# eraflash - Efficiency through maximum safety and measuring speed

## Introduction – What is a flash point?

The flash point is a key property not only for typical samples of the petroleum industry such as diesel or jet fuel. It is also measured for every-day products and intermediates in the flavor & fragrance and beverage industries. The main reason for determining the flash point of a material is safety concerns: the lower a flash point the easier it is to ignite the substance, hence the higher the potential hazardous risks.

By definition, the flash point is *the lowest temperature at which vapors of a material will ignite, when given an external ignition source*. This definition describes the basic principle how a flash point is measured, see Figure 1 and a general description below:

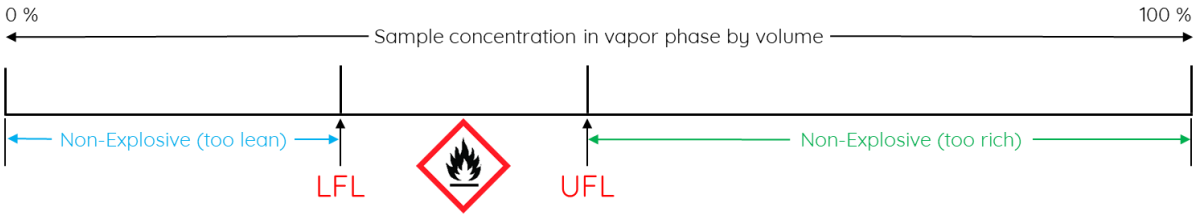


Figure 1: Flammability limits displayed in dependence of the sample concentration in the vapor phase

1. A starting temperature is chosen well below the expected flash point of the sample.
2. The test specimen's vapor concentration is continuously enriched in the headspace by increasing the temperature.
3. An ignition source is brought into contact with the vapors multiple times in temperature intervals defined by the used flash point test method.
4. As soon as the vapor concentration reaches the lower flammability limit (LFL) an ignitable concentration is reached in the headspace atmosphere, and therefore the vapors will ignite.
5. This temperature is then reported as the flash point.

A flash point measurement will therefore produce circumstances which are otherwise strictly avoided in lab environments, namely flammable materials in combination with an open ignition source. This potential risk of setting the lab on fire was the original reason why modern flash point test methods were developed. In contrast to older flash point test methods such as ASTM D93 (Pensky-Martens) or ISO 13736 (Abel closed-cup) the *Continuously Closed Cup Flash Point* test methods (CCCFP) ASTM D6450 & D7094 available on era**flash** do not use an open ignition source and are therefore considered as inherently safe.

**ASTM D6450** is the preferred flash point test method in the **flavor & fragrance industry** due to its small sample volume of only 1 mL, making it ideal for relatively precious materials such as perfumes. Typical samples for these applications are alcohols, esters or mixtures and aqueous dilutions thereof, with flash points ranging from slightly below ambient temperature up to 80 °C.

On the other hand, the **petroleum industry** relies on ASTM **D7094** due to its versatility and outstanding precision. It used for many products such as diesel, kerosene, lube oils and fuel oils, having typical flash points from 38 °C up to 250 °C.

# Measurement speed – era**flash** vs. ASTM D93

Apart from the general apparatus design, the modern flash point test method ASTM D7094 differs from D93 in another aspect: while both test methods require the test specimen to be continuously heated during the measurement, the heating rate is different. While D93 increases the temperature by 5.5 °C/min, D7094 uses a slower rate of 2.5 °C/min for improved precision.

| | |
|---|---|
| Does that mean that D93 is faster than D7094 in theory? | **Yes** |
| Is a flash point measurement on a D93 tester faster in practice? | **No** |

The total time required for a flash point measurement defining the turnaround time does not consist of just the net measuring time alone. Instead, the measurement procedure has accompanying time-consuming phases as depicted in Figure 2:

1. During the INITIALIZATION PHASE, the sample is regulated slightly below the starting temperature and the heating rate is stabilized.
2. The second step is the actual MEASURING PHASE which is primarily defined by the heating rate.
3. Eventually, the apparatus needs to be cooled down during the FOLLOW-UP PHASE to reset the system to be ready for the next measurement.

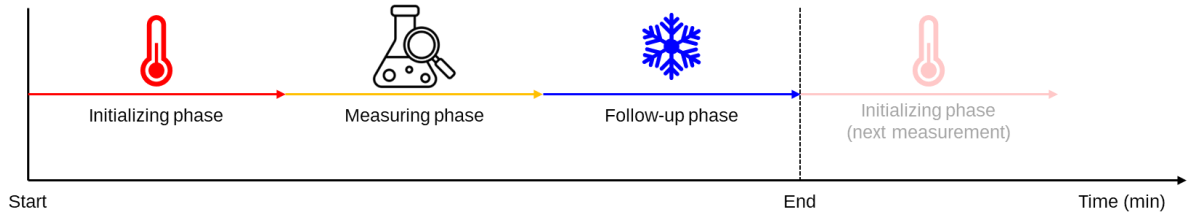


Figure 2: Phases of a flash point measurement

To determine the turnaround time for ASTM D7094 & D6450, a series of 10 sequential measurements (5 each) was performed on an era**flash S10** autosampler flash point tester.

- **Anisole** exhibits a flash point of 44 °C and is therefore a good representative material for the flavor & fragrance industry. Furthermore, it is a reference material stated in **ASTM D6450**.
- **Diesel** is the economically most important product in the petroleum industry beside Jet Fuel, of which the flash point is determined according to **ASTM D7094**.

The preparation of the samples and the instrument apparatus includes selecting and filling the individual sample cups up to the mark as shown exemplarily in Figure 3. The test method D6450 requires the smaller sample cup with a liquid volume of 1 mL, whereas D7094 uses the larger 2 mL cup.

The cups can either be filled directly on the analyzer or on the carousel and/or the sample cups can be conveniently removed and reinstalled by hand in a matter of seconds.

Figure 3: Carousel and sample cups of era**flash S10**

The corresponding test method is selected for each of the activated positions in the user interface directly on the analyzer. The screenshot below (Figure 4) shows the display for both the ASTM D6450 anisole & D7094 diesel measurements.

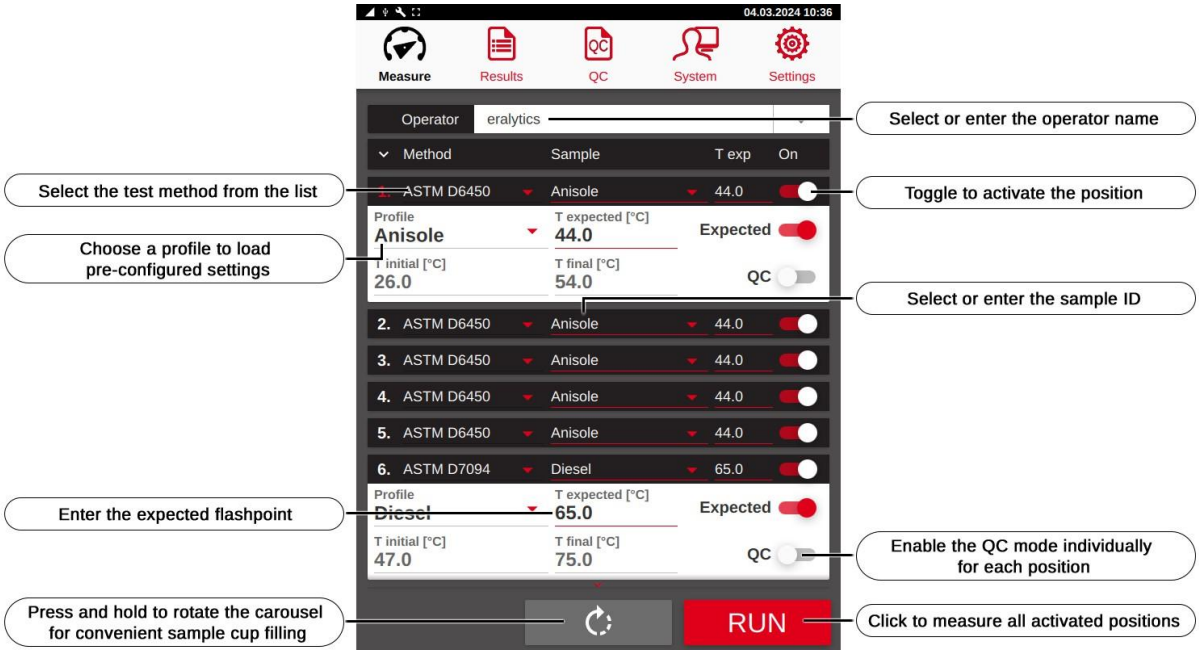


Figure 4: Screenshot of the *"Measure"* menu on the analyzer for the ASTM D6450 Anisole & D7094 Diesel measurements.

The expected flash point of a sample is entered, or the suitable temperature settings can be loaded by choosing a pre-configured *profile* from the list. Anisole was measured on positions 1-5 with an expected flash point of 44 °C, and the diesel measurements were carried out on the positions 6-10, setting the expected flash point to 65 °C. The current time was recorded immediately before starting the measurement series by pressing the *RUN* button on the screen. The analyzer then automatically and sequentially measured all sample cups (positions 1-10) placed on the carousel. The flash point results and the measuring time individually for each position, as well as the total measuring time are listed in Figure 5 below:

| Cup # | Method | Sample | Time per cup | Flash point | Total time | Comment |
|---|---|---|---|---|---|---|
| | | | | | 0 min | --- Start --- |
| 1 | D6450 | Anisole | 6 min | 43.6 °C | 6 min | Cup 1 finished |
| 2 | D6450 | Anisole | 7 min | 43.6 °C | 13 min | Cup 2 finished |
| 3 | D6450 | Anisole | 7 min | 43.6 °C | 20 min | Cup 3 finished |
| 4 | D6450 | Anisole | 6 min | 43.6 °C | 26 min | Cup 4 finished |
| 5 | D6450 | Anisole | 7 min | 43.6 °C | 33 min | Cup 5 finished |
| 6 | D7094 | Diesel | 12 min | 65.6 °C | 45 min | Cup 6 finished |
| 7 | D7094 | Diesel | 11 min | 64.6 °C | 56 min | Cup 7 finished |
| 8 | D7094 | Diesel | 11 min | 64.6 °C | 67 min | Cup 8 finished |
| 9 | D7094 | Diesel | 11 min | 64.6 °C | 78 min | Cup 9 finished |
| 10 | D7094 | Diesel | 12 min | 65.6 °C | 90 min | --- End --- |

Figure 5: Anisole and diesel flash point results according to ASTM D6450 and D7094. As specified by the test methods, the flash point results are corrected to an ambient pressure of 101.3 kPa.

All three test methods – ASTM D93, D6450 & D7094 – require a starting temperature of at least 18 °C below the expected flash point. Therefore, the net measuring time equals **3.5 min** for D93 & D6450 and **8 min** for D7094 as defined by their heating rates of 5.5 °C/min (D93 & D6450) and 2.5 °C/min (D7094), respectively.

The measurement sequences, consisting of the INITIALIZATION PHASE, the MEASURING PHASE and the FOLLOW-UP PHASE are plotted in Figure 6 according to these findings. The ASTM D93 data were determined according to the performance specifications of commercially available Pensky-Martens testers:

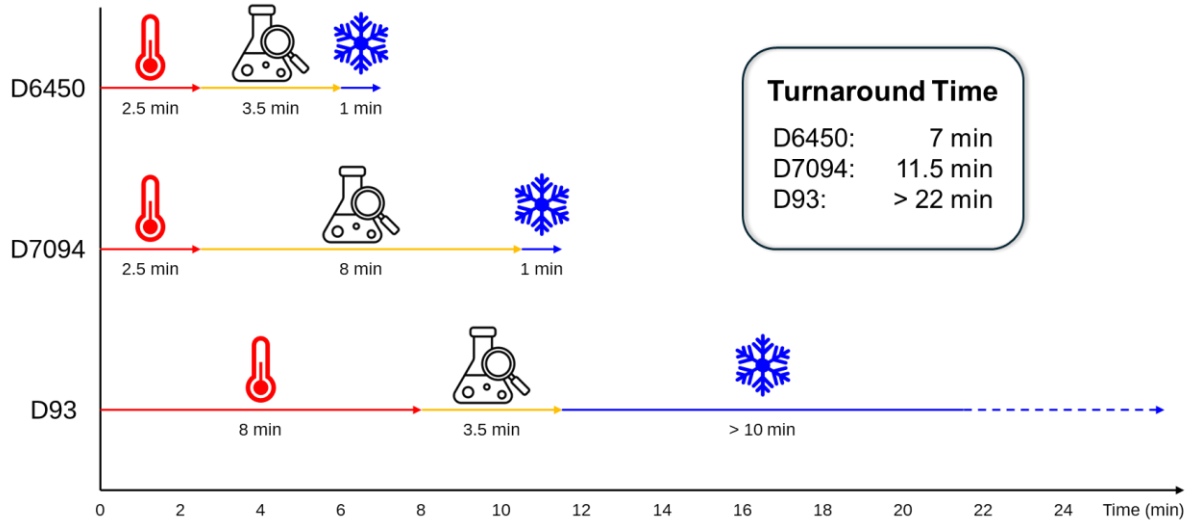


Figure 6: Turnaround times plotted for flash point test methods ASTM D93, D6450 & D7094

The average turnaround time for a typical diesel sample measured according to ASTM D7094 was found to be approximately **11.5 min**, and an anisole measurement took close to **7 min**. Taking into consideration the net measuring time of 8 min (D7094) and 3.5 min (D6450), respectively, the analyzer roughly required **3.5 min** per measurement for the initialization and follow-up phase.

The tremendous difference between the turnaround times of D93 and era**flash**'s CCCFP methods ASTM D6450 & D7094 can be unambiguously explained:

- D93 requires a sample volume of 75 mL per measurement, which in turn requires a massive and inertial heating system of a thermal mass easily exceeding 2-3 kg.
- In contrast to that, a sample volume as small as 1-2 mL is needed for era**flash**, and the patented eralytics PBT® - Peltier Boost Technology allows for otherwise unreached heating and cooling power.

This fundamental difference in apparatus design limits the overall speed for an ASTM D93 measurement. While the net measuring time is short due to the relatively fast heating rate of 5.5 °C/min, the turnaround time exceeds **22 min** for a typical diesel sample. Both the INITIALIZATION PHASE (heating) and especially the FOLLOW-UP PHASE (cooling) take considerably longer due to the vastly increased thermal mass of the analyzer and the test specimen itself.

# Conclusions

- era**flash** is inherently safe

  The apparatus design – no open ignition source (gas flame or glowing wire) – eliminates the risk of fire hazards for flash point measurements in the laboratory.

- ASTM D7094 is twice as fast as D93

  The turnaround time of a typical D7094 diesel measurement on era**flash** is 11.5 min compared to 22 min on a D93 flash point tester. This allows for up to 5 measurements per hour (instead of only 2-3).

- The turnaround time on era**flash** can be as low as 7 min

  By using the test method ASTM D6450 featuring a faster heating rate of 5.5 °C/min, the turn-around time can be reduced down to less than 7 min per measurement which allows for up to 8 measurements per hour.

- era**flash** is the most innovative and fastest flash point tester on the market

  The patented PBT® - Peltier Boost Technology allows for otherwise unreached heating and cooling power on era**flash**. Furthermore, a temperature range of -25 °C to 420 °C is covered with one single analyzer.

- CCCFP methods reduce waste

  Due to the small sample volume of only 1-2 mL, a flash point measurement on era**flash** produces practically no waste. In contrast, a ASTM D93 measurement requires a sample volume of 75 mL, which produces a considerable amount of liquid waste at the end of the working day.

- era**flash** features a user-friendly interface

  Setting up individual measuring tasks is very convenient on era**flash**. Several test methods, suitable for different applications or industries, are available, and pre-configured profile settings can be created and loaded directly on the analyzer.

- era**flash S10** is the only stand-alone autosampler flash point tester on the market

  Neither the operation nor the automatic data export to a LIMS server via ethernet require the use of a PC.