AN ANALYSIS OF THE THERMAL STABILITY OF CONVENTIONAL AND ALTERNATIVE AVIATION FUELS

by

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Institute for Aerospace Studies
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Abstract

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An experimental apparatus was used to examine the thermal stability of conventional and alternative aviation fuels. The apparatus is a simplified but controllable representation of an aircraft fuel system consisting of a preheating section and a test section. The preheating section simulates the fuel conditions as it acts as a coolant on board of the aircraft while the test section simulates the conditions of the fuel injection nozzles. The apparatus measures the accumulated deposit by taking the pressure drop data across the heated test section. After thermal stressing, the pressure drop data is verified by a carbon burnoff apparatus. The fuel chemical composition is evaluated by nuclear magnetic resonance spectroscopy. Experimental results are presented and discussed in this thesis for four different types of aviation fuels to show the relationship between fuel chemical composition and coking propensity. The experiments show that fuels with aromatic content tend to produce more deposits and the alternative fuels are potentially more thermally stable than their conventional counterparts.
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Chapter 1

Introduction

The aviation industry in recent years has shown no sign of slowing down. With almost 3.1 billion passengers in 2013 and accounting for about 2% of global greenhouse gas (GHG) emissions [1], aircraft manufacturers are scrambling for innovations that will further reduce the GHG emissions and the cost of travel in order to open up new markets. Recent developments in lighter structures made of composites have significantly reduced fuel consumption while improved engines are burning hotter to increase thermal efficiency. However to date, there are only a small handful of types of fuels that are used in commercial aviation (Jet A and Jet A-1 being the most dominant), due to the stringent requirements of fuels’ physical and chemical properties [2]; and any aviation fuel must also maintain backward compatibility with older engines. As conventional fuels are nonrenewable and contribute to global warming, it becomes prudent to begin exploring alternative fuels.

One of the challenges associated with finding appropriate alternative fuels is consequence of newer engine designs that are progressively burning hotter, which puts a strain on the material limits of the turbine blades. One of the ways used to cool the turbine blades is to use jet fuel to absorb the excess heat before the fuel is injected into the combustor. However, prolonged exposure to high temperatures will cause the fuel to
thermally degrade and prematurely break down via a host of mechanisms collectively known as coking. These reactions eventually produce insoluble precipitates that clog fuel pipes or collect at fuel spray injectors. If the pipe channels become narrower, the fuel pumps would have to expend more energy to keep a constant flow rate and if the spray injectors were obstructed, the fuel spray pattern would no longer be uniform; both of which cause an overall reduction in operating efficiency. Hence, for any new fuel to be approved for commercial use, it must pass through a multitude of tests and standards.

This thesis plans to examine the coking propensities of conventional and alternative aviation fuels via a test setup which was designed and built at the University of Toronto Institute of Aerospace Studies (UTIAS). The coking rig uses two independent diagnostics, a pressure drop measurement and a carbon burnoff technique to determine the mass of accumulated deposit in the test section.
Chapter 2

Background and Literature Review

2.1 Chemistry of Deposit Formation

In order to properly characterize the coking propensities of alternative aviation fuels, the chemical kinetics of how insoluble deposits form needs to be examined. Numerous studies have been conducted to describe the thermal stability of jet fuels; Balster et al [3] showed that thermally stressed fuels decompose at a rate dependent on the temperature.

Commodo et al [4] used UV-visible absorption and fluorescence spectroscopy to examine the change in the chemical composition of fuels as they are heated prior to entering the engine. It was found that as the fuels were exposed to higher temperatures, more aromatic compounds formed. As temperature increased, the compounds were composed of more rings [5]. These large complex aromatics with molecular masses upwards of 600-900 g/mol [6] are sufficiently heavy as to render them insoluble resulting in their precipitation out of the fuel and depositing on the inner surfaces of the pipes.

Liu [7] modeled the chemical and thermal rate kinetics of the formation of insoluble deposits in Jet A-1 using eighteen intermediate reactions within the fuel for the formation of radicals and three terminating reactions for the formation of deposits. A pair of wall reactions were also considered as the autooxidation of fuel could be catalyzed by the
metal wall. The simulation showed that increasing temperatures also increased the rate of deposit formation, which corroborates [6] and [8]. This model simulated flow in the laminar regime and used parabolic velocity profiles, which Liu notes could be influenced by steep temperature gradients and create a significant buoyancy effect.

## 2.2 Conditions that Affect Deposit Formation

### 2.2.1 Fuel Temperature Regime

There are two temperature regimes, autoxidation and pyrolysis where the type of fuel degradation and deposit formation markedly differs from one another. Autoxidation is the cooler of the two regimes and Hazlett [9] showed that dissolved O$_2$ reacts with the fuel to produce hydroperoxide radicals, which act as precursors to deposit formation. The pyrolytic regime on the other hand dominates when the temperature is much higher and/or the dissolved oxygen has been depleted. This regime is characterized by the fuel molecules breaking into free radical components themselves due to the heat and act as precursors to deposit formation. The approximate temperature regimes are shown in Figure 2.1.

Grinstead and Zabarnick [11] conducted tests to examine the oxidation rates of fuels when exposed to high temperatures as part of a program goal to increase the heat sink capacity of Jet Propellant 8 (JP-8), a widely used conventional fuel in the military of North Atlantic Treaty Organization countries. An apparatus was designed that bubbled O$_2$ into the fuel as it was heated, as recommended by ASTM D4871 [12] in order to provide an excess of dissolved O$_2$ for autoxidative degradation to take place. It was found that doping the fuel with antioxidant additives such as metal deactivators reduced the oxygen consumed by the fuel by as much as 80%.

The pyrolytic regime was investigated by Andresen et al [13] who showed that alkanes (paraffins) in fuels exposed to temperatures higher than the autoxidation regime or in
Figure 2.1: Graph of carbon deposit formation as a function of temperature in the autoxidation and pyrolysis regimes [10].

the absence of oxygen tend to form cycloalkanes which then form aromatics which finally combine to larger polyaromatics eventually rendering them insoluble.

### 2.2.2 Fuel Flow Regime

Previous studies into fuel thermal stability as well as the designated standard ASTM D1655 test [2] for determining thermal stability are conducted in the laminar flow regime. However, the actual operating regime may be not be laminar. Pande et al [14] examined fuel flow in three different test rigs, one of which was in a turbulent regime. The results showed similar correlations for deposit formation with respect to temperature whether turbulent or laminar flow was considered. However, the laminar flow conditions showed a higher concentration of deposits while deposit formation rate were similar between laminar and turbulent conditions. This is likely due to less mixing and an increased residence time in the laminar regime compared to the turbulent, where fuel close to the tube walls were exposed to high temperatures for a longer time than in the core of the flow. The apparent activation energies of deposit formation in both the laminar and turbulent flow
were also calculated in the study, and the activation energies of both regimes were within measurement errors of each other, leading the authors to conclude that temperature, not Reynolds number nor flow condition was the critical factor in determining how much deposit was formed. Finally, the authors noted that oxygen depletion in the Jet Fuel Thermal Oxidation Test (JFTOT) could be alleviated by increasing the flow rate.

A contrasting study by Spadaccini et al. [10] showed that deposit formation can be affected by the flow regime. The study showed that in the autoxidation temperature regime, increasing the turbulence by reducing the tube inner diameter resulted in higher overall amounts of deposit formation. The study notes that this is likely due to the increased heat and mass transfer of precursors to the inner wall surface.

2.2.3 Pipe Materials

Venkataraman and Eser [15] used transmission electron microscopy (TEM) to examine the structure of deposits formed from thermally stressing fuels. It was found that metal sulfides play an important role in deposit formation, as the pipe materials used such as Inconel 600 and Stainless Steel 316 acted as substrates for the deposition reactions. This was confirmed when TEM showed metallic atoms from the wall in the chemical makeup of the deposits. Unfortunately, the sulfur content in jet fuel cannot be totally removed, resulting in the formation of these sulfide precursors.

2.2.4 Fuel Storage Conditions

While the prime concern for fuel thermal stability are fuel conditions during flight, another potential source of thermal degradation could come from long term storage of jet fuels. Pande and Hardy [16] thermally stressed JP-5 at much lower temperatures (20°C to 90°C) and stored the fuel with copper additives such as copper(II) ethyl acetoacetate (C_{12}H_{18}CuO_6) over a period ranging from 24 hours to 6 months. Thermal stability was evaluated in [16] by a modified JFTOT procedure. The study found that the presence
of copper additives, longer storage conditions and higher temperatures both promoted deposit formation, despite exposing the fuels to temperatures much lower than can be seen in jet engines.

2.3 Thermal Stability Diagnostic Techniques

A review of the common diagnostics used in quantifying the amount of coking deposits in various experimental rigs will be given in this section.

2.3.1 Quartz Crystal Microbalances

Zabarnick et al [17] used a Quartz Crystal Microbalance (QCM) to measure the amount of deposits formed after thermal stressing. In Zabarnick’s study, a QCM was immersed into a Jet A fuel which was heated and stirred in a sealed reactor, while the air above the fuel fed into a gas chromatograph. The temperature ranges examined were 140°C to 180°C which are in the autoxidation regime and over time, deposit would accumulate on the crystal. When the crystal is excited at its resonant frequency, it shears with maximum displacement at the crystal faces. However, if mass were to accumulate on the crystal surface (such as insoluble deposits produced from thermal degradation), this would cause a detectable decrease in the crystal’s resonant frequency. The study showed that the QCM method could determine the amount of deposit formed, which was verified by monitoring the O₂ via gas chromatography, where increased O₂ consumption indicated a greater rate of deposit formation. The study also used auger spectroscopy on the electrodes of the quartz crystal to examine the atoms and types of chemical bonds present on the electrode surface. Fuels that had a greater sulfur concentration formed more deposit on the electrode surfaces, indicating that the presence of sulfur increases the rate of deposit formation as well, which corroborates with Venkataraman and Eser [15].
2.3.2 Smoke Point

An alternative method for characterizing the thermal stability of a fuel sample was presented by Aksoy et al [18] and utilized the smoke point of a fuel. The smoke point is defined as the maximum height of a smokeless flame fed by a wick lamp of specific design. It was found that the smoke point of a fuel was inversely proportional to the concentration of high molecular mass reactive species, these species act as precursors to deposit formation. The study by Aksoy compared conventional jet fuels, and modified their aromatic contents by doping with Fischer-Tropsch isoparaffin jet fuel and other polar organic compounds such as phenol and indole. Smoke point tests were conducted in accordance with ASTM D1322.

2.4 Alternative Aviation Fuels

2.4.1 Fuel Blends

Among the many considerations for adopting alternative fuels in aviation is the manufacturing process and the type of fuel it produces. Alternative fuels developed by the Fischer-Tropsch (FT) process have been examined and it has been determined that such fuels require aromatics so that seals in the piping system will swell properly to prevent leakage. FT fuels doped with aromatics was the focus of Link et al [19]; where nitrile rubber o-rings were immersed into a batch of FT fuel spiked with various types of aromatic compounds, such as benzyl alcohol. The performance of the additive was measured by the volume and mass increase of the rubber o-rings over a period of 48 hours as per ASTM D1414 and D471. Among the aromatic compounds added, benzyl alcohol produced the best results among the compounds tested with a 17% volume increase of the o-rings. Thermal stability of the fuels with additives was also examined by heating at 350°C for up to 12 hours. Benzyl alcohol was also shown not to increase the formation of precursor
compounds that lead to insoluble deposits, despite being an aromatic compound itself.

Similarly, Amara et al [20] examined nine organic compounds ranging from linear alkanes, cyclic alkanes and aromatics and their effects on the oxidative and thermal stability when blended to alternative fuels. The fuel examined in this study was synthetic paraffinic kerosene produced from hydroprocessed esters and fatty acids (HEFA-SPK), which does not contain any aromatic compounds. Thermal stability was assessed according to ASTM D3241. It was discovered that adding small amounts of xylene (the three isomers, 1,2-dimethylbenzene, 1,3-dimethylbenzene and 1,4-dimethylbenzene were used), tetralin (also known as 1,2,3,4-tetrayhydronaphthalene, which is a double ringed compound where one of the two rings are saturated) or decalin (also known as decahy-dronaphthalene, which is a double ringed compound where both rings are saturated) increased the stability of HEFA-SPK. However, increasing the concentration of either xylene or tetralin ended up reducing the thermal stability of HEFA-SPK. In contrast, adding n-propylbenzene, n-propyl-cyclohexane or n-decane had a detrimental effect on thermal stability regardless of concentration. It appears that compounds (whether aromatic or not) with longer alkyl chains reduce the thermal stability of fuels and serve as precursors for deposit formation.

Doping with fuel additives has been shown to increase thermal stability by reducing oxygen consumption, but only up to a certain temperature. Morris et al [21] studied the effect of the following five different types of additives.

1. Butylated hydroxytoluene (also known as Ionol or 2,6-Di-tert-butyl-4-methylphenol)

2. \(N, N'\)-di-sec-butyl-p-phenylenediamine (also known as PDA).

3. FOA-3 (a commercial additive from DuPont whose active ingredient is a tertiary aliphatic amine)

4. FOA-310 (a commercial additive from DuPont made up of FOA-3 and a metal deactivator from DuPont)
5. \(N, N'-\text{disalicylidine-1,2-propanediamine}\) (a type of metal deactivator or MDA)

Ionol, PDA, FOA-3, FOA-310 were added at concentrations of 24 mg/L while MDA was added at 5.8 mg/L. These concentrations were chosen by the authors since these are the maximum allowable additive concentrations in the military specification MIL-T-5624 for JP-5 fuel. The presence of these five additives were able to reduce deposit formation at 260 °C. However, at higher temperatures, only the MDA additive reduced deposit formation while the other four additives increased deposit formation. Another interesting result of the tests conducted at temperatures higher than 260 °C was that two of the additives, FOA-3 and PDA reduced peroxide concentrations but were ineffective in reducing the buildup of insoluble deposit. This suggests that at higher temperatures, peroxides are no longer the dominant precursors in deposit formation, and that additives must be able to counteract the formation of precursors in both autoxidation and pyrolytic temperature regimes.

2.4.2 Properties of Alternative Fuels

Quintero et al [22] examined numerous types of alternative fuels for microturbine applications, which are engines typically seen in hybrid-electric vehicles. The study examined both the thermal stability of the alternative fuels and the swelling behavior of nitrile o-rings when immersed in these alternative fuels [19]. The tests on thermal stability were done on a custom-built rig with a single heated test section and showed that thermally stressed biofuels produced significantly more deposits. Carbon deposits for various types of fuels and pipe materials are shown in Figure 2.2 (a) while the behavior of o-rings immersed in alternative fuels is shown in Figure 2.2 (b).

Quintero et al also tested these fuels in a T63 engine test bench to determine the specific fuel consumptions normalized by thrust as seen in Figure 2.2 (c), and the biofuels performed just as well as the conventional fuels.
Figure 2.2: Summary of results from Quintero et al [22].


2.4.3 Alternative Fuel Production and Certification

For a fuel to be approved to be used on aircraft, it has to pass many globally recognized standards, for example ASTM D1655 [2], which specify many physical and chemical properties such as the thermal coefficients of expansion, surface tension and flammability limits. Many recent studies have shown that blends of conventional fuel with biomass derived components satisfy the requirements set by ASTM D1655, D4054, D3241 among others, with those studies implying that these blends are suitable for use in aircraft as drop-in fuels or fuel blends. However Wilson et al [23] strongly oppose this conclusion, noting that there are inherent limitations of the ASTM standards. For example, ASTM D1655 does not set a minimum requirement for the concentration of aromatic compounds in the fuel, but aromatic compounds aid in the swelling of pipe seals [19][22], and the absence of aromatics reduce the effectiveness of these seals. Wilson et al claim that alternative fuels and fuel blends must pass through many more certifications, and not to misconstrue ASTM standards that were approved using conventional feedstocks and refining pathways as the same exact standard to be applied in approving alternative fuels. It is also important to collaborate with the ASTM committee to ensure that all aspects, be it fuel properties, source feedstock, refining or storage pose minimal risk in operation.

An example of such a collaboration is the National Jet Fuels Combustion Program [24], consisting of 34 researchers in the US and Canada testing alternative fuels in cooperation with corporate partners such as Safran, Boeing and Airbus. The studies currently in progress range from measuring laminar flame speeds, creating chemical kinetic models, determining spray characteristics and measuring lean blow out to name a few.

Radich [25] examined numerous fuel varieties and production pathways currently under research and/or pending review by the ASTM committee. The report also showed the fuel production facilities operational and currently under construction, showing an optimistic outlook where increased supply could lower alternative fuel prices in the future and allow it to compete with conventional fuels.
2.4.4 Thesis Objectives

The current ASTM standards may not be appropriate when determining the thermal stability of alternative or synthetic fuels. The test apparatus designed at UTIAS with its quantitative analysis could be more suitable for alternative fuels. The main objective of this thesis is to study two types of biojet fuels on the coking rig to demonstrate the viability of the UTIAS test apparatus for quantitatively describing fuel thermal stability by verifying the results with two independent measurement methods.
Chapter 3

Experimental Apparatus

Before describing the experimental apparatus used in the current work, a brief review of the existing rigs reported in the literature is given.

3.1 Existing Test Rigs

There are two main types of jet fuel thermal stability experiments documented in the literature: continuous flow dynamic tests and pressurized static tests. Dynamic tests range from bench-top single pipe heat exchangers to full-size gas turbines, fuel tanks, or nozzle simulators [9]. Dynamic tests have shorter fuel residence times, but a continuous supply of fresh fuel (which possibly carries dissolved oxygen) is needed. Dynamic tests also require a characterization of the fuel temperature profile through the heated test section to determine the actual fuel temperature inside the test section [26]. In contrast, pressurized static tests have much longer residence times, require less fuel and the fuel is heated at a fixed temperature. There have been a variety of dynamic flow apparatuses developed and each of them have differences from one another that can make data comparison between different research groups difficult, even if the experimental designs are similar. There were also standardized apparatuses for determining the thermal stability of a sample fuel, such as the Coordinating Research Council (CRC) coker (ASTM
D-1660, now a withdrawn standard) and the subsequent JFTOT (ASTM D-3241). The CRC coker consists of a 330 mm long electrically heated aluminum pipe within a larger pipe, followed by an electrically heated housing containing a 25 µm filter [9]. The laminar fuel flow is along the outside of the aluminum pipe, and contained within a larger pipe. The fuel temperature at the end of the aluminum pipe is set to 149 °C and the filter housing is kept at 205 °C, simulating the passage size within fuel spray nozzles. The fuel volumetric flow rate is 60 mL/min for 5 hours at a system pressure of 1.0 MPa, causing the fuel residence along the aluminum pipe to be 10 s. The fuel is given a pass or fail based on the visual discoloration of the aluminum pipe by deposit buildup as long as the pressure drop across the filter did not exceed 10 kPa. The JFTOT improved upon this and replaced the CRC coker in 1973 as ASTM D-3241 [9, 27]. Instead of the 19 L of fuel required in ASTM D-1660, JFTOT uses only 0.6 L at a volumetric flow rate of 3 mL/min over 2.5 hours. Fuel still flows along the outside of an aluminum pipe, and heating was improved to achieve a 260 °C pipe wall temperature 38.7 mm from the starting end of the pipe. Other changes included an increase of system gauge pressure to 3.45 MPa with a nitrogen gas blanket, a non-heated filter housing, and a reduction in filter pore size to 17 µm. The same visual discoloration criterion is still used, but the maximum allowable pressure drop across the filter is reduced to 3.3 kPa. One of the main limitations of the JFTOT test is that it is only capable of testing a fuel up to 260 °C, and today’s research is interested in higher fuel temperatures since engines are burning ever hotter to improve thermal efficiency [26, 28]. Another limitation is that much like the earlier ASTM D-1660, it judges the fuel by discoloration which is a highly subjective method of evaluation, and it is potentially problematic when different people see colors differently or in different lighting conditions. The most common apparatus used in universities or small laboratories consists of a single, heated pipe with fuel flowing through it to simulate a heat exchanger. There have been many examples of such test rigs such as in NASA Glenn Research Center’s Heated Pipe Facility [29], the USAF’s Phoenix Rig, the
Australian Defence Science and Technology Organisation (DSTO) Thermal Stability Rig [30, 31], and others by United Technologies Research Center (UTRC) [32], Rocketdyne [33], and PSU Energy Institute’s flow reactor [34]. Different methods have been used to heat the fuel pipe. Resistively heated isothermal copper blocks surrounded and conductively heat the fuel pipes in UTRC rig and the Phoenix Rig [6, 10, 35, 36]. Australia’s Airframes and Engines Division of DSTO used a fluidized sand bath to conductively heat their submerged fuel pipe [30, 31]. PSU’s flow reactor used an electrically heated pipe furnace that provided radiative heating of the fuel pipe that was in the centre of the furnace [28, 37].

More recently, there has also been an interest in changing the test section geometry to generate eddies and determine their effects on deposit formation. Alborzi et al used a test section with a sudden expansion and contraction as shown in Figure 3.1 and compared it to a tube with a circular cross section. Both the standard cylindrical tube and the expansion/contraction tube were heated with radio frequency heaters under similar heating power input conditions. A computational fluid dynamics (CFD) simulation was used to predict the flow streamlines in the vicinity of the expansion and contraction regions. Thermocouples that were used to monitor the test section temperatures detected a sharp rise in temperature near the contraction portion of the test section, with peak temperatures about 50°C higher than the test section without expansion/contraction. Alborzi notes that this contraction location created a recirculation zone where flow velocities stagnated and was thus heated for longer periods of time. This particular area was found to have the largest concentration of deposits, as expected due to the higher temperatures. The amount of deposit accumulation from thermal stressing was examined by direct measurement via a scanning electron microscope and by the carbon burnoff method.

Another study was conducted Pei et al [39] using a curved test section whose layout is depicted in Figure 3.2. CFD was also used to predict the flow streamlines in the curved
Figure 3.1: Standard cylindrical and expansion/contraction test sections used in Alborzi. Thermocouples were positioned at A to E and dimensions are in mm [38].

section. The amount of deposit formation from thermal stressing was calculated via the weight change of the test section before and after thermal stressing. There was a sharp temperature drop of about 100 K as the fuel flowed around the curve. Thus, the point immediately prior to the curve contained the highest concentration of deposits as it was the hottest portion of the test section. Likewise, there was a temperature concentration on the inner side of the curve.

Figure 3.2: S-Bend test section layout used in Pei et al [39].
This thesis will characterize deposit formation by means of a pressure drop measurement across a heated test section, an $^1$H NMR of the thermally stressed fuel and a carbon burnoff on the test section.

3.2 Previous UTIAS Test Setup

Previous masters studies done by three UTIAS students have focused on the design and operation of the testing rig used to examine the coking of aviation fuel. The apparatus designed by Wong [40] thermally stressed the fuels inside a 91 cm (36 in) long and 1.8 mm (0.069 in.) inner diameter test section made of stainless steel 304 tubing. The fuels were pumped at a constant volumetric flow rate through the test section using a syringe pump (Teledyne ISCO model 500D) while the flow back pressure was regulated by Swagelok regulator. A 0.5 $\mu$m filter was placed downstream of the heated section to avoid clogging. After a test run, the stainless steel tube was flushed with nitrogen gas to clear it of liquid fuel left behind. Cleaning the tube was done in preparation for heating with Grade 2.6 oxygen gas to a temperature of 900°C to oxidize any deposits. The resulting amount of CO$_2$ was collected and analyzed via an infrared analyzer to determine how much CO$_2$ was present. From the CO$_2$ levels, the carbon deposit that was present inside the stainless steel test section after the experimental run could be calculated. The study by Wong found that with increasing temperatures, there was an increase of deposit formation for a given fuel.

3.3 Current Test Setup

The rationale for the design of the current coking setup at UTIAS was summarized by Liang [41] and consists of two sections, a preheating section and a test section. The general layout of the apparatus is shown in Figure 3.4. This apparatus differs from the setup in ASTM D3241-15 [27] as the ASTM standard only heats up the fuel in one stage.
which is not representative of on-board conditions. The standard determines fuel thermal
stability via a color comparison test of the thermally stressed fuel (shown in Figure 3.3)
and a visual examination of the heated section. Such metrics are prone to subjectivity
and Wilson et al. [23] notes that if an alternative fuel passed the ASTM standard for
conventional fuels, it is no guarantee that it will be compatible with aircraft systems. It
is possible that thermally stressing alternative fuels will produce different colors and will
render ASTM3241 moot for assessing thermal stability.

This new setup was designed to simulate better the conditions that fuel would expe-
rience prior to entering the combustion chamber of the jet engine, where the fuel absorbs
waste heat from the avionics, electronics and the surrounding air. As such, this test
setup has fuel residence times in the preheating section of about 50 s, similar to what
is present on aircraft [9]. Afterwards, the fuel passes through the test section in a much
shorter time (on the order of 0.05 s), which simulates the timescale of the fuel passing
through the spray nozzle.

![Figure 3.3: ASTM D3241 color comparison metric.](image)

The preheating section consists of a stainless steel pipe with a length of 1 m, an
inner diameter of 4.57 mm and an outer diameter of 6.35 mm. This 1 m long section
is immersed in a heated oil bath to simulate the fuel acting as a coolant; onboard an
aircraft, the fuel would be absorbing extra heat from the engine, electronics as well as
the surrounding air.
The test section is made of stainless steel with a length of 5.08 cm, an outer diameter of 3.18 mm and an inner diameter of 0.686 mm. The test section simulates the conditions inside the fuel injection nozzles of an engine combustor.

Also, a quicker method was devised to determine the amount of deposit within the pipe and uses pressure transducers which measured the pressure drop across the test section. To monitor and measure deposit formation within the test section, two static pressure taps were used, one upstream and another downstream of the test section to determine the pressure drop. The pressure transducer (Omega, MMDWU001V5P3A0T1A1) has a range of 0-6.89 kPa (0-1 psi), accuracy of ±0.08% of its full dynamic range, which is equivalent to ±5.5 × 10⁻³ kPa (±0.8 × 10⁻³ psi). The differential pressures as a function of time were recorded by a multifunction data acquisition device (National Instruments, USB-6210). Temperature and pressure differential data were taken at 30 second intervals.

Larger pressure drops indicated a greater reduction of the flow-through area, implying a greater buildup of insoluble deposit within, by the Hagen-Pouiseille Law in Equation 3.1.

\[ \Delta P = \frac{8\mu LQ}{\pi r^4} \]  (3.1)

Where \( \Delta P \) is the difference in pressure, \( \mu \) is the dynamic viscosity, \( L \) is the length of the pipe, \( Q \) is the volumetric flow rate and \( r \) is the pipe radius. Using Equation 3.1, it is possible to calculate the overall reduction in radius from and thickness of the deposit layer by taking the initial and final values of the pressure drop (denoted by \( \Delta P_i \) and \( \Delta P_f \)) to yield Equation 3.2.

\[ \frac{\Delta P_i}{\Delta P_f} = \frac{R_i^4}{R_f^4} \]  (3.2)

This method is a more objective metric compared to the color test of ASTM D3241. However, this relation is only valid when the flow is laminar since its derivation from
the Navier-Stokes equations requires that the radial and swirl components of the velocity be zero to give a parabolic velocity profile (such a profile will not occur in a turbulent flow). It also assumes that the deposit uniformly accumulates along the length of the test section. Later carbon burnoff experiments will show that this is a slightly inaccurate assumption.

The fuel to this test apparatus is supplied by a syringe pump (Teledyne, ISCO model 500D) whose flow pressure was controlled by a back pressure regulator downstream of the flow.

The fuel from the supply tank is pumped at a constant volumetric flowrate. The test section is enclosed in a solid brass casing 38.1 mm in diameter and 50.8 mm long consisting of two halves. The brass block is heated by a 300 W band heater and insulating foam surrounds the brass blocks to reduce heat loss to the environment and allowing for better temperature control. The temperature on the outer wall of the pipe is monitored by six Type K thermocouples with an accuracy of $\pm 2.2^\circ C$ labeled $T_{ts,1}$ through to $T_{ts,4}$, $T_{in}$ and $T_{out}$ in Figure 3.4. Temperature data are recorded by a thermocouple datalogger.
Chapter 3. Experimental Apparatus

(Omega, OM-CP-OCTTEMP2000) and fed to a PC. $T_{\text{in}}$ records the pipe outer wall temperature upstream of the test section and immediately after the oil bath section while $T_{\text{out}}$ monitors the temperature downstream of the test section. $T_{ts,1}$ through to $T_{ts,4}$ are four thermocouples spaced 1.3 cm apart and are used to monitor the outer wall temperature of the test section to ensure that the test section temperature is as uniform as possible. In particular, $T_{ts,1}$ is used by temperature controller (Omega, CN7523) to control the band heater and set the test section temperature. It should be noted that the outer wall temperature of the test section is higher than the inner wall temperature. A simulation code developed by Liu [7] was used to determine the outer wall temperature set by the controller to get the desired inner wall temperature that the fuel is exposed to.

After passing through the test section, the fuel is still at temperatures where it is expected to coke, hence a 0.5 $\mu$m metal filter was installed to collect deposits that do not accumulate at the test section wall. This was done to minimize clogging within the remaining sections of the test apparatus. After passing through the metal filter, cold water at 10°C is pumped around a cooling coil to ensure that the waste fuel temperature is lower than the autoignition temperature as the fuel flows into the waste tank. The back pressure regulator maintained the fuel flow pressure to about 689 kPa (100 psi) for all tests. The optional N$_2$ could be used for clearing and cleaning the pipes between tests, however it was used only when tests would not take place for extended periods of time as this could add air bubbles into the system.

3.4 Diagnostic Tools

3.4.1 Nuclear Magnetic Resonance

The UTIAS test rig documented in the thesis of Liang [41] was dependent on pressure drop measurements across the test section to estimate the amount of deposits. In the
current study, a carbon burnoff apparatus is used in addition to pressure drop measure-
ments. Further, the fuels were analyzed by proton nuclear magnetic resonance ($^1$H NMR)
before and after thermal stressing to see, if any, changes in the fuel molecular structure
and composition. Brief descriptions of $^1$H NMR and carbon burnoff techniques are given
below.

Charged particles generate a magnetic field when they move by translation or rotation
relative to a reference point. Certain species of nuclei have a net spin, which in of itself
generates its own magnetic field, allowing such nuclei to be split into discrete energy
states. When the nucleus’s own field aligns with an applied external field, it is at a
lower energy state while opposing the external applied field would result in a higher
energy state. In order for nuclei to have a net spin however, the nuclei need to have
an odd number of protons or neutrons, which is why NMR spectroscopy can detect the
bonds involving hydrogen atoms. Both stable isotopes of hydrogen $^1$H and $^2$H satisfy the
odd-number requirement, making it detectable in NMR spectroscopy. The vast majority
(98%) of carbon atoms however, are carbon-12 $^{12}$C which have an even number of protons
and neutrons, resulting in zero net magnetic moment for these nuclei and thus do register
in NMR spectroscopy. In this thesis, only Hydrogen NMR ($^1$H NMR) was used.

When hydrogen atoms are subjected to an external magnetic field, their nuclei re-emit
electromagnetic (EM) waves at a specific frequency. This EM wave interacts with the
magnetic fields generated by the electrons in the bonds between atoms, resulting in a
specific frequency. This frequency can be compared to a standard to produce a chemical
shift $\delta$ away from the standard. The standard in $^1$H NMR is commonly tetramethyl
silane Si(CH$_3$)$_4$, whose $^1$H NMR frequency of 300 MHz is used to normalize all other the
other frequencies via Equation 3.3, where $\nu_r$ is the frequency of Si(CH$_3$)$_4$ and $\nu_s$ is the
specific frequency of a carbon-hydrogen bond.

$$\delta = \frac{\nu_s - \nu_r}{\nu_r}$$ (3.3)
Table 3.1: Hydrogen bond type classifications [42].

<table>
<thead>
<tr>
<th>Hydrogen Type</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>$H_A$</td>
<td>Hydrogens on aromatic rings (typically benzene and naphthalene-based compounds)</td>
</tr>
<tr>
<td>$H_o$</td>
<td>Hydrogens on olefinic or alkene/alkyne carbon atoms</td>
</tr>
<tr>
<td>$H_\alpha$</td>
<td>Hydrogens on $\alpha$-position to aromatic rings. $\alpha$ position indicates adjacent to an aromatic ring.</td>
</tr>
<tr>
<td>$H_{C1}$</td>
<td>Paraffinic and naphthenic hydrogens attached to carbon atoms that have no other hydrogens attached to it (typically a CH group), or hydrogens attached to carbon atoms at the $\beta$-positions to aromatic rings. $\beta$ position indicates one step removed from the $\alpha$ position. Hydrogens on cycloalkanes also belong to the $H_{C1}$ category</td>
</tr>
<tr>
<td>$H_{C2}$</td>
<td>Paraffinic hydrogens attached to carbon atoms that have one other hydrogen bonded to it (typically a $CH_2$ group), or hydrogens attached to $CH_2$ groups at the $\gamma$ or further position to aromatic rings. $\gamma$ position indicates two steps removed from the $\alpha$ position. Hydrogens on cycloalkanes in $\beta$-positions away from any functional group also belong to this category</td>
</tr>
<tr>
<td>$H_D$</td>
<td>Paraffinic hydrogens attached to carbon atoms that have two hydrogens attached to it (typically $CH_3$ group), or hydrogens attached to $CH_3$ groups at the $\gamma$- or further position to aromatic rings. Hydrogens in cycloalkanes in $\gamma$- or further position away from any functional group also belong to this category</td>
</tr>
</tbody>
</table>

This then allows determination of the type of bond that the hydrogen is participating in, as well as the relative percentage of the number of such bonds in the sample.

**Hydrogen Bond Types**

Using $^1H$ NMR and Si(CH$_3$)$_4$ as the reference standard allows determination of the type of bond that the hydrogen is participating in, as well as the relative percentage of the number of such bonds in the sample. Table 3.1 describes the various types of hydrogen classifications [42]. Various combinations of hydrogen bond types can also be seen in Figure 3.5.

Hydrogen bond scores are calculated as a value out of 100 for each of the six hydrogen bond types, either via $^1H$ NMR or calculating via known chemical structure. As an example, let us take n-octane C$_8$H$_{18}$ in Figure 3.6, which has 18 C-H bonds. It has
Table 3.2: List of ppm ranges used in $^1$H NMR to classify hydrogen bond types [42].

<table>
<thead>
<tr>
<th>Hydrogen bond type</th>
<th>Range of ppm</th>
</tr>
</thead>
<tbody>
<tr>
<td>$H_A$</td>
<td>9.2 - 6.2</td>
</tr>
<tr>
<td>$H_o$</td>
<td>6.2 - 4.0</td>
</tr>
<tr>
<td>$H_\alpha$</td>
<td>4.0 - 2.1</td>
</tr>
<tr>
<td>$H_{C1}$</td>
<td>2.1 - 1.4</td>
</tr>
<tr>
<td>$H_{C2}$</td>
<td>1.4 - 1.05</td>
</tr>
<tr>
<td>$H_D$</td>
<td>1.05 - 0.5</td>
</tr>
</tbody>
</table>

6 hydrogens on $H_D$-type bonds which are seen as CH$_3$ groups on either end and 12 hydrogens on $H_{C2}$-type bonds, giving a $H_D$ score of 6/18*100 = 33.33 and a $H_{C2}$ score of 12/18*100 = 66.66. This means that the Hydrogen bond types of $H_A$, $H_o$, $H_\alpha$ and $H_{C1}$ all have a score of 0 for n-octane, as expected since n-octane is not aromatic or olefinic.

The parts per million (ppm) ranges for each of the hydrogen types were taken from a study by Glavinčevski et al [42], shown in Table 3.2. It is useful to note that these ppm ranges were found experimentally, as other studies such as by Sarathy et al [43] have slightly different ppm ranges and other compound classifications. Since the exact composition and chemical structure of the fuels used in this study are unknown, $^1$H NMR was used to determine the various percentages of hydrogen types.

### 3.4.2 Carbon Burnoff

One of the two main methods for determining deposit buildup in the test section (the other is via a pressure differential) is using a carbon burnoff apparatus. The carbon burnoff apparatus in this study (Eltra SC-800) has a range of 5-50,000 µg of carbon and accuracy of ±1%. The carbon burnoff test involves passing heated O$_2$ at 700°C through a test section inside a sealed chamber. The hot O$_2$ will oxidize any solid carbonaceous residue inside the tube into CO$_2$ which is then collected and detected in the same apparatus via an infrared cell. The motivation for using the carbon burnoff apparatus is
the supposed improved accuracy as well as recommendations from previous studies done at UTIAS [40], since the pressure drop measurements assume a uniform deposit buildup through the length of the test section; an assumption that will be shown to not be entirely accurate.

### 3.5 Fuels used in this study

There were four types of fuels examined in this thesis, and these were acquired from the National Research Council in Ottawa, Canada via the research collaboration of BioFuel Net. The four fuels, their compositions and their respective percentage of aromatic compounds by mass are shown in Table 3.3. The percentage of aromatic compounds was found by the Fluorescent Indicator Adsorption Test (ASTM D1319 [44]) conducted by the National Research Council. The fuel codes are nomenclature used internally within BioFuel Net, which is a consortium of researchers across Canada investigating the production pathways, physical and chemical properties, and the use of biofuels throughout all sectors, not just in aviation.

The four fuels JF1, JF2, BJ1 and BJ2 were chosen for their variety in aromatic content, being conventional or alternative, and availability from the National Research Council in large enough quantities for the pressure drop experiment. JF1 is conventional Jet A used in commercial aircraft worldwide. JF2 is Jet Propellant 5 (JP-5) used for
aircraft on board aircraft carriers, hence why JP-5 is also designated as Aviation Carrier Turbine Fuel (AVCAT) and carries a North Atlantic Treaty Organization (NATO) code of F-44. JP-5 is a kerosene based fuel similar to Jet A, compatible with non-carrier based aircraft but has a slightly higher flash point to reduce the risk of accidental fires on aircraft carriers. BJ1 and BJ2 are alternative aviation fuels whose properties are being researched by BioFuel Net for the purpose of blending them with regular jet fuel as future drop-in aviation fuels.

In order to compare and contrast the four fuels, a test and metric for determining the chemical composition is required. Unfortunately, it is exceedingly difficult and impractical to identify every single unique compound present and their respective proportions in each fuel sample. Each batch of fuel could also be different depending on the feedstock and/or refining method/location. To determine the percentage of aromatic compounds in JF1, JF2, BJ1 and BJ2, the National Research Council used the Fluoresce Indicator Adsorption Test (ASTM D1319 [44]). This test involves passing the fuel into an adsorption column containing silica gel and fluorescent indicator dyed gel under pressure. Once the fuel is in the column, it is exposed to ultraviolet (UV) light, and the individual components of the fuel interact with the gel and UV light to produce a color pattern similar to Figure 3.7.

The volume percentages in each of aromatics, olefins and saturates (paraffins) are found by taking the particular height of each zone divided by the total length of all zones. The ASTM D1319 standard state a repeatability of about 1.4 % by volume and reproducibility of about 3.0 % by volume for aromatic compounds. Unfortunately, ASTM D1319 can only differentiate between three types of organic compounds. Its accuracy could also be affected by the subjective nature of color determination which demarcate the start and end of each zone. Finally, ASTM D1319 also notes that some batches of gel can cause the color boundary between olefins and aromatics to fade, and that the reason behind this is unknown but will certainly affect accuracy and repeatability [44],
since the measured height will be different depending on the degree of fading.

In order to accurately describe the fuel composition, each fuel was thus examined via $^1$H NMR, which yielded a consistent metric on the percentage composition of various types of hydrocarbons.
Figure 3.5: Positions and hydrogen types on arbitrary compounds.

Figure 3.6: \( n \)-octane
Figure 3.7: Color boundaries used in ASTM D1319 to identify organic compounds in mixtures. The column shows the aromatic, olefin and saturate (paraffin) zones [44].
Chapter 4

Experimental Procedure

4.1 Preparations for the Pressure Drop Test

4.1.1 Nuclear Magnetic Resonance of the Fuels

Fuel chemical composition was evaluated by $^1$H NMR spectroscopy, which is based on a phenomenon that is observed when the nuclei of some species of atoms are subjected to a magnetic field as described in Section 3.4.1. $^1$H NMR spectra for each of the four fuels are in Figures 4.1 to 4.4. Each of the four spectra have notations for the relevant peaks and a table of hydrogen bond types, as described in Section 3.4.1. The peak at 0 ppm is the signal from the reference compound tetramethyl silane (Si(CH$_3$)$_4$) since the shift from the reference compound away from itself will be 0. The peak at 7.26 ppm is the signal from the chloroform (CDCl$_3$) solvent (D is the deuterium isotope of hydrogen). Since the solvent peak occurs in the middle of the aromatic hydrogen bonds ($H_A$) range, the integration was done in two sections to avoid this peak. This splitting is also shown in the tables of hydrogen bond types within Figures 4.1 to 4.4.

As expected for the BJ1 fuel in Figure 4.3, there are no peaks in the $H_A$, $H_\alpha$ and $H_\alpha$ ranges as it consists purely of isoparaffins. The BJ2 fuel on the other hand in Figure 4.4 has the highest percentage of $H_A$ type hydrogen bonds due to the large presence of
Figure 4.1: $^1$H NMR of JF1 fuel.

aromatic compounds. JF1 and JF2 which are conventional fuels showed similar percentages of aromatic hydrogens, which correspond to Table 3.3, which lists the percentage of aromatic compounds measured via ASTM D1319 [44].

These $^1$H NMR spectra were obtained at the University of Toronto Lash Miller Chemical Laboratories with chloroform (CDCl$_3$) as a solvent and tetramethylsilane (Si(CH$_3$)$_4$) as a reference signal which is set at 0 ppm. $^1$H NMR data analysis was done using MestReNova software. Upon receiving the raw data, the following steps were done to ensure that the resulting $^1$H NMR data analysis was consistent.

$^1$H NMR Pre-processing Step 1: Tetramethylsilane Referencing

The spectra was first examined for the tetramethylsilane (Si(CH$_3$)$_4$) peak, which by $^1$H NMR convention is set to 0 ppm. On occasion, the raw spectrum would have a
small horizontal shift to the left. Unprocessed data would sometimes show that the Si(CH₃)₄ value was slightly above zero. Adjusting the spectra for this reference was critical because the integration limits for determining the hydrogen bond types in Table 3.2 depend on accurate ppm markings. The MestReNova software was used to shift the spectra horizontally when needed so that the TMS is centered on 0 ppm. If the TMS signal was too low or absent (at times this is due to negligence), the solvent used in ¹H NMR, chloroform (CDCl₃) could also be used as a reference.

Note that CDCl₃ is a type of chloroform used in ¹H NMR that has deuterium (a hydrogen isotope with a single neutron) instead of regular hydrogen which would have been CHCl₃. The standardized value for CDCl₃ on an ¹H NMR spectrum is 7.26 ppm. A raw ¹H NMR spectrum with a tetramethylsilane reference prior and after to correction can be seen in Figure 4.5.

Figure 4.2: ¹H NMR of JF2 fuel.


### Chapter 4. Experimental Procedure

**Figure 4.3:** $^1$H NMR of BJ1 fuel.

$^1$H NMR Pre-processing Step 2: Phase Corrections

Aside from adjusting the reference value of Si(CH$_3$)$_4$ and/or CDCl$_3$, the $^1$H NMR spectrum requires phase corrections. An $^1$H NMR spectrum shows positive valued peaks where the hydrogen bond types are present. If the hydrogen bond types are not present in the compound for analysis, the spectrum should show a value of zero, but a raw $^1$H NMR spectrum may register negative values. Thus, a phase correction is applied to shift the spectrum upwards until no negative values are present. This correction has a minimal impact on the integration process as the relative intensity of the peaks are on the order of 500-600 or higher while the negative intensity values go only as far as $-1$, a difference of two or more orders of magnitude. A raw $^1$H NMR spectrum prior to and after phase corrections can be seen in Figure 4.6.
Chapter 4. Experimental Procedure

Figure 4.4: $^1$H NMR of BJ2 fuel.

$^1$H NMR Pre-processing Step 3: Baseline Corrections

After performing phase corrections, the final step of pre-processing prior to integrations, baseline correction was performed on the $^1$H NMR spectrum. As discussed earlier, the phase corrections ensure that no negative values exist. However, this does not ensure that there is a flat, horizontal zero line consistent with a processed $^1$H NMR spectrum. Without the baseline correction, the integral values would have a systematic increase. For this thesis, a Whittaker Smoother algorithm (the standard smoothing algorithm of the MestReNova software) was applied to a phase-corrected data set to produce the final processed spectrum show in Figure 4.7.

Finally, after the three pre-processing steps are completed, integration with the bounds from Table 3.2 were applied. The sum of all integrated regions were normalized to 100 so as to give a percentage composition of each hydrogen bond type. Performing
Chapter 4. Experimental Procedure

Figure 4.5: $^1$H NMR Spectrum of JF1 before and after referencing.

Figure 4.6: $^1$H NMR Spectrum of JF1 before and after phase corrections.

Figure 4.7: $^1$H NMR Spectrum of JF1 before and after baseline corrections.
the pre-processing steps are meant to ensure consistency since the phase shifts differ between each $^1$H NMR test run. Failing to perform these steps resulted in minor ($<0.05\%$) value changes in the percentage compositions of the hydrogen bond types.

4.1.2 Cleaning Equipment

As the four types of fuels used in this thesis are quite chemically different (see Table 3.3), it was important to avoid cross-contamination in the testing equipment. Hence, prior to pouring a new type of fuel into the supply tank (as described in Figure 3.4), it is first cleaned, wiped and rinsed repeatedly with 99% ethanol and left to dry for at least one day, ensuring that residual fuel from the previous test dried out. Since ethanol contains both polar and nonpolar regions in its molecular structure, it was capable of dissolving any of the four types of fuels in use. The addition of ethanol during cleaning also enhanced the evaporation rate of the ethanol-fuel mixture due to the low boiling point of ethanol. In parallel, the syringe pump was completely drained of the previous fuel. Unfortunately, it was impractical to remove the previous batch of fuel left inside the preheating tubing without inadvertently introducing air bubbles or other impurities. Thus, the new type of fuel is run for at least 30 minutes before heating the test section in order to cleanse the entire apparatus of the old batch of fuel. Each test run also required the manufacture of a new test section, described in Section 4.1.3 and a new filter.

4.1.3 Test Section

Test sections were manufactured prior to full scale runs. These test sections are made of stainless steel 304 tubes procured from McMaster-Carr with an outer diameter of 3.18 mm and an inner diameter of 0.686 mm. The tubes were cut with a hacksaw blade that has 24 teeth per 25 mm, also known as 24 teeth per inch (TPI). The tubes were cut into lengths of 82.55 mm as measured by rulers. The tubes were then filed and deburred with a cobalt steel countersink, single flute, 90° angle with a 1/8 inch (3.175 mm) body
diameter. The newly cut tubes were then purged with compressed air to ensure that no metal filings were inside a tube as a result of the deburring.

After the tubes were cleaned, the following Swagelok ferrules and fittings, SS-200-SET and SS-202-1 were installed for the purpose of screwing the tubes into the test apparatus. The brass blocks were then placed along with the band heater to wrap around the test section. Finally, four thermocouples were attached to the brass blocks to monitor the temperature of the test section throughout the steady state run as shown in Figure 4.8.

A new Swagelok 0.5 micron metal filter SS-4F-K4-05 was also installed prior to every test. The filter is locked into its holder using a 1" torque wrench and 62.5 Nm of torque was applied. This metal filter was positioned after the heated test section to collect any deposits formed from thermal decomposition and prevent clogging of the test apparatus. After the complete installation of the new test section, fuel filter and the refilling of the fuel supply tanks, preliminary trial runs were performed at room temperature, flow pressure of 689 kPa and a flow rate 20.408 mL/min to ensure that there were no leaks throughout the entire apparatus. When no leaks were detected, insulating foam was wrapped on the sides of the brass blocks to minimize heat loss. The prepared test section shown in Figure 4.9 is now ready for a full experimental run.

![Figure 4.8: Diagram of the test section.](image-url)
4.1.4 Determining Flow Regime, Fuel Viscosity and Flow Rate

Flow Regime

For four of the five experimental runs, the fuel flowed in a laminar flow regime. To determine the flow regime, the Reynolds number had to be evaluated via Equation 4.1, where $\rho$ is the density of the fluid, $v$ is the characteristic velocity, $l$ is the length scale and $\mu$ is the dynamic viscosity of the fluid. For pipe flow, the characteristic velocity is the fluid velocity and the characteristic length is the pipe diameter. The Reynolds number for laminar flow in a pipe is approximately $< 2000$ [45], though other sources state the threshold is $Re = 2300$ [46]. Reynolds numbers between 2000 and 4000 constitute the transition regime while those above 4000 are in the turbulent regime.

$$Re = \frac{\rho v l}{\mu}$$ (4.1)

Rewriting Equation 4.1 gives Equation 4.2, where $\dot{Q}$ is the fuel volumetric flow rate, $d$ is the pipe diameter and $\nu$ is the kinematic viscosity (kinematic viscosity is also expressed as $\mu/\rho$).

$$Re = \frac{4\dot{Q}}{\pi d \nu}$$ (4.2)
Fuel Viscosity

The kinematic viscosity of a liquid is a function of its temperature only and is independent of pressure. The kinematic viscosity of Jet A (JF1 fuel) has been measured for temperatures between $-60^\circ C$ to $-20^\circ C$ (213 K to 253K), which is unfortunately too cold for the conditions in these experiments. The ASTM standard for the chemical and physical properties of aviation fuel (ASTM D1655 [2]) require an upper limit of 8.0 mm$^2$/s at $-40^\circ C$ for kinematic viscosity but has no lower limit for kinematic viscosity, since lower viscosities mean that the fuel flows more easily, leading to more favorable operation.

Measurements for the viscosities of pure compounds are available. A study by Kim et al [47] with the aim of modeling jet fuel surrogates from pure hydrocarbons showed that the resulting viscosity from a mixture is on the same order of magnitude as their individual components. The fuel viscosities also decreased with increasing temperature, modeled using the Grunberg-Nissan relation [48] shown in Equation 4.3 for the viscosity of a mixture. $\eta_m$ is the mixture viscosity, $\eta_1$ and $\eta_2$ are the individual component viscosities, $N_1$ and $N_2$ are the component mole fractions and $d$ is a characteristic constant of the system.

$$\log \eta_m = N_1 \log \eta_1 + N_2 \log \eta_2 + N_1 N_2 d \quad (4.3)$$

However, empirical measurements of the kinematic viscosity of Jet A were unavailable at temperature ranges near $340^\circ C$ (613 K), as heating jet fuel above $210^\circ C$ in order to measure fuel viscosity using a viscosimeter would likely result in autoignition [49]. In order to estimate what the kinematic viscosity would be at the temperature ranges of $340^\circ C$, an approximation of dynamic viscosity known as the Lewis-Squires approximation [50] was used. The Lewis-Squires relation is shown in Equation 4.4, and is an empirical approximation for the dynamic viscosity where $\mu_T$ is the dynamic viscosity at temperature
\( T, \mu_{T_1} \) is the dynamic viscosity at another temperature \( T_1 \) and the temperatures are either in Celsius or Kelvin.

\[
\mu_T^{0.2661} = \mu_{T_1}^{0.2661} + \frac{T - T_1}{233} \tag{4.4}
\]

Using the measured value for the viscosity of Jet A at \(-20^\circ C \) [49], the resultant dynamic viscosity at the test temperature of \( 346^\circ C \) is expected to be \( 1.015 \) mPa-s. The closest available data to compare with the chemical composition of Jet A and the elevated temperature condition was from kerosene [51]. The measured dynamic viscosity of kerosene at \( 300^\circ C \) \( 1.59 \) mPa-s, which is on the same order of magnitude as the Lewis-Squires approximation. Other approximations or correlations were also available, and they likewise predict that dynamic viscosity decays with increasing temperature. A comparison was performed by Liang [41] and shown in Figure 4.10 to verify the predictions of the Lewis-Squires approximation using available data from two handbooks [51, 52]. The Lewis-Squires relation appears to be inaccurate at lower temperatures, but the approximation converges to the higher temperature viscosity values in the two handbooks, showing that it is accurate for the purposes of this thesis.

The measured viscosity values of JF2, BJ1 and BJ2 were similarly unavailable at \( 346^\circ C \). However their constituent components’ viscosity values at \( 346^\circ C \) were likewise predicted using of the the Lewis-Squires relation in Equation 4.4 from known viscosity values at lower temperatures [51]. The predicted viscosity values are shown in Table 4.1, and they show that their kinematic viscosities are all in the same order of magnitude.

The kinematic viscosity values were used only in determining the flow Reynolds number so that the flow rate could be properly set. From the Hagen-Pouiseuille Law in Equation 3.1, the reduction in pipe diameter is a function of the pipe length, volumetric flow rate, pressure drop and dynamic viscosity. Since the test conditions keep the volumetric flow rate, temperature (by extension viscosity) and length constant, the pipe diameter is only a function of the pressure drop.
Flow Rate

Four of the five test runs were conducted in the laminar flow regime and the last test run was done in the transition regime. Experiments performed in the laminar regime used a flowrate of 20.408 mL/min ($3.4013 \times 10^{-4}$ L/s), resulting in a Reynolds number of approximately 1670.

An exploratory case was done in the transition regime using reserves of Jet A fuel from a previous study unrelated to BioFuel Net. Tests in the transition regime would need higher flowrates than those in the laminar regime as shown in Equation 4.2, thus a larger supply of fuel would be needed. However, the supply of JF1, JF2, BJ1 and BJ2 were limited. The transition regime case was performed to examine the effect of flow regime on the propensity of fuel coking since it is likely that commercial aircraft engines pump fuel at flowrates higher than the laminar regime.

The experiment performed at the transition regime used a higher flowrate of 31 mL/min ($5.1667 \times 10^{-4}$ L/s) to give a Reynolds number of approximately 2540. Un-
**Table 4.1:** Predicted dynamic and kinematic viscosities of JF1, JF2, BJ1 and BJ2 at the test temperature of 346 °C.

<table>
<thead>
<tr>
<th>Fuel code</th>
<th>Fuel component</th>
<th>Measured dynamic viscosity (mPa-s)</th>
<th>Predicted dynamic viscosity at 346 °C (mPa-s)</th>
<th>Predicted kinematic viscosity at 346 °C (mm²/s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>JF1</td>
<td>Jet A</td>
<td>3.3 (@−20 °C) [49]</td>
<td>1.015</td>
<td>0.389</td>
</tr>
<tr>
<td>JF2</td>
<td>JP-5</td>
<td>3.6 (@−20 °C) [54]</td>
<td>1.16</td>
<td>0.42</td>
</tr>
<tr>
<td>BJ1</td>
<td>C-10 iso-paraffin</td>
<td>0.907 (@20 °C) [51]</td>
<td>0.434</td>
<td>0.570</td>
</tr>
<tr>
<td>BJ2</td>
<td>Trimethyl benzene</td>
<td>1.112 (@20 °C) [51]</td>
<td>0.512</td>
<td>0.591</td>
</tr>
</tbody>
</table>

fortunately, the pressure drop data used to measure deposit thickness was no longer valid since the fuel velocity profile is not parabolic in the transition regime. However, the amount of accumulated deposit could still be measured using the carbon burnoff apparatus.

### 4.1.5 Determining Test Section Temperature Setting

In order to determine the desired temperatures for the test section, two things need to be considered.

The first consideration is the physical limitations of the liquid fuel. In order to produce the most pronounced results of thermal decomposition and to minimize the impact of measurement errors, higher temperatures are preferred. However, if the temperatures are too high, the fuel will start to boil inside the apparatus, which causes large (∼5 psi or 34.47 kPa) pressure fluctuations and compromise the readings of the pressure transducer. It is possible to increase the flow pressure to prevent the fuel from boiling, however, high pressures would damage the transducers. Similarly, no material data sheet exists that describe the behavior of the four fuels’ boiling points as a function of pressure, especially at such a high flow pressure of 100 psi (689 kPa). Hence, it was decided that the test temperature would be the highest temperature that Jet A (JF1 fuel) could tolerate at 100 psi (689 kPa) without boiling. The other three fuels (JF2, BJ1 and BJ2) were described
to be physically similar to the JF1 fuel, however, no research had yet tried to heat the other three fuels at a flow pressure of 100 psi (689 kPa), and there wasn’t an abundant supply to test out the boiling temperature at high pressures in addition to the coking study.

The second consideration is the temperature controller setting for the test section. The thermocouples that feed data into the controller and data logger are only in contact with the outer wall of the test section, which means that the controller only sets the outer wall temperature, and there is a temperature drop from the outer wall towards the inner wall. In order to estimate the temperature of the inner wall from the outer wall setting, an in-house simulation code was developed as part of the masters thesis of Liu [7]. The simulation predicts the inner wall and fuel temperature from the set temperature of the band heater, taking into account the thermal conductivity of the test section wall and the fuel flowrate.

It was found through benchmark trial tests that a fuel temperature of 346 °C was the highest tolerable temperature to prevent Jet A from boiling at a fuel flow rate of 20.408 mL/min (0.3401 mL/s) and 100 psi (689 kPa). This temperature is in between the two regimes, where autoxidation and pyrolytic reactions both come into play. Unfortunately, the BJ2 fuel required a slightly higher pressure of 110 psi (758 kPa) to keep from boiling. Using the code developed by Liu [7], this corresponded to a set temperature of 365 °C at the temperature controller to correspond to a test section temperature of 346 °C. Lower temperatures could also have been used, however this would result in less thermal decomposition since deposit formation is a function of fuel temperature [13][17]. A sample result of an experiment using a different batch of JF1 at a lower fuel temperature of 246°C performed by previously at UTIAS with the same apparatus is shown in Figure 4.11. The test, conducted over 25 hours shows reductions in deposit thickness at the 10, 20 and 30 hour mark and hardly any deposit accumulated. This is likely a result of an insufficiently high temperature to induce coking, where the measurement error and noise
of the apparatus was larger than the detected pressure drop.

![Figure 4.11: Pressure drop data for Jet A at a test section temperature of 246°C.](image)

A different set temperature was also calculated for the experiment conducted at the transition regime since the higher fuel flowrate through the apparatus meant that the fuel would take heat away from the test section at greater rate. Thus, a temperature controller setting of 367 °C was used for the test performed at the transition regime.

### 4.2 Pressure Drop Test Main Procedure

The main test involves three phases, a warm-up phase which lasts 1.5 to 2 hours, a steady state phase which lasts 5 hours and pressure drop data is taken at 30 second intervals and a cool down phase which lasts about 30 minutes. For logistical purposes, the steady state phase was run for 5 hours due to the fuel supply.

#### 4.2.1 Phase 1: Warm-up Phase

The first phase of the test is the warm-up phase; at the start of this phase fuel is not yet being pumped and the test section is not actively heated.

The oil bath was set at the desired temperature, which usually takes 1.5 to 2 hours depending on the ambient conditions. Once the oil bath reaches its target temperature, the
syringe pumps are started and temperature recorded every 30 seconds. The temperature is recorded via an Omega Data Acquisition card (Omega, OM-CP-OCTTEMP2000) and recorded via Omega v2.01 software. The water recirculator used for the cooling coil is activated as well. Once the fuel pump is started, the pressure transducer is set to active state where it can detect pressure drops when the National Instruments Data Acquisition Card records data later.

At this stage, the fuel throughout the apparatus is not yet under any significant pressure. Once the flow controller on the pump finishes the equilibrating phase and one of the two tanks drops below 350 mL, the downstream back pressure regulator is slowly turned to increase the flow pressure to 100 psi (689 kPa). This flow pressure was chosen to ensure that in the later steady-state phase, the heated fuel would not boil.

Over time, the test section will heat up since the fuel that enters the test section was preheated by the oil bath. As the test section heats up it expands, and the rate of thermal expansion is proportional to the rate of temperature increase. At a later point (usually around 30-45 minutes after initial fuel flow depending on ambient conditions and the fuel type), the rate of temperature increase decayed to the point where the temperature rise was negligible. This temperature was around 85°C, but depended on the ambient temperature as well as the quality of the insulation applied to the test section.

It is at this point where a 15 minute pressure monitoring is started, where pressure drop data is taken every 30 seconds. This 15 minute run is performed before every steady state run and the day after a steady state run (see Section 4.2.4). This was done for two reasons, the first is that the pressure drop data before and after thermal stressing is independent of the thermal expansion of the test section. The second reason is that the viscosity dependence is removed when the data is taken at the temperature before the steady state phase and the day after the steady state phase.
4.2.2 Phase 2: Steady State Phase

After the 15 minute pressure monitoring is complete, the test section is heated to the desired temperature. Once the test section temperature reaches to within 20°C of the desired temperature, the MatLab code for recording pressure drop every 30 seconds is initiated with a set running time of 5 hours. It would have been impractical to wait until the temperature of the test section reaches the desired set point (as this could last nearly an hour) as the fuel was already being pumped at this phase and exposed to temperatures high enough to cause thermal decomposition.

The temperature of the test section is also recorded during this phase purely for monitoring purposes. As discussed in Section 4.1.4, the temperature will affect the viscosity (and thus pressure drop) of the test section if the temperature profile isn’t uniform. A graph of the thermocouple data over a single test run is shown in Figure 4.12, where the pressure fluctuations are 5 – 6°C at most.

Figure 4.12: Temperature as recorded by thermocouples $T_{ts,1}$ to $T_{ts,4}$.

As mentioned in Section 3.3, the diameter of the preheating section (4.57 mm) is larger
than the test section (0.686 mm), which would cause a slight and relatively consistent drop in pressure between the fuel pump and the back pressure regulator.

Over the duration of the test, the pressure sensors of the Teledyne syringe pumps would gradually detect an increase in pressure even when the downstream back pressure regulator was maintained at a constant pressure setting. This occurs because the test section progressively narrows from deposit accumulation. To compensate and keep the flow pressure consistent in the test section, the downstream back pressure regulator was periodically adjusted so that the Teledyne pump pressure sensors were kept at a reading of 100 psi (689 kPa).

### 4.2.3 Phase 3: Cool Down Phase

Once the steady state run is complete, the test section and oil bath heaters are switched off. The back pressure regulator is slowly turned to reduce the flow pressure until it reaches zero. The pressure transducers are switched to neutral position after which the pumps are shut off. The water recirculator is also shut off since fuel is no longer flowing in the apparatus. Once the experiment is completed, the apparatus is left to cool overnight.

### 4.2.4 Phase 4: Pickup Test

The day after a steady state run, Phase 1 is repeated to collect 15 minutes of pressure drop data after thermal stressing. The 15 minute pressure drop data in Phase 4 must be collected at the same test section temperature that occurred in Phase 1 so as to remove the dependence in viscosity in Equation 3.1. This pickup test is done so that Equation 3.2 can be used; where $\Delta P_1$ is the average pressure drop before the steady state run in Phase 1 and $\Delta P_f$ is the average pressure drop data in Phase 4.

After the 15 minute run, fuel flow is stopped, the oil bath is switched off and the apparatus is left to cool.
4.2.5 Waste Fuel $^1$H NMR

After the apparatus has cooled, a small sample from the waste fuel tank is taken and passed through $^1$H NMR for chemical analysis. The fuel filter is also stored in a clear glass container for comparisons.

4.2.6 Equipment Cleaning

Prior to the next test, the supply and waste tanks are thoroughly rinsed with pure ethanol ($C_2H_5OH$) and left to dry for at least one day to ensure that there was no contamination when a different batch of fuel was used for the next test.

4.3 Carbon Burnoff Test Procedure

Once the test apparatus has cooled, the test section is removed from the apparatus for carbon burnoff analysis so as to determine how much deposit was accumulated in Phase 2. Prior to passing the test section into the carbon burnoff apparatus, the test section is first cut, cleaned, dried in a vacuum oven and then weighed.

4.3.1 Test Section Cutting

The test section is removed from the apparatus and is cut into six pieces using a tube cutter. The two outermost pieces of the test section are 0.625 inches (1.5875 cm) long and the four inner pieces are 0.5 inches (1.27 cm) long. These lengths were chosen as a balance between equal length cuts and the constraint of the gripping rollers of the tube cutter that were blocked by the Swagelok ferrules, as the ferrules were locked into place during the manufacturing of the test section. Tube cutters were used instead of saws or other cutting tools as the tube cutters did not cause blockages of the inner pipe section. The cutting diagram for the test section is shown in Figure 4.13.
4.3.2 Vacuum Oven

After the test section was cut into six pieces, they were lightly rinsed in \( n \)-hexane \( (C_6H_{14}) \) so as to remove as much of the liquid jet fuel remaining inside while preserving the deposit that has accumulated within the test section. Once the pieces have been rinsed, they were dried in a vacuum oven overnight under -90 kPa vacuum gage pressure (approximately 10 kPa of absolute pressure) and 120 °C (393 K). These pressure and temperature conditions ensured that any liquid inside the test section pieces are vaporized prior to the carbon burnoff as any liquid compound (either \( n \)-hexane or fuel) would erroneously add to the detected carbon buildup.

4.3.3 Carbon Burnoff Apparatus

The carbon burnoff apparatus (Eltra SC-800) was used to determine the amount of deposits collected inside the test section by burning the sample in a pure \( O_2 \) environment at a temperature of 700 °C (973 K). The apparatus is equipped with infrared sensors to detect the amount of \( CO_2 \) produced from burning the sample, which then uses it to calculate the original carbon deposit on the sample.

The carbon burnoff apparatus’s infrared cells were initially set to a warm-up phase for 1-2 hours. Once the IR cells were ready, the internal furnace was heated up to 700 °C (973 K), which took between 30 to 45 minutes depending on the ambient temperature of the day. An operating temperature of 700 °C was chosen based on a series of trial runs with older specimens from previous studies as a balance between responsiveness and
reliable results. If the temperature was too high, then the carbon deposits on the sample would start to burn as soon as it was near the furnace and before the recording process was started. This would result in less CO$_2$ detected by the IR cells. If the temperature was too low, it may not burn the accumulated deposit as the carbon burnoff apparatus integrated the CO$_2$ concentration over time, and if there isn’t enough heat to burn the carbon deposit, the IR cells would not detect any and cause the analysis to stop. Using a temperature of 700°C provided a good balance for this as the furnace temperature would also fluctuate by about ± 5°C during the test.

Once the furnace reached 700°C, pure O$_2$ of at least 99.5% purity was fed into the apparatus at pressure of 300 kPa. Calibration runs were performed using pure CO$_2$ fed at a pressure of 100 kPa. During the calibration procedure, the specimens’ mass were measured using a mass balance. Once the calibration was completed, the specimens were passed into the apparatus for analysis, and care was taken to ensure that minimal time passed from feeding the specimen into the furnace and initiating analysis. Most analyses were completed within 3-4 minutes. After taking the specimens out, their masses were once again measured in the balance.
Chapter 5

Results and Discussion

In order to characterize the coking propensity of the fuels, three metrics were used:

The first metric is the pressure drop measurement, discussed in Section 3.3. The test setup is equipped with pressure transducers to measure the pressure drop between the inlet and outlet of the test section. Higher pressure drops indicate that the test section has narrowed due to deposit buildup.

The second metric is the $^1$H NMR of the thermally stressed fuel and fuel filter. As the fuel is thermally stressed, the precursor compounds for deposits begin to form, and over time these precursors become large enough to be caught by the 0.5 micron metal filter. However, some precursors still have a relatively low atomic mass and size which allow them to slip past the filter and into the waste tank. Thermally stressed fuel was also examined with $^1$H NMR to determine the change in chemical composition, particularly the increase in aromatic compounds.

The third metric is the carbon burnoff method, described in Section 3.4.2 which oxidizes the accumulated deposit inside the test section and measures the resulting CO$_2$ to calculate the mass of the deposit. This metric will be used to corroborate the results from the pressure transducers.
5.1 Pressure Drop Results

During the steady state phase of the experiment, the pressure transducers transmitted pressure drop data to a datalogger and a PC recorded them at 30 second intervals. Data from the steady state phase of the fuels in the laminar regime are shown in Figure 5.1. The four graphs indicate a progressively increasing pressure drop, which likely means that the inner diameter of the test section was slowly being reduced due to the buildup of insoluble deposits.

All four of the runs have a similar shape in the pressure drop vs time graphs. The graphs show pressure drop fluctuations in the range of 20 Pa (0.02 kPa) which is likely caused by the syringe pumps pneumatic action when it refills and/or switches pumps to maintain the continuous flow operation mode.

The pressure drop data taken before thermal stressing and the day after thermal stressing were used to calculate the average thickness of deposits via Equation 3.2 for the four tests in the laminar regime. Table 5.1 shows the overall average deposit thickness after five hours of thermal stressing at a test section inner wall temperature of 346°C and $T_{in}$ of 163°C.

The fuel that produced the largest pressure drop values was conventional JF1, while the fuel with the least pressure drop was BJ1, an alternative fuel composed only of paraffins and had no aromatic compounds. The result for BJ1 was expected since aromatics serve as precursors to deposit formation [9], and with fewer aromatics, it becomes more difficult to initiate the formation of deposits. However, the other three fuels have at least 18% aromatics by mass but only JF1 showed a significantly higher pressure drop and resultant deposit thickness.

Fuels JF2 and BJ2 in Figure 5.1 b) and d) showed progressively increasing pressure drop during the steady state phase, meaning that during the thermal stressing portion, the test section diameter was slowly being constricted. However, the pressure drop data taken the day after thermal stressing (where the test section should have had accumulated
Chapter 5. Results and Discussion

Table 5.1: Thickness of accumulated deposit for each fuel type from thermal stressing.

<table>
<thead>
<tr>
<th>Fuel code</th>
<th>Fuel</th>
<th>Thickness of carbon deposit after thermal stressing measured by pressure drop across the test section ($\mu$m)</th>
</tr>
</thead>
<tbody>
<tr>
<td>JF1</td>
<td>Jet A</td>
<td>3.23 $\pm$ 0.18</td>
</tr>
<tr>
<td>JF2</td>
<td>JP-5</td>
<td>-0.847 $\pm$ 0.039 (non-valid result)</td>
</tr>
<tr>
<td>BJ1</td>
<td>Alcohol to jet 99% iso-paraffin</td>
<td>0.440 $\pm$ 0.023</td>
</tr>
<tr>
<td>BJ2</td>
<td>73% C10 iso-paraffin + 23% trimethyl benzene</td>
<td>0.668 $\pm$ 0.043</td>
</tr>
</tbody>
</table>

Deposits did not present a marked increase from the pressure drop taken before thermal stressing. These do not corroborate with the data from the subsequent verification tests. The carbon burnoff tests discussed Section 5.3 show that JF2 and BJ2 produced a similar amount of deposits. The waste fuel $^1$H NMR also showed an increase in aromatics in JF2 and BJ2. The fuel filters used in the JF2 and BJ2 tests became darker as well, which showed that thermal decomposition took place.

5.1.1 Alternative Pressure Drop Measurement and Thickness Calculation

The fuels JF2 and BJ2 have around 18% and 30% aromatics by mass respectively. Curiously, they both presented minimal amounts of deposit accumulation when calculated using the pressure drop before and after the steady state phase. An alternative calculation to find the accumulated deposit was performed. Equation 3.2 requires the terms $\Delta P_i$ and $\Delta P_f$. Instead of using pressure drop data before and after the 5 hour steady state phase, we now use the pressure drops within the steady state phase. Hence, the new $\Delta P_i$ is now pressure drop at the first instance of reaching a stable test section temperature of 346°C while the new $\Delta P_f$ is the pressure drop at the end of the steady state phase which
Figure 5.1: Pressure drop data for JF1, JF2, BJ1 and BJ2 fuels in the laminar regime at a test section temperature of 346°C, $T_{\text{in}}$ of 163°C.
Table 5.2: Thickness of accumulated deposit for each fuel type from thermal stressing using pressure drop data during the 5 hour thermal stressing steady state phase.

<table>
<thead>
<tr>
<th>Fuel Code</th>
<th>Fuel</th>
<th>Thickness of carbon deposit after thermal stressing measured by pressure drop across the test section (µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>JF1</td>
<td>Jet A</td>
<td>24.2 ± 1.3</td>
</tr>
<tr>
<td>JF2</td>
<td>JP-5</td>
<td>7.38 ± 0.39</td>
</tr>
<tr>
<td>BJ1</td>
<td>Alcohol to jet (99% iso-paraffin)</td>
<td>9.41 ± 0.46</td>
</tr>
<tr>
<td>BJ2</td>
<td>73% C10 iso-paraffin + 23% trimethyl benzene</td>
<td>15.4 ± 0.8</td>
</tr>
</tbody>
</table>

was also at 346°C. Keeping the initial and final temperatures identical is key to eliminate the effect of viscosity in Equation 3.1 and to allow the use of Equation 3.2. The results of this alternative calculation are presented in Table 5.2.

The initial problems with JF2 and BJ2 presenting negligible deposit thickness has been rectified as they both now show some accumulation of deposits, however two problems arise from this calculation. The first is that BJ1 fuel which has no aromatics was found to have a thicker deposit accumulation than JF2 which has about 18% aromatics by mass. The other issue is that BJ2 fuel which had the most aromatics (about 30% by mass) had accumulated less deposit than the JF1 fuel which only had 20% aromatics by mass. Both issues contradict the amount of accumulated deposit found by the carbon burnoff apparatus. One possible reason for this is that deposit accumulation in the test section is not uniform along the length of the pipe, as shown in Figure 5.2 and further elaborated in Section 5.3. Another possible reason may be a glitch in the experiment as the steady state run both JF2 and BJ2 fuels have a sudden sharp reduction of about 25 kPa in the pressure drop measurement around the 2 hour mark. A sharp reduction could mean that some of the accumulated deposit was detached from the inner surface of the test section during the test.
Chapter 5. Results and Discussion

5.1.2 Exploratory Case: Pressure Drop Data of Jet A in the Transition Regime

The Hagen-Poiseuille equation described in Section 3.3 is only valid for flows in the laminar regime. This means that calculating the deposit thickness accumulation from the pressure drop data is not valid in the transition regime as there is no guarantee that the flow streamlines are still parallel. Pressure drop data were still recorded and are shown in Figure 5.3 and during the test. It can be seen that the pressure drop continued to grow, indicating that there is some constriction in the test section due to deposit accumulation. Carbon burnoff data also verifies that there is some accumulated deposit.

5.2 Waste Fuel Results

Immediately after the heated test section, the fuel passes through a Swagelok metal filter, ensuring that any deposits larger than 0.5 µm do not flow into the remainder of the test apparatus and cause clogging. However, this means that the fuel contains dissolved
compounds formed from thermal stressing which can be detected via $^1$H NMR.

The $^1$H NMR spectrum describes the percentage of various hydrogen bond types present in the sample. Since thermal stressing to produce more aromatic compounds, we expect an increase in the number of hydrogens in the $H_A$ region of the spectrum. A comparison of the $H_A$ values before and after thermal stressing is presented in Table 5.3. The waste fuels from JF2, BJ1 and BJ2 show a marked increase in aromatics after thermal stressing. However, JF1 showed a slight decrease in aromatics after thermal stressing, which is likely due to the aromatic compounds in the stressed fuel collecting in the fuel filter. Photographs of the filters used in each of the test runs are in Figure 5.4, where it can be seen that the JF1 and JF2 fuel filters are much darker than the fuel filters used for BJ1 and BJ2. The filters are rated for 0.5 microns meaning they collect any precipitate larger than 0.5 microns. With the JF1 filter being the darkest, this is the likely reason why the $^1$H NMR of the thermally stressed JF2 fuel has fewer aromatic compounds than before thermal stressing.
Table 5.3: Aromatic content for fuels before and after thermal stressing.

<table>
<thead>
<tr>
<th>Fuel code</th>
<th>Fuel composition</th>
<th>Percentage composition of aromatic hydrogens before thermal stressing</th>
<th>Percentage composition of aromatic hydrogens after thermal stressing</th>
</tr>
</thead>
<tbody>
<tr>
<td>JF1</td>
<td>Jet A</td>
<td>3.97</td>
<td>3.92</td>
</tr>
<tr>
<td>JF2</td>
<td>JP-5</td>
<td>3.82</td>
<td>4.04</td>
</tr>
<tr>
<td>BJ1</td>
<td>Alcohol to jet 99% iso-paraffin</td>
<td>0.41</td>
<td>0.94</td>
</tr>
<tr>
<td>BJ2</td>
<td>73% C10 iso-paraffin + 23% trimethyl benzene</td>
<td>5.81</td>
<td>6.10</td>
</tr>
<tr>
<td>JF1 (transition regime)</td>
<td>Jet A</td>
<td>5.28</td>
<td>5.21</td>
</tr>
</tbody>
</table>

5.3 Carbon Burnoff Results

During the steady state phase, the fuels were subjected to thermal stressing, resulting in an accumulation of insoluble deposits within the test section. At the conclusion of the experimental runs, the test sections were cut, cleaned and dried in a vacuum oven prior to examining each of them in a carbon burnoff apparatus. The carbon burnoff apparatus heated the test sections to a temperature of 700°C in a pure O₂ environment to oxidize the deposits into CO₂ which is detected in an infrared cell. The results for the five experimental runs are shown in Table 5.4.

5.3.1 Laminar Regime

Comparing the four experiments in the laminar regime, the BJ1 fuel composed of pure paraffins produced the least amount of deposits, which is expected since BJ1 had 0%
Chapter 5. Results and Discussion

Figure 5.4: Filters positioned after the test section at a temperature of 346°C, $T_{in}$ of 163°C.
Table 5.4: Total carbon deposit for each fuel type from thermal stressing.

<table>
<thead>
<tr>
<th>Fuel code</th>
<th>Fuel composition</th>
<th>Total carbon deposit via carbon burnoff ($\mu g$ of Carbon)</th>
</tr>
</thead>
<tbody>
<tr>
<td>JF1 (laminar regime)</td>
<td>Jet A</td>
<td>159.7 ± 37.7</td>
</tr>
<tr>
<td>JF2 (laminar regime)</td>
<td>JP-5</td>
<td>153.2 ± 37.7</td>
</tr>
<tr>
<td>BJ1 (laminar regime)</td>
<td>Alcohol to jet 99% iso-paraffin</td>
<td>82.42 ± 37.7</td>
</tr>
<tr>
<td>BJ2 (laminar regime)</td>
<td>73% C10 iso-paraffin, 23% trimethyl benzene</td>
<td>157.7 ± 37.7</td>
</tr>
<tr>
<td>JF1 (transition regime)</td>
<td>Jet A</td>
<td>122.2 ± 37.7</td>
</tr>
</tbody>
</table>

aromatic content; and deposit formation tends to begin with precursor aromatic compounds [9]. The other three fuels in the laminar regime JF1, JF2 and BJ2 all had similar amounts of deposits despite their varying aromatic contents. A possible reason for this is that although aromatics serve as the precursors to deposit formation, once their concentrations are high enough, other aspects in the experiment become the rate-limiting factor(s). The fuel temperature and/or fuel residence time also play a role in how fast fuels thermally decompose.

The deposit profile within the test section for JF1 fuel is shown in Figure 5.5. The measurement or systematic error of the carbon burnoff apparatus was stated as 1% from the manufacturer. However, the random error is much more problematic to evaluate, as it is difficult to repeat measurements of the deposit buildup as the apparatus is supposed to oxidize all of carbon within the test section. In order to approximate the random error, three test sections that were not used in the coking experiments were likewise cleaned, cut, placed in a vacuum oven tested in the carbon burnoff apparatus. As these test sections were unused, the carbon burnoff apparatus should have detected no deposit.

However, the carbon burnoff apparatus did register some carbon deposit, hence the random error was taken to be the standard deviation of these trace readings, which on average were 5-6 $\mu g$ of carbon for an unused test section. For comparison, the deposit
Figure 5.5: Graph of carbon deposit along the test section for JF1 in the laminar regime at a test section temperature of 346°C and $T_{in}$ of 163°C.

profile within the test section for the four experimental runs conducted in the laminar regime are shown in Figure 5.6, and error bars describing the random error were omitted for clarity. In these experiments, the amount of deposit appears to be highest in the initial portion of the test section even though Figure 4.12 shows that the temperature is almost uniform through the length of the test sections. This would imply that deposit accumulation should be uniform through the length of the test section, which is not the case here. In this work, it is important to note the hydraulic and thermal boundary layers. The hydraulic boundary layer may be at steady state by the time the fuel enters the test section but the thermal boundary layer has an ”entrance length” through the test section, where the temperature profile has yet to reach steady state.

The accumulation of deposit in near the inlet of the test section can also be explained by the large temperature gradient experienced by the fuel as it leaves the oil bath which had a fuel temperature of 163°C and into the test section that kept the fuel temperature at 346°C. Deposit formation is strongly linked to temperature, and once the fuel is exposed
to a sudden increase in temperature, precursors would begin to form and deposits would start to accumulate on the inner wall. Since the inlet region of the test section is the first area that exposes the fuel to 346°C, the fuel would begin to undergo decomposition there. Once the inner wall near the test section inlet has some accumulated deposit, it could have served as a nucleus for further precursors and deposits to attach to.

Figure 5.6: Graph of carbon deposit along the test section for JF1, JF2, BJ1 and BJ2 in the laminar regime at a test section temperature of 346°C and $T_{in}$ of 163°C. Error bars are omitted for clarity.

5.3.2 Exploratory Case: Carbon Burnoff in the Transition Regime

The Jet A experiment performed in the transition regime resulted in deposit accumulation of roughly 20% less than the Jet A in the laminar regime. This possibly a result of the change in the temperature and precursor compound profiles in the test section. In the laminar case, the fuel streamlines would have been parallel causing the fuel closest
to the wall to be consistently hotter than the fuel in the center of the test section. When a laminar flow transitions, these streamlines can possibly start mixing, resulting in temperature gradients that are less steep and preventing the precursor compounds that later form insoluble deposits to accumulate at the test section wall. The deposit profile within the test section used for the experiment conducted in the transition regime with Jet A fuel is shown in Figure 5.7. Like the four tests in the laminar regime, the highest concentration of deposits is near the inlet of the test section. This supports the claim of Spadaccini et al [10] where increasing turbulence in the pyrolytic temperature regime can reduce deposit formation.

![Figure 5.7: Graph of carbon deposit along the test section for JF1 in the transition regime at a test section temperature of 346°C and $T_{in}$ of 163°C.](image-url)
5.4 Sources of Experimental Errors

One of the largest sources of uncertainties in this work is the supply of aviation fuel. In order to produce repeatable data, the aviation fuels’ composition would need to be almost identical across batches. However this is hardly the case as the fuel feedstock cannot be guaranteed to come from the same location, and even fuels from the same refinery but processed at different times of the year have different compositions. For example, the percentage of aromatic hydrogens $H_A$ (see Table 3.2) in the Jet A batch procured in March 2015 was 4.89%, while the batches from June and August of 2015 had $H_A$ of 3.81% and 4.18% respectively.

5.4.1 Pressure Drop Experiment

The pressure drop experiment used to determine the thickness accumulated thermal deposit has three main sources of errors, the pressure transducer measurements, the thermocouple measurements and the manufacturing of the test section.

The pressure transducers have a rated accuracy (measurement error) of $\pm 0.08\%$ of its full range, which is equivalent to $\pm 5.5$ Pa ($\pm 0.8 \times 10^{-3}$ psi). The larger source of error in the pressure drop experiment however, comes from the fluctuations during the steady state phase as a result of the Teledyne ISCO pumps. The continuous flow mechanism of the syringe pumps works by having two pumps alternately pump and refill, such that when one pump is almost empty, the controller automatically switches to the other pump to give time for the first pump to refill its contents. These fluctuations lead to random error that can be as high as 8-10 Pa.

The Type K thermocouples used to monitor the test section temperature have a rated accuracy of $\pm 2.2^\circ C$ and the temperature controllers of the oil bath and test section managed to keep the temperature fluctuations to within $\pm 1^\circ C$ for the duration of the test. Another source of error was the application of insulating foam on the test section. The
temperature may directly influence the calculations for the deposit thickness if it is not kept constant as viscosity is dependent on temperature. However, for the temperatures used in this work, minor variations in temperature on the order of ±2.2°C result negligible variations in fuel viscosity as described in the Lewis-Squires relation in Equation 4.4.

The test section was made from Stainless Steel 304 tubing with a length of 82.55 mm, outer diameter of 3.18 mm and nominal inner diameter of 0.686 mm (0.027 in). After cutting and fitting with Swagelok ferrules the inner diameters were once again measured using gage pins that have an accuracy of ±0.0005 in (±0.0127 mm). For a single test section, the inlet and outlet diameters were measured to be nearly identical by gage pins. However, from one test section to another, it was found that the inner diameters ranged from 0.027 to 0.033 in, indicating that the inner diameter may vary slightly along the length of a single test section. To minimize the potential error in the uniformity of the inner diameter, newly cut test sections were rejected if the inner diameter between the inlet and outlet differed by more than 0.0127 mm (0.0005 in) or if the gage pins could enter the inlet but could not fall through to the other end at the outlet.

Another possible issue with the test sections is thermal expansion due to the increase in temperature. The average thermal expansion coefficient of stainless steel is $17.3 \times 10^{-6}/\text{K}$ for the temperature ranges of (0°C-315°C). As the temperature of the test section increases, its inner diameter along with outer diameter and length are expected to increase. Using the average thermal expansion coefficient, it is expected that an internal diameter of 0.686 mm would rise to 0.690 mm or about a 0.5% increase in diameter. To mitigate excessive increases in the inner diameter, the brass blocks that surround the test section are restrained.

Measuring the inner diameters of the test sections using gage pins was performed for verification and the results of which are presented in Table 5.5. Unfortunately, due to the limited resolution of the gage pins (± 0.0005 in or ±0.0127 mm), it makes the diameter increase is difficult to ascertain. The gage pins could also expand once in contact with
Table 5.5: Test section inner diameters at various temperatures.

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>Test section inner diameter (mm)</th>
<th>Inner diameter % increase from 20°C</th>
</tr>
</thead>
<tbody>
<tr>
<td>20</td>
<td>0.8382</td>
<td>0</td>
</tr>
<tr>
<td>110</td>
<td>0.8382</td>
<td>0</td>
</tr>
<tr>
<td>140</td>
<td>0.8382</td>
<td>0</td>
</tr>
<tr>
<td>170</td>
<td>0.8382</td>
<td>0</td>
</tr>
<tr>
<td>200</td>
<td>0.8636</td>
<td>3.0</td>
</tr>
<tr>
<td>225</td>
<td>0.8636</td>
<td>3.0</td>
</tr>
</tbody>
</table>

the heated test section which would further compromise the measured inner diameter.

5.4.2 \textsuperscript{1}H NMR of Waste Fuels

The accuracy of nuclear magnetic resonance would depend greatly on the chosen ranges of the types of compounds. As discussed in Section 3.4.1, the ppm ranges of the various carbon-hydrogen bonds are determined experimentally and different studies could present alternative ranges [42, 43].

5.4.3 Carbon Burnoff Apparatus

The stated measurement error of the carbon burnoff apparatus from the manufacturer is ±1%. However, the random error is more difficult to evaluate, as once the test section has been heated to 700°C, it cannot be used in a repeat test to check for precision since no carbon deposit will remain. To estimate the random error, three unused test sections were cut, cleaned, dried in a vacuum oven and passed through the carbon burnoff apparatus. Since the three test sections were not exposed to any fuel or thermal stressing, it should have negligible carbon deposits. The random error was then calculated from the standard deviation of all the cut sections, leading to an estimated random error of approximately 6 \mu g.
Chapter 6

Conclusion

A novel experimental apparatus was used to examine the thermal stability of conventional and alternative aviation fuels. The apparatus simulates the residence time, pressure and temperature conditions experienced by the fuel as it acts as a heat sink and in the fuel spray nozzle prior to burning in a combustor.

Three metrics were used to characterize the thermal stability. The first metric was by using a pressure transducer with a range of 1 psi (6.89 kPa) to monitor and record the pressure drop across a heated test section. Larger pressure drops indicated that the test section was progressively narrowing due to a buildup of insoluble deposits. The second metric was the chemical analysis of the thermally stressed fuel by Hydrogen Nuclear Magnetic Resonance (\(^{1}\)H NMR) and examination of the fuel filters. A higher concentration of aromatics in the thermally stressed fuel indicated greater thermal decomposition. The last metric was by passing the test section into a carbon burnoff apparatus, which heated the test sections to 700°C in a pure oxygen environment to oxidize the accumulated deposits into CO\(_2\) which is then detected by IR cells and used to calculate the amount of carbon deposit. Four different types of fuels were examined with these metrics to determine the effect of chemical structure and composition of the fuel on its coking propensity. Using three metrics allows a comparison between their results, so as to
determine the most reliable and accurate method of determining the thermal stability.

The experiments showed that fuels with aromatic compounds produced more deposits when thermally stressed than fuels with no aromatic compounds. However, when comparing fuels with aromatic compounds, the amount of accumulated deposit did not vary as much. This is likely due to the aromatics being present in excess amounts and other factors such as the fuel temperature or fuel residence time serving as the limiting factors to deposit formation. This is supported by the experiment of JF1 (Jet A) in the transition regime at the same test section temperature as the JF1 in the laminar regime. The higher flowrate resulted in a shorter residence time in the test section, which produced fewer deposits detected by the carbon burnoff test.

The experiments also showed that the pressure drop method could produce erroneous data that showed a negative thickness of accumulated deposit. This is possibly a consequence of the non-uniformity of deposit accumulation along the length of the test section or sudden glitches that present as large reductions of pressure drops. If more fuel were available, more steady state runs could be done to investigate this problem further.

6.1 Recommendations for Future Work

The following recommendations are given to improve the accuracy of the experiments.

1. Alternative aviation fuels are currently (Jan 2017) still too expensive to compete with conventional jet fuel, which causes great difficulty in obtaining large quantities of alternative fuels for experiments. When alternative fuels become more affordable, it is recommended that more be procured for these experiments. Further tests could also be performed in the turbulent regime and deposit buildup could still be reliably measured by the carbon burnoff apparatus; however this will require more than triple the fuel supply used in this experiment to bring the Reynolds number into the turbulent regime. When performing the pressure drop test in the turbulent
regime, the Darcy-Weisbach equation may be substituted for the Hagen-Poiseuille equation.

2. Conventional aviation fuel (Jet A and Jet A-1) procured from the same location or refinery but a different times of the year have varying aromatic contents. This variation makes comparisons difficult since aromatic compounds are the precursors to deposit formation. It is therefore recommended that a suitable substitute fuel be used that is made from known pure compounds in known proportions so that the resulting data is reproducible. The substitute fuel ideally should fulfill the requirements listed in ASTM D1655 [2].

3. It would be possible to test the fuels at even higher temperatures, but this would require higher flow pressures. Tests from a previous project with this same apparatus showed issues with the pressure transducers produced erroneous readings when the flow pressure was too high (about 150 psi). Newer and/or improved pressure transducers could solve this issue.

4. Performing the pressure drop experiment with different pipe materials could also be worth a look. Venkataraman et al [15] showed that metal sulfides which form from dissolved sulfur in the fuel act as substrates for deposit formation reactions.

5. Examining the oxidation characteristics of alternative aviation fuels is a possible project as their chemical structure is unlike conventional fuels, with oxygen chemically bonded to the fuel compounds. This will likely entail pumping O₂ into the fuels and heating solely in the autoxidation temperature regime.
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