# Shock-Tube Study of Lubricating Oil Ignition Delay Times

Matthew Abulail, Sean P. Cooper, Matthew G. Sandberg, and Eric L. Petersen
Texas A&M University
College Station, Texas, USA

## 1 Introduction

Lubricating oils are essential in the operation of modern power generation systems. Oils maximize efficiencies in these systems by dispersing heat throughout the system while lowering the power lost by friction [1]. With increasing regulations, the engine combustion systems need to run at higher temperatures and pressures to meet these new efficiency requirements [2]. In addition, downtime for systems such as gas turbines is costly, resulting in lubricants not being changed within the proper time intervals. With these harsh operating conditions, lubricating oils are being used to their limits, resulting in rapid oil degradation, oil ignition, and possible combustion. With oil degradation becoming a reoccurring issue within mechanical and electrical systems, a fundamental chemical kinetics study on lubricating oils would be beneficial. Quantifying the ignition delay time (IDT) of the oils would provide a necessary comparison between oils [3-7]. This comparison would provide necessary information for informed lubricant selection for systems with high pre-ignition characteristics [4-7].

To accomplish such a study, shock tubes are ideally suited. It is well known that shock tubes are able to observe and demonstrate the chemical kinetics for a given gaseous fuel-oxidizer mixture [8, 9]. Cooper and coworkers developed an injector-based lubrication injection system to test fluids in liquid or solid form at standard temperature and pressure [4-7]. In the system described by Cooper et al., an automotive injector is backed with 40 psi of air, with the lubricant material placed in the barrel of the injector. Before the incident shock wave, the air injector will disperse the oil as a spray, effectively suspending droplets of lubricants within the shock tube, allowing the incident shock wave to vaporize the lubricant [6, 7]. This method provides a nearly homogenous dispersion of lubricant in the shock tube while providing an effective method to obtain chemical kinetic data. The initial work conducted by Cooper et al. focused on equivalence ratios around 1.2 [4, 5, 7]. Using the method described by Cooper et al., n-hexadecane, mineral oil, Mobil DTE 732, and Castrol Perfecto X32 were tested in the present work at equivalence ratios around 2.5. In this present work, an overview of the experimental setup, results for each oil are provided and investigated, including comparisons of different IDT definitions.

## 2 Experimental Setup

The High-Pressure Shock Tube (HPST) available at Texas A&M University (TAMU) was utilized for these experiments. The HPST is constructed with a 4.92-m-long driven section with an inner diameter of 15.24 cm and a 2.51-m-long driver with an inner diameter of 7.62 cm. Attached to the end of the

Correspondence to: am370236@tamu.edu 

shock tube, Cooper et al. developed an injector system to allow for the testing of fluids which remain in liquid or solid form under vacuum [4-7]. Further information can be found within the authors' previous work [4-7]. Based on the injector system, Cooper and coworkers determined that the uncertainty for this system is ± 30% for IDT and ± 1.5% for temperature readings [4-7, 10, 11].

In the present work, there are two different sets of IDTs used. These two definitions are shown in Fig. 1. Emission diagnostics, namely OH* chemiluminescence, were utilized at the sidewall of the shock tube to observe the OH radical produced during combustion [7-9]. The difference in time between the arrival of the reflected-shock front and the maximum rate of change in OH* signal is used to define one type of IDT. Similarly, the pressure-based IDT is the intercept between the steepest slope of the ignition-induced pressure rise and the pressure after the reflected shock ($P_5$). Since the signals are taken at the sidewall, additional time is added to offset the time the reflected shock wave takes to get to the pressure transducer [4-7, 12]. This time, typically 50 µs for the conditions of the present study, is added to both the OH* and pressure IDT definitions.

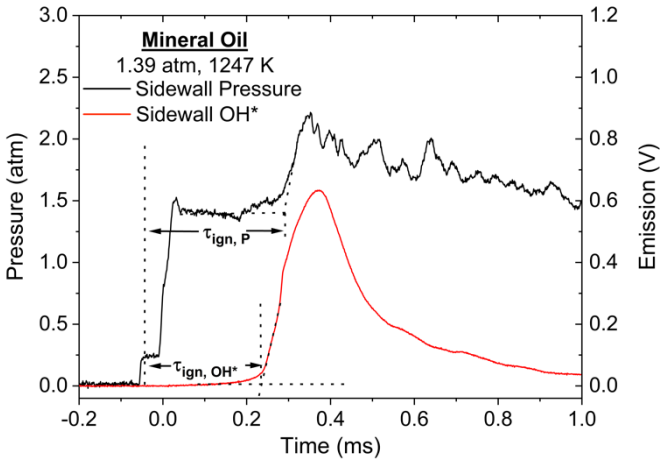


Figure 1: A representative time-history results of a mineral oil test which depicts the two separate definitions of IDTs used.

## 3 Results

### 3.1 n-Hexadecane and Mineral Oil

IDT for n-hexadecane was recorded at reflected-shock pressures of 1.33 ± 0.04 atm. The test temperatures ranged between 1158 and 1263 K. The data points are plotted along with hexadecane from Cooper et al., which were taken at an equivalence ratio near 2.5 using the same spray injector system [7]. Similarly, the IDT for mineral oil was recorded at reflected-shock temperatures and pressures of 1152 to 1287 K and reflected-shock pressures of 1.4 ± 0.1 atm, respectively. These data sets are shown in Fig. 2. Based on the OH* and pressure IDT definitions of n-hexadecane, two separate temperature-dependent correlations were developed. The OH* correlation combines the data from Cooper et al. and this study, since the same method for defining OH* IDT was used. The correlations for OH* and pressure IDT are shown in Eqns. 1 and 2, respectively. For these equations, R is the universal gas constant which equates to $1.987 \times 10^{-3}$ kcal/mol-K, and T is the temperature in K. Additionally, the activation energy is represented by the value in the numerator of the exponential in units of kcal/kmol. From the regressions for n-hexadecane (Eqns. 1 and 2), the $R^2$ values (coefficient of determination) are 0.866 and 0.782 for the OH* and pressure IDT regression, respectively. Additionally, the standard errors for the OH* and pressure IDT regressions are 38.2% and 32.8%, respectively. Similarly, the regressions for mineral oil are shown in Eqns. 3 and 4. Regression analysis of the mineral oil data (Eqns. 3 and 4) led to $R^2$ values of 0.945 for OH* IDT and 0.934 for pressure IDT, respectively. The standard error is

22.8% and 21.8% for OH* and pressure IDT, respectively. The statistical regression values for n-hexadecane and mineral oil are evaluated in the discussion section.

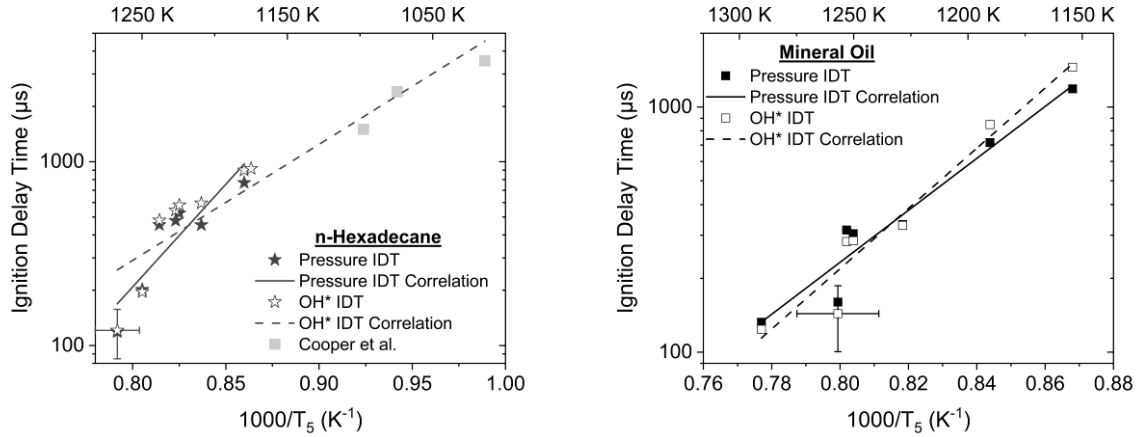


Figure 2: A comparison between OH* and pressure IDT definitions for n-hexadecane (left) and mineral oil (right) at an equivalence ratio near 2.5. The results for n-hexadecane are compared to results from Cooper et al. [7]. The estimated uncertainties of ±1.5% and ±30% in temperature and IDT, respectively, are shown using error bars on both plots.

$$\tau_{ign}(\mu s) = 2.59 \times 10^{-3}\exp(28.9/RT) \quad (1)$$

$$\tau_{ign}(\mu s) = 2.41 \times 10^{-7}\exp(51.1/RT) \quad (2)$$

$$\tau_{ign}(\mu s) = 3.23 \times 10^{-8}\exp(56.2/RT) \quad (3)$$

$$\tau_{ign}(\mu s) = 7.75 \times 10^{-7}\exp(48.5/RT) \quad (4)$$

## 3.2 Mobil DTE 732 and Castrol Perfecto X32

Mobil DTE 732 and Castrol Perfecto X32 are plotted with regression correlations in Fig. 3. The testing conditions for Mobil DTE 732 were at reflected-shock pressures of 1.36 ± 0.05 atm over a reflected-shock temperature range of 1173 to 1292 K. Castrol Perfecto X32 has not been previously tested using the spray injector system, therefore equivalence ratios near 1.2 and 2.5 were tested. The testing conditions for Castrol Perfecto X32 were at reflected-shock pressures of 1.22 ±0.13 atm and 1.35 ±0.09 atm for equivalence ratios near 1.2 and 2.5, respectively. Additionally, the reflected-shock temperature ranges from 1225 to 1383 K and 1193 to 1252 K for equivalence ratios near 1.2 and 2.5, respectively. In addition to this, two-stage ignition phenomena described by Cooper et al. are observed with 4 cases of Castrol Perfecto X32 and were sufficiently small to not affect the sidewall pressure, and therefore used in this work [4, 5]. Similar to n-hexadecane and mineral oil, the correlations created for the IDT of OH* and pressure are provided in Eqns. 5 and 6. The correlations corresponding to Mobil DTE 732 resulted in $R^2$ values of 0.967 and 0.969 for OH* and pressure IDT, respectively. In addition to this, the standard error for OH* IDT is 10.3% and pressure IDT is 10.4%. For Castrol Perfecto X32 at an equivalence ratio of ~1.2, the OH* and pressure IDT correlations are shown in Eqns. 7 and 8, respectively. Similarly, for an equivalence ratio of ~ 2.5, Eqns. 9 and 10 depict the correlations for the OH* and pressure definitions of IDT. For the OH* ignition delay time correlations of Castrol Perfecto X32, the $R^2$ values are 0.875 and 0.613 for equivalence ratios of ~1.2 and ~2.5, respectively. Additionally, the standard errors for OH* IDTs are 15.1% and 23.8% for equivalence ratios of ~ 1.2 and ~ 2.5, respectively. Similarly, the $R^2$ values for Castrol Perfecto X32 pressure ignition delay times are 0.880 and 0.809 for equivalence ratios of ~ 1.2 and ~ 2.5, respectively. Also, the standard error for pressure IDT at an equivalence ratio ~ 1.2 is 16.7%, and at an equivalence ratio ~ 2.5 it is 14.7%.

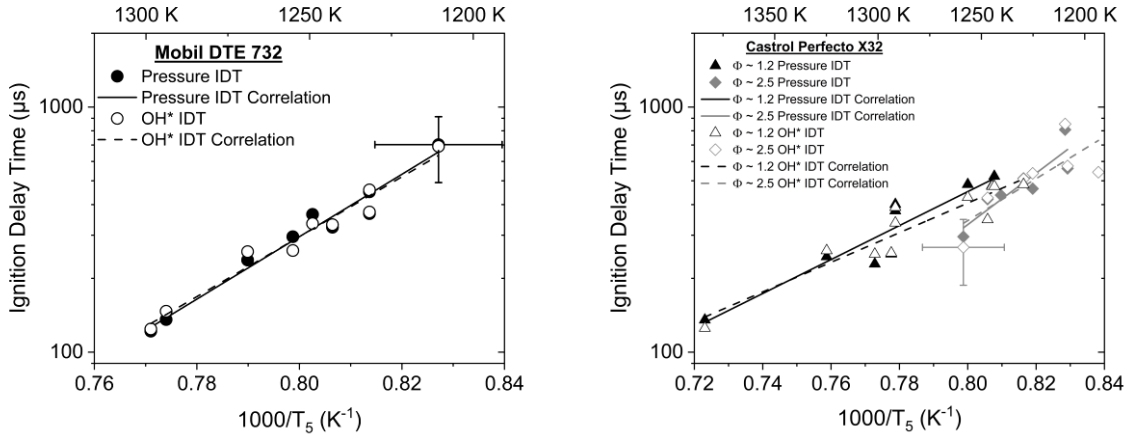


Figure 3: Mobil DTE 732 (left) data taken at an equivalence ratio near 2.5 using the spray injector system described in the experimental setup. Castrol Perfecto X32 (right) data were taken at equivalence ratios of 1.2 and 2.5.

$$\tau_{ign}(\mu s) = 4.96 \times 10^{-8}\exp(55.9/RT) \quad (5)$$
$$\tau_{ign}(\mu s) = 1.92 \times 10^{-8}\exp(58.3/RT) \quad (6)$$
$$\tau_{ign}(\mu s) = 5.96 \times 10^{-3}\exp(27.6/RT) \quad (7)$$
$$\tau_{ign}(\mu s) = 1.29 \times 10^{-3}\exp(31.7/RT) \quad (8)$$
$$\tau_{ign}(\mu s) = 4.96 \times 10^{-5}\exp(39.1/RT) \quad (9)$$
$$\tau_{ign}(\mu s) = 1.33 \times 10^{-6}\exp(48.0/RT) \quad (10)$$

## 3.3 Overall Comparison

Many oils are a combination of similar base oils and additives. As a result, the combustion data for each of the oils are likely similar with minor deviations. Therefore, a grand correlation for all the lubricants are included for each definition of IDT are shown in Fig 4.

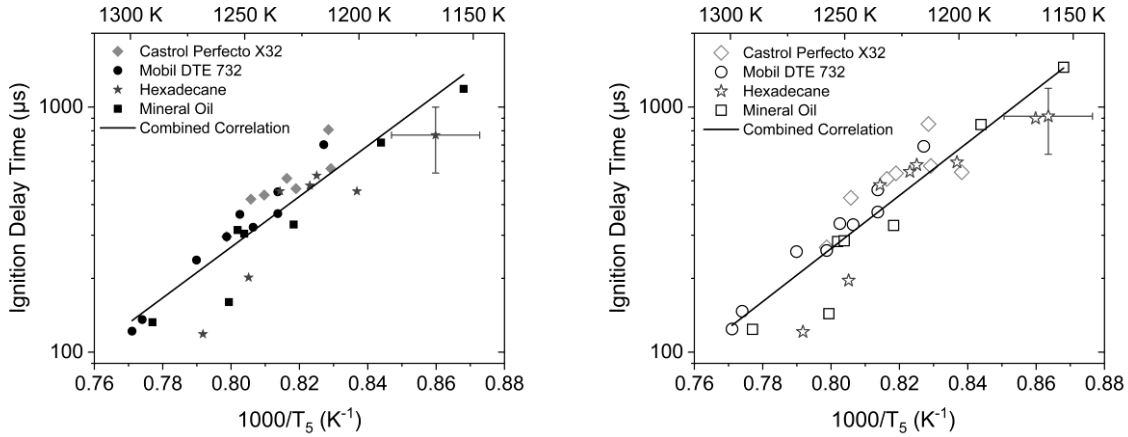


Figure 4: Overall correlation utilizing all the pressure IDT (left) and OH* IDT (right) data for mineral oil, n-hexadecane, Mobil DTE 732, and Castrol Perfecto X32 at an equivalence ratio near 2.5.

As shown in Fig. 4, Mobil DTE 732 and Castrol Perfecto X32 IDT values are all nearby one another. A noteworthy concept observed is the inclusion of n-hexadecane and mineral oil. From Fig. 4, mineral oil and hexadecane seem to stray away from the cluster of data points representing Mobil DTE 732 and

Castrol Perfecto X32. This trend suggests that there might be some additives in the latter two (brand-name) oils allowing for higher combustion temperatures, implying that these compositions are more resilient to combustion than their base oil and surrogate counterparts. Similar to the individual analysis of the lubricating oils, a general correlation was developed for the combined data sets of the lubricating oils. Equation 11 depicts the correlation coefficients for the OH* definition of IDT. The OH* correlation results in an $R^2$ value of 0.845. Likewise, the pressure definition of IDT is shown in Eqn. 12 and provides an $R^2$ value of 0.811. The standard errors for OH* and pressure IDTs are 26.2% and 26.0%, respectively. Further analysis is provided in the following section.

$$\tau_{ign}(\mu s) = 5.83 \times 10^{-7} \exp(49.5/RT) \quad (11)$$

$$\tau_{ign}(\mu s) = 1.43 \times 10^{-6} \exp(47.3/RT) \quad (12)$$

## 4 Discussion

Observing the grand correlation between all the lubricating oils, Castrol Perfecto X32 and Mobil DTE 732 agree well with the uncertainty of the IDT data. A notable trend is the deviation between both the surrogate, n-hexadecane, and mineral oil from the Mobil DTE 732 and Castrol Perfecto X32 brand oils. This difference likely stems from the additives in off-the-shelf products. Additives are meant to increase the performance of the base oils by increasing their resistance to oxidation at high temperatures. As such, IDT data points for the Castrol and Mobil lubricants exist at higher temperatures than mineral oil and hexadecane (by about 16 to 45 K), indicating Castrol Perfecto X32 and Mobil DTE 732 are better at resisting combustion compared to mineral oil and n-hexadecane.

For the spray injector system, a comparison between different ignition delay time definitions was conducted. Such a comparison will set a precedent or guide for all additional data collection using the same system going forward. The correlations regarding mineral oil, Mobil DTE 732, and Castrol Perfecto X32 at an equivalence ratio near 1.2 resulted in differences in $R^2$ values of only 0.011, 0.002, and 0.005, respectively. A higher $R^2$ value is more desirable since the correlation is much stronger, therefore making OH* definition more reliable for mineral oil and pressure definition better for Castrol Perfecto X32 (equivalence ratio ~ 1.2) and Mobil DTE 732. Similarly, for the combined lubricant correlations, the two $R^2$ values differed by 0.034. Lastly, n-hexadecane and Castrol Perfecto X32 produced the largest differences in $R^2$ value. n-Hexadecane produced a difference of 0.084 between definitions, with OH* IDT having a stronger correlation strength than pressure IDT. However, the differences in these data are partly due to the fact that more data points were used for the OH* IDT data set. For Castrol Perfecto X32, the pressure definition for IDT produced an $R^2$ value 0.20 higher than its OH* counterpart. The differences between the two lubricants show the inconsistencies between both definitions. Therefore, OH* definition might not be the best method to describe the ignition delay time for this data set.

An important distinction to make for future use of the spray injector system is a consistent definition for IDT. Based on the sets of lubricant IDT data provided, the pressure- and OH*-based definitions are comparable except for the case of Castrol Perfecto X32 at an equivalence ratio near 2.5. Note that both definitions of ignition delay time are usually found near one another (on a time-history plot), so as long as there is consistency in the data acquisition, similar conclusions may be met moving forward, as shown from the similarities between OH* and pressure correlations for n-hexadecane, mineral oil, Mobil DTE 732, Castrol Perfecto X32 (at an equivalence ratio ~ 1.2), and the combined data sets.

## 5 Conclusion

Four different lubricants (mineral oil, n-hexadecane, Mobil DTE 732, and Castrol Perfecto X32) were tested in a shock tube using a spray injector method developed previously in the authors' laboratory [4-

7]. The tests were conducted at ~ 1.3 atm and at temperatures between 1152 and 1383 K. All of the oils were tested at equivalence ratios near 2.5, with the addition of Castrol Perfecto X32 also being tested at an equivalence ratio near 1.2. Each data set produced reliable correlations of IDT on an Arrhenius plot. Furthermore, IDT was defined using two different definitions, sidewall OH* and pressure. The resulting ignition delay time correlations were compared to one another using a metric for strength of correlation, $R^2$. An overall comparison between the different definitions of ignition delay time was also conducted. Both OH* and pressure definitions resulted in correlations with $R^2$ values within 0.085 of one another. From the IDT for all four oils, it was seen that the two brand-name oils produced longer IDT values than the mineral oil and n-hexadecane. In addition to the refined definitions of ignition delay time, the data can be used to update chemical kinetic models and ultimately produce a reliable model for predicting ignition for the lubricating oils.

## 6 Acknowledgments

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