Auto-ignition of CH_4/air, C_3H_8/air, CH_4/C_3H_8/air and CH_4/CO_2/air using a 1 l ignition bomb

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Abstract

The distinction between auto-ignition and hot-surface ignition of a given gas is emphasized. In ideal auto-ignition there is no diffusion of heat or matter.

Published information on auto-ignition temperatures (AIT) of multi-component fuels in air is scarce. This also applies to North Sea natural gas, of which CH_4, higher alkanes and CO_2 are essential components.

In the present experimental laboratory-scale study, AIT of four types of hydrocarbon mixtures (CH_4/air, C_3H_8/air, CH_4/C_3H_8/air and CH_4/air/CO_2) have been measured using a 1 l ignition bomb. The experimental method ensured that the gas mixtures studied were of known composition and homogeneous in concentration. The gas mixture was admitted to the pre-evacuated ignition bomb in the form of a turbulent jet when the bomb wall had reached the desired temperature. Ignition was recognized as a sudden pressure rise in the bomb a few seconds after the gas flow into the bomb had stopped.

The minimum AITs for CH_4/air and C_3H_8/air were found to be 640 °C and 500 °C, respectively. The AIT of CH_4/C_3H_8/air decreased with increasing propane content and total fuel concentration. A fuel concentration region was discovered for which CH_4/C_3H_8/air and C_3H_8/air with the same ratio of propane to oxygen gave the same AIT. Reducing the oxygen content of a CH_4/air mixture by adding CO_2 gave, under the present experimental conditions, a systematic increase of AIT with increasing CO_2 content. The role of the CO_2 was probably essentially that of an inert diluent.

It has been known for a long time that the 'minimum hot-surface ignition temperature' is not a constant for a given gas mixture, but highly dependent, by several hundred degrees centigrade, on the dynamic state of the gas, the geometry and material of the ignition surface, and the mode of heat supply to the surface. The direct application of AIT values to assess industrial hot-surface ignition risks may therefore be unduly conservative. Consequently there is a need for general mathematical models that can predict minimum ignition temperatures for various practical situations in industry. Such models will have to contain sub-models of ignition chemistry, fluid mechanics and heat and mass transfer.

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1. Introduction

Literally 'auto-ignition' means 'ignition by itself'. In the present context it means the lowest temperature of a given quantity of an explosible premixed gas, at which the entire gas quantity ignites spontaneously. This implies that the mixture is ignited by the energy released by a chemical 'induction process' within the gas itself, and conditions must exist at which the pre-ignition chemical reactions can take place which finally lead to 'thermal explosion'. Normally heat must be supplied to the ignition zone during the early phase. The heat source can be a compression process or a hot surface embracing a well-stirred gas. Ideally, auto-ignition also implies that during the whole ignition process, the concentration of chemical species and the temperature are uniform throughout the mixture. The process thus contains chemical reaction only, transfer of species and heat being absent.

Ignition of premixed combustible gases, vapours and air by hot surfaces has long been recognized as a major cause of fires and explosions in the process industry handling such materials. However, as pointed out by Mullins [1], the hot-surface ignition temperature of a given gas mixture is not an unambiguous inherent property of that mixture. It also indeed depends on the dynamic state of the gas in relation to the hot surface, the hot-surface geometry, the way in which heat is supplied to the hot surface, and to some extent also on the material of which the hot surface is made. This is a process involving not only chemical reactions, but also heat and mass transfer within the mixture.

In most practical situations of hot-surface ignition, the majority of the bulk of the gas mixture is initially at ambient temperature, and the ignition process takes place in a comparatively thin preheated gas layer adjacent to the hot surface. The established flame front must therefore be able to propagate by itself into a gas at ambient temperature. Consequently, hot-surface ignition temperatures will generally be higher than auto-ignition temperatures for identical premixed gases. The difference can be very significant. This has been discussed by Eckhoff and Thomassen [2]. For example for propane in air, the auto-ignition temperature is lower than 500 °C, whereas the hot-surface ignition temperatures found by Alfert and Fuhre [3] were in the range 800–1000 °C, depending on the geometry of the hot surface.

Nevertheless, the auto-ignition temperature can serve as a conservative, relative measure of the 'minimum ignition temperature' of combustible mixtures. Whereas a fair amount of standard test data have been published on auto-ignition of single-component fuels in air, information on multi-component fuels in air is more scarce. In the present work, described in greater detail elsewhere by Kong and Alfert [4], auto-ignition temperatures (AITs) of mixtures of methane and propane in air, and of methane and CO_2 in air, all three gases being typical components of North Sea natural gas, were determined experimentally.

2. Apparatus and experimental procedure

According to Mullins [1] the present test method for measuring AIT can be categorized as a 'bomb method' because of the ignition space being virtually closed.

The apparatus is illustrated in Fig. 1. The entire system consists of three parts, i.e. the ignition bomb, the gas mixture preparation system and the diagnostics.

A standard stainless steel sampling cylinder was used as the ignition bomb. Its geometry and dimensions are given in Fig. 2. The internal volume is 1.0 l. The bomb was mounted inside a 10 kW oven with automatic temperature control.

In order to prepare a homogeneous gas mixture of the desired composition, the constant flow method was used to mix the various gases and air before admitting the
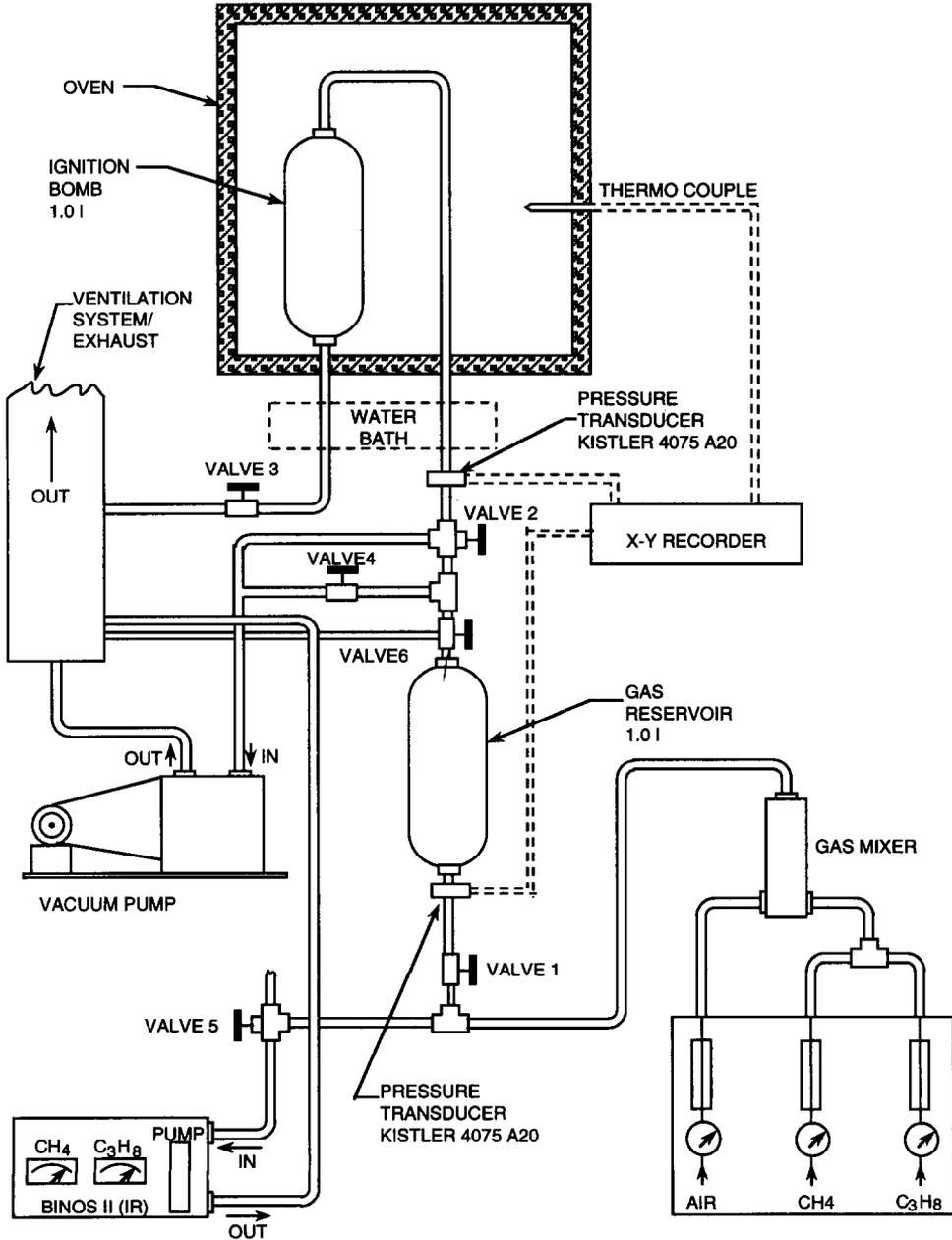
Fig. 1. Diagram of the 'bomb' apparatus used for determining auto-ignition temperatures of gas mixtures.
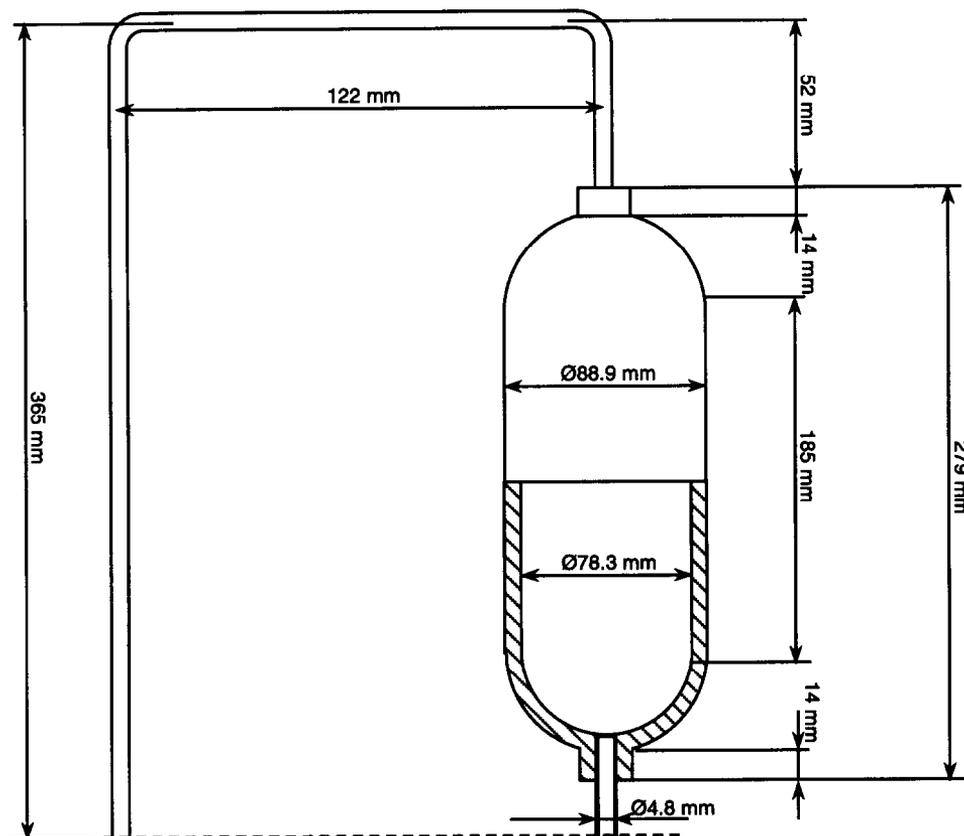
Fig. 2. Diagram of the 1 l ignition bomb.

mixture to the ignition bomb. The flows of gas and air were controlled by rotameters and manometers. Continuous mixing was accomplished in the gas mixer. The concentrations of methane and propane in the flow out of the mixer were monitored by an infrared gas analyser.

A thermocouple was used to monitor the oven temperature, whereas the pressure change in the ignition bomb, during the experiment, was recorded by a pressure transducer.

Details of the experimental procedure are given in the Appendix. The oven temperature was set at a value slightly below the expected AIT, as estimated from the literature or from results of preceding tests. The oven temperature was allowed to stabilize for at least 30 min. Fig. 3 shows a typical time history of pressures inside the ignition bomb and the gas reservoir during an experiment. Just before admitting premixed gas mixture from the reservoir to the bomb, the pressure in the reservoir (A) was typically 1.2–1.4 bar (abs) whereas vacuum was kept inside the ignition bomb (A'). Following the opening of valve 2, cold gas mixture flowed into the bomb, causing the bomb pressure to increase and the pressure in the reservoir to decrease. The
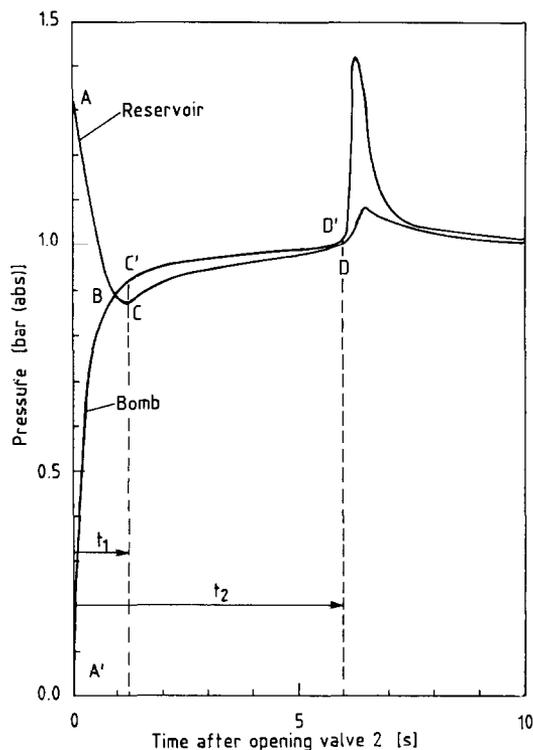
Fig. 3. Diagram of pressure–time histories in the bomb and reservoir during an auto-ignition experiment.

connection between the ignition bomb and the gas reservoir was left open. At about 1.2–1.5 s (time t_1) after valve 2 had been opened, the pressure in the reservoir reached its minimum (C) and started to increase. The reason is that the well-stirred cold gas having just entered the bomb, expanded due to being rapidly heated by the bomb wall, and started to flow back into the reservoir. The bomb pressure at this time (C') was typically 0.9 bar (abs). During the subsequent self-heating of the gas in the ignition bomb, the pressure there reached about 1.0 bar (abs) just prior to ignition.

The induction times (t_2) for ignition of the various gas mixtures investigated were mostly in the range 2.5–15 s depending on the mixture composition (see [4]). If ignition did not occur within 10 min, the test was terminated and another test was conducted at a higher bomb wall temperature. If ignition occurred within 10 min, the test was repeated at a lower temperature. The AIT was identified to within a bomb wall temperature interval of $\leqslant 5\,°C$. (Long-term irreversible changes of the apparatus caused a moderate systematic drift towards somewhat higher AIT values, as mentioned in the note at the end of Section 3.3.)

A theoretical analysis by Kong and Alfert [4] showed that the cooling of the internal ignition bomb wall by admitting the cold gas mixture to the evacuated bomb was negligible. Therefore the assumption that the internal bomb wall temperature at the moment of ignition was equal to the overall oven temperature is valid.

3. Experimental results

3.1. AITs of CH_4/air and C_3H_8/air

Measured AITs of mixtures of CH_4/air with equivalence ratios Φ_1 in the range 0.14–1.68, and of C_3H_8/air with Φ_3 in the range 0.168–4.04 are summarized in Fig. 4. The equivalence ratios Φ_1 and Φ_3 are defined as:

$$\Phi_1 = \left(\frac{V_{CH_4}}{V_{O_2}}\right)\bigg/\left(\frac{V_{CH_4}}{V_{O_2}}\right)_{\text{stoich.}} , \tag{1}$$

$$\Phi_3 = \left(\frac{V_{C_3H_8}}{V_{O_2}}\right)\bigg/\left(\frac{V_{C_3H_8}}{V_{O_2}}\right)_{\text{stoich.}} , \tag{2}$$

where V denotes volume percentage of the component indicated.

The non-linear dependence of AIT on fuel concentration is recognized clearly in Fig. 4. For CH_4/air the minimum AIT was 640 °C, in the range $V_{CH_4} = 3.0$–8.0 vol.%, which corresponds to $\Phi_1 = 0.30$–0.83. AIT increased to 680 °C when Φ_1 decreased to 0.145 or increased to 1.68.
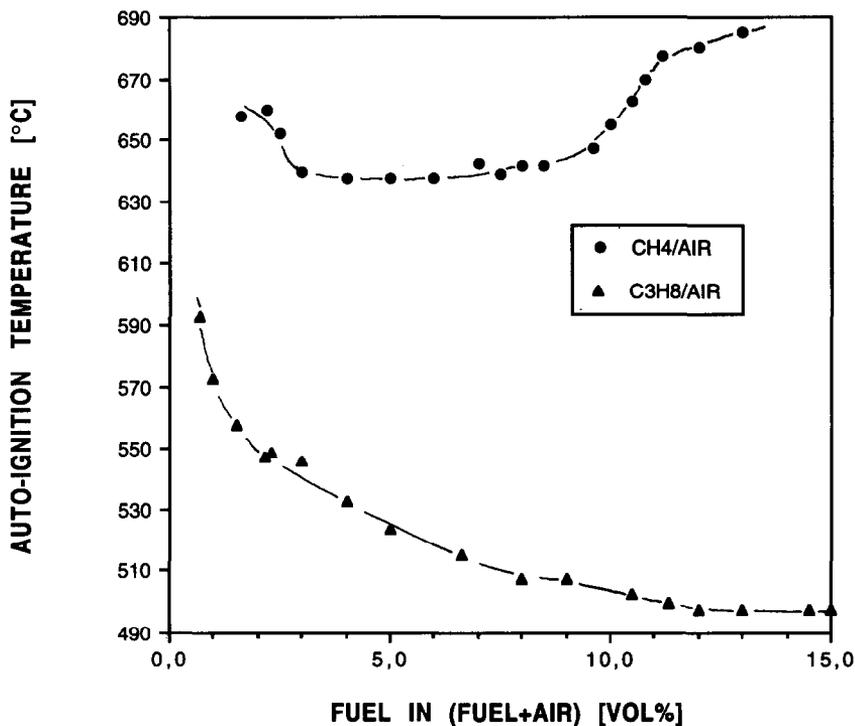
Fig. 4. Influence of fuel concentration on the auto-ignition temperatures of CH_4/air and C_3H_8/air mixtures in the 1 l ignition bomb.

For C_3H_8/air AIT decreased monotonically in the experimental range $V_{C_3H_8} = 0.7$–14.5 vol.%, corresponding to $\Phi_3 = 0.168$–4.04. The lowest AIT ($= 500$ °C) was found in the fuel-rich range $V_{C_3H_8} = 10.5$–14.5%, corresponding to $\Phi_3 = 2.79$–4.04.

These results show that the lowest AIT of CH_4/air occurs in the lean range of 3.0–8.0 vol.% CH_4, which is in part below the lower flammability limit at normal temperature. For C_3H_8/air the lowest values occur at very rich fuel concentrations, beyond the upper flammability limit at normal temperature. It is important to keep in mind that the flammable limits are widened when the initial temperature of the entire mixture is raised, as it will be in the closed-bomb apparatus used. In the present work the lower flammability limit of CH_4/air was found to be 1.5–2.0 vol.% at 680 °C, compared with 5.0 vol.% at 25 °C [5]. The explosible range of C_3H_8/air was widened from 2.1–9.5 vol.% at 25 °C [5] to a lower limit of less than 1.0 vol.% and an upper limit of more than 15% at 680 °C. Table 34 of Ref. [6] confirms this trend for the lower flammability limit.

3.2. AIT of CH_4/C_3H_8/air mixtures

The measured values of AIT of CH_4/C_3H_8/air at various overall equivalence ratios and fuel ratios are summarized in Fig. 5. As can be seen, AIT decreases monotonically with increasing overall equivalence ratio Φ and with increasing propane/methane ratio. The overall equivalence ratio, for any given $V_{C_3H_8}/(V_{CH_4} + V_{C_3H_8})$ is defined as

$$\Phi = \left(\frac{V_{CH_4} + V_{C_3H_8}}{V_{O_2}}\right) \bigg/ \left(\frac{V_{CH_4} + V_{C_3H_8}}{V_{O_2}}\right)_{stoich.}, \tag{3}$$

It is also seen from Fig. 5 that there is a steep drop of AIT as the propane fraction increases from 0 to 30 vol.% of the total fuel. Apparently, the AIT is a function of both Φ and Φ_3.

The results in Fig. 5 show that the use of linear interpolation on the basis of volume percentages between the AIT of CH_4/air and C_3H_8/air will lead to over-estimating of AITs of intermediate mixtures of propane and methane.

It was found by Griffiths et al. [7] that CH_4/C_4H_{10}/air and C_4H_{10}/air, with the same volume ratio of C_4H_{10} to oxygen, had the same AIT. In other words, the AIT apparently was independent of the presence of methane, as long as the C_4H_{10}/oxygen ratio remained constant. This was attributed to the fact that, throughout the induction period leading to ignition of CH_4/C_4H_{10}/air mixtures, the consumption of methane is small (2.5%), whereas that of butane is high (> 75%), methane therefore plays only a minor role in the ignition process.

It is interesting to apply the same line of thought to the present results for CH_4/C_3H_8/air, because the oxidation of higher alkanes, such as propane and butane, have similar chemical kinetics. The measured AITs of C_3H_8/air and CH_4/C_3H_8/air versus their corresponding volume ratios of propane to oxygen are given in Figs. 6–8 for $\Phi = 0.75$, 1.0 and 2.34, respectively. When Eq. (2) for Φ_3 is applied to mixtures of various hydrocarbons, it relates propane to the total amount of oxygen in the mixture.
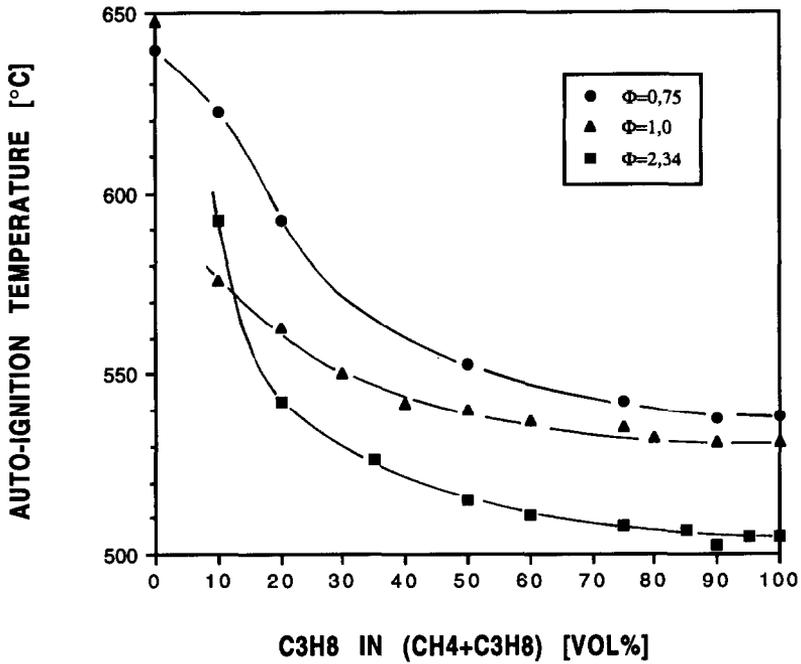
Fig. 5. Influence of fuel composition on the auto-ignition temperatures of CH_4/C_3H_8/air mixtures for three different overall equivalence ratios ($\Phi = 0.75$, 1.0 and 2.34).
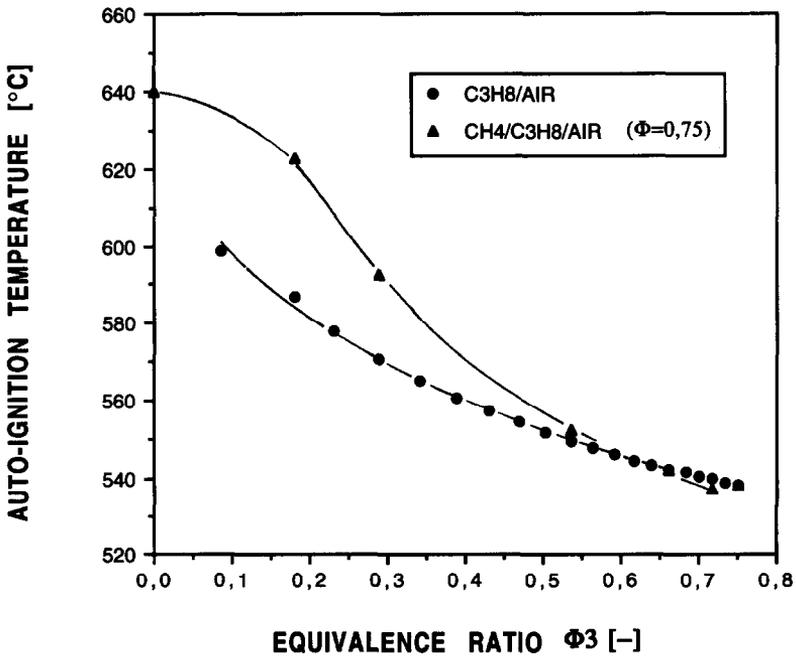
Fig. 6. Dependence of the auto-ignition temperature on the equivalence ratio Φ_3 of C_3H_8/air, and CH_4/C_3H_8/air mixtures of $\Phi = 0.75$.
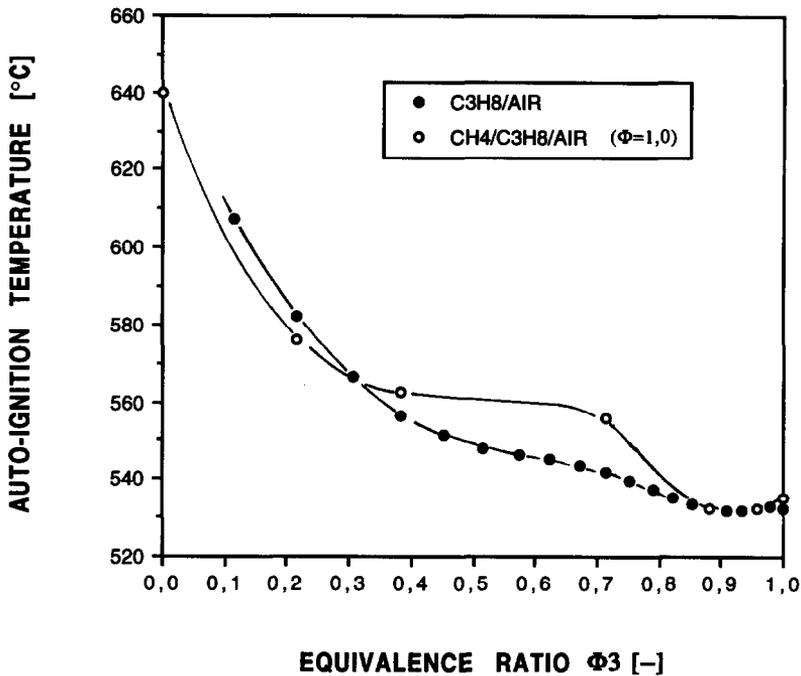
Fig. 7. Dependence of the auto-ignition temperature on the equivalence ratio Φ_3 of C_3H_8/air, and CH_4/C_3H_8/air mixtures of $\Phi = 1.0$.

As can be seen, there is a certain range of Φ_3 within which the AITs of CH_4/C_3H_8/air are the same as those of C_3H_8/air with corresponding Φ_3 values. These ranges are:

$\Phi = 0.75$: $\Phi_3 = 0.55$–0.75,

$\Phi = 1.0$: $\Phi_3 = 0.1$–1.0,

$\Phi = 2.34$: $\Phi_3 = 1.0$–2.34.

Fig. 9 summarizes Figs. 6–8.

3.3. AIT of CH_4/air/CO_2 mixtures

The possible influence of small fractions of CO_2 in the CH_4 on the AIT was studied. Three CH_4/air/CO_2 mixtures of $\Phi_1 = 0.83$ (Eq. (1)), with CO_2 concentrations of 2.0, 5.1 and 7.4 vol.%, were investigated. The corresponding volume ratios of CO_2 to CH_4 were 0.25, 0.67 and 1.0, respectively.

As shown in Fig. 10, the influence of CO_2 was small in the concentration range studied. The AIT rose from 663 °C to 668 °C as the CO_2 concentration increased from 0.0 to 2.0 vol.%, i.e. the volume ratio of CO_2 to CH_4 increased from 0.0 to 0.25. It increased further to 677 °C as the volume ratio of CO_2 to CH_4 reached 0.67. However, no further rise in AIT was observed when this ratio was increased further to 1.0.
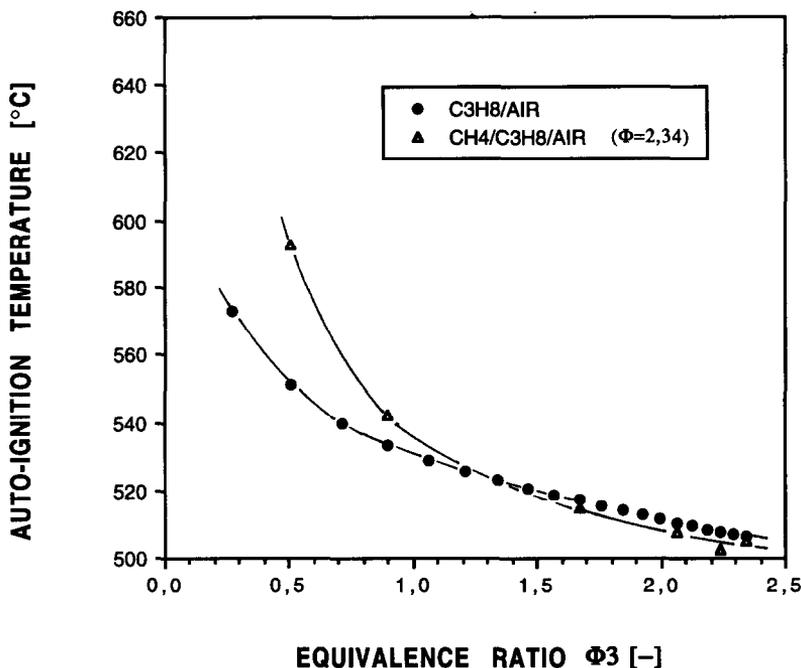
Fig. 8. Dependence of the auto-ignition temperature on the equivalence ratio Φ_3 of C_3H_8/air, and of CH_4/C_3H_8/air mixtures of $\Phi = 2.34$.

It should be mentioned that the AIT of CH_4/air, with no CO_2 addition, ($\Phi_1 = 0.83$) was found to be modestly (23 °C) higher in this part of the investigation, than in the part reported in Sections 3.1 and 3.2, which was conducted several months earlier. The reason for this was not traced. However, as long as the effect of adding CO_2 is the prime concern, this modest deviation is of little significance.

The moderate increase of AIT with added CO_2 that was found, may be explained in terms of straightforward dilution of the explosive mixture (CH_4/air) by an inert gas (CO_2).

4. Discussion

4.1. Influences of the experimental conditions

As pointed out in the Introduction, the temperature and chemical species concentration distributions in the gas mixture in an ideal auto-ignition process are uniform in space at any time during the induction period leading to ignition. However, this ideal requirement is not easy to satisfy fully in realistic bomb experiments.

The present method satisfies this requirement to some extent. Turbulent jet mixing generated by the large initial pressure difference between the reservoir and the ignition
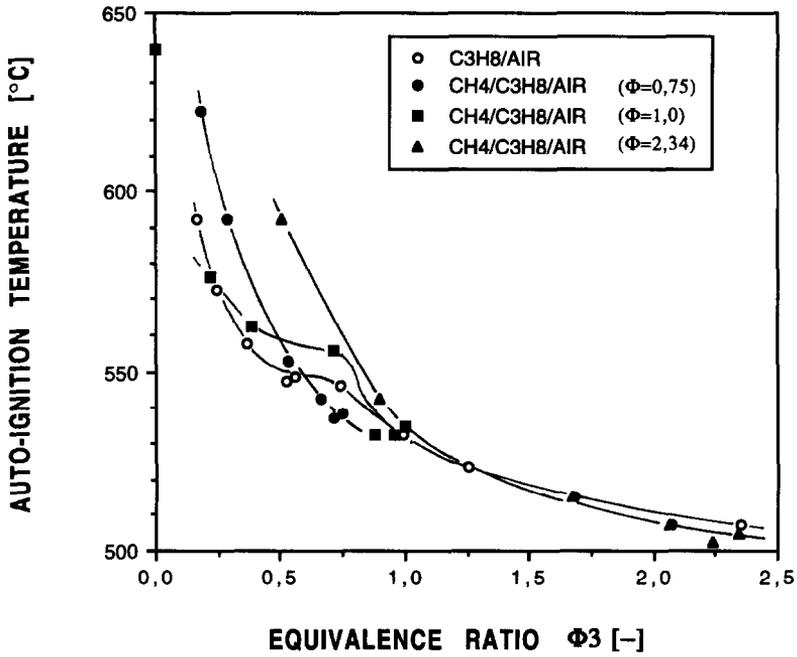
Fig. 9. Dependence of the auto-ignition temperatures of C_3H_8/air and CH_4/C_3H_8/air mixtures on the equivalence ratio Φ_3.
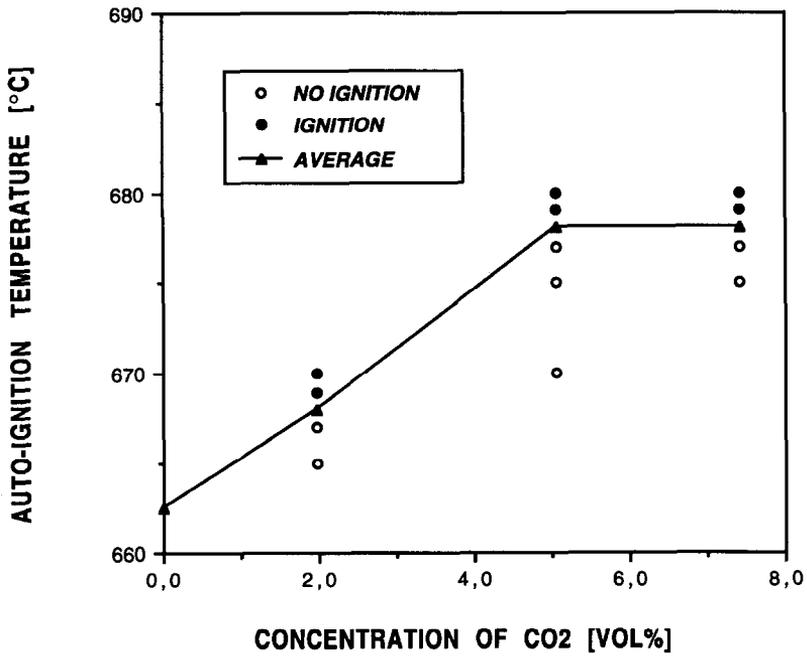
Fig. 10. Influence of carbon dioxide on the auto-ignition temperature of CH_4/air/CO_2 mixtures.

bomb provides a fairly uniform temperature distribution and species concentration distribution throughout the induction period leading to the first pressure equilibrium between bomb and reservoir (point B in Fig. 3). Assuming that the gas is ideal and that its temperature in the bomb equals the bomb wall temperature, 0.25–0.35 l of gas mixture would have entered the ignition bomb within 1.0–1.5 s (t_1). With an internal tube diameter of 4.8 mm, this means that the average velocity of the cold gas jet entering the bomb during this period was of the order of several meters per second. Such a jet would ensure effective turbulent mixing within the bomb as long as the jet persisted. Up to this point it would seem reasonable to assume that the mixing conditions resembled those of a well-stirred reactor. Hence, the temperature of the gas in the bomb was probably close to the bomb wall temperature at time t_1. As shown theoretically by Kong and Alfert [4], the drop of the wall temperature of the ignition bomb due to the admission of cold gas was negligible.

The induction times (t_2) for mixtures at critical temperatures for ignition were mostly in the range 2.5–15-s, thus significantly exceeding the times (t_1) of 1.0–1.5 s needed to establish pressure equilibrium between bomb and reservoir. Therefore, the main part of the chemical induction process in the gas mixture in the bomb from t_1 to t_2 probably occurred in a fairly quiescent gas. As the gas temperature increased beyond that of the bomb wall, the wall would act as a heat sink. Mixing during this phase would be by buoyancy-driven currents. The extent to which uniform temperature and chemical species concentration distributions existed in the bulk of the gas during this phase is unknown.

The present method ensured reasonably accurate control of the gas mixture composition, as opposed to standard test methods such as ASTM E 659-78 and IEC 79-4. In the latter methods a small amount of the gaseous fuel is injected into an open spherical glass vessel or an Erlenmeyer flask, preheated to a given wall temperature, and becomes mixed with the air in the vessel flask by convection and diffusion. Therefore, it is more difficult to control the homogeneity of the mixture.

4.2. Influence of adding higher hydrocarbons to methane/air

Guirguis et al. [8] performed numerical calculations using the chemical kinetics scheme of Westbrook et al. and found that in the induction process of auto-ignition of methane, the following reaction plays an important role:

$$CH_4 \rightarrow CH_3 + H. \tag{4}$$

At comparatively low temperatures reaction (4) is the 'bottle neck' of the pre-ignition chemical reactions. This is because of its comparatively large activation energy and a correspondingly low reaction rate at low temperature. Therefore the AIT of CH_4/air is relatively high.

However, H atoms can be generated through reactions other than reaction (4), e.g. by adding higher hydrocarbons, which produce significant quantities of H atoms even at relatively low temperatures. Then the overall reaction will bypass reaction (4), and

decomposition/oxidation of CH_4 becomes much faster, and ignition occurs at a lower temperature than in pure CH_4/air, according to the reactions

$$H + O_2 \rightarrow OH + O, \tag{5}$$

$$O + CH_4 \rightarrow CH_3 + OH. \tag{6}$$

The rate of generation of H atoms, and hence the overall reaction rate of propane oxidation, increases as the fraction of C_3H_8 increases. This explains why the AITs of C_3H_8/air decrease as the mixture becomes fuel-richer, as shown in Fig. 5.

Adding C_3H_8 to CH_4 will therefore provide H atoms and CH_3 radicals at a lower temperature than in pure methane/air. Thus, in general, the AIT of a CH_4/C_3H_8/air mixture will be lower than that of a CH_4/air mixture with the same Φ_1, and higher than that of a C_3H_8/air mixture with the same Φ_3. However, for some mixtures of CH_4/C_3H_8/air, the oxidation of methane in the induction period leading to ignition plays a very minor role compared with the oxidation of propane, and the oxidation kinetics are virtually identical to the kinetics of C_3H_8/air mixtures of the same Φ_3.

4.3. Influence of adding CO_2 to methane/air

The increase of AIT of CH_4/air due to addition of small amounts of CO_2 may be explained qualitatively in the following way. As shown in Section 3.1 the AIT of CH_4/air remains constant as the methane concentration falls to the range 3.0–8.0 vol.%. So, in the case of the present experiments, the change of methane concentration caused by adding CO_2 would not be expected to influence AIT. However, adding CO_2 lowers the O_2 concentration; therefore a higher initial temperature is needed for ignition to occur. Besides, CO_2 acts as an inert heat sink in addition to the nitrogen of the air.

4.4. Application of the present experimental data

The AIT values obtained from the present measurements cannot be applied directly to evaluate hot-surface ignition hazards in practical situations. They can only provide a relative measure of the ignitability of gas mixtures. However, the present work shows that for CH_4/C_3H_8/air mixtures, there exists a wide range of C_3H_8/air ratios, within which the CH_4/C_3H_8/air mixtures have the same minimum ignition temperatures as C_3H_8/air mixtures of identical C_3H_8/air ratios. This relationship probably also holds for various hot-surface ignition situations.

For a general quantitative treatment of the hot-surface ignition problem as appearing in industrial practice, a comprehensive tailor-made analysis of all the influential factors will be required. Using a suitable CFD code together with a chemical reaction kinetics package accounting for transfer of both momentum, heat and mass during the induction period is the appropriate way of treating hot-surface ignition problems in the future.

5. Conclusions

Measurements of auto-ignition temperatures (AITs) of CH_4/air, C_3H_8/air, CH_4/C_3H_8/air and CH_4/air/CO_2 mixtures were performed with various equivalence ratios and fuel compositions using a 1 l ignition bomb. The method used ensures accurate control of the composition of the gas mixture to be investigated, which is not necessarily the case with common standard test methods. The method further implies that the entire test gas mixture is heated to the ignition source temperature prior to ignition. The following conclusions can be drawn:

(1) For the mono-fuel/air mixtures, a strong non-linear dependence of AIT on equivalence ratio (fuel/oxygen volume ratio) was found. The most favourable fuel concentrations for ignition were 3.0–8.0 vol.% for CH_4/air and 10.0–15.0 vol.% for C_3H_8/air. The lowest AITs measured for the two fuels were 640 °C and 500 °C, respectively. The corresponding values obtained by other investigators, using the standard open flask method ASTM E 659-78, arc 630 °C and 450 °C.

(2) For the binary fuel/air mixture CH_4/C_3H_8/air, AIT decreased with increasing overall equivalence ratio and increasing C_3H_8/O_2 ratio.

(3) For a given overall equivalence ratio

$$\Phi = \left(\frac{V_{CH_4} + V_{C_3H_8}}{V_{O_2}}\right) \bigg/ \left(\frac{V_{CH_4} + V_{C_3H_8}}{V_{O_2}}\right)_{stoich.},$$

a 'coincidence range' was identified, within which CH_4/C_3H_8/air and C_3H_8/air of the same C_3H_8/O_2 ratio gave the same AIT value. This range could be quantified in terms of a range of the partial equivalence ratio of propane:

$$\Phi_3 = \left(\frac{V_{C_3H_8}}{V_{O_2}}\right) \bigg/ \left(\frac{V_{C_3H_8}}{V_{O_2}}\right)_{stoich.},$$

(4) Only a very modest rise of AIT was found when CO_2 was added to CH_4/air ($\Phi = 0.83$ was kept constant). By increasing the CO_2 content from 0 to 5.1 vol.%, AIT rose by only 14 °C, and no further significant rise in AIT was found when CO_2 content was increased further to 7.4 vol.%.

(5) In general, 'minimum ignition temperatures' of gas mixtures depend on the specific experimental apparatus used. When standing alone, without being related to general theory, data from such methods can only serve as a relative measure of the ignitability of various gas mixtures in that specific test situation. Published data show that experimental minimum hot-surface temperatures for igniting a given propane/air mixture can differ by at least 500 °C, depending on the test conditions.

(6) The essential theoretical elements exist, by which comprehensive dynamic simulation models of hot-surface ignition of explosive gas mixtures may be formulated. The essential elements include both chemical kinetics, heat transfer, and fluid dyamics.

Appendix

With reference to Fig. 1, in the main text, the experimental procedure for determin-ing AIT was as follows:

(1) The oven temperature was set at a value close to the expected AIT, as estimated from the literature or from results of preceding tests.
(2) The oven temperature was allowed to stabilize for at least 30 min.
(3) The ignition bomb was evacuated by closing valves 4 and 3, and switching valve 2 to the pump. Then the bomb was sealed by closing valve 2.
(4) The two-way valve 6 was then switched to the ventilation system, and the flows of air and fuels were adjusted until the desired gas mixture had been obtained.
(5) Then the gas reservoir was evacuated by closing valves 1 and 2, opening valve 4 and switching valve 6 in line with valve 4.
(6) After evacuation, valve 4 was closed and valve 1 was opened, until the pressure in the gas reservoir had reached atmospheric (several seconds). Then valve 6 was switched back in line with the ventilation system.
(7) The gas mixture, of the desired composition, was allowed to purge the gas reservoir, using an amount of gas equal to three times the volume of the reservoir (1 l).
(8) Then valve 1 was closed, and valve 6 was again switched to become in line with valve 2, whereby the gas reservoir became completely sealed. The pressure in the gas reservoir at this point was typically 1.2–1.4 bar (abs). Vacuum was kept inside the ignition bomb.
(9) Valve 2 was then switched to open the connection between the gas reservoir and the evacuated ignition bomb. Initial pressure equilibrium was established within 1.2 s at typically 0.9 bar (abs). During further, slower heating of the gas in the ignition bomb, the equilibrium pressure rose to about 1.0 bar (abs). (The connec-tion between the ignition bomb and the gas reservoir was kept open.)
(10) If ignition did not occur within 10 min, the test was terminated and another test was conducted at a higher oven temperature. If ignition occurred within 10 min,

the test was repeated at a lower oven temperature. The AIT was identified within a temperature interval $\leqslant 5\,°C$. (Long-term irreversible changes of the apparatus caused a systematic drift towards somewhat higher AIT values, as mentioned in the note at the end of Section 3.3.)

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