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Flash Point: Evaluation, Experimentation and Estimation

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Abstract The flash point is an important indicator of the flammability of a chemical. For safety purposes, many data compilations report the lowest value and not the most likely. This practice, combined with improper documentation and poor data storage methods, has resulted in compilations filled with fire-hazard data that are inconsistent with related properties and between members of homologous chemical series. In this study, the flash points reported in the DIPPR® 801 database and more than 1,400 other literature values were critically reviewed based on measurement method, inter-property relations, and trends in chemical series. New measurements for seven compounds illustrate the differences between experimental flash points and data commonly found in fire-hazard compilations. With a critically reviewed set of experimental data, published predictive methods for the flash point were evaluated for accuracy.

Keywords Data evaluation · Database · DIPPR · Flash point · Prediction

1 Introduction

The flash point (*FP*) is defined as the lowest temperature, corrected to 101.3 kPa, at which application of an ignition source causes the vapors of a specimen to ignite under specified conditions of a test [1]. An important indicator of the flammability of a substance, the flash point is frequently used as a parameter in process, storage, and fuel

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design. Additionally, the U.S. Department of Transportation requires the flash point of a liquid be known for shipping and transportation purposes.

Not surprisingly, flash points of common chemical substances are widely reported. Differences in apparatuses and experimental methods may influence the measured flash point by tens of degrees. Poor data storage and reporting procedures combined with circular referencing have further exacerbated this problem, resulting in many hazardous property and chemical data compilations that report flash-point values inconsistent with other chemical properties. These inconsistent data are then often used in regressing parameters for flash-point estimation methods.

In this study, we illustrate the use of proper data evaluation techniques to evaluate the flash-point data in the DIPPR® 801 database [2]. Experimental measurements are made to illustrate the effect of apparatus on the flash point and to supplement the critical review. With this consistent set of data, the accuracy of published prediction methods is evaluated.

2 Flash-Point Data Evaluation

Because flash-point values are dependent on the apparatus and experimental method, it is often difficult to determine the "best" or most probable value when more than one measured value is available for a given compound. For safety purposes, many fire-hazard data compilations have adopted the policy of publishing the lowest reported value. This practice results in unnecessary and costly process restrictions, ignores fuel design as an application of flash-point data, and disregards the important relationships between the flash point and several other properties. To aid researchers and engineers in flash-point data evaluation, we suggest several guidelines for selecting the most probable flash point.

2.1 Interproperty Relations

The most fundamental relationship is between the flash point and the lower temperature limit (*LTL*). The lower temperature limit is defined as the minimum temperature, corrected to a pressure of 101.3 kPa, below which the mixture of air and vapors in equilibrium with a solid or liquid will not support flame propagation. This definition is essentially identical to that of the flash point, and in fact, the two properties differ only in their measurement method [3,4]. Lower temperature limit experiments are typically performed at a fixed temperature, in a vessel with a volume of at least 1 L. The standard ignition source is a high energy spark or fuse-wire, positioned at least halfway down the vessel to allow for upward flame propagation.

The flash point, on the other hand, is meant to be a small-scale approximation of the lower temperature limit. Flash-point experiments are performed in small cups with an unheated lid, and conditions are rarely at equilibrium. Flash-point measurements use a small, weak flame as the ignition source at the top of the cup, resulting in downward and outward flame propagations.

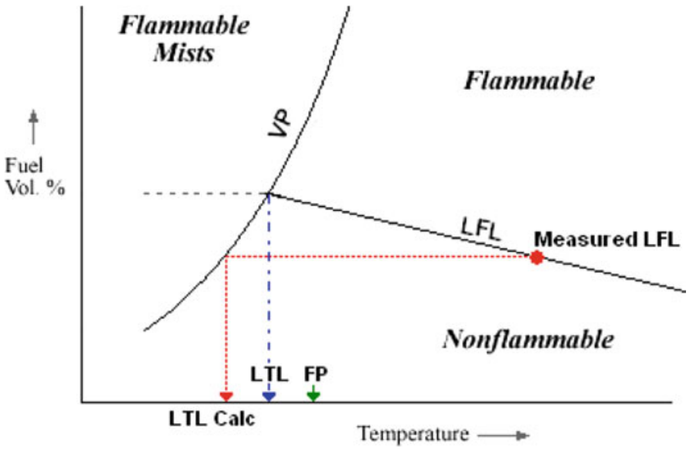

Fig. 1 Relationship among *FP, LTL*, and the *LTL* if *LFL* is assumed to be independent of temperature

These differences in experimental methods lead to our first criterion,

$$FP \geq LTL \tag{1}$$

Evlanov [5] tried to quantify this difference, regressing flash-point data against lower temperature limit points:

$$FP - LTL = 0.4FP + 2 \tag{2}$$

The coefficients for this correlation certainly depend upon the measurement apparatuses, but unfortunately Evlanov gives little information about how either the flash-point or lower temperature limit data were obtained.

More commonly reported than the lower temperature limit, the lower flammability limit (*LFL)* is defined as the minimum percent volume of a combustible substance in air that is capable of propagating a flame upward and outward from an ignition source. Lower flammability limits are determined similarly as the lower temperature limit, but typically above saturation so that no liquid is present.

As shown in Fig. 1, the lower flammability limit is related to the lower temperature limit through the vapor–pressure curve (*VP*). The lower temperature limit is the intersection of the vapor-pressure curve with the temperature-dependent lower flammability limit curve. Thus, the flash point is related to the measured lower flammability limit by

$$\frac{VP(FP)}{101.3\,\text{kPa}} \times 100\,\% > LFL \tag{3}$$

The inequality in Eq. 2 has changed to show that the flash point at the vapor pressure is greater than the lower flammability limit, and not greater than or equal to, as would be expected from Eq. 1. This is a result of the temperature dependence of the lower flammability limit, a characteristic of the property that is often overlooked. As mentioned above, flammability limits are measured at some temperature above saturation to avoid condensation in the test vessel. Because lower flammability limits decrease monotonically with increasing temperature (T),

$$\frac{VP(LTL)}{101.3\,\text{kPa}} \times 100\,\% > LFL(T) \tag{4}$$

unless the temperature dependence of the flammability limit is known and the measured value has been extrapolated back to the lower temperature limit. This is rarely the case with reported data, however. Instead, the lower flammability limit is usually assumed to be independent of temperature, as shown by the horizontal dashed-line in Fig. 1.

Several investigators have tried to derive a more exact relationship between the flash point and lower flammability limit. Affens [6] suggested an empirical relation between the flash point and lower flammability limit of *n*-alkanes:

$$(FP + 277.3)^2 = \frac{77291}{LFL} - 3365 \tag{5}$$

Similarly, Kueffer and Donaldson [7] established an empirical formula for a wider range of chemicals:

$$\frac{VP(FP)}{101.3\,\text{kPa}} = 1.5 \cdot LFL(298\,K) + 0.00198 \tag{6}$$

Oehley [8] derived a semi-empirical equation relating the flash point and the lower flammability limit to the normal boiling point (T_b):

$$LFL = \frac{14400}{(T_\text{b} - FP)^2} \tag{7}$$

Kanury [9] took a more theoretical approach. Starting with the Clapeyron equation, he derived an expression relating the flammability limit, the enthalpy of vaporization (H_v), the boiling point, and flash point:

$$\ln(LFL) \leq \frac{H_\text{v}}{R}\left(\frac{1}{T_\text{b}} - \frac{1}{FP}\right) \tag{8}$$

which may also be written as

$$FP = \frac{T_\text{b}}{1 - \frac{RT_\text{b}}{H_\text{v}} \ln(KLFL)} \tag{9}$$

According to Kanury, the value K is apparatus dependent to account for differences in fuel vapor dispersion and mass transfer. For an apparatus with uniform vapor dispersion and no fuel loss to the atmosphere, K is unity. Without a consistent set of

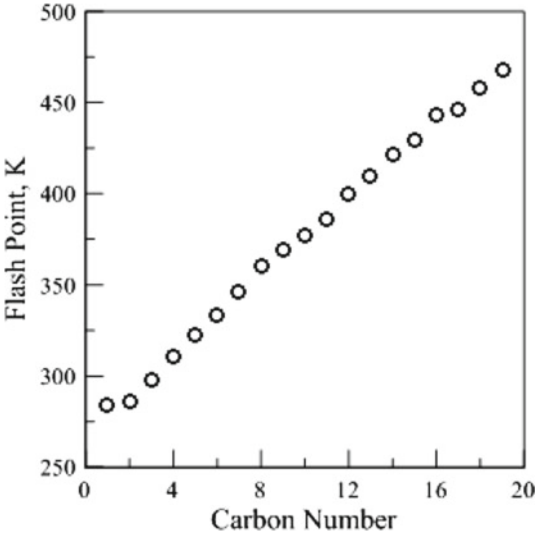

Fig. 2 Flash point as a function of carbon number for a homologous series, shown here for the *n*-alcohols; values taken from DIPPR® 801 database

experimental flash-point and lower flammability limit data, however, values of K cannot be determined.

2.2 Chemical Series Trends

Plots of the flash point for homologous chemical series, such as the *n*-alkanes, show clear patterns in the data with respect to carbon number (Fig. 2). The flash points for broader classes of chemicals may be evaluated similarly by plotting the flash point against the normal boiling point, enthalpy of combustion (*HCOM*), and enthalpy of vaporization at the flash point, and plotting the vapor pressure at the flash point against the enthalpy of combustion (Fig. 3).

A comparison of two or more similar compounds may also be used to obtain a rough estimate of a flash point. For example, it is expected that *sec*-butylbenzene, isobutylbenzene, and *n*-butylbenzene would all have similar flash points. Experimental data in the DIPPR® 801 database report the flash point of these compounds as 325 K, 325 K, and 323 K, respectively.

2.3 Common Errors/Mistakes

Evaluation of thousands of flash-point values from hundreds of sources reveals several recurring errors in the published data:

- Certain reported values appear repeatedly in fire-hazard data compilations such that the following values should always be considered suspect: 110 °C(230 °F), 100 °C(212 °F), 0 °C(32 °F), and − 20 °C(−4 °F). These values are actually extrema of the expected flash point, i.e., >110 °C or <−20 °C, but unfortunately the inequality sign is often absent. The most frequent of these values is 110 °C,

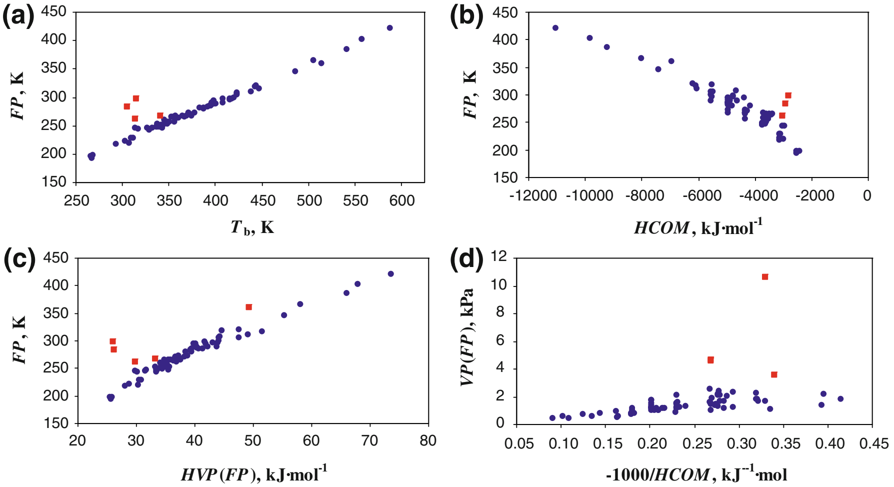

Fig. 3 Flash-point trends for the alkenes and alkynes by plotting (**a**) *FP* versus T_b, (**b**) *FP* versus *HCOM*, (**c**) *FP* versus *HVP*(*FP*), and (**d**) *VP*(*FP*) versus -1000/*HCOM*; *squares* indicate points with possible errors

which is the upper range of the ASTM 9C thermometer commonly used in flash-point experiments.

- Because the measured flash point depends significantly on the apparatus, information pertaining to measurement method/apparatus is crucial in evaluating flash-point data. Unfortunately, compilations often make general statements pertaining to the measurement method that are inaccurate for many of the reported values. For example, the *Aldrich Handbook of Fine Chemicals* [10] states that the reported flash points “are determined with the ‘Setaflash’ apparatus recommended by the DOT using ASTM Procedure D3278.” Personal communication with Aldrich confirmed that this statement is true when the flash point is actually measured at Aldrich, but these data are in no way differentiated from the points that are predicted or cited, or that were measured previous to the standardization of the Setaflash apparatus.

  Rather than specify a specific apparatus, most compilations generalize by stating that measurements are “closed-cup unless otherwise noted.” It does not take long to find inconsistencies among the compilations with regard to this statement. For example, DIPPR cites several compilations reporting the flash point of 1-pentene as 255 K. Two compilations claim this value was determined in an open-cup, two references make no statement pertaining to the apparatus, and two list the value as being determined in a closed-cup apparatus.
- Unit conversion is a perpetual problem in data compilations, and flash points are no exception. Compilations often neglect to convert a value reported in °C to °F and vice versa.

It is not difficult to imagine how the common practice of reporting the lowest point available instead of the most probable value only serves to aggravate the problems

Table 1 Flash-point data statistics in the original and reviewed databases showing the difference in the number of flash-point data, grouped by data type

	Original		Reviewed	
Data type	Number of DIPPR recommended values	Total	Number of recommended values	Total
Experimental	935	2974	1008	3884
Unknown	97	794	133	1478
Smoothed	19	20	34	35
Predicted	674	869	568	966
Total	1725	4657	1743	6363

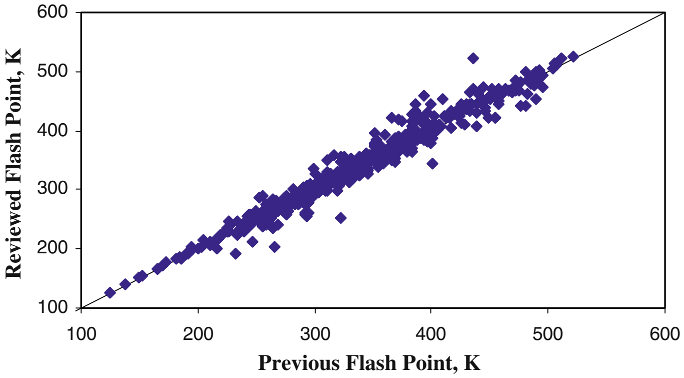

Fig. 4 Reviewed flash points versus previously accepted DIPPR values

Table 2 Statistics relevant to the change in the accepted flash-point value

	Average	Median
$FP_{\mathrm{old}} - FP_{\mathrm{new}}$ (K)	−1.57	0.00000
Abs ($FP_{\mathrm{old}} - FP_{\mathrm{new}}$) (K)	4.29	0.15000
% Diff	−0.47	0.00
% Abs. diff	1.31	0.045

caused by these common errors, especially when unintentional circular referencing occurs. The result is compilations filled with erroneous data.

2.4 Evaluation of DIPPR® 801 Flash-Point Data

Applying the evaluation techniques and criteria above, flash-point data stored in the DIPPR® 801 database for over 2,000 chemical components were critically evaluated with more than 1,400 other values found during a large-scale literature search. A comparison of database statistics before and after the review is shown in Table 1.

A plot of the old flash-point data accepted by DIPPR against the new values shows no obvious trend in the changes recommended by the review (Fig. 4). Statistics in Table 2 also appear to indicate the lack of a significant bias in the review.

Table 3 Flash-point apparatuses commonly used today

Apparatus	Temperature uniformity	Sample volume (mL)	Primary use
Tagliabue (Tag)	Liquid bath	50	Compounds with viscosity $<5.8 \times 10^{-6}\,\mathrm{m^2 \cdot s^{-1}}$ at 38 °C
PM	Stirred; metal shell	75	Compounds with viscosity $>5.8 \times 10^{-6}\,\mathrm{m^2 \cdot s^{-1}}$ at 38 °C
Cleveland	Metal plate across cup base	70	Open-cup tests
SS (Setaflash)	Preheated to fixed temperature	2 or 4	SS and flash/no-flash tests
Abel	Water bath and air gap	79	European tests

Table 4 Differences between ASTM methods using the PM and SS apparatuses

Method	Apparatus	Heating rate	flash point range (°C)	Repeatability	Reproducibility
D 93	PM	$5–6\,°\mathrm{C \cdot min^{-1}}$	40–104	2 °C	3.5 °C
			104–370	5.5 °C	8.5 °C
D 3278	SS	Fixed temperature	0–110	1.7 °C[a]	3.3 °C
				3.3 °C[b]	5 °C
D 3828	SS	Fixed temperature	20–70	0.5 °C	$0.03(M + 29\,°\mathrm{C})$
			70–300	$0.022M$[c]	$0.083M$
D 3941	PM	$0.5\,°\mathrm{C \cdot min^{-1}}$	0–110	2 °C	3 °C
D 3934	PM	Fixed temperature	0–110	Unknown	Unknown

[a] For viscosity $<5.8 \times 10^{-6}\,\mathrm{m^2 \cdot s^{-1}}$ at 38 °C
[b] For viscosity $>5.8 \times 10^{-6}\,\mathrm{m^2 \cdot s^{-1}}$ at 38 °C
[c] M is the mean of two results

3 Experimental Determination

Because the measurement method and apparatus may significantly affect the flash point, a basic understanding of the differences among these apparatuses helps in evaluating differences in reported data.

3.1 Standardized Flash-Point Apparatuses

There are five main flash-point apparatuses used internationally today (Table 3), the majority of which are closed-cup. The two most widely used flash-point apparatuses in the USA are the Pensky-Martens (PM) and small-scale (SS). Details of the differences between the ASTM test methods that utilize these apparatuses are listed in Table 4.

Flash points measured in open-cup apparatuses may differ by tens of degrees from closed-cup values. Open-cup values are dependent on the distance of the ignition source from the liquid surface, and therefore may not represent any meaningful hazard property [11].

The difference between automated and manual apparatuses was examined by Montemayor et al. [12]. Despite finding no statistical difference between flash points determined using automated versus manual apparatuses, ASTM still gives preference to values measured manually.

Probst [13] investigated the differences in flash points for 26 compounds found when using Tag and PM apparatuses. Values found in the Tag apparatus were on average 0.8 °C higher than those found using the PM device, though there appeared to be a linear correlation between the values obtained in the two apparatuses.

For this study, measurements were made using PM (ASTM D 93) and SS (ASTM D 3828) apparatuses. The most notable difference between these two methods is that the SS is set to a fixed temperature for each flash-point determination, whereas the PM apparatus is ramped at a constant heating rate (5 to 6) $^\circ\mathrm{C} \cdot \mathrm{min}^{-1}$). There is evidence that the heating rate employed in method D 93 does not yield equilibrium conditions between the vapor and liquid, and may result in an elevated flash point. To overcome this shortcoming, ASTM developed methods D 3941 and D 3934 for the PM apparatus which use a slower ramping rate ($0.5\,^\circ\mathrm{C} \cdot \mathrm{min}^{-1}$) and fixed temperature, respectively. However, these methods are substantially more time consuming than D 93, and consequently there are few data points reported that were obtained using them.

3.2 Experimental Results

Table 5 lists the previously reported literature flash-point data with the value recommended by DIPPR listed first, the measured flash point using both the PM and SS apparatuses for seven compounds, and the difference between the measured values found using the two apparatuses. At least four replicates were made using both apparatuses, except where noted. The minimum purity of the compounds studied was 98 %.

Although by no means a thorough study, the data show differences up to 7 K between values determined in the PM and SS apparatuses. Two-sided t-tests ($\alpha = 0.05$) indicated statistical differences between the measured values for all compounds except 2-nonanone. The data also indicate that there are often significant differences between experimental data and values commonly found in compilations.

Table 5 Comparison of literature values found in the DIPPR® 801 database, and experimental measurements for the flash point using PM and SS apparatuses

Compound	Literature values (K)	PM (K)	SS (K)	PM-SS (K)
Diisobutyl phthalate	434, 458[a], 458	447.6 ± 2.1	444.6 ± 1.1	3.0
Isopropyl myristate	423[b]	432.3 ± 3.7	424.4 ± 0.8	7.9
n-Dodecanoic acid	430[b], > 428 but < 438	440.9 ± 2.6	437.1 ± 0.4	3.8
2-Nonanone	337, 344	345.7 ± 6.7[c]	345.4 ± 0.8	0.3
n-Butanol	306[b], 302, 310	310.5 ± 0.6	313.1 ± 0.7	−2.6
n-Octanol	357[a], 354	361.8 ± 0.5	359.7 ± 1.1	2.1
n-Hexadecanol	427[a], 408, > 383, 383	446.7 ± 3.2	442.9 ± 0.7	3.8

DIPPR recommended values are listed first

[a] Reported as open-cup

[b] Predicted by DIPPR

[c] Only two replicates taken in the PM device

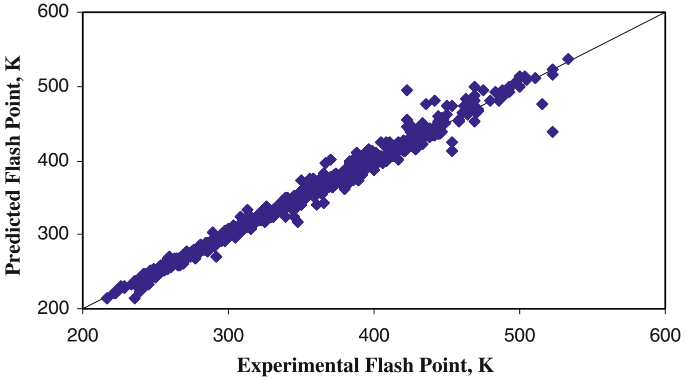

Fig. 5 Flash-point values predicted by Leslie and Geniesse versus experimental values

4 Estimation

Using the DIPPR® Information and Data Evaluation Manager (DIADEM) software [14], we evaluated the performance of 33 published flash-point estimation methods against data in the DIPPR® 801 database for more than 1,000 compounds, and against the primary experimental points mentioned above. The average absolute percent deviation between the predicted and database values are shown in Table 6 for each method.

In all cases, the method of Leslie and Geniesse resulted in the lowest absolute average percent deviation. They related the flash point to the moles of oxygen required for stoichiometric combustion (β):

$$\frac{VP(FP)}{101.3\,\text{kPa}} = \frac{1}{8\beta} \tag{10}$$

Figure 5 shows the value predicted by the method of Leslie and Geniesse plotted against the reviewed flash-point value for the evaluated compounds.

It is interesting that the three most accurate methods all utilize the vapor pressure to estimate the flash point and the two most accurate methods both use the stoichiometric moles of oxygen for combustion. When accurate knowledge of the vapor pressure is not available, the empirical equation proposed by Ishiuchi may be used instead:

$$FP = \left[T_\text{b}^{a} + b\left(\frac{\beta}{760}\right)^{-a} - c\right]^{1/a} \tag{11}$$

where the constants a, b, and c are 0.105, 0.0570, and 0.142, respectively, for associating liquids, and 0.119, 0.0656, and 0.185 for liquids that do not associate.

Table 6 Absolute average deviations of flash-point prediction methods against the experimental and unknown accepted values in the DIPPR® 801 database, and the experimental values obtained using the PM and SS apparatuses

	% Absolute deviation		
Method	Overall	PM	SS
Leslie–Geniesse [15]	1.54	1.21	1.62
Modified Thornton [16]	1.62	1.74	1.98
Pintar [17]	1.83	1.85	2.28
Ishiuchi [18]	1.92	2.46	2.30
Catoire–Naudet [19]	2.16	2.53	2.63
Prugh [20]	2.30	3.89	3.74
Metcalfe and Metcalfe [21]	2.57	4.14	3.73
Blinov [22]	2.74	1.34	5.04
Shebeko et al. [22]	3.08	2.72	3.02
Butler Approximation [23]	3.17	3.27	3.47
Affens (from VP) [6]	3.23	3.34	3.54
Korol’chenko et al. [24]	3.37	2.42	3.13
Modified Satyanarayana–Kakati [25]	3.37	5.62	5.33
Patil [26]	3.42	3.71	2.73
Wang–Sun [27]	3.51	3.85	3.58
Hshieh [28]	3.53	4.72	4.50
Oehley [8]	4.09	3.75	4.29
Bodhurtha [29]	4.61	6.42	6.14
Butler et al. [23]	4.62	6.43	6.15
Affens [6]	4.65	6.29	6.07
Möller et al. [30]	4.70	5.57	6.61
Albahri [31]	4.80	25.15	7.20
Li–Moore [32]	4.85	3.76	3.35
Riazi–Daubert [33]	4.90	6.77	6.90
Satyanarayana–Kakati [34]	5.10	6.80	6.56
Satyanarayana–Rao [35,36]	5.11	4.54	3.98
Fujii–Hermann [37]	5.77	10.26	12.37
Suzuki et al. [38]	9.72	10.19	10.91
Akhmetzhanov et al. [39]	10.70	16.16	16.61
Shimy [40]	13.26	11.28	12.25
Katritzky et al. [41]	20.05	28.27	27.39
Affens (from Carbon Number) [6]	22.27	21.95	21.52
Pan et al. [42]	28.04	27.09	26.95

5 Summary

When evaluating flash-point data to select the most probable value, it is important to remember:

- The flash point is greater than or equal to the lower temperature limit
- The vapor pressure at the flash point divided by 101.3 kPa is greater than the flammability limit at the lower temperature limit
- Trends in the flash point exist among chemical series when plotted against the enthalpy of combustion, the normal boiling point, and the heat of vaporization at the flash point. The vapor pressure at the flash point plotted against the heat of combustion is also helpful
- Compilations are full of erroneous values because of typographical errors and poor documentation standards
- The experimental apparatus and method used to determine an flash point may influence the data significantly

The flash point may be predicted with considerable accuracy using the vapor pressure and the moles of oxygen required for stoichiometric combustion, as demonstrated by Leslie and Geniesse.

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