MATRIX ACIDIZING PARALLEL CORE FLOODING APPARATUS

A Thesis

by

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ABSTRACT

Matrix acidizing is a well stimulation procedure where acid is injected down the wellbore or coil tubing and into the reservoir near the wellbore region. Wellbore damage is a common issue in the oil field. The primary goal of matrix acidizing in carbonate reservoirs is to bypass wellbore damage by creating highly conductive channels that go several feet into the formation, known as wormholes.

The goal of laboratory experiments is to find an optimum injection rate to create dominant wormholes and provide this information to the field. To conduct various experiments, core flooding setups are created. The setup consists of a core holder, accumulator, overburden pump, injection pump, accumulator, pressure sensors, and a back pressure regulator. Results from matrix acidizing core flooding in laboratory conditions provide an understand for wormhole growth, acid diversion, injection rates, and adds a variety of liquid chemicals for testing at reservoir pressures and temperatures.

The first objective was to design, assemble, and test a matrix acidizing parallel core flooding apparatus. The apparatus was rated for 5,000 psi and 250 ºF. The design uses 36 valves to configure small, medium, and large core holders. The key feature is the ability to run parallel core floods at high pressure and high temperature while providing resistance against corrosion from acid. Every flow line on the apparatus is sealed and acid is contained in closed flasks to provide a longer lifespan. With a narrow frame

design, the apparatus is readily portable. Electrical components were enclosed and wiring was shielded. The heating system was designed to maintain temperature. The pressure transducers were appropriately calibrated. A LabVIEW VI file was written with proper function and calibration. A transformer was provided to allow the apparatus to function with different voltage. The apparatus was tested and proved to have repeatability with accurate calculations. The second objective was to create a documented method for conducting experiments.

DEDICATION

To my mother and father.

ACKNOWLEDGEMENTS

I would like to thank my committee chair, Dr. A. D. Hill, and my committee members, Dr. Ding Zhu and Dr. Yuefeng Sun, for their guidance and support. Thank you for allowing me the wonderful opportunity to design and built an apparatus that involved mechanical, electrical, software, and petroleum engineering. I truly enjoyed working on this research for Dr. A. D. Hill and I gained valuable knowledge and skills. Thank you for giving me the opportunity to visit Doha, Qatar. Thank you for teaching and inspiring me.

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NOMENCLATURE

$\emptyset$	Porosity
ρ	Density
μ	Viscosity
D	Diameter
k	Permeability
L	Length
m_{dry}	Dry Mass
$m_{saturard}$	Saturated Mass
NPT	National Pipe Thread
ΔP	Differential Pressure
T	Temperature
t	Time
PV	Pore Volume
PVBT	Pore Volume to Breakthrough
Q	Flow Rate
V	Volume
v_i	Interstitial Velocity
Wt	Weight Percent of Acid or Water

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1. INTRODUCTION

1.1 Objective

The first objective of this research project was to design, assemble, and test a matrix acidizing core flooding apparatus with parallel core flood capabilities and a three core setup. The second objective was to provide users with an instruction manual which includes a procedure for conducting experiments and equipment troubleshooting solutions.

1.2 Matrix Acidizing

Matrix acidizing is a well stimulation procedure in which acid solution is injected into formation to dissolve minerals resulting in the increase of permeability in the wellbore region. Formation damage is a common issue in the oil field and it occurs during drilling, cementing, perforating, gravel packing, production, well workovers, chemical treatments, and injection operations. During drilling, mud may enter high permeable zones and becomes difficult to remove. During perforating, permeability around the perforated hole may be reduced 2 to 20 times less than the original permeability. Formation movement can cause damage to a producing well (O. McLeod Jr. 1984). The concept of matrix acidizing is to improve the permeability of the wellbore region without fracturing the production region (Economides et al. 1994). The goal of matrix acidizing in carbonate reservoirs is to bypass wellbore damage by creating highly conductive channels that go several feet into the formation, known as wormholes. The type of acid used depends on the formation. Hydrofluoric acid is recommended for sandstone formations and Hydrochloric acid is recommended for carbonate formations. With carbonate reservoirs the following chemical reactions take place:

Calcite: $CaCO_3 + 2HCl \rightarrow CaCl_2 + CO_2 + H_2O$

Dolomite: $CaMg(CO_3)_2 + 4HCl \rightarrow CaCl_2 + MgCl_2 + 2CO_2 + 2H_2O$

Wormholes can vary in growth based on acid type and acid concentration, mineralogy of the rocks, temperature of the formation, and most importantly, the injection rate of acid. Each matrix acidizing stimulation conducted in wells have an optimum injection rate. When the injection rate is too low then dissolution occurs on the wellbore and the acid cannot enter the damaged zone (Wang et al. 1993). With an injection rate too high, the wormhole branches out.

1.3 Core Flooding

The goal of laboratory experiments for carbonate acidizing is often to find an optimum injection rate to create dominant wormholes. To conduct various experiments, core flooding setups are created. The setup consists of a core holder, accumulator, overburden pump, injection pump, accumulator, temperature sensors, heaters, pressure sensors, and a back pressure regulator. Rock samples are obtained either during drilling with a core drill or from outcrops. The rock sample cut into the appropriate size and placed inside the core holder. The sides of the core holder are pressurized with hydraulic oil from the overburden pump. The injection pump provides hydraulic oil or water to the accumulators which inject brine, acid, or chemicals into the core holder. A back pressure regulator allows pressure to build inside the core holder to match reservoir conditions.

By using heating tape around the core holder, reservoir temperature is matched. Pressure sensors monitor the inlet and outlet pressure of the core holder and pressure gauges monitor various sections of tubing. This system is used for matrix acidizing, relative permeability tests, formation damage tests, enhanced oil recovery tests, water flooding, and various stimulation studies. The core flooding apparatus studies acid diversion as well with a parallel core holder configuration.

1.4 History of Matrix Acidizing in Core Floods

Matrix acidizing core flooding in laboratory conditions provides an understanding for wormhole growth, the effect of injection rates, and adds testing capability for a variety of chemicals. Core size dependencies seen in laboratory acid-flood experiments also change the wormhole characteristics (Furui et al., 2010). Research was conducted to find optimum injection rates which were the primary source to creating optimum wormholes (Wang et al., 1993). The effects of phase saturation conditions on wormhole propagations in carbonate acidizing were studied by Shukla, Zhu, and Hill (2006). Matrix acidizing core flooding apparatus were described by Gomez (2006), and Grabski (2012). Grabski (2012) discusses the equipment and procedure for a two core holder apparatus. A parallel core flood setup was described by Alghamdi (2010). This research combines design knowledge from previous work.

1.5 Design Requirements

1.5.1 Pressure

Insufficient back pressure is a common problem in matrix acidizing. During acidizing, carbon dioxide is produced and stays in solution as long as adequate back pressure is applied. Without adequate back pressure, carbon dioxide changes into the gas phase. The back pressure regulator was designed up to 6,000 psi of pressure provided from a nitrogen source. The fluid flow system is rated for a maximum of 5,000 psi based on valve and tubing pressure limits. The injection pump provides up to 7,500 psi to the flow lines. Overburden pressure is required to confine the rock sample inside the core holder.

1.5.2 Temperature

Temperature is required during experiments to match reservoir conditions. Heating tapes and a fluid heater were used to add heat to core holders and liquids respectively. Thermocouples and a thermo-probe monitor the temperature in each core holder and the liquids. The apparatus was rated up to 250 ºF.

1.5.3 Cost of Material

Material was selected based on corrosion resistance, high pressure, and high temperature ratings. HCl and HF are corrosive and high temperature accelerates corrosion inside the components. Hastelloy C-276 was selected to be used on the apparatus due to high resistance against acid. Hastelloy is ten times the cost of stainless steel 316. Therefore, components with minimal acid contact were designed with stainless steel 316. A combination of the various materials was chosen appropriately to meet the budget requirements. Valves, fittings, and tubing were placed on the frame with easy to reach locations to allow quick replacement.

1.5.4 Air and Liquid Leaks

The apparatus was designed with bleed valves to release trapped air from flow lines. Trapped air affects pressure readings during experiments. The system may also have leaks due to fittings not connected properly or due to corrosion. Air bleed ports were created to ensure air did not get trapped in the flow lines, accumulators, injection pump, and core holders. The apparatus was fully tested with no leaks after assembly was completed.

1.5.5 Mobility

The apparatus required a limited frame design to allow shipment to Texas A&M University in Qatar. The frame was designed at 31 inches wide x 71 inches height x 85 inches length. The narrow design allows transportation through laboratory doors, elevators, and meets requirements for air fright. Electronics on the apparatus were designed for both 110 Volts and 220 Volts using a transformer.

1.5.6 Parallel Core Flood Capability

The apparatus holds three size core holders and required the capability of running two core floods simultaneously to test acid diversion. This was accomplished with a 36 valve system and fraction collectors to record flow rate.

1.5.7 Design Safety

The apparatus frame was designed to minimize hazards. Components were set to eye level and reachable without effort. A plastic cover protects liquids from escaping outside the protected area. Electrical wiring was encased and closed to prevent corrosion. The apparatus requires placement under ventilation. Safety of the user is the first priority of any laboratory. Users must familiarize themselves with all laboratory procedures when dealing with acid and equipment at high pressure and high temperature. Laboratories must provide safety symbols and statements warning of potential hazards. Users are recommended to wear steel toe shoes during transportation of core holders. Personal protective equipment is provided, which includes lab coat, gloves, eye glasses, face shield, and gas mask. Long pants and covered shoes must be worn. Information on proper safety was provided in the procedure.

1.6 Approach

The project began with understanding equipment from a previously built two core holder apparatus. User inputs were gathered to understand effective techniques to run experiments. Improvements for the three core holder apparatus were designed. Literature review was conducted to understand the assembly of parallel core flooding and equipment. Components were listed and vendors were contacted to gain knowledge on the specifications, application, and cost. A visit to Texas A&M University in Qatar was conducted to understand laboratory conditions. Once the final design was confirmed, components were ordered from vendors. Each vendor had a purchase order or bid placed. Next, components were received and assembled. The frame and electrical system required service assistance. New LabVIEW software code was written for controlling four pressure transducers, three heating tapes, and one fluid heater. Once the apparatus was assembled, parallel core floods were successfully conducted and a laboratory procedure was written. Finally, shipment was planned.

2. MECHANICAL EQUIPMENT

2.1 Equipment Introduction

This chapter describes components required in the assembly of a core flooding apparatus for acidizing experiments. The components were selected based on cost and specifications given by vendors. Components satisfied the system requirements of 5,000 psi pressure rating, 250 ºF temperature rating, and corrosion resistance. A 36 valve system was designed to control fluid flow. The fundamentals of this chapter are to provide the user with a complete schematic, component specifications, safe operational procedure, and troubleshooting solutions. Chapter 3 provides further details on the heating system and the data acquisition system.

2.2 Components and Schematic

A basic matrix acidizing core flooding apparatus consists of core holder, injection pump, overburden pump, nitrogen tank, back pressure regulator, accumulator, pressure transducer, acid and brine accumulators, refill tank, pressure gauges, the heating system, and the data acquisition system. The apparatus is placed on a frame and components are connected with tubing, unions, fittings, and electrical wires. **Figure 1** displays a basic schematic of a core flooding apparatus. **Figure 2** displays a schematic of the current apparatus at Texas A&M University in College Station with a completed valve configuration.

Figure 3 displays the three core holder final schematic used to design a parallel core flooding apparatus for Texas A&M University in Qatar. The parallel core flooding apparatus is the basis for procedure instructions shown in Chapters 3-7. The frame holds three core holders: large (4 inch diameter by 20 inch length), medium (1.5 inch diameter by 20 inch length), and another medium which is switched out for a small size (1 inch diameter by 6 inch length). Two medium size core holders run parallel core flood experiments. The parallel core flooding apparatus requires two fraction collectors in addition to the previous specified list of components. **Table 1** shows the summary list of major components, vendors, and specifications required for parallel core flooding apparatus.

2.3 Fluid Flow

Based on schematic shown in **Figure 1** fluid flow is analyzed. The starting point is from the injection pump and ends as the flow lines exit the back pressure regulator. The injection pump provides hydraulic oil or water to the top of each accumulator. The accumulators are filled with brine or acid by the use of a refill tank. Brine or acid is injected into the flow lines, through a fluid heater into the core holder. During the experiment, the overburden pump provides hydraulic oil to the core holder and the nitrogen tank provides pressure to the back pressure regulator. Liquid exits the flow lines when the injection pump pressure is higher than the back pressure.

The parallel core flood schematic is based on the same principle but requires liquid to be collected after exiting the back pressure regulator into two fraction collectors. The flow lines are controlled with seven valve sets: injection/refill, core holder selection, back pressure, overburden, fraction collector, bypass, and pressure transducer.

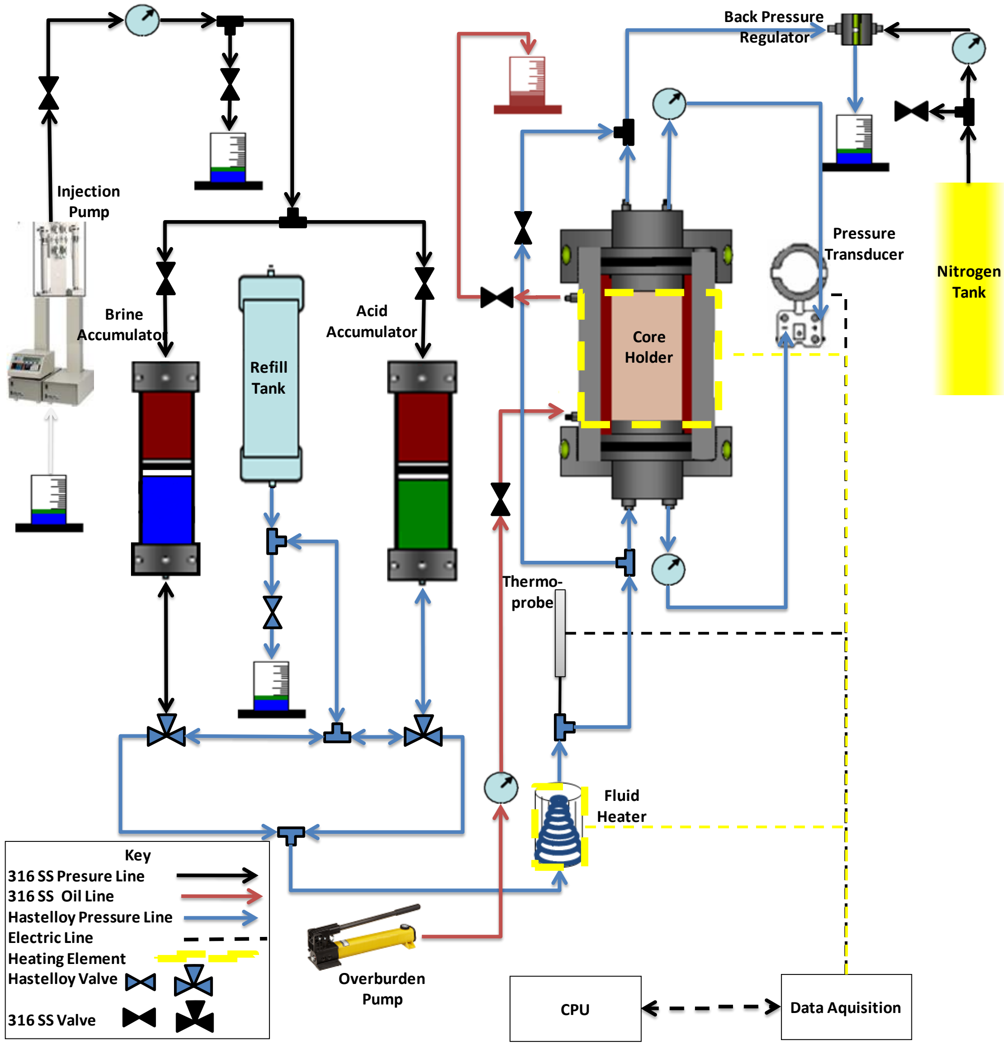


Figure 1: Matrix Acidizing Single Core Holder Schematic

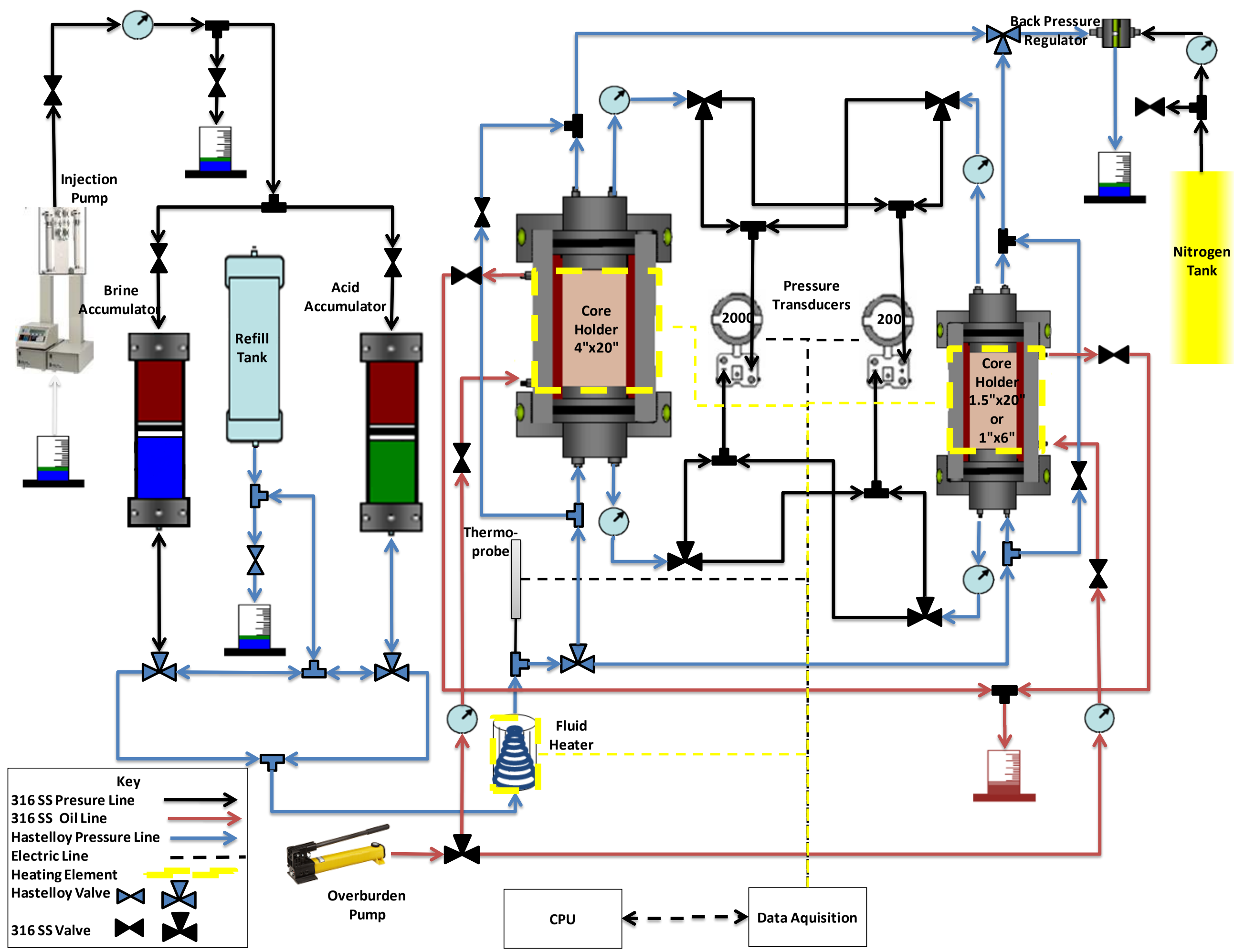


Figure 2: Matrix Acidizing Two Core Holder Schematic

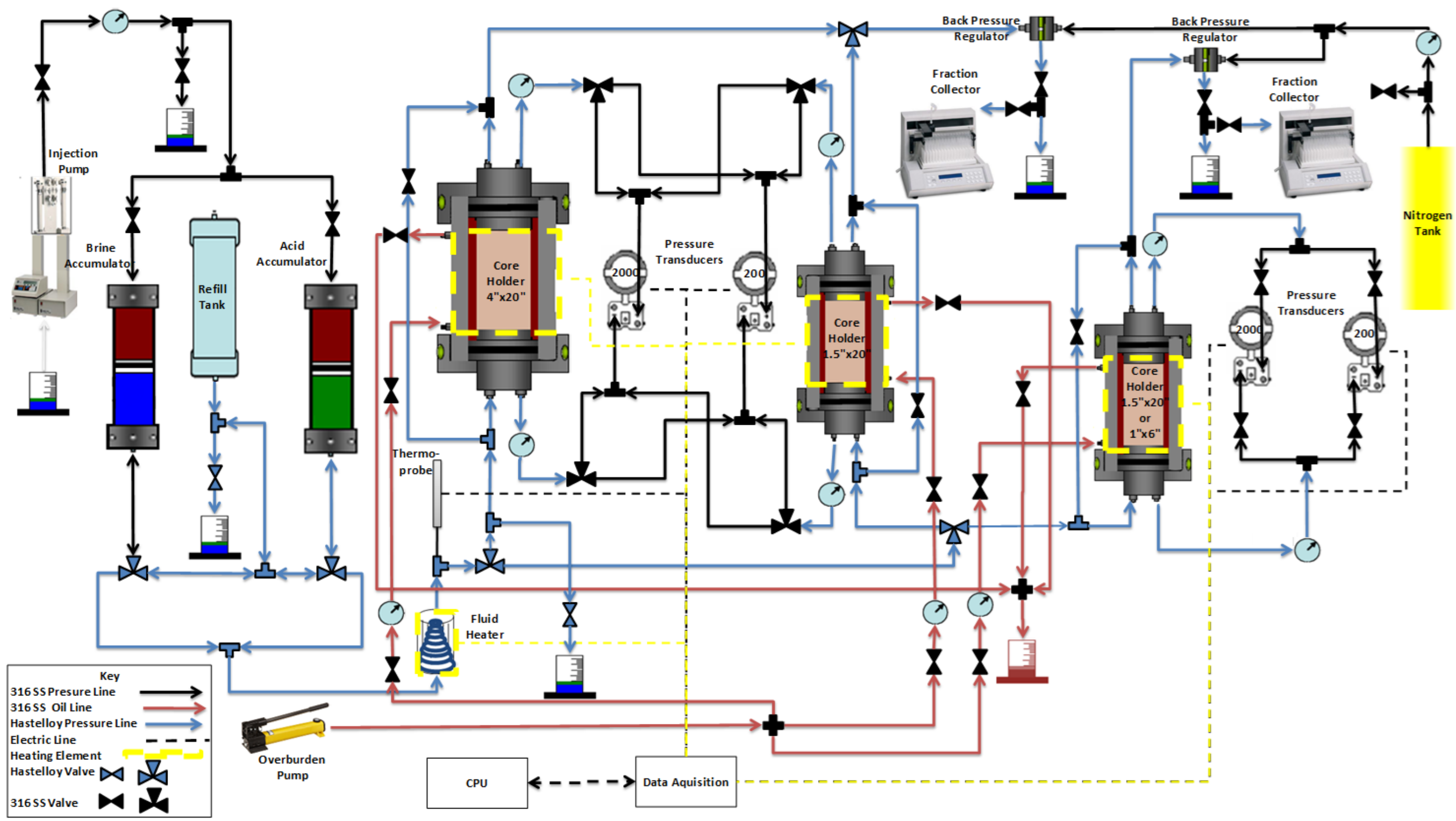


Figure 3: Matrix Acidizing Three Core Holder Schematic

Table 1: Components

Item	Material	Pressure psi	Temp F	Vendor	Unit Price	Quantity	Price
Back Pressure Regulator	SS 316/HC276	6,000	464	SunSource	$ 3,293.00	2	$ 6,586.00
Acid Accumulator	HC276	5,000	250	Phoenix Instruments	$ 5,175.00	1	$ 5,175.00
Brine Accumulator	SS 316	5,000	250	Phoenix Instruments	$ 3,925.00	1	$ 3,925.00
Small Core Holder (1"x6")	HC276	5,000	300	Phoenix Instruments	$ 5,185.00	1	$ 5,185.00
Medium Core Holder (1.5"x 20")	HC276	5,000	300	Phoenix Instruments	$ 7,945.00	2	$ 15,890.00
Large Core Holder (4"x 20")	HC276	5,000	300	Phoenix Instruments	$ 9,350.00	1	$ 9,350.00
PVC Refill Tank	PVC	100		Phoenix Instruments	$ 475.00	1	$ 475.00
Pressure Gauge	SS 316	6,000	212	AWC	$ 80.44	11	$ 884.84
Overburden Pump		10,000		Enerpac	$ 424.50	1	$ 424.50
Injection Pump		7,500		Teledyne Isco	$ 24,299.19	1	$ 24,299.19
80/20 Frame	Aluminum			Shepherd Controls	$ 5,826.55	1	$ 5,826.55
Fraction Collector	PTFE			Gilson	$ 2,821.00	2	$ 5,642.00
Valves	SS 316/ HC 276	5000	250	Swagelok, HIP	-	36	$ 8,554.00
Tubing and Fittings	SS 316/ HC 276	7,882	300	Swagelok, AWC	-	-	$ 10,585.00
Pressure Transducer 200 psi	SS 316	200		Fox Boro	$ 1,639.25	2	$ 3,278.50
Pressure Transducer 2000 psi	SS 316	2,000		Fox Boro	$ 1,707.33	2	$ 3,414.66
Heating Elements	Duo Tape, Fiberglass		1,400	HTS/Glascol	-	-	$ 1,046.00
Data Acquisition / Transformer	Fiberglass Enclosure			Britt Rice, NI	-	-	$ 5,640.00
Shipping	Air Freight Crate			LogNet Worldwide	-	-	$ 4,949.25
Total							**$ 121,130.49**

2.4 Frame

3-D CAD software, SolidWorks, was used to design the frame for the parallel core flood apparatus. The previous two core holder apparatus shown in **Figure 4** was modified by shortening the height and extending the length. **Figure 5** shows the modified frame for the three core holder apparatus. The new frame is on top of four wheels to allow mobility from one location to another and all four wheels rotate. Both frames can replace medium size core holders with small size.

The two core holder apparatus was designed with 1.5 inch x 1.5 inch square aluminum material and aluminum plating, purchased from Industrial Machines. The two core holder apparatus footprint is 77 inches height x 65 inches length x 32 inches width. The three core holder apparatus was designed with aluminum 1515 from the 80/20 Company. The 80/20 type frame enables use of mechanical connections to the beams and plates, providing a secured and aesthetic design compared to the two core holder apparatus. Dimensions are 71 inches height by 85 inches length by 31 inches width.

The frame includes a swivel system for the large core holder for easier access inserting the core horizontally. The swivel mechanism (**Figure 6)** rotates the core holder 90 degrees from the horizontal to the vertical position and is secured by a pin. All core holders are placed vertically during experiments to minimize gravity effects. The frame minimizes corrosion to injection pumps and electrical components by using a 1/8 inch aluminum plate. Core holders and accumulators mount on to the frame using stainless steel 316 clamps and pins as shown in the final assembly **(Figure 7)**.

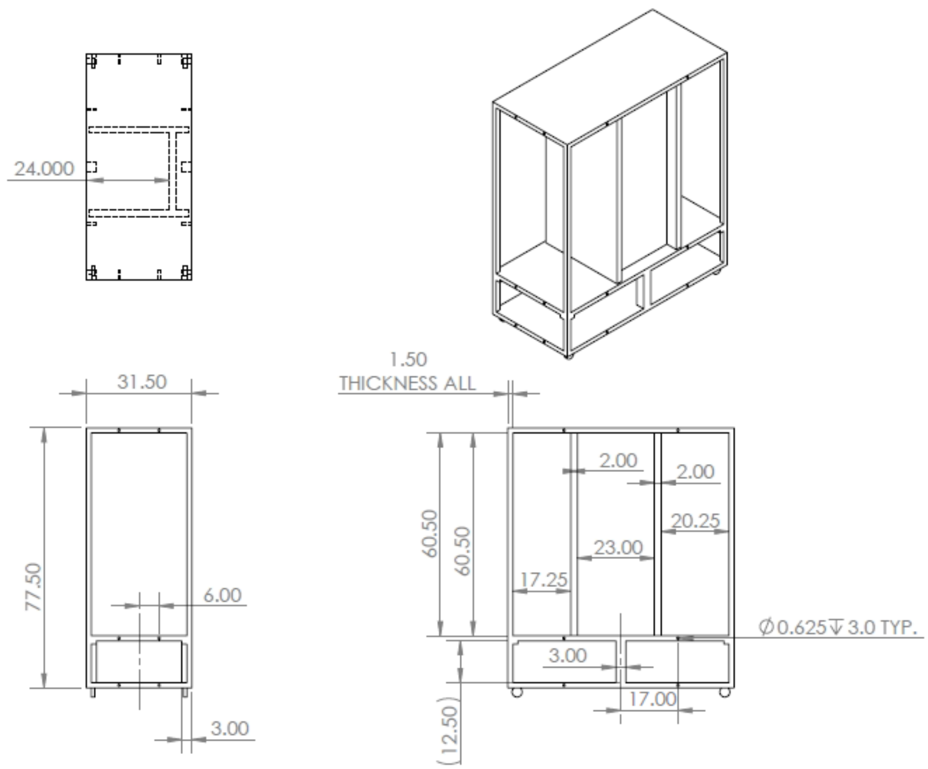


Figure 4: Two Core Holder Apparatus Frame

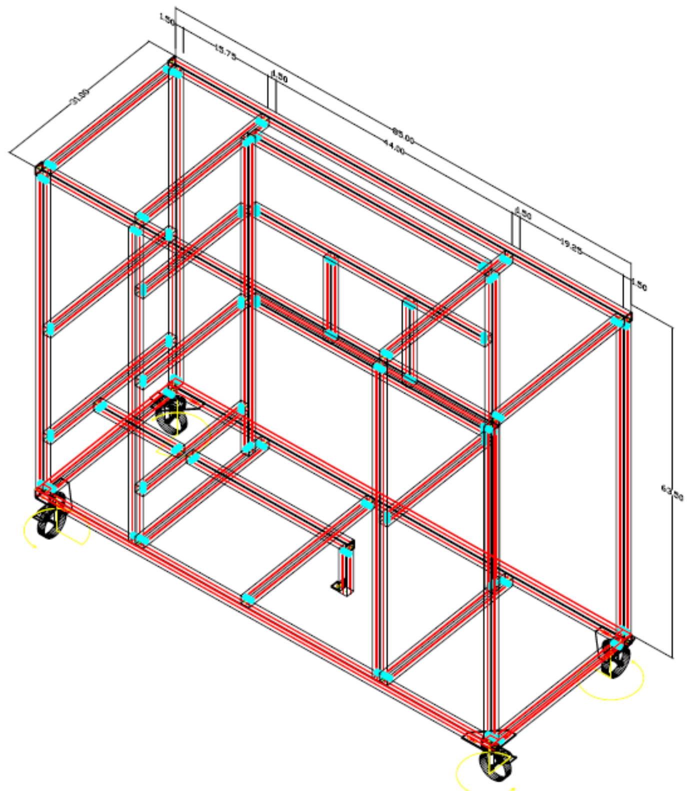


Figure 5: Three Core Holder Apparatus Frame for Texas A&M Qatar

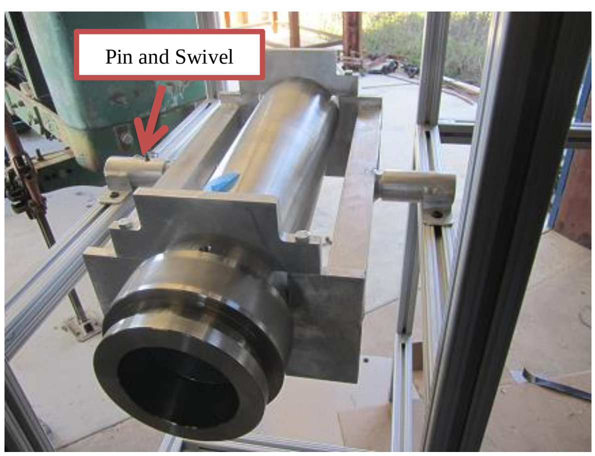


Figure 6: Large Core Holder Swivel Mechanism

Figure 7: Final Assembly of Frame

2.5 Core Holders

Core holders are cells where core samples are confined allowing for liquid flow testing. Core holders are made of Hastelloy C276 for internal components and stainless steel 17-4 for the body material. Phoenix Instruments provided three sizes of core holders: small - 1 inch diameter by 6 inch length, medium - 1.5 inch diameter by 20 inch length, and large - 4 inch diameter by 20 inch length (**Figure 8**). The core holders are rated up to 5,000 psi working pressure and 300 ºF working temperature.

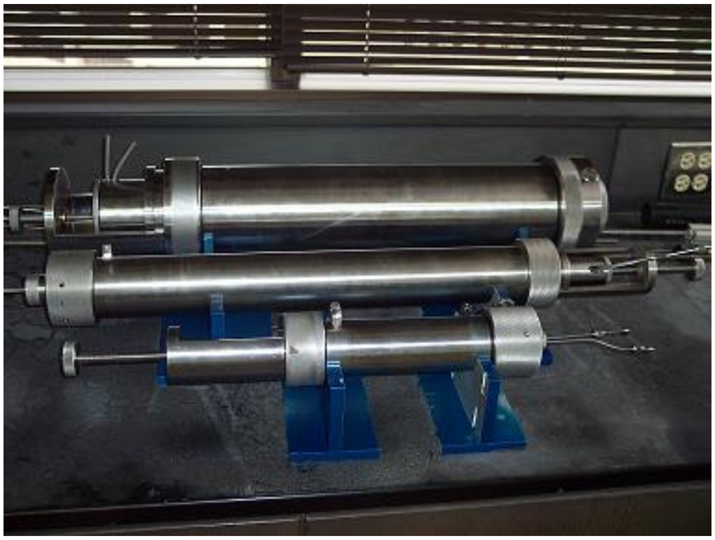


Figure 8: Small, Medium, and Large Core Holders

2.5.1 Core Holder Assembly

The core holder is composed of a bottom plug for inlet, top plug for outlet with spacer, cylinder, top cap, and two valves to control overburden, and clamp for frame connection as shown in **Figure 9**.

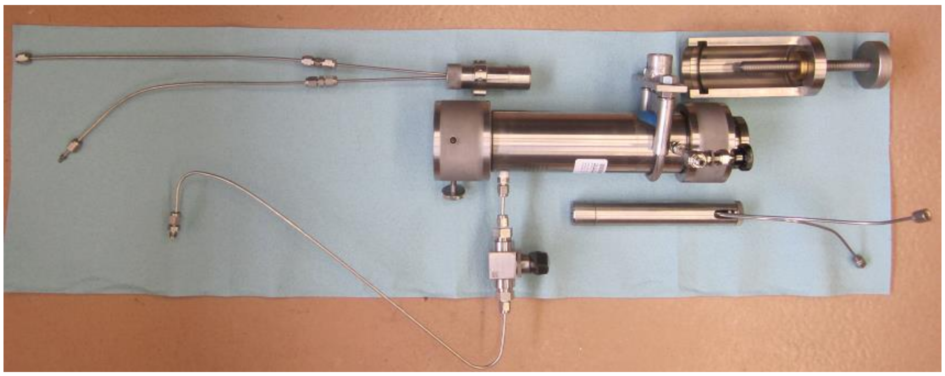


Figure 9: Core Holder Assembly

Based on **Figure 9**, a core is inserted into the bottom of the core cylinder. Then the bottom plug is pushed into place and twisted clockwise. A set screw holds the bottom plug in place. The top plug including spacers is pushed in from the top and the top cap aligns into place and seals the entire core holder. The core holder is mounted onto the frame and six connections are made: two for inlet, two for outlet, and two for overburden inlet and outlet. The core holder assembly is the same for small, medium, and large sizes. Spacers are required based on the length of the core sample. Spacers attach to the top plug by disconnecting tubing connections, attaching spacers, and reconnecting tubing connections. The large core holder is permanently fixed on the frame.

2.5.2 Core Holder Connections

The top and bottom plugs in each core holder have two 1/8 inch tubing ports on the top and two 1/8 inch tubing ports on the bottom. The bottom ports connect to flow inlet and pressure transducer low end. The top ports connect to the back pressure regulator and the high end of a pressure transducer. Each core holder has two 1/8 inch NPT connections on the body of the cylinder to allow hydraulic oil to enter. A Viton sleeve inserts into the core holder to provide confinement around the cylindrical body of the core sample. The sleeve is kept in place with two end caps. Valves are attached to the two ports to control hydraulic oil pressure during experiments.

2.5.3 Viton Sleeve Replacement

A common mistake occurs when the core holders are not closed properly with the top cap and the spacers do not touch the top and bottom of the core sample. Due to this error, when overburden pressure is applied, the Viton sleeves rupture and hydraulic oil flows out of the core holder. In the event of a rupture, a procedure is followed to fix the sleeve:

1. Remove the core holder from the frame or rotate the large core holder to the horizontal position.
2. Release hydraulic oil from the core holder.
3. Remove spacers with top plug from the core holder.
4. Mount core holder onto large vice clamp. Large core holder is left on the frame.
5. Unscrew the top and bottom threaded connections to the cylinder shown in **Figure 10**.

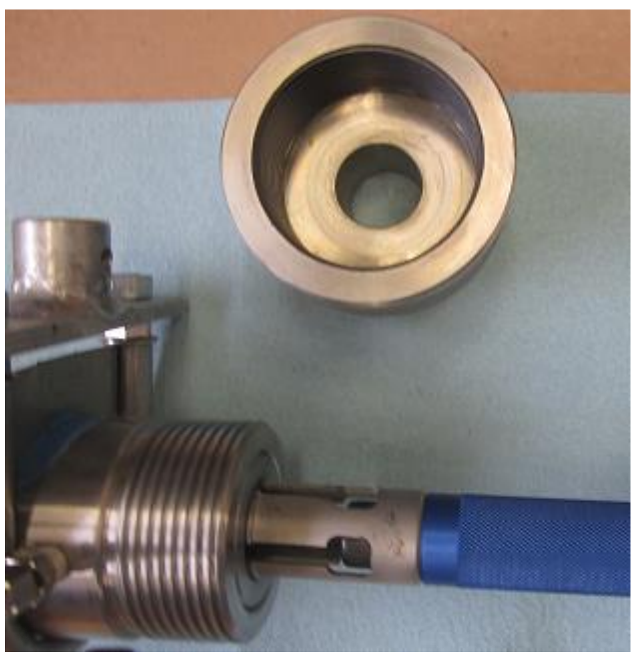


Figure 10: Removal of Viton Sleeve

6. Insert the grip tool shown in **Figure 10** to pull out metal ring seal from the top and bottom of the cylinder.
7. Remove damaged Viton sleeve and insert new Viton sleeve with insertion tool. Note that there are different size tools shown in **Figure 11**.

Figure 11: Viton Sleeve Removal and Insertion Tools

8. Place metal ring seal one on the top and one on the bottom of the cylinder.
9. Screw the top and bottom threaded connections to the cylinder.

2.6 Injection Pump

The injection pump provides controlled flow to the accumulators, hence providing pressure to liquid entering the core holder. A 260D dual syringe pump was purchased from Teledyne ISCO. Using an actuated valve system, the pump refills liquid from the reservoir and injects the liquid at a continuous steady flow rate. Minimum flow rate is 0.001 mL/min and maximum flow rate is 107 mL/min. Flow should not exceed more than 50 mL/min to allow continuous refill. Maximum pressure applied is 7,500 psi. A program allows the pump to turn off at a maximum set pressure to avoid damage to the accumulators and tubing. The pumps may be configured to work with 110 Volts or 220 Volts through a transformer.

Teledyne Isco recommends using hydraulic oil or distilled water for the reservoir. Any liquid higher than a viscosity of 2 cp cannot be placed into the syringe pump due to the tubing and valve inner diameter limitation of 1/8 in. The pump is unable to provide suction to high viscosity liquids. Reservoir liquid exiting the pump into the accumulator should never be placed back into the reservoir. The internal walls of the accumulator contact acid and brine from experiments and residue is left. **Figure 12** shows the injection pump.

Figure 12: Teledyne Dual Syringe Pump

2.6.1 Pump Operation Procedure

To operate refill and injection of the pump:

1. Turn on the pump switch on pump A and B, then switch on the pump controller shown in **Figure 13**.

Figure 13: Pump Control Panel

2. Check reservoir liquid and keep pump suction port in the reservoir. To take out air, confirm pump A and B read 0 mL.
3. Press “REFILL” and then press “A” on controller to flow liquid into the pump.
4. Press “REFILL” and then “B” on controller to flow liquid into the pump.
5. Press “MENU”, “A”, “4”, and then “1” to set liquid at continuous flow.
6. Confirm that bottom options read “NORMAL”, “NORMAL PRESSURE”, and “deliver”.
7. Press “RUN” to inject liquid, type desired flow rate between 0.001 – 107 mL/min, and press “ENTER”. Note that recommended flow should not go over 50 mL/min.

8. Always check reservoir and add liquid when low. Do not let the reservoir empty as air enters the pump.

A common mistake occurs when the pump runs out of liquid due to high refill flow rate. To refill liquid, continuous flow is stopped and each pump is selected by pressing the “B” button first. Note that to calibrate the pump to atmospheric pressure, press “ZERO PRESS”.

2.7 Accumulators and Refill Tank

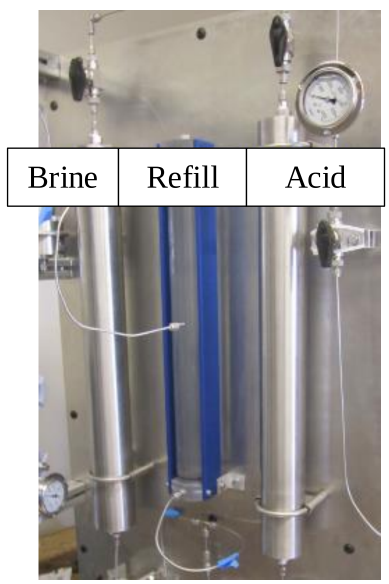


Figure 14: Brine Accumulator, Refill Tank, and Acid Accumulator

Phoenix Instruments manufactured all three components shown in **Figure 14**. Components are clamped onto a 1/8 in aluminum plate. The accumulators store brine and acid for injection into the core holder. Inside each accumulator, a Teflon piston moves up and down based on direction of applied pressure. The accumulators and the refill tank hold 1,000 mL maximum capacity. 1,000 mL capacity is the limitation for injecting brine into the large core. Therefore it is recommended to get a 4000 mL accumulator to avoid pausing experiments to refill brine. The brine accumulator is made of stainless steel 316 material. The acid accumulator is made of Hastelloy C276 material.

Each accumulator consists of one 1/8 inch NPT port on the top and on the bottom. The top section has a stainless steel 316 1/8 inch NPT to 1/8 inch tubing adaptor connecting to a valve configuration. The injection pump and air bleed tubing connect to the injection valve configuration. The bottom section of the acid accumulator uses a Hastelloy C276 1/8 inch NPT to 1/8 inch tubing connection which connects to the injection valve configuration. The bottom section of the brine accumulator uses a stainless steel 316 1/8 inch NPT to 1/8 inch tubing adapter which connects to the injection valve configuration. The accumulator valve configuration allows refill, acid injection, brine injection, and fluid bleed. The refill tank is attached with a 1/8 inch tubing port at the top for air injection. Air pressure provided from the laboratory should not exceed 100 psi to avoid the top bolt to come off. During refill the top bolt is removed and a funnel is placed.

A common error with accumulators occurs when air gets trapped under the piston. Air is compressible and causes fluctuations in pressure readings if trapped inside the accumulators. To make sure that all the air is bled out, the injection pump should fill the accumulators with liquid until pressure builds significantly in the pump controller. The piston inside the accumulator is then confirmed to rest at the bottom. During refill, air is removed out of the flow lines through a bleed port. The user must take caution during refilling each accumulator and make sure acid never gets injected into the brine accumulator. Before and after experiments, make sure that no liquid is left inside the acid accumulator. It is recommended to flush out both accumulators with water before and after experiments to avoid liquid contamination from previous experiments.

2.8 Overburden Pump

The overburden pump provides hydraulic oil between the inner cylinder of the core holder and Viton sleeve. An ENERPAC-293 hand pump was purchased for this application. The pump provides a maximum of 10,000 psi pressure and has a 900 mL capacity. The pump is controlled by hand. To use the pump, first confirm that the pump is set to vent mode and push down on the handle. Air enters the pump once oil is displaced to maintain pressure inside. Pull the handle upward to build pressure to inject hydraulic oil again.

Before running experiments, the reservoir is checked to ensure AW-32 hydraulic oil is available. To refill the reservoir, open the black cap, pour in hydraulic oil, and place the cap back into vent mode. Overburden pressure is constantly controlled throughout the experiment by monitoring overburden pressure gauges. Note with an increase of temperature, overburden pressure increases rapidly and should be bled out of the system.

Too much overburden pressure may crack the core and break the Viton sleeve. Overburden pressure always remains 300-500 psi above the back pressure.

The pump (**Figure 15**) has a 3/8 inch NPT connection which connects to a 3/8 inch NPT to 1/8 inch reducing union for tubing. The tubing then goes through a series of valves to the bottom 1/8 inch NPT connection of the core holder. Hydraulic oil should always be filled from the bottom of the core holder to the top to avoid air being trapped inside the Viton sleeve.

Figure 15: AW-32 Hydraulic Oil and Overburden Pump

2.9 Back Pressure Regulator

Back pressure is required to ensure liquid entering the core holder matches the reservoir conditions. In addition, back pressure keeps carbon dioxide in solution while the core acidizes. If back pressure is too low during acidizing, carbon dioxide is released from solution as gas and differential pressure builds. The back pressure regulator chosen was BPR21 from Circle-Seals. The regulator can handle 25-6,000 psi. The body material is made of stainless steel 316 and the internal seats are made of Hastelloy C276 to be resistant against acid, carbon dioxide, and various fluids. The regulator is rated to 400 ºF.

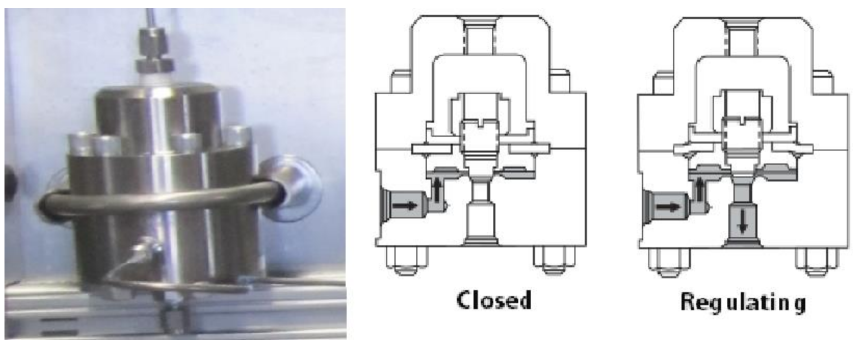


Figure 16: Back Pressure Regulator

The back pressure regulator is shown in the open and closed position (**Figure 16**). Pressurized gas from the nitrogen tank enters the top port of the regulator. Liquid entering the inlet arrow shown in **Figure 16** must exceed the nitrogen tank pressure in order to exit. The three ports on this regulator are 1/4 inch NPT. To connect to tubing, 1/4 inch NPT to 1/8 inch tubing reducers were attached. The regulators were attached to 3 inch round clamps and mounted onto the apparatus. Parallel core floods required two back pressure regulators since liquid is measured separately for each core holder. Flow lines exiting each back pressure regulator connect into two fraction collectors through a series of valves. The nitrogen tank provides 3,000 psi to both regulators equally. During experiments it is important to never lower back pressure as this causes a pressure drop in the flow lines of the apparatus causing liquid to leak out of the core holders.

2.10 Nitrogen Source

One nitrogen tank provides a pressure source for the back pressure regulator. Nitrogen can also be used as a source for gas flow back experiments. The setup includes a nitrogen tank, pressure regulator, and a bleed valve (**Figure 17**). The back pressure regulator connects to 1/8 inch tubing and a Swagelok valve is used to release nitrogen. Nitrogen tanks and pressure regulators vary in laboratory settings but should have a universal NPT port for tubing connection.

Figure 17: Nitrogen Tank, Pressure Regulator, and Bleed Valve

2.10.1 Opening and Closing Nitrogen Tank

Losing or gaining back pressure during experiments is a common mistake. Proper procedure should be followed:

1. Check that bleed valve is closed and regulators are reading 0 psi.
2. Check the valve directly above the nitrogen tank to be in the closed position.
3. Unscrew the regulator handle shown by hand in **Figure 17**.
4. Fully open the valve above the nitrogen tank. Notice that pressure on the right gauge reads the maximum pressure left inside the nitrogen tank such as 3,000 psi.
5. Start closing the regulator handle until pressure on the left gauge reaches the desired back pressure required for experiments.
6. Do not close the valve on top of the nitrogen tank during experiments as that causes back pressure to drop. Do not change the position on the regulator handle.
7. After the end of experiment, close the valve above nitrogen tank.
8. Slowly control bleed valve to release all nitrogen trapped in the flow line.

To prevent leaks in the nitrogen flow lines, use soap water on a piece of paper and place near each connection to see if bubbles form. Those connections are then changed or tightened.

2.11 Fraction Collector

The fraction collector gathers spent liquid from the exit port of the back pressure regulator. The purpose is to measure flow rate collected over a period of time. This is useful to determine acid diversion for parallel core floods. Two FC 203B fraction collectors were purchased from Gilson Inc. In addition, 80 x 10 mL test tubes and 108 x 3 mL test tubes were purchased. The max flow rate is limited to 20 mL/min. A back pressure bypass valve configuration was created to collect liquid into a large flask before and after the use of fraction collectors.

Figure 18: Fraction Collector

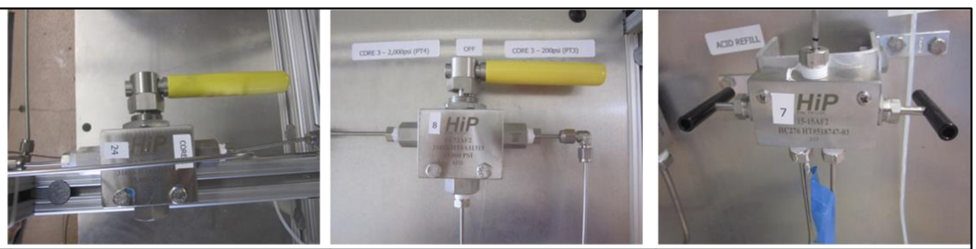
Fraction collector operates from the control panel (**Figure 18**). The timer is set in decimals for the desired minutes. The fraction collector moves from each test tube in an S shape pattern starting from the top left and moving down. A three-way valve controls the inlet of the flow to test tubes and to a bleed outlet. Data acquisition is available but was not required for the apparatus. The issue with using fraction collectors is corrosion. Acid corrodes the flow lines entering the three-way valve. Therefore, brine is flushed through tubing to remove acid after the experiment. The electrical outlet connection is set for 110 Volts or 220 Volts by changing the back fuses.

Follow instructions for proper use:

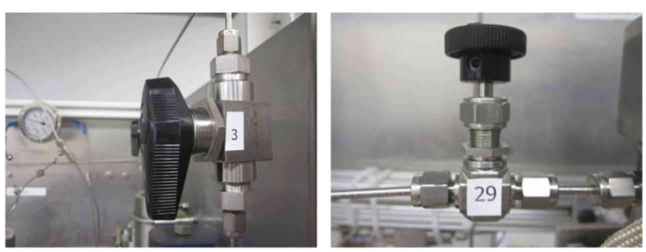
1. Place clean 80 x 10 mL test tubes on tray.
2. Check to see if program is on “RACK 1” on main screen.
3. Program time units ranging from 0.01 to 99.99 min per tube based on injection flow rate.
4. Attach fraction collector bleed bypass tubing to a beaker or flask.
5. Observe liquid going to the bleed bypass tubing.
6. Press “START” to start the fraction collector.
7. After samples are collected, Press “END”. Liquid goes out to the bleed bypass.
8. Flow water or brine and make sure no acid is left.
9. Observe when liquid stops flowing into the fraction collector.
10. Remove test tubes and measure with graduated cylinder.
11. Clean test tubes and wipe fraction collector.

2.12 Valve System

The purpose of all 36 valves in this apparatus is to control fluid flow and regulate pressure. Three core holders and parallel core flooding capabilities had increased the complexity of valve configurations. These configurations were broken down into sets: accumulator (**Figure 21**), pressure transducer (**Figure 22**), core holders (**Figure 23**), overburden (**Figure 24**), fraction collectors (**Figure 25**), and nitrogen. Understanding each set is critical for conducting experiments. Valve type, connection, material, pressure rating, temperature rating, and vendor information are represented in **Table 2**.



Vendor	HIP	HIP	HIP
Type	2 Way Open Close	3 Way Open Close	3 Way Needle



Vendor	Swagelok	Swagelok
Type	2 Way Open Close	2 Way Needle

Figure 19: Valve Type and Pictures

Different types of valves are available as shown in **Figure 19**: a two-way open close valve, a three-way open close valve, a two-way needle valve, and a three-way needle valve. The first type is a simple open and close function with handles. To open, shift the handle towards the flow in a 90 degree rotation. The second type is a needle valve, rotated open after many turns. The third type is a three-way open and close where flow can be diverted from one path to two other paths individually based on rotating the handle 0, 90, and 180 degrees. The fourth type is a three-way needle valve which flows from one direction into two directions. Flow is controlled based on having two stems controlling the valve individually. With a completed valve design (**Figure 20)**, the user does not have to create any new connections with tubing and fittings.

Note that it is recommended to never use a three-way Hastelloy ball valve with a PEEK seat. Acid corrodes the PEEK seat inside the ball valve during experiments. Three-way needle valves from HIP were a superior replacement to the older three-way ball valves.

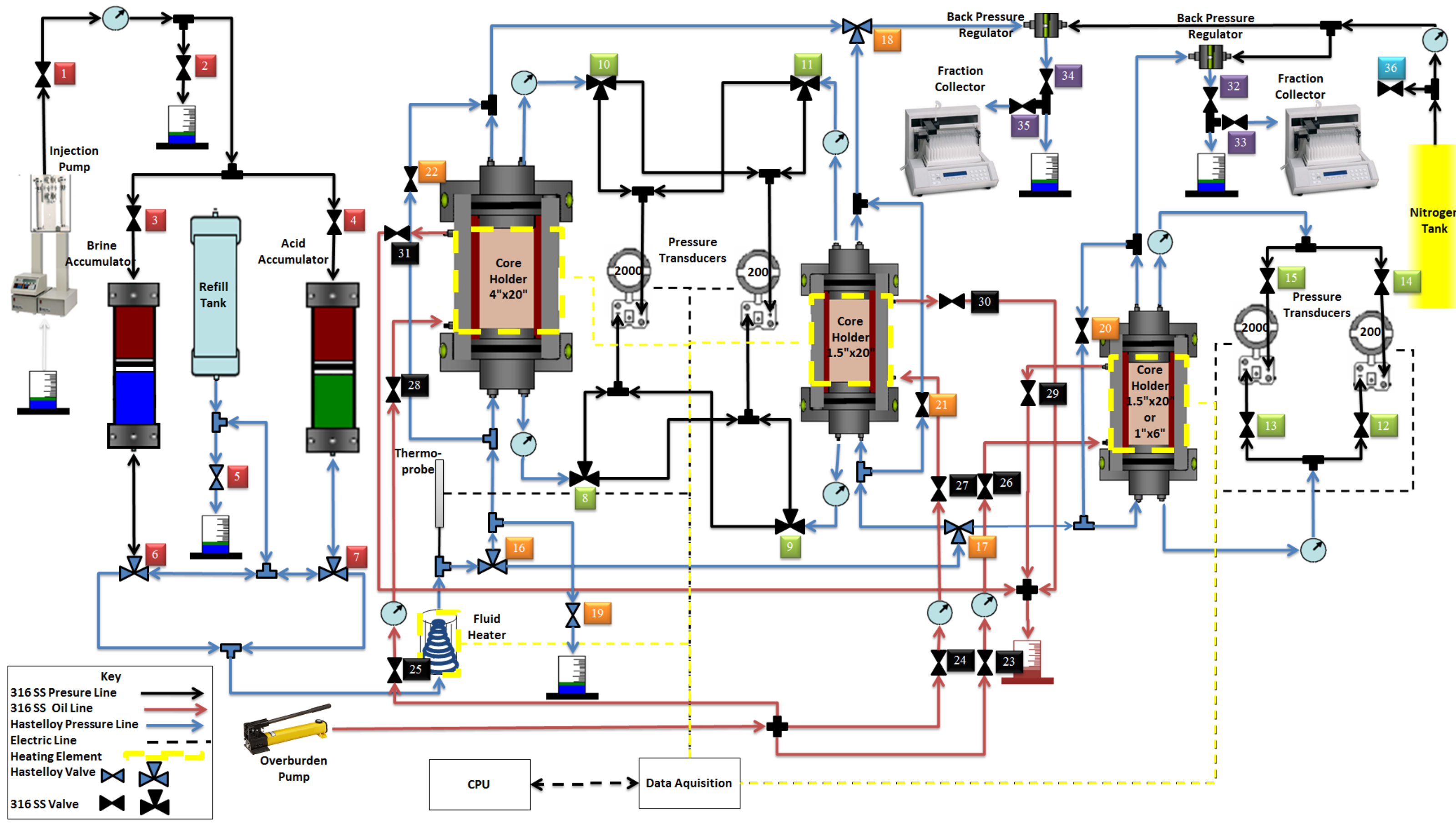


Figure 20: Valve Configuration for Parallel Core Flood Apparatus

Table 2: Valve List

#	Valve	Type	Connection	Material	Pressure psi	Temp F	Part no	Vendor
Accumulator Set								
1	Injection Pump	2	1/8" tube	SS 316	6000	300	SS-83PS2	Swagelok
2	Air and Liquid Bleed	2	1/8" tube	SS 316	6000	300	SS-83PS2	Swagelok
3	Injection Pump to Acid	2	1/8" tube	SS 316	6000	300	SS-83PS2	Swagelok
4	Injection Pump to Brine	2	1/8" tube	SS 316	6000	300	SS-83PS2	Swagelok
5	Acid / Brine / Air Bleed	2 needle	1/4" AF2	HC276	10000	650	10-11AF4	HIP
6	Refill Acid / Acid Injection	3 needle	1/8" AF2	HC276	10000	650	15-15AF2-HC276	HIP
7	Refill Brine / Brine Injection	3 needle	1/8" AF2	HC276	10000	650	15-15AF2-HC276	HIP
Pressure Transducer Set								
8	Core 3 bottom to PT 2 200/2000	3	1/8" AF2	SS 316	15000	650	15-71AF2	HIP
9	Core 2 bottom to PT 2 200/2000	3	1/8" AF2	SS 316	15000	650	15-71AF2	HIP
10	Core 3 top to PT 2 200/2000	3	1/8" AF2	SS 316	15000	650	15-71AF2	HIP
11	Core 2 top to PT 2 200/2000	3	1/8" AF2	SS 316	15000	650	15-71AF2	HIP
12	Core 1 bottom PT 1 200	2	1/8" tube	SS 316	6000	300	SS-83PS2	Swagelok
13	Core 1 bottom PT 1 2000	2	1/8" tube	SS 316	6000	300	SS-83PS2	Swagelok
14	Core 1 top PT 1 200	2	1/8" tube	SS 316	6000	300	SS-83PS2	Swagelok
15	Core 1 top PT 1 2000	2	1/8" tube	SS 316	6000	300	SS-83PS2	Swagelok
Core Holder Set								
16	Core 1+2 and Core 3	3 needle	1/8" AF2	HC276	10000	650	15-15AF2-HC276	HIP
17	Core 1 and Core 2	3 needle	1/8" AF2	HC276	10000	650	15-15AF2-HC276	HIP
18	Core 2 and 3 BPR	3 needle	1/8" AF2	HC276	10000	650	15-15AF2-HC276	HIP
19	Core Bleed Valve	2 needle	1/4" AF2	HC276	10000	650	10-11AF4	HIP
20	Core 1 Bypass	2	1/8" tube	SS 316	3000	250	SS-2P4T	Swagelok
21	Core 2 Bypass	2	1/8" tube	SS 316	3000	250	SS-2P4T	Swagelok
22	Core 3 Bypass	2	1/8" tube	SS 316	3000	250	SS-2P4T	Swagelok
Overburden Set								
23	Core 1 Flow Inlet	2	1/8" AF2	SS 316	15000	650	15-71AF2	HIP
24	Core 2 Flow Inlet	2	1/8" AF2	SS 316	15000	650	15-71AF2	HIP
25	Core 3 Flow Inlet	2	1/8" AF2	SS 316	15000	650	15-71AF2	HIP
26	Core 1 Inlet	2	1/8" tube	SS 316	6000	300	SS-83PS2	Swagelok
27	Core 2 Inlet	2	1/8" tube	SS 316	6000	300	SS-83PS2	Swagelok
28	Core 3 Inlet	2	1/8" tube	SS 316	6000	300	SS-83PS2	Swagelok
29	Core 1 Exit	needle	1/8" tube	SS 316	5000	450	SS-ORS2	Swagelok
30	Core 2 Exit	needle	1/8" tube	SS 316	5000	450	SS-ORS2	Swagelok
31	Core 3 Exit	needle	1/8" tube	SS 316	5000	450	SS-ORS2	Swagelok
Fraction Collector Set								
32	Core 1 Exit	needle	1/8" tube	SS 316	5000	450	SS-ORS2	Swagelok
33	Core 1 Fraction Divert	needle	1/8" tube	SS 316	5000	450	SS-ORS2	Swagelok
34	Core 2/3 Exit	needle	1/8" tube	SS 316	5000	450	SS-ORS2	Swagelok
35	Core 2/3 Fraction Divert	needle	1/8" tube	SS 316	5000	450	SS-ORS2	Swagelok
Nitrogen								
36	Nitrogen Bleed	needle	1/8" tube	SS 316	6000	450	SS-ORS2	Swagelok

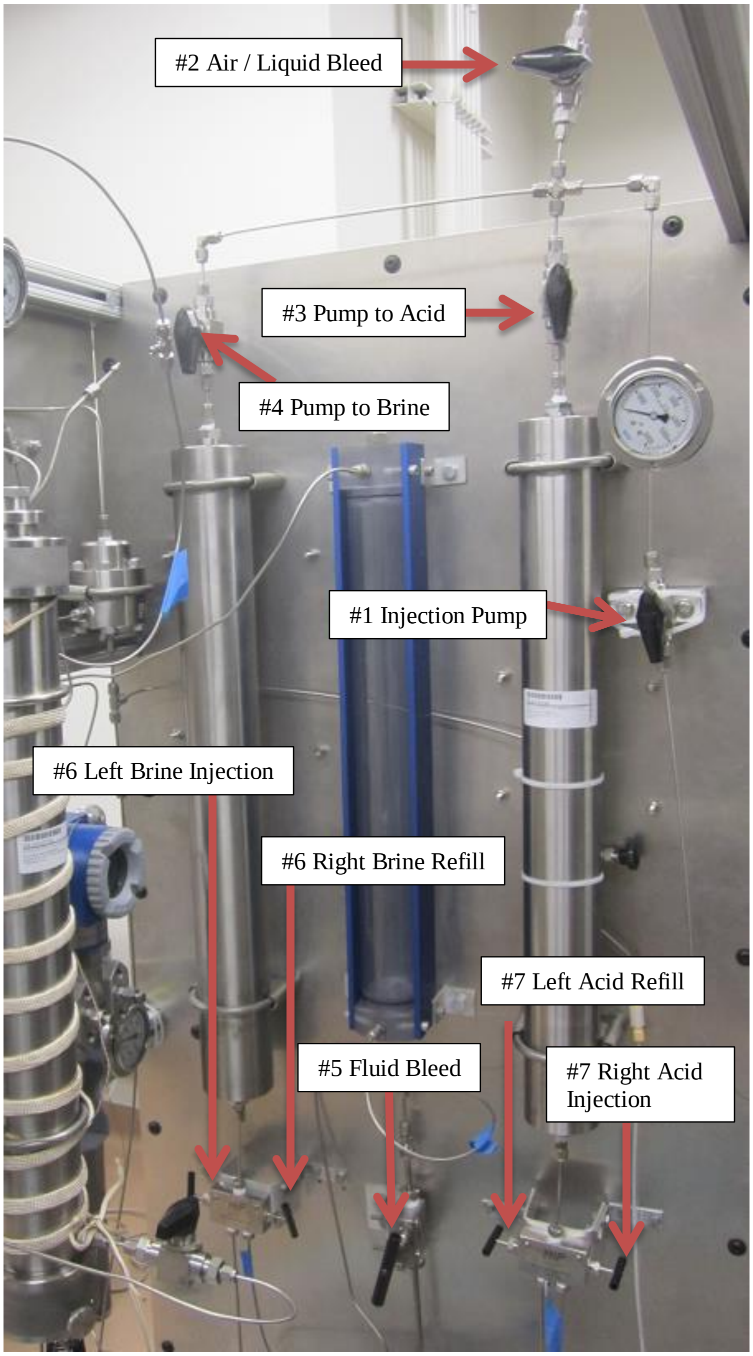


Figure 21: Accumulator Set

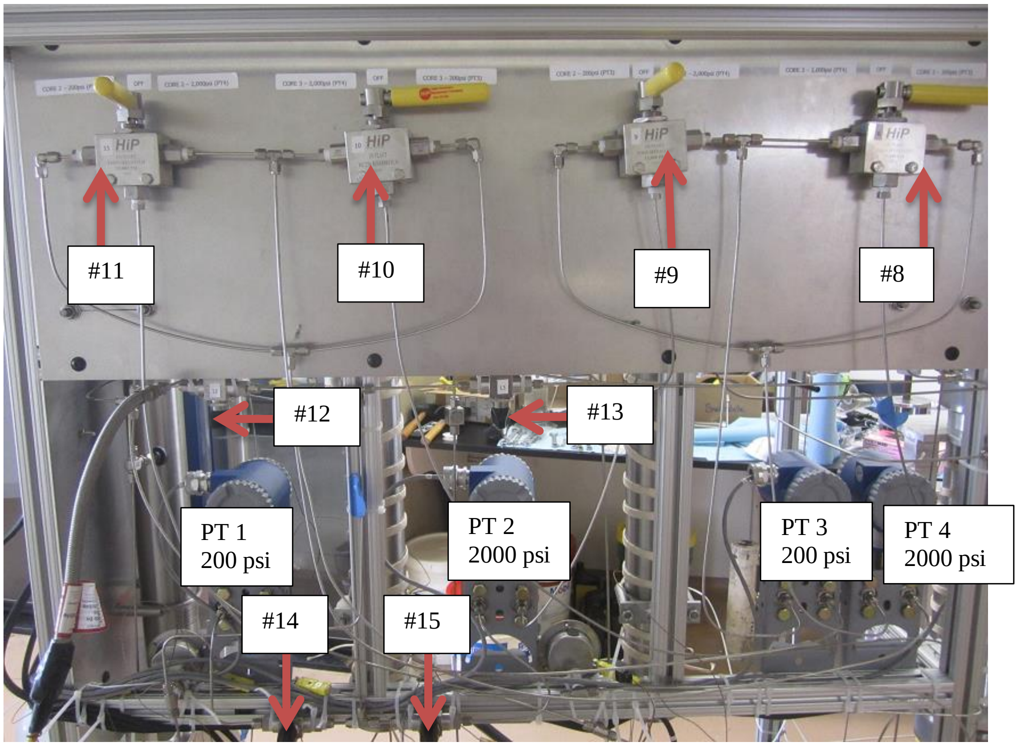


Figure 22: Pressure Transducer Set

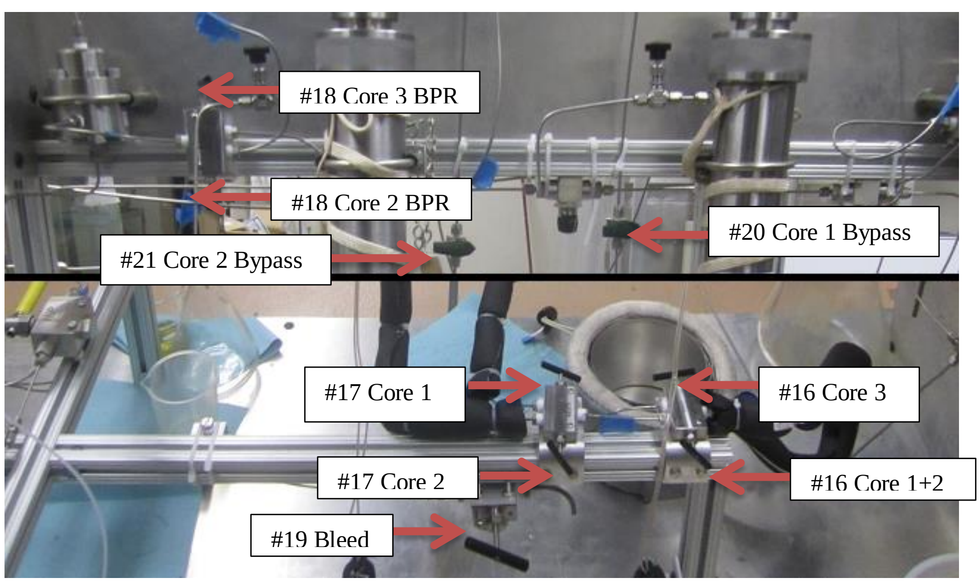


Figure 23: Core Holder Set

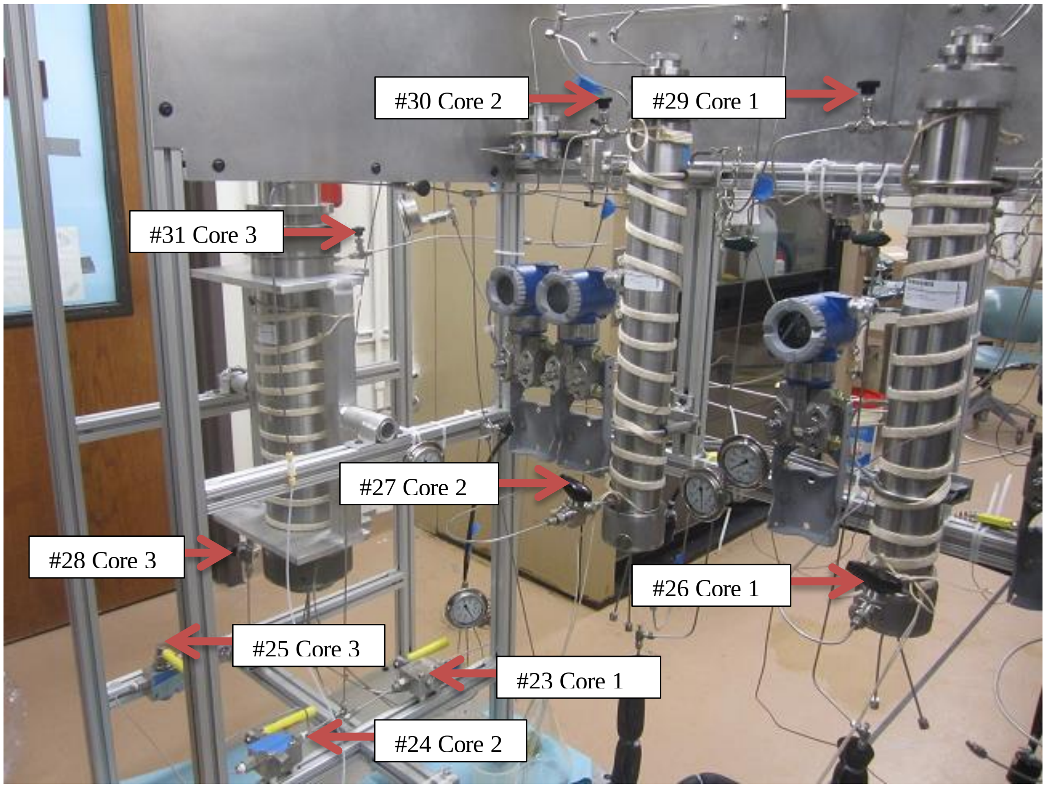


Figure 24: Overburden Set

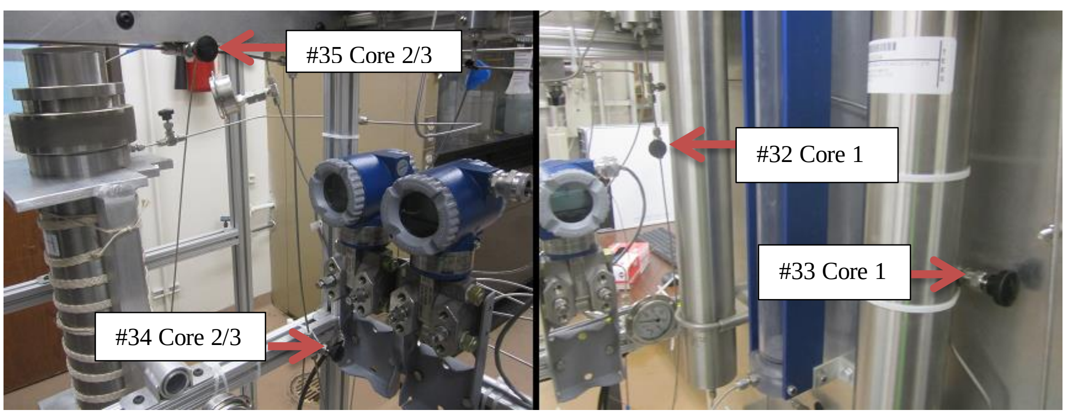


Figure 25: Fraction Collector Set (Flow Line Change)

2.13 Fittings

Fittings are used to connect valves, tubing, unions, and are required throughout the flow lines. Typical fittings consist of three parts: seat, ferrule, and nut. Two materials for fittings were used: Hastelloy C276 from AWC and stainless steel 316 from Swagelok. Four types of fittings exist: Swagelok, Gyrolok, NPT, and A-lock HIP. A-lock and NPT connections require Teflon tape. NPT type is common for accumulators, core holders, and back pressure regulators. Note that Gyrolok nuts attach into Swagelok unions but Swagelok nuts cannot attach to Gyrolok unions.

Fittings connections are sensitive to torque and should not be over tightened. It is highly recommended to use a torque wrench when connecting fittings, especially for A-lock HIP valve connections. **Figure 26** shows types of fittings and tubing connections. To create a fitting, the following steps were used:

1. Place a union vertically positioned on a clamp and tighten clamp.
2. Place the nut with a seat in first and then a ferrule.
3. Drop the tubing from the top into the union.
4. Tighten the nut fully onto the union clockwise by hand.
5. Use a wrench to tighten the nut onto the union. Note that only 3 9- degree turns are required with 1/8 inch fittings and 5 90 degree turns are required for 1/4 inch fittings. Do not tighten any further or the tubing shears.
6. Unscrew the nut and inspect to confirm the new connection is secured.

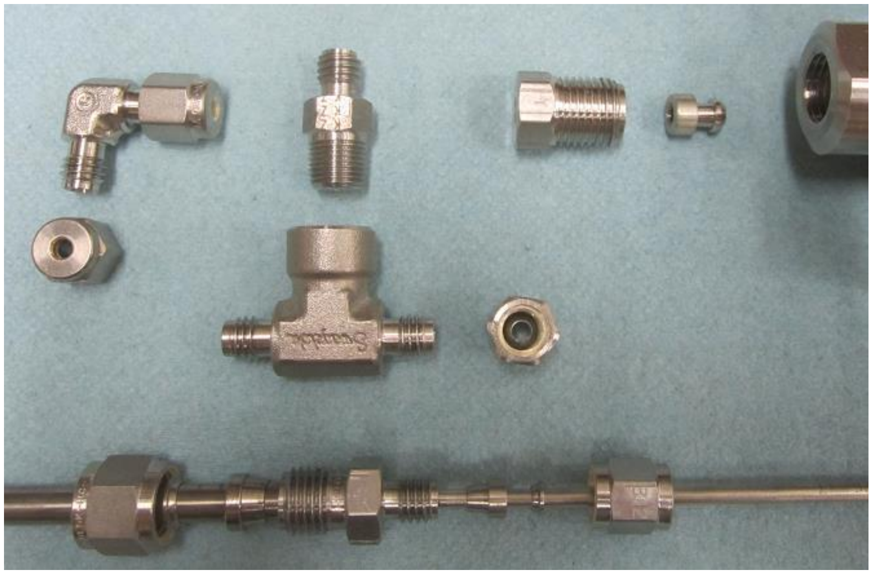


Figure 26: Fittings and Assembly

2.14 Tubing

Tubing connects all flow lines with valves and the major components. Two materials for tubing were used: Hastelloy C276 from Vindum Engineering and stainless steel 316 from Swagelok. Hastelloy tubing is rated at 9,175 psi and 300 ºF for 1/8 inch tubing with 0.02 inch thickness. For 1/4 inch Hastelloy C276 tubing is rated at 7,882 psi and 300 ºF. Hastelloy C276 is acid corrosion resistant but not cost effective. Stainless steel 316 tubing was used in other sections of the apparatus as it was approximately ten times more cost effective compared to Hastelloy C276. Hastelloy C276 was used in sections of the apparatus where acid travels and may remain stagnant. The schematic in **Figure 3** shows flow lines colored in blue for acid. Tubing length and the inner diameter of tubing are two important variables in calculating the time it takes acid to reach the core holders.

Stainless steel 316 tubing was purchased from Swagelok for 1/8 inch tubing (SS-T2-S-028-20) and 1/4 inch tubing (SS-T4-S-049-20D). 1/8 inch tubing has a wall thickness of 0.028 inch with rating of 8,500 psi and 300 ºF. 1/4 inch tubing has a wall thickness of 0.049 inch with rating of 7,500 psi and 300 ºF. Overall 1/8 inch tubing is suitable for the apparatus due to a higher bend radius. 1/4 inch tubing was used on the large core holder.

2.15 Pressure Gauge

Pressure gauges are setup throughout the flow lines for the purpose of monitoring core holder inlet pressure, core holder outlet pressure, back pressure, overburden pressure, and syringe pump injection pressure. Pressure gauges are useful to locate fluid leaks. WIKA Model 233.55 from AWC, Inc. was purchased as shown in **Figure 27**. Up to 6,000 psi is measured by the gauges. The gauges have a 1/4 inch female NPT end connection which attaches to a 1/4 inch NPT union. The union is then attached to 1/4 inch NPT with two 1/8 inch T-union which connects to the tubing.

Figure 27: Pressure Gauge

3. DATA ACQUISITION AND HEATING EQUIPMENT

3.1 Equipment Introduction

The experiments record differential pressure between the inlet of the core holder and the outlet. To achieve reservoir conditions, heating elements were added and controlled through the data acquisition software. Heaters are composed of fluid heater and heating tape, which go through solid state relays to the data acquisition board. A signal from LabVIEW generates the on and off mechanism on the solid state relays based on set temperature and is monitored by thermocouples. Pressure transducers send out current signals to the data acquisition board. **Figure 28** shows a layout of the data acquisition board and the electrical lines are shown in **Figure 3**. The parallel core flood apparatus was designed to function in both 110 and 220 Volt configurations using a transformer.

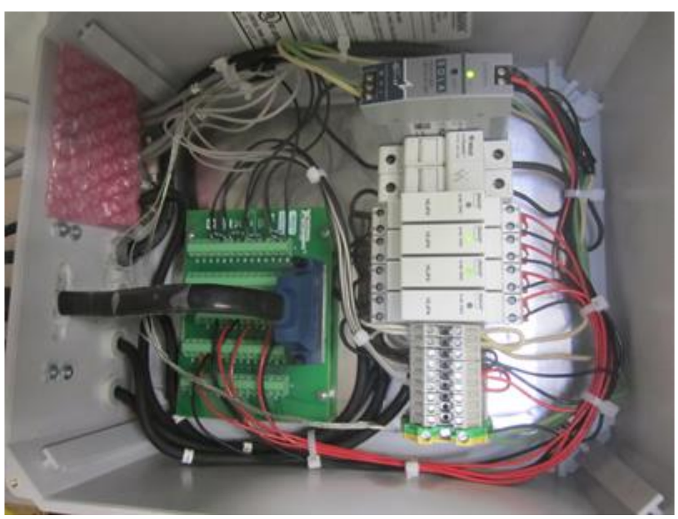


Figure 28: Data Acquisition

3.2 Electrical Enclosures

Two electrical enclosures were purchased. The small box houses a National Instruments USB 9162 reader for four temperature sensors. The larger electrical box contains a 24 Volt power supply, solid state relays, National Instruments CB 68-LP board, and two fuses as shown in **Figure 28**. Two enclosures were purchased from Britt Rice. The Wiegmann designed electrical enclosures are made of fiberglass and provide high resistance against acid corrosion. The enclosures were bolted to the frame shown in **Figure 29**. Placing the electrical components behind the 80/20 aluminum plate of the frame provides additional resistance from acid. Enclosures increase the life of electrical components significantly since the wires, board, and ports are sensitive to corrosion.

Figure 29: Electrical Enclosures

3.3 Fluid Heater

The fluid heater (**Figure 30**) raises the temperature of liquid passing through a series of coiled tubing. The heater was designed in a cylindrical form allowing Hastelloy tubing to coil around the center. Heat is transferred from the fluid heater to the liquid, and a calculation estimates the length of tubing required. The assumptions were using 1/8 inch tubing to achieve a max operating temperature of 300 ºF. Liquid would need to travel a minimum of 217 inches based on a 288 Watt heater at a flow rate of 10 mL/min. Hence 20 feet of 1/8 inch tubing was coiled around the fluid heater. The fluid heater was purchased from Glas-Col. Heater specifications were 7 inch inner diameter, 5 inch height, 350 Watt power, and 115 Volts. It takes one hour to raise the temperature of pressurized liquid to 300 ºF flowing at 10 mL/minute. Fiberglass fabric with silicone coated fabric covers the heater. Velcro allows a secure connection for the heater around the tubing.

Figure 30: Fluid Heater with Hastelloy Tubing

3.4 Heating Tape

Heating tape raises the temperature of the core holders and the core. The tape is wrapped around the core holder body and tied on both ends. Three different lengths of tapes were purchased for each core holder from HTS/Amptek Company. The large core holder uses 16 feet length with 312 Watts AWH-051-160 DL tape. Both medium core holders use 10 feet length with 195 Watts AWH-051-100 DL tape. The small core holder uses a 6 feet length with 117 Watts AWH-051-060 DL tape. Maximum temperature is rated at 1,400 ºF. Once the core holder is mounted onto the frame, the heating tape is wrapped around and tightened at both ends as shown in **Figure 31**.

Figure 31: Heating Tape on Core Holder

The heating tape wrapped around the core holder should not touch as this may cause a short in the circuit. To minimize heat loss, it is recommended to insulate the cylinder with fiber glass after the heating tape is secured in place. Fiberglass insulation and pipe insulation tape was purchased from Grainger. The heating tapes connect to solid state relays. Thermocouples are attached to the center of the cylinder and temperature is controlled with LabVIEW software.

3.5 Temperature Sensors

Three thermocouples and a thermo-probe measure the temperature of core holders and liquid respectively. The data is then transferred to a USB 9162 board which is read into LabVIEW. Type J thermocouples were purchased from Omega. They have a range of -40 ºF to 1,382 ºF. Since the thermocouple cannot reach the core sample directly, it is mounted onto the surface of the core holder (**Figure 32**). The thermo-probe directly contacts liquid coming out of the fluid heater.

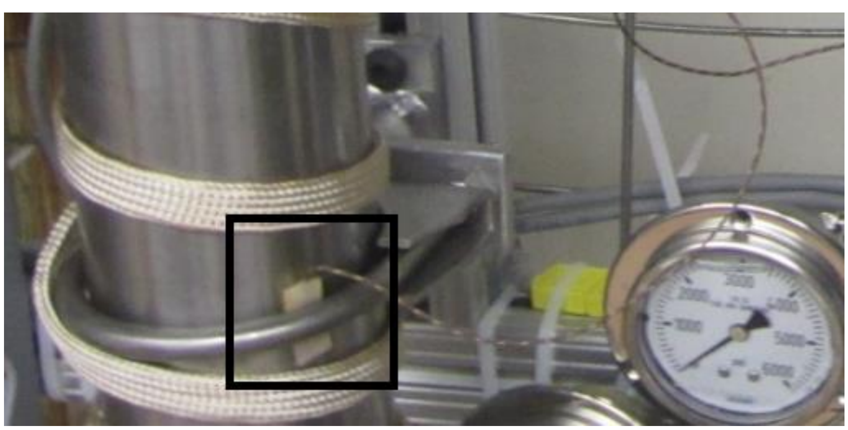


Figure 32: Thermocouple Placement

3.6 Pressure Transducers

Pressure transducers provide differential pressure data into LabVIEW. The locations where liquid is injected into the core holder and liquid exits the core holder are recorded. Four pressure transducers were used for this apparatus: two Foxboro model IDP10-A26EF-M1 of 0 – 2,000 psi and two Foxboro model IDP10-A26D11F-M1 of 0 – 200 psi. The 200 psi pressure transducer is used for high permeability cores. Pressure transducers are connected with 1/4 inch NPT connections which are attached to 1/4 inch to 1/8 inch tubing reducers. Note that it is important to fill the tubing flow lines with water to remove any trapped air. To remove air, open the port opposite of the tubing connection while having a constant injection of water. Water remains in the tubing and prevents acid from entering the flow lines during experiments. Electrical wires are attached to the pressure transducers connecting to the data acquisition board and power supply. The first wire is a ground and the other two wires are positive and negative lines. These lines are read through the data acquisition board into LabVIEW.

3.6.1 Scale Setup

The pressure transducer is scaled based on the voltage readings from the data acquisition board. The scale is based on 249 ohm resistors and a current of 4 - 20 amps. Once the pressure transducers are scaled, this procedure does not require further change unless the electrical components are altered. To perform the scale setup:

1. Open the measurements and automation software on the computer and go to scales option as shown in **Figure 33** and **Figure 34**.

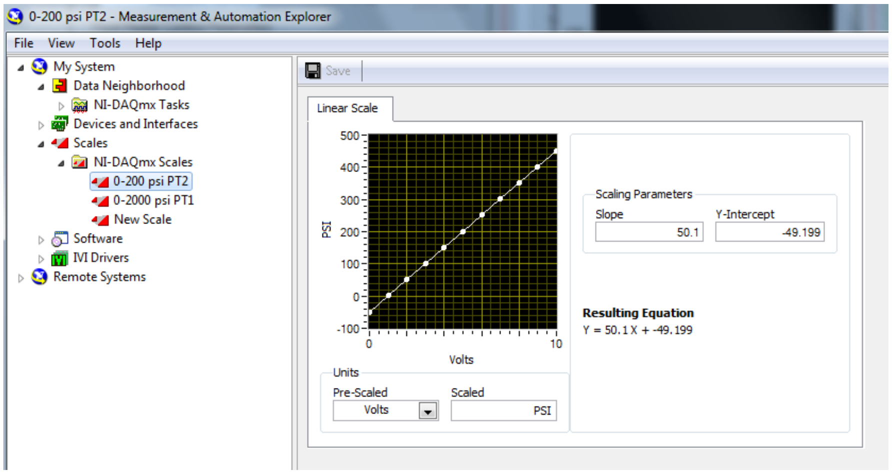


Figure 33: 0-200 psi Scale

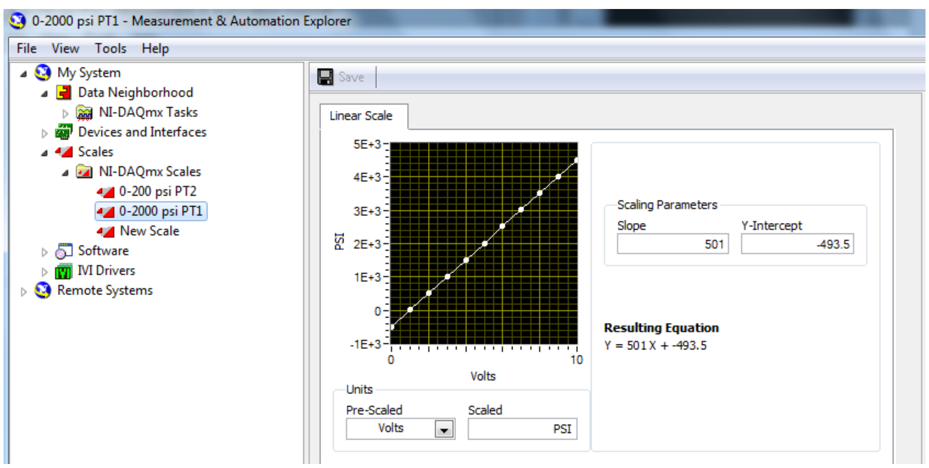


Figure 34: 0-2,000 psi Scale

2. Go to Devices and Interfaces > NI PCI-6221 > Click on Test Panel.
3. Select the pressure transducer from 0 to 3 under Channel Name (**Figure 35**). Hit "Start" and a voltage should appear. Observe that the pressure transducer should read 0 psi. Record the value for the voltage.

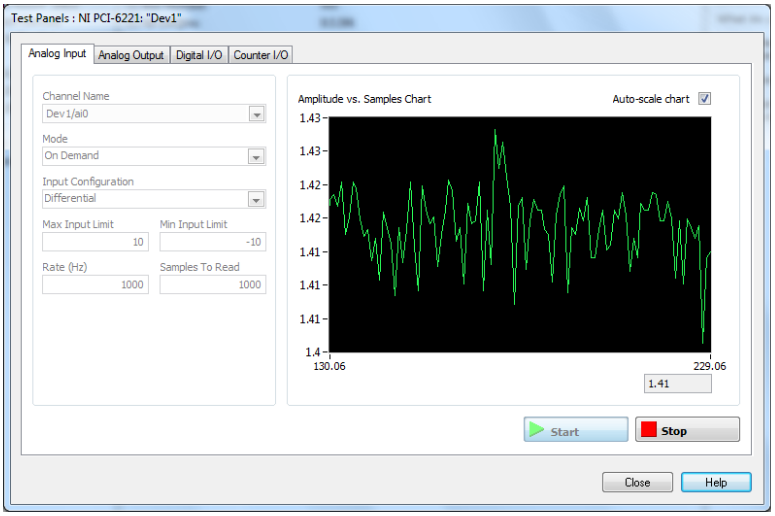


Figure 35: Transducer Test Panel

4. Attach a pressure source such as injection pump or nitrogen tank to the positive port on the pressure transducer and inject. Bleed out all air if using injection pump as a source.
5. Build pressure to 200 or 2,000 psi based on respective pressure transducers.
6. Run the test panel calibration once more and record the voltage. Observe this new value to be higher.
7. Take the difference between the two changes of voltage and create a linear correlation between each scale. **Figure 36** shows the approximate scale for 200 psi pressure transducers. **Figure 37** shows the approximate scale for 2,000 psi pressure transducers.

8. Insert the slope and Y-intercept into the scale shown in **Figure 33** for 200 psi and **Figure 34** for 2,000 psi and click “SAVE”.

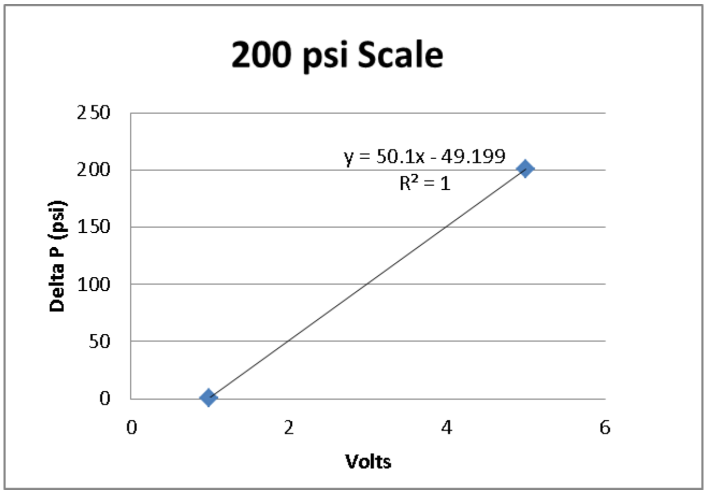


Figure 36: 200 psi Scale

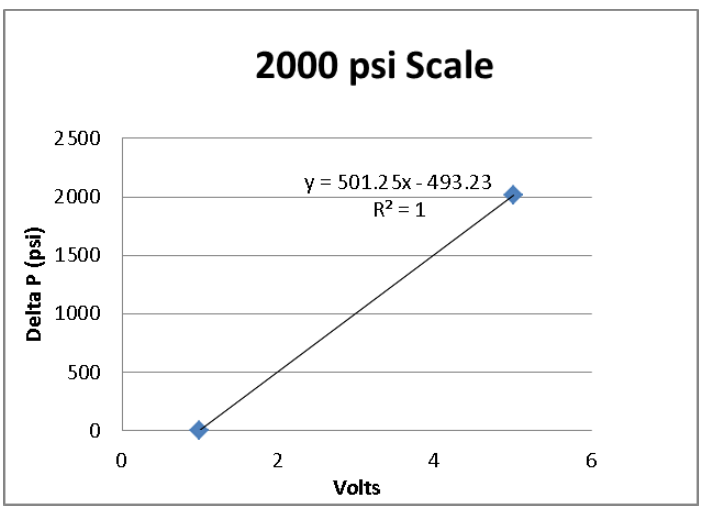


Figure 37: 2,000 psi Scale

3.6.2 Experimental Calibration

Pressure transducers are calibrated during experiments to accurately read 0 psi once a wormhole is created. Wormholes represent infinite permeability, hence there should not be any difference between the inlet and outlet pressure on the core holder. The transducer is reset once back pressure and overburden pressure are applied and a steady flow is achieved. This procedure occurs before the bypass valve is closed. The following steps are used to calibrate the pressure transducer:

Figure 38: Pressure Transducer Panel

1. Confirm overburden, back pressure, and injection pressures are steady. Bypass valve should be in the open position.
2. Open the front cover of the pressure transducer by hand (**Figure 38**).
3. Press “NEXT” and then press “ENTER”. The setting reads “CALIB”.
4. Press “ENTER” and then press “NEXT”. The setting reads “ATM0DONE”.

5. Keep pressing “NEXT” until the “SAVE” option appears and press “ENTER”.
6. Pressure is now set to 0 psi. Close front cover of pressure transducer by hand.
7. Observe when bypass valve is closed, pressure reading increases and reach a steady state. This maximum pressure is used to calculate permeability of the core sample.

3.7 National Instruments USB 9162

The purpose of the NI USB 9162 is to convert signals from the thermocouples and the thermo-probe into temperature data for LabVIEW. The device was purchased from National Instruments and comes with a 12 foot USB adapter. Each thermocouple has a positive and negative wire which insert into ports 0 to 7. Software was installed with an updated driver to use this device. The device is read using measurements and automation. With one self-test, the device is ready to function for LabVIEW and requires no calibration. **Figure 39** shows the setup for thermocouples attached to the ports.

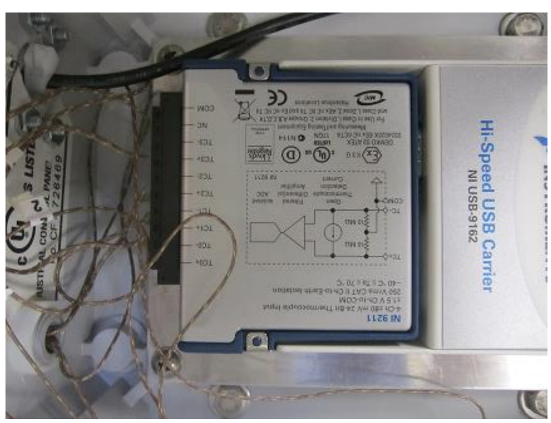


Figure 39: NI USB-9162

3.8 National Instruments CB-68LP and PCI-6221

The CB-68LP data acquisition board takes signals from pressure transducers and gives out signals for solid state relays to turn on and off. There are 68 ports consisting of input / output channels (**Figure 40**). The CB-68LP board connects to a PCI-6221 port built into the computer. A 4 foot SCSI 68 pin cable connects the board to computer port.

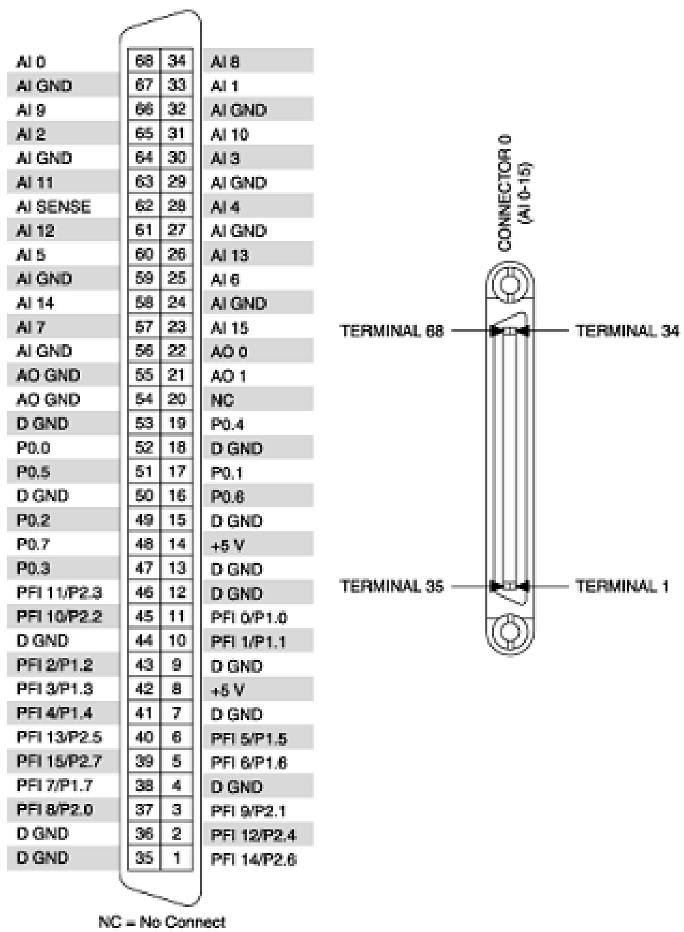


Figure 40: CB-68LP Ports

For wire connections, it is important to ground the data acquisition wires appropriately or noise is generated. 249 ohm resistors were used to reduce noise in voltage and provide a signal of 0 Volt with 4 amps coming from the pressure transducers. The signal would read 5 Volts with 2 amps when 200 or 2,000 psi was read on the pressure transducers respectively. To ground the system, a wire was connected to the negative wire of the power source with the analog ground. Wiring configuration is shown in **Figure 41** and port configuration is shown in **Table 3**.

Figure 41: Wiring Configuration

Table 3: Port Configuration

Port Number	Connection Type
68	200 psi PT 1 +
34	200 psi PT 1 -
33	2,000 psi PT 2 +
66	2,000 psi PT 2 -
65	200 psi PT 3 +
31	200 psi PT 3 -
30	2,000 psi PT 4 +
63	2,000 psi PT 4 -
17	Fluid Heater +
18	Fluid Heater GND
52	Rope Heater 1 +
53	Rope Heater 1 GND
49	Rope Heater 2 +
15	Rope Heater 2 GND
47	Rope Heater 3 +
13	Rope Heater 3 GND

3.9 Solid State Relays

Solid State Relays (**Figure 42**) are used by LabVIEW as an on and off mechanism for the fluid heater and the heating tapes. The input comes from the CB-68 LP board. The first wire is the positive signal and the second wire goes to a ground. The relays were compatible with the power source as they could handle 3 to 32 Volts DC. Two fuses provide a fail-safe in the case of short circuits. Each fuse takes 10 or 20 Amps breakers.

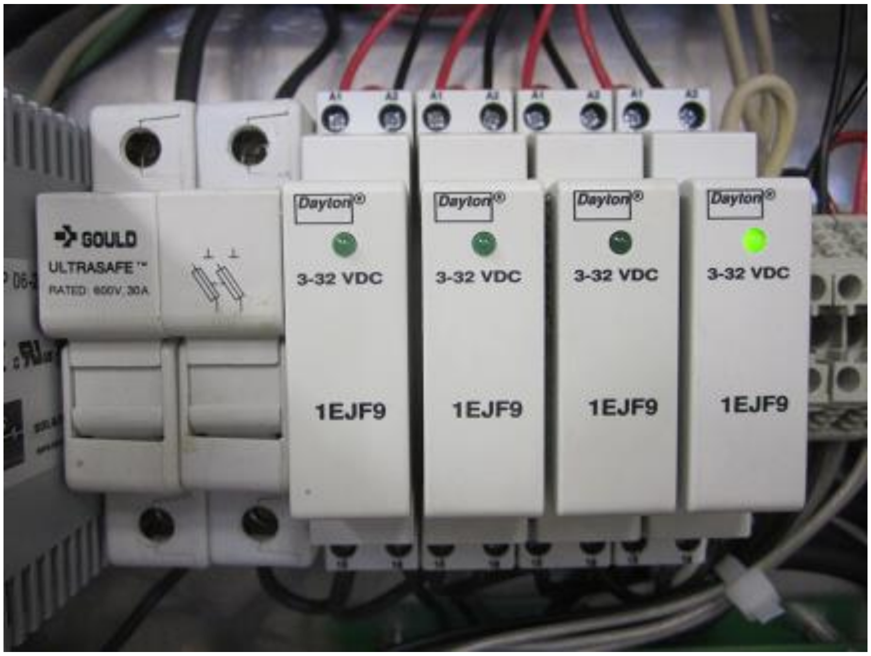


Figure 42: Solid State Relays

3.10 Power Supply and Transformer

Power supply (**Figure 43**) is provided by SOLA SDP 06-24-100T and provides 24 Volts DC for the pressure transducers and heaters. The pressure transducers are attached with a resistor which converts power into current for the Measurements and Automations software. The heating system uses a maximum of 952 Watts and the overall power was designed to go up to 1,800 Watts. A plug is connected to the laboratory outlet with a 125 Volt, 20 amp twist lock. For use in Qatar, a transformer was provided by Britt Rice. The transformer shown in **Figure 43** has a Qatar outlet BS-1363 plug and two outlet sockets for U.S. based straight blade plugs. The voltage is stepped down from 110 Volts to 220

Volts. The injection pump which uses 150 Watts is plugged into these inlets. Fraction collectors and computer are connected without requirement of a transformer.

Figure 43: Power Supply and Transformer

3.11 Computer

The computer provided by Texas A&M University operates on Windows 7 Professional with a Pentium D processor and 2 gigabytes of ram. The latest NI-DAQ device driver is version 9.5.5F4, Measurements and Automation is version 5.3.3F2, and LabVIEW is version 12.0.0. Hardware installation of a PCI 6221 port was required for the computer. A voltage switch is located on the back side. It is recommended to not modify further updates to the software on the computer.

3.12 Measurements and Automation

Measurements and Automation is an interface that receives data from the pressure transducers, the thermocouples, and the thermo-probe. The software is designed to test interfaces and calibrate the pressure transducers. LabVIEW can utilize scales and DAQmx temperature tasks via this software. To copy software into a new computer, the following procedure is used:

1. On old computer open Measurements and Automation.
2. Click on File > Import and follow the wizard.
3. Under file type choose the ".nce" format and click "Next".
4. Select all check boxes under the My System tree. These configurations transfer to the new computer. Click Export.
5. Transfer the ".nce" file to a new computer and run Measurements and Automation.
6. Click on File > Import and follow the wizard.
7. Select all check boxes under the My System tree and click Import.

3.13 LabVIEW

LabVIEW records pressure transducer data, temperature data, and turns the heaters on and off. The code is broken down into several sections: pressure transducer input (**Figure 44**), permeability and differential pressure graph outputs (**Figure 45**), thermocouple or thermo-probe input to graph **(Figure 47**), one fluid heater (**Figure 48**) and three heating tape signal outputs (**Figure 46**). To gain a better understanding of the code, refer to the annotations on the back panel of LabVIEW.

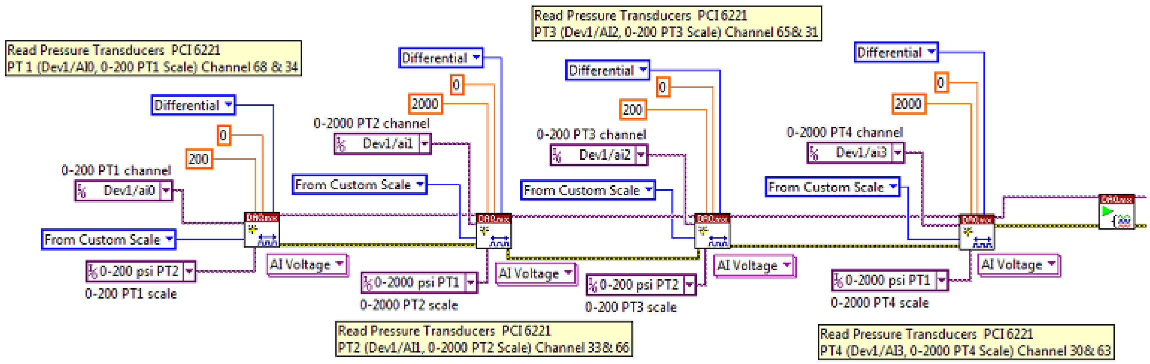


Figure 44: 4 Pressure Transducer Signal Inputs

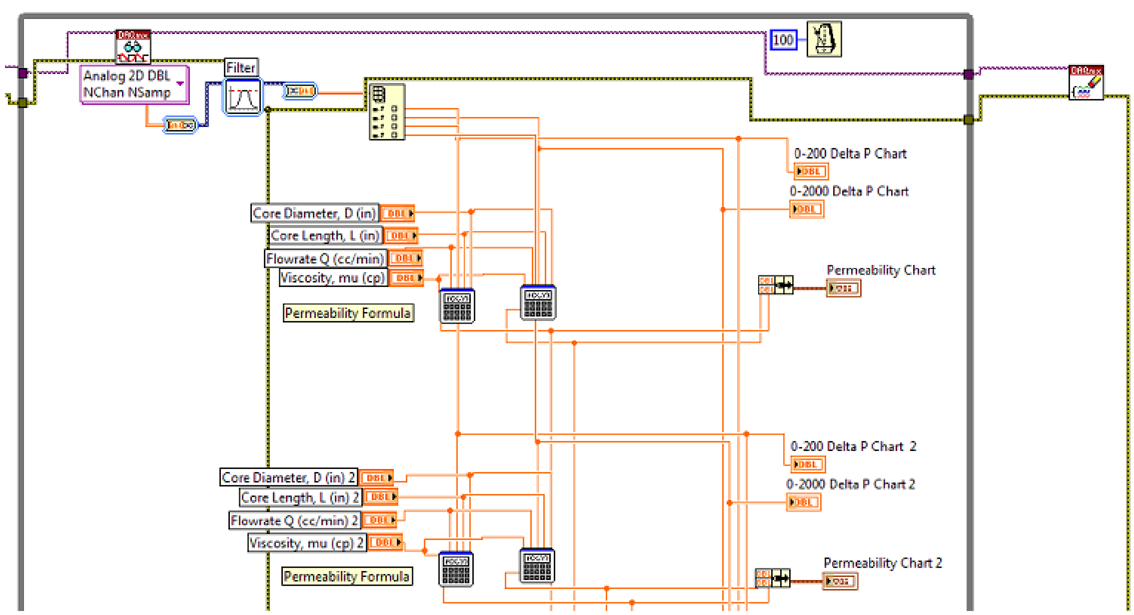


Figure 45: 4 Pressure and Permeability Graph Outputs

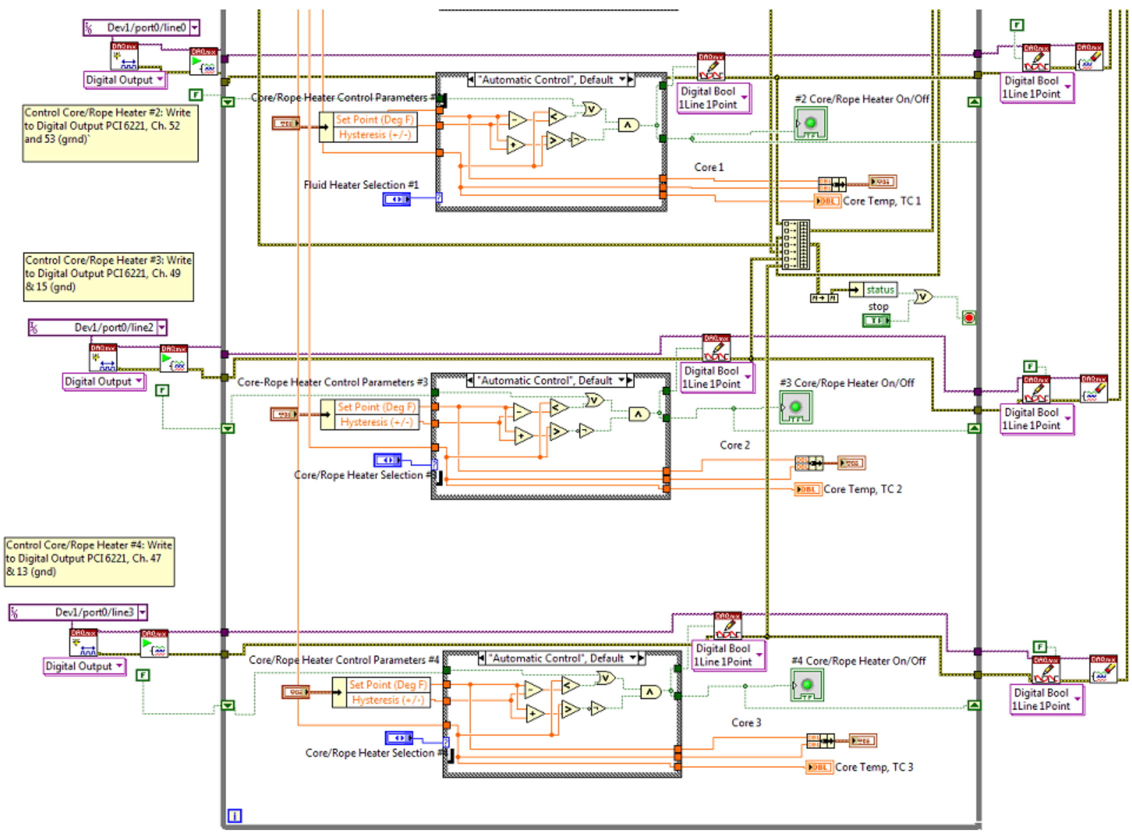


Figure 46: Heating Tape Outputs and Switches

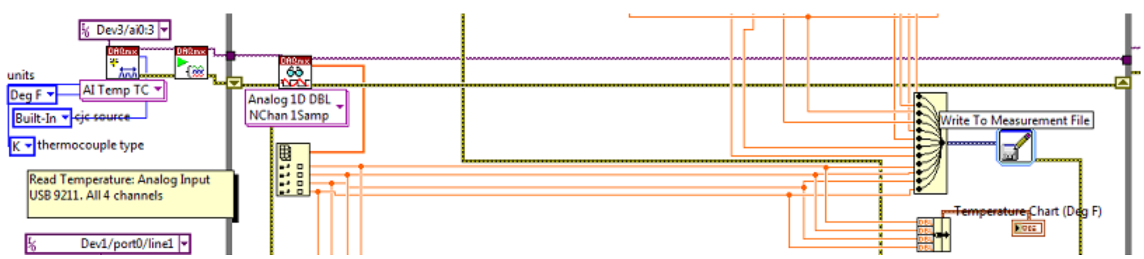


Figure 47: Temperature Inputs and Graph Outputs

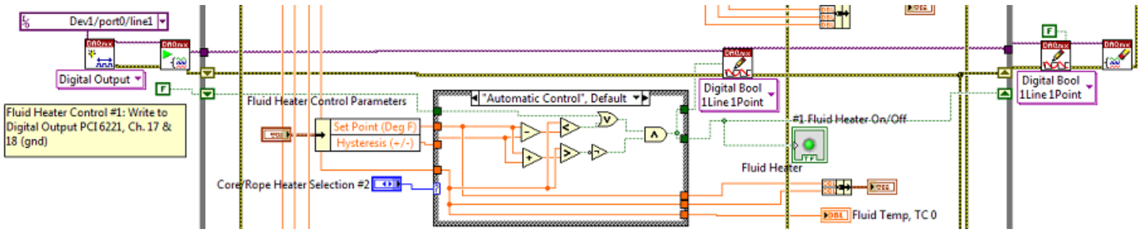


Figure 48: Fluid Heater Output and Switch

The front panel of LabVIEW (**Figure 49**) is where the user inputs core data for the experiment. Instructions are covered step by step during experimental procedure in chapters 5 and 6. It is recommended that the user spend time understanding how to turn the heaters on and off and to understand how to input core parameters and troubleshoot the code.

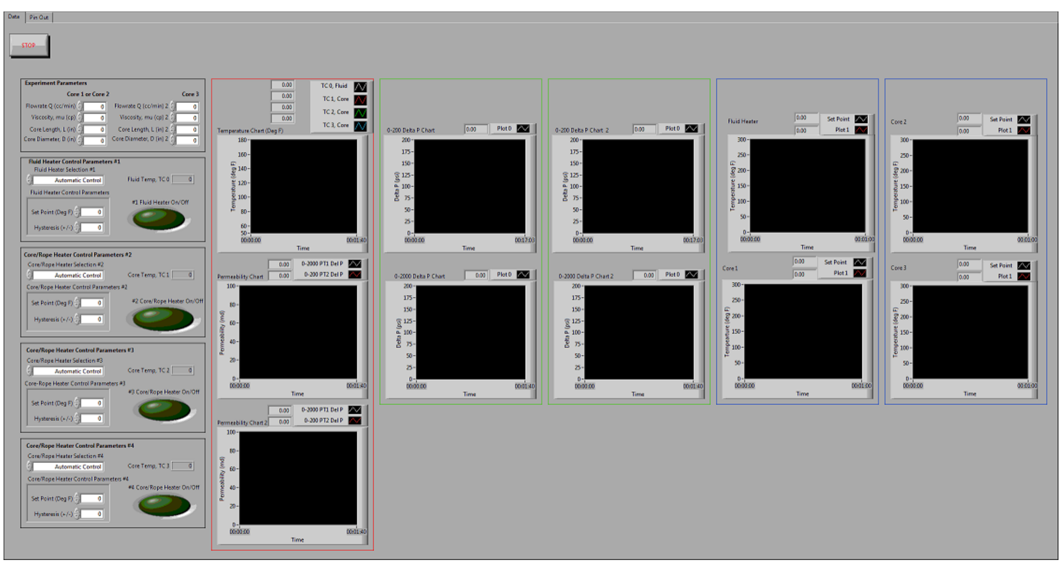


Figure 49: LabVIEW Front Panel

To input temperature, simply place the cursor on the set point box and type the desired temperature. The green circle turns light green once on. Note that it takes time for temperature to rise based on heat transfer time from heating tape or fluid heater to the core holder or liquid respectively. Core parameters for single and parallel core flood experiments are set such as flow rate, viscosity, core length, and core width. The user can click RUN to save the file to start the program and click STOP to end the program. Finally, a file is generated for excel or notepad. Details on analyzing recorded data are shown in chapter 7.

4. PRE-EXPERIMENTAL SETUP

4.1 Summary

To run an experiment, proper procedures are followed. The following instructions are summarized for preparing a matrix acidizing core flood experiment:

1. Porosity of the core is measured and the saturated core is kept on standby.
2. Apparatus is checked for working condition.
3. Acid mixture is created.
4. Brine and acid are filled into accumulators.
5. Core is inserted into core holder. Core holder is connected to flow lines.
6. Valve configuration is selected for the core holders, overburden, and pressure transducer sets.

4.2 Health, Safety, Environment Policy

Strict protocols are followed in laboratory due to use of high pressure, high temperature, hazardous chemicals, and heavy equipment. The user is required to be prepared, organized, and vigilant. The user must wear proper clothing, use caution with all fluids, and know where to seek help. Laboratory has no tolerance for eating, drinking, unchecked spills of chemicals, operation of equipment without permission, misuse of equipment by untrained users, or horseplay. In the event of rules broken, user must contact laboratory manager and instructor to take immediate action such that safety is not compromised.

In preparation for the experiment, it is recommended to have two individuals in the laboratory to ensure safety and keep vigilance on proper procedures. One of the users should be fully responsible for procedures and have experience with previous core flood experiments. The second user is then trained effectively. The laboratory should be clean and trip hazards should be noted. Proper clothing is mandatory: safety glasses, gloves, laboratory coat, full pants, and covered shoes. Steel toed shoes are preferred during placement and removal of core holders. An acid mask or face shield is recommended when creating acid mixtures and pouring acid into refill tank. Acid should be stored in a cool, dry, ventilated storage area with good drainage capabilities. Water should never be added into the acid solution as this causes rapid heat buildup.

In the event of accidents with acid, Material Safety Data Sheet covers procedures:

- Inhalation: Remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. Get medical attention immediately.
- Ingestion: Do not induce vomiting. Give large quantities of water or milk if available. Never give anything by mouth to an unconscious person. Get medical attention immediately.
- Skin Contact: In case of contact, immediately flush skin with plenty of water for at least 15 minutes while removing contaminated clothing and shoes. Wash clothing before reuse. Thoroughly clean shoes before reuse. Get medical attention immediately.

- Eye Contact: Immediately flush eyes with plenty of water for at least 15 minutes, lifting lower and upper eyelids occasionally. Get medical attention immediately.
- In the event that acid must be disposed, wear a face shield and go to the blue disposable container (**Figure 50**). Open container, dispose of acid, and close container. Do not breathe fumes near open container. The beaker or flask is cleaned immediately after disposal.

Figure 50: Spent Liquids and Acid Disposal

4.3 Core Sample Preparation

1. Inspect the core sample and measure diameter and length. Observe that diameter is based on core holder sizes and does not exceed 1 inch, 1.5 inch and 4 inch for

small, medium, and large core holders respectively. Do not harm the surface of the core during preparation.

2. Record the data based on provided core reports and use a marker to label the core on the cylindrical side. Note that all core samples should be labeled in the same position.
3. Dry the core in an oven to allow liquid to escape pore space. If the core is saturated with oil, use the Dean Stark extraction method to remove oil.
4. Weigh the dry core in grams. Reset scale to 0 grams before use (**Figure 51**).

Figure 51: Dry Weight Measurement

5. Saturate the core. Place the core inside a clean vacuum pump container filled with clean water. Observe liquid entering the pore space. For 1 inch and 1.5 inch

diameter cores, use the glass container shown in (**Figure 52**). For 4 inch diameter cores, use a PVC container.

6. The glass container requires grease to seal the top. Check for a proper seal for the vacuum chamber or PVC container. Pressure gauge reads 90-100 kPa for good seals.

Figure 52: Vacuum Pump

7. Observe oil level content inside pump to avoid motor damage, fill the pump if necessary. Turn on the Trivac pump for six hours.
8. Observe that no air bubbles are visible once fully saturated.
9. Stop the pump. Remove core and weigh in grams (**Figure 53**).

Figure 53: Saturated Core Weight

10. Record mass of saturated core to get porosity.
11. Pour water into a container and place the core inside.

4.4 Apparatus Preparation

The apparatus should be in full working condition prior to being filled with liquids and prior to inserting the core. The following check list is completed after the core is saturated.

1. Have a safety moment before working on the apparatus. Note where accident kit, shower, and eye wash are located. Wear proper safety gear.
2. Clear out all obstacles around or on the apparatus and check for trip hazards. Wheels of the apparatus must be in locked position to avoid movement.

3. In the event of liquid spills around apparatus, report to laboratory manager and instructor.
4. Check for hydraulic oil, water source, air source, and nitrogen source in laboratory.
5. Check fume hood vent for proper function. Apparatus must be under the closed area as shown in **Figure 54**.
6. Check the connection from nitrogen tank to back pressure regulator. It is connected with a 1/8 inch Swagelok tubing union.
7. Attach the 125 Volt, 20 amp twist lock connection to the laboratory outlet. With the Qatar configuration this connection goes into a transformer which is then connected to the laboratory outlet at 220 Volt.

Figure 54: Cleaned Apparatus in Locked Position

8. Check connection for the cable connecting from the large data acquisition board to the back of the computer with port PCI 6221. Measurements and Automation software can confirm that the board is being read on the computer.
9. Observe that pressure transducers are turned on.
10. Inspect the heating tapes and heater to be functioning. Use LabVIEW if necessary.
11. Check the air source from the laboratory source connection to the refill tank with a 1/8 inch Swagelok connection (**Figure 55**).

Figure 55: Air Connection for Refill Tank

12. Check the thermocouple connection to PC with USB outlet. Measurements and Automation confirms if the USB is being read on the computer.
13. Check the injection pump connection to the laboratory outlet or to the transformer.
14. Observe the injection pump reservoir to be filled with water or hydraulic oil of 1-2 cp. Do not allow the injection pump to run out of liquid. Keep a water beaker near the reservoir during experiment. Refer to chapter 2.6 for troubleshooting injection pump.
15. Check the fraction collector connection for parallel core flood experiments. Place test tubes on fraction collector and turn on.
16. Check overburden pump reservoir for hydraulic oil. Open the black cap on top and pour AW-32 hydraulic oil when low. Pump should be on vent mode.
17. Check all Swagelok and Gyrolok tubing connections to be secured to avoid leaks during experiment.
18. Place cleaned beakers or flasks into locations shown on the schematic (**Figure 3**) for liquids. Exiting liquid should be properly sealed prior to going into the flasks.

4.5 Acid Preparation

Acid is prepared in a safe manner for each experiment. Typical concentrations for acid are 15 weight percent or 28 weight percent. The maximum amount can be set up to 36.5 weight percent by mixing water and acid. The following steps are used to prepare the mixture:

1. Have a safety moment to understand the dangers of HCl. Check to make sure gloves, lab coat, safety glasses, safety mask, fully clothed pants, and covered shoes are worn. Check to make sure fume hood is functioning.
2. Note the desired weight percent required for experiments. The HCl purchased had 36.5 weight percent concentration and 1.18 g/mL for density. The density of 15 wt % HCl solution used is 1.07 g/mL. To calculate the weight percent:

$$\text{Volume HCl} = \frac{\text{Wt}_{\text{required}} * \rho_{\text{required}}}{\text{Wt}_{\text{given}} * \rho_{\text{given}}}$$

   For example to get 15 wt % HCl in 1 liter of water solution:

$$\frac{0.15 \text{ wt \%} * 1.07 \text{ g/mL}}{0.3646 \text{ wt \%} * 1.18 \text{ g/mL}} * 1000 \text{ mL} = 373.05 \text{ mL HCl}$$

   Water is calculated using the same method.

$$\frac{(1 - 0.15) \text{ wt \%} * 1.07 \text{ g/mL}}{0.3646 \text{ wt \%} * 1.18 \text{ g/mL}} * 1000 \text{ mL} = 626.95 \text{ mL water}$$

3. Clean a 1000 mL graduated cylinder. Maximum amount is determined by size of acid accumulator.
4. Use caution and always pour water first into the graduated cylinder. Water is placed first to avoid high energy release from the reaction with HCl.
5. Remove acid from storage and place under a vented chamber.
6. Place graduated cylinder under the vented chamber and pour the calculated HCl amount. Hold the acid container firmly when pouring HCl and avoid contact with skin and face shown in **Figure 56**.

Figure 56: Preparing Acid

7. Keep the mixture under the fume hood until ready to pour into accumulator for experiment.
8. Label the graduated cylinder for acid. Excess acid is disposed of properly (**Figure 50**).

4.6 Injecting Brine and Acid into Accumulator

Accumulators use the injection pump and air sources. Schematic shown in **Figure 57** is used as a guide for the operation. Details on Teledyne pump operation are shown in chapter 2.6.

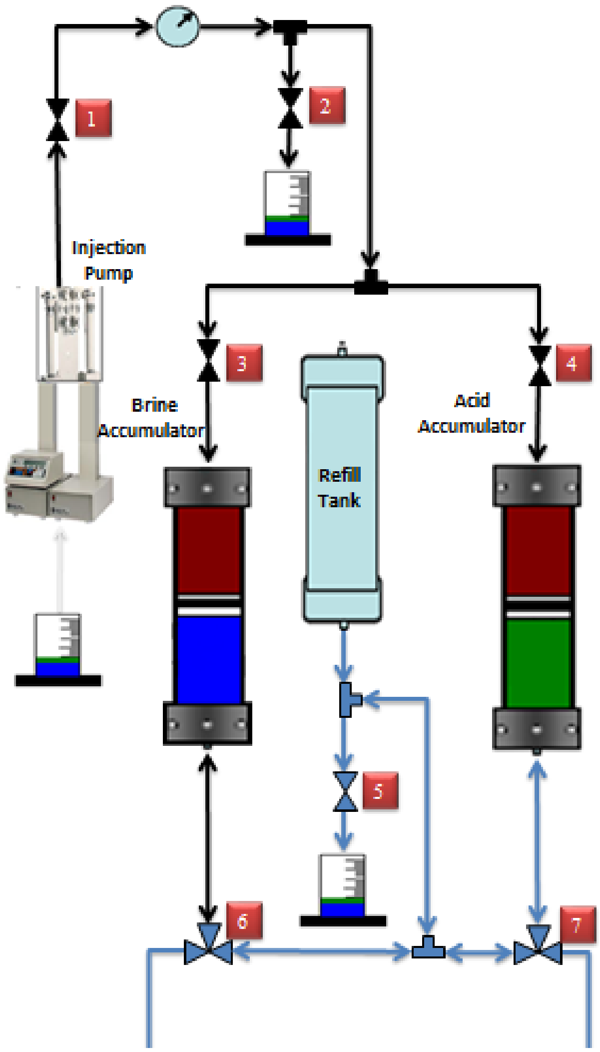


Figure 57: Accumulator Valve Schematic

4.6.1 Brine Injection

1. Confirm piston inside the accumulator is at bottom. This is noted when the pressure inside the syringe pump starts to build when the top of the accumulator is full of liquid. Open valve #6-right and valve #5 to bleed out liquid.
2. Close all valves except valve #3 and valve #4. These remain open at all times.

3. Open the top screw from the clear PVC refill tank and place a plastic funnel (**Figure 58**).

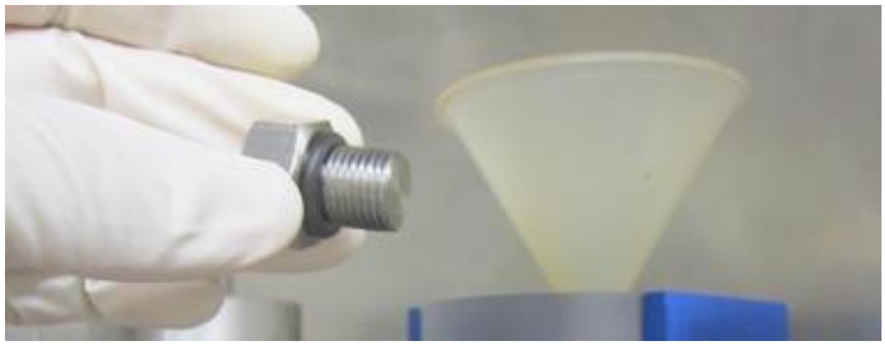


Figure 58: Top Screw and Funnel

4. Pour brine into the refill tank without spilling. The maximum amount is 1,000 mL.
5. Close the top screw and attach the air compressor tubing to top of refill tank.
6. Open valve #2 and valve #6 – right side.
7. Turn on air compressor and observe liquid level to drop inside refill tank.
8. Observe liquid starting to accumulate in the spent beaker coming out from the port connected to valve #2 (**Figure 59**). The beaker is contained in a larger bucket.

Figure 59: Liquid Bleed Observation

9. Observe until the liquid level stops dropping in the refill tank and liquid stops accumulating in the beaker. The accumulator is filled.
10. To prevent air in the accumulator, close valve #6 – right side before liquid empties in refill tank.
11. Turn off air compressor.
12. Open valve #5 to release liquid and compressed air inside refill tank into flask.

4.6.2 Acid Injection

1. Check for proper safety equipment when using HCl.
2. Confirm the piston inside the accumulator is at the bottom. This is noted when the pressure inside the syringe pump starts to build when the top of the accumulator is full of liquid. Open valve #7-left and valve #5 to bleed out liquid.
3. Close all valves except valve #3 and valve #4. They remain open at all times.
4. Open the top screw from the clear PVC refill tank and place the plastic funnel (**Figure 58**).

5. Remove acid from under the fume hood and pour into the refill tank without spilling (**Figure 60**). The maximum amount to pour is 1,000 mL.

Figure 60: Pouring Acid into Refill Tank

6. Close the top screw and attach the air compressor tubing to top of refill tank.
7. Open valve #2 and valve #7 – left side.
8. Turn on the air compressor and observe the liquid level to drop inside refill tank.
9. Observe liquid starting to accumulate in the spent beaker coming out from the port connected to valve #2 (**Figure 59**). The beaker is contained in a larger bucket.
10. Observe until the liquid level stops dropping in the refill tank and the liquid stops accumulating. The accumulator is full.
11. To prevent air in the accumulator, close valve #7 – left side before liquid empties in refill tank.

13. Turn off air compressor.
14. Open valve #5 to release liquid and compressed air inside refill tank into flask.
15. Flush the refill tank with water to remove acid. Never inject acid into the brine accumulator.
16. Throw away all waste liquid into the disposal container and wash the graduated cylinder.

4.7 Core Holder Preparation

The following instructions start with the core holder removed from the apparatus.

1. Remove the top and bottom plugs from the core holder. Inspect and clean the components with water. Confirm the overburden valves are closed.
2. Place the core into core holder from the bottom (**Figure 61**). Confirm the number on the core is facing up. If the core cannot enter, open overburden valve to release pressure in the Viton sleeve.

Figure 61: Core Inserted into Core Holder

3. Insert the spacer and the top plug (**Figure 62**). Open spacers based on the core length by pulling apart and disconnecting Swagelok union inside.

Figure 62: Spacer and Top Plug Inserted into Core Holder

4. Insert the bottom plug and twist (**Figure 63**). Attach set screw.

Figure 63: Bottom Plug Insert

5. Mount the core holder onto the frame and attach a pin to mechanically fasten (**Figure 64**). Confirm that the core holder is properly secured. The large core holder does not require this procedure.

Figure 64: Pin Fastener

6. Slide the screw cap on to the top of the core holder and firmly tighten (**Figure 65**). Confirm that there is no space between the spacer and core. Empty space causes Viton sleeve to rupture during experiments.

Figure 65: Secured Top Screw Cap

7. Connect 6 Swagelok / Gyrolok nuts to the unions. Two inlet connections at the bottom, two overburden valve connections on the cylinder, and two outlet connections at the top (**Figure 66**) are connected. Secure connections to avoid leaks during experiments.
8. Check to make sure the green bypass valve is in open position (**Figure 66**).
9. Attach heating tape by securing it from the bottom of the core holder. Tie the threads at end of the heating tape to the top of the core holder (**Figure 67**). For the small core holder, 6 feet of heating tape is used. The medium core holder uses 10 feet of heating tape and the large core holder uses 16 feet of heating tape.
10. Confirm that the heating tape does not touch to prevent a short circuit. Do not use any adhesive as it melts and damages the heating tape.

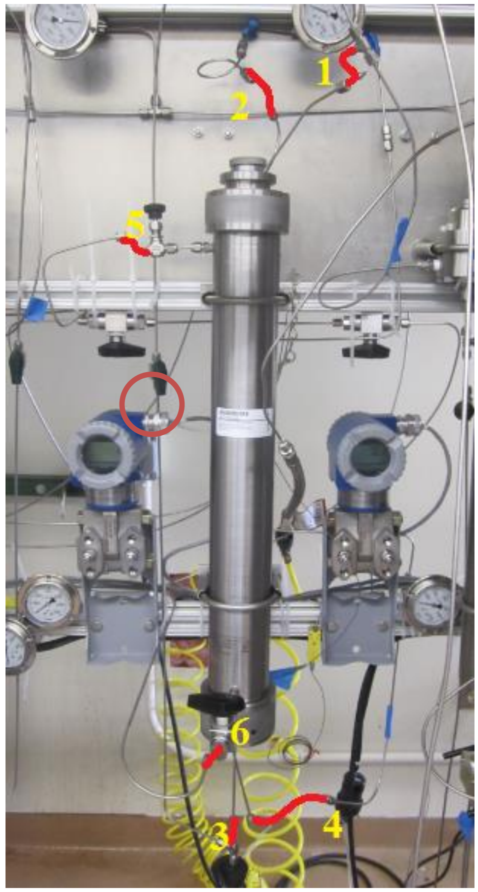


Figure 66: Inlet, Outlet, and Overburden Connections

Figure 67: Heating Tape Attached

11. Plug the heater into twist lock 1 and the heating tape into twist lock 2. Additional heating tape is plugged into twist lock 3 and 4 respectively for each core holder.
12. Attach the thermocouples to the fluid heater and the core holders respectively.
13. Use fiber glass insulation if necessary for higher temperature.
14. Repeat same process on second core holder for parallel core floods. Reverse the process to dismount and remove the core.

4.8 Core Inlet Valve Configurations

Configuration is based on referring to the inlet valve locations shown in **Figure 23** and the schematic in **Figure 20**. Four types of core floods are performed on this apparatus: core 1 (small or medium core holder), core 2 (medium core holder), core 3 (large core holder), or parallel core 1+2 (two medium core holders). Note that valve # 19 is used to bleed liquid in case of pressure build up as a safety precaution. **Table 4** shows the four options available for this apparatus.

Table 4: Core Holder Inlet Configurations

Option 1: Core 1	
Valve #	Configuration
16	Core 1+2 Open, Core 3 Close
17	Core 1 Open, Core 2 Close
18	n/a
Option 2: Core 2	
Valve #	Configuration
16	Core 1+2 Open, Core 3 Close
17	Core 1 Open, Core 2 Close
18	Core 2 Open and Core 3 Close
Option 3: Core 1+2 Parallel	
Valve #	Configuration
16	Core 1+2 Open, Core 3 Close
17	Core 1 Open, Core 2 Close
18	Core 2 Open and Core 3 Close
Option 4: Core 3	
Valve #	Configuration
16	Core 1+2 Open, Core 3 Close
17	Core 1 Open, Core 2 Close
18	Core 2 Close and Core 3 Open

4.9 Pressure Transducer Valve Configuration

Configuration is based on referring to pressure transducer valves shown in **Figure 22** and the schematic in **Figure 20**. Valves are connected to pressure transducers for three core holders. Core holders 2 and 3 share one pair of pressure transducers. The other pair is set specifically to run core holder 1. Four options are available: core 1+2 with 2000 psi, core 1+2 with 200 psi, core 3 with 2000 psi or core 3 with 200 psi. Note that core 1 and 2 can be run individually based on the configurations specified in **Table 5**.

Table 5: Pressure Transducer Valve Configuration

Option 1: Core 1 + 2 at 2,000 psi		Option 3: Core 1 + 2 at 200 psi	
Valve #	Configuration	Valve #	Configuration
8	Close	8	Close
9	Right	9	Left
10	Close	10	Close
11	Right	11	Left
12	Close	12	Open
13	Open	13	Close
14	Close	14	Open
15	Open	15	Close
Option 2: Core 3 at 2,000 psi		**Option 4: Core 3 at 200 psi**	
Valve #	Configuration	Valve #	Configuration
8	Left	8	Right
9	Close	9	Close
10	Left	10	Right
11	Close	11	Close

5. MATRIX ACIDIZING SINGLE CORE FLOOD PROCEDURE

5.1 Summary

The following steps are executed for a matrix acidizing core flooding experiment. Note that the experimental procedure involves acid at high pressure and high temperature. For this procedure, one trained user is required. This procedure is followed after the pre-experimental criteria are confirmed. The following steps are part of the procedure:

1. Pressurizing system
2. Temperature build up
3. Matrix acidizing
4. Depressurizing system
5. Post experimental clean up

5.2 Core Holder Configurations

The procedure discussed in this chapter uses a single core flood example for core holder 1. Similarly core holder 2 and core holder 3 are used based on valve configurations. **Table 6** shows alternative setup procedures for running single core experiments. Valves that are off are not used during each procedure.

Table 6: Core Selection for Procedures

#	Core 1 Configuration	Core 2 Configuration	Core 3 Configuration
Core Holder Set			
16	Core 1+2 on, Core 3 off	Core 1+2 on, Core 3 off	Core 1+2 off, Core 3 on
17	Core 1 on, Core 2 off	Core 1 off, Core 2 on	Core 1 off, Core 2 off
18	Core 2 and 3 BPR off	Core 2 on and 3 BPR off	Core 2 off and 3 BPR on
20	Core 1 Bypass	Core 1 Bypass off	Core 1 Bypass off
21	Core 2 Bypass off	Core 2 Bypass	Core 2 Bypass off
22	Core 3 Bypass off	Core 3 Bypass off	Core 3 Bypass
Overburden Set			
23	Core 1 Flow Inlet	Core 1 Flow Inlet off	Core 1 Flow Inlet off
24	Core 2 Flow Inlet off	Core 2 Flow Inlet	Core 2 Flow Inlet off
25	Core 3 Flow Inlet off	Core 3 Flow Inlet off	Core 3 Flow Inlet
26	Core 1 Inlet	Core 1 Inlet off	Core 1 Inlet off
27	Core 2 Inlet off	Core 2 Inlet	Core 2 Inlet off
28	Core 3 Inlet off	Core 3 Inlet off	Core 3 Inlet
29	Core 1 Exit	Core 1 Exit off	Core 1 Exit off
30	Core 2 Exit off	Core 2 Exit	Core 2 Exit off
31	Core 3 Exit off	Core 3 Exit off	Core 3 Exit

5.3 Pressurizing System

1. Select one option for the core inlet configuration based on **Table 4**. Valves are shown in **Figure 68**. Option 1 was selected as an example. Other configurations include:

   Option 1 – Small or Medium Core Flood on Core 1

   Option 2 – Medium Core Flood on Core 2

   Option 4 – Large Core Flood on Core 3

Figure 68: Core Selection

2. Select appropriate pressure transducer configuration Option 1 or Option 3 from **Table 5** when running core 1 or core 2. For core 3 select Option 2 or Option 4 (**Figure 69**). For this example, Option 3 was selected.

Figure 69: Core 1 and 2 at 2,000 psi

3. Open overburden valves inlet #23, core inlet #26, core outlet #29 for core holder 1. Close valve #24 and #25 (**Figure 24**).
4. Use overburden pump to apply hydraulic oil. Keep checking liquid levels in the pump to make sure hydraulic oil does not run out.
5. Observe when hydraulic oil comes out of the exit port (**Figure 70**). Note that it may take several pumps to fill the core holder if there is no oil inside the Viton sleeve.

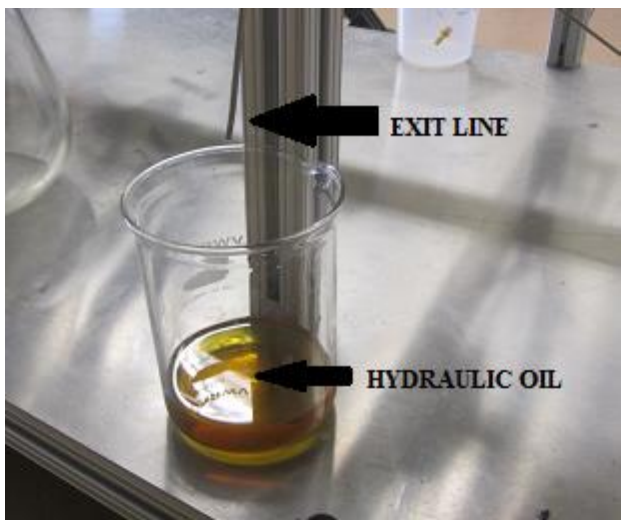


Figure 70: Hydraulic Oil Exit

6. Close needle valve #29 to seal the bleed port. Apply overburden until the pressure gauge (**Figure 71**) reads 300-500 psi. Do not apply further pressure to avoid breaking the Viton sleeve.

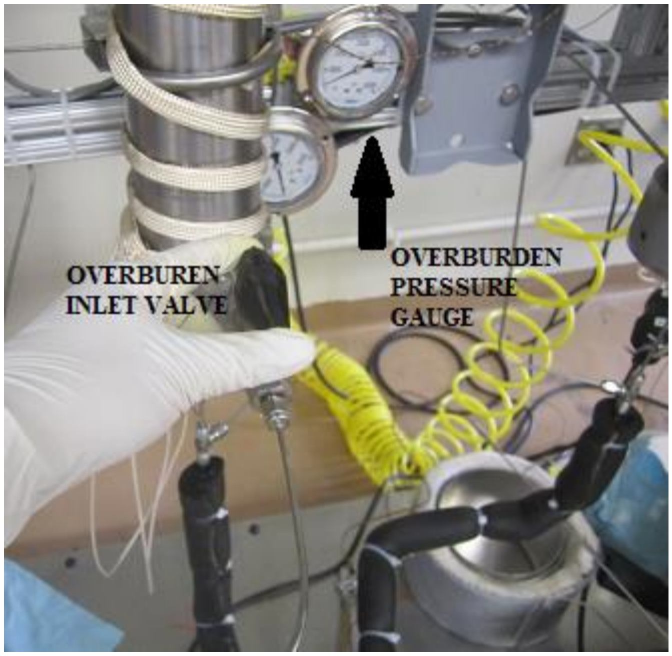


Figure 71: Overburden Pressure Gauge

7. Start injection pump at steady flow rate and open valve #1, 2, 3, and 4. Valve # 6 and #7 are closed. Note to pour liquid into injection pump reservoir throughout the experiment.
8. Observe a steady flow rate as this represents air is pushed out of the flow lines. Brine is now injected into the core holder. Close valve #2. Open valve #6 – left. Observe initial brine liquid level on injection pump and keep track until a maximum of 1 L of brine is spent. Follow refill procedure if liquid inside the accumulator is spent. Confirm bypass valve #20 is open.
9. Observe until liquid exits from the back pressure regulator into a flask (**Figure 72**). To speed the process, use a higher injection rate.

Figure 72: Liquid Exit from Back Pressure Regulator

10. Confirm that the nitrogen bleed valve #36 is closed. Apply back pressure from nitrogen tank as shown in chapter 2 and **Figure 17**. Observe that 3,000 psi or the highest amount of pressure builds in nitrogen tank. For example 1,500 psi was used **(Figure 73)**.

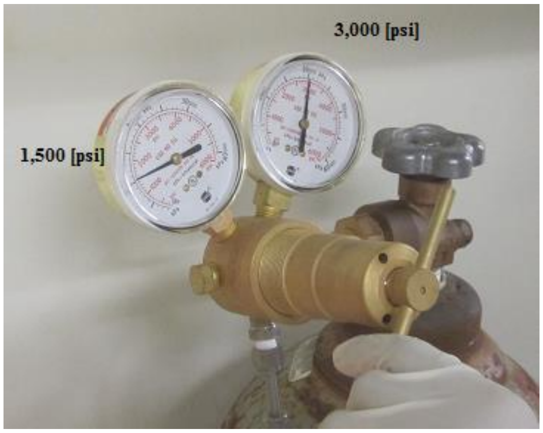


Figure 73: Setting Back Pressure

11. Observe the injection pump pressure as it rises to match the back pressure. Apply hydraulic oil with the overburden pump to make sure overburden pressure is 500 psi above injection pump pressure. Note that if overburden pressure drops below injection pressure, liquid leaks out of the core holder. This step is observed throughout the experiment.
12. Injection pressure should stabilize above back pressure and remain constant. Observe liquid coming out of the back pressure regulator port into flask.
13. Check the system for fluid leaks. If the injection pump pressure does not drop, there are no leaks.
14. Observe pressure transducer readings on core 1. Confirm that the flow rate is set to desired amount. If reading is not 0 psi, follow procedure shown in chapter 3.6.2 and reset the pressure transducer.
15. Start LabVIEW software file labeled "Matrix Acidizing Dual Core.vi" found in C:\Matrix Acidizing Dual Core Program\
16. Under the file menu > go to operate > run and an option to save the file appears.
17. Label the file name (**Figure 74**) and LabVIEW should now start recording data.
18. Input core data into the box under the experimental parameters section. Use viscosity of 1 cp for water. Note that the thermocouples should all read room temperature (**Figure 75**).

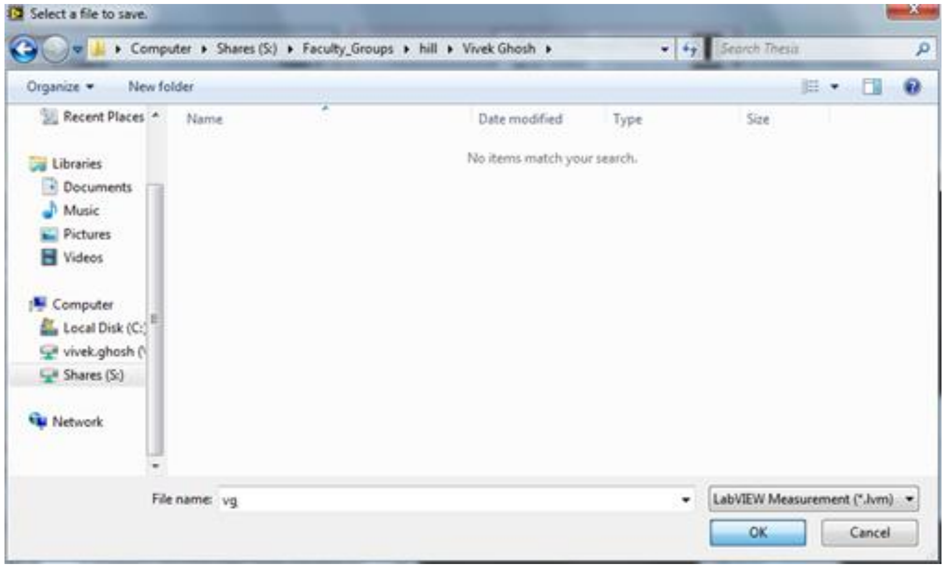


Figure 74: Save File Location

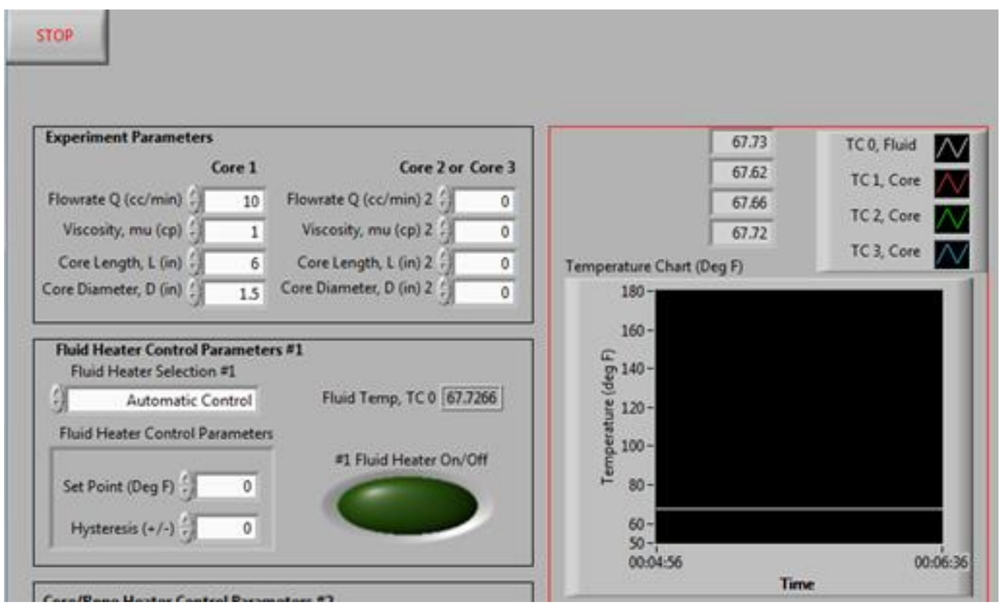


Figure 75: Core Data Inputs and Temperature Observation

19. Close bypass valve #20 and watch a pressure curve build on 0-2,000 psi Chart 1. If 200 psi pressure transducers were configured, pressure curve builds there.
20. Observe a differential pressure reading in LabVIEW. Allow pressure to stabilize and record to get permeability using Darcy's law.

5.4 Temperature Build Up

21. In LabVIEW, input a desired temperature in the set point box for Fluid Heater Control Parameters #1 and Core/Rope Heater Control Parameters #2. Place a Hysteresis of 1-2 in each box (**Figure 76**).

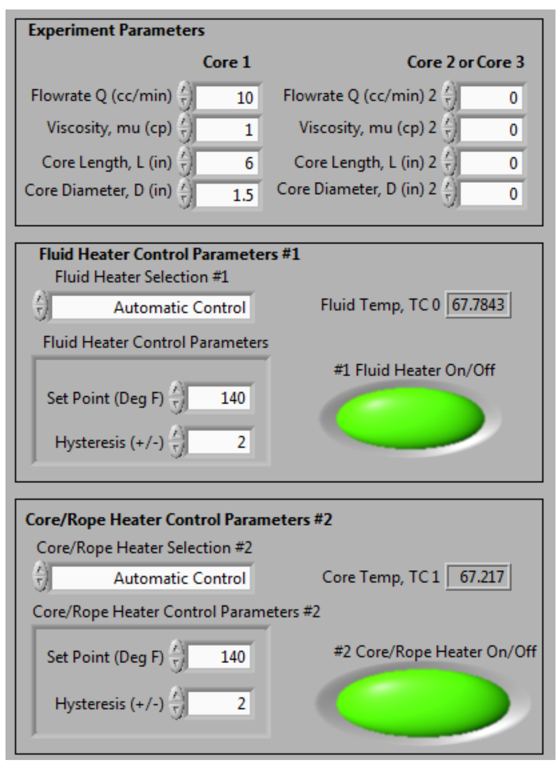


Figure 76: Temperature Control with 140 ºF

22. Observe the fluid heater temperature as it rises rapidly. The thermo-probe may be connected through a different port which is not being used. Note that the fluid heater (white) needs to go up higher than desired temperature and must be increased manually. **Figure 77** shows how temperature in the fluid heater turns on and off to achieve a desired set temperature for the core holders and liquid. This process may take up to 3 hours depending on set temperature.

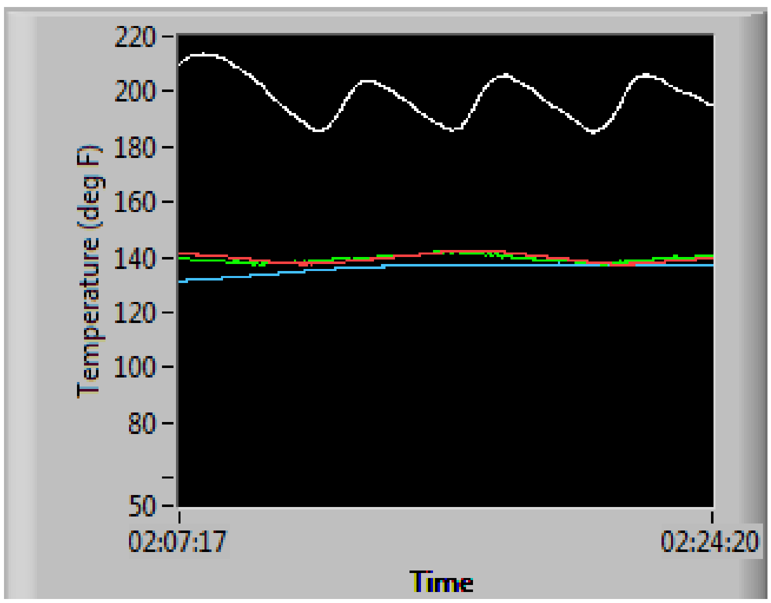


Figure 77: Steady Temperature Reading

23. Observe the overburden pressure. Temperature increases pressure in the Viton sleeve and therefore hydraulic oil should be bled out from valve #29.

24. Once the differential pressure and the temperature reach steady state, the experiment is ready for acidizing. Perform a final check for leaks, stabilized readings on pressure transducers, temperatures, back pressure, and overburden pressure. There should be sufficient reservoir liquid left in the injection pump and in the brine accumulator. Prepare a timer and a notepad for documentation.

5.5 Matrix Acidizing

25. Open acid injection valve #7-right (**Figure 78**). It is important to open this valve first before closing brine so that the injection pressure is kept constant inside both accumulators.

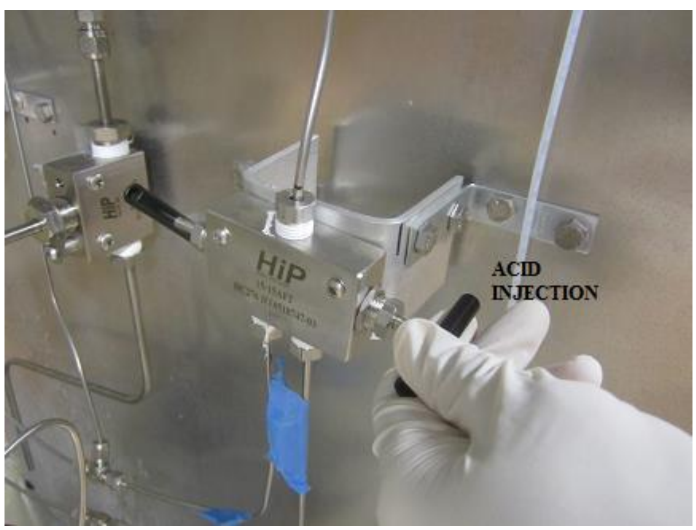


Figure 78: Acid Injection Open

26. Immediately close brine injection valve #6-left (**Figure 79**).

27. Start timer immediately. Note that it takes acid time to travel through the tubing into the core itself and this time is subtracted for calculations.
28. Observe the change in differential pressure with LabVIEW and observe the color of liquid in flask.

Figure 79: Brine Injection Close

29. Once differential pressure starts dropping rapidly, observe exit liquid with a pH reader and confirm that acid appears. Use caution and wear a mask if necessary.
30. Stop and record time when the differential pressure reaches approximately 0 psi.
31. Flush the flow lines with brine to remove acid. Open brine injection valve #6-right and close acid injection valve #7-left. Observe with pH strip to confirm all acid is removed from the flow lines.

32. LabVIEW is now completed with data. Type “0” for all temperature to turn off the heater and heating tape. Hit the “STOP” button on LabVIEW and close the software.

5.6 Depressurizing System

33. Press “STOP” button on injection pump.
34. Close brine injection valve 6-left and open valve #2.
35. Close the valve on top of the nitrogen tank fully.
36. Carefully open nitrogen bleed valve connected to the nitrogen tank (**Figure 80**). Observe that back pressure slowly drops. Observe that the spent liquid exiting flow lines are directed to beakers and flasks as liquid may flow out rapidly.

Figure 80: Open and Close Nitrogen Bleed Valve

37. Release the overburden needle valve #29 slowly and keep overburden pressure above back pressure at all times. It is important to keep about 100 psi with overburden pressure when the back pressure gauge reads 0 psi. Do not release all overburden pressure as liquid may leak out of the core holder from the bottom plug.
38. Open bypass valve #20.
39. Release bleed valve #2. Observe pressure gauges on the apparatus are read 0 psi.
40. Observe the apparatus until the core holder cools down to room temperature. This can take 1 hour at maximum.
41. Release all overburden from the needle outlet valve #29. Close the overburden inlet and outlet valves #26 and #29. This keeps hydraulic oil from spilling out of the core holder.

5.7 Core Removal and Clean Up

42. Remove heating tape and thermocouple from the core holder and disconnect.
43. Disconnect 6 connections on the core holder (**Figure 66**). Verify that the two overburden valves are still connected with core holder.
44. Unpin the back of the core holder clamp and pull out of the apparatus. Use caution as the core holder is heavy. The large core holder is rotated if used.
45. Remove the core from the core holder by reversing the procedure in chapter 4.
46. Observe the core and wash with water. Record observations and take pictures of the wormhole.
47. Wash all core holder equipment with water.

48. All remaining acid should be removed from the accumulator. Use the injection pump to run liquid into the acid accumulator and collect acid through bleed valve #5. Open valve #7-right to remove acid.
49. Flush the acid accumulator with water once by refilling process.
50. Dispose all spent liquid into liquid waste disposal.
51. Clean the apparatus and check to make sure there are no spills. Rinse beakers, check all pressure is out of the apparatus, and turn off all equipment.

6. MATRIX ACIDIZING PARALLEL CORE FLOODING PROCEDURE

6.1 Summary

The following steps are executed for a matrix acidizing parallel core flooding experiment. Note that the experimental procedure involves acid at high pressure and high temperature. For this procedure, one trained user is required. This procedure is followed after the pre-experimental criteria are confirmed. The following steps are part of the procedure:

1. Pressurizing system
2. Temperature build up
3. Matrix acidizing
4. Depressurizing system
5. Post experimental clean up

6.2 Pressurizing System

1. Select core inlet configuration **Option 3** from **Table 4** shown in **Figure 68**.
2. Select appropriate pressure transducer configuration **Option 1** or **Option 3** from **Table 5**. Example shows 2,000 psi selected (**Figure 69**).
3. Open overburden valves inlet #23, core inlet #26, core outlet #29 for core holder 1. Open overburden valves inlet #24, core inlet #27, core outlet #30 for core holder 2. Close valve #25 for third core holder (**Figure 24**).
4. Use the overburden pump to apply the hydraulic oil. Keep checking liquid levels in the pump to make sure the hydraulic oil does not run out.

5. Observe when hydraulic oil comes out of the exit port (**Figure 70**). Note that it may take several pumps to fill each core holder if there is no oil inside the Viton sleeve.
6. Close both needle valves #29 and #30 to seal the bleed ports. Apply overburden until the pressure gauge (**Figure 71**) reads 300-500 psi. Do not apply further pressure to avoid breaking the Viton sleeve.
7. Start injection pump at a steady flow rate and open valve #1, 2, 3, and 4. Valve # 6 and #7 are closed. Note to pour liquid into injection pump reservoir throughout the experiment.
8. Observe a steady flow rate as this represents air is pushed out of the flow lines. Brine is now injected into the core holders. Close valve #2. Open valve #6 – left. Observe initial brine liquid level on injection pump and keep track until a maximum of 1 L of brine is spent. Follow refill procedure if the liquid inside the accumulator is spent. Confirm bypass valve #20 and #21 are open.
9. Observe until liquid exits from the back pressure regulators into flasks (**Figure 72**). To speed the process, use a higher injection rate.
10. Confirm that the nitrogen bleed valve #36 is closed. Apply back pressure from the nitrogen tank as shown in chapter 2 and **Figure 17**. Observe that 3,000 psi or the highest amount of pressure builds in the nitrogen tank. For example 1,500 psi was used for the preliminary experiment **(Figure 73)**.
11. Observe the injection pump pressure as it rises to match the back pressure. Apply hydraulic oil with the overburden pump to make sure overburden pressure is 500

psi above injection pump pressure. Note that if overburden pressure drops below injection pressure, liquid leaks out of the core holder. This step is observed throughout the experiment.

12. Injection pressure should stabilize above back pressure and remain constant. Observe liquid coming out of the back pressure regulator into each flask.
13. Check the system for fluid leaks. If the injection pump pressure does not drop, there are no leaks.
14. Observe pressure transducer readings on core 1 and core 2. Confirm that the flow rate is set to desired amount. If reading is not 0 psi, follow procedure shown in chapter 3.6.2 and reset the pressure transducers.
15. Start LabVIEW software file labeled “Matrix Acidizing Dual Core.vi” found in C:\Matrix Acidizing Dual Core Program\
16. Under the file menu > go to operate > run and an option to save the file appears.
17. Label the file name (**Figure 74**) and LabVIEW should now start recording data.
18. Input core data into the both boxes under the experimental parameters. Use viscosity of 1 cp for water. Note that the thermocouples should all read room temperature (**Figure 81**).
19. Close bypass valve #20 and #21 for both core holders and watch a pressure curve build on 0-2,000 psi Chart 1 and 2. If 200 psi pressure transducers were configured, pressure curve builds there.
20. Observe that both pressure transducers read the same differential pressure.

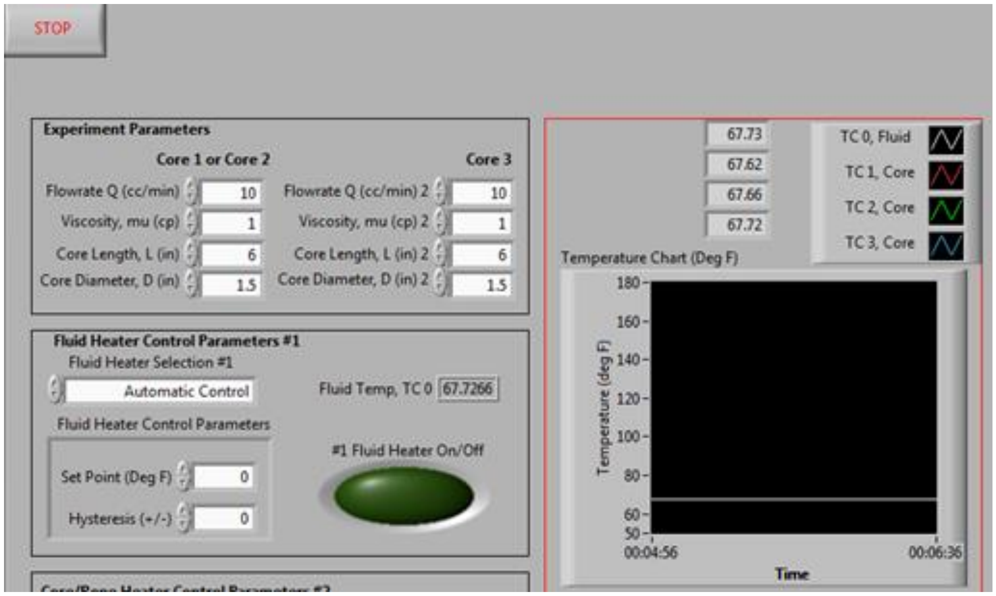


Figure 81: Input Parallel Core Data and Observe Temperature

21. Close inlet #17 for core 2. This raises the differential pressure on core 1. Allow pressure to stabilize and record maximum pressure for core 1 to get absolute permeability.
22. Open inlet #17 for core 2 and close inlet #16 for core 1. This raises the differential pressure on core 2. Allow pressure to stabilize and record maximum pressure for core 2 to get absolute permeability.
23. Reopen valve #17 for core 1 and 2 to reach steady state pressure.

6.3 Temperature Build Up

24. In LabVIEW, input a desired temperature in the set point box for Fluid Heater Control Parameters #1, Core/Rope Heater Control Parameters #2, and Core/Rope Heater Control Parameters #3. Place a Hysteresis of 1-2 in each box (**Figure 82**).

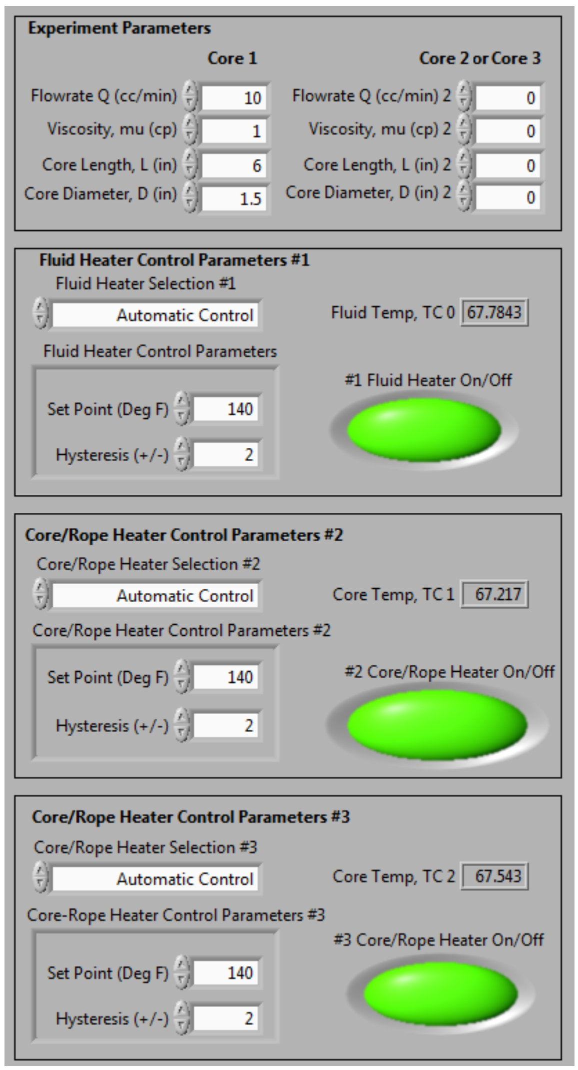


Figure 82: Temperature Control with 140 ºF

25. Observe the fluid heater temperature as it rises rapidly. The thermo-probe may be connected through a different port which is not being used. Note that the fluid heater (white) needs to go up higher than desired temperature and must be increased manually. **Figure 77** shows how temperature in the fluid heater turns on and off to achieve a desired set temperature for the core holders and liquid. This process may take up to 3 hours depending on set temperature.
26. Observe the overburden pressure. Temperature increases pressure in the Viton sleeve and therefore hydraulic oil should be bled out from valves #29 and #30.
27. Once the differential pressure and the temperature reach a steady state, the experiment is ready for acidizing. Perform a final check for leaks, stabilized readings on pressure transducers, thermocouples, back pressure, and overburden pressure. There should be sufficient reservoir liquid left in the injection pump and in the brine accumulator. Prepare a timer and a notepad for documentation.

6.4 Matrix Acidizing

28. Prepare the flow rate timer on both fraction collectors to collect spent liquid samples based on the injection rate. 80 test tubes should be cleaned and in place.
29. To set the timer, press "EDIT", "TIME", input time in decimals, "OK", and "OK". Capacity of a test tube is 10 mL. For example, if the injection flow rate is 10 mL/min, use 0.5 min/tube to get a maximum of 5 mL per test tube every 30 sec. Once set, the screen appears as shown (**Figure 83**).
30. Divert flow from spent liquid flasks to fraction collectors by closing valves # 32 and # 34. Open valves # 33 and #35 as shown in **Figure 25**.

Figure 83: Fraction Collector Panel Setting

31. Observe liquid collecting in the beaker for fraction collector (**Figure 84**). Use tape to hold tube in place on beaker due to movement from liquid flowing through.

Figure 84: Fraction Collector Diverted Liquid

32. Open acid injection valve #7-right (**Figure 78**). It is important to open this valve first before closing brine so that the injection pressure is kept constant inside both accumulators.
33. Immediately close brine injection valve #6-left (**Figure 79**).
34. Start timer immediately. Note that it takes acid time to travel through the tubing into the core itself and this time is subtracted for calculations.
35. Start fraction collectors together simultaneously. Press the “ENTER” button as shown (**Figure 85**).

Figure 85: Start Fraction Collectors

36. Observe that the liquid is properly being collected in each fraction collector. No liquid should spill outside of the test tubes (**Figure 86**).

37. Observe the change in differential pressure with LabVIEW and observe the color of liquid in each test tube. Observe overburden pressure. Bleed excess overburden pressure when necessary.
38. Once differential pressure starts dropping rapidly, observe the test tubes with a pH reader and confirm that acid appears. Use caution and wear a mask if necessary.

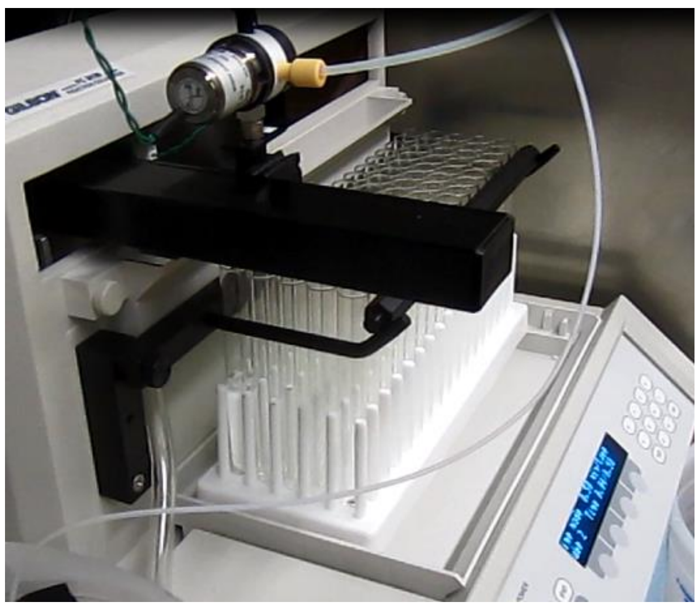


Figure 86: Liquid Sample Collection in Test Tube

39. Stop and record the time when differential pressure reaches approximately 0 psi. This means acid has broken through one of the two cores.
40. Press the "STOP" button on both fraction collectors.

41. Divert flow from fraction collectors to spent liquid flasks by closing valves # 33 and # 35. Open valves # 32 and #34 as shown in **Figure 25**.
42. Flush the flow lines with brine to remove acid. Open brine injection valve #6-left and close acid injection valve #7-right. Observe with pH strip to confirm all acid is removed from the flow lines.
43. LabVIEW is now completed with data. Type “0” for in all temperature boxes to turn off the heater and the heating tapes. Hit the “STOP” button on LabVIEW and close the software.

6.5 Depressurizing System

44. Press “STOP” button on injection pump.
45. Close brine injection valve #6-left and open valve #2.
46. Close the valve on top of the nitrogen tank fully.
47. Carefully open the nitrogen bleed valve #36 connected to the nitrogen tank (**Figure 80**). Observe that back pressure slowly drops. Observe that the spent liquid exiting flow lines are directed to beakers and flasks as liquid may flow out rapidly.
48. Release the overburden needle valves #29 and #30 slowly and keep overburden pressure above back pressure at all times. It is important to keep about 100 psi with overburden pressure when the back pressure gauge reads 0 psi. Do not release all overburden pressure as liquid may leak out of the core holder from the bottom plug.
49. Open the two bypass valves #20 and #21.

50. Release bleed valve #2. Observe pressure gauges on the apparatus are read 0 psi.
51. Observe the apparatus until the core holders cool down to room temperature. This can take 1 hour at maximum.
52. Release all overburden pressure from the needle outlet valves #29 and #30 once room temperature is achieved. Close the overburden inlet and outlet valves on both core holders #29, #30, #26, and #27. This keeps hydraulic oil from spilling out of the core holders.

6.6 Core Removal and Clean Up

53. Disconnect the fraction collectors and remove them from the apparatus. Record the volume in each test tube and clean the test tubes when completed.
54. Remove heating tape and thermocouple from each core holder and disconnect the plugs.
55. Disconnect 6 connections on the first core holder (**Figure 66**). Verify that the two overburden valves are still connected with core holder. Repeat this step on second core holder.
56. Unpin the back of the first core holder and pull core holder out of the apparatus. Repeat the same for second core holder. Use caution as core holder is heavy.
57. Remove core holder by reversing the procedure listed in chapter 4.
58. Observe the core and wash with water. Record observations and take pictures.
59. Wash all core holder equipment with water.

60. All remaining acid should be removed from the accumulator. Use the injection pump to run liquid into the acid accumulator and collect acid through bleed valve #5. Open valve #7-left to remove acid.
61. Flush the acid accumulator with water once by the refilling process.
62. Dispose all spent liquid into the liquid waste disposal.
63. Clean the apparatus and check to make sure there are no spills. Rinse beakers, check that all pressure is out of the apparatus, and turn off all equipment.

7. CALCULATIONS AND RESULTS

7.1 Summary

The time it takes for acid to break through a core is the primary output result achieved through the experiment. The result is matched with interstitial velocity to achieve the pore volume to breakthrough. The final results gained from matrix acidizing core floods are porosity, permeability, and pore volume to breakthrough. With parallel core floods, flow rate is measured for each core. The procedure to get the results is as follows:

1. recording experimental parameters
2. generate an excel graph from LabVIEW
3. calculations
4. output results

7.2 Recording Initial Parameters

Initial parameters are gathered during the pre-experimental setup to get porosity. Differential pressure is recorded before increasing the temperature and before acidizing during experiment to get permeability. **Table 7** and **Table 8** are the initial parameters for a preliminary parallel core flood experiment. Note that differential pressure is measured individually for each core. The same tables are applied for single core flood experiments.

Table 7: Pre-Experimental Data

Pre-Experiment Example			Reference
Parameters	Core 1	Core 2	
Lithology	Austin Chalk	Austin Chalk	4.3 step 2
Diameter inch	1.5	1.5	4.3 step 2
Length inch	6	6	4.3 step 2
Dry Mass g	330.7	339.3	4.3 step 4
Saturated Mass g	376.5	377.9	4.3 step 9
Acid wt %	15		4.5 step 2

Table 8: Experimental Data

Experiment Example			Reference	
Parameters	Core 1	Core 2	Single Core	Parallel Core
Injection Rate mL/min	10		5.3 step 7	6.2 step 7
Back Pressure psi	1,500		5.3 step 10	6.2 step 10
Differential Pressure psi for Perm.	110	700	5.3 step 20	6.2 steps 21,22
Temperature F	140		5.3 step 21	6.3 step 24
Acid Injection to Breakthrough s	374		5.5 step 30	6.4 step 36

7.3 Generating Excel Graph

The graph can be opened with excel once created with LabVIEW. From left to right each column represents data as follows: Time min, Core 1 200 pressure transducer psi, Core 1 2,000 pressure transducer psi, Core 1 permeability based on 200 pressure transducer md, Core 1 permeability based on 2,000 pressure transducer md, Core 2 200 pressure transducer 200 psi, Core 2 2,000 pressure transducer psi, Core 2 permeability based on 2,000 pressure transducer md, Core 2 permeability based on 200 pressure transducer md, fluid heater temperature ºF, Core 1 temperature ºF, Core 2 temperature ºF, Core 3 or thermo-probe temperature ºF. Graph is shown in **Figure 87**. Note that since the 200 psi pressure transducers were not used during this experiment, core 1 and core 2 permeability data for columns D and I do not exist.

A	B	C	D	E	F	G	H	I	J	K	L	M
Time min	Core 1 200 psi	Core 1 2000 psi	Core 1 perm 200	Core 1 perm 2000	Core 2 200 psi	Core 2 2000 psi	Core 2 perm 2000	Core 2 perm 200	Fluid Heater	Core 1 Temp	Core 2 Temp	Fluid Temp
111.7659	1.340964	63.397436	n/a	51.316481	0.26009	65.402101	49.74356	n/a	199.711379	137.526316	138.675721	138.330562
111.7746	1.294912	62.503464	n/a	52.050449	0.26009	65.483371	49.681824	n/a	199.66983	137.543544	138.766194	138.399934
111.783	1.313875	62.422194	n/a	52.118215	0.211329	65.510462	49.661279	n/a	199.536083	137.541003	138.783931	138.34897
111.7928	1.411397	62.991085	n/a	51.64752	0.379284	66.241894	49.112928	n/a	199.342773	137.499996	138.753283	138.346876
111.8018	1.365345	62.232564	n/a	52.277026	0.558075	66.241894	49.112928	n/a	199.188756	137.529744	138.832268	138.390276
111.8105	1.305748	61.230232	n/a	53.132795	0.517441	64.96866	50.075426	n/a	199.076304	137.570431	138.794728	138.386256
111.8212	1.216352	60.715521	n/a	53.583224	0.484934	64.074687	50.77408	n/a	198.883491	137.469464	138.680549	138.374294
111.8299	1.303039	61.419862	n/a	52.96875	0.357613	64.074687	50.77408	n/a	198.762431	137.533408	138.650118	138.384834
111.8385	1.297621	61.392772	n/a	52.992123	0.428045	64.237228	50.645606	n/a	198.675629	137.510328	138.682085	138.366311
111.847	1.262405	60.552981	n/a	53.727055	0.39012	63.695427	51.076404	n/a	198.553281	137.503432	138.774098	138.397079
111.8563	1.197389	59.686099	n/a	54.507387	0.298016	63.099445	51.558826	n/a	198.427717	137.456887	138.879444	138.381585
111.8648	1.197389	59.604829	n/a	54.581707	0.273635	63.045265	51.603135	n/a	198.451854	137.555546	139.003725	138.44794

Figure 87: LabVIEW Graph to Excel

7.3.1 Temperature Plot

Temperature for the heating tapes attached to core holders stabilizes evenly. During experimentation, medium core holders took 40 minutes to reach 140 ºF. To achieve max temperature rating of 250 ºF, the small and medium cores holder took 3 hours and the large core holder took 5 hours. Temperature for the fluid heater is raised higher due to heat transfer from the heater to tubing and then from tubing to liquid.

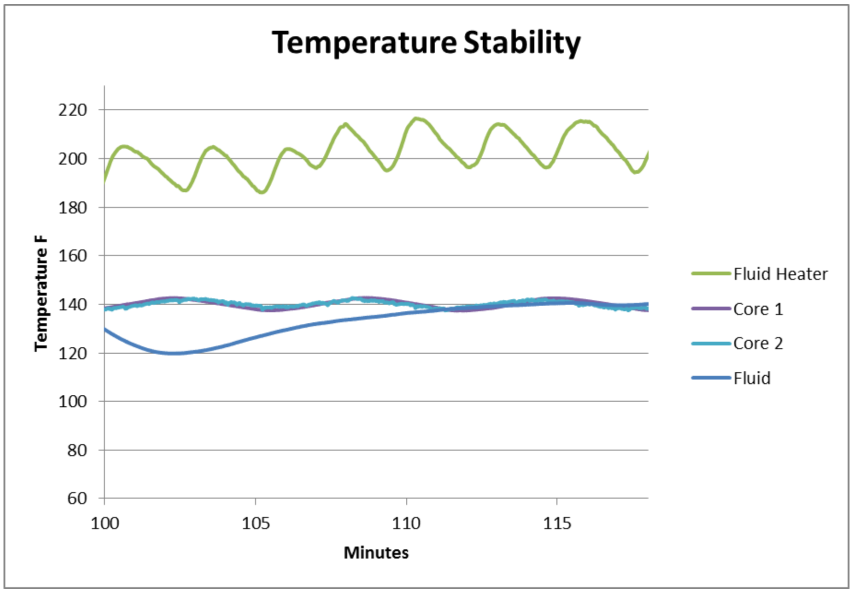


Figure 88: Temperature Stability

As shown in **Figure 88**, the fluid heater was set to 190 ºF for the liquid flowing at a rate of 10 mL/min to stabilize at 140 ºF. The fluid heater temperature is manually controlled during experiments depending on injection flow rate. A time lag occurs due to the thermo-probe being connected two feet after the fluid heater. Therefore at low flow rates liquid may appear to not increase rapidly. The fluid heater should not be set over 212 ºF at atmospheric pressures or liquid changes phases.

7.3.2 Differential Pressure Plot

Differential pressure plots are useful to show when acid breaks through the core. Note that in parallel core floods, the actual pressure drop is not valid but gives a time representation of when a wormhole was created. The following plot (**Figure 89**) shows results from the preliminary experiment.

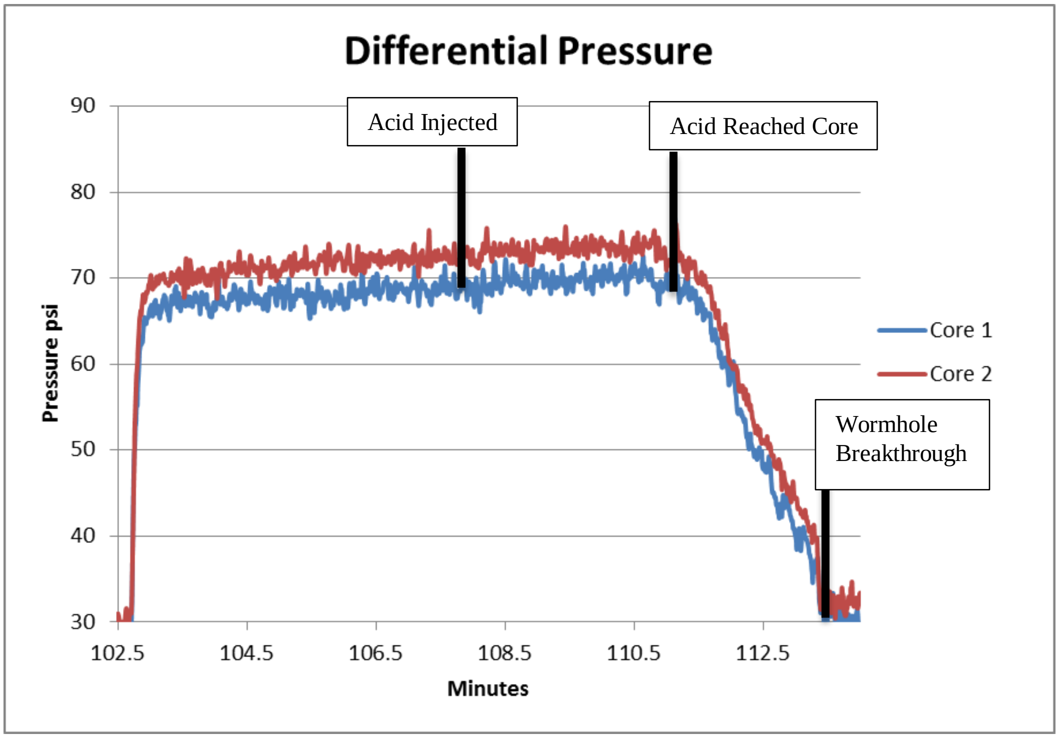


Figure 89: Differential Pressure for Parallel Core Flood

The time it takes acid to inject into the core and the time it takes to create a wormhole are explained in calculations. Note that acid injected does represent direct acid injection to core. Acid must travel through tubing to reach the core. Therefore, it is critical to time experiments as data significantly changes the final results of pore volume to breakthrough.

7.3.3 Diversion Flow Rate

Fraction collector test tubes are measured with a 10 mL graduated cylinder and recorded after the experiment is completed. Data is plotted into a graph (**Figure 90**). Note that the permeability ratio for core 1 to core 2 was 29.77:4.68 or 6.36:1. Flow rate was significantly higher in core 1 compared to core 2. Acid flowed into the high permeable core and flow rate in core 1 was observed to increase gradually until breakthrough occurred. It was observed that after breakthrough occurred, flow rate increased higher than injection rate in core 1 and no flow occurred in core 2.

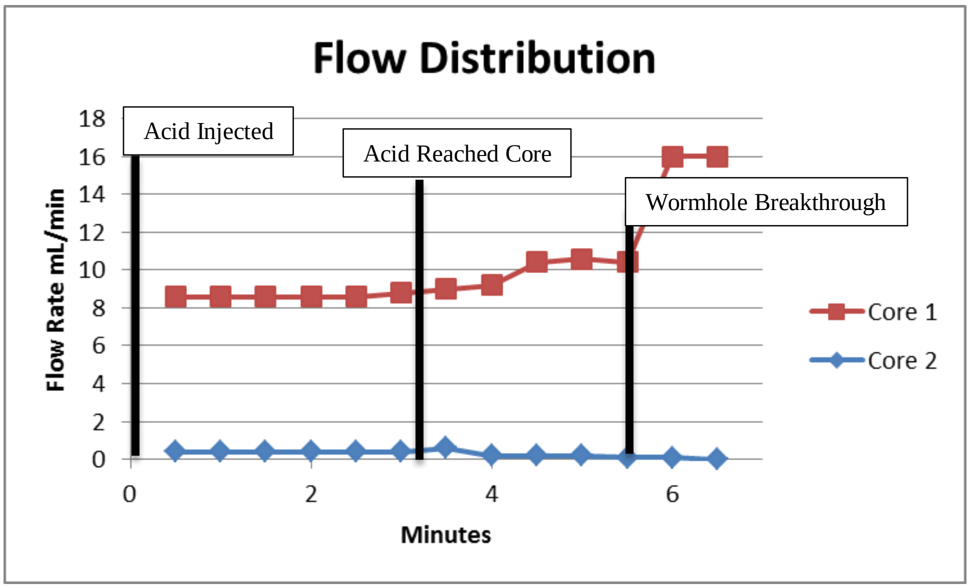


Figure 90: Distribution of Flow Rate in Each Core

7.4 Calculations

7.4.1 Porosity

$$\emptyset = \frac{(m_{saturard} - m_{dry})/\rho_{water}}{(2.54 * D)^2 * \frac{\pi}{4} * (2.54 * L)} * 100\%$$

Core 1: $\emptyset = \frac{367.5g - 330.7g/1g/cm^3}{(2.54*1.5\ in)^2*\frac{\pi}{4}*(2.54*6in)} * 100\% = 21.2\%$

Core 2: $\emptyset = \frac{377.09\ g - 339.03\ g/1g/cm^3}{(2.54*1.5\ in)^2*\frac{\pi}{4}*(2.54*6in)} * 100\% = 22.2\%$

7.4.2 Permeability

Based on steady state conditions and using pressure differential data, absolute permeability is calculated.

$$k = \frac{96.43 * 4 * Q * L * \mu}{\pi * D^2 * \Delta P}$$

Core 1: $k = \frac{96.43*4*10\frac{cm^3}{min}*6in*1cp}{\pi*1.5in^2*110\ psi} = 29.77\ md$

Core 2: $k = \frac{96.43*4*10\frac{cm^3}{min}*6in*1cp}{\pi*1.5in^2*700\ psi} = 4.68\ md$

7.4.3 Pore Volume to Breakthrough

Pore volume is calculated based on porosity:

$$Pore\ Volume = \emptyset * \frac{\pi}{4} * (D * 2.54)^2 * (L * 2.54)$$

Pore Volume Core 1:

$$Pore\ Volume = 21.2\% * \frac{\pi}{4} * (1.5\ in * 2.54)^2 * (6\ in * 2.54) = 36.8\ cm^3$$

Pore Volume Core 2:

$$Pore\ Volume = 22.2\% * \frac{\pi}{4} * (1.5\ in * 2.54)^2 * (6\ in * 2.54) = 38.6\ cm^3$$

Acid takes time to travel from the acid injection valve to core inside each core holder. The apparatus was designed with equal length of tubing between each core holder. To calculate the volume of tubing, Hastelloy tubing specification is used since the inner tubing diameter is a critical variable for volume.

Volume of tubing:

$$V = L * 2.54 * \frac{\pi}{4} * ((outer\ diameter - 2 * thickness) * 2.54)\text{^}2$$

$$V = 300\ in * 2.54 * \frac{\pi}{4} * ((0.125\ in - 2 * 0.02\ in) * 2.54)\text{^}2 = 27.9\ cm^3$$

Time for acid to get to the core is calculated with the volume of tubing and acid injection flow rate. Note that acid injection flow rate is based on data from fraction collector and not the total flow rate.

$$Time\ for\ Acid\ to\ get\ to\ Core = \frac{V}{Q} = \frac{27.9mL}{8.8mL/min} * 60sec/min = 190.2\ sec$$

Acid injection was 338.4 s. Subtracting 190.2 s of time traveling through tubing gives 148.2 s of time it took for acid to break through the core. Note that only core 1 had a wormhole therefore there is no time calculated for core 2. Pore volume to break through is calculated by integrating the acid flow rate with time and then dividing by pore volume. Data for calculating the acid flow rate with time is shown in **Figure 90**.

$$PVBT = \frac{\int_{t_1}^{t_2} Qdt}{\frac{60sec}{min} * PV} = \frac{\int_{190.2}^{338.4} Qdt}{\frac{60sec}{min} * 36.8} = \frac{29.2ccm}{36.8ccm} = 0.79$$

7.4.4 Interstitial Velocity

Interstitial velocity is the velocity of acid solution through the pore space cross sectional area of the core.

$$v_i = Q/(\frac{\pi}{4} * (2.54 * D)^2 * \emptyset)$$

For single core floods, the injection flow rate is used. Since flow rate varies in parallel core flood experiments, the flow rate where acid hits the core and the flow rate where acid breaks through the core are representatives of interstitial velocities:

$$v_i = 8.8\frac{mL}{min}/(\frac{\pi}{4} * (2.54 * 1.5in)^2 * 21.2\%) = 3.64\ \frac{cm}{min}$$

7.5 Result Output

A spreadsheet is created for every core flood. An example is shown with **Figure 91**.

Core Flooding Data Sheet

Core#	1		Date	April 15, 2013	
Core diameter	1.5	inch			
Core length	6	inch			
Porosity Measurement			Acid Core Flooding		
Weight (Dry)	330.7	g	Temperature	140	^oF
Weight (Sat)	367.5	g			
Porosity	21.2%		Acid Injection Rate	8.8	mL/min
Pore Volume	36.8	cm^3			
			Interstitial Velocity	3.64	cm/min
Permeability Measurement					
Brine Injection Rate	10	mL/min	Volume of Pipe to Core	27.90	cm^3
Pressure Differential	110	psi			
Permeability	29.77	md	Time for Acid to Get the Core	190.2	seconds
Acid Formulation	15%	wt HCl	Time for Acid Injection	338.4	seconds
Water	626.94	mL			
HCl	373.06	mL	Pore Volume Break Through	0.79	Pore volume
Core 2					
Permeability Measurement					
Brine Injection Rate	10	mL/min			
Pressure Differential	700	psi			
Permeability	4.68	md			

Figure 91: Parallel Core Flood Test Result

Based on calculations, the initial graphs in **Figure 89** and **Figure 90** were updated with acid injection timings and wormhole breakthrough timings. Pore volume to break through vs. interstitial velocity is plotted for single core floods.

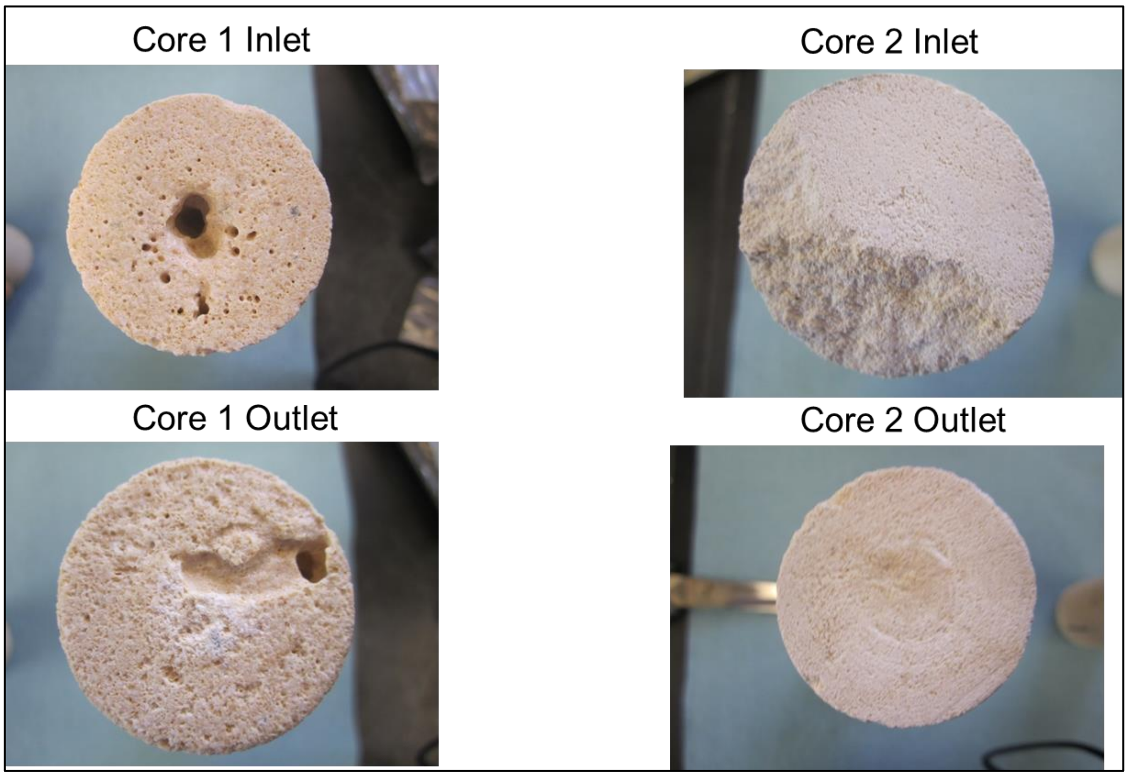


Figure 92: Austin Chalk Parallel Core Flood Pictures

Figure 92 shows the pictures of wormhole in core 1. There was no wormhole or propagation in core 2.

7.6 Repeatability Test

For repeatability, a second parallel core flood experiment was conducted. It is noted that during experiments, differential pressure did not immediately start from 0 psi. The conditions were the same conditions except for using Indiana Limestone cores and a back pressure of 2,000 psi. Core 1 broke through and the following results were recorded in **Figure 93** and **Figure 94**.

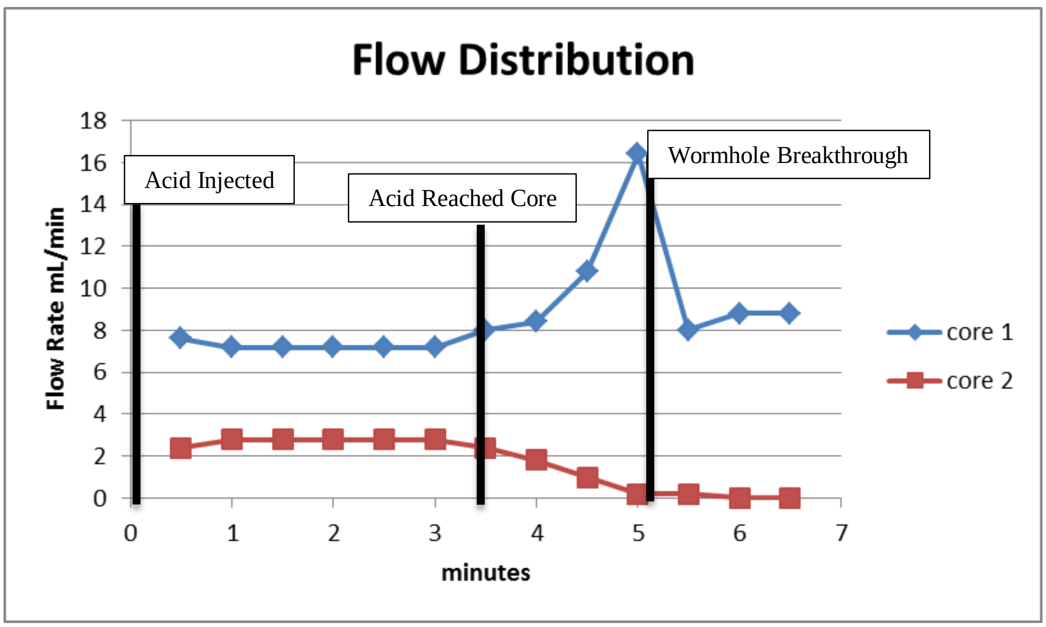


Figure 93: Distribution of Flow Rate in Each Core

Core Flooding Data Sheet

Core#	1		Date	April 30, 2013	
Core diameter	1.5	inch			
Core length	6	inch			
<u>Porosity Measurement</u>			<u>Acid Core Flooding</u>		
Weight (Dry)	380.2	g	Temperature	140	^{o}F
Weight (Sat)	405.7	g			
Porosity	14.7%		Acid Injection Rate	8	mL/min
Pore Volume	25.5	ccm			
			Interstitial Velocity	4.78	cm/min
<u>Permeability Measurement</u>					
Brine Injection Rate	10	mL/min	Volume of Pipe to Core	27.90	cm^3
Pressure Differential	68	psi			
Permeability	48.15	md	Time for Acid to Get the Core	209.2	seconds
<u>Acid Formulation</u>	15%	wt HCl	Time for Acid Injection	319.2	seconds
Water	626.94	mL			
HCl	373.06	mL	Pore Volume Break Through	0.58	Pore volume

Core 2		
<u>Permeability Measurement</u>		
Injection Rate	10	mL/min
Pressure Differential	245	psi
Permeability	13.36	md

Figure 94: Second Parallel Core Flood Test Result

During the experiment, the pressure transducers were reset to 0 psi before closing the bypass valves. In addition, a comparison was checked with the 2,000 psi and 200 psi transducers for core 1 (**Figure 95**). Note that 200 psi pressure transducer is more accurate. The two core samples were cut at 90 degree angles which is an explanation to having large differences in permeability.

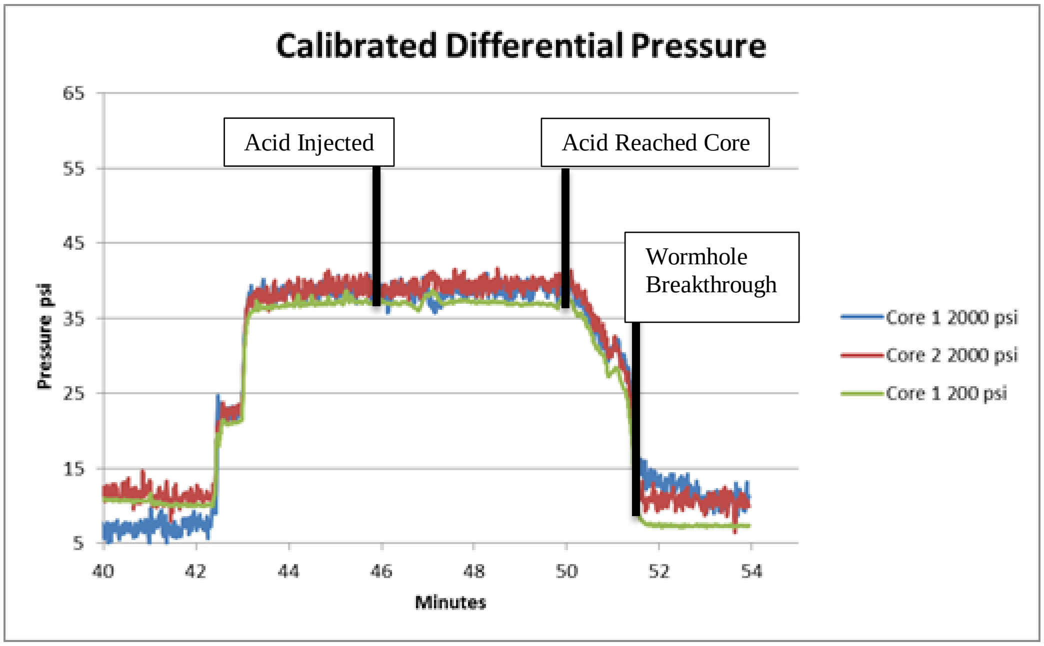


Figure 95: Second Parallel Core Flood with Calibrated Pressure Transducers

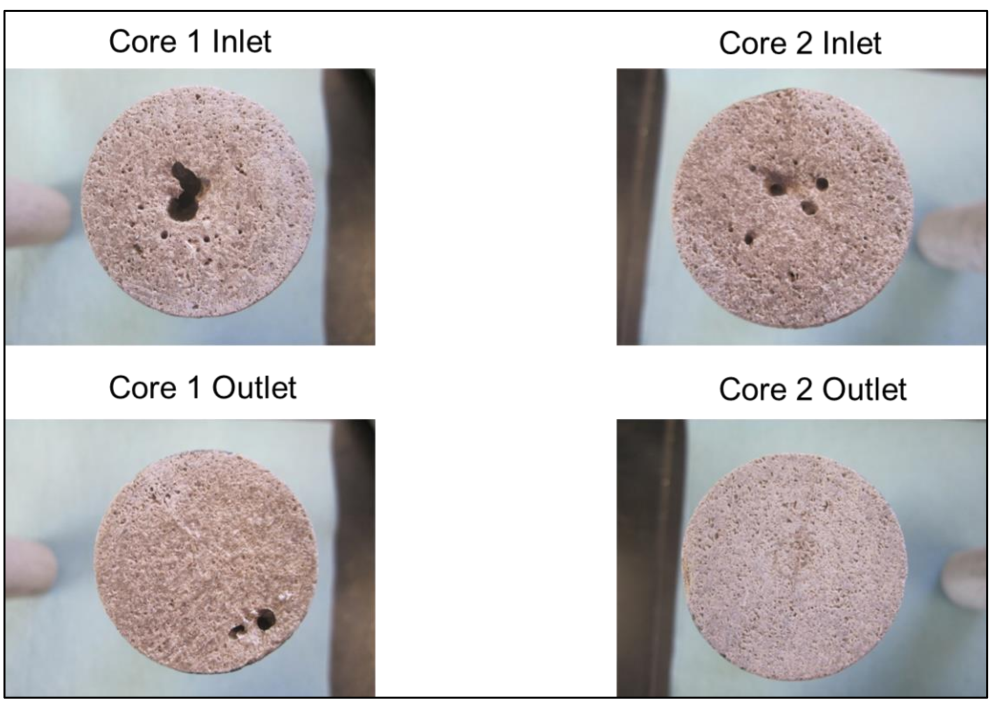


Figure 96: Indiana Limestone Parallel Core Flood Pictures

As shown in **Figure 96** a wormhole was created in core one. Core two had slight propagation but no breakthrough.

7.7 Qatar Core Samples

Three field cores of 1 inch diameter by 3.75 inch length were tested using the small core holder. The field cores had oil in place so the porosity data was calculated based on dry weight estimations. 1,500 psi back pressure was used during experiments. After completing the experiments, it was noted that a higher back pressure would maintain carbon dioxide in solution. The results are shown in **Figure 97** to **Figure 105**. The pore volume to break through data was verified by a Qatar Petroleum representative to be valid.

Core Flooding Data Sheet

Core#	1		Date	April 18, 2013	
Core diameter	1	inch			
Core length	3.75	inch			
Porosity Measurement			Acid Core Flooding		
Weight (Dry)	111	g	Temperature	140	°F
Weight (Sat)	118	g			
Porosity	14.5%		Acid Injection Rate	2	mL/min
Pore Volume	7	ccm cm^3	Interstitial Velocity	2.72	cm/min
Permeability Measurement					
Brine Injection Rate	10	mL/min	Volume of Pipe to Core	27.90	ccm cm^3
Pressure Differential	1608	psi			
Permeability	2.86	md	Time for Acid to Get the Core	836.9	seconds
Acid Formulation	15%	wt HCl	Time for Acid Injection	2004.0	seconds
Water	626.94	mL			
HCl	373.06	mL	Pore Volume Break Through	5.56	Pore volume

Figure 97: Single Core Flood Experiment 1

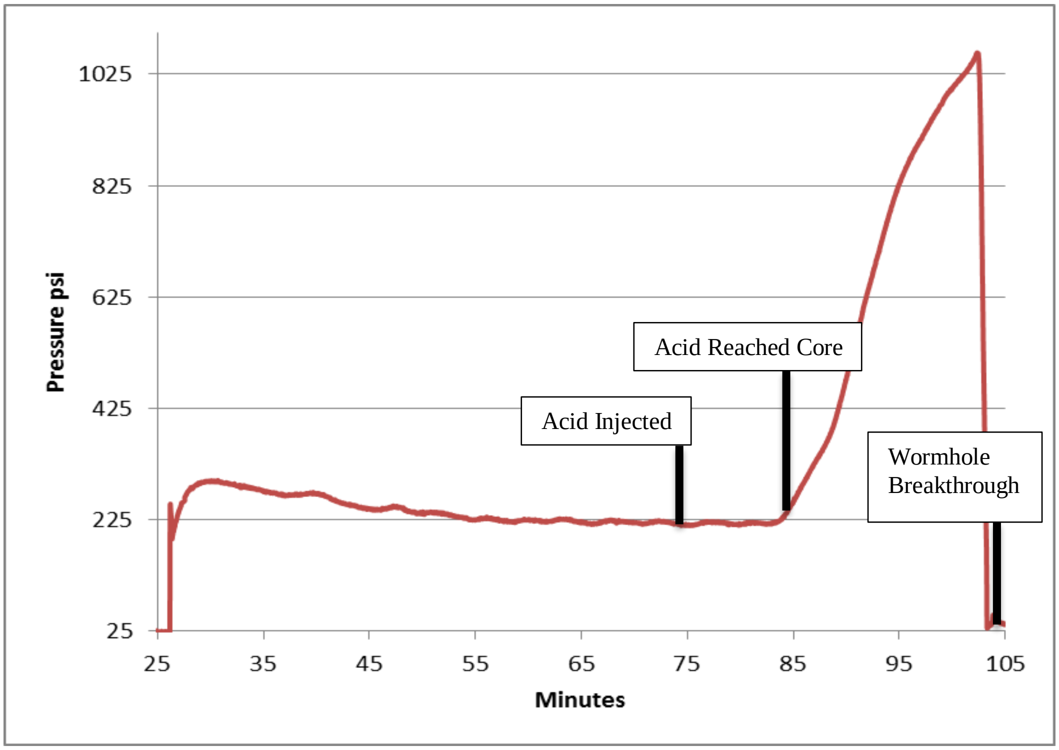


Figure 98: Single Core Flood Pressure Plot 1

Core Flooding Data Sheet

Core#	1		Date	April 15, 2013	
Core diameter	1	inch			
Core length	3.75	inch			
<u>Porosity Measurement</u>			<u>Acid Core Flooding</u>		
Weight (Dry)	110	g	Temperature	140	^oF
Weight (Sat)	118	g			
Porosity	16.6%		Acid Injection Rate	8	mL/min
Pore Volume	8	ccm			
			Interstitial Velocity	9.53	cm/min
<u>Permeability Measurement</u>	cm^3				
Brine Injection Rate	8	mL/min	Volume of Pipe to Core	27.90	ccm
Pressure Differential	450	psi			
Permeability	8.19	md	Time for Acid to Get the Core	209.2	secc cm^3
<u>Acid Formulation</u>	15%	wt HCl	Time for Acid Injection	546.0	seconds
Water	626.94	mL			
HCl	373.06	mL	Pore Volume Break Through	5.61	Pore volume

Figure 99: Single Core Flood Experiment 2

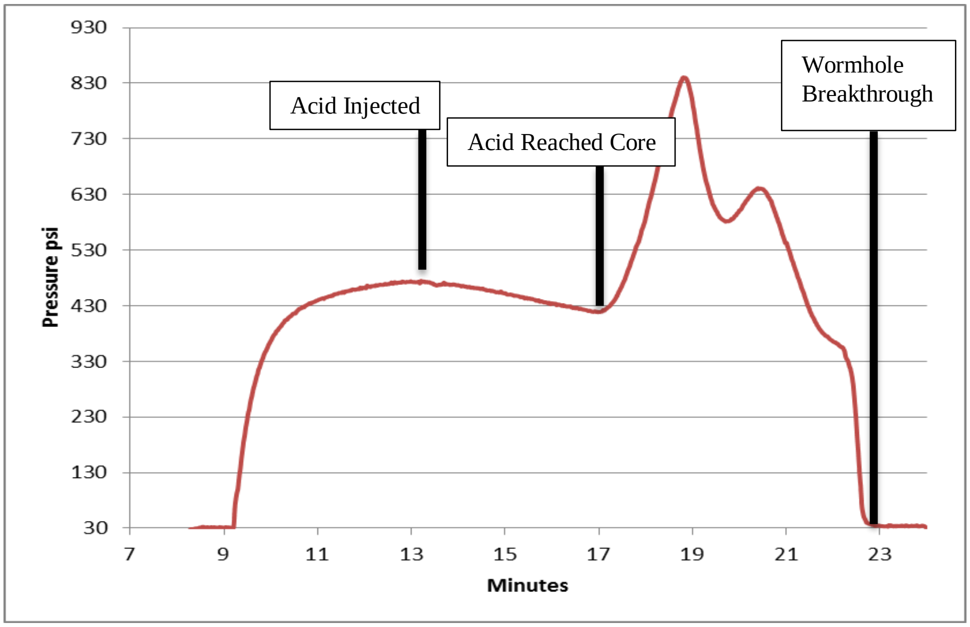


Figure 100: Single Core Flood Pressure Plot 2

Core Flooding Data Sheet

Core#	1	Date	April 15, 2013
Core diameter	1 inch		
Core length	3.75 inch		
Porosity Measurement		Acid Core Flooding	
Weight (Dry)	95 g	Temperature	140 °F
Weight (Sat)	113 g		
Porosity	37.3%	Acid Injection Rate	20 mL/min
Pore Volume	18 cm^3		
		Interstitial Velocity	10.58 cm/min
Permeability Measurement			
Brine Injection Rate	20 mL/min	Volume of Pipe to Core	27.9(cm^3
Pressure Differential	40 psi		
Permeability	230.22 md	Time for Acid to Get the Core	83.7 seconds
Acid Formulation	15% wt HCl	Time for Acid Injection	300.0 seconds
Water	626.94 mL		
HCl	373.06 mL	Pore Volume Break Through	4.01 Pore volume

Figure 101: Single Core Flood Experiment 3

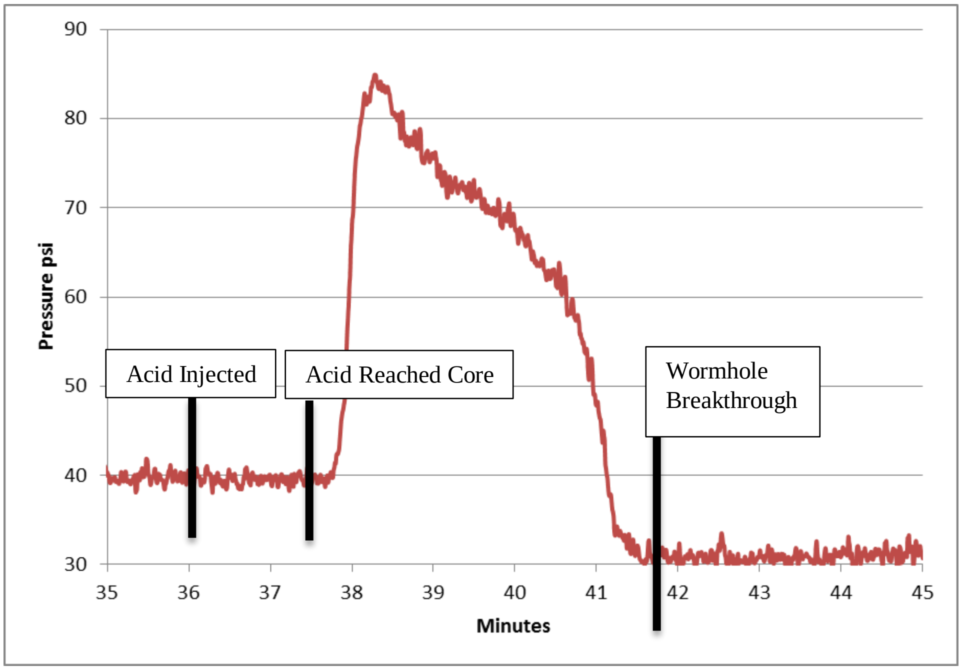


Figure 102: Single Core Flood Pressure Plot 3

Figure 103: Core 1 Inlet, Outlet, and Side View

Figure 104: Core 2 Inlet, Outlet, and Side View

Figure 105: Core 3 Inlet, Outlet, and Side View

8. CONCLUSION

The objective of this research to design, build, and test a parallel core flooding apparatus for matrix acidizing was fulfilled. The design uses 36 valves to configure small, medium, and large core holders. The key feature is the ability to run parallel core floods at high pressure and high temperature while providing resistance against corrosion from acid. Every flow line on the apparatus is sealed and acid is contained in closed flasks to provide a longer lifespan. With a narrow frame design, the apparatus is readily portable. Electrical components were enclosed and wiring was shielded. The heating system was designed to maintain temperature. The pressure transducers were appropriately calibrated. A LabVIEW VI file was written with proper function and calibration. A transformer was provided to allow the apparatus to function in Qatar. The apparatus was tested and proved to have repeatability with accurate calculations. Qatar field cores were tested and shipment plans were secured for the apparatus to be transported to Qatar.

Ultimately, the life of the apparatus depends on the user. It is highly recommended to have two users during experiments and follow procedure. Valves and core holders were placed in user friendly locations and a parts list was provided. Vendor contact information and troubleshooting guides were also provided. The procedures were written for pre-experimental setup, single core floods, parallel core floods, and for the data analysis of results. Finally, training was provided to a representative to continue experiments.

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