Flash Point determination in Food, Flavors and Fragrances

ERAFLASH™, an essential and reliable tool to guarantee both product quality and transportation safety.

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Introduction

Engineers and scientists of the food, flavors and fragrances industries are faced with many difficult tasks to control the quality of their processes, raw materials, intermediates and final products and to guarantee the consistency of production[1]. Identifying changes in process parameters that may lead to changes in quality, as well as detecting adulteration in any ingredient in cases of fraud and unfair competition are examples of these challenges. Quality in these industries is a fairly abstract term since it involves aromas, freshness, shelf live, compatibility with other volatile substances in fact it is a parameter that is very difficult to quantify. There is a need to establish a profile or finger print in which the measure of volatility through its flash point is becoming imperative.

Flash Point is by definition, the lowest temperature at which the vapors of a volatile material will ignite in the proximity of an ignition source. Flash Point serves as a tool to classify materials as flammable or combustible. The classification of your material will affect many requirements including labeling, storage requirements, shipping requirements and disposal.

Therefore, measuring the flash point is necessary to establish appropriate safety measures of transportation of volatile organic compounds, under which flavors, essential oils and fragrances fall into. The classification of fuels and other volatile materials by their flash point or flammability is a very old and well-established practice. Today the flash point is defined in numerous international standard test methods and measured by instruments using either 'open cup' or 'closed cup' apparatus as described in Table 1.

For many years the two traditional flashpoint methods ASTM D56 (TAG method) and ASTM D93 (Pensky-Martens method) were extensively utilized. Both methods share a common principle: 70-75 ml of sample is heated, in a specified interval, the lid of the cup is opened, and a test flame or glowing wire is lowered into the vapor space above the liquid to test whether the vapor ignites.

The ERAFLASH™ basic apparatus, shown in fig.1, or the automatic unit equipped with an S10 autosampler shown in fig.2 determine the flash point differently and in a modern way. It follows the ASTM D6450[2] and the newer ASTM D7094 Standard "Test Method for Flash Point by the Modified Continuously Closed Cup (MCCCFP) Tester"[3]. Due to its continuously closed cup design and the small amount of sample required for the test, ERAFLASH™ completely eliminates the risk of fire in the laboratory.

Figure 1. The ERAFLASH™ Flash Point analyzer basic unit.

Combined with the patented PBT - *Peltier Boost Technology*™, flash points for VOCs can quickly and safely be analyzed within minutes with quick cool down rates[4]. The flash point is detected by monitoring the pressure increase after the ignition inside the closed measuring chamber, completely preventing the risk of fire. The flame is extinguished automatically due to a lack of oxygen inside the chamber.

The Peltier elements are arranged above the measuring chamber following a revolutionary concept developed by eralytics. Paired with the conveniently small size (1-2 ml) of the continuously closed sample cup design (CCCFP) this new concept allows for extremely safe flashpoint tests over the full required temperature range of -25 to 420°C (-13 to 788°F) in a single portable ERAFLASH™ flashpoint tester. Additionally, the high-speed heating and cooling technology significantly reduces the run time, making flashpoint testing more economical than ever before[3]

The PBT technology is especially suited for flavors and fragrances where most of them are of very volatile nature, constituted by complex mixtures that are costly and produced in small quantities and kept in volatile liquid phase[5] as illustrated in Fig. 3. The requirement of just 1 – 2 mL of sample amount for flashpoint testing is indeed very beneficial for these types of products. ERAFLASH's results thoroughly correlate to D56, D93 and D3828 and their respective ISO standards 2719, 13736, 3679 and 3680.

Maximum Safety

Flash point, according to European and American Safety Regulations Agencies, is a vital parameter to be reported. 'Closed cup' instruments are more often specified as the instrument of choice, because the test results are less affected by laboratory conditions and give a more precise and safer result. To judge the fire hazard of a chemical also highly volatile components have to be taken into account. 'Closed cup' results take these volatiles into account and therefore always lower results than 'open cup' results where volatiles are lost during the heat up phase.

All these characteristics serve the most relevant purpose: a properly determined flash point avoids the risk of underestimating the hazardousness of the measured material and ensures the correct classification, which otherwise could lead to financial penalties.

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High Throughput

One additional benefit of the safest flash point testing method is that it is also safe when used for automating flash point measurements. ERAFLASH S10^{TM} is the bigger brother of ERAFLASH^{TM} enabling the unattended measurement of up to 10 samples. The samples are loaded on a tray and are always covered by a lid which can be seen in figure 2. This lid can optionally be cooled which further reduces the evaporation losses of highly volatile material prior to the measurement.

Figure 2. New ERAFLASH^{TM} with S10 Autosampler.

This automation is of special interest for QC labs of flavors and fragrances companies since they are producing many different substances all of which need measured flash point for the safety data sheet that must accompany every shipment. Bigger producers can easily face 100-200 flash point samples a day which can take up a significant amount of lab technician time if measured on a single position unit.

Automating these time-consuming measurements increases the efficiency in such environments significantly. And all that is possible without any bad feelings concerning safety issues.

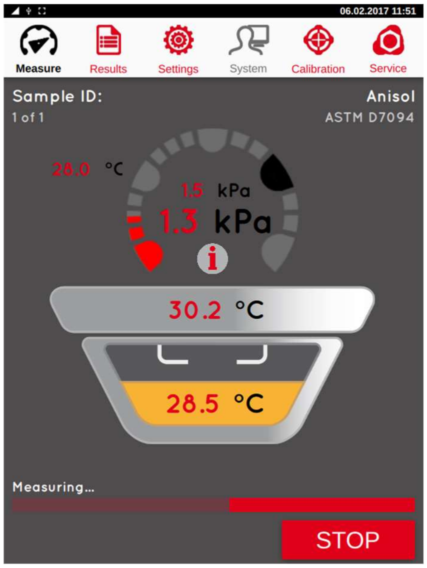

Figure 3. ERAFLASH™ software running a measurement

Figure 4. Essential Oils and Fragrances

Experimental Results

ERAFLASH™ displays and stores combustion characteristics of products facilitating detection of contamination and adulteration of products of enormous significance in the food, flavor and fragrances industry.

The Globally Harmonized System (GHS) of Classification and Labelling of Chemicals categorizes the flammable liquid criteria according the following flash point ranges:

Criteria	GHS Category	Transport Class/Packing Group
Flash Point <73°F (23°C) & Initial boiling point ≤95°F (35°C	1	3, I
Flash Point <73°F (23°C) & Initial boiling point ≤95°F (35°C	2	3, II
Flash Point ≥73°F (23°C) & ≤140°F (60°C)	3	3, III
Flash Point >140°F (60°C) & ≤200°F (93.3°C)	4	Combustible liquid, III

Table 1. Classification of Volatile materials

Two types of samples were analyzed with the ERAFLASH™ instrument according to D7094 method: A series of 5 essential oils presented in Table 2 and 9 raw materials like moisturizing and fragrance additives listed in Table 3. Both types of raw materials are used in the perfume and cosmetic industry. Flash point results of essential oils and fragrances listed in table 2 were determined in triplicates in order to display the dispersion and repeatability of the tests.

The results shown in Table 3, list a series of “No Flash” results primarily due to the considerable water content typical for hand & body creams or other additives & raw materials used in the cosmetic industry.

Table 3 also compares two different types of measurement results: SCAN and ASTM D7094. To measure a flash point according to ASTM D7094 an expected flash points needs to be known. If this is not possible then the SCAN method can be used over a broad temperature range to determine the approximate flash point to be used afterwards for the ASTM D7094 measurement.

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Run #	Sample ID	Pamb (kPa)	Flash Point (°C)	Corr. FP (°C)	Mean Value (°C)	STDEV	% RSD
1	Sample 1	99.6	59	59.42	59.08	0.58	0.99
2	Sample 1	99.6	59	59.42			
3	Sample 1	99.7	58	58.41			
4	Sample 2	98.6	59	59.66	60.33	0.58	0.96
5	Sample 2	98.6	60	60.66			
6	Sample 2	98.6	60	60.66			
7	Sample 3	98.7	58	58.65	58.65	0.01	0.02
8	Sample 3	98.8	58	58.64			
9	Sample 3	98.7	58	58.66			
10	Sample 4	98.9	59	59.59	59.54	0.05	0.08
11	Sample 4	99.2	59	59.53			
12	Sample 4	99.3	59	59.50			
13	Sample 5	99.1	50	50.55	49.88	0.58	1.16
14	Sample 5	99.1	49	49.56			
15	Sample 5	99.1	49	49.54			

Table 2. Flash Point on Essential Oils

			EXPERIMENTAL RESULTS	
Run #	**Sample ID**	**Sample Type**	**SCAN (°C)**	**ASTM D7094 (°C)**
1	Sample 6	colorless liquid	100	100.4
2	Sample 7	colorless liquid	104	104.5
3	Sample 8	white cream	No Flash	No Flash
4	Sample 9	orange- viscous liquid	No Flash	No Flash
5	Sample 10	beige cream	60	58.5
6	Sample 11	white pellets	196	188.5
7	Sample 12	beige cream	60	58.5
8	Sample 13	beige cream	No Flash	No Flash
9	Sample 14	beige cream	No Flash	No Flash
10	Sample 15	dark-blue cream	No Flash	No Flash

Table 3. Flash Points of Cosmetics

Conclusions

ERAFLASH™ and ERAFLASH S10™ testers are innovative, reliable, versatile, safe and easy to operate. The testers require a very small amount of sample and automatically perform the measurement in minutes, representing a throughput of up to 12 samples per hour. ERAFLASH™ can store more than 100000 detailed test reports and has pre-programmed correlation methods to the most significant ASTM, ISO and other international standard test methods. Furthermore, it provides for maximum efficiency under the amplest temperature ranges to allow for more applications including very volatile and valuable materials, such as fragrances and even hazardous waste materials.

References

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