DESIGN, SET-UP, AND TESTING OF A MATRIX ACIDIZING APPARATUS

A Thesis

by

JAVIER NEVITO GOMEZ

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

MASTER OF SCIENCE

August 2006

Major Subject: Petroleum Engineering

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Approved by:

Chair of Committee,	A. Daniel Hill
Committee Members,	Ding Zhu
	Zhengdong Cheng
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ABSTRACT

Design, Set-Up, and Testing of a Matrix Acidizing Apparatus. (August 2006)
Javier Nevito Gomez, B.Sc., Universidad Surcolombiana
Chair of Advisory Committee: Dr. A. Daniel Hill

Well stimulation techniques are applied on a regular basis to enhance productivity and maximize recovery in oil and gas wells. Among these techniques, matrix acidizing is probably the most widely performed job because of its relative low cost, compared to hydraulic fracturing, and suitability to both generate extra production capacity and to restore original productivity in damaged wells. The acidizing process leads to increased economic reserves, improving the ultimate recovery in both sandstone and carbonate reservoirs.

Matrix acidizing consists of injecting an acid solution into the formation, at a pressure below the fracture pressure to dissolve some of the minerals present in the rock with the primary objective of removing damage near the wellbore, hence restoring the natural permeability and greatly improving well productivity. Reservoir heterogeneity plays a significant role in the success of acidizing treatments because of its influence on damage removal mechanisms, and is strongly related to dissolution pattern of the matrix. The standard acid treatments are HCl mixtures to dissolve carbonate minerals and HCl-HF formulations to attack those plugging minerals, mainly silicates (clays and feldspars).

A matrix acidizing apparatus for conducting linear core flooding was built and the operational procedure for safe, easy, and comprehensive use of the equipment was detailed. It was capable of reproducing different conditions regarding flow rate, pressure, and temperature. Extensive preliminary experiments were carried out on core samples of both Berea sandstone and Cream Chalk carbonate to evaluate the effect of rock heterogeneities and treatment conditions on acidizing mechanisms.

The results obtained from the experiments showed that the temperature activates the reaction rate of HF-HCl acid mixtures in sandstone acidizing. The use of higher concentrations of HF, particularly at high temperatures, may cause deconsolidation of the matrix adversely affecting the final stimulation results. It was also seen that the higher the flow rate the better the permeability response, until certain optimal flow rates are reached which appears to be 30 ml/min for Berea sandstone. Highly permeable and macroscopic channels were created when acidizing limestone cores with HCl 15%. In carbonate rocks, there is an optimum acid injection rate at which the dominant wormhole system is formed.

DEDICATION

To my wife Elizabeth and my children, Ana María and Luis Guillermo, the source of my inspiration.

ACKNOWLEDGMENTS

I would like to thank my advisors Dr. A. Daniel Hill and Dr. Ding Zhu who guided me throughout the course of this research with extraordinary kindness and patience.

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CHAPTER I

INTRODUCTION

1.1 Matrix acidizing fundamentals

Matrix acidizing is one of the oldest well-stimulation techniques used to remove damage near the wellbore. It was initially applied in carbonate reservoirs and over the years it has been extended to more complex mineralogies. So far, matrix acidizing is often considered for many people as risky to undertake due primarily to heterogeneous nature of formation minerals and an appreciable degree of unpredictability of their response to acid formulations[1]; however, it is a relatively simple stimulation technique that has became one of the most cost-effective method to improve significantly the well productivity and hence the hydrocarbons recovery.

During matrix acidizing treatments the acid is injected at pressures below the parting pressure, to avoid fractures being created during the treatments, searching for improving the permeability in the vicinity of the wellbore rather than affecting the reservoir to a great extent; the acid reacts within a few inches form wellbore in sandstones and a few feet in carbonates[2]. In sandstone formations, matrix acidizing may enhance significantly the well performance by removing the near wellbore damage, primarily associated with plugging of pores by siliceous particles as the consequence of drilling, completion, stimulation, perforating, and production operations; therefore their natural permeability can be restored.

Hydrofluoric acid (HF) is the only common acid that dissolves clay, feldspar and quartz fines. For years mixtures of HF and HCl have been the standard acidizing treatment to dissolve the minerals that cause damage. These treatments are preceded by a preflush of either HCl varying between 7.5% to 15% or weaker acids such acetic

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This thesis follows the style of *SPE Journal*.

acid to dissolve carbonates and avoid precipitation of calcium fluoride[3]. The main readily HCl-soluble minerals are calcite, dolomite, and siderite which additionally do not generate precipitates. The reactions are:

Calcite 2HCl + $CaCO_3$ ------- $CaCl_2$ + H_2O + CO_2

Dolomite 4HCl + $CaMg(CO_3)_2$ ------- $CaCl_2$ + $MgCl_2$ + $2H_2O$ +$2CO_2$

Siderite 2HCl + $FeCO_3$ ------- $FeCl_2$ + H_2O + CO_2

Quartz, clay, and feldspars are the main siliceous particles involved in damage of sandstones. The primary chemical reactions in sandstone acidizing are:

Quartz SiO_2 + 4HF ------- SiF_4 (silicon tetrafluoride) + $2H_2O$
SiF_4 + 2HF ------- H_2SiF_6 (fluosilicic acid)

Clays (kaolinite) $Al_4Si_4O_{10}(OH)_8$ + 24HF + $4H^+$------- $4AlF_2$ + $4SiF_4$ + $18H_2O$
(montmorillonite) $Al_4Si_8O_{20}(OH)_4$ + 40HF + $4H^+$----- $4AlF_2$ + $8SiF_4$ + $24H_2O$

Feldspars (Mg, Na or K) $KAlSi_3O_8$ + 14HF + $2H^+$------ K^+ + AlF_2 + $3SiF_4$ + $8H_2O$

The major issues that determine design of matrix acid treatments are the reservoir characterization which comprises the understanding of formation mineralogy, permeability, porosity, and reservoir fluid dynamics. Formation damage assessment in turn includes laboratory analysis, fluid compatibility and core testing[4]. As a general basis, the treatments normally follow this procedure:

- Preflush, (HCl or Hac)
- Main acid (HF) stage
- Postflush stage
- Diverter stage
- Repeat previous stages (as necessary)
- Final flush

The main acid stage requires the greatest emphasis because of the damage mechanisms, directly associated to precipitation of products from the HF reactions. Secondary reactions may occur between fluosilicic acid H_2SiF_6, a byproduct of primary reaction, and aluminum-silicates, clays and feldspars. These reactions are considered to have adverse effects since silicon can be precipitated as hydrated silica, which contributes to damage if mobile even though the presence of HCl to reduce the pH to prevent silica and flousilicate precipitation. Conversely, some authors believe these reactions are beneficial because they retard HF reactions allowing deeper penetration. Additionally, HF dissolves native clays and feldspars, and when reacting with quartz may also cause formation deconsolidation by weakening the matrix.

Precipitation will always take place, associated with HF concentration among other things; however, it tends to be more severe if HF acid treatments are not properly displaced. Acid stimulation techniques have to account for both chemistry and treatment execution to accurately predict the effectiveness since the effect of these precipitates could be minimized if they are deposited far from the wellbore[4, 5]. Careful selection of mixtures, additives, acids formulations, and treatment volumes must be accounted to minimize these secondary adverse effects.

Reservoir geology and mineralogy are the relevant issues for successfully removing the acid-soluble particles present in reservoirs; removal mechanisms are strongly related to dissolution pattern of the matrix. Recent studies have shown that one of the most important factors that determines the etching pattern is the heterogeneity of the rock. In sandstones, the variations in permeability, porosity, and mineralogy may drive the acid to follow certain paths, the highly permeable channels called *wormholes*[6]. There is experimental evidence that wormhole dissolution patterns can be achieved particularly using high HF concentrations and elevated temperatures [4, 7]; however, the risk of precipitation and rock deconsolidation might significantly be increased.

Sandstone matrix acidizing can not be considered as an exact and predictable set of rules; thus, the appropriate design of treatments almost never has only one right answer. That is an inherent problem of the complex and heterogeneous nature of most sandstone matrices. The interactions between different minerals and the injected acid depend on the chemistry, as well as temperature, pressure, pore-size distribution, surface morphology, and pore- fluid composition[1].

1.2 Literature review and history of matrix acidizing

Acidizing may be considered as the oldest well stimulation technique, hydraulic fracturing has more recent development. The earliest acid jobs were performed by 1890 to stimulate limestone formations by using HCl. In the 1930's acidizing emerged as a suitable method for scale removal and the corrosion inhibitors were also developed.

As a direct consequence of successfully applications of acid in limestone formations, interest in sandstone treatments began growing. In 1933, Jesse Rusell Wilson patented the application of hydrofluoric acid for treating sandstone formations calling for HF generation either in the wellbore or in the formation to avoid the risk in handling this "extremely dangerous material". The most remarkable aspect of Wilson's work is its awareness of the damage caused by acid soluble solids plugging.

In the same year Halliburton conducted the first sandstone treatment using a mixture of hydrochloric and hydrofluoric acid with discouraging results of high sand production; therefore Halliburton did not offer this treatment during the next 20 years.

In late 1930's Dowell (Dow Well Service) introduced the "famous" mixture of 12% HCl and 3% HF, called "Regular Strength Mud Acid", whose main objective was initially the removal of the drilling mud filter cake from the wellbore[8]. In 1984 McLeod presented the basic guidelines for proper designing of acid treatments based on formation mineralogy, an important issue which is often overlooked. These guidelines are presented in Table 1.1.

Table 1. 1 Original McLeod's sandstone acidizing use guidelines

Formation	Main acid	Preflush
Solubility in HCl >20%	Use HCl only	
High Permeability (>100 mD)		
High quartz (> 80%); low clay (<5%)	12% HCl-3% HF	15% HCl
High feldspar (>20%)	13.5% HCl-1.5% HF	15% HCl
High clay (>10%)	6.5 HCl-1% HF	Sequestered 5% HCl
High iron chlorite clay	3% HCl-0.5% HF	Sequestered 5% HCl
Low Permeability (10 mD or less)		
Low clay (<5%)	6% HCl-1.5% HF	7.5% HCl or 10% Hac
High chlorite	3% HCl-0.5% HF	5% Hac

These guidelines have been modified since its introduction to fill certain gaps and must be considered as a starting point in treatment design. Kalfayan[8] presented a modification of the basic guidelines for conventional sandstone acidizing based on successive modification from McLeod's work and his own experience, as summarized in Table 1.2.

In 1999 Da Motta *et al*[3] presented evidence of successful application of fluosilicic acid to effectively stimulate sandstone in two injector wells in off-shore Brazil. They called to account for the relevant role of secondary reaction of H_2SiF_6 with clays to remove formation damage, emphasizing the fact that comparing with conventional HF treatments, the lower cost of fluosilicic acid as a by-product of sodium fluoride makes it a viable option to routine acidizing treatments.

Table 1. 2 Modified use guidelines from Kalfayan

Formation	Main acid	Preflush
Solubility in HCl > 15-20%	*Avoid use of HF, if possible*	
Calcite or dolomite	15% HCl only(1)	5% NH_4Cl
High iron carbonate (siderite, Ankerite)	15% HCl + iron control(1,2)	5% NH_4Cl + 3% Acetic
High permeability (> 100 mD) (3,4)		
High quartz (>80%); low clay (<5%)	12% HCL-3% HF	15% HCl
Mod. Clay (5-8%); low feldspar (<10%)	7.5 HCl, 1.5% HF	10% HCl
High clay (>10%)	6.5 HCl-1% HF	5-10% HCl
High feldspar (>15%)	13.5% HCL-1.5% HF	15% HCl
High feldspar (>15%) and clay (>10%)	9% HCL-1% HF	10% HCl
High iron chlorite clay (>8%)	3% HCl-0.5% HF or,	5% HCl
	10% acetic- 0.5% HF	10% acetic + 5% NH_4Cl
Medium permeability (10-100 mD) (3,4)		
High clay (>5-7%)	6% HCl- 1% HF	10% HCl
Lower clay (<5-7%)	9% HCl- 1% HF	10% HCl
High feldspar (>10-15%)	12% HCl-1.5% HF	10-15% HCl
High feldspar (>10-15%) and clay (>10%)	9% HCl-15 HF	10% HCl
High iron chlorite clay (> 8%)	3% HCl-0.5% HF or,	5% HCl
	10% acetic- 0.5% HF	10% acetic + 5% NH_4Cl
High iron carbonate (>5-7%)	9% HCl-1% HF	5% HCl
K< 25 mD	5% HCl-0.5% HF	10% HCl
Low permeability (1-10 mD) (3,4,5)		
Low clay (<5%); low HCl sol. (<10%)	6% HCl- 1.5% HF	5% HCl
High clay (>8-10%)	3% HCl- 0.5% HF	5% HCl
High feldspar (>10	9% HCl-1% HF	10%HCl
High iron chlorite clay (> 5%)	10% acetic-0.5% HF or,	10% acetic + 5% NH_4Cl
Very low permeability (<1 mD)	Avoid HF acidizing; non-HF matrix stimulation (dictated by damage) or hydraulic fracturing is preferred.	

Table 1. 2 Continued

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(1) Location of carbonate in matrix is important; it may be possible to include HF in naturally fractured formations with high carbonate content.

(2) HCl can be replaced by acetic or formic acid- partially or completely-especially at higher temperatures (250-300 °F).

(3) If zeolites (analcime) are present (> 3%), consider replacing HCl with 10% citric acid or special service company organic acids.

(4) For higher temperatures (> 225-250 °F), consider replacing HCl with acetic or formic acid.

(5) Although fracturing may be preferable, low permeability, low clay containing sands may respond favorable to HF acidizing-contrary to conventional wisdom.

In 2000, Kalfayan *et al.*[4] presented results of a job using high HF concentration up to 6% at 265 °F, achieving permeability increases as high as 200 %. Previous experimentation on cores showed the formation of larger channels although the integrity of the rock was reduced in a 40%. In 2002, Thomas *et al*[7] presented their results on core flood testing at temperatures as high as 300 °F; they observed channeling depending upon the type of preflush, HF concentration, core permeability, and heterogeneity of the core. These channels were enhanced with HCl instead of acetic acid, resulting in a positive impact on the field production.

More recently, some authors have called for the necessity of using non HF-based systems because of the nature of damaging potential inherent in the reactions between sandstone minerals and HF[9]. They insist in the capability of these new systems to stimulate effectively, specially those formations with high content of HCl soluble minerals.

1.3 Objective of the research work

The present research project aim to design and set up an apparatus to carry out matrix acid displacements in both, sandstone and carbonate cores, to determine the formation damage removal, permeability improvement, and minerals dissolution in the former by the acidizing processes, as well as evaluate the matrix acidizing behavior, the effect of rock heterogeneity, and to determine the optimal injection rates to create the acid created channels in the latter.

Conduct extensive experiments in both, sandstone and carbonate cores, to validate the operative suitability and reliability of the apparatus under a broad range of conditions, flow rate, temperature, volume pumped, and acid concentration. The new lab setup must reproduce as much as possible the characteristics of the treatments in field conditions to investigate the effects of matrix acidizing on diverse types of rock; therefore the results can be likewise escalated.

The new apparatus has been designed to be safe and easily operable, with the purpose of displacing diverse types of acid at different conditions; the operation procedure will be detailed. The wetted parts are made of Hastelloy C-276, an special alloy to prevent the attack of acid; three core holder will be available for different sizes of cores, two syringe precision pumps allow the wide range in flow rates (from 0.2 up to 204 cm^3/min) at high pressures (up to 3500 psi).

The experimental measured data are to be processed and interpreted; detailed procedures are to be developed for each experiment stage. The equipments and instruments specifications are to be listed. The major lab setups are illustrated and the future work about this project is discussed.

The complete test of matrix acidizing comprises the following steps:

- The core sample preparation, which includes sandstone and carbonate cutting at different sizes and further vacuum saturation with brine.
- Determination of initial porosity and permeability of the cores by using the helium porosimeter and the air permeameter.

- If possible, analyze the samples with CT-Scan equipment to determine their degree of heterogeneities and initial pore distribution.
- Brine and acid preparation, preflush and postflush, and injection through the core samples to conduct the experiment.
- Record the pressure data from the test, generation, and analysis of acid response curve of the specific sample to be analyzed.

CHAPTER II

DESIGN OF A MATRIX ACIDIZING SETUP

In this chapter we shall determine the components and specifications necessary to complete the proposed apparatus, which must be capable to deal with the required conditions for experimentation on different size core samples at flow rates and temperatures similar to those to be encountered at field conditions. Figure 2.1 is the schematic of the apparatus.

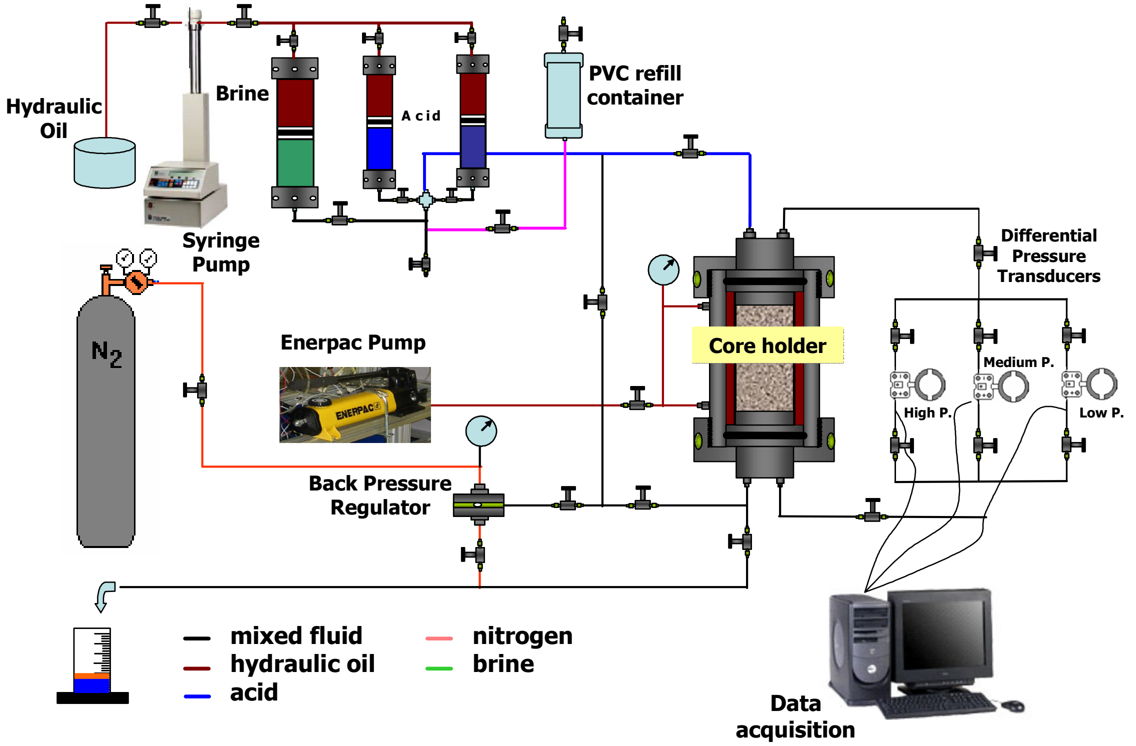


Figure 2. 1 Schematic of matrix acidizing laboratory set-up

As illustrated in fig. 2.1, brine and acid previously prepared and stored in the accumulators, are pumped and heated up, according to the test procedure and particular conditions, via the tubing network and flows through the cores samples within the core holder. The core sample is confined inside the core holder with a Viton sleeve by a hydraulic pump. The spent acid is gathered and measured in the beaker at the end of the process. The back pressure provided for nitrogen supply via the teflon diaphragm BPR maintain the CO_2 in solution.

During the matrix acidizing experiments, the flow rate, temperature, confining pressure (overburden), and back pressure are set and maintained constant; the differential pressure across the core during the process is measured by analog pressure transducers and recorded by the LABVIEW software to construct and analyze the acid response curve. All the equipment reside inside a laboratory exhaust system to vent the acid fume.

2.1 System requirements

The main parts considered essential for the purposes of the set up are the following:

- Pumps capable to displace at a broad range of flow rates and suitable to work on either constant flow rate or constant pressure modes.
- Core holders, special alloy made to prevent acid corrosion for a long time and high pressure and temperature resistant.
- Accumulators to store the acid mixtures required during the different tests at different concentrations.
- Heaters and temperature controllers to increase temperature up to desired levels and as occurring at field conditions.
- Back pressure regulator to provide the necessary pressure against the flow maintaining the CO_2, product of the acid reactions, in solution.

- Data acquisition system for monitoring and getting the data generated in the acidizing process for further analysis.
- Flow system to serve as a network for the different fluids to be used in the experimentation, oil, brine, and acids.
- Hydraulic pump to provide the necessary confining pressure to the core within the core holder.

2.2 System components

2.2.1 Pumps

In matrix acidizing experimentation, a versatile pumping system is required depending on the particularities of each type of rock to be treated and the desired conditions to affect the acidizing process. Among these conditions one of the most important is the flow rate at which the fluids are to be displaced; in the majority of the experiments this flow rate must be constant during the process to accurately determine the pressure changes in the rock while the fluids are passing through the core.

After visiting the laboratories of two majors services companies, BJ Services and Schlumberger, we defined that the best option for our application is the Teledyne ISCO 500D; see figure 2.2. This pump is a syringe precision type which makes it the ideal equipment for a wide range of chemical feed applications requiring flow rates up to 200 ml/min at pressures up to 3,750 psig; its 500 ml cylinder capacity allows delivering a precise 1 ml/minute for over 8 hours on a single fill.

The ISCO D500 pump uses a "Smart Key" controller that can operate any three pump modules, either independently or together. Operating modes include either single or dual pump independent constant flow or pressure and it additionally comes with a RS-

232 serial interface for computer control or monitoring of operating parameters using commercial LabVIEW software.

Figure 2. 2 Teledyne ISCO D500 precision syringe pump

The specifications of the equipment are:

- Capacity: 507 ml
- Flow range (ml/min): 0. 001 - 204
- Flow accuracy: 0.5% of set point
- Displacement resolution: 31.7 ml
- Motor stability: ± 0.001% per year
- Pressure range (psi): 0- 3,750
- Standard pressure accuracy: 0.5% FS
- Optional pressure accuracy: 0.1% FS
- Wetted materials (standard): Nitronic 50, PTFE, Hastelloy C-276

Plumbing ports:	1/8" NPT
Operating temperature:	0 - 40° C Ambient
Power required:	100 Vac, 117 Vac, 234 Vac, 50/60 Hz
Dimensions (HxWxD, cm):	103 x 27 x 45
Weight:	Pump module - 33 kg; controller - 3 kg

The set-up has two pumps installed in parallel which use hydraulic oil as a driving fluid to avoid direct contact with extremely corrosive conditions during testing. Inlet and outlet ports, that constitute the refill and dispense lines, are 1/8" NPT female threads connected to 1/8” stainless steel tubing forming the discharge manifold which in turn are connected using “gyrolok” type of compression fittings.

The cylinder of each pump must be filled first before any use by selecting REFILL mode in the controller; discharge and refill lines are independent and the valves in the manifold are to be operated carefully during any operation being done. Once the cylinder is empty after running the pump through a test, close the discharge valve and open the refill valve; select the pump in the controller and push the refill button. It is recommended refill at a flow rate of 30 to 40 ml/min. The refilling process may be performed at the same time the other pump is running. After refill is complete, close the refill valve and keeping the discharge valve closed, start the pump at 5 to 10 ml/min until reaching the discharge pressure, then open the valve and equalize the pressure.

2.2.2 Core holder

The core holder is perhaps the most important part of the system for matrix acidizing purposes; these are the cells where the core samples are confined to displace the fluids through them.

Because of the nature of fluids to be used, hydrochloric and hydrofluoric acids at different concentration and temperatures, the core holders need to be made of special alloy material. Our core holders were manufactured by Phoenix Instruments, made of Hastelloy C276, a corrosion resistant material and capable to withstand a working pressure of 3000 psi and temperatures of about 300 ^{o} F. Figure 2.3 shows the three core holders available in the matrix acidizing laboratory.

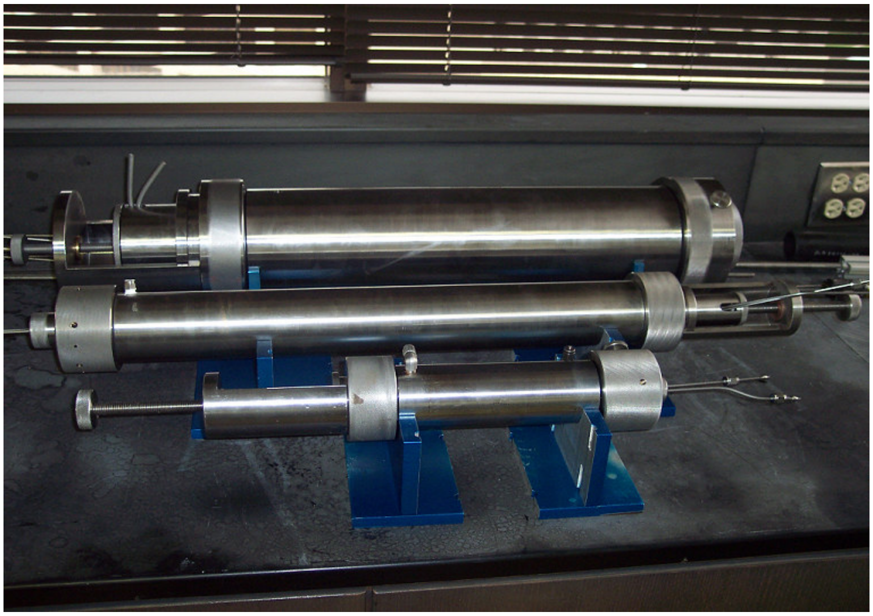


Figure 2. 3 Core holders 1", 1.5", and 4' diameter by 6" and 20" long.

Each core holder comes with the regular inlet and outlet tip with ports 1/8" and the distribution pattern to contact the total face area of the core; inside the cylinder a special rubber Viton sleeve, temperature resistant, has been adapted to confine the core once in use.

The core holders were though to allow the recirculation of mud and conduct different type of experiments on jetting acidizing; therefore additional set of tips with spacers were purchased to use in this particular application in which is necessary first to create a certain filter mud cake in the face of core sample. Figure 2.4 shows the drawings and the details of the core holders.

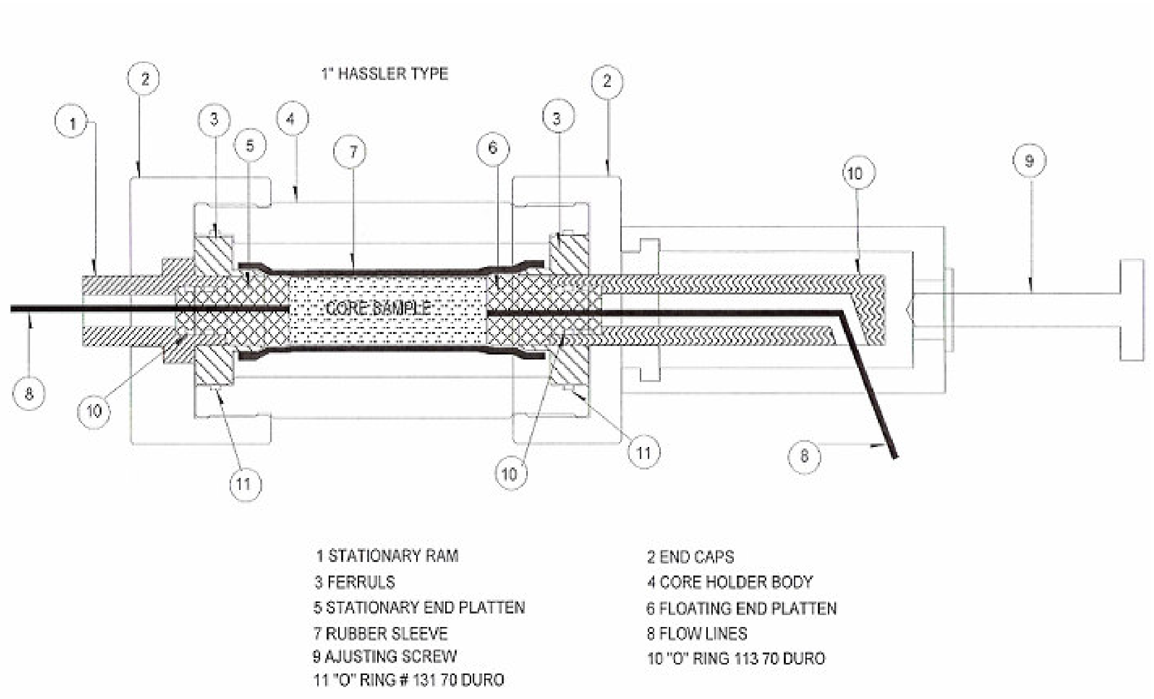


Figure 2. 4 Schematic drawing showing details of core holder

2.2.3 Accumulators

The accumulators were manufactured by Phoenix Instruments and they are type piston. These parts of the set up are the vessels where the products to be displaced through the cores during the acidizing experimentation are contained. Figure 2.5 shows the piston accumulators actually installed in the set up with their corresponding stands and entrance lines.

Three of these accumulators were designed to be corrosion resistant that means they are made of special alloy material, Hastelloy C-276, with capacities of 1000 ml and 2000 ml the largest one; there is also one accumulator stainless steel made because is tough to use for brine containment whose capacity is 1500 ml. All of the accumulators have inlet and outlet ports 1/8” NPT.

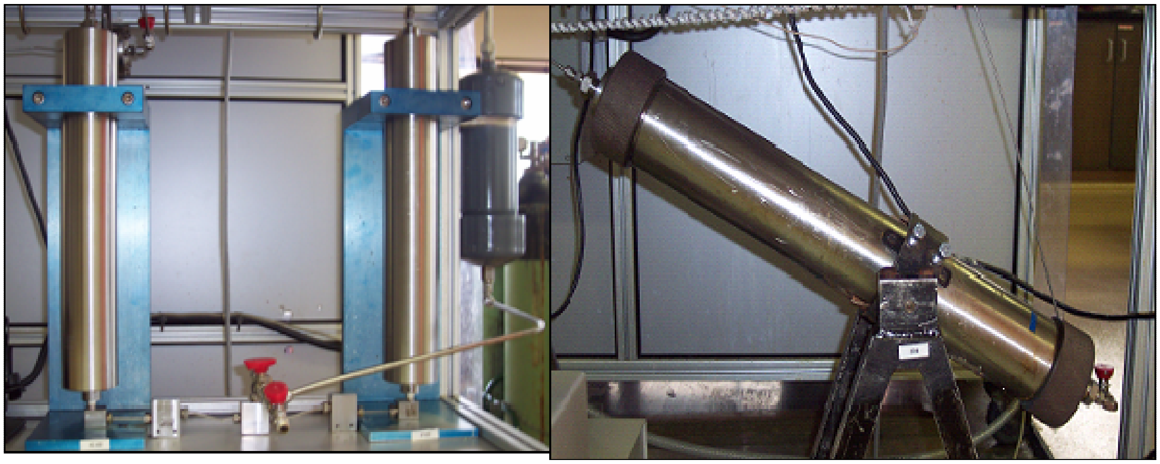


Figure 2. 5 Acid accumulators, PVC refill container, and brine accumulator

The displacement of the acid and brine is performed by the syringe pumps that pump the hydraulic oil which in turn acts on the Teflon piston pushing either the acid or brine out at necessary pressure and flow rate to carry them to the core via pipe network until reaching the core face into the core holder.

To refill the piston accumulators with either acid or brine, a PVC container is filled first with a funnel and then air at 100 psi from the laboratory air system, push the products into the accumulators to be ready for experiments. It is necessary to vent the oil line on top of the accumulator to allow either the acids or brine to be entered into the accumulators as the oil inside them is removed. This operation must be done independently for each accumulator. This procedure will be described in more detail in chapter III.

2.2.4 Heaters and temperature controller

Temperature is a relevant factor that affects the reaction rates and kinetics of chemical reactions between minerals and acids; in the case of HF with sandstone minerals the kinetics expressions can be represented by:

$$-r_{mineral} = E_f\,[1+K\,(C_{HCl})^{\beta}]\,C^{\alpha}_{\;HF} \qquad (2.1)$$

and

$$E_f = E_f^{\,o}\exp(-\Delta E/RT) \qquad (2.2)$$

It is also well known that actual reservoir temperatures are much higher than atmospheric or room temperature; therefore, temperature becomes an important aspect to be considered when simulating different conditions at which the acidizing experiments are conducted. To make the set up capable to reproduce real conditions on matrix acidizing and study the effect of temperature on mineral dissolution and wormholing patterns two different heaters were considered and installed on the apparatus.

A rope heater manufactured by OMEGA is used in the lab set-up, which is shown in Figure 2.6. This heater is capable to wrap around small diameter tubing, as small as 1/8" and for use in either conductive or non-conductive surfaces. Its maximum allowable temperature is 900 ^{o} F, the wattage is 500 w at 120 volts, and it is 10 feet long. The rope heater was installed wrapping the general flow line between the discharge of the accumulators and the inlet valve before entering the core holder.

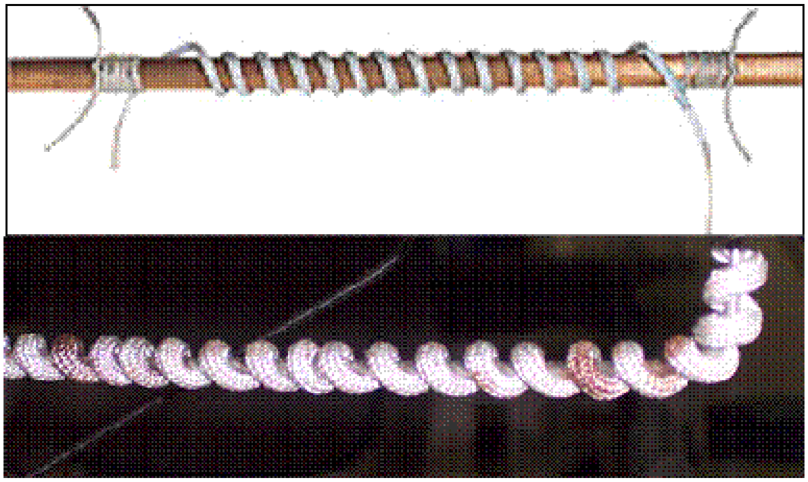


Figure 2. 6 Omegalux rope heater

Another different heater system has been in use acting directly on core holder wrapping the cylinder, it is ceramic type jacket. This device is much more powerful than rope heater one with a wattage up to 1600 at 120 volts, and capable to rise temperature also up to 900 ^{o} F, it is manufactured by Delta MFG. It can be seen in Figure 2.7.

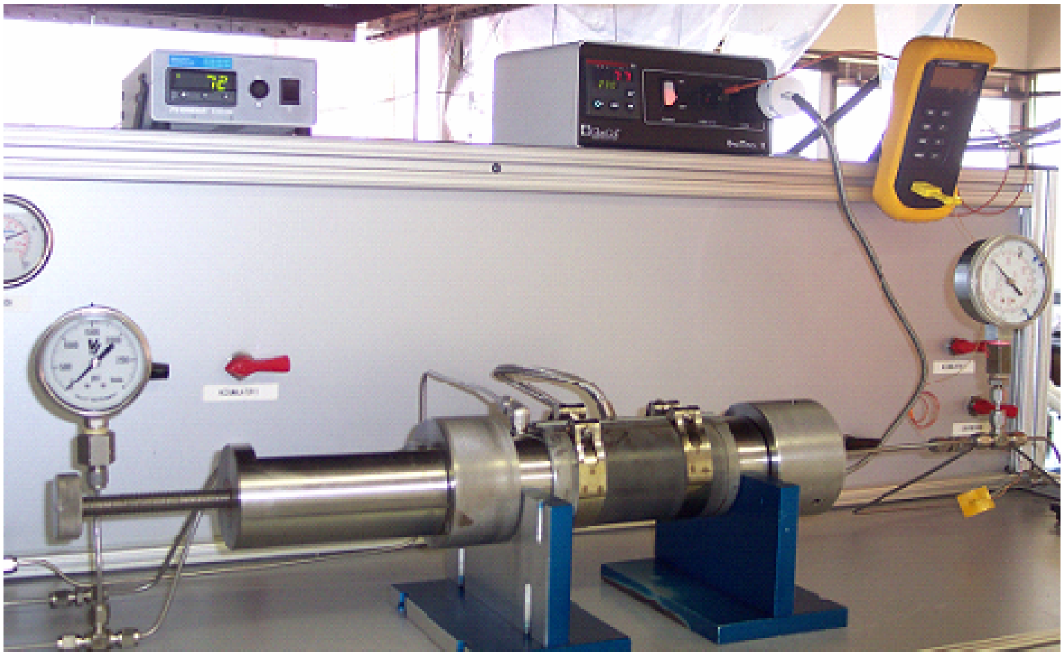


Figure 2. 7 Type jacket heater Delta MFG, wrapping the core holder

The temperature of the acidizing process must be maintained fairly constant to ensure the reliability of the results; so two controllers are then used. First the temperature of the preheated fluids coming from the accumulators is controlled by compact benchtop CSC32 series which has 4-digit display, 1 or 0.1° resolution with an accuracy of ± 0.25% full scale ± 1°C. It uses a type K thermocouple and two Outputs (5 A 120 Vac SSR); Figure 2.8 shows this type of device. The thermocouple located downstream, senses the temperature of the fluid displaying it in screen and maintain it constant.

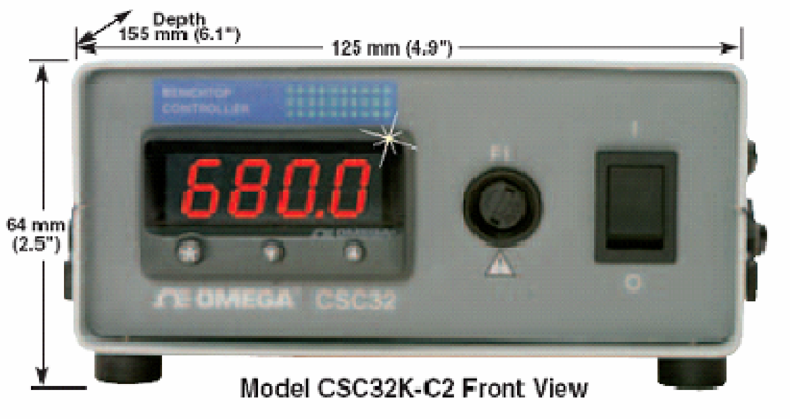


Figure 2. 8 OMEGA CSC32K-C2 benchtop controller

To control the temperature in the jacket wrapping the core holder is used a GLASCOL Digitrol II temperature controller which uses a 1/16 DIN microprocessor-based, user-selectable control mode temperature control. The control features a digital dual output display for set point and process temperature indication. The load and the thermocouple receptacles are located on the front panel; the thermocouple is type J installed upstream, so the set point is given by the temperature of the fluid coming out the core. The accuracy is 0.1% of span, it has up/down keys for set point and °F or °C can be displayed as necessary. Figure 2.9 shows the GLASCOL controller.

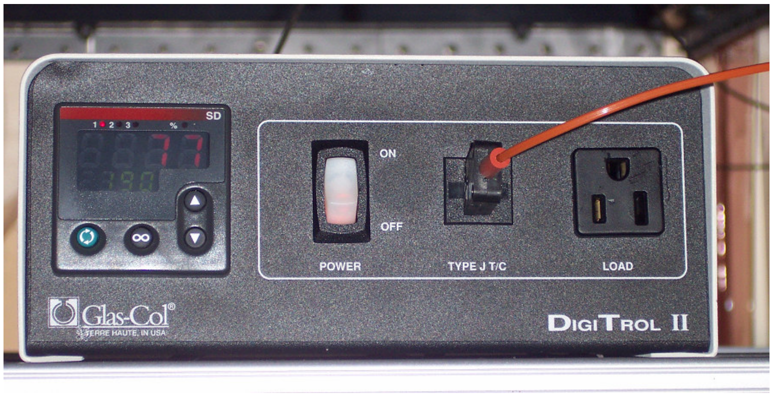


Figure 2. 9 GLASCOL Digitrol II temperature controller

These two heater rise temperature very fast and it is important never let the heater on for a long time when there is no fluid flow in the flow line, and exceeds the working temperature of the core holder, 300 ^{o} F to prevent early tubing and equipment metal failure, as well as risk of accident.

2.2.5 Back pressure regulator

Back pressure is necessary during the experiments at the core outflow to simulate down-hole conditions, as the pore pressure does in actual strata conditions, to avoid the presence of free CO_2 gas bubbles, which can lead to undesirable two-phase effects and might increase the fines migration phenomenon. The backpressure must be kept constant and it is desired to be 300 - 400 psi less than the overburden pressure.

One Mity-Mite back pressure regulator model S91-W is installed on the upstream line. The pressure in the line controls the effluent flow and exerts the resistance for the purpose described above, maintaining constant pressure upstream; the pressure is the same as the nitrogen dome pressure.

The connections to the flow lines are 1/4" NPT female threads and the connection to the charging line is 1/8" NPT female thread. In the front panel there is a gauge to control and watch the back pressure. The pressure range may vary between 100 and 2000 psi and temperature -65 ^{o} F and 200 ^{o} F; the material of body and dome is stainless steel and the diaphragm is Teflon. The maximum Cv is 0.38 and it weighs 4 lbs.

The type of BPR is externally dome loaded therefore it must be charged from an external source of gas pressure; in our case dome pressure is supplied by a nitrogen bottle with its correspondent regulators. Figure 2.10 shows the regulator as installed in the set up and figure 2.11 shows its detailed drawing.

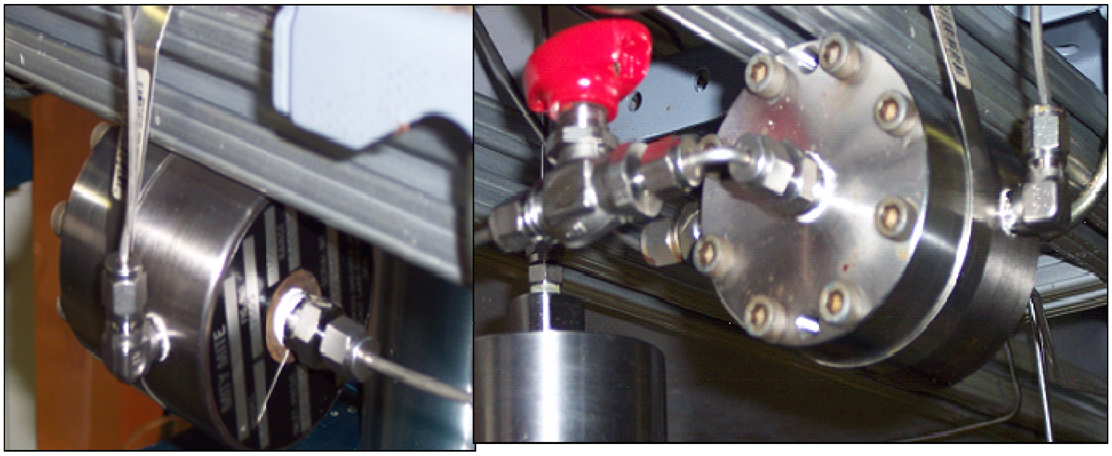


Figure 2. 10 Mity-Mite model S91-W back pressure regulator

Dome pressure acts over the exposed area of the diaphragm to seat it on the nozzle; when upstream pressure exceeds the desired level, it pushes the diaphragm up off the nozzle. The flow of fluid through the regulator relieves the pressure in the upstream system; as the upstream pressure drops off, the diaphragm moves down to reduce or shut off the flow, so that upstream line pressure is held constant.

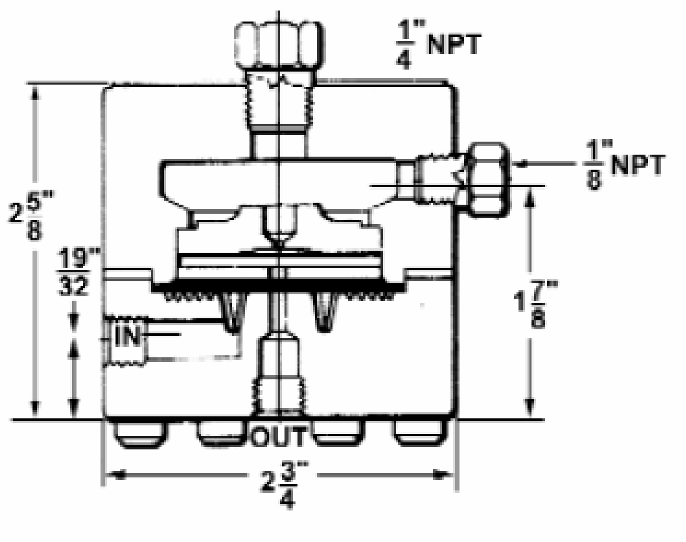


Figure 2. 11 Schematic drawing with details of Mity-Mite model S91-W BPR

2.2.6 Data acquisition

To exert a real time monitoring of behavior of the acidizing experiments, it is necessary to record all the parameters that define the process among others flow rate, temperature, and pressure drop along the core.

The flow rate is directly set and controlled by the ISCO syringe pumps controller, which can control up to three units; because the acidizing experimentation is normally carried out at constant flow rate the variable that changes as the process occurs is the discharge pump pressure, that in turn determines the pressure at the inlet face of the core.

The pressure drop is then sensed with a set of FOXBORO differential pressure gauges models, IDP10-T26(C-D-E) 21F-M2L1; which measures the difference between two pressures and transmits a proportional or square root (Flow) electrical Signal. The differential pressure gauges are energized by a 30 volts single DC power supply and display pressure data on LCD screens and output 4~20 mA DC current signals that travel to hardware via grade 16AWG electric cable.

There are three different gauges installed with ranges 0-30 psi, 0-300 psi, and 0-3000 psi; which permit to choose the most adequate one according to the expected pressure drop of each particular type of rock, related to their permeability. The sensor of these devices is made of hastelloy C276 and silicone fill fluid.

The connections of the transmitters are assembled with 1/8” hastelloy C276 tubing and Gyrolok compression fitting. Figure 2.12 illustrates the pressure transmitter setup and figure 2.13 shows the connection mode to capture the electric signals and convert to PC based data acquisition system.

The NI CB-68LP board is the device that receives the electric signal from the transducers, via the wires, and transfers the signals to the main board inside the computer; see figure 2.14.

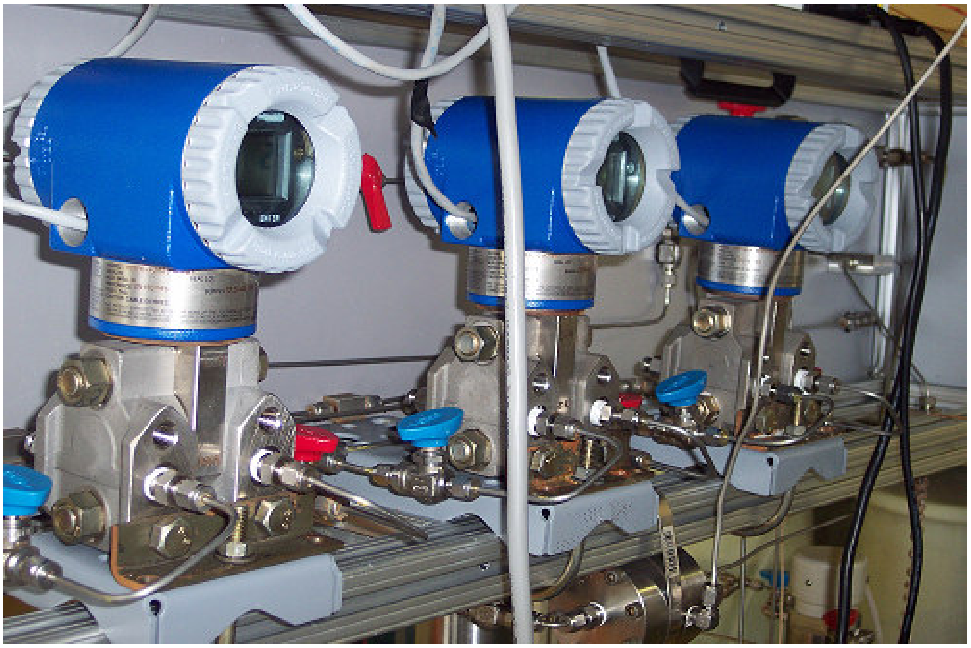


Figure 2. 12 FOXBORO pressure transducers

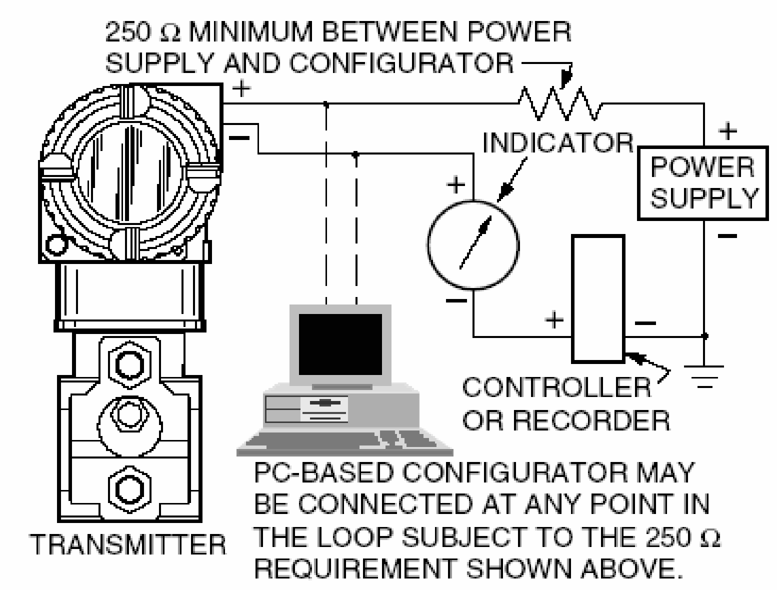


Figure 2. 13 Schematic of pressure signal acquisition

The interface board has a direct network interface to processes I/O signals up to 64 channels, eight of which are type analog. The channels entering to the board and its distribution and recognition are programmed by the software labVIEW from National Instruments.

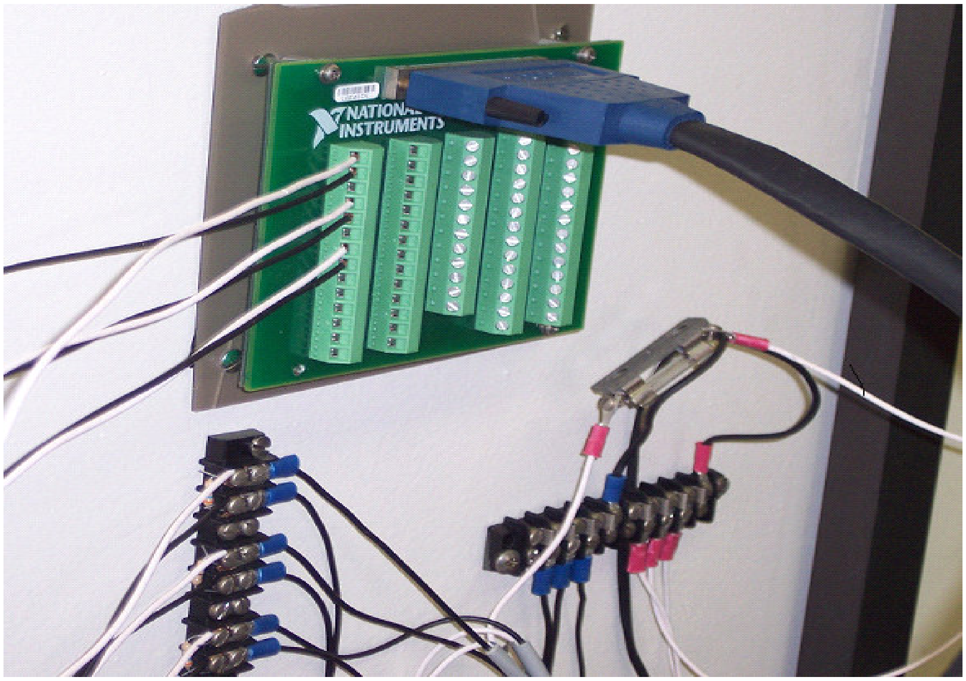
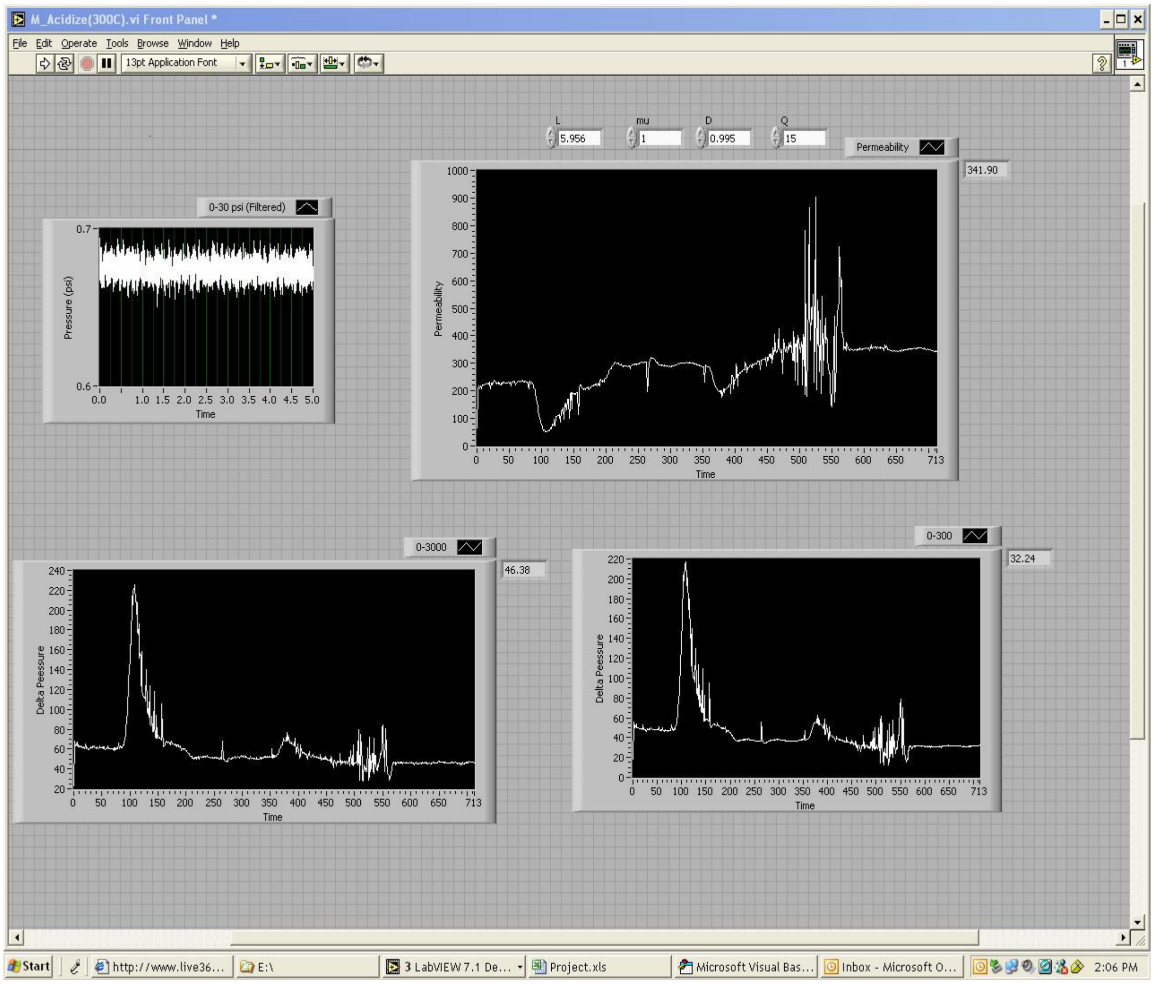


Figure 2. 14 NI Interface board CB-68LP DAQ

There are three pressure signals actually installed in the set up, which use independent analog channels in the interface board, these signals are then distributed and directed to the main board installed inside desktop. These three signals are processed by the software LabVIEW which automatically recognizes and display them on a wave chart in front panel as shown in figure 2.15.

The software works with specific commands for each tasks and it easily links the pressure signals with the workflow to calculate the variable permeability, which was previously formulated in the specific command. The pressure drop and permeability readings are finally sent to be recorded in a file in a time sequence determined as optimum depending on the duration of the test. The frequency of data acquisition must be set and normally one reading every two, five, or ten seconds is the standard.


M_Acidize(300C).vi Front Panel *

L 5.956

mu 1

D 0.995

Q 15

Permeability 341.90

0-3000 46.38

0-300 32.24

Figure 2. 15 Data acquisition LabVIEW program (front panel)

The files can be downloaded as a excel spread sheet to represent the differential pressure and permeability data. Figure 2.16 shows the block diagram of the LabVIEW with the program to acquiring and writing the data. In Appendix 2 the steps to create the program are described illustratively.

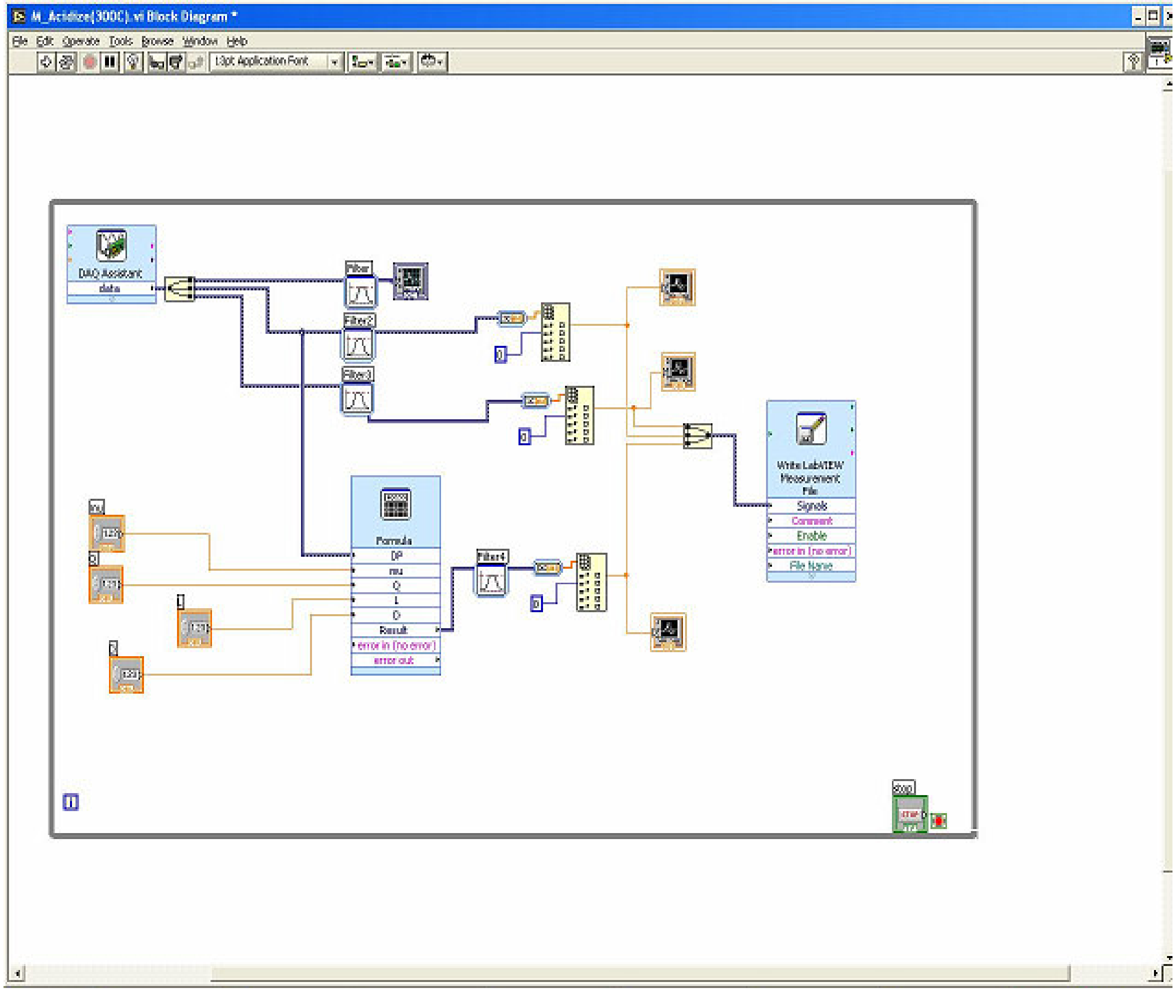


Figure 2. 16 Data acquisition LabVIEW program (block diagram)

2.2.7 Core preparation equipment and core description

Core samples used in matrix acidizing experiments are Berea sandstone and carbonates (Indiana Limestone, Dolomite, Cream Chalk). These cores are cut using the HILTI model DD200, a heavy duty and portable core driller and the three sizes of core bits required for use with the available core holders. A special frame was necessary to built to install the machine and make it more stable because the abnormal lengths of cores to be used; see figure 2.17.

Figure 2. 17 Heavy duty HILTI DD200 core driller and core bits

Core bits were specially designed for HILTI to be used in our application in a non-standard length 4” diameter by 20” long, 1.5” diameter by 20” long and the regular one 1” diameter by 6” long. Figure 2.18 shows the smallest cores obtained from Cream chalk (left), Indiana limestone (center), and Berea Sandstone (right).

The cores are saturated in the vacuum facility already existing in the Department. Vacuum bell is a large size glass vessel, in which the cores samples are submerged to completely remove air in the pore space of core samples and then saturate the cores with

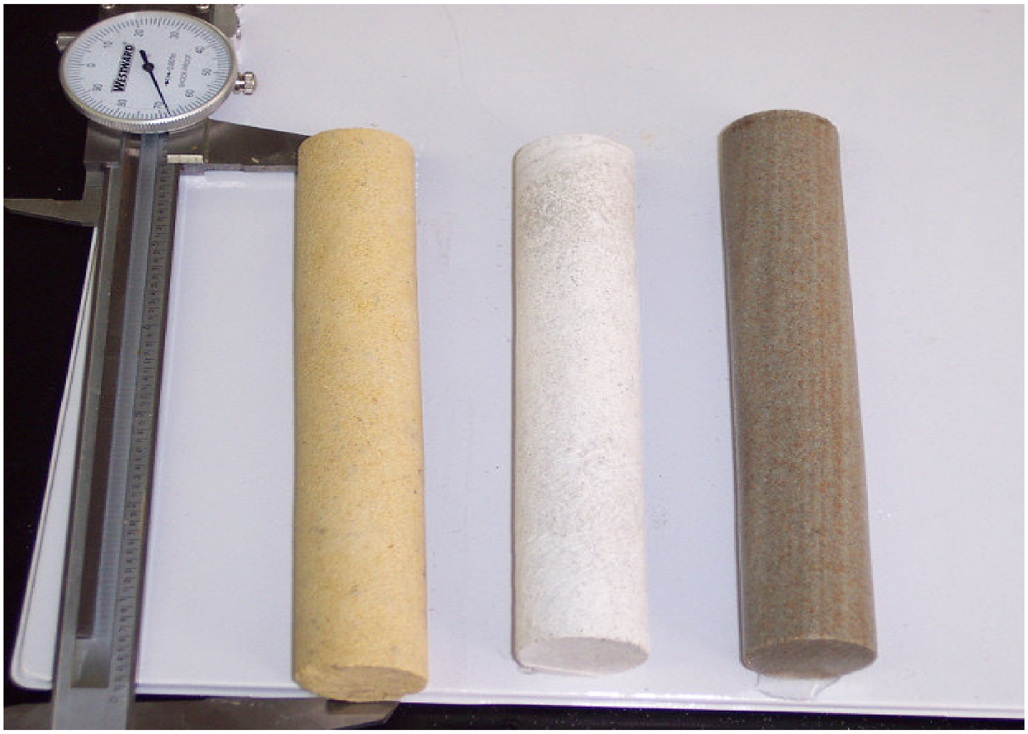


Figure 2. 18 Core samples of three different rocks

brine, ammonium chloride 3% or potassium chloride 1%. This pre-saturation process assures that the initial permeability is measured at 100% fluid saturated or absolute permeability.

2.2.8 Piping network, valves, and fittings

The tubing pipe installed in the set up to serve as flow line to displace the acids and brine during the experiments was designed to be 1/8” diameter with a wall thickness of 0.03”. Its effective inner diameter allows to flow at the maximum rates provided by the ISCO D500 pumps that is 204 ml/min; the burst pressure is greater than 7500 psi. Because the nature of the fluid to work with, this tubing is made of hastelloy C276 to resist the corrosive environment of HCl-HF mixtures. Regular stainless steel tubing 1/8” is used in tubing in which hydraulic oil is the fluid.

Two types of valves are installed in the piping network of the set up; needle valves HiP taper seal made of hastelloy, which are used for flow lines with acid; their fittings seal metal to metal as a compression fitting by a sliding sleeve fastened to the tubing. These valves are designed to withstand pressures up to 15,000 psi and temperatures of 450 ^{o} F. The ball valves are stainless steel made and bring with type gyrolock compression fittings; they are used in lines with either no dynamic flow as those for detecting pressure signals or hydraulic oil lines; they withstand up to 6000 psi and 300 ^{o} F.

2.2.9 Hydraulic pump

This pump is an ENERPAC hydraulic model P392 that provides the necessary pressure to confine the core sample inside the core holder to simulate the overburden pressure. Pressure, commonly 1,000-1200 psi, acts on the rubber Viton sleeve; which in turn contact the core sample. In any case it should be 300-400 psi greater than backpressure. Figure 2.19 shows the ENERPAC hydraulic pump.

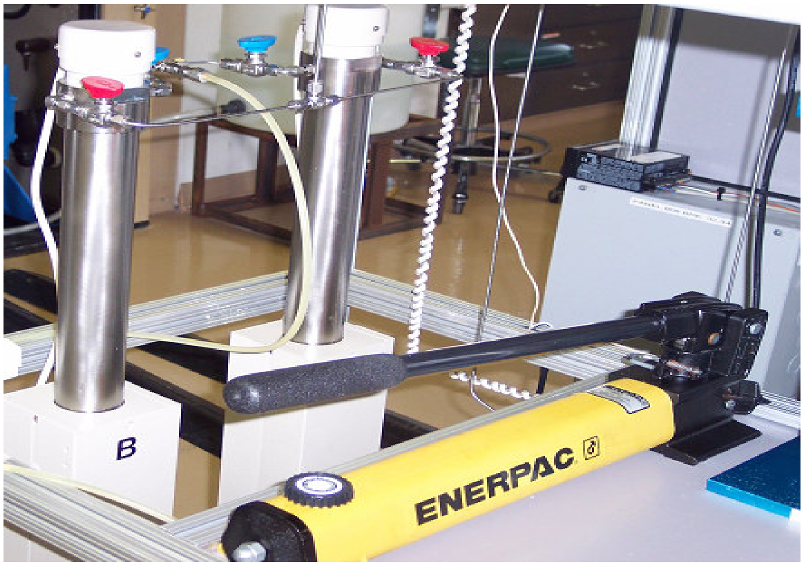


Figure 2. 19 ENERPAC model P392 hydraulic pump

CHAPTER III

EXPERIMENTAL DETAILS STUDY

3.1 Matrix acidizing experiment details

Acid core-flooding is a unique technique that mimics matrix acidizing effects on different types of rocks. Diverse phenomena associated with acid-mineral interactions during the linear core flooding can be observed and analyzed to simulate different treatment conditions and adjust available modeling to improve actual understanding on acidizing techniques.

Linear core-flood acidization in laboratory is extremely useful to understanding this complex process particularly in sandstone acidizing. Principles of flow in porous media, reaction kinetics and other phenomena reproduced by conducting linear flow experimentation of acid into the cores will allow us to provide highly quality experimental data well suited for petrophysical models, simulators or other tools that can be escalated to more complex geometries as occurring at field conditions

Core flood process begins once the core has been inserted inside the core and the confining pressure has been set at desired level. The objective of the project is to build the reliable equipment to conduct acid core-flooding at diverse conditions and to study their effect on different rocks once acidized.

Hydrochloric acid (HCl) is usually selected for carbonate acidizing; it reacts readily with carbonate minerals. HCl is used in carbonate formations to dissolve the matrix and bypass the damage. For this reason, both damage and rock characteristics must be taken into account when designing the treatment.

In carbonate rocks effective matrix stimulation rely on the formation of highly permeable channels called wormholes which are the result of flow and reaction of acids. The structure of these wormholes is strongly dependent upon the injection rate and the fluid/rock properties**[10]**. At low rates the wormhole structure result in a complete dissolution from the inlet face; while at high injection rates uniform dissolution and ramified wormholes can result, and at intermediate flow rates single a dominant wormhole are obtained.

The purpose of sandstone acidizing is to dissolve the damage; it is distinguished from carbonate acidizing in that it involves the dissolution of damage that is blocking or bridging the pore throats in the formation matrix, thus ideally recovering the original reservoir permeability. Siliceous minerals are dissolved by hydrofluoric acid and its chemistry is much more complex than the HCl when reacting with carbonates.

Reaction rates are affected for kinetics; among the factors that strongly influence the mineral reactions are acid concentration and temperature. Dissolution reaction rates are proportional to the HF concentration for most sandstone minerals. The dissolution of minerals is a thermally activated phenomenon; thus, the rates increase greatly as a function of temperature (approximately multiplied by 2 for quartz for a 25°C increment), and the penetration depths of live acid diminish accordingly **[11]**.

The broad range of flow rates that can be obtained with the set of pumps already installed, will allow to reach high acid flux as those encountered at field conditions or even higher; for example, when displacing acid in cores at 200 ml/min, the flux equals to 39.5 cm/min, 17.5 cm/min, and 2.5 cm/min for 1”, 1.5”, and 4” diameter respectively; thus, very realistic flow rates can be achieved to see the effect on the process of acidization of diverse rocks. When displacing acid through carbonate cores it is expected to form the wormholes at low injected volumes of acid, i.e. less than two pore-volumes; so it is so important to watch the pressure behavior until severe pressure drop can be observed and hence reached the breakthrough.

In sandstone cores the main result is the acid response curve in terms of pressure drop or permeability vs. time or amount of injected acid (pore-volume); it shows the permeability behavior as the acidizing process take place. Permeability is calculated by applying the Darcy's Law equation for a linear flow. This common expression as a follows:

$$k = \frac{Q\,A\,\Delta P}{\mu\,L} \tag{3.1}$$

The equation 3.1 can be rewritten in terms of variables used in laboratory as:

$$k = 122.8\,\frac{Q\,D\,\Delta P}{\mu\,L} \tag{3.2}$$

Where ***k*** is permeability in mD; ***D*** diameter of the core in inches; ***ΔP*** is pressure drop in psi; ***μ*** is the viscosity in cp, ***L*** is the core length in inches, and ***Q*** is the flow rate in ml/min.

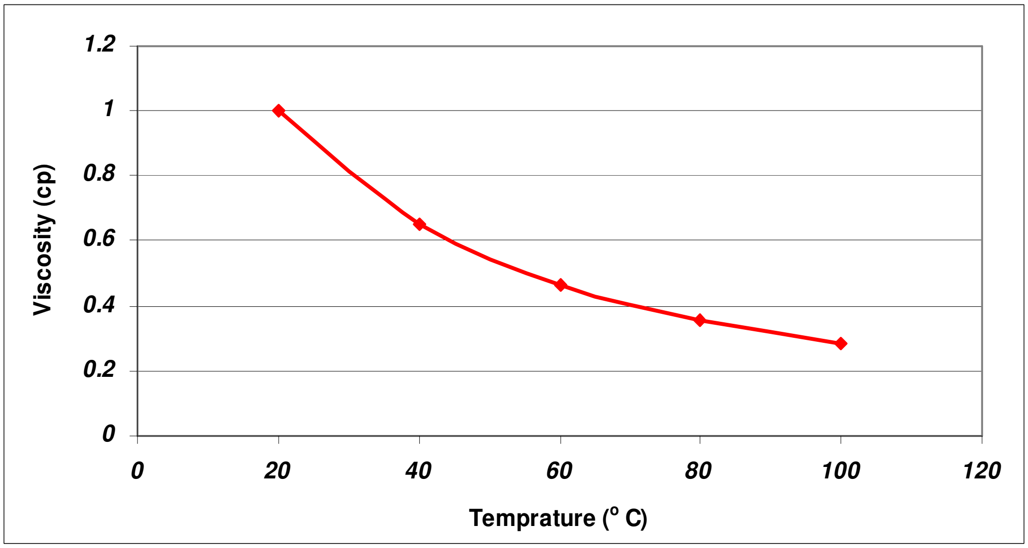


Figure 3. 1 Viscosity of brine

The values of permeability are computed directly for the LabVIEW, based on a constant viscosity to facilitate the calculation during the experiments; therefore, these calculations must be corrected by viscosity depending on the temperature at which the testing was carried out and the type of fluid being displaced at each stage once the file has been downloaded. Figure 3.1 shows the variation of viscosity of brine with temperature and Table 3.1 shows values of dynamic viscosity in centipoises for aqueous HCL.

Table 3. 1 Dynamic viscosity of aqueous solutions of HCl. * *extrapolation*

Concentration	**TEMPERATURE (^{o} F)**							
% wt	**14**	**32**	**50**	**68**	**104**	**140**	**176**	**212**
0.05	1.84	1.40	1.10	0.72*	0.51*	0.38*	0.30*	
0.10	2.58	1.94	1.50	1.19	0.79*	0.56*	0.41*	0.31*
0.15	2.64	2.02	1.51	1.28	0.87*	0.63*	0.48*	0.38*
0.20	2.73	2.13	1.71	1.39	0.98*	0.72*	0.56*	0.45*
0.25	2.96	2.32	1.87	1.54	1.10*	0.83*	0.65*	0.53*
0.28	3.43*	2.75	2.25	1.88	1.37*	1.06*	0.84*	0.70*
0.30	3.21	2.54	2.07	1.73	1.27*	1.00*	0.82*	
0.33	3.42*	2.72*	2.23*	1.87*	1.39*	1.10*		
0.36	3.74*	2.98*	2.44*	2.04*	1.51*			

HF aqueous solution also need to be corrected by viscosity, information about this property is limited; so as a good approximation the HCl viscosity at the concentration used with HF can be used because the % wt of HF normally is not greater than 6%. Figure 3.2 shows the viscosity of aqueous HF.

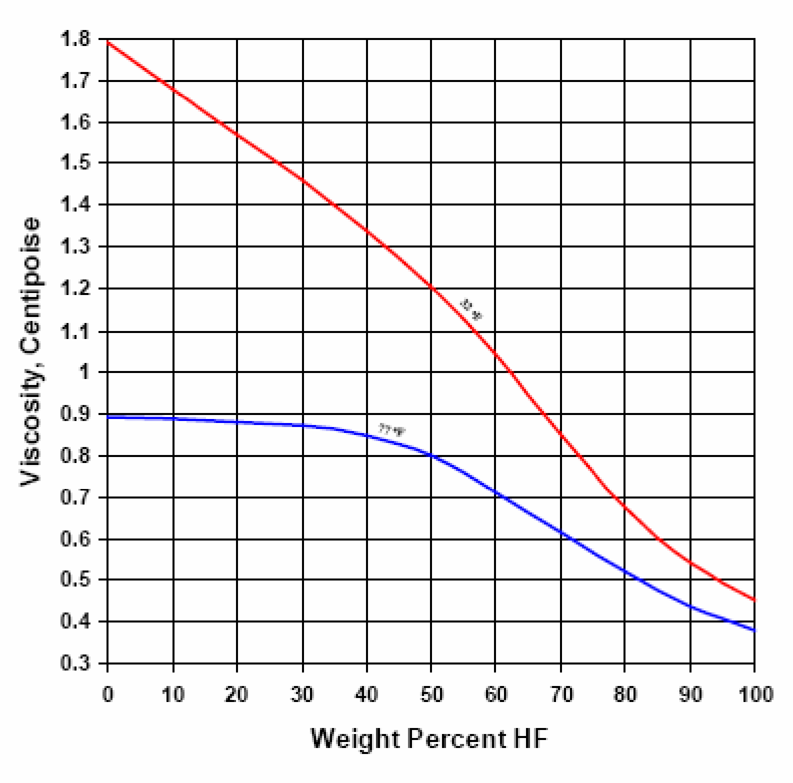


Figure 3. 2 Viscosity of HF-H2O system[12].

3.2 General matrix acidizing procedure

The general matrix acidizing procedure is:

- Cutting and preparation core samples;
- Saturation of core samples with brine:
- Prepare acid mixture and brine;
- Refill pumps with hydraulic oil and accumulators with acid and brine
- Assemble of core samples into core holder adjusting inlet and outlet face and pressurize the Viton sleeve;
- Set the desired back pressure from nitrogen supply;

- Pump the fluids in the correct sequence through the core sample. If temperature is required turn the heaters on once start pumping the brine, not before;
- Switch the pump of and close the inlet valve, release the back pressure to zero and the confining pressure as well;
- Disassemble the equipment and clean all the parts in contact with the fluids;

3.3 Core sample saturation procedure

The cores are previously cut in the desired size with the drilling machine HILTI DD-200 using the core bits. The core samples are saturated in a vacuum bell to get them saturated with brine before being flooded by the acid treatment; the air from the pore space in core samples is completely removed to avoid the presence of bubbles that may cause undesirable effect on acidizing process. Figure 3-3 shows the glass bell, the special vessel, vessel used to saturate the cores vacuum. The procedure to saturate the core samples is the follow:

- Connect the vacuum lines to the vacuum pump, glass vessel, buffer bottle and the switch.
- Put sufficient amount of brine into the vessel to completely submerge the samples.
- Apply the vacuum grease on glass vessel rim, put core samples in glass vessel, and then move glass lid to cover the vessel completely; make sure the whole core samples are submerged.
- Turn the vacuum pump on and keep it running for 24 hours to assure a good saturation.
- Shut off the vessel vacuum lines, take the core samples out of the vessel and put them in a container with similar type of brine while they are using in the experiments, avoiding long contact with air after saturated.

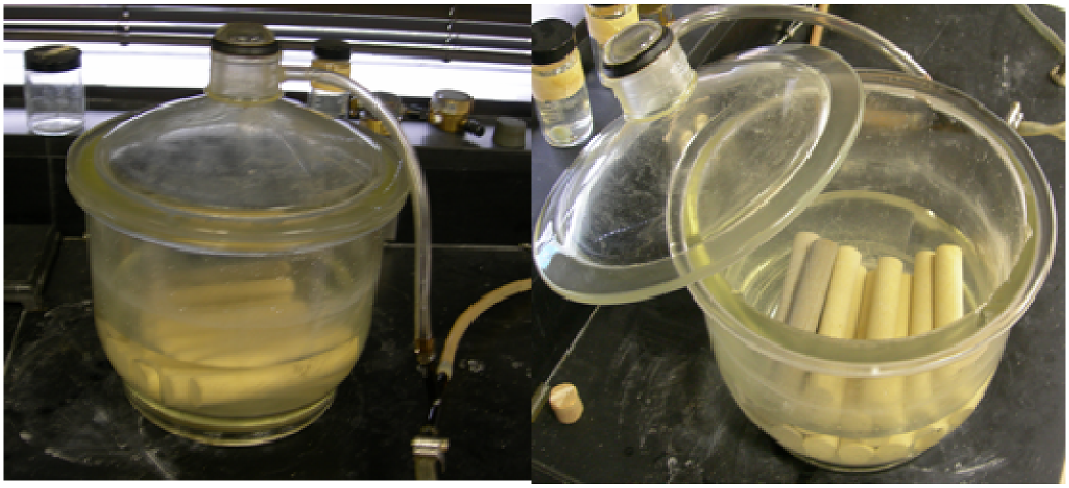


Figure 3. 3 Vacuum glass bell to saturate cores.

3.4 Acid preparation

Acid at different concentration and strengths must be prepared to be used in different types of matrix acidizing experimentation. For acidizing sandstone core samples either mixtures of HF and HCl or weaker acids are used; their concentration may vary depending the wanted effect on mineral dissolution and/or the severity of the damage to be removed. In the case of carbonates, HCl is the acid more commonly used to stimulate them and different concentration may be required depending on nature of the rock.

The most common commercial acid concentrations that can be found are hydrofluoric acid at 48% weight with density of 1.137 gm/cm^3 and HCl at 31.45% wt 20 ^{o} Baume, whose density is 1.16 gm/cm^3. To calculate the amount of each substance to be added to obtain the acid mixture to displace the cores next relationship must be used on the basis of preparing one liter of solution:

$$Vol\ add = \frac{\%\ wt_{final}\ \text{x}\ \rho_{final}}{\%\ wt_{initial}\ \text{x}\ \rho_{initial}}\ \text{x}\ 1000 \tag{3.3}$$

As an example, to prepare a solution of regular mud acid HF 3% wt and HCl 12% wt from commercial products, the proportion of each product must be calculated knowing that density of mud acid in 1.064 gm/cm^3, thus the calculation gives:

$$Vol_{\ HCl}\, add = \frac{0.12 \text{ x } 1.064}{0.3145 \text{ x } 1.16} \text{ x } 1000 = 350\ ml$$

$$Vol_{\ HF}\, add = \frac{0.03 \text{ x } 1.064}{0.48 \text{ x } 1.137} \text{ x } 1000 = 58\ ml$$

Thus volume of water must be added until the volume basis that is 1000 ml is completed. The balance in term of weight gives:

Volume (cm^3)	Density (gm/cm^3)	Weight (gm)	% weight
350	1.16	128	12
58	1.137	32	3
592	1	904	85

3.5 Accumulators charging

Once the acids and brine have been prepared according the formulation to be used and has been decide what type of treatment is going to be pumped, the accumulators must be charged appropriately with these fluids. Figure 3-4 shows the schematic diagram of accumulators and lines.

The procedure is quite simple but at the same time it demands to be carefully with the handling of the acids; therefore, proper safety equipment must be used such as neoprene gloves, face shield or glasses, and respirator. The procedure is as follows:

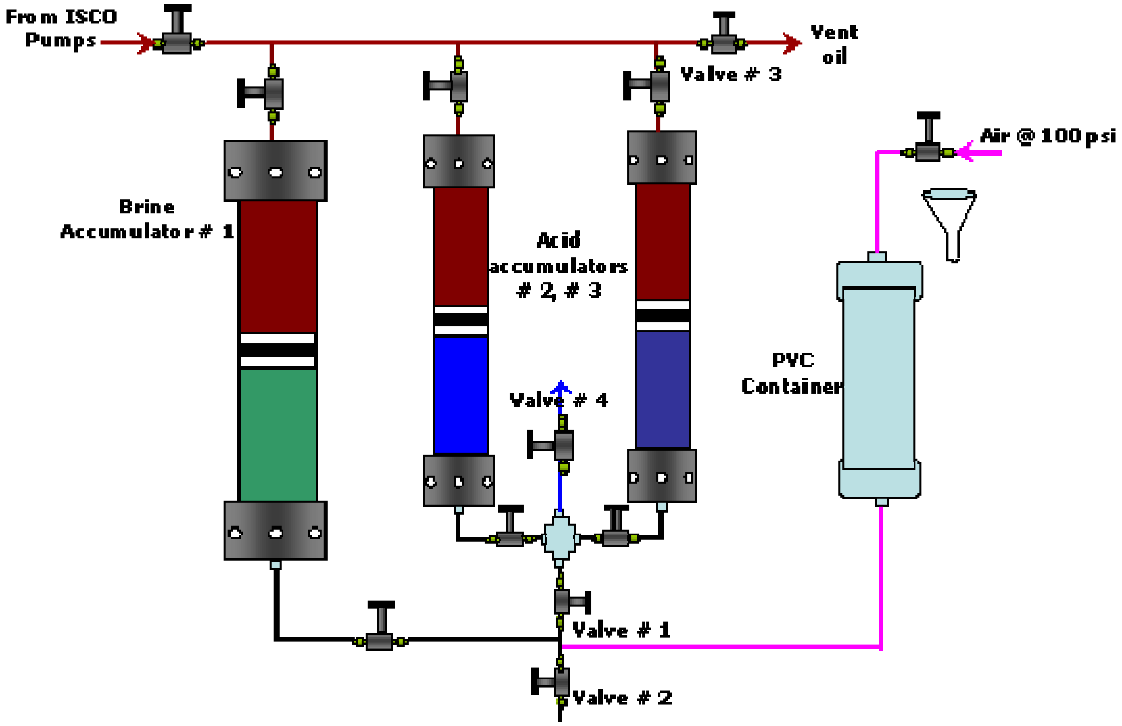


Figure 3. 4 Schematic of recharging system

- Remove the air quick connection on top of one liter PVC container;
- Check the valve # 1, # 2, and # 4 to be closed and by using the funnel, slowly put the acid into the PVC container; when filled this container, reconnect the air supply;
- Open the valve # 4 and those corresponding to the accumulator to be filled at this time, brine or acid, open the valve # 3 to vent the oil being displaced.
- Open the air supply, which at a pressure of 100 psi will displace the fluid into the accumulators while the oil is coming out and collected in other container;
- Watch this process to measure the volumes of hydraulic oil being recovered; it is extremely important when the accumulators is larger than one liter which will require a new operation.

- Once filled the accumulator close the valve of air supply, close the valve # 1 and those of the accumulator recently filled; Disconnect the air supply to release the remaining pressure.
- Open the valve # 2 to drain any remaining fluid from the PVC container taking care in handling the fluids.

3.6 Detailed matrix acidizing procedure

When the type of rock and the characteristics of the treatment to be pumped through the core sample are defined, and the accumulators have been filled with the appropriate fluids, then the experiment can be carried out; the detailed procedure is as follows:

- Insert the core inside the core holder, connect the flow tubing, inlet and outlet tips, and pressure access lines to core holder. Make sure all the connections are tighten and valves in proper positions. Check the nitrogen lines from nitrogen bottles to back pressure regulators.
- Check the valves of pressure to choose which of pressure gauges are going to be used to get the signals. As was previously mentioned, there are three different scales, so it is recommended to try first with the highest one, that means 0-3000 psi and once stabilized the pressure drop, and if this pressure is less than 300 psi the medium one can be used and so on if pressure goes down up to less than 30 psi to use then the lowest range gauge.
- Check the by-pass valves in the downstream line; which must be closed and open the inlet valve to allow the fluids to be displaced through the core.
- Connect the line of hydraulic oil to the ports in the core holder and apply the confining pressure with ENERPAC pump up to reach the desired pressure; it is

recommended 1200 psi, check the pressure gauge mounted in the front panel, labeled as Overburden Pressure, to see this pressure signal.

- Put in open position the inlet valves of the three accumulators to maintain the pressure as constant as possible during the experiments and open the outlet valve of brine accumulator.

- Open nitrogen regulator; for main effluent back pressure regulator, adjust the outlet pressure backpressure 300 psi less than overburden pressure; this is the pressure to be applied on the dome of BPR; check this pressure in the gauge mounted in the front panel.

- Open the discharge valve of both syringe pumps previously refilled with hydraulic oil; operate the key controller to select the pump which is going to start the process and set the working flow rate as well as the maximum pressures to protect the equipment. Turn on lab exhaust system and start the pump on.

- If the experiments are to be conducted at different temperature other than room temperature, turn the heater on by operating the heater controller and set the desired temperature. Do not operate the heaters before the fluids start pumping and monitor the fluid temperature constantly.

- As long the process is moving forward, open the LabVIEW software and run either "**M_Acidize(C3000).vi**" or acid "**M_Acidize(C300).vi**" program from the desktop. The wave chart for two pressure channels and the calculated permeability will display on screen. Input the flow rate, core diameter, core length, and the base viscosity; set the rate for acquiring the data and determine the file to store them.

- Check the flowing conditions and wait until the pressure downstream reach the backpressure, so the fluids will start to coming out and dropping into the container. Check the flow line to make sure no leakage at high pressure. When the process is stable enough, the data acquisition can start from the computer.

- Let the brine flow until the pressure drop becomes constant, reaching the plateau that means this is the original permeability.
- Switch the valves to change the fluids coming into the core; that is, open the HCl preflush valve and close the brine valve. Depending how much carbonate minerals are present is the amount of HCl necessary, in general it s recommended to pump at least twice necessary the volume to dissolve the carbonate mineral based on dissolving power at used acid strength.
- Once displaced the preflush, switch the valves again opening the brine line and closing the HCl line. Thus brine will displace the possible reaction by-products before pumping the main acid stage. It is recommended to pump eight to ten pore volumes of brine at this stage.
- When pumped the brine volume, switch the valves opening the HF/HCl accumulator and closing the brine accumulator; thus the main acid stage will be pumped into the core. The volume must be decided in accordance with the effect that is required to achieve in the rock.
- Finally once pumped the volume corresponding to main acid stage, switch the valves again opening the brine accumulator and closing the HF/HCl accumulator to allow brine to flow through the core an determine the final permeability.

Note: During the process of pumping and changing the type of fluids, depending on the flow rate, it will be necessary to change the pump switching them once the cylinder get empty and the operator must be ready to change. When the cylinder’s volume of working pump goes to zero, immediately in the controller select the new pump and push the button corresponding to this one and press start, making sure the flow rate has been set the same as previous. The empty pump must be refill by closing the discharge valve and opening the refill line, in the controller select the refill mode and choose the pump to be refilled and press start; it is recommended a maximum 40 ml/min as the rate for this refilling operation.

- When the brine postflush displacement is finished, switch the pump off and close the inlet valve. Close the nitrogen valves and release the back pressure. Stop LabVIEW program to acquiring data. Wait for a while, until flow in no longer coming out to release the overburden pressure.
- Disconnect all fittings and get the core out. Rinse all components with water.

3.7 Safety issues and precautions

During acidizing experimentation people must be aware of they are dealing with extremely dangerous and highly corrosive fluids as HF 48%. Testing is regularly conducted at moderate to high temperature and pressure; therefore, the safety is a priority and all of related issues as special clothes and carefully planned procedures deserve special attention.

HF is a colorless liquid with a strong irritating odor at low concentrations. Exposure to HF has dangerous potential effects, very hazardous to human health and the environment. It may cause severe burns to eyes, skin, and respiratory tract; these burns may not be immediately painful or visible, onset can be delayed up to 24 hours. It can be absorbed through the skin in fatal amounts causing destruction of the deep tissue, with high risk of cardiac and nervous disorders. Inhalation causes severe irritation of the nose and the throat, spasmodic cough and difficulty in breathing. Specialized medical treatment is required for any exposure to HF.

HF should be used with adequate ventilation to minimize inhalation of vapor. It must be handled inside a properly functioning chemical fume hood. Chemical splash goggles together with a face shield when handling concentrated HF, if risk of vapors is resent a respirators must be used with the adequate vapor gases cartridge.

Shorts or open-toed shoes never should be worn when handling HF or other corrosive chemicals; medium or heavyweight viton, nitrile, or natural rubber gloves are needed to be worn when working with HF. As first aid, calcium gluconate gel, a topical antidote for HF skin exposure must be kept nearby whenever working with HF. It. should be stored in a refrigerator if possible. Even after applying calcium gluconate, it is essential that a medical evaluation be made.

As a safe work practice if possible, avoid working alone when using HF. Do not eat, smoke, or drink where HF is handled, since the chemical can be swallowed. Wash hands thoroughly after handling HF. Store all HF and HF waste in labeled chemically compatible containers (e.g., polyethylene or Teflon). Glass, metal, and ceramic containers are not compatible with HF. HF should never be stored with incompatible chemicals such as ammonia or other alkaline materials.

Always place HF on a low protected shelf or other location where it will not be accidentally spilled or knocked over. HF waste should be placed in a chemically compatible container with a sealed lid and clearly labeled.

CHAPTER IV

PRELIMINARY EXPERIMENTS AND RESULTS

A set of preliminary experiments of matrix acidizing were conducted on cores of Berea sandstone, Cream Austin chalk and Indiana limestone to adjust the lab set-up and check the capacity and reliability of the installed equipment and testing its operating range. The experimental results were recorded and preliminarily analyzed.

Matrix acidizing in Berea sandstone is conducted to see the effect of mixtures of HF/HCl on acid response curve, permeability/ΔP Vs. injected pore volume; while in carbonates, Cream Chalk and Indiana limestone, the goal was to observe the evolution of wormholing patterns created by mineral dissolution through highly permeable pathways by effect of HCl mixtures.

4.1 Experiments parameters

Diverse acid mixtures were prepared according the formulation previously discussed and stored in the fume hoods conditioned for this purpose. Cores of Berea sandstone and some carbonates (Cream Chalk and Indiana limestone) were first completely vacuum-saturated with ammonium chloride brine.

Sandstone cores were acidized at three different flow rates for acid-displacement, 15 ml/min, 30 ml/min, and 45 ml/min. The acid concentration was the regular mud acid, 12% HCl and 3% HF for the main acid and the preflush was HCl 15%. Different temperatures were used at each flow rate; changing the variables, several displacements were done at room temperature, 72 ^{o}F, 100 ^{o}F, 150 ^{o}F, and 200 ^{o}F.

A certain volume of preflush is required in all sandstone treatments to dissolve the HCl soluble minerals. Based on the reaction stoichiometry between HCl and these minerals the concept of dissolving power[2] was applied. The dissolving power expresses, on a mass or volume basis, the amount of mineral that can be consumed by a given amount of acid. Thus, the gravimetric dissolving power β is defined as:

$$\beta_{100} = \frac{v_{\min eral} \; MW_{\min eral}}{v_{acid} \; MW_{acid}} \tag{4.1}$$

Where the ν terms are the stoichiometric coefficients and MW are the molecular weights; thus, for the reaction between 100% HCl and CaCO3, β_{100} = 1.37 lb $CaCO_3$/lb HCl. The dissolving power of any other concentration is β_{100} times the weight fraction of acid in the acid solution. For an acid 15% HCL, dissolving power β_{15} equals to 0.21. The volumetric dissolving power X, is defined as the volume of mineral dissolved by a given volume of acid and it is related to the gravimetric dissolving power by:

$$X_{100} = \beta \frac{\rho_{acid\ solution}}{\rho_{\min eral}} \tag{4.2}$$

A 15% HCl solution has a specific gravity of about 1.07 and $CaCO_3$ has a density of 169 lbm/ft^3. For the reaction of these species, the volumetric dissolving power is 0.082 ft^3 $CaCO_3$/ft^3 $HCl_{15\%}$.

Table 4.1 shows the typical mineral composition of Berea sandstone. As it can be seen HCl soluble minerals are Dolomite and Siderite whose volumetric dissolving power are 0.0628 and 0.0945 respectively; thus for a core of 6” long and 1” diameter, bulk volume 77 ml, with a porosity of 21%; the volume of HCl to dissolve the total dolomite and siderite was calculated as 75 ml, which means about 5 pore volumes of the core. The porosity of Berea sandstone core samples was measured at existing laboratory with the helium porosimeter as 21.15%.

Table 4. 1 Mineral composition of Berea sandstone[3].

MINERAL	VOLUME FRACTION (%)
Quartz	75
Feldspar	5
Dolomite	5
Siderite	5
Chlorite	5
Mica/Illite	5

4.2 Preliminary experiments results on sandstone cores

To acidize Berea sandstone cores, regular mud acid (HCl 12% HF 3%) was used at different flow rates and temperatures. Diverse effects were observed once the displacement was completed as can be seen in figure 4.1, which shows the inlet face of three cores of Berea sandstone recently acidized with regular mud acid.

Figure 4. 1 Inlet face of acidized Berea sandstone cores

The general procedure is followed in all cores previously saturated with brine 3% NH_4Cl at vacuum conditions.

- Displace the cores with brine 3 % NH4Cl until stable enough to measure initial permeability.

- Inject the preflush, 100 to 140 ml of HCl 15%, to remove HCl soluble minerals
- Inject NH4Cl as a spacer, generally 170 ml which corresponds to 10 pore volumes.
- Inject the treatment fluid, HF 3% - HCl 15%, at different temperatures and flow rates. Initially was pumped the maximum capacity available, up to 60 pore volumes.
- Inject the postflush, 3% NH_4Cl brine, to determine final permeability, sufficient amount until reach this value as constant as possible.

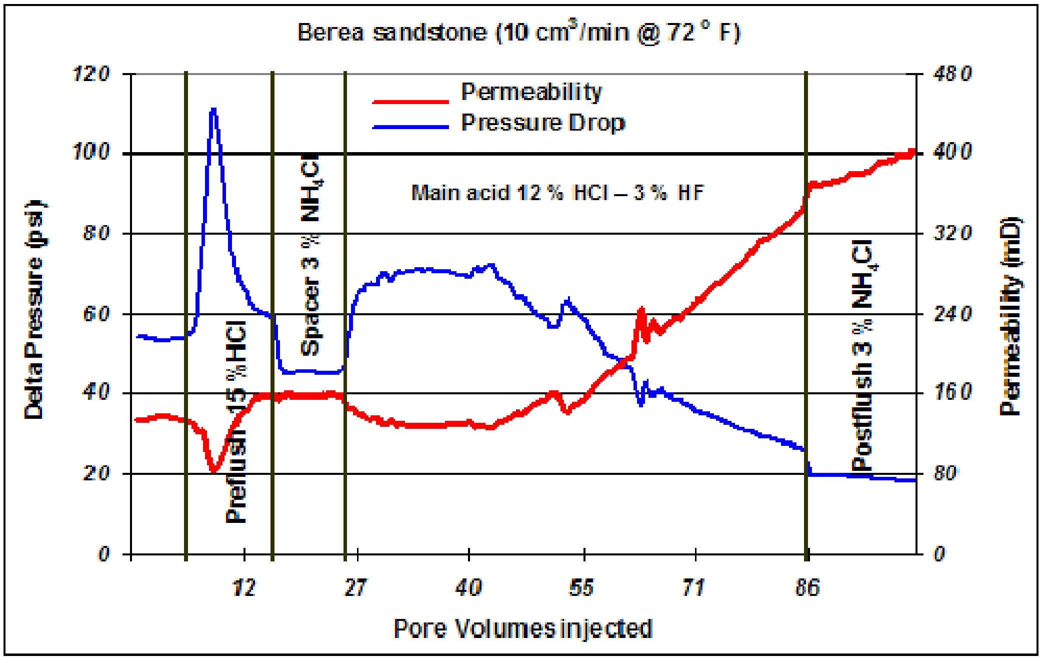


Figure 4. 2 Acid response curve from a Berea core

Figure 4.2 presents the acid response curve for a typical sandstone acid core flooding. In this curve, the pressure drop and the calculated permeability are plotted and the different stages of the process are detailed; the improvement in permeability can be observed clearly.

The core was scanned before and after acidizing process to see the difference between the two stages; after the process of acidization it appears to be present some sort of a preferential flow channel at the middle of the core based on the difference in density, see figure 4.3.

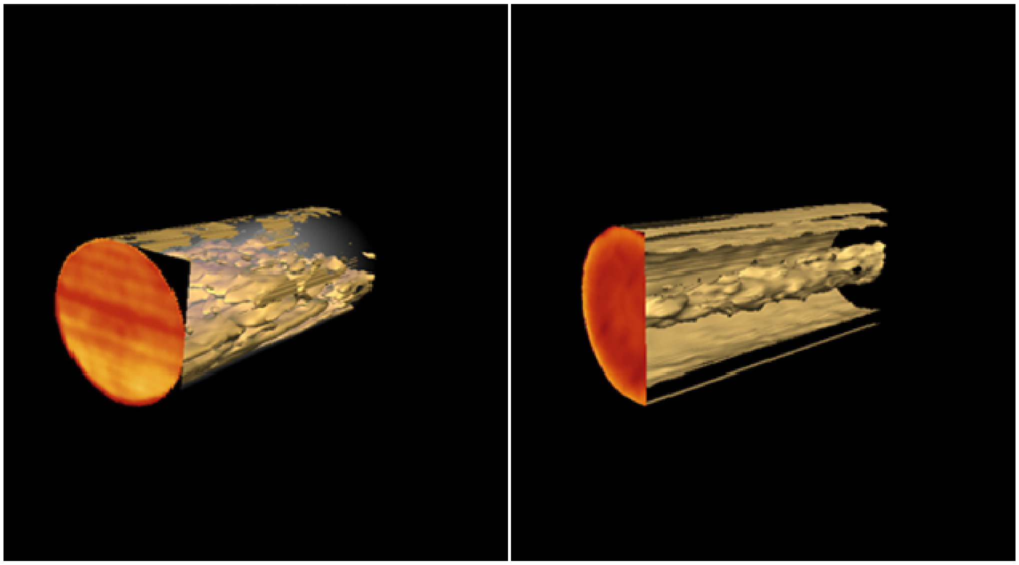


Figure 4. 3 Scanning visualization of the Berea sandstone core

During the preflush stage, a significant increase in pressure drop occurred, hence reducing the permeability; this may be a result of either viscosity contrast between the fluids in the porous media or the precipitates generated when HCl start reacting with the HCL soluble minerals; however, after one period proximately equal to the volume initially calculated as necessary to dissolve all of these minerals, the pressure drop goes down to the normal level. In the main acid stage a less severe pressure drop increase can be seen at the beginning of the stage and it gradually improves until switching to the postflush stage when the possible precipitates and any by-product are flowed out the core. Figure 4.4 shows the raw data as presented in the front panel in LabVIEW.

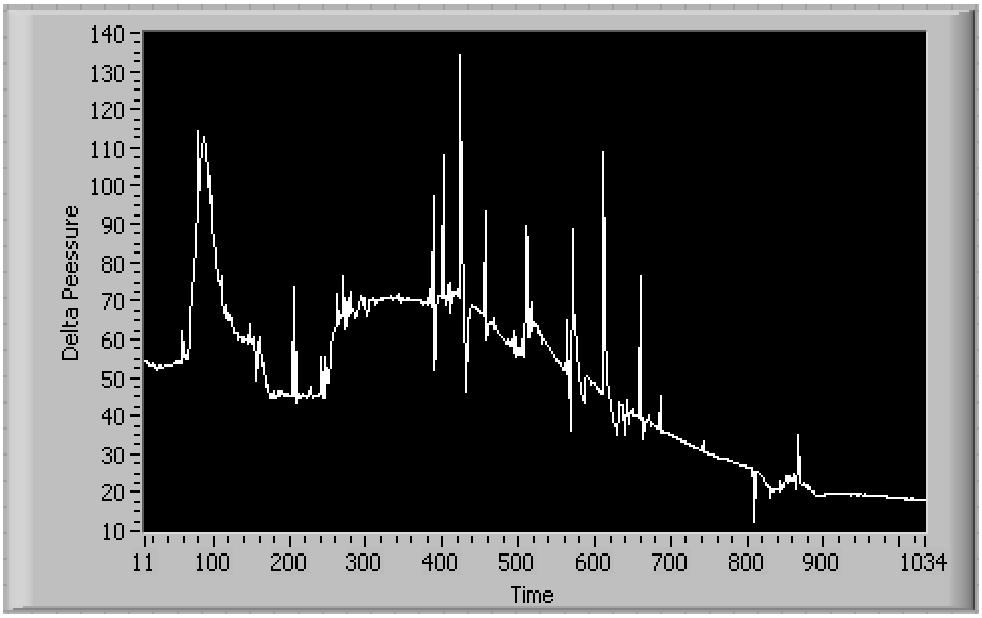


Figure 4. 4 Pressure drop data as recorded in LabVIEW program

The effect of temperature was studied at constant flow rate of 15 ml/min; temperature increases the reaction rate between clay minerals and regular mud acid as that used in the experiments. See figure 4.5, in which only the data corresponding to the main acid and postflush stage were plotted to isolate from the effect of HCl in preflush to dissolve carbonates.

Notice that the higher the temperature the more permeability improvement can be achieved. At room temperature could be achieved the same result in permeability enhancement as achieved at 100 ^{o} F but being necessary 40 % of additional acid to be injected as can be observed in curves 72 ^{o} F (a). In curve 72 ^{o} F can be observed that a large volume of acid was necessary to get optimal results.

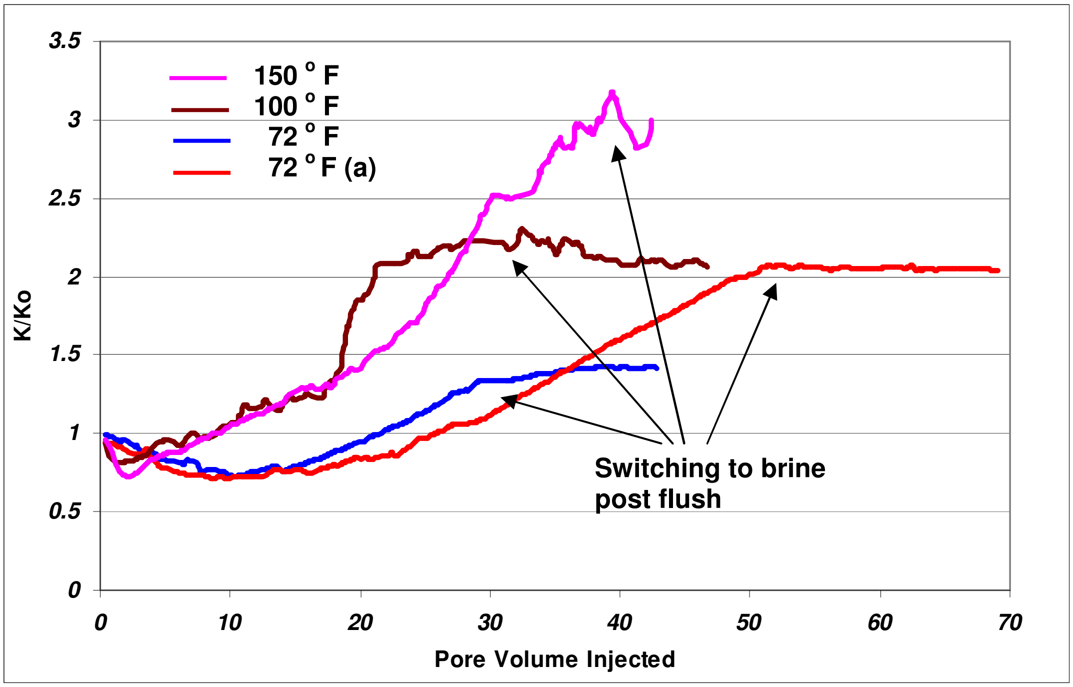


Figure 4. 5 Effect of temperature on acidizing Berea sandstone

Experiments were conducted at different flow rates to determine first the capability of the installed equipment and see the effect on matrix acidizing process. As it is shown in figure 4.6, in three core displacements at room temperature using Regular Mud Acid, the permeability response could be significantly improved when the flow rate increases. This may be explained by the fact of the reaction products are being rapidly displaced.

However, it is clearly notorious that when using very lower flow rates, as low as 5 ml/min, it seems that the permeability response to the stimulation process occurs slowly and just a very little enhancement in permeability could be obtained. This effect could be due to lack of displacing capacity of precipitates which may be present as products of the reaction between acid and minerals, leading possibly to cause an additional damage.

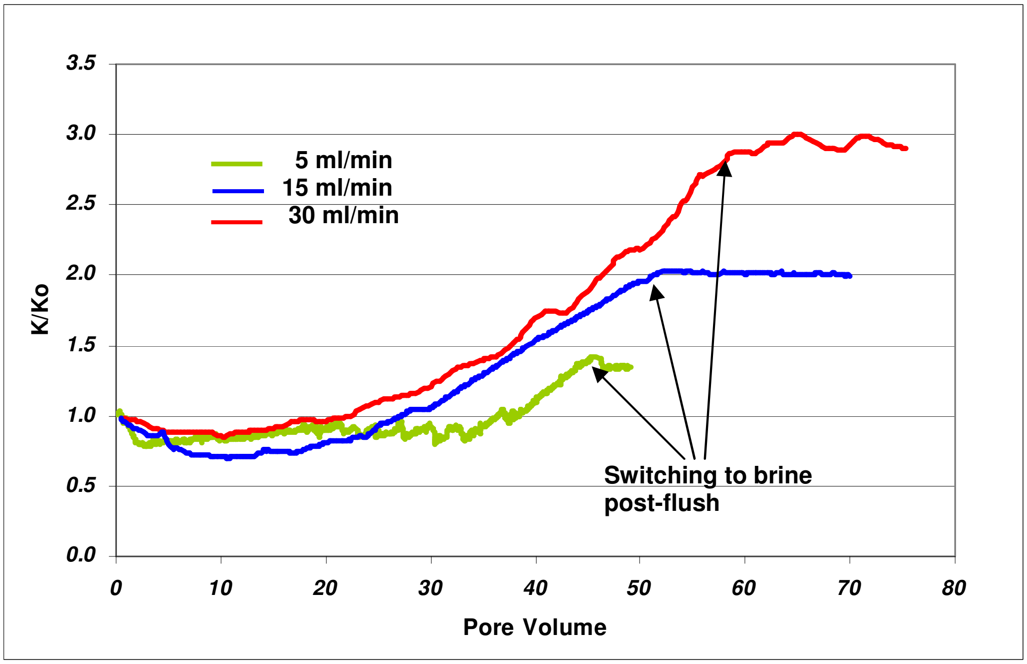


Figure 4. 6 Effect of flow rate on acidizing Berea sandstone at room temperature

The effect of flow rate was besides determined by analyzing the results from four core displacements using the Regular Strength Mud Acid, which were carried out at 100 ^{o}F; in figure 4.7 were plotted these different curves. It is shown that at lower flow rates the permeability response is still relatively poor compared to those obtained at higher ones. However, it looks like there could be determined an optimum flow rate, for Berea sandstone cores, based on the results at 30 ml/min, whose permeability response looks better than those achieved at 15 ml/min and 45 ml/min.

When the cores samples are acidized at relatively high temperatures, more than 100 ^{o}F, and large volumes of acid are used, more than 30 pore volumes, a severe change in the slope of the trend generally occurs in anywhere during the main acid stage leading to have a more rapid increase in permeability, see figure 4.8; that could means the complete dissolution of siliceous minerals present in bedding planes as can be seen in figure 4.9.

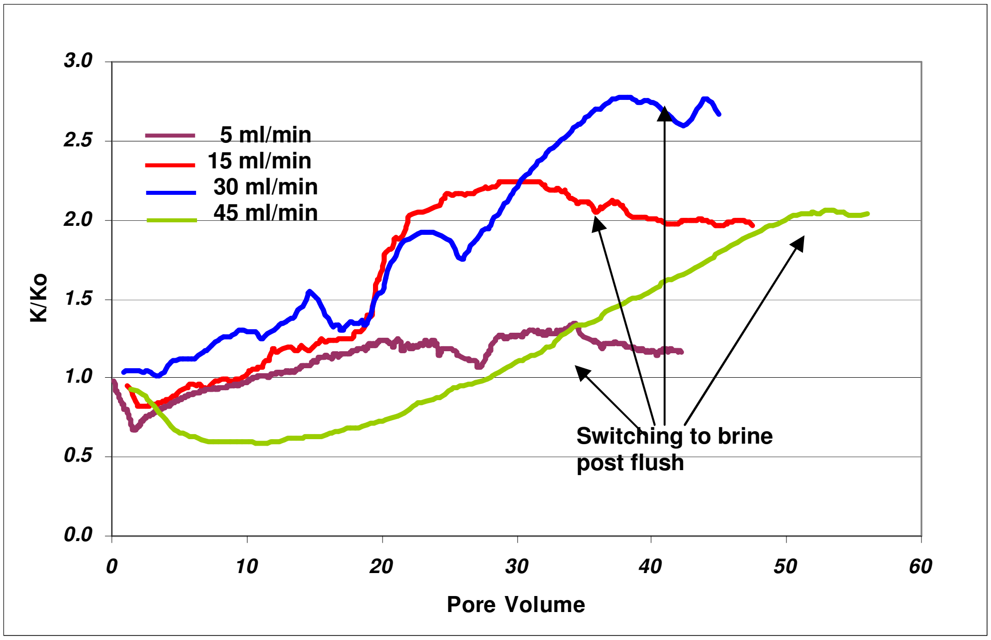


Figure 4. 7 Effect of flow rate on acidizing Berea sandstone at 100 ^{o}F

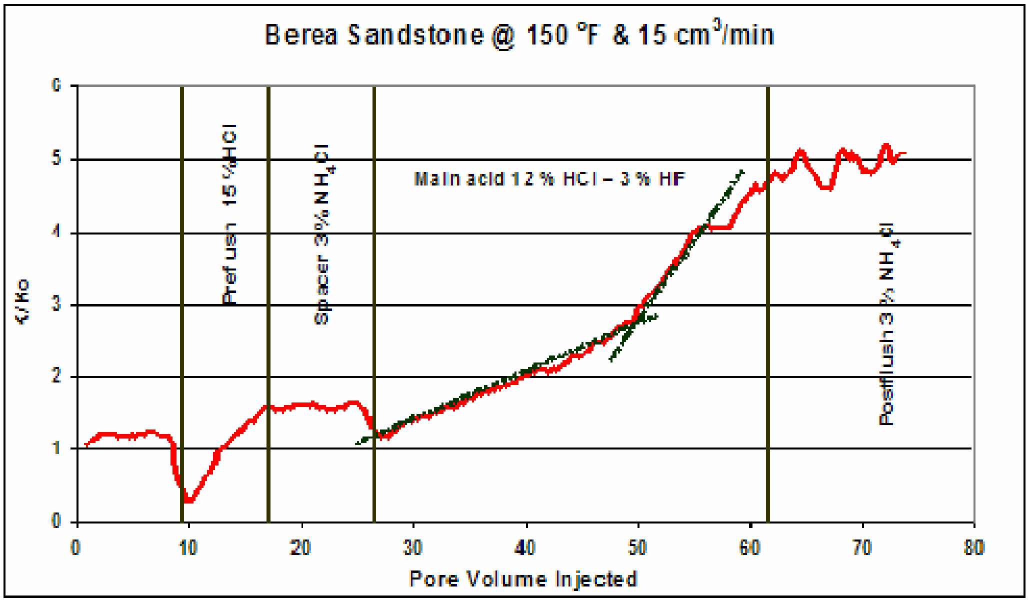


Figure 4. 8 Slope change during main acid stage

Figure 4. 9 Dissolved minerals in bedding planes of the cores

Acid concentration is one of the most important parameters to take into account when designing matrix acidizing. Figure 4.10 shows some sandstone core samples acidized with a high strength acid mixture, HF 6%- HCl 9%. As can be observed, by using this concentration may causes a significant matrix deconsolidation.

Figure 4. 10 Inlet face of cores acidized with 9% HCl- 6% HF

Because of this undesirable effect, an experiment was conducted, injecting only 10 pore volume of acid, whose acid response curve is shown in figure 4.11, it can be observed that not much permeability improvement has been reached due perhaps to additional migration of fines from deconsolidated matrix.

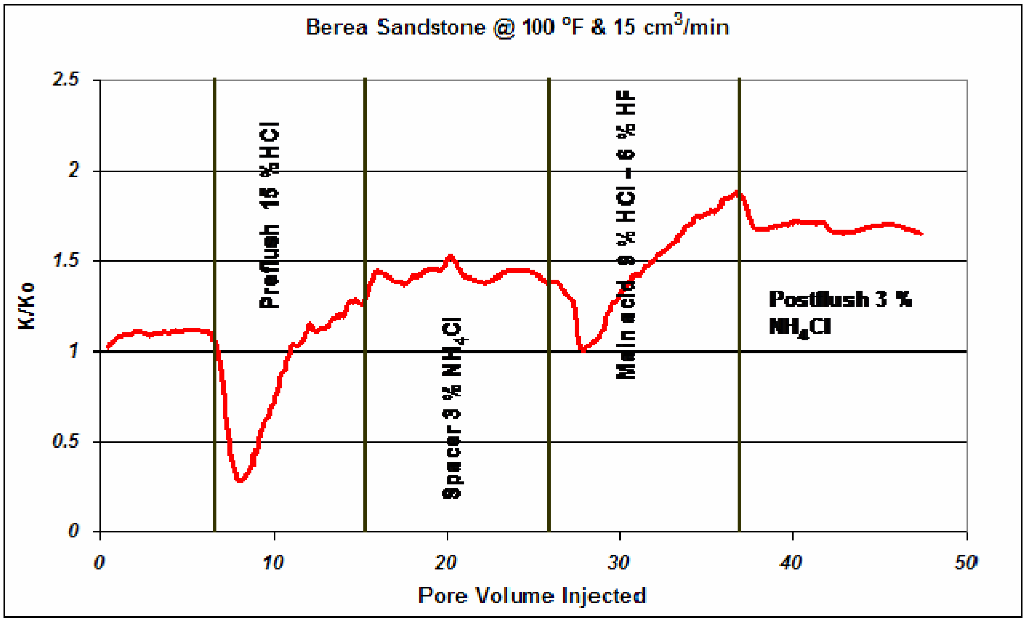


Figure 4. 11 Acid response curve of Berea acidized with 9% HCl- 6% HF

Lower acid concentration, HF 1% - HCl 9%, were also displaced in Berea sandstone cores; this concentration is the recommended by McLeod and Kalfayan for those permeability greater than 100 mD. Figure 4.12 shows the effect of acid concentration on acidizing results.

The acid response curve for the mixture 1% HF- 9% HCl does not show much success after injecting 55 pore volumes; however, the trend in the curve would allow to extrapolate a better permeability improvement by injecting more acid. The curves for Regular Mud Acid, 3% HF – 12% HCl, show similar behavior and the difference is due to the amount of displaced acid in each experiment. Only the data during the main acid stage and postflush has been plotted to isolate from the effect on permeability of HCl in preflush stage dissolving the carbonates.

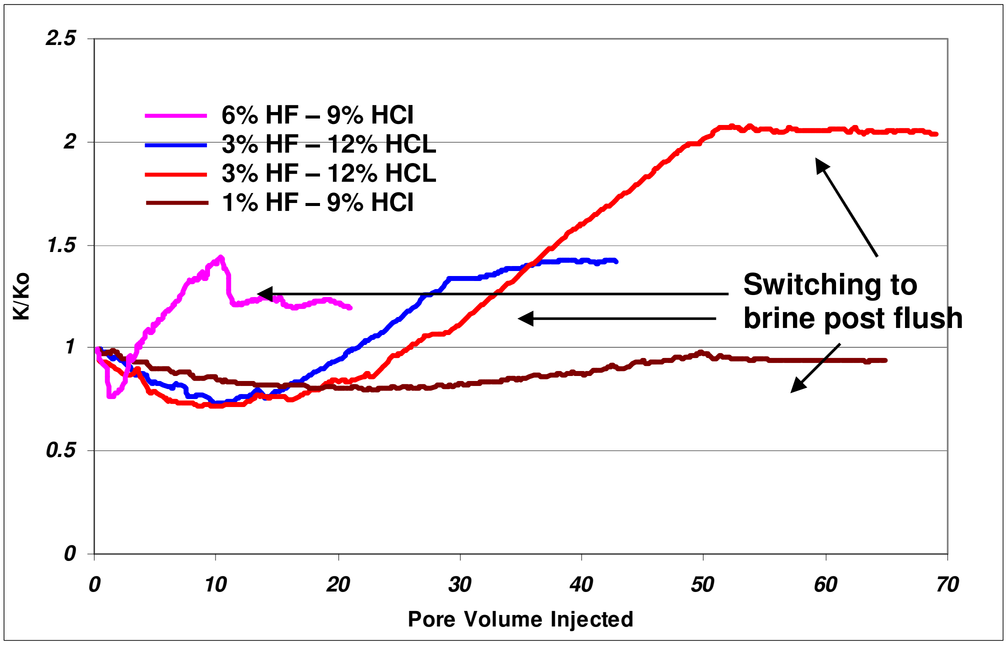


Figure 4. 12 Effect of acid concentration on Berea sandstone

4.3 Preliminary experiments results on carbonate cores

Matrix acidizing experimentation in carbonates differs enormously from that process in sandstone. In linear core-flooding of carbonates, the principal parameter to be evaluated is the volume necessary to reach the breakthrough, which means that wormholing phenomenon has occurred. This experimentation reveals the optimum rate to stimulate the rocks and the wormholing patterns development.

Cores of Cream chalk and Indiana limestone were used to do the core displacements; the porosity, measured at helium porosimeter, was 26.82%. Lower flow rates than those used in previous sandstone displacements were used. First a certain volume of brine was pumped, sufficient to get the original permeability of the rock. Once reading the permeability stable enough, switch the accumulator to displace the acid through the core and observe carefully the process until observing the sharpness in pressure drop indicating the breakthrough has occurred, immediately switching again to displace the brine. Figure 4.13 shows the pressure drop during carbonate acidizing; in this experiment the breakthrough occurred at 0.845 pore volume.

Figure 4.14 shows the inlet face of Cream chalk carbonate samples after they were acidized with HCl 15% wt; as it can be seen, clearly the wormholes product of mineral dissolution are present. The flow rates for acidizing these cores were 2, 4, and 1.5 ml/min from left to right. As can be seen the lesser the flow rate more uniform appears to be the inlet face dissolution.

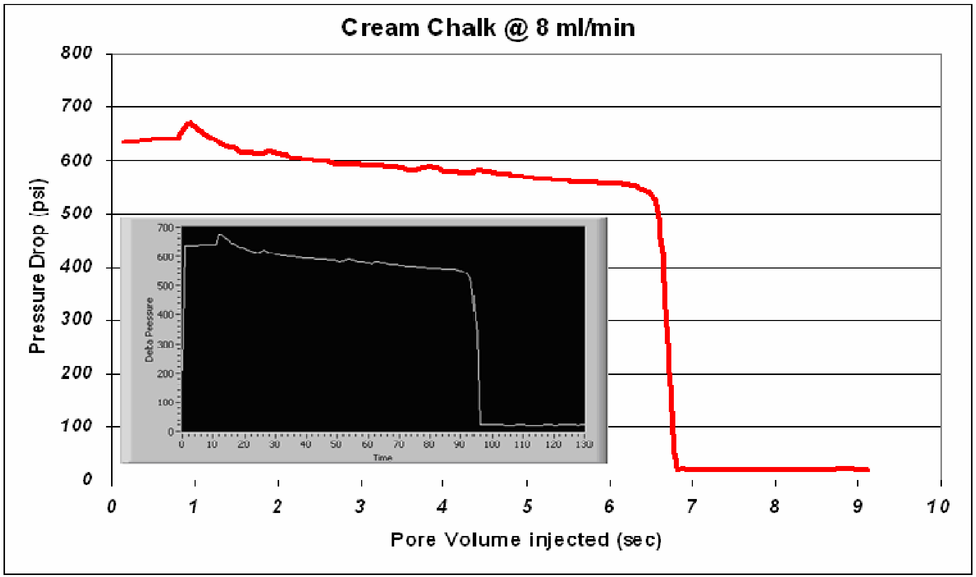


Figure 4. 13 Pressure drop during acidizing carbonate cores

In Figure 4.15 are plotted the injected pore volume at the breakthrough at different flow rate for Cream chalk cores at room temperature; it can be seen the trend to determine the optimum injection rate that represents the conditions at which a minimum volume of fluid is required to form the dominant wormhole channels.

Figure 4. 14 Wormholes at inlet and outlet face of carbonate cores

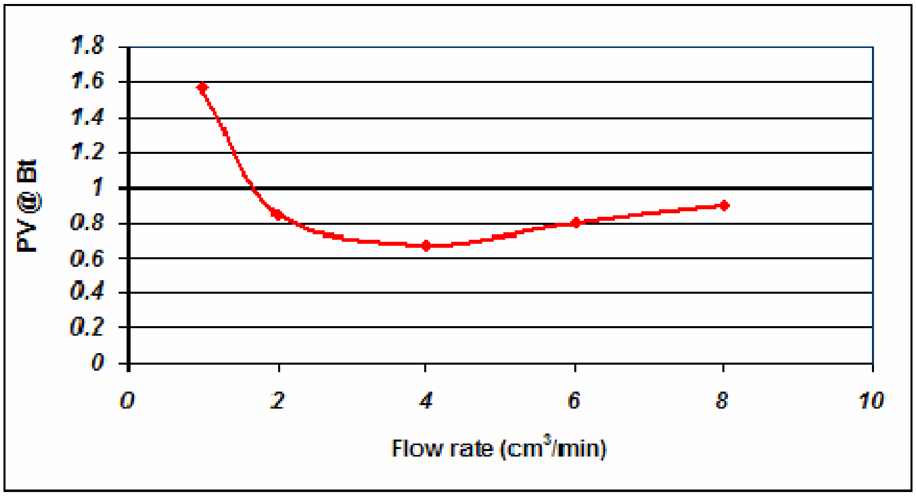


Figure 4. 15 Optimum injection rate for wormholing in Cream chalk

Some cores were scanned to see the patterns of created wormhole in Cream chalk. In Figure 4.16, the highly permeable channels can be observed, after acidizing the cores with HCl 15% wt at flow rates of 1, 1.5, and 2 ml/min.

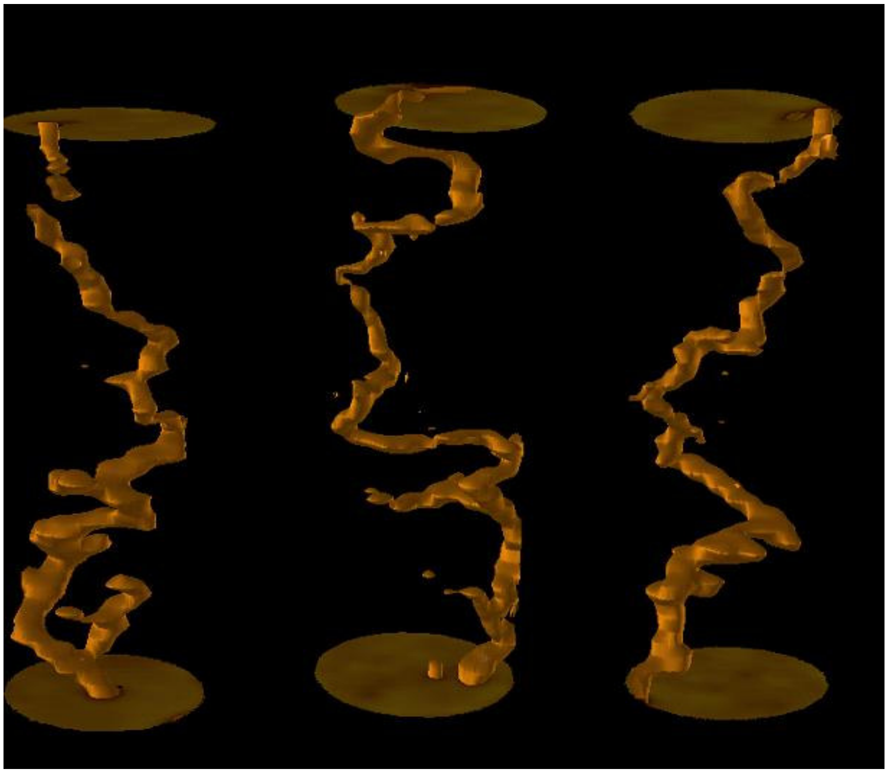


Figure 4. 16 Wormholes in Cream chalk

CHAPTER V

CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusions

A compact matrix acidizing apparatus was designed and built to conduct core-flooding experimentation on both sandstone and carbonate cores. The new equipment is located at the acidizing laboratory in Petroleum Engineering Department.

The acid apparatus was developed to be capable to work at a wide range of operational conditions in terms of flow rate, from 0.5 to 204 ml/min, temperature up to 250 ^{o} F, and pressure up to 3500 psi, to carry out extensive experimentation on matrix acidizing process.

The detailed operational procedure of the apparatus, as well as the main equipment and instrument features and capabilities were described; likewise the safety issues related to hydrofluoric acid handling were referred to account during experimentation.

Preliminary matrix acidizing experiments were carried out in both Berea sandstone and Cream chalk. Regular mud acid, HF 3% - HCl 12%, 6% HF – HCl 9%, and 1% HF – HCl 9% mixtures were used with the former and HCl 15% with the latter. From these preliminary experimental results, some conclusion can be drawn:

- Temperature increases the reaction rate of HF-HCl acid mixtures when acidizing Berea sandstone cores; thus, at higher temperatures better results could be expected from acidizing stimulation.
- Higher concentration of HF in acid mixtures to stimulate sandstones may cause deconsolidation of the rocks, adversely affecting the final result of

acidizing process because of post-stimulation fines migration along the core.

- From the limited testing with HF 3% - HCl 12%, in Berea sandstone at room temperature, it was observed that the higher the flow rate the better the permeability response, being significantly diminished the improvement at very low rates. At higher temperatures, 100 ^{o}F, the flow rate of 30 ml/ml appears to be the optimum rate.

- Acidizing limestone cores with HCl 15% showed the formation of highly permeable and macroscopic channels, wormholes.
- In carbonate rocks, the acid injection rate affects the amount of acid required for breakthrough. The lesser the flow rate the more uniform the pattern dissolution in the inlet face of the core samples.

5.2 Recommendations for future research work on matrix acidizing

Complement the matrix acidizing experimentation with sampling of the effluent to watch the mineral concentration during the coreflooding; exerting constant monitoring of the minerals product of the reactions. If possible, support interpretations of core testing with thin section analysis and SEM-DRX.

Test new non HF acid systems in sandstone matrix acidizing; which, although do not dissolve significantly some of silicates, may cause less negative effects on deconsolidation of the matrix. Another interesting area to go deeply into is using of retarded systems as those based on blends of phosphonic and citric that would allow reaching deeper penetration, being at the same time less corrosive and much safer to handle, reducing the common problems of conventional acid systems.

Investigate how using of different additives, regularly used in field applications, can affect the acidizing performance. Additives such as corrosion inhibitors, iron control agents, clay stabilizers, and surfactants are examples of the wide spectrum of products that can either positively or adversely influence the stimulation process.

Study in depth the impact of flow rate on acidizing performance in sandstone core samples, conducting more experimentation at rates lower than 5 ml/min to look for evidence of channeling, wormholing, as reported in some previous studies.

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APPENDIX A Matrix acidizing experiment data sheet sample

MATRIX ACIDIZING COREFLODING

Rock Type: ____________________ **Experiment Date:** ________________

Core Length: _____________ inch

Core Diameter: _____________ inch

Porosity: _____________ %

Acid Type: ____

Overburden Pressure: _____________ psi **Backpressure:** ______________ psi

Temperature: _____________ °F **Flow Rate:** ______________ ml/min

Pump Schedule: Preflush ______________ ml

Spacer ______________ ml

Main Acid ______________ ml

Postflush ______________ ml

Total Volume: __________________ ml

Observations: ____

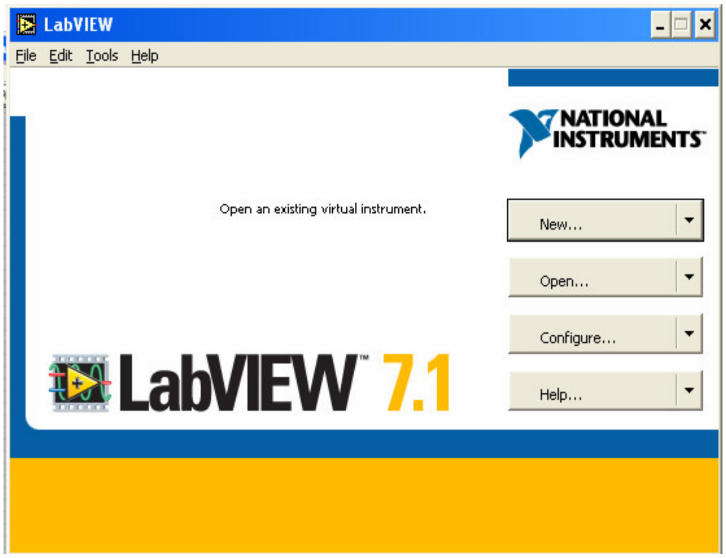
____

____

____

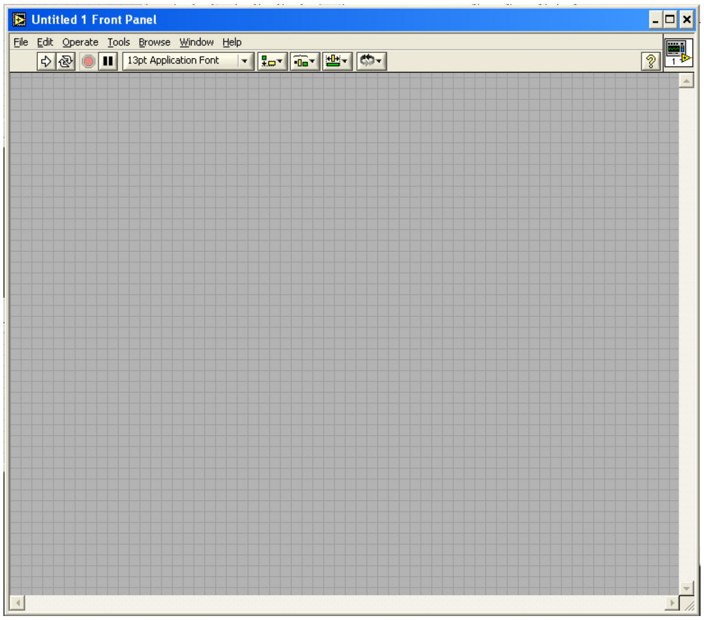
APPENDIX B Detailed procedure to program the LabVIEW DAQ

Open the software LabVIEW following the sequence “Start” - “Programs” - LabVIEW”; it displays the next screen.

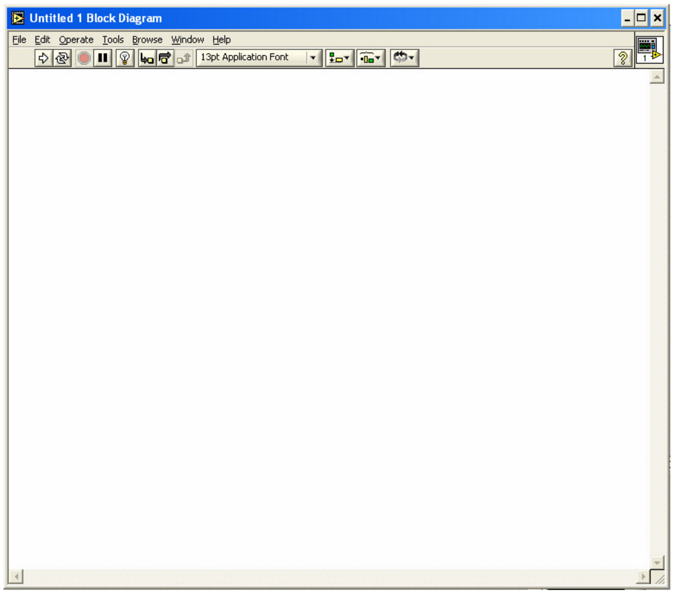


Open an existing virtual instrument.

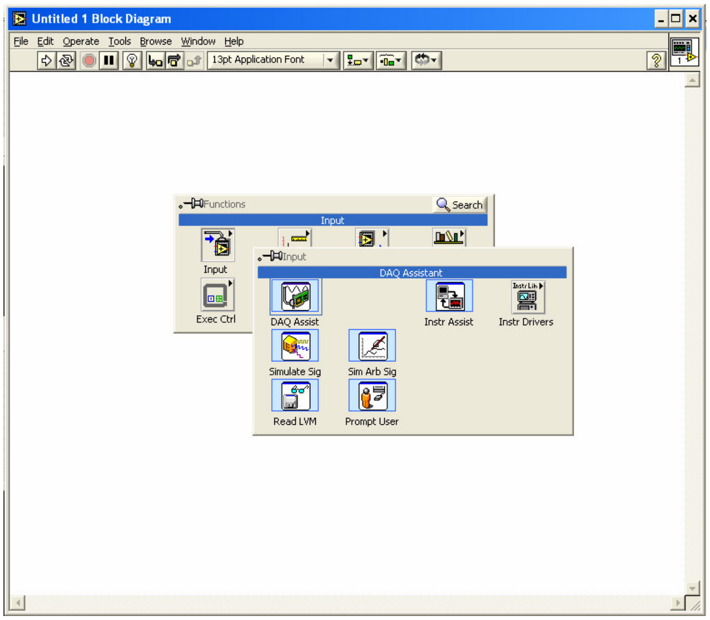
By clicking on New the “Front Panel” is displayed to start programming



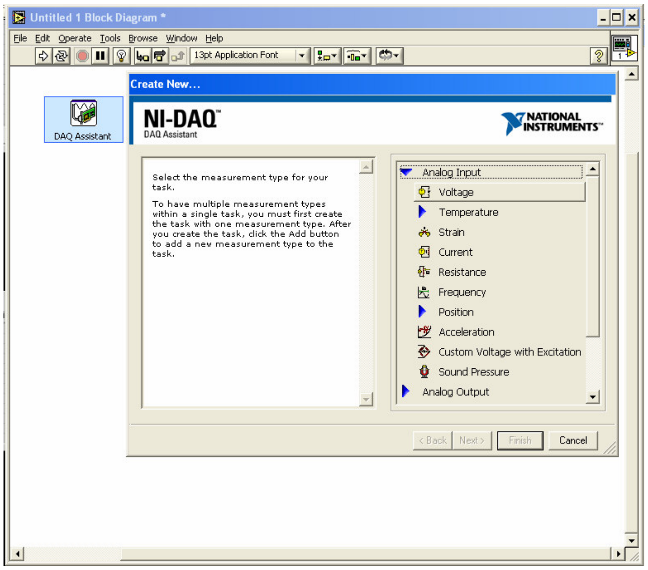
The platform to build the program is the Block Diagram view, which displays by selecting **Window** and then show **Block Diagram**.



Right click and by clicking the icon “Input” get the icon “DAQ Assistant”; drag the icon and put it somewhere in the **Block Diagram**. See next screen.



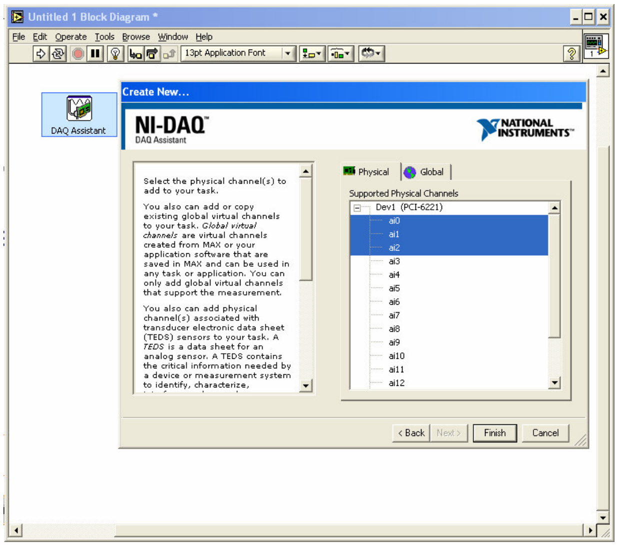
Double click on the “DAQ Assistant” and observe the new window in which **Analog Input** and then **Voltage** palette must be selected, see the screen.



Select the measurement type for your task.

To have multiple measurement types within a single task, you must first create the task with one measurement type. After you create the task, click the Add button to add a new measurement type to the task.

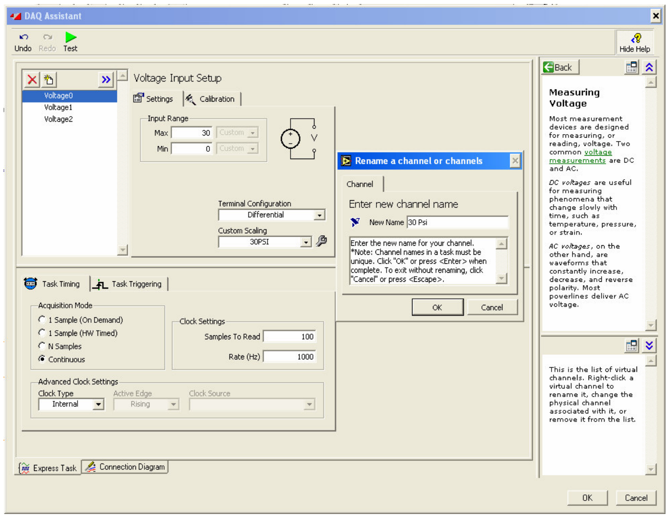
Select the analog input channels connected in the CB-68LP board; aI0 reads inputs 68 and 34, aI1 reads inputs 66 and 33, and aI2 reads inputs 65 and 31which correspond to differential pressure gauges of 30 psi, 300 psi, and 3000 psi as connected to the board. See the next screen.



Select the physical channel(s) to add to your task.

You also can add or copy existing global virtual channels to your task. *Global virtual channels* are virtual channels created from MAX or your application software that are saved in MAX and can be used in any task or application. You can only add global virtual channels that support the measurement.

You also can add physical channel(s) associated with transducer electronic data sheet (TEDS) sensors to your task. A *TEDS* is a data sheet for an analog sensor. A TEDS contains the critical information needed by a device or measurement system to identify, characterize, Once selected the analog channels, click "Finish" and it display a new window showing the control to adjust the scale and rename the channels as convenience to identify them easily, see the next screen.



Input Range
Max 30 Custom
Min 0 Custom

Terminal Configuration: Differential

Custom Scaling: 30PSI

Enter new channel name

New Name: 30 Psi

Enter the new name for your channel. *Note: Channel names in a task must be unique. Click "OK" or press <Enter> when complete. To exit without renaming, click "Cancel" or press <Escape>.

Acquisition Mode
○ 1 Sample (On Demand)
○ 1 Sample (HW Timed)
○ N Samples
● Continuous

Clock Settings
Samples To Read 100
Rate (Hz) 1000

Advanced Clock Settings
Clock Type: Internal
Active Edge: Rising
Clock Source

Measuring Voltage

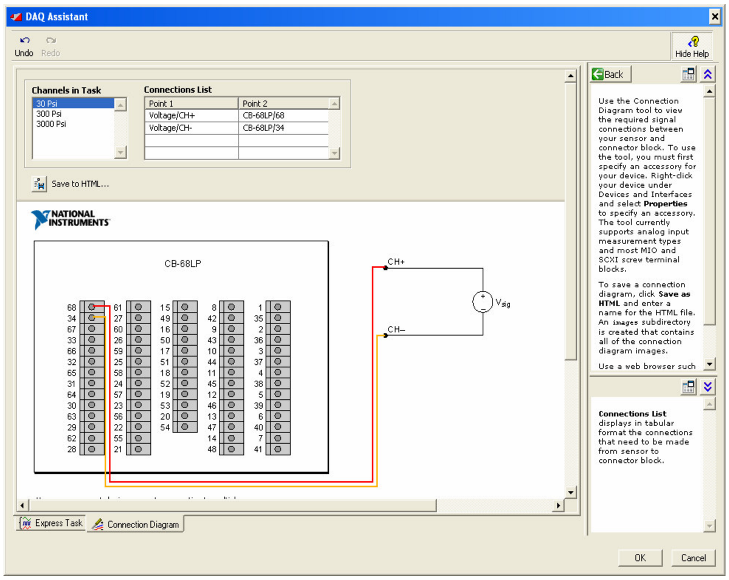
Most measurement devices are designed for measuring, or reading, voltage. Two common voltage measurements are DC and AC.

DC voltages are useful for measuring phenomena that change slowly with time, such as temperature, pressure, or strain.

AC voltages, on the other hand, are waveforms that constantly increase, decrease, and reverse polarity. Most powerlines deliver AC voltage.

This is the list of virtual channels. Right-click a virtual channel to rename it, change the physical channel associated with it, or remove it from the list.

By clicking the palette "Connection Diagram", the signal flow can be seen. The next screen shows the connection for the pressure signal up to 30 Psi, which means the inputs 68 and 34. See next screen.



Channels in Task
30 Psi
300 Psi
3000 Psi

Connections List

Point 1	Point 2
Voltage/CH+	CB-68LP/68
Voltage/CH-	CB-68LP/34

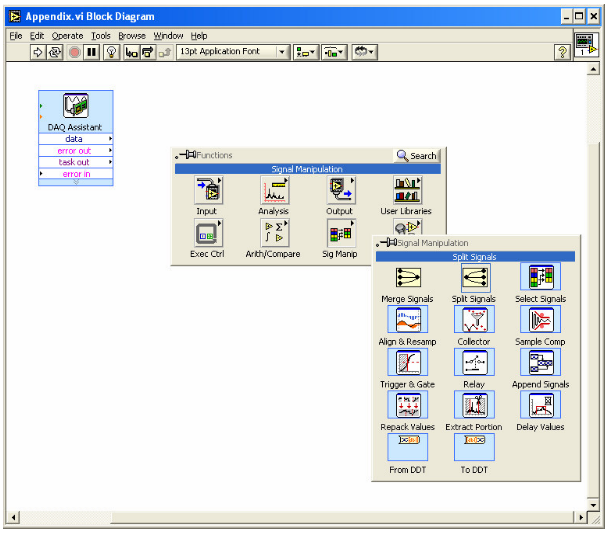
Use the Connection Diagram tool to view the required signal connections between your sensor and connector block. To use the tool, you must first specify an accessory for your device. Right-click your device under Devices and Interfaces and select **Properties** to specify an accessory. The tool currently supports analog input measurement types and most MIO and SCXI screw terminal blocks.

To save a connection diagram, click **Save as HTML** and enter a name for the HTML file. An `images` subdirectory is created that contains all of the connection diagram images.

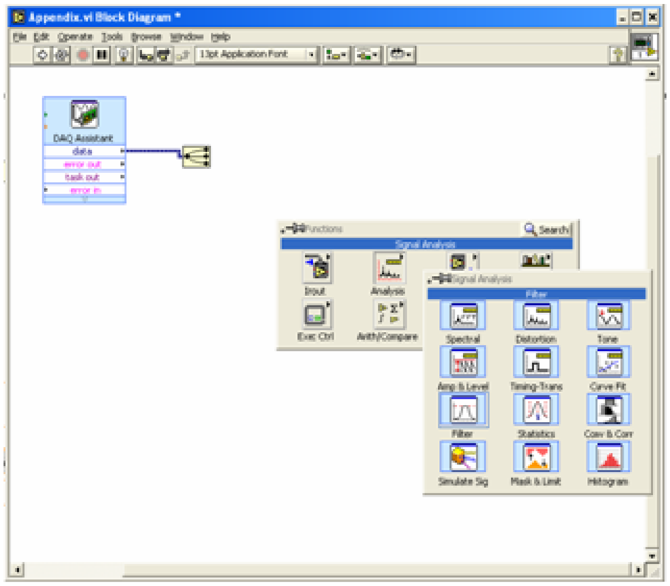
Use a web browser such

Connections List displays in tabular format the connections that need to be made from sensor to connector block.

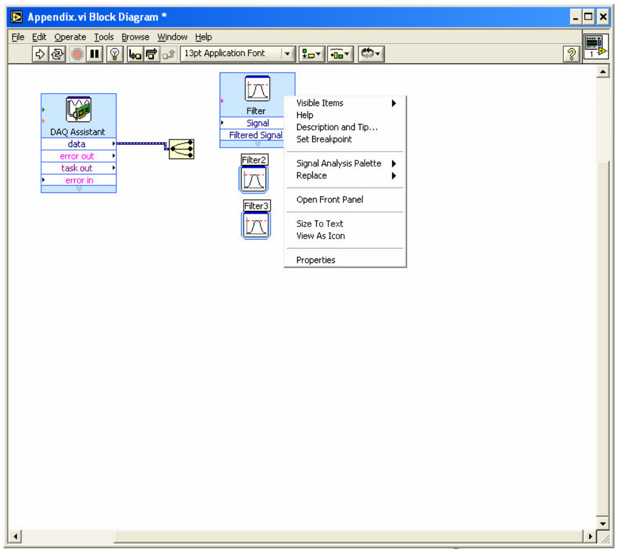
Going back to **Block Diagram** window, right click and select "Signal Manipulate" and then select the command "Split Signals", see the next screen.



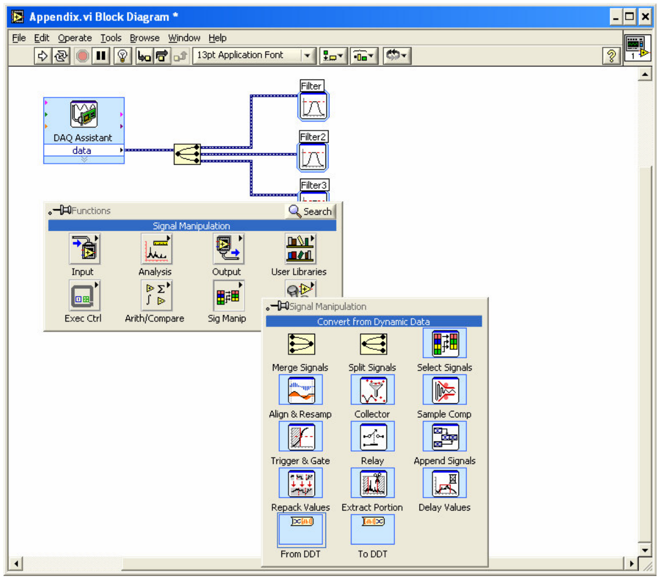
Drag the icon "Split Signal" next to the "DAQ Assistant", expand the icon up to the exact number of signals, in this case three pressure signals are coming from the set up; link the data box with the "Split Signal". Now, right click and select "Analysis" icon and "filter" command; see next screen.



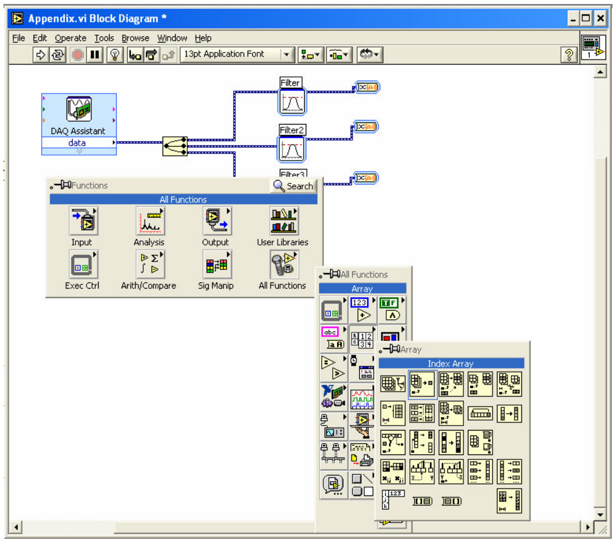
Drag three “Filter” icons corresponding to the three pressure signals and right clicking on each click on properties and select the option “smoothing” and “View as Icon” to become them smaller. See next screen.



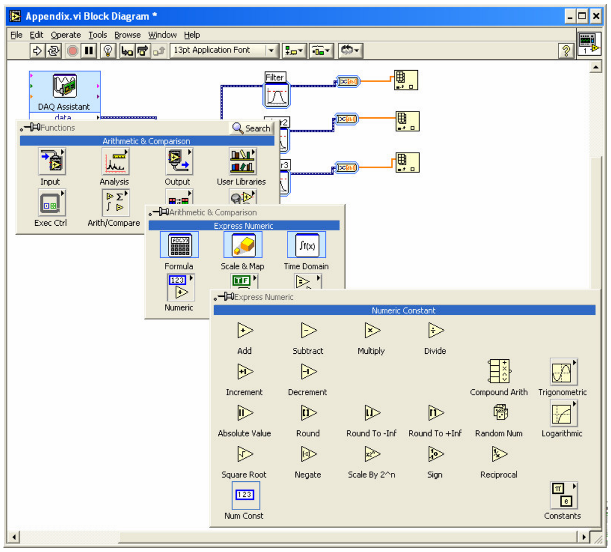
Link “Filter” icons with the “Split Signal” icon. Next, by right clicking, select “Signal Manipulate” and then select “From DOT” to convert from dynamic data.



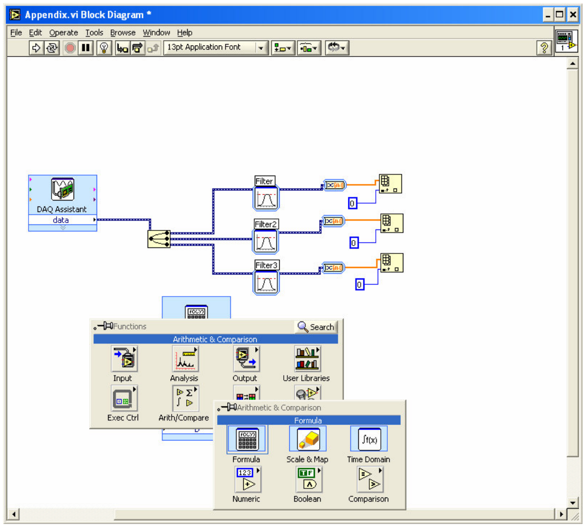
Drag the icons “From DOT” put them and link them next to the “Filter” icon. Next step is right click on Block diagram window, click on “All Tools” select “Array” and “Index Array”; see next screen.



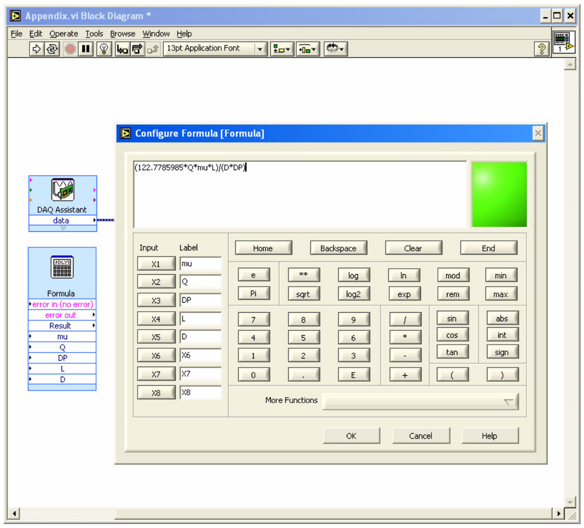
Drag “Index Array” icons and link them with the icons “From DOT”. Now right click on **Block Diagram** window, select the icon “Arith/Compare” next select “Numeric” and finally select “Numeric Constant”, see the next screen.



Drag three “numeric Constant” icons and link them with the icons “Index Array”. Next, to create the graph showing the calculated permeability, right click and select “Arith/Compare” and select “Formula”, see next screen.



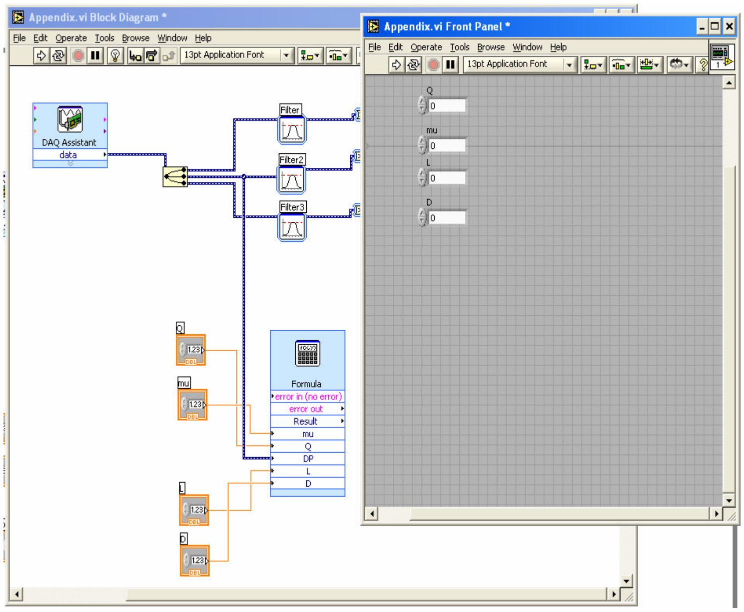
Drag the icon “Formula” and double click on it, it displays the windows in which the variables involved in the calculation are defined and the equation is written. By labeling each variable, automatically they become available to put into the equation. See next screen.



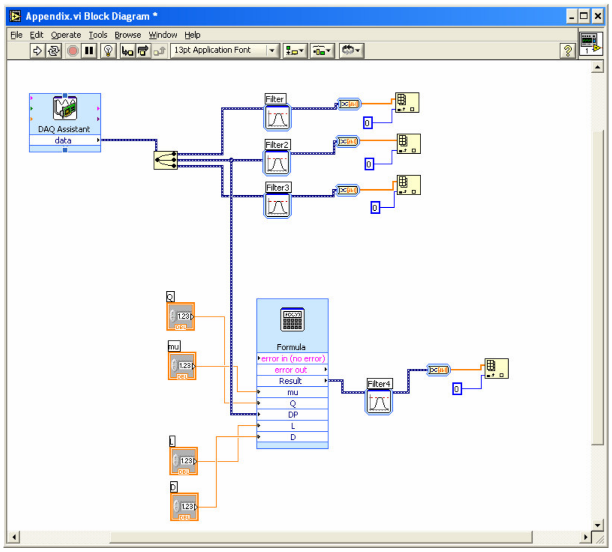
```
(122.7785985*Q*mu*L)/(D*DP)
```

Input	Label
X1	mu
X2	Q
X3	DP
X4	L
X5	D
X6	X6
X7	X7
X8	X8

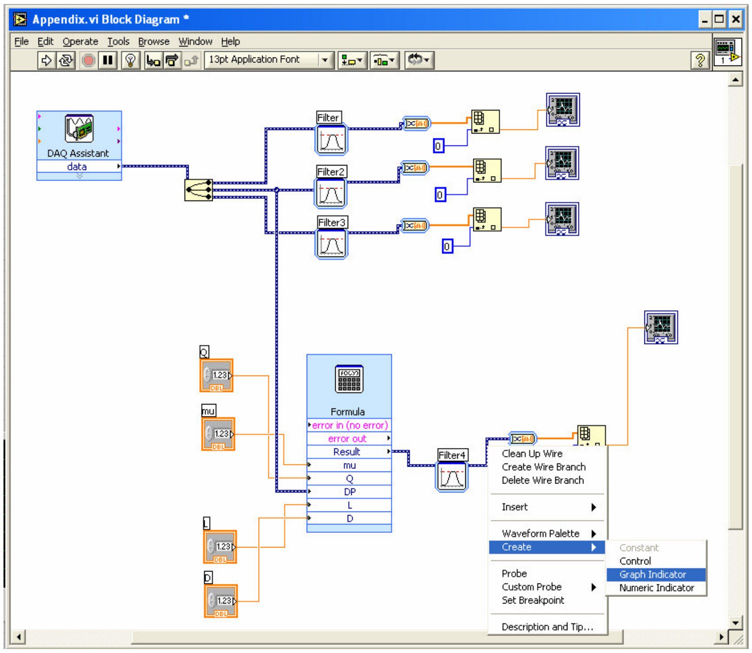
Once written the equation into the command “formula” and defined the variables, right click on each input, that is Q, L, D, and mu, and click **create control**, they automatically will appear linked to each input and in the **Front Panel** view as well; DP is taken from the differential pressure signal specifically chosen for each experiment, that means 0-30 psi, 0-300 psi, or 0-300 psi depending the conditions of the core sample and the measurements given during the core-flooding. See next screen.



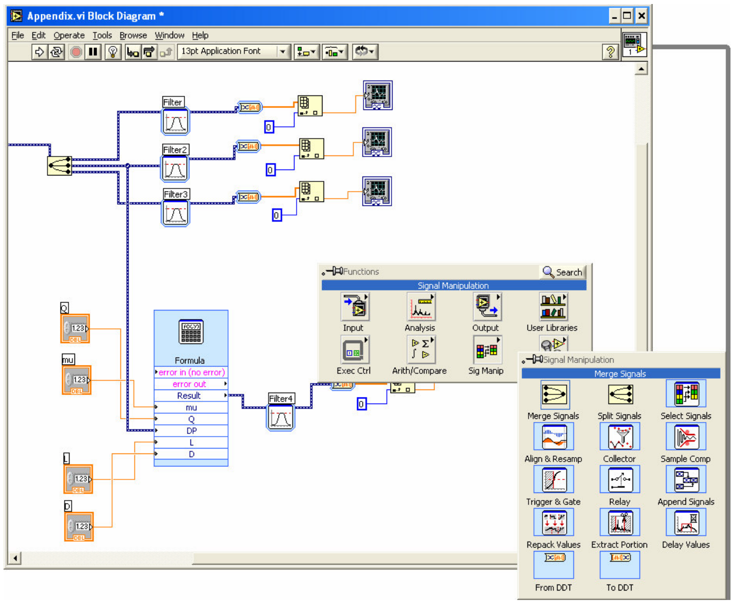
The result form calculated permeability is going to be filtered and put in same type of array previously described for the pressure signals, see next screen.



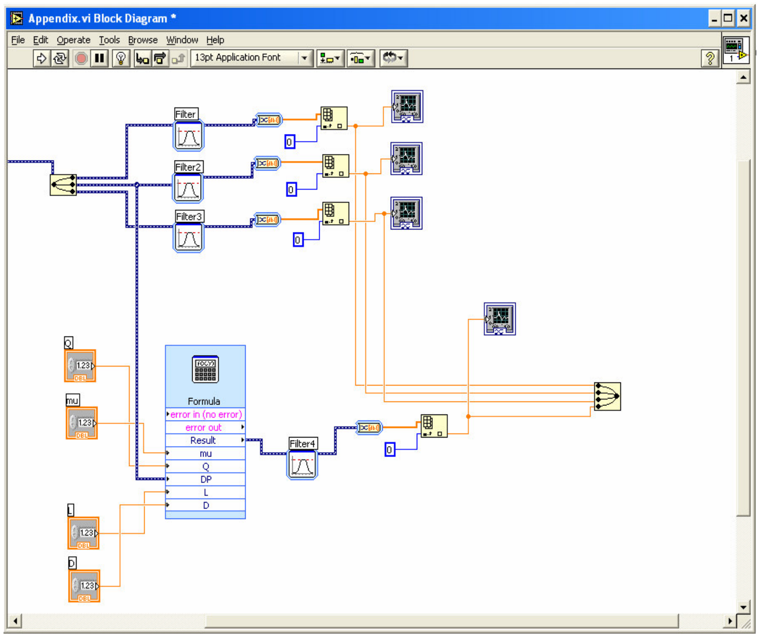
By right clicking, create "Graph Indicator" the wave form charts appear and they must be linked to signals from "Index Array"; see next figure. The wave form charts automatically display in the front panel view and they may be relocated as convenience by the users.



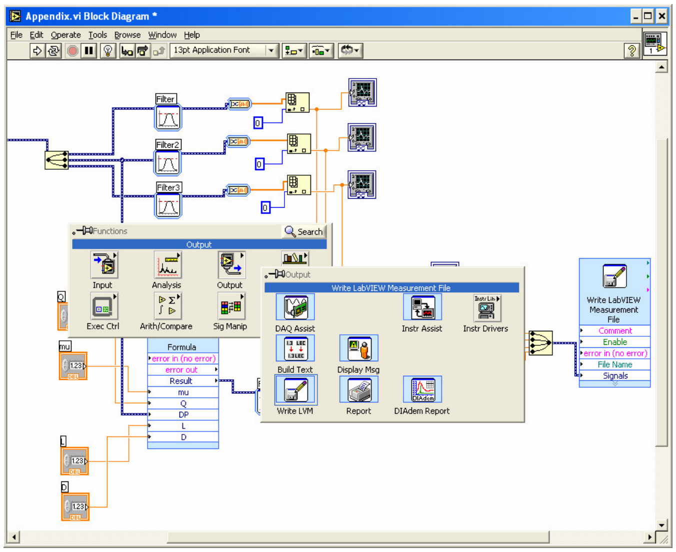
Right click and select “Signal manipulate” and then select “Merge Signals” to get the appropriate icon, see next screen.

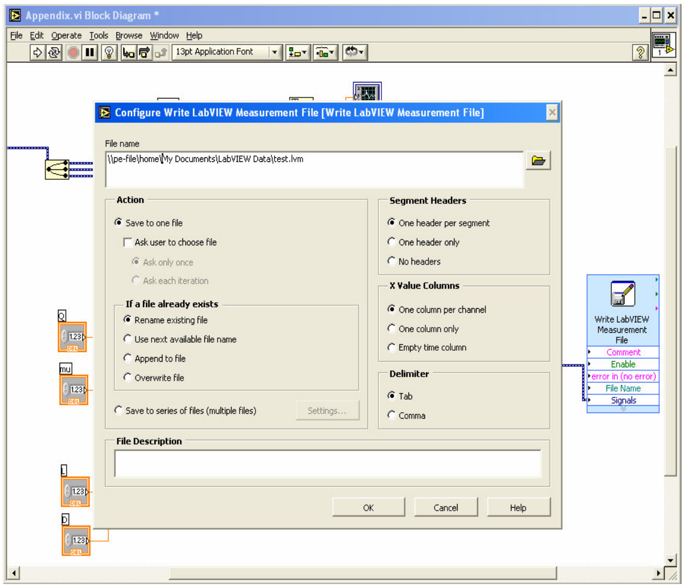


The four signals, three pressure and the calculated permeability, are merged to create the path to save the data in a defined file, see next screen.



By right clicking, select “Output” and select “Write LVM” to display the command to send the data to a file, this path may be changed by double clicking and selecting the new route easily.





Configure Write LabVIEW Measurement File [Write LabVIEW Measurement File]

File name: \\pe-file\home\My Documents\LabVIEW Data\test.lvm

Action
- ● Save to one file
  - ☐ Ask user to choose file
    - ● Ask only once
    - ○ Ask each iteration
- If a file already exists
  - ● Rename existing file
  - ○ Use next available file name
  - ○ Append to file
  - ○ Overwrite file
- ○ Save to series of files (multiple files) Settings...

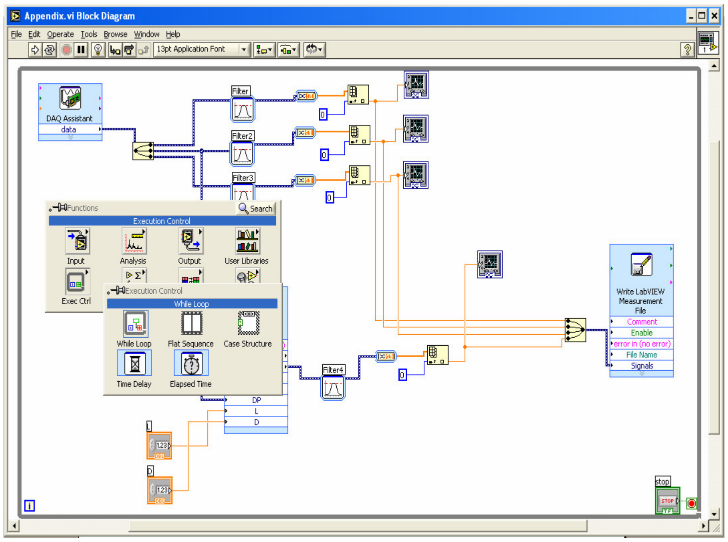
Segment Headers
- ● One header per segment
- ○ One header only
- ○ No headers

X Value Columns
- ● One column per channel
- ○ One column only
- ○ Empty time column

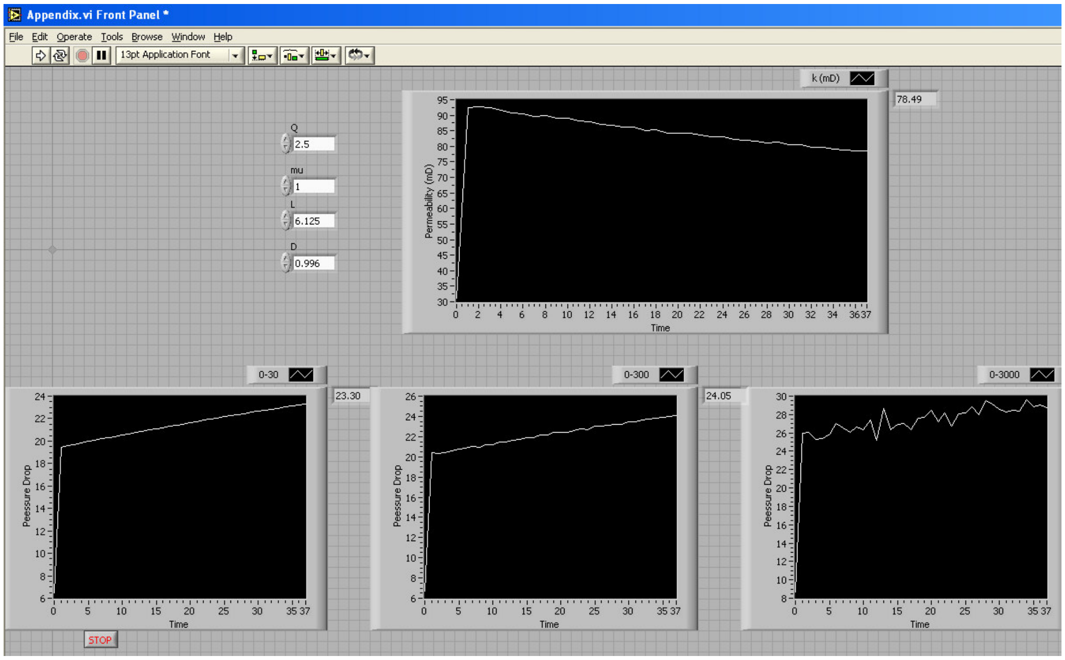
Delimiter
- ● Tab
- ○ Comma

File Description

Finally right click, select “Exec Control” and then select “While Loop” to create the limit for the program and select all the commands previously generated. See the final block diagram showing the command, and the final appearance of the front panel.



To start running the program first the values for core length, core diameter, flow rate and viscosity must be entered in the appropriate "Control" cells displayed on front panel. Once defined and connected the pressure gauge to be used for calculating permeability, defined the file where to store the data, click on the run button to start acquiring the data.



Q 2.5
mu 1
L 6.125
D 0.996
k (mD) 78.49
0-30 23.30
0-300 24.05

VITA

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