

Ignition of Lubricating Oils using a Novel Spray Injection Technique in a Shock Tube

Sean P. Cooper and Eric L. Petersen
J. Mike Walker '66 Department of Mechanical Engineering
Texas A&M University
College Station, Texas, USA

1 Introduction

Lubricants have long been known to be a fire hazard in propulsion and power generation systems [1-3]. Additionally, lubricant droplets are also a known source of low-speed pre-ignition (LSPI) in internal combustion engines (ICEs) [4, 5]. Notably, n-hexadecane (nC_{16}) has been used as a surrogate for lubricating oil to model the effect of lubricant addition on gasoline ignition in ICEs [6]. Explosions have also been caused by heavy oils in *in-situ* combustion (ISC) and air injection assisted steam recycling (AASR) [7]. Aside from combustion systems, lubricants are a fire hazard in air conditioners and wind turbines [8, 9]. Even though the safety issues with lubricating oils are well known, little has been done to understand the fundamental characteristics of lubricant combustion. Recently, Teigte experimentally examined lubricant droplet ignition on a hot plate and discussed similar techniques [10]. A summary of the limited lubricant auto-ignition studies has been presented previously [11].

In choosing the appropriate lubricant for system implementation, auto-ignition properties require consideration. Qualitative comparisons of lubricant ignition over a range of temperatures would provide much needed information about relative reactivity of lubricating oils. Shock tubes are particularly useful for producing homogeneous, high-temperature and –pressure regions for studying ignition delay times (τ_{ign}). However, utilizing a shock tube to observe lubricant ignition poses several issues, chief among them being getting the lubricant to vaporize. Lubricants thermally decompose before vaporization, so traditional methods are inadequate to study lubricant ignition in shock tubes. Additionally, aerosolization and simple spray injection of lubricants is difficult due to their high viscosity. To this end, a novel injection technique was devised for collecting lubricating oil τ_{ign} data at high temperatures. The experiment is characterized with Jet-A experiments, and τ_{ign} data are compared with those from the literature. An off-the-shelf lubricant, Mobil DTE 732, was tested at near atmospheric pressure for temperatures between 1220 and 1353 K. Herein, initial efforts to model this behavior use nC_{16} as a surrogate for the lubricant using kinetics models from Lawrence Livermore National Laboratory (LLNL) and the Chemical Reaction Engineering and Chemical Kinetics (CRECK) modeling group [12, 13]. These topics are discussed in the following sections.

2 Experimental Technique

The high-pressure shock tube (HPST) facility at Texas A&M University was utilized to generate high-temperature conditions. The shock tube's driven section has an inner diameter of 15.24 cm and is 5.03 m in length. Incident-shock velocity (v_{isw}) is collected using four pressure transducers (PCB Piezotronics, Inc. model 113B22) evenly spaced along the last 1.44 m of the driven section. Using v_{isw} , and the initial driven gas temperature, pressure, and composition (room air for all experiments) the temperature and pressure behind the reflected shock can be calculated to within $\pm 0.8\%$ and $\pm 1.0\%$ uncertainties, respectively. Pressure is monitored at the sidewall location 1.6 cm upstream from the endwall of the shock tube using an RTV silicone-shielded pressure transducer (PCB Piezotronics, Inc. model 113A22). The driver section of the shock tube has an inner diameter of 7.62 cm and a length of 5.11 m which, using helium and air to tailor the driver gas composition, allows for a test time of up to 7 ms. The driver and driven sections of this pressure-driven shock tube are separated using a 0.25-mm thick polycarbonate diaphragm to generate near-atmospheric post-reflected shock pressures. Two rotary vane vacuum pumps are used to evacuate the driver and driven sections (Agilent DS 102 and DS 402, respectively). The driver section is only evacuated to the pressure required for the pressure of air needed to tailor the driver gas (~ 7 -20 kPa), then is filled until diaphragm rupture (~ 480 kPa) using helium. A sharpened cutting blade is on the driven side of the diaphragm to aid in repeatable diaphragm rupture and to minimize fragmentation. Additional information on the shock-tube facility has been provided previously [14, 15].

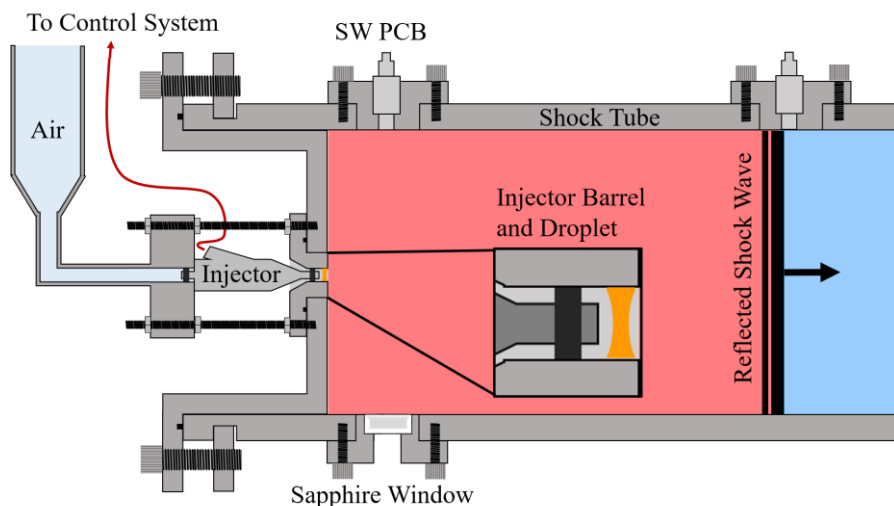


Figure 1: Schematic of the spray injection setup. A magnified view of the injector barrel is shown as well as the pressure diagnostics and optical access for OH* emission and laser scattering.

Generally, shock tubes are used strictly with gaseous mixtures, but low-vapor pressure fuels require special treatment. Usually this involves a heated shock tube, which raises the mixture's temperature so the fuel's vapor pressure is considerably higher than the partial pressure of the fuel in the mixture. This method works well for intermediate-vapor pressure fuels such as components of gasoline surrogates (toluene, iso-octane, n-heptane, etc.) [15]. However, this method meets its limit when dealing with higher-order hydrocarbons such as kerosene-based fuels (diesel, Jet-A, C₁₀ and higher). While success has been made in the past running experiments with these fuels, they are incredibly difficult and require heating up to 200 °C [14]. At these high initial temperatures, the time scales for thermal cracking are considerably shorter, limiting the amount of time a mixture can be used before the fuel no longer represents its initial composition, which for many off-the-shelf oils can also include various additives. However, aerosol shock tubes have been used successfully to avoid this issue.

In an aerosol shock tube, the fuel is aerosolized and introduced to the shock tube in liquid suspension form, utilizing the incident shock wave to vaporize the fuel [16, 17]. However, aerosol generators are limited by the fuel's viscosity. So, heated shock tubes are inadequate to observe lubricant ignition as the lubricant

would break down before vaporization, and the lubricant's viscosity is arguably too high for practical aerosolization in an aerosol shock tube.

Therefore, a new method was developed to introduce the lubricant to the reflected-shock region [11, 18]. A 550-cc/min automotive injector (RC Fuel Injection model SL4-550) is attached to the endwall of the shock tube, shown in Fig. 1. Unlike in its standard form where fuel is pressurized behind the injector, here, 275-kPa (abs) air is supplied to the injector. In this way, the injector acts as a fast-acting valve that opens once the reflected shock reaches the sidewall location. Figure 1 shows a magnified view of the injector "barrel," which is internal to the shock tube. A 0.2-mL droplet of the lubricant is placed within this barrel prior to each experiment. Once the injector is triggered by upstream shock wave detection, 275-kPa air is forced through the droplet for a duration of 250 μ s, causing a fine mist of lubricant to be introduced to the post-reflected-shock region where the mist vaporizes and combusts in the high-temperature environment. Note, since the droplet is internal to the shock tube before each experiment, the driven section is vacuumed only to the initial pressure needed for the experiment. If a high vacuum were achieved, the smaller components of the lubricant would vaporize and be evacuated from the shock tube, ruining the representative composition. In this way, room air is used as the working fluid in the driven section.

For the initial characterization of the spray injection method, a laser scattering setup was used to observe timing and duration of the lubricant spray. A JDS Uniphase HeNe laser (model 1508P-0) produces a continuous, 633-nm, 1-mW beam through the shock tube via two sapphire windows at a sidewall location into a New Focus, Inc. detector (model 2032). As the lubricant is sprayed into the post-reflected-shock region, the aerosolized lubricant scatters the beam, reducing the amount of signal reaching the detector. A representative laser trace is shown in Fig. 2. Here, a reduction in laser signal is observed as the reflected shock passes the sidewall location which continues to reduce until the end of the 250- μ s spray duration, then the signal returns to the initial value, indicating the mist has vaporized. Note, laser extinction continues after the spray duration as some of the aerosolized lubricant remains after the injector has closed, but complete vaporization is seen shortly after.

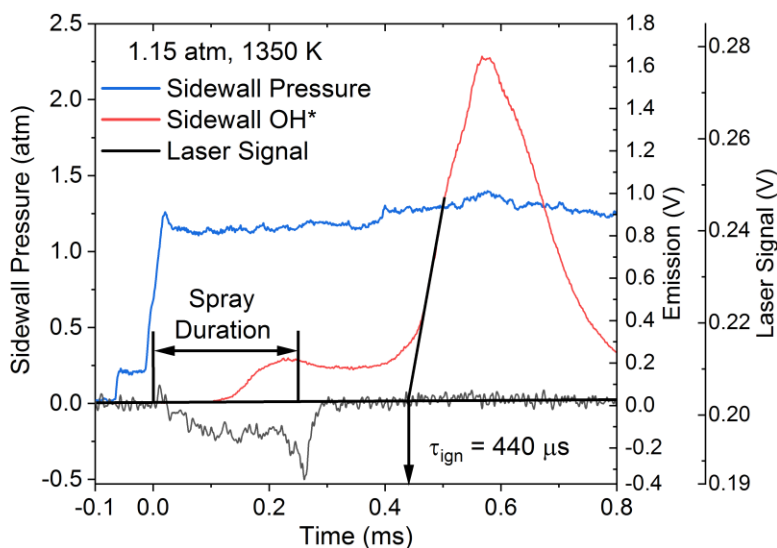


Figure 2: Representative signals from sidewall pressure, sidewall OH* emission, and laser scattering diagnostics for a lubricant experiment at 1.15 atm and 1350 K [11]. Ignition delay time is defined based on the secondary increase, which is 440 μ s in this case.

From here, an OH* chemiluminescence diagnostic is used to observe lubricant ignition. OH* light emission from combustion is emitted through the sapphire window at the sidewall location and is focused into a filtered photomultiplier tube (Hamamatsu model 1P21, centered at 307 nm). A representative emission profile is also shown in Fig. 2. Two-stage ignition is frequently observed for these experiments. Essentially,

combustion initially occurs during lubricant spray, then after additional lubricant vaporizes a stronger, secondary combustion occurs. The stronger ignition event is used for determining τ_{ign} .

Generally, for a $\pm 0.8\%$ uncertainty in post-reflected-shock temperature, $\pm 20\%$ uncertainty in τ_{ign} is considered conservative [19]. However, additional variables require consideration due to the added complexity from the injection method. Added variables include heat transfer from air injection, added mass from air injection, and the heat loss due to lubricant vaporization. The first two were found to be negligible due to the small amount of air needed for injection. The latter variable, vaporization heat loss, was found to reduce the temperature by a conservative -2% , using $n\text{C}_{16}$ as a model for heat loss. Therefore, the overall uncertainty in the temperature is $\pm 1.5\%$, which translates to a root-mean-square error in τ_{ign} of $\pm 30\%$ [18].

3 Results and Discussion

Initial characterization of the spray injection method was conducted with Jet-A. The results using Jet-A compared to literature models and experiments are shown in Fig. 3. Note, only one ignition event was observed for the Jet-A experiments. The equivalence ratio for the results was estimated by calculating the amount of air in the driven section and the molar concentration of fuel assuming total vaporization, which is near stoichiometric for both Jet-A and the lubricant assuming $n\text{C}_{16}$ for the lubricant. Good agreement is seen especially with the experimental results from Mullins [20]. Good agreement is also seen with model predictions made by the Malewicky et al. model, which performed well with previous, heated shock-tube experiments [14, 21]. This agreement demonstrates the experiment's repeatability and adds to its credibility.

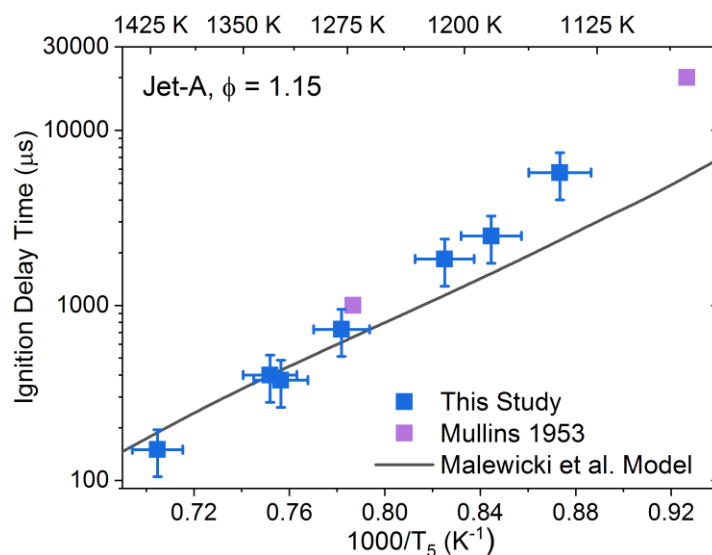


Figure 3: Ignition delay time results collected using the spray injection method with Jet-A. Test conditions were near-atmospheric pressure and between 1145 and 1419 K. Model predictions made by mechanisms from Malewicky et al. as well as experimental data from Mullins are also shown [20, 21].

Finally, experiments were conducted with an off-the-shelf lubricant, Mobil DTE 732, and the results are shown in Fig. 4. Unlike the strictly Arrhenius-like behavior of Jet-A, the lubricant exhibits a changing temperature dependency within the temperatures investigated (1220 – 1353 K). For temperatures between 1230 and 1275 K, no change in τ_{ign} is observed. $n\text{C}_{16}$ has been used as a surrogate for lubricants in the literature, so $n\text{C}_{16}$ kinetics mechanisms are compared to the data collected herein in Fig. 4. Coincidentally, CRECK agrees well with the data in the intermediate regime in both slope and magnitude. LLNL closer represents the overall slope of the data, but neither model exhibits the overall behavior shown by the data. This disagreement does not suggest the models are inadequate, but rather that additional efforts are required to model these new results. Particularly, the 0-D assumption generally used in shock tubes is likely not

applicable to this technique. Additional modeling work using heat transfer modeling may be required. Also, the lubricant itself has been found to be a much larger molecule than nC_{16} ($>C_{30}$), and chemical kinetics models are only really available for molecules up to C_{20} [18].

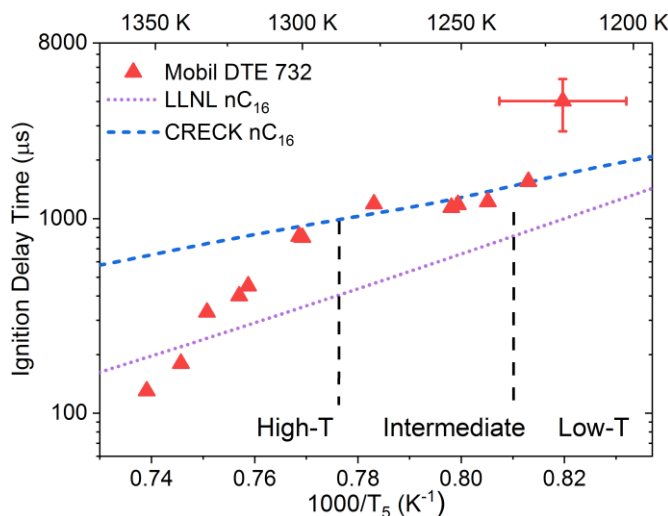


Figure 4: Ignition delay time results for Mobil DTE 732 using the spray injection method. Three regimes are observed: a high-temperature, Arrhenius-like regime; an intermediate regime with no change in τ_{ign} with temperature; and a lower-temperature regime which continues the temperature dependence of τ_{ign} . Models for nC_{16} by LLNL and CRECK, under a 0-D assumption, are unable to capture these features.

4 Conclusions and Future Work

A novel spray injection technique was developed to investigate ignition characteristics of lubricants. The method is applied to produce τ_{ign} results for Jet-A and an off-the-shelf lubricant Mobil DTE 732 near atmospheric pressure. The Jet-A results reproduced data from the literature well, and agreement was seen with a well-established kinetics mechanism from the literature, demonstrating the reliability of the technique. Ignition delay time results for Mobil DTE 732 show little temperature dependence for temperatures between 1230 and 1275 K. Initial efforts were made to model these results using nC_{16} , however, under a 0-D assumption, chemical kinetics mechanisms by LLNL and CRECK are unable to capture these trends. Moving forward, considerably more work will need to be conducted to model the results found herein. *n*-Hexadecane ignition should also be examined using this technique. Additionally, efforts need to be taken to understand the effect of lubricant additives, as much of the composition of Mobil DTE 732 is unknown due to the proprietary nature of the recipe. However, this technique expands upon the range of molecules for which shock tubes can study, allowing for additional work to be conducted on the reactivity of larger hydrocarbons like lubricants and other exotic fuels.

References

- [1] Zabetakis MG, Scott GS, Kennedy RE. (1962). Autoignition of lubricants at elevated pressures. U.S. Department of the Interior, Bureau of Mines, Pittsburgh, PA.
- [2] Kuchta J, Cato R. (1968). Ignition and Flammability Properties of Lubricants. SAE Transactions 77: 1008.
- [3] Loomis WR. (1976). Aircraft engine sump-fire studies. NASA Aircraft Safety and Operating Problems Conference, Langley Research Center, Hampton, VA.

- [4] Williams R, Landis J. (1954). Some Effects of Fuels and Lubricants on Autoignition in Cars on the Road. SAE Transactions 62: 57.
- [5] Wang Z, Liu H, Reitz RD. (2017). Knocking combustion in spark-ignition engines. Prog. Energ. Combust. 61: 78.
- [6] Mitsudharmadi H, Maharjan S, Elbaz AM, Qahtani YA, Roberts WL. (2019). Auto-Ignition of a Hexadecane Droplet Mixed with Different Octane Number Fuels at Elevated Pressures To Investigate the Pre-Ignition Behavior. Energ. Fuel 34: 806.
- [7] Huang L, Wang Y, Li Z, Zhang L, Yin Y, Chen C, Ren S. (2021). Experimental study on piloted ignition temperature and auto ignition temperature of heavy oils at high pressure. Energ. 229: 120644.
- [8] Kim CJ, Choi HH, Sohn CH. (2011). Auto-ignition of lubricating oil working at high pressures in a compressor for an air conditioner. J. Hazard. Mater. 185: 416.
- [9] Sun W, Lin WC, You F, Shu CM, Qin SH. (2019). Prevention of green energy loss: Estimation of fire hazard potential in wind turbines. Renew. Energ. 140: 62.
- [10] Teitge D. (2021). DESIGN AND CHARACTERIZATION OF A HOT-SURFACE IGNITION EXPERIMENT. Master's Thesis, J. Mike Walker '66 Department of Mechanical Engineering, Texas A&M University.
- [11] Cooper SP, Browne ZK, Alturaifi SA, Mathieu O, Petersen EL. (2021). Auto-Ignition of Gas Turbine Lubricating Oils in a Shock Tube using Spray Injection. J. Eng. Gas Turbine. Power 143: 051008.
- [12] Westbrook CK, Pitz WJ, Herbinet O, Curran HJ, Silke EJ. (2009). A comprehensive detailed chemical kinetic reaction mechanism for combustion of n-alkane hydrocarbons from n-octane to n-hexadecane. Combust. Flame 156: 181.
- [13] Ranzi E, Frassoldati A, Stagni A, Pelucchi M, Cuoci A, Faravelli T. (2014). Reduced kinetic schemes of complex reaction systems: fossil and biomass-derived transportation fuels. Int. J. Chem. Kinet. 46: 512.
- [14] Alturaifi SA, Rebagay RL, Mathieu O, Guo B, Petersen EL. (2019). A Shock-Tube Autoignition Study of Jet, Rocket, and Diesel Fuels. Energ. Fuel. 33: 2516.
- [15] Cooper SP, Mathieu O, Schoegl I, Petersen EL. (2020). High-Pressure Ignition Delay Time Measurements of a Four-Component Gasoline Surrogate and its High-Level Blends with Ethanol and Methyl Acetate. Fuel 275: 118016.
- [16] Hargis JW, Guo B, Petersen EL. (2020). A new high-pressure aerosol shock tube for the study of liquid fuels with low vapor pressures. Rev. Sci. Instrum. 91: 124102.
- [17] Hargis JW, Cooper SP, Mathieu O, Guo B, Petersen EL. (2021). High-temperature ignition behavior of conventional and GTL fuels using an aerosol shock tube. Combust. Flame 226: 490.
- [18] Cooper SP, Petersen EL. (2021). High-Temperature Ignition Kinetics of Gas Turbine Lubricating Oils. J. Eng. Gas Turbine Power. 143: 111020.
- [19] Zander L, Vinkeloe J, Djordjevic N. (2021). Ignition delay and chemical-kinetic modeling of undiluted mixtures in a high-pressure shock tube: Nonideal effects and comparative uncertainty analysis. Int. J. Chem. Kinet. 53: 611.
- [20] Mullins BP. (1953). Studies on the spontaneous ignition of fuels injected into a hot air stream. V. Ignition delay measurements on hydrocarbons. Fuel, Land. 32: 363.
- [21] Malewicki T, Gudiyella S, Brezinsky K. (2013). Experimental and modeling study on the oxidation of Jet-A and the n-dodecane/iso-octane/n-propylbenzene/1,3,5-trimethylbenzene surrogate fuel. Combust. Flame 160: 17.