



Reference Manual Book 5: Laboratory Policies and Procedures
Part B: Test Method Section

Method C22: Determination of the Flash Point of Fluids by the Closed Cup Test Method

1 SCOPE

- 1.1 This method describes a general procedure for the determination of the flash point of consumer and workplace products.
- 1.2 This method is applicable:
 - 1.2.1 To evaluate a consumer product to the Consumer Chemicals and Containers Regulations (CCCR), 2001, sections 49, 50, and 51 of Part 3, SOR/2001-269.
 - 1.2.2 To evaluate a workplace product to the Workplace Hazardous Materials Information System (WHMIS), Hazardous Products Regulations (HPR), section 7.6.1, SOR/2015-17.

2 SAFETY

- 2.1 Canopy with on/off interceptor.
- 2.2 Use grounding wire when transferring solvents from metallic containers.

3 APPLICABLE DOCUMENTS

- 3.1 SOPC-47, Standard Operating Procedure for the Tag 4 Flash Point Tester
- 3.2 LBC-47, TAG4 Closed Cup Flash Point Tester
- 3.3 SOPC-48, Standard Operating Procedure for the Pensky-Marten Automatic Flash Point Tester
- 3.4 LBC-48, Pensky-Marten Automatic Flash Point Tester
- 3.5 Method C27: Automatic Determination of the Kinematic Viscosity of Transparent and Opaque Liquids by miniAV Automatic Viscometer
- 3.6 Method C00.13, "Method for the Verification of Barometric Pressure" as per Book 5 of the Product Safety Laboratory.
- 3.7 ASTM D 56-05, Standard Test Method for Flash Point by Tag Closed Cup Tester.
- 3.8 ASTM D 93-02a (CCCR, HPR) Standard Test Methods for Flash Point by Pensky-Martens Closed Cup Tester.

4 DEFINITIONS

- 4.1 Flash point: The lowest temperature corrected to a barometric pressure of 101.3 kPa (760 mm Hg), at which application of an ignition source causes the vapors of a specimen of the sample to ignite under specified conditions of test (*ASTM D 56-05, D 93-02a*).



Reference Manual Book 5: Laboratory Policies and Procedures

Part B: Test Method Section

Method C22: Determination of the Flash Point of Fluids by the Closed Cup Test Method

5 REAGENTS AND APPARATUS

5.1 Reagents

- 5.1.1 n-decane (C₁₀H₂₂), 99+% or equivalent¹, CAS 124-18-5, Acros
Secondary working standard used for a flash point around 51°C
- 5.1.2 n-undecane (C₁₁H₂₄), 99% or equivalent¹, CAS 1120-21-4, Acros
Secondary working standard used for a flash point around 67°C
- 5.1.3 n-octane (C₈H₁₈), 99% or equivalent¹, CAS 111-65-9, Acros
Secondary working standard used for a flash point around 13-16°C
- 5.1.4 Acetone (C₃H₆O), HPLC grade or equivalent¹, CAS 67-64-1, Fisher Scientific
Secondary working standard used for a flash point around -18°C
- 5.1.5 Acetone (C₃H₆O) /Methanol (CH₄O), reagent grade or other suitable solvent¹, Fisher Scientific
Used for cleaning the test cup, temperature probe or thermometer, and lid
- 5.1.6 Certified reference material (CRM) TCC D56, ASTM-P-133-04, AccuStandard or equivalent¹
Nominal flash point 67°C
- 5.1.7 CRM PMCC D93, ASTM-P-132-01, AccuStandard or equivalent¹
Nominal flash point 60°C
- 5.1.8 CRM PMCC D93, ASTM-P-132-02, AccuStandard or equivalent¹
Nominal flash point 93°C

5.2 Labware

- 5.2.1 Thermometer having a precision of 1°C
- 5.2.2 Tester cups and lids
- 5.2.3 Temperature probe
- 5.2.4 Low-lint, non-abrasive cloth

5.3 Equipment

- 5.3.1 Fridge at 4°C, Freezer at -28°C or equivalent¹

5.4 Analytical Instruments

- 5.4.1 Tag 4 Closed Cup Flash Point Tester (automatic) or equivalent¹
- 5.4.2 Pensky-Martens Closed Cup Flash Point Tester or equivalent¹

¹ Where applicable, suitable equivalent reagents, materials, or equipment may be used.



Reference Manual Book 5: Laboratory Policies and Procedures

Part B: Test Method Section

Method C22: Determination of the Flash Point of Fluids by the Closed Cup Test Method

6 EXPERIMENTAL PROCEDURE

6.1 Flash point measurements are made under strictly controlled conditions and prescribed techniques. The specific details of operation vary significantly for the different types of Flash Point testers prescribed for a particular sample. Depending on the physical properties and characteristics of the test sample², the procedure shall be conducted according to the following criteria; unless a specific test is requested by the client:

ASTM D56: For liquids that have a viscosity below 5.5 mm²/s at 40°C and a flash point below 93°C.

ASTM D93: For liquids that have a viscosity of 5.5 mm²/s or more at 40°C, a flash point of 93°C or higher, a tendency to form a surface film under test condition; or containing suspended solids.

6.2 Precaution

6.2.1 Turn on the exhaust to ventilate the flash point tester while heating the solution and following analysis. Analysis for flash point should be conducted in a draft-free environment. Conduct the sampling and pipetting of test samples and oils in the fume hood.

6.2.2 All samples and solvents must be properly stored and disposed of according to the applicable procedures.

6.3 Preparation of Solutions

6.3.1 N/A

6.4 Preparation of Standards

6.4.1 N/A

6.5 Subsampling

6.5.1 When the quantity of sample is sufficient and when the flash point is unknown, a test portion should be used to determine the approximate flash point for future replicates.

6.5.2 Under no circumstances should a repeat test be conducted on the same sample portion. A fresh portion of sample must always be used for each determination.

6.6 Sample Preparation

² Determine the viscosity following method C27, if required.



Reference Manual Book 5: Laboratory Policies and Procedures
Part B: Test Method Section

Method C22: Determination of the Flash Point of Fluids by the Closed Cup Test Method

- 6.6.1 Clean the sample cup, the temperature probe or thermometer, and the lid with acetone and/or methanol depending on the sample type and dry them with a low-lint, non-abrasive cloth.
 - 6.6.2 Select the appropriate tester having a scope of operation covering the estimated flash point of the test sample.
 - 6.6.3 Mix the sample thoroughly and ground the container, if required. Transfer the sample into the cup in the manner dictated by the design of the apparatus. The sample should be at least 10°C below the expected flash point using the fridge or the freezer.
 - 6.6.4 Turn on the external cooling system if the instrument requires it.
 - 6.6.5 Place the sample cup in the instrument and enter the expected flash point³.
 - 6.6.6 Record the flash point temperature.
 - 6.6.7 If the instrument does not automatically record the ambient barometric pressure, record it at the time of the test.
 - 6.6.8 After the sample has been tested, dispose of it with caution. Clean the sample cup, the temperature probe or thermometer, and the lid with acetone and/or methanol depending on the sample type and dry them with a low-lint, non-abrasive cloth.
- 6.7 Blank Preparation
- 6.7.1 N/A
- 6.8 Control Preparation
- 6.8.1 The following table includes the expected flash points for secondary working standards and certified reference materials (CRMs)⁴.

³ Do not confuse the true flashpoint with the bluish halo which sometimes surrounds the test flame during applications preceding the one that causes the actual flash.

⁴ The expected flash point for CRMs should be taken from the certificate of analysis for each lot tested.



Reference Manual Book 5: Laboratory Policies and Procedures

Part B: Test Method Section

Method C22: Determination of the Flash Point of Fluids by the Closed Cup Test Method

Control Fluid	Flash Point Experimental Values (°C)	
	D93	D56
	Pensky-Martens	Tag 4
n-decane	53.3	52.6
n-undecane	69.2	68.6
Acetone	---	-18.0
TCC D56, ASTM-P-133-04	---	67 (nominal)
PMCC D93, ASTM-P-132-01	65.0 (nominal)	---
PMCC D93, ASTM-P-132-02	---	93 (nominal)

- 6.8.2 For each sample tested, select a secondary working standard from the table above that has a flash point nearest to the expected flash point of the sample. For samples tested to evaluate a workplace product with a high expected flash point, PMCC D93, ASTM-P-132-02 may be used as the control fluid.
- 6.8.3 Follow procedure 6.6.
- 6.8.4 Once a year, run a certified reference material (PMCC D93, ASTM-P-132-01; PMCC D93, ASTM-P-132-02; or TCC D56 ASTM-P-133-04) to ensure the validity of the instrument.

7 CALIBRATION

7.1 N/A

8 DETERMINATION

- 8.1 When the quantity of sample is sufficient and when the flash point is unknown, determine the flash point of a test portion to be used as the expected flash point for future replicates.
- 8.2 Determine the flash point of the secondary working standards, as prepared in section 6.8, before and after the analysis of the samples.
- 8.3 Determine the flash point of the samples, in triplicate, as prepared in section 6.6, using the flash point tester according to the instrument manufacturer's instructions.
- 8.4 Determine the flash point of the CRM, as prepared in section 6.8, on each flash point tester annually.



Reference Manual Book 5: Laboratory Policies and Procedures

Part B: Test Method Section

Method C22: Determination of the Flash Point of Fluids by the Closed Cup Test Method

9 CALCULATIONS AND REPORTING

9.1 For instruments that do not automatically record the barometric pressure or provide the corrected flash point, complete the following calculation:

9.1.1 When the barometric pressure at the time of the test differs from 101.3 kPa (760 mm Hg), correct the observed flash point according to the following equations:

$$\text{Flash Point (corrected), } ^\circ\text{C} = C + 0.25 (101.3 - P) \text{ or,}$$

$$\text{Flash Point (corrected), } ^\circ\text{C} = C + 0.033 (760 - p)$$

Where:

C = Observed flash point ($^\circ\text{C}$)

P = Ambient barometric pressure (kPa)

p = Ambient barometric pressure (mm Hg)

9.2 If applicable, add the value of the bias to the corrected flash point (calculated in section 9.1.1 or provided automatically by the instrument) following the equation:

$$\text{Flash Point (corrected with bias), } ^\circ\text{C}_B = ^\circ\text{C} + (\text{Bias})$$

Where:

$^\circ\text{C}$ = Corrected flash point

(Bias) = Found in section 11.3

9.3 Where the quantity of a sample available for testing is sufficient and where practical, the result of analysis will be reported as the average of a minimum of three replicate determinations having a precision which meets or exceeds the specifications defined in section 11.1.

9.4 Where applicable, the average (\bar{x}) of replicate determinations plus or minus Student's t (for a confidence level of 0.975) times the standard deviation (s) of replicate determinations (s for $n > 2$) will be calculated, and the result of analysis reported in the following format

Sample no.	Specimen no.	Test Method	Flash Point ($^\circ\text{C}$)
S20xx-0xxxx	A1	ASTM DXX	$\bar{x} \pm \frac{t_{0.975[95\%]} \times s}{\sqrt{n}}$

Where:

$t_{0.975(95\%)}$ is the value for t, obtained from Appendix A, Figure 4 in Method C00

s = standard deviation for the sample

n = number of values in the data set



Reference Manual Book 5: Laboratory Policies and Procedures

Part B: Test Method Section

Method C22: Determination of the Flash Point of Fluids by the Closed Cup Test Method

- 9.5 When the flash point is below or exceeds the limitations of the instrument, the flash point may be recorded as less than or greater than the limitations of the instrument. For consumer products, the flash point may be recorded as greater than 60°C. For workplace products, the flash point may be recorded as greater than 93°C.

10 QUALITY CONTROL PROCEDURE

In order to ensure the proper operation of the analytical instrument and that the precision and accuracy of the analytical measurements meet the specifications of the method, the following quality control procedure shall be conducted concurrently with the analysis of the test sample.

- 10.1 The normal and correct operation of the analytical equipment shall be verified according to the following guidelines:
- 10.1.1 Record the flash point results of the non-certified control sample in the electronic control chart. Verify that the measurements are within the warning limits ($\pm 2s$) and do not exceed the control limits ($\pm 3s$). If the non-certified control sample results are outside of the control limits, try with the certified flash point standard. If the certified flash point standard is outside of the control limits, the entire analytical procedure shall be repeated. Do not use the instrument until the source of the problem is identified and the instrument is repaired.
 - 10.1.2 Record the flash point results of the certified flash point control in the electronic control chart (annually). Verify that the measurements are within the warning limits ($\pm 2s$) and do not exceed the control limits ($\pm 3s$). If the certified flashpoint control results are outside of the control limits, the entire analytical procedure shall be repeated. Do not use the instrument until the source of the problem is identified and the instrument is repaired.
 - 10.1.3 If the instrument is found in a state of disrepair or out of calibration, the analytical instrument shall immediately be repaired and/or re-calibrated to meet the prescribed operating conditions prior to proceeding with the analysis. Determine the flash point of a certified flash point control after the repair of the instrument.
- 10.2 If the repeatability of the result of a given sample ($t_{0.975(95\%)} * s \sqrt{n}$) for a confidence interval of 95 % is equal or less than the repeatability limit of the method, the test result is considered within the limitations of the method.

If the repeatability of the result of a given sample does not meet the repeatability limit of the method (section 11.1) the analyst must consult with the section head before proceeding. It may be necessary to repeat the test method in triplicate, where the quantity of sample is sufficient and where practical. Unless an error in the original test is documented or a result is determined to be an outlier, all replicates should be used to determine the mean and the repeatability of the results for a given sample for a confidence interval of 95 %.



Reference Manual Book 5: Laboratory Policies and Procedures

Part B: Test Method Section

Method C22: Determination of the Flash Point of Fluids by the Closed Cup Test Method

11 PRECISION AND BIAS

11.1 Repeatability

The repeatability of the method was determined by conducting seven or more successive analyses on the identified reference materials for the method, with the exception of the repeatability indicated for PMCC D93 ASTM-P-132-01, which was estimated based on three replicates. The repeatability limit at 95% confidence level is calculated as follows:

$$\text{Repeatability limit} = 1.960 \times \sqrt{2} \times \text{CV}$$

Where: CV = Coefficient of variation (%)

Control Fluid	ASTM Standard Test Method	
	D56 Tag 4	D93 Pensky-Martens
n-decane	---	4.8%
n-undecane	---	3.4%
Acetone	7.6%	---
PMCC D93 ASTM-P-132-01	2.2%	3.9%
PMCC D93 ASTM-P-132-02	3.7%	---

11.2 Reproducibility

The reproducibility of the method was determined by conducting independent analyses, by different analysts on different days, on the identified reference materials for the method. The reproducibility limit at 95% confidence level is calculated as follows:

$$\text{Reproducibility limit} = 1.960 \times \sqrt{2} \times \text{CV}$$

Control Fluid	ASTM Standard Test Method	
	D56 Tag 4	D93 Pensky-Martens
n-decane	6.8%	4.9%
n-undecane	5.0%	6.0%
Acetone	8.7%	---
PMCC D93 ASTM-P-132-01	---	3.5%

Note: The reproducibility for the PMCC D93 ASTM-P-132-02 will be presented in a revised copy of the method.

11.3 Bias

The bias of the test method obtained from the average of a set of test results of acetone minus the accepted reference value is 0.5°C.



Reference Manual Book 5: Laboratory Policies and Procedures
Part B: Test Method Section

Method C22: Determination of the Flash Point of Fluids by the Closed Cup Test Method

There is no bias observed for the analysis of the n-decane and the n-undecane

12 LIMITS OF DETECTION AND QUANTIFICATION

12.1 N/A

13 REFERENCES

- 13.1 A. Soucy, C. Watson "Update Method C-22 Determination of the Flashpoint of Fluids by the Closed Cup Test Method", (PSL 2000-0581).
- 13.2 M. Charette, C. Watson, Update Method C22, "Determination of the Flashpoint of Fluids by the Closed Cup Test Method", Project report # 2007-1026.
- 13.3 M. Charette, Update Method C22, "Determination of the Flashpoint by the Closed Cup Test Method", Project report # 2011-1324.
- 13.4 C. Gudgeon, Update Method C22, "Determination of the Flashpoint by the Closed Cup Test Method", Project report # 2015-2108.
- 13.5 M. Charette, Method Update: C22, Determination of the Flashpoint of Fluids by the Closed Cup Test Method, Project report # P2017-00017.
- 13.6 Griffiths, K., Method Updates: C13, Determination of the pH of Consumer Products in Aqueous Solutions; C22, Determination of the Flashpoint of Fluids by the Closed Cup Test Method; and C27, Automatic Determination of the Kinematic Viscosity of Transparent and Opaque Liquids for the WHMIS Survey 2018-2019, PSL Report P2018-00093.
- 13.7 Grenier, G., Method Update: C22 Determination of the Flash Point of Fluids by the Closed Cup Test Method, PSL Report # P2019-00064.
- 13.8 Grenier, G., Method Update: C22 Determination of the Flash Point of Fluids by the Closed Cup Test Method, PSL Report P2021-00005, March 2021.