Experimental Investigation of Liquid Fuel Autoignition: Insights from Advanced Optical Diagnostics

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1 Introduction

The ignition of flammable gases, combustible liquids, or powders in a hot air atmosphere is a significant safety concern across various industries, including power generation, petroleum, automotive, chemical, and aerospace. To assess ignition hazards, standardized tests have been established to determine the autoignition temperature (AIT), a critical temperature threshold. AIT is defined as the minimum temperature at which self-sustaining combustion occurs without the need for an external ignition source, such as a spark or flame [1]. The most widely adopted methods for determining AIT are the ASTM-E659 standard [2] and the international standard ISO/IEC standard [3]. The protocol consists of injecting a small fuel quantity into a 500 mL round-bottom flask heated at a constant temperature in a furnace and observing for 10 minutes the presence or absence of flame. Following a specific procedure for varying the temperature and fuel volume in the test, a minimum temperature at which the ignition occurs is used to specify the AIT.

Previous research highlights that the AIT result depends on the experimental conditions used during measurements. Working on the early version of the ASTM-E659 apparatus, Setchkin [4] identified key parameters influencing AIT, which have been extensively studied since. Their findings showed that AIT decreases with increasing vessel size and that the ratio of the combustible substance to air plays a crucial role. [4] also highlighted the importance of maintaining uniform temperature within the flask. Recent studies [5, 6] have once again emphasized the impact of temperature gradients on the flow field within the apparatus and their influence on AIT measurements. Several other factors are known to influence AIT, including the shape of the flask, the rate and duration of heating, and the flask material [1]. Recent studies have further explored the impact of the combustion vessel, as well as the effects of increased pressure and nitrogen dilution on AIT [7, 8]. The influence of flask material was highlighted by Chen *et al.* [9], who found that the AIT of ethanol measured in a quartz flask was 20°C higher than in a borosilicate flask. The same study also examined the effect of ambient temperature, revealing a quadratic relationship in which a 25°C increase in ambient temperature led to an approximately 10°C rise in AIT. Additionally, flask cleanliness has been shown to affect AIT, as multiple uses can result in higher AIT readings [10].

The main objective of this work is to improve our understanding of the autoignition process of liquid fuels under ASTM standardized conditions and to investigate the key factors influencing AIT measurements. Tests were conducted in a transparent square cell under experimental conditions closely matching

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ASTM standards. The ignition process was divided into four distinct phases: injection, vaporization, dispersion, and the chemical reactions leading to potential ignition. Various measurement techniques, including Particle Image Velocimetry (PIV), Background-Oriented Schlieren (BOS), infrared light absorption, direct imaging, and chemiluminescence visualization, were employed to characterize each phase.

2 Material and methods

The ignition testing setup (see Figure 1) consists of a compact, enclosed test chamber within a larger enclosure that provides thermal insulation and ensures a uniform temperature environment. Stainless steel vertical bars and an aluminum lower platform secure the cell windows in place. Quartz, which is optically transparent, is used to allow the transmission of UV light during ignition. The cell can be equipped with thermocouples to monitor temperature both inside and on the walls of the testing chamber. A hatch is located on the top of the test cell, enabling the operator to flush combustion products using an aluminum cylinder and a heat gun after each test. The cell is thermally insulated with glass wool, and the top and bottom are separately heated to the desired temperature using electric heaters.

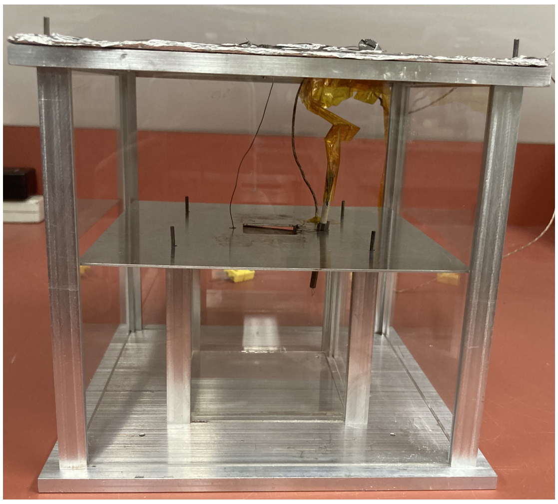


Figure 1: Experimental square cell

Multiple measurement techniques were employed to characterize the ignition testing process, covering the flow conditions before injection, the injection and vaporization of the fuel, and the exothermic chemical processes leading to ignition.

Direct light visualization was used to capture the injection process, including the fuel jet, the interaction of droplets with the hot surface, and the vaporization of the droplets. This was synchronized with Background Oriented Schlieren (BOS), which enhances density gradients within the cell, making the dispersion of fuel vapor visible. Particle Image Velocimetry (PIV) was initially applied with titanium oxide particles and a pulsed laser sheet to characterize the flow field velocity before, during, and after injection, as well as before and after ignition. The titanium oxide particles were later replaced by incense particles, which are capable of burning in high-temperature environments, thus highlighting areas of exothermic reactions. The infrared laser light absorption technique was used to study the dispersion of fuel vapor, relying on the fact that C-H bonds in hydrocarbon molecules strongly absorb infrared (IR) light at specific wavelengths. Finally, thermocouples type K, gauge 34, from Omega, with an acquisition module (NI 9213) from National Instruments, connected to a cDAQ-9171 CompactDAQ Chassis are used to measure the temperature inside the cell and on the walls at 75 Hz. The module has one internal autozero channel, 1 internal cold-junction compensation channel, and 16 thermocouple channels.

Tests were conducted with Hexane, Jet A POSF-4658, and another Synthetic Paraffinic Kerosene (SPK). The studied fuel is injected with a 500 μL hypodermic syringe with a 6-inch long (152.4 mm) 26 gauge needle.

3 Results and discussions

INJECTION AND VAPORIZATION: The injection of fuel consists of a jet impacting a hot plate, resulting in small droplets and an impact puddle. The droplets size depends on the diameter of the needle, the surface tension of the fuel, the velocity of injection, and the surface temperature. Leidenforst effect is involved when the surface temperature goes over the Leidenforst temperature of the fuel, which results in smaller vaporization and longer evaporation times.

Synchronized direct light visualization with the BOS technique captures the dispersion processes in the cell. Figure 2 gives an example of synchronized visualizations of the injection of 100 µL of SPK (high concentration of fuel vapor represented by bright colors).

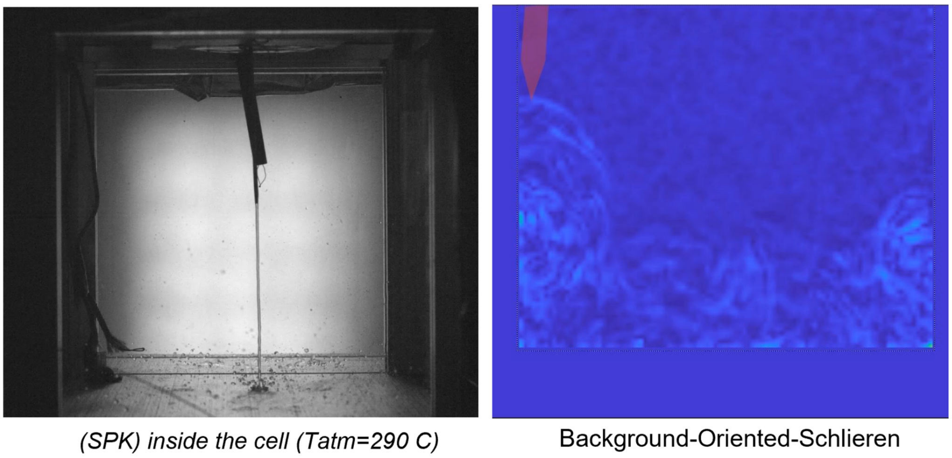
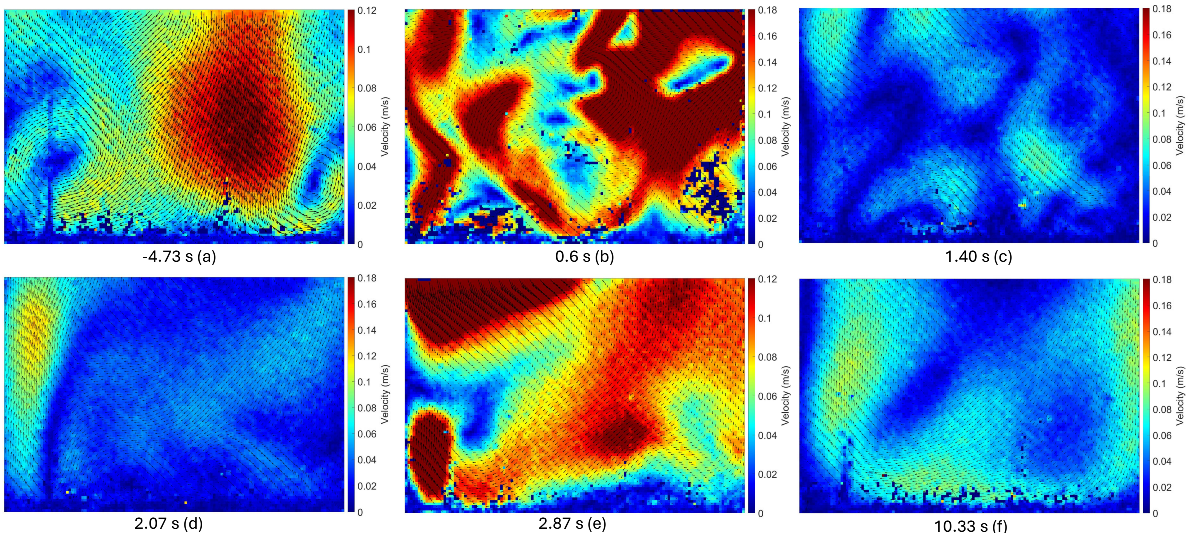


Figure 2: Direct light visualization and BOS images during the injection of SPK inside a 290 C cell

Additionally, the results show that the fuel jet phase depends on the needle temperature, which in turn is influenced by the time the operator takes to start the injection. If the injection occurs immediately (i.e., within approximately 5 seconds, depending on the fuel and atmosphere temperature), the jet is fully liquid. However, if the operator waits longer and leaves the needle filled with fuel inside the hot cell, the jet becomes multiphase, consisting of both liquid and vapor. If the delay exceeds about 10 seconds (again, depending on the fuel and atmosphere temperature), the fuel overheats inside the needle, resulting in a vapor jet or even the ejection of a significant amount of fuel without operator intervention due to increased pressure inside the needle.

DISPERSION: The dispersion is assessed with BOS and infrared light absorption. The methods help to characterize the characteristic time of the vaporization and dispersion and compare it with ignition delay time. The results suggest that ignition can occur before the fuel droplets have fully vaporized if the cell temperature is sufficiently high. This indicates that the amount of fuel participating in ignition is not always equal to the total amount injected. Additionally, ignition takes place in a non-homogeneous atmosphere, as fuel dispersion occurs over a longer timescale than ignition.

FLOW CONDITIONS DURING A TEST: PIV was successfully performed during autoignition testing. An example of post-processed images is given in Figure 3. Results reveal convective motion inside the cell before fuel injection (a). Fuel injection and dispersion generate intense turbulence within the cell (b), but the flow field quickly stabilizes (c). A hot gas pocket is observed rising on one side of the cell (d), possibly due to the exothermic reaction of the fuel. Shortly after, ignition is triggered (e), and the flow stabilizes once again into a natural convection pattern (f).



-4.73 s (a) 0.6 s (b) 1.40 s (c)

2.07 s (d) 2.87 s (e) 10.33 s (f)

Figure 3: Particle Image Velocimetry with titanium oxide particles, t=0s at injection time, 100μL of Hexane

EXOTHERMIC REACTION AND IGNITION: The hot gas pocket was also observed using incense particles instead of titanium oxide particles. Figure 4 provides an example, showing three consecutive images captured at 15 Hz. The images highlight a black pocket going up, indicating burned incense caused by an exothermic reaction in that region. These images were taken just before the ignition event. This hot gas pocket is observed for all the tests, even when the cell temperature is lower than the AIT of the tested fuel.

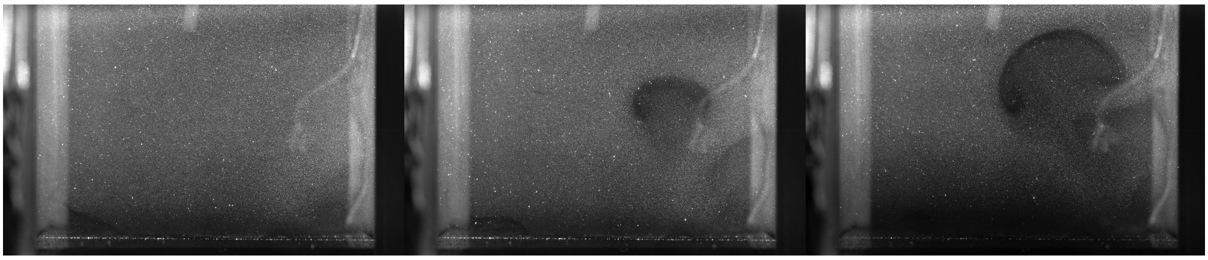


Figure 4: Particle Image Velocimetry with incense, 100μL of Hexane

4 Conclusion

This study provides insights into autoignition testing through experimental investigations. Our findings highlight injection, dispersion, and exothermic reaction as the three key components impacting autoignition behavior in an ASTM apparatus.

Results indicate that fuel injection dynamics influence droplet formation and vaporization, with factors such as needle temperature and injection delay affecting the jet phase. Dispersion measurements reveal turbulent mixing that stabilizes into a convective flow, while localized exothermic reactions contribute to ignition dynamics. Notably, ignition can occur before full vaporization in a non-homogeneous atmosphere, emphasizing the complex interplay between fuel distribution and thermal conditions.

These findings enhance the understanding of fuel-air interactions under autoignition conditions, offering valuable insights for optimizing combustion systems and refining ASTM testing methodologies.

Acknowledgments

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