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EXPERIMENT (6)

flash and fire point

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Flash- and fire point measurement

The flash point test can be summarized as a procedure in which a test specimen is introduced into a temperature-controlled test cup and an ignition source is applied to the vapors produced by the test specimen. The aim of the test is to determine whether the vapor/air mixture is flammable or at what temperature the vapor/air mixture is flammable.

Definition of flash- and fire point

There are many, slightly different, definitions of flash point. However, the following definition is widely used in standard test methods: The lowest temperature of the test specimen, corrected to a barometric pressure of 101.3 kPa, at which the application of an ignition source causes the vapor of the test specimen to ignite momentarily and the flame to propagate across the surface of the liquid under the specified conditions of test. It is important to realize that the value of the flash point is not a physical constant but is the result of a flash point test and is dependent on the apparatus and procedure used.

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.

Fire point may be considered as the lowest temperature of the liquid at which vapor combustion and burning commences. A fire point happens when an ignition source is applied and the heat produced is self-sustaining, as it supplies enough vapors to combine with air and burn even after the removal of the ignition source. The flammability of a substance is therefore characterized by:

- The conditions under which a substance can be ignited and those under which it continues to burn – known respectively as the flash point and fire point.
- The concentration range over which the vapor/oxidant mixture is flammable, i.e. the upper and lower flammability limits. These limits vary depending on the oxidant, pressure and the minimum oxygen concentration required for the combustion reaction.

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.

Purpose 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 can indicate the presence of potentially dangerous volatile contaminants or the adulteration of one product by another. The classification of chemicals including petroleum products helps to identify the hazards of a substance or preparation. It is important that the classification of the hazard is correct, otherwise the label, safety data sheet and the packaging may be incorrectly assigned. This can have potentially serious consequences for the end user. There are many health and safety legislations which require such classification:

- CPSC: United States Consumer Products Safety Commission
- DOT: United States Department of Transportation
- EPA: United States Environmental Protection Agency
- NFPA: National Fire Protection Agency
- OSHA: United States Occupational Safety and Health Administration
- UN: Class 3 Dangerous goods
- ADR: The European agreement concerning the international carriage of dangerous goods by road
- ECHA: European Chemicals Agency, guidance to regulation on classification, labelling and packaging (CLP) of substances and mixtures

The presence of contaminations can have a significant effect on the flash point, particularly if the contaminant is relatively more volatile. For example, pure ethylene glycol has a flash point of 111 °C. However, the flash point is reduced to 29 °C when acetaldehyde at a level of only 2 % is present. Another common example would be ethanol; water addition has a significant effect on flammability.

% Ethanol in water	Approx. flash point in °C
100	13.0
90	14.5
75	20.5
50	23.5
25	31.5
10	47.5
5	61.5

For solvents which are immiscible in water, the flash point will be largely unaffected by the addition of water.

Flash point methods

If a flash point method has been specified in a product specification or regulation, then that method should be the first choice. 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 may not apply. 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. Many flash point testers are available with stainless steel test cups.

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

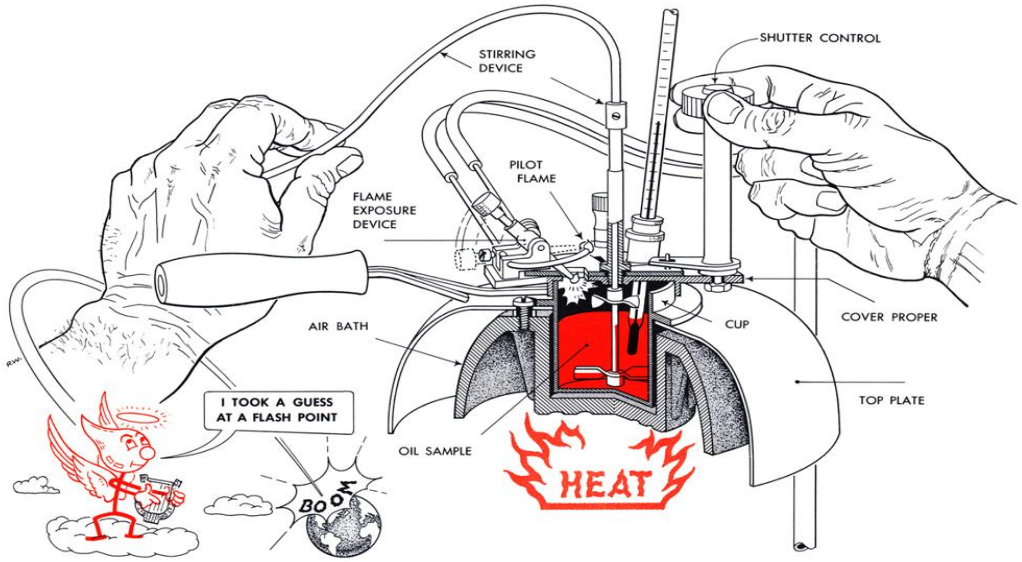
The **open-cup** test CLA 5 was initially developed to assess the potential hazards of liquid spillage. An ignition source is passed horizontally over the surface of the liquid, while the cup and liquid are being heated, to test if the vapors 'flash'. If the test is repeated at increasing test specimen temperatures a point may be reached at which the specimen 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 vapors produced by heating the test specimen are free to escape to the atmosphere and are more affected by local conditions in the laboratory. When open-cup tests are carried out at temperatures above ambient the result is usually higher than a result from a closed-cup test due to the reduced concentration of vapors.

The **closed-cup** test PMA 5 contains any vapors produced and essentially simulates the situation in which a potential source of ignition is accidentally introduced into a container. In this test a test specimen is introduced into a cup and a close-fitting lid is fitted to the top of the cup. The cup and test specimen is heated. Subsequently, apertures are opened in the lid to allow air into the cup and the ignition source to be dipped into the vapors to test for a flash.

The closed cup is mostly used in product specifications and regulations due to its better precision. The following table shows the comparative flash points measured in open and closed cup apparatus for some common pure liquids.

Comparison of flash points measured in open- and closed-cup apparatus

Liquid	Flash point [°C]	
	Closed Cup	Open Cup
Acetic acid	40	43
Acetone	-17	-9
n-Butanol	29	44
Methanol	10	16
Toluene	4	7



Flash point automation

In a manual flash point test the operator is in control throughout the test, ensures that the temperature, stirring and ignition requirements are at all times and determines when and if a flash has occurred. This is why manual tests are the reference in cases of dispute.

Automatic 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 can significantly reduce operator time and involvement, greatly increasing laboratory efficiency. In general automatic instruments are accepted in test methods provided that the instrument can be shown to conform to the method requirements

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. However, flash point methods employ different apparatus, heating and stirring rates, procedures and sample handling. All of these have an effect on relative biases, especially when the liquid is volatile or if volatile components are present. Therefore, it is not possible to claim correlation or a fixed relative bias between different test methods for all test samples.

Apparatus

- Pensky-Martens closed tester
- Thermometer

Pensky-Martens closed cup tester contains testing cup, lid, stirrer device, shutter, and flame exposure device. Thermometer of specified range generally 0°C to 350°C with sensitivity of 0.1°C should be used

Pensky-Martens Apparatus

Test Procedure

1. Heat the bitumen to above its softening point generally 75°C to 100°C and stir this softened bitumen thoroughly to remove air bubbles.
2. Fill the cup with softened bitumen up to the filling mark provided on the cup. Now place the lid and close the cup.
3. Other accessories like thermometer and flame exposure are suitably fixed in their respective positions. Now lit up the flame and set the size of flame to 4mm in diameter.

4. The bitumen getting heated and preferred rate of heating should be 5°C to 6°C per minute.

5.Stirring of sample should be simultaneously done along with heating using stirrer device

7. The rate of stirring should be approximately 60 revolutions per minute.

8. Observe the thermometer carefully and when the temperature is 17°C below the actual flash point (175°C) lit up the test flame.

9. The test flame size should be of 4mm diameter and carry it close to the heating sample.

10. Apply the test flame for every 1^{O}C rise from this point and remember during application of test flame the stirring should be stopped.

11. When the sample catches the flame and forms Flash, note town the temperature at that point which is Flash point of the bitumen.

12. Heat the sample further with the same previous rate and apply the test flame for every 2^{O}C rise when the material catches the fire and burns at least for 5 seconds, note the temperature at this point which is the fire point of the bitumen.

13. Repeat the experiment for 2 more times and the average of the three readings should be taken as Flash point and Fire point of the given sample.

Results

- Flash point of the bitumen = _____ ^{o}C
- Fire point of the bitumen = _____ ^{o}C

Recommended Values

For any type of Bitumen grade

Minimum Flash point value should be = $175^{o}C$
Minimum Fire point value should be = $175^{o}C + 5^{o}C$.