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Preskusne metode za karakterizacijo trdnih matriksov z vidika okolja - Vodilo za preskušanje plamenišča

Test methods for environmental characterization of solid matrices - Guide to flash point testing

Charakterisierung von Abfällen - Anleitung zur Prüfung des Flammpunkts

Caractérisation des déchets - Lignes directrices pour la détermination du point d'éclair

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Test methods for environmental characterization of solid matrices - Guide to flash point testing

Caractérisation des déchets - Lignes directrices pour la détermination du point d'éclair

Prüfverfahren für die umweltbezogene Charakterisierung fester Matrices - Anleitung zur Prüfung des Flammpunkts

This Technical Report was approved by CEN on 19 November 2018. It has been drawn up by the Technical Committee CEN/TC 444.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
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European foreword

This document (CEN/TR 17309:2019) has been prepared by Technical Committee CEN/TC 444 "Test methods for environmental characterization of solid matrices", the secretariat of which is held by NEN.

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Introduction

Flash point values are used in transporting, storage, handling and safety regulations, as a classification property to define “flammable” and “combustible” materials. Precise definition of the classes is given in each particular regulation.

A flash point value can indicate the presence of highly volatile material(s) in a relatively non-volatile or non-flammable material and flash point testing can be a preliminary step to other investigations into the composition of unknown materials. For products material safety data sheets provide further information also for flash point, but e. g. material safety data sheets for waste do not exist.

It is not appropriate for flash point determinations to be carried out on potentially unstable, decomposable, or explosive materials, unless it has been previously established that heating the specified quantity of such material in contact with the metallic components of the flash point apparatus, within the temperature range required for the method, does not induce decomposition, explosion or other adverse effects.

Flash point values are not a constant physical-chemical property of material tested. They are a function of the apparatus design, the condition of apparatus used, and no general valid correlation can be guaranteed between results obtained by different test methods or with test apparatus different from that specified.

1 Scope

The flash point test can be summarised as a procedure where a test portion is introduced into a temperature controlled test cup and an ignition source is applied to the vapours produced by the test portion to determine if the vapour / air mixture is flammable or at what temperature the vapour / air mixture is flammable.

This document is not intended to be a comprehensive manual on flash point tests and the interpretation of test results, however it covers the key aspects on these subjects.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— IEC Electropedia: available at http://www.electropedia.org/

— ISO Online browsing platform: available at http://www.iso.org/obp

4 Outline

There are many, slightly different, definitions of flash point, however the following definition is widely used in standard test methods:

The flash point is the 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 test conditions.

It is important to realise that the value of the flash point is not a physical constant but it is the result of a flash point test and is dependent on the apparatus and procedure used. This fact is so important that a general statement similar to the following will be incorporated into all the main flash point methods:

Flash point values are not a constant physical-chemical property of materials tested. They are a function of the apparatus design, the condition of the apparatus used, and the operational procedure carried out. Flash point can therefore only be defined in terms of a standard test method, and no general valid correlation can be guaranteed between results obtained by different test methods or with test apparatus different from that specified.

Due to the importance of flash point test results for both safety and regulatory purposes, the test method identification should always be included with the test result.

In general specific products specifications indicate which standard test method should be employed.

5 Brief history

The discovery of petroleum and the increased use of flammable distillates in the 19th century, for lighting and heating in place of animal and vegetable oils, led to a large number of explosions and other fire related accidents.

Legislation, such as the UK Petroleum Act in 1862 and the German Petroleum Regulations in 1882, quickly spread around the world and led to the development of many types of test instruments. The following list shows the dates when the major surviving instruments were in a form probably recognisable today:

— 1870 – 1880 Abel closed cup, Pensky-Martens closed cup

— 1910 – 1920 Tag closed cup, Cleveland open cup

6 Flash point, and sustained combustion and burning

The flash point is essentially the lowest temperature of the liquid or semi-solid at which vapours from a test portion combine with air to give a flammable mixture and 'flash' when an ignition source is applied.

Sustained combustion and burning tests are usually carried out with the test portion at a fixed temperature and tests whether vapour combustion and burning commences when an ignition source is applied and thereafter is continuous and where the heat produced is self sustaining and supplies enough vapours to combine with air and burn even after the removal of the ignition source.

7 The need of flash point tests

The fundamental reason for the requirement of flash point measurements is to assess the safety hazard of a liquid or semi-solid with regard to its flammability and then classify the liquid into a group. The lower the flash point temperature the greater the risk. This classification is then used to warn of a risk and to enable the correct precautions to be taken when using, storing or transporting the liquid.

Specifications quote flash point values for quality control purposes as well as for controlling the flammability risk.

A change in flash point may indicate the presence of potentially dangerous volatile contaminants or the adulteration of one product by another.

8 Selection of flash point method

8.1 First considerations

Firstly, if a flash point method has been specified in a product specification or regulation, then that method should be the first choice. If a number of alternative methods are specified then the choice will be influenced by availability and other factors such as sample size requirements, speed of testing or precision. In certain circumstances the choice of the stated referee method may be of special importance. Annex A gives an overview of the most common methods and their use in specifications and regulations.

When testing specifically for contamination or contaminants, certain test methods and procedures are more appropriate than others. In general an equilibrium test method is recommended for testing samples that may contain traces of volatile contaminants.

When selecting a flash point method for incorporation into a product specification or regulation, it is important that the product type is included in the scope of the test method and that the temperature range of the product is covered by the test method. If the product is not included in the scope then the test may be unsuitable for the product or the quoted precision does not apply. Where the scope of a test method is general or not suitable it is recommended to contact an appropriate standardization body for advice.

When testing chemicals, mineral products or corrosive materials it is recommended to check that the test cup material is suitable and will not produce flammable gases or be damaged by any possible chemical reaction.

NOTE For example alkaline liquids or samples with aluminium cause problems.

8.2 Open or closed cup

There are two general classes of flash point tests - open cup and closed cup:

The open cup was initially developed to assess the potential hazards of liquid spillage. In this test a test portion is introduced into a cup that is open at the top. An ignition source is passed horizontally over the surface of the liquid, while the cup and liquid are being heated, to test if the vapours "flash". If the test is repeated at increasing test portion temperatures a point may be reached when the test portion continues to burn without further application of the ignition source, this is the fire point. The precision of open cup tests is somewhat poorer than closed cup tests as the vapours produced by heating the test portion are free to escape to the atmosphere and are more affected by local conditions in the laboratory. When open cup tests are made at temperatures above ambient the result is usually higher than a result from a closed cup test due to the reduced concentration of vapours.

The closed cup test contains any vapours produced and essentially simulates the situation where a potential source of ignition is accidentally introduced into a container. In this test a test portion is introduced into a cup and a close fitting lid is fitted to the top of the cup. The cup and test portion is heated and apertures are then opened in the lid to allow air into the cup and the ignition source to be dipped into the vapours to test for a flash.

The closed cup test predominates in specifications and regulations due to its better precision and ability to detect contaminants.

8.3 Non-equilibrium, equilibrium and rapid equilibrium tests

These three types of tests and associated instruments are characterised by the level of temperature stabilisation of the test portion and resultant vapours, and by the test portion size and test time.

Test methods such as Pensky-Martens, Tag, Abel and Cleveland are referred to as non-equilibrium tests as the test temperature of the test portion is increased during the test and the temperature of the vapours is not the same (not in equilibrium) as the test portion temperature when the ignition source is dipped at regular intervals into the cup. This type of test has the advantage that it produces a definitive flash point result. Under normal circumstances the increasing temperature is not a problem, but when volatile contaminants or components are present the short time between each dip of the ignition source, combined with the rate of temperature increase, does not allow enough time for flammable vapours to evolve and this may cause unreliable results. For this reason non-equilibrium tests with lower rates of heating usually perform better than those using higher rates of heating, when volatile contaminants or components are present in the test portion.

Equilibrium tests are preferred for liquids and semi-solids containing volatile components or contaminants and for confirmatory purposes in regulations as the sample temperature is constant or is increased at a very slow rate. This allows enough time for vapours to build up and for the vapours to be in equilibrium with the test portion before the ignition source is dipped into the cup. The ignition source is dipped in the cup at different test portion temperatures thus resulting in a measurement of a flash point, or the ignition source is dipped only once to carry out a flash no flash test to check conformance against specifications and flammability criteria. These equilibrium tests use any type of closed cup in a liquid bath and limits the difference of temperature between the test portion and the liquid bath. The liquid bath is specified because it gives a very even temperature distribution on the outside of the test cup thus ensuring that hot spots are not present on the cup surface that could cause the localised increase of flammable vapours and thus a low flash point. Unfortunately these procedures take a long time to complete.

Rapid equilibrium (small scale) tests are not primarily aimed to give the actual flash point of a test portion. The test is a flash no flash test to determine if the test portion's vapours flash at the test temperature. This is useful for checking conformance against specifications and flammability criteria. The test cup is heated to the test temperature, a small test portion is introduced into the cup, and when the test portion is deemed to be at the test temperature, the ignition source is used to test for a 'flash'.

Actual flash point temperature is determined by repeating the flash no flash test at different temperatures with a new test portion. The constant temperature of the test cup ensures that the test portion cannot be overheated and that there is a reasonable time for vapours to build up before the ignition source is applied.

8.4 Flash point automation

For a manual flash point test the operator is in control throughout the test and ensures that the temperature, stirring and ignition requirements are met throughout the test and determines when and if a flash has occurred. Some semi-auto instruments may assist the operator in detecting a flash or controlling the temperature, but the operator is in control. This is why manual tests are the referee in cases of dispute.

Automated flash point testers conform to all the specified requirements of the manual test method such as dimensions, heating rate and flash detection, however the electronics, software and mechanics mimic the manual operations. This may significantly reduce operator time but this does have the disadvantage that more frequent validation of tester operation is required as the instrument operates mainly unattended and is more complex.

Automatic flash point testers are not based on a manual test and often only key dimensions and parameters are defined in a test method written just for this instrument type. A unique type of test may be advantageous to the user but the complexity of the tester makes it difficult for conformance to the test method to be measured. More frequent validation of the tester parameters and operation is required.

Some automated and automatic instruments are available with carousels that allow a number of tests to be carried out unattended. This is particularly advantageous where large numbers of samples are tested. However, accurate and reliable measurements may be compromised if the sample temperature does not meet the recommendations stated in the test method. This is especially relevant to samples that are volatile or contain volatile contaminants.

In general automated instruments are accepted in test methods provided that the instrument shows conformation to the method requirements, including precision.

8.5 Correlation between methods

It is well known that open cup tests usually give higher flash point results than closed cup tests for test temperatures above ambient. Some specifications list equivalent flash point methods and sometimes relative bias information for specific products. However flash point methods employ different apparatus, heating and stirring rates, procedures and sample handling which have an effect on relative biases, especially when the liquid is volatile, or volatile contaminants or components are present. It is therefore not possible and not correct to claim correlation or a fixed relative bias between different test methods for all test samples.

8.6 Valid temperature ranges

Flash point instruments often have a wider temperature range than the temperatures covered by the precision of the test method. Temperatures outside those covered by the precision may result in different precision or give unpredictable results. The temperature ranges covered by the precision are shown in the test method; otherwise it may be necessary to consult the relevant standardisation body for advice.

Test method procedures include information on the required temperature of the test portion when the flash point test commences and usually defines a temperature band over which a result is valid. It is important to follow the specified procedure as failure to do so may result in an incorrect measurement.