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Experimental Determination and Re-examination of the Effect of Initial Temperature on the Lower Flammability Limit of Pure Liquids

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The lower flammability limits of 18 $C_xH_yO_zN_w$ liquids were measured as a function of initial temperature in an ASHRAE 12 L style apparatus. Results indicate that the calculated adiabatic flame temperature is not constant, as previously reported but rather decreases with increasing temperature. Consequently, the modified Burgess−Wheeler law does not accurately predict the effect of temperature on the lower flammability limit. Though few direct comparisons are possible, previously reported data agree well with the values measured in this study.

Introduction

Knowledge of the combustion potential of a chemical is crucial when designing safe chemical processes. The lower flammability limit (LFL), the lowest concentration of fuel in air that will support flame propagation, is perhaps the most useful property to consider when dealing with gases and liquids. Although LFL decreases with increasing initial temperature, flammability data as a function of initial temperature are rare, the large majority of the reported data having been determined at 298 K for gases and at a single arbitrary elevated temperature for liquids. These single points are often corrected for initial mixture temperature using what has become known as the modified Burgess−Wheeler law. This work reports LFL measurements at temperatures up to 500 K for a variety of $C_xH_yO_zN_w$ liquids and examines the validity of the modified Burgess−Wheeler law.

Experimental Method

Measurements were made in an ASHRAE style apparatus, as set forth in Appendix A of ASTM E 681[1] (Figure 1). The spherical 12 L flask was heated to the test temperature by an oven that regulated the temperature within the flask within ± 2 K. The flask was evacuated, and a specified volume of liquid sample was injected.

Once the sample was completely evaporated, air was slowly introduced through the inlet until the flask reached atmospheric pressure, sweeping any sample droplets from the inlet into the flask. The mixture was agitated for at least 7 min while the glassware above the oven was heated to avoid condensation. Two thermocouples, one positioned just below the neck of the flask and the other near the center, were used to verify achievement of thermal equilibrium within the flask.

Shortly before ignition, the stirring mechanism was shut off, and the mixture was allowed to rest to eliminate turbulence. The clamps on the flask cover were released to allow venting during ignition. Ignition of the mixture was attempted by a 15 kV (30 mA) power supply attached to tungsten electrodes 6.4 mm apart, one-third of the flask's height from the bottom, with a spark duration limited to 0.4 s. The ASHRAE 90° flame propagation criterion[2,3] was utilized to distinguish between a flammable and a nonflammable mixture.

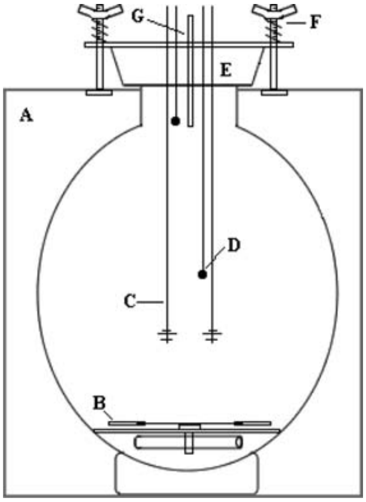

Figure 1. Schematic for 12 L flammability apparatus: A, oven; B, magnetic stir mechanism; C, electrode; D, thermocouple; E, silicon stopper; F, spring-loaded cover clamp; G, air/fuel inlet.

This process was repeated, varying the sample injection volume by (5 ± 2) mm^3 until consecutive trials resulted in both a flame that fulfilled the flame propagation criterion (commonly called a "go" trial) and one that did not (commonly called a "no go" trial). The lower limit was calculated from the average injection volume of these two tests, $\bar{V}_{\text{inj}}$, assuming ideal gas behavior:

$$\text{LFL} = \frac{\rho \bar{V}_{\text{inj}} RT}{PV_{\text{vessel}}} \tag{1}$$

where P is the lab pressure measured by a National Institute of Standards and Technology (NIST) traceable barometer (± 27 Pa), ρ is the liquid density at the temperature of the lab as given in the DIPPR 801 database,[4] R is the ideal gas constant, T is the test temperature, V_{vessel} is the calibrated volume of the flask, (12.59 ± 0.01) L, and the LFL is given in volume fraction, ϕ. The combined standard uncertainty was calculated for each measurement using the law of propagation of uncertainty, as outlined on the NIST Web site.[5]

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Table 1. Test Chemicals for LFL Measurements

chemical	CAS Number	purity	chemical	CAS Number	purity
1-hexyne	693-02-7	> 98 %	5-*o*-tolyl-2-pentene	6047-69-4	98 %
phenetole	103-73-1	99 %	isopropyl myristate	110-27-0	98 %
4-methyl-2-pentanol	108-11-2	99 %	methanol	67-56-1	99.80 %
2-methyl-1,3-propanediol	2163-42-0	99 %	butanol	71-36-3	99.90 %
dibutylamine	111-92-2	> 99.5 %	1-octanol	111-87-5	99 %
α-pinene	80-56-8	98 %	hexyl formate	629-33-4	> 98.5 %
2-nonanone	821-55-6	99 %	octyl formate	112-32-3	> 98 %
methyl benzoate	93-58-3	99 %	diisobutyl phthalate	84-69-5	99 %
decyl acetate	112-17-4	98 %	ethyl lactate	97-64-3	> 98 %

Table 2. Experimental LFL at Initial Mixture Temperature *T*

material	T/K	LFL/(100·ϕ)	material	T/K	LFL/(100·ϕ)
1-hexyne	342[a]	0.95 ± 0.01	5-*o*-tolyl-2-pentene	375	0.644 ± 0.009
	365	0.92 ± 0.01		423	0.537 ± 0.009
	448	0.79 ± 0.01		447	0.51 ± 0.01
	472	0.75 ± 0.01		471	0.48 ± 0.02
phenetole	355	0.949 ± 0.008	isopropyl myristate	450	0.377 ± 0.007
	379	0.888 ± 0.008		446	0.389 ± 0.004
	453	0.75 ± 0.01		471	0.426 ± 0.005
	481	0.72 ± 0.01		496	0.383 ± 0.005
4-methyl-2-pentanol	345	1.13 ± 0.01	methanol	301	7.03 ± 0.07
	374	1.06 ± 0.01		333	6.79 ± 0.04
	422	0.99 ± 0.01		372	6.43 ± 0.04
	471	0.91 ± 0.01		420	6.08 ± 0.05
2-methyl-1,3-propanediol	422	1.64 ± 0.02	butanol	328	1.67 ± 0.01
	447	1.56 ± 0.02		372	1.57 ± 0.02
	471	1.50 ± 0.01		422	1.48 ± 0.01
	496	1.43 ± 0.02		470	1.35 ± 0.02
dibutylamine	341	0.803 ± 0.007	1-octanol	375	0.82 ± 0.01
	365	0.731 ± 0.008		398	0.77 ± 0.01
	448	0.608 ± 0.008		470	0.68 ± 0.01
	472	0.580 ± 0.008		490	0.65 ± 0.02
α-pinene	351	0.680 ± 0.007	hexyl formate	352	1.03 ± 0.01
	374	0.644 ± 0.007		376	0.98 ± 0.01
	423	0.58 ± 0.01		423	0.89 ± 0.01
	472	0.52 ± 0.01		472	0.82 ± 0.01
2-nonanone	376	0.767 ± 0.008	octyl formate	376	0.777 ± 0.009
	400	0.713 ± 0.007		423	0.698 ± 0.009
	448	0.656 ± 0.008		474	0.62 ± 0.01
	473	0.68 ± 0.01	diisobutyl phthalate	452	0.496 ± 0.007
	483	0.686 ± 0.007		473	0.440 ± 0.005
	493	0.68 ± 0.02		493	0.414 ± 0.007
methyl benzoate	374	1.04 ± 0.01	ethyl lactate	352	1.64 ± 0.02
	399	0.97 ± 0.01		374	1.55 ± 0.01
	451	0.86 ± 0.01		447	1.37 ± 0.01
	472	0.82 ± 0.01		471	1.30 ± 0.02
decyl acetate	400	0.598 ± 0.006			
	424	0.556 ± 0.009			
	447	0.516 ± 0.007			
	472	0.502 ± 0.007			
	491	0.533 ± 0.009			

[a] A maximum error of ± 2 K is associated with measured temperatures.

At least three repetitions were performed at each temperature. Between runs the vessel was flushed with air and evacuated at least six times to remove remaining fuel and combustion products. The minimum purity for the liquids used in this study was 98 % (Table 1).

Results

Table 2 lists the experimental results for all 18 compounds, including the combined standard uncertainty at each temperature. As reported by Zabetakis,[6] LFL decreased linearly with increasing temperature for most compounds. There were three notable exceptions to this rule (Figure 2). LFL data for 2-nonanone and decyl acetate became nonlinear at temperatures exceeding (450 and 470) K, respectively. This change in behavior at elevated temperatures was thought to indicate the onset of slow autooxidation. Subsequent trials for decyl acetate at 492 K showed that doubling the mixing time raised the experimental LFL by at least 10 %, though the exact LFL was difficult to determine as the flame behavior became inconsistent between these trials. This result appeared to be consistent with our hypothesis. Extending the mixing time had no measurable effect, however, on the LFL of 2-nonanone at 472 K. It is possible that at some temperature between (450 and 480) K 2-nonanone decomposes into a mixture of flammable gases and thus exhibits a different flammability limit at higher temperatures. However, to our knowledge, no decomposition temperature has been reported for 2-nonanone.

The LFL of isopropyl myristate exhibited a unique relationship with temperature. At the two points below 470 K, the difference between a “go” and a “no go” run was less distinct

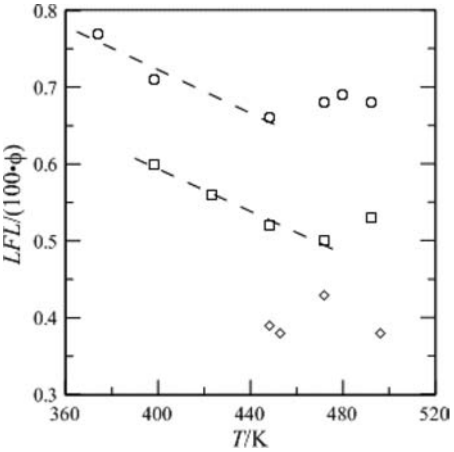

Figure 2. LFL data versus temperature for: ○, 2-nonanone; □, decyl acetate; ◇, isopropyl myristate. Linear fits are given by the dashed line.

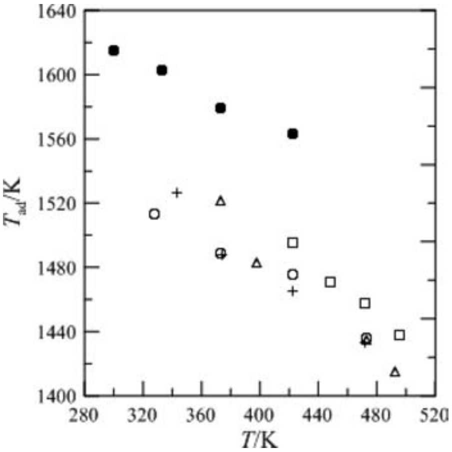

Figure 3. Calculated adiabatic flame temperature, T_{ad}, from experimental lower limit data as a function of initial mixture temperature: ●, methanol; ○, butanol; +, 4-methyl-2-pentanol; △, 1-octanol; □, 2-methyl-1,3-propanediol.

than with other compounds, and mixing time appeared to have a small effect on the measured values. Above 470 K, however, the distinction between a "go" and "no go" run became larger than with any of the other compounds, and the flammability limit was no longer affected by differences in mixing time. This sudden shift in flame behavior may be explained by the slow decomposition of isopropyl myristate below 470 K. Above 470 K, the decomposition reaction is complete, and the limits become independent of mixing time. Bonhorst et al.[7] reported a decomposition temperature for isopropyl myristate between (465 and 473) K.

Discussion

Adiabatic Flame Temperature. Analysis and prediction of flammability limits often employs the calculation of the adiabatic flame temperature (T_{ad}), the theoretical temperature of the flame assuming no heat loss, that is:

$$\sum_{\text{products}} H_i(T_{ad}) = \sum_{\text{reactants}} H_i(T^o) \tag{2}$$

where T^o is the initial mixture temperature and H is the enthalpy of species i. The combustion products are typically based on complete combustion with no disassociation:

$$C_xH_yO_zN_w + \left(x + \frac{y}{4} - \frac{z}{2}\right)O_2 = xCO_2 + \frac{y}{2}H_2O + \frac{w}{2}N_2 \tag{3}$$

White[8] and Zabetakis et al.[9] found that the calculated adiabatic flame temperature of a lower limit mixture was independent of the initial mixture temperature. Figure 3 shows T_{ad} as a function of initial mixture temperature for the alcohols studied in this work. Thermodynamic data used in calculating the flame temperature were taken from the DIPPR801 database.[4]

Contrary to the findings of White[8] and Zabetakis et al.,[9] T_{ad} is not independent of the initial mixture temperature but decreases significantly with increasing initial temperature. This is true for all compounds studied, though the magnitude of the effect of initial temperature varies by chemical. The majority of published data also correspond to flame temperatures that decrease with increasing initial temperature (Table 3). Exceptions to this rule were determined preponderantly in vessels with diameters less than 6 cm.

The effect of apparatus diameter on the slope of T_{ad} is easily explained by the relationship between the slopes of the LFL and T_{ad} versus temperature curves. A decrease in the LFL results in an increase of excess oxygen and nitrogen present during combustion and, accordingly, an increase in the specific heat of the products. Consequently, the slope of the T_{ad} temperature curve is directly related to the slope of the LFL, which, as White[8] observed, becomes more negative with increasing vessel diameter. According to Table 3, the vessel diameter affects the measured temperature dependence of the LFL for diameters at least up to 6 cm. The limiting diameter is likely larger, though the value likely depends on the selected flame-propagation criterion, with criteria requiring stronger flames also requiring larger diameters.

Modified Burgess−Wheeler Law. Burgess and Wheeler[26] showed that the heat liberated by a mole of a lower limit mixture at ambient temperature and pressure was approximately constant (Burgess−Wheeler law). On the basis of the findings of a constant adiabatic flame temperature, Zabetakis et al.[9] attempted to extend the law of Burgess and Wheeler by adding the enthalpy required to raise a limit mixture from ambient temperature to the initial test temperature:

$$\text{LFL} \cdot (-\Delta H_c) + c_p \Delta T = k \tag{4}$$

where c_p is the specific heat of the fuel−air mixture and ΔH_c is the heat of combustion of the fuel. When the LFL is known at a given temperature, T_0, eq 4 may be rewritten as:

$$\frac{\text{LFL}(T)}{\text{LFL}(T_0)} = 1 - \frac{c_p \cdot 100}{\text{LFL}(T_0) \cdot (-\Delta H_c)}(T - T_0) \tag{5}$$

For many hydrocarbons, this approximately corresponds to a 7 % decrease in LFL per 100 K, relative to the LFL at 293 K. The modified Burgess−Wheeler law (eq 5) works reasonably for data obtained in small-diameter vessels but underestimates the effect of temperature on the LFL for the experimental data obtained in this work (Figure 4).

Comparison with Reported Data. Equation 5 is often expressed in the more general form:

$$\frac{\text{LFL}(T_1)}{\text{LFL}(T_0)} = 1 - \frac{c}{100}(T_1 - T_0) \tag{4}$$

where c represents the decrease relative to the LFL at T_0, 293 K, per 100 K increase, that is, $c \approx 0.07\ \text{K}^{-1}$ according to the modified Burgess−Wheeler law. Gibbon et al.[15] found c to vary between (0.11 and 0.18) K^{-1}, while Goethals et al.[22] and Brandes et al.[13] found c values between (0.13 and 0.23) K^{-1}.

Table 3. Effect of Initial Temperature T on Calculated Adiabatic Flame Temperatures T_{ad} for Reported Lower Limit Data

name	ref	vessel[a]	T range/K	direction of calculated T_{ad} with increasing T
methanol	10	A	200	decreasing
	11	A	150	decreasing
	12	B	150	decreasing
	13	C	160	decreasing
	14	D	275	increasing
ethanol	10	A	200	decreasing
	11	A	150	decreasing
	15	E	200	constant
propanol	15	E	200	decreasing
butanol	11	A	150	decreasing
	13	C	80	decreasing
1-octanol	13	C	60	decreasing
acetone	11	A	150	decreasing
2-butanone	16	F	185	constant
	17	G	100	decreasing
methane	18	H	680	constant
	8	I	430	constant
pentane	8	I	280	constant
isopentane	11	A	150	decreasing
hexane	19	J	175	increasing
heptane	19	J	175	constant
octane	19	J	175	constant
2,2,4-trimethylpentane	19	J	175	constant
nonane	19	J	160	decreasing
decane	19	J	150	constant
cyclohexane	20	A	150	decreasing
cyclohexene	20	A	150	decreasing
benzene	21	B	230	decreasing
	20	A	150	decreasing
toluene	20	A	150	decreasing
	16	F	170	weakly decreasing
	21	B	230	decreasing
	22	K	200	decreasing
	23	A	100	decreasing
	23	L	175	constant
	23	M	200	decreasing
o-xylene	21	B	200	decreasing
tetrahydrofuran	16	F	185	constant
ethylene	8	I	380	constant
acetylene	8	I	280	constant
hydrogen	8	I	380	increasing
	8	N	380	increasing
ammonia	24	N	580	constant
carbon monoxide	8	I	380	decreasing
difluoromethane	25	O	70	decreasing
1,1-difluoroethane	25	O	70	weakly decreasing

[a] A: 9 cm × 45 cm cylinder; downward flame propagation. B: 7.5 cm × 150 cm tube. C: EN 1839 (T); 8 cm × 30 cm tube. D: 7.5 L spherical bomb; determined at 150 kPa. E: 13 L spherical bomb. F: 5 cm × 150 cm tube. G.: 7.6 cm × 180 cm steel tube. H: 2 cm × 15 cm closed tube; downward flame propagation. I: 2.5 cm × 150 cm tube; downward flame propagation. J: 5.7 cm × 122 cm tube. K: DIN 51649; 6 cm × 30 cm tube. L: 10.2 cm × 96 cm closed tube. M: 30.6 cm × 39 cm tube. N: 25 cm × 25.7 cm closed tube. O: ASTM E 681; 5 L sphere.

Data measured in this work correspond to c values between (0.12 and 0.25) K^{-1} (Table 3), though a lack of previously reported data only permits a direct comparison of experimental data for methanol, butanol, and 1-octanol (Figures 5 and 6).

Several striking similarities exist between reported and experimental data for methanol. The data reported by Briand et al.[11] (apparatus type A) and Fiumara[12] (apparatus type B) closely resemble the data measured in this work. These data also exhibit a similar slope with the data of Yannaquis[10] (apparatus type A) and the trend calculated from the data point and slope reported by Brandes et al.[27,13] (apparatus type C). The apparatus types are defined in Table 3. Data for butanol and 1-octanol also show similar slopes to those reported by Brandes et al.

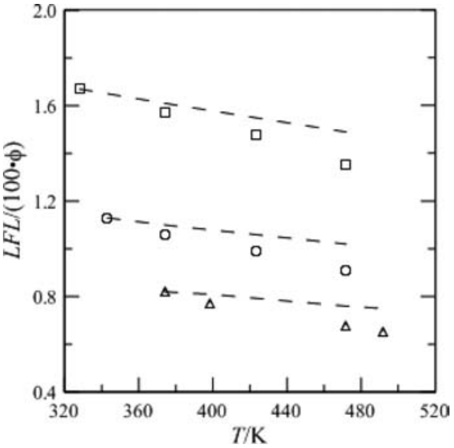

Figure 4. Effect of temperature on the lower limit as calculated by the modified Burgess−Wheeler law (eq 5), - - -, compared with experimental data for: ○, 4-methyl-2-pentanol; □, butanol; △, 1-octanol.

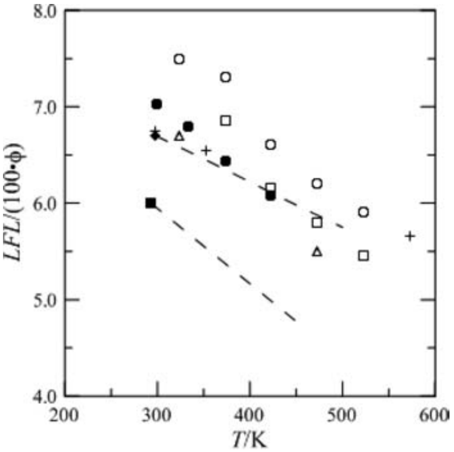

Figure 5. Experimental LFL data for methanol: ○, ref 10; □, ref 11; △, ref 12; ■- - -, calculated from a single data point from ref 27 and reported slope from ref 13; ♦- - -, ref 6 and calculated using the modified Burgess−Wheeler law (eq 5); +, ref 14; ●, this work.

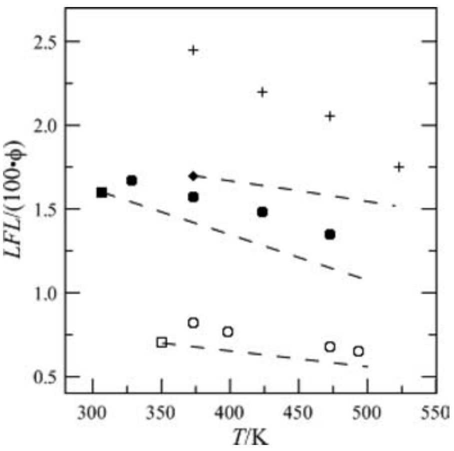

Figure 6. Experimental lower limit data for butanol (●, ■, ♦, +) and 1-octanol (○, □): ■/□- - -, calculated from a single data point from ref 27 and reported slope from ref 13; ♦- - -, ref 6 and calculated using the modified Burgess−Wheeler law (eq 5); +, ref 11; ●, ○, this work.

It appears likely that the offset between these data sets is, for the most part, merely a function of the propagation criteria utilized and the direction of flame travel. That the work of

Fiumara[12] closely resembles the data reported in this work is unsurprising, as the 90° ASHRAE propagation criterion was specifically created to yield results equivalent with full flame propagation in large tubes.[2,3] The EN 1839 method employed by Brandes et al.[27,13] requires only flame detachment and minimal flame propagation for a fuel/air mixture to be considered flammable, and so as should be expected, the LFL values are lower than those measured in this work, though parallel. It is also unsurprising that the LFL values of Yannaquis[10] are significantly higher than those reported here, as downward flame propagation was used rather than the upward propagation used in this work. What is surprising is the discrepancy between the results of Yannaquis[10] and Briand et al.[11] Both studies supposedly employed the same experimental method, yet the LFL values of Yannaquis are significantly higher.

Conclusions

This new set of LFL data and subsequent analysis show that the adiabatic flame temperature decreases with increasing initial temperature, contrary to the constant adiabatic flame temperature theory previously suggested in the literature. Consequently, the widely used modified Burgess−Wheeler law was shown to significantly under-predict the effect of temperature on the LFL. A new method to estimate the effect of temperature on the lower limit is needed.

The lower limit was found to decrease relative to the LFL at 293 K by (12 to 25) % per 100 K increase, which agrees closely with data obtained using other large-diameter apparatuses. Specifically, inspection of flammability curves for methanol, butanol, and 1-octanol show remarkable agreement with the slopes obtained using other large-diameter apparatuses. This agreement between measurements obtained in different geometries is encouraging, as heretofore the flammability limit was thought to be a function of the apparatus. If the offset between LFL data sets from large-diameter vessels can in fact be explained by differences in flame propagation criteria and propagation direction, this could imply the existence of a fundamental flammability limit.

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