Ignition Study in Rapid Compression Machine

by

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David E. Hardt
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To my family
ABSTRACT

As it becomes more and more difficult to find "easy" oil, various alternative fuels are introduced to the markets. These fuels have chemical properties that are different from the traditional gasoline and diesel fuels so that engine efficiency and other engine behaviors may be affected. To improve engine efficiency and to identify which alternative fuel is the cleanest fuel solution, it is necessary to compile information about the ignition delay, which governs auto-ignition in spark-ignition (SI), compression-ignition (CI) and homogeneous charge compression-ignition (HCCI) engines.

In this study, we measured ignition delay on the Rapid Compression Machine (RCM). RCM is a single-stroke device, which compresses uniform mixtures to engine-like condition. We can interpret from the pressure the detailed heat release process. A comprehensive ignition delay database of toluene/n-heptane mixtures and gasoline/ethanol mixtures was established. The data allow us to calculate the auto-ignition behavior in engines. Depending on application the correct choice of alternative fuels may be made.

Thesis Supervisor: Wai K. Cheng
Title: Professor of Mechanical Engineering
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Epictetus says decide who you must be, then do what you must do. The last five years at MIT was a valuable journey to find who I must be and to prepare for what I must do. It would be impossible to end this journey without helps and supports from my peoples at MIT as well as in Seoul.

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# NOMENCLATURE

## ACRONYMS

<table>
<thead>
<tr>
<th>Acronym</th>
<th>Description</th>
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<tr>
<td>ATDC</td>
<td>After Top Dead Center</td>
</tr>
<tr>
<td>BDC</td>
<td>Bottom Dead Center</td>
</tr>
<tr>
<td>CAD</td>
<td>Crank Angle Degree</td>
</tr>
<tr>
<td>CI</td>
<td>Compression Ignition</td>
</tr>
<tr>
<td>CN</td>
<td>Cetane Number</td>
</tr>
<tr>
<td>HCCI</td>
<td>Homogenous Charge Compression Ignition</td>
</tr>
<tr>
<td>ICE</td>
<td>Internal Combustion Engine</td>
</tr>
<tr>
<td>ID</td>
<td>Ignition Delay</td>
</tr>
<tr>
<td>IT</td>
<td>Intermediate Temperature</td>
</tr>
<tr>
<td>LLNL</td>
<td>Lawrence Livermore National Lab</td>
</tr>
<tr>
<td>MBT</td>
<td>Maximum Brake Torque</td>
</tr>
<tr>
<td>NTC</td>
<td>Negative Temperature Coefficient</td>
</tr>
<tr>
<td>ON</td>
<td>Octane Number</td>
</tr>
<tr>
<td>PRF</td>
<td>Primary Reference Fuel</td>
</tr>
<tr>
<td>RCM</td>
<td>Rapid Compression Machine</td>
</tr>
<tr>
<td>RPM</td>
<td>Revolutions Per Minute</td>
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<tr>
<td>SI</td>
<td>Spark Ignition</td>
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<tr>
<td>TDC</td>
<td>Top Dead Center</td>
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<tr>
<td>TRF</td>
<td>Toluene Reference Fuel</td>
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<tr>
<td>WOT</td>
<td>Wide Open Throttle</td>
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SYMBOLS

$\lambda$  Air fuel equivalence ratio

$\Phi$  Fuel air equivalence ratio

$\tau$  Ignition delay

$t$  Time

$\theta$  Crank angle

$N$  Revolution per second

$T$  Temperature

$T_{\text{ign}}$  Auto ignition time in engine

$P$  Pressure

$V_d$  Cylinder displacement volume
Chapter 1

INTRODUCTION

1.1 Motivation for the Fuel Research: The fuel challenge

The world needs more fuel for automobiles than it ever has before. Rapid growth among developing nations has significantly increased the global demand for oil, pushing the price of crude oil to record highs over the past years\(^1\). Concerns about high gas prices and the impact of anthropogenic carbon emissions motivate ways to reduce vehicle's fuel consumption or attempt to change petroleum based fuels to more sustainable ones\(^2\). Meanwhile, traditional internal combustion engines (ICEs) will continue to dominate the personal transportation sector for the next several decades. Due to the rigid fueling infrastructure for transportations, it is hard to make drastic changes toward replacing liquid based fuels. Distributed fueling infrastructure comprises 26% of total US energy consumption\(^3\); these significant changes in infrastructure entail high cost and long lead time. The heavy crude oils dependence also causes energy security concerns so that many countries actively invest in alternative energy research for energy independency. In particular, Canada has focused on fuels from oil sand and United State as well as Brazil have focused on ethanol\(^4\).
1.2 Backgrounds of the Fuel Research: *Focusing on ignition delay*

As it becomes more and more difficult to find crude oil, the properties of crude oil have been changing and various alternative fuels have been introduced to the markets. These fuels have various chemical properties that would affect engine efficiency and may alter engine behaviors. The possibility of engineered fuel may provide opportunities to improve engine efficiency and to reduce the carbon footprints. For examples, ethanol could be used as knock suppressants in spark-ignition (SI) engines. Also, various oxygenates could act as soot suppressants in compression-ignition (CI) engines. To explore these opportunities, it is necessary to compile combustion behavior of the evolving characteristics of fuels.

Since it is important to know combustion characteristics of fuels in the engine, not only to improve engine efficiency and identify the cleanest burning solution, but also to predict basic engine performances, it will be needed to setup the new guideline that can elucidate the combustion behaviors of various fuels mixtures. To understand the combustion behaviors in the engines, we must understand ignition delay that leads to autoignition in both SI and CI engines. Ignition delay governs knock that limits the maximum operating compression ratio so that it defines ultimate thermodynamic engine efficiency in SI engines. Also, ignition delay determines ignitability in CI and Homogeneous Charge Compression Ignition (HCCI) engines. For traditional fuels, either Octane Number (ON) or Cetane Number (CN) could be used as a meaningful guideline to predict the ignition delay. In particular, Prime Reference Fuel (PRF) that is mixture of iso-Octane and n-heptane has been denoted as guidance for conventional fuel design. Figure 1.1 shows ignition delay regarding various Octane Numbers in terms of tem-
perature. As it shown, ignition delay generally decreases with respect to temperature increase since chemical reaction rate is generally accelerated with increase in temperature and pressure. However, in the lower ON range, which represents higher n-heptane proportions, the Negative Temperature Coefficient (NTC) behavior is noticeable. In the NTC regime, due to the switching of oxidation paths in n-heptane as well as other large hydro-carbon based fuels, the ignition behavior in engine is complex. Furthermore, with introduction of various alternative fuels in the markets and altering properties of fuels, either ON or CN could not provide meaningful predictions of ignition delay of the fuels as they were used to. In particular, because of the NTC behavior, ignition delay at relatively low temperatures (<1000K) must be carefully defined and the temperature range that we are interested in for the engine combustion study overlap with possible NTC behavior regime.

Figure 1.1 Ignition delay regarding various ON. Points are data (Fieweger et al, C&F 109, 1997) and lines are LLNL model results.
Oxidation generally occurs through four steps; initiation, propagation, rapid multiplication of radicals through degenerated branching and termination. The degenerated branching process plays an important role in ignition delay. Table 1.1 shows a simplified scheme for hydrocarbon fuel oxidation. One of the main oxidation pathways is selected according to the temperature of the mixture. The first part of the propagation step at the low temperature oxidation pathway; \( R + O_2 \leftrightarrow RO_2 \) is reversible so that synthesis and decomposition occur simultaneously although synthesized products are the initiators for further propagation. When temperature is increased to enter the NTC regime, only the decomposition (\( R + O_2 \leftrightarrow RO_2 \)) occurs so that the oxidation propagation pathway for the low temperature is shutdown. On the other hand, at the high temperature, decomposition of branching agent (Hydrogen peroxide; \( H_2O_2 \rightarrow OH^- + OH^- \)) is the key reaction for further propagation, which takes places only enough high temperature. Therefore, in the NTC regime, temperature is high enough to turn off the low temperature oxidation pathway by shifting to the hydrocarbon decomposi-

\[
RH + O_2 \rightarrow \dot{R} + \dot{HO}_2
\]

<table>
<thead>
<tr>
<th>Low Temperature</th>
<th>NTC regime</th>
<th>High Temperature</th>
</tr>
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<tbody>
<tr>
<td>( R + O_2 \leftrightarrow RO_2 )</td>
<td>( RH + HO_2 \rightarrow R + H_2O_2 )</td>
<td>( H_2O_2 + M \rightarrow OH + OH + M )</td>
</tr>
<tr>
<td>( RO_2 \rightarrow \dot{QOOH} )</td>
<td>( HO_2 + HO_2 + M \rightarrow H_2O_2 + O_2 + M )</td>
<td></td>
</tr>
<tr>
<td>( \dot{QOOH} + O_2 \rightarrow \dot{OOQOOH} )</td>
<td></td>
<td></td>
</tr>
<tr>
<td>( \dot{OOQOOH} \rightarrow O=ROOH + \dot{OH} )</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Branching agent (hydroperoxyl carbonyl species)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>( O=ROOH \rightarrow O=RO + \dot{OH} )</td>
<td></td>
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</tbody>
</table>

Table 1.1 Characters of Hydrocarbon Chemical Oxidations, where RH, R, and QOOH are hydrocarbon species, \( O_2 \) and OH are oxidizer, and M is 3rd body.
\[ I = \int \frac{dt}{\tau} = \int_{\tau(T(\theta), P(\theta))}^{0} \frac{dT}{d\theta} \int_{0}^{T(\theta), P(\theta)} \frac{dT}{d\theta} = \frac{1}{2\pi N} \int_{0}^{T(\theta), P(\theta)} \frac{dT}{d\theta} \]

Equation 1.1 Auto-ignition criteria in engines, \( \tau \) = ignition delay, \( \theta \) = crank angle, \( T(\theta) \) = end gas temperature at \( \theta \), \( P(\theta) \) = pressure at \( \theta \), \( N \) = revolution per second.

But, the temperature is not high enough to turn on the high temperature oxidation by activating Hydrogen peroxide decomposition.

Once we compile the ignition delay as a function of temperature and pressure with respect to a specific fuel in a specific fuel equivalence ratio, the ignition delay data allows us to calculate auto-ignition in an engine by following the Auto-ignition criteria.

Auto-ignition criterion is a simple equation that enables us to estimate auto-ignition timing in an engine from the verifying temperature and pressure in an engine, as shown in Figure 1.2 (b). According to the crank angle, Figure 1.2 (b) displays the temperature and pressure in the engine at the moment. Ignition delay will be defined from

Figure 1.2 (a) Points are data (Fiehwege et al, C&F 109, 1997) and lines are LLNL model results, (b) Temperature and pressure profile in an one cylinder Ricardo WAVE engine.
the instant specific condition because ignition delay is a function of temperature and pressure with a specific fuel mixture. Auto-ignition commences when the integral defined at equation 1.1 reaches a threshold value (usually calibrated to be 1). Such auto-ignition leads knock in SI engines and initiates combustion in CI and HCCI engine.

As we discussed earlier, to find this auto-ignition point in engine, it is critical to have complied information for ignition delay with respect to temperature and pressure, like as Figure 1.2.(a). The ignition delay is caused by the hydro-carbon oxidation among the four steps (initiation, propagation, de-generated branching and termination). Because the reaction is fast once the degenerated branching process starts, there is no specific moment to define ignition precisely. One of the traditional ways to define ignition delay is find a point for 20% of total heat release amount as described in Figure 1.3.

The chemical processes that precede rapid heat release are also referred to pre-ignition reactions, $t_1$. Combustion for $n$-heptane or large hydro-carbon molecules fuels may

![Graph](image)

**Figure 1.3** the time interval to 20% of maximum pressure point as the ignition delay, ACCURACY - 95% confidence level: Ignition delay: ±0.14 ms (±1.6%), Max rate of pressure rise: ±7 bar/ms (±12%)
show a clear two-stage ignition. The 1st stage heat release may be up to the 10% of the total heat release. Therefore, the threshold is set to 20% of the maximum pressure to signify the major heat release point in a two-stage heat release process.

1.3 Objectives of This Study

As a partial fulfillment of the requirements for the Mechanical Engineer's Degree, this study focused on the practical interests of a research program. The practical interest of this study is to establish a working data set for predicting the knock in SI engine and ignitibility in CI and HCCI engines. The measurements are done with n-heptane/Toluene mixtures that is sometimes referred to as the Toluene Reference Fuels (TRFs), which is the simplest gasoline surrogate, and Gasoline/Ethanol mixtures. Fundamental interest of this study is to develop basic metrics for chemical kinetics model ignition characteristics of fuel mixtures.

Through this study, comprehensive data set for the Toluene/n-heptane mixtures was completed in terms of various temperatures and pressures. Experimental procedures of liquid fuel handing system for the Ethanol/Gasoline mixture data acquisition were established.
Chapter 2

EXPERIMENTAL SETUP

2.1 Rapid Compression Machine

Rapid Compression Machine (RCM), shown in Figure 2.1, is a single-stroke device that compresses uniform mixtures of fuels and oxidants to engine-like condition. Compression of RCM is induced by the piston movement like engines and the maximum holding force and duration of compression phase is adjustable by changing driving pressure. In this study, compression duration was set as 13ms. In contrast to engines, the piston of the RCM is locked at the end of the compression to produce a constant volume combustion environment. The mixture remains at the end of the compre-
Figure 2.2 RCM compression curve with $\text{N}_2/\text{O}_2$. Initial conditions: 39°C, 357.0 Torr, At the end of Compression: 780 K ($\pm 2$ K), 18 bar ($\pm 0.1$ bar)

...condition for a period of time as shown in figure 2.2. This period is only limited by heat losses. Figure 2.2 shows a compression period and the pressure drop due to the heat loss after the end of compression. However, right after the end of compression, the compressed gas inside the cylinder could be characterized by near iso-thermal processes. Compression with a fuel mixture may have a short delay period of chemical origin during the near iso-thermal processes. The chemical processes that precede ignition and heat release are also referred to as pre-ignition reactions. Combustion then takes place under constant volume condition and the rapid energy release results in significant pressure rise inside the combustion chamber. We measure inside cylinder pressure, which was created by the heat release, by a Kistler 6125A piezoelectric pressure transducer. Therefore, we can interpret ignition delay and maximum heat release rate. A RCM used for the purpose of this study was designed based on the Thornton design\textsuperscript{14} and was built by Kitsopanidis\textsuperscript{15} at 2004. The compression ratio of the RCM is flexible from 9 to 20.2. We can easily alter the compression ratio by changing a com-
bustion chamber head. In case of compression ratio for 16.5, the RCM has 5.08 cm of bore, 20.32 cm of stroke, and 1.27 cm of clearance height. Data acquisition speed for the RCM is 100 KHz so that it permits us to measure up to 0.01 ms accuracy. On the combustion chamber, heating tape is located to alter the initial temperature of the RCM. The combustion chamber could be heated up to 120 °C that corresponds to the temperature, 1100 K, at the end of compression depending on the compression ratio.

Figure 2.3 Picture of Rapid Compression Machine (RCM)

2.2 Experimental Procedure

To get a repeatable and reproducible RCM data, test should be done by a series of followed steps. The protocol of RCM operation is updated and modified based on the RCM manual written by Ioannis Kitsopanidis\textsuperscript{16}. The general guidelines for RCM maintenance and detailed operation manual are presented at Appendix A.
Initial preparations

Check hydraulic oil level: Checking the oil level through the sight tube is prerequisite for every RCM running. The oil level should locate at the recommended level indicated on the control panel shown in Figure 2.4. Use the oil pump and the oil flow regulation valve to pump more oil from the oil reservoir into the RCM. It is recommended to adjust oil flow regulation valve to prevent sudden increasing of the oil level in the sight tube. The oil supply valve should be opened after the oil pump turned on and closed before the oil pump turned off to prevent the oil from draining back to the oil reservoir.

Check driving tank pressure: Since the driving tank slowly leaks, it is recommended to check pressure from the tank pressure gauge on the control panel before preparing the mixture. For the most of experiments performed in this study, driving pressure was maintained at 200 psi, which results 13 ms compression time, and it sustains the maximum combustion chamber pressure up to 100 bar. The compressed air valve on the control panel is used to supply gas (usually inert gas, such as N₂) to the driving tank from gas bottles.
**Turn on heating system:** Since initial temperature, which is required to achieve the targeted condition at the end of the compression, is usually higher than the ambient temperature, we need to heat up the combustion chamber at first. Each fuel chambers, fuel tank, fuel lines and RCM combustion chamber has an independent heating control. In terms of RCM combustion chamber, there are 2 different electrical circuits (combustion chamber body and combustion head). All related controls (switches, temperature displays) are located on the heating panel as shown in Figure 2.6. To achieve and maintain required initial temperature, variable power output controllers, which located underneath the heating panel, need to be used. One should be careful not to exceed the maximum value marked on the controllers to prevent burning of the heaters. In particular, fuel lines should stay below 60 °C to avoid overheating of Baratron pressure gauge that may cause damage of valve seals inside the gauge. Since the heating of fuel lines is to prevent fuel condensation within the lines, it is sufficient to heat the fuel lines up to higher than the fuel dew point. Meanwhile, the temperatures of the fuel reservoirs are recommended to maintain lower than the temperature of the fuel lines.

*Retract and lock the piston at Bottom-Dead-Center position:* To secure the space in combustion chamber for fuel mixtures, the piston should bring to all the way back position that is corresponding to the Bottom-Dead-Center (BDC) in engines. By apply-
After ensuring the piston was fully retracted, around 100 psi pressure should be applied to the hydraulic chamber to prevent any movement of the piston from the BDC position even after the combustion chamber becomes a vacuum for mixture preparation.

**Evacuate the combustion chamber:** By applying the vacuum pump to the combustion chamber and mixture preparation units including fuel lines using the corresponding valves on the mixture preparation setup, we need to evacuate all the air inside combustion chamber as well as fuel lines. Usually it should take less than 5 minutes for a good vacuum to be achieved. If it takes more than 5 mins, need to check the corresponding valves' position on the control panel and the sealing in the combustion chamber whether it is properly maintained. Right after sufficient vacuum is achieved, close the vacuum valve at the end of the fuel lines at first and then shut down the vacuum pump. To make sure that the vacuum is well retained, one need to wait for couple of minutes.

**Mixture preparation**

*Meter the fuel by vapor pressure:* For neat fuels, such as n-heptane, Toluene, and pure ethanol, fuels would be metered by the partial vapor pressure using the fuel reservoirs in the mixture preparation units.

*Meter the fuel by liquid volume:* For gasoline mixtures, such as gasoline and
ethanol mixtures, fuels could not be metered in by the vapor pressure since the gasoline will fractionally distill during the vaporization in the fuel reservoir. Therefore, fuels should be metered volumetrically by micro-pipette using the fuel chamber.

*Mixture components introduction:* After introducing the fuels into the combustion chamber either by vapor pressure using the fuel reservoirs or by liquid volume using the fuel chamber, oxygen (O₂) and inert gas, either nitrogen (N₂) or Argon (Ar), should be fed to the combustion chamber. For each mixture component, the specific procedure should be followed to obtain good data. The detailed procedure will be described at the next chapters that is corresponding to the each fuel.

**Firing**

*Prepare force balance creation:* Before creating the drive force, hydraulic chamber should be pressurized from 100 psi to 1150 psi for 200psi driving pressure by opening slowly the compressed N₂ supply valve. Required final pressure of hydraulic chamber depends on driving pressure and this value could be easily calculated.

![Figure 2.7 Mixture preparation unit of RCM](image-url)
from piston area ratio between in the hydraulic chamber and in pneumatic (driving) chamber. Although the area ratio is little less than 5, it is recommended to multiply 5.1 for the safety reason.

Create driving force: To create driving force on the piston, driving tank should be connected to the pneumatic chamber by turning the compressed air valve to the by-pass position and opening a big yellow valve behind the control panel. Since pressure loss due to the volume change during supplying gas to the pneumatic chamber from the driving tank, the compressed air should supply to compensate the pressure back to 200 psi.

Destruct force balance: Before releasing the pressure in the hydraulic chamber, the pressure transducer should be turned on to its operational mode and the Labview data acquisition system should be activated. To fire the RCM, only action need to be done is the pressure release in the hydraulic chamber and this could be done by momentarily rotating the ball valve to vent the hydraulic chamber on the control panel.

Venting and Cleaning

The big yellow valve behind the control panel should be closed right after the firing. Then, the pneumatic chamber and the gas that pressurized the oil should be vent. It takes around 2 minutes if the valves are half open. During the venting, after saving the data, the pressure transducer should be reset and the mixture preparation setup would be better to vent. After venting is over, open slowly the oil-N2 separation and sight tube supply valves. If another experiment is planned, the experiment should be restarted from Check hydraulic oil level after flushing the combustion chamber with clean air several times. If there is no plan for the further experiments, turn off the
heaters and heating systems. Also, flushing the combustion chamber with clean air several times is recommended to minimize contamination inside the combustion chamber with combustion products after turning off heaters.

2.3 Defining Test Matrix

As we discussed in chapter 1.2, to apply the auto-ignition criterion, ignition delay for the specific fuel should be defined as a function of temperature, pressure and fuel equivalence ratio. Since SI engines in practice are operated at stoichiometric condition, we fixed the fuel equivalence ratio at 1. Then, we varied the fuel mixtures that could represent new alternated fuels. The temperature range in this study should cover from the low end gas temperature to the high end gas temperature. Figure 2.8 represents peak end gas temperature with respect to compression ratio. Green line ($T_{\text{inlet}}=25^\circ\text{C}$) and Red line ($T_{\text{inlet}}=40^\circ\text{C}$) represent results from different inlet gas tempe-

![Figure 2.8](image)

**Figure 2.8** Peak end gas temperature with respect to compression ratio for $V_d=500\text{cc}$, $\lambda=1$, $1500\text{ rpm}$, MBT timing
tatures. Dash line and solid line stand for results from different inlet pressure. Both dash lines are results from naturally aspirated engines with different inlet temperature. Solid line is a turbo charged engine that has higher inlet pressure. As shown in Figure 2.8, peak end gas temperature depends on compression ratio and inlet temperature rather than inlet pressure. The turbo charge engine's charge temperature is not sensitive the inlet pressure. Thus, the effect of turbo charging in auto-ignition is substantially via the pressure rather than the temperature dependence of the ignition delay. To alter pressure, molar density is used in stead of pressure because density is more fundamental variable and molar density is straightforward to control in experiments.

2.4 Sample Experimental Results

Figure 2.9 shows an experiment result of Toluene/n-heptane (50%/50%) mixture by liquid volume percentage for stoichiometric condition at 730 K and 11 bar. To

![Figure 2.9: Pressure trace of RCM for (50% Toluene/50% n-heptane)/N₂/O₂, 210 mol/ m³, λ = 1.](image)

Figure 2.9 Pressure trace of RCM for (50% Toluene/50% n-heptane)/N₂/O₂, 210 mol/ m³, λ = 1,
Initial conditions: 33°C, 240.0 Torr. At the end of Compression: 730 K (±2 K), 11 bar (±0.1 bar) achieve this condition at the end of compression, initial conditions of the RCM are 33°C and 240.0 Torr. The required initial condition of the RCM to achieve a targeted condition at the end of compression can be calculated from fundamental isentropic compression assumption for given the targeted condition at the end of compression by simply applying constitutive relation in the second thermodynamic law. In this study, Fortran code written by Professor Cheng is used to calculate detailed thermodynamic properties of various fuels and inert gas with respect to various RCM compression ratios.

The RCM pressure profiles of Toluene/n-heptane (50%/50%) mixture in Figure 2.9 clearly show two-stage ignition character due to the n-heptane in the mixture. From right after the end of compression, duration till detection of heat release is denoted as $t_0$. From $t_0$ around right after end of 1st heat release, pre-flame reaction takes a place and the amount of heat released during this period depends on the amount of n-heptane in mixture. We will discuss this dependency further more in chapter 3. Point c is 20% of total heat release point defined as ignition delay. Due to two stages ignition shown in Figure 2.9, 10% of heat release point or 5% of heat release point is considerably different from 20% of total heat release point in time domain. However, if the combustion does not have two stages combustion characteristic like as the Figure 2.10 that is relatively high temperature combustion for research-graded unleaded octane number 91 gasoline (UTG91) or like as Figure 2.11 that is relatively low temperature combustion for UTG91, the 10% of heat release point or 5% of heat release point I mostly the same with 20% of total heat release point since heat release propagates quickly once heat release starts. Therefore, to maintain the consistent definition of ignition delay in this
Figure 2.10 Pressure trace of RCM for Gasoline (UTG 91)/N₂/O₂, \( \lambda = 1 \),
Initial conditions: 107 °C, 574 Torr, At the end of Compression: 900 K (±1 K), 29.9 bar (±0.1 bar)

Figure 2.11 Pressure trace of RCM for Gasoline (UTG 91)/N₂/O₂, \( \lambda = 1 \),
Initial conditions: 55 °C, 495.8 Torr, End of Compression: 800 K (±1 K), 26.6 bar (±0.1 bar)
study, we define the ignition delay as 20% of total heat release point. Other plausible ways to define the ignition delay is discussed briefly in chapter 1.2 and it will be discussed further more at chapter 3 and chapter 4 regarding to the each fuel mixture. For some of experimental results for the higher temperature, it is difficult to define ignition delay due to very short delays since there may be significant reaction in the compression period. However, the little bump before starting rapid heat release process in Figure 2.10, caused by heat loss for thermal equilibrium process right after the compression process, allows us to know heat release does not start during the compression process.

2.5 Confirming the RCM performance: n-heptane

To confirm the performance of the RCM, we performed experiments for the simple neat fuel, n-heptane at first. Combustion process of n-heptane in the RCM is very repeatable as shown in Figure 2.12, and consistent with reference data as well as the verified LLNL modeling results as shown in Figure 2.13. In-cylinder temperature represented on the right axis is calculated based on the ideal combustion assumptions without heat loss during the combustion. For the simple estimation, the molar density change during the combustion was ignored. Due to the limit of data sample speed, the shorted ignition delay that could be measure is around 1ms. Order of millisecond is enough to calculate the auto-ignition point in engines. From Figure 2.14, NTC regime is expected to shift higher temperature with respects to pressure increasing. We will discusses further down at the end of the chapter 3.
Figure 2.12 Typical data & repeatability of n-heptane (0 ON), n-C₇H₁₆/N₂/O₂, 180 mol/m³, λ = 1,
Initial conditions: 29 °C, 206 Torr, At the end of compression: 705 K (±1 K), 9.5 bar (±0.1 bar)

Figure 2.13 Experimental data and LLNL simulation results, which consistent with reference data of
n-heptane (0 ON), n-C₇H₁₆/N₂/O₂. 180 mol/m³ corresponds to the end of compression pressure
from 10 bar to 13 bar depends on temperature
Figure 2.14 Ignition delay of n-heptane (n-C\textsubscript{7}H\textsubscript{16}; 0 ON) as very repeatable data used as reference to check RCM operation.
Chapter 3

TOLUENE and n-HEPTANE MIXTURES (TRFs)

3.1 Test Matrix

To establish the Toluene Reference Fuels (TRFs) dependence on the ignition delay, we blended toluene and n-heptane at room temperature by liquid volume percentage of Toluene 0, 25, 50, 60, 65 and 75. Mixture of 35% n-heptane and 65% toluene by liquid volume at room temperature corresponds to 28% n-heptane and 72% toluene by mole fraction. With Toluene Reference Fuels, the number that follows it usually is the Research Octane Number (RON). For example, TRF 84 represents the mixture of 35% n-heptane and 65% toluene by liquid volume that has 84 RON rate\textsuperscript{20,21} and TRF 64 represents the mixture of 50% n-heptane and 50% toluene by liquid volume\textsuperscript{22}. Temperature range of toluene and n-heptane blends in this study is set from 630K to 850K. The high temperature should cover the end gas temperature for knock study and the low temperature should cover the interests in ignitability for HCCI operation study. Density range covered in this study is from 150mol/m\textsuperscript{3} to 400 mol/m\textsuperscript{3}. As discussed in the previous chapter, we use density instead of pressure because density is the more fundamental variable and is more straightforward to control in experiments. We com-
pleted data matrix at 150, 180, 210, 300, and 400 mol/m³ to establish the density dependence of the ignition delay. These density correspond to 9.35, 11.2, 13.1, 16.7, 21.6 bar at 750K. For each initial condition, we repeat the experiments at least 2 times. If the error among the experiments is larger than 5%, we repeated experiments up to 9 times. Data set for TRFs generally show good repeatability except mixture of 60% Toluene and 40% n-heptane by liquid volume. Some challenging issues with regard to data acquisitions for TRFs will be discussed on the later part of this chapter.

3.2 Mixture Preparation Procedures for RCM

One of advantages in the RCM experiment is ability of precise fuel metering either single fuel or multiple fuels. In case of mixtures of neat fuels, we can meter fuels independently by vapor partial pressure. Toluene and n-heptane could be measured and delivered to the combustion chamber separately through vapor fuel metering system. Following process could be done anytime during the initial preparations process in the chapter 2.2. Once the filled fuel reservoirs are ready, they could be used up to three days as far as the reservoir contains the fuel enough to perform an experiment. The amount of fuel in the fuel reservoir could be interpreted from the pressure reader located above the heating control unit.

Prepare filled fuel reservoirs

Vacuumizing the fuel reservoirs and fuel lines using the vacuum pump is prerequisite for filling the fuel reservoirs. It is recommended to isolate the fuel lines and
fuel reservoirs from the rest parts of RCM to maximize the capacity of the vacuum pump since reaching and maintenance of vacuum state inside the fuel reservoirs are critical for the precise fuel metering. After confirming the pressure inside the fuel reservoirs and fuel lines remains zero torr through the Baraton pressure gauge reader on the heating panel, tightly close the needle valves of the fuel reservoirs. The liquid fuel will be filled into the fuel reservoir using a syringe through the syringe guide located on the top part of the fuel reservoir. Before injecting the fuel, make sure there is no air bubble inside the syringe. If any air bubble presents, up-right the syringe and remove the air bubble before injecting the fuel. One fuel reservoir should contain one neat fuel. Once liquid fuel is injected into the vacuum fuel reservoir, the fuel will be evaporated because the extremely low pressure curtails the boiling point of the fuel below the room temperature. If the planned experiment is designed for high density, it is recommended to heating the fuel reservoir to increase capacity of evaporation. However, the temperature of the fuel reservoir should be lower than either that of fuel lines or that of combustion chamber to prevent fuel condensation after leaving the fuel reservoirs.

Figure 3.1 Mixture preparation unit of RCM for vapor fuels metering system
The "Initial preparations process" suggested in chapter 2.2 should be done for starting the following process.

*Mixture components introduction:*

Fortran initial condition calculation code will give the accumulated partial pressure of each mixture components (Toluene, n-heptane, Oxygen, and inert gas (N₂/Ar)) required for aimed target condition at the end of compression. To produce repeatable data, the mixture components should be introduced into the combustion chamber in controlled stepwise manner. It is recommended to wait 5mins between introductions of each component to make sure the thermal equilibrium as indicated by pressure. If the pressure reduces after 5mins, add trim mixture components to get to correct pressure. If the pressure increases after 5mins, use the bumper to suction trim mixture component to get to correct pressure. If change of the pressure after 5mins is more than 5% of total pressure, it is recommended to evacuate the combustion chamber along with fuel lines and regulate the heater to maintain thermal equilibrium in the combustion chamber. Once the temperature is stable more than 15mins, restart the mixture components introduction procedure. After delivering all the mixture components to the combustion chamber, wait 15mins to confirm the equilibrium state of the mixtures inside the combustion chamber.
3.3 Typical Data and Repeatability

As discussed in chapter 3.1, the RCM results for TRFs are very repeatable and reproducible in general. Figure 3.2 shows a typical raw experimental result and a filtered data for toluene/n-heptane (50%/50%) blend by liquid volume. Blue line represents a raw data from the RCM and dashed line represents a filtered one. Temperature shown in the right axis was calculated for constant volume combustion and molar density in combustion chamber is assumed as un-burned molar constant density during the combustion for simple calculation. It shows the two stage heat release process and the temperature as well as time till 5% and 20% of total heat release points. Figure 3.3 shows repeatability of experimental data. This graph represents three repeat experiments and those experiments are consistent with each other.

**Figure 3.2** Raw and filtered pressure traces of RCM, TRF 64 (n-heptane 50%/toluene 50%)/N$_2$/O$_2$, 180 mol/m$^3$, $\varphi = 1$, Initial conditions: 33°C, 208.3 Torr, End of Compression: 730 K ($\pm$2 K), 9.7 bar ($\pm$0.1 bar)
3.4 Issues in Ignition Delay

As we discussed, large hydrocarbon fuels have two-stage heat release character. In oxidation process of fuels, the propagation process is usually rapidly preceded by the initiation process. Therefore, there is no significant interval from the first sign of heat release to either the maximum rate of pressure-rise \((dP/dt)_{\text{max}}\) point or 20% pressure rise point. On the other hand, in case of the heat release process that is involved in large hydrocarbon molecules, there is significant interval from the first sign of heat release to either the maximum rate of pressure-rise point or 20% pressure rise point. For combustion of n-heptane and Toluene mixtures, since the amount of heat release during the first stage heat release depends on the amount of n-heptane in mixtures, it would be better to choose a point that does not depend on amount of the first stage.
heat release to decouple dependence on amount of \( n \)-heptane in mixture regardless initial condition. Amount of heat released during the first stage heat release and duration till the second stage heat release considerably depend on toluene proportion and initial temperature and pressure. Figure 3.4 shows either 5% pressure rise point or 10% pressure rise point could be a function of amount of \( n \)-heptane in the mixture. Therefore, 20% pressure rise point or larger than 20% pressure rise point, such as maximum rate of pressure-rise point, would be meaningful. However, the temperature of mixtures at this point is not the same temperature which was designed for the end of compression temperature as initial temperature because temperature changes due to the first stage heat release. As shown in Figure 3.5, since there is no significant difference between the point for the maximum rate of pressure-rise and 20% heat release point, we will use 20% of heat release point as ignition delay.

![Figure 3.4 Ignition delay defined as 5%, 10%, 20%, 50% and 95% of total heat release points for the RCM results of TRF 0(pure \( n \)-heptane), TRF 64 (toluene 50% / \( n \)-heptane 50%), and TRF 84 (toluene 65% / \( n \)-heptane 35%) with respects to temperature.](image-url)
Figure 3.5 Ignition delay defined as points for the maximum rate of pressure-rise and 20% heat release points for the RCM results of n-heptane with respects to temperature at 150 mol/m$^3$ and 210 mol/m$^3$.

Now, we move on discussion for the relation between the amount of 1st stage heat release and toluene proportion in the mixture with respect to initial temperature. Figure 3.6 shows that the amount of heat that is released during the 1st stage combustion is proportional to the amount of n-heptane in the mixture as we expected. As shown in the figure, the two stage heat release sometimes leads to clear two stage ignition delays so that ignition delay is distinguishable although heat release starts before the end of compression. Therefore, the ignition delay should be carefully interpreted for 25% toluene/75% n-heptane mixture at 740K or 50% toluene/50% n-heptane mixture at 760K or 815K since the real temperatures at the end of the compression is not the targeted initial temperatures. It is recommended further study to assign the meaningful ignition delay of these specific cases. Although the heat release started before the end of compression, the secondary ignition delay may play a role to retard the knock in SI engine as well as the start ignition in CI and HCCI engine.
Figure 3.6 Dependence of amount of 1st stage heat release on temperature and the \( n \)-heptane contents in the fuels. Experimental results for 25\% toluene/75\% \( n \)-heptane mixture and 50\% toluene/50\% \( n \)-heptane mixture with respect to various temperatures at 210mol/m\(^3\).

Figure 3.7 shows data which have similar main heat release point for 60\% toluene/40\% \( n \)-heptane blend at 750K regardless pressure. Although these data have similar main ignition delay coincidentally, the figure reveals that 1\textsuperscript{st} stage ignition delay as well as the amount of heat released during the 1\textsuperscript{st} stage ignition depends on the pressure. However, the dependence is not straightforward. The same main ignition delay, which is defined as 20\% of total heat release point, for different mixtures or at different conditions does not indicate that the ignition character including the heat release process would be the same. So, we need to be careful when we exam only the graph of ignition delay data in the next section.
Figure 3.7 Dependence of the 1st stage ignition delay and the amount of heat released during the 1st stage ignition on the pressure, experimental results for 60% toluene/40% n-heptane mixture with respect to pressure at 210 mol/m$^3$.

Figure 3.8 Dependence of 1st stage ignition delay heat on the initial temperature, experimental results for 65% toluene/35% n-heptane at 210 mol/m$^3$. 
Figure 3.8 shows the relation between the duration till 1st stage heat release, which is referred as the 1st stage ignition delay, and initial temperature. Although the dependence is not significant, it shows that the 1st stage ignition delay also depends on initial temperature, like the overall ignition delay. The initial conditions of data shown in the Figure 3.8 may locate the NTC behavior regime for 65% toluene and 35% n-heptane mixture. The 1st stage ignition delay decreases as temperature increases in general and the relationship could be interpreted from the previous Figure 3.4 by focusing on the 5% of heat release point even though the amount of heat that is released during the 1st stage combustion for 65% toluene and 35% n-heptane mixture is smaller than 5% of total released heat. Also, Figure 3.9 shows the relation between the first stage ignition delay and n-heptane contents in the mixture. Although the main ignition delay of these experimental data was hard to tell the dependence of n-heptane contents,

![Graph showing heat release vs time for toluene mixtures](image)

**Figure 3.9** Dependence of 1st stage ignition delay heat on the n-heptane contents in the fuels, experimental results for toluene and n-heptane mixtures (60%/40% and 65%/35%) with respect at 210 mol/m³ and 745K
Figure 3.10: Inconsistence experimental results of toluene/n-heptane (60%/40%) mixture, in particular at 180 mol/m³ and 210 mol/m³. Each data point is collected spontaneously. Lines are interpolated based on the average of collected data.

the 1st stage ignition delay clearly shows dependence on n-heptane contents and the first stage ignition delay decreases as increase n-heptane contents in the fuels, in particular at this condition. On the other words, when we focusing on the same fuel at the same condition, the main heat release points are different from each other although the first stage heat release process was the same. Relatively non-repeatable data for high contents of Toluene mixture was issues during the data acquisition. Figure 3.10 shows inconsistence experimental results of toluene/n-heptane (60%/40%) mixture. According to the Figure 3.10, ignition delay is not proportional to pressure unlike other mixture results that show ignition delay is generally decreasing as pressure is increasing although the dependence of ignition delay on pressure is very complex. As shown in Figure 3.9, the different main heat release character in toluene/n-heptane (60%/40%) mixture and toluene/n-heptane (65%/35%) mixture are not caused by the inconsistent initial condition during the experiment since the compression process as well as
the 1st stage heat release process is identical among the repeat experiments. Also, because the data was not collected consecutively, the inconsistent main heat release was not likely cause by systematic error. Also, these inconsistence results were happened only at the specific pressure at the certain range of the temperature. Therefore, this phenomenon could be originated from a sensitive oxidation reaction of Toluene rather than n-heptane and it needs to be studied further more. The sensitive analysis of Toluene oxidation might be a good point to start.

3.5 Results for Toluene/n-heptane blends

Comprehensive set of ignition delay data are obtained for Toluene and n-heptane mixtures. By liquid volume, Toluene content in the mixtures is 0%, which is pure n-heptane, to 75%, which is 75% of toluene and 25% of n-heptane blend, and temperature covered on the data set is from 630K to 850 K. Density explored in the data set is from 150 mol/m$^3$ to 400mol/m$^3$. Based on the data set, ignition delay increases with Toluene content and decreases with change density. Dependence of ignition delay on toluene content and density is very complex since shape of ignition delay as a function of temperature changes with pressure. In following figures, symbols represent real initial temperatures where data collected and lines are interpolated based on collected data. The “real” initial temperature means that it is calculated back from the condition at end of compression from the experimental results. Because we assume the isentropic compression process when we calculate initial RCM experiment conditions from the targeted condition at the end of compression, the “real” initial condition at the end
of compression is different from the targeted condition at the end of compression. Moreover, when the 1st stage heat release happened during the compression process, the "real" initial condition at the end of compression is higher than the targeted condition at the end of compression. Following figures represent obtained RCM results sets.

**Figure 3.11** Ignition delay of toluene/\textit{n}-heptane mixtures by liquid volume for various temperatures at 150 mol/m$^3$.

**Figure 3.12** Ignition delay of Toluene/\textit{n}-heptane mixtures for various temperatures at 180 mol/m$^3$. 

50
Figure 3.13 Ignition delays of Toluene/\(n\)-heptane mixtures for various temperatures at 210 mol/m\(^3\)

Figure 3.14 Ignition delays of Toluene/\(n\)-heptane mixtures for various temperatures at 300 mol/m\(^3\)
Figure 3.15 Ignition delays of Toluene/\(n\)-heptane mixtures for various temperatures at 400 mol/m\(^3\).

Figure 3.16 Ignition delay of specific toluene/\(n\)-heptane blend in terms of different density with respect to temperature.
Figure 3.17 Ignition delay results with reference points\textsuperscript{23,24}, RCM experiment results were obtained at 180mol/m\textsuperscript{3} that is corresponding to 9.2 bar - 12.6 bar depending on temperature.

Figure 3.17 shows reference shock-tube experimental data from Herzler et al\textsuperscript{23} and numerical simulations using the semi-detailed chemical kinetic model\textsuperscript{24}. Although the temperature covered in those two studies is relatively higher then temperature ranges which we are interested in for the knock study, projection of previous studies agree well with experimental results in this study. Figure 3.18 shows ignition delay in terms of specific temperature. As we discussed earlier, although the dependence of ignition delay on toluene content and density is very complex since the shape of ignition delay as a function of temperature changes with pressure, the dependence of ignition delay ($\tau$) on the density can be represented as the power law dependence, $\tau \propto (\text{density})^{n}$ where $n$ is a function of temperature and $n$ proportionally increases as temperature increases.
Figure 3.18 Ignition delay in terms of specific temperature (680K, 730K and 780K) with respect to molar density.
4.1 Background of Ethanol Study

In recent years, considerable attentions to ethanol not only as a gasoline supplement in low concentrations (E10, E20) but also a major fuel (E85, E100) have increased dramatically because of concerns associated with conventional transportation fuels\textsuperscript{25}. Ethanol have been considered as one of the promising sustainable candidate for the next generation fuels and, at the same time, as a pathway to reduce energy dependency on middle east crude oils regardless of sources of Ethanol, either cone or switch grass. The commercial fuels in the gas station are already contained Ethanol up to 10%. However, fundamental ethanol ignition study has not been done extensively focusing on engine-like conditions, although broad performance data are introduced regarding the engine experiments. In particular, there is no available neat combustion data for the ethanol gasoline mixtures as well as ethanol/neat fuel mixture at high pressure. Therefore, there are numerous opportunities in ethanol study focusing IC engine applications. However, one of the challenges is that practical ethanol composition is not standardized. For example, Brazil’s E100 contains 95% Ethanol/5% water and E85 in win-
ter contains 80% Ethanol/15.8% gasoline/3.8% butane/0.4% water and E85 in summer contains 80% Ethanol/18.1% gasoline/1.5% butane/0.4% water. In this study, we will start from simple neat mixtures with Ethanol and gasoline (UTG 91), which is ON 91 unleaded test gasoline. Each mixture was prepared by liquid volume fraction such as E100 that means Pure Ethanol and E15 that means mixture of 15% Ethanol/75% UTG 91 based on liquid volume.

Because we have encountered many challenging issues with experiments, many of the RCM parts and experimental procedures has been improved and modified. There are two major improvements. One is with respect to fuel delivery into the combustion chamber by the liquid fueling procedure. The other one is regarding mixture component introduction procedure into the combustion chamber to enhance the mixing within the combustion chamber. We will discuss these improvements further down in the next section. Meanwhile, most of data that are represented in this chapter was performed without these modifications so that there may be substantial data quality issues. However, since most problems that was founded and improved was regarding the absolute amount of fuel delivered into the combustion chamber due to the changed fueling system, the mixture combustion profile and behavior would be worth to exam in terms of future experiments.

4.2 Mixture Preparation Procedures for RCM

4.2.1 Establish liquid fuel metering system

As briefly discussed in Chapter 2, because gasoline will be fractionally distilled if we use the fuel reservoir to make gasoline vapor, we need to meter the fuel other than
the vapor partial pressure. One of ways to meter the fuel is by liquid volume with a micropipette. By using a micropipette, the amount of fuels into the RCM could be controlled as a liquid forms although the fuel mixtures should be delivered into the combustion chamber after vaporized. Therefore, we designed a fuel chamber in the middle of the fuel lines as shown in Figure 4.1. The fuel chamber can be isolated from the RCM during the fuels metering by closing ball valves located on the both ends. Electrical heaters invaded the fuel chamber to vaporize the metered liquid fuels before it is delivered into the RCM combustion chamber. To make sure that all the metered fuels inside the fuel chamber is delivered into the combustion chamber; the clearance volume inside the fuel chamber should be minimized when it is designed. The liquid fuel metering system should be carefully examined further because of the high sensitivity depending on the processes. The remedies which had been taken to improve RCM performance will be discussed at the later part of this chapter. Following process should be done right after the completion of Initial preparations process suggested in chapter 2.2.

**Figure 4.1** Mixture preparation unit of RCM for liquid fuels metering system
4.2.2 Mixture preparation process

*Cooling the fuel chamber:* to minimize evaporation of fuel during the metering process, it is recommended to cool down the fuel chamber as well as the mixtures to around 5°C. To avoid possible water condensation inside fuel chamber, in particular under the humid experimental condition in the summer, it is recommended not to cool down to under 0°C. Liquid nitrogen and compressed air were used to perform the cooling in this experiment.

*Inject the fuel into the fuel chamber using a micropipette:* right before metering the fuel with a micropipette, it is recommended shaking the mixture to improve homogeneous of mixture. While handing the micropipette, maintaining same height with a tip of the micropipette and a mixture container is recommended by the manual of the micropipette. Right after injecting the fuel into the fuel chamber, close cap of the fuel chamber with a plug and turn on the heater for the fuel chamber.

*Heat up the fuel chamber:* to convert fuels in the fuel chamber from liquid to vapor before delivering the fuel to the combustion chamber, the fuel chamber is needed to be heated till above the boiling point of fuels using the electronic heater. It is recommended heat up to 100°C within 5mins and maintain the temperature during 3mins to make sure all the fuel injected into the fuel chamber is converted into vapor.

*Mixture components introduction:* blowing the vaporized fuel into the RCM should be performed in controlled stepwise manner. Before opening the ball valves which isolate the fuel chamber from the combustion chamber and fuel lines, fuel lines should be pressurized by O₂ to prevent back flow from the fuel chamber to the fuel lines. After pressurizing the fuel lines with O₂, open the ball valve connected to the
combustion chamber at first and then open the other ball valve connected to the fuel lines. After delivering the right amount of oxygen into the combustion chamber, fill the combustion chamber with inert gas (N₂/Ar) till required initial pressure. Strong final blow is recommended to enhance mixing inside RCM. During the steps, pressure could be read from the Baraton gauge through the heating panel and it is recommended to wait more than 5 mins between the introduction of oxygen and the introduction of inert gas for thermal equilibration as indicated by pressure and then add trim air to get to correct pressure. Detailed manual combined with RCM component numbers and suggested time lines will be attached at the Appendix.

4.2.3 Typical Data and Repeatability for UTG 91

Following figures shows typical test data for UTG 91. Data for UTG 91 have good repeatability, in particular at short ignition delay. Unlike the n-heptane combustion, UTG 91 of heat release process is rapid once it is initiated.

![Graph of UTG 91 data](image)

**Figure 4.2** Typical UTG 91 data for short ignition delay, Gasoline (UTG 91)/N₂/O₂, λ = 1, Initial conditions: 107 °C, 574 Torr, End of Compression: 900 K (±1 K), 29.9 bar (±0.1 bar)
4.3 Discussion of issues in ethanol mixture experiment on RCM

4.3.1 Cool Flame Combustion of Ethanol: Difficult to Define Ignition Delay

Figure 4.4 shows typical experimental data for E85, mixture of Ethanol 85% and UTG91 15% by liquid volume based. Unlike the UTG91 combustion profile in the previous section, high content of ethanol shows clear two stage ignition due to the cool flame combustion period of Ethanol oxidation. Therefore, defining the ignition delay as an end point at 20% pressure rise or maximum rate of pressure-rise: \((dP/dt)_{\text{max}}\) point would not be meaningful because temperature changes significantly during the 1st stage heat release process referred as cool flame combustion. If we use 5% pressure rise point as end point for defining ignition delay, temperature will be relatively well defined for the ignition delay as a function in terms of temperature and pressure expre-
Figure 4.4 Raw and filtered pressure traces of typical experimental data for E85

(Ethanol 85% + UTG91 15%)/N₂/O₂, 400mol m³, φ = 1, Initial conditions: 59 °C, 502 Torr,

At the end of Compression: 800 K (±1 K), 26.6 bar (±0.1 bar)

At the end of Compression although the ethanol mixture may not “fully auto-ignite” at that point. When we focus on the knock study in the engine, however, using (dP/dt)ₘₐₓ point as “ignition point” may be more appropriate because we concern more about the rapid heat release phase for engine knock behavior although the temperature of mixtures at this point may not be the same with the temperature for which the experiment was originally designed as the end of compression temperature. In fact, for E85 as shown in Figure 4.5, either using 5% pressure rise point as end point or (dP/dt)ₘₐₓ point as end point does not significantly effect on the results in terms of trends although ignition delay itself will have off-sets depending on the definition.

Because cool flame period depends on temperature, conventional ignition delay defined according to specific heat released point may not describe effectively combus-
Figure 4.5 Typical RCM data for E85 (Ethanol 85% + UTG91 15%)/N$_2$/O$_2$, 400mol/m$^3$, $\phi = 1$
Initial conditions: 44~75 °C, 479~526 Torr, At end of Compression: 770~830 K

Figure 4.6 Pressure profiles at different initial temperatures for Ethanol/N$_2$/O$_2$, 400mol/m$^3$, $\phi = 1$,
Initial conditions: 85~95 °C, 504~526 Torr, Various end of Comp. temperatures: 850~870K
tion of gasohol. Figure 4.6 shows two ignition delay result for the neat ethanol fuel for different temperatures. According to the experimental data shown in the figure, if we define the ignition delay as 5% pressure rise point for end point, shorter ignition delay does not guarantee that the real combustion occurred faster as well. Moreover, because heat release characteristics heavily depend on fuel compositions as well, the difficulty to defining ignition delay is more challenging when we explore different proportion of gasohols. Figure 4.7 shows combustion profiles for various ethanol and gasoline mixture under the same condition. Each pressure trace represents each fuel mixture under the same initial condition. Although the amount of heat released during the cool flame period is relatively proportional to the amount of ethanol in the mixtures, the ignition delay regardless of the definition is out of proportion to the fuel compositions.

Figure 4.7 Combustion profiles for various ethanol and gasoline mixture at 400 mol/m$^3$, $\phi = 1$ and 850 K at the end of compression
4.3.2 Post Heat Release During Heat Loss

One of challenging issues for the ethanol mixture experiments was the post heat release during heat loss process. The phenomena could monitor as a second pressure peak after the one associated with the primary heat release process. Since this issue was revealed with repeatability issue in the higher contents of ethanol mixture fuels, it was hard to exam whether the causation of these two issues would be the same or not.

Compression and fuel metering were unlikely the course of the variations because data with gasoline for checking repeatability were consistent regardless of acquisition time and compression process for gasohols was very repeatable although heat release process was not. In particular, the post heat release during the heat loss was not caused by liquid fuel metering process since the post heat release was happened regardless fuel metering method.

Also, compression and fuel metering were unlikely the cause heat release during heat loss regardless of methods of fuel metering. As discussed in the previous section, since the liquid fuel metering system introduced because of the gasoline in the mixtures, the neat ethanol could be metered by vapor pressure. Post heat release phenomenon was only frequently observed with ethanol mixtures although some changes rate of heat loss during the heat loss process was observed with toluene/n-heptane mixtures as shown in Figure 3.6. Therefore, the post heat release phenomenon was likely caused by one of oxidation characteristics of ethanol. According to pre-compression chemistry in literature search\(^{27}\), although oxidation for pure ethanol is very slow under 550K, reactions may occur in minutes when impurities such as oxidation products of HC are present. Figure 4.8 shows that the ignition delay as well as the
Figure 4.8 Different heat release profiles depending on the residential time within the combustion chamber after introduction of all the mixture components (E100/N2/O2) at 400mol/m$^3$, $\varphi = 1$, Initial conditions: 86 °C, 543.2 Torr., At the end of Compression: 850 K (±1 K), 26.6 bar (±0.1 bar),

post heat release depends on the residential time of the mixtures within the combustion chamber. Experiments were performed more than twice for each condition to confirm repeatability. Therefore, we established the intermediate species sampling system to assess any pre-oxidized components of ethanol exist in the combustion chamber during the residential period before compression. However, there was no significant species other than introduced mixture components. Another possible explanation for the post heat release is the post ignition due to the diffusion of residual ethanol within the clearance volume, such as between the piston and Vaton sealing. Enhanced oxidation of ethanol by impurities within the combustion chamber during the heat loss could be plausible cause for the fuel dependence on post heat release phenomenon$^{28}$. The fundamental mechanism why post heat release happened should be established further down.
4.3.3 Improved Features of RCM

**Liquid Fuel Metering System**

As we pointed, because the purpose of liquid fuel handling system was to prevent the partial distillation of gasoline in the mixtures, we experimented on pure ethanol both with vapor fuel metering system and with liquid fuel metering system under the same condition. As shown in Figure 4.10, the peak pressures from original liquid fuel metering results is not consistent with that from vapor fuel metering results. There could be two possible explanations for the inconsistency. One is fuels that is delivered by liquid fuel metering system could not fully ignite in the combustion chamber. The other is all the amount of fuels that is injected into the fuel chamber volumetrically by liquid fuel metering system could not be delivered into combustion chamber. As shown in Figure 4.9, 60% of volume inside fuel chamber is occupied by the clearance volume under the cap where the gaseous fuel, after the fuel chamber heating, could be trapped during introduction mixture components into the combustion chamber. By designing a plug that may eliminate the clearance volume inside the fuel chamber, deliver ratio of injected fuels was improved as indicated by improved peak pressure in the Figure 4.10 for new liquid fuel metering system.

![Figure 4.9](image)

*Figure 4.9*
Fuel chamber for liquid fuel metering system
Figure 4.10 Peak pressures improvements using a plug under the cap of the fuel chamber. E100/N2/O2, 400 mol/ m³, φ = 1, Initial conditions: 86 °C, 543.2 Torr At the end of Compression: 850 K (±1 K), 26.6 bar (±0.1 bar)

Mixing within the combustion chamber

After designing a new plug for the fuel chamber to reduce clearance volume inside the fuel chamber, although peak pressure has been improved, there is more than 10% of the peak pressures loss comparing with that of vapor fuel metering results. Since there is no more dummy volume inside the fuel tank besides the volume that is consistent with the fuel lines heading toward the combustion chamber, it is reasonable to assume that all the injected fuels into the fuel chamber as liquids would be delivered to the combustion chamber in so far as the fuels transform into vapor during the fuel chamber heating. The fuel chamber was heated up to 100 °C for a while above at the atmospheric pressure, although the boiling temperature of ethanol 78.4 °C at the atmospheric pressure. Therefore, it is unlikely there is remained liquid fuel after the heating. Also, because the fuel lines from the fuel chamber to the combustion chamber were heated appropriately, it is unlikely the compromised peak pressure was caused
by the condensation of the ethanol during the ways. Therefore, the loss of peak pressure is likely caused by the partial ignition of the delivered fuels within the combustion chamber. One possible explanation of the partial ignition is dependence on sequence of introduction of mixture components. When we meter the fuel by partial pressure, the order of introduction of mixture components is fuel, oxygen and inert gas. However, to enhance the first blowing out fuels inside the fuel chamber into the combustion chamber, the inert gas that occupies the largest partial pressure among the mixture components was introduced into the combustion chamber before the oxygen when we meter the fuel volumetrically using the micropipette. For the stoichiometric mixtures, inhibition of oxygen due to the inadequate mixing in the combustion chamber may lead incomplete combustion. To maximize the opportunity of fuel to contact with oxygen, we alter the procedure sequence for introduction of mixture components in liquid fuel metering process. As indicated in Figure 4.11, the peak pressure of the improved procedure with new plug is almost consistent with that of vapor fuel metering system.

![Figure 4.11](image)

Figure 4.11 Peak pressures improvements by changing the introduction order of mixture components into the combustion chamber. F:100/N₂/O₂, 400 mol/ m³, φ = 1, Initial conditions: 86°C, 543.2 Torr,
4.4 Ignition Delay Result of Ethanol and Gasoline Mixture

From this study, basic set of ignition delay data obtained for various Ethanol and Gasoline mixtures from the neat ethanol to the neat gasoline. Temperature range covered by the experiments was 630K to 850 K at 400 mol/m³. As we discussed before, since the fuels are not sufficiently delivered to the combustion chamber during the following data acquisition periods, experiments should be repeated to confirm the data. Still, the trends of ignition delay may represent lean mixture combustion because the performed improvements are aimed to enhance the delivery ratio to the combustion chamber when liquid fuel metering system is utilized. According to the results, although ignition delay of ethanol and gasoline mixtures was expected to be increased proportional to the portion of ethanol in the mixture, the ignition delay and the ethanol contents in the mixture do not show noticeable relation. Complexity of the dependency on Ethanol contents in the mixture is higher than commonly expected linear behaviors.

Figure 4.12 Ignition delay result of Ethanol and Gasoline mixtures, 400 mol/m³, φ = 1
Chapter 5

SUMMARY AND DISCUSSION

5.1 Overviews

Due to the increased concerns about the conventional oil supply, fuels from non-petroleum forms (such as plants and oil shale) are introduced either as supplement or as replacement. There are fuel optimization opportunities for engine applications. A significant attribute which would impact engine operation in the ignition delay that governs knocks in SI engines and limits ignitibility in CI and HCCI engines. As a partial fulfillment of the requirements for the Mechanical Engineer's Degree, this study focused on the practical interests in assessments of extensive ignition data set for Toluene/n-heptane mixtures and Ethanol/Gasoline mixtures. Experiments in this study were designed and performed on the Rapid Compression Machine (RCM). The RCM is a single stroke piston apparatus in which a fuel/air mixture is compressed up to engine-like condition to create constant volume combustion. Unlike engine experiments that are difficult to control or interpret due to the spatially as well as temporal change of fuel composition and thermal state during the experiments, the RCM is straightforward to control the thermal state and the fuel metering, in particular for the mixture of fuels. Also, unlike the shock tubes experiments, the RCM allow us to perform the experiment.
at the high pressure. Through this study, comprehensive data set for the Toluene/n-heptane mixtures was completed with respect to various temperatures and pressures and experimental procedures of liquid fuel handing system for the Ethanol/Gasoline mixture data acquisition were established.

5.2 Summary of Experimental Data

Toluene and n-heptane Mixtures

Comprehensive set of ignition delay data are obtained for Toluene/ n-heptane mixtures; 0%/100%, 25%/75%, 50%/50%, 60%/40%, 65%/35%, and 75%/25% by liquid volume. Temperature, from 630K to 850K, and density, from 150mol/m³ to 400mol/m³, was explored in the data set. Based on the data set, ignition delay increases with Toluene content and decreases with change density. Although dependence of ignition delay on toluene content and density is very complex since shape of ignition delay as a function of temperature changes with pressure, the dependence of ignition delay on the density could be interpreted as the power law dependent where the power constant is a function of temperature that proportionally increased as temperature increased.

Ethanol and Gasoline Mixtures

RCM liquid fuel handing system was developed and experimental procedures of liquid fuel handing system for the Ethanol/Gasoline mixture data acquisition were established. Ignition delay results of Ethanol and Gasoline mixtures from E0 to E100 was
obtained at 400 mol/m³. Although the results should be confirmed through further experiments, the results suggested the complexity of the dependency on Ethanol contents in the mixture is higher than commonly expected linear behaviors.

5.3 Recommended Future Works

For the experimental wise, comprehensive data sets for ethanol and gasoline mixtures should be completed with the established procedures in this study and then ignition measurements could be continued for ethanol/n-heptane and ethanol/iso-octane mixtures. In terms of modeling work, development and validation of chemical kinetic mechanisms that are consistent with obtained experimental results should be finished to enlarge the opportunities in application of experimental data set. Study for the calibrations of mixture kinetics focusing on engine-like conditions should be preceded.

Establish ignition delay set is critical since the data set could be an effective guideline as fuel design. Once we complete the data of ignition delay for the various fuel mixtures, it could be complied into the entire system-wise analysis to access strategic opportunities in improving fuel efficiency as a whole. It may permit us to optimize a fuel design with respect to the various contexts, such as environmental costs for developments/storage/transportation, economic impacts, and sustainability within society, rather than focusing on efficiency within vehicles. Therefore, study for the fundamental combustion characteristic, such as ignition delay, of fuel mixtures may augment a degree of freedom in terms of the possible application of the data set.
APPENDIX A

Rapid Compression Machine

1. RCM operation manual

Following example is suggested for a specific driving pressure, 200 psi, and a specific hydraulic pressure, 1150 psi. This operation manual only shows the stepwise order of procedure which should be done for the RCM data acquisition. Objectives of each procedure were explored at corresponded chapters. The names of RCM components on following manual were shown in Figure 2.1 and 2.7. Notation of valves and components on the manual was defined in Figure Appendix A.1. Default position of all the valves during operations is closed position unless it is defined as open.
<table>
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<tr>
<th><strong>Initial preparation</strong></th>
<th><strong>Check hydraulic oil level</strong></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>1. Open 1, 2, 5 and 8</td>
</tr>
<tr>
<td></td>
<td>2. Turn on oil pump, wait 30 sec</td>
</tr>
<tr>
<td></td>
<td>3. Open 4</td>
</tr>
<tr>
<td></td>
<td>4. Read the oil level through tube and adjust filling speed using 6</td>
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<tr>
<td></td>
<td>5. Once the oil is filled up to suggested level, close 4 and wait 1 min to confirm the oil level. If the oil level is getting lower, go back to step 3. If it is stable, turn off oil pump.</td>
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<table>
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<tr>
<th><strong>Check driving tank pressure</strong></th>
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<tbody>
<tr>
<td>6. If Tank pressure is lower than 200 psi, rotate 7 to supply position and fill the gas up to 200 psi.</td>
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<thead>
<tr>
<th><strong>Turn on heating system</strong></th>
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<tbody>
<tr>
<td>7. Turn on the H1 and H2</td>
</tr>
<tr>
<td>8. Turn on C1, C2, and F1. Adjust the rate of heating by heating regulators. Temperature regulate should be done before Mixture Components introduction process.</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th><strong>Retract and lock the piston</strong></th>
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<tbody>
<tr>
<td>10. If black marked line on piston is shown, close 2, 5 and 8.</td>
</tr>
<tr>
<td>11. Open 10 slowly while watching hydraulic pressure gauge and close 10 when the pressure reaches around 150 psi.</td>
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<tr>
<th><strong>Evacuate combustion chamber</strong></th>
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<tbody>
<tr>
<td>13. Close B4 and open B1, B2, and B3. Wait till pressure, read from the H2, reaches 0 torr.</td>
</tr>
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</table>

<table>
<thead>
<tr>
<th><strong>Mixture preparation</strong></th>
<th><strong>Vapor fuel metering</strong></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>1. Turn on heater for the fuel reservoirs.</td>
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<tr>
<td></td>
<td>3. Wait 1 min after pressure, read from the H2, reaches 0 torr. Then, close needle valves and open B2.</td>
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<tr>
<td></td>
<td>4. Fill a syringe with neat n-heptane and inject the fuel through syringe inlet. Repeat 2-3 times and then close B5.</td>
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<tr>
<td></td>
<td>5. Repeat step 4 for the toluene to the other fuel reservoir</td>
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</tbody>
</table>

<table>
<thead>
<tr>
<th><strong>Mixture components introduction</strong></th>
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</thead>
<tbody>
<tr>
<td>1. When the pressure, read from the H2, stays 0 torr. Close B1. Then, turn off vacuum pump.</td>
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<tr>
<td>2. Slightly open a needle valve connected to n-heptane fuel reservoir while monitoring the pressure on H2. When it reaches aimed pressure, close</td>
</tr>
<tr>
<td><strong>Introduction</strong></td>
</tr>
<tr>
<td>------------------</td>
</tr>
<tr>
<td>The needle valve and wait 3 mins to confirm the thermal equilibrium. Repeat the previous step 2 for the toluene.</td>
</tr>
<tr>
<td><strong>Liquid fuel metering</strong></td>
</tr>
<tr>
<td><strong>Inject fuel</strong></td>
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<tr>
<td><strong>Heat up fuel chamber</strong></td>
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<tr>
<td><strong>Mixture components introduction</strong></td>
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<tr>
<td><strong>Firing</strong></td>
</tr>
<tr>
<td>Prepare force balance creation:</td>
</tr>
<tr>
<td>Create driving force</td>
</tr>
</tbody>
</table>
| **Destruct force balance** | 4. Rotate 7 to supply and close 7 when the tank pressure reaches exactly at 200 psi.  
5. Make sure all the valve on the control panel directed at the final operation position indicated by red dots.  
7. Turn on the Amplifier, A1, and change the state of it as operation.  
8. Press run button of Labview program and rapidly open 11. |
| **Venting & Cleaning** | 1. Close the big yellow valve behind control panel  
2. Rotate 9 slightly to vent and slightly open the 2.  
3. Save Labview result. Change the state of Amplifier to reset before turning off. Turn off all the heaters. Open B2, B3, and B4.  
4. After compressed airs are completely vented from RCM, close 2 and open 1 and 5.  
5.1 If planned to another experiment, restart from *Check hydraulic oil level*.  
5.2 If not, turn on the vacuum pump and rotate 9 to vacuum. After piston is retracted, close 9/B4 and open B1. After 30 sec, wide open and close B4. Repeat several times, open/close B4. Turn off Vacuum pump. Close B1 and Open B4. |
## 2. RCM operation & maintenance tips

<table>
<thead>
<tr>
<th>Symptom</th>
<th>Check points</th>
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</table>
| Cannot achieve a vacuum of combustion chamber                         | 1. Check the valve position of 9, B1 and B4. They should be closed.  
                                                                 | 2. Check O-ring under the cap of the fuel chamber.  
                                                                 | 3. Check the oil level of vacuum pump. If it is below suggested level, add the vacuum oil.  
                                                                 | 4. Check the vacuum state of fuel lines only by close B3. If the vacuum is not achieved, leaks happened among the fuel lines.  
                                                                 | 5. Check the sealing on the piston inside combustion chamber. Only the cause is not found during the previous steps, disassemble the combustion chamber to check and change the sealing on the piston head.  
                                                                 | 6. Check the alignment of RCM. If the combustion chamber still leaks after the change sealing on the piston and 100% sure that sealing does not damaged during the combustion chamber assembly, the leaks may be cause by tilted alignment of RCM during the operations. |
| Cannot observe the hydraulic oil level through the tube on the main panel | 1. Check the valve position of 2, 5, 4 and 6. They should be opened.  
                                                                 | 2. Check the oil level of oil reservoir through the side tube. If the oil level is not shown, it is time to refill the silicon oil to the oil reservoir.  
                                                                 | 3. Increase pumping pressure and wait. Once the silicon oil is drawn back to oil reservoir for some reasons, it takes more than 15mins (~30mins) to fill the hydraulic chamber depending on the oil pumping pressure. |
| Cannot pressurize enough either tank pressure or hydraulic chamber pressure | 1. If you cannot hear the air sound when you rotate corresponding valve as open, check the regulator attached on the tank. (One may need to either open the regulator or change the tank.)  
                                                                 | 2. If you can hear the air sound, the sealing behind control panel or gauge may fail.                                                                                                                             |
| Fail to record data at Labview after firing RCM                        | 1. Exam the data which you got. You may fire the RCM either too late or too fast. Pay by ear to learn the right timing. It would be better to count one between running the program and firing the RCM.  
                                                                 | 2. Check the amplifier's status whether it was set as operates and there was an error sign.  
                                                                 | 3. Check the position of piston whether the piston had actually moved. Line was marked on the piston.                                                                                                        |
| Cannot get the repeatable data when fuel is metered by partial pressure. | 1. Fully evacuate the fuel reservoir by heating up the fuel reservoir while applying the vacuum pump. Calibration of the pressure gauge may need frequently. Change the silicon cap of the syringe inlet. Then, refill the fuel carefully to avoid accidentally inject the air bubble.  
2. Check leaks of the combustion chamber and fuel reservoirs as indicated by pressure.  
3. Check the heating components (thermo couples, heating tapes, etc.) for the combustion chamber. |
REFERENCES


[19] "Reduced mechanism for n-heptane," *the LLNL n-heptane mechanism version 38*. (Accessed 1/03/08) <http://www-cmels.llnl.gov/?url=science_and_technology-chemistry-combustion-nc7hl6_reduced_mechanism>


