DESIGNING A SYSTEM FOR UPGRADING OF HEAVY CRUDE OILS THROUGH ELECTRON BEAM TREATMENT

A Thesis

by

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ABSTRACT

Low-quality crude oil reserves require prohibitively high energy costs to extract and transport. The extreme viscosity and impurities of these oils prevents them from being transported via pipeline, requiring the use of more expensive trucks or trains. Light crude oil has a viscosity ranging up to 100 cP at 40˚C. In contrast, Crude Oil #1 under investigation measures 33,855 cP, and Crude Oil #2 is 4,570,000 cP at the same temperature as measured in the laboratory. Sulfur content of both exceeds 5% by mass. Effects of the exposure of these oils to an electron beam discharge are being researched to reduce viscosity with higher conversion factors, using less energy at low temperatures.

To facilitate this investigation, a flow loop was created with controls to adjust oil initial temperature with line heaters, radiation dose rate with height adjustment, flow shear rate through flow channel angle, and flow residence time through a gear pump. The flow loop uses stainless steel lines with a gear pump built to handle viscous oil at 230˚C, and makes extensive use of aluminum versus steel in a modular frame to prevent overheating from the e-beam. To support the flow test cart, a remote control station cart was created, along with a fire safety cart and mobile test cell for safe sample extraction and shakedown testing.

In designing the system and writing safety documentation, the test vehicles were further refined as new concerns were addressed and potential hazards mitigated.

Preliminary testing of the various system components yielded a successful design. The end result is a set of systems that allows for ease of variability in operating parameters such as dose rate, gas environment, and added hydrogenation.

DEDICATION

This thesis and the work standing behind it is dedicated to my late paternal grandmother, Mrs. Ella Louise Berman. Born September 21st, 1931, and raised through the era of the Great Depression, she lived a full and valiant life until her passing in February of 2014. Serving in the United States Air Force, she helped fly the President and was the first non-civilian woman with nuclear security clearance to work in the Pentagon. Her hard work put my father through New York University, where he studied to become a teacher and ultimately provide me with a better place to start my life and career one step ahead. Her passing saw me through to my first career interview, and her determination to survive and excel in the face of adversity inspires my continuing work through this day.

ACKNOWLEDGEMENTS

I would like to thank my committee chair, Dr. David Staack, for presenting me with the critical opportunity to work under his guidance as I bring my academic career to completion. I would like to thank my committee members Dr. Jerald Caton and Dr. Mark Holtzapple for their support and assistance. I would also like to thank all members of the plasma engineering lab with whom I have had the pleasure to work. Without the solid guidance of my senior lab members and invaluable teamwork with my peers, progressing this far would not have been possible.

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Lastly, I would like to thank my family and my girlfriend Irina Chen for their love and continued encouragement, and my friends for their support and warm welcoming to both the beautiful State of Texas and the Aggie family at Texas A&M University.

NOMENCLATURE

API	American Petroleum Institute
bbl	Barrel of Oil (42-gallon)
˚C	Degrees Celsius
CAPEX	Capital Expense
cP	Centipoise (unit of dynamic viscosity, 1000cP = 1Pa-s)
DRO	Diesel Range Organics
EBRF	Electron Beam Food Research Facility
EEP	Emergency Evacuation Plan
EROEI	Energy Return on Energy Invested
eV	Electron Volt
gpm	Gallons per Minute
GRO	Gasoline Range Organics
H_2S	Hydrogen Sulfide
HVAC	Heating, Ventilation, and Air Conditioning
hp	Horsepower
J	Joule
LINAC	Linear Accelerator
m	Meter
MKOPSC	Mary Kay O'Connor Process Safety Center
mol	Gram Mole

NFPA	National Fire Protection Association
NGC	Natural Gas Condensates
OPEX	Operating Expense
P&ID	Piping and Instrumentation Diagram
PPE	Personal Protective Equipment
PSA	Project Safety Assessment
psi	Pounds per Square Inch
RTC	Radiation Thermal Cracking
SDS	Safety Data Sheet (formerly MSDS – Material Safety Data Sheet)
SOC	Sample Oil Container
SOP	Standard Operating Procedure
TC	Thermal Cracking
USB	University Services Building
VFD	Variable Frequency Drive
VI	Virtual Interface
W	Watt

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CHAPTER I

INTRODUCTION

Brief Background

The ability to solve problems is a principle characteristic that defines an engineer. A mechanical engineer may enter a multitude of industries, each with its own set of challenges, and must be able to quickly adapt their skillset to overcome the tasks at hand. Each challenge has many facets that must be handled in unison to make a solution that is not only effective, but also safe, efficient, reliable, and cost-effective.

Many challenges are found in the oil industry. Oil is one of the most important natural resources on the planet, and the oil and gas industry is constantly searching for new sources of this finite resource. From the time an oil reserve is discovered to the time that oil is sold as a commercial product, many processes and types of machinery are required to refine crude oil into petroleum products. Ideally, integrating these systems would be seamless so oil is handled as a continuous flow. Although vast reserves remain available, they are becoming increasingly difficult to extract. Heavy, sour crude oil requires far more energy to extract and refine into petroleum products than lighter oils with fewer impurities. To make these oils viable for production, new methods are being explored to reduce their viscosity and remove impurities at reduced cost and increased efficiency.

One strategy for upgrading extra-heavy crude oils involves use of an electron beam. When the oil is exposed to the e-beam, the long carbon chains are broken up to reduce viscosity. As a byproduct of this processing, the sulfur impurities in the oil can also be released in gaseous forms, which requires safe handling of the toxic exhaust gases. Different procedures are under investigation on how to use the e-beam itself to find the most effective way of treating oils.

Thesis Statement

The goal of this research is to explore e-beam treatment of heavy crude oil. The objective of this thesis is to identify the process of developing a lab-scale engineering solution to enable this oil treatment with repeatable results in a safe manner. This will include discussion of the design, construction, preliminary testing, and finalization of the main test apparatus and its supporting systems.

Thesis Overview

In Chapter II, detailed background information will be presented on the challenges posed by unfavorable oil. The inner workings of the e-beam system and how it may benefit the industry will be discussed. Current solutions and treatment methods will be investigated and compared with the prospective results of e-beam treatment. Health and safety hazards of oil extraction and treatment will be shown. Lastly, patents and papers on the treatment of oil with e-beam technology will be reviewed to show progress made thus far on developing this solution.

Chapter III will show the process of developing a safe solution for e-beam treatment of heavy crude oil. This begins with defining the goal of the project to create a safe and reliable flow loop. After that, discussion will cover the establishment of physical size and working fluid property constraints, which determines the components of the system. Next, possible test apparatus development, fabrication of the chosen option, and troubleshooting the design through testing are covered. The development of a mobile test cell with a controlled environment will also be discussed.

Chapter IV will cover the finalization of the test apparatus. Precision control systems to ensure repeatable results, along with safety systems to safeguard against failure of any component in the system, will be reviewed. Documentation of the project in the form of a PSA and various SOP’s for certification will also be covered. Lastly, Chapter V will state conclusions about the viability of this research and work to be done in the future.

CHAPTER II

DETAILED BACKGROUND AND LITERATURE REVIEW

Chapter Overview

The oil and gas industry is turbulent because industry methods change with the availability of natural resources and the prices of the products. When the oil price is low, there is little motivation to pursue certain energy reserves that would cost more to extract than makes financial sense. As the price increases, more options become viable, but each option requires more complex tactics to be used.

Current methods are in use to refine extra-heavy crude oil into usable petroleum products. However, because of the properties of the crude oil, the process of bringing it to market is energy intensive and yields far less product per energy input than processing of lighter oils would. A significant portion of the increased cost comes from transportation, both of the oil and of diluents used in extraction. The oil is loaded into tank cars on both trucks and trains, but to do so the viscosity must be reduced for pumping. A diluent must therefore be shipped to the remote location of the oil well, pumped into the well, pumped back up with the oil, shipped with the oil to a refinery, and finally separated from the oil. Because refinery structures are already built around these processes, the cost of an alternative method must be amortizable by its energy savings.

The method under investigation is using electron beam exposure to treat extra-heavy crude oils. The excited electrons carry enough energy to break longer carbon bonds that cause extreme viscosity in the crude. In the presence of a hydrogen-rich atmosphere, the smaller hydrocarbon chains have the potential to become more saturated, bringing the crude one step closer to a finished product. Hydrogenation plays a key role to reduce the carbon double bonds that can be formed by the electron beam. These are unfavorable because of their unstable nature. Too high a dose negates the positive effects of exposure by polymerizing the oil, increasing its viscosity. The effects of varying parameters such as electron radiation dose rate, overall radiation dose, and flow shear rate are being researched to determine the viability of further development of this technique.

Understanding Crude Oils

Crude oil is a term that describes a wide variety of non-uniform substances. Crude oil is a mixture of various hydrocarbons varying in structure and degrees of saturation, with a number of possible impurities. The structure of the hydrocarbons inside the crude oil changes its bulk properties, most importantly its viscosity and density. Light, sweet crude is an ideal substance because of its low viscosity for ease of transport, and its low concentration of impurities for ease of refining. However, most sources of light sweet crudes have already been discovered and depleted.

Oil is rated on the American Petroleum Institute's (API) scale for specific gravity. The scale is in units of degrees on an inverse measurement. Water is used as the standard, set at 10 degrees. Oil that measures higher than 10 is lighter than water and will float on its surface, whereas oil with an API rating lower than 10 will sink. Light crude oil has an API rating of at least 22 degrees. Figure 1 below shows a spread of crude oils from various regions of the globe. There is a wide variety of densities and concentrations of sulfur found in crude oil.

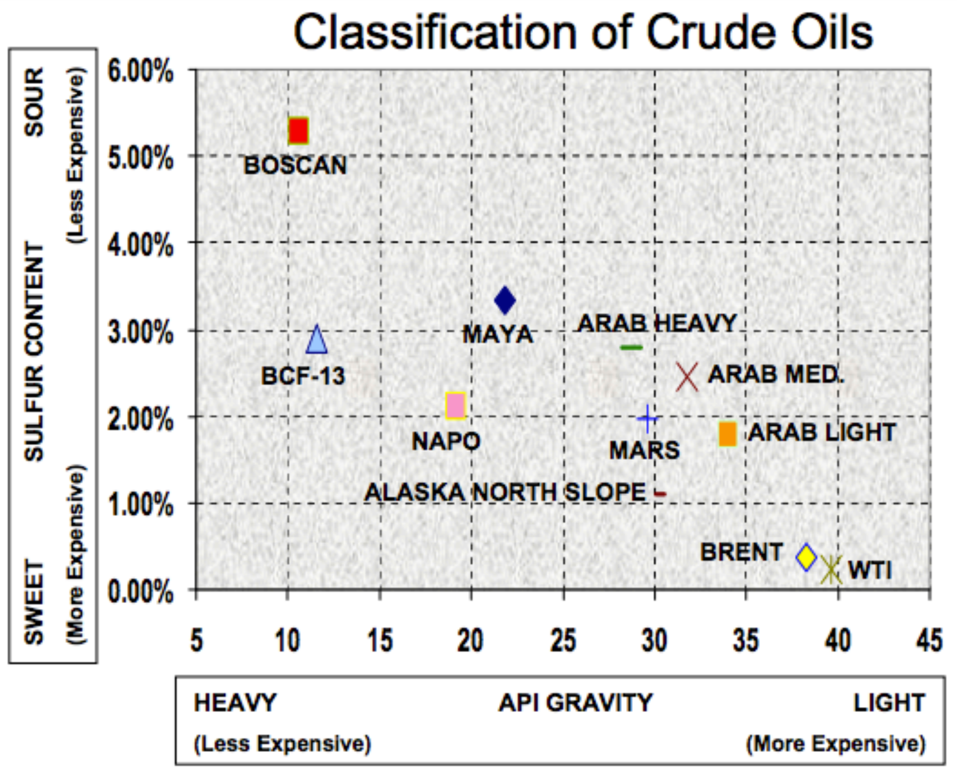


Figure 1 - ***Crude oil properties.*** **Reprinted from [1]**

Extra-heavy crude oil has an API gravity rating of less than 22. One type of this oil in particular is called bitumen, extra-heavy oil with a viscosity greater than 10,000 cP, of which 81% of the world's reserves are found in Alberta, Canada [2]. The bitumen that comes from natural sites is also generally a sour crude, meaning it has a high sulfur impurity content. Sulfur can exist in oil typically bonded to carbon atoms, as combustible pyrite, or in the form of deadly hydrogen sulfide gas (H_2S), and must be removed from the oil prior to transport in tankers. The sulfur must also be removed to meet stringent sulfur concentration limits for highway diesel fuel and fuel oil [3].

Risks involved with processing heavy crude oil are plentiful and must be carefully handled to ensure safety. Some impurities found in oil besides sulfur include iron, nickel, mercury, vanadium, and arsenic, which can pose significant health issues for exposed personnel [4]. Besides the constant flammability hazard of fumes and liquids at all stages of life of the crude oil, sulfuric gases pose severe health risks. These gases can build up in natural oil deposits, being released during exploration, or are formed during elevated temperature processing. Exposure to high concentrations of mercaptans and sulfides quickly deteriorates the nervous system and can lead to death. These gases must be handled properly, in this case through containment and controlled release in isolation.

The high viscosity of these oils is inconducive to pipeline transport, forcing the oil to be moved by truck, train, and ship. Common carrier companies that own and operate the pipelines have tariff rules, regulated by the Federal Energy Regulatory Commission, that dictate the properties of the products allowed to be shipped. Where available, oil tankers are price-competitive alternatives to pipelines, but most areas cannot be accessed by the large ships [5]. Because of the tar sands boom of recent years, oil tank cars on trains have seen a drastic increase in use. Dispatch numbers of unit trains, trains comprised of cars that solely transport one type of cargo, went from 207 in

2010 to 1,775 in 2012. The older design of the tank cars pose another risk in that they are not fit for the current level of service. Corrosion from chemicals added to the heavy crude oil during hydraulic fracturing and extraction weaken the tank cars, increasing the susceptibility of the crude to ignition because of containment issues.

Between the contaminants and the viscosity, the adverse qualities of these extra-heavy crudes translate to increased cost, making the oil a poor business choice when prices are low. The price of oil has steadily risen in recent years, meaning it is a matter of time until these oils are worth their effort, so new tactics are being developed to make the oil more energy effective to extract.

Current Methods

Presently, there are two major ways to prepare extra-heavy crude oil for distribution. One is by adding a diluent to reduce viscosity for pipeline transport, and the other is by using an upgrading facility to partially refine the oil. In the case of dilution, a lighter hydrocarbon is mixed with the heavy crude to reduce viscosity, and is later separated through distillation. Upgrading facilities near the extraction sites for extra-heavy crudes use extracted feedstock to create synthetic crude oil. The methods used in upgrading facilities include fractional distillation, vacuum distillation, de-asphalting of residue from vacuum distillation, cracking, and hydrodesulfurization. Each technique adds energy through heat, chemical catalyzing, or both, to reduce viscosity and sulfur content of the crude. One metric to judge viability of the entire process is energy return on energy invested, or EROEI. For bitumen, this can vary from as low as 1.8 to as high as 6.8, meaning one unit of energy in will yield 1.8 to 6.8 units of energy as products [6]. This energy is generated with the least expensive resource available, for example a BTU generated from cheap natural gas to create more BTU's of more valuable synthetic crude. While it may be economically justifiable, low EROEI processes are highly unfavorable from a conservation standpoint. Over time, greater efficiency has been achieved in these practices to allow more barrels of oil to be produced per barrel equivalent of energy input.

Dilution

Diluting extra-heavy crude oil is a current solution to moving the crude from a remote well to a refinery for processing. It is energy cost-intensive because of the extra transportation needs of moving the diluent around with the crude and on its own. The goal of this process is to mix the extra-heavy crude oil with a lighter hydrocarbon to reduce its density for transport in a pipeline, and to choose a diluent such that the mixture will be stable. If the mixture is unstable, heavy crude may precipitate out during transport and could lead to blockages in the pipeline. A blockage could be detrimental to the structure of the pipeline, as it could cause a spike in pressure that the fittings in the pipeline were not designed to safely handle.

One example of common application of diluting heavy crude is found at the Athabasca tar sands in Alberta, Canada. In the 1980's, the Alberta Energy Company developed a solution to moving bitumen through a pipeline by creating a fluid called "dilbit," a combination of diluent and bitumen. Dilbit is classified as a mixture of diluent and bitumen where the diluent density is lower than 800 kg/m^3. Natural gas condensates (NGC) are the most common diluent used, with the naphtha component playing a key role. However, the short supply of natural gas condensates in the area forced the company to use two separate pipelines to move the bitumen. One pipeline brings NGC in, and the other sends out the dilbit [7]. The costs of procuring the NGC, transporting it via pipeline to the well, pumping it into the well, pumping it back out as a component of the dilbit, sending it out via pipeline, and refining it back out of the dilbit are all added to the costs of pumping, transport, and refinement of the bitumen itself.

An alternative to the NGC is synthetic crude oil, which has been refined from other heavy crudes. This requires a higher concentration of diluent because of its higher density to reach the same API rating as with NGC, but has been shown to have higher stability as a mixture with the bitumen [8]. When bitumen is mixed with synthetic crude, the mixture is called "synbit," but synbit is used to classify any mixture of bitumen and diluent where the diluent density is greater than or equal to 800 kg/m^3. Presently, bitumen density is reduced to roughly 925 kg/m^3 before transport through a pipeline, using NGC with a density of roughly 700 kg/m^3 and a standardized bitumen density of 1015 kg/m^3 [9].

The positive side to using a diluent is that the diluent is comprised of hydrocarbons, just like the crude oil. This means it can be separated for reuse to create more dilbit. It also means it can be refined as a mixture and sold as petroleum products. Although some oil wells use dilution to send crude out for final refinement, others will use dilution on a short-term basis for transport to a nearby upgrading facility before it is moved to market.

Upgrading

Fractional distillation is the most common method used in industrial applications to separate components of crude oils [10]. Fractionation would be used at an upgrading facility to separate any lighter components that are present in the heavy crude oil. The fractionation system consists of a tower with an input feed, a heat source, and outlets at various heights along the tower. Figure 2 below shows the layout of a typical fractionation tower at an oil refinery.

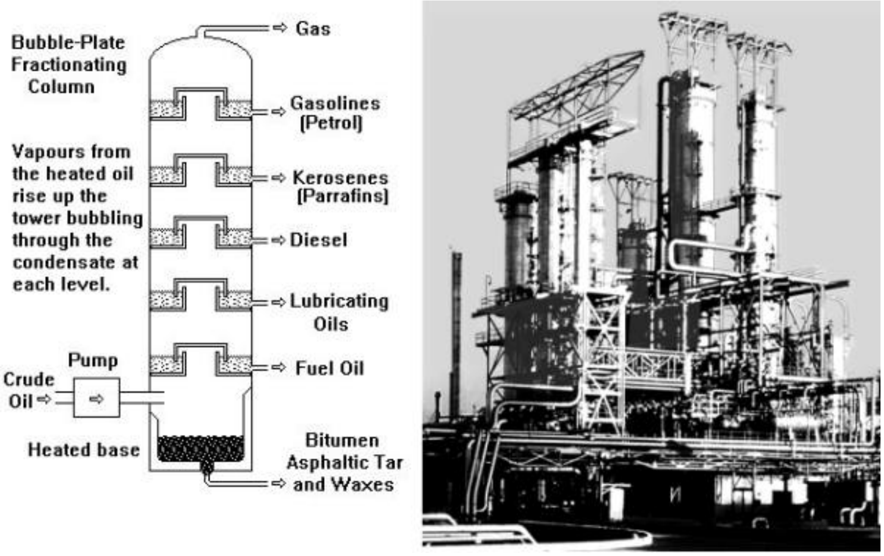


Figure 2 - ***Fractionation tower cross section.*** **Reprinted from [11]**

Because crude oil is comprised of various hydrocarbons, each component has a separate boiling range. The oil is heated to a temperature less than roughly 400˚C to avoid thermal cracking and the formation of petroleum coke. As the feed is heated, the hydrocarbons are boiled off and separated at different heights because of their varying densities. Heavy tar made of hydrocarbons with high boiling points is taken from the bottom of the tower, and light hydrocarbons that boil at room temperature exit through the top. The entire process is usually run at continuous steady state, where the mass flow rate of feed in is equal to the mass flow rate of the products being taken out.

Because fractional distillation does not separate the heavier hydrocarbons left at the bottom of the tower, other methods are required to refine the residuals. Vacuum distillation is generally the next step, which involves lowering the pressure in a separate distillation tower to below that of the vapor pressure of what is in the residuals. What remains after vacuum distillation is harder to process without high-energy input or chemical treatment. In the refinement of lighter crude oils, these vacuum residuals are most similar to the starting point of refining the oils under consideration for this project.

At this point, the vacuum residuals would be sent to a refinery for further processing. Visbreaking uses mild thermal cracking to break up long carbon chains in molecules to reduce viscosity. Feedstock, either residuum or bitumen, is heated to temperatures between 455˚C and 510˚C to begin thermal processing and is then quenched to halt the reactions before completion [12]. The visbreaker in a refinery is found in one of two styles, coil or soaker type, and comes before additional fractionation towers. A coil visbreaker is simply a coil in the feed line placed in the furnace that would normally heat the oil up for fractionation, leading to longer residence time for heat addition. A soaker visbreaker is a drum with lower temperature than the furnace where the oil is kept for much longer [13]. As mentioned, the cracked product then goes through another round of fractionating and vacuum distillation towers. At this point, most of the usable products have been extracted from the crude, and the residuals are non-volatile tar or wax. As mentioned previously, sulfur must be removed before certain petroleum products can be sold to market. Hydrodesulfurization is a process that treats oil after it is refined and turned into various grades of fuel. Through the use of catalytic metals, sulfur bonded to carbon atoms is replaced with a hydrogen atom, further saturating the hydrocarbon [14]. The sulfur is generally given off as hydrogen sulfide, which is later converted in the refinery to elemental sulfur or sulfuric acid.

This suite of refining technologies and strategies has served the industry for decades. As the characteristics of new and remaining crude oil reserves change, new methods are needed to adapt to the challenges they pose and maintain profitability. The process under investigation for this research is exposure of the oil to electron beam radiation. Cracking of large hydrocarbon molecules using radiation exposure has the potential to yield larger quantities of marketable petroleum products as these traditional methods. At the same time, it uses far less energy and has fewer steps, saving time as well as money.

Electron Beam

The electron beam is a powerful tool for a multitude of applications, from x-ray imaging to sterilization of food and medical supplies. The linear accelerator that generates the beam is simple in concept; it uses electric current to initiate the thermionic emission of electrons, a high-voltage accelerator, and a magnetic optical system for focusing and positioning the beam. Actual applications have more parts and supporting systems, including instrumentation and a vacuum enclosure. The components are operated in a vacuum to prevent air particles from interfering with the electrons inside the device. Figure 3 below shows a general diagram of a linear accelerator and electron beam. Electron beam energy varies depending on the individual accelerator; the one found at Texas A&M University's Electron Beam Food Research Facility (EBRF) is 10 MeV.

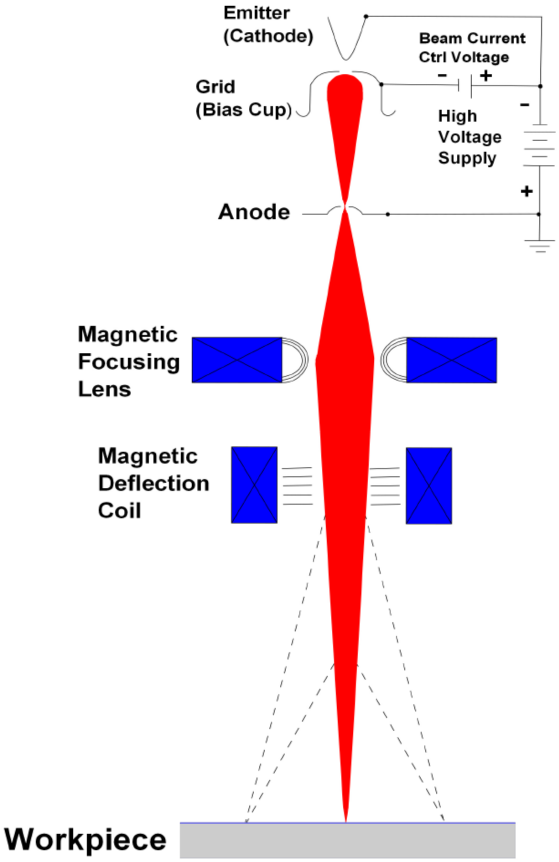


Figure 3 - ***Electron linear accelerator diagram.*** **Reprinted from [15]**

Understanding how to use the electron beam for specific applications is key to achieving desired results of processing. The beam at the EBRF is mainly used to sterilize food and medical products by passing them through a conveyor belt underneath the beam horn as it scans across a roughly 24-in-long, 2-in-wide treatment area. Altering the speed of the conveyor belt for these applications controls radiation dose. However, for the purposes of this research, the conveyor belt is not used, so control methods are different. Dose is defined as the energy absorbed by the sample from the beam, generally expressed in units of kilogray (kGy). One kGy is one kJ/kg, synonymous to specific energy input. In this application, it is dependent on dose rate and time of exposure; time is varied at the control station, but dose rate is a function of distance from the window of the electron beam horn. This variance is because of the expansion of the beam at roughly a 5-degree angle, where the same energy is spread out over a wider area as distance from the window increases. A graph was developed through prior research involving this accelerator to determine these numbers. Figure 4 is the graph used before each experimental run to estimate dose rate as a function of measured distance. The ability to precisely control dose rate and total dose are critical to having reliable results.

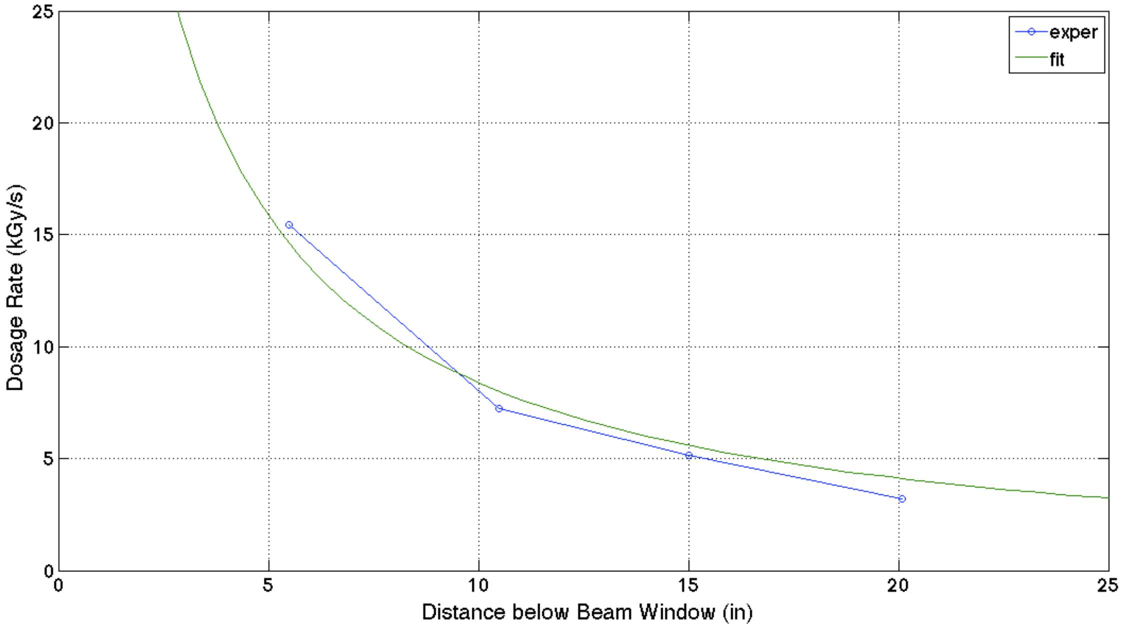


Figure 4 - ***E-beam dose rate vs window distance.***

When using the electron beam for processing oil, the desired effect is scission of long carbon bonds. The electrons carry enough energy to break molecular bonds, creating radicals and excited species. This initiates a chain reaction, which is then thermally propagated to ultimately break the carbon chains, reducing the viscosity of the oil. Too high a dose can lead to polymerization of the oil, which is undesirable. When a long hydrocarbon is broken, a reactive light radical and an unstable heavy radical are formed. The heavy breaks down further into another light radical and an olefin; the olefin initiates polymerization. Too high a concentration of olefins means the polymerization reaction rate is greater than the cracking reaction rate, leading to a decrease in light hydrocarbons and an increase in viscosity [16]. The beam also enables hydrogenation of the hydrocarbons if the oil is treated in a hydrogen-rich environment such as methane or hydrogen gas. The freshly broken carbon bonds will have a tendency to saturate with hydrogen and become more stable. Because the electron beam also raises the temperature of the oil while it is exposed, there are some thermal effects present.

Early Testing With Radiation

In the second half of the 20th century, the possibilities of harnessing atomic radiation were investigated. The new technology available held unknown promises and was applied as early as the 1950's to oil treatment. Early experiments dealt with low temperature irradiation using gamma isotope sources [21]. In 1962, a paper from the U.S.S.R. Academy of Sciences was published that tested the effects of an electron accelerator on vaporized hydrocarbons [17]. The test vehicle shares similarities with the system built during the research of this thesis, such as a thin metal sheet used as an airtight window for radiation to enter the system.

The testing setup was used to compare the effects of thermal processing and radiation thermal processing. In one experiment, gasoline with a boiling point of 200˚C and an isolated fraction of crude oil from the Tartar region with the same boiling point were used. Two samples of each were taken and processed through TC or RTC. The temperature of the treated products was varied, and boiling was monitored to observe the yield of the processing. Figure 5 below shows the results of this fractionation. In this graph, Lines 1 and 2 are from the RTC treated products, with 1 representing the crude fraction and 2 representing gasoline. Lines 3 and 4 are from the TC treated products, where Line 3 represents fractionation of the crude and 4 shows the treated gasoline.

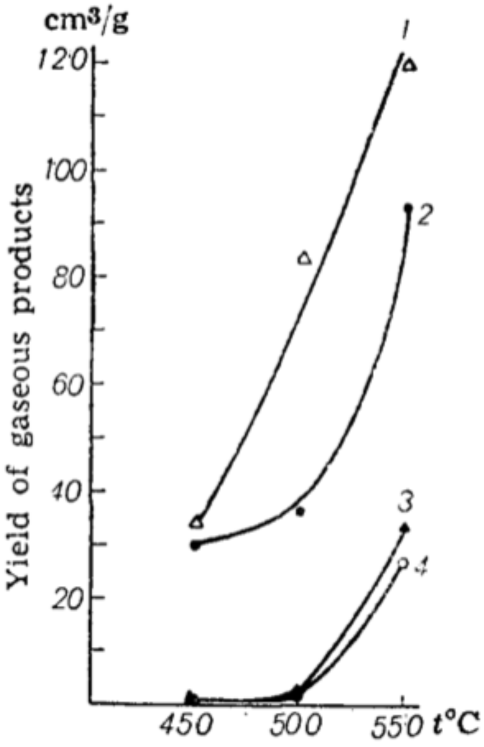


Figure 5 - ***RTC vs TC for gasoline and crude oil.*** **Reprinted from [17]**

As shown in Figure 5, the products of RTC have a significantly higher yield of lighter, gaseous hydrocarbons versus the products of TC. Also of note is that at a lower temperature of 450˚C, the yield for RTC treatment is greater than or roughly equal to those for TC at 100˚C higher temperature. For the lighter initial products, the properties of the liquid hydrocarbons remained similar; however, medium fuel oil was also processed and analyzed. The results of this processing, performed similarly as that behind the results shown in Figure 5, are below in Figure 6. The higher Line 1 represents RTC, and Line 2 represents TC processing of the fuel oil. In this case, the properties of the liquid hydrocarbons changed significantly, with RTC yielding 27% increase in gaseous products at 500˚C versus only 12% for TC at the same temperature [17].

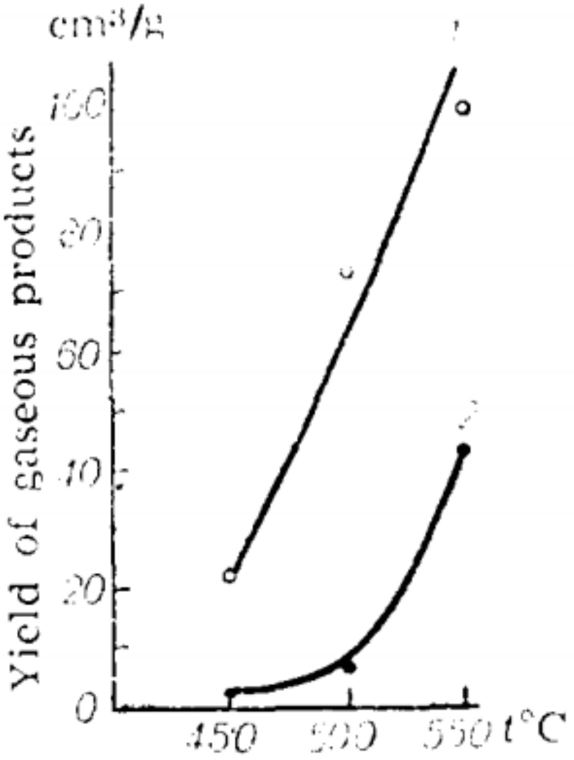


Figure 6 - ***RTC vs TC of medium fuel oil.*** **Reprinted from [17]**

From these results, there is promise of helpful benefits to using RTC on heavier oils. The cracking process was identified as having two major components, reaction initiation and chain propagation. The activation energy for initiation is much higher than that for propagation in TC, but RTC uses ionization to initiate the reaction with less energy. The study concluded that the activation energy for reaction initiation for RTC was roughly equal to that of reaction propagation in TC. It also found that RTC processing took less time than TC after initiation. Further investigation would help clarify these results, as this study did not include the effects of varying dose rate, and the 1-MeV accelerator could only produce a beam with 1 kW of power.

In the same time period elsewhere in the world, experiments were conducted using nuclear fission products and neutron radiation. The major drawback of this technique versus the use of an electron beam was the feedstock retaining residual radioactivity, but valuable results were obtained. In 1959, a process for hydrocarbon conversion using neutron bombardment was developed, using the air-cooled uranium graphite research reactor at the Brookhaven National Laboratory in New York. One key result of this research was the demonstration of a catalyst for hydrogenation becoming active below its effective temperature range. Hydrocarbons were hydrogenated to high saturation levels and the catalyst inhibited polymerization during exposure. It was found at this point that the same effects of higher temperature hydrocracking could be obtained with less energy expense [18].

Another study was performed in 1959 that directly relates to extra-heavy crude oil from the Athabasca tar sands. It was found that irradiation could be used to remove petroleum from petroleum-bearing sands, creating the possibility for new sources of oil to be explored. High levels of gamma radiation disturbed the molecular long-range forces that bound the petroleum to the sand, allowing it to be released. It also decreased the viscosity of the oil, making it easier to flow from the sand. The radiation source in this study was a cobalt-60 isotope, and in a proof-of-concept experiment, 500 g of oilbearing sand were irradiated to release 42 mL of oil. From this research, it was found that radiation was a useful tool in extracting oil from previously unusable sources [19].

The tendency of heavy crude oil to polymerize under long exposure to low dose rates of radiation was investigated in the 1980's and 1990's. In 1986, a heavy fraction of oil with a boiling point above 260˚C was mixed with hydrogenated coal and irradiated at room temperature. It was found that olefin bonds had increased after radiation, with further effects present after long-term storage. Over a period of two months, oxidation led to an increase in asphaltene concentrations from 7.2% to 16.7%. When products of coal hydrogenation were exposed to gamma radiation, a similar effect was observed. The yield of light fractions decreased from 23.1% to 14.5%, caused by a higher availability of olefin bonds and oxygen within the coal [20]. More recent studies would be performed that demonstrate the ability to avoid the formation of heavier species in favor of converting hydrocarbons to lighter fractions.

Recent Investigations

With the increasing need for new sources of oil since the turn of the millennium, extra-heavy crude oils have been a difficult alternative to use. The difficulty in processing these oils has motivated further research to find more energy-efficient methods, prompting a revisit of RTC experiments. Combining known progress made decades earlier with the more precise controls and analytical equipment available in the 2000's allowed researchers to better characterize the effects of electron beam exposure on crude oils.

As previously established, chemical kinetics of cracking can be summarized with two steps: initiation and propagation. Light radicals must be formed through dissociation to initiate a chain reaction that is carried out by excited molecules reacting with other radicals. The process of cracking is shown below, where R represents a radical, OL represents an olefin, and both H and RH are hydrocarbon species [21].

Recombination: $R + R \rightarrow H$

Disproportionation: $R + R \rightarrow RH + OL$

Dissociation: $R \rightarrow R_1 + OL$

Molecule Break: $R + RH \rightarrow R_2 + RH$

$R + OL \rightarrow$ Polymerization

In traditional thermal cracking (TC), the activation energy for initiation is 250 kJ/mol, meaning temperatures need to be in the range of 500 to 600˚C. The chain propagation step requires activation energy of 80 kJ/mol, meaning the entire process is very costly in terms of energy input. In radiation thermal cracking (RTC), the energy sufficient for initiation was below 0.4 kJ/mol through electron energy transfer [21]. This number is significantly lower than expected as a result of earlier testing, which believed the initiation activation energy to be two orders of magnitude greater. The propagation process is still thermally activated, but because of its lower required temperature, cracking temperatures for RTC are roughly 200˚C lower than that for TC [21]. Because of this, oil is heated to a set temperature before e-beam exposure begins. The fractional products of TC and RTC processing are shown in Table 1. As seen in the table, with a heavy crude feedstock, gasoline range organics (GRO) and diesel range organics (DRO) with lower boiling temperatures have a higher yield for RTC over TC.

Besides overall radiation dose, the effects of varying dose rate were studied. Earlier studies concluded that the cracking rate was proportional to the square root of the dose rate, based on an expected short life of radical species [16]. It was found, however, that the relationship was more proportional to $D^{0.5} + D^{1.5}$, where D is the dose rate [16]. This means that while the cracking rate would initially drop with increased dose rate, it would then come back. One note of concern with irradiation processing is that too high a dose can lead to polymerization [16]. This reduces the amount of light hydrocarbons created and can increase viscosity back to levels close to the feedstock properties. As shown in Figure 7, in the treatment of high-viscosity crude at different rates, the conversion factor of processed oil decreases after a peak. The slope of the two lines is the reaction rate, and the higher dose rate of 80 kGy/s clearly shows a higher initial reaction rate. The flattening of the curve occurs because of strong polymerization, but if the treatment is stopped before the dose gets too high, the same conversion factor can be achieved with significantly less time and energy than a lower dose rate [16]. From Zaikin and Zaikina, RTC applied to extra-heavy crude oils allows for efficient, reliable control of the composition of produced synthetic crude compared to more well-established refining methods. Combining a quicker path to desirable market products with nearly a 40% savings in energy makes radiation processing more than worthy of further investigation.

Table 1 - ***TC vs RTC product comparison.*** **Reprinted from [16]**

Boiling Temperature, ˚C	**Feed Fractions Mass%**	**TC Mass%**	**RTC Mass %**
<200	None	10	15
200 - 300	2	15	27
300 - 360	8	15	18
360 - 450	38	25	20
>450	52	30	10
Gases	None	5	10

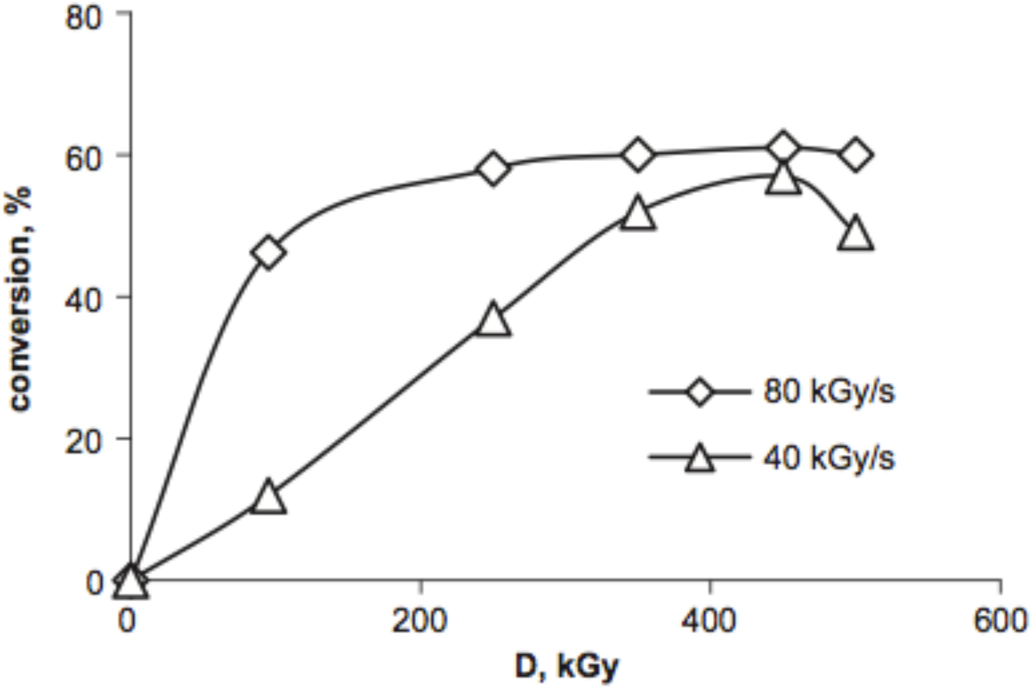


Figure 7 - ***Dose rate effects on conversion factors.*** **Reprinted from [21]**

Other aspects of electron beam processing of crude oils are also under investigation. The basic variable parameters are dose and dose rate, but different strategies have potential to yield the same results using less energy than what has been seen so far. The application of high shear to the oil during irradiation has been shown to better facilitate the conversion of crude oil to lighter hydrocarbons. If the oil is flowed within a controlled environment during treatment, that environment can be changed. In this research, an inert gas such as helium or argon was used to ensure the vaporized oil does not form a fuel-oxidizer mixture with air. The mixture would present a flammability hazard and was taken into consideration for safety. More beneficial for industrial use is a hydrogen-rich environment such as hydrogen gas or methane. These gases are available in a processing plant as head gas byproducts of other steps in refining crude oil. As the heavy oil undergoes RTC, hydrogen atoms will encourage hydrogenation of the carbon chains, leading to the production of more saturated hydrocarbons. Within this controlled gas method, the way the gas is supplied to the processing reactor has a significant effect on the results. Bubbling gas through the oil flow helps with mixing. The mixing further promotes hydrogenation, though it is desirable for the mixing to remain laminar from bubbling as opposed to a turbulent frothing of the oil [21].

Other e-beam strategies include pulsing the beam to achieve the same overall dose, varying oil flow from laminar to turbulent, and changing the shear rate of the flow. However, preliminary findings show that pulsing can lead to more polymerization since that occurs whether the beam is turned on or off, while cracking only occurs when the beam is on. It is also expected that turbulent flow will yield less favorable results because flow interruptions increase local viscosity of the oil [20]. Because heavy oil feedstock has widely different properties depending on its location of origin, an ideal processing system should allow control of dose rate, flow residence time, gas type and delivery, and oil temperature to accommodate oil from any source.

Economics

New technologies often need significant financial support to become working solutions. If the idea cannot make financial sense, especially when compared to conventional methods, it has a high risk of fading into obscurity. With past experiments showing the effectiveness of heavy crude processing through electron radiation, a simple economic analysis was performed to solidify the value in electron beam treatment.

Because the electron beam requires electrical energy, operating expense (OPEX) is dependent on the energy cost in dollars per kilowatt-hour. This cost is a commercial rate that is then converted to units of dollars per kilojoule for the purposes of the evaluation. For 1 kg of oil, if the required dose is 20 kGy, then 20 kJ of energy input is required for processing. Likewise, if the dose is 1 MGy, then 1 MJ must be administered. Scaling this out by how many kilograms of oil are in one 42-gallon barrel gives the OPEX per barrel. Capital expense (CAPEX) is determined by the size or production capacity of the hypothetical facility. For the purposes of this evaluation, large-scale facilities are considered, using large commercially available 500-kW accelerators. CAPEX takes into account both the accelerators and the physical facility and shielding required for operation. CAPEX is also broken down to a monthly payment using a simple interest formula in Excel. These numbers are scaled through a range of total dosages to determine cost per 42-gallon barrel and production capacity in barrels per day.

In this analysis, electrical energy is priced at a rough commercial average of \$0.09/kWh, although this is generally an overestimation. A production site dealing with extra-heavy crude oil will generally have its own power production because of its remote location, so energy costs may be significantly lower than that commercial rate. Density of the extra-heavy crude at room temperature is taken as 1,000 kg/m^3, meaning a 42-gallon barrel, or 0.2082 m^3, holds 208.2 kg of oil. Dosages range from 20 to 1,100 kGy to capture the full range of what may be effective. At 20 kGy, OPEX is \$0.08/bbl, while at the other end of the spectrum the cost rises linearly to \$4.37/bbl. CAPEX is shown below for facilities ranging from 500 kW to 3 MW in Table 2. For a given facility size, as the dose required increases, cost per barrel increases and the total barrels per day that can be processed decreases. However, cost per day remains constant given a facility. Because production was represented on a daily basis, total cost per barrel was calculated taking into account the monthly CAPEX cost divided across thirty days in a month, with promising results. For a given dose, cost per barrel decreases as facility size increases, down to \$0.19/bbl for a 3-MW facility running at 20 kGy. This scenario yields a production capacity of 62,247 bbl/day. On the other end, a 0.5-MW facility processing oil at 1,100 kGy dose will cost \$12.91/bbl and can only produce 247 bbl/day. In the State of Texas, an average oil well will yield 19.4 bbl/day [22], so the hypothetical small facility can only handle feed from 10 wells. Figure 8 below compares the cost of treating one barrel of oil with the number of barrels per day, which is directly dependent on dose required to treat the oil as desired. Both axes are on a logarithmic scale to more clearly show the data.

Table 2 - ***CAPEX analysis.***

Facility Size (MW)	CAPEX (Million $)	CAPEX ($)
0.5	$8	($63,263.49)
1	$11	($86,987.30)
1.5	$14	($110,711.11)
2	$20	($134,434.92)
3	$26	($181,882.53)
		($/mo, 5% interest for 15 yrs)

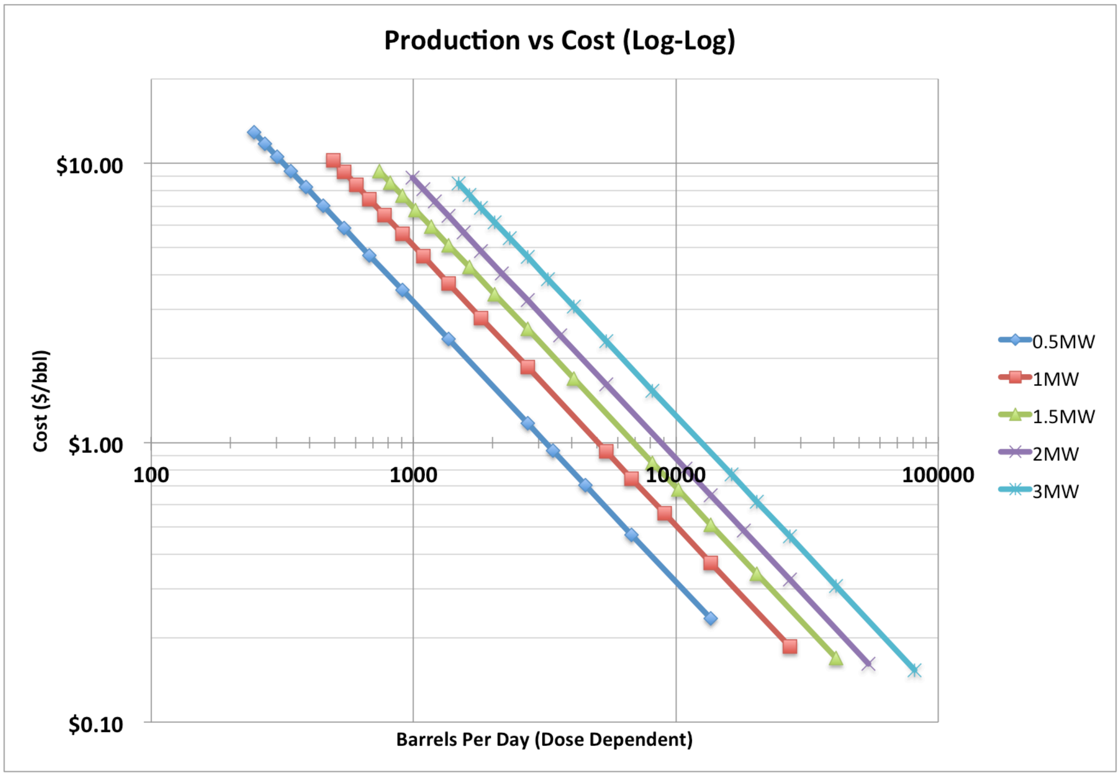


Figure 8 - ***Total cost analysis.***

From this graph, it can be seen that a wide range of operating parameters can make a strong financial case. Although the cost of processing oil at 1,100 kGy is nearly $16/bbl, that much energy input has been shown to go well past peak processing. Using Figure 7 as a reference, a peak at roughly 400 kGy for different dose rates is selected. At this level of energy input, processing costs range from $4.69 to $3.08/bbl. With the curve for the higher dose rate, if processing is done to 200 kGy, products are of similar quality but the cost range changes to $2.35 to $1.54/bbl. The percentage of the total cost dedicated to OPEX is constant regardless of barrels produced, but varies between facilities. OPEX for smallest facility at 0.5 kW is responsible for 33.9% of the total cost, while the largest facility at 3 MW shows OPEX taking 51.6% of the total cost per barrel. This is because the CAPEX on a larger scale facility is amortized by its extra production capacity. These numbers for total cost are small compared to the price of a barrel of oil, meaning e-beam processing is financially feasible.

The total cost per barrel can only be justified if the increase of value per barrel yields a sufficient profit margin. Prices of crude oil decrease for heavier and sourer specimens. Using Figure 9 as an example, the prices of Venezuelan Boscan crude oil can be compared with those of Texan WTI and Mexican Maya crude oil. Boscan oil is a heavy, sour crude with an API rating of 10.6 degrees and roughly 5% sulfur composition by mass. It is similar in both properties to Oil #1 and Oil #2 considered in this research, though they are slightly denser and more viscous, and would cost even less on the market. WTI is a light, sweet crude with an API rating of almost 39 degrees and sulfur concentration below 0.5%. Maya sits roughly in the middle of the two as a medium crude oil [1].

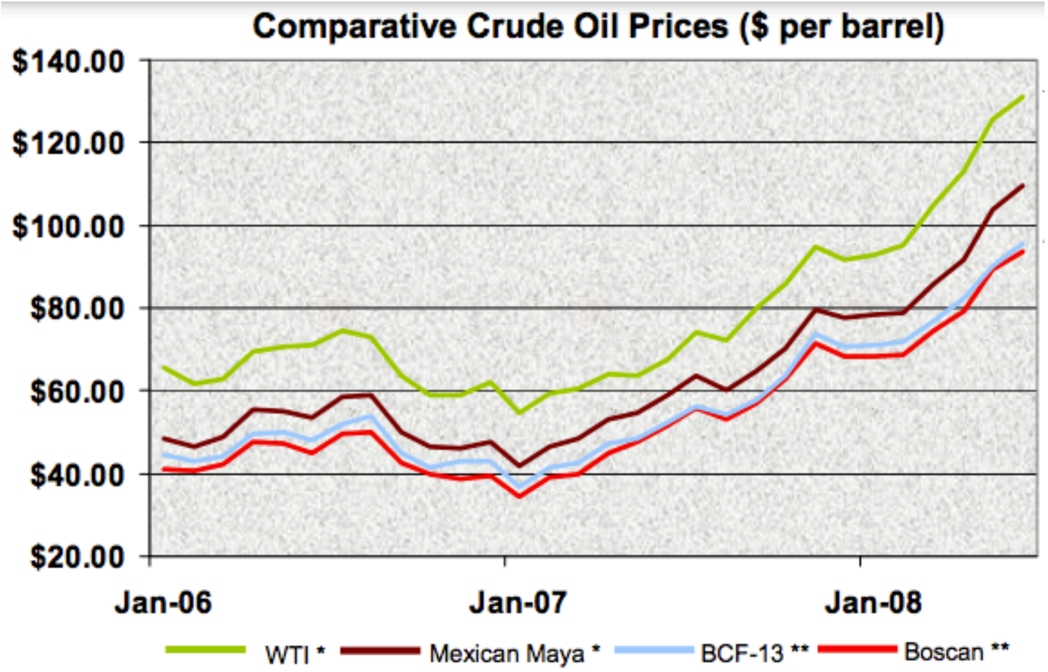


Figure 9 - ***Boscan-WTI crude oil price spread.*** **Reprinted from [1]**

The price spread over this two-year-long period between Boscan and WTI is a roughly constant $20/bbl difference, regardless of fluctuations in the market. Maya again sits in the middle of the two with around $10/bbl of spread between the other two. If the electron beam treatment can be used to upgrade oil with qualities similar to Boscan to a state more in line with Maya, the oil could then be sold for $10/bbl more than the heavier original crude. If it can be converted to a much lighter crude such as WTI, the profit margin increases by another $10/bbl, although this is a highly improbable outcome. With processing carrying a total cost of less than $6/bbl, the net result is a gain in profit of between $5 and $15/bbl. The extra-heavy crude considered in this research has an even lower API rating than Boscan, which means its price will be lower and the profit margin increases again. Combining a quicker, low-cost path to desirable market products with nearly a 40% savings in energy makes radiation processing more than worthy of further investment.

For this price difference in heavy versus medium crude of $10/bbl, a theoretical facility is considered. This pilot facility has a 1-MW capacity that can operate for any dosage between 20 and 1,100 kGy. Figure 10 below shows a graph of required dose versus production capacity and cost per barrel. As dose is increased, total cost per barrel increases and barrels per day decreases. If processing increases the value of the oil by $10/bbl, it must cost less than $10/bbl to perform that process. Not taking into account the cost of transporting the oil to the facility, it is shown that the maximum dose the facility can operate at is just under 1,100 kGy to break even, with a corresponding production capacity of around 500 barrels per day. Therefore, any dose requirement below 1,100 kGy will yield a profit and more than 500 barrels produced per day.

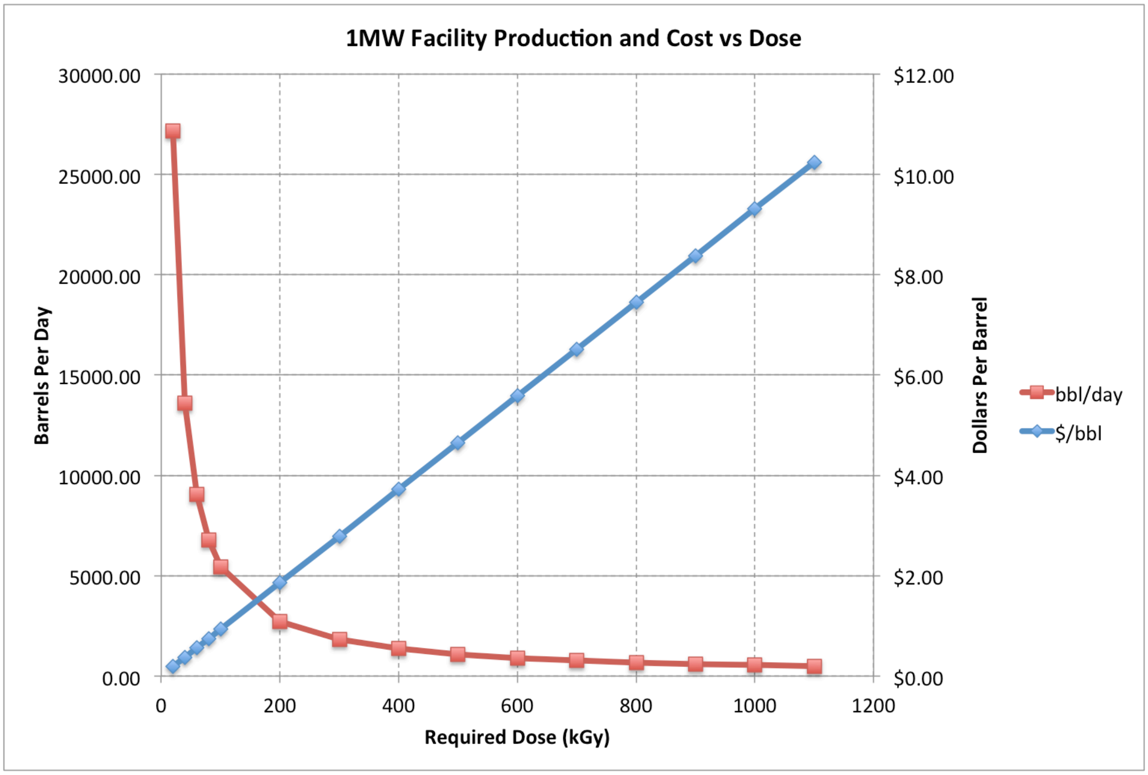


Figure 10 - ***Pilot facility economic analysis.***

Oil is a complex, non-uniform resource with properties depend on where it originates. As exploration turns to extra-heavy crude, conventional refining practices become more costly and wasteful of energy. New processes under development tailored to treating extra-heavy oil have the potential to outclass some traditional methods, saving time, energy, and money. Radiation thermal cracking by use of an electron beam has been shown to yield favorable results with 40% less energy than thermal cracking alone. Radiation dose required for a high conversion factor has been shown to fall within a reasonable range, with a peak before dropping off. At the estimated dosages, processing carries a total cost of between $2.01 to $6.15/bbl, including the cost of building the treatment facilities and running on residential electricity rates. Considering the change in density of oil after processing, the same mass prior to processing would occupy more volume afterwards, meaning more barrels are produced than are used as feedstock from a volumetric standpoint. These savings translate to a more viable profit margin for the industry, enabling new oil reserves to be tapped. These new reserves bring added stability in the energy industry, and in energy prices for consumers.

CHAPTER III

DEVELOPMENT OF SOLUTIONS

Chapter Overview

As with any engineering project, the start of creating a solution began with a set of goals. Vague parameters were set, requiring much further development before a final form could take shape. The full process of sound engineering judgement was employed from the start. With lateral and vertical thinking, a solution constrained by feasibility while remaining effective in completing the task at hand was created.

The initial instruction for this project was to create a flow loop. The loop would have to be safe for use with oils and solvents, and would have to be operable at extreme temperatures. Other initial constraints included extreme viscosity working fluids, the procurement of a positive displacement pump that would have jacketed heating and cooling, available materials, cost of new items, and time. One of the most significant design constraints is that the finished product must be operable within an e-beam vault, while under direct exposure to the beam. With no other information given, additional parameters and design constraints were sought out and organized into the first vision of the finished product. Limited knowledge of prior, similarly themed projects guided the initial design stages. Once measurements were taken of the intended operating area, along with dimensions of significant components to the flow loop repurposed from older projects on hand, space limits were defined. Thinking forward, it was chosen that the framework be modular and highly customizable, such that any necessary design alterations could be performed at haste.

Besides the flow loop itself, to support and maintain safety of the project, auxiliary test vehicles were developed. Although every component is of importance, the most significant of these vehicles is the mobile lab test cell. Other vehicles necessary to the project include the controls cart, fire extinguisher cart, and oil decanter cart. Starting from a vague expectation, view of a solution steadily came into focus until all of the systems developed meshed together. The finished product is a largely seamless solution with safety as paramount, and a reliable foundation for future development.

Defining Parameters

When starting this research, the goals were left vague as to not limit possibilities for how to meet those goals. As work progressed, parameters were defined, specific requests were made, and the first steps towards fabrication were taken. To start, the components of a flow loop were identified to include a container for loading a sample, a pump for moving the fluid, a reactor for the fluid, and then a three-way valve to direct flow back to the loading container or to a collection container for processed fluid, later changed for a more simplistic design. The specific components and the shape of the system would follow as requirements were clarified.

The first component to be specified was the pump. Properties of the working fluid were given that affect all parts of the loop, but if the pump is not compatible with the loop then the system will not function. Parameters were given as high viscosity oil that would be heated in excess of 300˚C to be flowed at 5 gpm at pressures up to 100 psi.

Because of the notable variation in viscosity of the oil at different temperatures, the pump itself had to be a positive displacement type. These pumps can handle a wider range of viscosities with somewhat constant flow versus a centrifugal pump. Preferentially, it would have a heating or cooling ability and run on household 120-V AC power. Multiple companies' product portfolios were researched, with Viking, Haight, and Oberdorfer having models of gear pumps that would suit the needs of the project on a condition. Because of the extremely high viscosity of the working fluid at room temperature, yet low viscosity at elevated temperatures, it would be nearly impossible to have a pump that could make a reliable seal for all viscosities and handle the high temperature requirement.

One idea was to have a pump for high viscosity fluid in series with a pump for low-viscosity fluid, but the tighter tolerances of the low-viscosity pump would not allow the thicker fluid to be forced through without having a significant total pressure loss. Thus, it was decided that a single pump for lower viscosity fluid would be used. This pump would not be operated if the fluid was cold to prevent damage to the pump or the motor. The high cost of pumps built to withstand sustained high temperature was somewhat prohibitive; however, there was a pump found in the lab from previous experiments that nearly exactly fit the needs of the project. A Chemsteel pump from Oberdorfer, with stainless steel gears built to withstand high temperature and sealed for use with oil, was paired with a ¾-hp, three-phase electric motor also found in the lab. The motor and pump work together with an Eaton VFD procured to provide variable flow control. The VFD runs on single-phase 120-V AC power, meaning a common household outlet will provide the necessary power. Physical measurements of the motor and pump system were taken for modeling, and a correlation between flow rate and VFD control frequency was made using water and mineral oil. A mock up flow loop was built using a stainless steel container and braided steel lines going to an older flow reactor, pictured below in Figure 11. A graduated container was held in the flow and the time necessary to fill to 600 mL was recorded. VFD frequency was increased until the flow was too violent to be contained in this manner. This allowed for verification that the pump could handle the desired flow rates, since the viscosity of mineral oil at room temperature is very similar to the viscosity of Oil #1 at 160˚C and Oil #2 at 200˚C.

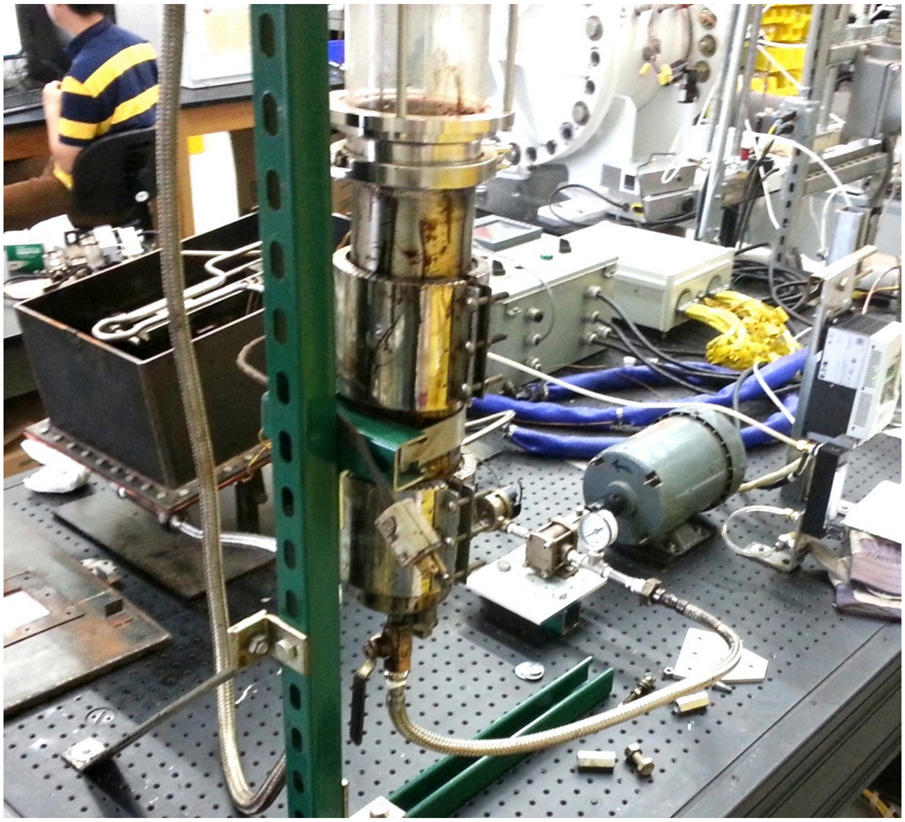


Figure 11 - ***Mock up flow loop.***

Because the pump would not be operable unless the fluid was heated, the container that would hold the oil sample would have to be heated as well. Because of the heat and the possibility for chemical leeching, a PVC container was quickly ruled out of the question. The container used for the mock flow loop proved to be a useful choice, and was made from 4-inch-diameter stainless steel with a sanitary fitting on the top and male pipe threads on the bottom exit. It already had a pair of heaters clamped on from a previous experiment, which would prove to be sufficient for heating the sample oil to desired temperatures. The lid was made from a sanitary fitting plate and had holes drilled for recirculating flow, a gas inlet, and a thermocouple. Devising a method of measuring the fluid level inside this container was attempted, but the equipment found either could not handle the temperatures, was too sensitive to electron beam interference, or was too cost-prohibitive. This container was labeled as the sample oil container (SOC) in the piping and instrumentation diagram (P&ID) made for the system.

After the pump and container were specified, the intended operating area was made known and visited to take dimensions in order to build the support structure for the loop. These dimensions were recorded in a SolidWorks drawing shown below in Figure 12. At the Texas A&M EBRF, there is a conveyor that goes down a vault made with concrete walls, whose thickness ranges between 4 and 8 feet, to the electron linear accelerators. The closest one to the access door in the control area is the tower LINAC, whose horn protrudes down from the ceiling to 23 inches above the conveyor belt. This is the accelerator used in this research, so dimensions of the vault were measured to make a system that would fit inside. It was found that the path alongside the conveyor was 2 feet wide at its minimum width, there was roughly 57 inches in allowable height, and the rails on the conveyor belt were just over 29 inches apart. On the floor underneath the horn is a pad with cooling coils run through to keep the floor from overheating when the beam is turned on. This pad measures 27 inches wide from the outside of the cooling coils on each side, and must be straddled by the support structure built for the flow loop. The maximum length allowable of the structure was 63 inches between the walls going down the vault because of equipment mounted inside. Finally, the structure had to be narrow enough to pass through doors that were 29 inches wide. The structure could not rest on the rollers of the conveyor belt since they have to be running when the beam is on, so the reactor box that goes underneath the beam horn would have to be cantilevered on the structure and possibly resting on the outside rails of the conveyor system.

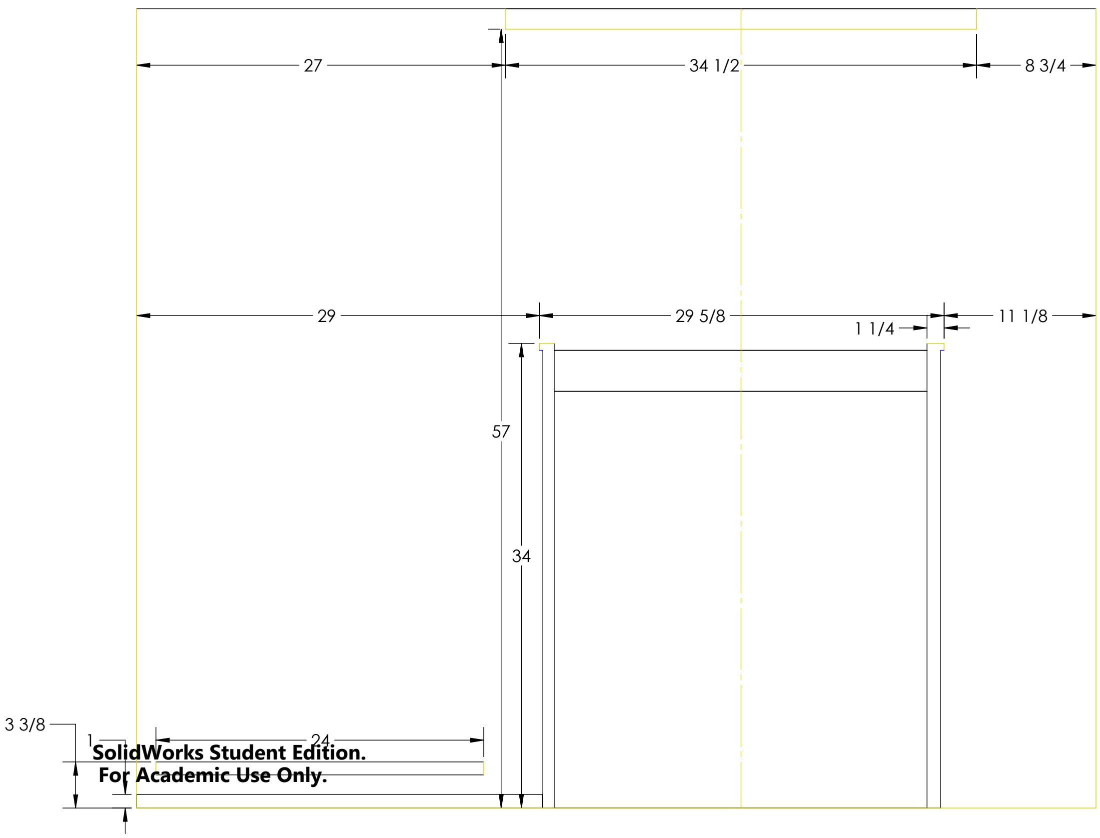


Figure 12 - ***EBRF vault profile.***

With all these numbers recorded, brainstorming led to the initial design of the framework. Previous test apparatus found in the lab were built out of various types of strut, the more versatile of which was Unistrut. It was chosen to build most of the framework from Unistrut pieces because of its ease of modification and ready availability. Slotted holes opposite the open side of the strut allow for hard supports for heavier items, while the open side serves as a track for finer adjustment. The original design for the structure made in SolidWorks is shown in Figure 13. The dimensions of the pump and motor, SOC, and the old reactor box were used to help visualize how well the components would fit within the size constraints.

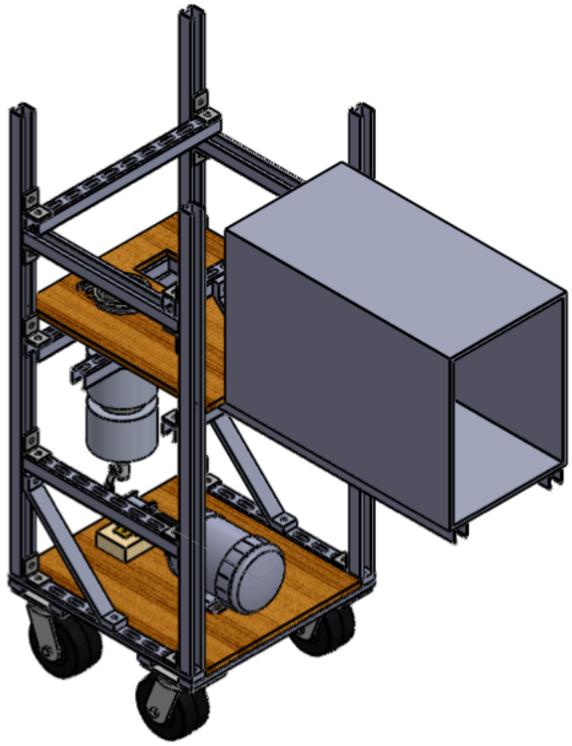


Figure 13 - ***Initial CAD model for oil cart.***

The original design had a squared base made from thinner strut members on large caster wheels for ease of transport, and its profile took up as much of the open space in the vault as possible. In the interest of stability, a wider base was chosen. The lengths of all strut members were measured for a weight estimate with the goal of keeping the total weight of the structure under 200 pounds. If steel was used for all members, the framework alone would weigh 98.1 pounds. It was decided to build the base plate out of steel strut and use aluminum for the uprights and reactor support members, since that would reduce the weight by 44% to 54.6 pounds. The structure housing the flow loop was named the oil cart, and as fabrication of the cart began, other parts of the system could be determined.

With the structure taking shape, the design of the reactor began. This is the most critical component of the whole cart, since the way the oil is handled underneath the e-beam horn determines the results of the entire experiment. On the outside, the box is 4 feet long, taking up as much length as the long cantilever supports on the cart gave. As with the oil cart, the reactor box was designed with variability in mind. The oil flow channel is mounted to the wall of the box through a breadboard of threaded holes to change the angle of the channel with ease. Special attention was given to drainage of the box during a lab meeting. In previous designs, the bottom of the box simply sloped down into a basin with a drainpipe. This caused oil to sit underneath the channel when it became blocked up from low flow. This is bad for reliability of experimental results, since the electron beam passes through the oil in the channel, the channel itself, and hits the pool of oil at the bottom again, overexposing the sample. The new box was designed to take any oil that had splashed out of the channel and drain it to the outside walls, outside the width of the electron beam, and then down to the box exit. This design is depicted in Figure 14. The exit on the new box was chosen to be a large, 3-inch diameter drain with a sanitary fitting on the outside for ease of changing processed sample collection tanks. This collector tank is known as the processed oil container (POC) in the P&ID.

Figure 14 - ***Processing box drainage surface.***

The lid of the box is a thin sheet of metal bolted between the box flange and a gasket to prevent gas from escaping. The lid is thin enough that the e-beam energy passes through it and into the oil stream. A diagram is shown in Figure 15 to further detail the physical interaction with the beam. The box, lid, and channel are constructed exclusively from aluminum to avoid overheating and transferring extra thermal energy to the oil. The lower density of aluminum lends to its higher transmissibility for e-beam radiation when compared to stainless steel. A beneficial side effect of this is that the cantilevered weight is greatly reduced compared to steel. In case of any oil pooling at the bottom, the aluminum would not overheat the stagnant oil as much as if it were steel.

Two windows were built into the side of the box to allow for observation of the flow channel, and multiple pass-throughs were made for instrumentation and gas.

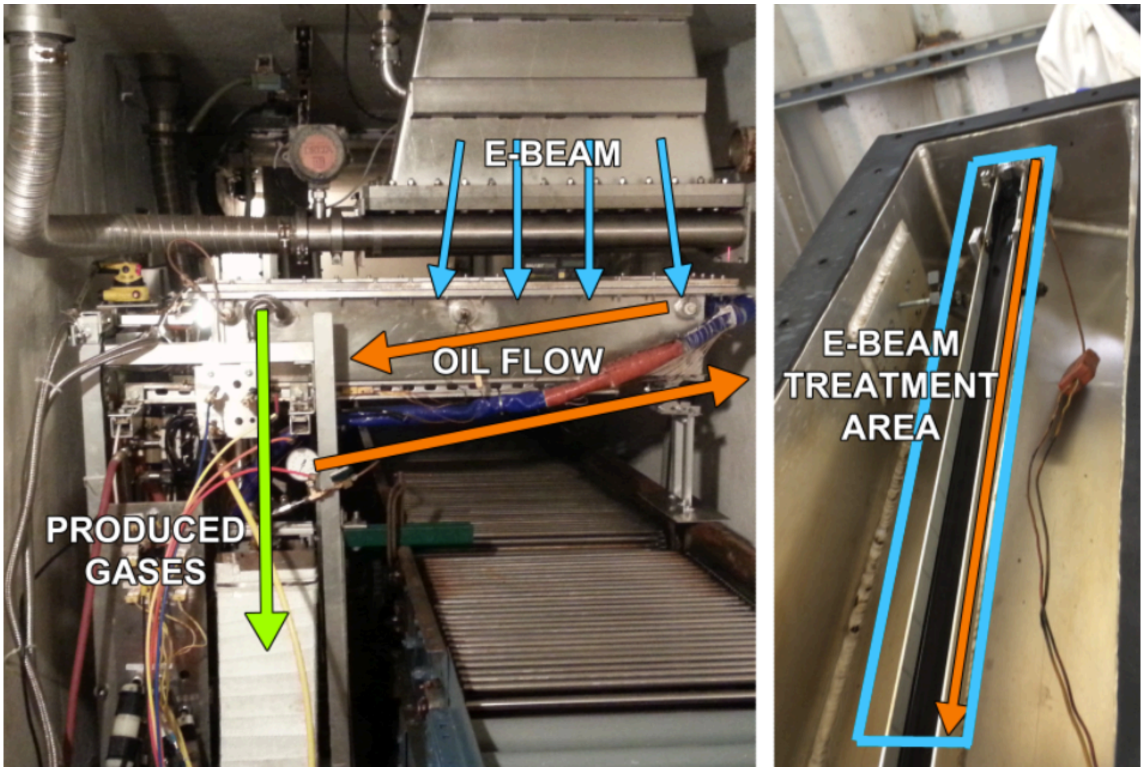


Figure 15 - ***Reactor box interaction diagram.***

A large gas condenser was required to accommodate the volume of gases produced by processing oil in expected quantities. The condenser was made from stainless steel sanitary pipe with a cap on the bottom and valving on top. It was decided that the condenser would use a liquid nitrogen bath, as used in other condensers in the lab, versus a dry ice interface. The liquid nitrogen is easier to handle when transferring to the condenser bath, and the Dewars necessary were already in possession at the lab.

Lastly, a remote-controllable valve was desired for control of flow timing. The issue with electronic equipment on the cart is that exposure to the e-beam will damage and destroy internal circuitry. Therefore, the valve chosen was a pneumatically driven, electrically controlled unit. The electrical control is analog on or off; when the cord is plugged in, a compressed air line is opened to open the valve, and when unplugged the air is closed and pressure is released, closing the valve. The valve is also be useful in case of an emergency to stop flow, since its normally-closed configuration means a loss of power stops the oil flow.

Fabrication

When the initial design was finished and components were designed, chosen, and sized out, the process of making the oil cart come together off paper began. With the lengths of strut previously calculated, the building materials were ordered. While the aluminum strut was in shipping, a previous lab apparatus was deconstructed and salvaged for its diagonal braces, angle brackets, bolts, square channel nuts, and caster wheels. The wheels were still easy to roll, so it was decided new ones were not needed. The base was constructed with special care given to ensuring the angles were square. A pair of 30-inch members were used on each side to hold a pair of wheels together. These two wheel rails were then joined together with three, 22-inch long transverse members. The middle member provides support for the motor and pump. Once the aluminum strut arrived, it was measured and cut with a chop saw in the USB lab's machining area. The grinder was not effective on aluminum because of its harder wheels made for steel, so a hand file was used to clean the cut edges and reduce the chance of skin cuts during handling of the cart.

During assembly, flow loop components were test fitted and mounted while they were more accessible. It was decided to orient the system so that components such as valves and the condenser bath were accessible in the e-beam vault. The motor and pump were both mounted on vibration-damping rubber pads to reduce wear on the frame. Flow components were joined by flexible braided lines or tubing to reduce interference and fitment issues with pipes. Stainless steel was used wherever possible because of its corrosion resistance; the e-beam rapidly oxidizes more reactive metals under exposure. With all lines in place, fiberglass-insulated heaters were installed between the SOC and the reactor box. The pump and flow valve were wrapped thoroughly because of their extra mass around the flow region. This reduces the likelihood of a flow blockage in the system, which is a potential fire hazard. The insulated heaters were wrapped with additional high-heat-resistant silicone insulation to protect against burns. A panel was made to organize all electrical connections for the heaters, the motor, and the valve. Another panel was made to organize gas inlets for backfill, bubbling, and purging lines. The oil cart is pictured below in Figure 16 inside the e-beam vault. At this point, the cart was at an early stage in need of refinement. More modifications would come in the future during shakedown testing, but with the cart at this state of completion, supporting systems took priority.

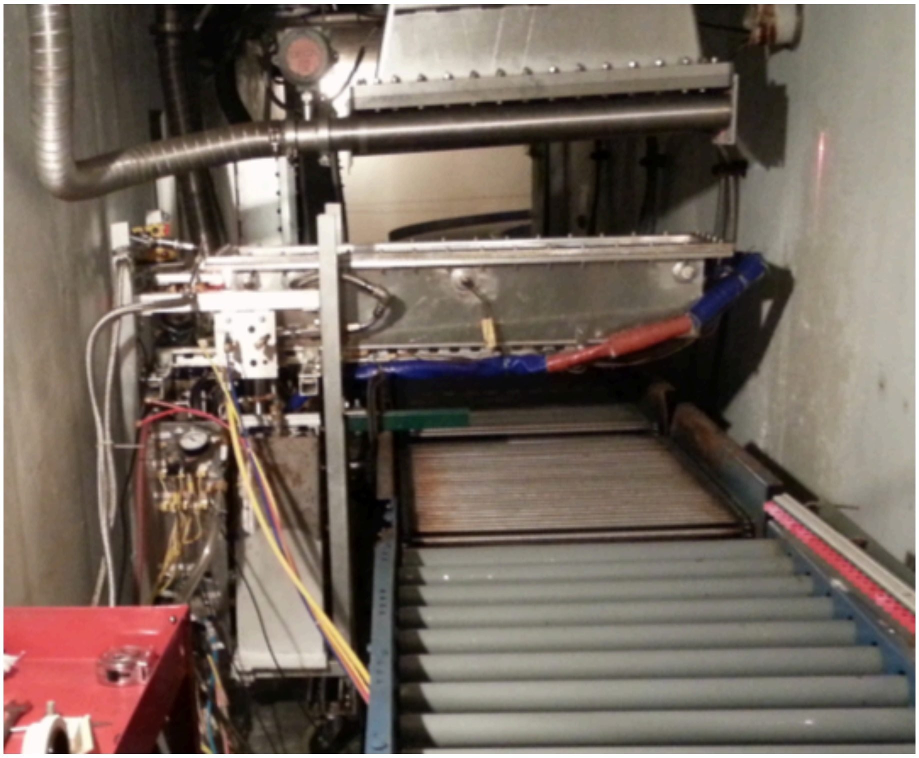


Figure 16 - ***Early oil cart at EBRF.***

Mobile Test Cell

The most important supporting system for this research is the mobile test cell. In order to do shakedown tests of the oil cart, the oil must be heated. When the oil is heated, it gives off harmful fumes that need to be controlled with proper ventilation. Because of the scale of the oil cart and the volume of oil being handled, a walk-in fume hood is required. However, in the timeframe of this research, it would not be possible to have one installed in the new laboratory. Therefore, plans to build a mobile walk-in fume hood were made, with a generator providing the added benefit of being able to conduct experiments on remote location.

The foundation of the mobile test cell is the flatbed trailer on which it was built. After researching multiple brands' product portfolios, the BigTex 14OA trailer was chosen; it is an over-axle flatbed with a 20-foot long, 8.5-foot wide deck. It has a class IV bumper-pull hitch as opposed to a gooseneck system to avoid extra insurance costs and paperwork. The 14OA weighs 3,510 pounds empty and has a GVWR of 14,000 pounds with two 7,000-pound axles in tandem. One crucial point to the entire cell is that the trailer itself has not been modified and retains Texas DOT legal compliance, so it is inspected and registered for use on the road. The certification document from the fabricator is shown in Appendix C. Welded to this foundation is a K-Line cargo container measuring 8.5 feet wide, 8 feet high, and 20 feet long. The Corten steel container weighs 5,180 pounds, and has a gritty, oil-resistant, high grip floor coating sprayed inside. There are also two rails of slotted Unistrut running the length of the container inside to help with securing equipment for transport. This union of container and trailer serves as the groundwork for the technical parts of the test cell.

When creating a fume hood out of the container, the National Fire Protection Association's NFPA 45 guidelines for 2015 were adhered to. This defines ventilation and lighting requirements, largely concerned with spark-proofing permanently mounted fixtures. There are two ventilation units attached to the container to provide a sufficient face velocity of at least 80 ft/min. Each is powered by a ¾ hp electric motor, and together they have a certified flow rate of 95 cfm and generate a certified face velocity of 120 ft/min.

Airflow testing was conducted after the trailer was delivered from Exosent, the fabrication company responsible for welding on the container, generator, and ventilation fans. A swing-vane anemometer was used to measure airflow at different distances from the door with both fans operating and with just one or the other. Both fans running produced an average face velocity of 131 ft/min, while just the larger fan running produced an average of 91 ft/min velocity. When running the small fan only, average velocity drops to 76 ft/min because of backflow in one area of the container. The results of these three tests are shown below, respectively, in Tables 3, 4, and 5. If the backflow in the last run is omitted from the average, it rises to a sufficient 86 ft/min. The small fan was originally intended to have a three-speed control system, however upon difficulty making a weather-tight electrical box outside the container and the lower average face velocity, it was decided to forego that route for simplicity. It was decided to permanently mount the anemometer in the region that experienced backflow as a safe way to ensure proper ventilation in the trailer.

Table 3 - ***Generator power - Both fans on, small fan 100% speed.***

Distance from door	**Upper-Left (ft/min)**	**Upper-Right (ft/min)**	**Lower-Left (ft/min)**	**Lower-Right (ft/min)**	**Average (ft/min)**	**Center (ft/min)**
1 ft	150	100	140	150	135	300
5 ft	110	120	130	150	127.5	200
10 ft	110	100	140	140	122.5	150
15 ft	150	130	130	150	140	150
Overall Average (ft/min)					131.25	

Table 4 - ***Generator power – Large fan only.***

Distance from door	**Upper-Left (ft/min)**	**Upper-Right (ft/min)**	**Lower-Left (ft/min)**	**Lower-Right (ft/min)**	**Average (ft/min)**	**Center (ft/min)**
1 ft	90	40	200	110	110	160
5 ft	100	100	90	90	95	90
10 ft	70	70	70	110	80	90
15 ft	80	70	80	90	80	80
Overall Average (ft/min)					91.25	

Table 5 - ***Generator power – Small fan only 100% speed.***

Distance from door	**Upper-Left (ft/min)**	**Upper-Right (ft/min)**	**Lower-Left (ft/min)**	**Lower-Right (ft/min)**	**Average (ft/min)**	**Center (ft/min)**
1 ft	80	-70	90	80	45	70
5 ft	80	60	70	110	80	100
10 ft	90	80	80	100	87.5	80
15 ft	80	100	80	100	90	90
Overall Average (ft/min)					75.625 (85.83)	

Lighting systems for the mobile test cell were also developed in compliance with NFPA 45. Lighting fixtures must vent to the exterior of the fume hood, so the lights are composed of fluorescent desk lights enclosed in a 5” acrylic tube whose ends stick outside the top of the container. Figure 17 gives a better visualization of the lighting system. With this configuration, all outlets and potential sources of electrical sparks are outside the environment of the fume hood. Power cords are routed up to the roof of the trailer to a length of 2-inch diameter electrical conduit for safeguarding from the elements. The conduit runs to the front of the trailer, and inside the conduit is an outdoor-rated extension cable with inline outlets. The cable comes out at the front near the ventilation fans for access to the mounted generator.

Figure 17 - ***Mobile test cell interior.***

Mounted to the front of the trailer is a generator to provide power for remote operation of the trailer's lighting and ventilation systems, as well as experimental equipment. The generator is a Generac GP17500e, producing 17.5 kW of power with a 992 cubic inch v-twin gasoline engine, weighing in at a total of 390 pounds. The generator has an idle speed control to use less fuel when less power is called for; full throttle consumption is 3.2 gallons per hour. With a myriad of outlets, the generator is capable of providing both 120-V and 240-V power in single- and three-phase configuration. The mobile test cell is shown with all equipment mounted in Figure 18 below. The container is kept at the loading dock of the USB, where vinyl curtains were installed to reduce excess load on the USB building HVAC systems from keeping one bay door open to access the inside. The trailer is parked close to the building to prevent as much draft as possible around the container, and blankets are used to seal the air gap at the bottom of the rolling door when closed so the ramp can be left tilted down.

Figure 18 - ***Mobile test cell exterior.***

From Exosent, the total weight of the mobile test cell with all permanently mounted equipment is 9,170 pounds, meaning an additional 4,830 pounds of testing equipment can be added and remain within legal weight limits. The construction of this mobile test cell allows experiments to be run without risk of exposure to hazardous fumes. Testing can continue in case of a power blackout with electricity from the generator, or while away from the lab at another location. Any spills that may occur are contained and easy to take care of with an absorbent cleaning compound. While not in use, the trailer has locks on both the rear doors and on the receiver hitch to prevent tampering or theft.

Support Systems

While the mobile test cell is the most important support system for the oil cart, the experiment would not be possible without other auxiliary systems for safety and control. These auxiliary systems are all cart-based for easy transport and high adaptability to different requirements and new equipment. First, a controls cart was made to facilitate remote operation of the oil cart. Next, a fire extinguisher cart was made to suppress any fires on the oil cart. Finally, an oil decanter cart was made to allow for the safe dispensing of oil samples at high temperatures. These systems work together with the oil cart to form a multi-faceted engineering solution and enable future research.

The controls cart is based on a welded steel cart procured from McMaster Carr, unlike other test apparatus constructed from Unistrut. The cart has two shelves, each of which can support 1,800 pounds. While that capacity is not used in full in this guise, it means the cart is more stable with the equipment that is loaded on. The top shelf is used for feedback and controls. A DAQ receives data from thermocouples and valve positioning, and sends it to a laptop that is hooked up to a mounted monitor that shows a LabView VI front panel. The VI controls heater cycles, valve state, and has a soft killswitch in case of emergency such as a fire. Next to the DAQ is a monitor for video and audio feed. This helps researchers see what is going on inside the vault, such as time of beam exposure, or if there are any major leaks or indications of fire. It also allows communication when running pre-test checks to ensure controls are functioning properly. Mounted beside the monitor is a switchbox controlling multiple electrical outlets in the vault, the most important of which are the two fire extinguisher switches. These two switches have lights to indicate whether they are on or off and are meant to be cycled as needed in case of a fire. Also on top is a box for the motor VFD, allowing researchers to remotely control motor speed as needed, while minimizing exposure to electrical terminals. Lastly, an array of large-digit thermocouple displays is mounted for quick reference.

The bottom shelf is used for equipment that is not part of the control and feedback interface. Multiple surge protectors are on the bottom, one of which has a battery backup in case of power failure. This allows data to be saved and proper emergency shutdown procedures to be followed. There is also an electrical switchbox for heater control that is controlled by the VI. A 4-gallon electric air compressor is mounted on the bottom shelf as well to operate the pneumatic valves. On the side of the cart, underneath the push handle, is a panel that supports rotameters and flow regulators for backfill, purge, and bubbling gas. The controls cart is pictured below in Figure 19.

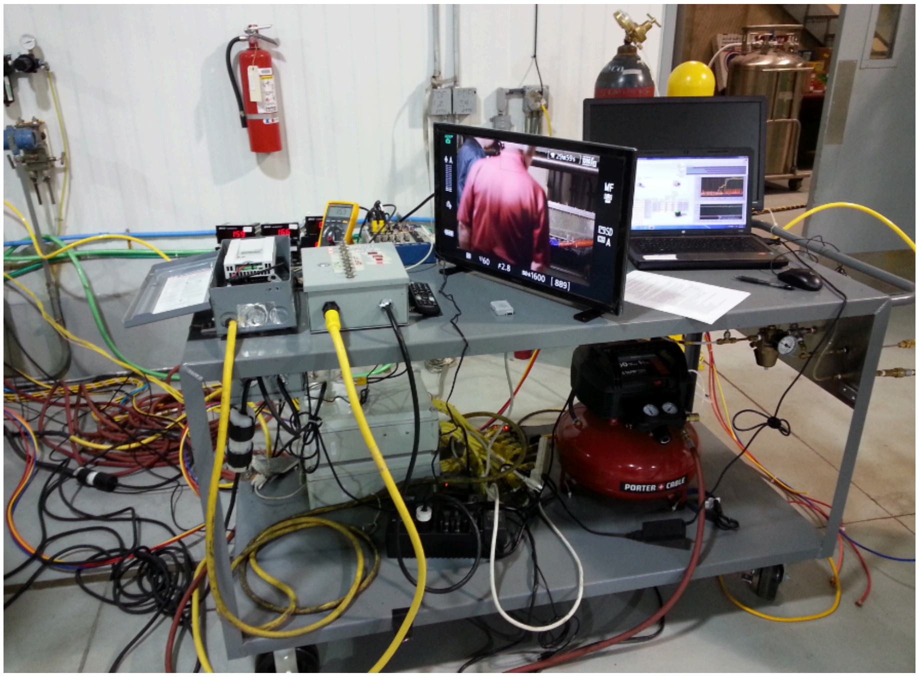


Figure 19 - ***Controls cart at EBRF.***

With the control cart outside of the e-beam vault, roughly 100 feet of length separates it from the oil cart. This caused issues for some electrical signal wires like the HDMI cable on the camera, which required an inline signal booster. It was also found that the gas lines had too much of a pressure drop to sustain the flow rate needed for purging the system, so larger diameter tubing had to be used. Otherwise, the control cart functions very effectively without feedback delay.

The fire extinguisher cart plays a critical safety role during experiments. At the e-beam facility, shutdown procedures prevent access to the vault until it is safe to enter. When the beam is on, ozone is generated and an exhaust fan is used to evacuate the gas. If the beam has been on for more than a short while, the exhaust fan needs more time to clear the air for safe entry. This means that the beam cannot be shut off if a fire breaks out and have personnel immediately enter to put out the flames. A remotely controlled extinguisher system is needed in conjunction with an emergency stop on the EBRF accelerator.

Initial thoughts for a remote fire suppression system led to the extinguishers used in racecars, where a button is pressed and an extinguisher will fire at the engine bay and other potential points for fire to start. The reason these were not used is because they are not controllable in how much extinguisher agent is dispensed. The goal of that system is to suppress the fire long enough for the driver to exit, at which point official fire crews would come to finish putting out the fire. If the extinguisher is emptied and the fire flares back up, there is no second round of extinguishing agent until the door is unlocked and a handheld extinguisher can be brought back. That is a risk in itself, because oil fires release toxic fumes that would be unacceptable to subject lab personnel to. Besides the single shot extinguisher, the control box for the racing systems is vulnerable to the electron beam. Exposure could disable the system, compromising safety of the experiment, or cause it to fire when not needed, resulting in unnecessary cleanup duty.

The attractive aspect of the racing type systems is that they can be plumbed to where the nozzles are needed, but to do this with a conventional fire extinguisher would be difficult. Pre-pressurized extinguishers are operated by squeezing a handle, meant for use by human hands. Developing a device that would depress this handle without the device or extinguisher slipping out of position, or the control systems being damaged by e-beam exposure, would carry too high a risk for such a critical application. On the other side, it is impossible to remove the handle and plumb the extinguisher hose into a different system since the extinguisher is pressurized up to the handle itself. Removing the handle would cause a violent release of pressure and extinguishing agent, and refilling the extinguisher would not be an option with a different system attached to the hose. There are pressurized extinguishers that can be hung above potential fire hazards with thermo bulbs that break at high temperatures, like a building fire sprinkler system. However, in case of an actual fire, by the time the bulb is hot enough to break, the fire has been burning for too long.

The solution to this problem is an alternate style of fire extinguisher. The cartridge-operated extinguisher functions similarly to the conventional handheld extinguisher, but it can do what other systems cannot. A cartridge extinguisher is a pressure-rated cylinder with extinguishing agent inside that is not pressurized until a CO2 or N2 cartridge is attached and activated. Cartridges are activated by pressing down a lever to punctures a foil in the cartridge, releasing pressure into the cylinder. Once activated, a handle on the nozzle is squeezed to release the pressurized extinguishing agent. While the system is not pressurized, however, the hand nozzle can be removed and the hose plumbed into a nozzle system mounted on the oil cart.

For the fire extinguisher cart, two 10-pound extinguishers were chosen from Ansul’s Red Line sub-brand. The cart itself is made of a Craftsman cart found in the lab and repurposed for this application; it happened to be red, which helps for quick identification. A single piece of shallow profile slotted Unistrut was mounted to the side, to which a cylinder holder was bolted to keep the extinguishers in place on the bottom shelf. With the extinguishers mounted, the control systems were installed.

Without a valve between the extinguisher and the nozzle with the hand lever removed, the extinguisher would start dispensing as soon as the cartridge was pressurized. A pair of pneumatic valves similar to the one used in the flow loop were scavenged from the lab and bolted to the bottom of the top shelf on the cart. These valves are analog electrically switched with compressed air driving the valve, and are used in a normally-closed configuration to prevent accidental discharge from a power interruption. A manual valve was installed inline between each extinguisher and its pneumatic valve to safeguard against equipment failure. A pressure gauge was also installed inline between the manual valve and the extinguisher to monitor pressure on an activated cartridge. The cart with all equipment mounted is shown below in Figure 20. The pneumatic valves were plumbed to a pair of nozzles mounted on the oil cart using braided stainless steel lines with a disconnect fitting in the middle.

Figure 20 - ***Complete fire extinguisher cart.***

The cartridge extinguishers are available with multiple types of extinguishing agent. There are ABC type powders which are good for wood, oil, and electrical fires, some of which are non-residue formulas to make cleanup easier. The type of extinguishing agent chosen for the oil cart is Purple-K, a dry chemical potassium bicarbonate agent with purple dye. It is a non-toxic BC agent found commonly in oil refineries, power plants, and airports that works by inhibiting the chemical reactions that propagate an oil fire. Purple-K is highly effective on oil fires and is safe for use on electrical fires, making it an ideal choice for this research, although cleanup can be difficult. When combined with water or hydrocarbons, the agent turns into a brittle dry mud, otherwise vacuuming is the easiest way to clean up the powder. A test discharge of the agent through the nozzles used on the oil cart is shown below in Figure 21; note the visible effectiveness of the mobile test cell ventilation units. The extinguishers can be refilled after operation when the pressure is depleted, however the Purple-K powder is extremely fine and poses a breathing hazard.

Figure 21 - ***Fire extinguisher test fire.***

The third supporting cart designed and built to facilitate safe research practices was the oil decanter cart. The oil supplied for study comes in small barrels that weigh roughly 40 pounds depending on how they were filled. Because of the extreme viscosity of both oils, they must be heated in order to flow. To heat the oil enough to flow, a band heater must be wrapped around the barrel and left on for a considerable amount of time, on the order of two or three hours. The surface temperature of the band heaters can exceed 300˚C in a short amount of time. Picking this barrel up by hand and holding it steady to pour the oil out is unsafe on many levels because of the temperature and weight of the barrel. Even with high-heat-resistant gloves, there is a risk of being burned from resting the barrel on a knee from fatigue. To eliminate this risk, a system was developed to make a largely hands-free process of extracting oil samples.

The basics of the system were defined as having wheels, a mechanism to securely hold the barrel, and a pivot to pour the oil out of the barrel. For the foundation, the electrical cart from which the fire extinguisher valves were taken was further scavenged since the wheels worked well and the base was larger than the oil barrel footprint. Multiple ideas were brainstormed on how to hold the barrel, such as a hinged pipe clamp with a handle. Ultimately, a framework was constructed around the barrel using Unistrut sections to lock the barrel in place.

For this cart, the front is the side of the barrel with the larger 2-inch diameter cap. To make the cradle, starting with four vertical strut members close to the barrel, the uprights on the left side were joined across the bottom of the barrel with the uprights on the right of the barrel. They were then joined together halfway up on the left and right side to serve as the mounting point for the pivot. The left and right side uprights were also joined at the bottom to prevent them from bowing out and allow the barrel to fall through the bottom. The uprights were then joined across the top of the barrel to keep it from sliding forward when tilted. A final member was added across the front-facing uprights to support the barrel when the cradle was tilted. The member joining the rearfacing uprights on the bottom of the barrel was made extra long to stick out past the vertical support between the cradle and the base to prevent the barrel from tipping backwards. This long member also functions as a handle to prevent contact with the heaters while maintaining control of the barrel angle. With the cradle completed, a pair of arches was made to hold it to the base. The decanter cart is shown below in Figure 22.

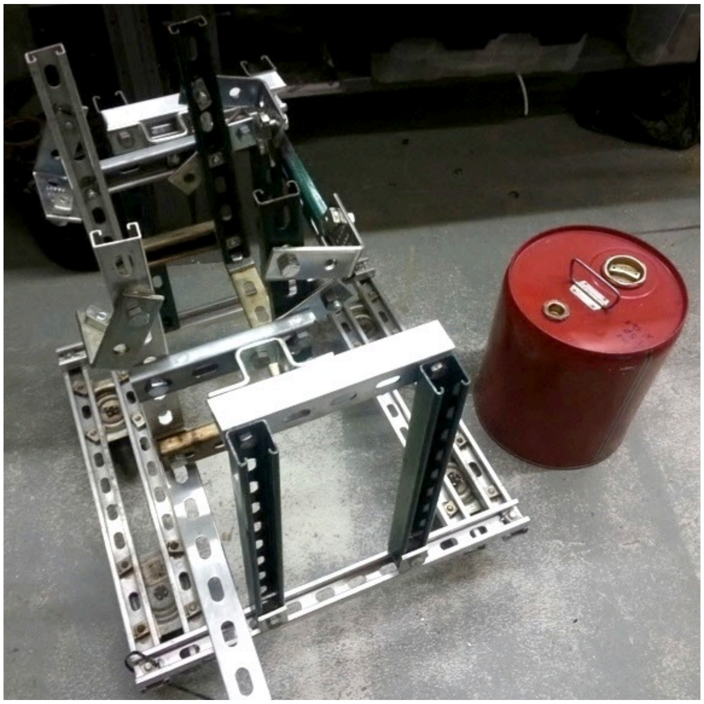


Figure 22 - ***Decanter cart.***

Standard operating procedures for the cart are found in Appendix B. When using the decanter cart, an extractor pipe is screwed into the 2-inch NPT hole in the barrel. This pipe allows the oil to be poured outside the footprint of the cart. In general, a sample collection canister will be placed on a scale next to the cart to ensure accurate mass control. It was found that for the less viscous of the two oils being considered, pouring at room temperature is possible. If the barrel were held by hand, this would not be a viable option because of the length of time needed to extract a sample. However, the more viscous oil must be heated to pour and will not flow at room temperature. Figure 23 below shows the cart in use with a heater attached but not engaged.

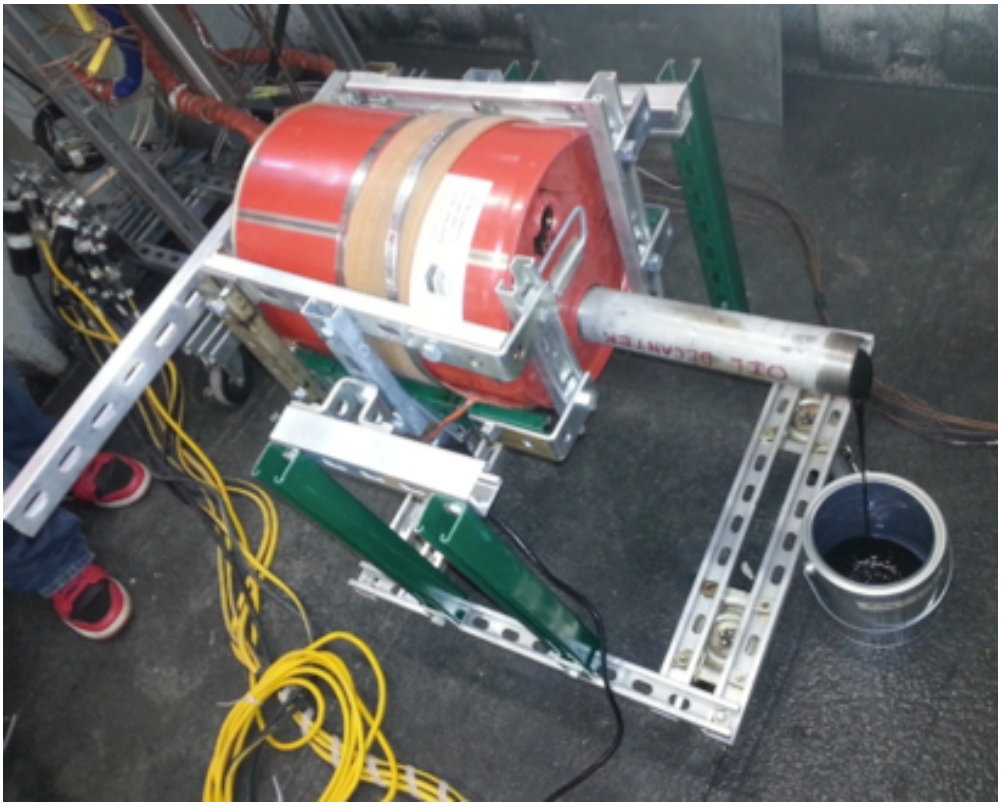


Figure 23 - ***Decanter in use.***

With all the auxiliary carts completed, the last supporting piece for the project was made. The oil cart weighs a significant amount, and tilting it back to help it roll over the cooling pad in the EBRF vault was unsafe. Rolling over the pad directly would damage its cooling coils, so an elevated platform was desired for ease of moving the oil cart into position. The ramp that was built was made from a single piece of sheet 1/8-inch thick aluminum measuring 4 feet long and 2 feet wide. It was cut to make a platform 38 inches long, leaving 10 inches as a ramp. The platform straddles the cooling pad with two stacked pieces of square profile steel Unistrut, with shallow steel strut sections running the length of the platform underneath. These shallow strut sections are run along the left, right, and middle, and keep the platform from bowing under the weight of the oil cart. There is slight bending under load of the platform, but not nearly enough for potential contact with the cooling pad. The ramp to the platform is attached using a piano hinge that was cut to size. The hinge is thin but showed no signs of deformation while rolling the cart over it. The main benefit to cutting the plate and using a hinge versus simply bending the plate is that the hinge will always rest on the ground. Bending the plate to an exact angle is difficult, and in non-precision fabrication, the numbers can change by small percentages from unforeseen deformation. This elevated platform is kept at the vault and moved into place before the oil cart is brought in.

From the first instruction to create a flow loop, a solution comprised of multiple systems was made. These systems work in unison to achieve the same goal of facilitating safe, repeatable experimentation with electron beam treatment of heavy crude oil. For the flow loop itself, necessary components were first identified and sized out to meet the needs of the project. Next, size constraints were sought and defined to bring understanding of how the individual parts might fit together. As development continued, other requirements were realized and addressed with modifications or the creation of auxiliary systems. A separate cart was made to house fire extinguishers, saving valuable space on the oil cart and ensuring safe research. A controls cart was outfitted to remotely operate the oil cart and fire extinguisher cart while they were in the e-beam vault, or otherwise unsafe to approach. The oil decanter cart was built for ease of oil sample preparation, mitigating a burn hazard by removing the need to directly hold the barrel while pouring. A mobile test cell was created to facilitate testing outside of the lab in a controlled, safe environment. It also serves as an easy, legally compliant way to transport all the systems to and from the EBRF. A ramp was also built to prevent damage to the EBRF cooling pad when moving the oil cart into place. These systems are fully integrated to the overall solution, while maintaining flexibility to be applied to other future projects, or to be modified to better suit the needs of the current one.

CHAPTER IV

FINALIZING AND DOCUMENTATION

Chapter Overview

After the initial design was completed, months of shakedown testing helped record and troubleshoot operating parameters. Special care was given to how the independent variables of experimentation could be fine-tuned to achieve the desired results. Precision systems were put in place to control alignment and positioning for repeatable results. As experiments were completed, significant design changes became less frequent until the parameters changed were largely from the control cart. Test runs and troubleshooting would continue until unforeseen issues were properly taken care of.

Documentation was developed to keep an official record of the project and to ensure its safe operation. The documents were organized into two project binders, one remaining with the engineering department and one to be kept in the lab. These documents include various standard operating procedures (SOP's) to instruct lab members on proper use of equipment. All chemicals used in the project have their according safety data sheet (SDS) included as well. For added safety, the emergency evacuation plan (EEP) for both the University Services Building (USB) and Electron Beam Food Research Facility (EBRF) buildings was procured and placed in the binder, along with safety training transcripts of all lab members involved in the project. The specifications and other documents for the trailer are also found in the binder for future reference. The most significant document is the project safety assessment (PSA), which was developed with guidance from the Mary Kay O'Connor Process Safety Center (MKOPSC) on campus. In writing the PSA, many safety concerns were pointed out, and the design of the project evolved to address these issues. The PSA has all safety information necessary to ensure that no experimenting students are at risk of injury. The contents of the PSA were written to be closely followed, such that safety can remain paramount.

Another consideration for safety comes in the form of safeguards and monitoring. When dealing with a new test apparatus, any number of unforeseen issues can occur from failure of various components. It is crucial to be prepared and have a complete plan of response for every possibility. Some of these are included in the PSA as "what-if" scenarios, but during testing a new issue can occur that was not previously thought of. Since this project deals with the potential for lethal fumes to be formed during processing of sample oils, those fumes must be diligently monitored. With all safety concerns addressed, finalization of the project can be completed.

Preliminary Testing

With all necessary systems built, there were still weeks of testing and finalizing before the first test run at the EBRF could be conducted. Through this shakedown testing, new requirements of the test vehicles were found and modifications were made to meet these needs. Some larger changes were made to the structure and layout of the oil cart, but most changes were concerned with precision adjustment and aligning components properly. Testing was conducted inside the mobile test cell to control any fumes or spills that may be released.

Few major design alterations were made on the oil cart, but the modular framework allowed these to be done without difficulty. One of the earlier modifications made to the oil cart was done in conjunction with the elevated platform built to protect the cooling pad in the vault. The maximum height that was measured and followed while the oil cart was being built went from the floor to a ventilation hose on the e-beam horn. This hose was just above the upright members on the cart, so when the elevated platform was built, four inches had to be cut off the top of the cart. All components had to be moved downward four inches as well to accommodate for the newly elevated floor. This was fortunately not a challenge, as the bottom of the SOC had clearance around the motor and pump, although not much to spare.

With this completed, flow testing began to correlate VFD control with oil residence time under the beam. Mineral oil was used to simulate how the more viscous oils would flow at high temperatures without the need to use the heaters. In some cases, the oil did not flow in a loop once the SOC was emptied and the POC was full. Without a second lift pump to help move the POC oil back to the SOC, it was apparent that continuous looped flow could not be reliably conducted. At the time, it was decided to run in a semi-batch processing configuration, where the oil would flow from the SOC to the POC and then stop there. The exit at the bottom of the POC was changed from a braided return line to a spigot for draining the oil out after processing. Once this was done, flow testing with the heavy crude oil began.

Running flow tests with the heated crude oil helped polish the control system as well as the flow correlations. The heaters successfully kept oil at the desired temperature, and the pump and valve performed without issue in high temperature operation. One issue that arose during testing concerned proper control of the manual ball valves in the flow loop. When the valve between the SOC and pump was left closed while the pump was on, a dangerous pressure spike occurred that caused oil to spray on the walls of the mobile test cell. This emphasized the need to closely follow a procedural checklist each time the cart was used.

In preparation for the first e-beam test, the oil cart was given a final check. All gas lines and gaskets were pressure checked for leaks, and the fire extinguisher test was conducted to ensure the nozzles would be effective. For the preliminary e-beam run, mineral oil was used instead of heavy crude. This was done because the results of processing mineral oil in the electron beam were much more well-anticipated and would give a more clear indication of its effectiveness. Hundreds of feet of wire and gas lines were uncoiled and laid out between the control cart and the oil cart, which would be kept in place at the EBRF for future runs to save a considerable amount of time. Previously written standard operating procedures (SOP) were followed to every detail, ensuring nothing was forgotten while also checking the clarity of the document. To record the radiation dose rate, alanine tablets were used on the reactor box. The alanine dosimeter absorbs radiation from the electron beam, which can be analyzed afterwards. The dosimetry run takes place before the processing run while the pump is off and oil is not flowing. Dividing the total radiation dose on the tablet by how long the e-beam was on yields the dose rate, which is checked against the correlation shown in Figure 4. A tablet taped on the box for a dosimetry run is shown below in Figure 24. The tablet is attached with the laser alignment tool to ensure it is in the center of the beam exposure area. This helps improve the accuracy of the dosimetry numbers.

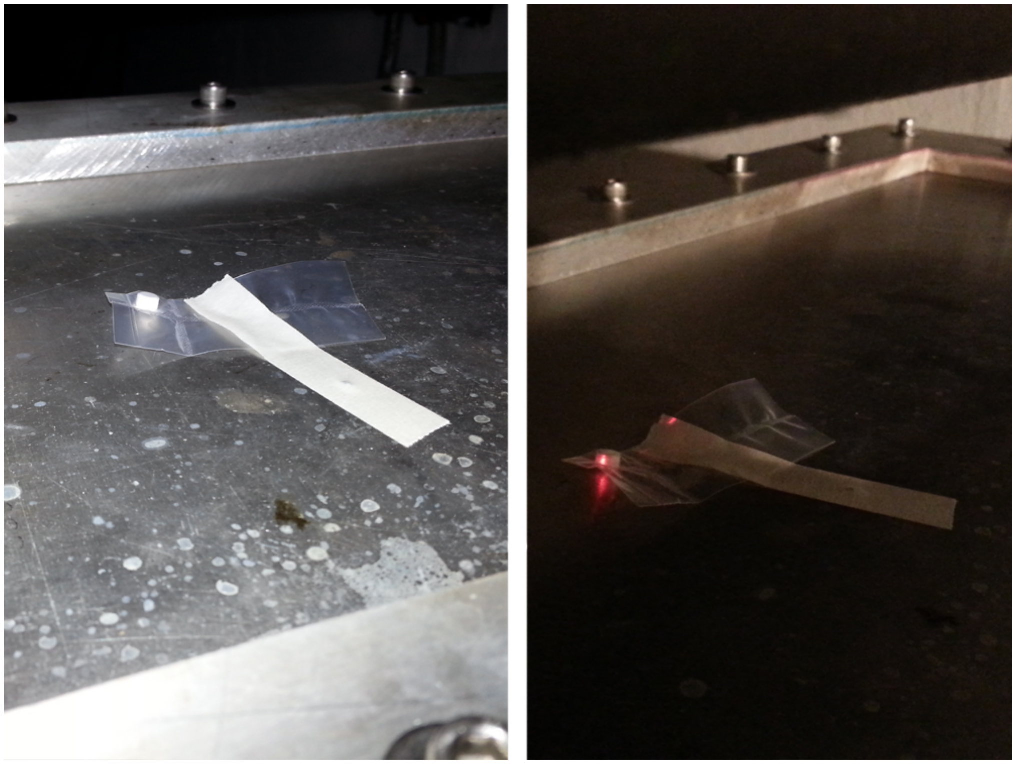


Figure 24 - ***Alanine tablet for dosimetry run.***

Since the tablets will only show dosages up to 80 kGy, the tablets are removed after the dosimetry run. During the processing run, thermocouples are used to monitor oil temperature and flow. Thermocouples are located in the SOC, on heated components such as the valve and pump, and inside the reactor. The thermocouple at the start of the flow channel indicates when the beam is on, and the one at the end of the channel indicates when oil is flowing over it. After the processing run, a meeting was held to draw conclusions on the events of the day.

Many lessons were learned from the initial test run, which was largely a success. What was found after the preliminary test was taken and used to further refine the procedures and equipment for future testing. The remote operation of nearly all components was successful, with a well-written control program and wired connections properly made. The heaters were able to bring up the mineral oil temperature to 160˚C as requested. Electrical supply was successfully divided across four accessible outlets in the EBRF without causing an interruption in power. When running multiple kilowatts of heaters along with the air compressor, computer and monitor systems, and the pump motor, careful power management is needed to avoid tripping a circuit breaker. A loss of power stops the experiment and can invalidate results if too much untreated oil has passed through to the POC, letting a day of preparation work go to waste. The dosimetry run also positively confirmed the dose rate estimates of 7 kGy/s calculated from the graph of distance versus dose rate. The mobile test cell had an uneventful maiden voyage, with the exception of a crest in the middle of one road near the USB laboratory causing the trailer to lean to the side more than would be comfortable. Upon arrival at the EBRF, all equipment that was bolted or tied down was found exactly where it was left. These positive outcomes were noted and further improved upon.

The experiment revealed a number of shortcomings that required remediation before an experiment with crude oil could be conducted. The most notable of these was that the mineral oil was not flowed through the system when the VFD was turned on. An early indication of this was the thermocouple at the end of the channel heating linearly from the e-beam, rather than a step from the hot oil coming in contact. The flow issue was a result of a misunderstanding of how twist-lock electrical plugs are connected. The plug to the pump motor was not twisted once the prongs were inserted, and the plug was loose after the run was concluded, indicating that no power was supplied to the motor. A solution to this is to have a final electrical check after filling the condenser liquid nitrogen bath and aligning the cart. It was also decided to visually monitor the flow channel when possible using the windows on the side of the box. In order to accomplish this, a mirror was mounted at a 45-degree angle to the windows. The mirror is made from highly polished stainless steel, since a conventional glass mirror would tarnish quickly under e-beam exposure. The upper window was used for a flashlight to shine through and illuminate the inside of the box. The lower window was visible through the mirror from down the hallway, where a camera with a telephoto prime lens was positioned. At this distance, the camera proved to hold up well to the e-beam exposure with no signs of data corruption thus far. The camera setup is pictured below in Figure 25. The video feed that allows researchers to monitor the reactor for signs of fire could now also be used to confirm flow in the channel.

Figure 25 - ***Camera monitor system.***

For purging the system with argon gas, the ¼-inch lines had too large a pressure drop across 100 feet to sustain the flow rates required for a timely purge process. The solution in this case was to use a larger diameter gas line for purging. Another issue found with the inert gas was that the backfill line to the SOC had no outlet other than the pressure relief valve, so an exhaust line from the lid of the SOC to the reactor box was installed. The gas would then travel through the condenser to atmosphere, improving the mass balance by condensing any oil evaporated because of heating. However, this untreated condensed oil vapor has potential to interfere with the treated condensed vapor properties.

A number of items were forgotten in the laboratory before driving to the EBRF, including a regulator for the argon gas, the power cable for the camera monitor, and an acrylic sheet to obtain more accurate measurements on the beam position. It is important to know exactly where the beam is relative to the cart alignment systems, since the oil flow channel is only as wide as the beam treatment area. After the test run, the oil cart had clearly moved out of position, likely during filling of the condenser bath, since maneuvering in the tight space without bumping into the equipment is difficult. To solve this, a kickstand was made out of a simple piece of strut that would bolt to the oil cart frame and attach to the conveyor belt rail with a c-clamp. Also of note was the HDMI cable being in the wrong orientation. At 100 feet long, the cable required an inline signal booster that has a specific input and output orientation. The cable was rerouted and functioned as intended upon realizing why it was not working. It was also found that some of the Purple-K extinguisher agent entered the nozzle lines between runs. When the extinguisher is pressurized and the manual valve opened, the line between the manual and pneumatic valve fills with pressurized extinguishing agent. At the conclusion of the experiment, the manual valves are closed. The next time the pneumatic valves are checked, they release this pressure through the line, though it is insufficient to carry the dry chemical through the nozzle. A cleanup and purge process was developed to remove as much of the powder from the lines as possible between days at the EBRF. Lastly, one of the emergency stops on the e-beam was tripped, forcing a hard reset on the EBRF computer systems. Extra care was taken in all future visits to the EBRF to avoid the button on the conveyor exit near where the control cart is stationed.

These issues were all corrected, but posed a problem for experimental procedures and accuracy more than for safety.

Safety issues were also present during the first test run. Before the experiment began, it was found that heater number 6 was on without command from the control program. This is a safety issue because the uncontrolled heater could easily overheat and cause a fire. The problem was diagnosed and remedied the following day as an issue on the control cart. Another safety issue was the condenser releasing gas within 5 minutes after the test was concluded. When processing heavy crude oil, the condenser could potentially contain hydrogen sulfide, which is lethal in relatively low concentrations. Solutions to this issue were to insulate the liquid nitrogen bath to better retain low condenser temperature, and to bring extra liquid nitrogen so the bath could be topped off before removing the oil cart from the vault. A large, 230-liter cylinder would be ordered for storage at the EBRF later.

With all shortcomings addressed, another run was scheduled the next week, where the mineral oil was successfully flowed through the system. In this run, a heater electrical cable for the SOC had issues shorting out on the frame, which was quickly remedied. Another issue that was found was the tendency of oil to pool around the drain of the reactor box without entering the drain. This is because of a weld on the inside of the box creating a raised surface around the drain. Potential solutions to this included using a slide hammer to create a depression in the bottom surface, or using a grinder to take off some of the material and chamfer the edge. However, the solution that was chosen was to create a shielded channel exit that would direct flow into the drain while minimizing splashing at the end. With this in place, no forceful alterations that could potentially damage the box had to be taken.

The week after the successful mineral oil run, the first crude oil test was conducted. Controls were verified as working properly, all items were brought, and testing was conducted successfully. From this run, more lessons were learned and applied to further refine the system. Extra liquid nitrogen supply successfully kept the condenser from exhausting during handling, however when the condenser was opened there was no oil inside. What was there appeared to be liquid nitrogen, so a quick leak test was performed. Placing the condenser pipe inside the liquid nitrogen bath with an open top and filling the bath around the pipe revealed that the gasket on the sanitary fitting shrank significantly under extreme cold temperatures. This allowed liquid nitrogen to leak in and potentially allowed condensed liquids to leak out. The solution to this was to weld the bottom cap onto the pipe, since a slight increase in difficulty extracting condensed liquids from the pipe was preferential to not having any liquids to extract at all.

While the oil was flowed and treated without issue in the first crude oil run, a meeting with Drs. Zaikin and Zaikina from PetroBeam concluded that a higher dose rate is preferable to see more meaningful results. Since the way to increase dose rate is to decrease distance from the e-beam horn, it was decided that the flow loop components would be moved upwards by 6 inches. Thanks to the modular frame, this was a simple procedure that occupied a minimal amount of time. The oil channel nozzle was also redesigned to bring the channel as high up as possible. The pass-through could not be made closer to the top without interfering with the existing hole. The new design uses a pair of 90-degree bends close to the wall to keep channel length the same.

With the increase in height of the box, the issue of controlling the angle of the flow channel became more apparent as the cantilever showed signs of sagging. A change in the angle of the flow channel throws off calculations regarding residence time, shear rate in the flow, and thickness of the flow. A support on the far side of the reactor box was needed to ensure accuracy. The support had to have enough adjustability to make up the difference in height between where the reactor box was sagging to where it would be level. A stainless steel scissor jack was procured that had an adjustable range of 2.5 inches, so a support between the jack and the conveyor belt rail had to be made. The support structure would have to be rigid to avoid bowing. The stainless steel jack and aluminum support structure would be in direct beam exposure, so the materials were chosen to avoid oxidation. Using a calibrated electronic level accurate to one tenth of a degree, angle control was successfully established. The jack, support structure, kickstand for cart stability, and digital level are all shown below in Figure 26.

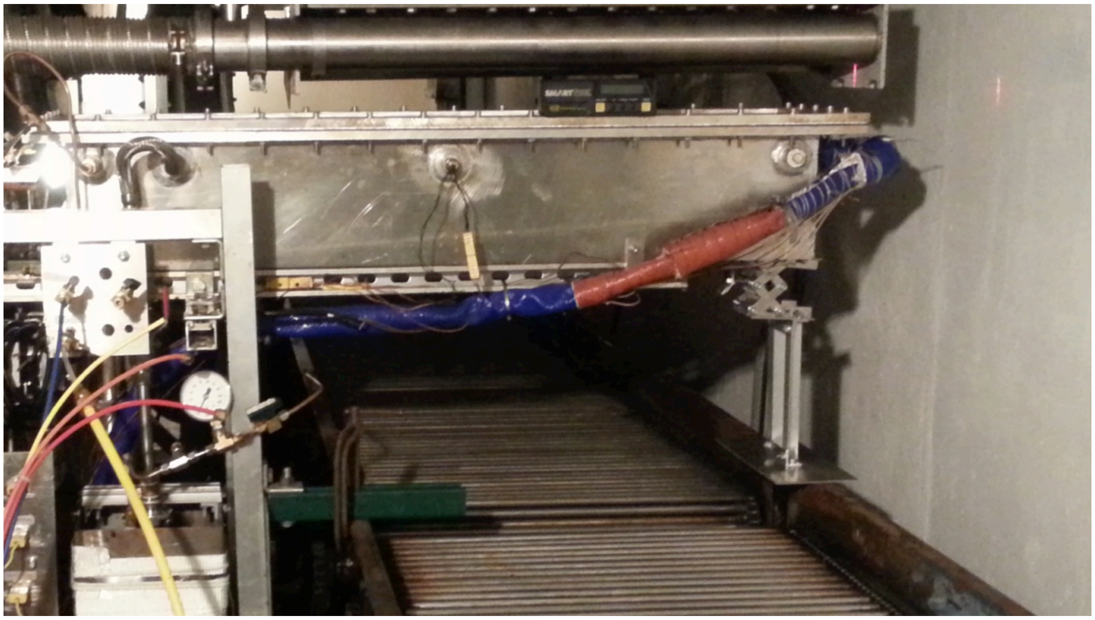


Figure 26 - ***Raised flow loop with angle control.***

Another issue plaguing the oil cart was the appearance of oil in the gas lines and the pressure relief valves. It was difficult at first to pinpoint the source of the oil, but a pressure relief valve becoming clogged is a serious safety issue. One possible source of the oil is hot evaporated oil vapors pushed through the gas lines by the inert gas flow and condensing inside the lines. To reduce the presence of the oil in the gas lines, high points were added in the lines that the vapors would have to overcome. The high point added in the inert gas line is shown below in Figure 27 along with the laser alignment tool. This has proven to be an effective solution, although it does not completely eliminate the presence of oil where it should not be.

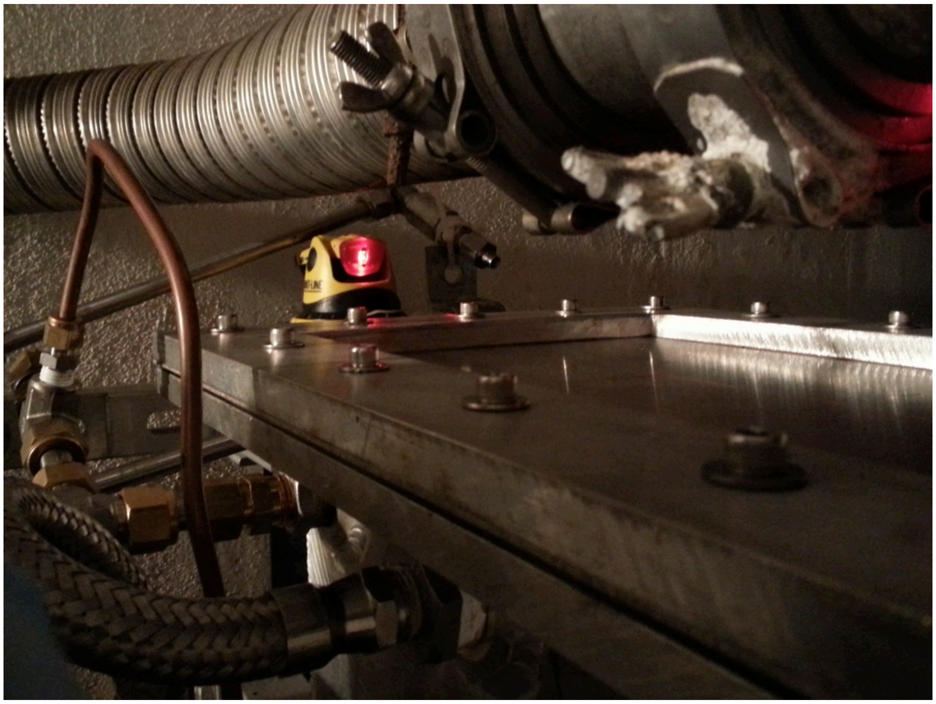


Figure 27 - ***Gas line high point and laser system.***

At this point, the alignment system was finely tuned for precise positioning of the oil cart in each e-beam run. If the oil channel is not entirely underneath the beam, the oil is not treated to the same dose as desired, so it is critical to have positioning correct. A laser alignment tool is mounted to the oil cart for alignment. Before the lid is placed on the reactor box, the laser is aligned with the channel. Using a separate test apparatus from a different experiment, a sheet of clear acrylic was exposed to the e-beam long enough to discolor and turn the plastic into a foam. This showed the position of the beam under the horn, to which the laser was realigned. With the new alignment done, a pair of marks was made on the flange of the e-beam horn. With the oil cart laser aligned to the channel of the box, the cart was then positioned to make the laser line up with the two marks on the horn. This process is shown below in Figure 28. Despite the narrow opening between the reactor and the horn, the laser sits at an angle such that the light is cast onto the far side mark with sufficient room to work with.

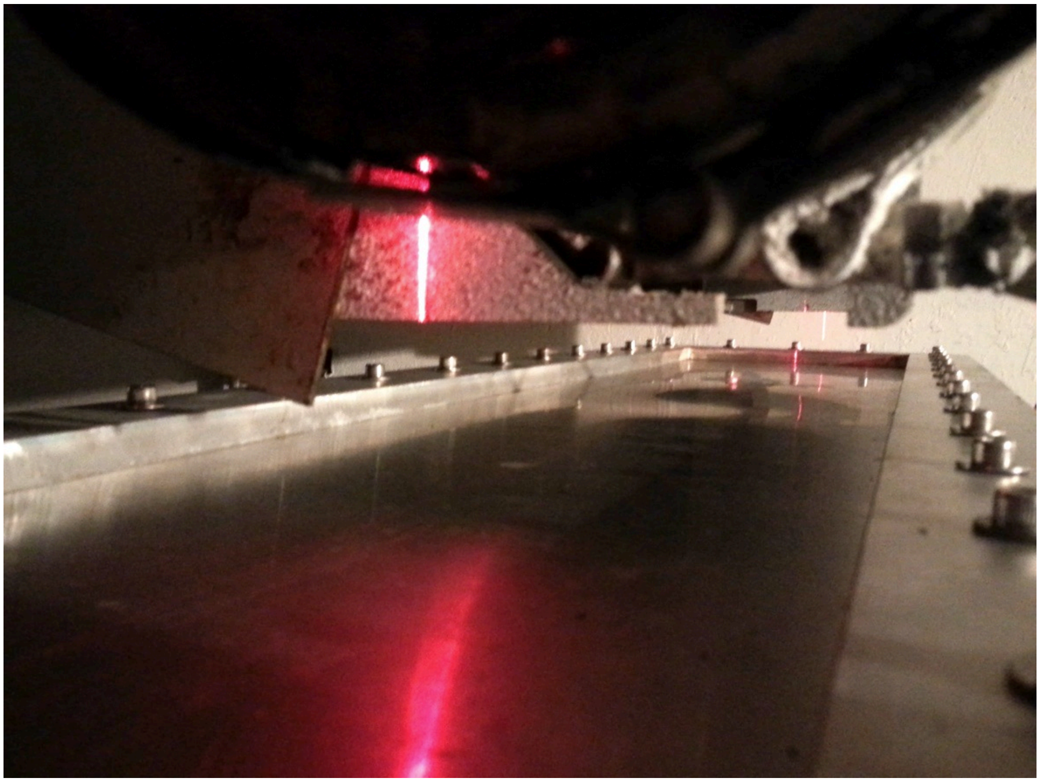


Figure 28 - ***Beam laser alignment.***

The final configuration change of the oil cart took place after more crude oil e-beam tests. While other changes made were to refine the precision controls of the system, this would be the last modification before most of the variation was done from the controls cart. To enable continuous flow in the system, the flow loop was reconfigured to eliminate the POC and recirculation line. The SOC was taken with heaters attached and affixed to the bottom of the reactor box in place of the POC. A 3-way valve was installed between the SOC exit and the pump to allow drainage of processed oil. The lines from the pump to the reactor were not changed, but the gas lines and SOC thermocouple had to be modified. With this new configuration, higher total dosages can be achieved, although the dose rate remains at the maximum achievable 16 kGy/s in this facility.

Each experimental run that was performed helped bring the oil cart and its supporting systems to a final state of completion. Major changes took place until the varying parameters were narrowed down to the controls cart for timing and temperature strategies. The result of months of shakedown testing is a system that can be used repeatedly for reliable results in experimentation. The testing has proven that the set of systems developed as a solution can handle a wide range of treatment parameters for experimenting with e-beam treatment of heavy crude oil.

Safety Documentation

Through the entire course of this research, safety has remained a paramount concern. Every test apparatus that was built has safeguards to minimize any risks associated with its operation, or was created for the sole purpose of making a process safe for all lab personnel to use. The most important aspect to having mitigated hazards in an experiment is ensuring they are documented and made well known. When information is unavailable that concerns an element of risk, procedures are based on guesses rather than verified knowledge. With this in mind, the project safety assessment was written.

The PSA contains every piece of available information about the safety of a project. Its appendices are composed of SDS's, diagrams, and training certificates that can be referenced in case of any doubts that may arise. The PSA itself is found in Appendix A of this document. The body of the PSA contains detailed explanations of each component of the experiment including what hazards are associated with it and how to minimize the accompanying risk. It also includes contact information of lab personnel involved with the experiment for future reference. The most important parts of the PSA, however, are the standard operating procedures and failure analyses. In the process of writing the PSA with feedback from the MKOPSC, safety concerns with the project that had previously been unknown were identified and fixed.

Identifying and describing individual components of the systems helped understand possible hazards associated with them. The hazards had varying severity and could be mitigated with solutions ranging from simply donning PPE, to complete and total avoidance by personnel. The aspects of the project with the highest risk factor were the electron beam itself and produced gases captured in the condenser. The electron beam is lethal on an acute timescale, but following the EBRF safety protocols ensures no chance of exposure to the beam. If a person were to remain in the vault, there is a multitude of emergency stop systems that can be activated well before the beam is activated to prevent exposure, besides walking around the corner of the 4-foot thick concrete walls. The oil used in the project has a high mass percentage of sulfur, which is converted to hydrogen sulfide during e-beam treatment. The gas is lethal in one or two breaths in concentrations of 100 ppm. An inert gas purge is used to eliminate sour gas inside the oil cart. Calculations were performed based on one volume change reducing concentrations of contaminants by 50 percent with a rough volume estimate of the flow loop. The starting condition was H_2S at the lethal concentration of 100 ppm, with the ending concentration at 5ppb; the concentration at which the human nose can detect the gas. It was found that 19 volume changes were required to reduce the concentration as desired. The calculations that determined this are documented in Appendix D. While hydrogen sulfide is in the condenser, temperature of the condenser must be kept low enough that the gas does not escape. If the supply of liquid nitrogen is depleted, the condenser shall be left inside a controlled area, such as the mobile test cell, to vent safely. Multiple gas alarms are used to ensure no personnel are exposed to the gas, including the personal clip-on alarm shown in Figure 29.

Figure 29 - ***Personal H_2S detector used in lab.*** **[23]**

Less lethal hazards are present in the apparatus of the project that require diligence of lab personnel to mitigate. Burn, shock, and high-pressure hazards are present in areas such as the heaters and metal surfaces exposed to the beam, electrical controls, and the oil and gas lines of the cart. There is no cart-side indication of when the heaters are engaged, so clear communication and heat-resistant gloves are required to avoid skin burns. The shaft between the pump and motor is not shielded, so long hair and loose clothing must be tied back as required in standard laboratory practices. Careful handling of electrical connections and shielding around open terminals prevents exposure to shock. Lastly, high-pressure hazards are up to lab personnel to avoid. When valves are incorrectly controlled, pressure from a gas cylinder or from the gear pump can build up to unknown levels. The lid of the reactor box visibly bows out when the gas purge line is open and the condenser exit is blocked. A high-pressure rupture of any component could have harmful effects, but following every step of written procedures while staying alert to possible flow blockages will greatly reduce the likelihood of over pressurization.

Operating limits of various flow loop components were also documented for safety. The system is designed around the lowest limit, but in case of temperature and pressure levels exceeding those limits, it is crucial to understand how the system will react. As mentioned, the lid of the box has a gasket that leaks at pressures elevated beyond 5 psi; therefore this is the lower pressure limit. The box is made from aluminum, which melts at a temperature of 660˚C. While this temperature is unlikely to be reached during experiments, high temperatures soften the metal, making it prone to deformation. As a result of the high box lid temperature while the beam is on and the pressurized environment inside the box, the lid had become deformed to a convex surface. Other components are made of stainless steel, which has a higher melting point and is less likely to deform from temperatures seen in practice. The fiberglass insulation on the line heaters were tested on a hot plate to reach 350˚C before the outer silicone insulation showed signs of burning. The o-rings on all sanitary fittings are Viton seals, able to withstand exposure to temperatures of 300˚C for short periods of time, and fittings are rated at 150 psi with a generous factor of safety. Pipe fittings and compression fittings are tightened to withstand pressures well above these limits. Table 6 below shows temperature and pressure limits for test components; knowing these limits affords some predictability as to what components may fail in case of an uncontrolled spike in temperature or pressure.

Table 6 - ***Component temperature and pressure operating limits.***

Component	Max Temperature (˚C)	Max Pressure (psi)
Gear Pump [24]	232	150
Viton Sanitary Gaskets [25]	200	150
Viton Reactor Gasket	200	5
NPT Pipe Fittings [26]	1,500	8,000
Compression Fittings [27]	1,500	4,100
Aluminum Reactor Box [28]	660	5
S.S. Sanitary Fittings [29]	1,500	200
PVC ¼ in. Gas Lines [30]	82	35
Silicone Heater Insulation	260	N/A

Environmental hazards of the mobile test cell were analyzed to see if extra protection was required for personnel working inside. Previous airflow evaluations proved that the test cell was safe to use as a walk-in fume hood. When handling fumes from hot oils, all personnel are to remain upstream of the ventilation to avoid exposure. For prolonged work, a sound test was required to see if hearing protection was necessary. A sound pressure meter was procured and used to measure an average sound reading at the same distances from the door as the airflow test, as well as outside near the generator. Measurements were taken at ambient levels with fans and the generator turned off as a control set. The same measurements were then taken with the generator on and fans in different configurations. The results of this test are shown below in Table 7. Hearing protection is required at sound pressure levels above 80 dB for an 8-hour workday. Sound levels exceed this threshold in some cases, so prolonged work in the container should be paired with earplugs for personnel safety.

Table 7 - ***Mobile test cell sound pressure test.***

Location	**Ambient, Generator Off**	**Generator On, Fans Off**	**Generator On, Large Fan Only**	**Generator On, Small Fan Only**	**Generator On, Both Fans On**
1 ft	51	62	74	78	78
5 ft	50	60	75	79	79
10 ft	50	61	76	79	81
15 ft	50	62	78	80	82
19 ft	50	64	82	84	86
Engine Side	50	76	90	90	93
Front of Generator	50	79	87	89	89
Exhaust Side	50	75	89	86	88

Standard Operating Procedures

When standard operating procedures are written, time is taken to test the entire process to ensure all concerns are addressed. The SOP written for conducting the e-beam experiments has been revised multiple times to clarify each step. This way, a new lab member or outside personnel not familiar with the project can read the document and understand that following it will keep them safe. For the e-beam experiment, the procedure is broken down to six sections: Sample Loading, Test Preparation, Testing, Standard Shutdown, Emergency Shutdown, and Test Conclusion. Within each section, cautionary notes are written where their mention is critical to safely conducting the experiment.

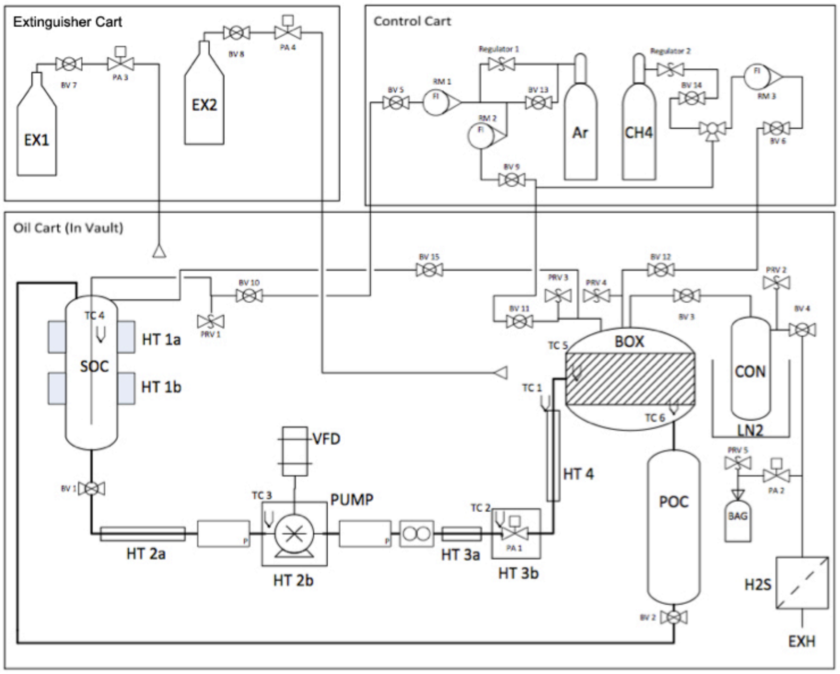
The Sample Loading section of the SOP covers the time between no work done and the oil sample loaded into the cart. It addresses using the oil extraction cradle and a mass balance to document how much oil is loaded into the system. The oil extraction cradle has its own SOP that was written to educate others on specific details of its use, which is included with the PSA. The Test Preparation section guides readers through transporting the test equipment to the EBRF and final system checks before beginning exposure. The Testing section is concise and emphasizes following the safety guidelines of the EBRF as the staff there is in charge of operating the electron accelerator.

The Standard and Emergency Shutdown procedures were given special care because of the added risks of handling the systems post-processing. The standard shutdown procedure helps ensure that the purge is performed correctly and powered systems are disengaged before personnel enter the area. The emergency shutdown procedure is referenced in most of the failure analyses to remedy any problem that may arise. It covers the use of fire extinguisher systems and refers to the standard shutdown procedure once emergency measures are taken. Finally, the Test Conclusion section covers processed sample extraction and cleanup. Care must be taken to ensure that no hazardous gases are released when handling the oil and condensed gas containers, and that the samples are not contaminated with cleaning solvents. It also addresses proper disposal of cleaning equipment for environmental concerns. The steps for the entire SOP are shortened into a checklist for use during experiments, while the SOP itself is more detailed to clarify any confusing instructions or cautionary notes.

The failure analyses are a key part of the PSA, taking longer to write than other sections of the document. A failure analysis is done for each component of the entire set of systems used for the project, and investigates how it could fail. The failure mode is noted, along with how it would affect other components of the system and what new risks would be present in case of failure. It was difficult to identify every piece that could have an issue until a new problem arose from a component that was thought to be free of risk. Most failure analyses defer to emergency shutdown procedures at some point in addressing the risk, unless the analysis covers a part not used inside the EBRF. For failure of electrical power, the VFD, heating systems, or EBRF central ventilation, the emergency shutdown procedure is the only instruction. For other issues such as the compressed air supply, inert gas, or mobile test cell ventilation, special steps are required and documented to ensure the safety of all personnel present.

System Diagram

The PSA and its accompanying documents are treated as living documents, such that they are updated as the project takes shape. As modifications were done to the oil cart, it was important to keep an updated P&ID for guidance. The P&ID was changed with each small detail altered in gas plumbing and reconfiguration of the flow loop. At the end of the project, when the oil cart was converted to a continuous flow system, the diagram was greatly changed to reflect this. To demonstrate the changes made to the system and the simplicity of the new layout, Figures 30 and 31 below show the diagram before and after this change. A text list is kept on the side for quick reference since alphanumeric nomenclature is not descriptive enough to quickly address an issue.



BV1 – Bottom of Storage Container
BV2 – Bottom of the Collector
BV3 – Box to Condenser
BV4 – Condenser Exhaust
BV5 – Control-side Argon Backfill
BV6 – Control-side CH4 Bubbling
BV7 – Extinguisher 1 Safety
BV8 – Extinguisher 2 Safety
BV9 – Control-side Argon Purge
BV10 – Cart-side Argon Backfill
BV11 – Cart-side Argon Purge
BV12 – Cart-side CH4 Bubbling
BV13 – Purge Bypass
BV14 – CH4 On/Off
BV15 – Argon exhaust line from SOC to inlet of BOX

PA1 – Flow Control
PA2 – Gas Sampling
PA3 – Extinguisher 1 Control
PA4 – Extinguisher 2 Control

RM1 – Argon Backfill Rotameter
RM2 – Argon Purge Rotameter
RM3 – CH4 Bubbling Rotameter

Figure 30 - ***P&ID for semi-batch processing.***

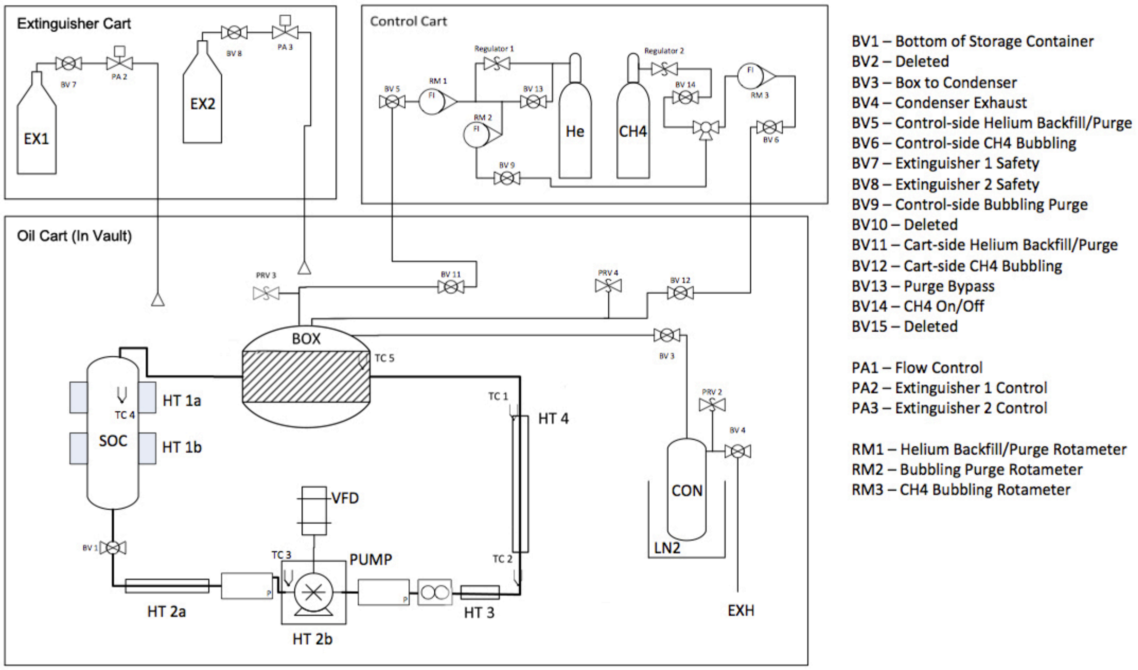


Figure 31 - ***Continuous flow loop P&ID.***

The diagrams were made in Microsoft Visio for ease of illustration. This program proved to be highly useful as it includes standard icons for various system components. The diagram for this project shows all three experimental carts and how they are connected together; thin lines indicate gas flow while thick lines are oil flow. Notable changes between the diagrams are the integration of the SOC and POC to eliminate the extra pressure drop of recirculating the flow in the old configuration. The flow control valve was also removed as a possible point of failure. The gas sample bag and hydrogen sulfide filter were also eliminated from the diagram because the condenser serves as both functions. The change of inert gas from argon to helium is another important change between diagrams. The removal of some gas lines is also apparent, as is the overall decrease in size of the system. Lastly, thermocouples 4 and 6 were combined with the omission of 6 since the drain of the box leads directly into the SOC in this configuration.

The PSA is kept inside a project binder, which is on hand during any experimentation for reference. The binder contains all supplemental information needed to safely conduct research with the heavy crude oils being used. The oil decanter cradle SOP and overall SOP checklist are in one section, with the inert gas purge calculations organized into a written paper. Documentation for the mobile test cell is in another section, which includes detailed specifications of all permanently mounted equipment. SDS's are kept in the back along with the training course transcripts of associated personnel, and emergency evacuation plans for both the USB and EBRF buildings. A second binder with identical documentation was made and is kept by Texas A&M University as an official safety record for the project.

CHAPTER V

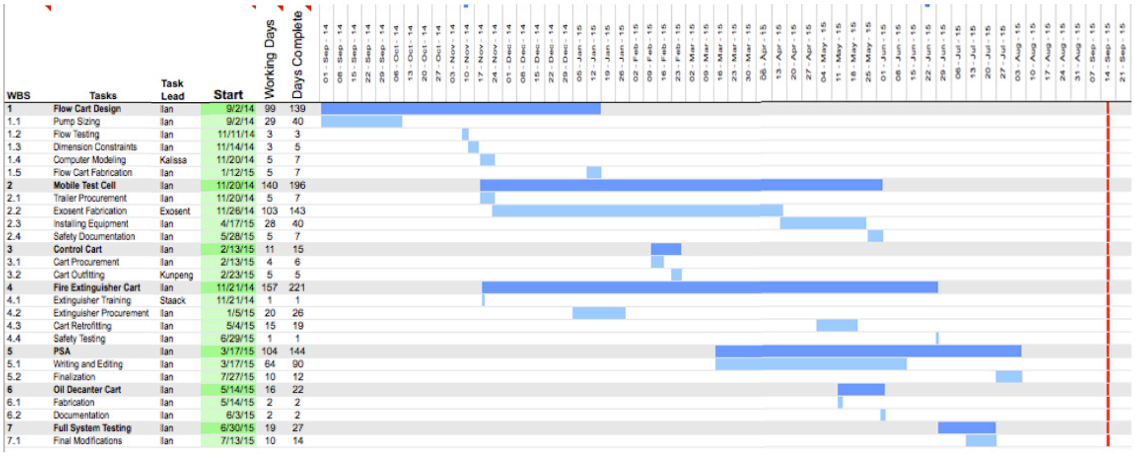
CONCLUSIONS

The goal of this research was to develop and build a system to facilitate further research on the treatment of extra-heavy crude oil with an electron beam. The focus of the solution would be the ability to yield reliable and repeatable results, with safety as an utmost concern at every stage of experimentation. Using sound engineering judgement throughout the design process, a foundation was laid to enable researchers to investigate the effects of e-beam processing. Through shakedown testing and modification of the developed systems, the flexibility of each component was demonstrated to allow for precise control of treatment variables. Altogether, the systems form an engineering solution that allows safe experimentation under a wide range of operating parameters.

Work Accomplished

Literature was reviewed to gain a better understanding of the motivation for the research this project would enable. Extra-heavy crude oil reserves require high energy costs to extract and refine. A higher efficiency, cost effective method of processing this oil is desired to make this oil more financially viable for development. The advent of the Atomic Age led to investigations of the effects radioactive energy used in a number of applications. One of these studies concerned the effects of exposure of crude oil to radiation. Early research on radiation thermal cracking demonstrated potential for favorable outcomes in processing. More recent research helped to solidify the benefits of RTC versus conventional thermal cracking, requiring far less energy to yield higher quantities of marketable petroleum products such as gasoline and diesel fuels. The research this project facilitates will continue to explore these effects.

From a loose set of initial conditions for a flow loop, design work began to create a solution to bring the requirements together. A Gantt chart was made to document the timeline of the project, shown below in Figure 32. Defining parameters of the system were organized and used to size components, including size constraints and working fluid properties. Fabrication began using a modular frame that could easily be modified as the needs of the project changed. Upon initial completion of the flow loop cart, other test vehicles were made as supporting systems. The first of these was a controls cart to remotely operate the flow loop inside the e-beam vault. A fire extinguisher cart was built to be remotely operated by the controls cart and provide fire suppression in case of emergencies with the flow loop. An oil decanter cart was made to safely extract oil that had to be heated to flow into sample collection canisters. Most importantly, a mobile test cell was created to allow remote experimentation in a controlled environment.



WBS	Tasks	Task Lead	Start	Working Days	Days Complete
1	**Flow Cart Design**	Ian	9/2/14	99	139
1.1	Pump Sizing	Ian	9/2/14	29	40
1.2	Flow Testing	Ian	11/11/14	3	3
1.3	Dimension Constraints	Ian	11/14/14	3	5
1.4	Computer Modeling	Kalissa	11/20/14	5	7
1.5	Flow Cart Fabrication	Ian	1/12/15	5	7
2	**Mobile Test Cell**	Ian	11/20/14	140	196
2.1	Trailer Procurement	Ian	11/20/14	5	7
2.2	Exosent Fabrication	Exosent	11/26/14	103	143
2.3	Installing Equipment	Ian	4/17/15	28	40
2.4	Safety Documentation	Ian	5/28/15	5	7
3	**Control Cart**	Ian	2/13/15	11	15
3.1	Cart Procurement	Ian	2/13/15	4	6
3.2	Cart Outfitting	Kunpeng	2/23/15	5	5
4	**Fire Extinguisher Cart**	Ian	11/21/14	157	221
4.1	Extinguisher Training	Staack	11/21/14	1	1
4.2	Extinguisher Procurement	Ian	1/5/15	20	26
4.3	Cart Retrofitting	Ian	5/4/15	15	19
4.4	Safety Testing	Ian	6/29/15	1	1
5	**PSA**	Ian	3/17/15	104	144
5.1	Writing and Editing	Ian	3/17/15	64	90
5.2	Finalization	Ian	7/27/15	10	12
6	**Oil Decanter Cart**	Ian	5/14/15	16	22
6.1	Fabrication	Ian	5/14/15	2	2
6.2	Documentation	Ian	6/3/15	2	2
7	**Full System Testing**	Ian	6/30/15	19	27
7.1	Final Modifications	Ian	7/13/15	10	14

Figure 32 - *Project timeline.*

During construction of all systems in the solution, safety was kept as a top priority. Writing safety documentation and standard operating procedures helped in the process of identifying possible risks in the project. Troubleshooting and shakedown testing of the systems also helped emphasize problem areas. Modifications were performed to the systems to address these concerns. Controls were kept easily accessible and used to minimize risk of exposure to any hazards associated with the project. A video monitoring system was established to watch for fire hazards, and gas monitors were used to ensure the safety of all personnel when handling possible sources of toxic fumes. Analyses were written to preemptively develop plans of action in case of failure of any component in the systems. All documentation is kept organized and clear in a project binder that accompanies the test apparatus at all times to ensure that any researchers associated with the project are aware of proper procedures.

Lastly, precision systems were finely tuned to ensure reliable experimental results. An alignment system was developed to accurately note the location of the electron beam treatment area, such that the oil flow channel of the oil cart could be positioned properly for maximum exposure. A kickstand clamped to the conveyor rail in the e-beam vault ensured A level control system was fabricated to accurately control flow characteristics in the channel. A final configuration change in the flow loop allowed for continuous flow processing instead of semi-batch runs.

Future Work

With construction of the test apparatus complete and all necessary safety measures taken, the systems can be used for further research on crude oil processing. Large modifications were performed on the oil cart to bring it to its present state, where the varying parameters of processing are mainly controllable from the controls cart systems. New strategies of exposure will be investigated, including the use of a pulsed electron beam versus continuous exposure. Another change to be researched is the effect of varying the motor speed to change flow shear rate and residence time in a continuous-flow configuration. Bubbling various gases through the oil flow will also be tested, as that technique has potential to significantly alter the resulting products of e-beam treatment. The systems created form an engineering solution that provides reliable and safe results for a wide array of varying parameters for future research.

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APPENDIX A – PSA

This document is the Project Safety Analysis. It contains safety information for the entire project listed in detail. It also contains operating procedures and plans of action in case of component failure.

Project Safety Analysis Plan

Master Agreement 11-1247/ Task 4: Plasma and Physical Technologies for Oil and Gas

Project #: C7730

7-23-2015

David Staack
Assistant Professor
Mechanical Engineering
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MECHANICAL ENGINEERING
TEXAS A&M UNIVERSITY

PROJECT IDENTIFICATION SECTION

Project Name: **Master Agreement 11-1247/ Task 4: Plasma and Physical Technologies for Oil and Gas**

TEES Project Number: C7730
TEES Proposal Number: ***

Contract Officer, Research Administration:

Sponsor:

Chevron Technology Ventures LLC

TEES/OSRS Administrator

Rebecca Gray

Project Description *(abstract or executive summary):*

Master Agreement 11-1247/ Task 4: Plasma and Physical Technologies for Oil and Gas

A descriptive title of this project is: Evaluation of electron beam technologies in flow conditions for upgrading of extra-heavy crude oils. Due to proprietary reasons the nature of the specific project should not be publically disclosed.

Electron beam irradiation is being investigated as a technique for vis-cracking and general upgrading of heavy crude oils, residues, and bitumen. Such *"non-traditional processing"* techniques are being investigated for use on *"non-traditional oils"* because of potential advantages in adding energy by non-thermal mechanisms. These advantages include increase efficiencies, reduced processing temperatures (and the avoidance of deleterious runaway reactions), increased selectivity, more scalable processes, and processes which are versatile (able to work with more variable oil sources).

In this particular project, funded by Chevron ETC, Texas A&M is working to demonstrate an irradiation processes originally developed by Zaikin and Zaikina ("Self-sustaining cracking of hydrocarbons", United States Patent 8192591, and owned by PetroBeam, Inc.). Texas A&M has already demonstrated for Chevron the batch processing of small quantities (~ 100g) of heavy crude oils at the Texas A&M National Center for Electron Beam Research (ebeam.tamu.edu). The success of the batch processing has led to continued efforts to implement a more efficient flow processing configuration, the topic of the current proposed research project. The proposed project will test the efficacy of flow configuration for various oils over a seven month program (ending in Q3, 2015).

The flow processing configuration will consist of an apparatus configured to be placed at the e-beam facility for process testing of 1 to 10 gallons of heavy crude oil (API ranges of 9 to 20). This apparatus will be built in a collaboration between PetroBeam, Inc. and Texas A&M University. In the apparatus the unprocessed oil will be pressure fed through a flow system such that a fluid sheet of oil passes into the treatment zone of the 10 MeV electron beam. Typically irradiation doses will be in the range of 50 to 500 kGy (1 kiloGray = 1 kJ/kg) and are deposited at a dose rate in the range of 3 kGy/s to 20 kGy/s. The oil may be preheated to as high as 200^{o}C prior to ebeam processing. Liquid, solid, and gaseous products will be collected for analysis locally by Texas A&M and also shipped to Chevron. The entire oil flow system will be a sealed system, without exposure to ambient. Liquid reactants and products must also be contained so as to not to contaminate the e-beam facility (which is also used for industrial food processing applications). Some incondensable gaseous products will be released into the e-beam chemical exhaust system.

Aside from irradiation safety precautions (generally accounted for at the electron beam facility) additional potential safety issues with the new system include the production of high temperature hydrocarbon liquids and gases and sulfur containing species (sulfur content in the crude oils can be as high as ~5% by mass). The MKO Process Safety Center will be employed to help in writing the project safety analysis and in revising safe operating procedures.

Principal Investigator:

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Location of Project Facilities:

Building No: **3400**
Building Name: **University Services Building (USB)**
Room No: **USB Room 127NB**

Project Duration *(projected dates):*

October 1, 2014 through August 31, 2015

REVIEW & AUTHORIZATION SECTION

The attached Project Safety Analysis has been reviewed by the undersigned. Any major modifications of equipment or changes in procedures will require additional review by the Departmental Safety Committee, and/or the Departmental Safety Officer, and the Department Head. In executing this work, you must abide by the Safety Procedures of the Department and University and must inform the Departmental Safety Officer of any changes in personnel or operations outside these procedures.

Primary Faculty/PI: **David Staack** Date:

Department Head: **Andreas Polycarpou** Date:

Department Safety Officer: **Robert Irving** Date:

Engineering Safety: **David Breeding** Date:

C. Informational Copies

Please send a copy of your final approved PSA document to the following TAMU Departments, for their information and use. Any comments or concerns will be conveyed to the PI and to the Engineering Safety before initiation date of the proposed project.

TAMU Environmental Health & Safety Department (EHSD)
Date:
Mail Stop: 4472 TAMU

(*A copy of the approved PSA is provided to EHSD for their information and use. EHSD will review and keep on file, and notify Engineering Safety & Security if any additional concerns are noted.)*

TAMU Office of Facilities Coordination, MS 1369 Date

(*To accompany requests for new space assignments or requests for use of sites at the Riverside Annex Campus or other outdoor sites.)*

Other… Date:

STRATEGY SECTION

Purpose of Project Safety Analysis:

PSA provides the Principal Investigator with the opportunity to review the environmental health, safety and security aspects of the research project to be undertaken, to identify known and potential hazards, to assess risks, and to select and implement necessary protective controls. This will help protect the researchers, graduate students, and staff involved with the project, reduce risk, ensure compliance, and conserve environmental resources, and protect facilities.

Scope:

All Principal Investigators shall file a written report on the safety analysis of each research project prior to the initiation of that exercise. The Project Safety Analysis (PSA) shall identify potential hazards and assess risks by the use of system safety analysis techniques, and shall detail the engineering and administrative controls that will be necessary to reduce risk to acceptable levels for the researchers, graduate students, and staff as well as the occupants of the building and the environment. The PSA will identify the costs, and the source of adequate funding, to implement necessary controls. It will identify necessary personnel training needs. The PSA will identify a plan for ultimate disposal of leftover equipment, materials and wastes, and the decontamination & clean up necessary to render the facility safe to reassign and reoccupy.

Extent of Applicability:

Recognizing that no activity is without some degree of risk, and that certain routine risks are accepted without question by the vast majority of persons (for example: machine shops that do not handle hazardous materials, cars used for personal transportation, etc,) the applicability of this analysis has been limited to those academic research projects that involve hazards not routinely encountered and accepted in the course of everyday living by the vast majority of the general public.

The analysis of a project which involves only hazards of a type and magnitude routinely encountered and accepted by the public will require justification which can be referenced to a recognized source.

Assistance in Conducting PSA

The Office of Engineering Safety is available to work with the Faculty/PI and research staff to identify hazards associated with the project, assess risks, and to identify necessary protective control measures.

APPARATUS AND PROCEDURE

A) Equipment Used in the Experiment

1. 10 MeV Electron Accelerator at the Electron Beam Center
    a. Used to alter composition of test oils, accelerates electrons through 10 MeV potential in a thin scanning motion.
    b. Risk of lethal radiation exposure. Risk of fire from heating of metal components in contact with spilled oil and exposed to air. Risk of hazardous ozone formation and exposure due to energy discharge.
    c. All personnel must use dosimeters inside facility and adhere to strict safety guidelines to eliminate risk of exposure to highly lethal radiation. System is leak-tested before experiment to mitigate fire risk. Facility procedures do not allow access to testing area until ozone is evacuated.

2. Chemicals
    a. Oils
        i. Heavy crude oil samples from Hamaca and McKay River, nearly solid at room temperature and will be heated up to 200˚C for processing and up to 50˚C for handling.
        ii. Mineral oil used in simulating elevated temperature test conditions using room temperature fluid.
    b. Mineral spirits and other solvents such as DCM and diesel used for cleaning equipment; cleaning rags disposed in proper container, waste liquid disposed in large labeled oil container.
    c. Fire extinguisher "Purple K" agent, used for eliminating oil-based fires. Requires clean up of residue with brushing and wet wiping.
    d. Health and fire risks from oil, solvents, respiratory risk from extinguisher.
    e. Oils and solvents to be handled in fume hood, spills controlled and kept away from ignition sources, waste disposal in large labeled oil container. Extremely fine powder poses respiratory risk on dispersal, clean up, and refilling of extinguishers. Recommended PPE includes gloves, breathing masks, and goggles.

3. Motor
    a. 3-phase motor rated at 0.75 hp, drives pump in flow loop, controlled remotely with VFD.
    b. Ventilated metal casing has potential for oil to enter and become exposed to electrical sparks, posing a fire risk. Risk of entanglement of loose hair and clothing when motor is running.
    c. Basic splash shield used around motor ventilation. Lab safety protocol followed regarding no loose clothing and tied back long hair.

4. Variable Frequency Drive (VFD)

    a. Allows fine-tuning of motor speed to adjust flow rate. Placed inside grounded electrical control box.
    b. Terminals at bottom are exposed and pose risk of electrical shock.
    c. Control panel reasonably isolated from terminal location, power shall be disconnected before modifying electrical connections.

5. Chemsteel Gear Pump (PUMP)
    a. Positive displacement pump for moving oil in flow loop. Made from steel alloy and designed to resist corrosion, potential for test fluid leak at pump exit if blockage occurs downstream from heavy oil solidification. Maximum pressure about 100 psi, fitting and system components upstream of final valve should be rated for such pressures.
    b. Risk of high pressure buildup downstream of pump if downstream valves remain closed.
    c. Valves shall be opened within a limited time of motor engagement.

6. Electrical Resistance Heaters (HT 1-4)
    a. Multiple ceramic container and fiberglass-wrapped wire hose heaters used to bring oil temperature to 200˚C.
    b. High surface temperature poses risk of burns on contact. Heaters operate in ambient air and pose risk of fire from oil leaks and spills.
    c. Heaters will be insulated for protection.

7. Instrumentation
    a. National instruments data acquisition system.
    b. Thermocouples (TC 1-6)
        i. Type-K thermocouples located throughout system to monitor heater temperature, processing oil temperature. Inside processing box, fiberglass-sleeved thermocouples used with ceramic connectors rated to 800°F and small junction to reduce error.
        ii. Risk of overheating and embrittlement when exposed to direct electron beam discharge.
        iii. Known accepted risk, thermocouples in direct exposure made with thin wires, fiberglass insulation, and ceramic connectors to mitigate damage.
    c. Camera System
        i. Located on test cart to monitor flow inside processing box, system integrity for leaks at pipe junctions, presence of fires.
        ii. Risk of failure when exposed to direct and indirect electron beam discharge; rendering fire monitoring systems blind.
        iii. Known accepted risk, backup camera used down hallway in case of cart camera failure.

8. Fire Extinguishers

   a. Two, 10 lb capacity, CO_2 cartridge-operated extinguishers allow for custom-fitted dispersal mechanisms (pneumatic valves) when depressurized. Extinguishers and associated valves are mounted to a red caster wheel cart. Reusable and filled with "Purple K" agent tailored to oil fires, will be highly effective at eliminating fire risks.
   b. Refilling extinguishers with fine powder agent, and removal of pneumatic valve before extinguisher is fully depleted, releasing any stored pressure and agent, poses respiratory risk.
   c. Breathing masks, goggles, lab coats, and gloves to be worn when handling extinguisher system.

9. Purge and Fill Gas
   a. Pneumatic valve controls the flow of inert gas (Ar) into the oil storage container to replace the removed oil during processing. Gas can be nitrogen, argon, or other. Gas is also used to purge the processing box from hydrocarbon gasses. Gas exit through the condenser.
   b. Risk of pressure build up if condenser clogs. Risk of air infiltration to hydrocarbon gas and combustible mixture if gas is depleted or not turned on.
   c. Pressure regulator to control pressure build up, flow controller to limit flow. Cylinder located near controls cart with pressure gauge to monitor levels and ensure system is operational when needed.

10. Bubbling Process Gas (CH4)
   a. Additional gas plumbed into processing box to bubble in flow. Can be methane, air, or other gas used to augment processing. Line leading to box connects with nitrogen purge line for safe handling at conclusion of experiments. Cylinder located near controls cart with pressure gauge to monitor levels.
   b. Risk of combustible fuel-air mixture forming from gas leaks, exhaust area.
   c. Extra care taken when tightening gas lines, exhaust is heavily diluted with air in controlled space.

11. Pneumatic Valves (PA1-4)
   a. Flow control and fire extinguisher valve actuated by compressed air, controlled by analog electrical signal. Allows for remote stoppage of oil flow in system, opening passage to gas sample bag, and remote on/off dispersal of extinguishing agent to conserve agent and suppress fires that self-reignite.
   b. Risk of failure due to power outage or insufficient air pressure.
   c. If power or air systems fail, valves will return to default position. Normally closed valve used for flow, gas sample bag, fire extinguisher.

12. Produced Gas Condenser (CON)
   a. All gases produced during processing are directed through the condenser. Condensable gases are trapped in the condenser housing. The condenser is

cooled using an insulated container of liquid nitrogen or dry ice. Incondensable gasses are not captured by the system. Condenser consists of a long stainless steel tube with sanitary fittings. NOTE: Condenser is not used as high-pressure container, pressure relief valve set at 20psi or lower, sanitary fittings rated to 250psi.

   b. Risk of pressure buildup as condensed gases return to room temperature, risk of hazardous gas exposure from captured species. Liquid nitrogen bath poses cryogenic risks.
   c. Pressure-sealed collection chamber rated to 100 psig with a low-threshold pressure relief valve installed between the condenser and box to ensure safe conditions. Gases captured can be hazardous to health and are handled under a fume hood and remotely purged. Cryogenic safety guidelines adhered to with long pants, closed toed shoes, and thermally insulated gloves when handling liquid nitrogen. Figure 33 below shows the condenser in current form:

Figure 33 - Produced Gas Condenser

13. H_2S Exhaust Filter (H2S)
   a. Located inline between condenser outlet and exhaust opening. Filters hydrogen sulfide from exhaust gases to minimize possibility of exposure.

14. Produced Gas Sample Bag (BAG)
   a. Clear plastic bag connected through a tee to condenser exhaust line between manual valve and low-threshold pressure relief exhaust valve. Fills with produced gases and allows exhaust to open when bag is full. Bag has built-in check valve, detaches from flow loop system and connects to gas analysis device after experiment completion.
   b. Risk of bag rupture and loss of collected sample if downstream pressure relief valve threshold too high. Rupture would also cause exhaust gases to bypass condenser and H2S filter.
   c. Pressure relief valve specifications carefully chosen, tested with compressed air to confirm desired operation.

15. Electron Beam Processing Box (BOX)
   a. Placed under electron beam during experiments, flows and exposes test oil to direct electron beam discharge. Designed to retain minimal amount of oil

inside box at all times with large exit to storage container/flow return. Box is made from aluminum to reduce energy absorption from electron beam, reducing heat transferred from the box to the oil. Maximum pressure not to exceed 10psig under operating conditions; regulated by exhaust through condenser. Must be pressure sealed to contain hazardous gases formed during experiments; opening handled under fume hood or remotely purged.

   b. Risk of skin burn from high temperature after processing, hazardous gases produced inside.
   c. Careful handling of cart when removing from test area prevents contact with heated sections. Hazardous gases remotely purged before entry to test area.

16. Electrical Air Compressor
   a. Portable compressor generates compressed air necessary to actuate pneumatic valves. Compressor pump generates heat and surface becomes hot to touch; located on control cart for ease of mobility and reduces risk of contact with hot surface.
   b. Risk of skin burn from hot compressor mechanism surface, risk of failure from power outage.
   c. Sign on compressor indicates hot area. In case of power outage, enough compressed air is stored to operate valves that are also set to safe default positions.

17. Flow Loop Test Cart
   a. Steel and aluminum structure on four rotating and locking caster wheels. Houses entirety of flow loop besides fire suppression system and controls. Also houses untreated sample oil storage container (SOC), heatable to 200˚C, with thermocouple and pressure relief valve, processed oil container (POC), condensed liquids container with pressure relief valve. See Figures 34 and 35 below for picture of cart in current form, and a piping and instrumentation diagram.
   b. Fully loaded weight exceeds 200 lb, some frame members have rough edges; risk of injury from toppling if mishandled.
   c. Gloves recommended when handling, though most members are smooth for safe grip. Care taken to balance cart when pushing along shorter base side to avoid toppling.

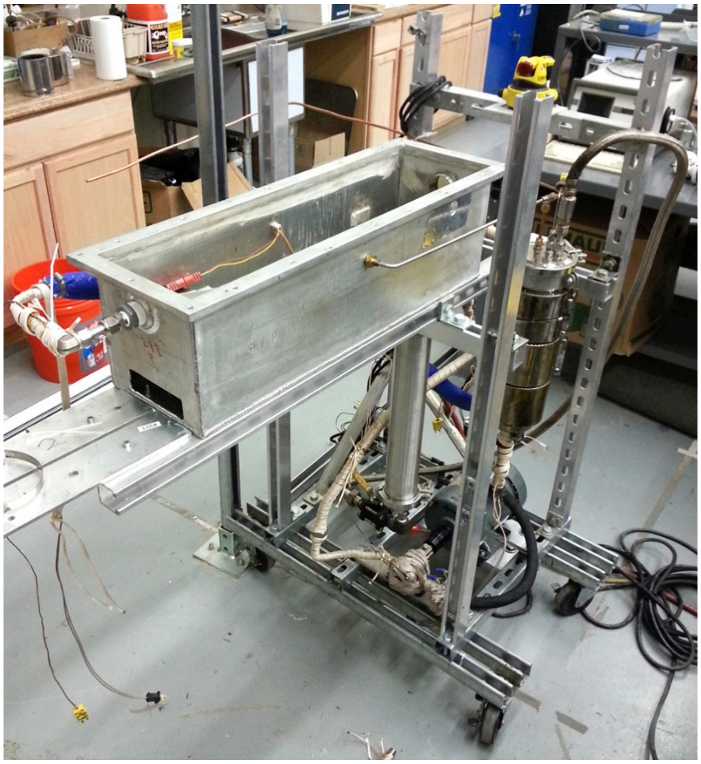


Figure 34 - Flow Loop Cart

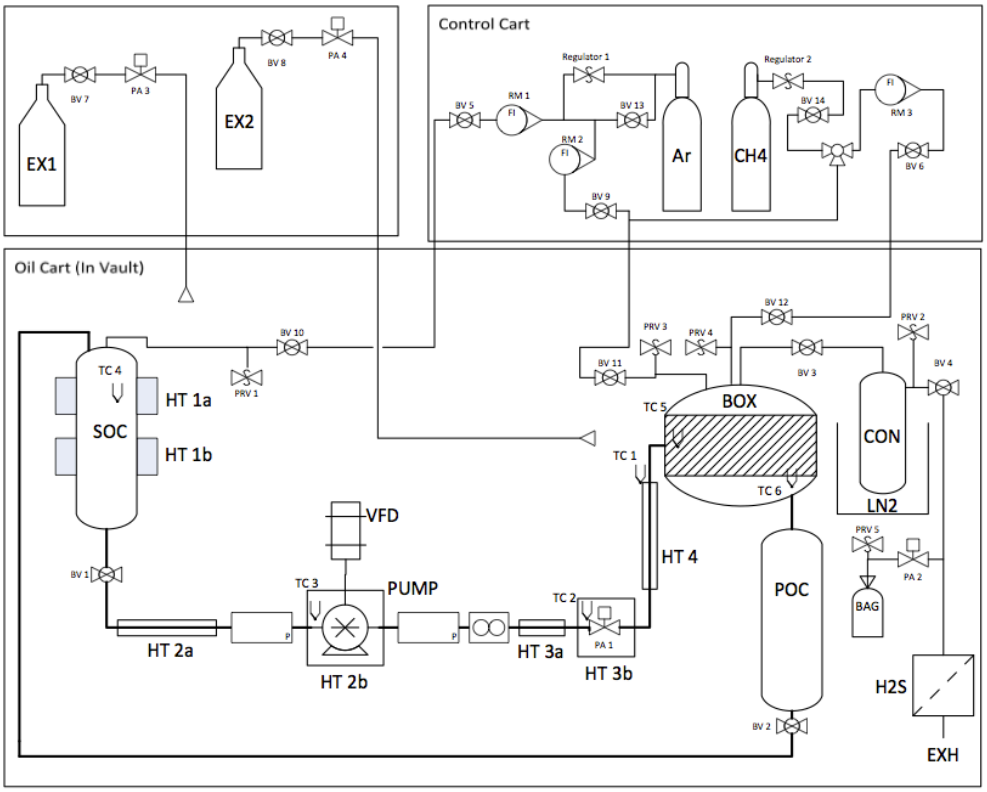


Figure 35 - P&ID

18. Auxiliary Test Cart
    a. Steel cart placed near test cart that houses fire extinguishers. Risk associated with compressed gas in fire extinguisher.

19. Inert Gas Cylinder
    a. Nitrogen cylinder located outside of processing area. Holds 300 scf and has safety measures in place including pressure relief valve and pressure gauge to monitor cylinder status. Connects to processing box and oil storage container for system backfill during experiments and purging hazardous gases after experiment completion.

20. Control Center Cart
    a. Large welded steel cart for housing independent control systems, instrumentation interfaces, air compressor, and electrical/signal/pneumatic lines. See Figure 36 below for current cart status.
    b. High weight capacity and excessive size requires cautious handling of loaded cart to avoid impacts with personnel and equipment. Electronic equipment on cart requires careful handling.
    c. Care taken when moving cart to avoid impacts.

Figure 36 - Control Cart

21. Mobile Lab Trailer
    a. 8 x 8.5 x 20 foot cargo container permanently mounted on 20 foot, over-axle deck, bumper-pull Class IV hitch trailer with 14,000 pound GVWR. Container has been fully and professionally retrofitted for lab use including an onboard externally mounted generator, ventilation system capable of

generating airflow with over 80 ft/min face velocity within the container, non-sparking enclosed lighting, and an oil-resistant floor coating oversprayed up the bottom of the walls.

b. Mobile fume hood retains DOT compliance and is registered and road-legal.
c. Figure 37 below shows the trailer near completion.

Figure 37 - Mobile Lab Trailer

B) Experimental Procedures

Standard Operating Procedure

This is a research project and facility and specific operation procedures change very frequently. Generally procedures are as follows.

I) Sample Extraction and Loading

1. Don PPE (gloves [high heat resistant when necessary], goggles, labcoats, work with a buddy).
2. When working in the mobile lab trailer, if possible, connect each fan to separate power circuits, i.e. one fan to generator power and one to building power. This will ensure that if one circuit goes down due to overload or power failure, such as running out of gas, one fan will remain operational at all times.
3. Select oil container from which to extract sample, bring into walk-in fume hood. Remove both caps of container.
4. Affix barrel heaters and 2" extraction pipe to oil barrel, and place in extraction cradle. Heat oil container in upright position to allow high-viscosity oil to flow; temperature should be about 140°C for McKay River or 170°C for Hamaca. This heating process can take roughly 2 hours. **CAUTION**: Heaters can reach temperatures in excess of 300˚C and will cause serious burns on contact with bare skin.
5. While heating, locate oil sample storage tank (SOC) on test cart. Close valve BV 1 at bottom and remove top lid for sample loading.

6. Obtain and clean an intermediate oil container for sample transferring from barrel to flow loop tank. Secure for sample loading. Don high heat resistant gloves at this time.
7. With intermediate container in place, use extraction cradle to slowly rotate barrel of heated oil to pour into container. Use a spatula to clear excess oil from the opening of the extraction pipe, rotate barrel back to upright position, and turn off barrel heaters.
8. While oil is still at elevated temperature and can flow, record mass of container with oil and immediately transfer to flow loop storage tank. Seal lid of storage tank and record mass of intermediate container with oil remains to calculate mass of oil placed in flow loop. Loosely replace cap of oil container to alleviate vacuum buildup from cooling; leave container in fume hood to cool naturally. **CAUTION**: Avoid inhalation of fumes from oil containers and contact with oil.
9. Record mass of intermediate container after transferring oil; calculate mass of oil sample extracted. Reinstall lid on sample oil container.
10. Isolate the system by closing the exhaust valve BV 4 on the condenser.
11. Perform leak test by filling processing box with inert gas such as nitrogen via gas pass-through on box. Increase pressure to 5 psig and allow to sit undisturbed for at least 1 hour. Check pressure after waiting; if differential is below 1 psig then the system is considered airtight. If differential is unacceptably large, check system for leaks in flow loop or at the box flange gasket and repeat test when issues are addressed.

II) Test Preparation Procedures

1. Carefully load flow loop test cart, auxiliary test cart (if applicable), and control center cart into mobile lab trailer and secure for transport to electron beam facility. Be sure all equipment is properly tied down to prevent toppling during transport. The route taken from the lab is shown below in Figure 38. Note: a left turn cannot be made from Old Ti Road onto University Drive, so a right turn must be made through the bank parking lot, followed by a left onto Copperfield Parkway, followed by a left onto University Drive at the traffic light.

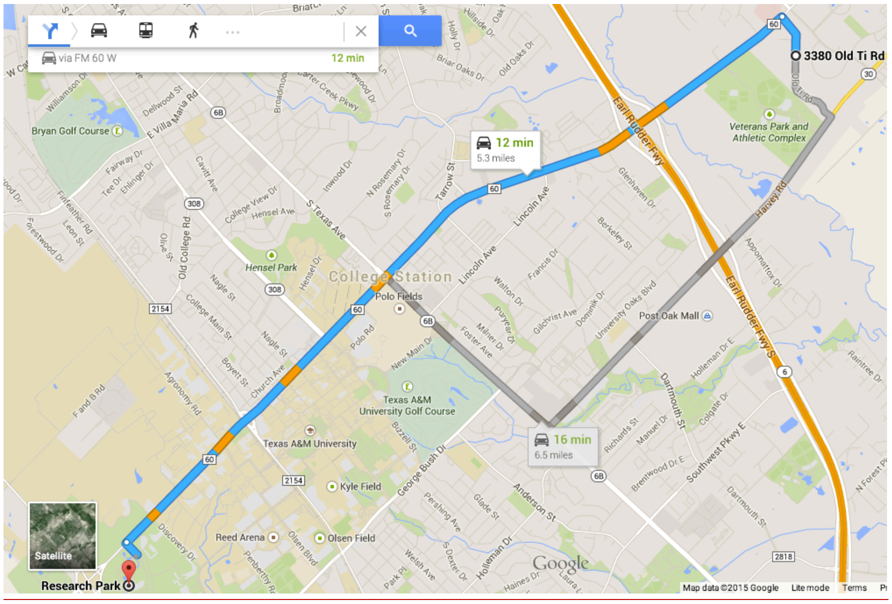


Figure 38 - Route from USB to E-Beam

2. Relocate flow loop test cart and auxiliary test cart to electron beam processing area. Relocate control center cart to control area on the south side of the exit conveyor. Move flow loop test cart so the wheels straddle the electron beam cooling pad on the floor. **CAUTION**: Take extra care not to damage any part of the electron beam device, or the exposed cooling coils of the floor pad.
3. Connect all applicable electrical power, electrical signal, gas, and pneumatic lines from control cart to test carts. These include connections to thermocouples 1-6, heaters 1-4, the camera system, pneumatic valves 1-4, the motor VFD, and the gas lines for inert gas and bubbling gas, if applicable. Check table of connection numbers to ensure proper wiring. Turn on control systems and air compressor, and open manual valve to condenser. With pump motor off, and fire extinguishers depressurized with manual valves closed, test for readings reported from all transducers, signal from cameras, and proper actuation of pneumatic valves. **CAUTION**: Make sure to close manual valves in series with pneumatic extinguisher valves prior to testing. If fire extinguisher cartridges were activated and manual valves are open, testing pneumatic valves for extinguisher control will release extinguishing agent.
4. When all controls are verified in good working order, engage all heaters on tanks, lines, valves, and pumps. Monitor temperature of fluid; when temperature is high enough to enable flow, continue to step 5. On practice runs to check integrity of system, when temperature is sufficiently high, open valves and engage motor for a short time to check for flow in processing box. Watch all connections downstream of pump for any leaks. When flow is verified, disengage motor and close flow loop pneumatic valve. Leave heaters engaged.

5. Open all manual valves being used except for BV 2 at the bottom of the processed oil tank if semi-batch processing is desired. Open manual valve BV 3 between processing box and condenser, and condenser exhaust valve BV4. Open BV 5, 10, 11, and 12 for gas flow, and open BV 7 and 8 for the fire extinguishers.
6. After completing previous steps, fill condenser with liquid nitrogen or other cooling agent. Activate fire extinguisher pressure cartridges by punching cartridge tab. Experiment is ready to begin, and all personnel shall evacuate processing area and return to test control room before startup procedures. **CAUTION**: Wear insulated rubber gloves rated for cryogenics when handling liquid nitrogen to avoid contact with skin or frostbite.

III) Test Procedures

1. **Safety is paramount; adhere strictly to all electron beam facility safety guidelines. Exposure to direct electron beam discharge is lethal on an acute timescale.**
2. When electron beam device is ready for activation, engage pump motor, open valve for inert gas to fill storage tank being emptied, and open flow loop pneumatic valve. If operating with gas bubbling on the processing trough, open valve BV 6 for the added gas cylinder as well. Have facility technicians engage the electron beam at this time. Monitor system integrity for any leaks and fire risks using camera system.
3. During e-beam operation, open valve PA 2 for a few seconds to fill the gas sample bag, then close the valve.
4. Continue running electron beam until POC is filled.
5. If no issues with flow stoppage, system leaks, or instrumentation/camera monitoring failure, continue to standard shutdown procedure. If camera system fails due to exposure, fire monitoring capabilities are no longer functional and experiment must be stopped. If system has significant leaks from seal failure, follow emergency shutdown procedure. Monitor leaks for fire. If fire occurs at any time, follow emergency shutdown procedure with fire option.

IV) Standard Shutdown Procedure

1. Follow facility shutdown protocols for turning off electron beam.
2. Turn off heaters, VFD. Motor is stopped after electron beam is disengaged to avoid over-processing of oil that would otherwise be stagnant. Small contamination of unprocessed oil is preferable. Leave air compressor on in case fire suppression is needed.
3. Initiate remote inert gas purge of flow loop with valve BV 9 to remove as much hazardous produced gas as possible. If bubbling gas was used, set the T-valve between the inert gas and bubbling gas to purge any combustible gases from the line as well. Gases displaced from flow loop by inert gas purge are evacuated by electron beam facility exhaust venting. Close valves BV 5, 6, and 9 when purge is complete.
4. When safe to enter electron beam processing area, use portable gas detector to ensure no lethal gases are present.

5. Leave all flow loop connections intact and allow test cart frame to cool down before removing from processing area. Leave valves BV 3 and 4 on condenser open to allow condenser and processing box to exchange gases while coming to room temperature, as well as allowing condenser to exhaust at atmospheric pressure.
6. Turn of air compressor, disconnect transducer electrical connections and pneumatic valve air lines, coil and place on control center cart in an organized fashion.
7. Release pressure from air compressor.
8. Continue to processed sample extraction.

V) Emergency Shutdown Procedure

1. Use kill switch in LabVIEW control to disengage all heaters, and set valve to safe position. Turn off pump motor. Ensure air compressor remains on and fire suppression system is still on standby.
2. Shut down electron beam as soon as possible through facility protocols; **do not enter electron beam processing area until it is safe and clear to do so.**
3. Monitor system with cameras, if functional, for any problems while waiting to enter processing area.
4. If no fire, follow standard shutdown procedure if facilities (power, ventilation) allow for it. Otherwise, wait for services to be restored.
5. **IN CASE OF FIRE:** Open valves PA 3 and 4 as needed for extinguishers to dispense agent until fire is out. Close pneumatic valves to conserve agent if fire goes out. Call fire department. Monitor system for at least 5 minutes for self-reigniting fires and open pneumatic valves as necessary. Repeat until fire is fully suppressed. Begin residue cleanup after allowing surfaces to cool down.

VI) Processed Sample Extraction, Test Conclusion

1. Ensure that the condenser outlet BV 4 and purge inlet BV 3 are sealed and that the system is isolated.
2. Disconnect the fire extinguishers and purge gas line from the test cart.
3. Relocate the auxiliary cart to the electron beam control room.
4. Relocate the test cart to the mobile lab trailer.
5. If necessary disconnect the controls cart from main power and lines to e-beam vault.
6. If necessary relocate the control cart to the mobile lab trailer.
7. Secure items in the trailer if moving.
8. If necessary drive to the USB lab. Otherwise work at the e-beam facility is possible.
9. Prior to opening flow loop to extract oils ensure that ventilation system in trailer is turned on and face velocity exceeds 80 ft/min.
10. Flow loop may still contain hazardous head gases under pressure in the processing box and lines; remote ventilation and purging is required. With trailer parked in open-air environment, make sure all personnel are standing upstream of ventilation system. Ensure PA 2 to sample bag is closed and remove bag. Open condenser inlet and exhaust valves, and remote purge flow loop with inert gas prior to opening flow loop system.

11. Condenser and processed sample storage tank may be disconnected after ventilation. Monitor gas detector at all times to ensure no dangerous quantities of hazardous gases are being released. Record mass of full processed sample storage tank and mass of SOC and compare to masses before the test to calculate mass of processed liquid oil in container. Discrepancies in mass to be accounted for in gas production and residue in flow loop lines. Processed oil should flow more easily at handling temperature about 50 C; pour into sample collection jars and test for change in properties.
12. Open processing box and storage containers and clean using approved solvents and paper towels; dispose of waste cleaning supplies and residual oils properly. Be gentile when cleaning delicate items or adjustment mechanisms in processing box.
13. Fire extinguishers are still pressurized if not used after activating the cartridges; monitor pressure for future usability. If pressure is low, discharge remaining pressurized agent in controlled environment and refill extinguisher agent as necessary.
14. Clean any spills that occur from disconnecting flow loop components and dispose of cleaning supplies properly.

HAZARD ANALYSIS

I) Uncommon Hazards Associated with Experiment

1) High Radiation
    a. Following all safety protocols of electron beam facility will nearly eliminate chance of exposure.
    b. Facility inspected regularly for safety compliance.
    c. All personnel wear dosimeters inside facility to monitor possible exposure.

2) Hazardous Gases
    a. H2S, CO, CHx, others can be produced from processing.
    b. Portable gas detectors used to prevent entering areas with high concentrations of hazardous gases.
    c. Remote purging of system reduces risk of exposure.

3) High Heat Surfaces
    a. Heaters used to aid extraction of oil sample, prevent solidification of oil within flow loop lines.
    b. Heat resistant gloves used when handling high temperature surfaces to reduce risk of burns.

4) Flammable Substances
    a. Oils, produced gases, and cleaning solvents are flammable.
    b. Spill trays used to prevent oil from contacting ignition sources.
    c. Fire extinguisher system used to quickly suppress any flame on test cart.

5) Chemicals Used in the Research Project

Crude oil, mineral oil, mineral spirits, Purple K extinguisher

Required chemical inventory current and posted? **Y**
{Attach a copy of the current chemical inventory for this facility}

Safety Data Sheets (SDS)? **Y**
{Are current SDS's available for all chemicals?}

All stored chemicals segregated by Hazard Class? **Y**
{Stored chemicals must be segregated by Hazard Class.}

Flammables stored away from potential oxidizers. No oxidizers are present in the lab.

II) Potential Hazards

A) List all Physical Hazards That May Cause:

Electrical Shock – **Electrical connections and terminals**
Cuts – **Rough edges on flow loop test cart frame members**
Burns – **Heating pads and resistance heaters on tanks, lines, pump**
Abrasions –
Slips – **Oil spilled during transfer or from leaks in system**
Trips – **Electrical power cables, diagnostic cables, pneumatic lines**
Falls –
Amputations –
Other…

B) List all Chemical Hazards

{Identify the name and characteristics of each chemical}
{Use the HazCom Engineering Chemical Inventory form}

Acids
Bases
Oxidizers
Flammables – **Oils, volatile gases**
Solvents – **Mineral Spirits**
Toxic Chemicals – H_2S, CO as byproduct gases
Reactives and Explosives – H_2, CO and gaseous hydrocarbons as byproducts

Nanoscale Particles

C) Biological Hazards

*** If Biological Hazards are present, OSHA Bloodborne Pathogen requirements and CDC Universal Precautions shall be implemented, and appropriate PPE shall be provided. Note: Please attach appropriate documentation of requisite approvals & permits***

NONE

Microbiologicals	
Bacteriologicals	
Bloodborne Pathogens	
CDC Select Agent	
Biological Toxins	
Pathogenic Organisms	
Recombinant DNA (rDNA)	
Viruses	
Genetically Engineered Organisms	
Biological Safety Level (BSL)	
Other…	
IRB Approval	n/a
Human Subjects Approval	n/a
Animal Use Protocol (AUP)	n/a
Other…	

D) Secure, Segregated Chemical Storage:

{Chemical storage areas shall not be accessible to students/passers-by}
{All stored Chemicals and other hazardous materials shall be provided with secure storage and segregated by Hazard Class}

Yes

Locations: USB 127NB
Quantities: See chemical inventory
Authorized Person(s) Accessing the Chemicals: All lab personnel

E) Hazardous Waste Disposal

{All hazardous chemical waste materials must be contained, labeled, tagged, and disposed of in compliance with the TAMU Hazardous Waste Management Program}

YES

Chemical: **Crude oil**
Disposal method: **Stored in flammable cabinet till pickup by HWM**

F) Monitoring and Detection

H2S Detector – BW Honeywell GasAlert Clip Extreme H2S Model#: GA24XT-H

Handheld detector – BW Honeywell GasAlert Micro 5 PID

CO, CH4, H2 detector – Industrial Test Equipment Co HIC-822

G) List all necessary Personal Protective Equipment (PPE)

{All PPE shall be ANSI/NIOSH/MSHA approved, as appropriate}
{All use of respiratory protection & SCBA, must comply with the TAMU Occupational Health Program}

Long Pants, Long Sleeved Shirts	**Yes**
No Shorts, No Skirts	YES
Closed-Toed Shoes	**Yes**
Rubber Soled Shoes	**Yes**
Aprons/Lab Coats	**Yes**
Goggles/Face Shields	**Yes**
Gloves	**Yes**
Dust Masks	**Yes**
SCBA	No
Other…	n/a

H) Personnel Training Needed for Specific Hazards

{Identify the specific hazard and the individuals affected}

Principal Investigator:
Researcher/Lab Technician:
Graduate Student:
Student Workers:
Other…

POTENTIAL ACCIDENTS

A) "What if" Failure Analysis

Utility:	Planned Response (SOP's):
Electricity	**Follow emergency shutdown procedure.**
Gas Cylinder Malfunction	**Follow emergency shutdown procedure; procure new purge gas cylinder before opening system in controlled area.**
Air(compressor)	**Follow emergency shutdown procedure; use manual valve on purge gas cylinder to control flow as needed.**
VFD	**Follow emergency shutdown procedure.**
Heater Pads	**Follow emergency shutdown procedure.**

Control System Failure	**Turn off VFD, return all pneumatic valves to neutral positions, and follow emergency shutdown procedure as possible.**
Trailer Ventilation	**Exit trailer, use detectors to monitor hazardous gases when attempting re-entry at later time..**
E-Beam Facility Ventilation	**Follow emergency shutdown procedure.**

B) Leaks and Spills

Oil leaks can be wiped up using paper towels and properly disposed of.

[M]SDS Available:	Yes
Spill Kit Available:	Yes
PPE Available:	Yes
Containment Procedures:	Yes
Disposal Procedures:	Yes
Personnel Training:	Yes

C) Equipment Failure

{Attach Documentation of All SOP's for Emergency Shutdown Procedures}

Following the emergency shutdown procedure (in SOP above)

1. **Disengage all systems besides fire suppression.**
2. **Shut down electron beam.**
3. **Monitor for fires.**
4. **Follow standard shutdown procedure**

D) Fire Prevention *{Attach the following}*

Fire Extinguisher Locations: **On location with test cart.**
Building Emergency Evacuation Plan:
Evacuation Routes:
Emergency Response Procedure:
Incident Reporting & Notification Procedure: Notify campus emergency services (911 or 9-911 from campus phone)

DOCUMENTATION AND MAINTENANCE

A) Utility Shut-offs labeled:

Electricity	**Yes**
Vacuum	**n/a**
Gas	**n/a**
Air	**n/a**
Hot Water	**n/a**
Cold Water	**n/a**

B) Identify all necessary Warning Signs:

Equipment
Instrumentation
Utilities
Personal Protective Equipment
Reagent Bottles
Secondary Containers
Refrigerators and Microwaves
Chemical Storage **Flammable/hazardous gases clearly labeled**
*Emergency Contact Information (*ECI*)
{Must be current and posted on all entry door(s)}

V) Noise

Will the project/ generate excessive noise? **No**

VI) Standard Operating Procedures (SOP) for each Planned Task & Activity

Standard Operating Procedures (SOP) Identified: **Yes**
Safe Work Practices (SWP) Identified: **Yes**
Affected Personnel Trained on SOP's & SWP's: **Yes**
(Refer to training recordkeeping requirements)

VII) Ultimate Disposal Plan

A detailed plan is required for the ultimate disposal of unused equipment, materials, chemicals and wastes following project conclusion; includes the plans for:

- *Clean up and decontamination of instrumentation, equipment & facilities,*
- *Laboratory decommissioning and closure,*
- *Waste Minimization,*

- *Pollution Prevention (P^2),*
- *Environmental Stewardship & sustainability*

All project operations will be planned and managed for environmental sustainability, waste minimization and pollution prevention, as well as health, safety and security. Following completion of this project, all materials and equipment will be evaluated for future productive use, wastes will be disposed in compliance with the university's Hazardous Waste Management Plan, and the facilities will be cleaned and decontaminated as necessary to return the space to safe and productive usage.

VIII) List & attach all necessary Emergency Planning

Emergency Response Plan
Building Emergency Evacuation Plan
Emergency Contact Information (*ECI*)
{Must be posted on entry door(s)}
Spill Control Plan
Decontamination & Clean Up Plan
Other…

IX) Internal Safety Reviews *{ List all internal, self-inspection mechanism(s) to ensure compliance, abatement & accountability }*

Procedure for Periodic Internal Safety Audit & Review:

1. The PI or designee will inspect the laboratory weekly, document findings, and implement corrective action within 24-hr.
2. The Department's designated Safety Officer will conduct monthly inspections, document findings, and implement corrective action within 24-hr.
3. EHSD will conduct an annual laboratory safety inspection, issue a documented safety inspection report, and conduct follow up inspections to ensure prompt corrective action.
4. EHSD will conduct periodic shop safety inspections, issue a documented safety inspection report, and conduct follow up inspections to ensure prompt corrective action.

X) NanoTechnology and Nanoscale Materials

All work with NanoTechnology and/or Nanoscale Materials must be conducted in accord with the TEES and Engineering "*Guideline for Working Safely with Nanotechnology, Nanoscale Materials & Particles*." By submitting this PSA document I/we declare my/our commitment to comply with best practices and with all provisions of this Guideline in order to prevent potentially harmful exposures to nanoscale materials.

All Engineering projects involving the use of nanotechnology and/or nanoscale materials must be conducted in accord with the Engineering Guideline for Working Safely with Nanotechnology and Nanoscale Materials." By signature on this PSA document, the PI and all affected project personnel confirm they have read and are familiar with all provisions of the Guideline, and pledge to conduct all project operations in compliance with the Guideline and with current Best Practices as published by the National Insititute for Occupational Safety & Health (NIOSH), the Occupational Safety & Health Administration (OSHA), and the U.S. Environmental Protection Agency (EPA)

NOTE: The Engineering Guideline for Working Safely with Nanotechnology and Nanoscale Materials is available on the Engineering SafetyNet web site at http://engineering.tamu.edu/safety/ , under “Guidelines.”

XI) Commitment to a Safe, Healthful and Secure Workplace Environment

By submitting this PSA document I/we declare our commitment to full compliance with federal & state law, and with TAMU & TEES rules and requirements for a safe, healthful, secure workplace environment, in support of our goal for safe and productive research outcomes.

XII) Checklist:

Checklist to be completed in following order, all steps required:

1) Sample Loading

- ○ Adorn PPE: gloves (high heat when needed), coat, goggles
- ○ Select oil container to be used, bring to fume hood
- ○ Loosen vent caps
- ○ Place barrel in cradle, secure heaters and extraction pipe
- ○ Turn on heaters
- ○ Close BV 1 and remove SOC top lid
- ○ Clean intermediate container for oil transfer
- ○ Tilt cradle to pour oil into intermediate container
- ○ Return barrel to upright position, turn off heaters with vents open
- ○ Record mass of intermediate container with oil

- Pour oil into SOC, replace top lid
- Record mass of intermediate container after oil deposited
- Calculate mass of oil sample loaded
- Close BV 4
- Perform leak test with inert gas

2) Test Preparation

- Load necessary carts and equipment on transport trailer
- Secure items for transport
- Relocate carts within e-beam facility, test cart straddles cooling pad
- Connect all wires and lines to cart
- Open BV 3, 4
- Turn on air compressor, control cart systems
- Close BV 7, 8
- Check for readings from all connections, actuation of pneumatic valves, VFD control of motor
    - PA1
    - PA2
    - PA3
    - PA4

    - TC1
    - TC2
    - TC3
    - TC4
    - TC5
    - TC6

    - Camera

    - VFD

- Open manual ball valves
  - If semi-batch processing, BV 1, 3, 4, 5, 7, 8, 10, 11, 12
  - If continuous flow, also open BV 2
  - If bubbling gas, also open BV 6
- Engage all heaters
- Fill condenser bath tank with LN2
- Activate fire extinguisher cartridges
- All personnel leave processing area

3) Test Procedure

- Adhere to e-beam facility safety guidelines
- Open PA 1
- Engage VFD and motor
- Switch on e-beam
- Momentarily open and then close PA 2
- Monitor cart for fire/failure with camera until test conclusion

4) Standard Shutdown

- Follow e-beam facility procedure for disengaging e-beam
- Turn off heaters, VFD
- Open BV 9 to purge flow loop
  - If bubbling gas, change T-valve to purge CH4 line
- Close all PA valves
- Use gas detectors to ensure safe entry to processing area
- Allow test cart to cool before moving, leave valves open between BOX, CON, and EXH
- Turn off air compressor, release pressure
- Disconnect all lines from test cart, organize in coils on control cart

4) Emergency Shutdown

- Use kill switch on control cart, turn off VFD
- Follow e-beam facility procedure for disengaging e-beam
- Monitor system for fires while waiting to enter
- In case of fire, call fire department immediately
- Use extinguishers to suppress flames
- Monitor for reigniting fire
- Follow standard shutdown procedure when safe

5) Sample Extraction

- Close BV 4
- Disconnect extinguishers, purge lines from test cart
- Move auxiliary cart to control room
- Move test cart to transport trailer
- Relocate control cart to trailer if necessary
- Secure for transport if moving
- Engage transport trailer ventilation
- Check for minimum 80ft/s face velocity
- Close PA 2, remove BAG
- Stand upwind of test cart, open BV 4
- Purge system with inert gas
- Close BV 3, disconnect CON from cart
- Retrieve processed oil from POC and CON, place in labeled collection jars for testing

- Clean system and any spills with approved solvents, paper towels
- Dispose of cleaning supplies properly

XIII) Safety Agreements *{Signatures are required to document the commitment of each participant in maintaining the safe, healthful, and secure project environment}*

Location of Project Records & Files:

USB 127NB – Oil & Soil Cabinet

Signed By:

Principal Investigator

Co-Principal Investigator

Co-Principal Investigator

Co-Principal Investigator

Graduate Students

Graduate Students

Graduate Students

Graduate Students

Graduate Students

Graduate Students

Graduate Students

Graduate Students

Graduate Students

Graduate Students

XIV) Training Plan

The following courses shall be taken by all lab personnel involved with this project before beginning work:

TrainTraq 2111131 – Responsible Conduct of Research
TrainTraq 2111743 – Lab Safety
TrainTraq 1211011 – Research Lab, Shop, and Chemical Safety
TrainTraq 211228 – Working Safely with Cryogenics
TrainTraq 211129 – Hands-on Fire Extinguisher Training
TrainTraq 211138 – On-Line Hazard Communication Training – EHS
TrainTraq 811013 – TEES Shop and Tool Safety Course

APPENDIX B – OIL DECANTER CART SOP

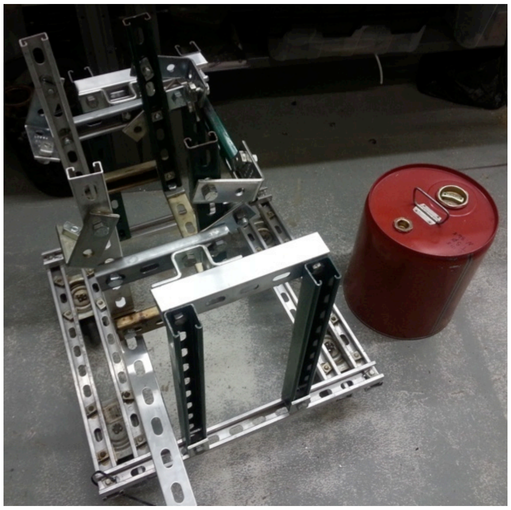
This is the operating procedure for safe use of the oil decanter cart. These instructions ensure that no personnel following them come into contact with any safety risks posed by heating a heavy container of oil.

Overview

The oil extraction cradle provides a safe method to create samples of high-viscosity oil. Some oils used in the lab are too viscous to flow at room temperature and must be heated before they can be poured, but the heaters become far too hot to safely handle the barrel by hand and pose a burn hazard. By using the extraction cradle, surface temperature and weight of the barrel are no longer an issue and all lab personnel can safely extract oil samples. When using the cradle, there are steps to follow to ensure proper operation.

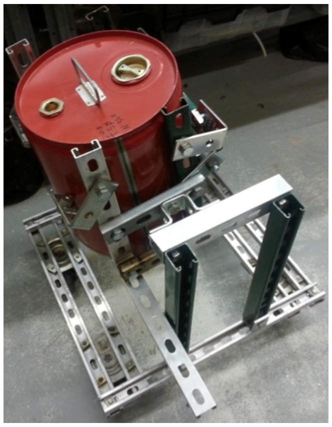
Preparation

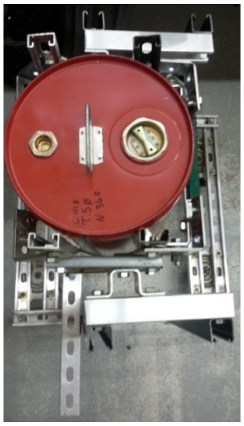
1) Adorn PPE, including safety glasses, vinyl gloves, and a lab coat.
2) Obtain heat resistant gloves for later use.
3) Obtain a clean, 1-gallon paint can for later use in sample extraction.
4) Select a barrel for extraction. Be sure that both the larger cap and the smaller vent cap can be opened.
5) Secure thin barrel heaters to the barrel.



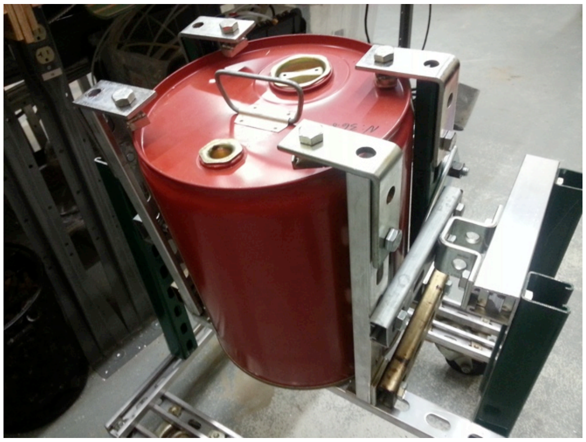
Procedure

1) Place the barrel with heaters in the cradle; note the orientation. The larger cap should face the green horizontal support, with the extended handle on the other side.

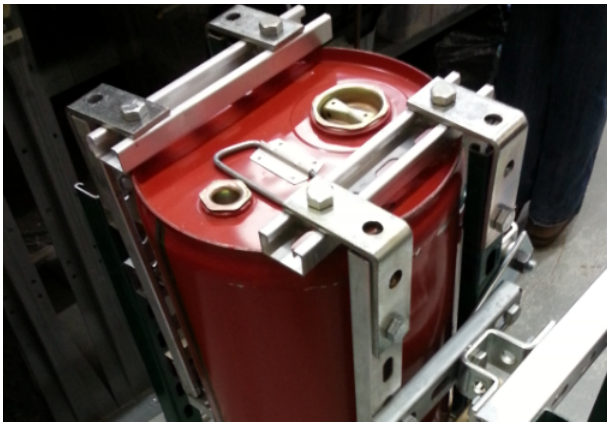




2) Rotate the four 90-degree brackets on top of the four barrel supports to the top surface of the barrel.



3) Insert the top crossbars and allow the flat surface of each crossbar to rest on as much of the lip of the barrel as possible. Be sure that the Unistrut rectangular nuts are grabbing the folded lip of the channel. Tighten the two bolts on each 90-degree bracket.



4) Rotate the barrel through the full range of motion to ensure it is firmly secured in the cradle before proceeding.

5) When the barrel is verified as being secure, lock the four cart wheels, remove the caps on the barrel, and install the 2” NPT extraction pipe on the larger hole.

6) Place a thermocouple in the smaller ventilation hole to monitor the temperature of the oil. With the barrel in an upright position, engage the heaters and wait for the oil temperature to rise to roughly 50 degrees Celsius for McKay River oil and 70 degrees Celsius for Hamaca oil.

7) Put on heat resistant gloves at this time. When the oil is warmed enough to pour, place the empty paint can on the ground under where the extraction pipe will be. Using the extended handle, slowly rotate the barrel to pour a sample into the paint can.

Shutdown

1) When extraction is complete, rotate the barrel back to an upright position and disengage the heaters.

2) Use a spatula to clean the lip of the extraction pipe. Wipe up any spills that may have occurred using paper towels and appropriate solvents while the oil is still warm enough to more easily be cleaned.

3) Allow oil barrel and heaters to cool off before removing barrel from cradle. Removal process is reverse of loading process:

    a. Loosen but do not remove bolts on the four top 90 degree brackets
    b. Slide out top crossbars
    c. Rotate 90 degree brackets out of the way of the barrel
    d. Remove barrel from cradle
    e. Remove heaters from barrel

APPENDIX C – TRAILER SPECIFICATIONS

This is the document that accompanied the mobile test cell from the fabricators who mounted the container on the trailer. This certifies that the test cell is fit for use as a fume hood and is also compliant with DOT regulations for use on the road.

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DESIGN · PREDICT · BUILD · INNOVATE

12600 Hwy 30, Suite 100
College Station, TX 77845

979-703-1949

www.exosent.com

Walk-in Hood Trailer

Customer : **Prof. David Staack**
Department of Mechanical Engineering
Texas A&M University
College Station, TX 77845
Date: May 18, 2015
Lead Engineer: **Yuval Doron**, M.S., E.I.T.
President and co-founder
Exosent Engineering, LLC

Exosent Engineering was tasked with mounting and modifying a container for a walk-in hood trailer.

- **Ventilation:** The installed fans have certified CFM of 95 and 120 fpm. This provides adequate ventilation.
- **Spark safety:** per NFPA 45 (2015 edition)
  - The fans are driven by externally mounted non-sparking motors (Dayton 43Y138)
  - All electrical switches are on the exterior of the trailer.
  - All lighting vents to exterior, isolated from interior through plexiglass.
  - Oil (up to 2 gallons) used is a category 4 flammable liquid, and is stored in a container during transport.
  - Solvents used are category 2 and 3, and not stored during transport.
- **Meets NFPA 45:** *Standard on Fire Protection for Laboratories using chemicals*
  - §2.2.1 Mobile lab is classified as Class D (Minimal Fire Hazard)
  - §3.1.1 As a class D with $< 10,000$ ft^2 area, no fire separation is required.
  - §3.1.9 Ventilation opening meets NFPA 90A *Standard for the Installation of Air-Conditioning and Ventilating Systems*.
  - §5.1.2 Explosion Hazard Protection is provided by minimization of explosive materials and the isolated nature of the mobile lab.
  - §6.2 Ventillation system minimizes fire hazards (see *Spark Safety* section) and ventilation system is not used to provide blast protection
  - §6.3 Isolated nature provides infinite supply of air
  - §6.4 Exhaust air is not recirculated.
  - §6.7 Exhaust fans were selected for fire, explosion, and corrosion requirements, with easy access for repair.

- Full compliance requires lab director to create, inspect and enforce the following:
    - §3.4.1 With no secondary exit, no fire or explosion hazard may be between workers and exit.
    - §3.4.5 Natural or external lighting must be provided to meet emergency lighting. requirements. All work performed with doors open.
    - §3.5 All furniture and equipment must be arranged so an exit can be easily reached.
    - §3.6 All electrical work must comply with NFPA 70 *National Electrical Code*.
    - §4.4 Class B Fire Extinguisher must be on hand.
    - §4.5 Fire detector must installed and tested every 6 months.
    - §4.6 Fire prevention and emergency plan procedures must be established.
    - §5.4 Properly post on doors "Laboratory contains an explosion hazard."
    - §5.5 Annual inspection of structure required.
    - §6.8.7 Flow measurement device in front of exhaust fans must be implemented to provide worker indication of adequate airflow.
    - §6.13 Annual inspection of exhaust system required.
    - §7.2 All chemicals shall be properly stored commensurate with quantities and hazards of chemicals involved.
    - §7.2.1.3 No category I or II liquid shall exceed capacity of 1.1 gal (4 L).
    - §7 Procedures for handling, transfer, and storage of hazardous chemicals shall be created and implemented per this section.
    - §8 Procedures for handling, transfer, and storage of compressed or liquefied gas shall be created and implemented per this section.
    - §9 Laboratory Operating Procedures shall be created and implemented per this section.

- **GAWR:** The total weight of trailer with permanent mounted items is 9,170 lbs. Listed GVWR from BigTex 140A-20 is 14,000 lbs.
- **DOT:** The trailer is unmodified and thus is still DOT compliant.

STATE OF TEXAS
ANDREW T. DUGGLEBY
107260
LICENSED
PROFESSIONAL ENGINEER

Certified by: Andrew Duggleby, Ph.D., P.E., C.W.I.
Title: Chief Technical Officer / Co-founder
Company: Exosent Engineering
Date: May 18, 2015

APPENDIX D – OIL CART PURGE

This is the document that was written to determine procedures for a safe inert gas purge of the flow system. Conditions were established and evaluated to calculate the number of air changes required to achieve safe concentrations of hazardous gases.

Introduction

During experiments performed using the flow loop at the electron beam facility, hazardous gases will be produced as a result of the treatment process. These gases include hydrogen sulfide (H_2S) and various hydrocarbon species (C_xH_x). Hydrocarbons are combustible when mixed with air and exposed to an ignition source, and hydrogen sulfide is especially hazardous to health by exposure. Before accessing processed samples from the flow loop, these gases must be purged from the system until concentrations are below a desired upper limit. Flushing the system with an inert gas for multiple volume changes is the chosen method of purging these hazardous gases.

Calculations

Hydrogen sulfide is a distinctly pungent gas that is detectable by the human nose at concentrations as low as 0.5 ppb [1]. An assumption for the upper limit of H_2S concentration will be made at 100 ppm at the conclusion of the electron beam process. It is desirable to bring concentrations below the smell limit. Hydrocarbon concentrations are assumed to be as high as 100%, and it is desirable to lower that concentration to 0.1%.

The flow loop system consists of four lengths of flexible hose, a sample storage tank, a processed sample collector, a processing box, and a gas condenser. The condenser is not included in the purge volume estimate because it will exhaust H_2S as it is produced during the experiment, and will be isolated at the conclusion of the experiment for analysis. The volumes of the smaller components such as valves and pumps are negligible on a scale of cubic feet, and the relevant volumes are shown below in Table 8. A simple diagram of the processing box dimensions is shown in Figure 39:

Table 8 - Component volumes

Component	I.D. (in)	Length (in)	Volume (in^3)
Hose from Storage to Pump	0.375	24	2.651
Hose from Pump to Valve	0.5	48	9.425

Table 8 - Continued

Component	I.D. (in)	Length (in)	Volume (in^3)
Hose from Valve to Box	0.25	63	3.093
Hose from Box to Storage	0.5	83	16.30
Storage Tank	4	15	188.50
Sample Collector	3	18	127.23
Processing Box			2051.3
Total			2398.5 = **1.39 ft^3**

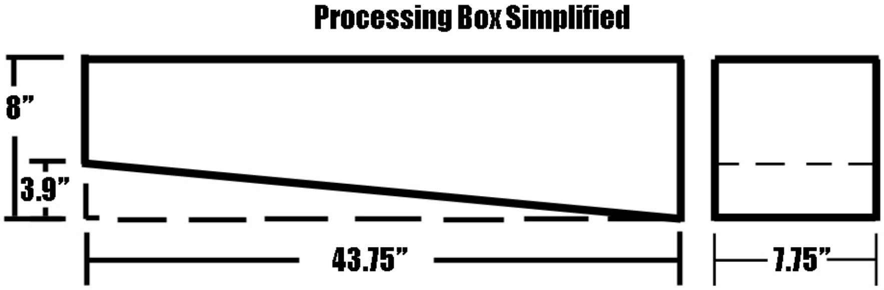

Figure 39 - Processing Box Diagram (not to scale)

It is estimated that one full volume change within the system reduces contaminant concentrations by 50%, so Equation 1 is used to determine the number of volume changes, N, needed to bring concentration, C, from an initial level to a desired final level:

$$C_f = C_i \left(\frac{1}{2}\right)^N \tag{1}$$

To reduce H_2S concentrations from 100 ppm to 0.5 ppb, it takes at least 18 volume changes, which equates to 25 cubic feet of inert gas given the volumes defined above.

Sources:

[1] CDC Agency for Toxic Substances and Disease Registry, "Public Health Statement – Hydrogen Sulfide," published October 2014, accessed 31 March 2015.
http://www.atsdr.cdc.gov/ToxProfiles/tp114-c1-b.pdf