa, at which the test liquid emits vapours in a closed vessel, under the conditions defined in the test method and in quantities such that a flammable mixture of vapour and air is produced in the test vessel.

4 GENERAL CONSIDERATIONS ON METHODS FOR MEASURING FLASHPOINT

4.1 General

As shown in annex A, methods for determining flashpoint in a closed cup have been standardized by several international organizations, such as the International Organization for Standardization (ISO), the European Committee for Standardization (CEN), the American Society for Testing and Materials (ASTM) or national bodies such as AFNOR, the British Standards Institution (BSI), the Petroleum Institute (IP), the Deutscher Normenausschuß (DIN), etc. for various products that can be regarded as related to essential oils.

Several of these overlap with regard to their field of application and are, in general, connected with the use of methods and measuring cups known as: "Abel", "Abel-Pensky", "Pensky", "Pensky-Martens", "Tag", "Sétaflash" and "Luchaire". It should be noted that methods using equipment of the Sétaflash type described in ISO 3679 and ISO 3680 have the obvious advantage of using a small amount of the product.

There are also international regulations given in ISO 1523 and ISO 1516 describing a general method connected with the restrictive use of the cups mentioned above.

In view of the large number of standards, the European Community has also decided to allow use of the choice between several methods regarded as acceptable. (See in annex B an extract from the European Community Directive referring explicitly to these methods.)

4.2 Equilibrium methods

These methods are described in ISO 1516, ISO 3680, ISO 1523 and ISO 3679.

4.2 Non-equilibrium methods

These methods are described in the following standards:

- BS 2000-170, NF M 07-011 and NF T 66-009 using the Abel apparatus,
- EN 57, DIN 51755 and NF M 07-036 using the Abel-Pensky apparatus,
- ASTM D 56 and ISO 2719 using the Tag apparatus,
- ISO 2719, DIN 51758, ASTM D 93-85(87), BS EN 22719 and NF M 07-019 using the Pensky-Martens apparatus.

5 DIFFERENCES BETWEEN THE TYPES OF APPARATUS

In practice, the following types of apparatus are available on the European market:

- a) Abel or Abel-Pensky apparatus: measurement without agitation, valid for temperatures below around 65 °C;
- b) Pensky-Martens apparatus: measurement with agitation, valid for temperatures greater than around 50 $^{\circ}\text{C}$;
- c) Sétaflash type apparatus: measurement without agitation, valid between ambient temperature and 110 °C;
- d) Luchaire type apparatus: measurement with agitation, valid between ambient temperature and 150 $^{\circ}\text{C}\,.$

6 TEST METHODS

6.1 Sample preparation

Refer to ISO 356:1977, Essential oils - Preparation of test sample.

6.2 Choice of test method

Choose one of the test methods given in annex B or clause 2 of this Technical Report and follow the instructions of the manufacturer of the apparatus.

7 TEST REPORT

The test report shall state the method used and the results obtained. It shall in addition mention all operating details not specified or regarded as optional, together with any events likely to have had an effect on the results.

The test report shall give all information necessary for the full identification of the sample.

ANNEX A

COMPARATIVE TABLE OF STANDARD METHODS FOR THE MEASUREMENT OF FLASHPOINT IN A CLOSED CUP

Standard and date	Method described of type of apparatus	Field of application or temperatures used	Comments or comparison
ISO 3679:1983	Equilibrium	< 110 °C	`"Sétaflash" type apparatus
ISO 1523: 1983	Equilibrium	5 °C to 110 °C	General method relating to different types of cup
ISO 3680: 1983	Equilibrium	< 110 °C	Flash/no flash, "Sétaflash" apparatus
ISO 1516: 1981	Equilibrium	5 °C to 65 °C	Flash/no flash, general method relating to different types of
ISO 2719: 1988	Pensky-Martens	12 °C to 110 °C and 107 °C to 379 °C	Suitable for petroleum products forming a surface film
NF T 60-103 December 1968	Non-equilibrium	for ambient temperature to 150 °C	Luchaire apparatus
NF T 66-009 March 1969	Abel	< 110 °C	With agitation, identical to IP 113-53
NF M 07-011 July 1988	Abel	< 55 °C	Without agitation, identical to IP 170/81
NF M 07-019 June 1989	Pensky-Martens	> 50 °C	Identical to IP 34/85(87), ASTM D 93-85(87) similar to DIN 51758
NF M 07-036 June 1989	Abel-Pensky	5 °C to 65 °C	Similar to DIN 51755 and identical to EN 57
IP 34/85(87)	Pensky-Martens	linked with viscosity	Identical to ASTM D 93-85(87)
IP 170/81	Abel	- 18 °C to + 71 °C	
IP 113/53	modified Abel	< 110 °C	With agitation
BS EN 22719:1994	Pensky-Martens	linked with viscosity	Identical to IP 34/85
DIN 51755: 1974	Abel-Pensky	5 °C to 65 °C	Comparable with NF M 07-036
DIN 51758: 1985	Pensky-Martens	65 °C to 200 °C	Comparable with NF M 07-019
ASTM D 56-87	Tag	linked with viscosity	
ASTM D 93-85(87)	Pensky-Martens	linked with viscosity	
ASTM D 3278-82	Sétaflash	0 °C to 110 °C	

ANNEX B

EXTRACT FROM THE COMMISSION DIRECTIVE OF 25 APRIL 1984 GIVING A SIXTH ADAPTATION TO THE TECHNICAL PROGRESS OF COUNCIL DIRECTIVE 67/548/EEC ON THE HARMONIZATION OF LEGISLATIVE, REGULATORY AND ADMINISTRATIVE PROVISIONS ON THE CLASSIFICATION, PACKAGING AND LABELLING OF DANGEROUS SUBSTANCES

[84/449/EEC - Annex A.9 (OJEC No. L 251/61 of 19 September 1984)]²

A.9 FLASHPOINT

1. METHOD

1.1 Introduction

It is useful, when carrying out this test, to have preliminary information on the flammability of the substance. The procedure is applicable to liquid substances, in their commercial form, the vapour from which can be ignited by ignition sources. The test methods described in this document are valid only for the flashpoint ranges specified in the individual methods.

1.2 Definitions and units

The flashpoint is the temperature, corrected for a pressure of $101,325~\mathrm{kPa}$, at which the test vapour in a closed test vessel, under the test conditions forms a flammable mixture of vapour and air in the test vessel.

Unit: °C

t = T - 273,15

(t in °C and T in K).

1.3 Reference substances

When examining new substances, reference substances should be used in all cases. They are used mainly for periodic calibration of the method and to permit a comparison of the results where another method is applied.

1.4 Principle of the method

The substance is placed in a test vessel which is heated gradually until the concentration of vapour in the air produces a mixture which can be ignited.

² References EN 11 and ASTM 8013 have been withdrawn.

1.5 Quality criteria

1.5.1Reproducibility

Reproducibility depends on the flashpoint range and test method used; maximum \pm 2 $^{\circ}\text{C}\,.$

1.5.2Sensitivity

Sensitivity depends on the test method used.

1.5.3Specificity

The specificity of certain test methods is limited to certain flashpoint ranges and depends on the data relating to the substance (e.g. high viscosity).

1.6 Description of the method

1.6.1Preparation

A sample of the test substance is placed in a test apparatus as described in 1.6.3.1 and/or 1.6.3.2.

1.6.2Test conditions

The apparatus shall preferably be installed away from draughts.

1.6.3Test procedure

1.6.3.1 Equilibrium method

See ISO 1516, ISO 3680, ISO 1523 and ISO 3679.

1.6.3.2 Non-equilibrium method

Abel apparatus:

See BS 2000 Part 170, NF M 07-011 and NF T 66-009.

Abel-Pensky apparatus:

See (EN 57), DIN 51755 Part 1 (for temperatures between 5 $^{\circ}$ C and 65 $^{\circ}$ C), DIN 51755 Part 2 (for temperatures less than 5 $^{\circ}$ C) and NF M 07-036.

Tag apparatus:

See ASTM D 56 and ISO 2719.

Pensky-Martens apparatus:

See ISO 2719, (EN 11), DIN 51758, ASTM 8013, ASTM D 93, BS 200-34 and NF M 07-019.

Comments:

Where the flashpoint determined by a method based on non-equilibrium (see 1.6.3.2) has the following values: 0 \pm 2 °C, 21 \pm 2 °C, 55 \pm 2 °C, it should be confirmed by a method based on equilibrium using the same apparatus.

Only methods capable of giving the temperature of the flashpoint may be used for notification.

For determining the flashpoint of viscous liquids (paints, gum, etc.) containing solvents, only apparatus and test methods for determining the flashpoint of viscous liquids shall be used. See ISO 3679, ISO 3680, ISO 1523 and DIN 53213 Part 1.

2. DATA

3. TEST REPORT

The test report shall, where possible, contain the following information:

- precise description of the substance (identification and impurities),
- description of the method used, together with all possible variants,
- the results and any information or comments which might be of use in interpreting the results.

4. REFERENCES

None

PD ISO/TR 11018:2001

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