# Determination of flash point in air and pure oxygen using an equilibrium closed bomb apparatus

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## Abstract

The standard closed testers for flash point measurements may not be feasible for measuring flash point in special atmospheres like oxygen because the test atmosphere cannot be maintained due to leakage and the laboratory safety can be compromised. To address these limitations we developed a new "equilibrium closed bomb" (ECB). The ECB generally gives lower flash point values than standard closed cup testers as shown by the results of six flammable liquids. The present results are generally in good agreement with the values calculated from the reported lower flammability limits and the vapor pressures. Our measurements show that increased oxygen concentration had little effect on the flash points of the tested flammable liquids. While generally regarded as non-flammable because of the lack of observed flash point in standard closed cup flash point testers, dichloromethane is known to form flammable mixtures. The flash point of dichloromethane in oxygen measured in the ECB is $-7.1\,^\circ\mathrm{C}$. The flash point of dichloromethane in air is dependent on the type and energy of the ignition source. Further research is being carried out to establish the relationship between the flash point of dichloromethane and the energy of the ignition source.
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## 1. Introduction

Flash point is the lowest temperature, corrected to the standard atmospheric pressure of 760 mmHg (101.3 kPa), at which application of a test flame causes the vapor of a specimen

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to ignite under specified test conditions. To form a flammable vapor–air mixture above the surface of a flammable/combustible liquid, the temperature of the liquid must be sufficiently high to produce vapor concentration above the liquid surface at or above the lower flammability limit (LFL) of the vapor. In other words, flash point is the temperature at which the vapor pressure divided by the pressure of the atmosphere is equal to the LFL expressed in mole fraction.

Flash point is used in transportation and safety regulations to define flammable and combustible liquids. Usually, flash point is measured in air. Flash points could be affected by the type of oxidant gases employed in a particular process. Many chemical processes may use or generate special oxidant gases, such as oxygen, chlorine and oxides of nitrogen, which may affect the flash point of the contents. Flash point is also used to determine the vapor explosion potential in a process. The risk of vapor explosions is generally greater in atmospheres of high oxidant concentration than in air. It is, therefore, necessary to determine the flash points of materials in special atmospheres.

Typically, flash points are measured using standardized testing methods in either open [1] or closed cup testers [2–7]. Both open and closed cup techniques involve heating a sample in a small vessel to a selected temperature. A pilot flame of hydrocarbon gas is used as the ignition source. The pilot flame is introduced over the surface of the sample and it is noted whether or not an ignition occurs. During a flash point test in an open cup tester, the more volatile components of a multi-component mixture may have been lost before the ignition source is applied. Consequently, open cup methods may overestimate the flash point of a sample containing multiple components. Nevertheless, open cup testers can provide flash point data representative to open vessels and spills. In contrasts, closed cup techniques prevent the loss of volatile components by keeping the sample enclosed until the ignition source is introduced. Additionally, equilibrium between the liquid and the vapor can be established within the test apparatus, provided the rate of temperature increase is small. Therefore, closed cup flash point data are more conservative than and generally preferred to open cup data.

There are several standard closed cup test methods for flash point measurements, which can be divided into non-equilibrium methods and equilibrium methods. Some of the parameters of the standard flash point test methods are summarized in Table 1. With non-equilibrium test methods, the specimen is heated at a specified heating rate. With equilibrium test methods, a closed test cup is placed in an isothermal liquid bath or other isothermal enclosure. As pointed out by ASTM E502 [8], with the exception of ASTM D3934 [6] and ASTM D3941 [7] and the Sataflash methods [4,5], most flash point tests are run at high enough heating rate that vapor concentrations are not representative of equilibrium conditions. All the standard flash point test methods use a hydrocarbon gas flame as the ignition source. The cup volume for the standard test methods is relatively small, the maximum cup volume is 0.11 liter in the case of Pensky–Martens method [2].

ASTM D1232 [9] defines the Lower Temperature Limit of Flammability (LTL) as the lowest temperature, corrected to the pressure of one atmosphere, at which application of an ignition source causes a homogeneous mixture of a gaseous oxidizer and vapors in equilibrium with a liquid (or solid) specimen to ignite and propagate a flame from the ignition source under the specified conditions of the test. A 5 l borosilicate glass flask equipped with a magnetic stirrer bar is used as the test vessel and is placed in a thermally-insulated chamber.

Table 1
Comparison of flash point test methods

| Test method | Test vessel diameter (cm) | Test vessel depth (cm) | Test vessel volume (l) | Heating method |
|---|---|---|---|---|
| ASTM D92 Cleveland open cup [1] | 6.4 | 3.4<br>1.0[a] | 0.11<br>0.032 | The temperature of the specimen is increased at 5–6 °C/min |
| ASTM D93 Pensky–Martens closed cup [2] | 5.085 | 5.6 | 0.11 | For ordinary liquids, the temperature of the specimen is increased at 5–6 °C/min |
| | | 2.184[a] | 0.043 | For liquids with solid suspensions or highly viscous materials, the temperature of the specimen is increased at 1–1.5 °C/min |
| ASTM D56 Tag closed cup [3] | 5.4 | 2.94[a] | 0.067 | For flash points below 60 °C, the temperature of the specimen is raised at a rate of 1 °C/min ± 6 s. For flash points above 60 °C, the temperature of the specimen is raised at a rate of 3 °C/min ± 6 s |
| ASTM D3278 Setaflash closed cup [4] | 5.0 | 1.0 | 0.02 | Sample cup is electrically heated or chilled and the sample temperature is kept constant |
| ASTM D3828 Setaflash closed cup [5] | 5.0 | 1.0 | 0.02 | Sample cup is electrically heated or chilled and the sample temperature is kept constant |
| ASTEM E1232 [9] | 22.2 | ~30.5 | 5.0 | Test vessel is placed in a temperature-controlled chamber |
| Equilibrium closed bomb of this work | 6.7 | 12.2 | 0.43 | Test vessel is placed in an isothermal liquid bath or a temperature-controlled oven |

[a] Volume between the lid and the sample surface.

Instead of a hydrocarbon gas flame, an electrical arc produced at an electrical gap by a 30 mA, 15 kV power source or a 19 mm loop of 40 gauge copper wire (fuse wire) is used as the ignition source. Provided the ignition sources used in the standard closed cup flash point testers (gas flames) [2–7], and the electrical arc and fuse wire used in ASTM E1232 [9] are sufficiently energetic, the LTL of a specimen measured using the method of ASTM E1232 [9] would be the same as the flash point measured using the equilibrium flash point test methods.

When measuring the flash point of a material in an oxidant gas other than air, a challenge common to the standard closed cup test methods is to establish and maintain a desired oxidant gas or oxidant gas mixture in the tester. Several limitations exist in the standard closed cup testers:

• Some of the closed cup testers are not leak tight. Therefore, the oxidant atmosphere in the testers is difficult to maintain, affecting the accuracy.

- As soon as the pilot flame is introduced into the closed cup testers, some portion of the flammable vapor is exposed to the surrounding air, again affecting the accuracy of the measurement.
- When using the standard closed cup testers, the oxidant gas may escape causing an oxidant-enriched atmosphere in the surroundings. This may lead to a more hazardous working environment in the laboratory especially when toxic gases are employed.

The method of ASTM E1232 [9] can provide a controllable oxidant atmosphere, however, the strength of the glass flask limits its application for pressurized or explosive atmospheres. What is needed is a closed test vessel with sufficiently high mechanical strength to withstand the explosion pressures in special oxidant atmospheres. For this reason we developed an "equilibrium closed bomb" (ECB) apparatus to measure the flash points of some flammable liquids in air and oxygen.

## 2. Test method

The ECB test apparatus (Fig. 1) is a cylindrical stainless steel vessel with an internal diameter of 67 mm and a depth of 122 mm. The internal volume of the apparatus is approximately 430 ml. The apparatus is placed in the liquid bath of a chiller/heater with an automatic temperature control. Three K-type thermocouples are used to measure the temperature of the liquid bath, the headspace of the bomb, and the liquid sample, respectively. A continuous electrical arc produced by a 10 kV, 2.5 mA dc power source is used as the ignition source.

When conducting a flash point test, the temperature of the liquid bath is stabilized at a desired temperature. The bomb is purged thoroughly with the oxidant gas (dry air or oxygen in this work) and the internal pressure is adjusted to slightly below one atmosphere. Then, a certain amount of sample is injected into the bomb to form a pool of sample at the bottom of the bomb. A sufficient period of time is allowed for the liquid–vapor system to reach equilibrium. As the sample temperature is stabilized, the pressure in the apparatus can be slightly higher than one atmosphere. Therefore, an exhaust valve is opened slightly as needed to adjust the pressure downward. A slight positive pressure is maintained in order to prevent air from entering the apparatus. Then, the ignition source is activated. If no ignition is observed, we try a second ignition. Typically, the interval between the two trials is 30–40 s. If no ignition is observed in the second trial, the temperature is increased typically by 1–2 °C and the ignition trials are repeated at the new temperature. If an ignition occurs, the temperature of the liquid is taken as the measured flash point. The pressure of the test atmosphere affects the measured flash point and the higher the pressure the higher the flash point. The effect of pressure on the measured flash point is corrected to atmospheric conditions (760 mmHg) using the following equation [2–9]:

$$FP_{Corrected} = FP_{Measured} - 0.033 \times (P - 760) \tag{1}$$

where $FP_{Measured}$ (°C) and $FP_{Corrected}$ (°C) are the measured flash point at pressure $P$ (mmHg) and the corrected flash point at 1 atm (760 mmHg), respectively.
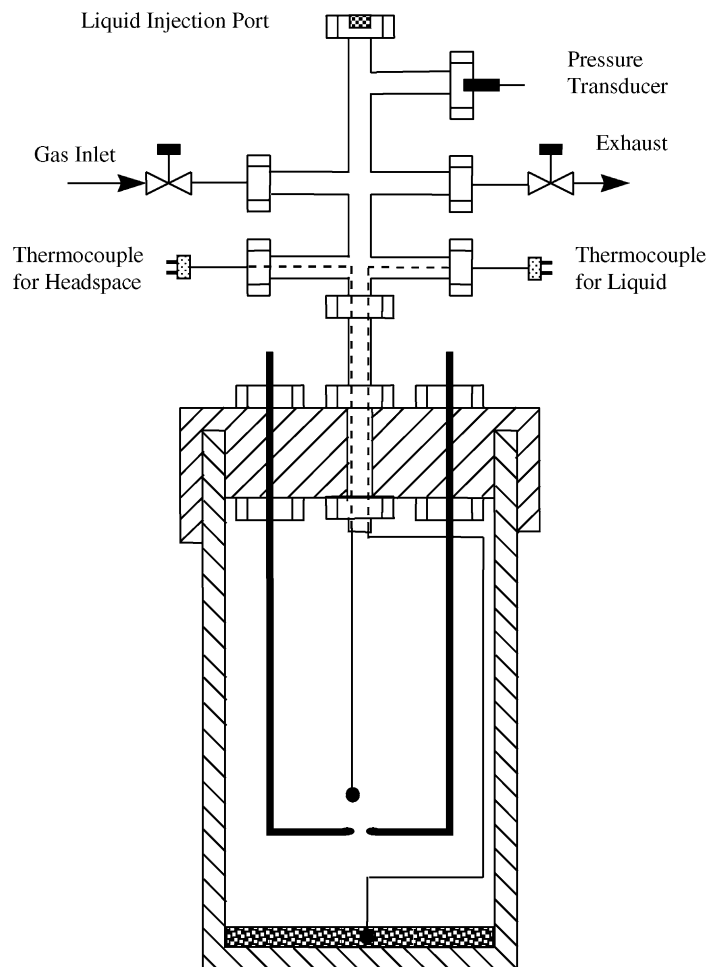
Fig. 1. "Equilibrium closed bomb" apparatus.

## 3. Results and discussions

The flash points of seven commonly used liquids in air and oxygen were measured using the ECB apparatus and the results are presented in Table 2. In this work, two or more trials were made to obtain a flash point of a material. As shown in Table 2, the difference between the results from different trials on identical materials is less than 1.0 °C. The temperature measurement unit that includes a thermocouple and a thermometer was calibrated using an ice bath. The temperature of the ice bath was measured using a liquid thermometer and the reading was 0 °C. The reading on the temperature measurement unit was 1.6 °C. Therefore, the measured flash point values were corrected by subtracting 1.6 °C from the readings of the temperature measurement unit. If the pressure at which the flash point was measured

Table 2
Flash points of some liquids in air and oxygen measured using the ECB apparatus and standard closed cup testers

| Liquids | Flash point in air (present work) (°C) | Flash point in oxygen (present work) (°C) | Flash point in air (reference) (°C) |
|---|---|---|---|
| Methyl alcohol | 7.0 to 7.7 | 7.3 to 8.1 | 11.0 [10] |
| Ethyl alcohol | 11.3<br>12.4[a] | 11.0 to 11.1 | 13.0 [10] |
| Isopropyl alcohol | 10.5 to 10.6 | 8.7 to 9.6 | 12.0 [10] |
| Toluene | 3.9 to 4.0 | 1.9 to 2.6 | 4.0 [10] |
| Tetrahydrofuran | −20.8 to −20.6 | −20.6 | −17.0 [10] |
| Ethyl acetate | −6.9 to −6.8 | −6.4 to −5.9 | −4.0 [10] |
| Dichloromethane | _[b] | −7.1 | 30.0 [15]<br>102.8 [16] |

[a] Measured using the Pensky–Martens closed cup tester.

[b] The flash point of dichloromethane in air was found to be dependent on ignition energy. This dependence is being investigated and will be reported in a subsequent paper.

was different from one atmosphere, the effect of pressure on the flash point was corrected using Eq. (1).

### 3.1. Advantages of the "equilibrium closed bomb" (ECB) method

Any special oxidant atmospheres can be conveniently established in the ECB provided the bomb material is compatible with the test atmosphere. Moreover, the flash points of all tested materials measured in the ECB are lower than those published in NFPA 325 [10]. For comparison, the flash point of ethyl alcohol in air obtained using the ECB and the Pensky–Martens closed cup test method were 11.3 and 12.4 °C, respectively.

In an equilibrium tester, e.g. the ECB, a lower flash point would be expected than in a non-equilibrium tester. This is due to the following reasons. Firstly, since the temperature of a non-equilibrium closed cup tester is increased at a finite heating rate, the vapor may not be in equilibrium with the liquid sample. Consequently, a higher temperature would be required for a non-equilibrium closed cup tester than for equilibrium apparatus in order to reach the same vapor concentration. Secondly, while the standard flash point test methods employ downward and horizontal propagation of flame, the ECB apparatus employs upward flame propagation (the electrodes are located at the lower part of the test apparatus). Flame propagation in downward and horizontal directions generally requires slightly higher vapor concentration than is required for upward flame propagation [8]. This means again that the flash point measured in a standard non-equilibrium tester will generally be higher than the flash point measured in the ECB.

At the flash point temperature, the saturated vapor concentration of a flammable/combustible liquid is equal to its LFL:

$$\text{LFL} = \frac{P_{\text{FP}}}{760} \tag{2}$$

where LFL (%v/v) and $P_{\text{FP}}$ (mmHg) are the lower flammability limit and vapor pressure at the flash point, respectively. This relationship can be used to check the accuracy of the flash

Table 3
Lower flammability limits in air and oxygen

| Liquids | LFL in air (measured) (%v/v) | LFL in oxygen (measured) (%v/v) | LFL in air (reference) (%v/v) | LFL in air (calculated) (%v/v) | LFL in oxygen (calculated) (%v/v) |
|---|---|---|---|---|---|
| Methyl alcohol | 6.71 | 6.43 | 6.0 [10] | 6.02 | 6.13 |
| Ethyl alcohol | 3.57[a] | 3.60[b] | 3.3 [10] | 3.39 | 3.32 |
| Isopropyl alcohol | 2.33 | ND | 2.0 [10] | 2.35 | 2.08 |
| Toluene | 1.38 | ND | 1.1 [10] | 1.14 | 1.01 |
| Tetrahydrofuran | 1.73 | 1.58[c] | 1.8 [10] | 1.82 | 1.84 |
| Ethyl acetate | 1.93 | 2.16[c] | 2.2 [10] | 2.06 | 2.13 |
| Dichloromethane | –[d] | 10.7[e] | 13.0 [13] | – | 13.0 |

The measured values in Columns 1 and 2 were obtained at 30 °C unless otherwise indicated. Values reported in last two columns were calculated from lowest flash point data measured in the ECB apparatus shown in Table 2.

[a] 33–37 °C.
[b] 30–38 °C.
[c] 50–60 °C.
[d] The LFL for dichloromethane was found to be dependent on ignition energy. This dependence is being investigated and will be reported in a subsequent paper.
[e] 18–20 °C.

point measurements. The LFLs of the tested liquids in air and oxygen were measured in this work as shown in Table 3. Using the measured flash points in air and oxygen obtained in the ECB apparatus and the vapor pressures of the liquids [11], the LFLs in air and oxygen were calculated and the results are presented in Table 3.

For methyl alcohol, ethyl alcohol, isopropyl alcohol and toluene, the LFLs in air measured in this work are slightly higher than the LFLs reported in NFPA 325 [10]. While for ethyl acetate and tetrahydrofuran, the measured LFLs of this work are lower. The data presented in NFPA 325 [10] were selected from different sources and the conservative values are selected where differences exist in reference sources. Therefore, the calculated LFLs in air were compared with the literature values or measured LFLs of this work, whatever is lower. The calculated LFLs in air deviate from the lowest measured LFLs by less than 6.7% except for isopropyl alcohol, the deviation is 18%. For LFLs in oxygen, the calculated LFLs for ethyl acetate, methyl alcohol and ethyl alcohol deviate from the measured LFLs by 1.4, 5 and 8%, respectively. For tetrahydrofuran, the deviation is 17%. This general agreement between the calculated and the measured LFLs in air and in oxygen supports that the flash point measured in the ECB apparatus are generally lower than the flash point measured in a non-equilibrium tester.

Another advantage of the ECB is that the diameter and volume of the ECB are larger than those of the standard closed cup methods (Table 1). This enables mixtures to be tested that would otherwise not be possible with the standard closed cup methods. The absence of a flash point does not ensure freedom from flammability [8]. Included in this category are materials that require large volumes for flame propagation, such as trichloroethylene. These materials will not propagate a flame in apparatus of the size of the standard flash point testers, however, their vapors are flammable and will burn when ignited in a vessel of adequate size. Some materials having very dense vapors, a narrow range of flammability, or the requirement for being somewhat superheated to burn will not exhibit a standard flash

point but can form flammable vapor–air mixtures if heating and mixing are optimized. Notably, a closed vessel with an adequate size is required for all of these materials in order to determine the flash point.

### 3.2. The effect of oxygen concentration on flash point and LFL

As in Table 2, the oxygen concentration has little effect on the flash points of the tested liquids with the exception of dichloromethane, as discussed later. Consistently, the LFLs of the flammable liquids measured in oxygen are essentially the same as the LFLs in air as shown in Table 3. The LFL of dichloromethane in oxygen measured using the method of ASTM E681 [12] is 10.7%, which is lower than its LFL in air (13%) reported in NFPA 49 [13]. The LFL of dichloromethane in air was found to be dependent on the energy of the ignition source.

A reason for the insensitivity of the LFL to oxygen concentration is that in order for flame to propagate in a gaseous fuel/oxygen/nitrogen mixture, the heat released from the burned layer must be sufficient to raise the temperature of the unburned gas mixture above the ignition temperature. In other words, the product of the vapor concentration (i.e. LFL) and the heat of combustion should be equal to or greater than the heat that is required to heat the unburned mixture above the ignition temperature, which is determined by the heat capacity of the mixture. When the fuel concentration is close to the LFL (typically under 5%), the heat capacity of the mixture is mainly determined by those of oxygen and nitrogen. Furthermore, since oxygen and nitrogen have similar heat capacities, the heat capacity of the fuel/oxygen/nitrogen mixture is not affected by the change of the oxygen concentration near the LFL. Therefore, the change of oxygen concentration has little effect on the LFL of a fuel/oxygen/nitrogen mixture as long as the oxygen concentration is in stoichiometric excess.

### 3.3. Flammability of dichloromethane

Inconsistent flammability data has propagated confusion about the relative safety of dichloromethane and in fact it is classified as a non-flammable liquid in 29 CFR 1910.106, Section 8 [14]. "Non-flammable" in this case means that it is neither a flammable liquid nor a combustible liquid. The NFPA defines liquids having a closed cup flash point below 37.8 °C (100 °F) as flammable liquids; and liquids having a closed cup flash point at or above 37.8 °C (100 °F) as combustible liquids. Since dichloromethane has been known to be combustible, it should be classified at least a "combustible liquid". It has been known that dichloromethane can form flammable vapor–air mixtures in larger volumes in a confined space and its LFL and UFL in air have been reported to be 13 and 23% by volume, respectively [13]. However, no flame has been observed in standard closed cup testers [13]. On the contrary, dichloromethane had been ignited in oxygen in the ECB apparatus using the 10 kV, 2.5 mA ignition source at temperatures as low as −7.1 °C. Dichloromethane in air had not been ignited in the ECB using the same ignition source, but ignited in 5 and 20 l test vessels at ambient temperatures with more energetic ignition sources.

The inconsistency in reported flash point data for dichloromethane in the literature is dramatic. Dichloromethane has no flash point in standard closed testers [13]. In 1968,

Kuchta et al. [15] reported that temperatures above 25 °C were needed to form flammable vapor–air mixtures. Coffee et al. [16], only 4 years later, published flammability diagrams of dichloromethane in air and other atmospheres and reported that the lowest temperature at which an ignition of dichloromethane–air mixture could occur was above 102.8 °C (217 °F). In the present work, the flash point of dichloromethane in oxygen was measured to be −7.1 °C. In 1983, Downey [17] estimated that dichloromethane is flammable at temperatures as low as −9 °C, which is lower than all the reported flash point values including the flash point in oxygen (−7.1 °C) measured in this work.

The primary cause for the discrepancies of reported flammability data for dichloromethane, in particular, we believe, is variation in ignition sources and energies used in the experiments. First, the type of ignition source used in the experiments may affect the results of flash point measurements. Standard closed cup test methods use a small hydrocarbon gas flame as the ignition source, but no ignition of dichloromethane–air mixture has been observed in standard closed cup testers. Kuchta et al. [15] used electric sparks and fuse wire as the ignition source. Coffee et al. [16] used 12 kV arc as the ignition source. In the present work, a continuous electrical arc produced by 10 kV, 2.5 mA dc power source was used as the ignition source.

Second, the energy of the ignition source can affect the ignition and thus the flash point of dichloromethane. The exact energy of the ignition sources used in the above references is not known. The minimum ignition energy (MIE) for most commonly used non-chlorinated liquids and flammable gases is of the order of less than a few millijoules. The MIE is much higher for chlorinated liquids. Because most commonly used ignition sources have energies well above a few millijoules, the energy of the ignition source does not affect the measured flash point for most flammable/combustible liquids. For chlorinated liquids, ignition energy appears to be very important. Preliminary tests have confirmed that the flash point of dichloromethane is dependent on the energy of the ignition source. Further work is currently underway to calibrate the ignition source used in the ECB apparatus and to quantify the effect of ignition energy on flash point determination for dichloromethane.

The wall of the vessel serves as heat sink for the combustion. For a flame to propagate, the heat released from the combustion must surpass the heat loss at the wall so that the unburned fuel-oxidant mixture ahead of the flame front can be heated to the ignition temperature. The larger the ratio of surface area to volume, the greater the quenching effect. If the volume of the vessel is sufficiently small, flame can be quenched due to the increased heat loss on the wall. On the other hand, the heat of combustion of the combustible also affects the quenching effects on flame and the smaller the heat of combustion the larger the vessel volume below which flame can be quenched. The heat of combustion for dichloromethane (106.8 kcal/gmol) is low compared with other liquids tested, e.g. 173.64 kcal/gmol for methyl alcohol and 326.68 kcal/gmol for ethyl alcohol [18]. Therefore, the influence of vessel volume on the flash point of dichloromethane needs to be investigated.

Since dichloromethane is labeled as non-combustible, it is often regarded as an inert chemical and is sometimes mixed with flammable liquids in order to suppress the flammability of the flammable liquid. Actually, mixing dichloromethane with a small quantity of a flammable liquid will significantly reduce the minimum ignition energy of the vapor

mixture. As a result, the vapor mixture of dichloromethane and the flammable liquid can be ignited by ignition sources that are not sufficient to ignite pure dichloromethane vapor. For example, Coffee et al. [16] determined that dichloromethane becomes flammable at 10 °C and 1 atm at a vapor concentration of 17% (by volume) with the addition of less than 0.5% (by volume) of methyl alcohol. Thus, mixing dichloromethane with methanol renders the entire mixed-liquid system flammable and significantly more sensitive to ignition than pure dichloromethane. Therefore, the flammability hazards of dichloromethane should be assessed not only based on the flash point but also taking into account of the energy of potential ignition sources and whether it is presented together with other flammable or combustible materials.

## 4. Conclusions

(1) The "equilibrium closed bomb" (ECB) apparatus provides reliable and accurate flash point data for various oxidant atmospheres with the following advantages:
   (a) In the ECB, flash point is measured under equilibrium condition.
   (b) It has a relatively large volume compared with most of the standard flash point measurement apparatus. Therefore, it can be used to measure the flash points of those materials whose flash points may not be able to be measured using other closed cup methods due to the quenching effects of the small vessel diameter on flame.
   (c) It is safer to measure the flash points of materials in hazardous oxidant atmospheres such as oxygen, chlorine and oxides of nitrogen as the ECB is totally closed.
   (d) It can be used to measure the lower temperature limit of flammability defined in ASTM E1232 [9] in special oxidant atmospheres and at higher pressures.

   For the flammable liquids tested, the LFLs calculated using the flash points measured in the ECB and the vapor pressure data [11] are generally in good agreement with the lowest LFLs reported in the literature and in this paper thus providing an internal check of the flash point measurements in the ECB.
(2) The present test results show that the flash points of the tested liquids (except for dichloromethane) are insensitive to oxygen concentration as long as the oxygen concentration is in stoichiometric excess.
(3) The flash point of dichloromethane in oxygen measured in the ECB is −7.1 °C. However, the flash point of dichloromethane in air has been shown to be dependent on the energy of the ignition source. Further studies to quantify the dependence of the flash point of dichloromethane on the energy of the ignition source are currently being carried out. The potential influence of vessel volume on the flash point will also be investigated.

Based on the available flammability data, dichloromethane should at least be treated as combustible liquid. In addition to temperature, the energy of potential ignition sources and the presence or absence of other flammable/combustible materials are important factors in the assessment of the flammability hazards associated with use of dichloromethane.

## Acknowledgements

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