38th Annual 2023 Environmental Measurement Symposium

Flashpoint Measurement by MCCCFP Method

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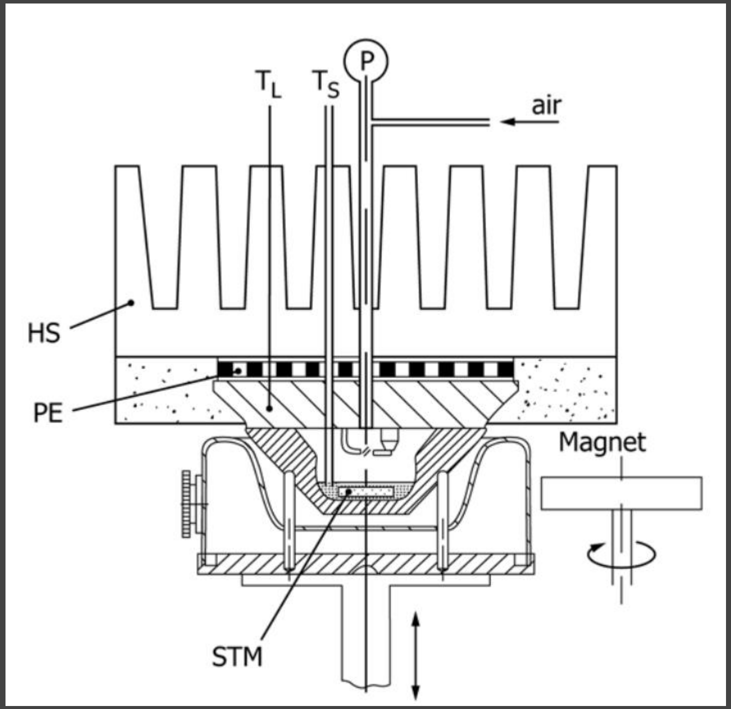
MCCCFP

- First Developed in 1998
- Original Methods for Petroleum Industry
- Innovative *Continuously C*losed *C*up *F*lash *P*oint (CCCFP)
- ASTM approved since year 2004 as D6450
- D6450, further developed for optimization of low level contamination, Modified *Continuously C*losed *C*up *F*lash *P*oint (MCCCFP)
- Lead to development of new method; D7094
- Today used in many different industries, one of the many applications is liquid waste testing



MCCCFP - Applications

ASTM D7094:

- Diesel
- Jet Fuel
- Kerosene
- Fuel Oil
- Lube Oil
- Gasoil

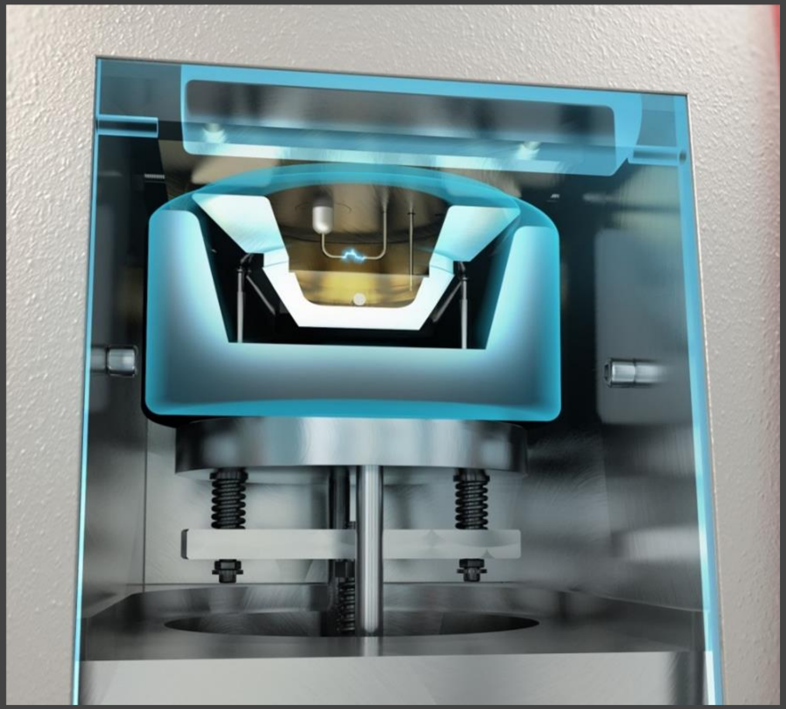
Various other samples:

- Chemicals
- Solvents
- Liquid Waste
- Flavors
- Fragrances
- Varnish
- Vegetable oil

MCCCFP – Measuring principle

<u>Continuously closed cup method:</u>

1. 1-2 mL of sample are filled into the sample cup
2. The sample cup is installed in the analyzer, lifted to heating plate and regulated to the starting temperature
3. A stable heating rate of 2.5 °C/min is adjusted
4. An ignition attempt (high voltage arc) is done every 1 °C
5. The pressure inside the sample cup is continuously monitored
6. As soon as flame occurs in the headspace of the cup the pressure peak during an ignition distinctively increases
    - A flashpoint is detected!

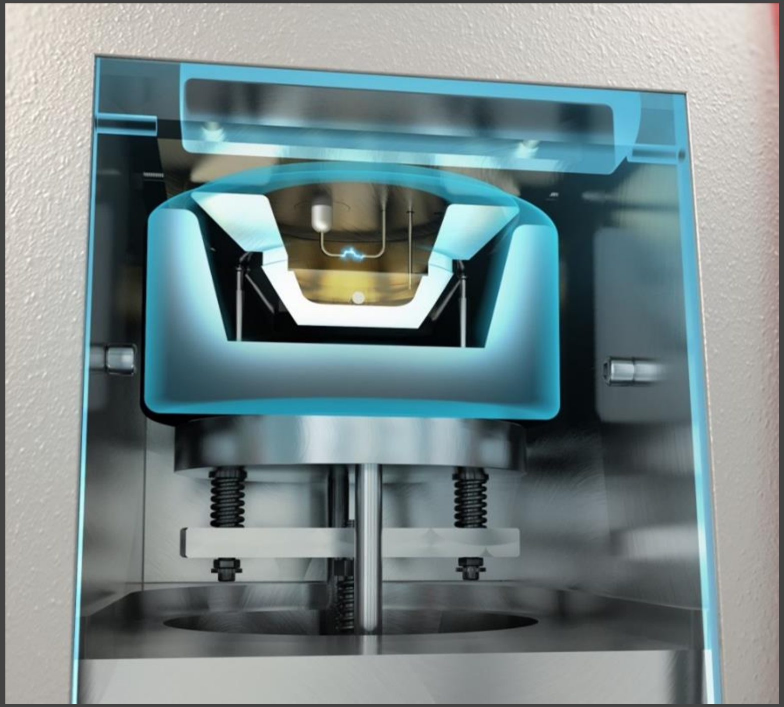


Established method ASTM D8175 (D93)

Pensky Martens Tester:

- Sample quantity: 75 mL
- Open flame or filament
- Danger of fire when measuring contaminated samples

MCCCFP Advantages - Safety

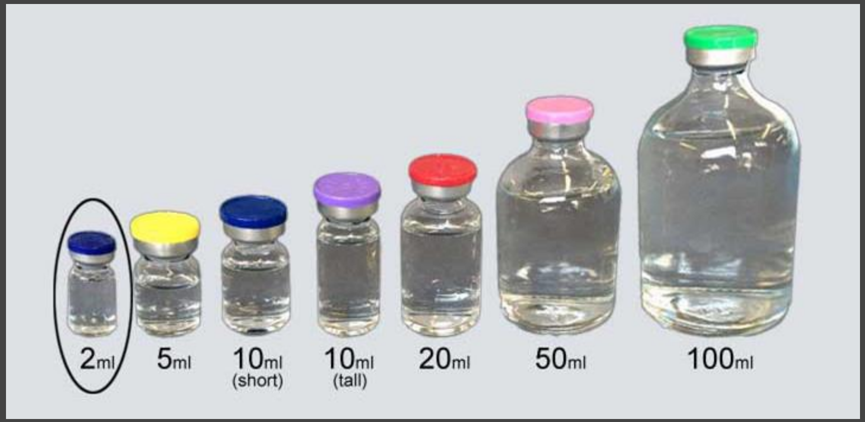


- Continuously closed
- No open flame
- Small sample quantities
- Intrinsically safe

No safety concern even for unknown samples!

MCCCFP Advantages – Testing Range

- Wide flashpoint testing range from -40°F to 790°F (-40°C to 420°C)
- Small sample quantities of 2mL
- Fast turnaround time



Test Samples used for D8174 & D8175

TABLE X4.1 Sample Reference, Corresponding Descriptions, and Number of Phases

Sample ID	Components and Their Volumetric Fractions	Phases
X50B50	50 % Xylenes, 50 % Butan-1-ol	1
B60D40	60 % Butan-1-ol, 40 % *n*-Decane	1
D70U30	70 % n-Decane, 30 % *n*-Undecane	1
A10H90	10 % Acetone, 90 % *n*-Heptane	1
Multi-Phase	50 % Diesel, 47.5 % Water, 2.5 % Acetone	2

Test Results of MCCCFP, D8174 and D8175

Sample	Flash Point	MCCCFP average	r	D8175 average	r	D8174 average	r
A10H90	-18,6	-18,5	1,9	no result		-21,4	2,34
A10H90	-18,6						
A10H90	-18,6						
A10H90	-19,6						
A10H90	-17,6						
A10H90	-19,6						
A10H90	-17,6						
A10H90	-18,6						
A10H90	-18,6						
A10H90	-17,6						
Multi-Phase aqueous layer	30,2	30,6	1,4	29,5	2,0	32,3	1,49
Multi-Phase aqueous layer	31,2						
Multi-Phase aqueous layer	30,2						
Multi-Phase aqueous layer	30,2						
Multi-Phase aqueous layer	30,2						
Multi-Phase aqueous layer	31,2						
Multi-Phase aqueous layer	31,2						
Multi-Phase aqueous layer	30,2						
Multi-Phase aqueous layer	30,2						
Multi-Phase aqueous layer	31,2						
Multi-Phase organic layer	33,4	33,9	2,2	34,1	2,0	31,1	2,21
Multi-Phase organic layer	32,4						
Multi-Phase organic layer	33,4						
Multi-Phase organic layer	34,4						
Multi-Phase organic layer	34,4						
Multi-Phase organic layer	33,4						
Multi-Phase organic layer	35,4						
Multi-Phase organic layer	34,4						
Multi-Phase organic layer	33,4						
Multi-Phase organic layer	34,4						

Test Results of MCCCFP, D8174 and D8175

Sample	Flash Point	MCCCFP average	r	D8175 average	r	D8174 average	r
X50B50	25,4	25,1	1,8	26	2,3	24,1	0,88
X50B50	25,4						
X50B50	25,4						
X50B50	24,4						
X50B50	25,4						
X50B50	24,4						
X50B50	24,4						
X50B50	24,4						
X50B50	25,4						
X50B50	26,4						
D70U30	53,3	53,9	1,4	54,7	1,3	52,8	0,97
D70U30	53,3						
D70U30	54,3						
D70U30	54,3						
D70U30	53,3						
D70U30	54,3						
D70U30	54,3						
D70U30	53,3						
D70U30	54,3						
D70U30	54,3						
B60D40	35,3	34,8	1,4	34,9	0,9	no result	
B60C40	35,3						
B60D41	34,3						
B60C41	34,3						
B60D42	35,3						
B60C42	35,3						
B60D43	35,3						
B60C43	34,3						
B60D44	34,3						
B60C44	34,3						

MCCCFP – Method Consensus Development

- ASTM approved since 1999 as D6450
- D7094 published in 2003 after successful ILS in 2001 with direct comparison to D93 using neat hydrocarbons like diesel, jet and lube oil + with gasoline contaminated hydrocarbons.
- Comparison to D93 done to show our MCCCFP can successful test difficult samples as well (such contaminated samples can be found in waste waters as well).
- 2018 the first European flash point method was developed based on D7094, namely IP620
- European work started in 2021 to develop new standard method based on D7094 as an EN ISO method.

MCCCFP – Method Consensus Development Cont'

- The new 2022 ILS and its resulting precision will be used together with the British method IP620 for the development.
- Currently draft of new EN ISO method is written which will be circulated in the ISO/TC28 and in WG9 flash point work group together with research report of ILS2022.
- In 2022, the largest ever flashpoint ILS was performed for MCCCFP using labs in North America and in Europe.
- 20 labs with 25 samples tested in duplicate were included in the ILS. More than 1000 test measurements were used to produce new precision for this advanced method

MCCCFP -

New Developments:

- New draft method circulated
- ASTM Committee D34; WK84158 - Standard Test Method for Finite Flash Point determination of Liquid Wastes by Modified Continuously Closed Cup (MCCCFP) Tester (Technical Contact: Andreas Schwarzmann)
- Ballot currently issued
- Results to be discussed during December, 2023 Meetings in Orlando, Florida
- Expecting method publication in 2023, early 2024
- as the safest flash point test method for waste samples

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Any Questions?

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