HEATED SHOCK TUBE DESIGN AND CHARACTERIZATION FOR LIQUID FUEL COMBUSTION EXPERIMENTS

A Thesis

by

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Submitted to the Office of Graduate and Professional Studies of
Texas A&M University
in partial fulfillment of the requirements for the degree of

MASTER OF SCIENCE

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August 2017

Major Subject: Mechanical Engineering

ABSTRACT

Studying the combustion properties of liquid fuels and low vapor pressure fuels is important for power generation, propulsion, and aviation. Determining these properties, requires facilities like heated shock tubes that can ensure the fuel can remain in the vapor phase during combustion experiments. To better study the properties of liquid fuels, modifications were made to the high-pressure shock tube at Texas A&M University using custom heating jackets from BriskHeat Corporation and heating tapes controlled by temperature controllers to heat the facility to temperatures up to 150°C. Experiments were conducted to ensure the driven section of the shock tube, where combustion measurements are collected, was kept at uniform temperatures to prevent speeding up or slowing down of the shock wave as it passes through the test mixture. A stoichiometric methane-oxygen mixture in 98% argon was used to validate the heated facility for high-temperature gas mixtures, and an n-nonane, 4% oxygen mixture was used to validate the heated facility for liquid fuel mixtures. These results were compared to previous studies of the same mixtures.

Temperature uniformity experiments resulted in a ±1°C temperature distribution for the 10 feet closest to the measurement location of the driven section for 50°C, 75°C, and 100°C set point temperatures. A temperature distribution of ±2°C was achieved for the 150°C maximum operating temperature of the facility. Ignition delay time measurements were made for the stoichiometric methane-oxygen mixture over a temperature range of 1784 K to 2134 K at 1.13 atm using an OH* diagnostic. These

results matched well with previous studies of the same methane mixture, hence validating that the heated shock tube performs as well as it did for such mixtures prior to heating it. Ignition delay time measurements were also made for the n-nonane mixture over a temperature range of 1320 K to 1444 K at 1.7 atm, and these aligned well with previous studies of the same mixture. Therefore, the heated shock tube was validated for use with both gas and liquid baseline fuel mixtures, giving confidence that it will perform as intended for all future studies of low vapor pressure fuels.

ACKNOWLEDGMENTS

I would like to thank my committee chair, Dr. Eric Petersen, for his support, guidance, and patience throughout the course of this research, and for introducing me to the world of gas dynamics and combustion. I would also like to express my thanks to Dr. Andrea Strzelec and Dr. Rodney Bowersox for serving on my thesis committee and for adding to my knowledge of thermodynamics and aerospace applications through their classes.

My sincerest gratitude goes to my shock-tube group colleagues Sulaiman Alturaifi, Joshua Hargis, Olivier Mathieu, Clayton Mulvihill, and all of my other lab colleagues for teaching me how to run the shock tube, helping me modify the shock tube, providing advice, and always being willing to help.

Thanks also go to my family and friends for their constant support and encouragement of all my academic and non-academic endeavors. Finally, I would like to thank my grandmother for paving the way for my love of science and learning in a time where there were very few female scientists in the field and setting a wonderful example for my career in STEM.

CONTRIBUTORS AND FUNDING SOURCES

Contributors

This work was supervised by a thesis committee consisting of Professors Eric Petersen and Andrea Strzelec of the Department of Mechanical Engineering and Professor Rodney Bowersox of the Department of Aerospace Engineering.

All work for the thesis was completed by the student, under the advisement of Dr. Eric Petersen of the Department of Mechanical Engineering.

Funding Sources

Graduate study was supported by a Graduate Diversity Fellowship from Texas A&M University. This work was also made possible in part by Qatar National Research Fund (QNRF) under Grant Number 8-1358-2-579 and the Defense Threat Reduction Agency under Grant Number HDTRA1-16-1-0031.

Its contents are solely the responsibility of the authors and do not necessarily represent the official views of the Department of Defense or QNRF.

NOMENCLATURE

HPST	High-Pressure Shock Tube
MT	Mixing Tank
P	Static Pressure
ST	Shock Tube
t	Time
T	Static Temperature
φ	Equivalence Ratio
τ_{ign}	Ignition Delay Time
Subscripts	
1	Initial condition of the driven section of the shock tube at t = 0
2	Condition behind the incident shock wave
3	Condition in the driver section behind the contact surface and the expansion wave
4	Initial condition of the driver section at t = 0
5	Condition behind the reflected shock wave in the driven section

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CHAPTER I

INTRODUCTION AND LITERATURE REVIEW

When it comes to studying combustion properties of fuels, shock tubes have become a useful tool in creating a test environment in which ignition delay times can be measured at a range of specified pressures and temperatures. However, certain liquid fuels require initial heating to change to the gas phase before combustion occurs. These include gas-to-liquid fuels, which are used in power generation, aviation, and propulsion applications [1]. Most of these gas-to-liquid jet fuels, liquid hydrocarbon propellants, and some auto diesel fuels are referred to as kerosenes, which have a boiling range of 200°C to 300°C [2]. Different applications require different types of fuels, and most fuels are selected based on tradeoffs between performance, physical characteristics, safety, cost, and availability [3]. For this reason, jet fuels like JP-7 and JP-8 are used for military applications, jet fuels like Jet-A are used for commercial applications, and fuels like RP-1 and RP-2 are used as kerosene rocket propellants [4].

Previous studies have proved the effectiveness of using a heated shock tube facility to understand combustion properties of a variety of fuels. For example, heated shock tube facilities have been used to study shock-induced oxidation of higher-order hydrocarbons to improve upon higher-order alkane models [5] [6]. These higher-order hydrocarbons can then be used in surrogate mixtures for kerosene fuels such as diesel and jet fuels [7], since surrogate mixtures tend to be easier to model than real fuels. Heated shock tubes have also been used to study kerosenes and other gas-to-liquid fuels, whose kinetic processes have not been validated computationally at higher temperatures [8]. Additionally, new kerosene fuels are being developed to reduce pollution by burning cleaner and reducing sulfur content [9]. By studying the combustion properties of kerosene aviation fuels like Jet-A or RP-1, these fuels can be better implemented in high-temperature, supersonic, and detonation applications.

To understand the properties of these fuels at high pressures and temperatures, it is necessary to create a heated facility in which the fuels can be vaporized for combustion experimentation. For this reason, the high-pressure shock tube at Texas A&M University was upgraded to include a system capable of heating a gas-to-liquid fuel mixture up to an operating temperature of 150°C, which was the focus of this thesis. This temperature range ensures that gas-to-liquid fuels are in the vapor phase during shock-tube experiments and allows the measurement of ignition delay times for these fuels.

1.1 High-Pressure Shock Tube Description

The high-pressure shock tube (HPST) at Texas A&M University, described by Aul [10], is composed of a stainless steel driver section and driven section, separated by a diaphragm, which is shown in Figure 1. For a general shock-tube experiment, a test gas mixture is created in a stainless steel mixing tank. Using a manifold, the driven section is filled with the test gas mixture to a specified pressure. Other gases may be used to pressurize the driven section further to reach a certain dilution level and equivalence ratio. Then, the driver section is filled with a helium mixture until it reaches a pressure at which the diaphragm bursts. This sudden bursting of the diaphragm creates a shock wave, which travels down the driven section [11]. When the shock reaches the end wall of the driven section, it reflects back towards the driver section, creating a region of a specific pressure and temperature where combustion measurements are collected. Laser and optical diagnostics can be used to collect information about species concentrations, rates of reaction, and ignition delay times for the test mixture.

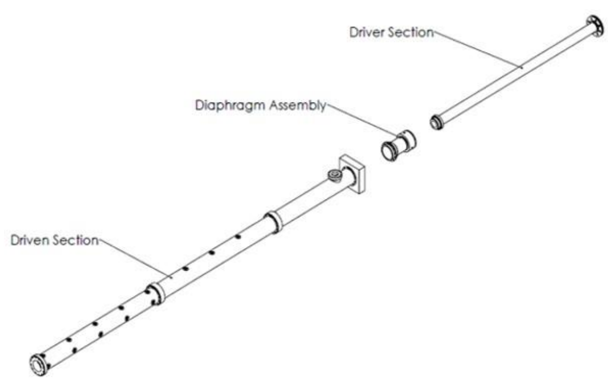
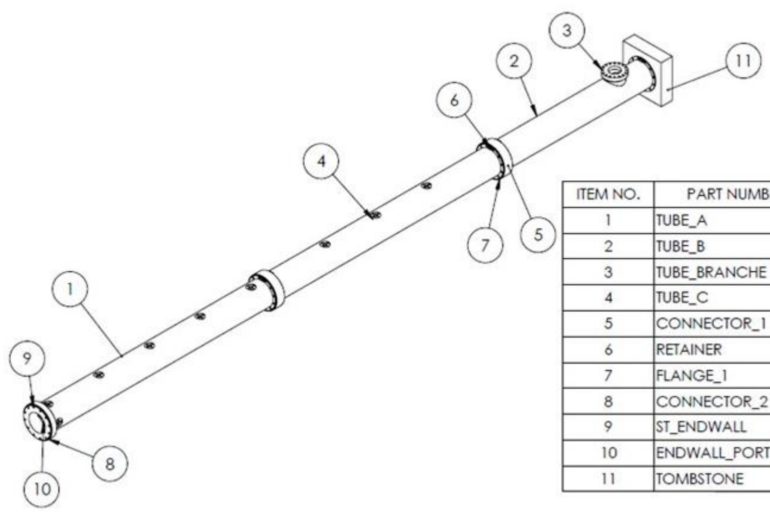


Figure 1- High-pressure shock tube main components.

Since combustion studies of the fuel occur in the shock tube driven section, this section was the focus of the heated shock tube design. Figure 2 shows an assembly of the driven section, which is composed of three, 7-in diameter tube sections, connected by flanges. The shock tube is anchored to a large counter weight via the square "tombstone", noted as item 11 on Figure 2. On the opposite side of the driven section is item 9, the end wall, which closes off the tube and is the location at which the shock wave is reflected. The tube section closest to the end wall has several ports which allow optical diagnostic techniques to be utilized through windows to measure the conditions behind the reflected shock. Ports can also be used for pressure measurements or for sensors to mark when the shock wave passes that location of the shock tube. These sensors are connected to time-interval counters which help to determine the shock speed and ultimately the pressure, P_5 and the temperature, T_5 behind the reflected shock wave.



ITEM NO.	PART NUMBER	QTY.
1	TUBE_A	1
2	TUBE_B	1
3	TUBE_BRANCHE	1
4	TUBE_C	1
5	CONNECTOR_1	2
6	RETAINER	6
7	FLANGE_1	12
8	CONNECTOR_2	1
9	ST_ENDWALL	1
10	ENDWALL_PORT	5
11	TOMBSTONE	1

Figure 2- Driven section parts identification.

1.2 Heated Shock-Tube Facilities

When it comes to measuring combustion properties of liquid fuels with very low vapor pressures like jet fuel, diesel, and kerosene, many researchers have turned to the use of aerosol shock tubes, which disperse tiny droplets of liquid fuel into the parent gas [12]. The mixture ignites when it is exposed to a shock wave. Although creating mixtures with low vapor pressure fuels such as Jet-A and JP-8 can be challenging, there are several advantages to using heated shock tubes instead. These include reliable, reproducible measurements of gas-phase ignition delay times and constant-volume modeling for a wide range of fuel mixtures and temperatures [13].

Dean et al. performed one of the first shock-tube studies of gas-phase jet fuel ignition with a Jet-A mixture in a heated shock tube in 2007 [8]. They sought to validate fuels with high numbers of hydrocarbons and their associated chemical kinetic processes at higher temperatures. Because of limited resources when it comes to analyzing complex kerosene fuels, surrogate mixtures were sought after to simplify computations. Using nichrome wire, the shock tube used by Dean et al. was heated to initial temperatures between 75°C and 100°C with mixing tank temperatures between 80°C and 174°C, depending on the fuel being tested [8]. Vasu et al. conducted similar studies of jet fuel surrogates and ignition delay times of Jet-A and JP-8 with a heated, high-pressure shock tube using OH* emission and side wall pressure [13]. Their facility was heated to 100°C using 13 separate heating zones for a ±3°C temperature uniformity along the driven section of the shock tube.

Heated shock tubes have been used to characterize Fischer-Tropsch surrogates of gas-to-liquid fuels as well. Aul et al. created a heated shock tube facility with HTS Amptek heating jackets which had a temperature uniformity of ±5°C along the shock tube [14]. This heated facility was used to study fuels like RP-1, RP-2, JP-8, and Fischer-Tropsch surrogates by using OH* chemiluminescence to measure ignition delay time. Wang et al. also collected OH* chemiluminescence ignition delay time measurements of Jet-A, JP-8, and Fischer-Tropsch fuels [15]. This was achieved using a shock tube facility heated to 140°C with a temperature uniformity of ±2.2°C and a mixing tank temperature of 150°C. Dagaut et al. used a heated shock tube set to 120°C and a mixing tank set to 180°C to study ignition delay times of n-decane, iso-octane, and other surrogate blends for gas-to-liquid fuels using CH* emission [16].

Heated shock tubes have also been used to study ignition delay times of aromatic fuels, such as the study done by Shen et al. [17] via OH* emission and pressure measurements. In this study, the shock tube was held at a temperature of 70°C with a temperature uniformity of ±2°C, which was deemed to be very small compared to incident shock velocity uncertainty. Other properties besides ignition delay times can also be studied using heated shock tubes. For example, soot induction delay times for heavy hydrocarbons were studied by Douce et al. using a shock tube heated to 130°C with a ±2°C temperature distribution [18].

Previous heated shock tubes used at Texas A&M University include the one constructed by Rotavera et al. This shock tube and associated mixing tank are heated using insulated cloth heating jackets from HTS Amptek with a ±2.5°C uncertainty at 112°C and a ±0.6°C uncertainty at 52°C [5]. The diaphragm section and the end wall section are covered with extra heating tape and insulation to produce a uniform temperature distribution inside the shock tube. Furthermore, the manifold piping which connects the mixing tank to the driven section is covered with heating tape and fiberglass insulation to keep liquid test fuels from condensing. Although this heated shock tube setup works well for low pressure experiments [19], a new heated facility was needed at Texas A&M University to test higher-pressure conditions. For this reason, the high-pressure shock tube was selected to be modified into a heated shock tube facility. With the design of the heated shock tube setup from Rotavera et al. in mind, a heating system was designed for the high-pressure shock tube at Texas A&M University.

This thesis outlines the design, characterization, and validation of the heated high-pressure shock-tube facility at Texas A&M University. Chapter II outlines the temperature compatibility of the shock-tube components, leak checks of the unheated facility, the heating jacket design for the shock tube and mixing tank, and additional heating for the shock-tube manifold. Chapter III discusses the characterization and validation of the heated shock-tube facility through temperature uniformity experiments as well as ignition delay time experiments for a methane gas mixture and a low vapor pressure n-nonane mixture. Finally, chapter IV summarizes results from this project and recommends improvements for future studies involving the heated high-pressure shock-tube facility and low vapor pressure fuels.

CHAPTER II

HEATED SHOCK TUBE DESIGN

2.1 Temperature Compatibility of Shock-Tube Components

Before heating elements were installed on the shock tube, it was important to consider the operating temperature of all the shock-tube components. This task included the stainless steel bulk material of the shock tube, as well as components such as o-rings, valves, pressure transducers, gages, supports, and wires. Components not compatible with temperatures of 150°C or higher included the MKS Baratron 626A manometers, Setra pressure transducers, and the PCB P113B22 pressure transducer on the end wall. Therefore, an MKS Baratron 631 heated manometer was installed to replace the current 0-to-1000-torr manometer; the Setra pressure transducers will not be used at temperatures higher than 85°C; and the end wall PCB pressure transducer port will be replaced with a blank port for experiments with temperatures higher than 135°C. A full list of shock-tube components and their operating temperatures can be found in Appendix A.

With a design temperature of 150°C, the nitrile o-rings used in the original design of the high-pressure shock tube needed to be replaced to prevent melting of the material and reduced integrity of the seals. Due to the corrosive fuels that are often tested in the shock tube and the vacuum grease products used to seal the o-rings, chemical resistance was another important factor in choosing high temperature o-rings for the shock tube. Therefore, VG-109 Viton o-rings, shown in Figure 3, were selected to replace the original o-rings due to their operating temperature of up to 204°C and their chemical resistance to many of the fuels that are tested in the shock-tube facility [20].

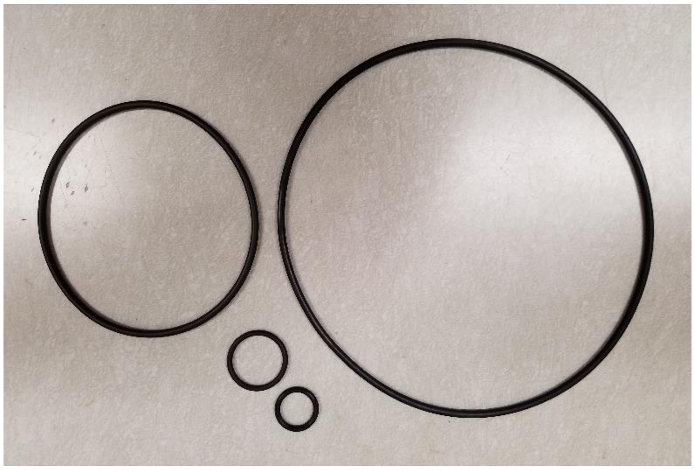


Figure 3- VG-109 Viton o-rings.

Replacing o-rings on the shock tube required knowledge of the original shock-tube design. Therefore, the original drawings of the high-pressure shock tube were consulted for o-ring sizes and can be found in Aul [10]. However, one of the o-ring sizes on the actuator connector's original drawing was found to be incorrect. A summary of the correct o-ring sizes on the high-pressure shock tube can be found in Appendix B.

Replacement of the original nitrile o-rings required a complete disassembly of the shock-tube components on the driven section and the vacuum section. For ports and the end wall, the bolts holding the components in place were removed, and the o-rings were replaced with Viton o-rings. For the diaphragm section, the o-rings were accessed easily from the assembly used to set the diaphragm in place. For larger components such as the 7-in diameter tube sections that compose the bulk of the body of the driven section, roller supports were installed underneath the shock tube at points on the optics table and underneath the vacuum section. The supports, shown in Figure 4, were held on top of wooden blocks and then raised and leveled to the correct height using adjustment threads.

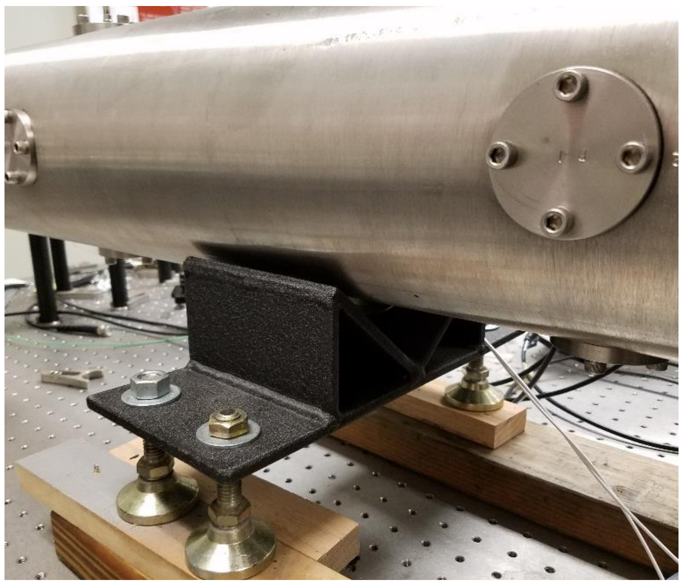


Figure 4- Shock-tube roller supports.

The bolts on the connecting flanges were removed, and the tube sections were separated, allowing enough space to replace the nitrile o-rings with Viton o-rings. When putting the tube sections back together, it was important to make sure that the ports on the top of the driven section were aligned well. Also, the tube sections themselves needed to be aligned vertically to ensure that there were no protruding lips inside the shock tube where the boundary layer could be tripped during a shock.

O-ring replacement required the disassembly of the nozzle from the shock tube and re-alignment of the nozzle with the driver section once the o-ring had been replaced. The vacuum section also needed to be disassembled to replace o-rings in the poppet valve and other components. Note that low vapor pressure krytox was used to install o-rings in the vacuum section to ensure a good seal between the o-rings and metal components. While replacing o-rings, copper gaskets were also replaced to ensure proper resealing of vacuum components. The o-rings on the end caps of the mixing tank were also replaced by taking off the end caps and installing the o-rings around the welded rod on the inside of the mixing tank. The driver section is not heated since the liquid fuel mixture does not reach that tube section until the experiment is over. Therefore, the o-ring on the far side of the driver section was not replaced with a Viton o-ring.

2.2 Leak Rate Checks

Before implementing the heating system onto the high-pressure shock tube, it was important to make sure that the o-rings were properly installed by checking for leaks in the driven section, vacuum section, and mixing tank. This leak checking was done by vacuuming out the desired section with the rough pump and the turbomolecular pump for at least 30 minutes. Then, the section was shut off to the vacuum, and the current pressure was recorded. After 20 minutes, the pressure was recorded and the leak rate was calculated by dividing the pressure difference by the time that had passed. Leak rates were also determined for the manifold section using both the vacuuming technique described above and by pressurizing the gas lines to check for leaks across valves over a

20-minute period. Necessary fixes to the manifold were made to minimize the leak rate such as tightening joints, replacing o-rings in valves, and replacing a valve. After o-ring replacements, the final leak rate for the shock tube was 1.7 mTorr/min, and the final leak rate for the mixing tank was 0.38 mTorr/min. Leak rates of other regions of the shock-tube facility can be found in Appendix C.

2.3 Heating Jacket Design

Due to the large size of the mixing tank and the driven section, insulated heating jackets were designed to uniformly heat these cylindrical components. Solidworks models of the driven section and the mixing tank were created, and designs for custom heating jackets to fit the dimensions of the shock tube were finalized with the help of BriskHeat Corporation. Three separate jackets were designed to cover the mixing tank, and five heating jackets were designed to cover the driven section of the shock tube, shown in Figure 5. Multiple jackets covering the mixing tank and shock tube allow finer control of the temperature of the tube as it is being heated and allows easier installation of the jackets.

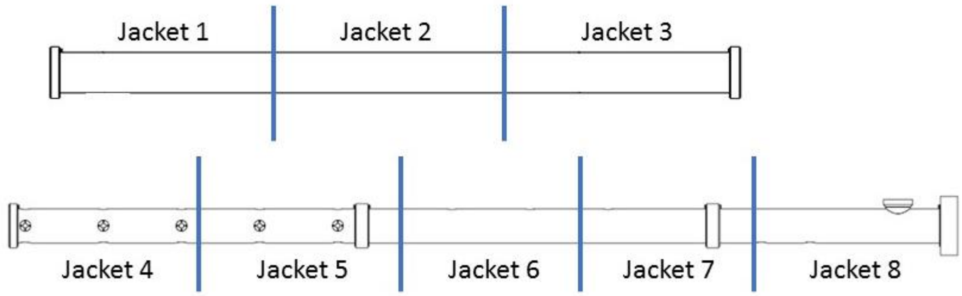


Figure 5- Heating jacket numbering system for the mixing tank (top) and shock tube (bottom).

During fabrication at BriskHeat Corporation, heating elements and a type J theromocouple were sewn into each heating jacket. When the jackets are properly installed, the heating elements and the thermocouple are in contact with the shock tube, allowing the shock tube to be heated to a specified temperature. The heating jackets selected for this facility were made from PTFE, which has an exposure temperature of up to 260°C, and a layer of fiberglass insulation to prevent heat loss [21]. Figure 6 shows heating jackets installed on the driven section of the shock tube. Note that the tube on top is the mixing tank, and the jacket-covered portion of the tube on the bottom is the driven section.

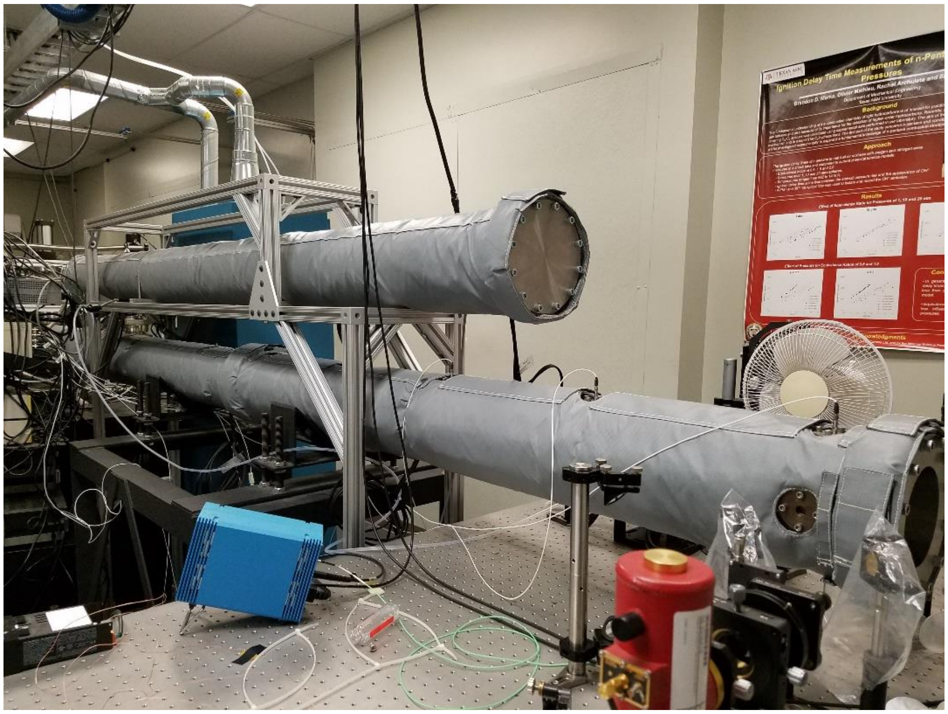


Figure 6- Heating jackets installed on the mixing tank and the driven section.

Each heating jacket is controlled by a temperature controller, which heats the shock tube to a set point temperature using feedback from the thermocouple in contact with the outside wall of the shock tube. Detailed procedures for using the heating jackets can be found in Appendix D. Note that when setting the jackets to temperatures higher than 50°C, it is advised to allow the heating jacket to warm up in 25°C increments to prevent large overshooting of temperatures. This level of caution is particularly important as temperatures reach the operating limit of shock-tube components such as transducers and gages.

The heating jackets were installed on the high-pressure shock tube and mixing tank by fitting each jacket around a designated tube section. Velcro straps sewn into each jacket were used to secure the heating jacket in place. Note that U-bolt supports were repositioned on the mixing tank to install the heating jackets properly. The location of the thermocouple on each jacket is marked with a yellow triangle. It is important that the thermocouple on the jacket is not resting on a support, which becomes a cold spot and may also cause overheating of the heating jackets.

Although the heating jackets were custom designed for the driven section of the shock tube and the mixing tank, holes were left in the heating jackets to allow access to window ports, pressure ports, the end wall, and end cap regions of the mixing tank. Even though these areas need to be accessible between experiments, the exposed surface area of these components can contribute to significant heat loss. For this reason, silicone foam with woven fiberglass was used to provide further insulation. Holes were cut in the pressure port insulation to allow cables to be connected to their corresponding pressure ports, shown in Figure 7.

Figure 7- Pressure port insulation.

To allow light to pass through for collecting optical measurements, holes were cut in window port insulation as well. Insulation was also created to fit around the welded pipes feeding into and out of the mixing tank and to fit onto the driven section end wall region and mixing tank end caps. This added insulation helps to prevent heat loss from areas that are not directly heated by heating jackets or heating tapes.

2.4 Additional Heating

Additional components of the shock tube facility also require heating to ensure the gas-to-liquid fuels remain in the vapor phase when transferring them from the mixing tank to the driven section. Because of smaller and more-complex geometries, heating tapes were selected to heat the manifold, the stainless steel piping, the vacuum section, the nozzle, and the tombstone counterweight which holds the shock tube in place during experiments. Note that for the experiments conducted in this project, it was not necessary to heat the vacuum section, nozzle, or the tombstone. However, these components will need to be heated when conducting experiments with more-complex, multi-component fuels. The heating tapes used on the heated HPST are Omega FGH Series fiberglass heating tapes, which are capable of heating up to temperatures of 482°C (900°F). The heating tapes were installed by coiling the tape around the component being heated, being careful not to overlap the heating tape on top of itself to prevent overheating. The tapes are secured in place using strings that are attached to both ends of the tape. To further secure the heating tape to the surface being heated, fiberglass insulation strips were wrapped on top of the heating tape to prevent heat loss from the component being heated to the surrounding environment. Pictures of the manifold section before and after being wrapped with heating tapes are featured in Figure 8 and Figure 9.

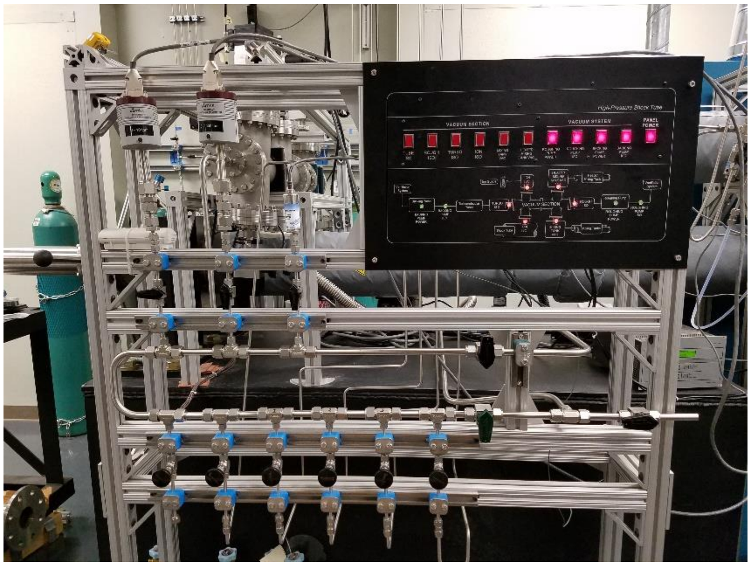


Figure 8- Manifold section before heating.

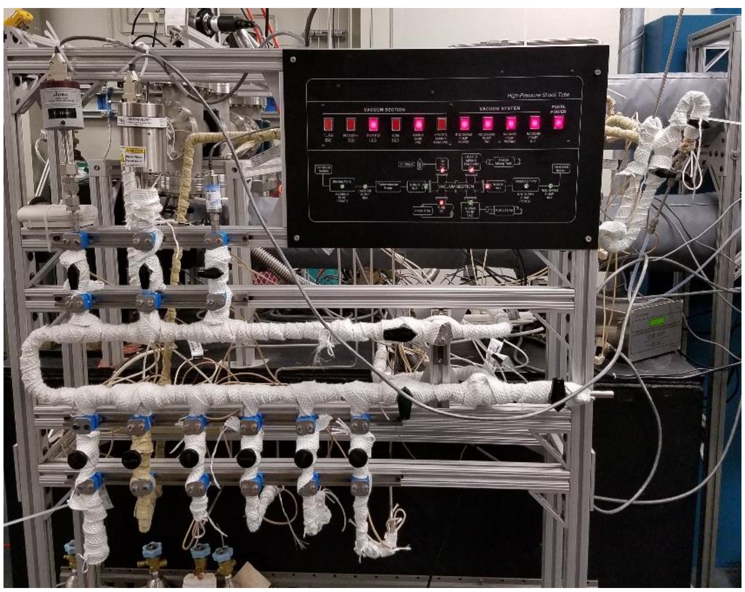


Figure 9- Manifold section with heating tape and insulation.

These heating tapes are controlled using temperature controllers and type K thermocouples, similar to the process used to control the heating jackets. Each controller receives feedback from only one thermocouple, so it is recommended to secure the thermocouple to a cold spot to make sure that the rest of the components being heated are at least at the set point temperature. The heating tapes on the manifold are divided into three temperature zones, which are shown in Figure 10. Each zone has one temperature controller, which are all labeled with their zone numbers for reference.

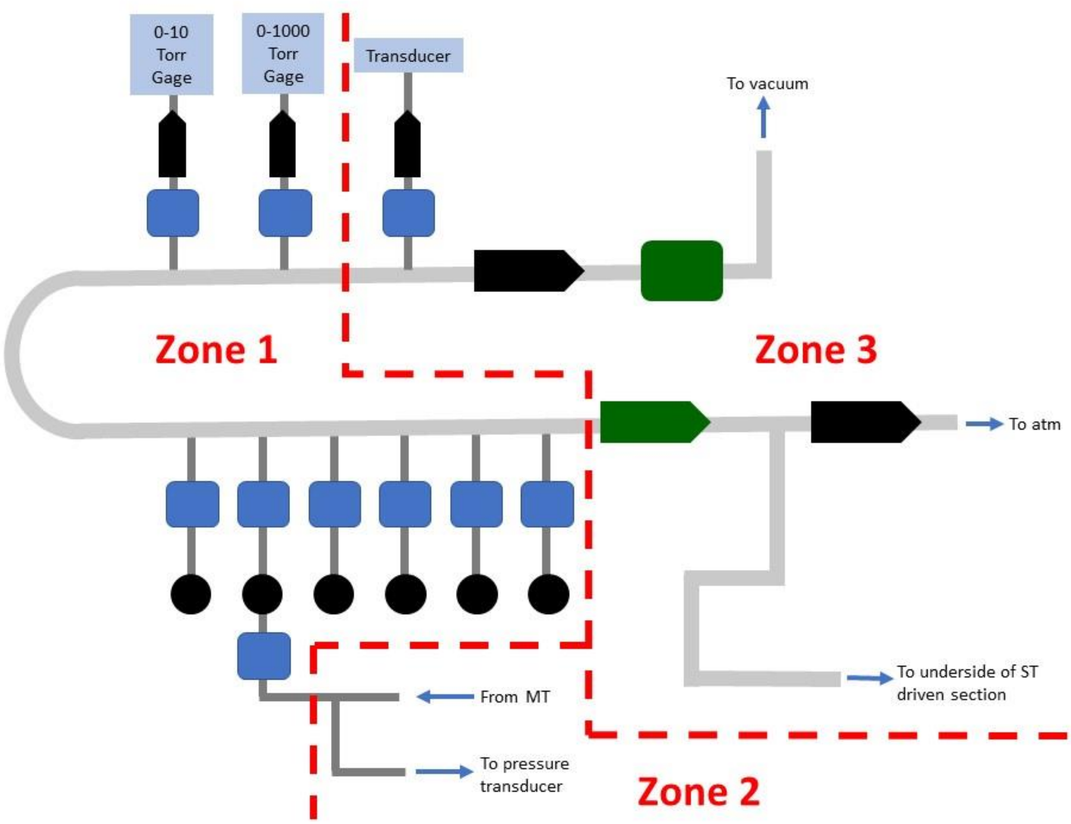


Figure 10- Manifold heating zones.

Note that temperature uniformity is not as much of a concern for the manifold and vacuum sections as it is for the driven section since test measurements do not occur in these areas. However, it is important to keep in mind that every spot covered with heating tape should be at a temperature higher than that required to keep the fuel in the vapor phase while it is being moved in and out of the mixing tank and driven section.

The controllers selected to control the temperature of the heating tapes on the manifold are Omega CN7800 series controllers, which are pictured in Figure 11. Instead of being benchtop controllers, like the X2 models used to control the heating jackets, the CN7800 controllers require additional electronic setups to perform correctly. However, buying these components separately instead of purchasing them as a unit inside of a benchtop controller proved to be more cost effective. Like the X2 controllers, the CN7800 controller gathers temperature data from a thermocouple, which is connected to the controller. The CN7800 controllers are also connected to a power source and a solid-state relay, which only provides power to the heaters when the controller reads a temperature that is not the same as the set point temperature. Multiple heaters can be controlled by a single controller as long as their power requirements do not exceed the maximum 1800 W that the wall plug can provide. All the components of the controller are connected using DIN rail terminal blocks.

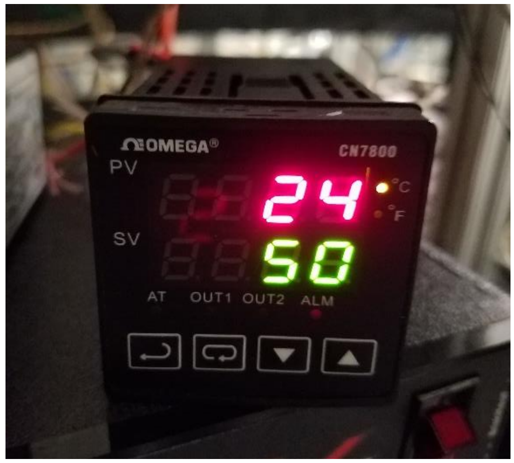


Figure 11- Omega CN7800 temperature controller.

CHAPTER III

HEATED SHOCK TUBE CHARACTERIZATION AND VALIDATION

3.1 Temperature Uniformity Experiments

To ensure that the driven section is being heated uniformly, temperature uniformity experiments were conducted by measuring the air temperature inside the shock tube. As mentioned earlier, it is important that the air temperature inside the driven section is uniform since shock speed is affected by changes in temperature. By keeping the temperature uniform, there is less chance of the shock wave speeding up and slowing down as it progresses along the tube during experiments. The heated shock tube modified by Aul et al. showed a temperature distribution of ±5°C [14], and the heated shock tube modified by Rotavera et al. at Texas A&M University showed a temperature distribution of ±2°C [5]. Therefore, the temperature distribution of this shock tube was expected to match or have lower amounts of variance, particularly near the end wall where measurements are made. Aul et al. characterized the uniformity of the temperature inside the shock tube using an Omega roller temperature probe [14]. Air temperature and wall temperature measurements were collected by pulling the roller temperature probe along the length of the shock tube and recording temperatures every 15 cm. Rotavera et al. characterized a square shock tube using similar techniques as Aul et al., but at 10-cm increments along the shock tube [5].

Using previous studies as a guide, temperature uniformity experiments were conducted for the present study to characterize the heated shock-tube setup in this thesis.

A first set of experiments included heating up the shock tube with the end wall open over the span of a few hours and then measuring the inside wall and air temperatures using a roller temperature probe, as done in previous studies by Aul et al. [14]. However, the data collected from these experiments showed a large heat loss near the end wall. Also, it was difficult to keep track of exactly where inside the shock tube measurements were being collected due to the spring back of coils in the wires connecting to the roller temperature probe. Because of these challenges, the temperature measurement process was modified by creating a metal rod assembly with type K thermocouples attached at one foot increments along the rod, shown in Figure 12. Note that fiberglass insulation was used to elevate the metal rod while inside the shock tube to prevent scratching the inside of the shock tube. Detailed procedures of calibration experiments are included in Appendix E.

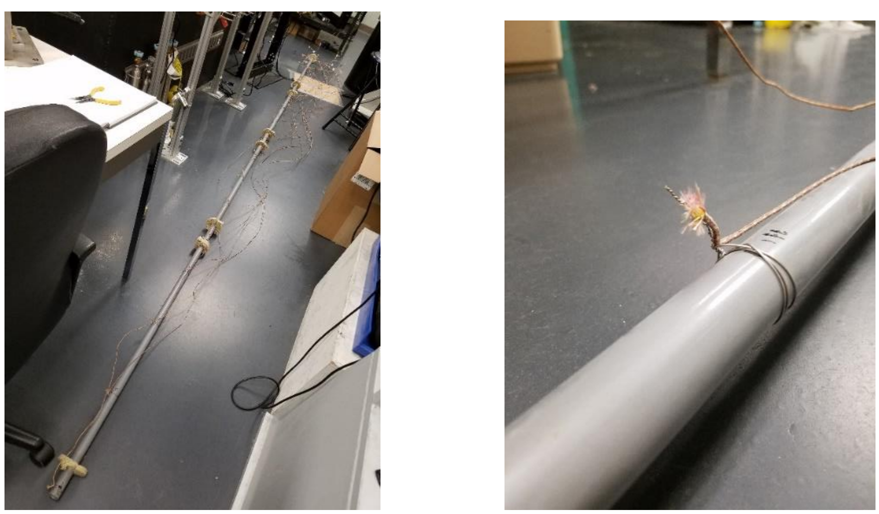


Figure 12- Metal rod assembly used to measure the air temperature inside of the driven section of the shock tube (left), Type K thermocouples fixed at 1-ft increments along the metal rod (right).

The metal rod assembly was inserted into the driven section of the shock tube, and the thermocouple wire leads were fed through a port on the bottom of the driven section, near the end wall, which is shown in Figure 13. Insulation was added to the port hole to minimize heat loss. The zero position of the thermocouple closest to the end wall was located directly in line with the windows and pressure ports, which is the location at which measurements are gathered during typical shock-tube experiments. Once the assembly was positioned in place, the end wall was closed, as it would be during shock-tube experiments. This closing off of the end wall allowed more-accurate temperature measurements to be made in the region closest to the end wall.

Figure 13- Thermocouple and metal rod assembly placement inside the driven section.

Calibration experiments were conducted by setting the driven-section heating jackets to a specific set point temperature and allowing the shock-tube temperature to reach steady state over the course of several hours. From observation, it was determined that the settling time required for the air temperature inside the driven section to reach a steady state temperature was approximately 7 hours from when the temperature controllers were set to a specific temperature. After a steady state temperature was reached, air temperature inside the driven section was measured at one-foot increments along the length of the driven section, using the thermocouple wire leads coming out of the port near the end wall. To reach a desired air temperature inside the shock tube, the heating jacket controllers must be set to a temperature higher than the inside air temperature due to the temperature controllers adjusting the temperature of the jackets according to the outside wall temperature of the shock tube. In other words, the air inside the shock tube is at a lower temperature than the temperature of the outside wall of the shock tube. Table 1 shows the heating jacket controller set point temperatures for the initial temperature distribution experiments.

Table 1- Heating jacket set point temperatures for driven section temperature distribution experiments.

Air Temperature (°C)	Jackets 5 through 8 Controller Set Point Temperature (°C)	Jacket 4 (near end wall) Controller Set Point Temperature (°C)
50	56	60
75	84	90
100	113	121
125	143	155
150	175	183

Note that for this temperature calibration setup, Jacket 4, nearest to the end wall, needed to be set to a higher temperature than the rest of the heating jackets to offset the heat loss that was occurring through the end wall. Figure 14 shows the temperature uniformity results for the closed end wall experiments with the thermocouples coming out of the port near the end wall.

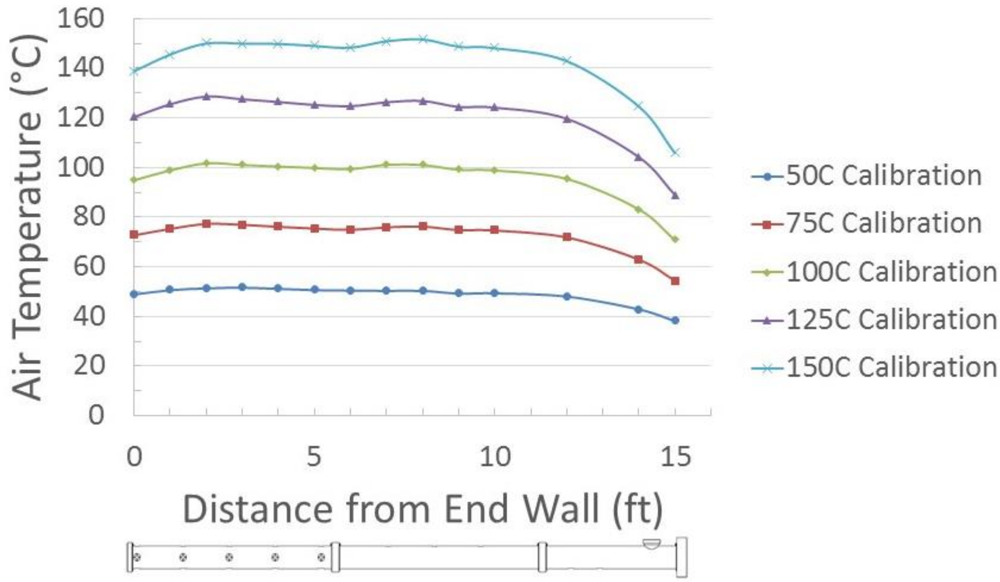


Figure 14- Initial driven section temperature distribution.

As shown in Figure 14, the driven section of the shock tube showed acceptable temperature uniformity between approximately 2 feet and 10 feet from the end wall. Near the end wall and the tombstone however, there was heat loss due to the decreased amount of heating elements near both ends of the driven section. The temperature distribution across the driven section also proved to be more uniform at lower temperatures than at high temperatures, where the temperature gradient between the calibration temperature and the temperature of the room is greater. This difference makes sense because the ends of the driven section are the most exposed to the lower room temperature, allowing heat transfer to occur between the air outside the shock tube and the driven section. The heat loss near the tombstone (closer to the diaphragm) is not as much of a problem since the shock is still forming in this region. However, the heat loss near the end wall needed to be addressed since shock tube experiment measurements occur close to the end wall.

Several adjustments were made to address the heat loss near the end wall. First, additional insulation was added to shock-tube components which were not previously covered by heating jackets and were not directly in contact with heating elements. These components include ports, windows, and the end wall of the driven section. High-temperature silicone foam with fiberglass facing was cut to fit these components to minimize heat loss from their exposed surface area. End wall insulation is pictured in Figure 15.

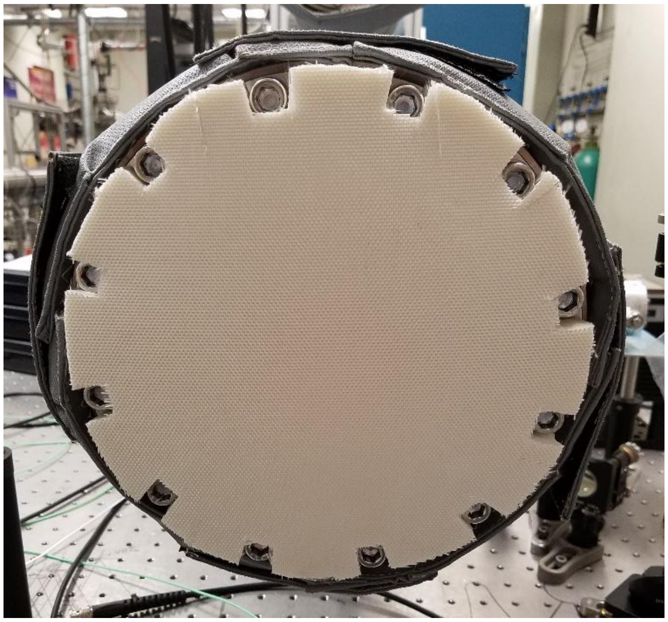


Figure 15- Custom end wall insulation.

Secondly, the metal rod assembly used to measure the air temperature inside the shock tube was modified to allow the thermocouple wire leads to come out of the nozzle of the shock tube instead of at a port near the end wall. This modification allowed for a fully closed shock tube at the end wall and thus eliminated heat loss that may have been caused by having an open port. Although heat loss would still occur at the open nozzle, insulation was added to the open area where the thermocouple wire leads were fed through to minimize heat transfer between the air inside the driven section and the air in the room. Because the open nozzle (near the diaphragm) is 16 feet away from the end wall, the heat loss occurring through the nozzle was not affecting the temperature of the end wall region. The temperature measurement setup for further calibration of the end wall region is pictured in Figure 16.

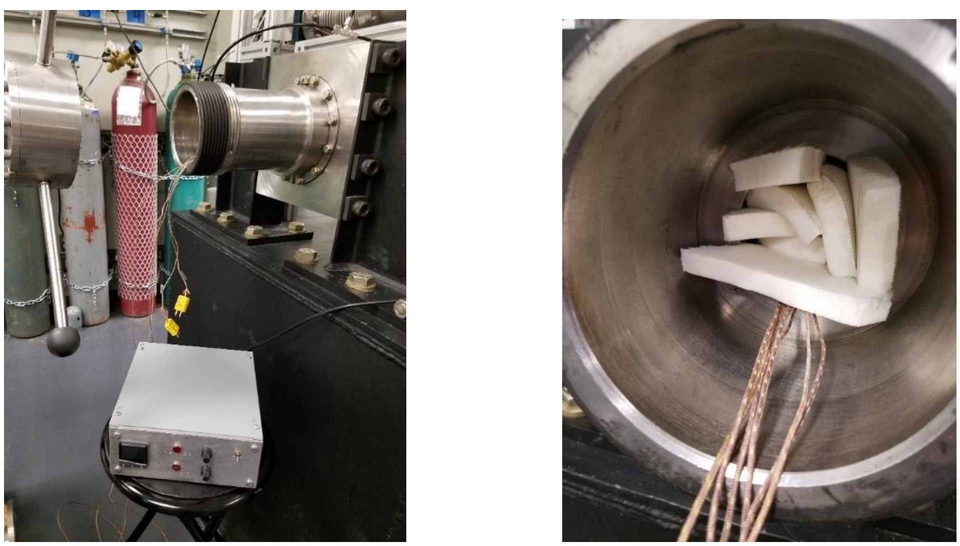


Figure 16- Temperature measurement setup with thermocouples coming out of the nozzle (left), Insulation added to the open nozzle (right).

Heating tape was also added to the end wall flange on the driven section, which is subjected to a large amount of heat loss due to the large surface area which is not in direct contact with the heating jacket. By wrapping the heating tape around the end wall flange and controlling the temperature of the heater with a controller and type K thermocouple setup, shown in Figure 17, the region near the end of the tube could be held at a near-uniform temperature.

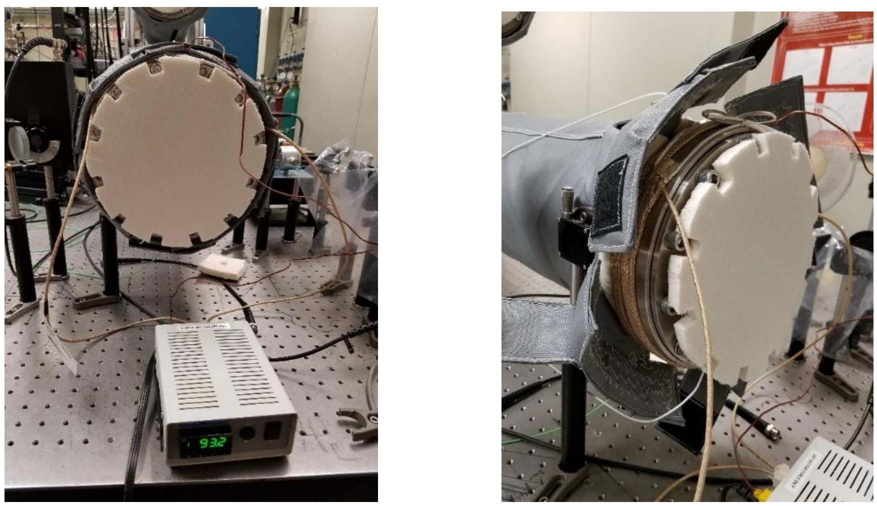


Figure 17- End wall heating tape setup (left), End wall heating tape wrapped under jacket 4 (right).

Adjusting the amount of insulation on the driven section, changing the measurement setup to have thermocouples coming out of the nozzle instead of at a port near the end wall, and adding extra heating tape to the end wall resulted in a highly uniform temperature distribution within 10 feet of the end wall, as shown in Figure 18.

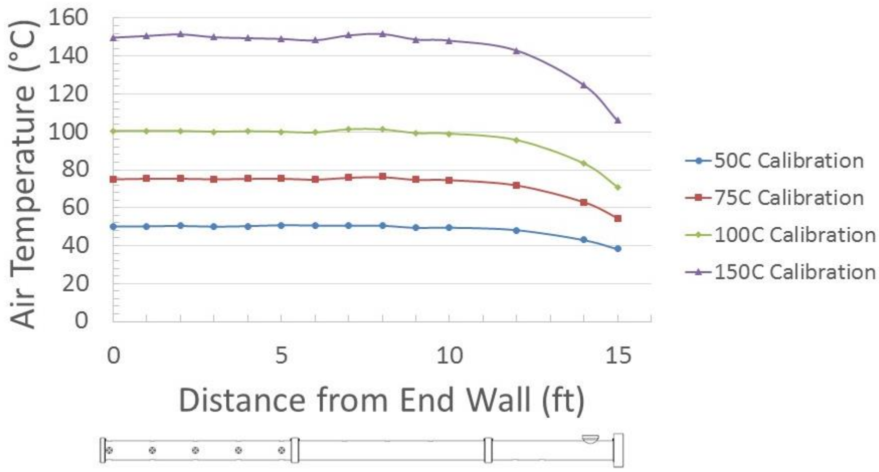


Figure 18- Driven section temperature distribution.

The temperature distribution shown in Figure 18 was created by measuring the air temperature when the heating jackets and the end wall heating tape were set to specific temperatures. A summary of the temperature settings of the heating jackets and the end wall heating tape is listed in Table 2.

Table 2- Temperature controller set points for uniform end wall temperature distribution.

Calibrated Air Temperature (°C)	**Heating Jackets Controller Set Point Temperature (°C)**	**End Wall Heating Tape Controller Set Point Temperature (°C)**
50	56	60
75	84	93
100	113	128
150	175 (180 for Jacket 4)	195

Compared to previous air temperature measurements with the thermocouple wire leads coming out of the port near the end wall, the data collected in Figure 18 show uniform temperature within ±1°C for the region of 0 to 10 feet from the end wall for calibration temperatures of 50°C through 100°C. At the maximum calibration temperature of 150°C, the temperature distribution is ±2°C for the region of 0 to 10 feet from the end wall, but is still within a distribution of ±1°C for the region of 0 to 7 feet. The temperature distribution for the region closest to the end wall is shown in Figure 19.

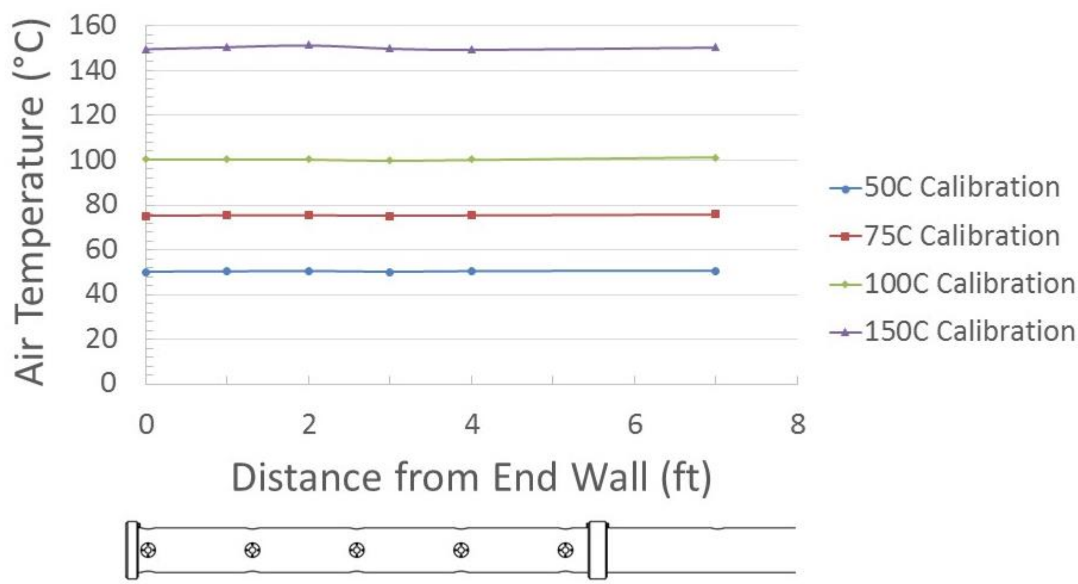


Figure 19- End wall region temperature distribution.

From the temperature settings required of the heating jackets and the end wall heating tape to reach an inside air temperature of 150°C, this is the maximum advisable limit for the operation of the heated HPST. Most of the components on the shock tube are rated to about 200°C, and once that limit is surpassed, measurement instruments start malfunctioning and the integrity of the o-ring seals is severely decreased. Therefore, the end wall heating tape set point temperature of 195°C is about the highest temperature any part of the shock tube should be set to in order to stay within operating conditions.

Note that temperature uniformity is not as crucial for the mixing tank and other heated components on the HPST as it is for the driven section since data are collected near the end wall. Temperature uniformity is also crucial near the end wall region since this region is where the critical incident-shock velocity is measured. However, the steady state temperature of the mixing tank and the manifold must be kept at a temperature high enough to prevent condensation of the fuel mixture on the way to the driven section. Further testing will be needed to determine the exact temperature of the inside of the mixing tank, but for now, it can be assumed to have heating properties similar to the driven section. It is advised to keep the mixing tank at a temperature approximately 25°C higher than the driven section during experiments to ensure that the fuel remains in the vapor phase once introduced into the shock-tube facility. Also, since the diameters of the tubing on the manifold and the hoses connecting the mixing tank to the vacuum section are significantly smaller than those of the mixing tank or the shock tube, it can be assumed that using temperatures similar to those of the driven section or higher will keep the inside temperature of these components above the temperature required to keep liquid fuels in the vapor state. The temperature the heating jackets, mixing tank, and manifold should be set to will depend on the fuel being studied and can be found by choosing a temperature higher than the temperature associated with the vapor pressure of the fuel.

3.2 Rate of Heating

Although temperature uniformity studies were conducted to measure the inside air temperature of the shock tube, these were all performed when the inside of the shock tube was at atmospheric pressure. During shock-tube experiments, the driven section is vacuumed down, close to zero gage pressure, and then filled to a specific pressure with the test mixture. With the heated shock-tube setup, the test mixture would be at a higher starting temperature in the mixing tank than the temperature of the heated driven section. Because the use of shock waves in the driven section do not allow thermocouple use on the inside of the shock tube, the initial temperature of the driven section will mostly be based off of the temperature uniformity studies conducted in the previous section. The time that it takes for the mixture to reach this calibrated driven section temperature is important for making sure that the correct initial temperature is accounted for when analyzing reflected shock conditions. Therefore, a rate of heating experiment was designed to measure the temperature of the driven section at specific fill pressures using a port modified with a thermocouple wire, shown in Figure 20.

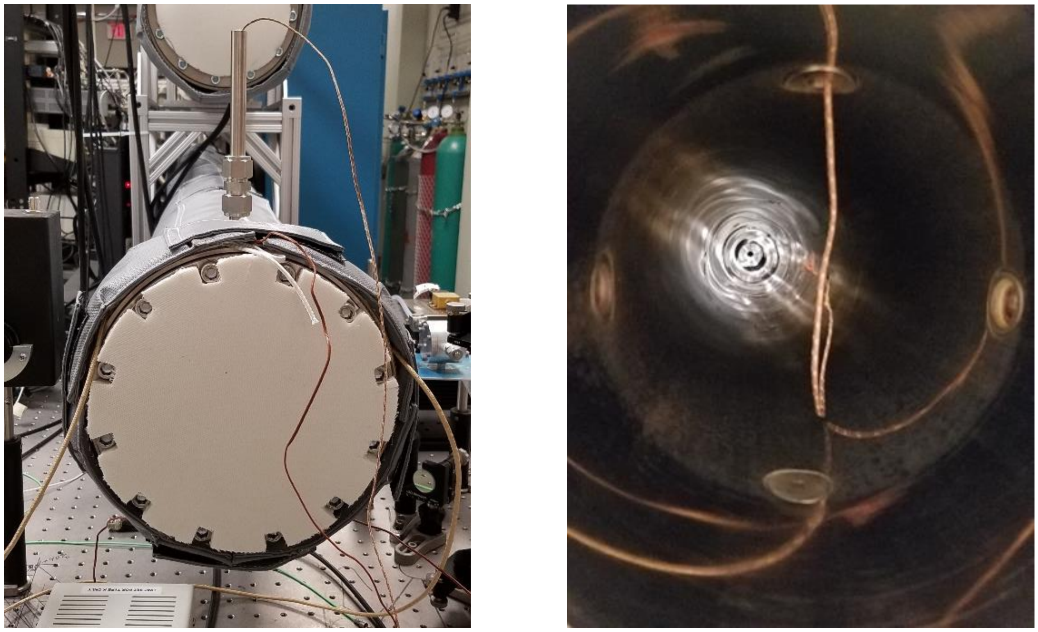


Figure 20- Temperature measurement port (left), Type K thermocouple wire inside the driven section (right).

A type K thermocouple wire was threaded through a piece of 0.5-in Swagelok tube and was filled with epoxy to create a seal. The piece of 0.5-in tube was connected to another 0.5-in Swagelok tube, which had already been welded to a port on the driven section. Next, the modified port was secured onto the shock tube near the end wall, and the thermocouple wire was positioned to measure the air temperature near the radial center of the tube. The end wall was then secured in place and the driven section was vacuumed to near zero torr conditions. Note that at vacuumed conditions, the temperature inside the shock tube did decrease slightly with the exit of the heated air inside. However, the difference in temperature was within 2°C of the temperatures measured during the temperature uniformity experiments.

In a normal heated shock-tube experiment, the driven section would be filled with a gas mixture that was at a higher temperature from sitting in the mixing tank than the temperature of the driven section. This temperature difference between the mixing tank and the driven section would be approximately 25°C. In anticipation of performing studies with a room-temperature methane mixture where heating the mixture beforehand was not as crucial since condensation would not be an issue at room temperatures, room temperature air was used to determine the time that it takes for a gas mixture to reach the set temperature of the heated driven section. For these experiments, the driven section was set to 75°C, and the room temperature air being used to fill the driven section was at a temperature of 23°C. Since this temperature difference is higher than the one expected for a normal heated shock tube experiment, the settling time for room temperature air to reach the driven section temperature would be greater than that of a heated mixture.

The heated driven section was filled with room temperature air at pressures from 10 torr to 300 torr. The temperature of the mixture in the driven section was measured through the modified temperature-measurement port. As the room temperature air was introduced to the heated driven section, the temperature inside the shock tube increased to a maximum temperature, and then decreased back to the original inside temperature of the driven section. The increase in temperature tended to be higher with increasing fill pressures. This process of temperature increase and then decrease back to the original temperature occurred within 5 minutes for pressures of 10 torr to 300 torr. Also, the settling time for the mixture to reach within 0.2°C of the original temperature usually occurred within about 3 minutes. Therefore, it was determined that for room temperature mixtures being introduced into the driven section, the user should wait approximately 3 minutes after filling before running the shock. This modest delay allows enough time for the temperature of the mixture to equilibrate to that of the heated shock tube.

Further studies should be conducted to calculate the settling time needed for other heating jacket set point temperatures as well as for mixtures already heated by the mixing tank heating system. However, for the validation of the heated shock-tube setup, 3 minutes was used as a baseline settling time for the mixture to reach the 75°C inside temperature of the shock tube based on the results described above.

3.3 Methane Studies

Once the shock tube was calibrated and a sufficient settling time was determined for test mixtures, it was important to demonstrate that the shock tube was still capable of performing ignition delay time experiments and producing accurate results, despite the changes made to the shock tube with the heated setup. For this reason, methane, a well-studied gaseous fuel, was selected to validate the working setup for high-initial-temperature gas mixture experiments. A stoichiometric, φ=1.0 mixture of methane and oxygen with 98% argon (by volume) was selected for the study. Note that φ is the equivalence ratio of the fuel mixture and represents the ratio of the fuel-to-oxidizer mass ratio at the current conditions to the fuel-to-oxidizer ratio at the stoichiometric case. Since the conditions selected are already at the stoichiometric case, the equivalence ratio is 1.0. For the stoichiometric case, where there is enough fuel and oxidizer present to completely be turned into water and carbon dioxide products, the overall theoretical chemical reaction can be summarized as follows: $CH_4 + 2O_2 \rightarrow CO_2 + 2H_2O$. From this chemical reaction, the ratio of oxygen to methane is 2:1. Knowing that the total mixture will have 98% argon and a set total mixture pressure of 25.0 psi, the mole fractions of the mixture components and the partial pressures of each component can be calculated and are shown in Table 3.

Table 3- Methane mixture components for φ=1.0 and 98% argon.

Component	**Component Mole Fraction**	**Component Fill Pressure**
Methane, CH_4	0.00667	8.621 torr
Oxygen, O_2	0.01330	17.2 torr
Argon, Ar	0.98	24.5 psi

The methane mixture was made at room temperature by filling the mixing tank with 8.621 torr of methane, adding an additional 17.2 torr of oxygen, and then filling the rest of the mixing tank with argon until the total mixing tank pressure was 25.0 psi. Then, the heating jackets on the mixing tank were set to 113°C to reach an inside temperature of about 100°C and were left to heat the mixture overnight.

For the methane experiments, 0.01-in lexan diaphragms were selected to create near atmosphere reflected shock conditions. Pressure gages were connected to the Gage Scope computer-based oscilloscope board to record side-wall pressure traces during the shock experiments. Additionally, an OH* diagnostic was used to record the concentration of OH* during combustion, initiated by the shock wave. Using a photomultiplier, the amount of light emitted by OH* during combustion was recorded on Gage Scope and used to calculate ignition delay time. In engines, the ignition delay time is the amount of time between the fuel injection and the start of combustion, indicated by a pressure rise [22]. For these experiments, ignition delay time is defined as the time between the reflected-shock side-wall pressure rise and the steepest slope of the OH* emission trace. An example of a plot used to calculate ignition delay time is shown in Figure 21.

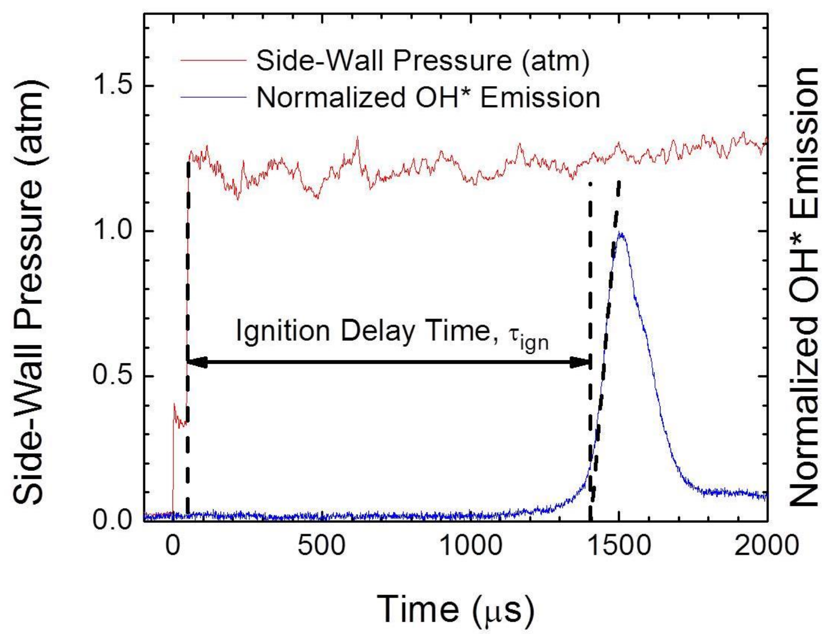


Figure 21- Ignition delay time analysis.

The side-wall pressure trace shows two step rises in pressure. The first rise in pressure is due to the incident shock wave, and the second rise in pressure is due to the reflected shock wave. The OH* trace shows the concentration of OH* in the test section as a function of the amount of light being emitted at 307 nm from this chemical reaction. By tracing a line along the steepest slope of the second pressure rise, the time at which the shock creates ignition conditions is found. By tracing a line along the steepest slope of the OH* trace, the point at which the trace reaches the time axis is the point of ignition. Therefore, ignition delay time is the time between the two slopes, as shown in Figure 21. For this example, the ignition delay time is about 1350 μs.

Using the OH* diagnostic to measure ignition delay time, experiments were conducted at an average pressure of 1.13 atm over a range of temperatures from 1784 K to 2184 K. A summary of the data collected is shown in Table 4.

Table 4- Methane results for φ=1.0, 98% argon.

Temperature (K)	**Pressure (atm)**	**τ_{ign} (μs)**
1784	1.161	1357
1821	1.190	689
1865	1.149	621
1904	1.143	435
1922	1.115	388
2022	1.098	201
2124	1.089	119
2130	1.093	109
2134	1.096	129

The results of the current study were compared with those of Mathieu et al.'s study of the same mixture at 0.9 atm and at 1.7 atm [23]. The plot in Figure 22 shows the results of both studies as well as a GRI 3.0 Model at 1.13 atm.

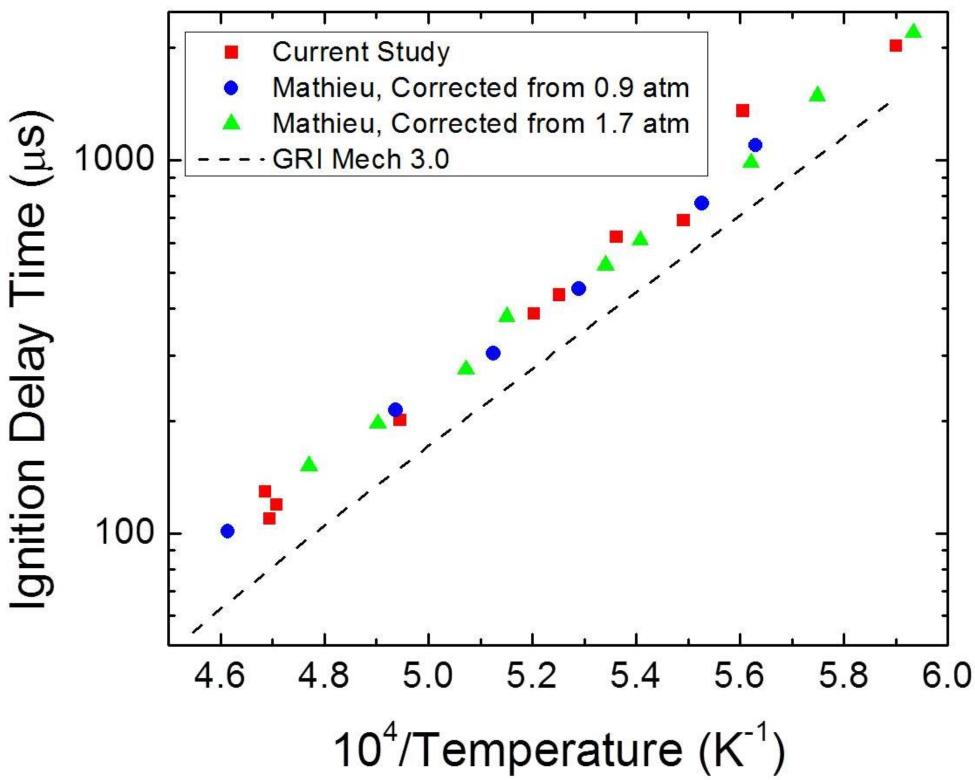


Figure 22- Methane, φ=1.0, P=1.13 atm comparison.

The results from the current study match well with those collected by Mathieu. Note that the study done by Mathieu was conducted at pressures slightly different than the pressures for the current study. Therefore, using pressure correction methods, the 0.9 atm and 1.7 atm experiments conducted by Mathieu were scaled to 1.13 atm. When scaled to 1.13 atm, the Mathieu studies align well with the current study. The GRI Mech 3.0 model seems to have ignition delay times that are slightly lower than the current study, but still shows overall agreement [24]. From the agreement of the current study to previous studies and models created for this specific mixture, it can be concluded that the heated facility on the HPST is still able to produce accurate results for gas phase mixtures.

3.4 n-Nonane Studies

The methane study proved that the heated HPST could perform well for high-temperature gas experiments, but testing liquid fuels is the main purpose of the heated facility. Therefore, a baseline liquid fuel was selected to prove that the heated facility could keep liquid fuels in the vapor phase for combustion experiments. After searching through the literature, two studies on n-nonane with 4% oxygen were selected as comparisons for the present study. In 2010, Davidson et al. performed a study on normal alkanes at Stanford University using their heated shock tube facility [25]. The manifold and mixing tank were set to a temperature of 70°C to accommodate the low vapor pressure of n-nonane at room temperature. Ignition delay time measurements were collected using OH* sidewall emission diagnostics. In 2016, He et al. performed a study of n-nonane and n-undecane at the Institute of Atomic and Molecular Physics using a heated facility [26]. Their facility included a mixing tank set to 150°C and a shock-tube temperature of 80°C to prevent the low vapor pressure fuels from condensing. CH* emission was used to collect ignition delay time data for the He et al. study. Both the

Davidson study as well as the He studies collected data at equivalence ratios of 0.5 and 1.0 for the n-nonane, 4% oxygen mixture at pressures near 2 atm.

With these studies for comparison, a 1000 torr, φ=0.5 mixture of n-nonane, 4% oxygen, and argon was created using the mole fractions and partial pressures found in Table 5. The balanced stoichiometric equation for this mixture is $C_9H_{20} + 14O_2 \rightarrow 9CO_2 + 10H_2O$. Therefore, the φ=0.5 lean mixture can be summarized by the equation, $C_9H_{20} + 28O_2 \rightarrow 9CO_2 + 10H_2O + 14O_2$. For the methane study mentioned previously, the mixture was made at room temperature and then left to heat to 100°C in the mixing tank overnight. However, with the liquid n-nonane fuel, this was not an option for creating the mixture. Instead, the mixing tank was set to 100°C before making the mixture. The manifold heating tapes were also set to 100°C. A vial of the liquid n-nonane fuel was attached to the manifold and covered with heating tape to allow the liquid fuel to turn to vapor. The first few vapors created from the n-nonane vial were vacuumed out to ensure that air or other components that may have gotten into the vial did not contaminate the mixture. Then, using a heated Baratron gage, 12.5 torr of n-nonane was added to the mixing tank, followed by 350 torr of oxygen. This mixture was then vacuumed to 41.4 torr and 958.6 torr of argon was added to the mixing tank. While filling, careful attention was paid to the amount of gas going into the mixing tank, especially as it heated up while sitting in the mixing tank. Then, the mixture was left overnight for uniform heating.

Table 5- n-Nonane, φ=0.5 mixture components.

Component	Mole Fraction	Component Fill Pressure
n-Nonane, C_9H_{20}	0.001429	1.43 torr
Oxygen, O_2	0.04	39.97 torr
Argon, Ar	0.9586	958.6 torr

The driven section of the shock tube was set to 85°C using the temperature calibration trends of set point temperatures of the heating jackets and the end wall heating tape. An OH* diagnostic, side-wall pressure, and end-wall pressure were used to collect ignition delay time data. Using the combination of a 0.02-in lexan diaphragm, an average pressure of 1.7 atm was achieved for these experiments. For the φ=0.5 condition, the data in Table 6 were gathered for a temperature range of 1320 K to 1444 K.

Table 6- n-Nonane results for φ=0.5.

Temperature (K)	Pressure (atm)	Ignition Delay Time (μs)
1320	1.536	587
1327	1.699	520
1356	1.713	393
1377	1.735	285
1444	1.768	122

The ignition delay times collected here were plotted and compared to the studies conducted by Davidson et al. [25] and He et al. [26] in Figure 23.

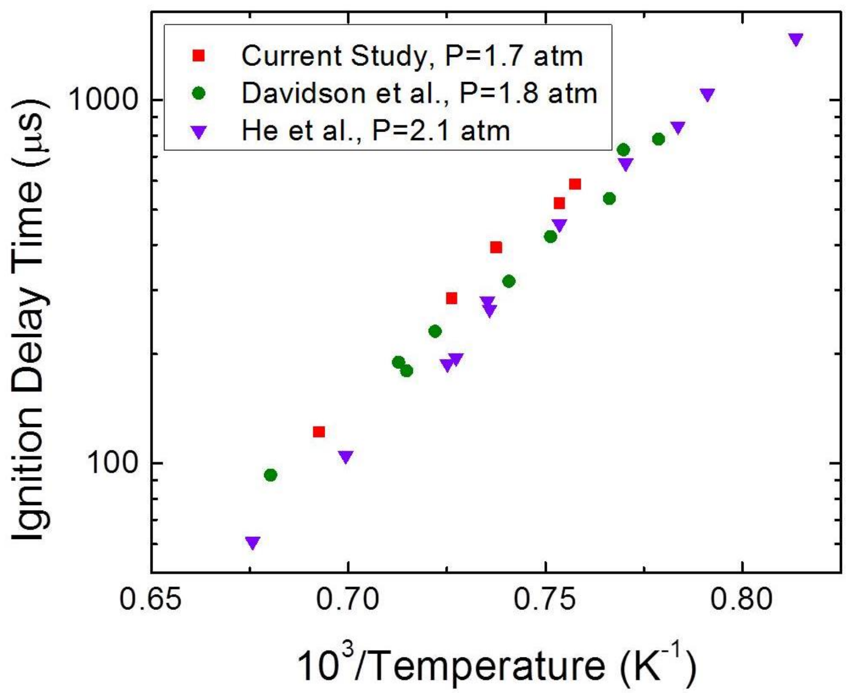


Figure 23- n-Nonane ignition delay time comparison for φ=0.5.

Comparison of the φ=0.5 cases to the current study shows that the current study falls in line with both the Davidson and He studies well. The current study seems to have slightly higher ignition delay times than the Davidson or He studies, but this is because the current study was conducted at a slightly lower pressure. Therefore, it can be said that the heated HPST facility is capable of collecting ignition delay time measurements for baseline liquid fuels.

To further prove the accuracy of the data collected by the heated shock tube facility, typical side wall pressure traces from the n-nonane experiments conducted here were compared to side wall pressure traces at similar pressures. Figure 24 shows a side wall pressure trace from an experiment with a reflected shock pressure, P_5 = 2.17 atm. Note that there are two distinct step rises in pressure, which indicate the arrival of the incident shock wave and then the reflected shock wave. The reflected-shock pressure levels off at P_5=2.17 atm and begins to decrease after approximately 2 ms of test time.

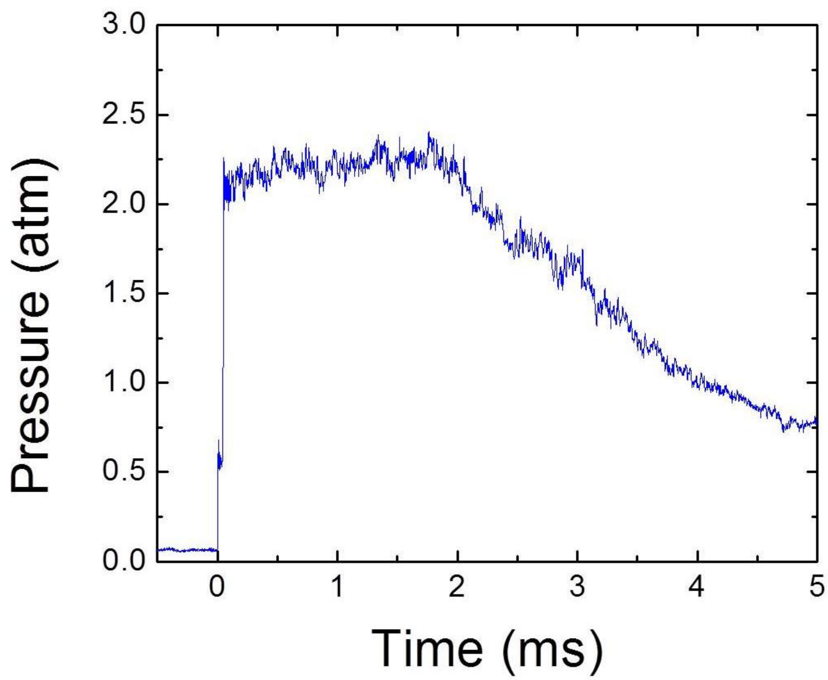


Figure 24- Side wall pressure trace from unheated HPST, P_5=2.17 atm.

Figure 25 shows the side wall pressure trace from a heated HPST experiment with P_5=1.67 atm. Although the P_5 pressure is not the same as the unheated condition, the shape of the heated trace is similar to the unheated pressure trace. The two-step rises in pressure are still very distinct, the second pressure rise levels out at the P_5 pressure, and there is still 2 ms of test time before the pressure begins to decrease at the end of the experiment. Therefore, it can be concluded that the heated setup did not affect the side wall pressure measurement capabilities of the shock-tube facility.

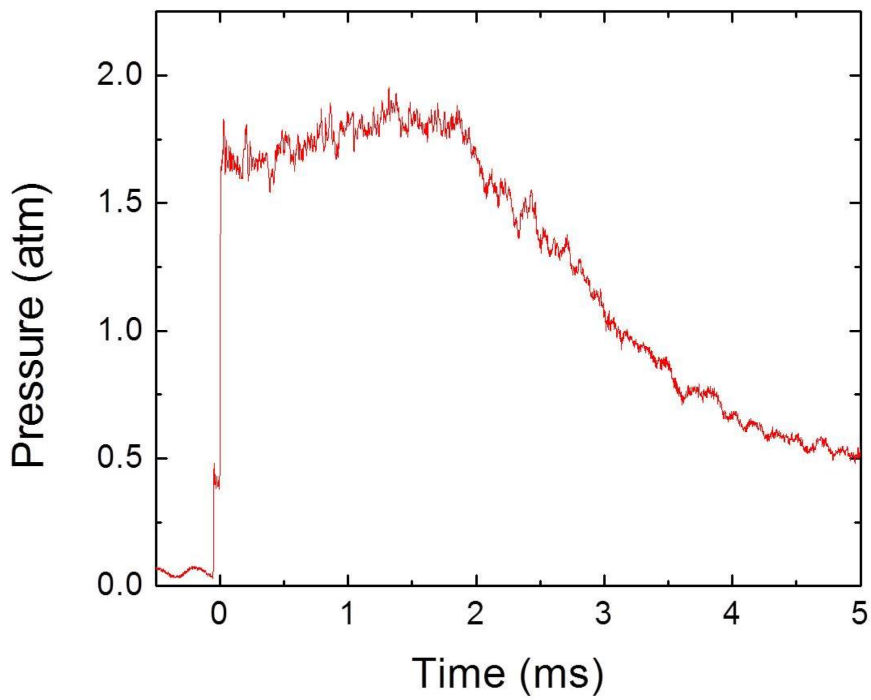


Figure 25- Side wall pressure trace for heated HPST, P_5=1.67 atm.

3.5 Uncertainty

Temperature measurements, pressure measurements, and creating mixtures were the biggest sources of uncertainty for this project. The uncertainty of the type K thermocouples used is 0.75%, so for high initial temperatures close to 150°C, the ±1°C temperature distribution measurements may not be as accurate [27]. However, errors in temperature distribution measurements were minimized by using the same thermocouple wires and temperature read out for each measurement. Each thermocouple was also used at room temperature to make sure that they all had a similar baseline temperature before being exposed to the heated shock tube. The measurements from these experiments were used as the initial T_1 temperatures for the methane and n-nonane experiments since the temperature inside of the shock tube cannot be easily measured while under a vacuum or while a shock is moving through the driven section. Therefore, the temperature inside the shock tube, despite the same heater settings, may be different than what it was for the temperature uniformity experiments due to the difference in pressure of the gas mixture inside of the shock tube. However, rate of heating studies showed that this difference in temperature is within 2°C of the temperature uniformity experiment measurements.

For shock tube experiments, the temperature and pressure in the reflected shock region was calculated by measuring the velocity of the shock wave instead of directly measuring T_5 and P_5. These reflected shock calculations were done through a program called FROSH, which calculates reflected shock conditions based on chemical composition, initial temperature, and shock speed [28]. However, the measurement of this shock velocity has uncertainties related to counter times. Overall, these uncertainties lead to a T_5 uncertainty of about ±6 K at 1800K and ±19K at 3500K [29].

The n-nonane study also brought to light a few challenges that need to be addressed with the heating system, particularly when using liquid fuels. It is important to keep all of the lines that the fuel is exposed to at a temperature above that associated with the vapor pressure limits of the fuel. Also, the fill pressure of the low vapor pressure liquid fuel is limited to the temperature at which the facility can be heated. Particularly at low fill pressures, leak rates of the heated facility start to come into play. With air leaking into the manifold due to heating, it is difficult to know exactly how much fuel and oxidizer are being added to the mixing tank. Working with limited amounts of heated pressure gages also decreases the accuracy of the filling process since the 0-to-1000-torr gage only reads to one tenth of a torr instead of the one thousandth of a torr that the unheated setup allowed. Therefore, some details of the heated HPST facility will need to be improved before conducting experiments with more complex liquid fuels. Some of these improvements are highlighted in the following chapter.

CHAPTER IV

CONCLUSIONS AND RECOMMENDATIONS

4.1 Summary

To study the combustion properties of liquid fuels and low vapor pressure fuels, it was necessary to create a heated shock tube facility. The high-pressure shock tube facility at Texas A&M University was modified to reach temperatures up to 150°C. Components of the shock tube such as measurement gages, transducers, o-rings, and valves were either confirmed to be compatible with high temperatures or were replaced with high-temperature components. Most notable of the replaced components included the 0-to-10-torr and 0-to-1000-torr MKS Baratron manometers, which were replaced with a heated 0-to-1000-torr manometer, and the nitrile o-rings which were replaced with high temperature VG-109 Viton o-rings. Once the high temperature o-rings were installed on the tube, leak checks were conducted to ensure the integrity of the new seals. Custom heating jackets from BriskHeat Corporation were designed to fit the dimensions of the HPST and mixing tank. These jackets are controlled by temperature controllers, which monitor the temperature of the outside wall of the shock tube through a J-type thermocouple placed on each heating jacket. To ensure uniform heating on the driven section and mixing tank, extra silicone foam insulation was added to cutouts in the jackets to cover ports, windows, the end wall, and end caps. An extra heating tape was added to the end wall of the driven section to make up for the heat loss occurring at the end of the shock tube. Heating tape and fiberglass insulation were added to the manifold to keep fuel from condensing as it is added to mixtures in the mixing tank and as it fills the driven section.

Temperature uniformity experiments were conducted to determine the settings needed for the heating jackets and end wall heating tape to heat the shock-tube driven section to 50°C, 75°C, 100°C, and 150°C. Using a metal rod assembly with thermocouples fixed at 1-foot increments along the driven section, the air temperature of the inside of the shock tube was measured. Settings were found which assured uniformity of the temperature of the shock tube to ±1°C for the last 7 feet, closest to the end wall where measurements during combustion experiments occur.

To ensure that the components of the shock tube were still working under heated conditions, ignition delay times were measured for a stoichiometric methane-oxygen mixture with 98% argon at 1.13 atm using an OH* diagnostic. These measurements were similar to the previous study done by Mathieu et al. [23], which proved the accuracy of the heated shock tube facility for gas mixtures.

A baseline liquid fuel mixture of n-nonane and 4% oxygen was also tested for ignition delay times at 1.7 atm, measured by an OH* diagnostic. These measurements were compared to studies by Davidson et al. [25] and He et al. [26] at φ=0.5. The current study proved to have similar ignition delay times to these previous studies. Therefore, the n-nonane study showed the heated shock tube's ability to conduct experiments with low vapor pressure and liquid fuels.

4.2 Recommendations

Before the heated HPST facility is ready to test more-complex liquid fuels such as Jet-A, JP-8, and RP-1, several modifications should be made to the current facility. For complex fuels, all of the lines on the manifold which come in contact with the fuel will need to be heated to temperatures above what is necessary to keep the fuel in the vapor phase. This modification includes the vacuum section and all the pipes and hoses leading to the vacuum section from the manifold and the mixing tank. Heating tape will also need to be added to the tombstone, nozzle, and diaphragm sections to ensure that condensation is not occurring at points further away from the end wall measurement region and to ensure that condensation on the diaphragm is not affecting the shock formation. Additional temperature controllers will be needed to keep these components at a set point temperature.

Additionally, more time should be spent trying to improve the uniformity of heating the driven section further away from the end wall. Although the end wall region is uniform to ±1°C for the last 10 feet for most temperatures, it would be better to have uniformity in the entire driven section. Heating the vacuum section, nozzle, and tombstone should help to increase uniformity of the temperature in this region of the driven section.

Leak rate checks should be performed on the manifold, mixing tank, and driven section under heated conditions to minimize sources of impurities in the mixtures. Steps should be taken to replace leaking components and to tighten places which may be leaking while the heated setup is in use.

Also, to improve the accuracy of making mixtures, it would be recommended to order more pressure gages which have operating temperatures more suited to the heated HPST temperatures. This remedy includes purchasing a heated 0-to-10-torr manometer to increase the current level of filling precision from one tenth of a torr on the 0-to-1000-torr manometer to one thousandth of a torr. It would also be ideal for the Setra pressure transducers to be replaced with high temperature pressure transducers. This switch would further aid in the driven section filling process at high pressures, help with making mixtures, and give the user a better knowledge of how much mixture is remaining in the mixing tank.

Furthermore, this heated shock tube could be used at temperatures up to 200°C with the replacement of even more components on the shock tube. This additional work includes pressure transducers, o-rings, and valves, whose maximum operating temperatures are currently close to 200°C. However, the heating jackets themselves, the heating tapes, and the controllers used on the current heated setup are fully capable of heating the shock tube to 200°C temperatures.

With adjustments to the current heated shock-tube setup, this heated HPST facility will be used to study liquid fuels for the improvement of power generation and aviation fuels which have not been fully characterized through ignition delay time studies.

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APPENDIX A

TEMPERATURE COMPATIBILITY OF SHOCK-TUBE COMPONENTS

The following table shows the operating temperatures for the components of the heated shock-tube setup. Note that most of the components can operate at temperatures up to 200°C, except for the PCB P113B22 pressure transducer located on the end wall. For experiments requiring the shock tube to be heated above 135°C, this transducer should be removed and the port on the end wall should be replaced with a blank port.

Table 7- Operating temperature of heated shock-tube components.

Description	**Tube Section**	**Operating Temperature**
VG109 O-Rings	Driven and Vacuum	204°C
SS-4P4T-BK Swagelok Valves	Manifold	204°C
SS-8P6T Swagelok Valves	Manifold	204°C
SS-1VS4 Swagelok Needle Valves	Manifold	232°C
PCB P113A Pressure Transducer	Driven sides	204°C
PCB P113B22 Pressure Transducer	End wall	135°C
Kistler 603B1 Pressure Transducer	Driven underside	200°C
MKS Baratron 631 Heated Manometer	Pressure Gage	150°C or 200°C
PCB 002C10 Coaxial Cables	Driven	204°C

Table 8 shows the operating temperatures of the high-pressure shock-tube components before it was modified with the heated setup. Note that the original shock-tube setup had several components which were not compatible with high temperatures, specifically pressure transducers, manometers, and o-rings. The pressure transducers and manometers were replaced with high-temperature models which are better suited to the operating temperatures of the heated shock tube. Also, all the nitrile o-rings on the shock tube driven and vacuum section were replaced with VG109-90 Viton o-rings, which are rated to higher temperatures. Some of the supports on the manifold and vacuum section also need to be replaced with high temperature silicone and metal U-bolts. Note that the operating temperatures of the vacuum pumps are also listed, but since they do not need to be heated for the operation of the heated shock tube, they were not replaced.

Table 8- Operating temperatures of the high-pressure shock tube components before the heated setup.

Description	Tube Section	Operating Temperature
Nitrile O-Rings	Driven and Vacuum	100°C (shorter life at 121°C)
SS-4P4T-BK Swagelok Valves	Manifold	204°C
SS-8P6T Swagelok Valves	Manifold	204°C
SS-1VS4 Swagelok Needle Valves	Manifold	232°C
Setra Model 225, 250 psia	Pressure Transducer	85°C

Table 8 Continued.

Description	Tube Section	Operating Temperature
Setra GCT-225, 200 atm	Pressure Transducer	85°C
PCB P113A Pressure Transducer	Driven sides	204°C
PCB P113B22 Pressure Transducer	End wall	135°C
Kistler 603B1 Pressure Transducer	Driven underside	200°C
MKS Baratron 626A Manometers	Pressure Gage	50°C
PCB 002C10 Coaxial Cables	Driven	204°C
Routing Clamps (Polypropylene)	Manifold and Vacuum	87°C (160°C melting point)
Varian NW25 Aluminum Block Valve	Vacuum	150°C
DS102, DS302, DS402 Vacuum Pumps	Vacuum	40°C
Varian 551 Turbo Pump	Vacuum	70°C

APPENDIX B

O-RING SIZES FOR THE HIGH-PRESSURE SHOCK TUBE

Table 9 shows a list of o-ring sizes for the high-pressure shock tube. These sizes were determined from the original drawings of the high-pressure shock tube, detailed by Aul [10]. Note that the 2-125 o-rings on the ACTUATOR_CONNECTOR part are mismarked on the original drawings as 2-215. When replacing the nitrile o-rings with Viton o-rings for the heated setup, 2-219 o-rings were used, but had a very snug fit. Since the replacement of the o-rings, it was determined that the correct size for this part is 2-125, as marked in the table. Also note that the nitrile o-rings in the driver section of the shock tube were not replaced with Viton o-rings. This is because it is not necessary to heat the driver section for the heated shock tube experiments.

Table 9- High-pressure shock tube o-ring sizes.

Section	**O-Ring Size**	**Quantity**	**Parts Involved**
Driven	2-260	6	TUBE_A, TUBE_B, TUBE_C, TUBE_D, ST_ENDWALL, TOMBSTONE
Mixing Tank	2-260	2	MT_ENDWALL_STING, MT_ENDWALL_BLANK(O-RING)
Windows	2-122	25	BLANK_PORT (25)
End Wall Ports	2-119	5	ENDWALL_PORT (5)

Table 9 Continued.

Section	O-Ring Size	Quantity	Parts Involved
Poppet Valve	2-125*	2	ACTUATOR_CONNECTOR (2)
Poppet Valve	2-114	2	CONFLAT_FLANGE_A, ACTUATOR_CONNECTOR
Poppet Valve	2-238	1	PISTON
Vacuum/ Poppet	2-241	1	CONFLAT_FLANGE_B
Diaphragm	2-239	6	CUTTER_RING, BLANK_RING, SPACER (2), LOCKER (2)
Driver	2-343	1	FLANGE_2

*Note that this size is not what is found on the original drawing for the ACTUATOR_CONNECTOR. The original drawing is mismarked.

APPENDIX C

LEAK RATE RESULTS

The following table shows the final leak rates of the various shock-tube components before the heating system was applied.

Table 10- Leak rate results.

Section	**Leak Rate (mTorr/min)**
Shock Tube Driven Section	1.70
Mixing Tank	0.38
Manifold	0.85
Vacuum Section	0.10

APPENDIX D

PROCEDURES FOR HEATING SYSTEM USE

D.1 Procedures for Heating Jacket Use

The following list describes procedures for using the heating jackets on the driven section of the high-pressure shock tube as well as the mixing tank.

1. Ensure that the tube is closed, all insulation is in place, and that all jackets are securely fastened. Also make sure that extra wires or materials that may be affected by heat are cleared away from the heating jackets and heated facility.
2. If not already plugged in, plug in the controller to a power source. Plug in the heating jacket power cord and the heating jacket thermocouple plug into the temperature controller.
3. Turn on the controller. Once turned on, the controller will display the temperature of the thermocouple at the designated position on the heating jacket. Note that the location of the thermocouple is marked with a yellow triangle on the corresponding heating jacket.
4. Press and hold the * button on the front of the controller to view the current set point temperature. To change the set point temperature, press and hold the * button while using the ↑↓ keys to increase or decrease the temperature. Note that when changing the set point temperature by a large amount, pressing and holding the ↑ or ↓ key will allow the temperature to increase or decrease faster.

5. Once the temperature is set to the correct set point, release hold on all keys. Press and hold the * button to ensure that the set point is correct. The thermocouple temperature should start to reflect the set point temperature change.

6. Repeat steps 2 through 5 for all temperature controllers and corresponding heating jackets. Note that there are 5 driven section jackets and 3 mixing tank jackets. The naming convention is as follows:

   a. Mixing tank heating jackets are named 1, 2, and 3, where 1 is closest to the driven section end wall, 2 is the middle jacket, and 3 is closest to the vacuum section.

   b. Driven section heating jackets are named 4, 5, 6, 7, 8, where 4 is closest to the end wall of the driven section, and the jackets are numbered sequentially by position as they get closer to the tombstone. Note that the jacket closest to the tombstone is jacket 8.

   c. The corresponding temperature controllers for each heating jacket are labeled according to the heating jacket number, where the mixing tank jackets also have “MT” in front of their jacket number.

7. Allow time for the controller to reach the set point temperature. Note that although the controller may say that the tube is at the set point temperature, but the air inside the tube may not be at that temperature yet. Settling time for most temperature set points is approximately 7 hours.

8. When the heated facility is no longer being used, switch off the temperature controllers and allow the shock tube to cool.

9. Refer to the BriskHeat corporation “X2 Benchtop PID Digital Temperature Controller Instruction Manual” for further procedures regarding the operation of the temperature controllers [30].

When using temperature settings higher than 50°C, it is advised to use the following additional procedures for turning on the heating jackets and interacting with the heated facility:

1. If setting the jackets to temperatures above 50°C, extra caution must be used during operation. Ensure that high temperature gloves are worn when handling the jackets and the heated facility and that temperature sensitive items are removed from the vicinity of the heated facility.

2. When trying to achieve a set point temperature of above 50°C, allow the jackets to heat up in 25°C increments. For example, if the target temperature is 100°C, first set the controllers to 50°C. Once all controllers have reached the set point temperature, set the controllers to 75°C. Wait for the temperature controllers to settle at the set point, and then set the controllers to 100°C. By heating in increments, it prevents overshooting the set point temperature, which is particularly important at high temperatures close to the operating limits of shock tube components. For the temperature limits of the shock tube components of this facility, see the “Temperature Compatibility” charts in Appendix A. This also decreases the amount of power used by the

temperature controllers at one time since higher temperature gradients (relative to the set point temperature) require more power to reach the set point temperature.

3. At very high temperatures, make sure that all personnel working near the heated shock tube facility are aware of the safety precautions associated with heated shock tube use.

D.2 Procedures for End Wall Heating Tape Use

To provide a more-uniform temperature distribution along the driven section near the end wall, an extra heating tape was installed on the end wall flange underneath the end of heating jacket 4. The following procedures detail the use of this heating tape and the Omega CSi32 series miniature bench-top controller associated with it. Remember to use caution at high temperatures, utilizing the appropriate protective gloves to prevent burns when needed.

1. Ensure that the heating tape is wrapped tightly around the end wall, making sure that the heating tape is not overlapping itself.
2. Ensure that a type K thermocouple is plugged into the back of the controller and that the heating tape is inserted in the "output 2" plug.
3. Position the thermocouple between the heating tape and the surface of the end wall flange at the top of the flange. Secure the heating jacket around the heating tape and thermocouple.

4. Turn on the controller. The display should read the current temperature of the thermocouple input. To change the set point temperature of the controller, press the left-most circular arrow button to display “SP1.” Press the enter button on the far right. Use the ↑ or ↓ buttons to increase or decrease the set point to the desired temperature. Note that pressing and holding the arrow buttons will allow the temperature setting to change at a faster rate. Press the circular arrow button on the far left. The display should now read “SP2”. Press the enter button on the far right and use the ↑ or ↓ buttons to change the set point to the desired temperature. Press the circular arrow button twice to return to the current temperature display. The temperature should be adjusting to the set point.

D.3 Procedures for Manifold Heating Tape Use

The manifold section of the heated high-pressure shock tube is covered with heating tapes labeled in zones. The zones explained earlier in this thesis are each controlled by a single temperature controller, labeled with their respective zone number. The following outlines procedures for manifold heating tape and CN7800 temperature controller use. Remember to use caution around the heated manifold at high temperatures and wear the appropriate protective gloves to prevent burns. Also, do not touch the terminals of the CN7800 temperature controllers or their associated terminal blocks and solid-state relay components while the circuit is energized by the wall plug. Use caution to prevent electric shock and make sure to be fully grounded when plugging in the controllers.

1. Ensure that the thermocouple attached to the controller is reading the temperature from a cold spot in the zone. This ensures that the entire heated zone is at the set point temperature or higher.
2. To turn on the CN7800 controllers, plug the controller into a wall outlet. The controller should also be connected to a power strip with several heating tapes. Ensure that the power strip is flipped to the on position. The controller display should light up with a top and bottom read out of temperature.
3. Ensure that the controller is set for a type K thermocouple input. Press and hold the left-most arrow button until the input is flashing. It should be set to the “y” setting. If not, use the ↑ or ↓ buttons to reach this setting. Then, press the left-most button twice to return to the main screen. Refer to the CN7800 Manual [31] for more details on this procedure.
4. On the main display, the controller should have two read outs of temperature. The top temperature displays the temperature being measured by the thermocouple in red and the bottom temperature shows the set point temperature in green. To change the set point temperature, use the ↑ or ↓ buttons to increase or decrease the temperature. Note that pressing and holding the arrows will increase the speed at which the temperature increases or decreases. Once the desired set point temperature is reached, press the left most button on the face of the controller twice. To ensure that the temperature is set, make sure that the set point display is not flashing.

5. Set the controller to start heating by selecting the circular arrows button, second from the left on the controller. The “r-5” menu should be displayed with the setting “off”. Use the ↑ or ↓ buttons to change “off” to “run”. Press the left most button twice. The top temperature read out should now be changing to match the bottom temperature set point.
6. Repeat this process for each temperature zone controller on the manifold section.

APPENDIX E

PROCEDURES FOR TEMPERATURE CALIBRATION EXPERIMENTS

E.1 Roller Probe Temperature Uniformity Experiment Procedures

The following procedure outlines temperature uniformity experiments using the insulation from heating jackets only and the end wall off the shock tube. The main tool used to collect data was a roller thermocouple probe from Omega. Note that this was not the method used to collect the final temperature distribution of the driven section.

1. Prepare the roller thermocouple probe for use by ensuring that the thermocouple connections are secure. Make sure that the end of the thermocouple attached to the top of the probe (used to measure air temperature) is not touching the probe itself. Note that thermocouple B is measuring the wall temperature via the roller thermocouple probe and that thermocouple A is measuring the air temperature via the thermocouple attached to the top of the probe.
2. Plug thermocouple A and thermocouple B into temperature reading devices. For these experiments, temperature controllers were used to read out the measured temperature (without being plugged into a heater). To ensure the thermocouples are working, warm one of the thermocouples with your fingers so that the temperature of that thermocouple increases accordingly.
3. Remove the end wall from the shock tube. Ensure that the diaphragm section of the shock tube is closed.

4. Place the roller probe into the shock tube. Use connecting sticks to push the roller probe down the shock tube until the probe is at the nozzle of the shock tube.
5. Carefully remove the connecting sticks from the shock tube, making sure that the roller probe does not get pulled back. A flashlight is useful in this process.
6. Record the temperature of the wall of the tube and the air inside the tube with the probe close to the nozzle.
7. Using a ruler, pull the thermocouple probe one foot towards the end of the tube (where the end wall is normally placed). Allow the temperature to settle, then record wall and air temperature measurements.
8. Repeat step 7 until the thermocouple probe reaches the end wall position.
9. Remove the thermocouple probe.
10. Set the temperature controllers to the desired set point temperature and allow the heated shock tube to reach steady state with the end wall off.
11. Place the roller probe into the shock tube. Allow the temperature to settle, then record the wall and air temperature at the point of the pressure transducer on the bottom of the tube. This was used as the “zero” point for when the end wall is on the tube during normal shock tube operation. Use caution and appropriate protection when dealing with heated surfaces such as high temperature gloves to prevent burns.

12. Use appropriate connecting sticks to push the roller probe into the tube 1 foot. Allow the temperature to settle, then record measurements for the air and wall temperatures. Note that for high temperatures, the material of the connecting sticks must be considered:

    a. At temperatures up to 50°C, the connecting sticks normally used to clean the shock tube were used to push the probe further into the tube.

    b. At temperatures between 50°C and 100°C, the connecting sticks were modified by wrapping fiberglass insulation around the duct taped areas of the stick. This acted as a barrier between the duct tape and the walls of the shock tube to prevent the duct tape from melting and sticking onto the inside of the shock tube.

    c. At temperatures higher than 100°C, the plastic connecting sticks began to soften due to the heat. A metal connecting stick assembly was created to push the probe down the tube. Instead of twisting together via screw-like components (as used for the plastic connecting sticks), the metal connecting sticks fit inside one another and are secured with a nut and bolt. To prevent the metal sticks and connecting bolts from scratching the inside of the tube, fiberglass insulation was wrapped around the metal connecting sticks to cushion and elevate the metal while inside the tube. Greater caution should be taken with the metal sticks so as not to scratch the inside of the shock tube while being assembled to increase its length inside the tube.

13. Collect measurements at one foot increments until the probe reaches the nozzle.
14. Carefully remove the connecting sticks from the tube, disassembling each piece. Note that gloves should be used during this process to prevent burns.
15. Carefully pull the roller probe out of the shock tube.

Although these experiments give a good idea of how the jackets work, they did not give accurate temperature results due to the heat loss through minor exposed areas, not covered by jackets and the end wall. Because of this, closed end wall experiments for characterization were conducted using the procedures below. Also, the shock tube was further insulated to prevent heat loss through gaps in the heating jackets on the shock tube.

E.2 Closed End Wall Temperature Uniformity Experiment Procedures

For closed end wall temperature uniformity experiments, a metal connecting rod assembly was created and outfitted with thermocouples to allow temperature measurement while the end wall was closed. The metal connecting rod assembly was created as follows:

1. Connect the metal rods together by fitting each rod inside the next and securing with a nut and bolt.
2. Wrap fiberglass insulation around the end of each individual rod to create an insulation disc, which provides height to the rod and prevents it from scratching the inside surface of the tube.

3. Secure the fiberglass insulation by tying a piece of wire through the middle of the disc and twist the wire at the edge of the disc. Make sure that the wire twists are on the top of the disc, to prevent it from scratching the bottom or side walls of the tube.
4. Cover the ends of the metal rod assembly with fiberglass insulation to prevent the edges from scratching the inside of the tube.
5. Measure one foot increments down the metal rod assembly and mark with a permanent marker.
6. Cut type K thermocouple wires to extend from one end of the rod assembly to each 1 foot marking with at least 4-ft of slack at the end to allow the thermocouple to reach to a temperature measurement device. Note that there should be a thermocouple for every foot marked. However, in the assembly used in these experiments, the 11-ft and 13-ft thermocouples were missing.
7. Attach each thermocouple at the designated foot marking using a piece of wire. Bend the end of the thermocouple upwards to ensure that the thermocouple will measure the air temperature instead of the wall temperature inside the tube.
8. Take the end wall off the shock tube and remove the measurement port on the bottom of the tube closest to the end wall.
9. Disassemble the rod assembly into the individual rods.
10. Carefully feed the first rod into the tube, making sure that no wires are scratching the inside of the tube and that the thermocouples are as vertical as

possible to ensure accurate air temperature measurement. Keep the long end of the thermocouple outside the tube

11. Attach the next rod using a nut and bolt and feed the rod assembly down the tube, paying attention to wire and thermocouple position. Continue this procedure until all the connecting rods are attached and all of the ends of the thermocouple wires are coming out from the end wall side of the tube.
12. Feed all the excess thermocouple wires through the bottom port on the tube so that temperature measurements can be recorded while the end wall is on the tube. Insert insulation into the hole to prevent heat loss and to secure the thermocouple wires in place.
13. Position the last thermocouple so it sits directly above the bottom port of the tube.
14. Put on the end wall, making sure that the end wall will not be contacting the end of the metal rod assembly or push it further into the tube. Secure the end wall in place.
15. Attach the appropriate thermocouple connectors to the ends of the thermocouples so that temperature measurements can be recorded.
16. Record a baseline temperature measurement for all the thermocouples to get a temperature profile at room temperature and to make sure that all of the thermocouples are working properly.

17. Heat the tube according to a desired set point. Once the inside of the tube reaches a steady state temperature, record measurements for each foot along the tube using the thermocouples coming out of the shock tube.

E.3 Closed End Wall Temperature Uniformity Experiments with Thermocouples Coming out of the Nozzle

To prevent heat loss in the end wall region and to increase the accuracy of the measurements in the end wall region, temperature uniformity experiments were conducted using the metal rod assembly described in the "Closed End Wall Temperature Uniformity Experiment Procedures" from above.

1. Modify the metal rod assembly so that only the last 7 feet are outfitted with type K thermocouples at one foot increments. If the metal rod assembly from previous experiments is available, use the longest thermocouple wires available.
2. Secure the thermocouple wires so that they are fixed to the metal rod, and re-label the ends of the thermocouple wire leads to reflect the location at which the thermocouple is positioned on the metal rod assembly between 0-ft and 7-ft from the end wall.
3. Open the end wall and the diaphragm section of the shock tube. Use a string to tie together the ends of the thermocouple wires together. Feed the string through the length of the shock tube driven section so that the thermocouple wire leads are coming out of the nozzle. Feed the metal rod assembly into the driven section from the end wall, connecting metal rods with bolts as needed.

4. Position the 0-ft thermocouple directly above the pressure port nearest to the end wall at the zero position where shock tube measurements occur. Close the end wall.
5. Add thermocouple connectors to the wire thermocouple leads coming out of the nozzle. Insert foam insulation into the nozzle to prevent heat loss through the nozzle opening.
6. Set the heating jackets and heating tapes to the desired set point temperature, making sure to heat the shock tube at increments of 25°C if a temperature of higher than 50°C is desired. Allow the temperature of the shock tube to reach steady state temperature over the course of approximately 7 hours.
7. Record temperature data at each of the 7 one-foot increments from the end wall by plugging the thermocouples into a temperature reading device.
8. Adjust the temperature of the heating jackets and heating tapes as needed until a uniform temperature distribution is achieved for the desired set point temperature.