**U.S. Department of Energy**

**Small Business Innovation Research Program**

**Final Project Report**

# Acoustic Energy: An Innovative Technology for Stimulating Oil Wells

**DOE Contract No. DE-FG02-01ER83336**

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## FOREWORD

This report was prepared as the final deliverable of a Small Business Innovation Research Phase II project funded by the U.S. Department of Energy. This research project, as proposed, consisted of a total of ten tasks. This final report contains an account of all activities conducted for the project along with recommendations for additional work necessary to refine and further demonstrate the technology before full commercialization can begin. In addition to the work conducted under the Phase II Project, this report also contains a discussion of the results obtained under Phase I that provided the proof-of-concept basis for the second phase of research and development. This final project report contains a complete and comprehensive description of all activities conducted under the project along with all data, interpretations, conclusions, and recommendations associated with the project.

## THE PROJECT TEAM

The Project Team consisted of a number of individuals from three private sector organizations and one educational institution. The lead organization was TechSavants, Inc. (TSI), a small business located in Wheaton, Illinois. Dr. Donald O. Johnson, President, was the Principal Investigator of the project. Mr. Michael L. Wilkey, Vice President of Engineering, and Dr. Dorland E. Edgar, Vice President of Science and Technology, also worked on several aspects of the project.

Furness-Newburge, Inc. (FNI) is a small business that is located in Versailles, Kentucky. FNI has collaborated with TechSavants on several other projects involving sonication. Mr. James C. Furness, Jr., President, and Dr. P. David Paulsen, Vice President and Technical Director, and Mr. Michael Watts, Senior Fabricator and Field Technician, all provided their expertise on several of the project tasks.

Dr. Robert W. Peters, Professor, Department of Civil and Environmental Engineering, University of Alabama at Birmingham (UAB), was instrumental in overseeing most of the laboratory tests that were conducted during the project at UAB facilities. Dr. Peters has collaborated successfully with both TSI and FNI on other R&D projects focused on energy and environmental issues.

A third entity was added to the project team at the beginning of second phase of the project. Mr. Wayne Roberts, Vice President of Armmco (Allied Resource Management and Marketing), Bakersfield, California was asked to join the project team because of his extensive experience within the petroleum industry. He provided expertise in conducting some of the economic analyses and commercialization considerations as well as other important input.

Contact information for the Project Team members is given below.


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## EXECUTIVE SUMMARY

The objective of this investigation was to demonstrate the effectiveness and viability of sonication in reducing the viscosity of heavy crude oils. Sonication is the use of acoustic or sound energy to produce physical and/or chemical changes in materials, usually fluids. The acoustic energy was generated by actuators or transducers containing magnetostrictive crystalline material that rapidly changes shape in the presence of a magnetic field. In this application, the magnetic field is created within the actuator by an external electrical power supply. When the magnetic field is cycled up to several thousands of times per minute, the actuator converts the electrical energy into mechanical energy that, in turn, is converted to sound energy. A "horn" is attached to the actuator to transmit the acoustic energy into the surrounding fluid medium.

The project was conducted in two phases. The goal of the first project phase was to demonstrate a proof of concept for the project objective. During this first phase of this study, batch tests of three commercially-available, single-weight oils (30-, 90-, and 120-wt) were performed in the laboratory. Initially, tests using all three oils were performed to determine the amount and rate of viscosity reduction when heated. These data were used subsequently to separate empirically the viscosity changes due to heat from those observed during sonication, which also tends to add some heat during the process. Large beakers containing oil samples were sonicated at various frequencies (1.8, 3.1, 6.8, and 13.1 kHz), using three different horn designs (fin spacing of 0.25 in/6.35 mm, 0.75 in/19.1 mm, and 1.05 in/31.8 mm). All individual tests were performed for 30 minutes or less. Oil samples were collected at five-minute increments during each test and the temperature and viscosity of each were measured. Viscosity (dynamic viscosity expressed in units of centipoises, cP) was determined using dip viscosity cups.

Several observations and conclusions were made from the results of the first phase of experiments. These include the following:

1) In general, the lower the acoustic frequency, the greater the efficiency in reducing the viscosity of the oils.

2) There appears to be somewhat more error and therefore less confidence in the results obtained with the less-viscous, 30-wt. oil than with the other two oils. This is due primarily to the method of viscosity determination employed.

3) Of the three horn designs with different horn spacings that were evaluated, the horn design with medium spacing generally provided greater viscosity reductions than either the small or large spacing.

4) Sonication treatment of the three oils resulted in reductions in viscosity that ranged from a low of 31% to a high of 75%.

5) After sonication treatment, when the oil samples were allowed to equilibrate to room temperature, the viscosity returned to approximately the pre-treatment values.

6) The results of the first phase of the project successfully demonstrated that sonication could reduce the viscosity of oils of differing viscosity, providing the proof of concept and the basis for Phase II of the project.

The goal of the second phase of the project was to demonstrate the ability of sonication to reduce the viscosity of three crude oils ranging from a light crude to a heavy crude. The experiments were designed to test this hypothesis and also to examine the benefits of two proprietary chemical additives used in conjunction with sonication to determine if they would enhance the sonication effects. Acoustic frequencies ranging from 800 Hz to 1.6 kHz were used in these tests. In addition, experiments were designed to evaluate acoustic horn design (1 in/2.5 cm fin spacing vs. 2 in/5 cm fin spacing), reduction in the input electrical power (normal power vs. 25% reduction), and the amounts and rates of viscosity change or recovery during a 30-day rest period following treatment. All individual experiments were conducted for a maximum of 120 minutes, with data points collected at 30-minute intervals beginning at zero minutes at the beginning of the test. Viscosity was measured with a digital viscometer that measures the viscous drag of rotating spindles immersed in the oil samples.

A reactor chamber was designed for flow-through operation with a capacity of one gallon (3.8 liters). Acoustic energy was added by way of three actuators with horns, all operating in the same plane within the reactor chamber. Two of the actuators were inserted into the chamber in opposing, horizontal, parallel positions with the horns facing each other. A third actuator was inserted vertically, perpendicular to the other two. Water was circulated through portions of the apparatus to keep the actuators cool and to eliminate the effects of heat within the oil chamber. The reaction chamber apparatus was designed and manufactured by the project team, and all components of the experimental system were thoroughly tested before the experiments began. The three crude oils selected for use in the testing program were: 1) a heavy crude from California with a viscosity of approximately 65,000 cP (API gravity about 12°), 2) a crude from Alabama with a significant water content and a viscosity of approximately 6,000 cP (API gravity about 22°), and 3) a light crude from the Middle East with a viscosity of approximately 700 cP (API gravity about 32°).

The principal observations and conclusions derived from the second project phase include the following:

1) The application of acoustic energy (sonication) was demonstrated to significantly reduce the viscosity of crude oils under laboratory conditions. The amount of viscosity reduction resulting from sonication is greater for more viscous, heavy crude oils than it is for less viscous, light crude oils.

2) Test results showed that after being heated to nearly 100°C, the "cooling" viscosity values were somewhat less than the "heating" cycle values at the same temperature. Reductions in viscosity due to heating were not sustained following treatment to the extent that post-sonication reductions were sustained.

3) The maximum viscosity reductions in Oils 1, 2, and 3 due to sonication were 43%, 76%, and 6%, respectively. The large reduction in Oil 2 was likely due to the large but variable amount of water present in samples of this crude oil; samples associated with larger viscosity reductions often exhibited a definite water separation layer follow the tests. Maximum reductions on the order of 23% were measured when this separation was not observed.

4) The best results obtained with the flow-through test equipment were with two actuators operating at different frequencies, aligned in parallel and adding energy to the oil from opposite sides. Better results were obtained when the two actuators were operated at 0.8 and 1.2 kHz or at 0.8 and 1.6 kHz than when other frequencies were used.

5) Of the two horn designs evaluated during the experiments, the design using the narrow fin spacing (1 in/2.5 cm) produced somewhat better results than did the design using the wider spacing. However, in most cases the differences were relatively small.

6) It was observed that reducing the input power by 25% had very little effect on the ability of sonication to alter crude oil viscosity.

7) The chemical additives used in the investigation were employed in concentrations ranging from 13% to 17% by volume. When added to the three oils, the range of viscosity reduction was from 37% to a maximum of 94% with the largest reductions being facilitated by the abundant water present Oil 2. If the Oil 2 results are not considered, the maximum reduction was 73%.

8) When crude oil samples containing the chemical additives were sonicated, the viscosities were reduced further. Final viscosity reductions at the conclusion of these tests were greater than those attained under comparable conditions by either sonication or the addition of the chemical mixes used alone. Thus, the effects of the additives and sonication are complementary in that one enhances the viscosity-reducing abilities of the other.

9) The viscosity of the crude oils tends to recover with time following sonication treatment. However, in no case did the viscosity return to as much as 50% of the pre-treatment value during a period of 30 days following treatment. Therefore, more than half of the viscosity reduction was maintained for a month without additional treatment.

10) Preliminary and very conservative estimates were made of the possible applications, market potential, and economic value of the implementation of a mature sonication technology within the petroleum industry. If all of these prospective applications were fully developed and implemented, it is conservatively estimated that several billion barrels of oil could potentially be upgraded or produced annually generating between $400 million and possibly more than $20 billion in annual revenue.

In terms of the project goals, the results that were obtained successfully demonstrated that sonication can effectively reduce the viscosity of crude oils having a broad range of viscosity/API gravity values. The project also showed that the use of chemical additives in conjunction with sonication can significantly enhance viscosity reduction. However, this project was the first step in the process leading to full-scale integration of the technology within the petroleum industry. Several specific recommendations are made for follow-on work that is required before the technology can be considered mature and ready for full commercial implementation.

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## FIGURES

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## APPENDICES

## ACKNOWLEDGEMENTS

The Project Team would like to express its appreciation and gratitude to the U.S. Department of Energy (DOE), Small Business Innovation Research Program (SBIR) for providing the funding for this project (DOE Contract No. DE-FG02-01ER83336). Mr. Chandra M. Nautiyal, a member of the DOE's National Energy Technology Laboratory (NETL) in Tulsa, Oklahoma, was the Project Officer. Mr. Nautiyal's guidance, technical support, encouragement, and patience in seeing the project through to completion are sincerely appreciated. In addition, Mr. John P. Motz and Mr. Michael D. Hill, both on the staff of DOE's Chicago Operations Office (DOE-CH) provided valuable and important contractual and administrative guidance throughout the duration of the project. Their individual and collective support was very much appreciated. The project team thanks all three of these individuals for being knowledgeable resources and for their consistent willingness to provide assistance and insight whenever it was requested.

# 1 INTRODUCTION

As witnessed by the recent upward spike of gasoline pump prices, jet fuel, diesel fuel, and the price of crude oil on the international market, the availability and pricing of petroleum and petroleum products clearly have a dramatic impact on our daily lives and on our nation's economy. The consistent and affordable supply of energy is the foundation of modern society and it will only become more critical as developing nations continue to develop a larger appetite for energy. At present, and for the immediate future, fossil fuels provide, and will continue to provide, the majority of the world's energy supply. In order to meet these ever increasing demands, additional sources and/or supplies of petroleum will be required. The research described in this report is focused on one technology that can help to meet these critical needs.

## 1.1 Problem Statement

### 1.1.1 Heavy Oil

One of the problems facing the oil industry is the cost-effective production of heavy crude oil. The term "heavy crude" refers to the viscosity of the oil, i.e., the internal friction within the oil due to cohesive forces between the oil molecules, which result in a resistance of the oil to flow. A significant portion of the world's reserves are heavy crude; Venezuela, for example, in the Orinoco Belt, has an estimated 1.2 trillion barrels of thick, heavy, costly-to-produce crude oil. Some additional key areas with large quantities of heavy crude are Mexico, the North Sea, Canada, and Kuwait. The U.S. also has a considerable portion of its oil reserves as heavy crude, primarily in California. Since current and past practices have concentrated on developing more easily produced lighter crude oil reserves, producers in the future will be dealing with an increasing percentage of heavier crudes.

In California, the bulk of the oil underlying the San Joaquin Valley, especially in Kern County's Midway-Sunset and Kern River fields, is heavy crude. In addition, the coastal areas from the Santa Maria basin to Oxnard appear to have extensive heavy crude reserves.

To produce these reserves, the oil must be made to flow, i.e., the viscosity must be changed. The American Petroleum Institute (API) has developed a numerical standard for expressing the specific weight of crude oil; the higher the specific gravity of an oil, the lower the API gravity number. Heavy crude has an API gravity of 20 or less, crude oil has an API gravity of 20-40.1, and light crude has an API gravity greater than 40.1.

### 1.1.2 Augmented Petroleum Recovery Methods

To facilitate pumping and enhance resource recovery, it is necessary to raise the API gravity (lower the viscosity) so that the oil will flow allowing it to be pumped to the ground surface. A number of approaches have been used for this purpose. Probably the most common and least expensive practice that has been used since the 1880s is waterflooding, where water is

injected into the production zone through injection wells. The injection system is designed so that the water increases formation pressure and "pushes" the oil to the production wells. In a similar approach, gas (e.g. air, natural gas, liquefied petroleum gases, nitrogen, carbon dioxide) can be injected for the same purpose. Another approach involves the injection of chemicals (chemical flooding) such as polymers, surfactants, and other chemicals to alter the physical properties of the petroleum and other formation fluids and thereby increase the mobility of the petroleum through the geologic medium. Chemical flooding has proven to be successful, but the economics of the process are a consideration, particularly if the resulting increase in production is small. Another option is *in situ* combustion, where a fire is started within the oil-bearing unit and air is injected to sustain the combustion process. The heat generated from the combustion will vaporize formation water present and generate steam and the heat will facilitate a viscosity reduction in the petroleum. In some cases, water can also be injected along with the air to increase the rate of steam formation. The most utilized approach to reduce crude oil viscosity *in situ* is the addition of heat through the injection of hot water, steam, or superheated steam with steam (steamflooding or cyclic steaming) being the most common choice. An informative discussion of augmented petroleum production approaches can be found on the Society of Petroleum Engineers website (SPE, 2005).

Although the process for making steam and injecting it into the well is simple, the cost components of the process have risen – some dramatically – in the past few years. In California, increased demand for water has raised its cost and, in many areas, constrained its availability. Natural gas costs in California's deregulated energy market have soared in response to a whole series of issues, including pass-through cost restrictions, air quality and emissions issues, and availability. Finally, California has become very concerned about water-contaminant problems and is taking a much harder look at petroleum industry produced-waters disposal. As a result of these circumstances, the increase in the cost to produce steam has raised the cost of produced oil and impacted the position of the industry to compete in the world market. If the industry continues to move more of its operations overseas, the domestic petroleum industry will decline, jobs will be lost and the U.S. economy will suffer.

Technology developments are needed to assist the domestic petroleum industry in addressing the heavy crude production problem. One development needed is a more efficient and economical technology for reducing the viscosity of oil to allow the development and production of more of the world's heavy crude reserves. Sonication, the use of acoustic (sound) energy to elicit physical and/or chemical changes in a fluid or a solid, has the potential to be a breakthrough technology in this regard.

## 1.2 Acoustic Technology Background

### 1.2.1 R & D History

The physics of acoustics and the science of sonication have been studied for more than 200 years. Early experimentalists used tuning forks (frequency) to show how acoustic/sound energy could cause ripples on the surface of water, and they also noted the extreme agitation caused when a tuning fork came in contact with the water. By the 1840's, materials had been developed which allowed the conversion of electrical and electromagnetic energy into mechanical energy. In 1842, James Joule discovered that an applied magnetic field (coil) could change the length of a bar of iron by "constricting" it. This magnetostrictive effect, named the Joule effect, is measurable and can be repeated virtually without fatigue in the metal. The physical dimension changes in such a bar of magnetostrictive material can be transformed into sound energy. Magnetostriction became the basis for numerous acoustical devices, including naval sonars. The materials favored in magnetostrictive devices, mainly nickel, became somewhat scarce during the period of the First World War due to demand for nickel for use in gun barrels and barrel liners. There was substantial incentive to develop other materials for transduction and these efforts led to investigations into piezoelectric (pressure-electric) materials and effects.

In a piezoelectric material, the application of a force or stress results in the development of an electrical charge in the material. Conversely, the application of a charge to the same material will result in a change in physical dimensions (strain) of the object. This movement can be converted from mechanical to sound energy. The development of piezoelectric ceramic sonar and the use of nickel as an energy converting material (transducer) reached their peak during World War II and for the ensuing 30 years, but eventually the physical limits of these materials were reached.

In the early 1970's, scientists at the Naval Ordnance Laboratories (now the Naval Surface Warfare Center) began experimenting with using the rare earth metals in magnetostrictive devices. Certain metal alloys of the lanthanide series showed tremendous potential for extremely high levels of magnetostriction. When a magnetostrictive rod is activated by a magnetic field produced by an alternating current, the oscillations (250-400 times a second) create an intense acoustic energy pressure wave that can be transmitted through a material.

Following the declassification of various sonication technology materials and data by the military in the early 1990's, considerable scientific and engineering innovations have been made in the application of acoustic energy to systems in order to affect physical and/or chemical changes in system components. Equipment and materials have evolved to the point that much larger amounts of energy can be generated for sonication purposes permitting larger and more efficient applications for a variety of different uses.

The power available in today's generation of magnetostrictive sonication materials and equipment – 1,000-6,000 watts – dwarfs what was being used in the laboratory only a few years ago, i.e., units with 350-500 watts of power. The tremendous increase in power, plus the much smaller size of sonication equipment, allows users to apply sonication technology to a number of situations at power levels previously unavailable. Thus, the technology can be used in new applications in various industrial sectors.

### 1.2.2 Physical Basis of the Technology

The physics of sound and sonication are fairly well known. Sound is a mechanical wave that consists of a pressure disturbance transmitted by means of molecular collisions in a fluid (gas or liquid). The term sonication refers to the application of sound waves (acoustic energy) to a system and this energy is transmitted through a liquid medium (water, oil, etc.) as a wave of alternating cycles of increasing and decreasing pressure. An analogy to visualize the movement of sound through a fluid is that of a stone tossed into a pond or pool of quiet, standing water. Waves radiate outward in all directions from the point where the stone hit the water (Figure 1). These are surface waves consisting of two parts – a peak or elevated portion and a trough or depressed portion. If a cork or other floating object were in the water as a wave passed, it would move up and down (perpendicular to the direction of wave motion) as each peak and trough passes its location. These types of waves are termed transverse waves where the particles of the transmitting medium move perpendicular to the wave direction; light waves are transmitted in this form.

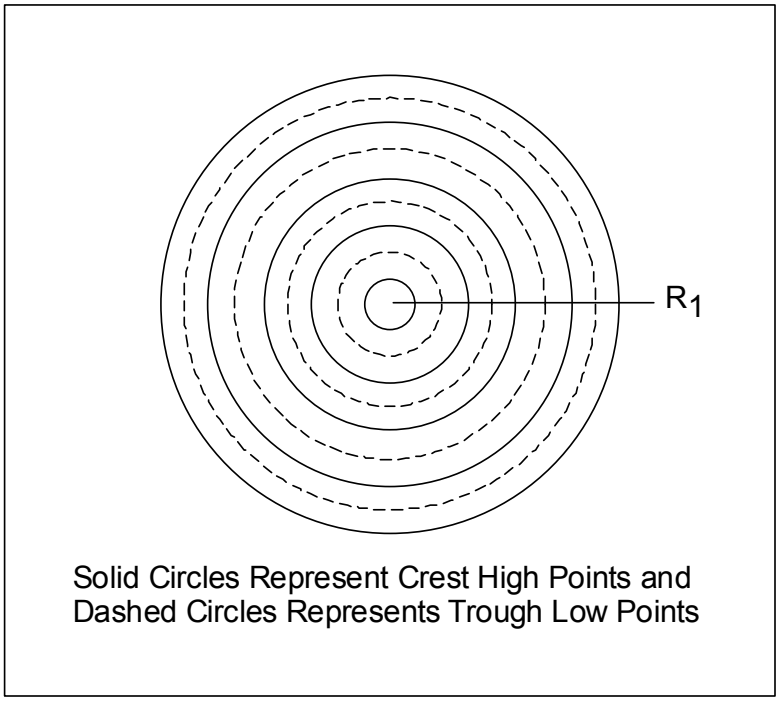


**Figure 1 Illustration of Surface Waves on Water**

Figure 1 is drawn from a perspective of being above the liquid surface looking down at the waves. If a cross-section of this system were observed along any radius from the center outward (for example $R_1$ in the above drawing), it would look like the drawing in Figure 2. This illustration shows a cross-section of a single wave with the wavelength and amplitude labeled. Here the water surface is shown as a plane where the pressure is atmospheric ($P_a$).

Beneath the liquid surface, within the liquid itself, sound waves take on a longitudinal (compressional) form meaning that the particle motion is in the direction of wave propagation. Compression cycles exert a positive pressure on the liquid, pushing molecules closer together, while expansion cycles exert a negative pressure, pulling molecules away (rarefaction) from each other. These conditions are represented by the spacing of the vertical lines and the horizontal arrows in Figure 2. The molecules tend to be pulled apart (pressure decreases) as the trough of a wave passes and pushed closer together or compressed (pressure increases) as a wave crest passes. Thus, within the fluid, the passage of a single wave of sound energy represents an alternating decrease and increase in pressure, which can be visualized to be like the sine wave representation of a surface wave shown here.

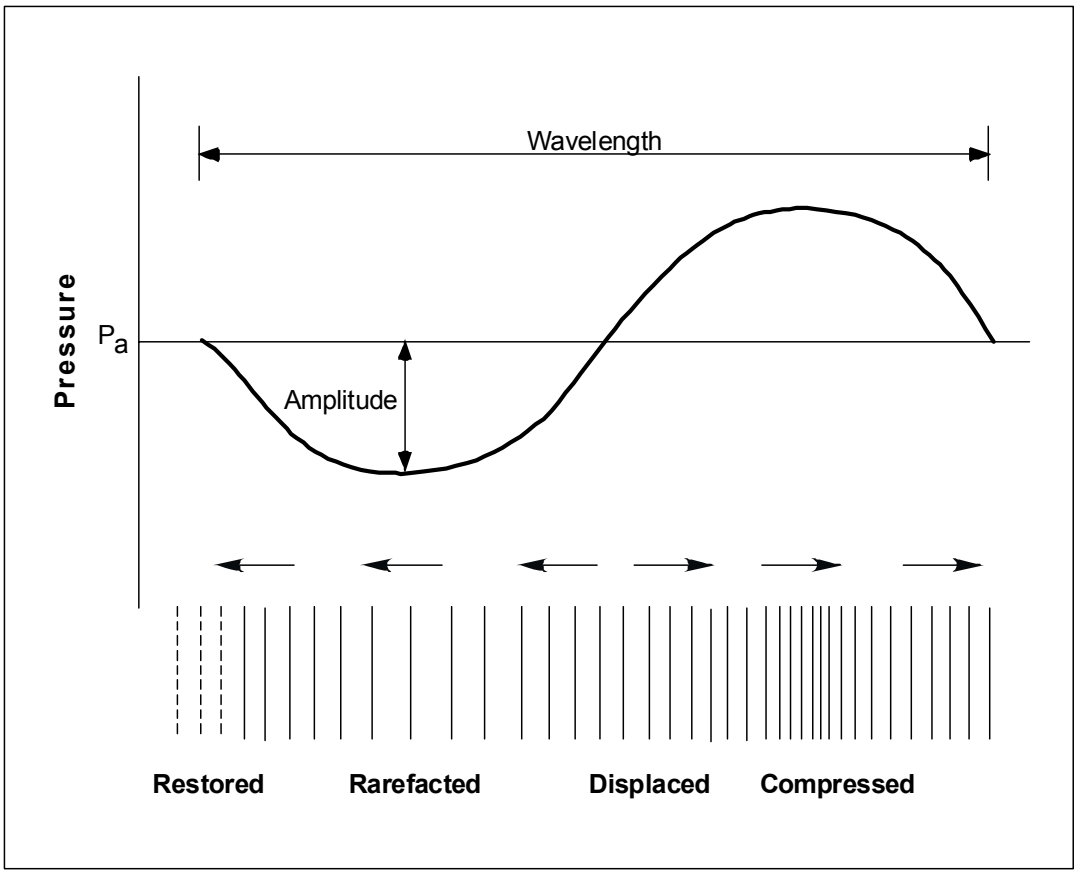


**Figure 2 Illustrations of a Single Sound Wave and the Alternating Increase and Decrease in Pressure**

The unit of measure of sound frequency is the Hertz (Hz), which is one cycle of compression and expansion or rarefaction (passage of one wavelength) in one second; a kilohertz (kHz) is one thousand cycles per second and a megahertz (MHz) is one million cycles per second. Where sound energy falls within the spectrum ranging from below the threshold for human hearing (16 Hz) to the upper level (18 kHz) is determined by the sound frequency. Ultrasound is defined as that sound above the threshold of hearing with frequencies between 20 kHz and up to 500 MHz. Sonochemistry, a rapidly growing area of research and technology development, refers to the discipline and phenomena of affecting chemical reactions by the application of sound waves (see Mason, 1999; Mason and Lorimer, 2002). Figure 3 illustrates the sonic spectrum and some applications of sound energy of various frequencies.

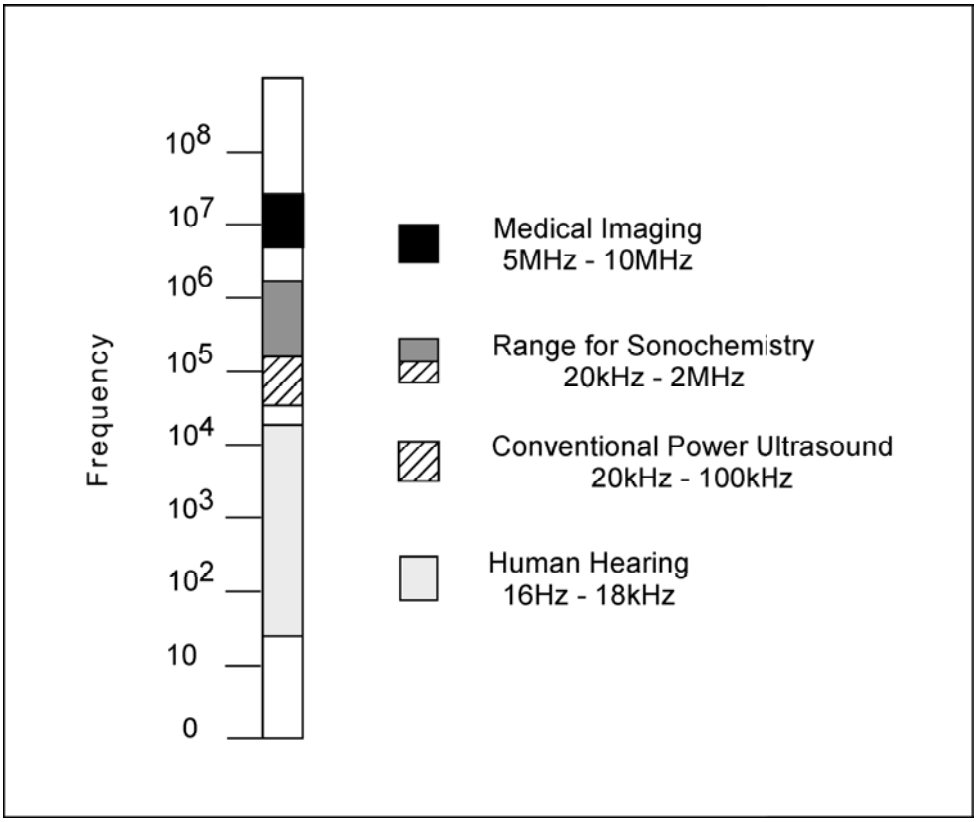


**Figure 3 Sound Frequencies**

When the amount of energy added to the system is increased, the amplitude of the sound waves will increase as the frequency (wavelength) is held constant. As this occurs, localized pressure in the sonicated liquid may drop below its vapor pressure during the rarefaction portion of individual sound waves (Figure 4). This will initiate the formation of microbubbles in the rarefaction zone when the liquid is locally vaporized and a bubble forms around the vapor pocket. These bubbles initially are very small, on the order of 1 μm ($1 \times 10^{-6}$m, 0.001mm). This phenomenon of bubble formation is called cavitation and is the basis for many of the physical and/or chemical changes that occur in the liquid medium during the sonication process. In

addition to the vaporization process due to pressure drops, the rarefaction or extension phase of the cycle causes molecules of the medium to pull apart when the negative pressures exceed the tensile strength of the material or the distance between the molecules exceeds the critical molecular distance necessary to hold the liquid intact. This forms cavities or voids in the liquid medium that produce additional bubbles during cavitation. During the alternating cycles of pressure increase and decrease, the microbubbles fluctuate in size, growing in rarefaction phases and shrinking in compression phases. Eventually, some of the individual bubbles grow to a critical size and then implode violently (collapse to zero size), releasing a large amount of localized energy (Figure 5).

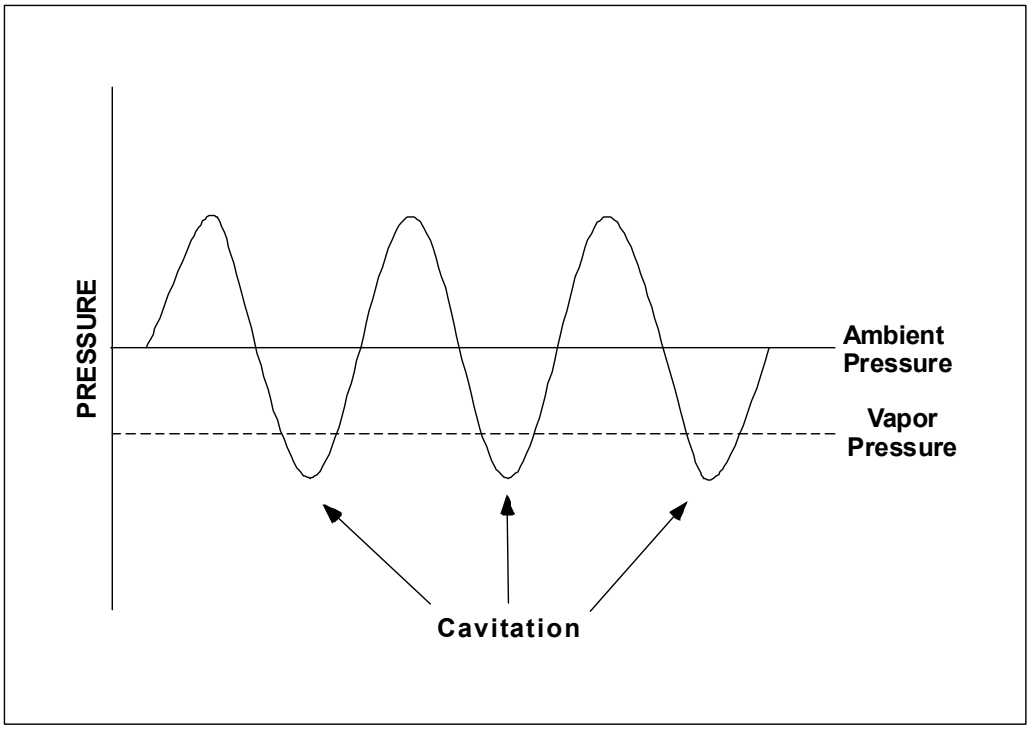


**Figure 4 Illustration of Pressure Drop Below Vapor Pressure of a Liquid Causing Cavitation**

Energy released when cavitation bubbles collapse occurs in three forms. Temperatures on the order of 5,000 $^{o}$K (8500 $^{o}$F) and pressures in excess of 1,000 atmospheres have been calculated to occur at the collapsing bubble interface during implosions (see Suslick, 1994). Furthermore, under some circumstances, light emissions also have been observed during sonication (sonoluminescence), which further indicates the release of intense energy from the cavitation process (Crum, Mason, Reisse, and Suslick, 1997; Beckett and Hua, 2001). It is also possible to generate strong, but small-scale shock waves within the sonicated fluid resulting from the sudden input/pulse of increased pressure when a bubble collapses. It must be remembered that all of these cavitation-related phenomena are on a very small scale and the energy dissipates very quickly in the immediate vicinity of the bubble. Consequently, the overall physical

properties (e.g. temperature) of the ambient fluid tend to remain relatively unchanged. However, the very large intensities of energy involved do have the capacity to produced dramatic, localized changes in the chemistry and physics of the sonicated medium (Mason and Lorimer, 2002; Mason and Peters, 2002).

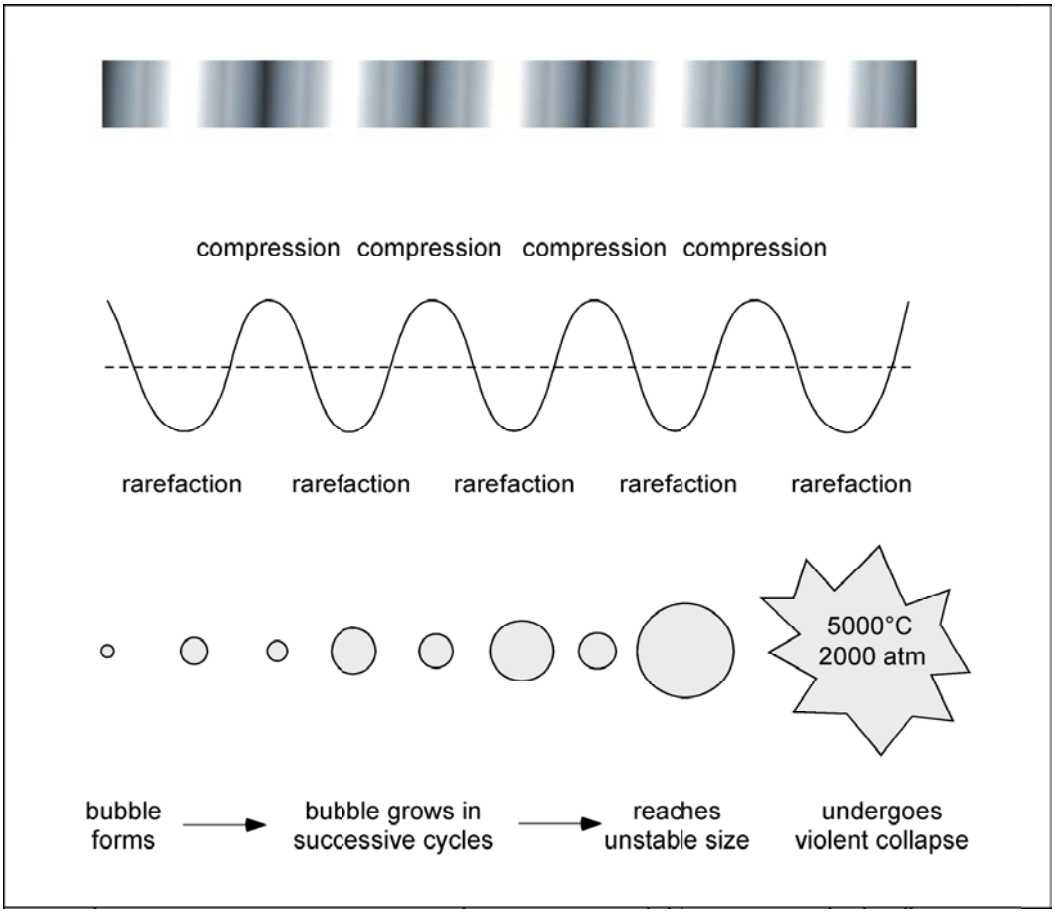


**Figure 5 Schematic Illustration of Bubble Growth and Collapse During Cavitation**

In water, the reactions within and adjacent to a collapsing bubble result in the formation of hydroxyl (•OH) and hydrogen (H•) radicals. Although these chemical species are extremely short-lived, they are very reactive and effective in destroying organic compounds contained within the water. The intensity of cavity implosion and the nature of the reactions involved can be controlled by process parameters such as the sonic frequency, sonic intensity (power per unit volume of liquid), static pressure, temperature, and the addition of reactive oxidants such as hydrogen peroxide ($H_2O_2$), ozone ($O_3$), and metal catalysts. Cavitation reactions supplemented by these additives produce an advanced oxidation system that has many potential environmental and industrial applications.

## 1.3 Applicability to the Problem

The field of sonochemistry, involving the effects of sound waves on chemical reactions and chemical processing, and the ability of sound waves to effect physical changes in materials have been a topics of investigation for more than 100 years. However, the levels of interest and corresponding number of scientific investigations into this complex subject have increased dramatically during the last 10-15 years. The number of applications of sonication technologies has also increased during this time and more will follow as the materials and equipment become more sophisticated with expanded capabilities. The project team believes that this technology s great promise in reducing the viscosity of heavy oil allowing more of this valuable resource to be recovered and used. Because of the ability of an acoustic/sonic device to add significant quantities of energy to fluids, it is logical that the process of sonication should be examined as a new technology capable of positively changing the physical properties, in particular the viscosity, of heavy crude to facilitate pumping from a reservoir at depth to the land surface. This would allow increased production from stripper wells and other wells producing heavy, high-viscosity petroleum. If successful, sonication would provide a viable alternative to existing heat, steam, and surfactant technologies with potentially large environmental and economic improvements. That premise is the basis for this investigation.

# 2 PROJECT PHASE I ACTIVITIES AND RESULTS

## 2.1 Technical Objectives

The objective of this initial phase of the project was to test and evaluate an integrated acoustic system under laboratory conditions to determine the ability of sonication to reduce the viscosity of oil. Parameters to be evaluated included acoustic frequency, power intensity, and treatment time. The specific technical objectives of these experiments were:

1. Conduct laboratory testing and evaluation to determine the ability of acoustic technology to reduce the viscosity of oils;
2. Perform a preliminary optimization of the acoustic technology through a series of laboratory experiments;
3. Conduct laboratory experiments on oils of varying viscosities; and
4. Develop a conceptual prototype design for a more advanced system that would lead to a downhole design and application.

## 2.2 Experimental Materials and Methods

### 2.2.1 Materials and Equipment

The Project Team decided that the initial Phase I testing to evaluate the ability of sonication to reduce the viscosity of oils would be conducted with commercially available motor oils. Three different single-weight oils were selected and procured: a 30-weight oil, a 90-weight oil, and a 140-weight oil. The initial sonication test apparatus employed was very simple in design. In its simplest form, the equipment necessary to generate acoustic/sonic energy and transfer it to a fluid consists of a power supply, wiring, an actuator/transducer to convert electrical energy into mechanical and acoustic energy, and a horn or similar device to transfer the energy into the fluid.

The power supply utilized in the laboratory experiments was a Titan Manual Oscillator MOS-01, with three different frequency output ranges with a possible output from a minimum of 20 Hz to a maximum of 20 kHz (Figure 6). This power supply unit has a maximum output of 1,000 watts and permits control of the voltage and current delivered to the actuator. Typically power is supplied at about 75-80% of maximum. The supply unit provides electrical power to the actuator by way of electrical wires.

The sonication system used in these experiments utilized two different transducers, one capable of operating at a lower frequency (<4 kHz) and a second that produces higher frequencies in the range of approximately 4 up to 20 kHz. Both transducers were manufactured by and purchased from Etrema Products, Inc. in Ames, Iowa. A photograph of one of the transducers is shown in Figure 7. Note in this photograph that the wires that attach to the power


**Figure 6  Power Supply Used in the Experiments**


**Figure 7  Actuator Used in the Experiments**

supply enter the actuator on the right, and the rod on the left end contains threads allowing horns to be easily attached. One of the objectives of the tests was to determine which acoustic frequencies had the most profound effect on oil viscosity. As part of this preliminary optimization process, different horn designs were developed and provided by Furness-Newburge, one of the project team members. The basic design consisted of a titanium horn with horizontal slotted fins (Figure 8). These were fabricated in the Furness-Newburge facilities from titanium bar stock. Due to the modular nature of the horn design, the number and spacing between the fins could be adjusted easily before a test. Three different horns were employed having fins with different spacing. The small spacing was 0.25 in (6.4 mm), the medium spacing was 0.75 in (19.1 mm), and the largest spacing was 1.25 in (31.8 mm).

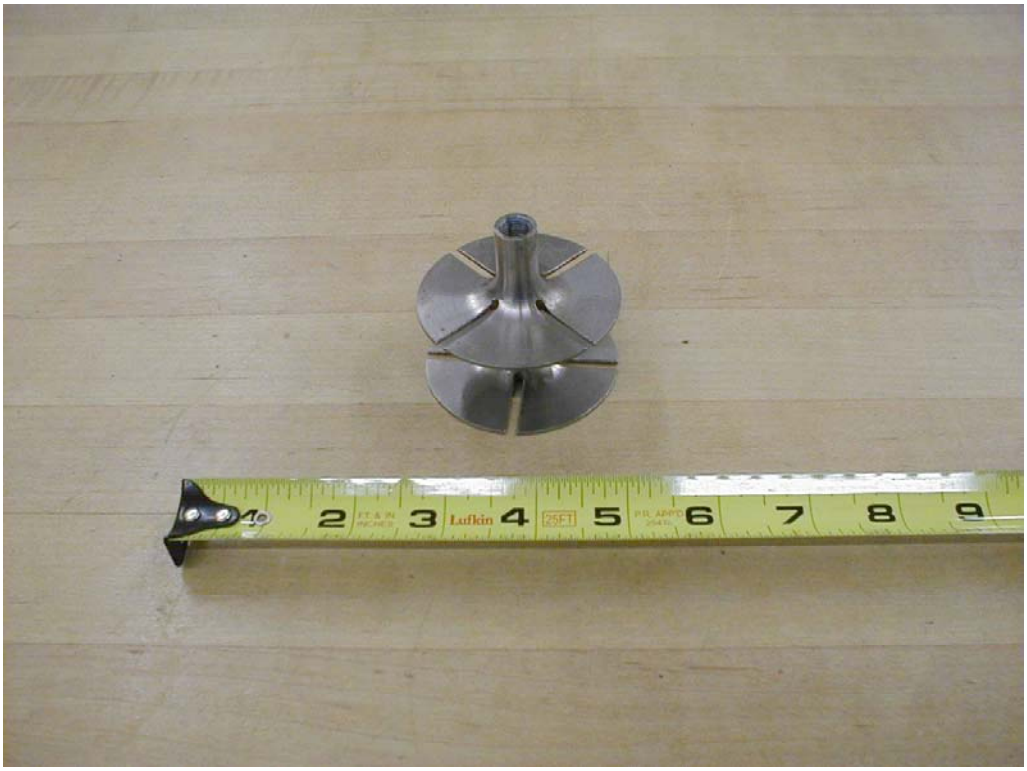


**Figure 8 Example of One Type of Acoustic Horn Used in the Experiments Showing Two Slotted Fins**

Viscosity is the measure of the internal molecular friction of a fluid exerted when layers of the fluid try to move past one another. Thus, it is the resistance of that fluid to shear stress and to flow. The greater the internal friction, the greater the amount of force required to cause movement. Therefore, highly viscous fluids, such as heavy crude oil, require a significant force or stress to move them from one location to another. Although that concept is simple, several different measures of viscosity can be obtained. One measure of viscosity is the ratio of the shearing stress to the velocity gradient within a fluid. This fluid property is called dynamic or absolute viscosity and is expressed in units of dyne seconds per square centimeter, which is

given the name poise (P). More commonly dynamic viscosity is expressed in units of centiPoises (cP) or 0.01 P. At 20.2 ºC, water has a dynamic viscosity of 1 cP. The second common measure of viscosity is kinematic viscosity which is the ratio of dynamic viscosity to the density of the fluid which results in units of square meters per second or Stokes (St) where 1 St = $10^{-4}$ $m^2$/sec. Because the Stoke is a very large unit, the more common unit is centistokes (cSt) where 1 St = 100 cSt. Because the specific gravity of water at 20.2 ºC is approximately one, the kinematic viscosity of water at this temperature is essentially 1.0 cSt.

Viscosity data were collected during the Phase I experiments using dip viscosity cups manufactured by Cole-Parmer Instrument Company. Two different viscosity cups were used: 1) Dip Viscosity Cup No. 2 (VI-EZ2), and 2) Dip Viscosity Cup No. 5 (VI-EZ5). These dip or EZ cups were calibrated by the manufacturer at 25 ºC, with a drainage time of 47.92 sec for a viscosity of 118.6 centistokes (cSt) for the No. 2 cup, and a drainage time of 38.06 sec for a viscosity of 877.2 cSt for the No. 5 cup. Each cup can measure viscosity through a defined range greater than and less than this calibration value. The process of obtaining viscosity data for a liquid using these cups is straightforward. The cup is dipped into a liquid being tested or otherwise filled completely with the liquid. A stopwatch is used to measure the time starting when the cup begins to drain in a steady stream through the hole in the bottom of the cup and ending when the liquid filament draining from the cup first breaks from a solid stream. The temperature of the liquid must also be noted in order to obtain an estimate of the viscosity by using calibration curves or equations for each cup. A photograph of one of the dip cups used in the experiments is shown in Figure 9. The ASTM standard test method utilizing dip cups is presented in Appendix A along with the calibration and computation procedures provided by the manufacturer of the dip cups.

**Figure 9 Photograph of Dip Cup for Measuring Viscosity**

### 2.2.2 Methods and Procedures

Batch experiments were conducted using variable frequency acoustic treatment of the three single-weight motor oils. Prior to beginning the testing program using the three single-weight motor oils, preliminary frequency evaluations were made using water and monitoring the performance of the various actuators and power inputs. Based on these preliminary observations and on past experiences with other acoustic applications, it was decided that four frequencies would be used during the tests: 1.8, 3.1, 6.8, and 13.1 kHz. In addition to acoustic frequency, there were two additional independent variables in the testing program. Horn design was also a variable to be evaluated to facilitate the delivery of acoustic energy to the oil. Three different horn fin spacings (see Figure 8 above) were examined: small 0.25 in (6.4 mm), medium 0.75 in (19.1 mm), and large 1.25 in (31.8 mm). In addition, the experiments were designed to segregate the effect of sonication from that of heat. It is well known that the viscosities of liquids are inversely related to temperature. Therefore, temperature was also included as a variable in the experimental planning. The test matrix utilized in the Phase I testing program is given in Table 1 below. Fewer tests were performed on the 30-wt oil because the primary interest was in determining the effects on more viscous oils, but the test plan included some tests using the less-viscous oil to provide information across a broader range of oil viscosities.

**Table 1 Test Matrix for the Phase I Experiments**

| Single-Weight Motor Oil | Heat Only Experiments | Acoustic Treatment Experiments | |
|---|---|---|---|
| | | Acoustic Frequency (kHz) | Horn Fin Spacing |
| 30-wt | Yes | 6.9<br>13.1 | small, medium, large<br>small, medium, large |
| 90-wt | Yes | 1.8<br>3.1<br>6.9<br>13.1 | small, medium, large<br>small<br>small, medium, large<br>small, medium, large |
| 140-wt | Yes | 1.8<br>3.1<br>6.9<br>13.1 | small, medium, large<br>small<br>small, medium, large<br>small, medium, large |

Prior to performing the acoustic frequency experiments to determine the effect of acoustic frequency and horn design on the resulting viscosity of the oil, a series of experiments was performed with each of the three single weight motor oils using heat alone. The oil was heated on a hot plate, with the mixing accomplished by a magnetic stirring bar. Samples were collected at various times, with the resulting temperature noted at the time of collection. The sample's viscosity as reflected in the dip cup drainage time was then determined. This approach enabled the drainage time and viscosity change to be determined using heat energy input alone. These

data served as the baseline to compare the results from the variable acoustic energy experiments against.

Prior to performing an experiment, the drainage time of the oil to be tested was measured using the viscosity drainage cups, measuring the time required for the full cup to drain (see Appendix A for procedures). The sample of oil to be tested was placed in a container at room temperature over night before testing began. The drainage time was determined for a minimum of three separate analyses and a maximum of five or six measurements in which the drainage times were within a few tenths of a second of one another. The temperature at which these oil drainage times were measured was also measured and recorded. These tests provided information on the initial viscosity/drainage time prior to acoustic testing as well as an estimate of the precision and reproducibility of this method.

When a particular experiment was to be performed, the acoustic frequency was selected, the horn design (small-, medium-, or large-spacing) was selected, and the initial temperature and drainage time of the oil was determined. Approximately 4.0 L (1.06 gallons) of oil was poured into a 5.0-L container and left over night at room temperature before the experiment began. All sonication experiments were performed within a fume hood as a precaution in case noxious gases were generated during the experiments. The power supply was connected to the combined actuator and horn and this tool was placed in the container of oil (Figure 10). The

**Figure 10 Experimental Setup During Acoustic Treatment of Oil**

power supply was turned on and the output frequency was tuned to provide the desired frequency for the test. Power output was slowly increased to the desired level to keep from damaging the actuator from the sudden input surge of large amounts of power. At this point, the experiment was begun.

During the course of an acoustic treatment test on the oil, the treatment time was noted. Samples were collected at various treatment times, but most typically in increments of 5 minutes. Power to the sonication unit was shut off in order to collect the oil samples for analysis. The temperature of the oil in the container was monitored and recorded at these sampling times. Additionally, the highest temperature observed on the transducer horn was likewise monitored (using an infrared thermometer) and recorded, in order to keep an operating log of the use of the acoustic equipment. After collecting the sample and noting the temperature of the oil in the container, the sample was transferred to the viscosity cup in order to determine the drainage time reflecting viscosity as a function of the treatment time. The drainage time was normally determined within one minute after collecting the oil sample. After these tasks were complete and the data recorded, the sonication unit was again turned on and operated for another time increment of about five minutes, after which the power supply unit was again turned off for the next sample collection and analysis event. This procedure was repeated until the desired overall treatment time was achieved and/or when it was observed that the drainage time appeared to have reached a relatively constant value from one test to the next. A typical experiment lasted from 30 minutes to 60 minutes (actual treatment time).

Several replicate experiments were performed to verify the reproducibility of the test procedures. The data collected during these tests are provided in Appendix B. These data clearly show that comparable results were obtained in all such tests indicating that the procedures provide reproducible results. Other experiments were conducted to determine the effects of performing the tests in a water bath to maintain a relatively constant temperature in the oils while they were treated with sonication. These data are also provided in Appendix B, and they indicate that results obtained are nearly the same whether or not the oil test vessel is surrounded by a water bath or not. Given this result, the majority of the tests were conducted without utilizing a water bath to simplify the equipment set-up and test procedure.

### 2.2.3 Data Analysis Rationale and Approach

A few points regarding data analysis decisions and the presentation of observations need to be discussed prior to presenting the experimental results in the following sections of this report. As noted above, the effects of heat alone on the three test oils were measured prior to sonication testing in order to separate the effects of heat from those of sonication. This allowed conclusions to be made about the ability of sonication to alter the viscosity of the oils separate from the well-know effects of heat. In reality, sonication usually generates some heat within the liquid receiving the energy during the process. Therefore, this is somewhat of an academic exercise

because the application of a full-scale sonication technology to the heavy crude problem would benefit simultaneously from the added heat as well as the effects of sonication by itself.

Given the limitations associated with the dip cup method of viscosity measurement, it was decided that the changes in viscosity during the experimental tests would be evaluated based on the measured drainage times of the individual samples instead of a calculated value of viscosity using the methods contained in Appendix A. There were several reasons for this decision. The information in Appendix A allows one to obtain an estimate of viscosity expressed in centistokes. However, these methods are not without some errors and uncertainty, thus the conversion from time to viscosity would introduce some additional inaccuracies into the results. Furthermore, the manufacturer did not provide data on the initial viscosity characteristics of the commercial motor oils used in the test, and each has a number of additives that affect the response of the oils to heat. Because of these conditions, it seemed to be best to look at viscosity in terms of drainage time because this variable could be measured directly for each oil sample at the beginning of a test and the values during the tests could be measured and compared to the initial value to quantify changes resulting from the test conditions. Using the procedures contained in Appendix A, it is possible to convert each drainage time into a viscosity value, but no additional information about the effects of heat and sonication within each experiment would be gained.

Because the testing program was designed to evaluate the effects of sonication (and heat) on the three test oils, the results are analyzed and presented to illustrate the change in viscosity (as reflected in dip cup drainage time) resulting from the individual treatment conditions. Given that objective, the primary interest is in the amount of change in viscosity from the initial conditions or the fractional amount of change in viscosity as opposed to the absolute value of viscosity before, during, and after treatment. Therefore, the data are presented in terms of the relative viscosity (drainage times) calculated as the ratio of drainage time after a specified treatment time to the initial drainage time ($t/t_0$). This yields a number equal to or less than one representing the fractional (percentage) amount of the original drainage time (viscosity) that is measured after the specific treatment time at the time of sample collection. For example, if this ratio is 0.40, this indicates that the viscosity has been reduced by 60% or that the value is 40% of the initial value before the test began. This means that 40% of the original viscosity value remains. Given the objective of the study, this latter value was of most interest because it reflected the relative amount of residual (remaining) viscosity that could be subjected to additional treatment. This variable was termed “residual viscosity” or “fractional residual viscosity” and was the primary measure used to reflect the effectiveness of the various treatment scenarios for the treatment time when sampled.

Given the above rationale, the experimental data were analyzed in terms of the resulting drainage times and oil temperatures for the total treatment time at the time a sample was collected. The resulting reduction in viscosity due to heat alone was determined from the

preliminary oil viscosity reduction experiments involving heat alone. The resulting reduction in oil viscosity due to combined acoustic energy and heat was determined using the experimental drainage times for samples collected periodically throughout the run. The reduction in viscosity due to sonication alone was estimated by subtracting the oil viscosity reduction effect due to heat alone, as calculated from the relationship obtained earlier from the heating tests, from the oil viscosity reduction due to combined sonication + heat. The resulting viscosity reductions due to the three effects (heat alone, sonication alone, and combined sonication + heat) were thereby segregated for individual evaluation.

Another concern exists that pertains to the accuracy of the viscosity measurements collected during testing of the 30-weight oil with the dip cups available at the time the tests were completed. Because this oil is the least viscous of the three oils tested, the drainage times utilizing the dip cups were very short compared to the other two. The fast drainage time made it more difficult to obtain accurate and reliable drainage times during the experiments, which introduced additional errors into the 30-weight oil data set. Furthermore, because the viscosity of this oil was fairly low to begin with, incremental changes with treatment were difficult to observe and measure, especially given the rapid drainage times with the available equipment. While this does not negate the utility of these data and observations, it is believed that the data from the two more viscous oils are more reliable. Therefore, a direct comparison of results across all three oils should be viewed in light of this probable limitation. For this reason, the results presented for Phase I of this study tend to emphasize the data collected from the 90-weight and 140-weight oils.

Most test data were examined, plotted, and analyzed by regression analysis. Although other options were considered, the best results were obtained when viscosity reductions due to the independent variables used in the study (heat, frequency, time, etc.) were fit to a first-order reduction model. This approach provides useful information and interpretations given the relatively good fit of the regression results as reflected by the coefficients of determination in the majority of the analyses. The coefficient of determination is represented by $r^2$ or $R^2$ depending on whether there is only one (simple regression) or more than one (multiple regression) independent variables in the analysis. This coefficient, which is also the squared value of the correlation coefficient, indicates the proportion of the observed variance (fluctuation) in the dependent variable that is predictable from the other (independent) variables as expressed in the regression equation. It is therefore a measure of how well the regression equation/line represents the data, and it can have a value ranging from 0 to 1. A value of $r^2 = 0.90$ would indicate that 90% of the variance in viscosity (Y variable) would be explained by the regression equation. A value of 1.0 would indicate a perfect fit and that the regression line would pass through each and every data point.

A final point regarding the data interpretations and graphical representations should be mentioned. As noted above, the total duration of individual tests typically ranged from about 30

minutes to about 60 minutes. Individual experiments were usually terminated after the drainage time for a given sample compared to that for the previous sample or previous several samples collected during the course of a test reflected very little or no change in drainage time/viscosity. This situation was interpreted as indicating that further testing would result in minimal or no additional viscosity (drainage time) reductions. In examining the data from these tests, it is apparent that, in most tests, the majority of the viscosity changes occurred within the first 20 minutes of testing. After that time, most viscosity values reflect small variations about an average value (fluctuations around a straight line). Therefore, when comparisons were made between the results obtained with the different frequencies, horn designs, and other independent variables, the data reflecting the initial portion of the tests when the viscosities were actively decreasing were used. The data reflecting the second phase of the tests when very little changes in viscosity (drainage times) were observed were considered to be of lesser usefulness. This was also true for the regression analyses performed on the test data. Unless stated to the contrary, the results reported are for the early phase of the experiments when viscosities were changing with treatment time. When all of the data for an individual test are included in a regression analysis, a more complex relationship (polynomial) is obtained that is not easily interpreted and that tends not to describe the variation in the data as well as the results obtained by using the data from the initial 20 minutes of the experiments. Consequently, the viscosity reduction relationships obtained and reported in subsequent sections of the report should be interpreted in terms of these restrictions. If one uses the equations to predict viscosity changes for treatment times significantly in excess of 30 minutes, unreliable results may be obtained.

## 2.3 Experimental Results

### 2.3.1 Viscosity Reduction Due to Heat Alone

To determine the viscosity reduction associated with increasing temperature (due to heat input only), experiments were conducted using a hot plate to increase the temperature of each of the single weight motor oils (30-, 90-, and 140-weight). During these tests, samples were collected at various times, with the temperature of the oil noted at the time of sample collection. Each of these samples was quickly evaluated with the dip cups to obtain an estimate of viscosity as reflected in the drainage time. All of the data resulting from these tests are presented in Tables B-1 through B-3 in Appendix B. The data plots from these three tests – drainage time (viscosity reduction) vs. temperature relationships – are shown in Figures 11, 12, and 13 immediately following. The temperature range over which these measurements were taken varied from about 20$^{o}$C to approximately 100$^{o}$C.

In each test, the individual samples that were tested for drainage time were allowed to return to room temperature. The temperature was recorded and drainage time was again measured with the dip cups. These values were compared to the pre-test data. In almost every case, the drainage time (viscosity estimate) returned to very close to the original value prior to

heating. Much of differences between these measurements are probably attributable to errors in the dip cup methodology. These data are also presented in Tables B-1, B-2, and B-3 in Appendix B.

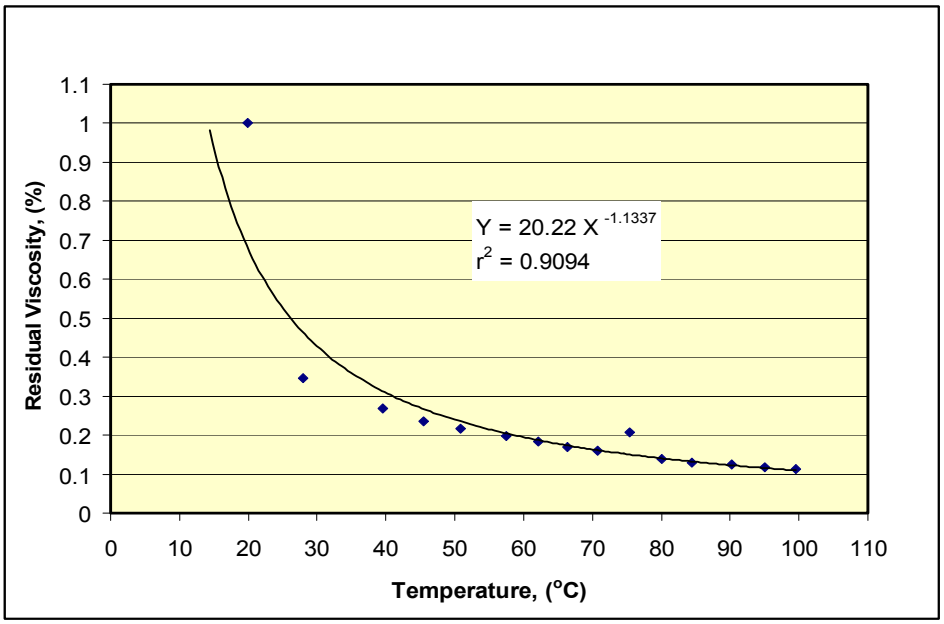


**Figure 11 Residual Viscosity as a Function of Temperature for the 30-Weight Oil**

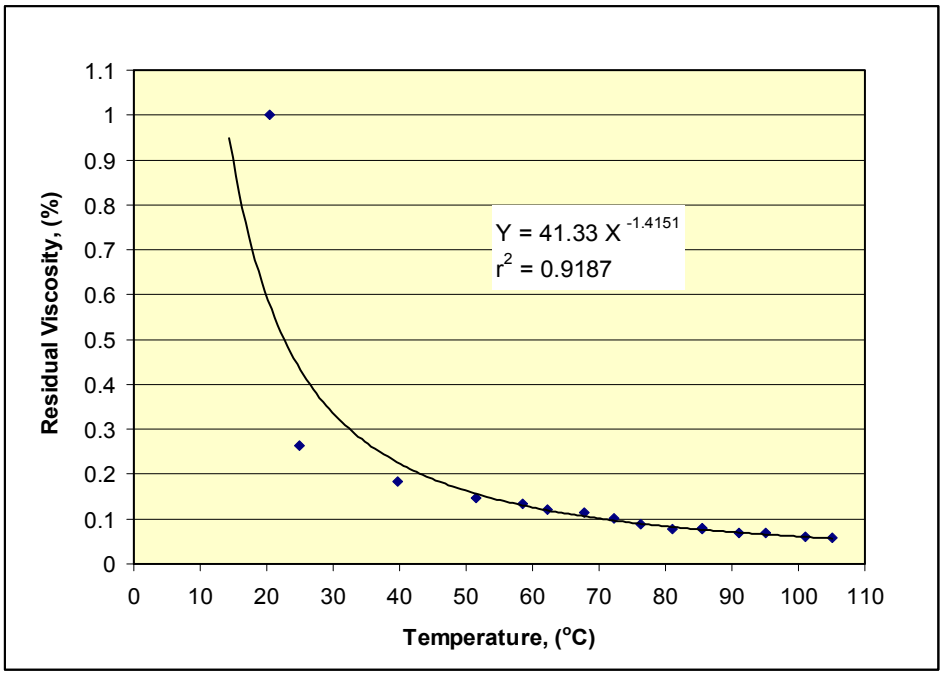


**Figure 12 Residual Viscosity as a Function of Temperature for the 90-Weight Oil**

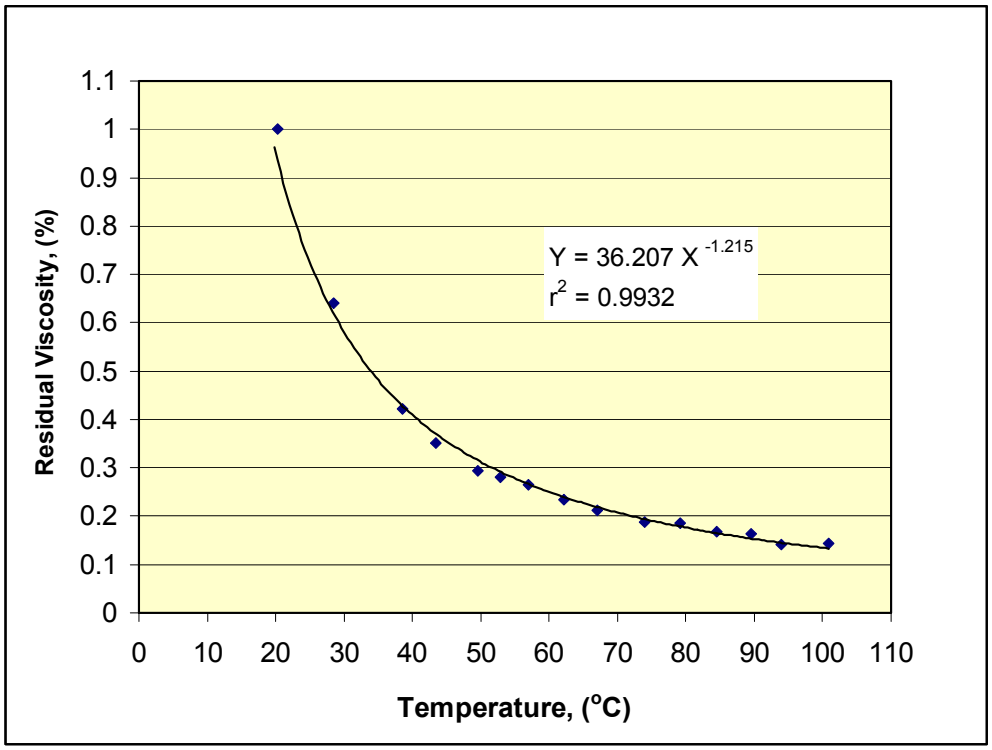


**Figure 13 Residual Viscosity as a Function of Temperature for the 140-Weight Oil**

As reflected in the three graphs above, the residual viscosity was modeled (regression analysis) as a function of temperature using a power-law relationship. These results for the three single weight oils are summarized in the following table.

**Table 2 Regression Results of Viscosity Reduction as a Function of Temperature**

| Weight of Motor Oil | Viscosity Reduction (Y) vs. Temperature (T) Regression | Coefficient of Determination ($r^2$) |
|---|---|---|
| 30 | $Y = 20.217\ T^{-1.1337}$ | 0.9094 |
| 90 | $Y = 41.330\ T^{-1.4151}$ | 0.9187 |
| 140 | $Y = 36.207\ T^{-1.2150}$ | 0.9932 |

These equations were used to separate the effects of heat and sonication on viscosity reduction within the three test oils. As noted previously, sonication also adds heat to the oils as they are treated. The equations in Table 2 were used to calculate the heat effects at the temperatures of the individual samples collected during the sonication + heat experiments. By

subtracting the heat only viscosity reduction value from the combined sonication + heat value, the difference is an estimate of the effects of sonication alone.

### 2.3.2 Viscosity Reduction Due to Combined Sonication and Heat

The experimental procedures used to conduct the series of tests that provided data on the combined effects of sonication and heat on the viscosities of the three oils used in this study are described fully in Section 2.2.2 of this report. All of the data collected during these tests are tabulated and presented in Appendix B (Tables B-4 – B-34). Selected results and observations using these data are presented and discussed in this section of the report. These results that are highlighted are representative of and analogous to the overall data trends and relationships resulting from additional analyses of the entire data set.

Figures 14 and 15 show representative results of the viscosity reduction achieved using sonication + heating on the 30-weight oil; the sonication frequencies employed in these examples are 6.9 kHz and 13.1 kHz, respectively.

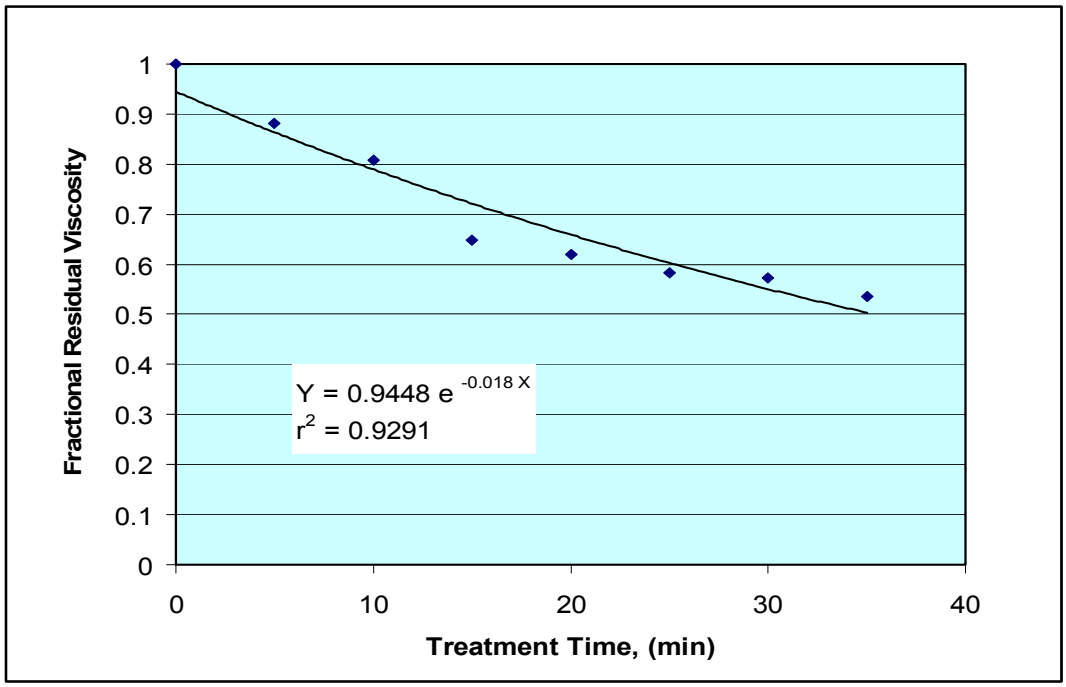


**Figure 14 Fractional Residual Viscosity as a Function of Treatment Time for 30-Weight Oil Employing Sonication at 6.9 kHz with the Medium Horn Spacing**

Results analogous with those illustrated above were obtained for the remaining tests of the 30-weight oil using other combinations of acoustic frequencies and horn designs (see Tables B-4 through B-9, Appendix B). These results show that sonication treatment of the oil decreases the

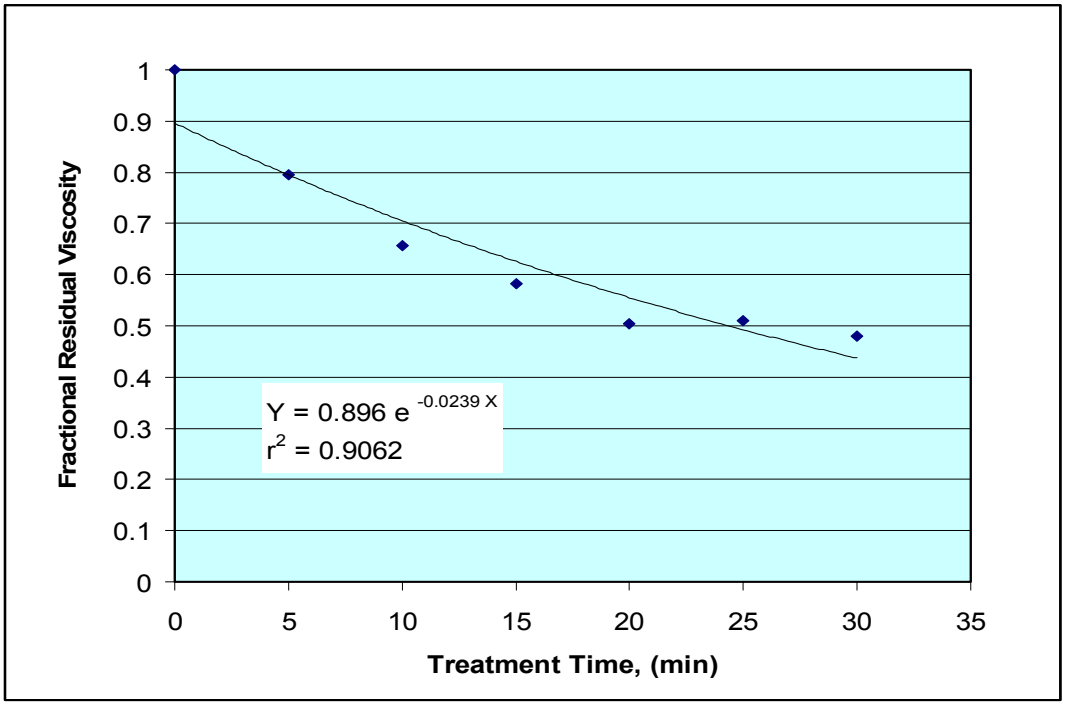


**Figure 15 Fractional Residual Viscosity as a Function of Treatment Time for 30-Weight Oil Employing Sonication at 13.1 kHz with the Large Horn Spacing**

viscosity with increasing treatment time, but the rate of reduction generally diminishes with time. As discussed previously, most of the viscosity reduction is accomplished during the first 20 minutes of treatment.

Figures 16 through 18 show typical results on the viscosity reduction achieved using sonication + heating on the 90-weight oil. In these examples, the sonication frequencies employed were 1.8, 6.9, and 13.1 kHz, respectively, using the small spacing transducer horn in all tests. Results using the other horn spacings and acoustic frequencies are presented in Appendix B. The behavior of the viscosity reduction in all of these tests generally follows a 1st-order decay. For the small spacings transducer horn over the range of acoustic frequencies studied, the 1st-order rate constants ranged from 0.0390 $min^{-1}$ to 0.0937 $min^{-1}$. For the same range of ultrasonic frequencies for the medium spaced transducer horn, the 1st-order rate constants ranged from 0.0211 $min^{-1}$ to 0.0696 $min^{-1}$. Similarly, for the large spaced transducer horn, the 1st-order rate constants ranged from 0.0251 $min^{-1}$ to 0.0700 $min^{-1}$. For the 90-weight oil, the small spaced transducer horn resulted in the largest 1st-order rate constants, indicating this spacing of the horn disks produced the greatest decrease in viscosity obtained during this set of experiments.

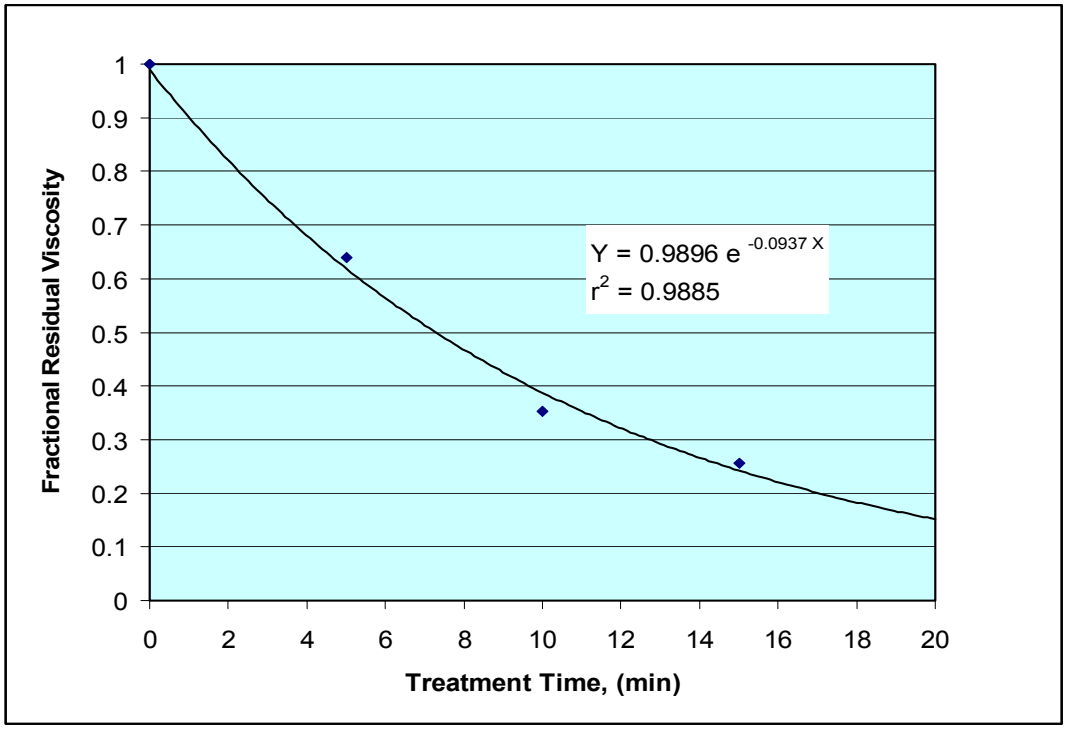


**Figure 16 Fractional Residual Viscosity as a Function of Treatment Time for 90-Weight Oil Employing Sonication at 1.8 kHz with the Small Horn Spacing**

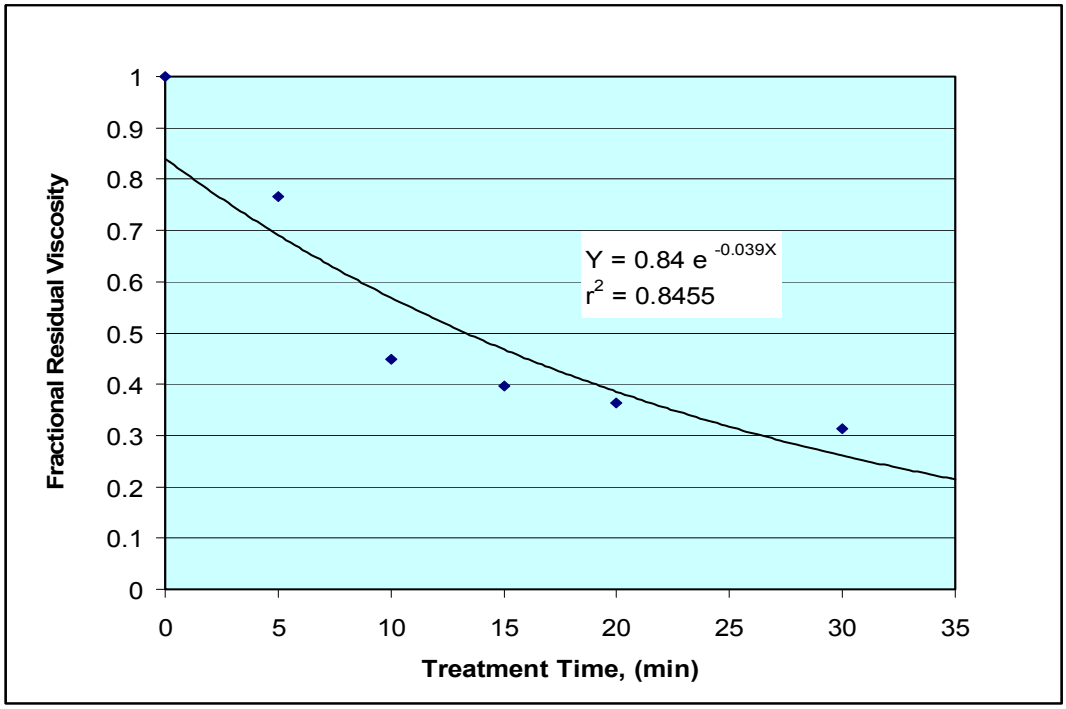


**Figure 17 Fractional Residual Viscosity as a Function of Treatment Time for 90-Weight Oil Employing Sonication at 6.9 kHz with the Small Horn Spacing**

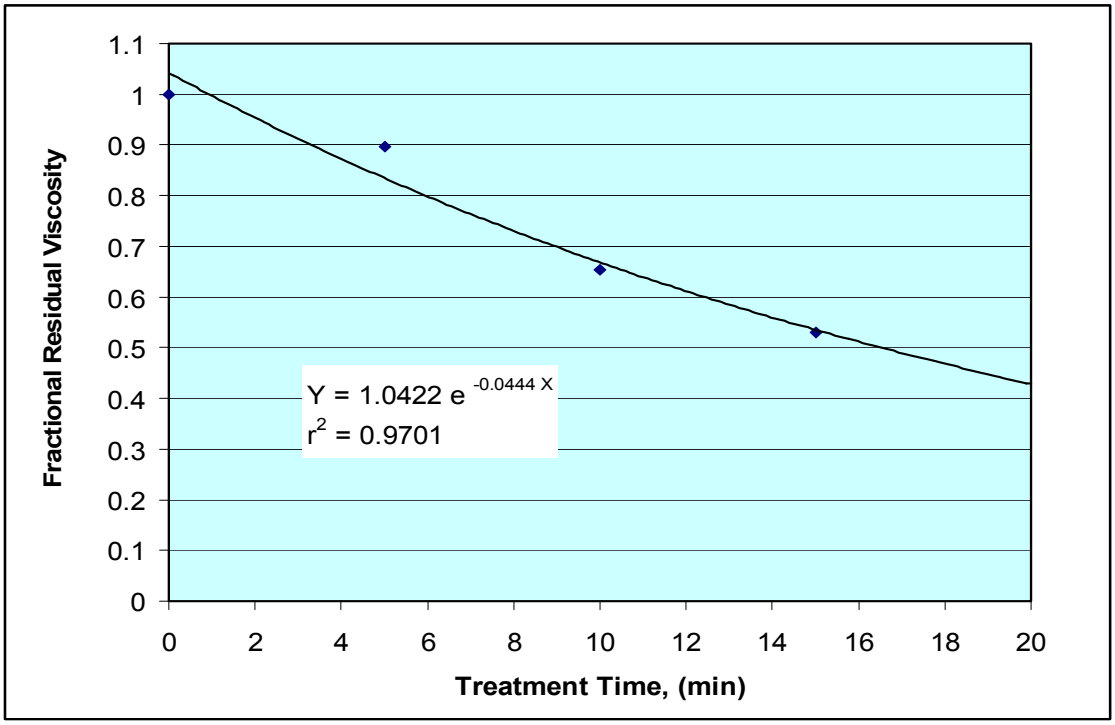


**Figure 18 Fractional Residual Viscosity as a Function of Treatment Time for 90-Weight Oil Employing Sonication at 13.1 kHz with the Small Horn Spacing**

From a review of the results presented in Figures 16 through 18 reflecting the effects of increasing acoustic frequency while maintaining a constant horn spacing, one can observe that, in general, the lower the frequency, the larger the 1st-order rate constant and the greater the percentage viscosity reduction. For example, Figure 16 shows that the viscosity reduction was about 75% (residual viscosity about 25%) at 1.8 kHz. At 6.9 kHz, the viscosity reduction after about 20 minutes treatment time was approximately 55%, while at 13.1 kHz, the viscosity reduction approached 60% after 20 minutes treatment time. As noted above, the change in the exponent of the regression line in these same graphs indicates that the rate of change (decrease) in the viscosity is greatest when the lowest frequency is applied to the 90-weight oil.

Figures 19 through 22 show the results of tests conducted on the 140-weight oil using the medium transducer horn spacing. Additional results obtained with this oil are presented in Table 3 following these illustrations. Data for the experiments using small and large transducer horn spacings, as well as the data used to generate the plots in Figures 19-22, are contained in Appendix B. The values of ultrasonic frequencies employed in these tests were 1.8, 6.9, and 13.1 kHz. Figure 20 shows the viscosity reduction results for treatment at 1.8 kHz using the medium spacing transducer horn (with the oil vessel contained in a water bath). The reduction in viscosity was about 56% (fractional residual viscosity approximately 44%) after 20 minutes

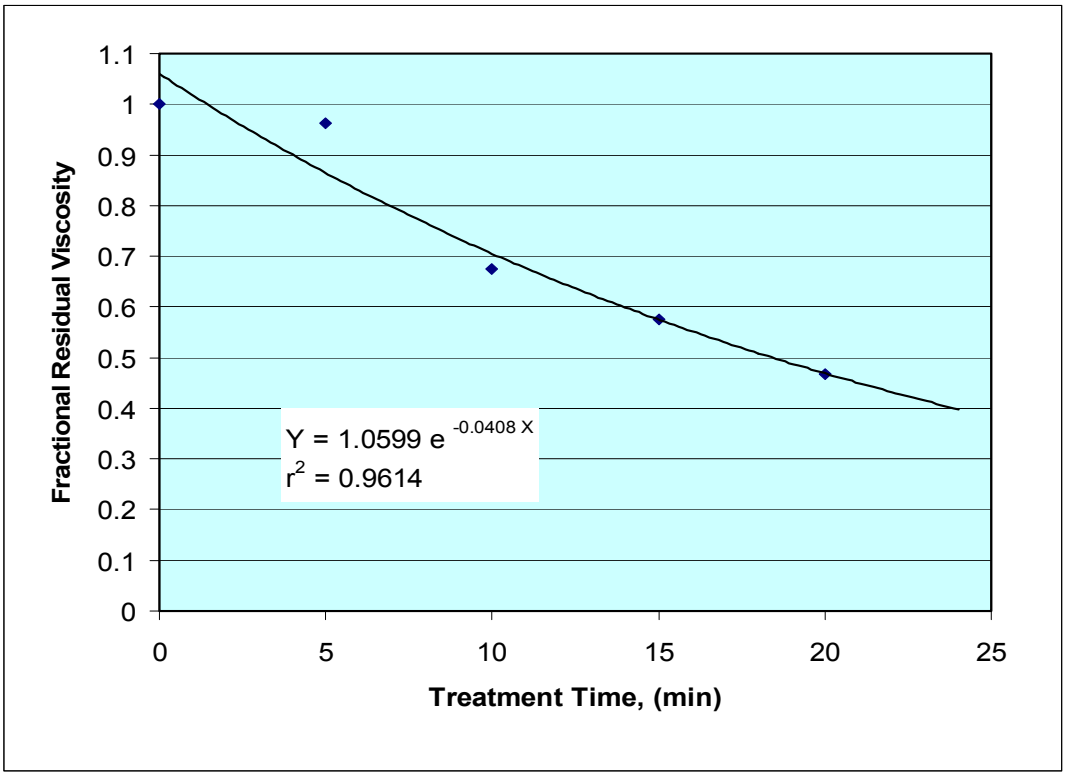


**Figure 19 Fractional Residual Viscosity as a Function of Treatment Time for 140-Weight Oil Employing Sonication at 1.8 kHz with the Medium Horn Spacing**

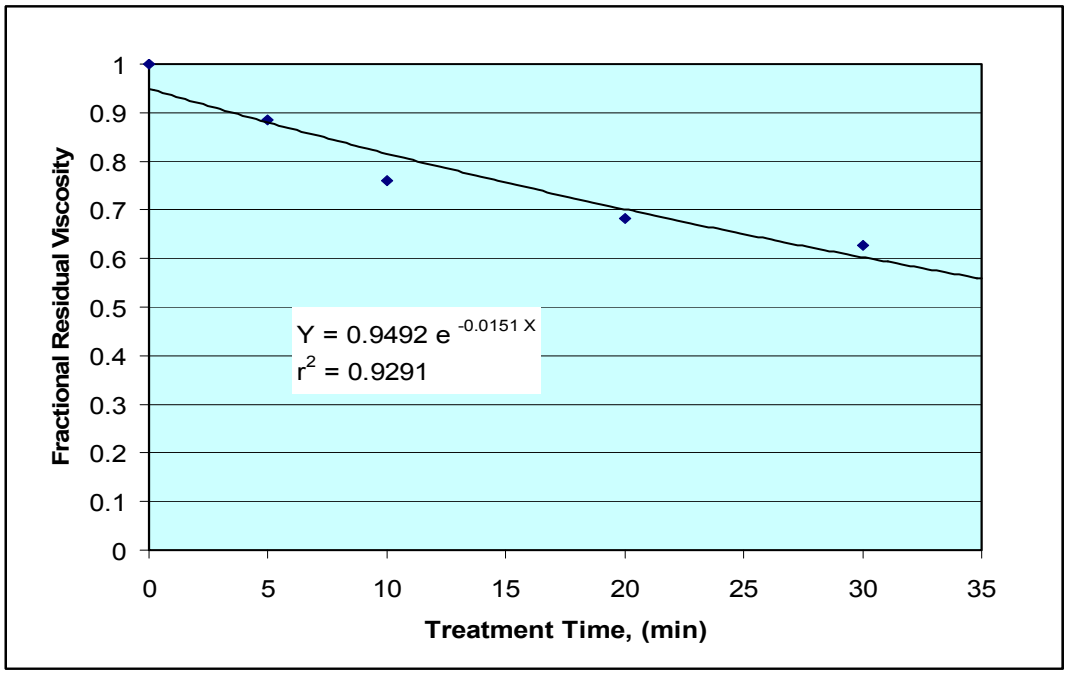


**Figure 20 Fractional Residual Viscosity as a Function of Treatment Time for 140-Weight Oil Employing Sonication at 6.9 kHz with the Medium Horn Spacing**

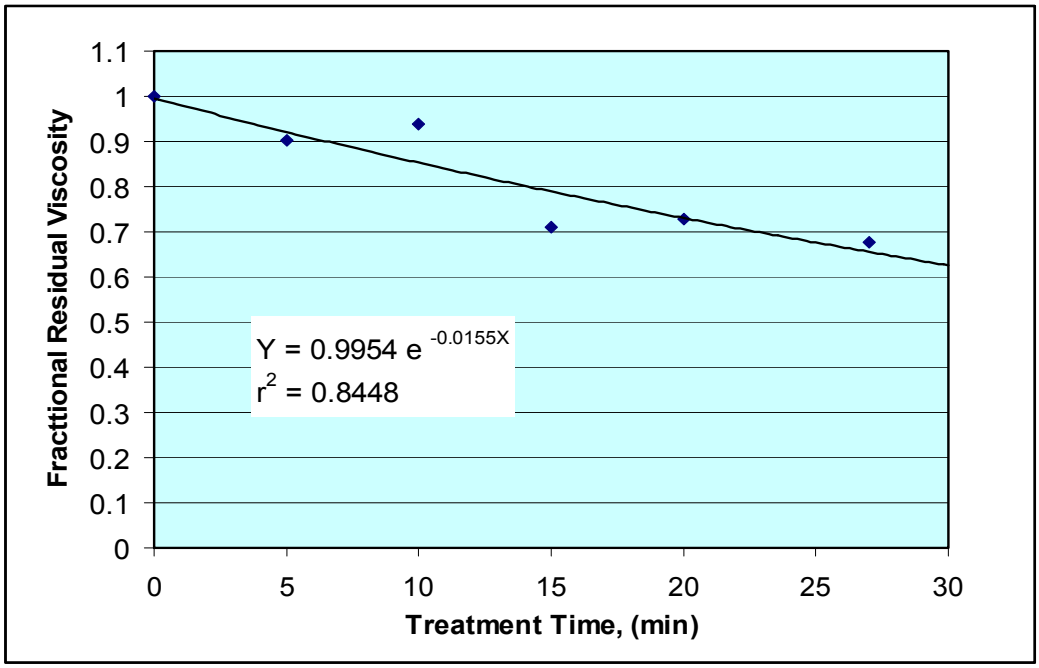


**Figure 21 Fractional Residual Viscosity as a Function of Treatment Time for 140-Weight Oil Employing Sonication at 6.9 kHz with the Medium Horn Spacing (Replicate Test of Figure 20 Conditions)**

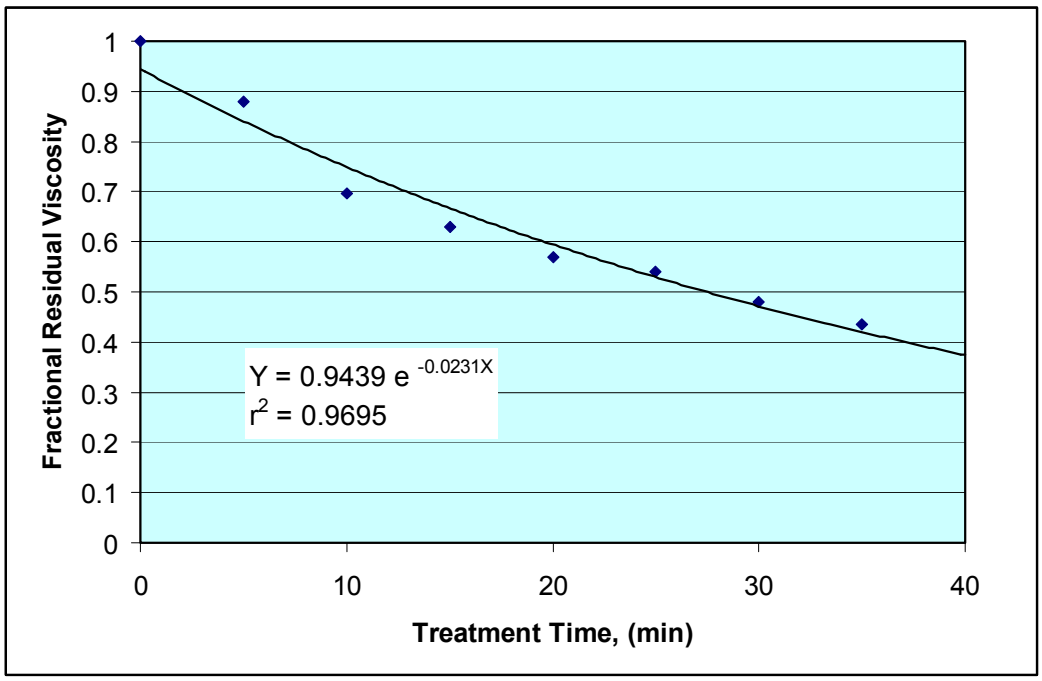


**Figure 22 Fractional Residual Viscosity as a Function of Treatment Time for 140-Weight Oil Employing Sonication at 13.1 kHz with the Medium Horn Spacing**

treatment time. Figures 20 and 21 show the results from replicate experiments performed at 6.9 kHz using the medium spacing transducer horn. The 1st-order rate constants are virtually identical, being 0.0155 $min^{-1}$ and 0.0151 $min^{-1}$, respectively. From both experiments, the reduction in viscosity after 30 minutes treatment time was nearly 40%. Figure 22 shows similar behavior for the experiment conducted at 13.1 kHz. In this series of tests, the greatest amount of viscosity reduction was accomplished with the lowest frequency while the least reduction was associated with the intermediate frequency. For the 140-weight oil, using the medium spacing transducer horn, the 1st-order rate constants ranged from 0.0151 to 0.0408 $min^{-1}$ with the largest

**Table 3 1st-Order Rate Constant Values for Viscosity Reduction using Combined Sonication + Heat**

| Weight of Oil | Measured Frequency, (kHz) | Fin Spacing of Horn | 1st-Order Rate Constant, ($min^{-1}$) |
|---|---|---|---|
| 140 | 1.75 | Small | 0.0385 |
| | 1.77 | Medium ($H_2O$ Bath) | 0.0408 |
| | 1.72 | Large | 0.0226 |
| | 3.09 | Small | 0.0219 |
| | 6.92 | Small | 0.0084 |
| | 6.93 | Medium | 0.0151 |
| | 6.84 | Medium | 0.0155 |
| | 6.88 | Large | 0.0242 |
| | 13.09 | Small | 0.0272 |
| | 13.05 | Small | 0.0308 |
| | 13.06 | Medium | 0.0231 |
| | 13.06 | Large | 0.0272 |
| 90 | 1.76 | Small | 0.0937 |
| | 1.74 | Medium | 0.0696 |
| | 1.76 | Large | 0.0700 |
| | 6.86 | Small | 0.0390 |
| | 6.78 | Small ($H_2O$ Bath) | 0.0408 |
| | 6.66 | Medium | 0.0319 |
| | 6.78 | Medium | 0.0211 |
| | 6.90 | Large | 0.0379 |
| | 13.08 | Small | 0.0300 |
| | 13.07 | Medium | 0.0353 |
| | 13.06 | Large | 0.0251 |

rate of viscosity reduction occurring with the lowest frequency. For the small spacing transducer horn, the 1st-order rate constants ranged from 0.0084 $min^{-1}$ to 0.0385 $min^{-1}$, while for the large spacing transducer horn, the 1st-order rate constants ranged from 0.0226 $min^{-1}$ to 0.0302 $min^{-1}$. Therefore, for the 140-weight oil, the medium spacing transducer horn appeared to provide somewhat greater 1st-order rate constants and larger viscosity reduction that the other two transducer horn designs used in the study.

Table 3 above summarizes the 1st-order rate constants determined from each of the viscosity reduction experiments performed using the 140- and 90-weight oils. The results using the 30-weight oil are not presented in this table for reasons described in Section 2.2.3; however, all of the 30-weight data are contained in Appendix B. The table lists the acoustic frequency used in each case and the fin spacing in the transducer horn employed in each experiment. Recall that small spacing is 0.25 inch (6.4 mm), medium spacing is 0.75 inch (19.1 mm) and large spacing is 1.25 inches (31.8 mm). For the 90-weight oil, the 1st-order rate constant ranges from 0.0211 $min^{-1}$ to 0.0937 $min^{-1}$, while for the 140-weight oil, the 1st-order rate constant ranges from 0.0084 $min^{-1}$ to 0.0408 $min^{-1}$. These results represent the viscosity reduction achieved by sonication, which includes effects due to both sonication and increased temperature from heating during the sonication treatment.

### 2.3.3 Viscosity Reduction Due to Sonication Alone

As described earlier, the residual viscosity was determined by regression analysis as a function of temperature using a power-law relationship. The results for the three single weight oils were presented in Table 2. These relationships were used to separate the sonication + heat viscosity reduction effects from the viscosity reduction effects resulting from heat alone in order to estimate the viscosity reduction effects due to sonication alone. The heat effect estimated from the power-law regression expression of viscosity reduction as a function of temperature for each weight of oil, was subtracted from the combined effect of the viscosity reduction as a function of time (noting the temperature of the oil for each sample collected), in order to estimate the viscosity effect due to sonication alone (see also Section 2.2.3). Figures 23 and 24 show the segregated viscosity reduction results associated with heat alone, sonication alone, and the combined sonication + heat on the 30 weight oil employing acoustic frequencies of 6.9 kHz and 13.1 kHz, respectively as obtained by this analytical technique. Both of these tests used the medium transducer horn spacing. The results shown in these two figures indicate that for the 6.9 kHz and 13.1 kHz frequencies, the majority of the viscosity reduction appears to be caused by sonication, with much less contribution associated with heat.

Figures 25 through 27 present the viscosity reduction results associated with heat alone, sonication alone, and the combined sonication + heat on the 90-weight oil employing 1.8 kHz, 6.9 kHz and 13.1 kHz, respectively, with the small horn spacing. When the oil was treated with

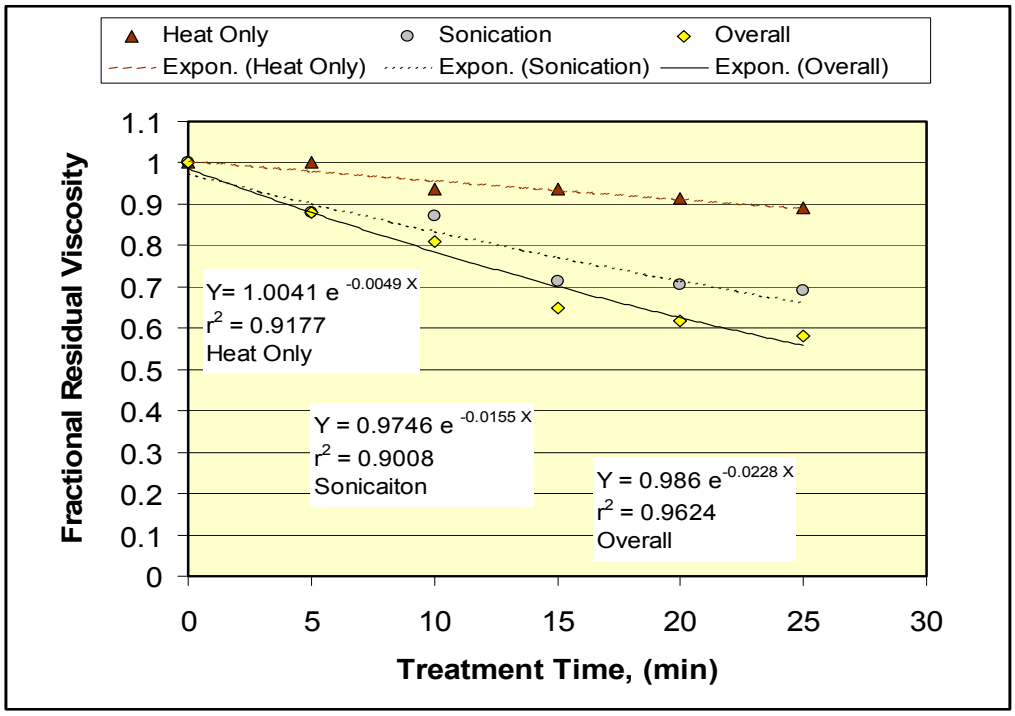


**Figure 23 Fractional Residual Viscosity in 30-Weight Oil as a Function of Treatment Time Employing Sonication at 6.9 kHz with the Medium Horn Spacing due to Heat Only, Sonication Only, and Combined Sonication + Heat**

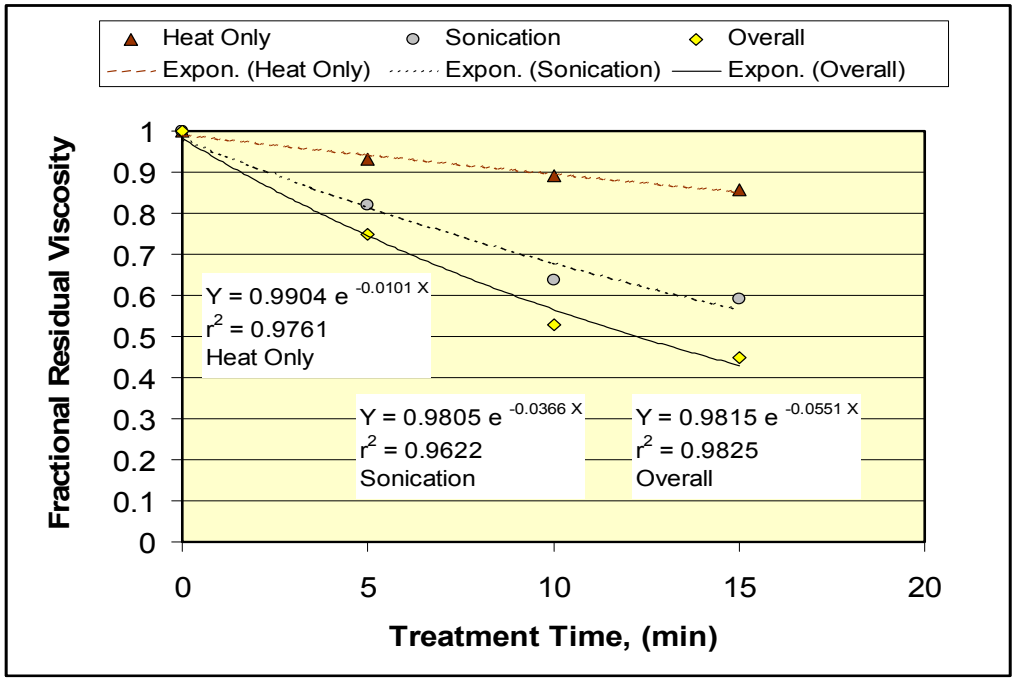


**Figure 24 Fractional Residual Viscosity in 30-Weight Oil as a Function of Treatment Time Employing Sonication at 13.1 kHz with the Medium Horn Spacing due to Heat Only, Sonication Only, and Combined Sonication + Heat**

sonication at 1.8 kHz, the majority of the viscosity reduction was associated with heat. On the other hand, using sonication frequencies of 6.9 kHz and 13.1 kHz resulted in viscosity reduction due to both sonication and heat effects in roughly equal contributions. The graphical results from these three tests also indicate that the total amount of viscosity reduction due to both heat and sonication progressively decreases with increasing sonication frequencies. In the test using 1.8 kHz, the fractional residual viscosity at the end of the test was approximately 0.25 indicating that the initial viscosity had been reduced by approximately 75%. When 6.9 kHz was employed, the viscosity was reduced by about 65%, and the reduction was only about 50% when the highest frequency of 13.1 kHz was used.

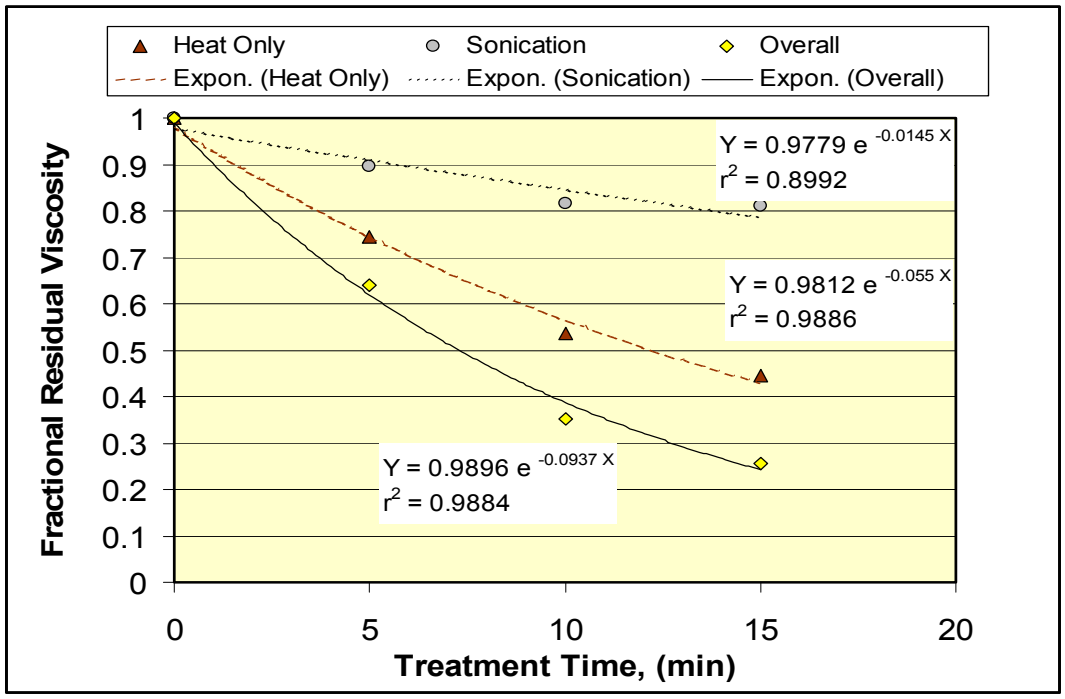


**Figure 25 Fractional Residual Viscosity in 90-Weight Oil as a Function of Treatment Time Employing Sonication at 1.8 kHz with the Small Horn Spacing due to Heat Only, Sonication Only, and Combined Sonication + Heat**

Figures 28 through 30 present the viscosity reduction results associated with heat alone, sonication alone, and the combined sonication + heat on the most viscous (140-weight) of the three oils tested in the Phase I experiments. Sonication frequencies were again 1.8 kHz, 6.9 kHz and 13.1 kHz, respectively and the medium transducer horn spacing was employed in each of these tests. In this case, for the 1.8 kHz sonication treatment, the majority of the viscosity reduction is associated with sonication effects. For the 6.9 kHz treatment, viscosity reduction is due to both sonication and heat effects, in roughly equal contributions. For the 13.1 kHz treatment, most of the initial viscosity reduction as shown in Figure 30 is due primarily to sonication effects; however, at larger treatment times, both sonication and heat effects are

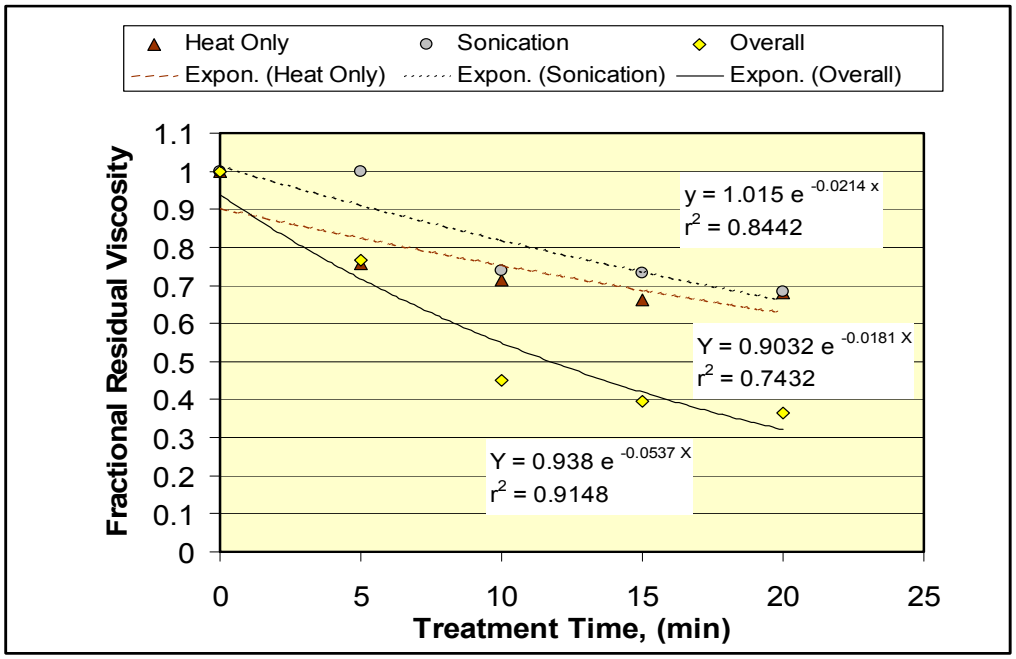


**Figure 26 Fractional Residual Viscosity of 90-Weight Oil as a Function of Treatment Time Employing Sonication at 6.9 kHz with the Small Horn Spacing due to Heat Only, Sonication Only, and Combined Sonication + Heat**

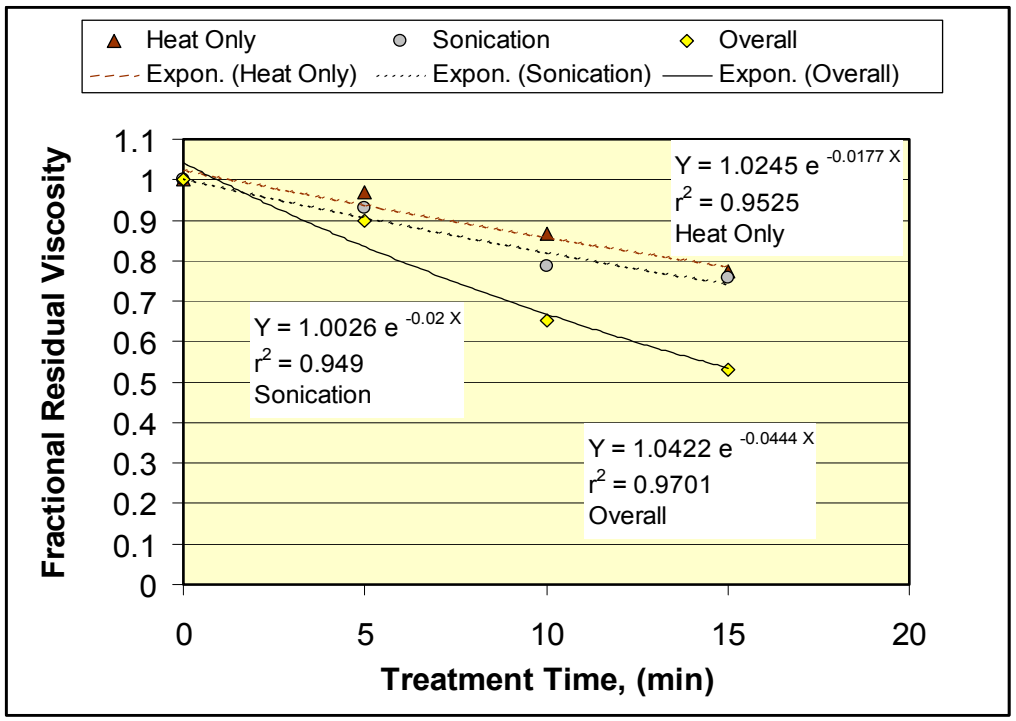


**Figure 27 Fractional Residual Viscosity of 90-Weight Oil as a Function of Treatment Time Employing Sonication at 13.1 kHz with the Small Horn Spacing due to Heat Only, Sonication Only, and Combined Sonication + Heat**

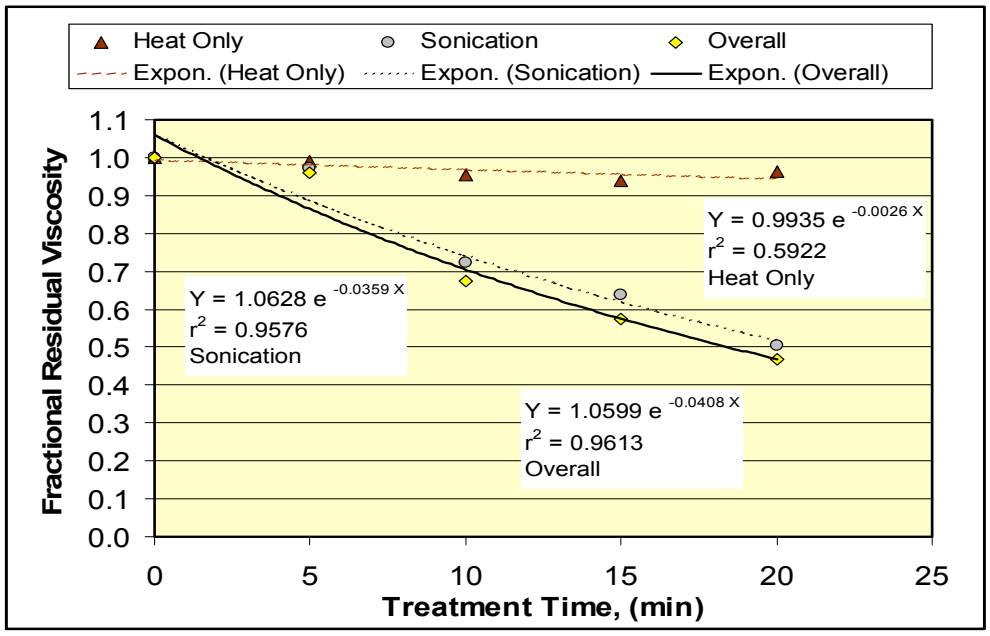


**Figure 28 Fractional Residual Viscosity of 140-Weight Oil as a Function of Treatment Time Employing Sonication at 1.8 kHz with the Medium Horn Spacing due to Heat Only, Sonication Only, and Combined Sonication + Heat**

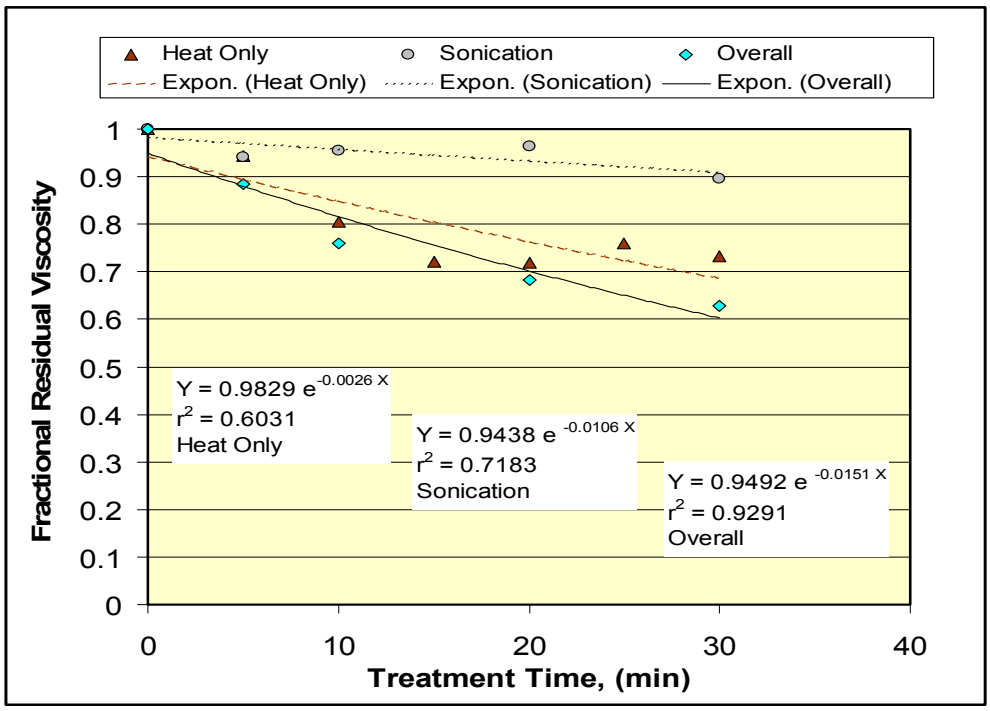


**Figure 29 Fractional Residual Viscosity of 140-Weight Oil as a Function of Treatment Time Employing Sonication at 6.9 kHz with the Medium Horn Spacing due to Heat Only, Sonication Only, and Combined Sonication + Heat**

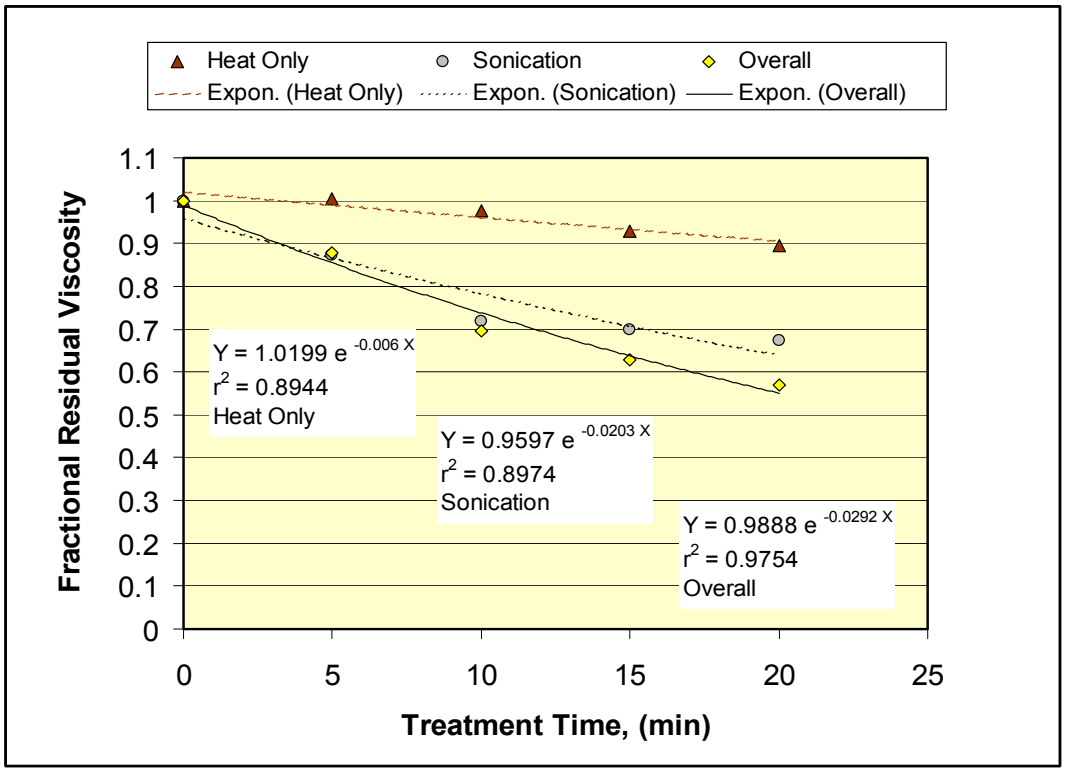


**Figure 30 Fractional Residual Viscosity of 140-Weight Oil as a Function of Treatment Time Employing Sonication at 13.1 kHz with the Medium Horn Spacing due to Heat Only, Sonication Only, and Combined Sonication + Heat**

important, in roughly equal contributions. As was the case with the 90-weight oil, the lowest sonication frequency (1.8 kHz) resulted in the greatest reduction in initial viscosity (~55%) after approximately 20 minutes of treatment. The intermediate frequency (6.9 kHz) resulted in the least amount of reduction (about 35%), while the tests involving the largest frequency (13.1 kHz) produced an intermediate value (~45%) for viscosity reduction during the test. All of the test results for the 140-weight oil are presented in Appendix B.

## 2.4 Comparison of Process Performance

Results of the 1st-order rate constants determined from each experiment using 90- and 140-weight oils, associated with the combined sonication + heat, are summarized in Table 4 below. The table also contains the acoustic frequency used and the transducer horn spacing employed in each experiment. For the 90-weight oil, the 1st-order rate constant ranges from 0.0211 $min^{-1}$ to 0.0937 $min^{-1}$, while for the 140-weight oil, the 1st-order rate constant ranges from 0.0084 $min^{-1}$ to 0.0408 $min^{-1}$. These results represent the viscosity reduction achieved by sonication, which includes effects due to both sonication and increased temperature from heating during the sonication treatment. When the data are tabulated in the manner contained in Table 4, one can look at trends associated with changing sonication frequencies while maintaining the same fin

spacing on the horns. The data in this table indicate that in all cases, the lowest sonication frequency resulted in the largest rate constant (most rapid decrease in viscosity) for each horn design. The results for the 30-weight oil are not tabulated for this comparison because of the reasons and rationale discussed in Section 2.2.3.

**Table 4 Rate Constant Values for Small, Medium, and Large Horn/Fin Spacings and Variable Sonication Frequencies**

| Weight of Oil | Horn/Fin Spacing | Acoustic Frequency, (kHz) | 1st-Order Rate Constant, ($min^{-1}$) |
|---|---|---|---|
| 140 | Small | 1.75 | 0.0385 |
| | | 3.09 | 0.0219 |
| | | 6.92 | 0.0084 |
| | | 13.05 | 0.0308 |
| | | 13.09 | 0.0272 |
| | Medium | 1.77 | 0.0408 |
| | | 6.84 | 0.0155 |
| | | 6.93 | 0.0151 |
| | | 13.06 | 0.0231 |
| | Large | 1.72 | 0.0226 |
| | | 6.88 | 0.0242 |
| | | 13.06 | 0.0272 |
| 90 | Small | 1.76 | 0.0937 |
| | | 6.78 | 0.0408 |
| | | 6.86 | 0.0390 |
| | | 13.08 | 0.0300 |
| | Medium | 1.74 | 0.0696 |
| | | 6.66 | 0.0319 |
| | | 6.78 | 0.0211 |
| | | 13.07 | 0.0353 |
| | Large | 1.76 | 0.0700 |
| | | 6.90 | 0.0379 |
| | | 13.06 | 0.0251 |

Table 5 summarizes the 1st-order rate constants obtained using various ultrasonic frequencies in treating the 90- and 140-weight oil, for each transducer horn/fin design. When the data are tabulated in this manner, it is possible to look for trends in the rate of viscosity reduction associated with changing horn/fin design while maintaining the same acoustic frequency. These data will be examined and discussed in more detail in a subsequent section of this report.

During the heat only experiments, samples were allowed to cool to room temperature and the viscosity was measured again for comparison with the viscosity measured before heating. In

most cases, the viscosity returned to very close to the same values after cooling. Similar data were collected with the sonicated samples. The data in Appendix B indicate that when the samples were allowed to equilibrate to room temperature after being treated using sonication techniques, the viscosity returns to approximately the same viscosity level as it was prior to the sonication treatment, although some variations are present in this trend. This suggests that the acoustic energy has not severely altered the fundamental chemical make-up of the oil, although additional and more sophisticated chemical analyses would be necessary to verity this inference.

**Table 5 Rate Constant Values for Changing Horn/Fin Configurations Compared to Acoustic Frequencies**

| Weight of Oil | Acoustic Frequency, (kHz) | $1^{st}$-Order Rate Constant, ($min^{-1}$) | | |
|---|---|---|---|---|
| | | Horn/Fin Spacing | | |
| | | Small | Medium | Large |
| 140 | 1.72 | | | 0.0226 |
| | 1.75 | 0.0385 | | |
| | 1.77 | | 0.0408 | |
| | 3.09 | 0.0219 | | |
| | 6.84 | | 0.0155 | |
| | 6.88 | | | 0.0242 |
| | 6.92 | 0.0084 | | |
| | 6.93 | | 0.0151 | |
| | 13.05 | 0.0308 | | |
| | 13.06 | | 0.0231 | 0.0272 |
| | 13.09 | 0.0272 | | |
| 90 | 1.74 | | 0.0696 | |
| | 1.76 | 0.0937 | | 0.0700 |
| | 6.66 | | 0.0319 | |
| | 6.78 | 0.0408 | 0.0211 | |
| | 6.86 | 0.0390 | | |
| | 6.90 | | | 0.0379 |
| | 13.06 | | | 0.0251 |
| | 13.07 | | 0.0353 | |
| | 13.08 | 0.0300 | | |

## 2.5 Comparison of Performance at 20 Minutes Treatment Time

Using the data obtained for the viscosity reduction due to heat alone, sonication alone, and combined sonication + heat, the fractional residual viscosity was determined at 20 minutes treatment time for each of these three conditions for each experiment performed (Table 6). As

**Table 6 Fractional Residual Viscosity at 20 Minutes Treatment Time for Various Test Conditions**

| Motor Oil Weight | Sonication Frequency, (kHz) | Transducer Horn Spacing | Fractional Residual Viscosity at 20 Minutes Treatment Time | | |
|---|---|---|---|---|---|
| | | | Heat Only | Sonication | Combined |
| 140 | 1.8 | Small | 0.8185 | 0.6540 | 0.4725 |
| | | Medium | 0.9628 | 0.5038 | 0.4666 |
| | | Large | 0.7351 | 0.7215 | 0.4566 |
| | 3.1 | Small | 0.8285 | 0.7259 | 0.5544 |
| | 6.9 | Small | 0.8070 | 0.9355 | 0.7425 |
| | | Medium | 0.7188 | 0.9641 | 0.6829 |
| | | Medium | 0.8862 | 0.8412 | 0.7274 |
| | | Large | 0.8798 | 0.6915 | 0.5713 |
| | 13.1 | Small | 0.7520 | 0.8846 | 0.6368 |
| | | Small | 0.6526 | 0.8921 | 0.5447 |
| | | Medium | 0.8958 | 0.6727 | 0.5685 |
| | | Large | 0.8818 | 0.6213 | 0.5031 |
| 90 | 1.8 | Small | 0.4124 | 0.8456 | 0.2580 |
| | | Medium | 0.4454 | 0.8286 | 0.2740 |
| | | Large | 0.5647 | 0.6961 | 0.2608 |
| | 3.1 | Small | 0.3545 | 0.8915 | 0.2460 |
| | 6.9 | Small | 0.6795 | 0.6841 | 0.3636 |
| | | Small | 0.8763 | 0.7209 | 0.5972 |
| | | Medium | 0.7692 | 0.6731 | 0.4422 |
| | | Large | 0.8251 | 0.6717 | 0.4968 |
| | | Large | 0.5684 | 0.9029 | 0.4713 |
| | | Large | 0.6546 | 0.8151 | 0.4697 |
| | 13.1 | Small | 0.7519 | 0.7890 | 0.5409 |
| | | Medium | 0.6563 | 0.8485 | 0.5048 |
| | | Large | 0.5992 | 0.9448 | 0.5440 |
| | | Large | 0.3618 | 0.9685 | 0.3303 |
| 30 | 6.9 | Small | 0.9362 | 0.7516 | 0.6878 |
| | | Medium | 0.9129 | 0.7060 | 0.6189 |
| | | Large | 0.8766 | 0.7324 | 0.6091 |
| | 13.1 | Small | 0.7665 | 0.8487 | 0.6152 |
| | | Medium | 0.8883 | 0.5748 | 0.4631 |
| | | Large | 0.7479 | 0.7568 | 0.5047 |

discussed in more detail in Section 2.2.3, twenty minutes was selected as a reasonable time period when the viscosity was still decreasing (had not reached a pseudo-steady state behavior in which the residual viscosity had reached a relatively constant value) and it provided a benchmark

by which one could compare the performance of the various experimental conditions studied. These data for all three to the oils used in this investigation are summarized in Table 6. These data reflect the percentage of the initial viscosity remaining after the treatment. Table 7 contains

**Table 7 Percentage of Viscosity Reduction After 20 Minutes Treatment Time for Various Test Conditions**

| Motor Oil Weight | Sonication Frequency, (kHz) | Transducer Horn Spacing | Fractional Viscosity Reduction at 20 Minutes Treatment Time | | |
|---|---|---|---|---|---|
| | | | Heat Only | Sonication | Combined |
| 140 | 1.8 | Small | 0.1815 | 0.3460 | 0.5275 |
| | | Medium | 0.0372 | 0.4962 | 0.5334 |
| | | Large | 0.2649 | 0.2785 | 0.5434 |
| | 3.1 | Small | 0.1715 | 0.2741 | 0.4456 |
| | 6.9 | Small | 0.1930 | 0.0645 | 0.2575 |
| | | Medium | 0.2812 | 0.0359 | 0.3171 |
| | | Medium | 0.1138 | 0.1588 | 0.2726 |
| | | Large | 0.1202 | 0.3085 | 0.4287 |
| | 13.1 | Small | 0.2480 | 0.1154 | 0.3633 |
| | | Small | 0.3474 | 0.1079 | 0.4553 |
| | | Medium | 0.1042 | 0.3273 | 0.4315 |
| | | Large | 0.1182 | 0.3787 | 0.4969 |
| 90 | 1.8 | Small | 0.5876 | 0.1544 | 0.7420 |
| | | Medium | 0.5546 | 0.1714 | 0.7260 |
| | | Large | 0.4353 | 0.3039 | 0.7392 |
| | 3.1 | Small | 0.6455 | 0.1085 | 0.7540 |
| | 6.9 | Small | 0.3205 | 0.3159 | 0.6364 |
| | | Small | 0.1237 | 0.2791 | 0.4028 |
| | | Medium | 0.2308 | 0.3269 | 0.5578 |
| | | Large | 0.1749 | 0.3283 | 0.5032 |
| | | Large | 0.4316 | 0.0971 | 0.5287 |
| | | Large | 0.3454 | 0.1849 | 0.5303 |
| | 13.1 | Small | 0.2481 | 0.2110 | 0.4591 |
| | | Medium | 0.3437 | 0.1515 | 0.4952 |
| | | Large | 0.4008 | 0.0552 | 0.4560 |
| | | Large | 0.6382 | 0.0315 | 0.6697 |
| 30 | 6.9 | Small | 0.0638 | 0.2484 | 0.3122 |
| | | Medium | 0.0871 | 0.2940 | 0.3811 |
| | | Large | 0.1234 | 0.2676 | 0.3909 |
| | 13.1 | Small | 0.2335 | 0.1513 | 0.3848 |
| | | Medium | 0.1117 | 0.4252 | 0.5369 |
| | | Large | 0.2521 | 0.2432 | 0.4953 |

data representing the amount of (percentage) reduction in the initial viscosity at the end of twenty minutes for the individual treatments. The data within either of these tables reflect the effectiveness of the individual treatments in reducing the viscosity of the test oils. Obviously, the viscosity remaining and viscosity reduction values in these two tables for corresponding treatment conditions sum to one. The reduction in viscosity ranges from a low of 31.2% to a high of 75.4%; the residual or remaining amount of viscosity after treatment ranges from a maximum of 68.8% to a minimum of 24.6%. The data in Table 6 are presented in graphical form in Figures 31 through 33 for the 30-, 90-, and 140-weight oils.

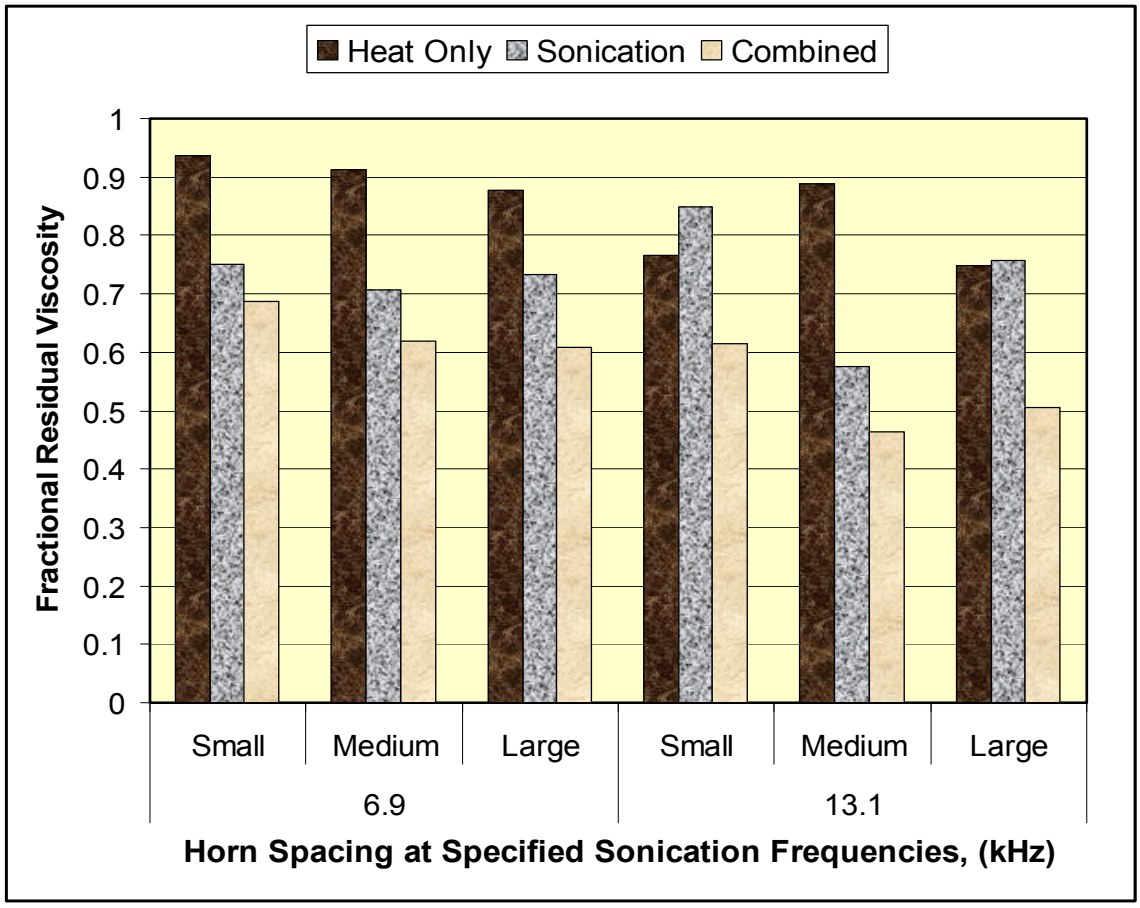


**Figure 31 Comparison of Fractional Residual Viscosity of 30-Weight Oil Obtained after 20 Minutes Treatment from Heat Only, Sonication Only, and Combined Sonication + Heat**

For treatment of 30 weight oil, Figure 31 shows that while the sonication performed at 13.1 kHz resulted in a greater overall reduction in viscosity (about 50% reduction) than sonication operated at 6.9 kHz (overall viscosity reduction was about 40%), the lower frequency sonication (at 6.9 kHz) had a slightly greater average effect on the viscosity reduction due to sonication alone than the higher frequency (13.1 kHz). The figure also indicates that the medium horn spacing was somewhat more effective than the small or large spacing transducer horns.

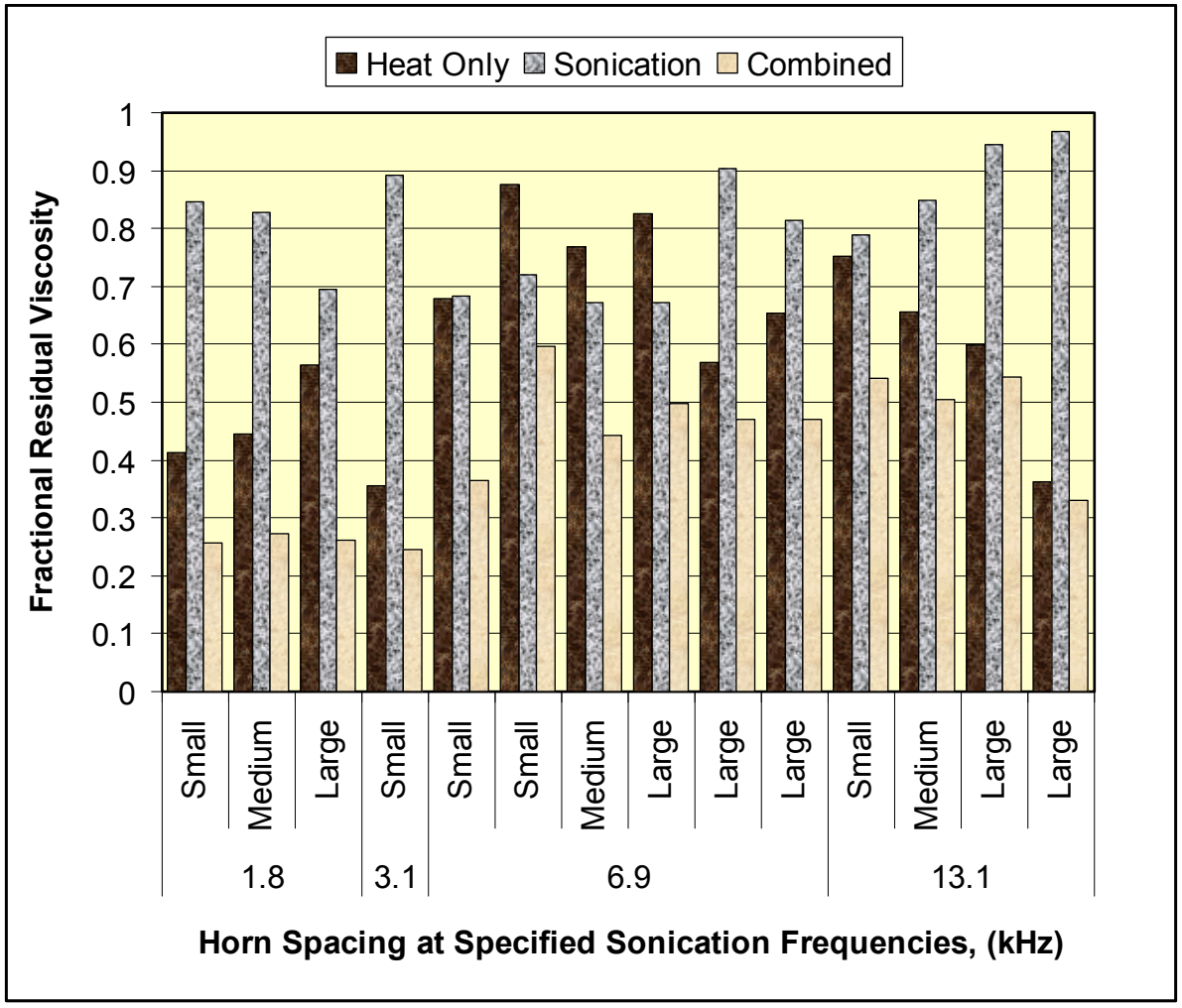


**Figure 32 Comparison of Fractional Residual Viscosity of 90-Weight Oil Obtained after 20 Minutes Treatment from Heat Only, Sonication Only, and Combined Sonication + Heat**

For treatment of 90-weight oil, Figure 32 shows that the lowest ultrasonic frequencies (1.8 and 3.1 kHz) resulted in the greatest reduction in viscosities (greater than 72% reduction) as compared to the higher frequencies; typical viscosity reductions were approximately 50-60% at 6.9 kHz and 46-67% at 13.1 kHz. However, at the lower frequencies, the dissipation of heat from the sonication into the 90-weight oil appears to have been the primary effect responsible for the reduction of the viscosity of the oil. Similar behavior was also observed at the higher frequency (13.1 kHz). For sonication performed at 6.9 kHz, the large spacing transducer horn likewise had a similar behavior. Using the small and medium spacing transducer horns at 6.9 kHz however, resulted in sonication having more of a relative effect on the viscosity reduction.

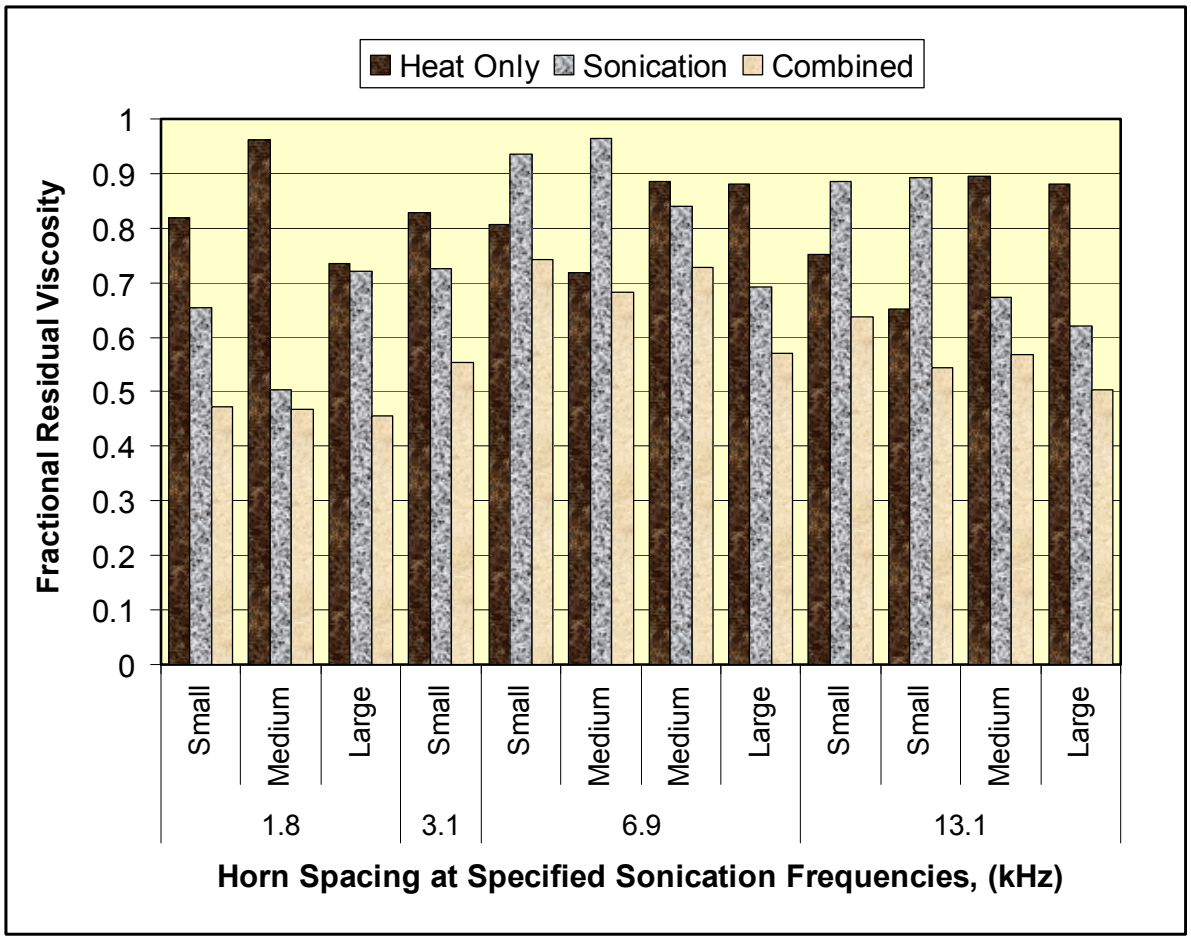


**Figure 33 Comparison of Fractional Residual Viscosity of 140-Weight Oil Obtained after 20 Minutes Treatment from Heat Only, Sonication Only, and Combined Sonication + Heat**

For treatment of the more viscous oil (140-weight oil), Figure 33 illustrates that at the lower acoustic frequencies (1.8 and 3.1 kHz), sonication appears to have had more of an effect in causing the viscosity reduction than did the dissipation of heat into the oil. Overall reductions in viscosity at the lower frequencies ranged from ~45% to ~54%. For the higher frequencies (6.9 and 13.1 kHz), heat dissipation into the oil was primarily responsible for the reduction in viscosity using the small transducer horn spacing, whereas for the medium and large horn spacings, sonication appears to have had a greater role in causing the reduction in viscosity than did heat dissipation into the oil. However, some exceptions to these trends do exist. For the higher ultrasonic frequencies (6.9 and 13.1 kHz), overall reductions in viscosity typically were in the range of 30% to 50%, somewhat less than the lower frequencies.

## 2.6 Phase I Summary and Conclusions

As stated previously, the objective of this Phase I investigation was test and evaluate an integrated acoustic system under laboratory conditions to determine the ability of sonication to

reduce the viscosity of oil. The results of this study clearly prove the validity of this concept and demonstrate that the concept is worthy of further study and evaluation. In addition to this over-arching conclusion, a number of more specific conclusions and observations were derived from the testing program. These are summarized in the following list.

1. The reduction in viscosity observed in the tests was due both to sonication effects and dissipation of heat (resulting from sonication) into the oil.

2. The reduction in viscosity due to heat input only was successfully defined by regression analysis invoking a power-law relationship.

3. The greater the temperature, the more fluid the oil becomes (and hence a resulting reduction in viscosity).

4. Sonication frequencies examined were 1.8, 3.1, 6.9, and 13.1 kHz. Generally, the lower the acoustic frequency, the greater the efficiency in reducing the viscosity of the oil. The reduction in viscosity was greatest for the 1.8 kHz and 3.1 kHz frequencies.

5. The reductions in viscosity due to sonication and the combined sonication + heat dissipation were successfully defined by regression analysis using a $1^{st}$-order decay relationship in viscosity with increasing treatment time.

6. For the 90-weight oil, the $1^{st}$-order rate constant ranged from 0.0211 $min^{-1}$ to 0.0937 $min^{-1}$, while for the 140-weight oil, the $1^{st}$-order rate constant ranged from 0.0084 $min^{-1}$ to 0.0408 $min^{-1}$. The results for the 30-weight oil ranged from 0.0223 $min^{-1}$ to 0.0551 $min^{-1}$. There is less confidence in the results from the tests using the less-viscous oil tests than those from the other two oils.

7. Three different spacings for the fins on the acoustic transducer horns were tested in this study. In general, the transducer horn with the medium spacing resulted in the greatest reduction in viscosity. The efficiencies tended to decrease with both the smaller and larger distances between horn fins.

8. While the sonication at 13.1 kHz resulted in a greater overall decrease is viscosity (~50% reduction) in the 30-weight oil than sonication performed at 6.9 kHz (overall viscosity reduction ~40%), the data from tests employing the lower frequency sonication indicate that the effects of sonication were greater than those resulting from heat dissipation. The reverse situation is suggested by the data obtained from the higher frequency (13.1 kHz) tests.

9. For the case of the 90-weight oil, the lowest acoustic frequencies (1.8 and 3.1 kHz) resulted in the greatest reduction in viscosities (greater than 72% reduction) as compared to the higher frequencies. Typical viscosity reductions were approximately 50%-60% at 6.9 kHz and 46%-67% at 13.1 kHz.

10. At the lower frequencies, the dissipation of heat into the 90-weight oil from the sonication treatment was the primary effect responsible for the reduction in viscosity of the oil as compared to the effects from sonication alone. Similar behavior was also observed at the higher frequency (13.1 kHz).

11. For the case of the 140-weight oil, the tests using lower acoustic frequencies (1.8 and 3.1 kHz) indicate that sonication has more of an effect in causing the viscosity reduction than does the dissipation of heat into the oil. Overall reductions in viscosity at the lower ultrasonic frequencies ranged from ~45% to ~54%.

12. Tests on the 140-weight oil using the higher acoustic frequencies (6.9 and 13.1 kHz) indicate that heat dissipation into the oil was primarily responsible for the reduction in viscosity using the small spacing transducer horn, whereas for the medium and large spacing transducer horns, sonication appeared to have a greater effect in causing the reduction in viscosity than did heat dissipation into the oil. For the higher frequencies (6.9 and 13.1 kHz), overall reductions in viscosity typically were between 30% and 50%.

13. Sonication treatment of the three oils resulted in a reduction in viscosity that ranged from a low of 31.2% to a high of 75.4%. The viscosity reductions measured for each of the test oils were: 31.2% – 53.7% for the 30-weight oil, 40.3% – 75.4% for the 90-weight oil, and 25.8% – 54.3% for the 140-weight oil.

14. After sonication treatment, when the samples were allowed to equilibrate to room temperature, the viscosity returned to approximately the same condition as it was prior to the sonication, suggesting that the acoustic energy has not appreciably altered the oil's chemical structure. However, it would require additional testing and chemical analyses to verify this hypothesis.

# 3 PROJECT PHASE II ACTIVITIES AND RESULTS

Based on the results obtained from the first phase of the project, Phase II was funded and conducted to expand upon the preliminary results and proof of concept from Phase I. The second project phase was designed to evaluate the effects of sonication on three crude oils with significantly varying viscosity/gravity characteristics.

## 3.1 Technical Objectives

The technical objectives defined for the second project phase were:

1. To design, fabricate, and test a three-actuator, integrated, acoustic energy system suitable for laboratory testing and to serve as the basis for engineering scale-up for larger-scale applications if warranted;

2. To collect and analyze data on the performance of the acoustic energy system on three heavy crude oils with different API gravity values;

3. To develop a commercialization plan for the technology including process and component economics, scale-up factors, and market potential; and

4. To prepare a comprehensive final project report containing the system design, all test data, data analyses, and the commercialization plan.

## 3.2 Work Plan

In order to meet the technical objectives, the following work plan, consisting of ten individual tasks, was developed.

Task 1. Project kickoff meeting

Task 2. Design and fabricate three-actuator sonication system

Task 3. Test and debug the sonication system

Task 4. Test the sonication system on heavy crude oil #1; analyze data

Task 5. Mid-project review meeting

Task 6. Test the sonication system on heavy crude oil #2; analyze data

Task 7. Test the sonication system on heavy crude oil #3; analyze data

Task 8. Develop process economics, market potential, and scale-up factors

Task 9. Develop commercialization plan

Task 10. Prepare final report

## 3.3 Project Coordination

TechSavants, Inc. (TSI) was the Lead Principal Investigator for both phases of the project. TechSavants, Inc., Furness-Newburge, Inc. (FNI), and the University of Alabama at Birmingham (UAB) conducted the first phase of the project. For the second phase of work, Armmco, a firm supplying solution-oriented technologies to the petroleum industry, was added to the project team to ensure that the commercialization planning and market potential estimates were realistic and based on an extensive knowledge and understanding of the petroleum industry.

The initial Phase II kickoff meeting was held at UAB and staff members from all team organizations attended. The results and lessons learned from Phase I were discussed at length. The specific activities planned for Phase II were defined in detail along with the anticipated schedule for completion of the individual activities as well as the resources required and team member responsibilities for completion. Because the experiments were going to be conducted at UAB, the laboratory facilities and equipment were examined and a meeting was held with the Safety Coordinator for the Department of Civil and Environmental Engineering to discuss any general safety aspects and concerns associated with the experimental plan. It was agreed that all individuals working on the project experiments would receive a safety briefing and that a set of procedures would be developed and approved for receiving, handling, storing, and disposing of all crude oil needed for the project. The standard operating procedures developed for properly managing the crude oils during the laboratory experiments are presented in Appendix C.

During the time that Tasks 2-5 were being conducted, close coordination between TSI and the other project team members was regularly maintained. Any difficulties or items that could impact the results of or schedule for the project were discussed and a resolution obtained. After Task 5 was completed, a second team meeting, the mid-project coordination meeting was held at UAB. As during the kickoff meeting, personnel representing all team member organizations were present. During this meeting, results obtained during Tasks 2-5 were discussed and, in particular, the experimental procedures and results obtained from the first crude oil were evaluated. Based on this information and lessons learned during the first set of experiments, some modifications to the procedures were identified and recommended for the tests involving the remaining two oils. It was decided that even though this would mean that all three oils were not tested in precisely the same manner, it was reasoned that the overall results would be significantly improved by the new plan of action. These changes will be discussed in more detail in subsequent sections of this report where the experimental results are presented.

Lastly, TSI was responsible for preparing this final project report with considerable input and assistance from the individual project team members that were primarily responsible for

individual elements of the work plan. All individuals listed as contributing authors on the report title page provided written material for this report.

### 3.4 Design and Fabricate Sonication Test System

A series of designs incorporating three actuators was evaluated for use in fabricating the reaction chamber. The designs considered were: 1) a design where all three actuators would act in series along a section of pipe; 2) the three actuators were attached to three of the four vertical walls of a reactor, with the actuators in the same horizontal plane or in differing planes; and 3) the actuators positioned in a cross arrangement, i.e., two actuators acting horizontally and one vertically, all in the same plane and all of their acoustic energy focused on a central point. The location of the ports to get the oil in and out of the chamber and the issue of heat build-up in the actuators also had to be addressed.

The first design was ruled out, because it could be deduced that the total energy produced by the three actuators would never be concentrated on one area. Although the oil might be subjected to a longer sonic treatment time, the magnitude of the energy at any point during the treatment would be far less than what was available in total, since the oil would be primarily exposed to the acoustic energy from only one reactor at a time. The Furness-Newburge team felt that the energy needed to maximize the change in viscosity would not be reached with this design, thus increased flow resulting from viscosity reduction would be limited.

The second design was discarded because the heavy crude could not be readily handled during experiments with this chamber design. Concern was raised that the oil would have to be injected vertically into the chamber and that pipe-edge liquefaction and the effect of gravity would minimize viscosity reduction time. The specific concern was that the oil in contact with the pipe interior would become liquefied (viscosity reduced) before the oil in the center of the pipe, and the oil mass would flow not from an overall reduction in viscosity, but from the reduced friction from the liquefied oil on the inside surface of the pipe. Given these conditions, it was estimated that the residence time needed for maximum oil viscosity reduction would not be realized.

Ultimately, the third option was selected. With this design, oil is pumped into the reaction chamber, oil is exposed to acoustic energy, and the liquefied oil (viscosity-reduced oil) rises to the oil surface (uppermost part of the chamber) and is removed through outlet ports while the remainder of the oil is subjected to further acoustic activity until it also becomes liquefied and rises. Untreated heavy crude oil is constantly pushed into the chamber using a pump. This approach allowed for the most even treatment of the oil inside the acoustic chamber and was the closest to achieving continuous flow stirred tank reactor (CSTR) behavior. The treatment time could be modified in the flow through system by changing the pumping rate, although this was limited by the viscosity of the crude oil. This design also provides for water cooling of the

actuators to dissipate the heat build-up resulting from the actuators' operation, thereby decreasing the amount of heat transferred to the oil. In this way, the temperature effects on viscosity reduction in the oil would be minimized.

The reaction chamber, as designed and fabricated, consisted of a four-flanged, cross design with a volume capacity of approximately one gallon (3.8 liters) of crude oil (see Figure 34). Three of the flanges were machined for sealed mounting of the actuators; the fourth flange was drilled and tapped for the oil inlet via a pump system. Three holes were drilled and tapped for use as oil outlets, and a stand was constructed for mounting the reaction chamber. This configuration had the flexibility of examining the effect of acoustic stimulation with the actuators in parallel or in perpendicular geometry.

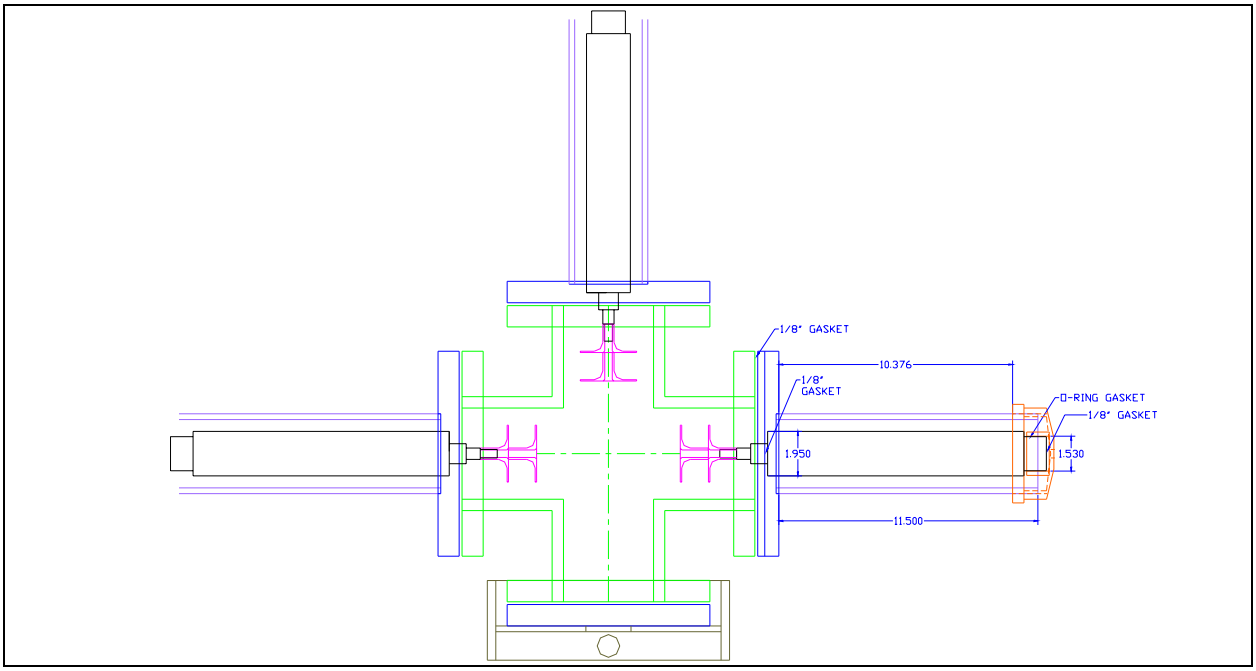


**Figure 34 Cross-Sectional Drawing of Reaction Chamber Designed and Fabricated for Phase II Experimental Testing of Crude Oils (Dimensions in Inches)**

The pumping system for moving the oil through the reaction chamber consisted of a positive displacement pump rated at three to six gallons per hour (3-6 gph) (ll.4-22.3 liters/hr), with a variable speed drive. Because of the crude oil's high viscosity, a significant amount of time was spent in testing the pump inserts and system tubing. Proper flow was finally obtained by using more rigid tubing and increasing the suction pressure to assist flow.

Each of the three actuators utilized in the reactor design had its own 1000-Watt power supply for independent operation and the three power supplies were attached to a mobile test stand. Initially the actuators were fitted with double-disked horns (see Figure 34), although final

horn configurations would be determined from subsequent testing (Task 4). The horns were manufactured into a modular system of spacers, slotted fins, and non-slotted/solid fins. In this way, different configurations altering such things as fin spacing and number of fins could easily be achieved. The cooling system was designed to be completely separate, independent and sealed from the oil reactor. Each actuator sits in a water-cooled chamber fabricated with pipe and pipe caps; plastic tubing brings water to the chamber. The water flows around the actuator, removing the heat build-up from the actuator, and exits the chamber. The chambers were machined with fittings for the tubing and for the electrical connections between the actuators and the power supplies.

## 3.5 Test and Debug Sonication System

### 3.5.1 System and System Component Testing

The system component testing occurred in parallel with whole system fabrication. Tests of horn design – the spacing between the disks, the optimum thickness of the disks, and the decision to use whole or slotted disks – continued past the fabrication and debugging period. The modular horn system supplied to UAB permitted them to perform this testing on heavy crude oil. It was not possible to conclusively rule out several horn configurations during the water testing because one could not be certain that the more viscous medium would react the same. The optimal thickness of the horns was limited to some extent by durability considerations. The thinner the horn's titanium disk, the greater the deflection and apparent transfer of acoustic energy into the fluid. However, the thinner disks were not as durable and did not survive as long during testing. Based on these preliminary evaluations, the optimum thickness for the disks appears to be 0.0625 inches (~1.6 mm.). At this thickness the disk flexes well without breaking. Based on results subsequently obtained by UAB, this held true when the horns are used for a longer period on heavy crude. During acoustic actuator life testing, thinner horns of ~0.030 inch (0.8 mm) thickness broke after 30-50 hours of use. No breakage occurred in the actuators themselves from long-term use. In this testing phase, no actuators failed during more than 100 hours of testing. No degradation of performance occurred during two-week tests of actuator-horn systems (using 0.0625 in/1.6 mm thick disks).

Water flow tests demonstrated that the system was watertight, and the design selected for use in the project eliminated the concern over heat buildup causing actuator failure or causing interference with the test results through thermal viscosity effects on the oil. Flow testing resulted in the decision to determine flow rates by measuring the volume of oil removed from the oil-feed system during a measured time interval. Similarly, viscosity measurements and measuring the volume of treated (changed viscosity) oil collected over a fixed time interval gave a post-treatment flow rate. To ensure that the system, and in particular its pump, could handle the worst case, initial testing of system was performed with a heavy Californian crude (viscosity > 100,000 centiPoises at test temperatures). This thick crude was also used during training of

UAB personnel. Further, system tests using surrogate oils conducted as a check of the equipment prior to beginning the testing program did not present any challenge or problems for the system or its pump.

### 3.5.2 Optimization Testing

The goal of the optimization testing was to learn as much as possible by collecting data on different horn configurations, actuator frequencies, etc. in water. While changes in crude oil were the focus of the project, the water medium had the advantages of actually being able to observe the horns during stimulation, being an easier test, being a quicker test, and not having special handling and disposal issues. Water testing allowed for a larger test matrix to be explored. It was initially proposed that water tests would be performed using red dye. Unfortunately, the system's power and frequency combinations were insufficient to pyrolize the dye and; furthermore, no conclusions could be drawn from observing the dye's mixing behavior in the system under different operating conditions. It was possible to make some judgments through direct observation of cavitation bubble formation and movement, but these observations introduced too much subjectivity and they drew too few distinctions between different treatments. Therefore, the proposed dye evaluation system was replaced with a method of observing sand motion and node formation to evaluate the sonication system that was identified simply as the "sand tests".

All the sand tests were performed in an aquarium 30 in (76.2 cm) long by 12 in (30.5 cm) wide by 18 in (45.7 cm) tall (deep). At the beginning of a test, the aquarium was filled with water 16-17 in (40.6-43.2 cm) deep. Silica sand was spread evenly across the floor of the aquarium approximately 1/8 in (3.2 mm) deep before each test. The actuator was placed in the center of the aquarium with the bottom of the horn 1 inch (2.5 cm) from the bottom. The actuator was run for approximately 11 minutes, and photographs were taken before, during, and after each test to document the manner in which the acoustic stimulation moved the sand. Visual observations of sand motion, cavitation, etc. were recorded during each test. The narratives of these observations for each test are presented in Appendix D.

The test matrix consisted of four different frequencies, single and double finned horns, two different distances between fins for the double finned horns, and solid vs. slotted fins. Table 8 below documents the input conditions and horn configurations tested. Table 9 summarizes the observations made during the tests (see also Appendix D). Holes in the sand would form at the bottom of the aquarium indicating where the sonication pushed the sand away. These features were interpreted as being the location of acoustic standing wave nodes. To document and compare each treatment's effect, the total number of holes in the sand formed in the aquarium, the total number of holes formed greater than six inches away, and the location of the hole closest and furthest from the actuator were recorded for each test.

**Table 8 Sand Test Conditions and Horn Configurations**

| Test Number | Horn Configuration | Frequency (Hz) | Output Current (%) | Voltage (volts) |
|---|---|---|---|---|
| **A1** | 2 fins, 1 inch apart, 4 slots. 45 deg offset, 0.050 inch thick | 900 | 99.9 | 187 |
| **A2** | 2 fins, 1 inch apart, 4 slots, 45 deg offset, 0.050 inch thick | 1136 | 100.0 | -- |
| **A3** | 2 fins, 1 inch apart, 4 slots, 45 deg offset, 0.050 inch thick | 1291 | 99.5 | -- |
| **A4** | 2 fins, 1 inch apart, 4 slots, 45 deg offset, 0.050 inch thick | 1560 | 87.0 | -- |
| **B1** | 2 fins, 2.0625 inches apart, 4 slots, 45 deg offset, 0.050 inch thick | 900 | 99.5 | 187 |
| **B2** | 2 fins, 2.0625 inches apart, 4 slots, 45 deg offset, 0.050 inch thick | 1139 | 99.8 | 239 |
| **B3** | 2 fins, 2.0625 inches apart, 4 slots, 45 deg offset, 0.050 inch thick | 1288 | 98.0 | 260 |
| **B4** | 2 fins, 2.0625 inches apart, 4 slots, 45 deg offset, 0.050 inch thick | 1563 | 85.4 | 278 |
| **C1** | 1 fin with 4 slots and 0.050 inch thick | 900 | 99.4 | 185 |
| **C2** | 1 fin with 4 slots and 0.050 inch thick | 1178 | 99.7 | 278 |
| **C3** | 1 fin with 4 slots and 0.050 inch thick | 1247 | 98.3 | 249 |
| **C4** | 1 fin with 4 slots and 0.050 inch thick | 1563 | 85.4 | 278 |
| **D1** | 1 fin with no slots, 0.050 inch thick | 902 | 99.4 | 186 |
| **D2** | 1 fin with no slots, 0.050 inch thick | 1147 | 98.8 | 238 |
| **D3** | 1 fin with no slots, 0.050 inch thick | 1347 | 94.6 | 278 |
| **D4** | 1 fin with no slots, 0.050 inch thick | 1534 | 92.0 | 276 |
| **E1** | 2 fins, 1 inch apart, no slots, 0.050 inch thick | 901 | 99.2 | 186 |
| **E2** | 2 fins, 1 inch apart, no slots, 0.050 inch thick | 1146 | 98.6 | 240 |
| **E3** | 2 fins, 1 inch apart, no slots, 0.050 inch thick | 1295 | 96.2 | 276 |
| **E4** | 2 fins, 1 inch apart, no slots, 0.050 inch thick | 1538 | 92.2 | 275 |
| **F1** | 2 fins, 2.0625 inches apart, no slots, 0.050 inch thick | 903 | 99.0 | 186 |
| **F2** | 2 fins, 2.0625 inches apart, no slots, 0.050 inch thick | 1147 | 99.6 | 242 |
| **F3** | 2 fins, 2.0625 inches apart, no slots, 0.050 inch thick | 1280 | 95.1 | 276 |
| **F4** | 2 fins, 2.0625 inches apart, no slots, 0.050 inch thick | 1420 | 95.0 | 276 |

**Table 9 Overview of Sand Test Observations**

| Test No. | Total No. of Holes | No. Holes, Distance> 6 in. | Hole Nearest Actuator | | | Hole Furthest from Actuator | | |
|---|---|---|---|---|---|---|---|---|
| | | | Location | Distance (in.) | Size (in.) | Location | Distance (in.) | Size (in.) |
| **A1** | 6 | 2 | (2.5,0) | 2.5 | 2.5 | (12,4) | 12.65 | 1.0 |
| **A2** | 13 | 8 | (2,0) | 2 | 1.0 | (-13.5,-4.5) | 14.23 | 1.5 |
| **A3** | 20 | 12 | (-2,-2) | 2.83 | 1.5 | (14,0) | 14.00 | 1.5 |
| **A4** | 6 | 3 | (-2.75, -0.5) | 2.80 | 3 | (10.5,-2) | 10.69 | 0.5 |
| **B1** | 1 | 0 | (0,0) | 0 | 1.5 | (-13.5,-4.5) | (0, 0) | 0 |
| **B2** | 14 | 9 | (-2,0) | 2 | 1.0 | (12,4) (-12,4) | 12.65 | 0.5, 1.0 |
| **B3** | 1 | 0 | (-1.5,-1.5) | 2.12 | 1.5 | (-1.5,-1.5) | 2.12 | 1.5 |
| **B4** | 0 | 0 | | | | | | |
| **C1** | 0 | 0 | | | | | | |
| **C2** | 5 | 1 | (1.5,0) | 1.50 | 1.5 | (6,2) | 6.32 | 0.5 |
| **C3** | 14 | 11 | (-3,-0.5) | 3.04 | 2.0 | (-12.5, -5.75) | 13.76 | 0.75 |
| **C4** | 0 | 0 | | | | | | |
| **D1** | 0 | 0 | | | | | | |
| **D2** | 8 | 4 | (-2.5,-1.5) | 2.92 | 0.5 | (-5.75,-4) | 7.00 | 0.5 |
| **D3** | 12 | 5 | (-1.5, -1.25) | 1.95 | 0.25[a] | (8,4) | 8.94 | 1.0 |
| **D4** | 20 | 13 | (-4.5,0) | 4.50 | 1.0 | (-12.75, -5.5) | 13.89 | 1.5 |
| **E1** | 3 | 3 | (-9.5,0) | 9.50 | 0.5 | (10,3.5) | 10.59 | 1.5 |
| **E2** | 13 | 10 | (-2,0) | 2.00 | 0.75 | (-13.5,-4) | 14.08 | 3x1.5[a] |
| **E3** | 18 | 14 | (-0.5,-2.5) | 2.55 | 2x1[a] | (-14.75, 5.75) | 15.83 | 0.5 |
| **E4** | 28 | 18 | (-1,-2) (-1,2) | 2.24 | 0.5 0.75 | (-14.5,-5.5) | 15.51 | 1.5 |
| **F1** | 8 | 3 | (0,0) | 0.00 | 0.75 | (-10.5,-0) | 10.50 | 2x5[b] |
| **F2** | 15 | 10 | (0,0) | 0.00 | 0.75 | (12,4) | 12.65 | 1.5x3[b] |
| **F3** | 12 | 8 | (-0.5,-2.5) | 2.55 | 1.5x0.75[b] | (15,-0.5) | 15.01 | 7x3[c] |
| **F4** | 16 | 11 | (3,-0.5) | 3.04 | 5.5x2[c] | (13.25,-4.5) | 13.99 | 3.5x3[d] |

The Notation used in Table 9 immediately above is as follows: a = Ellipse, b = Ellipse (E-W), c = Ellipse (N-S), and d = Rectangle. Location: based on coordinates measured in inches to the left (negative), right (positive), above (positive), or below (negative) of the actuator position when viewed from above

Figures 35-38 contain selected photographs taken during the series of optimization tests. Figure 35 shows the test set-up at the beginning of a test illustrating the placement of the actuator, horn with two slotted fins, and sand layer on the bottom of the aquarium. In all photographs, the fins have a diameter of 2.5 in (6.4 cm). Figure 36 is a photograph showing a cluster of cavitation bubbles on the upper surface of the slotted horn fin. The other bubbles visible in the photo are air bubbles. Note also the holes formed in the bottom sand layer along with a small amount of fine sediment suspended beneath the horn. Figure 37 illustrates sand movement resulting in several holes being formed where sand moved into and adjacent mound beneath the actuator horn. In this test the horn is solid (no slots). Figure 38 contains a photograph taken during a test that illustrates the effects of very vigorous (effective) sonication of the test system as evidenced by the large amount of fine sediment that has been suspended within the water along with the large number of holes that have formed in the bottom sand layer as a result of extensive sand movement.

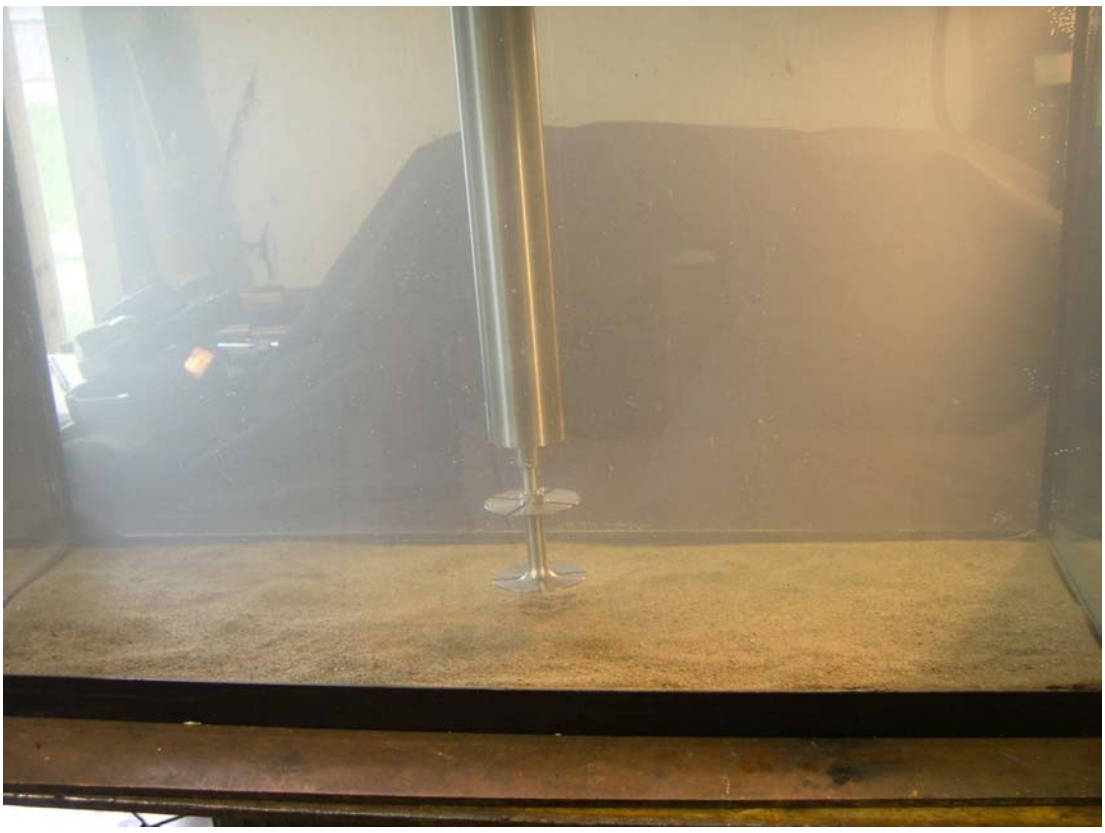

**Figure 35 Photograph of Sand Test Equipment at the Beginning of a Test**

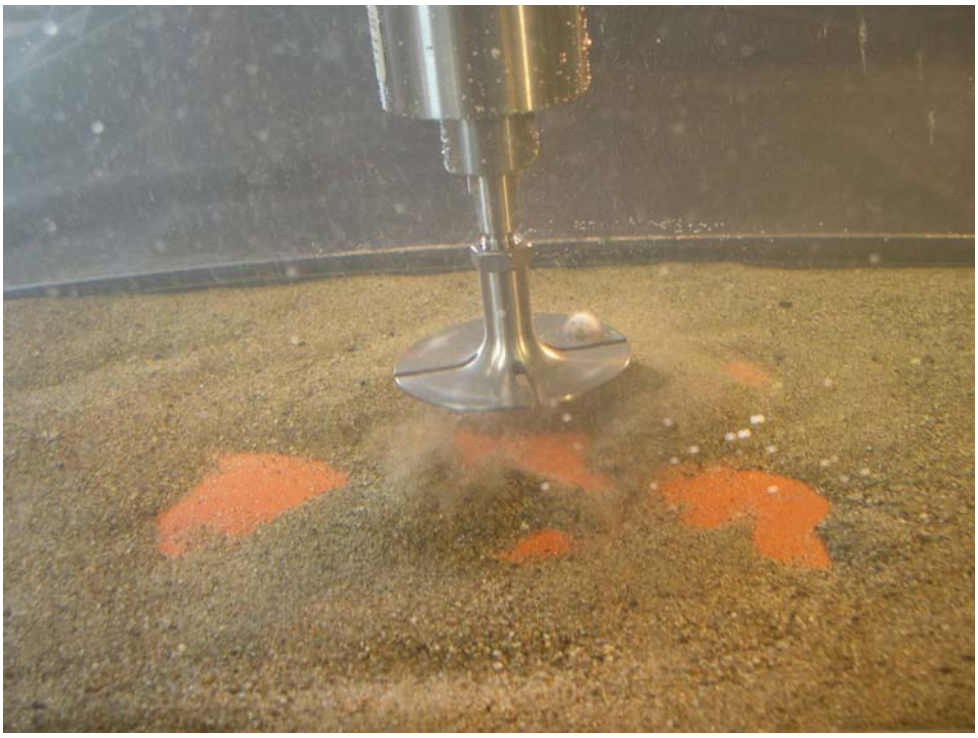

**Figure 36 Photograph Showing Cluster of Cavitation Bubbles, Holes in the Sand Layer, and Minor Amount of Fine Sediment Suspension**

**Figure 37 Close-Up Photograph of a Sand Mound Beneath the Solid Horn and Several Holes where Sand Removed**

**Figure 38 Photograph Illustrating Major Fine Sediment Suspension and a Complex Pattern of Several Holes Where Sand Removed**

Based on a qualitative evaluation of the observations made during the sand tests, it appeared that the double-finned horns worked much better than the single-fin horns. Further, the closer spacing of 1 inch (2.5 cm) worked better than the 2 inch (5 cm) spacing, and the solid fins created somewhat more sand motion than the slotted fins. More flexure was created in the horn through slotting, but it appeared that this design dissipated more of the energy near the fin. This greater intensity near the horn may actually prove to be better for chemical alteration of a fluid, but these results suggest that it is probably at the expense of reduced distribution of the sound energy away from the energy source. The solid fins worked best at the highest frequency, whereas the slotted fins worked best in the middle frequencies.

### 3.5.3 Preliminary Crude Oil Viscosity Testing

Preliminary brief tests were performed on heavy crude at Furness-Newburge facilities to evaluate the viscosity change resulting from sonication using the system components. These shop tests were performed to help evaluate the new horns as well as the performance of the rotational viscometer (Brookfield Digital Viscometer Model DV-E) that was purchased for use in the second phase of the project. The rotational viscometer is more precise and accurate than the viscosity/dip cup approach and better suited to evaluate thicker, more viscous fluids.

The initial viscosity of the heavy crude in the first test was 115,000 cP (centipoises) at 63.0 degrees Fahrenheit (°F). Multiple spindle types and speeds were used with the Brookfield Viscometer to evaluate the operation of the unit and the results compared to the documentation provided with the instrument. The initial test was done at 1.30 kHz (kilohertz) frequency at 880 Watts of power. A double-finned horn was used for 12 minutes on the 1.5-liter sample. The viscosity following treatment was 48,000 cP at 70°F.

A second oil sample that was tested had an initial viscosity of 153,000 cP at 61.0°F. The treatment conditions were 1.30 kHz frequency under 900 Watts of power, double- finned horn, for 10 minutes on a 1.5-liter sample. Following treatment, the viscosity was 54,000 centipoises at 70°F. This sample was further treated with a frequency of 1.31 kilohertz and 890 Watts of power, using a double-finned horn for 10 minutes on the same 1.5-liter sample. Following this additional treatment the viscosity was further reduced to 39,500 cP at 74°F. A final treatment was performed on the sample, using 1.035 kHz frequency under 860 Watts of power, double-finned horn for 10 minutes. Post-treatment viscosity was 36,500 cP at 76°F.

### 3.5.4 Crude Oil Testing Evaluation by Headspace Sampler-GC-FID

It was observed that sonication had reduced the viscosity of various processed and crude oils during Phase I and the initial testing during Phase II that was independent of any thermal viscosity effects. The investigators hypothesized that the sonication treatment had chemically changed the oils by creating a greater amount of lighter hydrocarbons. In this test series, a combination of headspace analysis, gas chromatographic (GC) separation, and flame ionization detection (FID) was used to determine if indeed the percentage of lighter hydrocarbons had increased thereby reducing the viscosity of the oil. For these preliminary tests, a 30 weight refined oil, a 140 weight refined oil, and the same heavy California crude oil that was used in the viscosity tests described above were used.

In these tests, a quadruplicate set of oil samples was collected before treatment, the samples were sonicated a 1.5 liter oil volume for 10 minutes at 1250 Hz and 990 watts, and another set of quadruplicate oil samples was collected after treatment. The samples consisted of 12 mL of oil in a sealed 20 mL vial. The headspace sampler heated and pressurized the vial to increase the volatilization of the lighter hydrocarbons. The headspace sampler supplied the GC with the resultant gas for separation and the FID quantified the amount of each analyte released by the sample.

Figures 39 through 44 show sample chromatograms of oils before and after treatment obtained during these tests. As would be expected, the two processed oils had far fewer volatile hydrocarbons (Figures 39 and 41) than the crude oil (Figure 43) resulting in much cleaner chromatograms with fewer peaks. The 140-weight oil's peaks occurred later in the chromatogram than did the 30-weight oil's peaks, which likely indicates that the 30-weight oil

had a larger fraction of lighter, more volatile hydrocarbons as would be expected from a thinner oil.

Statistical comparison of the peak areas before and after treatment demonstrated with a 95% confidence level that the sonic treatment of the 30-weight oil did significantly increase the volatile hydrocarbon represented by the peak at 1.15 minutes (Figures 39 and 40). The area of this peak increased by an average of 18%. Sonication did not result in a significant difference (to a 95% confidence level) in any of the peak areas in the 140-weight oil (Figures 41 and 42). The GC-FID identified five separate peaks representing volatiles in the 30-weight oil and six peaks in the 140-weight oil.

GC-FID analysis of the crude oil samples identified as many as 75 different peaks. Statistical comparison of all areas of these peaks indicated that areas for 8 different peaks (1.08, 1.21, 1.65, 2.07, 2.48, 2.63, 2.72, and 3.42 minutes) were statistically different at a 95% confidence level (Figures 43 and 44). Thus, even though the areas of many of the peaks increased after treatment, only about 10% of these increases were statistically significant at the 95% level. The areas of those peaks that were significantly different after treatment showed increases between 8% and 32%.

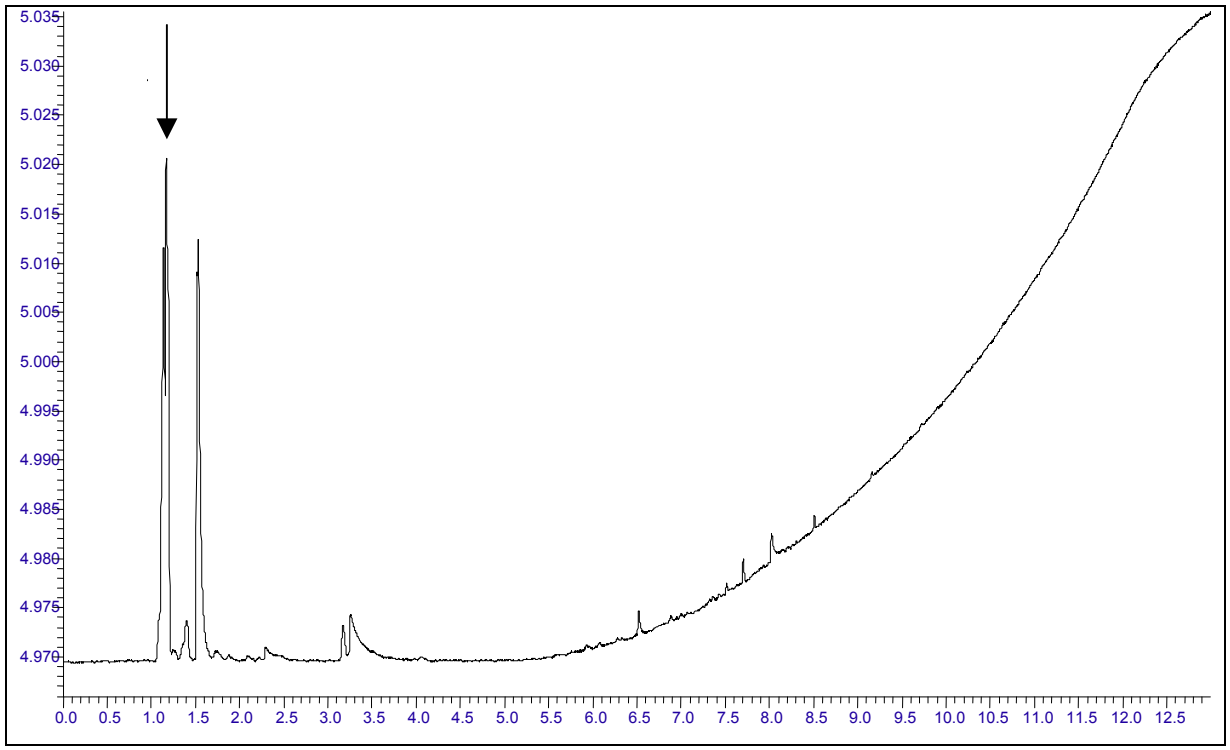


**Figure 39 Chromatogram of SAE 30-Weight Oil Sample Before Acoustic Treatment**

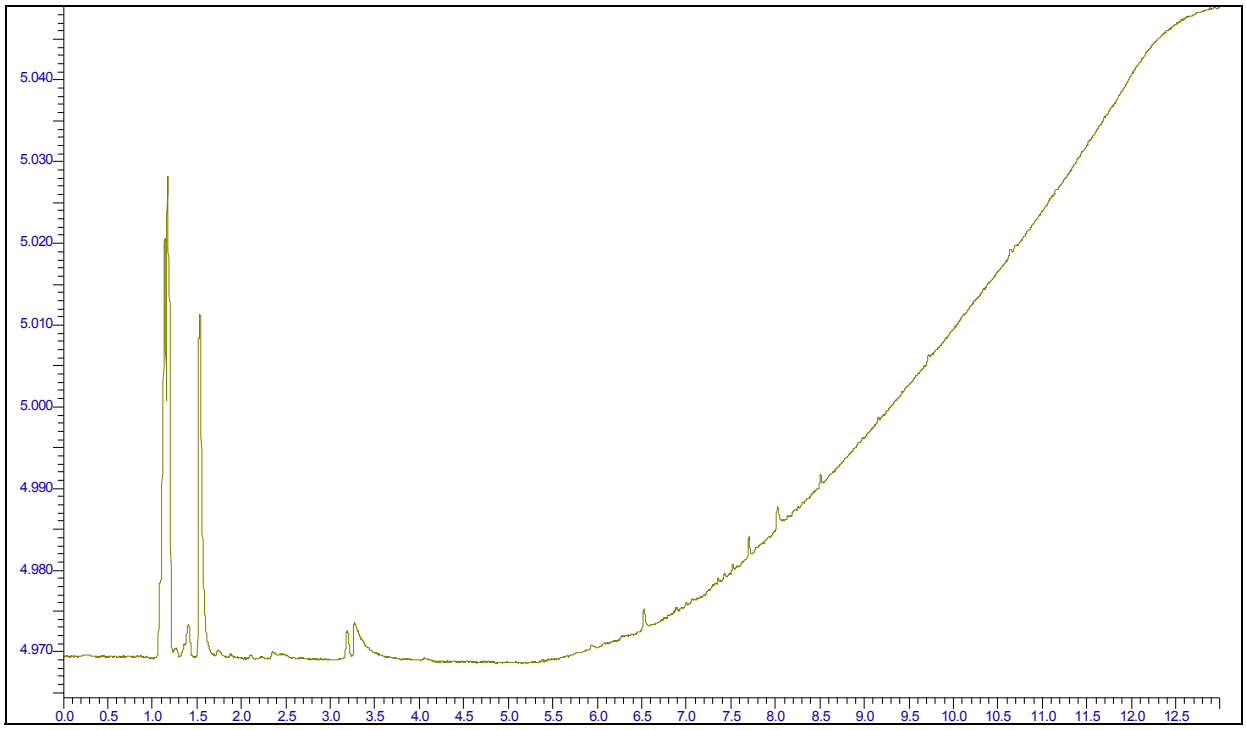


**Figure 40 Chromatogram of SAE 30-Weight Oil Sample After 10 Minutes of Acoustic Treatment**

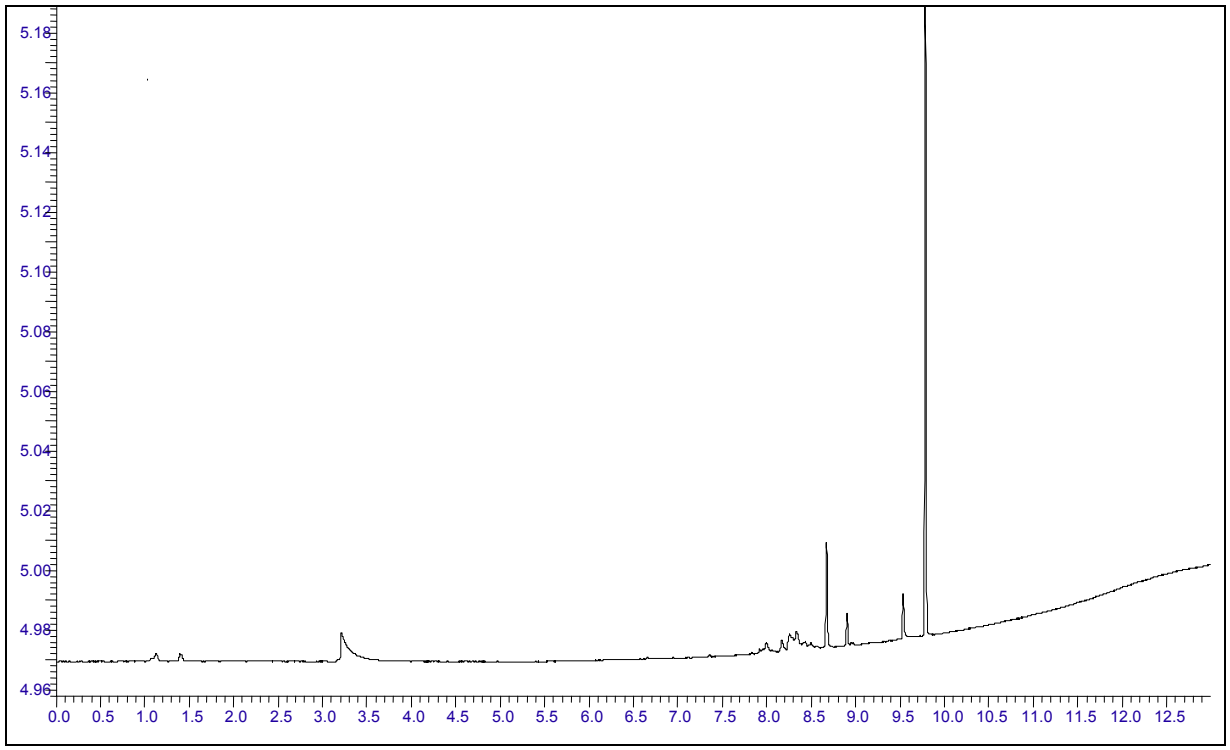


**Figure 41 Chromatogram of EP 140-Weight Oil Sample Before Acoustic Treatment**

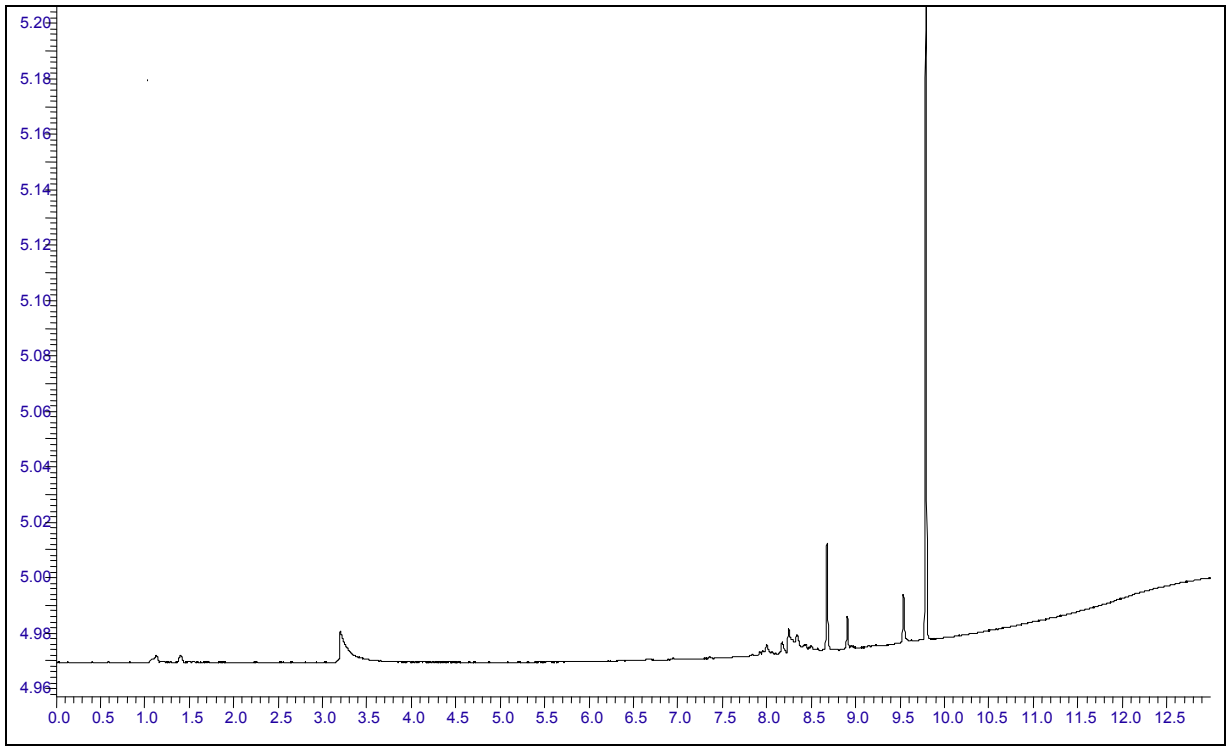


**Figure 42 Chromatogram of EP 140-Weight Oil Sample after 10 Minutes of Acoustic Treatment**

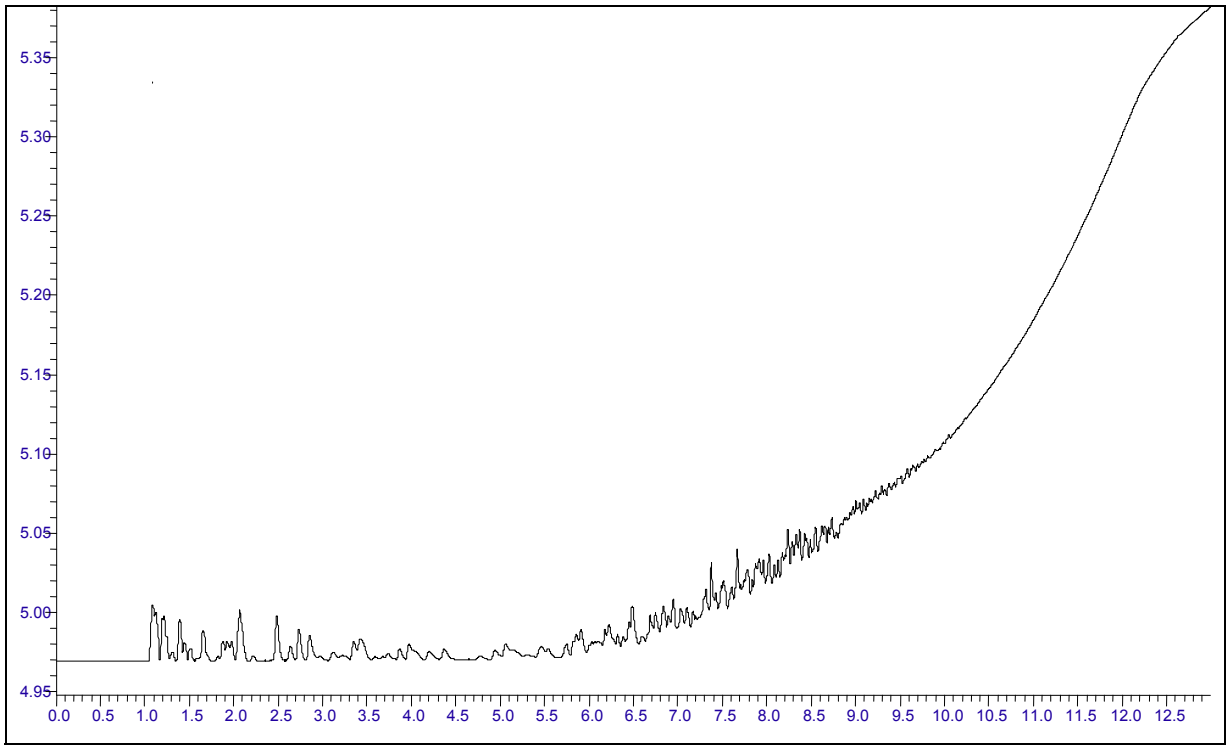


**Figure 43 Chromatogram of Crude Oil Sample Before Acoustic Treatment**

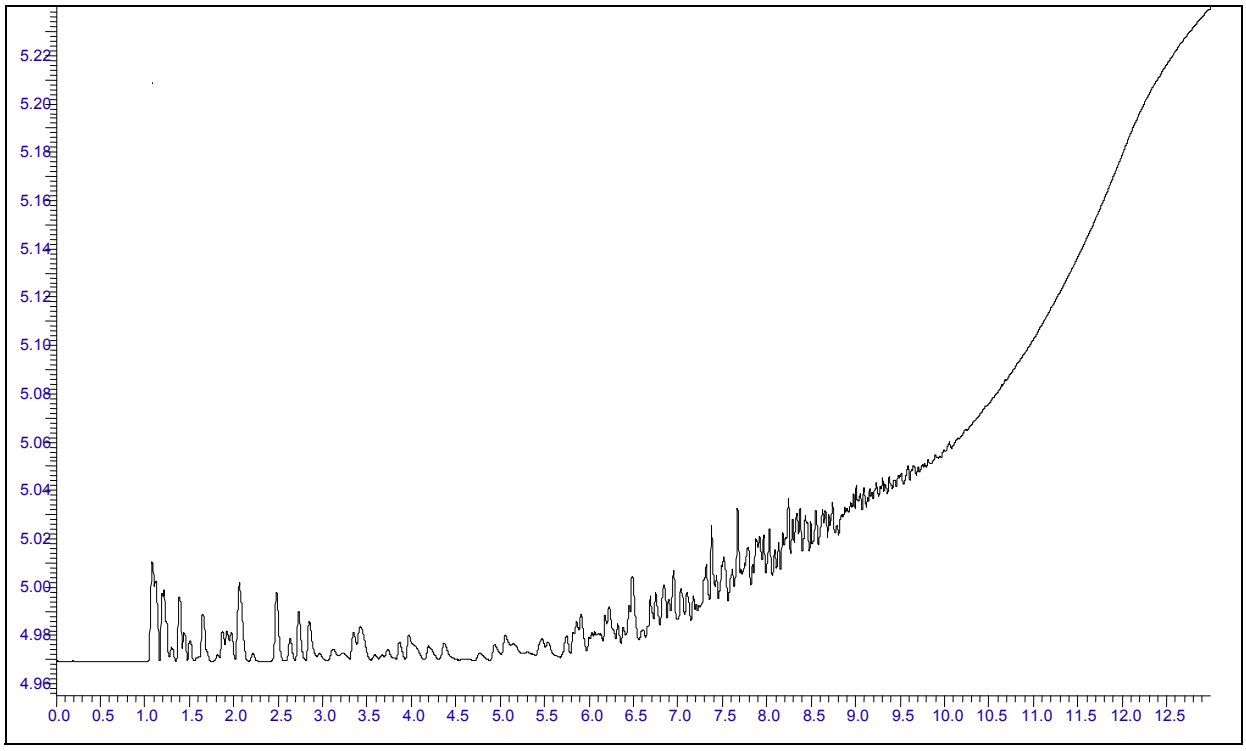


**Figure 44 Chromatogram of Crude Oil Sample After 10 Minutes of Acoustic Treatment**

Figure 45 compares the areas of the eight significantly different peaks observed in the crude oil chromatogram; Figure 46 shows the amount that the areas of these peaks increased in absolute terms (counts) and in percentage terms. The later peaks (greater than 3.5 minutes) in the heavy crude oil's chromatogram showed no significant difference and were poorly resolved. Although these test results are not completely conclusive, the observed increase in these lighter hydrocarbon concentrations provide plausible evidence that sonication can reduce oil viscosity through chemical change.

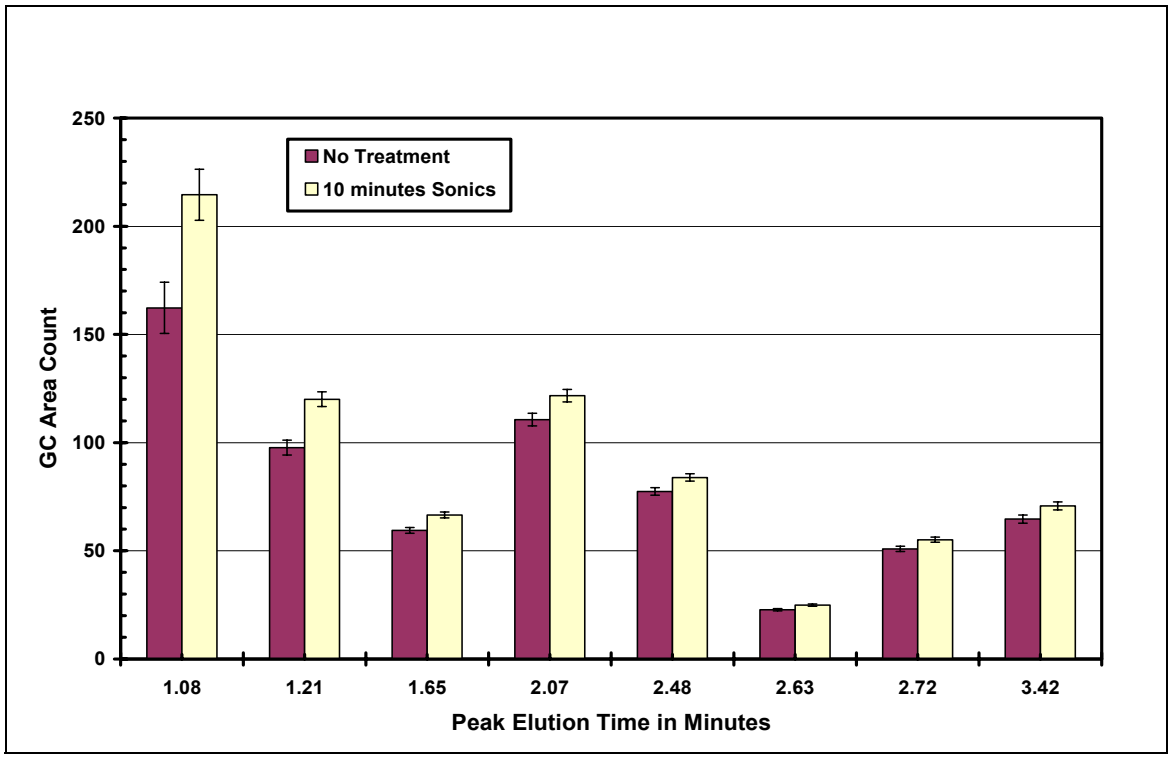


**Figure 45 Areas of Peaks Obtained by GC-FID Analysis of Heavy Crude Oil Before and After Sonication**

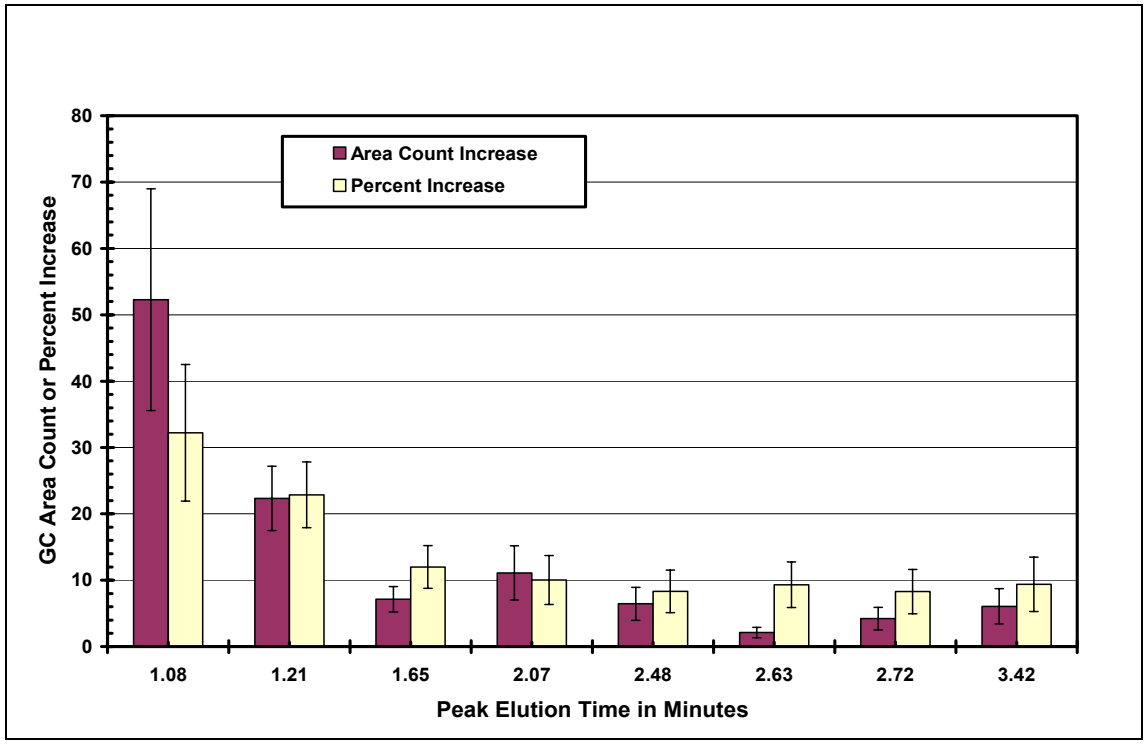


**Figure 46 Absolute (Area Counts) and Percent Increases in Peak Areas Obtained by GC-FID Analysis of Heavy Crude Oil Before and After Sonication**

## 3.6 Experimental Methods and Materials

### 3.6.1 Materials and Equipment

All of the Phase II laboratory experiments were performed at the University of Alabama at Birmingham (UAB) with guidance and oversight from TechSavants and the other project team members. The sonication system that was designed, fabricated, and evaluated at Furness-Newburge facilities in Versailles, Kentucky was shipped to UAB for use during the experimental program. Figure 47 shows the reaction chamber in a fume hood where all of the experiments were conducted. Compare this photograph with the design drawing given in Figure 34. As shown in the drawing, each of the three cylindrical members of the reaction chamber is designed to contain an actuator for producing acoustic energy. As shown in Figure 34, the horns attached to actuators extend into the central chamber containing the crude oil being exposed to the acoustic energy. The same power supplies, actuators, and horn designs that were used during the Phase I experiments (see Figures 6, 7, and 8) were used in Phase II. Each of the cylindrical members is designed to allow water to circulate around the actuator for cooling purposes, but this space is sealed off from the central chamber so that water does not mix with the oil during testing. The apparatus shown below is approximately 30 inches (76 cm) tall and each of the cylinders measures approximately 1 ft. (30.5 cm) in length from the end cap to flange base and about 3.5 in. (9 cm) in diameter.

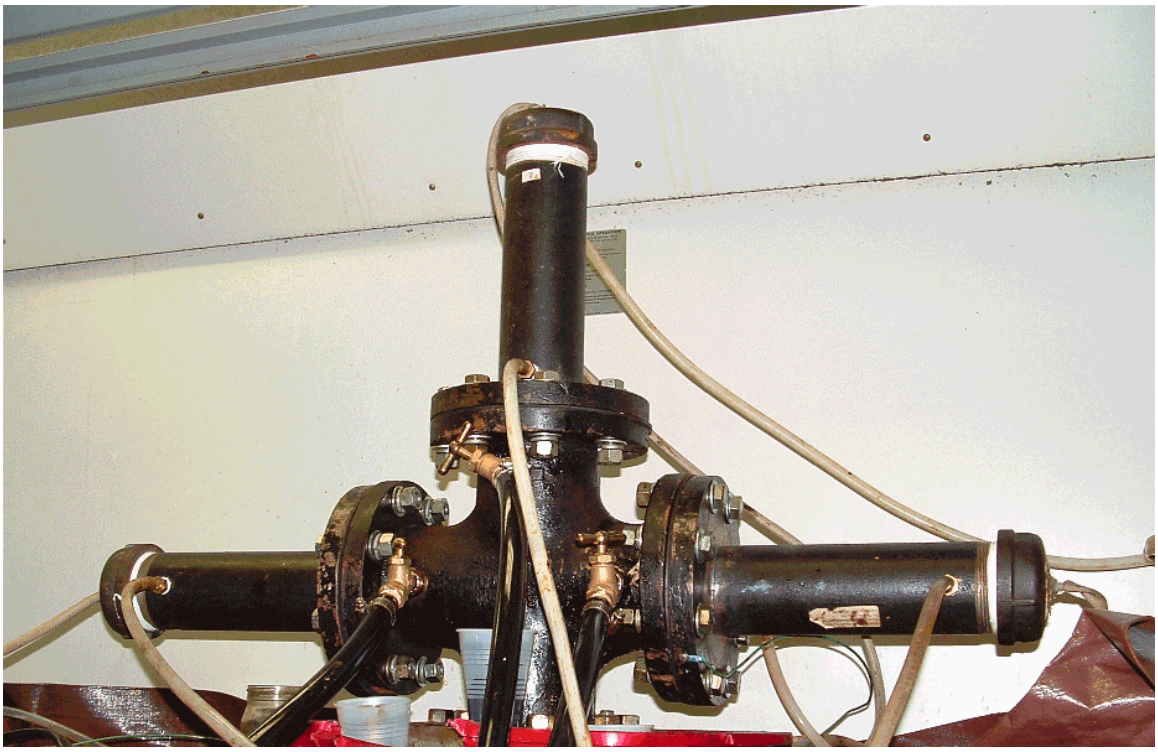

**Figure 47 Crude Oil Reaction Chamber used during the Phase II Testing Program**

Two water lines connect to each of the three cylindrical members to circulate the cooling water to and from each member. Each actuator in the apparatus is connected to a power supply via two electrical wires contained within a protective fabric cladding. These can be seen entering through the three end caps of the cylinders in Figure 47. In addition, plastic tubing is attached to the central reaction chamber to transfer oil into and out of the chamber during testing. Each tube passes through a shut-off valve mounted on the outer surface of the chamber to control the flow of oil.

Figure 48 contains a schematic drawing of the experimental apparatus used in Phase II for testing crude oils. Note that water lines are connected to each of the three tubular chambers that house the actuators, but to keep the drawing simple, water lines to only one chamber are shown. Also, the power supplies and power lines to the actuators within each tubular chamber are also omitted from the drawing for simplicity. The supply tank has a capacity of approximately 10 gallons (38 liters). During a test, oil is moved from the supply tank to the reaction chamber by the positive displacement pump that has a maximum rating of six gallons (22.7 liters) per hour. The oil moves through the reaction chamber where sonic energy is delivered to the fluid via the actuator horns. The oil then moves through tubing to the post-treatment container where it is stored temporarily until the experiment is complete. Samples to measure the sonication effects on the crude oil are collected after the oil leaves the reactor and before it enters the post-treatment container. The supply tank and pump were attached to a fabricated metal stand constructed with unitstrut (http://www.unistrut.com) and with castors/wheels on the bottom to allow the unit to be easily moved from one location to another. These components were connected to the reaction chamber housed in a fume hood during testing by the plastic tubing used to move the crude oil through the system.

It will be recalled that viscosity was measured in Phase I using dip cups. It was decided that a more accurate method for obtaining viscosity data would be employed in Phase II. A Brookfield Digital Viscometer (Model DV-E) was purchased for this purpose from Brookfield Engineering Laboratories in Middleboro, Mass. A photograph of the viscometer in operation during the testing program is shown in Figure 49. This instrument is provided with several spindles of differing shapes. The viscometer operation involves rotating one of the spindles that is immersed in the test fluid through a calibrated spring that measures the viscous drag against the spindle by the spring deflection. The amount of deflection in the spring is measured with a rotary transducer that provides a torque signal. Viscosity data are provided in units of centipoises (cP). Additional information on viscosity and viscosity measurement methods and equipment can be found on the Brookfield web site at http://www.brookfieldengineering.com and a copy of the operating instructions manual for the Model DV-E is given in Appendix E.

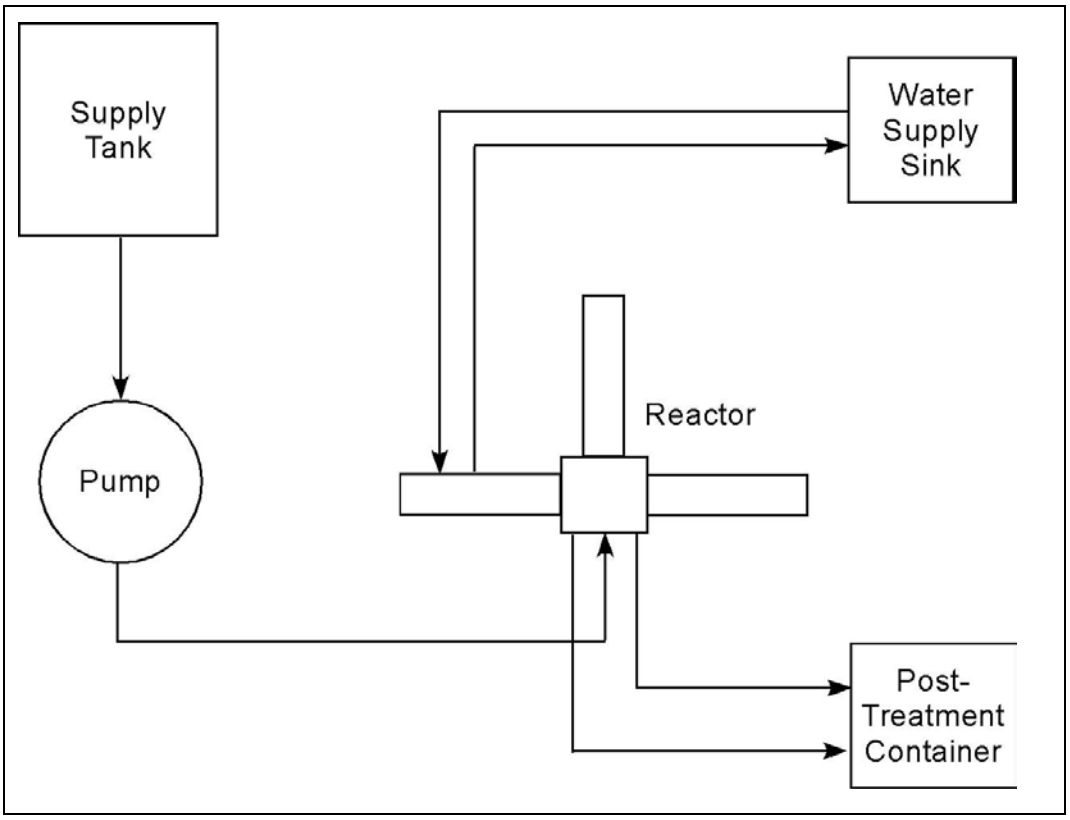


**Figure 48 Schematic Drawing of the Phase II Experimental Apparatus**

**Figure 49 Brookfield Digital Viscometer in Use in the Laboratory**

Three different crude oils were used in the Phase II experiments. These were selected to represent oils with low, medium, and high values of viscosity in order to evaluate the ability of sonication to change viscosity with a range of initial viscosity conditions. The identification numbers, source locations, and representative viscosities of the three crude oils used in these tests are given in Table 10. Each experiment that was conducted required approximately eight gallons (30 liters) of oil.

**Table 10 The Three Crude Oils used in the Phase II Testing Program**

| Crude Oil Identification No. | Source Location of Oil | Initial Viscosity, (cP) |
|---|---|---|
| 1 | Bakersfield, California | 65,300 |
| 2 | Gilbertown, Alabama | 6,000 |
| 3 | Middle East | 700 |

### 3.6.2 Methods and Procedures

A series of tests was designed to evaluate the effects of sonication/acoustic frequency, horn design, arrangement of actuators within the reaction chamber, chemical additives, and input power levels on each of the three crude oils. The experimental plan that was implemented was based on the results of Phase I of the project, the content of the proposal as submitted to the sponsor as well as the results from the initial testing of the equipment during the early tasks of Phase II. Furthermore, the experimental plan called for performing the initial suite of tests using Crude Oil 1 to be followed by a project team meeting to evaluate the experimental work and results to determine if changes should be implemented in order to improve the utility of the results obtained during the testing of the remaining two oils (see discussion in Section 3.3).

The following Phase II experimental procedures, unless noted to the contrary, were utilized to evaluate the effects of the identified independent variables on the viscosity of the three crude oils. At the outset of each test, crude oil was placed in the supply tank. The pump was started and operated at a constant flow rate. As oil began flowing into the reaction chamber, the power supplies attached to the actuators in the reactor were turned on initially to a 5-10% power level. The transducers were operated for a short period of time at this low power level to allow them to warm up thereby preventing damage that could result from a quick input of high power. After the reaction chamber was filled and oil began to flow from the reactor to the collection/post-treatment container, the power to each actuator was gradually increased to a maximum of approximately 90% of total power, which was the normal operating condition. This point in time was noted as the beginning of the experiment.

For each independent variable that was being evaluated (frequency, horn design, etc.), the initial test plan called for collection of an oil sample at 0 minutes (background/initial sample),

and at 30-minute intervals thereafter until an individual test was terminated after a total duration of 120 minutes. Thus, for each experiment five individual samples were collected in plastic cups at times of 0, 30, 60, 90, and 120 minutes. At the start of each experiment, a sufficient quantity of crude oil for the test was added to the supply tank. The initial (time = 0 minutes) sample was collected, the oil temperature was recorded, and the viscosity was measured. After 30 minutes, the first sample was collected and the oil temperature and viscosity were measured. These steps were repeated for the remainder of the samples.

Each sample collected was approximately 12 ounces (0.35 liters) in volume. Immediately after each sample was collected, its temperature was measured and recorded and the viscosity was measured using the Brookfield viscometer. The initial plan called for the first sample that was collected immediately before a test began and the final sample collected at time 120 minutes to be analyzed using a Gas Chromatograph/Mass Spectrometer (GC/MS) after the temperature and viscosity were determined. This was intended to provide insight into any chemical changes in the oil that would have occurred as a result of the sonication treatment. Unfortunately however, due to the large molecular weight of the oil and other factors, the attempts to use the available GC/MS equipment and analytical resources in place at UAB to analyze the crude oil samples were unsuccessful, and the results were inconclusive. As a result of these difficulties, no direct evidence regarding chemical changes in the crude oils resulting from sonication could be collected from these tests.

It will be recalled that the Phase I experiments were performed in batch mode. A quantity of oil was added to a container, it was sonicated for five minutes, and a sample was collected and analyzed. The container of oil was sonicated for another five minutes, and a second sample was collected and analyzed. This process was repeated until the experiment was concluded. The project team decided that the testing conducted during Phase II would be done in a manner to approximate a continuous flow reactor because this would, in all likelihood, be the preferred mode for implementing the technology within full-scale operations. The reactor system was designed with this approach in mind, and each Phase II experiment was performed in a flow-through mode as described above.

As noted previously, the pump rate was set at 2.5 gallons/hr (9.5 liters/hr). This was a constant for all tests. It also was previously stated that the volume of the reaction chamber was approximately one gallon (3.8 liters). Therefore, during each test, the reaction chamber would be filled with incoming oil 2.5 times each hour of testing (five times during a 120-minute test), which equates to a residence time for the oil in the reaction chamber of 24 minutes. In order to facilitate the interpretation of test results, it must be assumed that the flow through the reaction chamber was uniform and consistent throughout the chamber volume; that is, as fresh oil moves into the chamber, an equivalent volume simultaneously moves out. It must also be assumed that the residence time of each particle of oil that enters the chamber is 24 minutes so that none of the oil resides longer nor leaves the chamber sooner (equivalent to an idealized plug flow reactor).

Although these ideal conditions are probably not actually obtained in all of the experiments, the assumptions are not unrealistic and do not introduce significant bias into the data interpretation. As a consequence, the maximum exposure of any unit volume (chamber volume) of oil to the acoustic energy during an experiment was approximately 24 minutes. Looking at these test conditions another way, one can assume that during any one-minute time period, approximately 4.2% (1/24) of the chamber volume of oil enters the chamber as an equivalent volume leaves the chamber. Under the assumed ideal conditions, after 23 more minutes this fractional volume leaves the chamber after having been exposed to sonication for a period of 24 minutes. This process is repeated every minute that the test continues. Therefore, given the assumption of ideal conditions, each sample of oil that is collected after leaving the reaction chamber has been exposed to sonication for 24 minutes. This explains the fact that, as will be seen, most test results indicate that the viscosity did not change a large amount as individual tests progressed as reflected in the samples collected from times 30 through 120 minutes. One can argue that if the experiments had been designed with a lesser flow rate, such as one gallon per hour (3.8 liters/hr) for example, the amount of viscosity reduction could have been greater because the residence time of the oil in the reaction chamber would have increased from 24 to 60 minutes. On the other hand, the results from the Phase I tests indicate that the bulk of the reduction in viscosity was accomplished during the first 20-25 minutes of the tests with the viscosity changing only modest amounts after that time. This observation was a significant factor in developing the Phase II experimental procedures that could provide additional data to corroborate this conclusion and demonstrate the stability of the acoustic reaction system and its ability to maintain the viscosity reductions under continuous flow conditions.

Finally, it is generally agreed that for any continuous flow system, it normally takes at least three residence times for the system to approach steady state conditions. In the system used in the Phase II experiments as described immediately above, one residence time was about 24 minutes; therefore, three residence times equate to approximately 72 minutes. Given this relationship, the data from samples collected at 90 and 120 minutes should be the most indicative of the performance of the reactor system utilized in the tests.

## 3.7 Experimental Results Using Crude Oil 1

### 3.7.1 Temperature Effects on Viscosity

Before the Phase II testing with the sonication system began, the effects of heat on the crude oils were measured. In each case the oil was heated and the viscosity measured in samples collected at pre-determined temperatures. In addition, the personnel at Brookfield Engineering Laboratories agreed to analyze a sample of Crude Oil 1 to demonstrate the operational robustness and reproducibility of their viscometer. The data obtained by Brookfield are presented in Table 11.

**Table 11 Viscosity of Crude Oil 1 as a Function of Spindle Speed and Temperature (Data Collected by Brookfield Engineering Laboratories)**

| Temperature, (ºC) | Spindle Speed, (rpm) | Viscosity, (cP) |
|---|---|---|
| 20.8 | 1.0 | 60,200 |
| 20.7 | 0.5 | 60,400 |
| 20.7 | 1.0 | 60,200 |
| 20.6 | 2.5 | 60,160 |
| 20.6 | 1.0 | 60,400 |
| 20.7 | 0.5 | 60,400 |
| 20.7 | 1.0 | 60,400 |
| 20.6 | 2.5 | 60,320 |
| | | |
| 50.1 | 2.0 | 1,773 |
| 50.1 | 10.0 | 1,773 |
| 50.1 | 15.0 | 1,766 |
| 50.1 | 10.0 | 1,766 |
| 50.2 | 5.0 | 1,766 |
| | | |
| 93.9 | 50.0 | 89.0 |
| 93.9 | 100.0 | 89.0 |
| 93.9 | 200.0 | 88.3 |
| 93.9 | 100.0 | 89.0 |
| 93.9 | 50.0 | 89.0 |

The data in this table reflect the consistency and reproducibility of viscosity measurements for three different temperatures and with several spindle speeds for each. It can be seen that very little variation in the measured viscosity values is present as spindle speeds change at a given temperature. Further, repeat measurements at given temperature-spindle speed conditions produce consistent and reproducible results. To illustrate the measured change in viscosity with temperature, values indicative of each of the three temperature-viscosity groups from the table are plotted in Figure 50. This data plot clearly shows the negative exponential relationship between the two variables as one might expect. This observation is consistent with the data collected during Phase I of the project.

Similar tests to those described above were also performed in the UAB laboratory. However, the UAB tests included both heating and cooling tests and measurements to determine

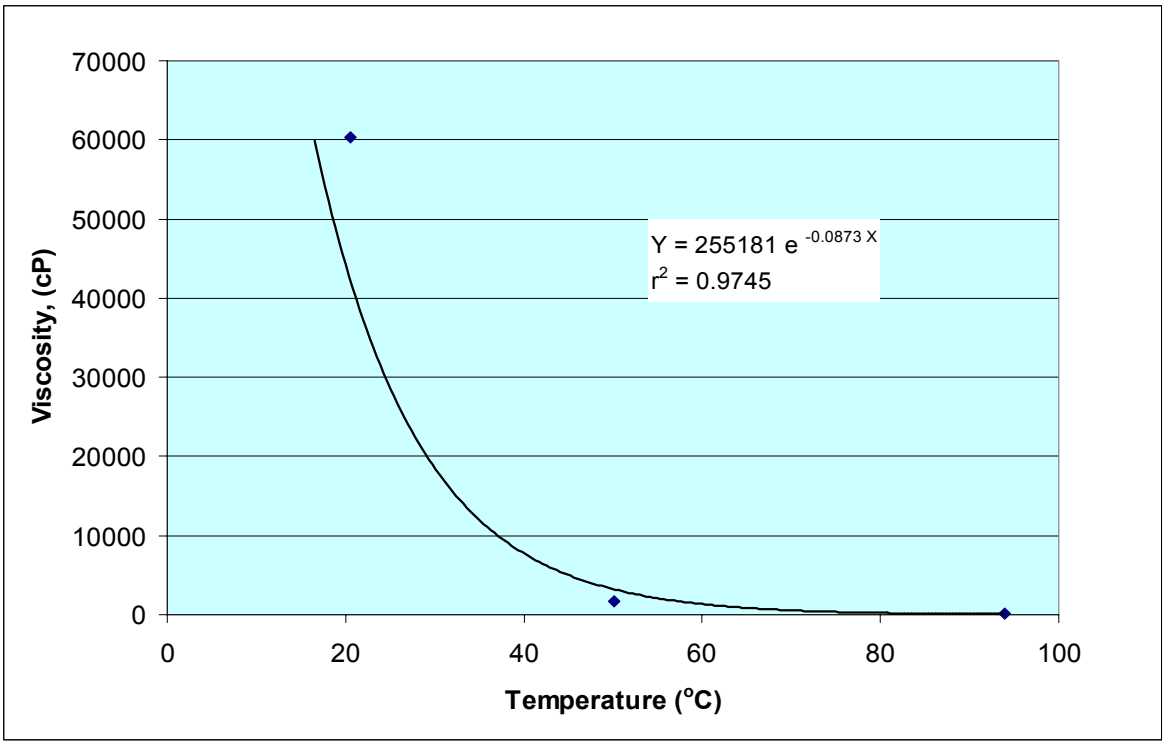


**Figure 50 Relationship Between Viscosity and Temperature for Crude Oil 1 (Data Collected by Brookfield Engineering Laboratories)**

if there were hysteresis effects in addition to comparing the results of the UAB heating test results with those provided by Brookfield. The UAB data are contained in Table 12. Figure 51 is a graph of the temperature-viscosity data and Figure 52 contains the exponential regression results from these same data. These data indicate that the initial viscosity prior to beginning the heating tests was about 68,900 cP at a temperature of 21.5ºC. After heating the oil to a maximum of 100ºC, the viscosity was reduced to only 144 cP, a reduction of 99.79%. The most significant decrease in viscosity was associated with the temperature range of approximately 20ºC to 40ºC; at 40ºC the viscosity was reduced to 7,350 cP, a reduction of 89.33%. Figure 51 also indicates that there is an observable hysteresis effect between the heating and cooling viscosities at temperatures less than 60ºC; viscosity values at temperatures less than 60ºC are less at the same temperatures during the cooling cycle than those during the heating cycle. This graph also indicates that the final viscosity after cooling to 22.5ºC was appreciably less (13.91%) than the viscosity at the same temperature at the beginning of the heating cycle. In addition, a comparison of the regression curve for the Brookfield data (Figure 50) to those for the UAB data (Figure 51) indicates that the results are quite similar, adding credence to the data obtained at UAB using the Brookfield viscometer.

**Table 12 Viscosity of Crude Oil 1 as a Function of Temperature During Heating and Cooling (Data Collected by UAB)**

| Heat Applied | | Cooling Cycle | |
|---|---|---|---|
| Temperature, (°C) | Viscosity, (cP) | Temperature, (°C) | Viscosity, (cP) |
| 21.5 | 64,870 | | |
| 22.5 | 51,107 | 22.5 | 44,000 |
| 25 | 16,700 | 25 | 16,400 |
| 30 | 12,000 | 30 | 9,360 |
| 35 | 11,170 | 35 | 7,260 |
| 40 | 7,350 | 40 | 3,220 |
| 50 | 6,600 | 50 | 957 |
| 60 | 1,270 | 60 | 629 |
| 70 | 440 | 70 | 302 |
| 80 | 376 | 80 | 243 |
| 90 | 188 | | |
| 100 | 144 | | |

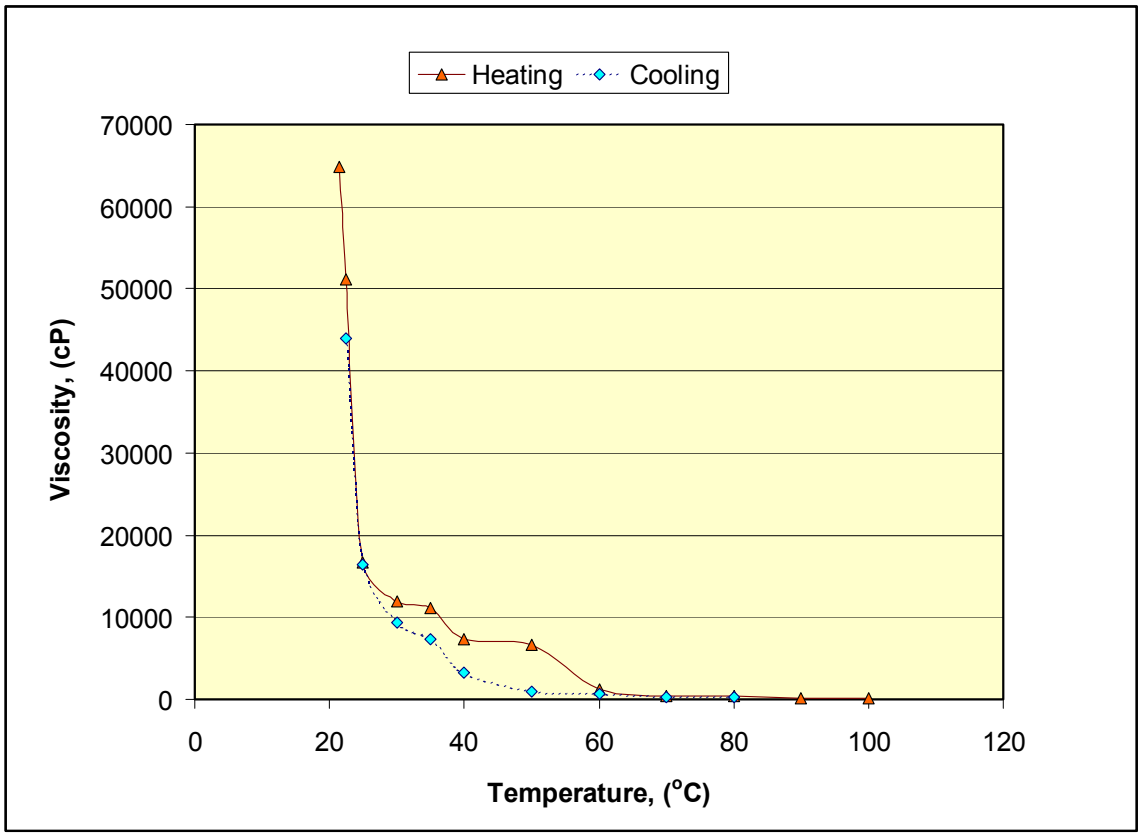


**Figure 51 Plot of Viscosity vs. Temperature for Crude Oil 1 Collected by UAB**

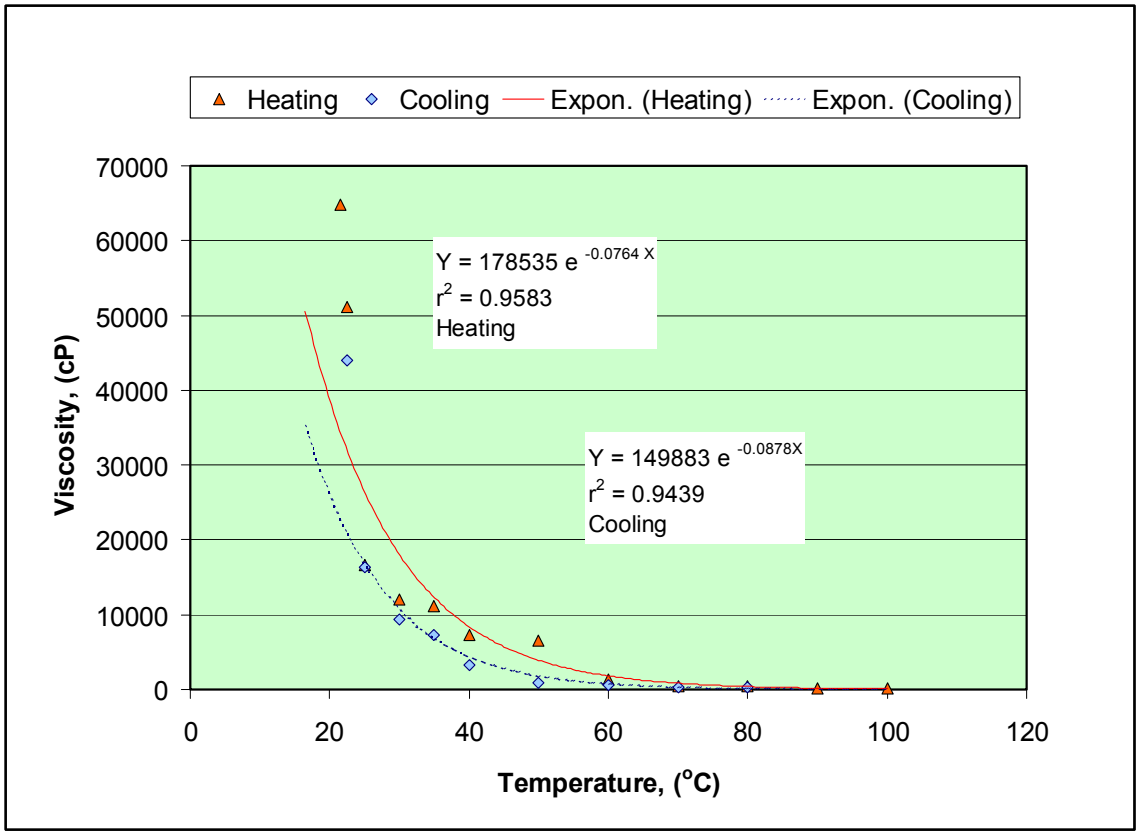


**Figure 52 Plot of the Regression Results of Viscosity and Temperature Data for Crude Oil 1 (Data Collected by UAB)**

### 3.7.2 Sonication Effects on Viscosity

A series of tests was designed to evaluate the effects of number and positions of actuators within the reactor, acoustic frequency, horn design, reduced power levels, the addition of chemical additives, and treatment time. Because the volume of Crude Oil 1 available for testing was limited, and due to the serious difficulties (both logistically and in terms of project schedule) involved in obtaining additional large volumes of crude oil, it was necessary to use some of the oil in multiple tests. It was reasoned that after the results of Oil 1 were evaluated it should be possible to optimize subsequent experiments by eliminating some test conditions that had minimal or no effects on the viscosity of Oil 1. Therefore, it seemed likely that some of the projected tests involving the second and third crude oils could be eliminated, thereby eliminating the necessity to reuse oil samples during experiments on Oils 2 and 3. Given this less than ideal, but necessary condition, it was decided that the initial tests using Oil 1 to examine the effects of sonication frequency and actuator arrangements would be conducted using only one fresh Crude Oil 1 sample. After the first test was completed, the same oil was used in the second test and all subsequent tests until the series of ten individual experiments (see Table 13) was completed. The oil was allowed to sit for a minimum of 12 hours between tests. Although this was less

desirable that using a fresh sample of oil for each individual test, this experimental design also provided data on the effects of exposing the oil to sonication for a longer period of time. At the conclusion of this series of tests, the oil sample had been exposed to sonication for a total of 20 hours (1200 minutes).

Table 13 presents the basic test conditions of number of actuators, position/locations of actuators within the three possible locations in the oil reaction chamber (see Figure 47), and acoustic frequencies that were used during experiments with Crude Oil 1. All tests used the standard one-inch (2.5 cm) spacing between two slotted horn fins. This test design formed the basis for the remaining tests to evaluate the added effects of horn design, power levels, time, and additives. It should be noted that due to the sensitivity of the equipment and difficulty in controlling precisely the acoustic frequency output with the power supplies used in these tests, there were occasions when the frequency was not exactly equal to these test conditions. In these cases, the actual frequency used during an experiment is reported, not the standard frequency in the test plan. For this reason, the reader may note that some test conditions reported in some of the data tables do not match exactly the conditions in Table 13.

**Table 13 Sonication Test Conditions for Crude Oil 1**

| Sonication Treatment Conditions Investigated |
|---|
| 3 actuators @ 0.8 kHz |
| 3 actuators @ 1.4 kHz |
| 3 actuators @ 1.8 kHz |
| 2 actuators parallel @ 0.8 kHz* |
| 2 actuators parallel @ 1.4 kHz* |
| 2 actuators parallel @ 1.8 kHz* |
| 2 actuators parallel @ 0.8 kHz and 1.2 kHz* |
| 2 actuators parallel @ 0.8kHz and 1.6 kHz* |
| 2 actuators parallel @ 1.2 kHz and 1.6 kHz* |
| 3 actuators @ 0.8 kHz, 1.2 kHz, and 1.6 kHz# |
| * Acoustic transducers/actuators placed in a horizontal arrangement, facing each other with no transducer in the vertical position |
| # Acoustic transducers/actuators operating at 0.8 and 1.6 kHz placed in a horizontal arrangement facing each other; the transducer operating at 1.6 kHz placed in the vertical position facing downward |

The results of acoustic testing on the viscosity of Oil 1 are shown in Tables 14 and 15. Table 14 contains the viscosity values for the various treatment conditions as measured in the samples collected at 30-minute increments during each test. Table 15 contains data expressed in terms of fractional residual viscosity (as utilized in Phase I of the project). In this case, all viscosity values are divided by the initial viscosity measured at the beginning of each test.

**Table 14 Summary of Viscosity Results for Acoustic Treatment of Crude Oil 1**

| Treatment Conditions | Initial Temp. (°C) | Viscosity, (cP) | | | | |
|---|---|---|---|---|---|---|
| | | Time, (min) | | | | |
| | | 0 | 30 | 60 | 90 | 120 |
| 3 actuators @ 0.8 kHz | 21.9 | 69,800 | 67,547 | 68,160 | 66,773 | 65,403 |
| 3 actuators @ 1.4 kHz | 21.9 | 49,270 | 45,930 | 44,400 | 44,800 | 42,270 |
| 3 actuators @ 1.8 kHz | 21.7 | 63,930 | 45,577 | 44,223 | 43,467 | 42,643 |
| 2 actuators parallel @ 0.8 kHz | 21.9 | 64,070 | 37,470 | 39,300 | 38,560 | 39,280 |
| 2 actuators parallel @ 1.2 kHz | 21.9 | 48,470 | 39,240 | 38,320 | 36,880 | 37,480 |
| 2 actuators parallel @ 1.6 kHz | 21.9 | 47,330 | 42,070 | 38,480 | 40,600 | 34,840 |
| 2 actuators parallel @ 0.8 kHz and 1.2 kHz | 22.5 | 39,520 | 25,360 | 23,040 | 22,920 | 22,520 |
| 2 actuators parallel @ 0.8 kHz and 1.6 kHz | 22.2 | 48,500 | 42,470 | 39,320 | 37,800 | 32,600 |
| 2 actuators parallel @ 1.2 kHz and 1.6 kHz | 22.8 | 42,930 | 38,130 | 38,270 | 37,670 | 37,200 |
| 3 actuators @ 0.8 kHz, 1.2 kHz, and 1.6 kHz | 21.5 | 47,530 | 41,800 | 41,930 | 40,200 | 39,600 |

**Table 15 Summary of Fractional Residual Viscosity Results for Acoustic Treatment of Crude Oil 1**

| Treatment Conditions | Initial Temp. (°C) | Fractional Residual Viscosity | | | | |
|---|---|---|---|---|---|---|
| | | Time, (min) | | | | |
| | | 0 | 30 | 60 | 90 | 120 |
| 3 actuators @ 0.8 kHz | 21.9 | 1 | 0.9677 | 0.9765 | 0.9566 | 0.9370 |
| 3 actuators @ 1.4 kHz | 21.9 | 1 | 0.9322 | 0.9012 | 0.9093 | 0.8579 |
| 3 actuators @ 1.8 kHz | 21.7 | 1 | 0.7129 | 0.6918 | 0.6799 | 0.6670 |
| 2 actuators parallel @ 0.8 kHz | 21.9 | 1 | 0.5848 | 0.6134 | 0.6018 | 0.6131 |
| 2 actuators parallel @ 1.2 kHz | 21.9 | 1 | 0.8096 | 0.7906 | 0.7609 | 0.7733 |
| 2 actuators parallel @ 1.6 kHz | 21.9 | 1 | 0.8889 | 0.8130 | 0.8578 | 0.7361 |
| 2 actuators parallel @ 0.8 kHz and 1.2 kHz | 22.5 | 1 | 0.6417 | 0.5830 | 0.5800 | 0.5698 |
| 2 actuators parallel @ 0.8 kHz and 1.6 kHz | 22.2 | 1 | 0.8757 | 0.8107 | 0.7794 | 0.6722 |
| 2 actuators parallel @ 1.2 kHz and 1.6 kHz | 22.8 | 1 | 0.8882 | 0.8915 | 0.8775 | 0.8665 |
| 3 actuators @ 0.8 kHz, 1.2 kHz, and 1.6 kHz | 21.5 | 1 | 0.8794 | 0.8822 | 0.8458 | 0.8332 |

Consequently, each initial viscosity has a fractional residual viscosity value of 1.0 and all other measurements reflect a percentage of that initial viscosity value remaining for each sample. Thus, a fractional residual viscosity 0.90 indicates that the viscosity has been reduced by 10%, or that 90% of the original viscosity is remaining in the sample. In some cases, it will be seen that the viscosity actually increased during a test because the fractional residual viscosity is greater than 1.0. It also should be stated that during this initial series of tests with Oil 1, the temperature of each sample was measured along with the viscosity, since it is known that temperature has a significant effect on viscosity. However, because the design of the reactor permitted each actuator to be cooled with water during operation, the temperature of the oil remained fairly constant during each test. Consequently, temperature is not reported for these tests beyond the initial temperature, and temperature was concluded not to be a factor in the experimental results.

The data in Table 14 are plotted in Figure 53. Both of the above tables and Figure 53 clearly show that the viscosity of Crude Oil 1 is reduced by all of the various treatments that were evaluated; but some treatments are obviously more effective than others. It is also possible to examine the changes in viscosity associated with various treatment conditions by looking only at the initial and final (120 minutes) viscosity values. These data are plotted in Figure 54 with the initial viscosity values shown in solid black and the 120-minute values shown with a pattern. Further insight into the effectiveness of the various treatments under the test conditions can be gained by looking at the total fractional amount of viscosity reduction (or amount of viscosity remaining) measured at the conclusion of each test. These data are shown in Figure 55. Note that in both Figures 54 and 55 the term “actuators” has been omitted from the test condition descriptions to save space within the illustration (compare to the legend in Figure 53). This convention will continue throughout the remainder of this report where appropriate.

Several observations can be made regarding the effects of the various sonication treatment conditions on Crude Oil 1. First, as Figure 55 illustrates, the reductions in viscosity after 120 minutes of testing range from a minimum of 6.3% (93.7% residual viscosity) to a maximum of 43.0% (57% residual viscosity). The test results indicate that treating the crude oil with three transducers all operating at the same frequency was relatively ineffective in reducing the viscosity of the crude oil for acoustic frequencies in the range of 0.8 to 1.4 kHz (reduction in viscosity <14.2 %). However, at the higher frequency (1.8 kHz), treatment with the three acoustic transducers showed an improved reduction in viscosity (33.3% reduction). An interesting feature shown in the data is the fact that treatment with two acoustic transducers arranged horizontally facing each other generally produced greater reductions in viscosity than treatment with three transducers arranged in a T-arrangement, with two transducers arranged horizontally, and one transducer located midway between these two horizontal transducers arranged vertically. Reductions in viscosity using the two horizontal transducers facing each other were in the range of 13.3% to 43.0%, with most experiments having reductions exceeding 25%. For this arrangement, operation at the lower frequencies (e.g., both transducers operating at 0.8 kHz, or one transducer operating at 0.8 kHz and the other at 1.2 kHz) gave the best

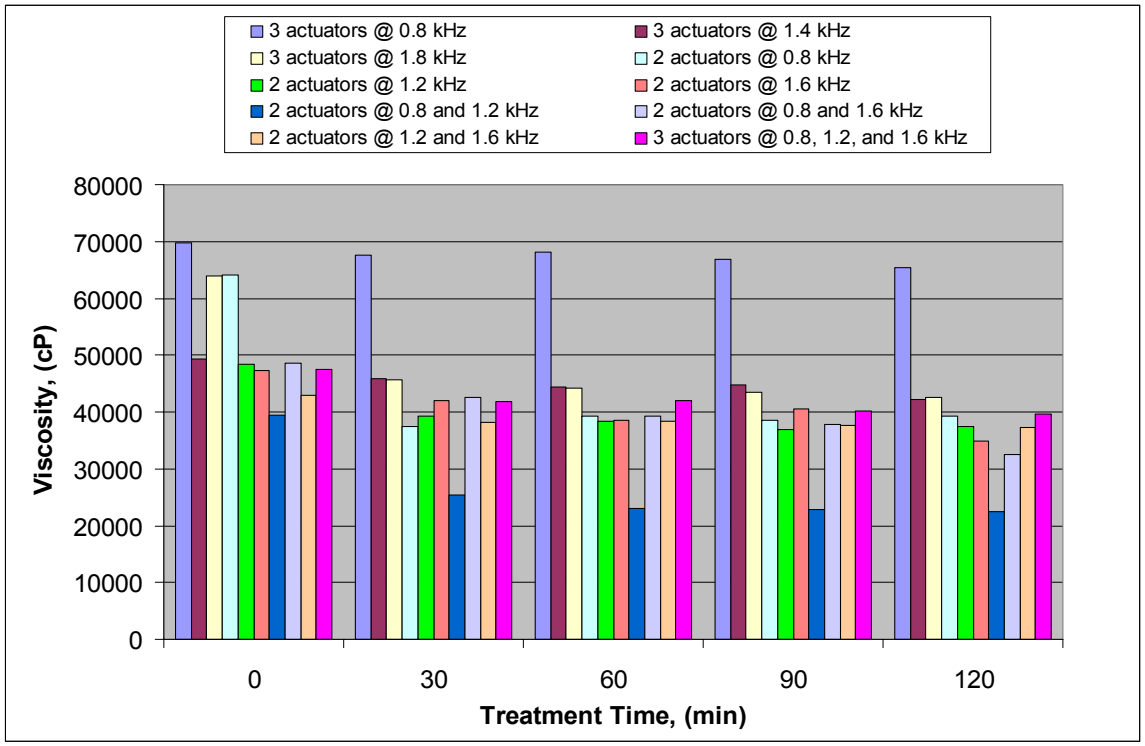


**Figure 53 Plot of Viscosity of Crude Oil 1 as a Function of Treatment Time for Various Acoustic Treatment Conditions**

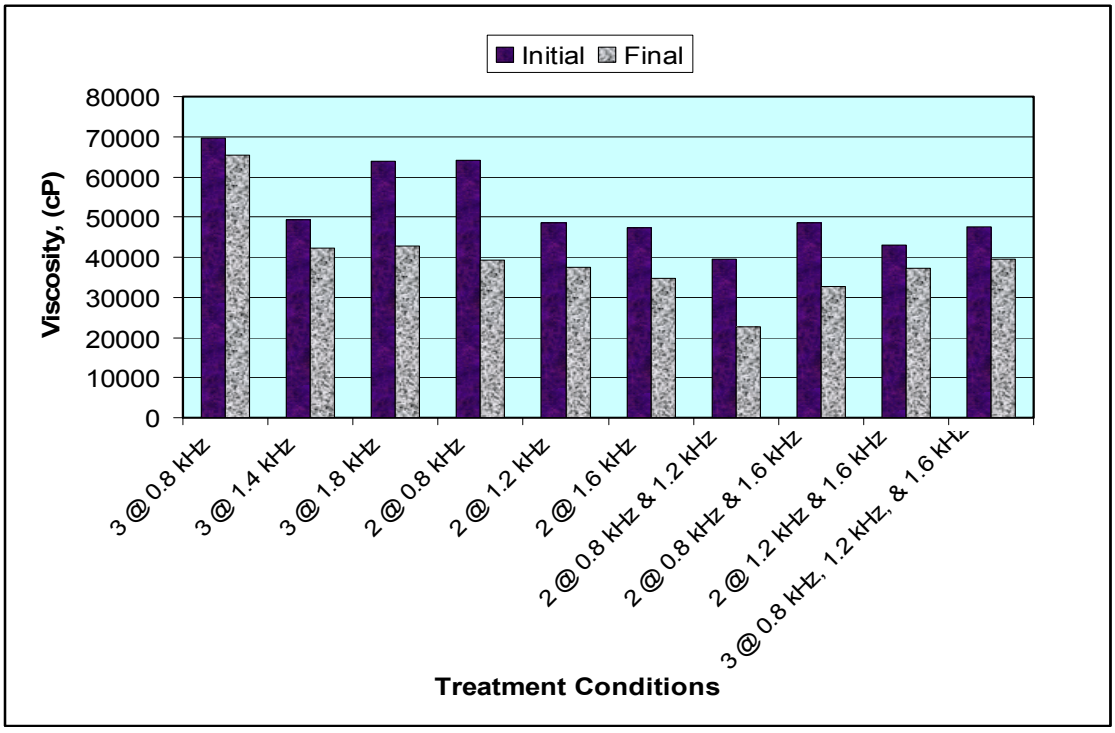


**Figure 54 Comparison of the Crude Oil 1 Initial Viscosity and Viscosity after 120 Minutes for the Various Treatment Conditions**

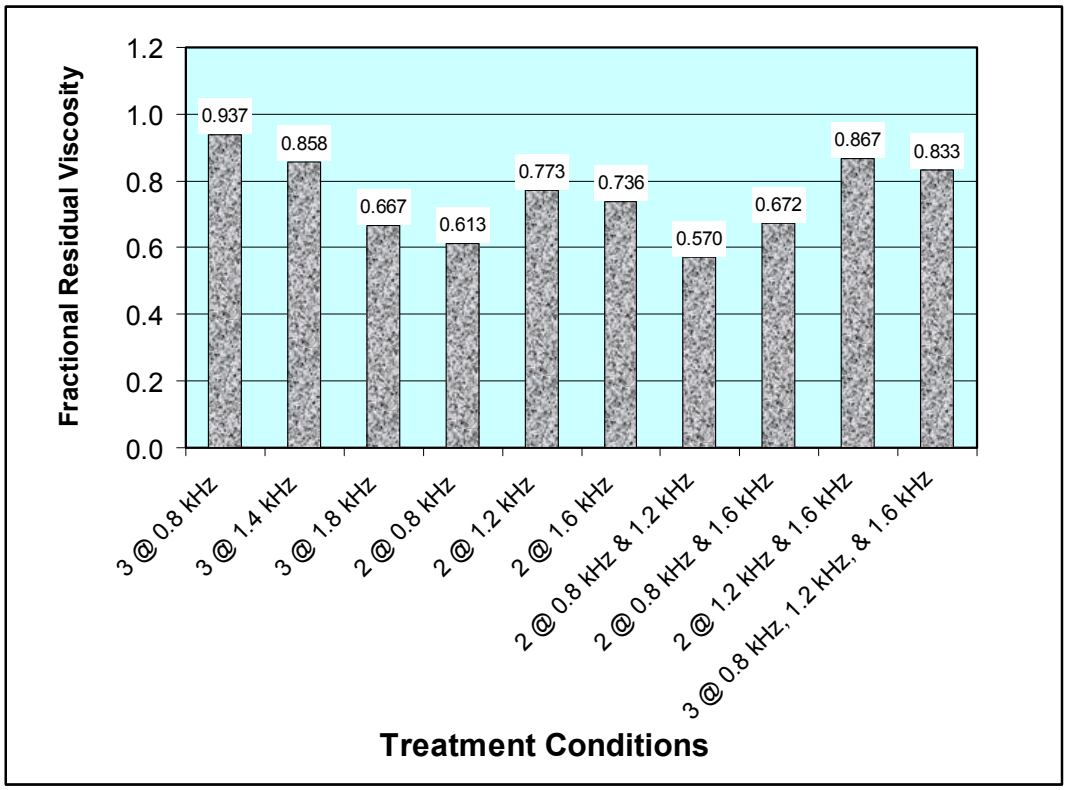


**Figure 55 Fractional Residual Viscosity of Crude Oil 1 after 120 Minutes of Treatment for the Various Treatment Conditions**

reductions in viscosity observed during these tests. Operating one transducer at 1.2 kHz and the second transducer at 1.6 kHz was not highly effective (13.3% reduction in viscosity). In summary, there were four treatment conditions from this series of tests that resulted in greater viscosity reduction than the remaining six: 1) three actuators operating at 1.8 kHz, 2) two parallel actuators operating at 0.8 kHz, 3) two parallel actuators operating at 0.8 and 1.2 kHz, and 4) two parallel actuators operating at 0.8 and 1.6 kHz.

As noted previously, due to the limited quantity of Crude Oil 1 available for use in the experiments, it was necessary to use some samples of oil for more than one test. Given this situation, the data described immediately above was further analyzed to assess the viscosity effects due to performing multiple tests on one sample of the oil.

The initial viscosity measurements obtained at the beginning of each of the tests shown in Table 14 were arranged in chronological sequence and are presented in Table 16. The treatment conditions for each test are also tabulated. The first experiment in this table used the fresh/untreated sample of Crude Oil 1. The remaining nine experiments are listed in the order in which the tests were conducted using the same oil that was used in the previous experiment. As one would expect, there is an obvious trend of decreasing viscosity as the oil is exposed to more

sonication energy with increasing number of tests and greater exposure duration. Comparing the data in Table 14 and Table 16, one can observe that the viscosity tends to recover somewhat during the idle period of time between tests. That is, the final viscosity at 120 minutes for a given experiment is consistently less than the initial viscosity for the next experiment. This issue of viscosity recovery after sonication was a point of interest during the investigation, and a series of tests was devised to examine this phenomenon. These results are presented in a later section of this report.

**Table 16 Reduction in Viscosity Resulting from Repeated Testing of a Sample of Crude Oil 1**

| Experiment No. | Viscosity, (cP) | Treatment Conditions |
|---|---|---|
| 1 | 69,800 | 3 actuators @ 0.8 kHz |
| 2 | 64,070 | 2 actuators @ 0.8 kHz |
| 3 | 63,930 | 3 actuators @ 1.8 kHz |
| 4 | 49,270 | 3 actuators @ 1.4 kHz |
| 5 | 48,500 | 2 actuators parallel @ 0.8 and 1.6 kHz |
| 6 | 48,470 | 2 actuators parallel @ 1.2 kHz |
| 7 | 47,530 | 3 actuators @ 0.8, 1.2, and 1.6 kHz |
| 8 | 47,330 | 2 actuators parallel @ 1.6 kHz |
| 9 | 42,930 | 2 actuators parallel @ 1.2 and 1.6 kHz |
| 10 | 39,520 | 2 actuators parallel @ 0.8 and 1.2 kHz |

The data from Table 16 are plotted in Figure 56 below. Two regression analyses were performed on the data and the results are shown on the graph. Both the exponential (solid line and upper equation) and the power function (dashed line and lower equation) provide a good fit to the observed data. The coefficients of determination for both regression equations are close to 90%. These results clearly demonstrate the effect of repeated and extended treatment of Crude Oil 1 with acoustic sonication. The viscosity at the beginning of the tenth experiment (Table 16, No. 10) was reduced to almost one-half of the viscosity measured at the beginning of the series of tests, even after a minimum of twelve hours transpired between the ten individual experiments when the viscosity could recover. If one were to look at the final viscosity value for experiment 10 (22,520 cP, Table 14), this represents a reduction of 67.7% in the initial viscosity. In these tests, experiment number and time are interchangeable because each experiment lasted for a total of 120 minutes (two hours). Consequently, one could insert time for experiment number in this data set and within Figure 56 and similar results would be obtained.

### 3.7.3 Effects of Horn Design on Viscosity

Experiments were conducted to evaluate various combinations of acoustic frequency, number of actuators, horn design, and power level in terms of the ability to reduce the viscosity Crude Oil 1. In addition to actuator arrangements and frequencies used in the previous set of

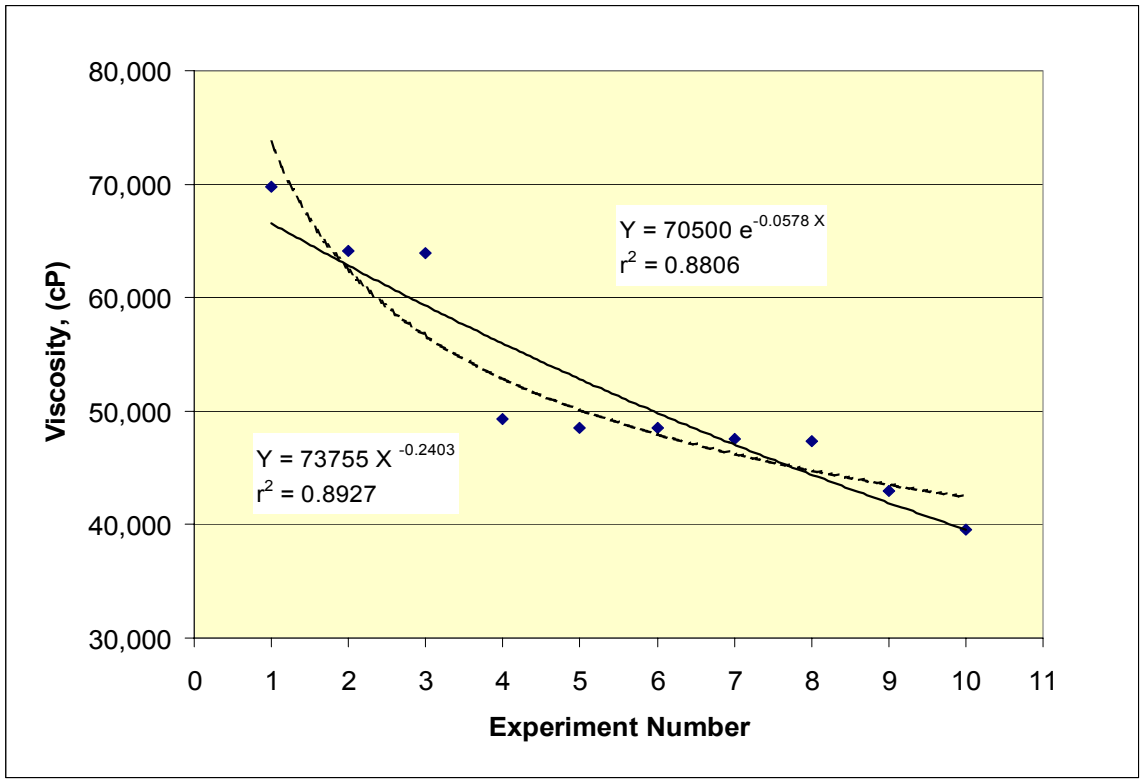


**Figure 56 Viscosity Reduction as a Result of Repeated Sample Use During Testing**

tests described above, two different horn designs were evaluated: one with two disks/fins spaced approximately one inch (2.54 cm) apart, and a second design with two disks/fins spaced approximately two inches (5.0 cm) apart. The previous results presented in Section 3.7.2 were all obtained with a one-inch (2.54-cm) spacing. In addition, tests were conducted to ascertain if reducing the normal operating power levels by 25% would have an effect on the observed viscosity changes. These results are presented in the following report section. As in previous experiments, all individual tests were performed for a total of 120 minutes.

Figure 57 illustrates the effects of increasing the fin spacing on the sonication horns from one inch (2.54 cm) to two inches (5.0 cm) on the viscosity measured at the end of each test. This plot indicates that the horn spacing has a small, but observable effect on viscosity changes during sonication. However the results are mixed. The data show that in three of the ten tests, the greatest reductions in viscosity after 120 minutes of sonication were obtained with the smaller fin spacing on the horns. In the remaining seven tests, the differences in the results obtained with the two horn designs are so small that they could conceivably be attributed to measurement error. Interestingly, the tests producing the largest differences between the two horn designs tend to be the same test conditions that produced the best overall reductions in viscosity by sonication observed in the first series of tests on Oil 1. The maximum difference in viscosity obtained by using a different horn spacing in these tests was 19.5%, but the average difference for all ten

tests was only 6.5%. Based on these results, one must conclude that the variation in horn disk/fin spacing between one and two inches (2.5 and 5.0 centimeters) has a minimal effect on the observed viscosity of Crude Oil 1 after two hours of sonication, with the smaller spacing appearing to be somewhat more effective than the larger spacing.

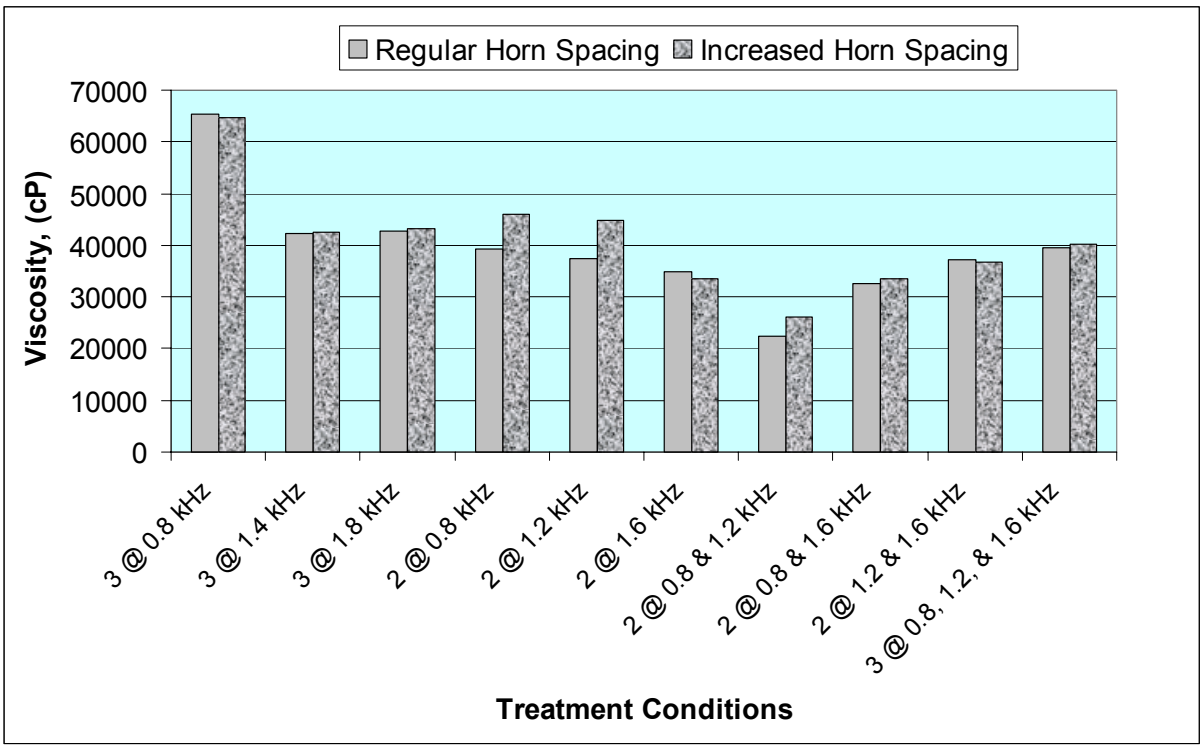


**Figure 57 Viscosity Variation of Crude Oil 1 due to Increased Horn Fin Spacing**

### 3.7.4 Effects of Power Level on Viscosity

Data obtained during sonication tests using the same actuator-frequency conditions as reported above but with reduced electrical power input are given in Table 17. In Figure 58, these data obtained with power reduced by 25% are compared to the viscosities obtained using 100% of the normal power levels (Table 14). The reduced-power tests were also performed for a total of 120 minutes, and the smaller horn spacing of one inch (2.54 cm) was used in each test. As one can see in Figure 58, the results are inconsistent. Under some frequency-actuator conditions, the data showed an improved reduction in viscosity with reduced power whereas other conditions resulted in a lesser reduction with reduced power or a final viscosity that was greater than that obtained using normal power input. In seven of the ten tests, reduced power input was associated with a larger viscosity (smaller reduction) after 120 minutes of sonication compared to the full power results; in the remaining three tests, reduced power was associated with a greater reduction in viscosity. However, in two of these three tests, the differences are extremely small.

**Table 17 Oil 1 Viscosity Data for Various Treatment Times and Treatment Conditions with Power Decreased by 25%**

| Treatment Conditions | Viscosity, (cP) | | | | |
|---|---|---|---|---|---|
| | Time, (min) | | | | |
| | 0 | 30 | 60 | 90 | 120 |
| 3 actuators @ 0.8 kHz | 65,200 | 49,270 | 39,640 | 39,800 | 39,880 |
| 3 actuators @ 1.4 kHz | 64,500 | 41,870 | 41,670 | 41,730 | 41,470 |
| 3 actuators @ 1.8 kHz | 64,700 | 42,200 | 40,670 | 40,270 | 40,000 |
| 2 actuators parallel @ 0.8 kHz | 65,200 | 47,870 | 46,800 | 46,000 | 45,870 |
| 2 actuators parallel @ 1.2 kHz | 65,330 | 53,130 | 50,470 | 49,070 | 47,400 |
| 2 actuators parallel @ 1.6 kHz | 65,400 | 51,130 | 46,070 | 45,200 | 44,730 |
| 2 actuators parallel @ 0.8 kHz and 1.2 kHz | 53,100 | 45,600 | 45,400 | 45,000 | 44,270 |
| 2 actuators parallel @ 0.8 kHz and 1.6 kHz | 53,070 | 40,870 | 40,930 | 40,800 | 40,470 |
| 2 actuators parallel @ 1.2 kHz and 1.6 kHz | 65,600 | 48,930 | 43,870 | 41,730 | 41,800 |
| 3 actuators @ 0.8 kHz, 1.2 kHz, and 1.6 kHz | 65,270 | 53,270 | 50,500 | 49,400 | 49,800 |

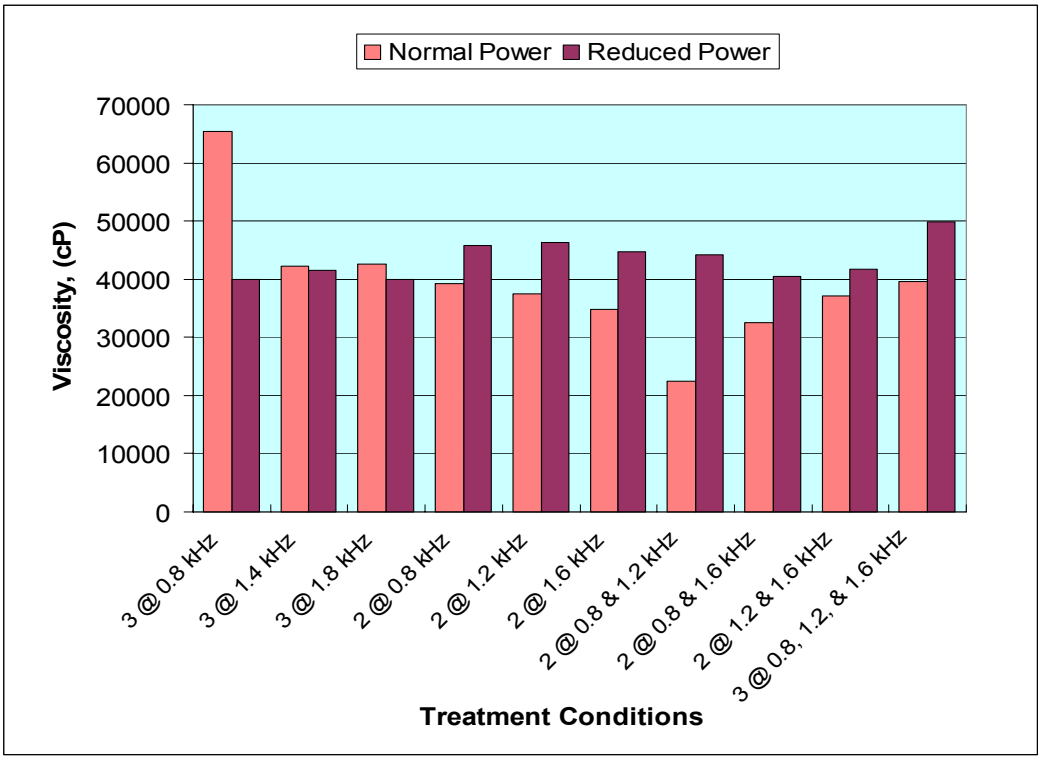


**Figure 58 Viscosity Variation of Crude Oil 1 due to Reduced Power Input**

It will be recalled that the initial sonication tests using Crude Oil 1 were performed by reusing an initial sample of oil. The tests involving reduced power used fresh oil for each test; therefore, the viscosity values obtained from the two series of tests are not directly comparable because of the discussed effects of oil reuse on viscosity. Consequently the data relationships depicted in Figure 58 should be viewed with this limitation in mind. One approach to deal with the effects of oil reuse and to make the results from the two series of tests somewhat more comparable is to examine the fractional viscosities in each case. By employing these data, the viscosity values are "normalized" by expressing the final viscosity as a percentage of the initial viscosity for each test. This allows one to view the relative effects of each test on the viscosity, instead of using the absolute viscosity values; an imperfect but better alternative. The fractional residual viscosity values for the reduced power tests are given in Table 18. These data indicate that all treatment conditions resulted in viscosity reductions with the greatest reduction being 38.8% and the least being 16.6%.

**Table 18 Oil 1 Fractional Residual Viscosity Data for Various Treatment Times and Treatment Conditions with Input Power Reduced by 25%**

| Treatment Conditions | Treatment Time, (min) | | | | |
|---|---|---|---|---|---|
| | 0 | 30 | 60 | 90 | 120 |
| 3 actuators @ 0.8 kHz | 1 | 0.7557 | 0.6080 | 0.6104 | 0.6117 |
| 3 actuators @ 1.4 kHz | 1 | 0.6492 | 0.6461 | 0.6470 | 0.6430 |
| 3 actuators @ 1.8 kHz | 1 | 0.6522 | 0.6286 | 0.6224 | 0.6182 |
| 2 actuators parallel @ 0.8 kHz | 1 | 0.7342 | 0.7178 | 0.7055 | 0.7035 |
| 2 actuators parallel @ 1.2 kHz | 1 | 0.8133 | 0.7725 | 0.7511 | 0.7256 |
| 2 actuators parallel @ 1.6 kHz | 1 | 0.7818 | 0.7044 | 0.6911 | 0.6839 |
| 2 actuators parallel @ 0.8 and 1.2 kHz | 1 | 0.8588 | 0.8550 | 0.8475 | 0.8337 |
| 2 actuators parallel @ 0.8 and 1.6 kHz | 1 | 0.7701 | 0.7713 | 0.7688 | 0.7626 |
| 2 actuators parallel @ 1.2 and 1.6 kHz | 1 | 0.7459 | 0.6688 | 0.6361 | 0.6372 |
| 3 actuators @ 0.8, 1.2, and 1.6 kHz | 1 | 0.8162 | 0.7752 | 0.7569 | 0.7630 |

Table 19 contains treatment conditions and the resulting fractional residual viscosity values in Crude Oil 1 obtained after 120 minutes of sonication with the normal or standard power input and the values obtained with a 25% reduction in power. Reduced power was less effective in that the residual viscosity values after 120 minutes were greater than those at standard power in three of the ten tests. Conversely, the reduced power tests resulted in a greater reduction in viscosity (smaller values of fractional residual viscosity) for seven of the ten tests. The maximum difference in the residual viscosity values for standard vs. reduced power levels is 32.5% while the average difference is 14.6%. This means that by reducing the power levels by 25%, the average difference between the final residual viscosity values averaged about 15%. The differences in six of the ten pairs of final residual viscosity values were less than 10%. These results shown graphically in Figure 59 suggest that, if necessary, input power could be

reduced up to 25% without significantly reducing the effectiveness of the sonication system on viscosity reduction under the majority of the test conditions evaluated.

**Table 19 Comparison of Fractional Residual Viscosity of Oil 1 After 120 Minutes of Sonication with Standard and Reduced Power Input Conditions**

| Treatment Condition | Fractional Viscosity Remaining | |
|---|---|---|
| | Standard Power | 25% Power Reduction |
| 3 actuators @ 0.8 kHz | 0.9370 | 0.6117 |
| 3 actuators @ 1.4 kHz | 0.8579 | 0.6430 |
| 3 actuators @ 1.8 kHz | 0.6670 | 0.6182 |
| 2 actuators parallel @ 0.8 kHz | 0.6131 | 0.7035 |
| 2 actuators parallel @ 1.2 kHz | 0.7733 | 0.7256 |
| 2 actuators parallel @ 1.6 kHz | 0.7361 | 0.6839 |
| 2 actuators parallel @ 0.8 and 1.2 kHz | 0.5698 | 0.8337 |
| 2 actuators parallel @ 0.8 and 1.6 kHz | 0.6722 | 0.7626 |
| 2 actuators parallel @ 1.2 and 1.6 kHz | 0.8665 | 0.6372 |
| 3 actuators@ 0.8, 1.2, and 1.6 kHz | 0.8332 | 0.7630 |

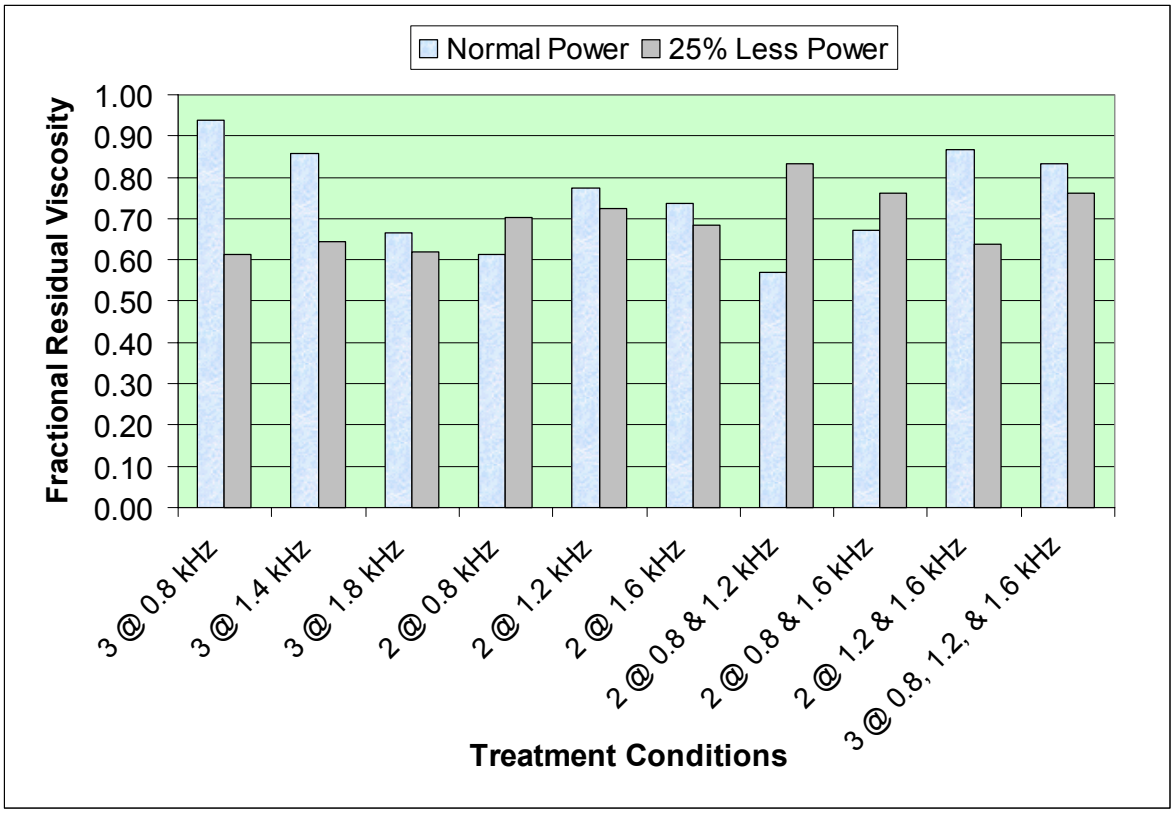


**Figure 59 Fractional Residual Viscosity Data for the Treatment Conditions Using Standard and Reduced Power Input**

### 3.7.5 Combined Effects of Power Level and Horn Design on Viscosity

Figure 60 contains a plot of viscosity data collected after 120 minutes of testing under conditions of reduced power input and changing horn fin spacing for each of the frequency-actuator conditions. Power was reduced by 25% for both sets of tests. One set of tests used the one-inch (2.54-cm) horn spacing, while the second series of tests, noted as Large Horn Spacing, utilized the 2-inch (5.0-cm) fin spacing on the horns. These results indicate that there is very little difference in the final viscosity of samples treated under reduced power input conditions and variable horn fin spacing. The average difference between the two viscosity values for the ten test conditions is only about 2.5%. In other words, changing the horn design/fin spacing from one inch (2.54 cm) to two inches (5.0 cm) had little effect on viscosity reduction when input power is reduced by 25%.

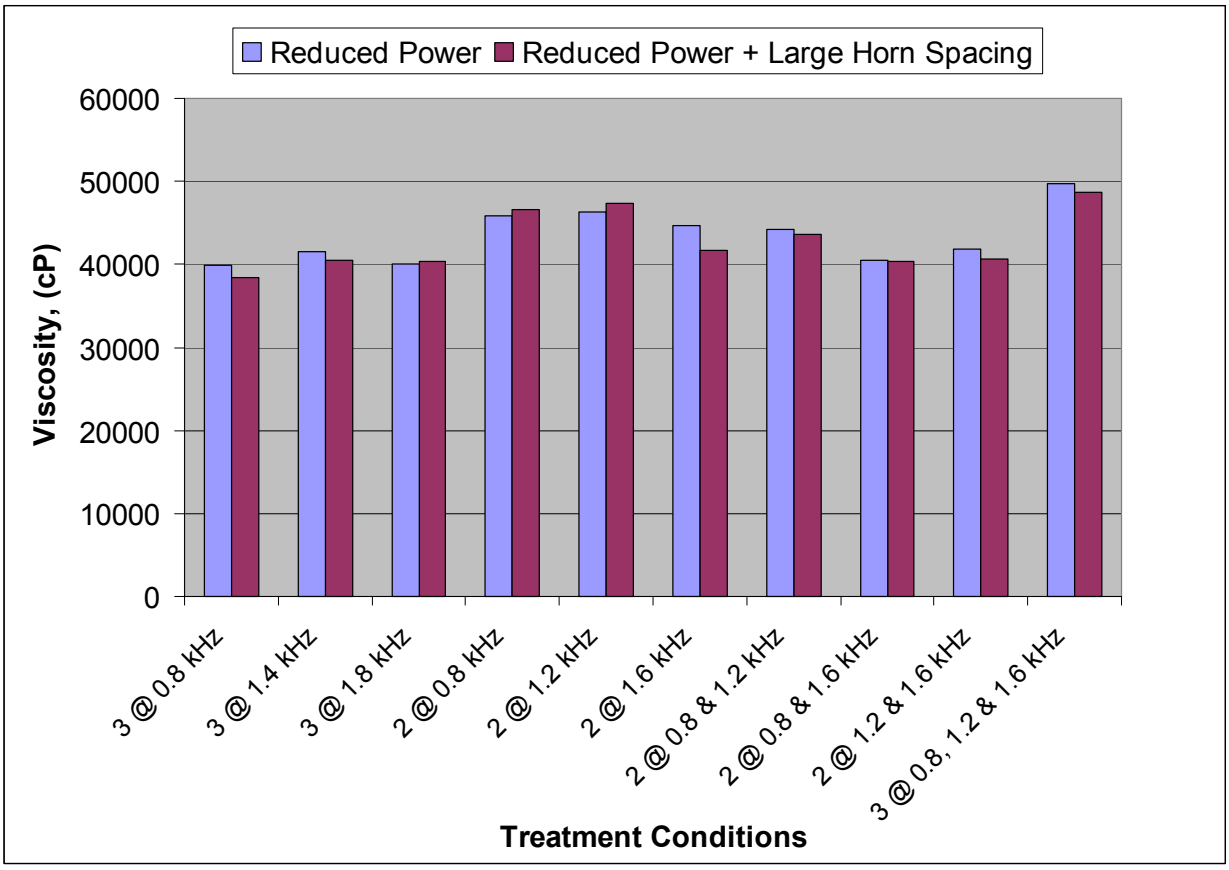


**Figure 60 Variation in Viscosity of Crude Oil 1 due to Horn Spacing at Reduced Power**

Figure 61 shows the variation in viscosity values obtained after 120 minutes of testing Crude Oil 1 under conditions of large-spacing horn design and varying power input levels. In each test, the two-inch (5.0-cm) horn fin spacing was used. One series of tests was performed at 100% or normal power and the second series was conducted with power reduced by 25%. These data show somewhat mixed results in that there is no consistent trend throughout the range of test

conditions. These results are quite similar to those obtained by reducing the input power level while using the smaller horn spacing (Figure 58). The data illustrated in Figure 61 indicate that, while using the larger horn fin spacing, reduced power conditions resulted in a lower viscosity in only three of the ten experiments, whereas reduced power resulted in a larger viscosity (less viscosity reduction) in seven out of ten test conditions. The average difference between viscosity values for the normal vs. reduced power using the larger horn spacing is approximately 20.3%. The average difference in the viscosity values using the small horn spacing was 16.6% for the same test conditions. One could conclude that the effects of reducing power by 25% are somewhat greater while using the larger horn spacing than when using the smaller spacing.

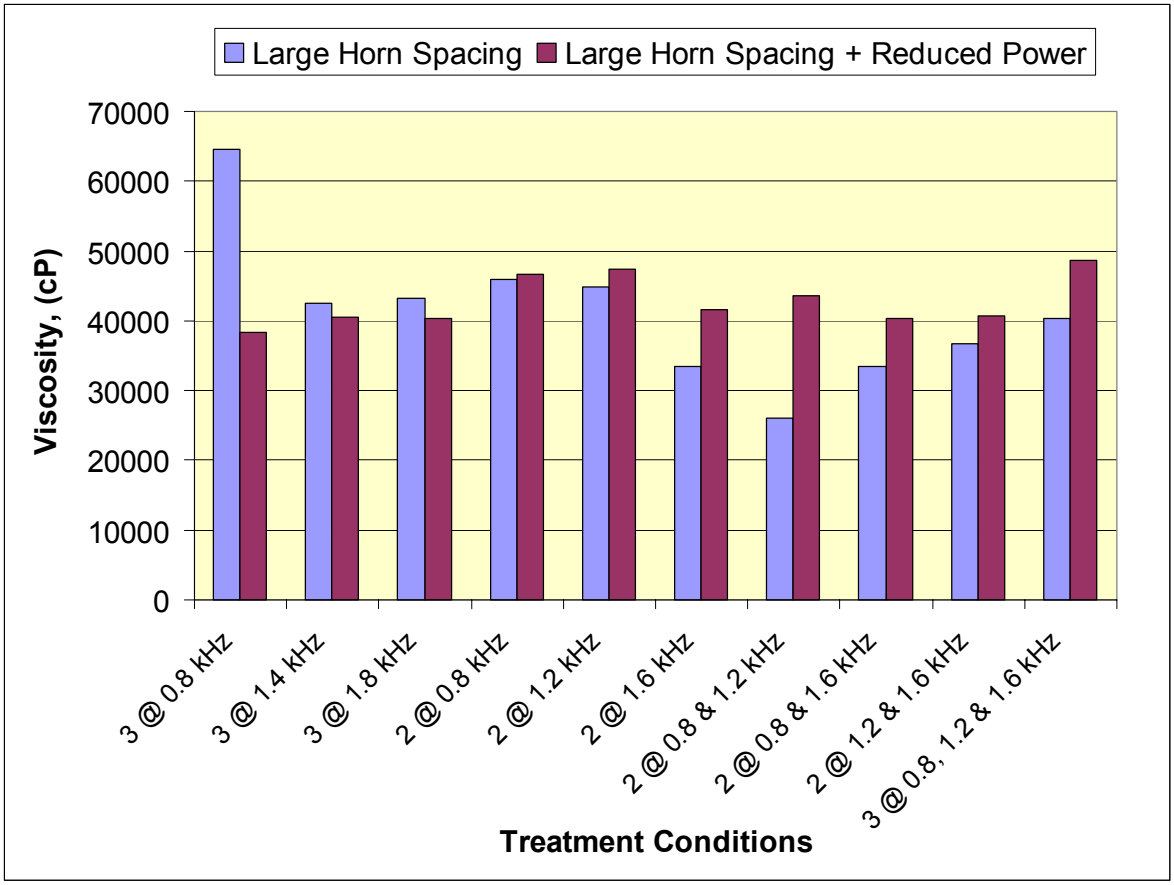


**Figure 61 Variation in Viscosity of Crude Oil 1 due to Reduced Power at Increased Horn Spacing**

Based on the results obtained in these series of tests examining the effects of horn design/fin spacing and input power levels, it is possible to make some general observations. Although the results are certainly not conclusive, it appears that the smaller horn spacing is somewhat more effective at reducing oil viscosity than is the larger spacing with standard power input. The results from testing the effects of reduced power input are likewise poorly defined throughout the range of test conditions with only two of the ten test conditions showing a large

difference between the full- and reduced-power results. Interestingly, these two results indicate opposite responses. While there is evidence to suggest that under some of the test conditions, reduced power may be slightly more effective in reducing viscosity than is the higher power input, the physical rationale for this observation is unclear. When comparing measured viscosity values, reduced power correlates with a diminished capacity to reduce oil viscosity in the majority of the tests. However, when the fractional residual viscosity data for individual treatment conditions are compared for full and reduced power, reduced power is more effective in altering viscosity in seven of the ten experiments. Consequently, if there is a desire to reduce the power input for future applications of this technology for cost savings or other reasons, these data suggest that this option should be explored as a reasonable design and optimization parameter. The differences between fractional viscosity values using standard power versus reduced power range from a maximum of 32.5% to a low of 4.8%. Results from the two series of tests looking at combined effects of input power levels and horn design suggest that the effects of input power level on viscosity reduction are greater than those due to altering the horn design/fin spacing.

### 3.7.6 Effects of Chemical Additives on Viscosity

The project team selected two proprietary chemical additives, termed A and B, for inclusion in the suite of tests to evaluate their effects on viscosity both with and without sonication. These substances were selected because they have been demonstrated to effectively reduce the viscosity of petroleum materials under a variety of circumstances, and because they can be formulated from readily available materials that are relatively inexpensive compared to other materials developed for the same purposes and applications.

The same procedures, with only minor modifications, were used in testing the viscosity effects of additives on all three crude oils. Initially the necessary volume of crude oil was added to the supply tank (see Figure 48). Immediately following, the calculated volume of chemical additives to attain the appropriate concentration, measured as a volume percent, was added and manually mixed in the supply tank. Because the viscosity of Crude Oil 1 was so great, the additive-oil mixture was allowed to sit for about two hours before each test to facilitate interaction. After this reaction time was complete, the pump was operated for an additional two hours and the oil was recycled through the system without turning on the sonication equipment. This was done to further enhance the mixing of the additives and oil. After this mixing period, the actuators were turned on and the tests commenced. Because Crude Oils 2 and 3 were much less viscous, mixing the additives with the oils was much easier. In these instances, it was not necessary to allow the mixture to stand for two hours, and the mixture of oil and additives was circulated through the equipment system for approximately 90 minutes before individual experiments were started. Samples of each oil-additives mixture were collected after mixing, and their viscosities were measured before being exposed to sonication. The viscosity values for Oil 1 illustrating the effects of the chemical additives without sonication are contained in Table

20. The experimental results utilizing both chemical additives and sonication are presented in Table 21.

**Table 20 Effects of Chemical Additives on Viscosity of Crude Oil 1 Without Sonication**

| Additives, A + B, (%) | Treatment Description | Lowest Residual Viscosity Achieved, (cP) | Maximum Reduction in Viscosity, (%) |
|---|---|---|---|
| 3.0 + 10.0 | Additive blend added to attain 3% concentration and mixed | 43,730 | 37.35 |
| 5.0 + 10.0 | Additive blend added to raise the concentration to 5.0 % and mixed | 19,940 | 71.43 |
| 7.0 + 10.0 | Additive blend added increasing the concentration to 7% and mixed | 38,560 | 44.76 |

Experiments to determine the combined effects of chemical additives and sonication were performed in which an additive blend of 3%A and 10%B (Mixture 1) was added to Crude Oil 1. The initial viscosity of the crude oil prior to addition of the additive mixture was ~69,800 cP. As shown in Table 20, the viscosity was reduced to 43,730 cP prior to sonication. The oil was then subjected to low frequency treatment (800 Hz) in four separate experiments (Table 21). After treatment during one experiment, the oil was allowed to set for a period of time; this oil was then used as the initial feedstock in the next experiment. No additional additives were added during this series of four experiments after the initial amount for the first test. In all tests involving Crude Oil 1, the viscosity recovered/increased fairly rapidly during the first three days following one of these tests. During the subsequent three or four days, the viscosity changed very slowly and remained fairly stable after about seven days. Consequently, in all of the tests shown in Table 21, the oil samples were set aside for approximately seven days before the next test on that sample was conducted. The approach utilized in the first series of four tests illustrated the effects from a continued recycle and reuse of the crude oil containing the 3% additive blend (3%A and 10%B). Each experiment lasted 120 minutes, so that the crude oil was treated for a total time of 480 minutes (8.0 hours) at the conclusion of this first set of four tests. Samples were collected every 30 minutes during each experiment for viscosity measurement.

In the second set of experiments (5, 6, and 7 in Table 21), the oil from the first set of experiments was used again after several days time following the conclusion of the first set of tests. During this time, viscosity was observed to increase to a stable value. Sufficient quantities of the additives were added to the crude oil to bring the concentration of additives in the crude oil to 5.0%A and 10%B (5% Mixture). In this second set of experiments, the effect of low acoustic frequency combined with the additive mixture was studied. Three separate experiments were performed using different acoustic frequencies: 800 Hz, 1.2 kHz, and 1.6 kHz (Table 21). After the additional increment of additives was mixed with the crude oil, the viscosity of the

resultant mixture was 19,940 cP before the sonication began. Again, viscosity measurements were performed on samples collected every 30 minutes, and each test lasted for 120 minutes.

**Table 21 Viscosity Values for Crude Oil 1 Treatments Involving Both Sonication and Chemical Additives**

| Additives A+B, (%) | Treatment Description | Lowest Residual Viscosity Achieved, (cP) | Maximum Reduction in Viscosity, (%) |
|---|---|---|---|
| 3.0 + 10.0 | Crude oil subjected to 800 Hz treatment (Experiment 1) | 11,070 | 84.14 |
| | Crude oil subjected to 800 Hz treatment with no further addition of additives (Experiment 2) | 9,530 | 86.35 |
| | Crude oil subjected to 800 Hz treatment with no further addition of additives (Experiment 3) | 19,570 | 71.96 |
| | Crude oil subjected to 800 Hz treatment with no further addition of additives (Experiment 4) | 7,928 | 88.64 |
| 5.0 + 10.0 | Additive added to oil used in Experiment 4; crude oil subjected to treatment at 800 Hz (Experiment 5) | 11,450 | 83.60 |
| | Crude oil from above experiment subjected to treatment at 1.2 kHz (Experiment 6) | 8,890 | 87.26 |
| | Crude oil from above experiment subjected to treatment at 1.6 kHz (Experiment 7) | 9,150 | 86.89 |
| 7.0 + 10.0 | Additives added to fresh Crude Oil 1; oil subjected to treatment at 800 Hz (Experiment 8) | 30,300 | 56.59 |
| | Crude oil from above experiment subjected to treatment at 1.2 kHz (Experiment 9) | 35,400 | 49.28 |
| | Crude oil from above experiment subjected to treatment at 1.6 kHz (Experiment 10) | 38,680 | 44.58 |

In the third set of experiments (8, 9, and 10, Table 21), fresh Crude Oil 1 was used, to which a third chemical blend (7%A + 10%B) was added. Experiments were again performed using the three frequencies of 800 Hz, 1.2 kHz, and 1.6 kHz. After the experiment was completed for the 800 Hz frequency, the crude oil was allowed to set for several days until the viscosity stabilized, and then that same oil was treated at the next higher frequency (1.2 kHz). This approach was then repeated for the 1.6 kHz frequency. The results from all ten experiments involving sonication as well as the results obtained with the chemical additives alone prior to

sonication are presented graphically in Figure 62. As noted previously, the initial viscosity of the oil before any chemicals were added was approximately 69,800 cP.

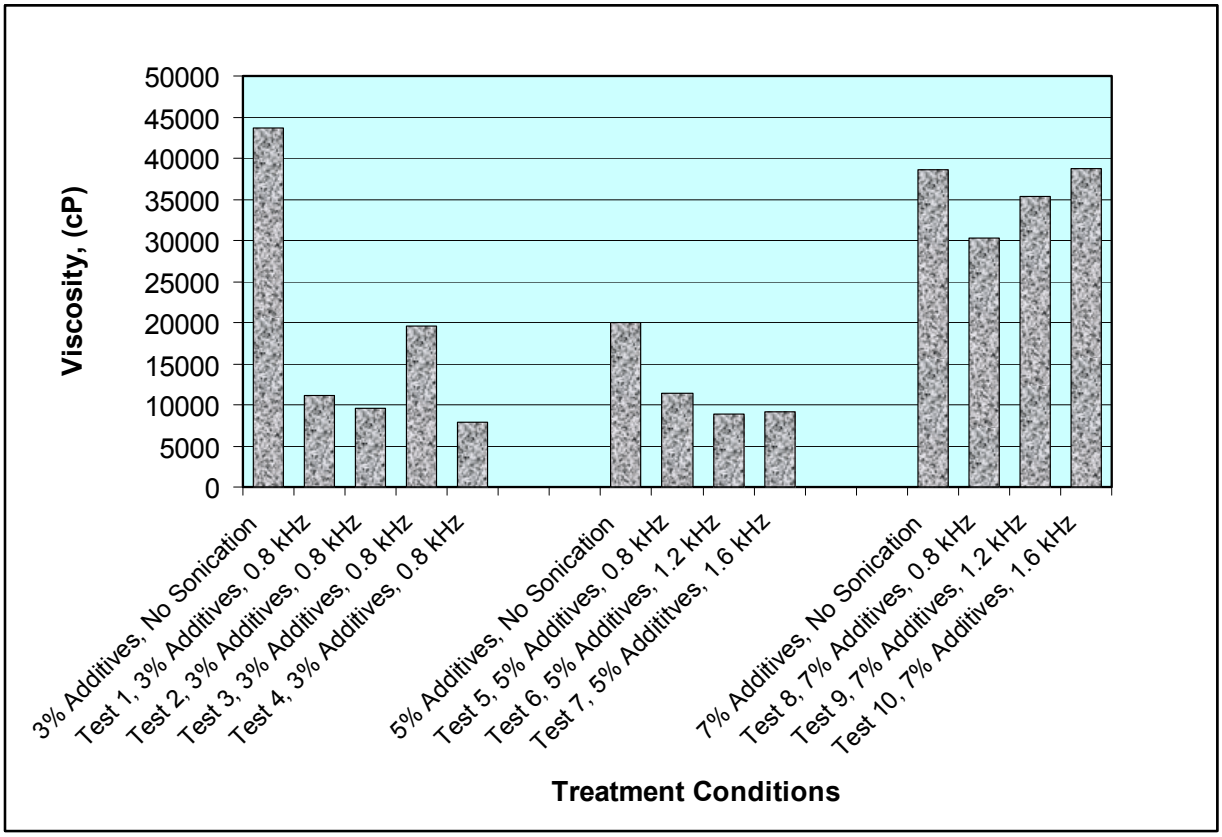


**Figure 62 Maximum Viscosity Reduction in Crude Oil 1 Resulting from Chemical Additives and Sonication**

Several observations can be made about the effects of the chemical additives on the viscosity of Crude Oil 1 under the treatment scenarios employed. The addition of the 3% mixture of chemical additives reduced the viscosity by 37.4%, but that value was further reduced to a more than 84% reduction after exposing the oil mixture to sonication at 800 Hz for 120 minutes. Similarly, the 5% mixture showed a reduction of 71.4% by itself, but this reduction was increased to 83.6% after sonication for 120 minutes. Given the fact that this second series of tests (5%) was conducted on the same oil sample that was used in the first (3%) series to tests, it is likely that some of the initial viscosity reduction with the 5% mixture was due to the previous exposure to 480 minutes of sonication. Additives of the 7% mixture reduced viscosity by 44.8%, which was increased to 56.6% after the first 120 minutes of sonication.

Further examination of Table 21 and Figure 62 indicates that all tests using the 3% mixture resulted in viscosities below 20,000 cP, which is less than 30% of the initial viscosity value. At the conclusion of the four tests using the 3% mixture and 800 Hz, the viscosity was reduced by more than 88%. A similar result is indicated for the tests involving the 5% mixture, but the

viscosity reduction was less (from about 44% to 57%) when the 7% mixture of additives was used. The 5% mixture had an apparently greater effect on viscosity than either the 3% or 7% mixture before exposure to sonication (Table 20), but this may be attributed to the fact that that the oil used in this test was also used in the previous four tests. After sonication in the presence of the additives, the end results are basically the same with both the 3% and 5% mixtures. Increasing the concentration of additives to produce the 7% mixture resulted in a somewhat improved viscosity reduction prior to sonication compared to the 3% mixture (Table 20), but this mixture provided the poorest results when combined with sonication. These data suggest that the most effective conditions for reducing the viscosity this oil would most likely be the addition of a 3% mixture of chemicals and sonication at 1.2 kHz, although no test was performed with this specific combination (3% additives mixture and 1.2 kHz) of treatment conditions.

### 3.7.7 Summary of Crude Oil 1 Results

Tests involving Crude Oil 1 were performed to evaluate the effects of heating, acoustic frequency and actuator arrangement, horn design, input power level, and the addition of chemicals on the oil's viscosity. In addition, the effect of reusing a sample of oil for several tests (longer-term exposure to sonication) was also examined because there was a limited quantity of this crude oil available for the planned experiments.

Tests were performed to quantify the reduction in viscosity with the addition of heat. The viscosity was observed to decrease from an initial value of approximately 68,900 cP to only 144 cP after heating to a maximum of 100°C, a reduction of 99.8%. The rate of decrease was not uniform in that the most significant decrease was in the range of 20°C to 40°C. A hysteresis effect between heating and cooling cycles was observed, and it was also observed that the final viscosity after the sample returned to room temperature was almost 14% less than the initial viscosity at the same temperature before heating.

It was observed that sonication was also an effective means of reducing the viscosity of this crude oil. The viscosity reductions after 120 minutes of testing ranged from a minimum of 6.3% (93.7% remaining viscosity) to a maximum reduction of 43.0% (57% residual viscosity), depending upon the arrangement of actuators in the T-shaped test apparatus and the operating frequencies. Treatment with two parallel actuators facing each other within the reaction chamber was generally more effective than using three actuators in a T-shaped arrangement, regardless of acoustic frequency employed. Operation of the two actuators at lower frequencies (0.8 kHz) or one at 0.8 kHz and the other at 1.2 kHz produced the best reductions in viscosity.

Changes in acoustic horn design, as determined by the spacing between fins, had only minimal effect on the observed viscosity results. In three of the ten tests, the greatest reductions in viscosity were clearly associated with the smaller fin spacing. However, in the remaining seven tests, the differences in results obtained with the two horn designs were so small that the

results must be considered inconclusive. The differences between the viscosities obtained with the two horn designs averaged only 6.5% for all ten tests performed, with the maximum difference being about 20%. Similarly, the effects of reducing electrical input power by 25% from the normal/maximum level resulted in somewhat inconsistent results. Interestingly, reduced power input was associated with a greater reduction in fractional residual viscosity in seven of the ten tests conducted. The greatest difference in viscosity values obtained with the two different input power levels was 32.5%, the minimum difference was 4.9%, and the average difference for the ten test conditions was 14.3%. While these results are not well defined, they do suggest that, if necessary, input power probably could be reduced without significantly reducing the effectiveness of the sonication system on viscosity reduction. The results obtained from two series of tests looking at the combined effects of input power and horn design also show mixed results. In looking at the effects of different horn design/fin spacing under reduced power conditions, one observes that there are only minor differences in the results of the two data sets with the average difference in viscosity obtained under these circumstances being only 2.5%. The results of comparing the viscosity data obtained with the large horn spacing and normal vs. reduced power show results that are very similar to the data obtained with the small horn spacing and normal vs. reduced power. The average difference obtained with changing input power and large horn design was approximately 20%. These combined results suggest that the effects of input power level on viscosity reduction are somewhat greater than those due to changing the horn design/fin spacing.

Lastly, a suite of experiments was conducted to evaluate the effectiveness of adding mixtures of two proprietary chemicals in different concentrations on viscosity reduction both in the presence and absence of sonication. Three different mixes were evaluated: 3% A and 10% B, 5% A and 10% B, and 7% A and 10% B. The initial viscosity values were reduced by 37.4%, 71.4%, and 44.8%, respectively, by the addition of these mixtures prior to sonication. In all tests, the combined effects of the chemical additives and sonication were better than the effects resulting from the additives or sonication individually. When the crude oil with chemical additives was sonicated, the maximum reduction in viscosity ranged from 44.6% to 88.6%. The reduction in six of the ten tests was greater than 80%, and the average for the ten tests was 73.9%. The best results were obtained with the lesser concentrations of chemical additives and sonication frequencies in the range of 0.8 kHz to 1.2 kHz.

## 3.8 Experimental Results Using Crude Oil 2

Based on the results obtained during the experiments involving Crude Oil 1, some modifications were made to the experimental procedures used to test Crude Oil 2. First, the use of three transducers arranged in a T-shape was eliminated. All sonication testing of Oil 2 was performed with two transducers arranged in parallel (horns facing each other) within the two horizontal components of the reaction vessel (see Figure 47). Second, testing using 25% input power reduction was eliminated because in almost all cases, the change in viscosity reduction as

compared to using the normal power levels was relatively small. A third change involved only using the two best combinations of horn design and acoustic frequency in conjunction with testing the effects of chemical additives. This action was deemed necessary in order to reduce the amount of crude oil necessary to complete the entire suite of tests. A fourth change involved the addition of a series to tests to systematically examine viscosity recovery over a period of 30 days. Some observations of viscosity recovery were made during the tests with Crude Oil 1, but this variable was not part of the Oil 1 experimental plan. The project team decided that viscosity recovery should be evaluated more systematically using Oils 2 and 3.

### 3.8.1 Temperature Effects on Viscosity

As was the case with Oil 1, the first tests performed on Oil 2 were heating experiments to determine the relationship between heat/temperature and measured viscosity. The results of these tests are presented in Figure 63. These results are similar to those obtained with Oil 1, except that the hysteresis effect noted in the plot for Oil 1 is not present in the Oil 2 results. In both series of tests, the final viscosity of the cooling cycle is less than the initial viscosity of the oil measured prior to heating.

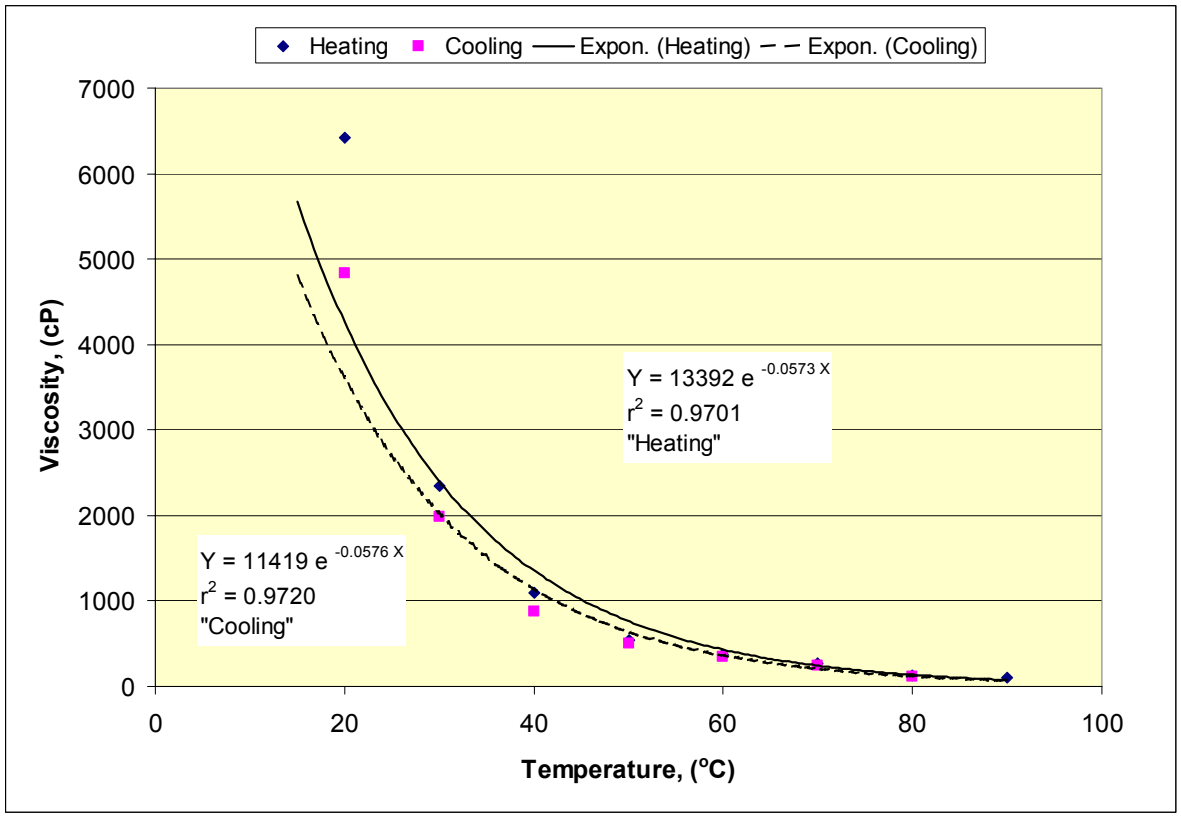


**Figure 63 Viscosity of Crude Oil 2 as a Function of Temperature**

### 3.8.2 Sonication Effects on Viscosity

The test matrix designed to evaluate the effects of sonication on the viscosity of Crude Oil 2 was modified based on the results from testing Crude Oil 1. As noted above, the number of test conditions was reduced by eliminating the tests using three actuators; consequently, the total number of actuator/frequency test conditions was reduced from ten to six. As in the previous experiments with Oil 1, individual tests were conducted for a total of 120 minutes with individual samples collected for viscosity determination after 0 (pre-treatment), 30, 60, 90, and 120 minutes.

The viscosity data obtained by treating Oil 2 with the selected acoustic frequencies and two parallel actuators fitted with horns having a two-inch (5-cm) fin spacing are presented in Table 22. The corresponding "normalized" viscosity values, or fractional residual viscosity, are given in Table 23, and a plot of these data is presented in Figure 64.

**Table 22 Summary of Viscosity Results for Acoustic Treatment of Crude Oil 2**

| Treatment Conditions | Viscosity, (cP) | | | | |
|---|---|---|---|---|---|
| | Time, (min) | | | | |
| | 0 | 30 | 60 | 90 | 120 |
| 2 actuators @ 0.8 kHz each | 4553 | 6784 | 6167 | 6067 | 5093 |
| 2 actuators @ 1.2 kHz each | 4827 | 4887 | 4547 | 4493 | 3957 |
| 2 actuators @ 1.6 kHz each | 4943 | 4373 | 3988 | 4107 | 5667 |
| 2 actuators @ 0.8 and 1.2 kHz | 4983 | 4120 | 3768 | 3888 | 3828 |
| 2 actuators @ 0.8 and 1.6 kHz | 4577 | 4733 | 4333 | 4213 | 3552 |
| 2 actuators @ 1.2 and 1.6 kHz | 4633 | 3920 | 3808 | 5187 | 3768 |

**Table 23 Summary of Fractional Residual Viscosity Results for Acoustic Treatment of Crude Oil 2**

| Treatment Conditions | Fractional Residual Viscosity | | | | |
|---|---|---|---|---|---|
| | Time, (min) | | | | |
| | 0 | 30 | 60 | 90 | 120 |
| 2 actuators @ 0.8 kHz each | 1 | 1.4900 | 1.3545 | 1.3325 | 1.1186 |
| 2 actuators @ 1.2 kHz each | 1 | 1.0124 | 0.9420 | 0.9308 | 0.8198 |
| 2 actuators @ 1.6 kHz each | 1 | 0.8847 | 0.8068 | 0.8309 | 1.1465 |
| 2 actuators @ 0.8 and 1.2 kHz | 1 | 0.8268 | 0.7562 | 0.7803 | 0.7682 |
| 2 actuators @ 0.8 and 1.6 kHz | 1 | 1.0341 | 0.9467 | 0.9205 | 0.7761 |
| 2 actuators @ 1.2 and 1.6 kHz | 1 | 0.8461 | 0.8219 | 1.1196 | 0.8133 |

These data exhibit a pattern of variation similar to that shown in the tests with Oil 1. One can see that there is a general trend for viscosity to decrease with time. That is, in most cases, the viscosities observed after 120 minutes are less than the initial viscosity values at the start of the tests. However the data in Tables 22 and 23 and Figure 64 indicate that in some cases, the viscosity actually increases after exposure to sonication (values of fractional residual viscosity greater than 1.0), a condition not observed in the test results using Oil 1. The reason for this appears to be the fact that Oil 2 had appreciable water content whereas Oil 1 did not. Several samples collected during these sonication tests with Oil 2 showed an evident water layer that had separated from the oil as a result of the treatment. The presence of water also tends to reduce the viscosity of the oil (e.g. Rivas and others, 1985), which also explains the fact that the initial viscosities in Tables 22 and 23 are less than the average value of 6,000 cP that was reported in Table 10. Water content appeared to vary among the large storage containers of oil obtained for the study.

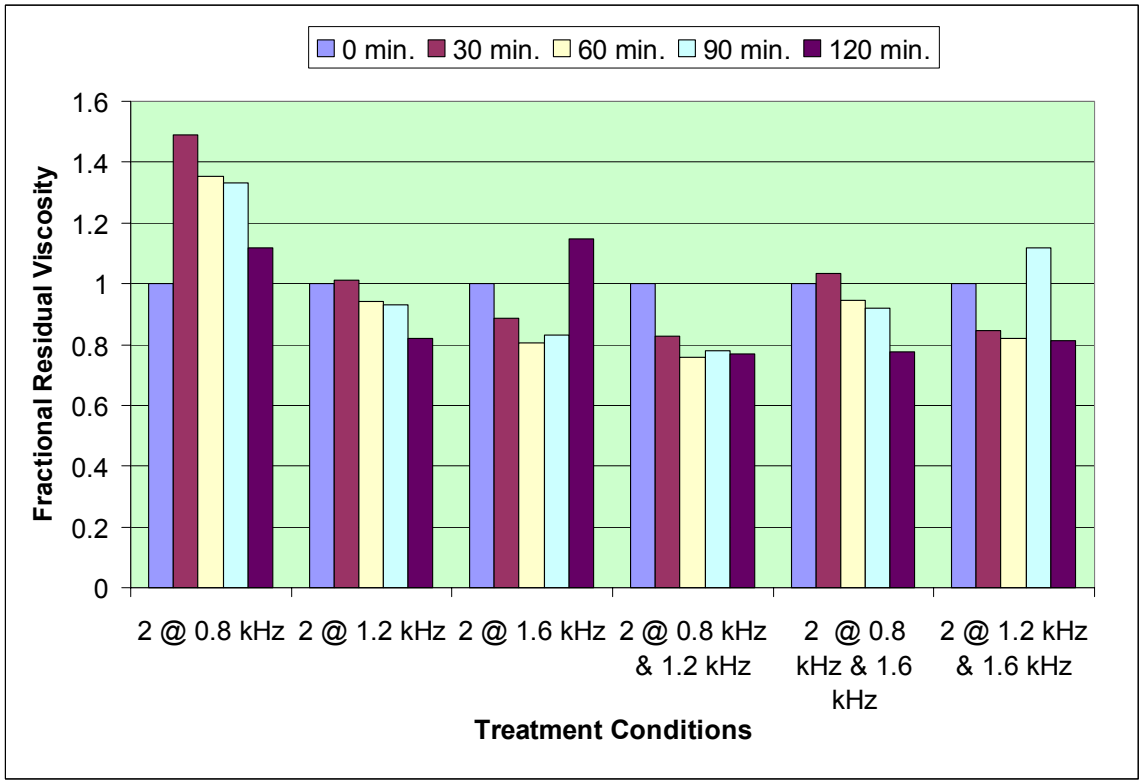


**Figure 64 Fractional Residual Viscosity Variation of Oil 2 with Changing Acoustic Treatment Conditions**

The maximum amount of viscosity reduction attained in Oil 2 after 120 minutes of testing with sonication was 23.2% (fractional residual viscosity = 0.7682). In addition, it appears that two transducers operating at 0.8 kHz and 1.2 kHz was the most effective in reducing viscosity, although there is not a great deal of difference in the results from four of the six treatment

conditions shown in Figure 64. This set of frequencies also produced the best viscosity reduction in Oil 1.

### 3.8.3 Effects of Horn Design on Viscosity

All of the experiments described in the preceding report section were performed with the wide, 2-inch (5-cm) horn fin spacing. The experiments were repeated using the reduced, 1-inch (2.5-cm) fin spacing on the acoustic horn. The data obtained with the reduced horn spacing are presented in Table 24, Table 25, and Figure 65.

**Table 24 Summary of Viscosity Results for Acoustic Treatment of Crude Oil 2 using Reduced Horn Spacing**

| Treatment Conditions | Viscosity, (cP) | | | | |
|---|---|---|---|---|---|
| | Treatment Time, (min.) | | | | |
| | 0 | 30 | 60 | 90 | 120 |
| 2 actuators @ 0.8 kHz each | 6440 | 3356 | 4347 | 3600 | 3080 |
| 2 actuators @ 1.2 kHz each | 6633 | 3060 | 3736 | 2233 | 2333 |
| 2 actuators @ 1.6 kHz each | 6220 | 2650 | 2490 | 2050 | 1982 |
| 2 actuators @ 0.8 and 1.2 kHz | 5927 | 1970 | 2560 | 1993 | 1758 |
| 2 actuators @ 0.8 and 1.6 kHz | 6864 | 1770 | 3068 | 1734 | 1660 |
| 2 actuators @ 1.2 and 1.6 kHz | 6333 | 1958 | 2070 | 1938 | 1882 |

**Table 25 Summary of Fractional Residual Viscosity Results for Acoustic Treatment of Crude Oil 2 with Reduced Horn Spacing**

| Treatment Conditions | Fractional Residual Viscosity | | | | |
|---|---|---|---|---|---|
| | Treatment Time, (min.) | | | | |
| | 0 | 30 | 60 | 90 | 120 |
| 2 actuators @ 0.8 kHz each | 1 | 0.5211 | 0.6750 | 0.5590 | 0.4783 |
| 2 actuators @ 1.2 kHz each | 1 | 0.4613 | 0.5632 | 0.3367 | 0.3517 |
| 2 actuators @ 1.6 kHz each | 1 | 0.4261 | 0.4003 | 0.3296 | 0.3187 |
| 2 actuators @ 0.8 and 1.2 kHz | 1 | 0.3324 | 0.4319 | 0.3363 | 0.2966 |
| 2 actuators @ 0.8 and 1.6 kHz | 1 | 0.2579 | 0.4470 | 0.2526 | 0.2418 |
| 2 actuators @ 1.2 and 1.6 kHz | 1 | 0.3092 | 0.3269 | 0.3060 | 0.2972 |

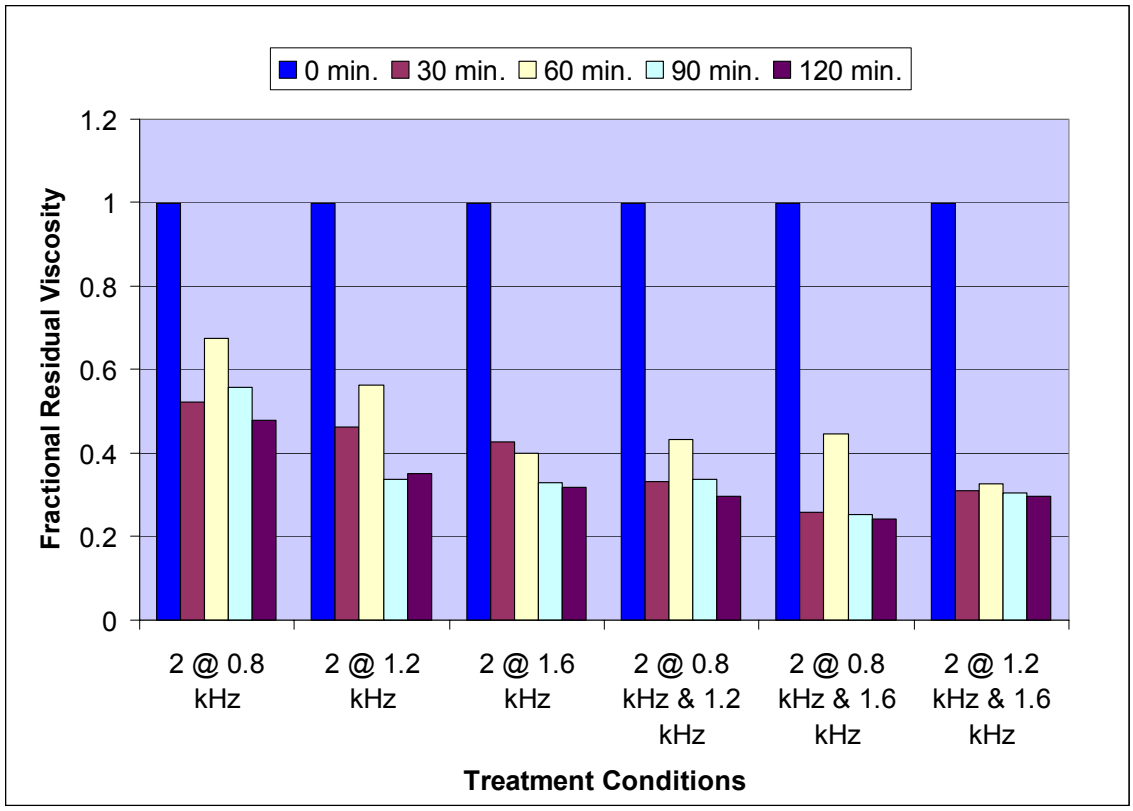


**Figure 65 Fractional Residual Viscosity Variation of Oil 2 with Changing Acoustic Treatment Conditions and Reduced Horn Spacing**

Once again, the data reflect a general trend of decreasing viscosity as the treatment time increases. In these tests involving a reduced horn fin spacing, using two actuators operating at frequencies of 0.8 + 1.2 kHz and 0.8 + 1.6 kHz provide the greatest reductions in viscosity. With the 1-in. (2.5-cm) horn spacing, the maximum reduction in viscosity was 75.8%, which results in a fractional residual viscosity of 24.2%. This result is a significant improvement over the maximum 24.4% reduction obtained using the wider, 2-in (5-cm) horn spacing. A comparison of Figures 64 and 65 indicates that in every instance, the viscosity reduction using the smaller horn spacing produced superior results compared to those produced by the larger spacing. It is also interesting to note that in no case did the viscosity increase to a value greater than the initial value when the smaller horn spacing was employed as was the case with the larger spacing. The reason for this difference is not clear from these experimental results. However, these observations raise interesting questions about the physical effects of sonication on crude oil that should be explored further to better define the full potential of this technology on petroleum production and processing.

To facilitate a comparison of the results using both smaller (1 in; 2.5 cm) and larger (2 in, 5 cm) horn spacing, both sets of fractional residual viscosities are combined and presented in Table 26. As noted above, the smaller horn design resulted in a much greater viscosity reduction

in every combination of acoustic frequencies employed in the experiments. The amount of enhanced reduction in the fractional residual viscosity obtained by utilizing the smaller horn spacing ranged from a maximum of 82.8% to a minimum of 46.8%. The average difference for all tests was 57.6%. Thus, the smaller acoustic horn fin spacing appears to be much more effective at reducing viscosity than does the larger spacing.

**Table 26 Fractional Residual Viscosity of Crude Oil 2 Resulting from Two Different Horn Fin Spacings**

| Treatment Conditions | Fractional Residual Viscosity | | | | |
|---|---|---|---|---|---|
| | Treatment Time, (min.) | | | | |
| | 0 | 30 | 60 | 90 | 120 |
| 2 actuators @ 0.8 kHz each | 1 | 1.4900 | 1.3545 | 1.3325 | 1.1186 |
| 2 actuators @ 0.8 kHz each – Smaller Fin Spacing | 1 | 0.5211 | 0.6750 | 0.5590 | 0.4783 |
| 2 actuators @ 1.2 kHz each | 1 | 1.0124 | 0.9420 | 0.9308 | 0.8198 |
| 2 actuators @ 1.2 kHz each – Smaller Fin Spacing | 1 | 0.4613 | 0.5632 | 0.3367 | 0.3517 |
| 2 actuators @ 1.6 kHz each | 1 | 0.8847 | 0.8068 | 0.8309 | 1.1465 |
| 2 actuators @ 1.6 kHz each – Smaller Fin Spacing | 1 | 0.4261 | 0.4003 | 0.3296 | 0.3187 |
| 2 actuators @ 0.8 and 1.2 kHz | 1 | 0.8268 | 0.7562 | 0.7803 | 0.7682 |
| 2 actuators @ 0.8 and 1.2 kHz – Smaller Fin Spacing | 1 | 0.3324 | 0.4319 | 0.3363 | 0.2966 |
| 2 actuators @ 0.8 and 1.6 kHz | 1 | 1.0341 | 0.9467 | 0.9205 | 0.7761 |
| 2 actuators @ 0.8 and 1.6 kHz – Smaller Fin Spacing | 1 | 0.2579 | 0.4470 | 0.2526 | 0.2418 |
| 2 actuators @ 1.2 and 1.6 kHz | 1 | 0.8461 | 0.8219 | 1.1196 | 0.8133 |
| 2 actuators @ 1.2 and 1.6 kHz – Smaller Fin Spacing | 1 | 0.3092 | 0.3269 | 0.3060 | 0.2972 |

### 3.8.4 Effects of Chemical Additives on Viscosity

A series of experiments examining the effects of chemical additives on the viscosity of Oil 2 was performed. As noted at the outset of this discussion of the experiments involving Oil 2, the experimental procedures were modified somewhat from those used with Oil 1; only the two best combinations of horn design and acoustic frequency were used to test the effects of chemical additives. This modification was deemed necessary to reduce the amount of oil required for the experiments because the amount available for use was limited. The same additives used with Oil 1 were also used with Oil 2 and similar methods of mixing the additives

with the oil, operating the test equipment, sample collection, and data collection were employed during this series of tests. As in the case of Oil 1, the viscosity of an initial sample of oil was measured. This was the assumed initial value for all tests as shown in Table 27 below. The oil and additives A and B at initial concentrations (3% and 10%) were mixed, the first test was performed, samples were collected at 0, 30, 60, 90, and 120 minutes, and the viscosity of each sample was measured. The test apparatus was drained and cleaned, and the oil collected from the first experiment was then reused. The concentrations of chemicals A and B were adjusted to 5% and 10% and the second test was performed at the same frequencies. This procedure was repeated for the remaining tests in the series. The test conditions employed and the corresponding viscosity results for Oil 2 are presented in Table 27. All tests were performed with the horn fins spaced at 1 in (2.5 cm).

**Table 27 Viscosity Values for Oil 2 Reflecting the Effects of Chemical Additives and Sonication**

| Treatment Conditions | Viscosity, (cP) | | | | | |
|---|---|---|---|---|---|---|
| | Time, (min) | | | | | |
| | Avg. Initial | 0 | 30 | 60 | 90 | 120 |
| 0.8 & 1.2 kHz, 3% A + 10% B | 6402 | 492 | 419 | 389 | 343 | 278 |
| 0.8 & 1.2 kHz, 5% A + 10% B | 6402 | 525 | 491 | 471 | 413 | 367 |
| 0.8 & 1.2 kHz, 7% A + 10% B | 6402 | 382 | 365 | 329 | 308 | 298 |
| 0.8 & 1.6 kHz, 3% A + 10% B | 6402 | 519 | 448 | 413 | 367 | 299 |
| 0.8 & 1.6 kHz, 5% A + 10% B | 6402 | 463 | 437 | 419 | 361 | 293 |
| 0.8 & 1.6 kHz, 7% A + 10% B | 6402 | 427 | 384 | 352 | 314 | 278 |

One apparent observation to be made from Table 27 is the fact that a very large reduction in viscosity is attained with the addition of the chemical mix alone without sonication. The minimum viscosity obtained with only the chemical additives is 382 cP which represents a 94% reduction in the initial viscosity. When sonication is added to the treatment conditions, viscosity is reduced even further. Longer treatment times resulted in lower viscosity values for all treatment conditions. The changes resulting from sonication after the chemical mix was added can best be seen when examining the fractional residual viscosities when the initial viscosity is taken to be that value after the chemicals have been added but before beginning sonication (time = 0 minutes in Table 27). The normalized viscosity values are given in Table 28 and are shown graphically in Figure 66. These data indicate that sonication of samples of Oil 2 in the presence of chemical additives A and B can further reduce the viscosity by as much as approximately 44%. When compared to the initial (before any chemical mix was added) the final viscosity values are only about 4%-5% of the pre-treatment value. This large reduction is, at least in part, due to the large quantity of water contained in Oil 2. In terms of chemical additives alone, the largest viscosity reduction was produced by the addition of a mixture of 7% A and 10% B,

although the difference in viscosity reduction among the various mixes of chemical additives is very small. The greatest final reduction in fractional residual viscosity resulted from chemical additives in 3% and 10% concentrations with both combinations of sonication frequencies (Table 28 and Figure 66). This illustration also clearly shows the pattern of reducing viscosity with increasing treatment times for all treatments.

**Table 28 Fractional Residual Viscosity Data for Oil 2 Illustrating the Effects of Chemical Additives and Sonication**

| Treatment Conditions | Fractional Residual Viscosity | | | | |
|---|---|---|---|---|---|
| | Time, (min) | | | | |
| | 0 | 30 | 60 | 90 | 120 |
| 0.8 & 1.2 kHz, 3% A + 10% B | 1 | 0.8510 | 0.7900 | 0.6968 | 0.5650 |
| 0.8 & 1.2 kHz, 5% A + 10% B | 1 | 0.9352 | 0.8982 | 0.7877 | 0.7000 |
| 0.8 & 1.2 kHz, 7% A + 10% B | 1 | 0.9545 | 0.8609 | 0.8054 | 0.7803 |
| 0.8 & 1.6 kHz, 3% A + 10% B | 1 | 0.8627 | 0.7947 | 0.7073 | 0.5764 |
| 0.8 & 1.6 kHz, 5% A + 10% B | 1 | 0.9438 | 0.9049 | 0.7796 | 0.6326 |
| 0.8 & 1.6 kHz, 7% A + 10% B | 1 | 0.8996 | 0.8238 | 0.7339 | 0.6497 |

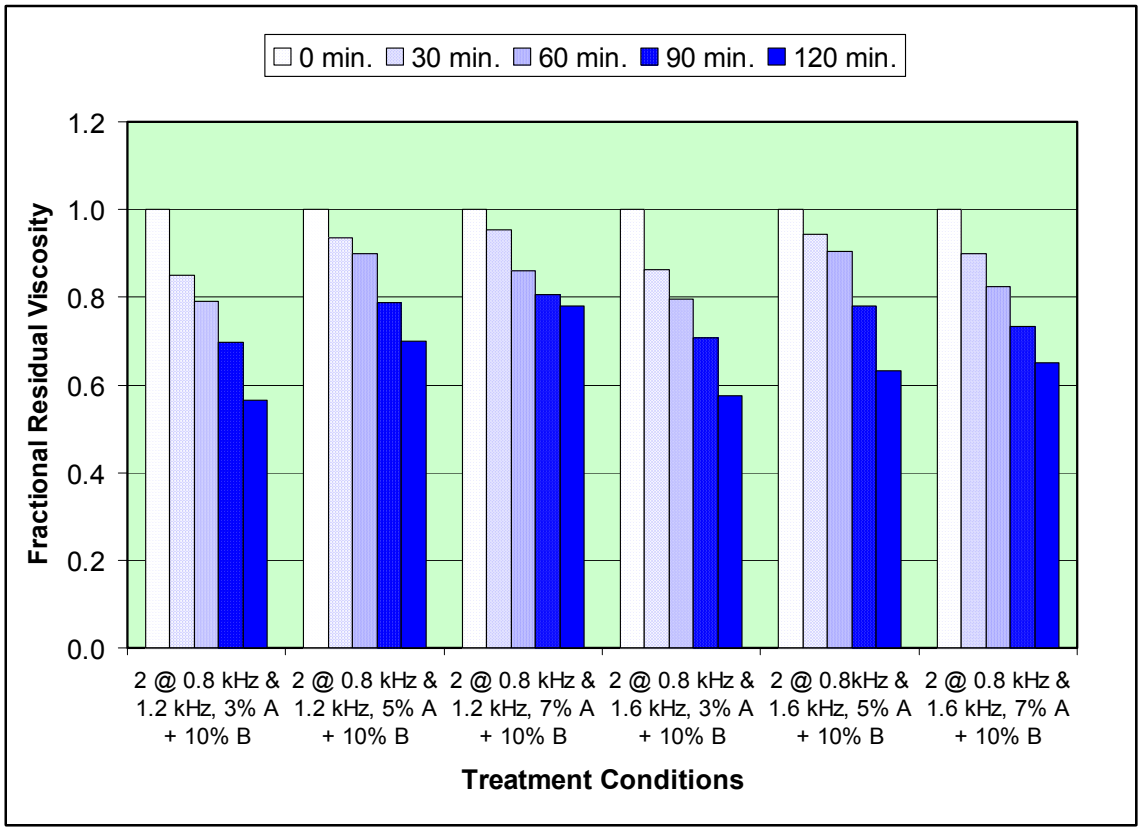


**Figure 66 Variation of Fractional Residual Viscosity of Oil 2 with Changes in Sonication Frequencies and Amount of Chemical Additives**

### 3.8.5 Viscosity Recovery

Based upon some initial observations during Phase I and during the testing of Crude Oil 1, the project team decided that the issue of viscosity recovery should be examined systematically to determine if the reductions in viscosity were short-lived or longer-term. A set of experiments was designed using Oil 2 wherein oil samples collected after the various treatments (acoustic frequencies, horn design, chemical additives) would be monitored for a period of 30 days to obtain data on the amount and rate of viscosity recovery. These tests would also provide information as to the relative effectiveness of the various treatments in terms of how long the resulting viscosity reductions might be expected to remain. These questions clearly have a bearing on the ultimate applications of the technology within commercial operations.

Tables 29 and 30 contain the viscosity recovery data for Crude Oil 2 following sonication treatment using the wider, 2-in (5-cm) horn fin spacing. The actual measured viscosity values are presented in Table 29, and Table 30 contains the fractional residual viscosity recovery data. In both cases, as well as the other tables presented in this section, the initial viscosity values represent the viscosity of the oil prior to the beginning of the sonication treatment experiments. The data for 0 days following treatment are the data obtained after 120 minutes of sonication testing during the initial experiments. The viscosity of each sample was subsequently measured after the sample was allowed to sit undisturbed for periods of 1, 3, 7, 14, and 30 days. Tables 31 and 32 contain analogous data collected using the narrow (1 in, 2.5 cm) horn spacing.

**Table 29 Viscosity Recovery in Crude Oil 2 Following Sonication Using the Wide Horn Spacing**

| Treatment Conditions | Viscosity, (cP) | | | | | | |
|---|---|---|---|---|---|---|---|
| | Time Following Treatment, (days) | | | | | | |
| | Initial | 0 | 1 | 3 | 7 | 14 | 30 |
| 2 actuators @ 0.8 kHz each | 4553 | 5093 | 5123 | 5467 | 5483 | 5472 | 5464 |
| 2 actuators @ 1.2 kHz each | 4827 | 3957 | 4233 | 4397 | 4423 | 4435 | 4429 |
| 2 actuators @ 1.6 kHz each | 4943 | 5667 | 5643 | 5650 | 5680 | 5674 | 5682 |
| 2 actuators @ 0.8 and 1.2 kHz | 4983 | 3828 | 3880 | 3893 | 3923 | 3942 | 3935 |
| 2 actuators @ 0.8 and 1.6 kHz | 4577 | 3552 | 3710 | 3747 | 3763 | 3748 | 3765 |
| 2 actuators @ 1.2 and 1.6 kHz | 4633 | 3768 | 3942 | 4080 | 4167 | 4151 | 4159 |

Tables 29 and 30 indicate that the viscosity of Oil 2 increased during the rest period after sonication treatment. However, the amount of change is very small. When the data for 0 and 30 days following treatment are compared, the viscosity increases by a maximum of 11.9% and a minimum of less than one percent. It is interesting to note that the resting viscosity tends to increase in all cases, even the two tests that resulted in increased viscosity following exposure to sonication. When the 30-day viscosity data are compared with the initial (pre-sonication) values,

the maximum change is 21.0% and the minimum is 8.2%. Consequently, the changes that occurred as a result of sonication are sustained during the rest period.

**Table 30 Fractional Residual Viscosity Recovery in Crude Oil 2 Following Sonication Using the Wide Horn Spacing**

| Treatment Conditions | Fractional Residual Viscosity, (cP) | | | | | | |
|---|---|---|---|---|---|---|---|
| | Time Following Treatment, (days) | | | | | | |
| | Initial | 0 | 1 | 3 | 7 | 14 | 30 |
| 2 actuators @ 0.8 kHz each | 1 | 1.1186 | 1.1252 | 1.2001 | 1.2043 | 1.2018 | 1.2001 |
| 2 actuators @ 1.2 kHz each | 1 | 0.8198 | 0.8770 | 0.9109 | 0.9163 | 0.9188 | 0.9176 |
| 2 actuators @ 1.6 kHz each | 1 | 1.1465 | 1.1416 | 1.1430 | 1.1491 | 1.1479 | 1.1495 |
| 2 actuators @ 0.8 and 1.2 kHz | 1 | 0.7682 | 0.7787 | 0.7813 | 0.7873 | 0.7911 | 0.7897 |
| 2 actuators @ 0.8 and 1.6 kHz | 1 | 0.7761 | 0.8106 | 0.8187 | 0.8222 | 0.8189 | 0.8226 |
| 2 actuators @ 1.2 and 1.6 kHz | 1 | 0.8133 | 0.8509 | 0.8806 | 0.8994 | 0.8960 | 0.8977 |

**Table 31 Viscosity Recovery in Crude Oil 2 Following Sonication Using the Narrow Horn Spacing**

| Treatment Conditions | Viscosity, (cP) | | | | | | |
|---|---|---|---|---|---|---|---|
| | Time Following Treatment, (days) | | | | | | |
| | Initial | 0 | 1 | 3 | 7 | 14 | 30 |
| 2 actuators @ 0.8 kHz each | 6440 | 3080 | 3347 | 3443 | 3477 | 3468 | 3459 |
| 2 actuators @ 1.2 kHz each | 6633 | 2333 | 2923 | 2830 | 2847 | 2839 | 2854 |
| 2 actuators @ 1.6 kHz each | 6220 | 1982 | 2290 | 2313 | 2320 | 2332 | 2319 |
| 2 actuators @ 0.8 and 1.2 kHz | 5927 | 1758 | 2087 | 2133 | 2147 | 2142 | 2136 |
| 2 actuators @ 0.8 and 1.6 kHz | 6864 | 1660 | 1924 | 2268 | 2310 | 2321 | 2314 |
| 2 actuators @ 1.2 and 1.6 kHz | 6333 | 1882 | 2140 | 2264 | 2288 | 2291 | 2304 |

The data in Tables 31 and 32 illustrate somewhat different results. First, as discussed previously, the sonication treatment using the smaller horn spacing resulted in a dramatically reduced viscosity under all treatment conditions compared to the results obtained with the wider horn spacing (compare initial and 0 days data in these two tables). During the recovery period, viscosity obtained with the narrow horn spacing similarly increased in all cases, ranging from a minimum increase during the 30 days of 12.3% to a maximum of 39.4%. However, these increases were small when compared to the amount of viscosity reduction achieved by sonication. When the data for 30 days are compared to the initial (pre-sonication), the minimum reduction in viscosity is 46.3% whereas the maximum reduction is 66.3%. Therefore, it is safe to conclude from these data that the viscosity was reduced by approximately 50% for a period of 30 days after the conclusion of the sonication treatment using the small horn spacing.

**Table 32 Fractional Residual Viscosity Recovery in Crude Oil 2 Following Sonication Using the Narrow Horn Spacing**

| Treatment Conditions | Fractional Residual Viscosity, (cP) | | | | | | |
|---|---|---|---|---|---|---|---|
| | Time Following Treatment, (days) | | | | | | |
| | Initial | 0 | 1 | 3 | 7 | 14 | 30 |
| 2 actuators @ 0.8 kHz each | 1 | 0.4783 | 0.5198 | 0.5346 | 0.5399 | 0.5385 | 0.5371 |
| 2 actuators @ 1.2 kHz each | 1 | 0.3517 | 0.4407 | 0.4267 | 0.4292 | 0.4280 | 0.4303 |
| 2 actuators @ 1.6 kHz each | 1 | 0.3187 | 0.3682 | 0.3719 | 0.3730 | 0.3749 | 0.3728 |
| 2 actuators @ 0.8 and 1.2 kHz | 1 | 0.2966 | 0.3521 | 0.3599 | 0.3622 | 0.3614 | 0.3604 |
| 2 actuators @ 0.8 and 1.6 kHz | 1 | 0.2418 | 0.2803 | 0.3304 | 0.3365 | 0.3381 | 0.3371 |
| 2 actuators @ 1.2 and 1.6 kHz | 1 | 0.2972 | 0.3379 | 0.3575 | 0.3613 | 0.3618 | 0.3638 |

The data in Table 31 are presented graphically in Figure 67. This plot clearly illustrates the large decrease in viscosity attained and maintained with sonication using the narrow horn fin spacing. This figure also shows that the small amount of viscosity recovery occurs primarily in the first seven days or less following the treatment, and that it remains fairly constant during the last three weeks of the recovery period.

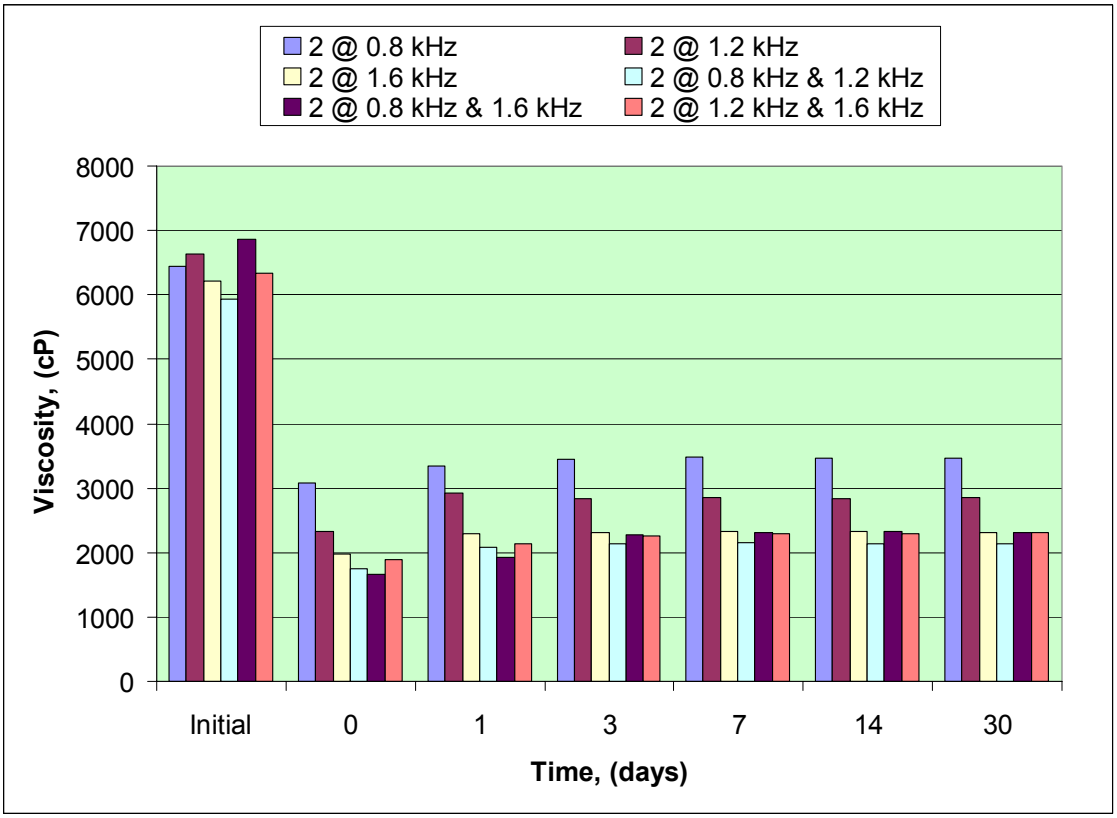


**Figure 67 Viscosity Recovery with Time Following Sonication with Reduced Fin Spacing for Crude Oil 2**

Finally, a third set of viscosity recovery data was collected using the samples collected at the conclusion of the series of tests involving sonication and chemical additives. These data are given in Tables 33, 34, and 35. The first of these tables contains both the viscosity data following the addition of the chemical mixes to Oil 2 and the average pre-additives viscosity in addition to the viscosity recovery data. The column labeled "Initial A+B" contains the viscosity

**Table 33 Crude Oil 2 Viscosity Recovery with Time Following Treatment with Sonication Using Small Horn Spacing and Chemical Additives**

| Treatment Conditions | Viscosity, (cP) | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | Time Following Treatment, (days) | | | | | | | |
| | Avg. Initial | Initial A+B | 0 | 1 | 3 | 7 | 14 | 30 |
| 0.8 & 1.2 kHz, 3% A + 10% B | 6402 | 492 | 278 | 360 | 416 | 421 | 439 | 443 |
| 0.8 & 1.2 kHz, 5% A + 10% B | 6402 | 525 | 367 | 401 | 433 | 466 | 472 | 479 |
| 0.8 & 1.2 kHz, 7% A + 10% B | 6402 | 382 | 298 | 362 | 423 | 429 | 434 | 437 |
| 0.8 & 1.6 kHz, 3% A + 10% B | 6402 | 519 | 299 | 369 | 384 | 407 | 416 | 422 |
| 0.8 & 1.6 kHz, 5% A + 10% B | 6402 | 463 | 293 | 360 | 392 | 390 | 394 | 393 |
| 0.8 & 1.6 kHz, 7% A + 10% B | 6402 | 427 | 278 | 300 | 339 | 375 | 289 | 395 |

**Table 34 Recovery of Fractional Residual Viscosity of Oil 2 with Time Following Treatment with Sonication and Chemical Additives Using Post-Additives Viscosity as Initial Value**

| Treatment Conditions | Fractional Residual Viscosity, (cP) | | | | | | |
|---|---|---|---|---|---|---|---|
| | Time Following Treatment, (days) | | | | | | |
| | Initial A+B | 0 | 1 | 3 | 7 | 14 | 30 |
| 0.8 & 1.2 kHz, 3% A + 10% B | 1 | 0.5650 | 0.7325 | 0.8459 | 0.8553 | 0.8931 | 0.9006 |
| 0.8 & 1.2 kHz, 5% A + 10% B | 1 | 0.7000 | 0.7644 | 0.8245 | 0.8878 | 0.9003 | 0.9131 |
| 0.8 & 1.2 kHz, 7% A + 10% B | 1 | 0.7803 | 0.9461 | 1.1067 | 1.1219 | 1.1355 | 1.1438 |
| 0.8 & 1.6 kHz, 3% A + 10% B | 1 | 0.5764 | 0.7110 | 0.7385 | 0.7845 | 0.8011 | 0.8123 |
| 0.8 & 1.6 kHz, 5% A + 10% B | 1 | 0.6326 | 0.7783 | 0.8481 | 0.8422 | 0.8517 | 0.8485 |
| 0.8 & 1.6 kHz, 7% A + 10% B | 1 | 0.6497 | 0.7014 | 0.7924 | 0.8769 | 0.9092 | 0.9237 |

data for Oil 2 following the addition of the chemicals (post-additives) but before that sample was treated with sonication. As with the previous tests of viscosity recovery, the viscosity at time 0 days is the value measured after the sample was exposed to sonication for 120 minutes and at the beginning of the recovery period. As discussed previously, the chemical additives are very effective in reducing Oil 2 viscosity without sonication. When the oil with additives is exposed to sonication, the viscosity is reduced even further. In terms of recovery, the data in Table 33

show that the viscosity values change only by small amounts during the 30 days following treatment. This situation is further illustrated by examining the fractional residual viscosity values presented in Table 34. These normalized values are based on the post-additives viscosity (Initial A+B). The changes in viscosity during the recovery period are clear in these data. In all cases, the residual viscosity increased during the recovery period, and in one case, the recovery at the end of the rest period exceeded the initial viscosity value after adding the chemical mix and prior to sonication. The remaining five values indicate that the final viscosity was approaching the pre-sonication, post-additives value. However, as one can determine from Table 33, the absolute value of viscosity change in all tests during the recovery period was very small.

**Table 35 Recovery of Fractional Residual Viscosity of Oil 2 with Time Following Treatment with Sonication and Chemical Additives Using Pre-Additives Viscosity as Initial Value**

| Treatment Conditions | Fractional Residual Viscosity | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | Time Following Treatment, (days) | | | | | | | |
| | Avg. Initial | Initial A+B | 0 | 1 | 3 | 7 | 14 | 30 |
| 0.8 kHz & 1.2 kHz, 3% A + 10% B | 1 | 0.0769 | 0.0434 | 0.0562 | 0.0650 | 0.0658 | 0.0686 | 0.0692 |
| 0.8 kHz & 1.2 kHz, 5% A + 10% B | 1 | 0.0820 | 0.0573 | 0.0626 | 0.0676 | 0.0676 | 0.0737 | 0.0748 |
| 0.8 kHz & 1.2 kHz, 7% A + 10% B | 1 | 0.0597 | 0.0465 | 0.0565 | 0.0661 | 0.0670 | 0.0678 | 0.0683 |
| 0.8 kHz & 1.6 kHz, 3% A + 10% B | 1 | 0.0811 | 0.0467 | 0.0576 | 0.0600 | 0.0636 | 0.0650 | 0.0659 |
| 0.8 kHz & 1.6 kHz, 5% A + 10% B | 1 | 0.0723 | 0.0458 | 0.0562 | 0.0612 | 0.0609 | 0.0615 | 0.0614 |
| 0.8 kHz & 1.6 kHz, 7% A + 10% B | 1 | 0.0667 | 0.0434 | 0.0469 | 0.0530 | 0.0586 | 0.0608 | 0.0617 |

The relative significance of the viscosity recovery in Oil 2 under these test conditions can be judged from the data in Table 35 where the fractional residual (normalized) viscosity data are based on the viscosity of Oil 2 before any chemicals were added and before sonication. These data indicate that the residual viscosity is reduced to only about 5% to 8% of the initial value by the addition of the chemical mixture. During the recovery period, the residual viscosity increased by only about 1.5% or less of the pre-treatment viscosity in all cases.

### 3.8.6 Summary of Crude Oil 2 Results

Tests were conducted to evaluate the effects of heat on the viscosity of Crude Oil 2. Test results demonstrated that the viscosity decreased in an exponential/power function relationship with increasing temperature. As was the case with Oil 1, the viscosities during the cooling cycle were less than those at similar temperatures measured during the heating cycle.

Experiments to determine the results of sonication on viscosity were performed using two parallel actuators operating at six different combinations of acoustic frequencies. Initial experiments were conducted with a two-inch (five-centimeter) fin spacing on the horns attached to the actuators. Data obtained during these tests indicated that under one of the six test conditions, viscosity increased with increasing exposure time. In another test, viscosity initially increased above the pre-treatment value, but subsequently decreased to a value that was still in excess of the initial viscosity. These results were contrary to the results obtained with Oil 1. It was concluded that this situation resulted because of the appreciable and variable water content in the oil samples. Those tests producing increased viscosities also resulted in an observable separated water layer in the post-treatment samples. This separation was not readily apparent in those samples where viscosities were reduced. A maximum reduction of about 23% was measured. When the series of experiments was repeated using a one-inch (2.5-cm) horn spacing, all test results exhibited a dramatic reduction in viscosity following sonication with the maximum reduction being almost 76%; the minimum reduction with these test conditions was 52.2%. Thus the horn with the reduced spacing was far more effective than the one with the larger spacing. The combination of frequencies of 0.8 kHz and 1.2 kHz appear to provide consistently better viscosity reductions in Crude Oil 2 with the combination of 0.8 kHz and 1.6 kHz also providing good results. These observations agree with those obtained with Crude Oil 1.

Another series of tests was performed to evaluate the effects of chemical additives along with sonication using the narrow horn spacing. The same chemical mixes were used as were used with Oil 1. Interestingly, the addition of the chemicals reduced the viscosity reduced the viscosity of the oil by more than 90%, and subsequent exposure of the sample to sonication reduced viscosities even further. The combination of additives and sonication was extremely effective in reducing the viscosity of Oil 2. The mix of 7% additive A along with 10% additive B appears to be the most effective followed by 5% A and 3% A in combination with 10% B. However, the differences in results arising from varying the amount of chemical additives is very small.

Finally, a series of tests was conducted to evaluate viscosity recovery in samples allowed to sit undisturbed for 30 days following the individual tests. In all of the tests, viscosity increased during the recovery period. However, in all cases, the relative amounts of increase, compared to the pre-treatment values, were very small. When all recovery data are examined, the results obtained with the narrow horn spacing were clearly better than those obtained with the wide horn spacing. In no case did the viscosity at the end of the month-long recovery period return to a value approximating the pre-treatment value. The final fractional residual viscosity at the end of 30 days following sonication alone and using the narrow horn spacing ranged from 0.3371 to 0.5371. When the results involving both sonication and chemical additives are examined, one sample increased to a Fractional Residual Viscosity of 1.14 when the initial sample viscosity after adding the chemical mix is used as the point of reference; the rest of the samples remained below this reference value. When the initial pre-treatment (before adding

chemical and before sonication) viscosity is used as a reference, the maximum Fractional Residual Viscosity attained after 30 days was only 0.0748. Therefore, the experimental evidence indicates that viscosity reductions resulting from sonication alone and from sonication in combination with the tested chemical additives persist for at least 30 days following treatment.

## 3.9 Experimental Results Using Crude Oil 3

The experimental procedures used to test Crude Oil 3 were the same as those used to test Crude Oil 2. To summarize, all sonication testing of Oil 3 was performed with two transducers arranged in parallel (horns facing each other) within the two horizontal components of the reaction vessel (see Figure 47). Testing using 25% input power reduction was not performed because the results from Oil 1 indicated that differences in viscosity reduction using reduced power compared to results with normal power levels was relatively small. Third, only the two best combinations of horn design and acoustic frequency were used in conjunction with testing the effects of chemical additives in order to conserve the quantities of oil required for the experiments. Lastly, a number of measurements were collected to systematically examine viscosity recovery over a period of 30 days.

### 3.9.1 Temperature Effects on Viscosity

As was the case with Oils 1 and 2, the first tests performed on Oil 3 were heating experiments to determine the relationship between heat/temperature and measured viscosity. The results of these tests are presented in Figure 68. These results are similar to those obtained during the previous tests, except that the hysteresis effect noted in the plot for Oil 1 is not present in the Oil 3 results. This is similar to the results obtained with Oil 2 in that regard. In all three series of tests, the final viscosity of the cooling cycle is less than the initial viscosity of the oil measured prior to heating.

### 3.9.2 Sonication Effects on Viscosity

The test matrix designed to evaluate the effects of sonication on the viscosity of Crude Oil 3 was identical with that employed with Oil 2. In total, six different actuator/frequency combinations were evaluated. As in the previous experiments with Oils 1 and 2, individual tests were conducted for a total of 120 minutes with individual samples collected for viscosity determination after 0 (pre-treatment), 30, 60, 90, and 120 minutes.

The viscosity data obtained by sonicating Oil 3 with the selected acoustic frequencies and two parallel actuators fitted with horns having a two-inch (5-cm) fin spacing are presented in Table 36. The corresponding “normalized” viscosity values, or fractional residual viscosity, are given in Table 37, and a plot of these fractional residual data is presented in Figure 69.

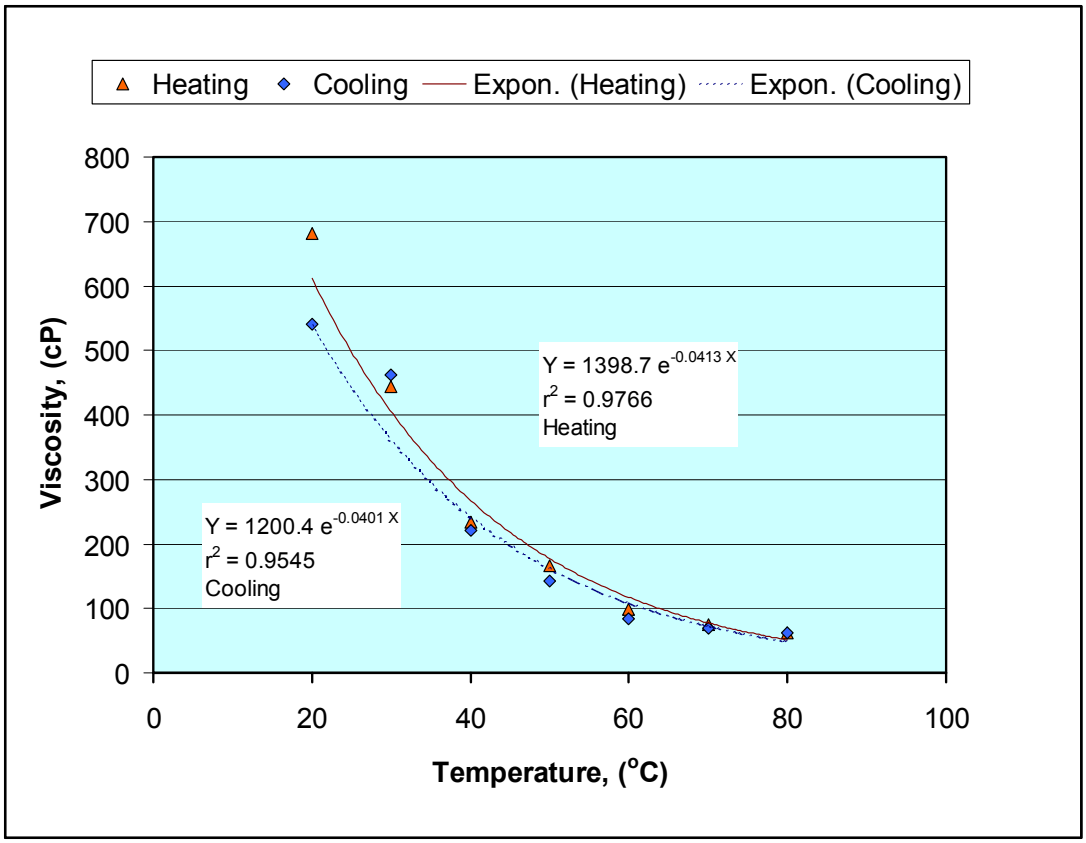


**Figure 68 Viscosity of Crude Oil 3 as a Function of Temperature**

**Table 36 Summary of Viscosity Results from Acoustic Treatment of Crude Oil 3**

| Treatment Conditions | Viscosity, (cP) | | | | |
|---|---|---|---|---|---|
| | Time, (min) | | | | |
| | 0 | 30 | 60 | 90 | 120 |
| 2 actuators @ 0.8 kHz each | 622 | 655 | 720 | 709 | 587 |
| 2 actuators @ 1.2 kHz each | 626 | 684 | 647 | 619 | 616 |
| 2 actuators @ 1.6 kHz each | 637 | 722 | 753 | 689 | 683 |
| 2 actuators @ 0.8 and 1.2 kHz | 633 | 712 | 678 | 652 | 594 |
| 2 actuators @ 0.8 and 1.6 kHz | 660 | 726 | 654 | 638 | 621 |
| 2 actuators @ 1.2 and 1.6 kHz | 646 | 679 | 694 | 666 | 646 |

These data exhibit a pattern of variation generally similar to that shown in the tests with Oils 1 and 2. One can see that there is a general trend for viscosity to decrease with time for each set of frequency conditions. That is, in most cases, the viscosities observed after 120 minutes are less than the initial viscosity values at the start of the tests. There was one exception to this trend (two actuators operated at 1.6 kHz). As was observed in the sonication tests of Oil 2, data in Tables 36 and 37 and Figure 69 indicate that in some cases the viscosity increases after

exposure to sonication before subsequently decreasing by the conclusion of the tests, a condition not observed in the test results using Oil 1. In the tests involving Oil 2, this situation attributed to the presence of an appreciable quantity of water in the samples of oil. However, water separation was not observed in any of the Oil 3 samples; therefore, no probable explanation for this phenomenon in the Oil 3 data can be provided at this time.

**Table 37 Summary of Fractional Residual Viscosity Results for Acoustic Treatment of Crude Oil 3**

| Treatment Conditions | Fractional Residual Viscosity | | | | |
|---|---|---|---|---|---|
| | Time, (min) | | | | |
| | 0 | 30 | 60 | 90 | 120 |
| 2 actuators @ 0.8 kHz each | 1 | 1.0526 | 1.1576 | 1.1395 | 0.9442 |
| 2 actuators @ 1.2 kHz each | 1 | 1.0927 | 1.0331 | 0.9891 | 0.9840 |
| 2 actuators @ 1.6 kHz each | 1 | 1.1335 | 1.1812 | 1.0808 | 1.0720 |
| 2 actuators @ 0.8 and 1.2 kHz | 1 | 1.1243 | 1.0712 | 1.0295 | 0.9386 |
| 2 actuators @ 0.8 and 1.6 kHz | 1 | 1.1006 | 0.9915 | 0.9673 | 0.9406 |
| 2 actuators @ 1.2 and 1.6 kHz | 1 | 1.0514 | 1.0749 | 1.0316 | 0.9994 |

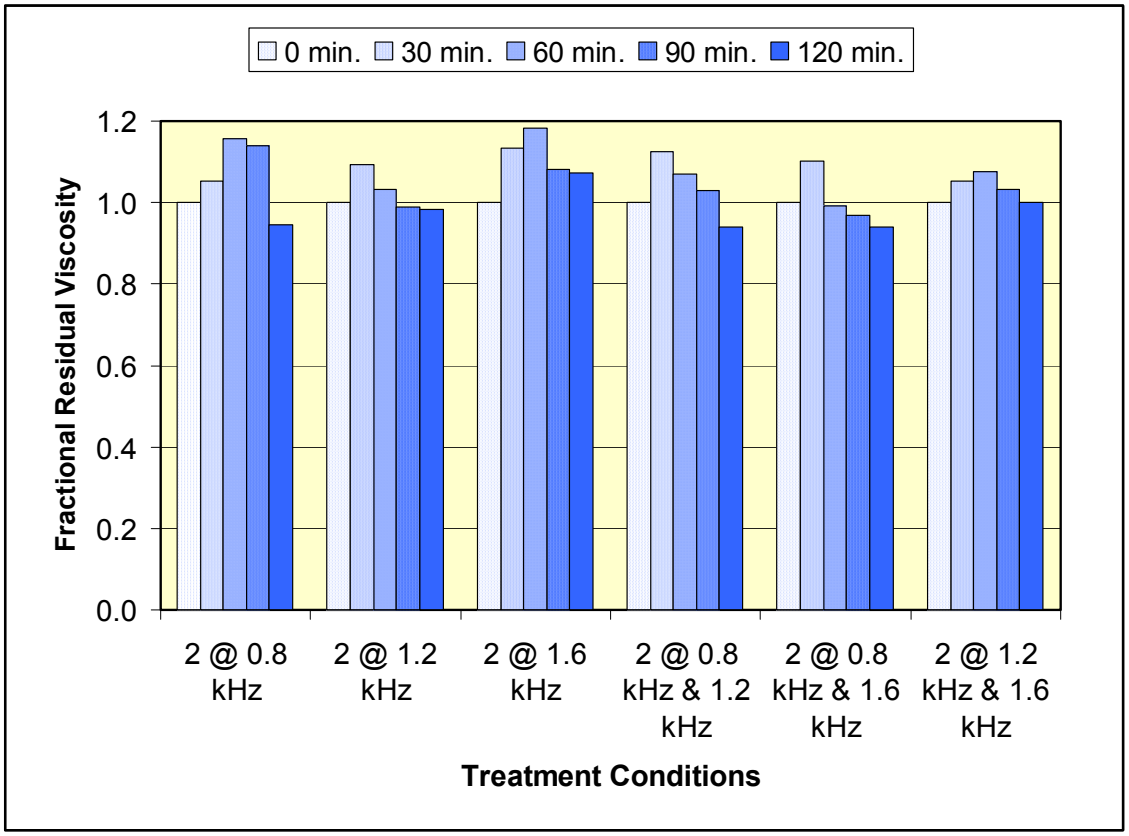


**Figure 69 Fractional Residual Viscosity Variation of Oil 3 with Changing Acoustic Treatment Conditions**

The data indicate that the maximum viscosity occurs on average after 30 to 60 minutes of treatment, followed by a reduction in the 90 and 120 minute samples. This lends support to the point made previously (Section 3.6.2) that it normally takes at least three residence times for a flow-through system to approach steady state conditions. The maximum amount of viscosity reduction attained in Oil 3 after 120 minutes of testing with sonication using the wide horn design was only 6.1% (fractional residual viscosity = 0.9386). This small effect of sonication on Oil 3 is to be expected because the oil naturally has a very low viscosity in the untreated state. One would expect the greatest effects to be observed in oils with greater initial viscosity values (which was the case in these tests). Figure 69 also shows that there is very little difference in the effects of the various treatment conditions employed during these experiments. Again, this result might be expected because of the very low viscosity of Oil 3.

### 3.9.3 Effects of Horn Design on Viscosity

All of the experiments described in the preceding report section were performed with the wide, 2-inch (5-cm) horn fin spacing. The experiments were repeated using the reduced, 1-inch (2.5-cm) fin spacing on the acoustic horn. These data reflecting the reduced horn spacing are presented in Table 38, Table 39, and Figure 70.

**Table 38 Summary of Viscosity Results for Acoustic Treatment of Crude Oil 3 using Reduced Horn Spacing**

| Treatment Conditions | Viscosity, (cP) | | | | |
|---|---|---|---|---|---|
| | Treatment Time, (min.) | | | | |
| | 0 | 30 | 60 | 90 | 120 |
| 2 actuators @ 0.8 kHz each | 627 | 764 | 747 | 724 | 605 |
| 2 actuators @ 1.2 kHz each | 628 | 691 | 645 | 616 | 625 |
| 2 actuators @ 1.6 kHz each | 627 | 729 | 733 | 705 | 649 |
| 2 actuators @ 0.8 and 1.2 kHz | 649 | 728 | 711 | 694 | 610 |
| 2 actuators @ 0.8 and 1.6 kHz | 648 | 696 | 662 | 645 | 619 |
| 2 actuators @ 1.2 and 1.6 kHz | 652 | 740 | 699 | 670 | 649 |

As in the previous examples, the data reflect a general trend of decreasing viscosity as the amount of treatment time increases following an initial viscosity increase at 30 minutes treatment time. In these tests with Oil 3, only a small amount of variation in the viscosity and fractional residual viscosity values is observed for the various treatment conditions. In other words, the pattern and magnitude of viscosity change is essentially the same, regardless of the treatment conditions employed. With the 1-in. (2.5-cm) horn spacing, the maximum reduction in viscosity was 6.1%, which results in a fractional residual viscosity of 93.9%. This result is nearly identical to that obtained using the wider, 2-in (5-cm) horn spacing. A comparison of Figures 69 and 70 indicates that there is very little difference in the results obtained by varying the horn design.

These results add further credence to the idea that the effects of sonication and the variables involved in delivering acoustic energy to the oil are dramatically less in low-viscosity oils than when the technology is applied to thicker, more viscous oils.

**Table 39 Summary of Fractional Residual Viscosity Results for Acoustic Treatment of Crude Oil 3 with Reduced Horn Spacing**

| Treatment Conditions | Fractional Residual Viscosity | | | | |
|---|---|---|---|---|---|
| | Treatment Time, (min.) | | | | |
| | 0 | 30 | 60 | 90 | 120 |
| 2 actuators @ 0.8 kHz each | 1 | 1.2179 | 1.1911 | 1.1542 | 0.9640 |
| 2 actuators @ 1.2 kHz each | 1 | 1.1006 | 1.0275 | 0.9809 | 0.9949 |
| 2 actuators @ 1.6 kHz each | 1 | 1.1629 | 1.1693 | 1.1246 | 1.0353 |
| 2 actuators @ 0.8 and 1.2 kHz | 1 | 1.1212 | 1.0953 | 1.0695 | 0.9389 |
| 2 actuators @ 0.8 and 1.6 kHz | 1 | 1.0741 | 1.0222 | 0.9951 | 0.9556 |
| 2 actuators @ 1.2 and 1.6 kHz | 1 | 1.1350 | 1.0724 | 1.0282 | 0.9951 |

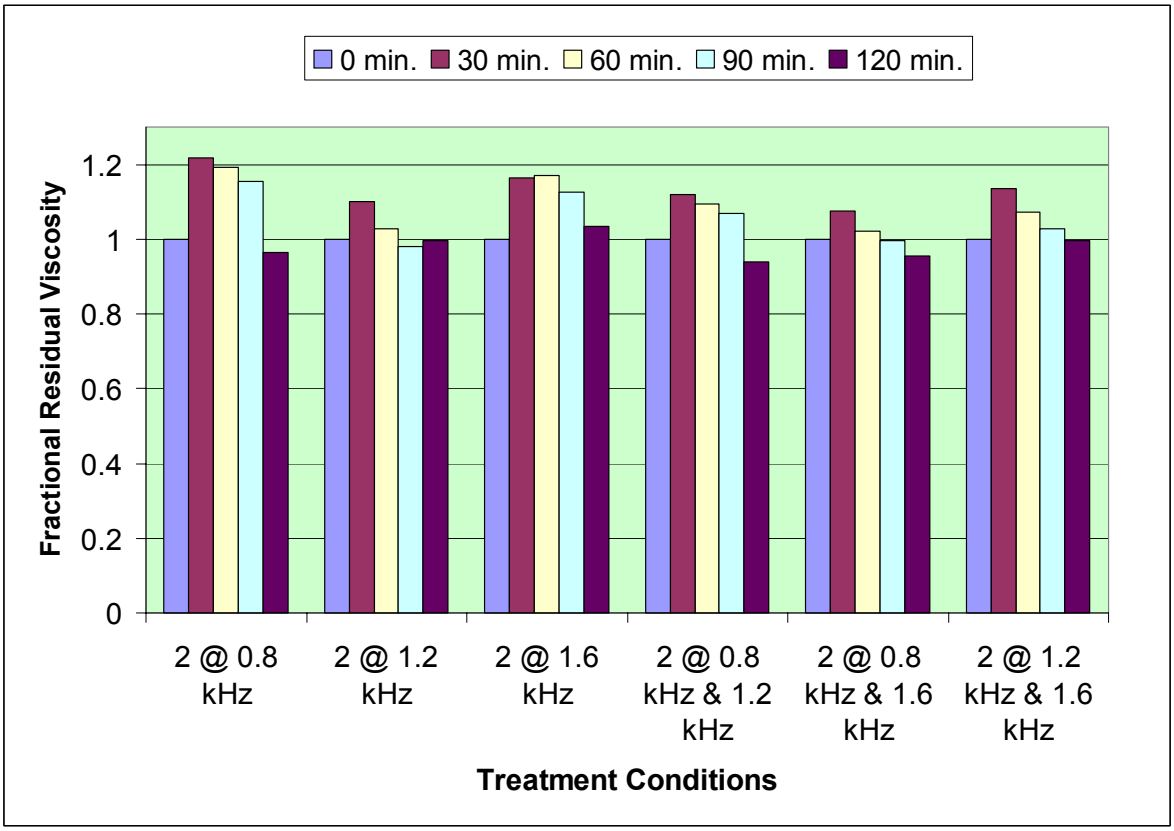


**Figure 70 Fractional Residual Viscosity Variation of Oil 3 with Changing Acoustic Treatment Conditions and Reduced Horn Spacing**

### 3.9.4 Effects of Chemical Additives on Viscosity

A series of experiments examining the effects of chemical additives on the viscosity of Oil 3 was performed. As noted at the outset of this discussion of the experiments involving Oil 3, the same experimental procedures that were used with Oil 2 were employed with Oil 3, with one exception. Because the quantity of Oil 3 that was available for testing was not as much of a limiting factor as it was with Oil 2, each test involving chemical additives presented in Table 40 below began with a fresh crude oil sample. The same additives used with Oils 1 and 2 were also used with Oil 3 and similar methods of mixing the additives with the oil, operating the test equipment, sample collection, and data collection were employed during this third series of tests. The viscosity of an initial sample of oil was measured prior to beginning the experiment. The oil and additives A and B at initial concentrations (3% and 10%) were mixed, and the first test was performed. Samples were collected at 0, 30, 60, 90, and 120 minutes, and the viscosity of each sample was measured. The test apparatus was drained and cleaned, and a fresh sample of crude oil was obtained. The concentrations of chemicals A and B were adjusted to 5% and 10% and the second test was performed at the same frequencies. This procedure was repeated for the remaining tests in the series. The test conditions employed and the corresponding viscosity results for Oil 3 are presented in Table 40. All tests were performed with the horn fin spacing of 1 in (2.5 cm).

**Table 40 Viscosity Values for Oil 3 Reflecting the Effects of Chemical Additives and Sonication**

| Treatment Conditions | Viscosity, (cP) | | | | | |
|---|---|---|---|---|---|---|
| | Time, (min) | | | | | |
| | Initial | 0 | 30 | 60 | 90 | 120 |
| 2 actuators @ 0.8 kHz & 1.2 kHz 3% A + 10% B | 627 | 359 | 295 | 275 | 253 | 238 |
| 2 actuators @ 0.8 kHz & 1.2 kHz 5% A + 10% B | 628 | 320 | 298 | 270 | 266 | 256 |
| 2 actuators @ 0.8 kHz & 1.2 kHz 7% A + 10% B | 626 | 232 | 230 | 220 | 190 | 182 |
| 2 actuators @ 0.8 kHz & 1.6 kHz 3% A + 10% B | 649 | 226 | 209 | 205 | 189 | 180 |
| 2 actuators @ 0.8 kHz & 1.6 kHz 5% A + 10% B | 648 | 174 | 139 | 118 | 117 | 98 |
| 2 actuators @ 0.8 kHz & 1.6 kHz 7% A + 10% B | 652 | 174 | 168 | 157 | 109 | 97 |

One readily noticeable observation that can be made from Table 40 is the fact that a very large reduction in viscosity is attained with the addition of the chemical mix alone without sonication. This result was also observed with the other crude oils that were tested previously. The minimum viscosity (maximum reduction) obtained with only the chemical additives is 174

cP which represents a 73% reduction in the initial viscosity. The minimum viscosity reduction obtained with only the chemical additives resulted in a viscosity of 359 cP which equates to a 43% reduction. Thus, the chemical additives that were evaluated were very effective in reducing the viscosity of even the low-viscosity Crude Oil 3. When sonication is added to the treatment conditions, viscosity is reduced even further. As in the other series of experiments, longer treatment times resulted in lower viscosity values for all treatment conditions. The changes resulting from sonication after the chemical mix was added can best be seen when examining the fractional residual viscosities when the initial viscosity is taken to be that value after the chemicals have been added but before beginning sonication (time = 0 minutes in Table 41). These normalized viscosity values for Oil 3 in Table 41 also are shown graphically in Figure 71. These data indicate that sonication of samples of Oil 3 in the presence of chemical additives A and B can further reduce the viscosity by as much as approximately 56%. In terms of chemical additives alone, the largest viscosity reduction was produced by the addition of mixtures of 5% A and 10% B and 7% A and 10% B, although the difference in viscosity reduction among the various mixes of chemical additives is relatively small. The greatest final reduction in fractional residual viscosity resulted from chemical additives in 7% and 10% concentrations with 0.8 and 1.6 kHz acoustic frequencies, with the mixture of 5% A and 10% B producing nearly identical results with the same frequencies (Table 41 and Figure 71). This figure also clearly shows the pattern of reducing viscosity with increasing treatment times for all treatments.

**Table 41 Fractional Residual Viscosity Data for Oil 3 Illustrating the Effects of Chemical Additives and Sonication**

| Treatment Conditions | Fractional Residual Viscosity | | | | |
|---|---|---|---|---|---|
| | Time, (min) | | | | |
| | 0 | 30 | 60 | 90 | 120 |
| 2 actuators @ 0.8 kHz & 1.2 kHz 3% A + 10% B | 1 | 0.822 | 0.766 | 0.705 | 0.663 |
| 2 actuators @ 0.8 kHz & 1.2 kHz 5% A + 10% B | 1 | 0.931 | 0.845 | 0.834 | 0.802 |
| 2 actuators @ 0.8 kHz & 1.2 kHz 7% A + 10% B | 1 | 0.990 | 0.948 | 0.819 | 0.786 |
| 2 actuators @ 0.8 kHz & 1.6 kHz 3% A + 10% B | 1 | 0.922 | 0.905 | 0.834 | 0.797 |
| 2 actuators @ 0.8 kHz & 1.6 kHz 5% A + 10% B | 1 | 0.7995 | 0.682 | 0.673 | 0.567 |
| 2 actuators @ 0.8 kHz & 1.6 kHz 7% A + 10% B | 1 | 0.968 | 0.903 | 0.628 | 0.559 |

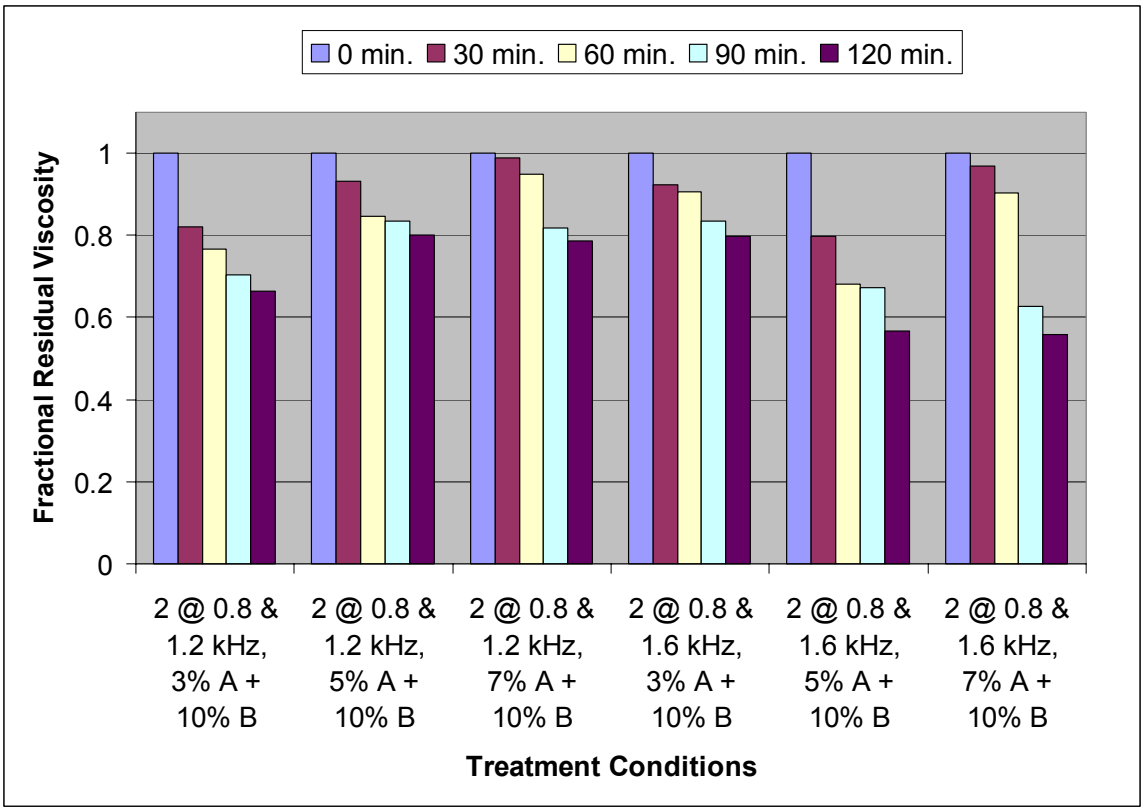


**Figure 71 Variation of Fractional Residual Viscosity of Oil 3 with Changes in Sonication Frequencies and Amount of Chemical Additives**

### 3.9.5 Viscosity Recovery

As was done during the experiments involving Oil 2, a set of experiments was designed using Oil 3 wherein oil samples collected after the various treatments (acoustic frequencies, horn design, chemical additives) were monitored for a period of 30 days to obtain data on the amount and rate of viscosity recovery. These data also provide information as to the relative effectiveness of the various treatments in terms of how long the resulting viscosity reductions might be expected to remain.

Tables 42 and 43 contain the viscosity recovery data for Crude Oil 3 following sonication treatment using the wider, 2-in (5-cm) horn fin spacing. The actual measured viscosity values are presented in Table 42, and Table 43 contains the fractional residual viscosity recovery data. In both cases, as well as the other tables presented in this section, the initial viscosity values represent the viscosity of the oil prior to the beginning of the sonication treatment experiments. The data for 0 days following treatment are the data obtained after 120 minutes of sonication testing during the initial experiments. The viscosity of each sample was subsequently measured after the sample was allowed to sit undisturbed for periods of 1, 3, 7, 14, and 30 days. Tables 44 and 45 contain analogous data collected using the narrow (1 in, 2.5 cm) horn spacing.

**Table 42 Viscosity Recovery in Crude Oil 3 Following Sonication Using the Wide Horn Spacing**

| Treatment Conditions | Viscosity, (cP) | | | | | | |
|---|---|---|---|---|---|---|---|
| | Time Following Treatment, (days) | | | | | | |
| | Initial | 0 | 1 | 3 | 7 | 14 | 30 |
| 2 actuators @ 0.8 kHz each | 622 | 587 | 591 | 590 | 593 | 597 | 596 |
| 2 actuators @ 1.2 kHz each | 626 | 616 | 618 | 615 | 622 | 623 | 623 |
| 2 actuators @ 1.6 kHz each | 637 | 683 | 692 | 694 | 696 | 695 | 692 |
| 2 actuators @ 0.8 and 1.2 kHz | 633 | 594 | 612 | 619 | 624 | 626 | 626 |
| 2 actuators @ 0.8 and 1.6 kHz | 650 | 621 | 631 | 635 | 639 | 641 | 642 |
| 2 actuators @ 1.2 and 1.6 kHz | 646 | 646 | 652 | 661 | 665 | 668 | 664 |

**Table 43 Fractional Residual Viscosity Recovery in Crude Oil 3 Following Sonication Using the Wide Horn Spacing**

| Treatment Conditions | Fractional Residual Viscosity, (cP) | | | | | | |
|---|---|---|---|---|---|---|---|
| | Time Following Treatment, (days) | | | | | | |
| | Initial | 0 | 1 | 3 | 7 | 14 | 30 |
| 2 actuators @ 0.8 kHz each | 1 | 0.9442 | 0.9503 | 0.9490 | 0.9529 | 0.9600 | 0.9585 |
| 2 actuators @ 1.2 kHz each | 1 | 0.9840 | 0.9875 | 0.9831 | 0.9943 | 0.9954 | 0.9946 |
| 2 actuators @ 1.6 kHz each | 1 | 1.0720 | 1.0865 | 1.0890 | 1.0923 | 1.0902 | 1.0860 |
| 2 actuators @ 0.8 and 1.2 kHz | 1 | 0.9386 | 0.9665 | 0.9774 | 0.9853 | 0.9885 | 0.9891 |
| 2 actuators @ 0.8 and 1.6 kHz | 1 | 0.9551 | 0.9709 | 0.9771 | 0.9837 | 0.9855 | 0.9875 |
| 2 actuators @ 1.2 and 1.6 kHz | 1 | 0.9994 | 1.0093 | 1.0237 | 1.0286 | 1.0344 | 1.0280 |

Tables 42 and 43 indicate that the viscosity of Oil 3 increased during the rest period after sonication treatment. However, the amount of change is very small. When the data for 0 and 30 days following treatment are compared, the viscosity increases by a maximum of 5.1% and a minimum of only about one percent. It is interesting to note that the resting viscosity tends to increase in all cases; that is when the post-treatment viscosity is both greater than and less than the initial viscosity prior to sonication. When the 30-day viscosity data are compared with the initial (pre-sonication) values, the maximum change is only 8.6% (both frequencies = 1.6 kHz) and the minimum change is less than one percent (both frequencies = 1.2 kHz). Consequently, the changes that occurred with the low-viscosity crude as a result of sonication are very small, but they are sustained during the rest period.

The data in Tables 44 and 45 illustrate similar results. These data, along with the observations discussed previously, indicate that changes in the horn design produce insignificant differences in viscosity reduction and recovery in this low-viscosity crude oil. With the narrow horn spacing, viscosity increased during the recovery period in all cases, ranging from a

minimum increase during the 30 days of 1.1% to a maximum of only 3.5%. When the data for 30 days are compared to the initial (pre-sonication), the maximum viscosity change remaining at the end of the recovery period is 4.5%. Therefore, one can conclude that sonication has very little effect on the viscosity of Oil 3 and that the effects of horn design are negligible. However, the changes that did occur were persistent during the month-long recovery period.

**Table 44 Viscosity Recovery in Crude Oil 3 Following Sonication Using the Narrow Horn Spacing**

| Treatment Conditions | Viscosity, (cP) | | | | | | |
|---|---|---|---|---|---|---|---|
| | Time Following Treatment, (days) | | | | | | |
| | Initial | 0 | 1 | 3 | 7 | 14 | 30 |
| 2 actuators @ 0.8 kHz each | 627 | 605 | 610 | 615 | 619 | 622 | 621 |
| 2 actuators @ 1.2 kHz each | 628 | 625 | 629 | 631 | 633 | 632 | 635 |
| 2 actuators @ 1.6 kHz each | 627 | 649 | 652 | 654 | 655 | 656 | 656 |
| 2 actuators @ 0.8 and 1.2 kHz | 649 | 610 | 615 | 619 | 622 | 626 | 626 |
| 2 actuators @ 0.8 and 1.6 kHz | 648 | 619 | 631 | 639 | 641 | 641 | 642 |
| 2 actuators @ 1.2 and 1.6 kHz | 652 | 649 | 654 | 659 | 661 | 660 | 663 |

**Table 45 Fractional Residual Viscosity Recovery in Crude Oil 3 Following Sonication Using the Narrow Horn Spacing**

| Treatment Conditions | Fractional Residual Viscosity, (cP) | | | | | | |
|---|---|---|---|---|---|---|---|
| | Time Following Treatment, (days) | | | | | | |
| | Initial | 0 | 1 | 3 | 7 | 14 | 30 |
| 2 actuators @ 0.8 kHz each | 1 | 0.9639 | 0.9729 | 0.9806 | 0.9868 | 0.9917 | 0.9906 |
| 2 actuators @ 1.2 kHz each | 1 | 0.9949 | 1.0022 | 1.0051 | 1.0081 | 1.0067 | 1.0110 |
| 2 actuators @ 1.6 kHz each | 1 | 1.0353 | 1.0402 | 1.0440 | 1.0453 | 1.0471 | 1.0462 |
| 2 actuators @ 0.8 and 1.2 kHz | 1 | 0.9389 | 0.9476 | 0.9535 | 0.9584 | 0.9637 | 0.9643 |
| 2 actuators @ 0.8 and 1.6 kHz | 1 | 0.9556 | 0.9741 | 0.9863 | 0.9892 | 0.9892 | 0.9906 |
| 2 actuators @ 1.2 and 1.6 kHz | 1 | 0.9951 | 1.0032 | 1.0107 | 1.0138 | 1.0124 | 1.0172 |

The data in Table 42 are presented graphically in Figure 72. A plot of the data in Table 44 illustrates an analogous pattern of variation. Figure 72 illustrates the inconsistent results obtained with Crude Oil 3 using sonication to further reduce the already low viscosity. In some cases, viscosity remained greater than the initial value after 30 days; in others the viscosity remained lower. Although viscosity changes were small, they were sustained during the month following treatment. This figure also shows that the small amount of viscosity recovery occurs primarily in the first seven days following the treatment and that viscosity remains fairly constant during the last three weeks of the recovery period. This observation is similar to that made during the experiments involving Oil 2.

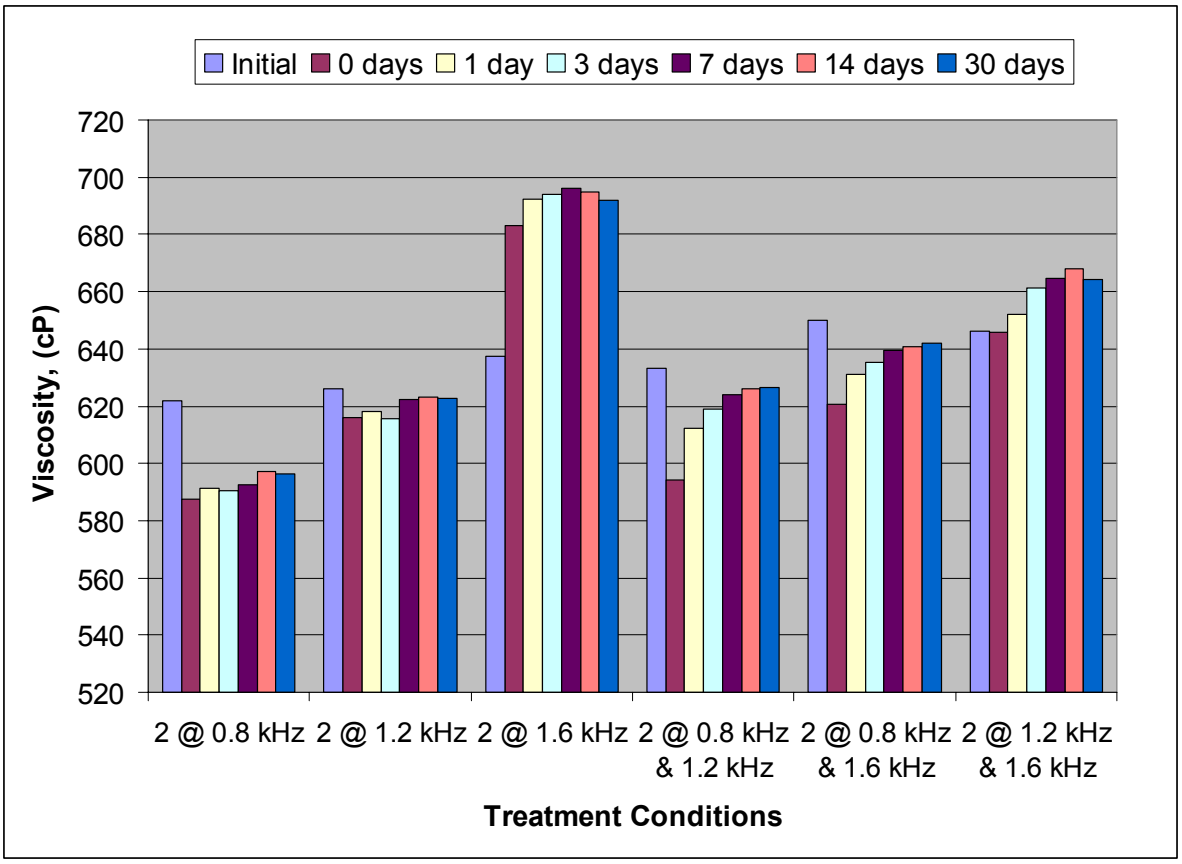


**Figure 72 Viscosity Recovery with Time Following Sonication with the Wide Fin Spacing for Crude Oil 3**

Finally, a third set of viscosity recovery measurements was made using the samples collected at the conclusion of the series of tests involving sonication and chemical additives. These data are given in Tables 46, 47, and 48. The first of these tables contains both the viscosity data following the addition of the chemical mix to Oil 3 and the initial, pre-additives viscosity. The columns labeled "Initial A+B" contain the viscosity data for Oil 3 following the addition of the chemicals (post-additives) but before that sample was treated with sonication. As with the previous tests of viscosity recovery, the viscosity at time 0 days is the value measured after the sample was exposed to sonication for 120 minutes and at the beginning of the recovery period. As noted previously, the chemical additives are very effective in reducing Oil 3 viscosity without sonication. When the oil with additives is exposed to sonication, the viscosity is reduced even further. In terms of recovery, the data in Table 46 show that the viscosity values change only by small amounts during the 30 days following treatment. This situation is further illustrated by examining the fractional residual viscosity values presented in Table 47. These normalized values are based on the post-additives viscosity (Initial A+B). The changes in viscosity during the recovery period are clear in these data. In all cases, the residual viscosity increased during the recovery period, and in one case, the final viscosity at the end of the 30-day rest period exceeded the initial viscosity value after adding the chemical mix and prior to

sonication. The remaining five values indicate that the final viscosity was approaching the pre-sonication, post-additives value. However, as one can determine from Table 46, the absolute value of viscosity change in all tests during the recovery period was very small.

**Table 46 Crude Oil 3 Viscosity Recovery with Time Following Treatment with Sonication Using Small Horn Spacing and Chemical Additives**

| Treatment Conditions | Viscosity, (cP) | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | Time Following Treatment, (days) | | | | | | | |
| | Initial | Initial A+B | 0 | 1 | 3 | 7 | 14 | 30 |
| 0.8 & 1.2 kHz, 3% A + 10% B | 627 | 359 | 238 | 248 | 256 | 263 | 266 | 268 |
| 0.8 & 1.2 kHz, 5% A + 10% B | 628 | 320 | 256 | 262 | 273 | 282 | 288 | 295 |
| 0.8 & 1.2 kHz, 7% A + 10% B | 626 | 232 | 182 | 206 | 213 | 221 | 224 | 224 |
| 0.8 & 1.6 kHz, 3% A + 10% B | 649 | 226 | 180 | 223 | 246 | 251 | 255 | 262 |
| 0.8 & 1.6 kHz, 5% A + 10% B | 648 | 174 | 98 | 146 | 153 | 161 | 164 | 168 |
| 0.8 & 1.6 kHz, 7% A + 10% B | 652 | 174 | 97 | 133 | 148 | 155 | 159 | 162 |

**Table 47 Recovery of Fractional Residual Viscosity of Oil 3 with Time Following Treatment with Sonication and Chemical Additives Using Post-Additives Viscosity as Initial Value**

| Treatment Conditions | Fractional Residual Viscosity, (cP) | | | | | | |
|---|---|---|---|---|---|---|---|
| | Time Following Treatment, (days) | | | | | | |
| | Initial A+B | 0 | 1 | 3 | 7 | 14 | 30 |
| 0.8 & 1.2 kHz, 3% A + 10% B | 1 | 0.6577 | 0.6912 | 0.7135 | 0.7330 | 0.7414 | 0.7469 |
| 0.8 & 1.2 kHz, 5% A + 10% B | 1 | 0.8010 | 0.8198 | 0.8554 | 0.8836 | 0.9018 | 0.9218 |
| 0.8 & 1.2 kHz, 7% A + 10% B | 1 | 0.7862 | 0.8897 | 0.9164 | 0.9526 | 0.9655 | 0.9655 |
| 0.8 & 1.6 kHz, 3% A + 10% B | 1 | 0.7968 | 0.9867 | 1.0866 | 1.1087 | 1.1263 | 1.1572 |
| 0.8 & 1.6 kHz, 5% A + 10% B | 1 | 0.5668 | 0.8422 | 0.8813 | 0.9274 | 0.9459 | 0.9700 |
| 0.8 & 1.6 kHz, 7% A + 10% B | 1 | 0.5586 | 0.7667 | 0.8506 | 0.8908 | 0.9149 | 0.9333 |

The relative significance of the viscosity recovery in Oil 3 under these test conditions can be judged from the data in Table 48 where the fractional residual (normalized) viscosity data are based on the viscosity of Oil 3 before any chemicals were added and before sonication. These data indicate that the residual viscosity is reduced to about 26% to 57% of the initial value by the addition of the chemical mixture. During the recovery period, the viscosity increased by a minimum of about 5.1% to a maximum of 12.6% of the pre-treatment viscosity. At the conclusion of the 30-day recovery period, the residual viscosity remained at 24.9% to 46.9% of the pre-treatment value. These results suggest that the combined treatment with additives and

sonication is effective in reducing viscosity even further in crude oils with an initially low viscosity. The most effective mixture of chemicals were 5% A and 7% A combined with 10% B.

**Table 48 Recovery of Fractional Residual Viscosity of Oil 3 with Time Following Treatment with Sonication and Chemical Additives Using Pre-Additives Viscosity as Initial Value**

| Treatment Conditions | Fractional Residual Viscosity | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | | | Time Following Treatment, (days) | | | | | |
| | Initial | Initial A+B | 0 | 1 | 3 | 7 | 14 | 30 |
| 0.8 & 1.2 kHz, 3% A + 10% B | 1 | 0.5722 | 0.3764 | 0.3955 | 0.4083 | 0.4195 | 0.4242 | 0.4274 |
| 0.8 & 1.2 kHz, 5% A + 10% B | 1 | 0.5089 | 0.4076 | 0.4172 | 0.435 | 0.4497 | 0.4589 | 0.4691 |
| 0.8 & 1.2 kHz, 7% A + 10% B | 1 | 0.3706 | 0.2914 | 0.3297 | 0.3396 | 0.3530 | 0.3578 | 0.3578 |
| 0.8 & 1.6 kHz, 3% A + 10% B | 1 | 0.3488 | 0.2780 | 0.3442 | 0.3790 | 0.3867 | 0.3929 | 0.4037 |
| 0.8 & 1.6 kHz, 5% A + 10% B | 1 | 0.2679 | 0.1519 | 0.2256 | 0.2361 | 0.2485 | 0.2534 | 0.2599 |
| 0.8 & 1.6 kHz, 7% A + 10% B | 1 | 0.2669 | 0.1491 | 0.2046 | 0.2270 | 0.2377 | 0.2442 | 0.2491 |

### 3.9.6 Summary of Crude Oil 3 Results

Tests were conducted to evaluate the effects of heat on the viscosity of Crude Oil 3. Test results demonstrated that the viscosity decreased in an exponential/power function relationship with increasing temperature. As was the case with all three crude oils tested, the viscosities during the cooling cycle were less than those at similar temperatures measured during the heating cycle.

Experiments to determine the results of sonication on viscosity were performed using two parallel actuators operating at six different combinations of acoustic frequencies. Initial experiments were conducted using horns with a two-inch (five-centimeter) fin spacing. Data obtained during these tests indicated that under some conditions, viscosity increased slightly upon initial exposure to sonication. However, subsequent exposure to sonication resulted in a decrease in viscosity to the extent that in only one case was the final viscosity after 120 minutes of testing greater than the initial viscosity. This was contrary to the results obtained with Oil 1, but this same phenomenon was observed with Oil 2. It was concluded that this situation resulted because of the appreciable and variable water content in the Oil 2 samples. However, this does not appear to be the situation with Oil 3, because water separation was not observed. The reason for this pattern of response in the least-viscous oil is not presently known. A maximum viscosity reduction of only about 6% was achieved with these test conditions. When the series of experiments was repeated using a one-inch (2.5-cm) horn spacing, the test results exhibited a similar pattern of change in viscosity following sonication with the maximum reduction again being approximately 6%. Thus the horn design had a minimal effect on the experimental results. The combination of frequencies of 0.8 kHz and 1.2 kHz appear to provide consistently better

viscosity reductions in Crude Oil 3 with the combination of 0.8 kHz and 1.6 kHz also providing good results. These observations agree generally with those obtained with the other two crude oils.

Another series of tests was performed to evaluate the effects of chemical additives along with sonication. Since the narrow horn spacing was used to test Oil 2, the same design was selected for testing Oil 3. The same chemical mixes were used as were used in the previous experiments. The addition of the chemicals reduced the viscosity by a minimum of 43% and a maximum of 73% prior to exposure to sonication. Subsequent exposure of the samples to sonication reduced viscosities even further; thus the combination of additives and sonication was more effective in reducing the viscosity of Oil 3 than either treatment used by itself. The mix of 3% additive A along with 10% additive B appears to be the most effective on this low-viscosity crude followed by 5% A and 7% A in combination with 10% B. However, these differences are very small.

Finally, a series of tests was conducted to evaluate viscosity recovery in samples allowed to sit undisturbed for 30 days following the individual tests. In all of the tests, viscosity increased during the recovery period. However, in all cases, the relative amounts of increase, compared to the pre-treatment values, were very small. When all recovery data involving Oil 3 are examined, the results obtained with the narrow horn spacing are nearly identical to those obtained with the wide horn spacing. The amount of viscosity increase during the month-long observations of these two suites of tests was always less than 10%. The changes in viscosity observed during the recovery period in samples treated with chemical additives and sonication were larger than those observed in the samples treated with sonication alone. However, after recovery, the viscosity values were only about 25% to 47% of the initial, pre-treatment values. In no case did the viscosity at the end of the month-long recovery period in any treated sample of Oil 3 return to a value approximating the pre-treatment value. Therefore, the experimental evidence indicates that although viscosity reductions resulting from sonication alone and from sonication in combination with the tested chemical additives in Oil 3 are small compared to the two more viscous crudes evaluated, the reductions in viscosity do occur and they persist for at least 30 days following treatment.

### 3.10 Results From Three Crude Oils Compared

The experimental results obtained with Crude Oils 1, 2, and 3 were described in the previous sections of this report. In Section 3.10, some of the results involving common tests of all three oils will be compared and evaluated. For this evaluation, the effects of acoustic frequencies, horn design, and chemical additives will be considered. Although the effects of input power levels along with the number and arrangement of actuators were also examined in this study, these conditions were not evaluated with all three crude oils. Consequently, the comparison of results in the following discussion is limited to the three treatment conditions that

were examined with all three oils. In each case, the two best results obtained for each crude oil, as reflected in the amount of viscosity reduction, are used in the comparison. Given this approach, it is possible that the same acoustic frequency conditions are not shown for all three oils for a given comparison because different frequencies may have produced the "best" results with different oils. In all instances discussed in this report section, the "best" results are deemed to be those where the smallest values of viscosity or fractional residual viscosity are observed at the conclusion (after 120 minutes) of testing.

### 3.10.1 Effects of Sonication

All three crude oils were exposed to a similar set of acoustic test conditions. It will be recalled that Crude Oil 1 was tested using a larger number of test conditions and equipment configurations, but the least effective options were eliminated from further testing, leaving the common conditions used in testing all three oils. It should be stated again that Crude Oil 1 was very viscous with the greatest viscosity of the three crude oils evaluated during this study. Oil 2 had an intermediate viscosity and contained a substantial amount of water that tended to separate from the oil during sonication. Oil 3 was a light crude with no apparent water content and a very low viscosity.

Table 49 contains the fractional residual viscosity values representing the two best results obtained from sonication alone for each of the three crude oils. These data are also presented graphically in Figure 73. One can see that the greatest reduction in viscosity as a result of

**Table 49 Comparison of Fractional Residual Viscosity Results Obtained During Sonication Testing of Crude Oils 1, 2, and 3**

| Treatment Conditions | Fractional Residual Viscosity | | | | |
|---|---|---|---|---|---|
| | Time, (min) | | | | |
| | 0 | 30 | 60 | 90 | 120 |
| 2 actuators @ 0.8 kHz & 1.2 kHz, Crude Oil 1 | 1 | 0.6417 | 0.5830 | 0.5800 | 0.5698 |
| 2 actuators @ 0.8 kHz, Crude Oil 1 | 1 | 0.5848 | 0.6134 | 0.6018 | 0.6131 |
| 2 actuators @ 0.8 kHz and 1.2 kHz, Crude Oil 2 | 1 | 0.8268 | 0.7562 | 0.7803 | 0.7682 |
| 2 actuators @ 0.8 kHz and 1.6 kHz, Crude Oil 2 | 1 | 1.0341 | 0.9467 | 0.9205 | 0.7761 |
| 2 actuators @ 0.8 kHz and 1.2 kHz, Crude Oil 3 | 1 | 1.1243 | 1.0712 | 1.0295 | 0.9386 |
| 2 actuators @ 0.8 kHz and 1.6 kHz, Crude Oil 3 | 1 | 1.1006 | 0.9915 | 0.9673 | 0.9406 |

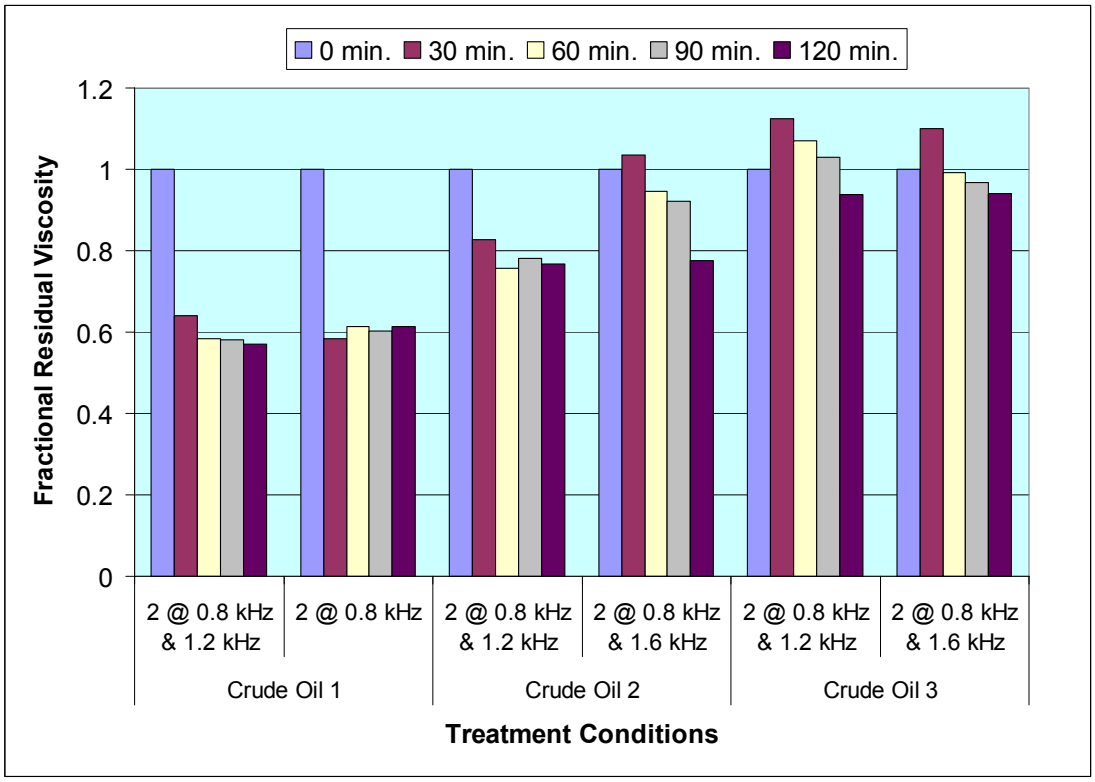


**Figure 73 Variation of Fractional Residual Viscosity in Crude Oils 1, 2, and 3 Resulting from Sonication Only**

sonication occurred in Oil 1, the most viscous oil of the three. The tests involving Oil 2 produced intermediate results, and the least reduction in viscosity, as evidenced by the largest values of fractional residual viscosity, occurred in Oil 3 which had a very low viscosity prior to testing. The viscosity of Oil 1 was reduced by approximately 42%, whereas that of Oil 3 was reduced by only about 6%. The viscosity of Oil 2 was reduced by approximately 23%. Operating two actuators in parallel at frequencies of 0.8 kHz and 1.2 kHz produced the greatest reduction in viscosity in all three oils; frequencies of 0.8 kHz and 1.6 kHz produced nearly the same results in two of the three oils. The trend of increasing fractional residual viscosity (reduced ability of sonication to decrease oil viscosity) as the initial, untreated oil viscosity decreases is well illustrated in Figure 73. In other words, the ability of sonication to reduce the viscosity of crude oil is inversely related to the initial viscosity. Sonication is more effective in reducing the viscosity of heavy, viscous crudes than lighter, less-viscous crudes.

### 3.10.2 Effects of Horn Design

The second independent variable that can be evaluated across all three crude oils is the effect of horn design. It will be recalled that two different horn designs were evaluated during the study: one involved a fin spacing of one inch (2.5 cm) (narrow fin spacing) and the second

utilized a spacing of two inches (5 cm) (wide fin spacing). Each of the three crude oils was tested with both horns while maintaining the same acoustic output from the actuators.

Table 50 contains the values of fractional residual viscosity for each of the three crude oils measured at the conclusion (120 min.) of testing under the specified treatment conditions. As discussed in the previous report section, the two "best" results from each crude oil are used for comparison. These data are presented graphically in Figure 74. In general, these data indicate that the acoustic horn design based on the narrow (1 in, 2.5 cm) spacing is more effective in reducing the viscosity of all three oils than is the wide (2 in, 5 cm) spacing, although there is essentially no difference in the results for the low-viscosity Oil 3. Clearly, the largest difference in residual viscosity due to changing horn design was in Oil 2. It will be recalled that this oil had a significant water content which may have influenced this response; however, this is only speculation at this time. There is no other obvious explanation for such a dramatic difference in the results from this oil compared to those obtained with Oils 1 and 3.

**Table 50 Fractional Residual Viscosity Values at the Conclusion of Testing the Three Crude Oils with Narrow Spacing and Wide Spacing Acoustic Horn Fins**

| Treatment Conditions | Fractional Residual Viscosity | |
|---|---|---|
| | Narrow Fins | Wide Fins |
| 2 actuators @ 0.8 kHz & 1.2 kHz, Crude Oil 1 | 0.5698 | 0.6612 |
| 2 actuators @ 0.8 kHz & 1.6 kHz, Crude Oil 1 | 0.6722 | 0.6892 |
| 2 actuators @ 0.8 kHz & 1.2 kHz, Crude Oil 2 | 0.2966 | 0.7682 |
| 2 actuators @ 0.8 kHz & 1.6 kHz, Crude Oil 2 | 0.2418 | 0.7761 |
| 2 actuators @ 0.8 kHz & 1.2 kHz, Crude Oil 3 | 0.9389 | 0.9386 |
| 2 actuators @ 0.8 kHz & 1.6 kHz, Crude Oil 3 | 0.9556 | 0.9406 |

The data for the wide fin spacing show a progressive decrease in the amount of viscosity reduction with decreasing viscosity of the untreated crude oils (recall that Oil 1 is highly viscous, Oil 2 has intermediate viscosity, and Oil 3 has a low viscosity). This trend is obviously related to the identical relationship in the sonication treatment data discussed previously (Table 49 and Figure 73). A similar trend in the narrow fin horn design data might also exist were it not for the large disparity in the results for Oil 2. In addition, one can see that the differences between the results of the two horn designs are larger in Oils 1 and 2 that in Oil 3. This suggests that the narrow fin design is more likely to be a better choice with heavier, more-viscous crudes than with lighter, less-viscous crude oils.

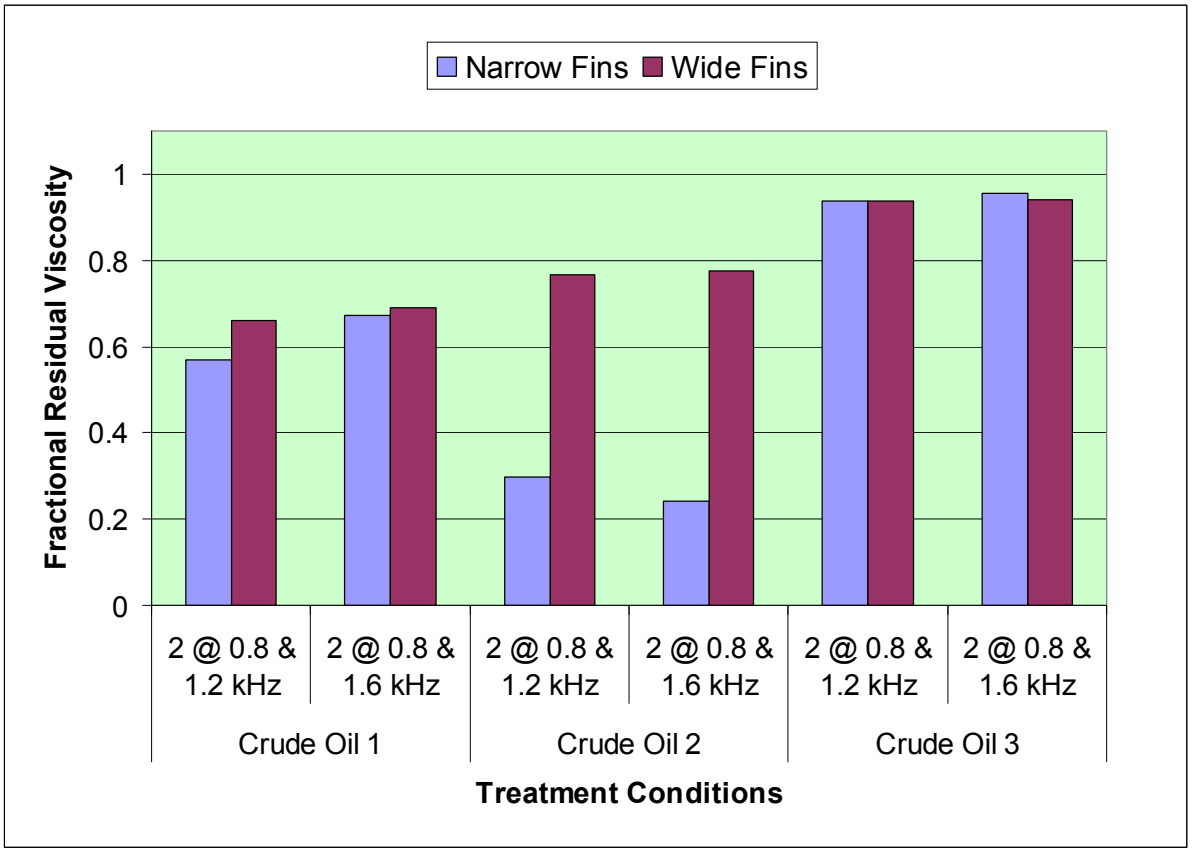


**Figure 74 Fractional Residual Viscosity Data for Oils 1, 2, and 3 Illustrating the Effects of Acoustic Horn Design**

### 3.10.3 Effects of Chemical Additives

The third set of treatment conditions to be compared across all three crude oils is the addition and concentrations of chemical additives used in conjunction with sonication. As discussed previously, the concentration of chemical A was varied from 3% (by volume) to 5%, and to 7% while the concentration of chemical B was maintained at 10% in each experiment. The two best results obtained with chemical additives and sonication for each of the three crude oils are given in Table 51 and Figure 75.

Based on these data, it is clear that the combined effects of sonication and chemical additives are much more effective in reducing the viscosity of Oil 1 than either Oil 2 or 3. One can also see that the results for Oils 2 and 3 are very similar both in the value of fractional residual viscosity at the conclusion of the tests, and the patterns of variation with time. The acoustic conditions/frequencies that produced the best results in conjunction with the chemicals in Oil 1 are different from those associated with the best results in Oils 2 and 3, but the best

**Table 51 Fractional Residual Viscosity Data for Crude Oils 1, 2, and 3 Treated with Chemical Additives and Sonication**

| Treatment Conditions | Fractional Residual Viscosity | | | | |
|---|---|---|---|---|---|
| | Time, (min) | | | | |
| | 0 | 30 | 60 | 90 | 120 |
| 2 actuators @ 0.8 kHz, 3% A + 10% B, Crude Oil 1 | 1 | 0.970 | 0.480 | 0.410 | 0.320 |
| 2 actuators @ 1.2 kHz, 5% A + 10% B, Crude Oil 1 | 1 | 0.330 | 0.390 | 0.399 | 0.320 |
| 2 actuators @ 0.8 kHz and 1.2 kHz, 3% A + 10% B, Crude Oil 2 | 1 | 0.851 | 0.790 | 0.697 | 0.565 |
| 2 actuators @ 0.8 kHz and 1.6 kHz, 3% A + 10% B, Crude Oil 2 | 1 | 0.863 | 0.795 | 0.707 | 0.576 |
| 2 actuators @ 0.8 kHz and 1.6 kHz, 5% A + 10% B, Crude Oil 3 | 1 | 0.799 | 0.682 | 0.673 | 0.567 |
| 2 actuators @ 0.8 kHz and 1.6 kHz, 7% A + 10% B, Crude Oil 3 | 1 | 0.968 | 0.903 | 0.682 | 0.559 |

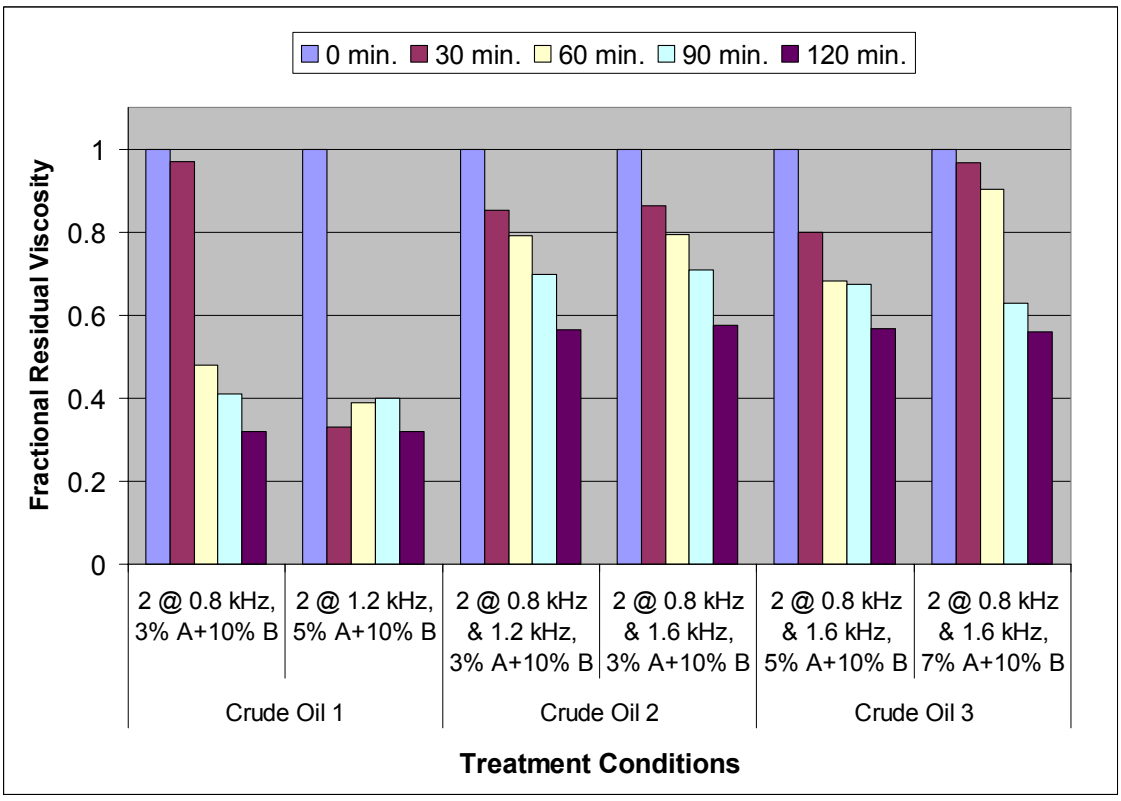


**Figure 75 Variation of Fractional Residual Viscosity of the Three Crude Oils with Changing Mixtures of Chemical Additives and Sonication Conditions**

frequencies for these latter two test oils are similar. Finally, it is apparent that the pattern of variation within the results obtained with both sonication and chemical additives (Figure 75) are similar to those obtained with sonication alone (Figure 73); however, the amount of viscosity reduction is greater (lower values of fractional residual viscosity) in all six tests when chemical additives are used than in the tests involving only sonication.

### 3.10.4 Summary of Comparisons of Three Crude Oil Results

The data and observations presented in this report section show that the viscosity of all three crude oils having viscosities ranging over two orders of magnitude (approximately 60,000 cP to 700 cP) can be reduced when exposed to sonication either in the presence or absence of chemical additives. The amount of viscosity reduction is greater when the chemicals are added than when sonication is used alone. The reductions in viscosity attained by the acoustic and chemical treatments are inversely related to the initial, pre-treatment viscosity of the crude oils. The greatest reduction in viscosity occurred in Oil 1 which was the most viscous of the three, whereas the minimum viscosity effects were observed with the Crude Oil 3 having the lowest initial viscosity. A very similar pattern of variation was observed in the results obtained with sonication only and with sonication plus chemical additives.

The results involving the acoustic horn design are inconclusive. In most tests, the smaller fin spacing design was more effective in reducing crude oil viscosity than was the wider spacing. However, there was essentially no difference in the results involving Oil 3. The pattern of variation in results involving the wide spacing is progressive in that the greatest effects were observed in Oil 1, intermediate results with Oil 2, and the least effects in Oil 3. A dramatic reduction in post-treatment viscosity in Oil 2 occurred with the narrow spacing. It is reasoned that this may be due to the larger water content of Oil 2 as compared to the other two oils. However, it is unclear why this condition would cause the narrow spacing to be so much more effective in separating the water and reducing the oil viscosity than when the wide horn design was employed.

# 4 PROCESS ECONOMICS, MARKET POTENTIAL, AND SCALE-UP FACTORS

## 4.1 Process Economics

Process economic data that can be developed at this time must be viewed as preliminary and subject to change as more tests are completed and additional data are obtained. Because this project is a "first of a kind", all economic data must be considered as estimates or projections. The project was a laboratory-based study, but the design used in the laboratory, a three-acutator "cross" design, is unsuitable for downhole use. However, for above-ground applications, such as moving heavy crude between the well site and a transportation vehicle (ship, rail tank car, etc.) and between the vehicle and crude user/processor facility (e.g. refinery), the design, with some modifications, may be acceptable. The modifications could entail enlarging the equipment, designing a series of reaction chambers to work in sequence, changing the cross design to a single-plane design where the number and spacing of actuators could be increased to address more power and reaction-time requirements, and similar engineering considerations. The cost of such a system would be dependent upon the size and complexity of the system along with the number of components, but it is estimated that an initial cost would be approximately $40,000 for a two-actuator system. Downhole systems would likely cost approximately $25,000 for a single actuator system. Multiple actuator systems for downhole use are possible, but further development of this concept is necessary.

## 4.2 Market Potential

### 4.2.1 Applications

Treating crude oil with acoustic energy may have a number of applications. These potential uses range from in-situ to surface applications.

1. **Surface crude oil viscosity reduction.** Reducing the viscosity of heavy crude oil will add value by increasing the API gravity (adds value differential) and by making the crude easier to pump (eliminate the cost of heating the crude while transporting through pipelines or on tankers). Surface treatment of crude may be enhanced by using additives such as the following:
    - Solvents
    - Chemicals/catalysts
    - Surfactants
    - Hydrogen

    The proprietary chemicals used in this study enhanced the reduction in viscosity of the three crude oils used in the investigation with the largest effects being evident in the most viscous oil.

2. **Subsurface crude oil viscosity reduction.** Similar to the benefits of treating crude oil on the surface, treating crude oil downhole provides the added benefit of increasing recovery factors (reserves) and minimizing surface treatment problems and associated costs. Subsurface treatment may also be enhanced by using additives such as the following:
    - Solvents
    - Chemicals/catalysts
    - Surfactants

    TechSavants, Inc. and its collaborators have demonstrated the increase in production using downhole acoustic energy (Johnson and others, 2004; Paulsen and others, 2005b). The chemicals used in this study increased appreciably the levels of viscosity reduction attained by sonication.

3. **Subsurface micro fracture propagation.** Treating low permeability reservoir rock with acoustic energy at specific frequencies and power levels may cause the rock matrix to fail locally, resulting in the formation of micro fractures (secondary permeability). Successful development of this technology would facilitate oil production from tight reservoirs currently not productive. Work in this area requires further research. This area of investigation was not part of this SBIR project.

4. **Sulfur removal from crude oil.** Sour crude production makes up 60% of the daily global crude oil production and 80% of the economically recoverable global oil reserves (Harvey, 2005). As the demand for crude oil increases, more sour crude will enter the market. These sour crudes contain sulfur contents greater than 0.5% by weight and may contain as much as 5% or 6% sulfur (Petroleumiran, 2005). There is limited global refining capacity to refine these high sulfur crude oils and environmental constraints on sulfur emissions continue to be tightened. A technology that can cost effectively reduce sulfur at the point of production would reduce the refining capacity problem and would add value to the raw product. The price spread between comparable sour and sweet API crudes is approximately $3.00 (Cooke, 2004). This spread will increase as more sour crude enters the market. Theoretically, treatment with acoustic energy applied at specific frequencies and power levels should be able to break molecular bonds within sour crudes, thereby facilitating sulfur removal. However, research on this topic was not part of this SBIR investigation.

5. **Well bore cleaning.** As producing wells age, the completions and the "near wellbore" reservoir plugs with fines, paraffin, asphaltenes, sand, iron silicate, and other substances. Acoustic energy applied at specific frequencies and power levels will remove or mobilize deposits in the completion interval. Cleaning the completion interval (screens, perforations, casing, etc.) will facilitate increased fluid flow that should increase oil/gas production. TechSavants, Inc. and Furness-Newburge, Inc.

recently participated in a study examining acoustic energy as a remediation tool in natural gas storage (injection/withdrawal) wells (Paulsen and others, 2005a, 2005b).

### 4.2.2 Market Projections

Since the applications mentioned above are either pre-commercial or conceptual in nature, market projections are speculative at the present time. However, a preliminary analysis of market size and value has been performed to provide an "order of magnitude" estimate. Table 52 below summarizes estimates based on conditions that exist today and assuming a reasonable acceptance of the technology by the petroleum industry.

**Table 52 Estimated Market Size and Value for Selected Petroleum Industry Applications**

| Application | Estimated Annual Volume, (MMBO) | Estimated Annual Revenue, ($ MM) |
|---|---|---|
| **Viscosity Reduction** | | |
| Surface | >1,000 | >$6,000 |
| Subsurface | >250 | >$1,500 |
| **Fracture Propagation** | | |
| Subsurface | >1,250 | >$12,500 |
| **Sulfur Removal** | | |
| Surface | >14,000 | >$6,300 |
| **Wellbore Cleaning** | | |
| Subsurface | >40 | >$400 |
| **Note:** MMBO is Millions of Barrels of Oil and $ MM is Millions of Dollars | | |

### 4.2.3 Assumptions

The following assumptions form the basis for the calculations used to obtain the data presented in Table 52. In all examples, the assumptions were developed to provide very conservative market potential values. Given the recent rise in the price of crude oil to more than $60/bbl associated with supply constraints, one could argue that the pricing and market penetration assumptions could be significantly increased compared to those used in this document. However, the authors believe that the approach employed is much more realistic and defensible. Because of the historical cyclic nature of petroleum supply-demand and pricing variations, being conservative in future projections and estimates seems prudent. This is also a major factor in the reasoning for using a price margin (sale price minus all costs/expenses) of

$10/bbl for estimating potential revenues. This represents a sound, conservative, and easily defended value.

Surface Viscosity Reduction Assumptions

- Heavy oil/Bitumen = API gravity is lower than 20 degrees and viscosity is greater than 1000 cP (Herron, 2000).
- Daily global crude oil production = 65-70 million BOPD (barrels of oil per day).
- Daily production of heavy oil/bitumen = 2.8 million BOPD (~ 1 billion bbls/yr) which is a very conservative estimate.
- Viscosity reduction equal to WTI (West Texas Intermediate benchmark standard for oil pricing). Current price spread between WTI and Kern River crude (API 13) is approximately $14/bbl. Conservatively use the historic spread = $6.00/bbl in these estimates.
- A majority of the value created by reducing crude oil viscosity is associated with realizing the API gravity value differential as compared to WTI. Therefore, only the API gravity value differential is used in the analysis and the value associated with making the crude easier to transport is not included (conservative approach).

Subsurface Viscosity Reduction

- Same assumptions used to calculate the value for "Surface Viscosity Reduction" above.
- Total world oil reserves = 1.5 trillion barrels (Cooke, 2004).
- Reserves applicable for this technology (assumed 10% of total) = 150 billion barrels.
- Increase recovery by 5% (conservative estimate) of the 150 billion barrels applicable to this technology over 30 years (7.5 billion bbls/30 years = 250 MMBO/yr).
- Use historic spread between heavy crude and WTI = $6.00/bbl ($1,500 MM/yr)

Fracture Propagation

- 10% of the world's oil reserves are in low permeability rock.
- Total world oil reserves = 1.5 trillion barrels (Cooke, 2004).
- Reserves applicable for this technology assumed to be 10% of total = 150 billion barrels.
- Recover 25% of the 150 billion barrels applicable to this technology over 30 years (37.5 billion bbls/30years = 1.25 billion bbls/yr).
- Average oil price margin = $10/bbl ($12.5 billion/yr).

Sulfur Removal from Crude Oil

- 60% of world production is classified as "sour" crude (Harvey, 2005).

- Daily global production = 65-70 million BOPD.
- Daily global sour production = 39-42 million BOPD (14-15 billion bbls/yr)
- Average price uplift for sulfur removal = $1.50/barrel (use 30% of $1.50 in estimate = $0.45/bbl).
- Revenue range of $6.3 billion/yr to $6.75 billion/yr. Use the smaller value as estimate.

Production Well Bore Cleaning

- Number of crude oil production wells in the U.S. = 500,000 (Energy Information Agency, 2003).
- Assume 10% of the U.S. wells could use his technology = 50,000 wells.
- Average production per U.S. well = 11 BOPD (Energy Information Agency, 2003).
- Assume 20% average production gain per well = 2.2 BOPD per well (110,000 BOPD = 39.6 MMBO/yr increased production).
- Average oil price margin = $10/bbl ($396 million/yr additional revenue).
- Estimated annual revenue is from incremental oil production only and does not include service company fees.

### 4.2.4 Geographical Use

The application of treating crude oil with acoustic energy can be used globally. This technology is simple in nature, relative small in size, portable, and only requires electric power to energize the system. It can be used in either a refinery or in an oil field setting. Once the applications mentioned in the market potential section are matured to a point for commercialization, deployment should be rapid due to the relatively low cost of the technology.

### 4.2.5 Potential Clients

Due to the projected low cost of the technology, both small and large oil companies and refineries, could afford to adopt the technology into their daily operating strategies. For several of the potential applications, service companies can be equipped with the technology for deployment in the field.

## 4.3 Scale-Up Factors

Because the technology is small, scale-up factors are not a major issue. In fact, a real issue was scaling down the technology to fit downhole without a significant loss in acoustic energy power. Furness-Newburge, Inc. and TechSavants, Inc. have designed and developed a downhole system to meet variable well constraints. During two previous projects, this system was deployed downhole six times, three times to stimulate oil production and three times to clean natural gas storage well perforations and casing. Oil production was increased a minimum of 15% within each well and the gas storage well “skin” improved from -2.5 to +1.3. However,

other applications will require scale-up engineering to accomplish necessary performance levels. Future scale-up will examine methods to increase the power output, expand the broadband capabilities of the actuators, and increase the efficiency of the horns. In addition, a multi-acutator system may be designed and developed for certain applications.

# 5 COMMERCIALIZATION PLAN

## 5.1 Background

TechSavants, Inc. is preparing an initial draft commercialization plan focusing initially on the downhole stimulation of marginal wells (stripper wells) for several reasons. These are:

1. The technology as currently developed is limited to operating to depths of less than approximately 7000 feet (2134 m) and 2000 pounds/in$^2$ (psi) (141 kg/cm$^2$) of pressure. Most marginal wells fall within these limitations.

2. The technology has been demonstrated in three downhole projects, two conducted with government funding and one with private sector support. In each case, the data indicated that production increased a minimum of 15%, although more precise measurements might have shown in increase of 20-25% (values suggested in some of the data).

3. TechSavants, Inc. is currently in discussions with three oil-field operators regarding future downhole stimulation projects. Most of the likely projects involve deploying the acoustic unit down two to four wells to validate the technology. Successful tests will lead to additional work in these three fields and the likelihood of similar work in nearby well fields.

4. TechSavants believes it has a niche market where it has few competitors. Most technology in acoustic or seismic stimulation is targeting deeper and more productive wells.

## 5.2 Strategy

Depending on the application, the technology could be commercialized following two different approaches: process enhancement or service company deployment. For the applications focused on "*surface crude oil viscosity reduction"* and "*sulfur removal from crude oil*" as discussed in Section 4.2, the technology could be sold as a process enhancement. These systems could be integrated into either a refinery or into oil field processing facilities for field treatment. The technology systems could be either leased to individual operators or the technology could be licensed to individual companies for their use within their operations and infrastructure. These options would most likely be accomplished through strategic alliances or partnering arrangements with firms within the petroleum industry.

As discussed previously (Section 4.2), other potential applications focus on "*subsurface crude oil viscosity reduction*" and "*subsurface micro-fracture propagation*". These two applications could be sold as either process enhancement or delivered to an end user through a service company. If sold as a process enhancement, the customer would integrate the technology

into the daily operating strategy of the oil field operations. This approach could involve an outright sale of the technology, a licensing arrangement, or an equipment lease. A service company approach would provide the customer with periodic treatments for a fee. As with the process enhancement option, the technology could be commercialized by selling it to a service company, licensing the technology to a service company, and/or leasing the equipment. The deployment specifics would depend on actual field performance (treatment time vs. benefit) and the cost-effectiveness of the treatment. The final application area, "*well bore cleaning*" would most likely be deployed via a service company, although the technology could be licensed or bought by other types of companies for subsequent integration within well service operations. Well cleaning does not require continuous treatment and therefore would not be permanently installed in individual wells. However, this is an option if subsequent tests demonstrate an obvious benefit to this approach. For reasons cited above in the previous section. TechSavants believes the initial commercialization efforts should be focused on the stripper well segment of the petroleum industry.

TechSavants' goal is to dominate the marginal oil well market by 2009 using strategic alliances in regional-to-national settings. Marginal oil well production provided 15% of total U.S. domestic crude oil production in 2003, averaging 860,000 BOPD (IOGCC, 2004). This percentage increases to about 25% if only onshore production from the lower 48 states is considered. An increase in production of 20% resulting from sonication stimulation would provide 175,000 additional barrels per day from marginal wells. At $30/barrel marginal oil price, this would result in daily revenues of $5.25 million. Assuming a 10% market penetration, this production increase would provide more than $500,000 in revenue per day from marginal wells.

The conceptual commercialization plan short-term activity (2006-2007) is to deploy the technology in as many downhole applications as is logistically feasible, documenting the success of increased production. TechSavants will act as the technology operator on a leased-unit cost basis. Strategic alliances may form where others will be trained in the operation of the systems. By 2008, the maturity and value of the technology will position TechSavants as a target to be acquired or for partnership with a major field service company.

# 6 CONCLUSIONS AND RECOMMENDATIONS

## 6.1 Conclusions

### 6.1.1 General Conclusions

The primary conclusions drawn from the project activities are as follow:

1. The initial phase of this investigation demonstrated that exposure of single-weight oils to sonication would reduce their viscosity. These findings supported the hypothesis that sonication could be used as an alternative method for reducing the viscosity of heavy crude oils and provided the basis for the larger, second phase of he project.

2. The application of acoustic energy (sonication) has been demonstrated to significantly reduce the viscosity of crude oils under laboratory conditions.

3. The amount of viscosity reduction due to sonication is greater for more viscous (greater initial viscosity) heavy crude oils than it is for less viscous light crude oils.

4. The viscosity reduction is further enhanced by the addition of two proprietary chemical mixtures studied in the experiments. The amount of viscosity reduction due to the chemical additives was inversely related to initial crude oil viscosity – greater viscosity reduction was obtained with a more viscous, heavy crude and lesser reductions in less viscous crudes.

5. Although the viscosity tends to recover with time following sonication treatment, in no case did the viscosity return to more than about 50% of the pre-treatment value during a period of 30 days following treatment. Therefore, in most cases, more than half of the initial viscosity reduction was maintained for a month without additional treatment.

6. As expected, heating tests demonstrated the effectiveness of this method of viscosity reduction. As the crude oils were allowed to cool, the viscosity returned to nearly the same values that were measured during the heating cycle except that the "cooling" values were somewhat less than the "heating" values at the same temperature. The reductions in viscosity were not sustained following heat treatment to the extent that the post-sonication reductions were sustained.

7. The best results obtained with the flow-through test apparatus were with two actuators operating at different frequencies, aligned in parallel and adding energy to the reaction vessel from opposite sides. This arrangement should be considered in subsequent engineering design evaluations for larger-scale systems.

8. Two acoustic horns were evaluated. One horn design using a narrow fin spacing (1 in, 2.5 cm) produced somewhat better results (improved viscosity reductions) than did the design based on a fin spacing of 2 in (5 cm). However, in most tests, the differences were not great.

9. It was observed that reducing the input power by 25% had very little effect on the ability of sonication to alter crude oil viscosity. Therefore, if input power were an issue (either availability or cost), it is probable that a lower input could be utilized without sacrificing the treatment efficacy.

10. Although a significant amount of additional work is needed, the project results indicate that sonication technology could serve several roles within the oil and gas industry. Conservative estimates indicate that if all of these prospective applications were fully developed, optimized, and implemented, several billion barrels of oil potentially could be upgraded or produced annually generating between $400 million and potentially more than $20 billion in estimated annual revenue.

11. With time, the oil being produced by the marginal or stripper well industry is becoming, on average, more viscous and difficult to produce because in many fields the lighter crudes have already been or are rapidly being withdrawn. In addition, newer, less-expensive methods are needed to clean the perforations and completion zones in these wells to sustain/enhance production rates. In 2003, marginal oil accounted for about 28% of production from onshore wells in the lower-48 states and about 15% of total domestic production (IOGCC, 2004; Stripper Well Consortium, 2005). The successful integration of sonication technology into this industry sector would quickly yield appreciable economic benefits.

### 6.1.2 Specific Conclusions

A number of more specific conclusions were drawn from the major tasks of the project. The first phase of the project involved experiments with three commercially available single-weight oils (30-weight, 90-weight, and 120-weight) that were conduced under "batch" treatment conditions. The second phase of the project utilized a flow-through experimental system using three different crude oils with very different viscosities. Crude Oil 1 was a heavy, highly viscous crude from California with an initial viscosity of approximately 65,000 cP. The second crude oil that was evaluated was produced in Alabama and had a viscosity of approximately 6,000 cP as well as a significant water content. The third crude oil included in the experiments was a very light crude produced in Middle East having an initial viscosity of only about 700 cP.

Conclusions drawn from the Phase I of the project include those listed below.

- The reduction in viscosity observed in the experiments was due to both sonication effects and the dissipation of heat into the oil. The reduction due to heat only was defined by regression analysis allowing the effects of the two energy sources (heat and acoustic energy) to be segregated.

- The sonication frequencies examined were 1.8, 3.1, 6.9, and 13.1 kHz. Generally, the lower the acoustic frequency, the greater the efficiency in reducing the oil viscosity.

- Three horn fin spacings were evaluated: small (0.25 in, 6.4 mm), medium (0.75 in, 19.1 mm), and large (1.25 in, 31.8 mm). In general, the horn design with medium spacing provided greater viscosity reductions than either the small or large spacing.

- The results obtained with the low-viscosity, 30-weight oil were subject to greater potential error in viscosity measurements than the data obtained with the other two, more viscous oils due to the method of viscosity measurement in Phase I. Consequently, there is less confidence in the results obtained from the 30-weight oil.

- For the case of the 90-weight oil, the lowest acoustic frequencies (1.8 and 3.1 kHz) resulted in the greatest reduction in viscosities as compared to the higher frequencies. Typical viscosity reductions were in the range of 46% to 67% for these two frequencies.

- At the lower frequencies, heat from the sonication process using the 90-weight oil appeared to be the primary effect responsible for viscosity reduction as compared to the effects of sonication alone.

- For the case of the 140-weight oil, the tests using lower acoustic frequencies (1.8 and 3.1 kHz indicate that sonication has more of an effect in causing viscosity reduction than does heat dissipation. Overall viscosity reductions at lower frequencies ranged from approximately 45% to 55%.

- Tests of higher frequencies involving the 140-weight oil suggest that heat input was more the cause of viscosity reduction than was sonication when the small horn spacing was employed; however, the reverse appeared to be the case with the medium and large horn spacings.

- After sonication treatment, the viscosity of oil samples allowed to equilibrate to room temperature returned to approximately the pre-treatment condition.

- Sonication treatment of the three oils resulted in a reduction in viscosity that ranged from a low of 31.2% to a high of 75.4%. The viscosity reductions measured for each

of the test oils were: 31.2% – 53.7% for the 30-weight oil, 40.3% – 75.4% for the 90-weight oil, and 25.8% – 54.3% for the 140-weight oil.

In Phase II of the project, a number of important observations and conclusions were obtained from the laboratory experiments with the three crude oils described above. They are summarized in the following list.

- All three crude oils were heated to temperatures exceeding 80°C and incremental measurements of viscosity were collected during both heating and cooling cycles. The most rapid decrease in viscosity as heat was applied occurred in the range of 20°C to 40°C, and the maximum viscosity reduction exceeded 90% in all three oils. In all tests, the viscosity during the cooling cycle was less than that during the heating cycle at the same temperatures.

Conclusions involving Crude Oil 1 follow.

- Sonication reduced the viscosity of Crude Oil 1 by a minimum of 6.3% and a maximum of 43.0%. Two parallel actuators operating at 0.8 kHz or one at 0.8 kHz and the second operating at 1.2 kHz provided the greatest viscosity reductions.

- The maximum reduction of 43% noted above occurred after 120 minutes. However, after only 30 minutes of sonication, the viscosity was reduced by 35.8%. Thus, about 80% of the total viscosity reduction occurred in the first 30 minutes. Similar trends were observed during other experiments.

- The results of altering the horn design (narrow fin spacing of 1 in/2.5 cm vs. wide fin spacing of 2 in/5 cm) in the Crude Oil 1 experiments indicated that this variable had only minimal effects on the observed viscosity results. Although the smaller spacing provided somewhat greater viscosity reductions than the larger one with the same frequencies, the average difference for all tests was only 6.5%.

- Experiments were conducted with Crude Oil 1 to evaluate the effects of reducing input electrical/acoustic power by 25%. The results were somewhat inconclusive in that some tests showed an increased viscosity reduction with reduced power and other results indicated the opposite. The average difference for these two series of tests was about 14% suggesting that, if necessary, power could be reduced by up to 25% without significantly reducing sonication effectiveness.

- Tests involving both power level and horn design suggest that the effects of input power level on viscosity reduction are somewhat greater than those due to changing the fin spacing of the acoustic horns.

- A suite of experiments examining the effects of two proprietary chemical additives (A and B) used alone and in conjunction with sonication. Volume concentrations of 3%, 5%, and 7% A in combination with 10% B in each case were evaluated for all three crude oils. These additives alone in these concentrations reduced the viscosity of Crude Oil 1 by approximately 37%, 71%, and 45%, respectively. When sonication was added to each treatment, maximum viscosity reduction ranged from 45% to 89%. Viscosity was reduced more than 80% in six of ten tests, and the average reduction for all tests with chemical additives and sonication was 74%.

- The best results using both chemical additives and sonication were obtained with lesser concentrations of chemical additives and sonication frequencies in the range of 0.8 kHz to 1.2 kHz.

Conclusions involving Crude Oil 2 follow.

- Crude Oil 2 had a significant water content that was not consistent among the various large containers of oil gathered for this project. In some tests of this oil examining the effects of sonication, viscosity was observed to increase with time. In most of these experiments, oil samples showed a distinct separation of water from the oil after sonication, thereby increasing the viscosity of the oil. This suggests that sonication could have potential as an alternative oil-water separation methodology.

- A maximum reduction in viscosity of 23% was measured in the sonication tests using the wide horn fin spacing. Using the narrow spacing, the maximum reduction reached almost 76%, indicating that this narrow horn design was much more effective at reducing the viscosity of Crude Oil 2.

- The two best acoustic frequency combinations employed with the two parallel actuators were 1) 0.8 kHz and 1.2 kHz, and 2) 0.8 kHz and 1.6 kHz. These observations agree with those obtained with Oil 1.

- Tests involving chemical additives with composition and concentrations the same as those used with Oil 1 indicated that the additives alone reduced the viscosity of Oil 2 by more than 90%. It appears that the effectiveness of the chemicals was enhanced by elevated water content within the oil. Sonication of these samples reduced the viscosity even further.

- The mix of 7% additive A plus 10% B appeared to be the most effective followed by 5% A and 3% A both in combination with 10% A. However, these differences are vary small.

- A series of tests was conducted to evaluate viscosity recovery in samples of Oil 2 allowed to sit undisturbed for 30 days following the individual tests. In all samples, viscosity tended to increase during this period of time, but the increases were relatively small when compared to the pre-treatment condition. In most cases, the majority of the recovery occurred in the first seven days or earlier following treatment.

- The viscosity recovery data from Oil 2 indicate that the samples exposed to sonication with the narrow horn spacing sustained those reductions much more effectively than the samples sonicated using the wide horn spacing. At the end of the rest period, viscosity values of samples exposed to sonication using the narrow horn design ranged from about 34% to 54% of the initial, pre-treatment values with an average value of 40%. In other words, in the worst case, the viscosity reduction at the end of the recovery period remained at about one-half of the pre-treatment value. On average, the viscosity reduction from sonication at the end of 30 days was 60%.

- The observed viscosity recovery in the samples containing chemical additives and receiving sonication treatment also increased slightly during the 30 days following treatment, but the change was very small. In the worst case, the viscosity remained at about 7.5% of the initial, pre-treatment value; thus, a reduction in viscosity in excess of 93% was observed after a month-long recovery period.

Conclusions involving Crude Oil 3 follow.

- Although test conducted by sonicating Crude Oil 3, a very light crude, indicated a general trend of reduction in viscosity with increasing treatment time, the results are highly variable and less well defined as in Oils 2 and 3. In some cases, viscosity increases somewhat initially during the tests. The reason for this is unclear.

- The maximum reduction in the viscosity Crude Oil 3 was only about 6% using both the narrow-fin and wide-fin horn designs. Therefore, horn design had only a minimal effect on the ability of sonication to reduce the viscosity of this low-viscosity oil.

- Combined frequencies of 0.8 kHz and 1.2 kHz provided better viscosity reductions in Oil 3 with the combination of 0.8 kHz and 1.6 kHz also producing enhanced results. These observations are in general agreement with those involving the other two crude oils.

- The addition of the proprietary chemicals to Crude Oil 3 reduced the viscosity by a minimum of 43% and a maximum of 73% prior to sonication. When the oil and additives are sonicated, the viscosity is further reduced by more than 50% based on the post-additives, pre-sonication values.

- The mix of 3% A and 10% B appears to be most effective in reducing the viscosity of Oil 3, although the differences in results obtained with the three mixes are small.

- Viscosity recovery in Oil 3 was evaluated during a month-long recovery period. The data indicate essentially no difference in the results obtained with the small fin spacing and the wide fin spacing horn designs. In both cases, the recovery was less than 10% during the 30 days.

- Increases in viscosity during the recovery period were greater in those samples treated with both chemical additives and sonication than in samples treated by sonication alone. However, the final viscosity values of samples receiving the chemicals were only about 25% to 47% of the pre-treatment values at the end of 30 days.

- The reductions in viscosity of Oil 3 are small compared to those observed in the more-viscous Crude Oils 1 and 2. However, even these small reductions in the light crude are persistent and sustained in large part for at least 30 days following treatment.

Conclusions involving all three crude oils follow.

- The viscosity (cP) of the three crude oils evaluated in this project varied over two orders of magnitude. The study demonstrated that sonication can effectively reduce the viscosity of each oil tested, either alone or in the presence of chemical additives included in the study. The greatest effects due to sonication alone are observed in the heavy crude with the least effects being produced in the light crude.

- A very similar pattern of variation was observed in the results obtained with sonication only and with sonication plus chemical additives. The greatest viscosity changes due to the addition of the chemical additives, both without sonication and with sonication, occurred with the heavy crude, whereas the least effects occurred with the lighter crude.

## 6.2 Recommendations

This investigation has demonstrated the potential utility of sonication/acoustic stimulation in reducing the viscosity of crude oil. These results have many potential applications and benefits to different sectors of the petroleum industry. However, before these potential applications can become reality, additional scientific and engineering work remains to be done. The recommendations given below are made with this need in mind.

- A brief follow-up laboratory investigation using the same equipment as in the study described herein should be undertaken to solidify the results obtained and to expand upon them. Three or four additional crude oils with viscosity values in the range of

approximately 5,000 cP to 50,000 cP (approximately 12º to 24º API gravity) should be included. The test procedures should focus on optimizing results with shorter treatment/residence times and lesser concentrations of chemical additives. Testing should be performed with the approximate acoustic range used in the present study as a maximum value, with a focus on lower frequencies and using more powerful, second-generation actuators.

- A simple laboratory investigation should be conducted to quantify the effects, if any, of sonication alone and sonication with chemical additives on the chemistry of crude oils. A range of acoustic conditions should be evaluated on heavy crude oils with different properties and characteristics.

- An extensive engineering evaluation and design activity should be undertaken to develop optimal equipment designs that could be used as a basis for various proposed applications within the petroleum industry. These would be both in-well, underground, *in situ* applications as well as above ground applications. The key design objectives would focus on directing the most powerful and intense acoustic energy of optimum frequency within a specific treatment area. Above-ground applications should also focus on the ability to obtain the desired oil flow conditions in the minimum amount of time.

- Efforts should be undertaken to identify or design next-generation, more powerful acoustic equipment that can be modified and optimized for the proposed petroleum applications and to integrate these items into the design activities recommended immediately above. Experiments with this more powerful equipment should evaluate the ability of sonication to change crude oil viscosity to handle paraffins, waxes, and to break sulfur compound chains.

- Additional laboratory testing should be performed in which sonication is applied in a hydrogen-rich environment or in the presence of a hydrogen source. The molecular structure of crude oil before and after the test needs to be determined in order to evaluate sonication's potential to upgrade crude by the addition of hydrogen and/or removal of carbon.

- Multiple in-well tests should be performed to verify viscosity reduction/production stimulation by sonication. Various items of sensor equipment that can be deployed downhole should be employed to evaluate the effects of variations in power, wave focus mechanisms, and acoustic characteristics under differing geologic and crude oil conditions.

- Several field demonstrations should be performed to evaluate the various methods of integrating sonication technology into surface petroleum applications such as

transportation (pipelines, storage tanks, tankers, etc.) and refining (e.g. sulfur removal). Data from these projects would be used to optimize the sonication applications, refine equipment designs, and provide information on changes in system efficiencies.

- As more data on the technology applications become available, a detailed economic assessment of the technology applications within various petroleum industry sectors must be completed to quantify the probable economic benefits given various commercialization scenarios.

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# APPENDIX A VISCOSITY MEASUREMENT SUPPORTING DOCUMENTATION

## A-1 ASTM Standard Test Method for Viscosity by Dip-Type Viscosity Cups (Designation: D 4212)

## A-2 Calibration Data and Computation Procedures Provided by the Manufacturer of the Cups Used in this Study

# APPENDIX SECTION A-1

Designation: D 4212 – 99

# Standard Test Method for Viscosity by Dip-Type Viscosity Cups[1]

This standard is issued under the fixed designation D 4212; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 This test method covers the determination of viscosity of paints, varnishes, lacquers, inks, and related liquid materials by dip-type viscosity cups. This test method is recommended for viscosity control work within one plant or laboratory and should be used to check compliance with specifications only when sufficient controls have been instituted to ensure adequate comparability of results.

1.2 Viscosity cups are designed for testing of Newtonian and near-Newtonian liquids. If the test material is non-Newtonian, for example, shear-thinning or thixotropic, another method, such as Test Methods D 2196, should be used. Under controlled conditions, comparisons of the viscosity of non-newtonian materials may be helpful, but viscosity determination methods using controlled shear rate or shear stress are preferred.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

## 2. Referenced Documents

2.1 *ASTM Standards:*

D 1200 Test Method for Viscosity by Ford Viscosity Cup[2]

D 2196 Test Methods for Rheological Properties of Non-Newtonian Materials by Rotational (Brookfield) Viscometer[2]

D 4287 Test Method for High Shear Viscosity Using the ICI Cone/Plate Viscometer[2]

E 1 Specification for ASTM Thermometers[3]

## 3. Terminology

3.1 *Definitions:*

3.1.1 *near-Newtonian liquid*—a liquid in which the variation of viscosity with shear rate is small and the effect on viscosity of mechanical disturbances such as stirring is negligible.

3.1.2 *Newtonian liquid*—a liquid in which the viscosity is independent of the shear stress or shear rate. If the ratio of shear stress to shear rate is not constant, the liquid is non-Newtonian.

## 4. Summary of Test Method

4.1 The cup is completely immersed in the material to be tested, withdrawn, and the time for the material to flow through a hole in the base of the cup is measured.

## 5. Significance and Use

5.1 Viscosity is a measure of the fluidity of a material. Viscosity data are useful in the determination of the ease of stirring, pumping, dip coating, or other flow-related properties of paints and related fluids.

5.2 This type of cup is used to measure viscosity because it is easy to use, robust, and may be used in tanks, reservoirs, and reactors.

5.3 There are other types of apparatus for measuring viscosity in the laboratory that provide better precision and bias, including the Ford viscosity cup (Test Method D 1200), and the Brookfield viscometer (Test Methods D 2196).

5.4 Certain higher shear rate devices such as cone/plate viscometers (Test Method D 4287) provide more information about sprayability, roll coatability, and other high-shear rate related properties of coatings.

## 6. Apparatus

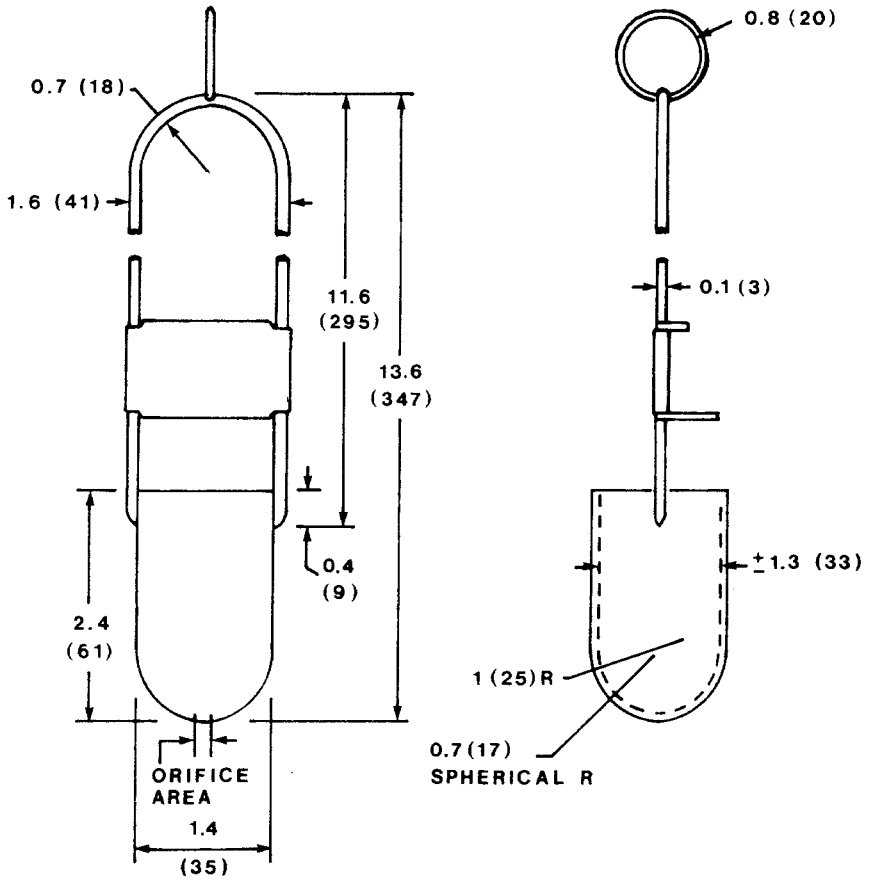
6.1 *Zahn Viscosity Cup*—No. 1 through No. 5 Zahn viscosity cups made of corrosion- and solvent-resistant materials. The nominal capacity of the cup is 44 mL, but may vary from 43 to 49 mL, depending on the manufacturer. A diagram of a Zahn cup is given in Fig. 1. The dimensions, including orifices, are only approximate because the cups are not made to a uniform specification. Each manufacturer produces a different cup and considerable variation between batches from some manufacturers has been noted in the past. This is a major

[1] This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee D01.24 on Physical Properties of Liquid Paints and Paint Materials.

Current edition approved May 10, 1999. Published July 1999. Originally published as D 4212 – 82. Last previous edition D 4212 – 93.

[2] *Annual Book of ASTM Standards*, Vol 06.01.

[3] *Annual Book of ASTM Standards*, Vol 14.03.




NOTE 1—Dimensions are approximate only and may vary with the manufacturer and from batch to batch

**FIG. 1 Zahn Cup Nominal Dimensions**

reason why Zahn cups should not be referenced in specifications between producer and user only when controls sufficient to ensure adequate cup-to-cup and operator-to-operator comparison are included. (See Appendix X1 for additional information on Zahn Cups.)

NOTE 1—The various cup numbers are for identification of the viscosity ranges within the series only and should not be used for comparison between different kinds of cups, that is, a No. 2 Zahn cup has no relationship whatsoever with a No. 2 Shell cup.

6.1.1 Nominal Zahn cup orifice diameters are listed in Table X2.1. Cup No. 1 with the smallest orifice is used for determining the viscosity of thin-bodied materials. Cup No. 2 is for use with clears, lacquers, enamels, and press-side adjustment of flexographic inks; cups Nos. 3 and 4 are for use with more viscous paints and inks (No. 3 for manufacturing of flexographic inks); and cup No. 5 is used for silk screen inks.

6.2 *Shell Viscosity Cup*[4]—No. 1 through No. 6 Shell viscosity cups made of stainless steel with a capacity of 23 mL and a 1-in. (25-mm) long capillary in the bottom and conforming to the dimensions shown in Fig. 2.

6.2.1 Nominal Shell cup orifice diameters are listed in Table X2.1. Cup Nos. 1 through 2½ are recommended for use with reduced rotogravure inks; No. 2 is for use with flexographic inks; Nos. 3 through 4 are used for industrial enamels, lacquers, flexographic, and gravure inks; Nos. 5 and 6 are used for heavy materials.

[4] Shell cups may be obtained from the Norcross Corp., 255 Newtonville Ave., Newton, MA 02158. This committee is not aware of any other source for flow cups having properties similar enough to the Shell cup to be included in this test method. If you have knowledge of a cup that should be considered, please provide details to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

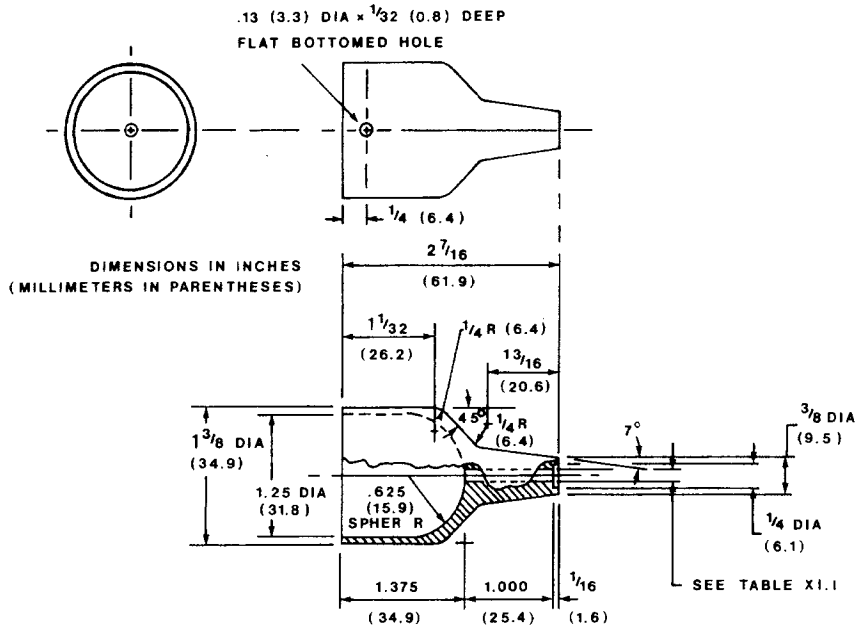



FIG. 2 Shell Cup

6.3 *Calibration Thermometer*—ASTM Saybolt Viscosity Thermometer 17F having a range of 66 to 80°F and subdivisions of 0.2°F, or 17C having a range of 19 to 27°C and subdivisions of 0.1°C, both conforming to the requirements of Specification E 1. Thermometers having subdivisions other than these may be used depending on the sensitivity of the material to be tested, the demands of the application, and the agreement between the purchaser and seller.

6.4 *Timer*—Any timing device may be used provided that the readings can be taken with a discrimination of 0.1 s or better.

## 7. Test Materials

7.1 The material to be tested should be visibly homogeneous and free from any foreign material or air bubbles.

## 8. Temperature of Testing

8.1 Measurements should be made at 77°F (25°C) unless otherwise specified. Temperature drift during the test should be kept to a minimum. The viscosities of paints and related materials are highly dependent on temperature. Differences in temperature between measurements can give substantially different viscosities (up to 5 % per °F). For careful work, the temperature should be taken in the efflux stream, but for process control (such as monitoring a dip tank), this is not necessary.

8.2 A temperature correction curve may be constructed for each liquid by plotting viscosity (seconds) against temperature over the expected temperature range. With this curve, a viscosity determined at one measured temperature may be converted quickly to a viscosity at another temperature.

NOTE 2—When dip cups are used for original purposes, that is thinning or monitoring of materials in tanks, coaters, etc., temperature is not important. This is because the key to good operation is to maintain the fluid within a certain range of dip cup-seconds regardless of the temperature of the fluid.

## 9. Checking and Calibration of Cups

9.1 Cups should be checked in accordance with the procedure described in Appendix X2. The frequency of this depends upon the amount of use and care that the individual cup receives, and the level of precision required.

9.2 Cups may be calibrated with standard fluids according to the procedure in Appendix X3. However, because the viscosity of standard fluids can vary significantly with temperature and due to difficulty in obtaining adequate temperature control with dip cups, calibration is a difficult procedure that must be done with great care and knowledge.

## 10. Procedure

10.1 Choose the proper cup so that the time of efflux will be between 20 and 80 s. See Table 1 for viscosity ranges for the various cups.

NOTE 3—The formulas used in this test method to describe the conversion from Zahn seconds to stokes are linear, the actual cup response

**TABLE 1 Approximate Viscosity Ranges, cST ($mm^2/s$) (Roughly Corresponding to 20 to 80 s Flow Time)**

| Cup Number | Zahn Cup | Shell Cup |
|---|---|---|
| 1[A] | 5–60 | 2–20 |
| 2 | 20–250 | 10–50 |
| 2½ | ... | 20–80 |
| 3 | 100–800 | 30–120 |
| 3½ | ... | 40–170 |
| 4 | 200–1200 | 70–270 |
| 5 | 400–1800 | 125–520 |
| 6 | ... | 320–1300 |

[A]The lower limit for the Zahn No. 1 cup is 35 s rather than 20 s.

is not. The range of 20 to 80 s covers the most linear portion of each cup. In addition, below 20 s, turbulent flow may cause additional inconsistencies. Above 80 s, factors that may impact on the precision include; loss of solvent (and therefore varying viscosity), "skinning" of the liquid in the cup, intermittent flow.

10.2 Immerse the cup in the container, which may be a can or beaker, but is more likely to be a thinning or mixing tank or even a resin reactor. Stir or agitate the fluid well to give uniform temperature and density. Allow the cup to remain in the fluid for 1 to 5 min to attain thermal equilibrium. (Because of their greater mass, Shell cups should remain in the fluid for the full 5 min.)

NOTE 4—Dip cups are not recommended for use with thixotropic (time dependent) materials but if used for them (such as gravure or flexographic inks), more vigorous agitation will be necessary to break up the structure before the measurement is made.

10.3 Lift the cup vertically out of the material in a quick, steady motion. As the top edge of the cup breaks the surface, start the timer. During the time of flow, hold the cup vertically no more than 15.2 cm (6 in.) above the level of the liquid. Stop the timer at the first definite break in the stream at the base of the cup. The efflux time in seconds constitutes the viscosity. It is common to make only a single measurement, but for greater precision and accuracy the mean of two or more measurements should be taken.

NOTE 5—The cup should not be held by the loop handle during the measurement process. Most manufacturers equip the cup with a ring through the loop handle. Holding the cup by this ring will help to ensure that the cup hangs vertically.

## 11. Care of Cups

11.1 Following each determination, clean the cup with a suitable solvent and a soft brush. Use no metal tools in contact with the instrument as nicks or wear of the drilled orifice affect the accuracy of the cup.

## 12. Report

12.1 Report the efflux time to the nearest 0.2 s for Zahn or Shell cup No. ___, manufactured by _____, (in the case of Zahn cups) the temperature of the fluid (where measured), and whether the result is from a single measurement or the mean of two of more measurements.

## 13. Precision and Bias

13.1 The most satisfactory results when using dip cups are obtained when viscosity is being controlled at a single location only. However, when comparisons between locations are made, cups from the same manufacturer must be used or other action taken to ensure compatibility of results. The following criteria can be used for judging the acceptability of results at the 95 % confidence level:

13.1.1 *Zahn Cups*—Precision was determined on the basis of an interlaboratory test in which six laboratories used new Zahn cups (all from the same set from the same manufacturer) to test eight paints covering a broad range of viscosities. The within-laboratory coefficient of variation was 3.7 % and the between-laboratories coefficient of variation was 11.5 %. Based on these coefficients the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

13.1.1.1 *Repeatability*—Two results, each the mean of two measurements, obtained by the same operator should be considered suspect if they differ by more than 11 % of their mean value.

13.1.1.2 *Reproducibility*—Two results, each the mean of two measurements, obtained by operators in different laboratories should be considered suspect if they differ by more than 33 % of their mean value.

NOTE 6—The values used to determine the precision were obtained under ideal conditions (a single set of cups), reproducibility in practice can be just as good, by employing strict controls and good techniques.

13.1.1.3 *Bias*—Bias does not apply to this test method as no acceptable standards exist.

NOTE 7—Since the precision values were obtained under ideal conditions (a single set of cups), reproducibility in practice probably is poorer than that given (perhaps as bad as 50 %).

13.1.2 *Shell Cups*—Precision was determined on the basis of an interlaboratory test in which four laboratories tested seven paints covering a broad range of viscosities. The within-laboratory coefficient of variation was 3.2 % and the between-laboratories coefficient of variation was 6.3 %. Based on these coefficients the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

13.1.2.1 *Repeatability*—Two results, each the mean of two measurements, obtained by the same operator should be considered suspect if they differ by more than 9 % of their mean value.

13.1.2.2 *Reproducibility*—Two results, each the mean of two measurements, obtained by operators in different laboratories should be considered suspect if they differ by more than 18 % of their mean value.

13.1.2.3 *Bias*—Bias does not apply to this test method as no acceptable standards exist.

## 14. Keywords

14.1 dip cup(s); flow cup(s); Shell cup(s); viscosity; Zahn cup(s)

# ANNUAL BOOK OF ASTM STANDARDS

## 2003

SECTION SIX

## Paints, Related Coatings, and Aromatics

VOLUME 06.01

### Paint–Tests for Chemical, Physical, and Optical Properties; Appearance

ASTM INTERNATIONAL
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*Revision Issued Annually*

## Appendix Section A-2

# EZ© VISCOSITY CUP #2
# DRAIN TIME - CENTISTOKES CONVERSION TABLE©

(Accurate for True Liquids Only)

| SECONDS | 0.0 | 0.1 | 0.2 | 0.3 | 0.4 | 0.5 | 0.6 | 0.7 | 0.8 | 0.9 |
|---|---|---|---|---|---|---|---|---|---|---|
| | < — — — — — — — — — VISCOSITY IN CENTISTOKES — — — — — — — — — > | | | | | | | | | |
| 20 | 18.7 | 19.1 | 19.6 | 20.0 | 20.5 | 21.0 | 21.4 | 21.9 | 22.3 | 22.8 |
| 21 | 23.2 | 23.7 | 24.1 | 24.6 | 25.0 | 25.5 | 25.9 | 26.3 | 26.8 | 27.2 |
| 22 | 27.6 | 28.1 | 28.5 | 28.9 | 29.4 | 29.8 | 30.2 | 30.7 | 31.1 | 31.5 |
| 23 | 31.9 | 32.3 | 32.8 | 33.2 | 33.6 | 34.0 | 34.4 | 34.8 | 35.3 | 35.7 |
| 24 | 36.1 | 36.5 | 36.9 | 37.3 | 37.7 | 38.1 | 38.5 | 38.9 | 39.3 | 39.7 |
| 25 | 40.1 | 40.5 | 40.9 | 41.3 | 41.7 | 42.1 | 42.5 | 42.9 | 43.3 | 43.7 |
| 26 | 44.1 | 44.5 | 44.8 | 45.2 | 45.6 | 46.0 | 46.4 | 46.8 | 47.2 | 47.6 |
| 27 | 47.9 | 48.3 | 48.7 | 49.1 | 49.5 | 49.8 | 50.2 | 50.6 | 51.0 | 51.3 |
| 28 | 51.7 | 52.1 | 52.5 | 52.8 | 53.2 | 53.6 | 54.0 | 54.3 | 54.7 | 55.1 |
| 29 | 55.4 | 55.8 | 56.2 | 56.5 | 56.9 | 57.3 | 57.6 | 58.0 | 58.4 | 58.7 |
| 30 | 59.1 | 59.5 | 59.8 | 60.2 | 60.5 | 60.9 | 61.3 | 61.6 | 62.0 | 62.3 |
| 31 | 62.7 | 63.1 | 63.4 | 63.8 | 64.1 | 64.5 | 64.8 | 65.2 | 65.5 | 65.9 |
| 32 | 66.3 | 66.6 | 67.0 | 67.3 | 67.7 | 68.0 | 68.4 | 68.7 | 69.1 | 69.4 |
| 33 | 69.8 | 70.1 | 70.5 | 70.8 | 71.2 | 71.5 | 71.8 | 72.2 | 72.5 | 72.9 |
| 34 | 73.2 | 73.6 | 73.9 | 74.3 | 74.6 | 74.9 | 75.3 | 75.6 | 76.0 | 76.3 |
| 35 | 76.7 | 77.0 | 77.3 | 77.7 | 78.0 | 78.4 | 78.7 | 79.0 | 79.4 | 79.7 |
| 36 | 80.1 | 80.4 | 80.7 | 81.1 | 81.4 | 81.7 | 82.1 | 82.4 | 82.7 | 83.1 |
| 37 | 83.4 | 83.7 | 84.1 | 84.4 | 84.7 | 85.1 | 85.4 | 85.7 | 86.1 | 86.4 |
| 38 | 86.7 | 87.1 | 87.4 | 87.7 | 88.1 | 88.4 | 88.7 | 89.1 | 89.4 | 89.7 |
| 39 | 90.0 | 90.4 | 90.7 | 91.0 | 91.4 | 91.7 | 92.0 | 92.3 | 92.7 | 93.0 |
| 40 | 93.3 | 93.7 | 94.0 | 94.3 | 94.6 | 95.0 | 95.3 | 95.6 | 95.9 | 96.3 |
| 41 | 96.6 | 96.9 | 97.2 | 97.6 | 97.9 | 98.2 | 98.5 | 98.8 | 99.2 | 99.5 |
| 42 | 99.8 | 100.1 | 100.5 | 100.8 | 101.1 | 101.4 | 101.7 | 102.1 | 102.4 | 102.7 |
| 43 | 103.0 | 103.3 | 103.7 | 104.0 | 104.3 | 104.6 | 104.9 | 105.3 | 105.6 | 105.9 |
| 44 | 106.2 | 106.5 | 106.9 | 107.2 | 107.5 | 107.8 | 108.1 | 108.4 | 108.7 | 109.1 |
| 45 | 109.4 | 109.7 | 110.0 | 110.3 | 110.7 | 111.0 | 111.3 | 111.6 | 111.9 | 112.2 |
| 46 | 112.6 | 112.9 | 113.2 | 113.5 | 113.8 | 114.1 | 114.4 | 114.8 | 115.1 | 115.4 |
| 47 | 115.7 | 116.0 | 116.3 | 116.6 | 117.0 | 117.3 | 117.6 | 117.9 | 118.2 | 118.5 |
| 48 | 118.8 | 119.1 | 119.5 | 119.8 | 120.1 | 120.4 | 120.7 | 121.0 | 121.3 | 131.6 |
| 49 | 122.0 | 122.3 | 122.6 | 122.9 | 123.2 | 123.5 | 123.8 | 124.1 | 124.4 | 124.8 |
| 50 | 125.1 | 125.4 | 125.7 | 126.0 | 126.3 | 126.6 | 126.9 | 127.2 | 127.5 | 127.8 |
| 51 | 128.2 | 128.5 | 128.8 | 129.1 | 129.4 | 129.7 | 130.0 | 130.3 | 130.6 | 130.9 |
| 52 | 131.2 | 131.5 | 131.8 | 132.2 | 132.5 | 132.8 | 133.1 | 133.4 | 133.7 | 134.0 |
| 53 | 134.3 | 134.6 | 134.9 | 135.2 | 135.5 | 135.8 | 136.1 | 136.4 | 136.8 | 137.1 |
| 54 | 137.4 | 137.7 | 138.0 | 138.3 | 138.6 | 138.9 | 139.2 | 139.5 | 139.8 | 140.1 |
| 55 | 140.4 | 140.7 | 141.0 | 141.3 | 141.6 | 141.9 | 142.2 | 142.5 | 142.9 | 143.2 |
| 56 | 143.5 | 143.8 | 144.1 | 144.4 | 144.7 | 145.0 | 145.3 | 145.6 | 145.9 | 146.2 |
| 57 | 146.5 | 146.8 | 147.1 | 147.4 | 147.7 | 148.0 | 148.3 | 148.6 | 148.9 | 149.2 |
| 58 | 149.5 | 149.8 | 150.1 | 150.4 | 150.7 | 151.0 | 151.3 | 151.6 | 151.9 | 152.2 |
| 59 | 152.5 | 152.8 | 153.1 | 153.4 | 153.7 | 154.0 | 154.3 | 154.6 | 154.9 | 155.2 |
| 60 | 155.6 | 155.9 | 156.2 | 156.5 | 156.8 | 157.1 | 157.4 | 157.7 | 158.0 | 158.3 |

Example: 53.8 Seconds = 136.8 Centistokes.



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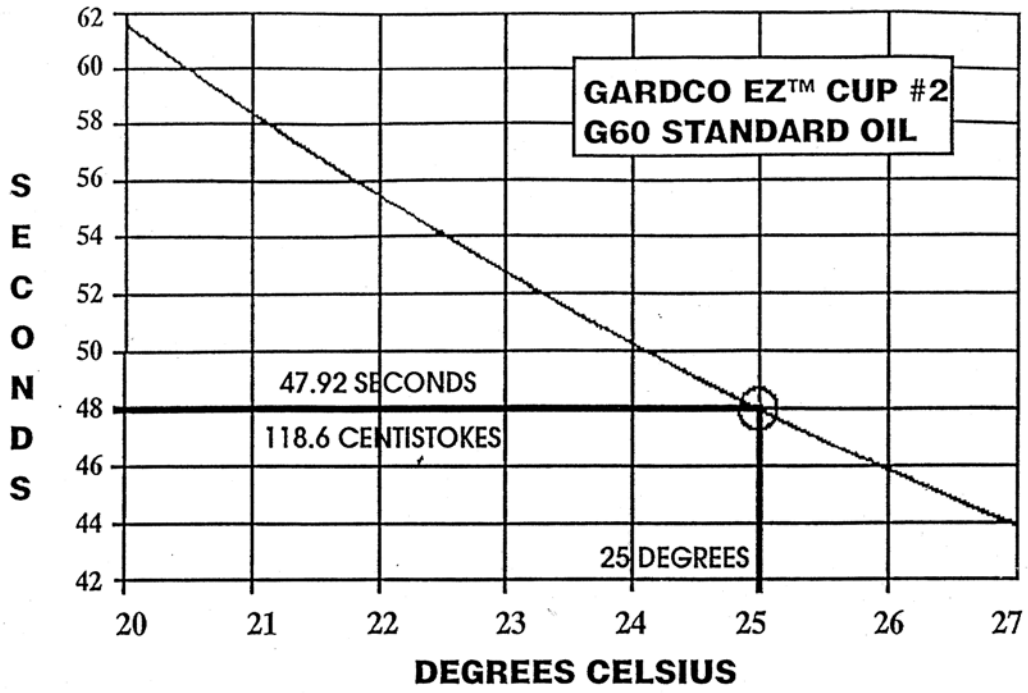
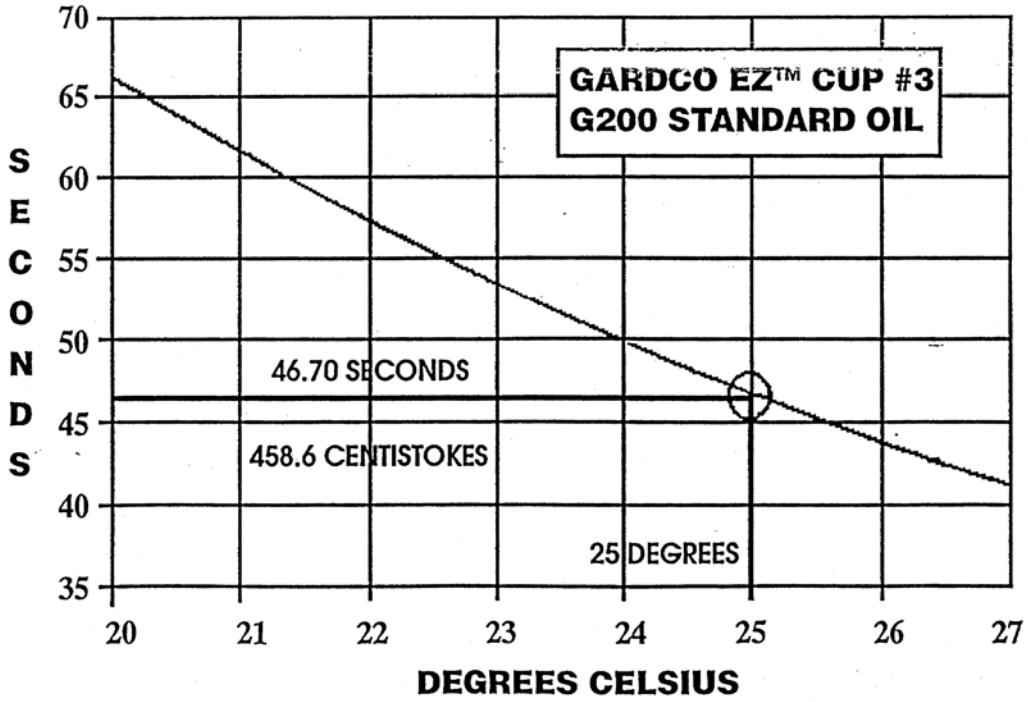
# GARDCO EZ™ VISCOSITY CUPS

## EFFLUX TIME IN SECONDS — TEMPERATURE

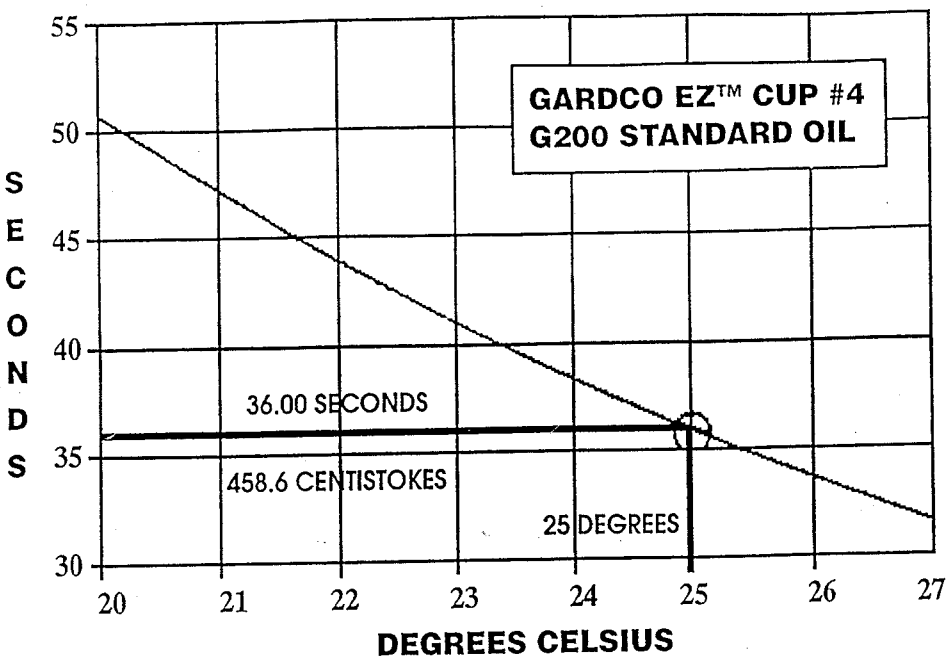


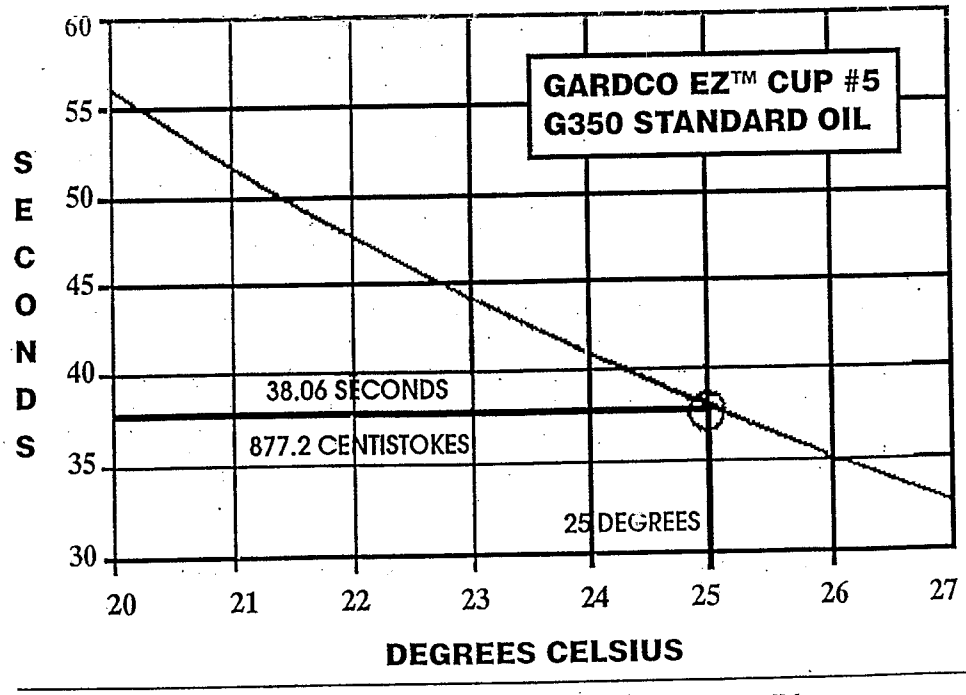




COLE-PARMER INSTRUMENT COMPANY
625 EAST BUNKER COURT
VERNON HILLS, ILLINOIS 60061

# GARDCO EZ™ VISCOSITY CUPS

## EFFLUX TIME IN SECONDS — TEMPERATURE

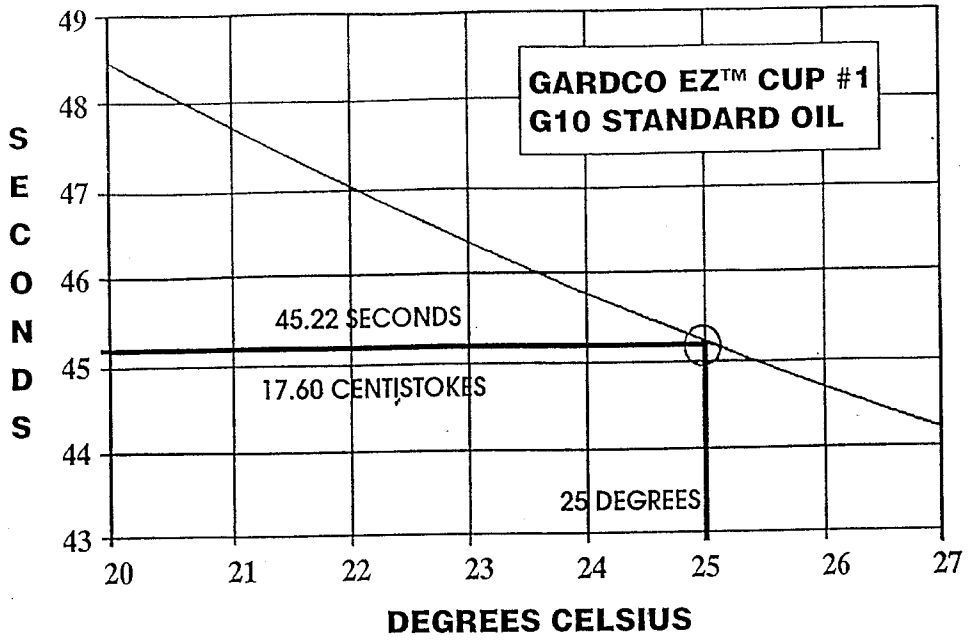



COLE-PARMER INSTRUMENT COMPANY
625 EAST BUNKER COURT
VERNON HILLS, ILLINOIS 60061

# GARDCO EZ™ VISCOSITY CUPS

## EFFLUX TIME IN SECONDS — TEMPERATURE




Gardco produced viscosity cups are calibrated with standard "G" Series oils. Centistoke viscosity of these oils is traceable to the National Institute of Standards and Technology. These oils are available from the Paul N. Gardner Company.

Shown in the above graph is the viscosity cup number and the standard "G" oil used for its calibration. Normally, cup calibration is at 25 degrees Celsius, shown on the graph by bold lines intersecting with the curve in the circle. Graphs for other numbered cups in the series are on following pages.

Viscosity of most liquids, including the standard oils, are dependent on temperature. Efflux time in seconds for the indicated cup-oil combination from twenty (20) to twenty seven (27) degrees Celsius is shown in the above graph. The cup may be checked with the indicated "G" oil with reasonable accuracy within these limits. for best accuracy, the standard oil label viscosity with temperature at 25 degrees Celsius shold be used. Conversion from viscosity to efflux time in seconds is by the formula or table furnished with the cup. Conversion between degrees Celsius and Fahrenheit is on the reverse side of this page.

This information, furnished as an additional customer service, is included with each viscosity cup sold by the Paul N. Gardner Company or by licensed distributors.

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COLE-PARMER INSTRUMENT COMPANY
625 EAST BUNKER COURT
VERNON HILLS, ILLINOIS 60061

VIBL-CP-12/10/96

# TEMPERATURE SCALE CONVERSION BETWEEN CELSIUS AND FAHRENHEIT

| DEGREES CELSIUS | DEGREES FAHRENHEIT |
|---|---|
| 20.0 | 68.0 |
| 20.1 | 68.2 |
| 20.2 | 68.4 |
| 20.3 | 68.5 |
| 20.4 | 68.7 |
| 20.5 | 68.9 |
| 20.6 | 69.1 |
| 20.7 | 69.3 |
| 20.8 | 69.4 |
| 20.9 | 69.6 |
| 21.0 | 69.8 |
| 21.1 | 70.0 |
| 21.2 | 70.2 |
| 21.3 | 70.3 |
| 21.4 | 70.5 |
| 21.5 | 70.7 |
| 21.6 | 70.9 |
| 21.7 | 71.1 |
| 21.8 | 71.2 |
| 21.9 | 71.4 |
| 22.0 | 71.6 |
| 22.1 | 71.8 |
| 22.2 | 72.0 |
| 22.3 | 72.1 |
| 22.4 | 72.3 |
| 22.5 | 72.5 |
| 22.6 | 72.7 |
| 22.7 | 72.9 |
| 22.8 | 73.0 |
| 22.9 | 73.2 |
| 23.0 | 73.4 |
| 23.1 | 73.6 |
| 23.2 | 73.8 |
| 23.3 | 73.9 |
| 23.4 | 74.1 |
| 23.5 | 74.3 |
| 23.6 | 74.5 |
| 23.7 | 74.7 |
| 23.8 | 74.8 |
| 23.9 | 75.0 |
| 24.0 | 75.2 |
| 24.1 | 75.4 |
| 24.2 | 75.6 |
| 24.3 | 75.7 |
| 24.4 | 75.9 |
| 24.5 | 76.1 |
| 24.6 | 76.3 |
| 24.7 | 76.5 |
| 24.8 | 76.6 |
| 24.9 | 76.8 |
| 25.0 | 77.0 |
| 25.1 | 77.2 |
| 25.2 | 77.4 |
| 25.3 | 77.5 |
| 25.4 | 77.7 |
| 25.5 | 77.9 |
| 25.6 | 78.1 |
| 25.7 | 78.3 |
| 25.8 | 78.4 |
| 25.9 | 78.6 |
| 26.0 | 78.8 |
| 26.1 | 79.0 |
| 26.2 | 79.2 |
| 26.3 | 79.3 |
| 26.4 | 79.5 |
| 26.5 | 79.7 |
| 26.6 | 79.9 |
| 26.7 | 80.1 |
| 26.8 | 80.2 |
| 26.9 | 80.4 |
| 27.0 | 80.6 |

$F° = (C° \times 1.8) + 32$

$C° = (F° - 32°) \div 1.8$

# EZ© VISCOSITY CUP #5
# DRAIN TIME - CENTISTOKES CONVERSION TABLE©

(Accurate for True Liquids Only)

| SECONDS | 0.0 | 0.1 | 0.2 | 0.3 | 0.4 | 0.5 | 0.6 | 0.7 | 0.8 | 0.9 |
|---|---|---|---|---|---|---|---|---|---|---|
| | < — — — — — — — — — VISCOSITY IN CENTISTOKES — — — — — — — — — > | | | | | | | | | |
| 10 | 161 | 164 | 167 | 170 | 173 | 177 | 180 | 183 | 186 | 189 |
| 11 | 192 | 194 | 197 | 200 | 203 | 206 | 209 | 212 | 215 | 218 |
| 12 | 221 | 224 | 226 | 229 | 232 | 235 | 238 | 241 | 243 | 246 |
| 13 | 249 | 252 | 255 | 257 | 260 | 263 | 266 | 268 | 271 | 274 |
| 14 | 277 | 279 | 282 | 285 | 288 | 290 | 293 | 296 | 298 | 301 |
| 15 | 304 | 306 | 309 | 312 | 315 | 317 | 320 | 323 | 325 | 328 |
| 16 | 330 | 333 | 336 | 338 | 341 | 344 | 346 | 349 | 352 | 354 |
| 17 | 357 | 359 | 362 | 365 | 367 | 370 | 372 | 375 | 378 | 380 |
| 18 | 383 | 385 | 388 | 390 | 393 | 396 | 398 | 401 | 403 | 406 |
| 19 | 408 | 411 | 414 | 416 | 419 | 421 | 424 | 426 | 429 | 431 |
| 20 | 434 | 437 | 439 | 442 | 444 | 447 | 449 | 452 | 454 | 457 |
| 21 | 459 | 462 | 464 | 467 | 469 | 472 | 474 | 477 | 479 | 482 |
| 22 | 485 | 487 | 490 | 492 | 495 | 497 | 500 | 502 | 505 | 507 |
| 23 | 510 | 512 | 515 | 517 | 520 | 522 | 524 | 527 | 529 | 532 |
| 24 | 534 | 537 | 539 | 542 | 544 | 547 | 549 | 552 | 554 | 557 |
| 25 | 559 | 562 | 564 | 567 | 569 | 572 | 574 | 577 | 579 | 581 |
| 26 | 584 | 586 | 589 | 591 | 594 | 596 | 599 | 601 | 604 | 606 |
| 27 | 609 | 611 | 613 | 616 | 618 | 621 | 623 | 626 | 628 | 631 |
| 28 | 633 | 636 | 638 | 640 | 643 | 645 | 648 | 650 | 653 | 655 |
| 29 | 658 | 660 | 662 | 665 | 667 | 670 | 672 | 675 | 677 | 680 |
| 30 | 682 | 684 | 687 | 689 | 692 | 694 | 697 | 699 | 701 | 704 |
| 31 | 706 | 709 | 711 | 714 | 716 | 719 | 721 | 723 | 726 | 728 |
| 32 | 731 | 733 | 736 | 738 | 740 | 743 | 745 | 748 | 750 | 753 |
| 33 | 755 | 757 | 760 | 762 | 765 | 767 | 769 | 772 | 774 | 777 |
| 34 | 779 | 782 | 784 | 786 | 789 | 791 | 794 | 796 | 799 | 801 |
| 35 | 803 | 806 | 808 | 811 | 813 | 815 | 818 | 820 | 823 | 825 |
| 36 | 828 | 830 | 832 | 835 | 837 | 840 | 842 | 844 | 847 | 849 |
| 37 | 852 | 854 | 856 | 859 | 861 | 864 | 866 | 868 | 871 | 873 |
| 38 | 876 | 878 | 881 | 883 | 885 | 888 | 890 | 893 | 895 | 897 |
| 39 | 900 | 902 | 905 | 907 | 909 | 912 | 914 | 917 | 919 | 921 |
| 40 | 924 | 926 | 929 | 931 | 933 | 936 | 938 | 941 | 943 | 945 |
| 41 | 948 | 950 | 953 | 955 | 957 | 960 | 962 | 965 | 967 | 969 |
| 42 | 972 | 974 | 977 | 979 | 981 | 984 | 986 | 989 | 991 | 993 |
| 43 | 996 | 998 | 1001 | 1003 | 1005 | 1008 | 1010 | 1013 | 1015 | 1017 |
| 44 | 1020 | 1022 | 1025 | 1027 | 1029 | 1032 | 1034 | 1036 | 1039 | 1041 |
| 45 | 1044 | 1046 | 1048 | 1051 | 1053 | 1056 | 1058 | 1060 | 1063 | 1065 |
| 46 | 1068 | 1070 | 1072 | 1075 | 1077 | 1080 | 1082 | 1084 | 1087 | 1089 |
| 47 | 1091 | 1094 | 1096 | 1099 | 1101 | 1103 | 1106 | 1108 | 1111 | 1113 |
| 48 | 1115 | 1118 | 1120 | 1123 | 1125 | 1127 | 1130 | 1132 | 1134 | 1137 |
| 49 | 1139 | 1142 | 1144 | 1146 | 1149 | 1151 | 1154 | 1156 | 1158 | 1161 |
| 50 | 1163 | 1166 | 1168 | 1170 | 1173 | 1175 | 1177 | 1180 | 1182 | 1185 |
| 51 | 1187 | 1189 | 1192 | 1194 | 1197 | 1199 | 1201 | 1204 | 1206 | 1208 |
| 52 | 1211 | 1213 | 1216 | 1218 | 1220 | 1223 | 1225 | 1227 | 1230 | 1232 |
| 53 | 1235 | 1237 | 1239 | 1242 | 1244 | 1247 | 1249 | 1251 | 1254 | 1256 |
| 54 | 1258 | 1261 | 1263 | 1266 | 1268 | 1270 | 1273 | 1275 | 1278 | 1280 |
| 55 | 1282 | 1285 | 1287 | 1289 | 1292 | 1294 | 1297 | 1299 | 1301 | 1303 |
| 56 | 1306 | 1308 | 1311 | 1313 | 1316 | 1318 | 1320 | 1323 | 1325 | 1327 |
| 57 | 1330 | 1332 | 1335 | 1337 | 1339 | 1342 | 1344 | 1347 | 1349 | 1351 |
| 58 | 1354 | 1356 | 1358 | 1361 | 1363 | 1366 | 1368 | 1370 | 1373 | 1375 |
| 59 | 1377 | 1380 | 1382 | 1385 | 1387 | 1389 | 1392 | 1394 | 1396 | 1399 |
| 60 | 1401 | 1404 | 1406 | 1408 | 1411 | 1413 | 1415 | 1418 | 1420 | 1423 |

Example: 45.3 Seconds = 1051 Centistokes.

# EZ© VISCOSITY CUPS
# (EQUIVALENT ZAHN)
# CUP #5
# CONVERSION FORMULAS AND TABLE©

EZ© viscosity cups are designed to comply with requirements of ASTM D-4212 and to take advantage of design changes known to provide best possible results. Cup dimensions are carefully controlled and cup calibration conditions comply with ANSI/NCSL Z540-1 or MIL-STD-45662A as applicable. Standard viscous oils traceable to the National Institute of Standards and Technology are used in calibration procedures to insure specified drain time tolerance.

Use this formula derived by Paul N. Gardner Company research to find viscosity (V) in centistokes when cup drain time in seconds (T) is known:

$$V = 23.56T - 744 \div T$$

Use this formula to find cup drain time in seconds (T) when viscosity (V) in centistokes is known:

$$T = (V + \sqrt{V^2 + 70115}) \div 47.12$$

Results from the above formulas, solved for each tenth second within the cup range, are shown on the reverse side of this page. To find centistoke viscosity for a given cup drain time in seconds, read down the column on the left to find the nearest second. Then, read to the right to the nearest tenth second column to find centistoke value. The chart may be read in reverse to find drain time seconds when viscosity is known.

The EZ© series of five viscosity cups are produced, calibrated and sold only by the Paul N. Gardner Company and licensed agents.



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VIBL-CP-12/10/96

# EZ© VISCOSITY CUPS
# (EQUIVALENT ZAHN)
# CUP #2
# CONVERSION FORMULAS AND TABLE©

EZ© viscosity cups are designed to comply with requirements of ASTM D-4212 and to take advantage of design changes known to provide best possible results. Cup dimensions are carefully controlled and cup calibration conditions comply with ANSI/NCSL Z540-1 or MIL-STD-45662A as applicable. Standard viscous oils traceable to the National Institute of Standards and Technology are used in calibration procedures to insure specified drain time tolerance.

Use this formula derived by Paul N. Gardner Company research to find viscosity (V) in centistokes when cup drain time in seconds (T) is known:

$$V = 2.80T - 747 \div T$$

Use this formula to find cup drain time in seconds (T) when viscosity (V) in centistokes is known:

$$T = (V + \sqrt{V^2 + 8366}) \div 5.60$$

Results from the above formulas, solved for each tenth second within the cup range, are shown on the reverse side of this page. To find centistoke viscosity for a given cup drain time in seconds, read down the column on the left to find the nearest second. Then, read to the right to the nearest tenth second column to find centistoke value. The chart may be read in reverse to find drain time seconds when viscosity is known.

The EZ© series of five viscosity cups are produced, calibrated and sold only by the Paul N. Gardner Company and licensed agents.



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625 EAST BUNKER COURT
VERNON HILLS, ILLINOIS 60061

# APPENDIX B

## Project Phase I Data

**Table B-1 Drainage Times as a Function of Temperature for the 30-Weight Oil**

30-Weight Oil

| | | |
|---|---|---|
| Initial Drainage Times (sec): | 176.46 | T=19.9°C |
| | 168.16 | (No.2 Dip Cup) |
| | 174.97 | |
| Mean: | 173.19 | |
| Standard Deviation: | 4.43 | |

| Temperature, (°C) | Drainage Time, (sec) | Drainage Time at Room Temperature | Oil Temperature, (°C) | $t/t_o$ |
|---|---|---|---|---|
| 19.9 | 173.20 | --- | 19.9 | 1 |
| 28.0 | 60.13 | 207.49 | 19.9 | 0.3472 |
| 39.5 | 46.53 | 195.24 | 19.9 | 0.2686 |
| 45.4 | 40.75 | 200.35 | 19.9 | 0.2353 |
| 50.8 | 37.39 | 193.97 | 20.4 | 0.2159 |
| 57.5 | 34.18 | 196.55 | 20.1 | 0.1973 |
| 62.1 | 31.67 | 197.29 | 20.1 | 0.1829 |
| 66.3 | 29.57 | 194.16 | 20.3 | 0.1707 |
| 70.7 | 27.74 | 190.01 | 20.3 | 0.1602 |
| 75.4 | 35.93 | 192.01 | 20.3 | 0.2074 |
| 80.1 | 24.18 | 195.91 | 20.3 | 0.1396 |
| 84.4 | 22.57 | 191.04 | 20.4 | 0.1303 |
| 90.2 | 21.51 | 191.94 | 20.5 | 0.1242 |
| 95.1 | 20.28 | 187.69 | 20.5 | 0.1171 |
| 99.5 | 19.69 | 190.26 | 20.7 | 0.1137 |
| | Mean: | 194.57 | | |
| | Standard Deviation: | 5.02 | | |

**Table B-2 Drainage Times as a Function of Temperature for the 90-Weight Oil**

90-Weight Oil

| | | |
|---|---|---|
| Initial Drainage Times (sec): | 412.20 | T=20.4°C |
| | 397.53 | (No.2 Dip Cup) |
| | 409.20 | |
| Mean: | 406.31 | |
| Standard Deviation: | 7.75 | |
| | 38.40 | T=20.4°C |
| | 39.33 | (No.5 Dip Cup) |
| | 39.16 | |
| Mean: | 38.96 | |
| Standard Deviation: | 0.50 | |

| Temperature, (°C) | Drainage Time, (sec) | Drainage Time at Room Temperature | Oil Temperature, (°C) | $t/t_o$ |
|---|---|---|---|---|
| 20.4 | 406.31 | --- | --- | 1 |
| 24.9 | 107.52 | 48.71 | 20.1 | 0.2646 |
| 39.7 | 74.89 | 38.21 | 20.1 | 0.1843 |
| 51.5 | 59.37 | 40.17 | 19.9 | 0.1461 |
| 58.5 | 54.48 | 38.09 | 20.1 | 0.1341 |
| 62.2 | 49.59 | 39.93 | 19.9 | 0.1220 |
| 67.8 | 46.46 | 37.75 | 19.9 | 0.1143 |
| 72.3 | 41.42 | 36.83 | 20.7 | 0.1019 |
| 76.3 | 35.85 | 33.71 | 21.5 | 0.0882 |
| 81.0 | 31.18 | 36.56 | 21.9 | 0.0767 |
| 85.5 | 32.64 | 33.73 | 21.9 | 0.0803 |
| 85.5 | 31.61 | --- | --- | 0.0778 |
| 91.1 | 27.67 | 32.95 | 21.7 | 0.0681 |
| 95.0 | 28.48 | 35.32 | 21.1 | 0.0701 |
| 101.1 | 24.45 | 36.89 | 21.3 | 0.0602 |
| 105.1 | 23.71 | 33.29 | 21.8 | 0.0584 |
| | Mean: | 37.30 | | |
| | Standard Deviation: | 4.04 | | |

**Table B-3 Drainage Times as a Function of Temperature for the 140-Weight Oil**

140-Weight Oil

| | | |
|---|---|---|
| Initial Drainage Times (sec): | 39.82 | T=20.3°C |
| | 36.48 | (No.5 Dip Cup) |
| | 38.30 | |
| | 38.72 | |
| Mean: | 38.33 | |
| Standard Deviation: | 1.39 | |

| Temperature, (°C) | Drainage Time, (sec) | Drainage Time at Room Temperature | Oil Temperature, (°C) | $t/t_o$ |
|---|---|---|---|---|
| 20.3 | 38.33 | 38.33 | 20.3 | 1 |
| 28.5 | 24.53 | 45.63 | 20.7 | 0.6400 |
| 38.5 | 16.20 | 46.65 | 20.3 | 0.4226 |
| 43.5 | 13.47 | 44.13 | 20.1 | 0.3514 |
| 49.6 | 11.26 | 43.04 | 21.1 | 0.2938 |
| 52.9 | 10.75 | 43.89 | 20.3 | 0.2805 |
| 57.0 | 10.12 | 41.05 | 20.7 | 0.2640 |
| 62.1 | 8.94 | 43.25 | 21.1 | 0.2332 |
| 67.1 | 8.12 | 42.38 | 20.9 | 0.2118 |
| 73.9 | 7.23 | 48.46 | 20.0 | 0.1886 |
| 79.1 | 7.13 | 47.67 | 19.3 | 0.1860 |
| 84.5 | 6.44 | 47.43 | 19.1 | 0.1680 |
| 89.6 | 6.27 | 48.89 | 19.9 | 0.1636 |
| 94.0 | 5.42 | 47.71 | 19.2 | 0.1414 |
| 100.9 | 5.49 | 49.01 | 19.1 | 0.1432 |
| | Mean: | 45.17 | | |
| | Standard Deviation: | 3.19 | | |

**Table B-4 Data from Testing 30-Weight Oil at 6.9 kHz with Small Horn Spacing**

30-Weight Oil:

Nominal Experimental Conditions:

Acoustic Frequency: 6.9 kHz
Horn Design: Small Spacing

Initial Drainage Times, (sec): 18.99, 18.03, 18.09, 18.47, 18.64 T=21.7°C (No.5 Cup)

Mean: 18.44
Standard Deviation: 0.40

| Acoustic Frequency Measured, (kHz) | Treatment Time, (min) | Oil Temperature (°C) | Drainage Time (sec) | Drainage Time After Cooling, (sec) | Temperature After Cooling, (°C) | Fractional Residual Viscosity |
|---|---|---|---|---|---|---|
| 6.88 | 0 | 21.7 | 18.44 | 18.67 | 21.5 | 1 |
| 6.88 | 5 | 22.6 | 13.98 | 19.17 | 21.7 | 0.75797 |
| 6.88 | 10 | 21.9 | 11.71 | 19.33 | 21.5 | 0.63489 |
| 6.88 | 14.5 | 23.1 | 10.14 | 18.93 | 21.5 | 0.54977 |
| 6.88 | 19 | 23.0 | 12.69 | 18.31 | 21.7 | 0.68776 |
| 6.88 | 23 | 24.2 | 11.54 | 18.39 | 21.6 | 0.62568 |
| 6.88 | 27.5 | 24.7 | 12.63 | 18.72 | 21.3 | 0.68478 |
| 6.88 | 31.5 | 25.1 | 12.49 | 18.07 | 21.7 | 0.67718 |
| 6.88 | 35 | 24.4 | 12.34 | 18.23 | 21.6 | 0.66905 |

**Table B-5 Data from Testing 30-Weight Oil at 6.9 kHz with Medium Horn Spacing**

30-Weight Oil:

Nominal Experimental Conditions:

Acoustic Frequency: 6.9 kHz
Ultrasonic Horn: Medium Spacing

Initial Drainage Times, (sec): 19.03 T=21.4°C
18.19 (No.5 Cup)
16.89
17.83
Mean: 17.99
Standard Deviation: 0.89

| Acoustic Frequency Measured, (kHz) | Treatment Time, (min) | Oil Temperature (°C) | Drainage Time (sec) | Drainage Time After Cooling, (sec) | Temperature After Cooling, (°C) | Fractional Residual Viscosity |
|---|---|---|---|---|---|---|
| 6.87 | 0 | 21.5 | 17.99 | 17.38 | 22.4 | 1 |
| 6.87 | 5 | 21.5 | 15.85 | 17.20 | 23.6 | 0.88129 |
| 6.87 | 10 | 22.8 | 14.54 | 16.44 | 23.8 | 0.80845 |
| 6.87 | 15 | 22.8 | 11.67 | 16.81 | 23.7 | 0.64887 |
| 6.87 | 20 | 23.3 | 11.13 | 16.94 | 23.6 | 0.61885 |
| 6.87 | 25 | 23.8 | 10.47 | 17.04 | 23.5 | 0.58215 |
| 6.87 | 30 | 23.8 | 10.30 | 16.13 | 23.7 | 0.57270 |
| 6.87 | 35 | 24.2 | 9.63 | 16.83 | 23.7 | 0.53545 |
| 6.87 | 40 | 25.2 | 10.37 | 16.57 | 23.8 | 0.57659 |

**Table B-6 Data from Testing 30-Weight Oil at 6.9 kHz with Large Horn Spacing**

30-Weight Oil:

Nominal Experimental Conditions:

Acoustic Frequency: 6.9 kHz
Horn Design: Large Spacing

Initial Drainage Times, (sec): 18.60 T=20.9°C
18.25 (No.5 Viscometer)
17.59
16.95
Mean: 17.85
Standard Deviation: 0.73

| Acoustic Frequency Measured, (kHz) | Treatment Time, (min) | Oil Temperature (°C) | Drainage Time (sec) | Drainage Time After Cooling, (sec) | Temperature After Cooling, (°C) | Fractional Residual Viscosity |
|---|---|---|---|---|---|---|
| 6.87 | 0 | 20.3 | 17.85 | 17.75 | 23.1 | 1 |
| 6.87 | 5 | 21.3 | 14.65 | 17.76 | 22.3 | 0.82084 |
| 6.87 | 10 | 21.8 | 10.5 | 17.60 | 22.7 | 0.58832 |
| 6.87 | 15 | 22.0 | 10.69 | 17.79 | 22.6 | 0.59896 |
| 6.87 | 20 | 22.8 | 10.87 | 17.65 | 22.9 | 0.60905 |
| 6.87 | 25 | 23.0 | 11.56 | 17.91 | 22.8 | 0.64771 |
| 6.87 | 30 | 23.6 | 11.51 | 17.60 | 22.9 | 0.64491 |

**Table B-7 Data from Testing 30-Weight Oil at 13.1 kHz with Small Horn Spacing**

30-Weight Oil:

Nominal Experimental Conditions:

Acoustic Frequency: 13.1 kHz
Horn Design: Small Spacing

Initial Drainage Times, (sec): 17.53, 17.94, 16.47, 16.46 — T=22.7°C (No.5 Cup)
Mean: 17.10
Standard Deviation: 0.75

| Acoustic Frequency Measured, (kHz) | Treatment Time, (min) | Oil Temperature (°C) | Drainage Time (sec) | Drainage Time After Cooling, (sec) | Temperature After Cooling, (°C) | Fractional Residual Viscosity |
|---|---|---|---|---|---|---|
| 13.07 | 0 | 22.7 | 17.10 | 17.24 | 23.0 | 1 |
| 13.07 | 5 | 28.7 | 12.85 | 17.44 | 23.1 | 0.75146 |
| 13.07 | 10 | 24.8 | 10.43 | 17.40 | 23.1 | 0.60994 |
| 13.07 | 15 | 28.1 | 11.13 | 17.27 | 23.0 | 0.65088 |
| 13.07 | 20 | 28.7 | 10.52 | 17.33 | 23.0 | 0.61520 |
| 13.07 | 25 | 29.5 | 10.27 | 17.17 | 23.2 | 0.60058 |
| 13.07 | 30 | 29.7 | 9.95 | 17.12 | 23.2 | 0.58187 |
| 13.07 | 35 | 30.7 | 10.39 | 16.94 | 23.2 | 0.60760 |

**Table B-8 Data from Testing 30-Weight Oil at 13.1 kHz with Medium Horn Spacing**

30-Weight Oil:

Nominal Experimental Conditions:

Acoustic Frequency: 13.1 kHz
Horn Design: Medium Spacing

Initial Drainage Times, (sec): 18.08 T=22.7°C
17.47 (No.5 Cup)
18.09
Mean: 17.88
Standard Deviation: 0.36

| Acoustic Frequency Measured, (kHz) | Treatment Time, (min) | Oil Temperature (°C) | Drainage Time (sec) | Drainage Time After Cooling, (sec) | Temperature After Cooling, (°C) | Fractional Residual Viscosity |
|---|---|---|---|---|---|---|
| 13.08 | 0 | 22.7 | 17.88 | 17.91 | 22.6 | 1 |
| 13.08 | 5 | 24.2 | 13.4 | 17.93 | 22.7 | 0.74944 |
| 13.08 | 10 | 25.1 | 9.46 | 18.33 | 22.7 | 0.52908 |
| 13.08 | 15 | 26.0 | 8.02 | 17.10 | 22.6 | 0.44855 |
| 13.08 | 20 | 25.2 | 8.28 | 18.14 | 22.6 | 0.46309 |
| 13.08 | 25 | 25.2 | 8.84 | 17.35 | 22.6 | 0.49441 |
| 13.08 | 30 | 28.6 | 8.57 | 18.19 | 22.7 | 0.47931 |
| 13.08 | 35 | 27.9 | 8.92 | 16.70 | 22.7 | 0.49888 |
| 13.08 | 40 | 29.1 | 9.81 | 17.14 | 22.7 | 0.54866 |

**Table B-9 Data from Testing 30-Weight Oil at 13.1 kHz with Large Horn Spacing**

30-Weight Oil:

Nominal Experimental Conditions:

Acoustic Frequency: 13.1 kHz
Horn Design: Large Spacing

Initial Drainage Times, (sec): 17.60, 17.52, 17.77, 17.49 — T=22.6°C (No.5 Cup)
Mean: 17.59
Standard Deviation: 0.13

| Acoustic Frequency Measured, (kHz) | Treatment Time, (min) | Oil Temperature (°C) | Drainage Time (sec) | Drainage Time After Cooling, (sec) | Temperature After Cooling, (°C) | Fractional Residual Viscosity |
|---|---|---|---|---|---|---|
| 13.07 | 0 | 22.6 | 17.59 | 17.76 | 22.7 | 1 |
| 13.07 | 5 | 22.5 | 13.98 | 17.39 | 22.7 | 0.79454 |
| 13.07 | 10 | 24.5 | 11.56 | 17.54 | 22.7 | 0.65700 |
| 13.07 | 15 | 27.9 | 10.24 | 17.49 | 22.8 | 0.58198 |
| 13.07 | 20 | 29.2 | 8.88 | 17.56 | 22.7 | 0.50469 |
| 13.07 | 25 | 26.9 | 8.97 | 17.57 | 22.8 | 0.50980 |
| 13.07 | 30 | 30.3 | 8.45 | 17.79 | 22.2 | 0.48025 |
| 13.07 | 35 | 28.7 | 8.49 | 16.33 | 22.6 | 0.48252 |
| 13.07 | 40 | 33.4 | 8.42 | 17.19 | 22.7 | 0.47855 |

**Table B-10 Data from Testing 90-Weight Oil at 1.8 kHz with Small Horn Spacing**

90-Weight Oil:

Nominal Experimental Conditions:

Acoustic Frequency: 1.8 kHz
Horn Design: Small Spacing

Initial Drainage Times, (sec): 39.35, 40.22, 40.71, 40.10, 40.77
Mean: 40.23
Standard Deviation: 0.57

T=22.3°C (No.5 Cup)

| Acoustic Frequency Measured, (kHz) | Treatment Time, (min) | Oil Temperature (°C) | Drainage Time (sec) | Drainage Time After Cooling, (sec) | Temperature After Cooling, (°C) | Fractional Residual Viscosity |
|---|---|---|---|---|---|---|
| 1.76 | 0 | 22.3 | 40.23 | 51.27 | 20.7 | 1 |
| 1.76 | 5 | 27.5 | 25.72 | 51.70 | 20.8 | 0.6393 |
| 1.76 | 10 | 34.6 | 14.19 | 50.42 | 20.9 | 0.3527 |
| 1.76 | 15 | 39.5 | 10.29 | 51.61 | 20.9 | 0.2558 |
| 1.76 | 20 | 41.7 | 10.38 | 47.03 | 21.5 | 0.2580 |
| 1.76 | 25 | 44.6 | 9.94 | 48.46 | 21.0 | 0.2471 |
| 1.76 | 30 | 48.1 | 9.48 | 47.48 | 20.9 | 0.2356 |
| 1.76 | 35 | 47.6 | 9.49 | 47.76 | 21.1 | 0.2359 |
| 1.76 | 40 | 43.6 | 10.94 | 50.30 | 21.0 | 0.2719 |

**Table B-11 Data from Testing 90-Weight Oil at 1.8 kHz with Medium Horn Spacing**

90-Weight Oil:

Nominal Experimental Conditions:

Acoustic Frequency: 1.8 kHz
Horn Design: Medium Spacing

Initial Drainage Times, (sec): 36.98 T=22.7°C
38.15 (No.5 Cup)
37.77
Mean: 37.63
Standard Deviation: 0.60

| Acoustic Frequency Measured, (kHz) | Treatment Time, (min) | Oil Temperature (°C) | Drainage Time (sec) | Drainage Time After Cooling, (sec) | Temperature After Cooling, (°C) | Fractional Residual Viscosity |
|---|---|---|---|---|---|---|
| 1.74 | 0 | 22.7 | 37.63 | 46.49 | 19.9 | 1 |
| 1.74 | 5 | 24.3 | 23.19 | 45.09 | 19.7 | 0.6162 |
| 1.74 | 10 | 25.1 | 12.26 | 40.66 | 20.0 | 0.3258 |
| 1.74 | 15 | 29.1 | 11.30 | 43.43 | 19.9 | 0.3003 |
| 1.74 | 19 | 40.2 | 10.31 | 45.21 | 19.9 | 0.2740 |
| 1.74 | 25 | 39.1 | 10.12 | 44.17 | 19.9 | 0.2689 |

**Table B-12 Data from Testing 90-Weight Oil at 1.8 kHz with Large Horn Spacing**

90-Weight Oil:

Nominal Experimental Conditions:

Acoustic Frequency: 1.8 kHz
Horn Design: Large Spacing

Initial Drainage Times, (sec): 40.11, 44.52, 37.66, 40.14, 41.16 (T=21.1°C, No.5 Cup)

Mean: 40.72
Standard Deviation: 2.49

| Acoustic Frequency Measured, (kHz) | Treatment Time, (min) | Oil Temperature (°C) | Drainage Time (sec) | Drainage Time After Cooling, (sec) | Temperature After Cooling, (°C) | Fractional Residual Viscosity |
|---|---|---|---|---|---|---|
| 1.76 | 0 | 21.1 | 40.72 | 47.37 | 20.7 | 1 |
| 1.76 | 5 | 21.7 | 25.67 | 39.06 | 20.8 | 0.6304 |
| 1.76 | 10 | 23.2 | 14.60 | 40.25 | 20.7 | 0.3586 |
| 1.76 | 15 | 30.3 | 11.38 | 42.53 | 20.7 | 0.2795 |
| 1.76 | 20 | 31.6 | 10.62 | 39.37 | 20.6 | 0.2608 |
| 1.76 | 25 | 35.2 | 11.43 | 38.62 | 20.7 | 0.2807 |
| 1.76 | 30 | 34.3 | 11.14 | 41.57 | 20.6 | 0.2736 |

**Table B-13 Data from Testing 90-Weight Oil at 3.1 kHz with Small Horn Spacing**

90-Weight Oil:

Nominal Experimental Conditions:

Acoustic Frequency: 3.1 kHz
Horn Design: Small Spacing

Initial Drainage Times, (sec): 51.36, 51.04, 50.10, 51.49, 51.93 (T=21.0°C, No.5 Cup)
Mean: 51.18
Standard Deviation: 0.68

| Acoustic Frequency Measured, (kHz) | Treatment Time, (min) | Oil Temperature (°C) | Drainage Time (sec) | Drainage Time After Cooling, (sec) | Temperature After Cooling, (°C) | Fractional Residual Viscosity |
|---|---|---|---|---|---|---|
| 3.10 | 0 | 21.0 | 51.18 | 51.59 | 21.5 | 1 |
| 3.10 | 5 | 28.9 | 22.07 | 51.23 | 21.6 | 0.4312 |
| 3.10 | 10 | 36.4 | 15.37 | 50.39 | 21.7 | 0.3003 |
| 3.10 | 15 | 39.1 | 12.73 | 49.77 | 21.7 | 0.2487 |
| 3.10 | 20 | 43.7 | 12.59 | 50.30 | 21.6 | 0.2460 |
| 3.10 | 25 | 48.2 | 11.19 | 48.76 | 22.0 | 0.2186 |
| 3.10 | 30 | 45.9 | 10.89 | 49.71 | 21.7 | 0.2128 |
| 3.10 | 35 | 46.7 | 11.37 | 49.94 | 21.7 | 0.2221 |

**Table B-14 Data from Testing 90-Weight Oil at 6.9 kHz with Small Horn Spacing**

90-Weight Oil:

Nominal Experimental Conditions:

Acoustic Frequency: 6.9 kHz
Horn Design: Small Spacing

Initial Drainage Times, (sec): 35.78 T=20.7°C
37.47 (No.5 Cup)
38.71
36.45
Mean: 37.10
Standard Deviation: 1.28

| Acoustic Frequency Measured, (kHz) | Treatment Time, (min) | Oil Temperature (°C) | Drainage Time (sec) | Drainage Time After Cooling, (sec) | Temperature After Cooling, (°C) | Fractional Residual Viscosity |
|---|---|---|---|---|---|---|
| 6.86 | 0 | 20.7 | 37.10 | 46.29 | 20.3 | 1 |
| 6.86 | 5 | 25.2 | 28.43 | 44.05 | 20.5 | 0.7663 |
| 6.86 | 10 | 26.3 | 16.68 | 41.86 | 20.0 | 0.4496 |
| 6.86 | 15 | 27.7 | 14.69 | 41.69 | 20.6 | 0.3959 |
| 6.86 | 20 | 27.2 | 13.49 | 41.03 | 20.5 | 0.3636 |
| 6.86 | 25 | 28.9 | | 39.56 | 20.9 | |
| 6.86 | 30 | 30.6 | 11.64 | 40.47 | 20.5 | 0.3137 |
| 6.86 | 40 | 30.8 | 11.65 | 43.69 | 20.4 | 0.3140 |

**Table B-15 Data from Testing 90-Weight Oil at 6.9 kHz with Small Horn Spacing Using a Water Bath**

90-Weight Oil:

Nominal Experimental Conditions:

Acoustic Frequency: 6.9 kHz
Horn Design: Small Spacing
(Performed in Water Bath)

Initial Drainage Times, (sec): 36.93 T=22.5°C
37.24 (No.5 Cup)
37.22
36.42
36.23
Mean: 36.81
Standard Deviation: 0.46

| Acoustic Frequency Measured, (kHz) | Treatment Time, (min) | Oil Temperature (°C) | Drainage Time (sec) | Drainage Time After Cooling, (sec) | Temperature After Cooling, (°C) | Fractional Residual Viscosity | Water Bath Temp., (°C) |
|---|---|---|---|---|---|---|---|
| 6.78 | 0 | 22.5 | 36.81 | 37.35 | 20.9 | 1 | 22.3 |
| 6.78 | 5 | 23.7 | 31.38 | 36.32 | 21.1 | 0.8525 | 22.1 |
| 6.78 | 10 | 24.8 | 23.71 | 36.87 | 21.2 | 0.6442 | 22.4 |
| 6.78 | 15 | 24.9 | 20.48 | 38.40 | 21.2 | 0.5564 | 22.4 |
| 6.78 | 20 | 24.7 | 21.98 | 35.81 | 21.6 | 0.5972 | 22.4 |
| 6.78 | 25 | 26.6 | | | | | 22.4 |
| 6.78 | 30 | 26.8 | 20.72 | 38.59 | 21.3 | 0.5629 | 22.3 |
| 6.78 | 35 | 26.5 | | | | | 22.3 |
| 6.78 | 40 | 27.9 | 18.15 | 35.23 | 21.5 | 0.4931 | 22.3 |

**Table B-16 Data from Testing 90-Weight Oil at 6.9 kHz with Medium Horn Spacing**

90-Weight Oil:

Nominal Experimental Conditions:

Acoustic Frequency: 6.9 kHz
Horn Design: Medium Spacing

Initial Drainage Times, (sec): 45.95, 44.54, 45.13, 45.91 — T=22.5°C (No.5 Cup)
Mean: 45.38
Standard Deviation: 0.68

| Acoustic Frequency Measured, (kHz) | Treatment Time, (min) | Oil Temperature (°C) | Drainage Time (sec) | Drainage Time After Cooling, (sec) | Temperature After Cooling, (°C) | Fractional Residual Viscosity |
|---|---|---|---|---|---|---|
| 6.81 | 0 | 21.1 | 45.38 | 48.50 | 20.7 | 1 |
| 6.81 | 5 | 20.3 | 44.07 | 37.94 | 20.5 | 0.9711 |
| 6.66 | 10 | 23.2 | 36.99 | 37.13 | 20.1 | 0.8151 |
| 6.66 | 15 | 24.3 | 25.31 | 35.89 | 19.9 | 0.5577 |
| 6.66 | 20 | 25.4 | 20.07 | 39.67 | 19.9 | 0.4422 |
| 6.66 | 30 | 26.4 | 15.77 | 43.80 | 20.2 | 0.3475 |
| 6.66 | 40 | 31.1 | 14.20 | 38.49 | 19.9 | 0.3129 |
| 6.66 | 50 | 27.8 | 12.01 | 35.50 | 20.3 | 0.2646 |
| 6.66 | 60 | 34.5 | 11.49 | 35.42 | 20.2 | 0.2532 |

**Table B-17 Data from Testing 90-Weight Oil at 6.9 kHz with Medium Horn Spacing (Duplicate Experiment with that Summarized in Table B-16)**

90-Weight Oil:

Nominal Experimental Conditions:

Acoustic Frequency: 6.9 kHz
Horn Design: Medium Spacing
(Duplicate Experiment)

Initial Drainage Times, (sec): 40.61, 41.76, 39.62, 41.36, 39.84, 40.52
Mean: 40.62
Standard Deviation: 0.83

T=21.3°C
(No.5 Cup)

| Acoustic Frequency Measured, (kHz) | Treatment Time, (min) | Oil Temperature (°C) | Drainage Time (sec) | Drainage Time After Cooling, (sec) | Temperature After Cooling, (°C) | Fractional Residual Viscosity |
|---|---|---|---|---|---|---|
| 6.78 | 0 | 21.3 | 40.62 | 38.68 | 21.2 | 1 |
| 6.78 | 5 | 21.6 | 35.77 | 35.21 | 20.7 | 0.8806 |
| 6.78 | 10 | 23.1 | 24.89 | 38.95 | 21.0 | 0.6128 |
| 6.78 | 15 | 24.5 | 22.78 | 40.31 | 21.5 | 0.5608 |
| 6.78 | 20 | 24.4 | 20.18 | 37.19 | 20.9 | 0.4968 |
| 6.78 | 25 | 31.1 | 19.10 | 36.13 | 21.5 | 0.4702 |
| 6.78 | 30 | 27.0 | 19.25 | 40.35 | 21.6 | 0.4739 |
| 6.78 | 35 | 27.6 | 17.67 | 37.65 | 21.7 | 0.4350 |
| 6.78 | 40 | 29.8 | 16.77 | 36.05 | 21.4 | 0.4129 |
| 6.78 | 45 | 29.5 | 17.19 | 38.78 | 21.5 | 0.4232 |
| 6.78 | 50 | 31.1 | 16.19 | 38.43 | 21.5 | 0.3986 |

**Table B-18 Data from Testing 90-Weight Oil at 6.9 kHz with Large Horn Spacing**

90-Weight Oil:

Nominal Experimental Conditions:

Acoustic Frequency: 6.9 kHz
Horn Design: Large Spacing

Initial Drainage Times, (sec): 57.93, 57.72, 58.18, 56.57, 56.36, 58.64
Mean: 57.57
Standard Deviation: 0.91

T=21.4°C (No.5 Cup)

| Acoustic Frequency Measured, (kHz) | Treatment Time, (min) | Oil Temperature (°C) | Drainage Time (sec) | Drainage Time After Cooling, (sec) | Temperature After Cooling, (°C) | Fractional Residual Viscosity |
|---|---|---|---|---|---|---|
| 6.84 | 0 | 21.4 | 57.57 | 60.66 | 21.3 | 1 |
| 6.84 | 5 | 22.9 | 48.63 | 57.83 | 21.6 | 0.8448 |
| 6.84 | 10 | 27.3 | 36.13 | 56.93 | 21.5 | 0.6276 |
| 6.84 | 15 | 28.5 | 27.09 | 61.19 | 21.4 | 0.4706 |
| 6.84 | 20 | 31.9 | 27.13 | 57.57 | 21.3 | 0.4713 |
| 6.84 | 25 | 30.3 | 24.81 | 56.97 | 21.5 | 0.4310 |
| 6.84 | 30 | 33.5 | 21.59 | 55.27 | 21.7 | 0.3750 |
| 6.84 | 35 | 31.5 | 26.75 | 57.46 | 21.6 | 0.4647 |
| 6.84 | 39.5 | 34.3 | 25.86 | 56.29 | 21.7 | 0.4492 |
| 6.84 | 44 | 31.8 | 26.65 | 57.01 | 21.5 | 0.4629 |

**Table B-19 Data from Testing 90-Weight Oil at 6.9 kHz with Large Horn Spacing Using a Water Bath**

90-Weight Oil:

Nominal Experimental Conditions:

Acoustic Frequency: 6.9 kHz
Horn Design: Large Spacing
(Performed in Water Bath)

Initial Drainage Times, (sec): 48.50 T=20.8°C
47.78 (No.5 Cup)
47.99
48.19
48.14
Mean: 48.12
Standard Deviation: 0.27

| Acoustic Frequency Measured, (kHz) | Treatment Time, (min) | Oil Temperature (°C) | Drainage Time (sec) | Drainage Time After Cooling, (sec) | Temperature After Cooling, (°C) | Fractional Residual Viscosity | Water Bath Temp., (°C) |
|---|---|---|---|---|---|---|---|
| 6.88 | 0 | 21.2 | 48.12 | 44.15 | 21.7 | 1 | 21.8 |
| 6.88 | 5 | 23.4 | 37.07 | 44.19 | 21.5 | 0.7704 | 22.1 |
| 6.88 | 10 | 27.6 | 29.91 | 43.97 | 21.6 | 0.6216 | 22.0 |
| 6.88 | 15 | 29.3 | 25.30 | 41.26 | 22.1 | 0.5258 | 22.0 |
| 6.88 | 20 | 28.6 | 22.60 | 43.67 | 21.8 | 0.4697 | 21.9 |
| 6.88 | 25 | 29.5 | 23.06 | 42.44 | 21.8 | 0.4792 | 22.0 |
| 6.88 | 30 | 31.0 | 22.77 | 43.28 | 22.0 | 0.4732 | 21.9 |
| 6.88 | 35 | 31.9 | 22.43 | 44.23 | 21.8 | 0.4661 | 21.9 |
| 6.90 | 40 | 31.9 | 21.38 | 42.68 | 21.9 | 0.4443 | 21.9 |
| 6.90 | 45 | 28.7 | 22.03 | 42.92 | 21.9 | 0.4578 | 21.9 |
| 6.90 | 50 | 31.5 | 21.93 | 42.12 | 21.9 | 0.4557 | 21.8 |

**Table B-20 Data from Testing 90-Weight Oil at 13.1 kHz with Small Horn Spacing**

90-Weight Oil:

Nominal Experimental Conditions:

Acoustic Frequency: 13.1 kHz
Horn Design: Small Spacing

Initial Drainage Times, (sec): 38.56, 38.40, 37.43, 39.73, 38.53 (T=21.5°C, No.5 Cup)

Mean: 38.53
Standard Deviation: 0.82

| Acoustic Frequency Measured, (kHz) | Treatment Time, (min) | Oil Temperature (°C) | Drainage Time (sec) | Drainage Time After Cooling, (sec) | Temperature After Cooling, (°C) | Fractional Residual Viscosity |
|---|---|---|---|---|---|---|
| 13.08 | 0 | 21.5 | 38.53 | 37.14 | 22.3 | 1 |
| 13.08 | 5 | 22.0 | 34.59 | 37.14 | 21.7 | 0.8977 |
| 13.08 | 10 | 23.8 | 25.19 | 35.95 | 22.0 | 0.6538 |
| 13.08 | 15 | 25.8 | 20.42 | 33.05 | 22.5 | 0.5300 |
| 13.08 | 20 | 26.3 | 20.84 | 36.49 | 23.1 | 0.5409 |
| 13.08 | 25 | 29.8 | 19.07 | 37.79 | 22.9 | 0.4949 |
| 13.08 | 30 | 32.3 | 18.58 | 35.80 | 22.9 | 0.4822 |
| 13.08 | 35 | 30.4 | 19.29 | 36.39 | 22.6 | 0.5006 |
| 13.08 | 40 | 32.5 | 17.435 | 36.49 | 24.3 | 0.4525 |
| 13.08 | 45 | 33.1 | 17.49 | 36.13 | 22.7 | 0.4539 |

**Table B-21 Data from Testing 90-Weight Oil at 13.1 kHz with Medium Horn Spacing and Reduced Power Input**

90-Weight Oil:

Nominal Experimental Conditions:

Acoustic Frequency: 13.1 kHz
Horn Design: Medium Spacing

71.7% current; 89.8 volts

Initial Drainage Times, (sec): 38.59, 37.97, 38.80, 38.90, 38.66, 37.54 — T=22.5°C (No.5 Cup)

Mean: 38.41
Standard Deviation: 0.54

| Acoustic Frequency Measured, (kHz) | Treatment Time, (min) | Oil Temperature (°C) | Drainage Time (sec) | Drainage Time After Cooling, (sec) | Temperature After Cooling, (°C) | Fractional Residual Viscosity |
|---|---|---|---|---|---|---|
| 13.07 | 0 | 22.5 | 38.41 | 45.13 | 20.7 | 1 |
| 13.07 | 5 | 23.1 | 33.29 | 40.26 | 20.7 | 0.8667 |
| 13.07 | 10 | 27.3 | 24.54 | 45.17 | 21.5 | 0.6389 |
| 13.07 | 15 | 32.6 | 22.35 | 45.07 | 20.6 | 0.5819 |
| 13.07 | 20 | 30.3 | 19.39 | 45.03 | 20.5 | 0.5048 |
| 13.07 | 25 | 35.7 | 18.31 | 46.05 | 20.4 | 0.4767 |
| 13.07 | 30 | 38.7 | 16.16 | 46.30 | 20.7 | 0.4207 |
| 13.07 | 35 | 41.1 | 14.75 | 46.33 | 20.7 | 0.3840 |

**Table B-22 Data from Testing 90-Weight Oil at 13.1 kHz with Large Horn Spacing**

90-Weight Oil:

Nominal Experimental Conditions:

Acoustic Frequency: 13.1 kHz
Horn Design: Large Spacing

Initial Drainage Times, (sec): 44.83, 44.44, 43.87, 44.12, 44.77, 44.87 (T=21.5°C, No.5 Cup)

Mean: 44.48
Standard Deviation: 0.42

| Acoustic Frequency Measured, (kHz) | Treatment Time, (min) | Oil Temperature (°C) | Drainage Time (sec) | Drainage Time After Cooling, (sec) | Temperature After Cooling, (°C) | Fractional Residual Viscosity |
|---|---|---|---|---|---|---|
| 13.05 | 0 | 21.1 | 44.48 | 38.79 | 22.7 | 1 |
| 13.06 | 5 | 22.7 | 35.01 | 40.07 | 22.5 | 0.7870 |
| 13.06 | 10 | 27.5 | 29.77 | 40.37 | 22.3 | 0.6692 |
| 13.06 | 15 | 29.7 | 25.75 | 40.84 | 22.6 | 0.5789 |
| 13.06 | 20 | 30.3 | 24.2 | 37.73 | 22.9 | 0.5440 |
| 13.06 | 25 | 31.5 | 21.23 | 39.54 | 22.6 | 0.4773 |
| 13.06 | 30 | 35.2 | 19.73 | 40.62 | 22.4 | 0.4435 |
| 13.06 | 35 | 34.7 | 17.3 | 40.49 | 22.5 | 0.3889 |
| 13.06 | 40 | 37.4 | 17.19 | 38.59 | 23.1 | 0.3864 |
| 13.06 | 45 | 37.4 | 16.88 | 39.68 | 22.7 | 0.3795 |
| 13.06 | 50 | 38.7 | 14.01 | 40.59 | 22.4 | 0.3149 |
| 13.06 | 55 | 38.5 | | | | |
| 13.06 | 60 | 35.7 | 14.97 | 40.48 | 22.6 | 0.3365 |

**Table B-23 Data from Testing 140-Weight Oil at 1.8 kHz with Small Horn Spacing**

140-Weight Oil:

Nominal Experimental Conditions:

Acoustic Frequency: 1.8 kHz
Horn Design: Small Spacing

Initial Drainage Times, (sec): 46.98 T=21.2°C
44.62 (No.5 Cup)
42.58
44.87
Mean: 44.76
Standard Deviation: 1.80

| Acoustic Frequency Measured, (kHz) | Treatment Time, (min) | Oil Temperature (°C) | Drainage Time (sec) | Drainage Time After Cooling, (sec) | Temperature After Cooling, (°C) | Fractional Residual Viscosity |
|---|---|---|---|---|---|---|
| 1.75 | 0 | 21.2 | 44.76 | --- | --- | 1 |
| 1.75 | 5 | 22.6 | 38.19 | 36.47 | 20.1 | 0.8532 |
| 1.75 | 10 | 24.6 | 26.83 | 35.73 | 20.3 | 0.5994 |
| 1.75 | 15 | 25.5 | 24.19 | 35.43 | 20.9 | 0.5404 |
| 1.75 | 20 | 25.0 | 21.15 | 40.53 | 21.1 | 0.4725 |
| 1.75 | 25 | 26.3 | 15.04 | 37.63 | 21.5 | 0.3360 |
| 1.75 | 30 | 26.2 | 14.99 | 36.30 | 21.7 | 0.3349 |

**Table B-24 Data from Testing 140-Weight Oil at 1.8 kHz with Medium Horn Spacing Using a Water Bath**

140-Weight Oil:

Nominal Experimental Conditions:

Acoustic Frequency: 1.8 kHz
Horn Design: Medium Spacing
(Performed in a water bath)

Initial Drainage Times, (sec): 36.59, 37.09, 37.34, 37.59, 37.11
Mean: 37.14
Standard Deviation: 0.37

T=22.0°C
(No.5 Cup)

| Acoustic Frequency Measured, (kHz) | Treatment Time, (min) | Oil Temperature (°C) | Drainage Time (sec) | Drainage Time After Cooling, (sec) | Temperature After Cooling, (°C) | Fractional Residual Viscosity | Water Bath Temp., (°C) |
|---|---|---|---|---|---|---|---|
| 1.767 | 0 | 22.1 | 37.14 | 46.36 | 20.7 | 1 | 22.4 |
| 1.767 | 5 | 22.3 | 35.72 | 37.13 | 20.7 | 0.9617 | 22.0 |
| 1.767 | 10 | 23.0 | 25.06 | 36.76 | 20.9 | 0.6747 | 22.0 |
| 1.767 | 15 | 23.3 | 21.36 | 36.6 | 21.1 | 0.5751 | 22.0 |
| 1.767 | 20 | 22.8 | 17.33 | 37.9 | 20.9 | 0.4666 | 22.2 |
| 1.767 | 25 | 23.5 | | --- | --- | | 22.3 |
| 1.767 | 30 | 24.2 | 18.22 | 35.79 | 20.9 | 0.4905 | 22.2 |
| 1.767 | 35 | 25.5 | 19.56 | 35.19 | 21.1 | 0.5266 | 22.3 |
| 1.767 | 41 | 25.4 | 18.43 | 38.28 | 20.7 | 0.4962 | 22.4 |

**Table B-25 Data from Testing 140-Weight Oil at 1.8 kHz with Large Horn Spacing**

140-Weight Oil:

Nominal Experimental Conditions:

Acoustic Frequency: 1.8 kHz
Horn Design: Large Spacing

Initial Drainage Times, (sec): 35.26, 34.09, 34.59
Mean: 34.65
Standard Deviation: 0.59

T=22.4°C (No.5 Cup)

| Acoustic Frequency Measured, (kHz) | Treatment Time, (min) | Oil Temperature (°C) | Drainage Time (sec) | Drainage Time After Cooling, (sec) | Temperature After Cooling, (°C) | Fractional Residual Viscosity |
|---|---|---|---|---|---|---|
| 1.72 | 0 | 22.2 | 34.65 | 45.11 | 20.3 | 1 |
| 1.72 | 5 | 24.1 | 29.26 | 46.29 | 20.7 | 0.8445 |
| 1.72 | 10 | 29.4 | 19.30 | 41.61 | 20.3 | 0.5571 |
| 1.72 | 15 | 27.5 | 18.82 | 40.17 | 20.3 | 0.5432 |
| 1.72 | 20 | 28.6 | 15.82 | 41.89 | 20.2 | 0.4566 |
| 1.72 | 25 | 29.8 | 14.87 | 40.26 | 20.3 | 0.4292 |
| 1.72 | 30 | 31.7 | 13.78 | 39.99 | 20.1 | 0.3977 |
| 1.72 | 35 | 32.5 | 13.53 | 40.92 | 20.5 | 0.3905 |
| 1.72 | 40 | 35.1 | 14.29 | 40.70 | 20.1 | 0.4124 |

**Table B-26 Data from Testing 140-Weight Oil at 3.1 kHz with Small Horn Spacing**

140-Weight Oil:

Nominal Experimental Conditions:

Acoustic Frequency: 3.1 kHz
Horn Design: Small Spacing

Initial Drainage Times, (sec): 38.75, 40.74, 39.50, 40.38
Mean: 39.84
Standard Deviation: 0.90

T=22.4°C (No.5 Cup)

| Acoustic Frequency Measured, (kHz) | Treatment Time, (min) | Oil Temperature (°C) | Drainage Time (sec) | Drainage Time After Cooling, (sec) | Temperature After Cooling, (°C) | Fractional Residual Viscosity |
|---|---|---|---|---|---|---|
| 3.09 | 0 | 20.9 | 39.84 | 38.72 | 20.4 | 1 |
| 3.09 | 5 | 22.3 | 26.11 | 36.90 | 19.9 | 0.6553 |
| 3.09 | 9 | 24.1 | 21.3 | 37.91 | 20.1 | 0.5346 |
| 3.09 | 15 | 23.8 | 25.15 | 34.81 | 20.3 | 0.6312 |
| 3.09 | 15 | 23.8 | 25.5 | --- | --- | 0.6400 |
| 3.09 | 20 | 24.4 | 22.09 | 37.66 | 19.9 | 0.5544 |
| 3.09 | 25 | 27.9 | 26.04 | 39.04 | 20.3 | 0.6536 |
| 3.09 | 25 | 27.9 | 25.63 | --- | --- | 0.6433 |

**Table B-27 Data from Testing 140-Weight Oil at 6.9 kHz with Small Horn Spacing**

140-Weight Oil:

Nominal Experimental Conditions:

Acoustic Frequency: 6.9 kHz
Horn Design: Small Spacing

Initial Drainage Times, (sec): 40.43, 40.02, 36.01, 35.65, 38.33 — T=22.8°C (No.5 Cup)

Mean: 38.09
Standard Deviation: 2.21

| Acoustic Frequency Measured, (kHz) | Treatment Time, (min) | Oil Temperature (°C) | Drainage Time (sec) | Drainage Time After Cooling, (sec) | Temperature After Cooling, (°C) | Fractional Residual Viscosity |
|---|---|---|---|---|---|---|
| 6.78 | 0 | 22.8 | 38.09 | 38.44 | 20.8 | 1 |
| 6.78 | 5 | 23.6 | | 37.71 | 20.4 | |
| 6.78 | 10 | 25.4 | 29.14 | 38.93 | 20.8 | 0.7651 |
| 6.87 | 15 | 28.8 | | --- | --- | |
| 6.87 | 20 | 27.2 | 28.28 | 40.00 | 20.7 | 0.7425 |
| 6.92 | 25 | 28.5 | | 40.53 | 20.7 | |
| 6.92 | 30 | 30.5 | 25.49 | 41.47 | 20.8 | 0.6692 |
| 6.92 | 35 | 28.3 | | --- | --- | |
| 6.92 | 40 | 30.9 | | --- | --- | |
| 6.92 | 45 | 28.7 | 25.20 | --- | --- | 0.6616 |

**Table B-28 Data from Testing 140-Weight Oil at 6.9 kHz with Medium Horn Spacing**

140-Weight Oil:

Nominal Experimental Conditions:

Acoustic Frequency: 6.9 kHz
Horn Design: Medium Spacing

Initial Drainage Times, (sec): 39.03, 41.19, 40.23, 41.16 — T=20.5°C (No.5 Cup)
Mean: 40.40
Standard Deviation: 1.02

| Acoustic Frequency Measured, (kHz) | Treatment Time, (min) | Oil Temperature (°C) | Drainage Time (sec) | Drainage Time After Cooling, (sec) | Temperature After Cooling, (°C) | Fractional Residual Viscosity |
|---|---|---|---|---|---|---|
| 6.93 | 0 | 20.5 | 40.40 | --- | --- | 1 |
| 6.93 | 5 | 21.5 | 35.73 | 40.67 | 20.8 | 0.8844 |
| 6.93 | 10 | 24.5 | 30.70 | 37.76 | 19.5 | 0.7599 |
| 6.93 | 15 | 26.8 | | --- | --- | |
| 6.93 | 20 | 26.9 | 27.59 | 37.07 | 19.7 | 0.6829 |
| 6.93 | 25 | 25.7 | | --- | --- | |
| 6.93 | 30 | 26.5 | 25.36 | 37.03 | 20.0 | 0.6277 |
| 6.93 | 35 | 26.3 | | --- | --- | |
| 6.93 | 40 | 29.5 | | --- | --- | |
| 6.93 | 45 | 31.1 | 25.46 | 38.24 | 20.5 | 0.6302 |
| 6.93 | 50 | 27.9 | | --- | --- | |
| 6.93 | 55 | 28.1 | | --- | --- | |
| 6.93 | 60 | 29.1 | 25.53 | 39.80 | 20.1 | 0.6319 |

**Table B-29 Data from Testing 140-Weight Oil at 6.9 kHz with Medium Horn Spacing (Continuation of Test Summarized in Table 28)**

140-Weight Oil:

Nominal Experimental Conditions:

Acoustic Frequency: 6.9 kHz
Horn Design: Medium Spacing

Initial Drainage Times, (sec): 39.03, 41.19, 40.23, 41.16
Mean: 40.40
Standard Deviation 1.02

T=20.5°C (No.5 Cup)

| Acoustic Frequency Measured, (kHz) | Treatment Time, (min) | Oil Temperature (°C) | Drainage Time (sec) | Drainage Time After Cooling, (sec) | Temperature After Cooling, (°C) | Fractional Residual Viscosity |
|---|---|---|---|---|---|---|
| 6.74 | 0 | 22.0 | 41.97 | 43 | 21.5 | 1 |
| 6.74 | 5 | 22.5 | 37.89 | 35.7 | 21.5 | 0.9028 |
| 6.74 | 10 | 22.2 | 39.37 | 34.38 | 20.7 | 0.9381 |
| 6.83 | 15 | 22.5 | 29.81 | 37.06 | 21.4 | 0.7103 |
| 6.83 | 20 | 24.3 | 30.53 | 32.97 | 22.1 | 0.7274 |
| 6.84 | 27 | 25.6 | 28.36 | 36.91 | 21.6 | 0.6757 |
| 6.84 | 32 | 25.8 | 28.71 | 33.38 | 21.6 | 0.6841 |

**Table B-30 Data from Testing 140-Weight Oil at 6.9 kHz with Large Horn Spacing**

140-Weight Oil:

Nominal Experimental Conditions:

Acoustic Frequency: 6.9 kHz
Horn Design: Large Spacing

Initial Drainage Times, (sec): 45.31 T=20.7°C
46.06 (No.5 Cup)
43.81
43.39
44.35
Mean: 44.58
Standard Deviation: 1.09

| Acoustic Frequency Measured, (kHz) | Treatment Time, (min) | Oil Temperature (°C) | Drainage Time (sec) | Drainage Time After Cooling, (sec) | Temperature After Cooling, (°C) | Fractional Residual Viscosity |
|---|---|---|---|---|---|---|
| 6.88 | 0 | 20.7 | 44.58 | 41.19 | 20.4 | 1 |
| 6.88 | 5 | 21.0 | 38.39 | 39.47 | 20.1 | 0.8611 |
| 6.88 | 10 | 21.6 | 33.10 | 39.28 | 19.9 | 0.7424 |
| 6.88 | 15 | 23.1 | 26.96 | 38.45 | 20.5 | 0.6047 |
| 6.88 | 20 | 23.0 | 25.47 | 39.58 | 20.3 | 0.5713 |
| 6.88 | 30 | 26.0 | 19.38 | 43.93 | 20.4 | 0.4347 |
| 6.88 | 39 | 23.5 | 17.80 | 39.09 | 20.5 | 0.3992 |

**Table B-31 Data from Testing 140-Weight Oil at 13.1 kHz with Small Horn Spacing with Reduced Power Input**

140-Weight Oil:

Nominal Experimental Conditions:

Acoustic Frequency: 13.1 kHz
Horn Design: Small Spacing

71% current output; 89 volts

Initial Drainage Times, (sec): 42.75, 42.73, 42.81, 41.91, 42.81 (T=20.5°C, No.5 Viscometer)

Mean: 42.60
Standard Deviation: 0.34

| Acoustic Frequency Measured, (kHz) | Treatment Time, (min) | Oil Temperature (°C) | Drainage Time (sec) | Drainage Time After Cooling, (sec) | Temperature After Cooling, (°C) | Fractional Residual Viscosity |
|---|---|---|---|---|---|---|
| 13.06 | 0 | 20.5 | 46.60 | 44.09 | 20.5 | 1 |
| 13.06 | 5 | 21.5 | 35.57 | 45.09 | 20.5 | 0.7633 |
| 13.09 | 10 | 26.4 | 29.01 | 45.87 | 20.4 | 0.6225 |
| 13.09 | 14 | 22.8 | 30.17 | 46.39 | 20.3 | 0.6474 |
| 13.09 | 18 | 26.8 | 27.81 | 46.27 | 20.3 | 0.5968 |
| 13.09 | 22 | 25.1 | 31.52 | 44.29 | 20.3 | 0.6764 |
| 13.09 | 26 | 26.1 | 28.69 | 44.94 | 20.4 | 0.6156 |
| 13.09 | 29 | 26.6 | 31.60 | 45.04 | 20.2 | 0.6781 |

**Table B-32 Data from Testing 140-Weight Oil at 13.1 kHz with Small Horn Spacing with Reduced Power Input (Duplicate of Test Summarized in Table B-30)**

140-Weight Oil:

Nominal Experimental Conditions:

Acoustic Frequency: 13.1 kHz
Horn Design: Small Spacing

71% current output; 89 volts
(Duplicate Experiment)

Initial Drainage Times, (sec): 45.70, 46.30, 45.14, 45.76, 44.37 — T=20.2°C (No.5 Cup)

Mean: 45.45
Standard Deviation: 0.73

| Acoustic Frequency Measured, (kHz) | Treatment Time, (min) | Oil Temperature (°C) | Drainage Time (sec) | Drainage Time After Cooling, (sec) | Temperature After Cooling, (°C) | Fractional Residual Viscosity |
|---|---|---|---|---|---|---|
| 13.05 | 0 | 20.2 | 45.45 | 45.43 | 21.3 | 1 |
| 13.05 | 5 | 21.0 | 40.83 | 43.02 | 21.1 | 0.8983 |
| 13.05 | 10 | 21.8 | 35.91 | 44.58 | 21.2 | 0.7900 |
| 13.05 | 15 | 27.7 | 29.47 | 44.02 | 21.1 | 0.6483 |
| 13.05 | 20 | 28.7 | 24.76 | 43.78 | 21.2 | 0.5447 |
| 13.05 | 25 | 29.0 | 25.30 | 43.79 | 21.2 | 0.5566 |
| 13.05 | 30 | 29.1 | 24.74 | 46.60 | 21.1 | 0.5443 |
| 13.05 | 35 | 30.1 | 24.16 | 46.96 | 21.3 | 0.5315 |
| 13.05 | 40 | 29.5 | 24.02 | 40.19 | 21.5 | 0.5284 |
| 13.05 | 45 | 31.1 | 21.77 | 40.91 | 21.3 | 0.4789 |
| 13.05 | 50 | 31.1 | 23.34 | 43.18 | 21.2 | 0.5135 |

**Table B-33 Data from Testing 140-Weight Oil at 13.1 kHz with Medium Horn Spacing with Reduced Power Input**

140-Weight Oil:

Nominal Experimental Conditions:

Acoustic Frequency: 13.1 kHz
Horn Design: Medium Spacing

70% current output; 88 volts

Initial Drainage Times, (sec): 43.74, 44.01, 43.99, 44.00, 44.04 T=21.1°C (No.5 Cup)

Mean: 43.96
Standard Deviation: 0.12

| Acoustic Frequency Measured, (kHz) | Treatment Time, (min) | Oil Temperature (°C) | Drainage Time (sec) | Drainage Time After Cooling, (sec) | Temperature After Cooling, (°C) | Fractional Residual Viscosity |
|---|---|---|---|---|---|---|
| 13.05 | 0 | 21.1 | 43.96 | 43.33 | 21.7 | 1 |
| 13.06 | 5 | 21.0 | 38.61 | 38.16 | 22.0 | 0.8784 |
| 13.06 | 10 | 21.5 | 30.62 | 37.88 | 22.1 | 0.6966 |
| 13.06 | 15 | 22.4 | 27.68 | 41.80 | 21.9 | 0.6297 |
| 13.06 | 20 | 23.1 | 24.99 | 42.33 | 21.7 | 0.5685 |
| 13.06 | 25 | 24.3 | 23.73 | 40.03 | 21.9 | 0.5399 |
| 13.06 | 30 | 26.5 | 21.13 | 39.66 | 21.9 | 0.4807 |
| 13.06 | 35 | 28.3 | 19.09 | 37.47 | 22.0 | 0.4343 |
| 13.06 | 40 | 24.7 | 23.48 | 40.23 | 22.1 | 0.5342 |
| 13.06 | 45 | 27.9 | 22.03 | 41.31 | 22.1 | 0.5012 |
| 13.06 | 50 | 28.2 | 21.99 | 40.03 | 22.1 | 0.5003 |

**Table B-34 Data from Testing 140-Weight Oil at 13.1 kHz with Large Horn Spacing with Reduced Power Input**

140-Weight Oil:

Nominal Experimental Conditions:

Acoustic Frequency: 13.1 kHz
Horn Design: Large Spacing

70% current output

Initial Drainage Times, (sec): 42.72, 40.69, 42.43, 43.82, 42.12 (T=22.0°C, No.5 Cup)

Mean: 42.36
Standard Deviation: 1.13

| Acoustic Frequency Measured, (kHz) | Treatment Time, (min) | Oil Temperature (°C) | Drainage Time (sec) | Drainage Time After Cooling, (sec) | Temperature After Cooling, (°C) | Fractional Residual Viscosity |
|---|---|---|---|---|---|---|
| 13.05 | 0 | 22.0 | 42.36 | 47.84 | 20.1 | 1 |
| 13.05 | 5 | 23.3 | 26.17 | 45.55 | 20.2 | 0.6179 |
| 13.06 | 10 | 23.1 | 25.96 | 46.02 | 20.3 | 0.6129 |
| 13.06 | 15 | 23.3 | 22.49 | 43.92 | 20.9 | 0.5310 |
| 13.06 | 20 | 24.4 | 21.31 | 44.08 | 20.5 | 0.5031 |
| 13.06 | 25 | 23.2 | 19.06 | 44.00 | 20.5 | 0.4500 |

**Table B-35 Fractional Viscosity Effects of Heat, Sonication, and Both for 30-Weight Oil, 6.9 kHz, and Small Horn Spacing**

30-Weight Oil:

Acoustic Frequency: 6.9 kHz
Horn Design: Small Spacing

| Treatment Time, (min) | Oil Temperature ($^{o}$C) | Calculated Fractional Residual Viscosity (due to heat) | Normalized Fractional Residual Viscosity (due to heat) | Viscosity Reduction due to Sonication | Normalized Fractional Residual Viscosity due to Sonication | Overall Fractional Residual Viscosity |
|---|---|---|---|---|---|---|
| 0 | 21.7 | 0.61741 | 1 | 0 | 1 | 1 |
| 5 | 22.6 | 0.58961 | 0.95497 | 0.19700 | 0.80300 | 0.75797 |
| 10 | 21.9 | 0.61102 | 0.98965 | 0.35476 | 0.64524 | 0.63489 |
| 14.5 | 23.1 | 0.57516 | 0.93157 | 0.38180 | 0.61820 | 0.54977 |
| 19 | 23.0 | 0.57800 | 0.93617 | 0.24841 | 0.75159 | 0.68776 |
| 23 | 24.2 | 0.54561 | 0.88372 | 0.25804 | 0.74196 | 0.62568 |
| 27.5 | 24.7 | 0.53311 | 0.86346 | 0.17868 | 0.82132 | 0.68478 |
| 31.5 | 25.1 | 0.52349 | 0.84788 | 0.17070 | 0.82930 | 0.67718 |
| 35 | 24.4 | 0.54055 | 0.87551 | 0.20646 | 0.79354 | 0.66905 |

**Table B-36 Fractional Viscosity Effects of Heat, Sonication, and Both for 30-Weight Oil, 6.9 kHz, and Medium Horn Spacing**

30-Weight Oil:

Acoustic Frequency: 6.9 kHz
Horn Design: Medium Spacing

| Treatment Time, (min) | Oil Temperature ($^{o}$C) | Calculated Fractional Residual Viscosity (due to heat) | Normalized Fractional Residual Viscosity (due to heat) | Viscosity Reduction due to Sonication | Normalized Fractional Residual Viscosity due to Sonication | Overall Fractional Residual Viscosity |
|---|---|---|---|---|---|---|
| 0 | 21.5 | 0.62392 | 1 | 0 | 1 | 1 |
| 5 | 21.5 | 0.62392 | 1 | 0.11871 | 0.88129 | 0.88129 |
| 10 | 22.8 | 0.58375 | 0.93561 | 0.12716 | 0.87284 | 0.80845 |
| 15 | 22.8 | 0.58375 | 0.93561 | 0.28674 | 0.71326 | 0.64887 |
| 20 | 23.3 | 0.56957 | 0.91288 | 0.29403 | 0.70597 | 0.61885 |
| 25 | 23.8 | 0.55602 | 0.89117 | 0.30902 | 0.69098 | 0.58215 |
| 30 | 23.8 | 0.55602 | 0.89117 | 0.31847 | 0.68153 | 0.57270 |
| 35 | 24.2 | 0.54561 | 0.87449 | 0.33904 | 0.66096 | 0.53545 |
| 40 | 25.2 | 0.52113 | 0.83525 | 0.25866 | 0.74134 | 0.57659 |

**Table B-37 Fractional Viscosity Effects of Heat, Sonication, and Both for 30-Weight Oil, 6.9 kHz, and Large Horn Spacing**

30-Weight Oil:

Acoustic Frequency: 6.9 kHz
Horn Design: Large Spacing

| Treatment Time, (min) | Oil Temperature ($^{o}$C) | Calculated Fractional Residual Viscosity (due to heat) | Normalized Fractional Residual Viscosity (due to heat) | Viscosity Reduction due to Sonication | Normalized Fractional Residual Viscosity due to Sonication | Overall Fractional Residual Viscosity |
|---|---|---|---|---|---|---|
| 0 | 20.3 | 0.66590 | 1 | 0 | 1 | 1 |
| 5 | 21.3 | 0.63057 | 0.94694 | 0.12610 | 0.87390 | 0.82084 |
| 10 | 21.8 | 0.61420 | 0.92236 | 0.33404 | 0.66596 | 0.58832 |
| 15 | 22.0 | 0.60787 | 0.91286 | 0.31390 | 0.68610 | 0.59896 |
| 20 | 22.8 | 0.58375 | 0.87663 | 0.26758 | 0.73242 | 0.60905 |
| 25 | 23.0 | 0.57800 | 0.86800 | 0.22029 | 0.77971 | 0.64771 |
| 30 | 23.6 | 0.56137 | 0.84302 | 0.19811 | 0.80189 | 0.64491 |

**Table B-38 Fractional Viscosity Effects of Heat, Sonication, and Both for 30-Weight Oil, 13.1 kHz, and Small Horn Spacing**

30-Weight Oil:

Acoustic Frequency: 13.1 kHz
Horn Design: Small Spacing

| Treatment Time, (min) | Oil Temperature ($^{o}$C) | Calculated Fractional Residual Viscosity (due to heat) | Normalized Fractional Residual Viscosity (due to heat) | Viscosity Reduction due to Sonication | Normalized Fractional Residual Viscosity due to Sonication | Overall Fractional Residual Viscosity |
|---|---|---|---|---|---|---|
| 0 | 22.7 | 0.58667 | 1 | 0 | 1 | 1 |
| 5 | 28.7 | 0.44969 | 0.76652 | 0.01506 | 0.98494 | 0.75146 |
| 10 | 24.8 | 0.53067 | | | | 0.60994 |
| 15 | 28.1 | 0.46059 | 0.78511 | 0.13423 | 0.86577 | 0.65088 |
| 20 | 28.7 | 0.44969 | 0.76652 | 0.15132 | 0.84868 | 0.61520 |
| 25 | 29.5 | 0.43589 | 0.74300 | 0.14242 | 0.85758 | 0.60058 |
| 30 | 29.7 | 0.43257 | 0.73733 | 0.15546 | 0.84454 | 0.58187 |
| 35 | 30.7 | 0.41663 | 0.71016 | 0.10256 | 0.89744 | 0.60760 |

**Table B-39 Fractional Viscosity Effects of Heat, Sonication, and Both for 30-Weight Oil, 13.1 kHz, and Medium Horn Spacing**

30-Weight Oil:

Acoustic Frequency: 13.1 kHz
Horn Design: Medium Spacing

| Treatment Time, (min) | Oil Temperature ($^{o}$C) | Calculated Fractional Residual Viscosity (due to heat) | Normalized Fractional Residual Viscosity (due to heat) | Viscosity Reduction due to Sonication | Normalized Fractional Residual Viscosity due to Sonication | Overall Fractional Residual Viscosity |
|---|---|---|---|---|---|---|
| 0 | 22.7 | 0.58667 | 1 | 0 | 1 | 1 |
| 5 | 24.2 | 0.54561 | 0.93003 | 0.18059 | 0.81941 | 0.74944 |
| 10 | 25.1 | 0.52349 | 0.89231 | 0.36323 | 0.63677 | 0.52908 |
| 15 | 26.0 | 0.50299 | 0.85738 | 0.40883 | 0.59117 | 0.44855 |
| 20 | 25.2 | 0.52113 | 0.88830 | 0.42521 | 0.57479 | 0.46309 |
| 25 | 25.2 | 0.52113 | 0.88830 | 0.39389 | 0.60611 | 0.49441 |
| 30 | 28.6 | 0.45148 | 0.76956 | 0.29025 | 0.70975 | 0.47931 |
| 35 | 27.9 | 0.46434 | 0.79149 | 0.29261 | 0.70739 | 0.49888 |
| 40 | 29.1 | 0.44269 | 0.75459 | 0.20593 | 0.79407 | 0.54866 |

**Table B-40 Fractional Viscosity Effects of Heat, Sonication, and Both for 30-Weight Oil, 13.1 kHz, and Large Horn Spacing**

30-Weight Oil:

Acoustic Frequency: 13.1 kHz
Horn Design: Large Spacing

| Treatment Time, (min) | Oil Temperature ($^{o}$C) | Calculated Fractional Residual Viscosity (due to heat) | Normalized Fractional Residual Viscosity (due to heat) | Viscosity Reduction due to Sonication | Normalized Fractional Residual Viscosity due to Sonication | Overall Fractional Residual Viscosity |
|---|---|---|---|---|---|---|
| 0 | 22.6 | 0.58961 | 1 | 0 | 1 | 1 |
| 5 | 22.5 | 0.59258 | 1 | 0.20546 | 0.79454 | 0.79454 |
| 10 | 24.5 | 0.53805 | 0.91255 | 0.25555 | 0.74445 | 0.65700 |
| 15 | 27.9 | 0.46434 | 0.78754 | 0.20556 | 0.79444 | 0.58198 |
| 20 | 29.2 | 0.44097 | 0.74791 | 0.24322 | 0.75678 | 0.50469 |
| 25 | 26.9 | 0.48396 | 0.82081 | 0.31101 | 0.68899 | 0.50980 |
| 30 | 30.3 | 0.42287 | 0.71720 | 0.23695 | 0.76305 | 0.48025 |
| 35 | 28.7 | 0.44969 | 0.76270 | 0.28018 | 0.71982 | 0.48252 |
| 40 | 33.4 | 0.37866 | 0.64222 | 0.16367 | 0.83633 | 0.47855 |

**Table B-41 Fractional Viscosity Effects of Heat, Sonication, and Both for 90-Weight Oil, 1.8 kHz, and Small Horn Spacing**

90-Weight Oil:

Acoustic Frequency: 1.8 kHz
Horn Design: Small Spacing

| Treatment Time, (min) | Oil Temperature (ºC) | Calculated Fractional Residual Viscosity (due to heat) | Normalized Fractional Residual Viscosity (due to heat) | Viscosity Reduction due to Sonication | Normalized Fractional Residual Viscosity due to Sonication | Overall Fractional Residual Viscosity |
|---|---|---|---|---|---|---|
| 0 | 22.3 | 0.51083 | 1 | 0 | 1 | 1 |
| 5 | 27.5 | 0.37972 | 0.74334 | 0.10404 | 0.89596 | 0.6393 |
| 10 | 34.6 | 0.27436 | 0.53708 | 0.18438 | 0.81562 | 0.3527 |
| 15 | 39.5 | 0.22747 | 0.44529 | 0.18949 | 0.81051 | 0.2558 |
| 20 | 41.7 | 0.21067 | 0.41241 | 0.15441 | 0.84559 | 0.2580 |
| 25 | 44.6 | 0.19155 | 0.37498 | 0.12788 | 0.87212 | 0.2471 |
| 30 | 48.1 | 0.17213 | 0.33696 | 0.10136 | 0.89864 | 0.2356 |
| 35 | 47.6 | 0.17470 | 0.34198 | 0.10608 | 0.89392 | 0.2359 |
| 40 | 43.6 | 0.19780 | 0.38721 | 0.11531 | 0.88469 | 0.2719 |

**Table B-42 Fractional Viscosity Effects of Heat, Sonication, and Both for 90-Weight Oil, 1.8 kHz, and Medium Horn Spacing**

90-Weight Oil:

Acoustic Frequency: 1.7 kHz
Horn Design: Medium Spacing

| Treatment Time, (min) | Oil Temperature (ºC) | Calculated Fractional Residual Viscosity (due to heat) | Normalized Fractional Residual Viscosity (due to heat) | Viscosity Reduction due to Sonication | Normalized Fractional Residual Viscosity due to Sonication | Overall Fractional Residual Viscosity |
|---|---|---|---|---|---|---|
| 0 | 22.7 | 0.49814 | 1 | 0 | 1 | 1 |
| 5 | 24.3 | 0.45237 | 0.90811 | 0.29191 | 0.70809 | 0.6162 |
| 10 | 25.1 | 0.43210 | 0.86743 | 0.54163 | 0.45837 | 0.3258 |
| 15 | 29.1 | 0.35052 | 0.70365 | 0.40335 | 0.59665 | 0.3003 |
| 19 | 40.2 | 0.22188 | 0.44542 | 0.17142 | 0.82858 | 0.2740 |
| 25 | 39.1 | 0.23077 | 0.46326 | 0.19436 | 0.80564 | 0.2689 |

**Table B-43 Fractional Viscosity Effects of Heat, Sonication, and Both for 90-Weight Oil, 1.8 kHz, and Large Horn Spacing**

90-Weight Oil:

Acoustic Frequency: 1.8 kHz
Horn Design: Large Spacing

| Treatment Time, (min) | Oil Temperature (ºC) | Calculated Fractional Residual Viscosity (due to heat) | Normalized Fractional Residual Viscosity (due to heat) | Viscosity Reduction due to Sonication | Normalized Fractional Residual Viscosity due to Sonication | Overall Fractional Residual Viscosity |
|---|---|---|---|---|---|---|
| 0 | 21.1 | 0.55242 | 1 | 0 | 1 | 1 |
| 5 | 21.7 | 0.53093 | 0.96110 | 0.33070 | 0.66930 | 0.6304 |
| 10 | 23.2 | 0.48302 | 0.87436 | 0.51576 | 0.48424 | 0.3586 |
| 15 | 30.3 | 0.33104 | 0.59924 | 0.31974 | 0.68026 | 0.2795 |
| 20 | 31.6 | 0.31193 | 0.56466 | 0.30386 | 0.69614 | 0.2608 |
| 25 | 35.2 | 0.26776 | 0.48471 | 0.20401 | 0.79599 | 0.2807 |
| 30 | 34.3 | 0.27776 | 0.50280 | 0.22920 | 0.77080 | 0.2736 |

**Table B-44 Fractional Viscosity Effects of Heat, Sonication, and Both for 90-Weight Oil, 3.1 kHz, and Small Horn Spacing**

90-Weight Oil:

Acoustic Frequency: 3.1 kHz
Horn Design: Small Spacing

| Treatment Time, (min) | Oil Temperature (ºC) | Calculated Fractional Residual Viscosity (due to heat) | Normalized Fractional Residual Viscosity (due to heat) | Viscosity Reduction due to Sonication | Normalized Fractional Residual Viscosity due to Sonication | Overall Fractional Residual Viscosity |
|---|---|---|---|---|---|---|
| 0 | 21.0 | 0.55615 | 1 | 0 | 1 | 1 |
| 5 | 28.9 | 0.35396 | 0.63644 | 0.20514 | 0.79486 | 0.4313 |
| 10 | 36.4 | 0.25536 | 0.45915 | 0.15885 | 0.84115 | 0.3003 |
| 15 | 39.1 | 0.23077 | 0.41494 | 0.16624 | 0.83376 | 0.2487 |
| 20 | 43.7 | 0.19716 | 0.35451 | 0.10851 | 0.89149 | 0.2460 |
| 25 | 48.2 | 0.17163 | 0.30860 | 0.09000 | 0.91000 | 0.2186 |
| 30 | 45.9 | 0.18392 | 0.33071 | 0.11791 | 0.88209 | 0.2128 |
| 35 | 46.7 | 0.17948 | 0.32272 | 0.10062 | 0.89938 | 0.2221 |

**Table B-45 Fractional Viscosity Effects of Heat, Sonication, and Both for 90-Weight Oil, 6.9 kHz, and Small Horn Spacing**

90-Weight Oil:

Acoustic Frequency: 6.9 kHz
Horn Design: Small Spacing

| Treatment Time, (min) | Oil Temperature (ºC) | Calculated Fractional Residual Viscosity (due to heat) | Normalized Fractional Residual Viscosity (due to heat) | Viscosity Reduction due to Sonication | Normalized Fractional Residual Viscosity due to Sonication | Overall Fractional Residual Viscosity |
|---|---|---|---|---|---|---|
| 0 | 20.7 | 0.56759 | 1 | 0 | 1 | 1 |
| 5 | 25.2 | 0.42968 | 0.75702 | 0 | 1 | 0.7663 |
| 10 | 26.3 | 0.40447 | 0.71261 | 0.26301 | 0.73699 | 0.4496 |
| 15 | 27.7 | 0.37585 | 0.66218 | 0.26628 | 0.73372 | 0.3959 |
| 20 | 27.2 | 0.38566 | 0.67947 | 0.31587 | 0.68413 | 0.3636 |
| 25 | 28.9 | 0.35396 | 0.62361 | | | |
| 30 | 30.6 | 0.32645 | 0.57516 | 0.26146 | 0.73854 | 0.3137 |
| 40 | 30.8 | 0.32346 | 0.56988 | 0.25588 | 0.74412 | 0.3140 |

**Table B-46 Fractional Viscosity Effects of Heat, Sonication, and Both for 90-Weight Oil, 6.9 kHz, and Small Horn Spacing (Test Performed Using a Water Bath)**

90-Weight Oil:

Acoustic
Frequency: 6.9 kHz
Horn Design: Small Spacing
(Test Performed in a Water Bath)

| Treatment Time, (min) | Oil Temperature (ºC) | Calculated Fractional Residual Viscosity (due to heat) | Normalized Fractional Residual Viscosity (due to heat) | Viscosity Reduction due to Sonication | Normalized Fractional Residual Viscosity due to Sonication | Overall Fractional Residual Viscosity |
|---|---|---|---|---|---|---|
| 0 | 22.5 | 0.50442 | 1 | 0 | 1 | 1 |
| 5 | 23.7 | 0.46866 | 0.92911 | 0.07661 | 0.92339 | 0.8525 |
| 10 | 24.8 | 0.43952 | 0.87133 | 0.22713 | 0.77287 | 0.6442 |
| 15 | 24.9 | 0.43702 | 0.86639 | 0.30999 | 0.69001 | 0.5564 |
| 20 | 24.7 | 0.44204 | 0.87633 | 0.27913 | 0.72087 | 0.5972 |
| 25 | 26.6 | 0.39803 | 0.78908 | | | |
| 30 | 26.8 | 0.39383 | 0.78076 | 0.21786 | 0.78214 | 0.5629 |
| 35 | 26.5 | 0.40016 | 0.79330 | | | |
| 40 | 27.9 | 0.37204 | 0.73756 | 0.24446 | 0.75554 | 0.4931 |

**Table B-47 Fractional Viscosity Effects of Heat, Sonication, and Both for 90-Weight Oil, 6.9 kHz, and Medium Horn Spacing**

90-Weight Oil:

Acoustic Frequency: 6.7 kHz
Horn Design: Medium Spacing

| Treatment Time, (min) | Oil Temperature ($^{o}$C) | Calculated Fractional Residual Viscosity (due to heat) | Normalized Fractional Residual Viscosity (due to heat) | Viscosity Reduction due to Sonication | Normalized Fractional Residual Viscosity due to Sonication | Overall Fractional Residual Viscosity |
|---|---|---|---|---|---|---|
| 0 | 21.1 | 0.55242 | 1 | 0 | 1 | 1 |
| 5 | 20.3 | 0.58348 | | 0.02890 | 0.97110 | 0.9711 |
| 10 | 23.2 | 0.48302 | 0.87436 | 0.05926 | 0.94074 | 0.8151 |
| 15 | 24.3 | 0.45237 | 0.81888 | 0.26118 | 0.73882 | 0.5577 |
| 20 | 25.4 | 0.42490 | 0.76915 | 0.32695 | 0.67305 | 0.4422 |
| 30 | 26.4 | 0.40230 | 0.72825 | 0.38075 | 0.61925 | 0.3475 |
| 40 | 31.1 | 0.31905 | 0.57755 | 0.26465 | 0.73535 | 0.3129 |
| 50 | 27.8 | 0.37394 | 0.67690 | 0.41230 | 0.58770 | 0.2646 |
| 60 | 34.5 | 0.27548 | 0.49868 | 0.24548 | 0.75452 | 0.2532 |

**Table B-48 Fractional Viscosity Effects of Heat, Sonication, and Both for 90-Weight Oil, 6.9 kHz, and Medium Horn Spacing (Duplicate Experiment)**

90-Weight Oil:

Acoustic Frequency: 6.8 kHz
Horn Design: Medium Spacing
(Duplicate Experiment)

| Treatment Time, (min) | Oil Temperature ($^{o}$C) | Calculated Fractional Residual Viscosity (due to heat) | Normalized Fractional Residual Viscosity (due to heat) | Viscosity Reduction due to Sonication | Normalized Fractional Residual Viscosity due to Sonication | Overall Fractional Residual Viscosity |
|---|---|---|---|---|---|---|
| 0 | 21.3 | 0.54510 | 1 | 0 | 1 | 1 |
| 5 | 21.6 | 0.53442 | 0.98040 | 0.09980 | 0.90020 | 0.8806 |
| 10 | 23.1 | 0.48598 | 0.89154 | 0.27874 | 0.72126 | 0.6128 |
| 15 | 24.5 | 0.44715 | 0.82032 | 0.25952 | 0.74048 | 0.5608 |
| 20 | 24.4 | 0.44975 | 0.82508 | 0.32828 | 0.67172 | 0.4968 |
| 25 | 31.1 | 0.31905 | 0.58531 | 0.11511 | 0.88489 | 0.4702 |
| 30 | 27.0 | 0.38971 | 0.71494 | 0.24104 | 0.75896 | 0.4739 |
| 35 | 27.6 | 0.37778 | 0.69304 | 0.25804 | 0.74196 | 0.4350 |
| 40 | 29.8 | 0.33892 | 0.62177 | 0.20887 | 0.79113 | 0.4129 |
| 45 | 29.5 | 0.34381 | 0.63073 | 0.20753 | 0.79247 | 0.4232 |
| 50 | 31.1 | 0.31905 | 0.58531 | 0.18671 | 0.81329 | 0.3986 |

**Table B-49 Fractional Viscosity Effects of Heat, Sonication, and Both for 90-Weight Oil, 6.9 kHz, and Large Horn Spacing**

90-Weight Oil:

Acoustic Frequency: 6.8 kHz
Horn Design: Large Spacing

| Treatment Time, (min) | Oil Temperature ($^{o}$C) | Calculated Fractional Residual Viscosity (due to heat) | Normalized Fractional Residual Viscosity (due to heat) | Viscosity Reduction due to Sonication | Normalized Fractional Residual Viscosity due to Sonication | Overall Fractional Residual Viscosity |
|---|---|---|---|---|---|---|
| 0 | 21.4 | 0.54150 | 1 | 0 | 1 | 1 |
| 5 | 22.9 | 0.49200 | 0.90858 | 0.06378 | 0.93622 | 0.8448 |
| 10 | 27.3 | 0.38366 | 0.70852 | 0.03192 | 0.96808 | 0.6766 |
| 15 | 28.5 | 0.36101 | 0.66668 | 0.19608 | 0.80392 | 0.4706 |
| 20 | 31.9 | 0.30779 | 0.56840 | 0.09710 | 0.90290 | 0.4713 |
| 25 | 30.3 | 0.33104 | 0.61133 | 0.18033 | 0.81967 | 0.4310 |
| 30 | 33.5 | 0.28719 | 0.53037 | 0.15537 | 0.84463 | 0.3750 |
| 35 | 31.5 | 0.31333 | 0.57864 | 0.11394 | 0.88606 | 0.4647 |
| 39.5 | 34.3 | 0.27776 | 0.51295 | 0.06375 | 0.93625 | 0.4492 |
| 44 | 31.8 | 0.30916 | 0.57093 | 0.10803 | 0.89197 | 0.4629 |

**Table B-50 Fractional Viscosity Effects of Heat, Sonication, and Both for 90-Weight Oil, 6.9 kHz, and Large Horn Spacing (Test Performed Using a Water Bath)**

90-Weight Oil:

Acoustic Frequency: 6.8 kHz
Horn Design: Large Spacing

(Test Performed in a Water Bath)

| Treatment Time, (min) | Oil Temperature ($^{o}$C) | Calculated Fractional Residual Viscosity (due to heat) | Normalized Fractional Residual Viscosity (due to heat) | Viscosity Reduction due to Sonication | Normalized Fractional Residual Viscosity due to Sonication | Overall Fractional Residual Viscosity |
|---|---|---|---|---|---|---|
| 0 | 21.2 | 0.54874 | 1 | 0 | 1 | 1 |
| 5 | 23.4 | 0.47719 | 0.86960 | 0.09920 | 0.90080 | 0.7704 |
| 10 | 27.6 | 0.37778 | 0.68844 | 0.06684 | 0.93316 | 0.6216 |
| 15 | 29.3 | 0.34714 | 0.63261 | 0.10681 | 0.89319 | 0.5258 |
| 20 | 28.6 | 0.35922 | 0.65463 | 0.18493 | 0.81507 | 0.4697 |
| 25 | 29.5 | 0.34381 | 0.62655 | 0.14735 | 0.85265 | 0.4792 |
| 30 | 31.0 | 0.32051 | 0.58408 | 0.11088 | 0.88912 | 0.4732 |
| 35 | 31.9 | 0.30779 | 0.56090 | 0.09480 | 0.90520 | 0.4661 |
| 40 | 31.9 | 0.30779 | 0.56090 | 0.11660 | 0.88340 | 0.4443 |
| 45 | 28.7 | 0.35745 | 0.65140 | 0.19360 | 0.80640 | 0.4578 |
| 50 | 31.5 | 0.31333 | 0.57100 | 0.11530 | 0.88470 | 0.4557 |

**Table B-51 Fractional Viscosity Effects of Heat, Sonication, and Both for 90-Weight Oil, 13.1 kHz, and Small Horn Spacing**

90-Weight Oil:

Acoustic Frequency: 13.1 kHz
Horn Design: Small Spacing

| Treatment Time, (min) | Oil Temperature ($^{o}$C) | Calculated Fractional Residual Viscosity (due to heat) | Normalized Fractional Residual Viscosity (due to heat) | Viscosity Reduction due to Sonication | Normalized Fractional Residual Viscosity due to Sonication | Overall Fractional Residual Viscosity |
|---|---|---|---|---|---|---|
| 0 | 21.5 | 0.53794 | 1 | 0 | 1 | 1 |
| 5 | 22.0 | 0.52072 | 0.96799 | 0.07029 | 0.92971 | 0.8977 |
| 10 | 23.8 | 0.46588 | 0.86604 | 0.21224 | 0.78776 | 0.6538 |
| 15 | 25.8 | 0.41561 | 0.77259 | 0.24259 | 0.75741 | 0.5300 |
| 20 | 26.3 | 0.40447 | 0.75189 | 0.21099 | 0.78901 | 0.5409 |
| 25 | 29.8 | 0.33892 | 0.63004 | 0.13514 | 0.86486 | 0.4949 |
| 30 | 32.3 | 0.30241 | 0.56216 | 0.07996 | 0.92004 | 0.4822 |
| 35 | 30.4 | 0.32950 | 0.61252 | 0.11192 | 0.88808 | 0.5006 |
| 40 | 32.5 | 0.29978 | 0.55727 | 0.10477 | 0.89523 | 0.4525 |
| 45 | 33.1 | 0.29212 | 0.54303 | 0.08913 | 0.91087 | 0.4539 |

**Table B-52 Fractional Viscosity Effects of Heat, Sonication, and Both for 90-Weight Oil, 13.1 kHz, and Medium Horn Spacing**

90-Weight Oil:

Acoustic Frequency: 13.1 kHz
Horn Design: Medium Spacing

| Treatment Time, (min) | Oil Temperature ($^{o}$C) | Calculated Fractional Residual Viscosity (due to heat) | Normalized Fractional Residual Viscosity (due to heat) | Viscosity Reduction due to Sonication | Normalized Fractional Residual Viscosity due to Sonication | Overall Fractional Residual Viscosity |
|---|---|---|---|---|---|---|
| 0 | 22.5 | 0.50442 | 1 | 0 | 1 | 1 |
| 5 | 23.1 | 0.48598 | 0.96344 | 0.09674 | 0.90326 | 0.8667 |
| 10 | 27.3 | 0.38366 | 0.76061 | 0.12171 | 0.87829 | 0.6389 |
| 15 | 32.6 | 0.29848 | 0.59172 | 0.00982 | | 0.5819 |
| 20 | 30.3 | 0.33104 | 0.65627 | 0.15147 | 0.84853 | 0.5048 |
| 25 | 35.7 | 0.26247 | 0.52035 | 0.04365 | 0.95635 | 0.4767 |
| 30 | 38.7 | 0.23415 | 0.46420 | 0.04350 | 0.95650 | 0.4207 |
| 35 | 41.1 | 0.21504 | 0.42631 | 0.04231 | 0.95769 | 0.3840 |

**Table B-53 Fractional Viscosity Effects of Heat, Sonication, and Both for 90-Weight Oil, 13.1 kHz, and Large Horn Spacing**

90 Weight Oil:

Acoustic Frequency: 13.1 kHz
Horn Design: Large Spacing

| Treatment Time, (min) | Oil Temperature ($^{o}$C) | Calculated Fractional Residual Viscosity (due to heat) | Normalized Fractional Residual Viscosity (due to heat) | Viscosity Reduction due to Sonication | Normalized Fractional Residual Viscosity due to Sonication | Overall Fractional Residual Viscosity |
|---|---|---|---|---|---|---|
| 0 | 21.1 | 0.55242 | 1 | 0 | 1 | 1 |
| 5 | 22.7 | 0.49814 | 0.90174 | 0.11474 | | 0.7870 |
| 10 | 27.5 | 0.37972 | 0.68737 | 0.01817 | 0.98183 | 0.6692 |
| 15 | 29.7 | 0.34054 | 0.61645 | 0.03755 | 0.96245 | 0.5789 |
| 20 | 30.3 | 0.33104 | 0.59924 | 0.05524 | 0.94476 | 0.5440 |
| 25 | 31.5 | 0.31333 | 0.56720 | 0.08990 | 0.91010 | 0.4773 |
| 30 | 35.2 | 0.26776 | 0.48471 | 0.04121 | 0.95879 | 0.4435 |
| 35 | 34.7 | 0.27324 | 0.49462 | 0.10572 | 0.89428 | 0.3889 |
| 40 | 37.4 | 0.24575 | 0.44486 | 0.05846 | 0.94154 | 0.3864 |
| 45 | 37.4 | 0.24575 | 0.44486 | 0.06536 | 0.93464 | 0.3795 |
| 50 | 38.7 | 0.23415 | 0.42386 | 0.10896 | 0.89104 | 0.3149 |
| 55 | 38.5 | 0.23587 | 0.42698 | | | |
| 60 | 35.7 | 0.26247 | 0.47513 | 0.13863 | 0.86137 | 0.3365 |

**Table B-54 Fractional Viscosity Effects of Heat, Sonication, and Both for 140-Weight Oil, 1.8 kHz, and Small Horn Spacing**

140-Weight Oil:

Acoustic Frequency: 1.8 kHz
Horn Design: Small Spacing

| Treatment Time, (min) | Oil Temperature ($^{o}$C) | Calculated Fractional Residual Viscosity (due to heat) | Normalized Fractional Residual Viscosity (due to heat) | Viscosity Reduction due to Sonication | Normalized Fractional Residual Viscosity due to Sonication | Overall Fractional Residual Viscosity |
|---|---|---|---|---|---|---|
| 0 | 21.2 | 0.88572 | 1 | 0 | 1 | 1 |
| 5 | 22.6 | 0.81950 | 0.925244085 | 0.07204 | 0.92796 | 0.8532 |
| 10 | 24.6 | 0.73928 | 0.834664449 | 0.23526 | 0.76474 | 0.5994 |
| 15 | 25.5 | 0.70770 | 0.799009148 | 0.25861 | 0.74139 | 0.5404 |
| 20 | 25.0 | 0.72493 | 0.818466598 | 0.34597 | 0.65403 | 0.4725 |
| 25 | 26.3 | 0.68163 | 0.769576569 | 0.43358 | 0.56642 | 0.3360 |
| 30 | 26.2 | 0.68479 | 0.77314687 | 0.43825 | 0.56175 | 0.3349 |

**Table B-55 Fractional Viscosity Effects of Heat, Sonication, and Both for 140-Weight Oil, 1.8 kHz, and Medium Horn Spacing**

140-Weight Oil:

Acoustic Frequency: 1.8 kHz
Horn Design: Medium Spacing

| Treatment Time, (min) | Oil Temperature ($^{o}$C) | Calculated Fractional Residual Viscosity (due to heat) | Normalized Fractional Residual Viscosity (due to heat) | Viscosity Reduction due to Sonication | Normalized Fractional Residual Viscosity due to Sonication | Overall Fractional Residual Viscosity |
|---|---|---|---|---|---|---|
| 0 | 22.1 | 0.84208 | 1 | 0 | 1 | 1 |
| 5 | 22.3 | 0.83292 | 0.98911 | 0.02741 | 0.97259 | 0.9617 |
| 10 | 23.0 | 0.80222 | 0.95265 | 0.27795 | 0.72205 | 0.6747 |
| 15 | 23.3 | 0.78969 | 0.93777 | 0.36267 | 0.63733 | 0.5751 |
| 20 | 22.8 | 0.81078 | 0.96282 | 0.49622 | 0.50378 | 0.4666 |
| 25 | 23.5 | 0.78153 | 0.92808 | 0.92808 | | |
| 30 | 24.2 | 0.75415 | 0.89557 | 0.40507 | 0.59493 | 0.4905 |
| 35 | 25.5 | 0.70770 | 0.84040 | 0.31380 | 0.68620 | 0.5266 |
| 41 | 25.4 | 0.71108 | 0.84443 | 0.34823 | 0.65177 | 0.4962 |

**Table B-56 Fractional Viscosity Effects of Heat, Sonication, and Both for 140-Weight Oil, 1.8 kHz, and Large Horn Spacing**

140-Weight Oil:

Acoustic Frequency: 1.8 kHz
Horn Design: Large Spacing

| Treatment Time, (min) | Oil Temperature ($^{o}$C) | Calculated Fractional Residual Viscosity (due to heat) | Normalized Fractional Residual Viscosity (due to heat) | Viscosity Reduction due to Sonication | Normalized Fractional Residual Viscosity due to Sonication | Overall Fractional Residual Viscosity |
|---|---|---|---|---|---|---|
| 0 | 22.2 | 0.83748 | 1 | 0 | 1 | 1 |
| 5 | 24.1 | 0.75795 | 0.90504 | 0.06054 | 0.93946 | 0.8445 |
| 10 | 29.4 | 0.59532 | 0.71085 | 0.15375 | 0.84625 | 0.5571 |
| 15 | 27.5 | 0.64566 | 0.77096 | 0.22776 | 0.77224 | 0.5432 |
| 20 | 28.6 | 0.61561 | 0.73508 | 0.27848 | 0.72152 | 0.4566 |
| 25 | 29.8 | 0.58563 | 0.69927 | 0.27007 | 0.72993 | 0.4292 |
| 30 | 31.7 | 0.54326 | 0.64868 | 0.25098 | 0.74902 | 0.3977 |
| 35 | 32.5 | 0.52705 | 0.62933 | 0.23883 | 0.76117 | 0.3905 |
| 40 | 35.1 | 0.48000 | 0.57315 | 0.16075 | 0.83925 | 0.4124 |

**Table B-57 Fractional Viscosity Effects of Heat, Sonication, and Both for 140-Weight Oil, 3.1 kHz, and Small Horn Spacing**

140-Weight Oil:

Acoustic Frequency: 3.1 kHz
Horn Design: Small Spacing

| Treatment Time, (min) | Oil Temperature ($^{o}$C) | Calculated Fractional Residual Viscosity (due to heat) | Normalized Fractional Residual Viscosity (due to heat) | Viscosity Reduction due to Sonication | Normalized Fractional Residual Viscosity due to Sonication | Overall Fractional Residual Viscosity |
|---|---|---|---|---|---|---|
| 0 | 20.9 | 0.90119 | 1 | 0 | 1 | 1 |
| 5 | 22.3 | 0.83292 | 0.92425 | 0.26895 | 0.73105 | 0.6553 |
| 9 | 24.1 | 0.75795 | 0.84106 | 0.30646 | 0.69354 | 0.5346 |
| 15 | 23.8 | 0.76958 | 0.85396 | 0.22276 | 0.77724 | 0.6312 |
| 15 | 23.8 | 0.76958 | 0.85396 | 0.21396 | 0.78604 | 0.6400 |
| 20 | 24.4 | 0.74664 | 0.82851 | 0.27411 | 0.72589 | 0.5544 |
| 25 | 27.9 | 0.63443 | 0.70399 | 0.05039 | 0.94961 | 0.6536 |

**Table B-58 Fractional Viscosity Effects of Heat, Sonication, and Both for 140-Weight Oil, 6.9 kHz, and Small Horn Spacing**

140-Weight Oil:

Acoustic Frequency: 6.9 kHz
Horn Design: Small Spacing

| Treatment Time, (min) | Oil Temperature ($^{o}$C) | Calculated Fractional Residual Viscosity (due to heat) | Normalized Fractional Residual Viscosity (due to heat) | Viscosity Reduction due to Sonication | Normalized Fractional Residual Viscosity due to Sonication | Overall Fractional Residual Viscosity |
|---|---|---|---|---|---|---|
| 0 | 22.8 | 0.81078 | 1 | 0 | 1 | 1 |
| 5 | 23.6 | 0.77751 | 0.95896 | | | |
| 10 | 25.4 | 0.71108 | 0.87704 | 0.11194 | 0.88806 | 0.7651 |
| 15 | 28.8 | 0.61042 | 0.75289 | | | |
| 20 | 27.2 | 0.65432 | 0.80703 | 0.06453 | 0.93547 | 0.7425 |
| 25 | 28.5 | 0.61824 | 0.76253 | | | |
| 30 | 30.5 | 0.56934 | 0.70221 | 0.03301 | 0.96699 | 0.6692 |
| 35 | 28.3 | 0.62355 | 0.76908 | | | |
| 40 | 30.9 | 0.56039 | 0.69118 | | | |
| 45 | 28.7 | 0.61301 | 0.75607 | 0.09447 | 0.90553 | 0.6616 |

**Table B-59 Fractional Viscosity Effects of Heat, Sonication, and Both for 140-Weight Oil, 6.9 kHz, and Medium Horn Spacing**

140-Weight Oil:

Acoustic Frequency: 6.9 kHz
Horn Design: Medium Spacing

| Treatment Time, (min) | Oil Temperature ($^{o}$C) | Calculated Fractional Residual Viscosity (due to heat) | Normalized Fractional Residual Viscosity (due to heat) | Viscosity Reduction due to Sonication | Normalized Fractional Residual Viscosity due to Sonication | Overall Fractional Residual Viscosity |
|---|---|---|---|---|---|---|
| 0 | 20.5 | 0.92260 | 1 | 0 | 1 | 1 |
| 5 | 21.5 | 0.87072 | 0.94377 | 0.05937 | 0.94063 | 0.8844 |
| 10 | 24.5 | 0.74294 | 0.80527 | 0.04537 | 0.95463 | 0.7599 |
| 15 | 26.8 | 0.66621 | 0.72210 | | | |
| 20 | 26.9 | 0.66320 | 0.71884 | 0.03594 | 0.96406 | 0.6829 |
| 25 | 25.7 | 0.70101 | 0.75982 | | | |
| 30 | 26.5 | 0.67538 | 0.73204 | 0.10434 | 0.89566 | 0.6277 |
| 35 | 26.3 | 0.68163 | 0.73881 | | | |
| 40 | 29.5 | 0.59287 | 0.64261 | | | |
| 45 | 31.1 | 0.55602 | 0.60267 | 0 | 1 | 0.6302 |
| 50 | 27.9 | 0.63443 | 0.68766 | | | |
| 55 | 28.1 | 0.62895 | 0.68171 | | | |
| 60 | 29.1 | 0.60279 | 0.65336 | 0.02146 | 0.97854 | 0.6319 |

**Table B-60 Fractional Viscosity Effects of Heat, Sonication, and Both for 140-Weight Oil, 6.9 kHz, and Medium Horn Spacing (Continuation of Test Summarized in Table 59)**

140-Weight Oil:

Acoustic Frequency: 6.9 kHz
Horn Design: Medium Spacing

| Treatment Time, (min) | Oil Temperature ($^{o}$C) | Calculated Fractional Residual Viscosity (due to heat) | Normalized Fractional Residual Viscosity (due to heat) | Viscosity Reduction due to Sonication | Normalized Fractional Residual Viscosity due to Sonication | Overall Fractional Residual Viscosity |
|---|---|---|---|---|---|---|
| 0 | 22.0 | 0.84674 | 1 | 0 | 1 | 1 |
| 5 | 22.5 | 0.82393 | 0.97306 | 0.07026 | 0.92974 | 0.9028 |
| 10 | 22.2 | 0.83748 | 0.98906 | 0.05096 | 0.94904 | 0.9381 |
| 15 | 22.5 | 0.82393 | 0.97306 | 0.26276 | 0.73724 | 0.7103 |
| 20 | 24.3 | 0.75038 | 0.88620 | 0.15880 | 0.84120 | 0.7274 |
| 27 | 25.6 | 0.70434 | 0.83183 | 0.15613 | 0.84387 | 0.6757 |
| 32 | 25.8 | 0.69771 | 0.82400 | 0.13990 | 0.86010 | 0.6841 |

**Table B-61 Fractional Viscosity Effects of Heat, Sonication, and Both for 140-Weight Oil, 6.9 kHz, and Large Horn Spacing**

140-Weight Oil:

Acoustic Frequency: 6.9 kHz
Horn Design: Large Spacing

| Treatment Time, (min) | Oil Temperature ($^{o}$C) | Calculated Fractional Residual Viscosity (due to heat) | Normalized Fractional Residual Viscosity (due to heat) | Viscosity Reduction due to Sonication | Normalized Fractional Residual Viscosity due to Sonication | Overall Fractional Residual Viscosity |
|---|---|---|---|---|---|---|
| 0 | 20.7 | 0.91178 | 1 | 0 | 1 | 1 |
| 5 | 21.0 | 0.89598 | 0.98267 | 0.12157 | 0.87843 | 0.8611 |
| 10 | 21.6 | 0.86583 | 0.94960 | 0.20720 | 0.79280 | 0.7424 |
| 15 | 23.1 | 0.79800 | 0.87522 | 0.27052 | 0.72948 | 0.6047 |
| 20 | 23.0 | 0.80222 | 0.87984 | 0.30854 | 0.69146 | 0.5713 |
| 30 | 26.0 | 0.69119 | 0.75807 | 0.32337 | 0.67663 | 0.4347 |
| 39 | 23.5 | 0.78153 | 0.85715 | 0.45795 | 0.54205 | 0.3992 |

**Table B-62 Fractional Viscosity Effects of Heat, Sonication, and Both for 140-Weight Oil, 13.1 kHz, and Small Horn Spacing with Reduced Power Input**

140-Weight Oil:

Acoustic Frequency: 13.1 kHz
Horn Design: Small Spacing

71% current output; 89 volts

| Treatment Time, (min) | Oil Temperature ($^{o}$C) | Calculated Fractional Residual Viscosity (due to heat) | Normalized Fractional Residual Viscosity (due to heat) | Viscosity Reduction due to Sonication | Normalized Fractional Residual Viscosity due to Sonication | Overall Fractional Residual Viscosity |
|---|---|---|---|---|---|---|
| 0 | 20.5 | 0.92260 | 1 | 0 | 1 | 1 |
| 5 | 21.5 | 0.87072 | 0.94377 | 0.18047 | 0.81953 | 0.7633 |
| 10 | 26.4 | 0.67849 | 0.73541 | 0.11291 | 0.88709 | 0.6225 |
| 14 | 22.8 | 0.81078 | 0.87880 | 0.23140 | 0.76860 | 0.6474 |
| 18 | 26.8 | 0.66621 | 0.72210 | 0.12530 | 0.87470 | 0.5968 |
| 22 | 25.1 | 0.72142 | 0.78195 | 0.10555 | 0.89445 | 0.6764 |
| 26 | 26.1 | 0.68798 | 0.74570 | 0.13010 | 0.86990 | 0.6156 |
| 29 | 26.6 | 0.67230 | 0.72870 | 0.05060 | 0.94940 | 0.6781 |

**Table B-63 Fractional Viscosity Effects of Heat, Sonication, and Both for 140-Weight Oil, 13.1 kHz, and Small Horn Spacing with Reduced Power Input (Duplicate of Test Summarized in Table B-62)**

140-Weight Oil:

Acoustic
Frequency: 13.1 kHz
Horn Design: Small Spacing

71% current output; 89 volts
(Duplicate Experiment)

| Treatment Time, (min) | Oil Temperature ($^{o}$C) | Calculated Fractional Residual Viscosity (due to heat) | Normalized Fractional Residual Viscosity (due to heat) | Viscosity Reduction due to Sonication | Normalized Fractional Residual Viscosity due to Sonication | Overall Fractional Residual Viscosity |
|---|---|---|---|---|---|---|
| 0 | 20.2 | 0.93927 | 1 | 0 | 1 | 1 |
| 5 | 21.0 | 0.89598 | 0.95391 | 0.05561 | 0.94439 | 0.8983 |
| 10 | 21.8 | 0.85619 | 0.91154 | 0.12154 | 0.87846 | 0.7900 |
| 15 | 27.7 | 0.64000 | 0.68138 | 0.03308 | 0.96692 | 0.6483 |
| 20 | 28.7 | 0.61301 | 0.65264 | 0.10794 | 0.89206 | 0.5447 |
| 25 | 29.0 | 0.60531 | 0.64445 | 0.08785 | 0.91215 | 0.5566 |
| 30 | 29.1 | 0.60279 | 0.64176 | 0.09746 | 0.90254 | 0.5443 |
| 35 | 30.1 | 0.57854 | 0.61595 | 0.08445 | 0.91555 | 0.5315 |
| 40 | 29.5 | 0.59287 | 0.63120 | 0.10280 | 0.89720 | 0.5284 |
| 45 | 31.1 | 0.55602 | 0.59197 | 0.11307 | 0.88693 | 0.4789 |
| 50 | 31.1 | 0.55602 | 0.59197 | 0.07847 | 0.92153 | 0.5135 |

**Table B-64 Fractional Viscosity Effects of Heat, Sonication, and Both for 140-Weight Oil, 13.1 kHz, and Medium Horn Spacing with Reduced Power Input**

140-Weight Oil:

Acoustic Frequency: 13.1 kHz
Horn Design: Medium Spacing

70% current output; 88 volts

| Treatment Time, (min) | Oil Temperature ($^{o}$C) | Calculated Fractional Residual Viscosity (due to heat) | Normalized Fractional Residual Viscosity (due to heat) | Viscosity Reduction due to Sonication | Normalized Fractional Residual Viscosity due to Sonication | Overall Fractional Residual Viscosity |
|---|---|---|---|---|---|---|
| 0 | 21.1 | 0.89082 | 1 | 0 | 1 | 1 |
| 5 | 21.0 | 0.89598 | 1.00579 | 0.12739 | 0.87261 | 0.8784 |
| 10 | 21.5 | 0.87072 | 0.97744 | 0.28084 | 0.71916 | 0.6966 |
| 15 | 22.4 | 0.82840 | 0.92993 | 0.30023 | 0.69977 | 0.6297 |
| 20 | 23.1 | 0.79800 | 0.89581 | 0.32731 | 0.67269 | 0.5685 |
| 25 | 24.3 | 0.75038 | 0.84235 | 0.30245 | 0.69755 | 0.5399 |
| 30 | 26.5 | 0.67538 | 0.75816 | 0.27746 | 0.72254 | 0.4807 |
| 35 | 28.3 | 0.62355 | 0.69998 | 0.26568 | 0.73432 | 0.4343 |
| 40 | 24.7 | 0.73564 | 0.82580 | 0.29160 | 0.70840 | 0.5342 |
| 45 | 27.9 | 0.63443 | 0.71219 | 0.21099 | 0.78901 | 0.5012 |
| 50 | 28.2 | 0.62624 | 0.70299 | 0.20269 | 0.79731 | 0.5003 |

**Table B-65 Fractional Viscosity Effects of Heat, Sonication, and Both for 140-Weight Oil, 13.1 kHz, and Large Horn Spacing with Reduced Power Input**

140-Weight Oil:

Acoustic Frequency: 13.1 kHz
Horn Design: Large Spacing

70% current output

| Treatment Time, (min) | Oil Temperature ($^{o}$C) | Calculated Fractional Residual Viscosity (due to heat) | Normalized Fractional Residual Viscosity (due to heat) | Viscosity Reduction due to Sonication | Normalized Fractional Residual Viscosity due to Sonication | Overall Fractional Residual Viscosity |
|---|---|---|---|---|---|---|
| 0 | 22.0 | 0.84674 | 1 | 0 | 1 | 1 |
| 5 | 23.3 | 0.78969 | 0.93262 | 0.31472 | 0.68528 | 0.6179 |
| 10 | 23.1 | 0.79800 | 0.94244 | 0.32954 | 0.67046 | 0.6129 |
| 15 | 23.3 | 0.78969 | 0.93262 | 0.40162 | 0.59838 | 0.5310 |
| 20 | 24.4 | 0.74664 | 0.88179 | 0.37869 | 0.62131 | 0.5031 |
| 25 | 23.2 | 0.79382 | 0.93751 | 0.48751 | 0.51249 | 0.4500 |

# APPENDIX C

## Standard Operating Procedures for the Storage, Handling, and Disposal of Crude Oil at the University of Alabama at Birmingham

## SOP Developed for Crude Oil Storage, Handling, and Disposal

1) The oil samples for an experiment were collected from the main storage area in the cage having controlled access and located at the Business and Engineering Complex (BEC). Approved hazardous waste containers were used to collect and transport the oil to the laboratory, and these same containers were later used to dispose of the waste.
2) The experimental equipment was placed in a kitty litter pan containing litter that was also covered with plastic sheets to contain any oil in case of a spill. The test chamber along with spill containment was placed inside a fume hood and maintained there while experiments were conducted.
3) The exit lines from the reaction chamber apparatus were used to drain the oil from the reaction chamber into an approved hazardous waste container. Waste oil that was no longer being used for testing was placed in 55-gallon drums for storage until final disposition. These drums were stored in a secure cage area outside of the laboratories and the BEC complex.
4) During an experiment, plastic Dixie cups were used to collect samples for viscosity analysis. After the viscosity measurement was taken, the cups were placed upside down in a funnel and the oil was drained as much as possible into the hazardous waste container, and the cups were disposed of in a separate hazardous waste container.
5) As soon as an experiment was completed, the parts of the equipment that required cleaning were first cleaned with paper towels, and the paper towels were disposed of in the same container that was marked "Hazardous Waste", which was previously used to dispose of the Dixie cups. The waste paper towels containing oil were placed in a plastic bag and the bag opening was tied to prevent leakage from the paper towels. The bag was then placed in the waste container.
6) After the first stage of cleaning, the equipment was cleaned further with paper towels that were soaked in mineral sprits. The waste was disposed of in the same container marked as "Hazardous Waste", as described above in Step 4.
7) After the oil was removed from the equipment and any parts of the hood necessary because of a spill, the cleaning materials were placed in the waste container that was properly sealed.
8) During all times of operation, the waste container was kept as near to the hood as possible or fully inside the hood) if there was sufficient room thereby eliminating any chance of volatile or gaseous emissions emitting from the stored waste.
9) When the waste container was nearly full, a chemical manifest was filled out and the UAB Chemical Safety Department or the Hazardous Waste Office was notified to request removal of the waste. At this point, the hazardous waste container was placed in a secondary container until it was collected and removed for disposal.
10) Records of the amount and the date of generation of the waste as well as the date when the material was picked up by the UAB Chemical Safety Department were prepared and maintained.

11) At the end of the experiments, all of the waste oil stored in 55-gallon drums was removed by a licensed firm for recycle and/or disposal.

# APPENDIX D

## Sonication System Testing: Sand Test Conditions and Observations

# Sand Test Methods

All of the sand tests were performed in an aquarium 30 in (76.2 cm) long by 12 in (30.5 cm) wide by 18 in (45.7 cm) tall (deep). At the beginning of a test, the aquarium was filled with water 16-17 in (40.6-43.2 cm) deep. Silica sand was spread evenly across the floor of the aquarium approximately 1/8 in (3.2 mm) deep before each test. The actuator was placed in the center of the aquarium with the bottom of the horn 1 inch (2.5 cm) from the bottom.

Photos of tank before test: 5 photographs taken.

## Test Series A (A1, A2, A3, A4)

Horn configuration was the following:

- Two fins.
- Four slots in both fins.
- Both fins have 2.5 inch diameter.
- Slots were offset by 45-degree difference between top and bottom fins.
- Fins were 1 inch apart (inside to inside dimension).
- Both fins were 0.050 inches thick.
- Bottom fin (bottom of horn) was 1 inch above aquarium floor.

**Test A1**: Output Conditions: Frequency = 900 Hz, Current = 99.9%, Voltage = 187 volts
Photos: 0 during and 5 after.

**Test A2**: Output Conditions: Frequency = 1136 Hz, Current = 100%, Voltage not recorded
Photos: 2 during and 4 after.

**Test A3**: Output Conditions: Frequency = 1291 Hz, Current = 99.5%, Voltage not recorded
Photos: 2 during and 4 after.

**Test A4**: Output Conditions: Frequency = 1560 Hz, Current = 87%, Voltage not recorded
Photos: 2 during, 5 after.

## Test Series B (B1, B2, B3, B4)

Horn configuration was the following:

- Two fins.
- Four slots in both fins.
- Both fins have 2.5 inch diameter.
- Slots were offset by 45-degree difference between top and bottom fins.
- Fins were 2.0625 inches apart (inside to inside dimension).
- Both fins were 0.050 inches thick.
- Bottom fin (bottom of horn) was 1 inch above aquarium floor.

**Test B1:** Output Conditions: Frequency = 900 Hz, Current = 99.5%, Voltage = 187 volts
Photos: 2 during and 4 after.

**Test B2:** Output Conditions: Frequency = 1139 Hz, Current = 99.8%, Voltage = 239 volts
Photos: 2 during and 6 after.

**Test B3:** Output Conditions: Frequency = 1288 Hz, Current = 98.0%, Voltage = 260 volts
Photos: 2 during and 4 after.

**Test B4:** Output Conditions: Frequency = 1563 Hz, Current = 85.4%, Voltage = 278 volts
Photos: 2 during and 4 after.

**Test Series C (C1,C2,C3,C4)**

Horn configuration was the following:

- One fin.
- Four slots in the single fin.
- The fin has a 2.5 inch diameter.
- Fin is 0.050 inches thick
- Bottom fin (bottom of horn) was 1 inch above aquarium floor.

**Test C1:** Output Conditions: Frequency = 900 Hz, Current = 99.4%, Voltage = 185 volts
Photos: 2 during and 5 after.

**Test C2:** Output Conditions: Frequency = 1178 Hz, Current = 99.7%, Voltage = 278 volts
Photos: 2 during and 4 after.

**Test C3:** Output Conditions: Frequency = 1247 Hz, Current = 98.3%, Voltage = 249 volts
Photos: 3 during and 4 after.

**Test C4:** Output Conditions: Frequency = 1563 Hz, Current = 85.4%, Voltage = 278 volts
Photos: 2 during and 4 after.

**Test Series D (D1,D2,D3,D4)**

Horn configuration was the following:

- One fin.
- No slots in the fin.
- Fin has 2.5 inch diameter.
- Fin is 0.050 inches thick
- Bottom fin (bottom of horn) was 1 inch above aquarium floor.

**Test D1:** Output Conditions: Frequency = 902 Hz, Current = 99.4%, Voltage = 186 volts
Photos: 2 during and 4 after.

**Test D2:** Output Conditions: Frequency = 1147 Hz, Current = 98.8%, Voltage = 238 volts
Photos: 2 during and 4 after.

**Test D3:** Output Conditions: Frequency = 1347 Hz, Current = 94.6%, Voltage = 278 volts
Photos: 4 during and 4 after.

**Test D4:** Output Conditions: Frequency = 1534 Hz, Current = 92.0%, Voltage = 276 volts
Photos: 4 during and 5 after.

**Test Series E (E1,E2,E3,E4)**

Horn configuration was the following:

- Two fins.
- No slots in the fins.
- Fins have 2.5 inch diameter.
- Both fins are 0.050 inches thick.
- Fins were 1 inch apart (inside to inside dimension).
- Bottom fin (bottom of horn) was 1 inch above aquarium floor.

**Test E1**: Output Conditions: Frequency = 901 Hz, Current = 99.2%, Voltage = 186 volts
Photos: 2 during and 4 after.

**Test E2:** Output Conditions: Frequency = 1146 Hz, Current = 98.6%, Voltage = 240 volts
Photos: 2 during and 4 after.

**Test E3:** Output Conditions: Frequency = 1295 Hz, Current = 96.2%, Voltage = 276 volts
Photos: 3 during and 4 after.

**Test E4:** Output Conditions: Frequency = 1538 Hz, Current = 92.2%, Voltage = 275 volts
Photos: 2 during and 4 after.

**Test Series F (F1,F2,F3,F4)**

Horn configuration was the following:

- Two fins.
- No slots in the fins.
- Fins have 2.5 inch diameter.
- Both fins are 0.050 inches thick.
- Fins were 2.0625 inches apart (inside to inside dimension).
- Bottom fin (bottom of horn) was 1 inch above aquarium floor.

**Test F1**: Output Conditions: Frequency = 903 Hz, Current = 99.0%, Voltage = 186 volts
Photos: 2 during and 4 after.

**Test F2:** Output Conditions: Frequency = 1147 Hz, Current = 99.6%, Voltage = 242 volts
Photos: 0 during and 5 after.

**Test F3:** Output Conditions: Frequency = 1280 Hz, Current = 95.1%, Voltage = 276 volts
Photos: 2 during and 6 after.

**Test F4:** Output Conditions: Frequency = 1420 Hz, Current = 95.0%, Voltage = 276 volts
Photos: 3 during and 5 after.

The observations made during each of these test are presented on the following pages of this appendix.

DATE 9/24/2003
TIME 1:34 PM EDT
TEST NUMBER A1

TEST CONDITIONS

FREQUENCY 900 Hz
CURRENT 99.9%
VOLTAGE 187

AQUARIUM CONFIGURATION

Actuator centered (6 inches from top and bottom 15" from sides)
Bottom of Horn is 1 inch above sand
Sand is 1/8 in. deep

HORN INFORMATION

Double Finned (fins are 0.050 inch thick)
Slotted (4 slots per fin)
Slots not aligned (45% out of alignment)
Fins are 1 inch apart

OBSERVATIONS DURING THE TEST

After 2 to 3 minutes, the sand clears out from immediately under the bottom fin. At 6 to 6.5 minutes, the hole (circle) under the bottom fin is becoming elongated (elliptical) on the east (right hand side as you are looking at the aquarium). Bubble continues to dance on bottom fin's west side (left) at the 8 to 8.5 minute mark. At the 9 minute mark, the bubble floats off. Then a bubble forms on the NE quadrant of the top fin. Sand begins to fill in around circle (hole) under the horn on the E and W sides. Twin circles begin appearing on both the E&W sides. The sand directly underneath the horn is not as cleared out as before. Bubbles at 11 minute mark switched to SW quadrant of top fin at the 12.5 minute mark. Three circles are now becoming one blob. Test was shutdown at approximately 13-14 minute mark. Five photos were taken before and 5 photos after test. 2 circles, one ellipse and one big blob observed at end of test (six holes).

DATE 9/24/2003
TIME 1:55 PM EDT
TEST NUMBER A2

TEST CONDITIONS

FREQUENCY 1136 Hz
CURRENT 100%
VOLTAGE

AQUARIUM CONFIGURATION

Actuator centered (6 inches from top and bottom 15" from sides)
Bottom of Horn is 1 inch above sand
Sand is 1/8 in. deep

HORN INFORMATION

Double Finned (fins are 0.050 inch thick)
Slotted (4 slots per fin)
Slots not aligned (45% out of alignment)
Fins are 1 inch apart

OBSERVATIONS DURING THE TEST

During the first thirty seconds, sand is drawn to the center of the bottom fin. Bubbles start appearing on all four quadrants of both top and bottom fins. Three distinct "holes" appear to the east (right of the actuator/horn at about the 2-minute mark. Sand is now being pushed away (clear spot) form directly underneath the bottom fin at the 2.5-minute mark. At 3 minutes, sand is being drawn into a cone shape underneath the bottom fin. At 3.5 minutes, there are four holes around the bottom fin and seven other "holes" appear throughout the aquarium. At 4 minutes, the cone disintegrates. At 4.5 minutes the buildup underneath the bottom fin begins again. Photos 11 and 1 are taken at the 5-minute mark. There appears to be more bubbles on the fins than at the 900 Hz (test A1). Asymmetry under the bottom fin is due to the bubbles attracting the sand particles. At the 10-minute mark several bubbles are appearing on all 8 quadrants. Five photos (#13-16) were taken after test. A total of thirteen "holes" was observed.

DATE 9/24/2003
TIME 2:22 PM EDT
TEST NUMBER A3

TEST CONDITIONS
FREQUENCY 1291 Hz
CURRENT 99.5%
VOLTAGE

AQUARIUM CONFIGURATION
Actuator centered (6 inches from top and bottom 15" from sides)
Bottom of Horn is 1 inch above sand
Sand is 1/8 in. deep

HORN INFORMATION
Double Finned (fins are 0.050 inch thick)
Slotted (4 slots per fin)
Slots not aligned (45% out of alignment)
Fins are 1 inch apart

OBSERVATIONS DURING THE TEST

During the first minute, sand is moving rapidly. A big "hole" appears just west (left) of the actuator. Lots of big bubbles with lots of activity (bubble action) were observed on underneath side of bottom fin. Very seldom was there a "hole" directly underneath the bottom fin. Typically there was a cone building up, then a flattening out of the pile of sand and then a build-up to a peak on the cone. This pattern kept on repeating several times during the test. Photos #17 & #18 were taken at the 10-minute mark. Test concluded at the approximately the 11 minute mark when photos # 19 to #22 were taken. Twenty "holes" were counted at the end of the test. This appeared to be the most aggressive movement of sand observed as part of this series of tests.

DATE 9/24/2003
TIME 2:46 PM EDT
TEST NUMBER A4

TEST CONDITIONS

FREQUENCY 1560 Hz
CURRENT 87%
VOLTAGE

AQUARIUM CONFIGURATION

Actuator centered (6 inches from top and bottom 15" from sides)
Bottom of Horn is 1 inch above sand
Sand is 1/8 in. deep

HORN INFORMATION

Double Finned (fins are 0.050 inch thick)
Slotted (4 slots per fin)
Slots not aligned (45% out of alignment)
Fins are 1 inch apart

OBSERVATIONS DURING THE TEST

During the first minute, 2 "holes" appeared on the west (left) side of the actuator/horn and one "hole" appeared on the east (right) side. The bubbles that appeared on all 4 quadrants of both fins were much smaller than those observed during test A3. At the 3-minute mark, two additional "holes" appeared on the east (right) side of the horn. Photos # 23 and #24 were taken at the 5-minute mark. Minimal activity on the underneath side of the bottom fin was observed throughout test A4. Not much really happening during test A4. Test was shut down at the 11-minute mark, when photos # 25 to #29 were taken. A total of six "holes" were observed at the end of test A4.

DATE 9/25/2003
TIME 11:01 AM EDT
TEST NUMBER B1

TEST CONDITIONS

FREQUENCY 900 Hz
CURRENT 99.5%
VOLTAGE 187

AQUARIUM CONFIGURATION

Actuator centered (6 inches from top and bottom 15" from sides)
Bottom of Horn is 1 inch above sand
Sand is 1/8 in. deep

HORN INFORMATION

Double Finned (fins are 0.050 inch thick)
Slotted (4 slots per fin)
Slots not aligned (45% out of alignment)
Fins are 2.0625 inches apart

OBSERVATIONS DURING THE TEST

Initially not much action. Even after 4 minutes, not much is happening. No sand appears to be moving. After 5 minutes, a small hole starts forming under the horn. A few bubbles are observed on the bottom fin. Test ended at 11 minutes. One hole, 1.5 inches in diameter, directly under the horns/actuator.

DATE 9/25/2003
TIME 11:32 AM EDT
TEST NUMBER B2

TEST CONDITIONS

FREQUENCY 1139 Hz
CURRENT 99.8%
VOLTAGE 239

AQUARIUM CONFIGURATION

Actuator centered (6 inches from top and bottom 15" from sides)
Bottom of Horn is 1 inch above sand
Sand is 1/8 in. deep

HORN INFORMATION

Double Finned (fins are 0.050 inch thick)
Slotted (4 slots per fin)
Slots not aligned (45% out of alignment)
Fins are 2.0625 inches apart

OBSERVATIONS DURING THE TEST

Immediately upon starting the test, a large 4-inch oval begins to appear on the east (right) side. During the first thirty seconds, two ovals 2.5 inches long and 1 inch wide start to appear on the north and south. At time equals one minute, a small (1-inch diameter) begins to appear northeast of the actuator/horn. One large bubble appears on all eight quadrants on the horns. One bubble appears to be moving sand on the underneath side of the bottom fin at time = 2 minutes. The bubbles on the top of both fins appear to pair up (NW and SE being large and NE and SW being smaller) before they bubble off towards the top of the aquarium. Between the t=5 and t=8 minute marks, several small holes opened up on the east and west sides. Test concluded at t=11 minutes. Fourteen holes were observed at the end of the test.

DATE 9/25/2003
TIME 1:12 PM EDT
TEST NUMBER B3

TEST CONDITIONS

FREQUENCY 1288 Hz
CURRENT 98.0%
VOLTAGE 260

AQUARIUM CONFIGURATION

Actuator centered (6 inches from top and bottom 15" from sides)
Bottom of Horn is 1 inch above sand
Sand is 1/8 in. deep

HORN INFORMATION

Double Finned (fins are 0.050 inch thick)
Slotted (4 slots per fin)
Slots not aligned (45% out of alignment)
Fins are 2.0625 inches apart

OBSERVATIONS DURING THE TEST

During the first minute, bubbles appeared on all four quadrants on both the top and bottom fins. During the first minute a 2-2.5 inch diameter hole opened up on the west. There is also activity on the underneath side of the bottom fin. At t=2 minutes, a 1.5 inch diameter hole begins NE of the actuator. At t=3, the west hole begins filling in and the NE hole expands to 2 to 2.5 inch diameter. At t=4.4 minute, a large oval 2-3 inches long and 2-2.5 inches in diameter is forming on west-southwest side. The NE hole now begins to diminish and begins filling in. At t=6.5 west-southwest oval has increased to include a hole to the south. Meanwhile sand is coned up directly under the horn. At t=8, a ridge begins to form around the west to south arc of the hole. At t=9, the crescent is moving north, with the southern portion filling in. The ridge is sort of heart shaped. Test concluded at t=10 minutes. Only one hole observed. The ridge is about 3/8 inch high.

DATE 9/25/2003
TIME 1:32 PM EDT
TEST NUMBER B4

TEST CONDITIONS

FREQUENCY 1563 Hz
CURRENT 85.4%
VOLTAGE 278

AQUARIUM CONFIGURATION

Actuator centered (6 inches from top and bottom 15" from sides)
Bottom of Horn is 1 inch above sand
Sand is 1/8 in. deep

HORN INFORMATION

Double Finned (fins are 0.050 inch thick)
Slotted (4 slots per fin)
Slots not aligned (45% out of alignment)
Fins are 2.0625 inches apart

OBSERVATIONS DURING THE TEST

Small bubbles forming mainly on the top fin. Very little action. At t=5 minutes no sand movement is observed. At t=7.5 minutes a slight increase in bubble size on top fin. Nothing else is happening. Tiny bubbles are forming (but not growing on the lower fin. Test concluded at t=10 minutes. No holes observed. Very little if any sand moved during this entire ten-minute test.

DATE 9/25/2003
TIME 2:47 PM EDT
TEST NUMBER C1

TEST CONDITIONS
FREQUENCY 900 Hz
CURRENT 99.4%
VOLTAGE 185

AQUARIUM CONFIGURATION
Actuator centered (6 inches from top and bottom 15” from sides)
Bottom of Horn is 1 inch above sand
Sand is 1/8 in. deep

HORN INFORMATION
Single Fin (0.050 inch thick)
Slotted (4 slots per fin)

OBSERVATIONS DURING THE TEST

Initially some sand started out on top of fin. Not much else is happening. Even after 5 minutes, not much is happening. Sand grains on top fin are moving/swaying, but no bubbles. Test ended after 10 minutes. No holes.

DATE 9/25/2003
TIME 3:04 PM EDT
TEST NUMBER C2

TEST CONDITIONS

FREQUENCY 1178 Hz
CURRENT 99.7%
VOLTAGE 278

AQUARIUM CONFIGURATION

Actuator centered (6 inches from top and bottom 15" from sides)
Bottom of Horn is 1 inch above sand
Sand is 1/8 in. deep

HORN INFORMATION

Single Fin (0.050 inch thick)
Slotted (4 slots per fin)

OBSERVATIONS DURING THE TEST

Immediately upon starting the test, 2 big bubbles appear on the SW and SE quadrants and sand begins pushing out and away from directly underneath the horn. At t=1 minute, 2 holes (1 inch diameter) appear 4" east on the centerline of the aquarium, plus a 2 inch diameter hole appears 4 inches north of horn. At time = 2 minutes, a big bubble is added to the NE quadrant. At time = 5 minutes, a second 1 inch hole appears on the east side of the centerline. At time = 8 minutes, a hole begins to appear directly under the NE quadrant of the horn. At time = 9, the NE quadrant hole starts expanding to the SW. There are now 3 big bubbles on the top, with a small bubble on the other (NE) quadrant of the horn. Test concluded at t=11 minutes. Five holes were observed at the end of the test.

DATE 9/25/2003
TIME 3:25 PM EDT
TEST NUMBER C3

TEST CONDITIONS
FREQUENCY 1247 Hz
CURRENT 98.3%
VOLTAGE 249

AQUARIUM CONFIGURATION
Actuator centered (6 inches from top and bottom 15" from sides)
Bottom of Horn is 1 inch above sand
Sand is 1/8 in. deep

HORN INFORMATION
Single Fin (0.050 inch thick)
Slotted (4 slots per fin)

OBSERVATIONS DURING THE TEST

Immediately, sand starts swirling. Holes begin to appear at 6 inches west of the centerline, 4 and 7 inches east of the centerline. A big oval begins to swirl from 2 inches east to 2 inches west of the centerline. This area includes heavy activity directly under the fin. There is only one big bubble on top of the NE quadrant of the fin. At time 4 min, and additional hole 6 inches north of the actuator appears. At time = 6 min, three one-inch holes appear at 4.5 inches east of the west edge of the aquarium, each one 0.5 to one inch in diameter. A big bubble is now on the shaft and one on the NE quadrant of the fin. Test concluded at 10.5 minutes. 14 holes were observed at the end of the test.

DATE 9/25/2003
TIME 3:58 PM EDT
TEST NUMBER C4

TEST CONDITIONS
FREQUENCY 1563 Hz
CURRENT 85.4%
VOLTAGE 278

AQUARIUM CONFIGURATION
Actuator centered (6 inches from top and bottom 15” from sides)
Bottom of Horn is 1 inch above sand
Sand is 1/8 in. deep

HORN INFORMATION
Single Fin (0.050 inch thick)
Slotted (4 slots per fin)

OBSERVATIONS DURING THE TEST

Not much happening at the beginning. No bubbles, no sand movement. At t=4 min, same as at the beginning. At t=10 min, no activity, no bubbles. Test concluded at 11 minutes. No holes observed. Very little if any sand moved during this entire test.

DATE 9/26/2003
TIME 9:40 AM EDT
TEST NUMBER D1

TEST CONDITIONS

FREQUENCY 902 Hz
CURRENT 99.4%
VOLTAGE 186

AQUARIUM CONFIGURATION

Actuator centered (6 inches from top and bottom 15" from sides)
Bottom of Horn is 1 inch above sand
Sand is 1/8 in. deep

HORN INFORMATION

Single Fin (0.050 inch thick)
No Slots

OBSERVATIONS DURING THE TEST

Some sand movement just west of midpoint at t= 0.5 minutes. At t=1.5 minutes, some clockwise movement of sand at center point. This type of motion continued throughout the test, but no real holes developed. Motion at (2, 0), but nowhere else. Test ended at t=11.5 minutes.

DATE 9/26/2003
TIME 10:08 AM EDT
TEST NUMBER D2

TEST CONDITIONS
FREQUENCY 1147 Hz
CURRENT 98.8%
VOLTAGE 238

AQUARIUM CONFIGURATION
Actuator centered (6 inches from top and bottom 15" from sides)
Bottom of Horn is 1 inch above sand
Sand is 1/8 in. deep

HORN INFORMATION
Single Fin (0.050 inch thick)
No Slots

OBSERVATIONS DURING THE TEST

Initially, tiny bubble is dancing around in the SE quadrant of the horn. Some sand is moving directly under the horn. At t=2 minutes, three one-inch diameter holes are forming at 1.5 inches from the south edge on the centerline and 2 inches SE of centerline and at north edge at the centerline. At t=4.5 minutes, a 1 inch diameter hole is forming, 6.5 inches east of and on the centerline. At t=5.5 min, 10 inches from the W edge of the aquarium, 1.5 inches from the southern edge, a 0.5 inch hole has opened up. The hole at the north edge on the centerline has become three holes. The hole on the centerline and 1.5 inches from the south edge has become an ellipse 2 inches long by 1 inch wide. A new hole has begun developing at 10.5 inches in line with the others. At t= 9 minutes, the three holes at the north edge have become one ellipse. Sand had mounded up under the horn. Test was concluded at t= 10 minutes. 8 holes were observed.

DATE 9/26/2003
TIME 10:27 AM EDT
TEST NUMBER D3

TEST CONDITIONS

FREQUENCY 1347 Hz
CURRENT 94.6%
VOLTAGE 278

AQUARIUM CONFIGURATION

Actuator centered (6 inches from top and bottom 15" from sides)
Bottom of Horn is 1 inch above sand
Sand is 1/8 in. deep

HORN INFORMATION

Single Fin (0.050 inch thick)
No Slots

OBSERVATIONS DURING THE TEST

Immediately big bubbles are underneath the horn, driving sand away forming a crescent/smile along the south edge of the fin. 2-2.5 inches south of that hole are two more holes near the south edge of the aquarium. A large (1.5 inch wide by 3 inch long) hole butts up against the north edge at the centerline. Two one-inch diameter holes 7 inches off the west edge and 1 inch north of the centerline and 2.5 inches north of the centerline. Two holes (half-circles) are at the north edge 3.5 inches from the centerline and a 1-inch diameter hole approximately 1.5 inches south of that previous hole. At t=5 minutes, this hole has sand swirling in cone shape—this stopped at t=6 minutes. A new hole 3 inches west of there. At t=7.5 minutes, the bubble underneath the fin continue to stir/swirl sand into a mound. Test concluded at t= 10 minutes. 12 holes were observed.

DATE 9/26/2003
TIME 11:08 AM EDT
TEST NUMBER D4

TEST CONDITIONS
FREQUENCY 1534 Hz
CURRENT 92.0%
VOLTAGE 276

AQUARIUM CONFIGURATION
Actuator centered (6 inches from top and bottom 15” from sides)
Bottom of Horn is 1 inch above sand
Sand is 1/8 in. deep

HORN INFORMATION
Single Fin (0.050 inch thick)
No Slots

OBSERVATIONS DURING THE TEST

Nine holes formed almost immediately. Sand movement is very active, with fine sand particles swirling up above the top of the horn towards the actuator. Movement so active that the aquarium is cloudy due to suspended solids (sand grains and other fine particles). Aquarium was too cloudy to count the number of holes during the 10 minutes of this test. Once test concluded at t=10 minutes, the aquarium was allowed to settle w/o taking photos. At 1:20 p.m. EDT, an attempt was made to count, photograph and locate the 20 holes formed during the test.

DATE 9/30/2003
TIME 11:34 AM EDT
TEST NUMBER E1

TEST CONDITIONS
FREQUENCY 901 Hz
CURRENT 99.2%
VOLTAGE 186

AQUARIUM CONFIGURATION
Actuator centered (6 inches from top and bottom 15" from sides)
Bottom of Horn is 1 inch above sand
Sand is 1/8 in. deep

HORN INFORMATION
Double Finned, each 0.050 inch thick
No Slots
Fins are 1 inch apart

OBSERVATIONS DURING THE TEST

At the beginning, a large bubble began traversing the bottom of the lower fin with one small bubble on top of each fin. At t=1.45 minute, two small bubbles appear and a 1-inch diameter hole appears just SW of the center point of the aquarium. At t=2 min, a 1-inch hole appears one inch west of the eastern edge of the aquarium on the centerline and a 1-inch diameter hole appears at (10, 4). At t=4, the hole just southwest of the center point is becoming elliptical. There is still one bubble on each of the fin's bottom surface and on the top fin a single bubble continues to move about. At t= 7 min, a large bubble on the underneath side of the bottom fin appears in its SE quadrant. At t= 8.5 min, the bubble on the underneath side of the bottom fin moves more to the east and sand begins filling in the ellipse to the west of the center point as the bubble moves to the northwest. Test ended at t=10 minutes. Only three holes observed at the end.

DATE 9/30/2003
TIME 12:10 PM EDT
TEST NUMBER E2

TEST CONDITIONS
FREQUENCY 1146 Hz
CURRENT 98.6%
VOLTAGE 240

AQUARIUM CONFIGURATION
Actuator centered (6 inches from top and bottom 15" from sides)
Bottom of Horn is 1 inch above sand
Sand is 1/8 in. deep

HORN INFORMATION
Double Finned, each 0.050 inch thick
No Slots
Fins are 1 inch apart

OBSERVATIONS DURING THE TEST

Initially, the sand begins mounding underneath the horn. Within the first two minutes, ten holes appear. Almost continuously throughout the test, one bubble appears on each fin and sand continues to mound up directly underneath the horn. At t=8.5 min, two bubbles are on the top fin. At t=9.5 min, three additional 0.5-inch diameter holes appear. A total of thirteen holes are observed when the test concluded at t=10 minutes.

DATE 9/30/2003
TIME 1:25 PM EDT
TEST NUMBER E3

TEST CONDITIONS

FREQUENCY 1295 Hz
CURRENT 96.2%
VOLTAGE 276

AQUARIUM CONFIGURATION

Actuator centered (6 inches from top and bottom 15" from sides)
Bottom of Horn is 1 inch above sand
Sand is 1/8 in. deep

HORN INFORMATION

Double Finned, each 0.050 inch thick
No Slots
Fins are 1 inch apart

OBSERVATIONS DURING THE TEST

Immediately two holes appear—a 2-inch diameter hole southwest of the center point of the aquarium and a 2" by 1" north-south ellipse at 23 inches from the west edge on the centerline. At t=1 min, all kinds of holes begin to form. One bubble is underneath the bottom fin, and 1-2 bubbles are racing around on the topside of each fin. At t=2 min, and at t=4.5 min, bubbles on the underneath side of the top fin are observed. Sand is constantly moving underneath the horn. At t=6 min, a big mound of sand is collecting underneath the horn. At t=9 min, bubble is observed on shaft above the horn. Test ended at t=10 minutes, when 17 holes were counted.

DATE 9/30/2003
TIME 2:15 PM EDT
TEST NUMBER E4

TEST CONDITIONS
FREQUENCY 1538 Hz
CURRENT 92.2%
VOLTAGE 275

AQUARIUM CONFIGURATION
Actuator centered (6 inches from top and bottom 15" from sides)
Bottom of Horn is 1 inch above sand
Sand is 1/8 in. deep

HORN INFORMATION
Double Finned, each 0.050 inch thick
No Slots
Fins are 1 inch apart

OBSERVATIONS DURING THE TEST

Within the first minute, 17 holes are formed. At t=2 min, four bubbles are observed on top of the bottom fin. Also a big mound of sand is forming underneath the horn. At t=5 min, lots of sediment fines are being kicked up to cloud up the aquarium. At t= 6 min, the bottom horn manages to unscrew itself. Test is suspended and horn is reattached. We are still observing four bubbles chasing each other counterclockwise around the top of the bottom fin. Lots of fine sediment being stirred up and aquarium is very cloudy. Mound directly underneath the horn is so high that bubbles on the bottom fin are picking up sand. At t=8.5 min, "clear" bubbles appear on both fins on the outer circumference surface. Very unusual observations were noted during this test. Test concludes at t=10 min with a very dark/cloudy aquarium. After some 45 minutes, 28 holes were counted.

DATE 10/01/2003
TIME 3:22 PM EDT
TEST NUMBER F1

TEST CONDITIONS

FREQUENCY 903 Hz
CURRENT 99.0%
VOLTAGE 186

AQUARIUM CONFIGURATION

Actuator centered (6 inches from top and bottom 15" from sides)
Bottom of Horn is 1 inch above sand
Sand is 1/8 in. deep

HORN INFORMATION

Double Finned, each 0.050 inch thick
No Slots
Fins are 2.0625 inches apart

OBSERVATIONS DURING THE TEST

Within the first 30 seconds, a 2-in hole appears under and southwest of the center point. At t=2 min, 5 more holes appear. One hole is north-northwest of center point. Three more holes are at 2 in, 3 in, and 5 in east of the west edge. The fifth hole is 5 in west of the east edge and 2 in south of north edge. At t=5.5 min, holes 2, 3 and 4 have merged into an ellipse 1.5 in by 3 – 3.5 in long running east to west. Test concluded at t=11 minutes. 8 holes observed.

DATE 10/01/2003
TIME 3:43 PM EDT
TEST NUMBER F2

TEST CONDITIONS
FREQUENCY 1147 Hz
CURRENT 99.6%
VOLTAGE 242

AQUARIUM CONFIGURATION
Actuator centered (6 inches from top and bottom 15" from sides)
Bottom of Horn is 1 inch above sand
Sand is 1/8 in. deep

HORN INFORMATION
Double Finned, each 0.050 inch thick
No Slots
Fins are 2.0625 inches apart

OBSERVATIONS DURING THE TEST

Within the first minute, 6 holes appear. There is a bubble on each side (top and bottom) of each fin. At t=2 min, 10 holes ar3e observed. At t=2.5 min, a bubble runs up and down on horn shaft between the two fins. At t=3 min, mounding of sand is noticed at the center point (directly underneath the horn/actuator). At t=4 min, 13 holes are now observed. At t=6 min, two bubbles are observed on the topside of each fin. At t=7.5 min, only a tiny bubble on the topside of the bottom fin is observed. At t=8 min, the only bubble observed is a big one on the underneath side of the top fin. Test concluded at t=10 minutes. 15 holes were counted after the test concluded.

DATE 10/02/2003
TIME 1:02 PM EDT
TEST NUMBER F3

TEST CONDITIONS

FREQUENCY 1280 Hz
CURRENT 95.1%
VOLTAGE 276

AQUARIUM CONFIGURATION

Actuator centered (6 inches from top and bottom 15" from sides)
Bottom of Horn is 1 inch above sand
Sand is 1/8 in. deep

HORN INFORMATION

Double Finned, each 0.050 inch thick
No Slots
Fins are 2.0625 inches apart

OBSERVATIONS DURING THE TEST

Immediately at the beginning of the test, 8 holes appear. At t=1 minute, sand is mixing on bottom fin all the way up to underneath side of upper fin. At t-=1.5 mon, holes elongate to ellipses running north-south, 4 – 5 inches long and 1 – 2.5 inches wide. At t=4 min, chaos is occurring in the tank. At t=5 min, there are bubbles racing around the topside of the top fin, both near the shaft and around the far edge of the fin (~1 inch from shaft). At t=5.5 minutes, three- to four-inch thick fog clouds the entire bottom of the aquarium. At t= 7.5 minutes, fog is now 3.5 to 4.5 inches thick. At t= 9 min, fog at center point of the aquarium is 5 inches thick and is as high as 6 inches above the bottom of the aquarium in several other spots. Test concluded at t=10 minutes. After the fog cleared, it was noticed that the bottom fin had come unscrewed during the 10-minute test. Due to the dense sediment cloud in the aquarium, it was not possible to observe exactly when it came off. Twelve large holes observed after fog settled sufficiently to document the results.

DATE 10/01/2003
TIME 4:06 PM EDT
TEST NUMBER F4

TEST CONDITIONS

FREQUENCY 1420 Hz
CURRENT 95.0%
VOLTAGE 276

AQUARIUM CONFIGURATION

Actuator centered (6 inches from top and bottom 15” from sides)
Bottom of Horn is 1 inch above sand
Sand is 1/8 in. deep

HORN INFORMATION

Double Finned, each 0.050 inch thick
No Slots
Fins are 2.0625 inches apart

OBSERVATIONS DURING THE TEST

Chaos. Immediately aquarium looks cloudy/murky. Many holes appear within the first minute. It looks like Swiss cheese. Sand is mixing with the bubbles on both sides of the bottom fin and creating a “dust devil” (sediment vortex). At t=5.5 min, sand is being kicked up so high that it is mixing with he bubbles on the topside of the top fin. Test over at t=10 minutes.

# APPENDIX E

## Brookfield Digital Viscometer Operating Manual

BROOKFIELD DIGITAL VISCOMETER

**MODEL DV-E**

Operating Instructions

Manual No. **M/98-350-E1203**

BROOKFIELD

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# I. INTRODUCTION

The Brookfield **DV-E** Viscometer measures fluid viscosity at given shear rates. Viscosity is a measure of a fluid's resistance to flow. You will find a detailed description of the mathematics of viscosity in the Brookfield publication *"More Solutions to Sticky Problems"* a copy of which was included with your **DV-E** and can be downloaded in pdf form from the Brookfield website.

The principle of operation of the **DV-E** is to rotate a spindle (which is immersed in the test fluid) through a calibrated spring. The viscous drag of the fluid against the spindle is measured by the spring deflection. Spring deflection is measured with a rotary transducer which provides a torque signal. The measurement range of a **DV-E** (in centipoise or milliPascal seconds) is determined by the rotational speed of the spindle, the size and shape of the spindle, the container in which the spindle is rotating, and the full scale torque of the calibrated spring.

There are four basic spring torque series offered by Brookfield:

| | **Spring Torque** | |
|---|---|---|
| Model | dyne-cm | milli Newton-m |
| LVDV-E | 673.7 | 0.0673 |
| RVDV-E | 7,187.0 | 0.7187 |
| HADV-E | 14,374.0 | 1.4374 |
| HBDV-E | 57,496.0 | 5.7496 |

The higher the spring torque, the higher the measurement range. The viscosity measurement range for each spring torque may be found in **Appendix A.**

**All units of measurement are displayed according to either the CGS (cP) system or the SI (mPa•s) system.**

1. Viscosity appears in units of centipoise (shown as "cP") or milliPascal-seconds (shown as "mPa•s") on the **DV-E** display.
2. Torque appears in units of dyne-centimeters or Newton-meters (shown as percent "%" in both cases) on the **DV-E** display.

The equivalent units of measurement in the SI system are calculated using the following conversions:

| | **SI** | | **CGS** |
|---|---|---|---|
| Viscosity: | 1 mPa•s | = | 1 cP |
| Torque: | 1 Newton-m | = | $10^7$ dyne-cm |

References to viscosity throughout this manual are made in CGS units. The **DV-E** Viscometer provides equivalent information in SI units (see Section II.4 AUTORANGE).

## I.1 Components

1) **DV-E** Viscometer
2) Laboratory Stand: Model A
3) Spindle Set with Case (4 spindles for **LVDV-E**; 6 Spindles for **RV, HA** and **HBDV-E**).
4) Power Cord
5) Guard Leg **(LVDV-E** and **RVDV-E** only)
6) Carrying Case
7) Shipping Cap

Please check to be sure that you have received all components, and that there is no damage. If you are missing any parts, please notify Brookfield Engineering or your local Brookfield agent immediately. Any shipping damage must be reported to the carrier.

## I.2 Utilities

| | |
|---|---|
| Input Voltage: | 115 VAC or 230 VAC |
| Input Frequency: | 50/60 Hz |
| Power Consumption: | Less than 20 WATTS |

| Power Cord Color Code: | United States | Outside United States |
|---|---|---|
| Hot (live) | Black | Brown |
| Neutral | White | Blue |
| Ground (earth) | Green | Green/Yellow |

## I.3 Specifications

Speeds: 0.3, 0.5, 0.6, 1.0, 1.5, 2.0, 2.5, 3.0, 4.0, 5.0, 6.0, 10, 12, 20, 30, 50, 60, 100

| Weight: | | | |
|---|---|---|---|
| | Gross Weight | 20 lb | 9 kg |
| | Net Weight | 17 lb | 7.7 kg |
| | Carton Volume | 1.65 cu ft | 0.05 $m^3$ |
| | Carton Dimension | 19 x 10 x 15 in | 48 x 25 x 38 cm |

Operating Environment: 0°C to 40°C Temperature Range (32°F to 104°F)
20% - 80% R.H.: non-condensing atmosphere

Accuracy: ±1.0% Full Scale Range in Use (See **Appendix D** for details)

Reproducibility: 0.2% of Full Scale Range

Electrical Certifications:
Conforms to CE Standards:
BSEN 50081-1: Emission Standard - Light Industrial
BSEN 50082-1: Immunity Standard - Light Industrial
BSEN 50081-2: Emission Standard - Industrial
BSEN 50082-2: Immunity Standard - Industrial
BSEN 61010-1: Safety requirements for electrical equipment, for measurement, control and laboratory use

Approved Standards:
CSA Std. C22.2 No. 151-M1986 - Laboratory Equipment
CSA Class 8721 81 - Laboratory Equipment

This product has been certified to the applicable CSA and ANSI/UL Standards, for use in Canada and the U.S.

Installation Category (over-voltage category) II: Classification of parts of installation systems or circuits in local level, portable equipment, appliances, etc..

## I.4 Set-Up

1. To assemble the Model A Laboratory Stand, place the upright rod into the base (refer to assembly instructions in **Appendix E**). The rack gear and clamp assembly should face the front of the base. The upright rod is held in place with the jam nut which is attached from the bottom of the base. Tighten this nut with a suitable wrench (spanner). Attach leveling feet.

2. Insert the mounting rod on the back of the **DV-E** Viscometer into the hole on the clamp assembly. Be sure that the clamp screw, VS-41Y, is loose.

3. Adjust the Viscometer to be as close to level as possible while tightening the clamp screw. Tighten the VS-41Y clamp screw.

4. The Viscometer must be leveled. The level is adjusted using the three leveling screws on the base. Adjust so that the bubble level on top of the **DV-E** is centered within the circle.

   **Note:** Check level periodically during use.

5. Remove the Viscometer shipping cap from the pivot cup. This cap is designed to protect the Viscometer spindle coupling nut during shipment. ***Do not attempt to operate the Viscometer with the shipping cap in place!***

6. Make sure that the AC power switch at the rear of the **DV-E** is in the **OFF** position. Connect the power cord to the socket on the back panel of the instrument and plug it into the appropriate AC line.

⚠ ***The AC input voltage and frequency must be within the appropriate range as shown on the name plate of the viscometer.***

***The DV-E must be earth grounded to ensure against electronic failure!!***

## I.5 Safety Symbols and Precautions

**Safety Symbols**

The following explains safety symbols which may be found in this operating manual.

⚠ Indicates hazardous voltages may be present.

⚠ Refer to the manual for specific warning or caution information to avoid personal injury or damage to the instrument.

**Precautions**

⚠ If this instrument is used in a manner not specified by the manufacturer, the protection provided by the instrument may be impaired.

⚠ This instrument is not intended for use in a potentially hazardous environment.

⚠ In case of emergency, turn off the instrument and then disconnect the elecrical cord from the wall outlet

⚠ The user should ensure that the substances placed under test do not release poisonous, toxic or flammable gases at the temperatures to which they are subjected to during the testing.

## I.6 Instrument Controls

The following describes each switch's function:

***MOTOR ON***

Turns the motor **ON** or **OFF**.

***AUTO RANGE***

Presents the maximum (100% torque) viscosity attainable using the selected spindle at the selected speed. This value is referred to as *full scale range*. The allowable error for the viscosity measurement is ± 1% of full scale range.

> **Note: Pressing and holding the AUTO RANGE key during power on will enable the viscosity display to be read in either CGS (cP) or SI (mPa•s) units.**

***SPEED/SPINDLE SWITCH***

Sets the viscometer in either speed select or spindle select (see Table C1 in Appendix C) mode. When set in the left position, the operator may select speed of rotation. When set in the right position, the operator may select spindle.

> **Note: This is a three (3) position switch. We recommend that the switch be set to the middle position when finished with spindle or speed adjustment. This will prevent an accidental change of parameters during a test.**

***SELECT KNOB***

This knob is used to scroll through the available speed or spindle selections (see Table C1 in Appendix C). This knob is active when the switch is set to the left (speed) or right (spindle) position.

Rotate the knob clockwise to increase value and counter-clockwise to decrease value.

## I.7 Cleaning

⚠ **Be sure to remove spindle from instrument prior to cleaning. Severe instrument damage may result if cleaned in place.**

| | |
|---|---|
| Instrument and Keypad: | Clean with dry, non-abrasive cloth. Do not use solvents or cleaners. |
| Immersed Components (spindles): | Spindles are made of stainless steel. Clean with non-abrasive cloth and solvent appropriate for sample material that is not aggressive to immersed components. |

⚠ **When cleaning, do not apply excessive force which may result in bending spindles.**

# II. GETTING STARTED

## II.1 Power Up

Turn the power switch (located on the rear panel) to the **ON** position. This will result in the following screen display:


Figure II-1

After a few seconds, the following screen appears:


Figure II-2

After a short time, the display will clear and the default screen is displayed:


Figure II-3

## II.2 Spindle Selection

LVDV-E Viscometers are provided with a set of four spindles and a narrow guardleg; RVDV-E Viscometers come with a set of six spindles and a "wider" guardleg; HADV-E and HBDV-E Viscometers come with a set of six spindles and no guardleg. (See **Appendix D** for more information on the guardleg.)

The spindles are attached to the viscometer by screwing them to the male coupling nut. Note that the spindles and coupling have a **left-hand thread**. The lower shaft should be held in one hand (lifted slightly), and the spindle screwed to the left. The face of the spindle nut and the matching surface on the coupling nut shaft should be smooth and clean to prevent eccentric rotation of the spindle. Spindles can be identified by the number on the side of the spindle coupling nut.

The **DV-E** must have a Spindle Entry Code number to calculate viscosity values. The **DV-E** memory contains parameters for all standard Brookfield spindles and the two digit entry code for each spindle (the complete list of spindle entry codes may be found in **Appendix C**).

**Note: The DV-E will display the Spindle Entry Code which was in use when power was turned off.**

Setting the SPEED/SPINDLE switch to the right position will allow the operator to adjust the spindle selection. The SELECT knob can be rotated until the desired spindle number is selected. Once the desired spindle number is shown on the display, set the SPINDLE/SPEED switch to the middle position.

**Note:** Verify the proper spindle entry code for the selected spindle found in Appendix C. Not all spindles have an entry code number that is the same as the spindle number. For example: the spindle entry code for spindle LV1 is 61 and the spindle entry code for UL Adapter is 00.

The **DV-E** will begin to calculate using the new spindle parameters after the spindle number is shown in the display.

Please see Brookfield publication, "More Solutions to Sticky Problems" (Chapter 3), for information on how to select a spindle.

## II.3 Speed Selection & Setting

There are 18 rotational speeds available on the **DV-E** Viscometer. These speeds correspond to the standard **LVF, LVT, RVF, RVT, HAT** and **HBT** models, and they are combined sequentially. See Table 1 below.

**Table 1: DV-E Speeds**

| | |
|---|---|
| 0.3 | 10 |
| 0.5 | 12 |
| 0.6 | 20 |
| 1.0 | 30 |
| 1.5 | 50 |
| 2.0 | 60 |
| 2.5 | 100 |
| 3.0 | |
| 4.0 | |
| 5.0 | |
| 6.0 | |

Setting the SPEED/SPINDLE switch in the left position will allow the operator to adjust the speed selection. The SELECT knob can be rotated until the desired speed is selected. Once the desired speed is shown on the display, set the SPINDLE/SPEED switch to the middle position.

The viscometer will rotate the spindle at the selected speed when the motor switch is in the **ON** position. A motor on condition is indicated on the display by **RPM** shown beside the speed. When the motor switch is in the **OFF** position, **OFF** will be displayed beside the speed.


(MOTOR ON)


(MOTOR OFF)

Figure II-4

**Note: When the motor switch is in the ON position, any change to the selected speed will be effective immediately. When collecting data at multiple speeds, you may wish to leave the SPEED/SPINDLE switch in the left position to facilitate speed changes. Also, when the motor switch is turned off, the display will hold the last measured torque value and measured viscosity.**

The DV-E Viscometer employs an optical signal pick-up inside the instrument to detect the torque value of the calibrated spring. This optical signal pick-up is recorded four times per revolution of the spindle. When the spindle begins to rotate at a defined speed, four torque values are recorded during the first full revolution of the spindle and averaged together. The display reports the average value for both torque (%) and viscosity (cP or mPa•s). Thereafter, the next torque value recorded by the optical signal pick-up is averaged together with the three preceding torque values and the newly calculated torque (%) and viscosity (cP or mPa•s) values are then displayed. This 4x revolution pick-up and display continues as long as the motor is on.

This algorithm in the instrument firmware is used for all viscosity and torque readings. Consequently, the wait time to observe the initial displayed readings for torque and viscosity increase as you go to lower speeds.

It may also be necessary to allow time for the indicated reading to stabilize.

**Note: At speeds of 1 RPM and lower, additional time may be required to allow for complete deflection of the torque sensor.**

The time required for stabilization will depend on the speed at which the Viscometer is running and the characteristics of the sample fluid. For maximum accuracy, readings below 10% should be avoided. Additional information on making viscosity measurements is available in Appendix B or the Brookfield publication *"More Solutions to Sticky Problems"* .

The **DV-E** Viscometer will remember the selected speed and spindle when power is turned off. On start-up, the Viscometer will be set to the previously selected spindle and speed.

Please see Brookfield publication *"More Solutions to Sticky Problems"* (Chapter 3) for information on how to select a speed.

## II.4 Autorange and CGS or SI Units Selection

The **AUTO RANGE** key allows you to determine the maximum calculated viscosity (full scale reading) possible with the **current spindle/speed setting**. Pressing the key *at any time* will cause

the current viscosity display to change and show *that* maximum viscosity. The screen torque display will now display "**%100**" to indicate this special condition. This maximum viscosity and **%100** value will be displayed for as long as the **AUTO RANGE** key is depressed. **Figure II-5** shows the AUTO RANGE function for the situation where the No. 1 RV spindle is rotating at 10 RPM. The full scale range is 1000 cP (or 1000 mPa·s).


Figure II-5

Pressing and holding the **AUTO RANGE** key during power on will enable the viscosity unit displayed to toggle between CGS (cP) and SI (mPa•s) units. To change the unit format:

1. Turn the power off.
2. Press and hold the **AUTO RANGE** key and turn the power **ON**.

The **DV-E** will retain the unit selection when the viscometer is turned **OFF**.

| | CGS | SI |
|---|---|---|
| Viscosity: | cP | mPa·s |

*1 cP = 1 mPa•s*

## II.5 Out of Range

The **DV-E** gives indications for out of specification or out-of-range operation. When **% (Torque)** readings *exceed* 100.0 % (over-range), the display changes to that shown in **Figure II-6**:


Figure II-6

You must change either speed or spindle to correct this condition. If you operate at spindle speeds that produce **% (Torque)** below 10.0 % (under-range), the **DV-E** displays both **% (Torque)** and **cP (Viscosity)** with flashing unit designations. The parameters of % (Torque) and cP (Viscosity) will also flash prior to one complete spindle revolution. It is not recommended that readings are taken while parameters are flashing.


Figure II-7

Negative **% (Torque)** will be displayed as shown in **Figure 8**:


Figure II-8

Viscosity values will be displayed as "- - - -" when the **% (Torque)** is below zero.

## II.6 Operation

The following procedure is outlined for making a viscosity measurement in a 600 mL low form Griffin beaker.

1. Mount the guardleg on the **DV-E** Viscometer (LV and RV series). Be sure that the motor is OFF before attaching the spindle. Select a spindle and attach it to the lower shaft. Lift the shaft slightly, holding it firmly with one hand while screwing the spindle on with the other (note **left-hand thread**). Avoid putting side thrust on the shaft.

2. Insert and center spindle in the test material until the fluid's level is at the immersion groove on the spindle's shaft. With a disc-type spindle, it is sometimes necessary to tilt the spindle slightly while immersing to avoid trapping air bubbles on its surface. (You may find it more convenient to immerse the spindle in this fashion before attaching it to the Viscometer.)

3. To make a viscosity measurement, select a speed and follow the instructions in Sections **II.2** and **II.3**. Allow time for the indicated reading to stabilize. The time required for stabilization will depend on the speed at which the Viscometer is running and the characteristics of the sample fluid. For maximum accuracy, readings below 10% should be avoided. Additional information on making viscosity measurements is available in Appendix B or the Brookfield publication *"More Solutions to Sticky Problems"*.

4. Switch the **MOTOR ON/OFF** switch to turn the motor **"OFF"** when changing a spindle or changing samples. Remove spindle before cleaning.

5. Interpretation of results and the instrument's use with non-Newtonian and thixotropic materials is discussed in the booklet, *"More Solutions to Sticky Problems"*, and in **Appendix B, Variables in Viscosity Measurements**.

## Appendix A - Viscosity Ranges

### LV and RV,HA,HB Viscometers

| Viscosity Range (cP) | | |
|---|---|---|
| **Viscometer** | **Minimum** | **Maximum** |
| LVDV-E | 15 | 2 M |
| RVDV-E | 100 | 13 M |
| HADV-E | 200 | 26 M |
| HBDV-E | 800 | 106 M |

### Small Sample Adapter and Thermosel

| SSA/Thermosel Spindle | Shear Rate (1/SEC) | Viscosity (cP) |
|---|---|---|
| | | LVDV-E |
| SC4-16 (SSA) | 0.29 N | 120 - 400 K |
| SC4-18 (SSA/Tsel) | 1.32 N | 3 - 10 K |
| SC4-25 (SSA) | 0.22 N | 800 - 1.60 M |
| SC4-31 (SSA/Tsel) | 0.34 N | 30 - 100 K |
| SC4-34 (SSA/Tsel) | 0.28 N | 60 - 200 K |
| HT-81 (Tsel) | 1.29 N | 3.5 - 10 K |
| SC4-82 (SSA) | 1.29 N | 3.5 - 10 K |
| SC4-83 (SSA) | 1.29 N | 11.0 - 38 K |

| SSA/ Thermosel Spindle | Shear Rate (1/SEC) | Viscosity (cP) | | |
|---|---|---|---|---|
| | | RVDV-E | HADV-E | HBDV-E |
| SC4-14 (SSA) | 0.40N | 1.25K - 4.2 M | 2.5 M - 8.3 M | 10 M - 33.3 M |
| SC4-15 (SSA) | 0.48N | 500 - 1.7 M | 1 M - 3.3 M | 4 M - 13.3 M |
| SC4-21 (SSA/Tsel) | 0.93N | 50 - 170 K | 100 - 300 K | 400 - 1.3 M |
| SC4-27 (SSA/Tsel) | 0.34N | 250 - 830 K | 500 - 1.7 M | 2 M - 6.7 M |
| SC4-28 (SSA/Tsel) | 0.28N | 500 - 1.7 M | 1 M - 3.3 M | 4 M - 13.3 M |
| SC4-29 (SSA/Tsel) | 0.25N | 1 K - 3.3 M | 2 M - 6.7 M | 8 M - 26.7 M |
| HT-81 (Tsel) | 1.29N | 36 - 10 K | 73 - 10 K | 292 - 10 K |
| SC4-82 (SSA) | 1.29N | 36 - 10 K | 73 - 10 K | 292 - 10 K |
| SC4-83 (SSA) | 1.29N | 121 - 50 K | 242 - 50 K | 970 - 50 K |

### UL Adapter

| UL Spindle | Shear Rate (1/SEC) | Viscosity (cP) | | | |
|---|---|---|---|---|---|
| | | LVDV-E | RVDV-E | HADV-E | HBDV-E |
| YULA-15 or 15Z | 1.224N | 1.0 - 2 K | 6.4 - 2 K | 12.8 - 2 K | 51.2 - 2 K |

*K = 1,000* *M = 1,000,000* *N = RPM*

## DIN Adapter Accessory

| DAA Spindle | Shear Rate (1/SEC) | Viscosity (cP) | | | |
|---|---|---|---|---|---|
| | | LVDV-E | RVDV-E | HADV-E | HBDV-E |
| 85 | 1.29N | 1.2 - 3.8 K | 12 - 5 K | 24 - 5 K | 98 - 5 K |
| 86 | 1.29N | 3 - 10 K | 36 - 10 K | 73 - 10 K | 292 - 10 K |
| 87 | 1.29N | 11 - 38 K | 12 - 50 K | 242 - 50 K | 970 - 50 K |

## Spiral Adapter

| DAA Spindle | Shear Rate (1/SEC) | Viscosity (cP) | | | |
|---|---|---|---|---|---|
| | | LVDV-E | RVDV-E | HADV-E | HBDV-E |
| SA-70 | 0.68 - 68 | 100 - 98 K | 1 M - 1 M | 2 M - 2 M | 8 M - 8.4 M |
| | *(1-100 RPM)* | | | | |

## Helipath with T-Bar Spindles

| T-Bar Spindle | Viscosity (cP) | | | |
|---|---|---|---|---|
| | LVDV-E | RVDV-E | HADV-E | HBDV-E |
| T-A | 156 - 62 K | 2 M - 400 K | 4 M - 800 K | 16 M - 3.2 M |
| T-B | 312 - 124 K | 4 M - 800 K | 8 M - 1.6 M | 32 M - 6.4 M |
| T-C | 780 - 312 K | 10 M - 2 M | 20 M - 4 M | 80 M - 16 M |
| T-D | 1.5 M - 624 K | 20 M - 4 M | 40 M - 8 M | 160 M - 32 M |
| T-E | 3.9 M - 1.5 M | 50 M - 10 M | 100 M - 20 M | 400 M - 80 M |
| T-F | 7.8 M - 3.1 M | 100 M - 20 M | 200 M - 40 M | 800 M - 160 M |

***K = 1,000*** ***M = 1,000,000*** ***N = RPM***

In taking viscosity measurements with the **DV-E** Viscometer, there are two considerations which pertain to the low viscosity limit of effective measurement.

1) Viscosity measurements should be accepted within the equivalent % Torque Range from 10% to 100% for any combination of spindle/speed rotation.

2) Viscosity measurements should be taken under laminar flow conditions, not under turbulent flow conditions.

The first consideration has to do with the accuracy of the instrument. All **DV-E** Viscometers have a full scale range accuracy of (+/-) 1% of any spindle/speed rotation. We discourage taking readings below 10% of range because the potential viscosity error of (+/-) 1% is a relatively high number compared to the instrument reading.

The second consideration involves the mechanics of fluid flow. All rheological measurements of fluid flow properties should be made under laminar flow conditions. Laminar flow is flow wherein all particle movement is in layers directed by the shearing force. For rotational systems, this means all fluid movement must be circumferential. When the inertial forces on the fluid become too great, the fluid can break into turbulent flow wherein the movement of fluid particles becomes random and the flow can not be analyzed with standard math models. This turbulence creates a falsely high viscometer reading with the degree of non-linear increase in reading being directly related to the degree of turbulence in the fluid.

For the following geometries, we have found that an approximate transition point to turbulent flow occurs:

1) No. 1 LV (optional) Spindle: 15 **cP** at 60 RPM
2) No. 1 RV (optional) Spindle: 100 **cP** at 20 RPM
3) UL Adapter: 0.85 **cP** at 60 RPM

Turbulent conditions will exist in these situations whenever the RPM/cP ratio exceeds the values listed above.

## Appendix B - Variables in Viscosity Measurement

As with any instrument measurement, there are variables that can affect a viscometer measurement. These variables may be related to the instrument (viscometer), or the test fluid. Variables related to the test fluid deal with the rheological properties of the fluid, while instrument variables would include the viscometer design and the spindle geometry system utilized.

### Rheological Properties

Fluids have different rheological characteristics that can be described by viscometer measurements. We can then work with these fluids to suit the lab or process conditions.

There are two categories of fluids:

**Newtonian** - These fluids have the same viscosity at different Shear Rates (different RPM's) and are called Newtonian over the Shear Rate range they are measured.

**Non-Newtonian** - These fluids have different viscosities at different shear rates (different RPM's). They fall into two groups:

1) Time Independent
2) Time Dependent

**Time Independent** means that the viscosity behavior does not change as a function of time when measuring at a specific shear rate.

**Pseudoplastic** - A pseudoplastic material displays a decrease in viscosity with an increase in shear rate, and is also known as "shear thinning". If you take viscometer readings from a low to a high RPM and then back to the low RPM, and the readings fall upon themselves, the material is time independent ,pseudoplastic and shear thinning.

**Time Dependent** means that the viscosity behavior changes as a function of time when measuring at a specific shear rate.

**Thixotropic** - A thixotropic material has decreasing viscosity under constant shear rate. If you set a viscometer at a constant speed recording viscosity values over time and find that the viscosity values decrease with time, the material is thixotropic.

Brookfield publication, "More Solutions to Sticky Problems", includes a more detailed discussion of rheological properties and non-Newtonian behavior.

### Viscometer Related Variables

Most fluid viscosities are found to be non-Newtonian. They are dependent on Shear Rate and the spindle geometry conditions. The specifications of the viscometer spindle and chamber geometry will affect the viscosity readings. If one reading is taken at 2.5 rpm, and a second at 50 rpm, the two viscosity values produced will be different because the readings were made at different shear rates. The faster the spindle speed, the higher the shear rate.

The shear rate of a given measurement is determined by: the rotational speed of the spindle, the size and shape of the spindle, the size and shape of the container used and therefore, the distance between the container wall and the spindle surface.

A repeatable viscosity test should control or specify the following:

1) Test temperature
2) Sample container size (or spindle/chamber geometry)
3) Sample volume
4) Viscometer model
5) Spindle used (if using LV**DV-E** (#1-4) or RV**DV-E** (#2-7) attach the guard leg)
6) Test speed or speeds (or the shear rate)
7) Length of time or number of spindle revolutions to record viscosity.

## Appendix C - Spindle and Model Codes

Each spindle has a two digit code which is scrolled via the select knob on the **DV-E**. The spindle code directs the **DV-E** to calculate viscosity for the spindle that is being used. The spindle multiplyer constant (**SMC**) is used to calculate full scale viscosity range for any spindle/speed combination (refer to **Appendix D**). Spindle codes are listed in **Table C-1**.

Table C-1

| SPINDLE | CODE | SMC |
|---|---|---|
| RV1 (optional) | 01 | 1 |
| RV2 | 02 | 4 |
| RV3 | 03 | 10 |
| RV4 | 04 | 20 |
| RV5 | 05 | 40 |
| RV6 | 06 | 100 |
| RV7 | 07 | 400 |
| HA1 (optional) | 01 | 1 |
| HA2 | 02 | 4 |
| HA3 | 03 | 10 |
| HA4 | 04 | 20 |
| HA5 | 05 | 40 |
| HA6 | 06 | 100 |
| HA7 | 07 | 400 |
| HB1 (optional) | 01 | 1 |
| HB2 | 02 | 4 |
| HB3 | 03 | 10 |
| HB4 | 04 | 20 |
| HB5 | 05 | 40 |
| HB6 | 06 | 100 |
| HB7 | 07 | 400 |
| LV1 | 61 | 6.4 |
| LV2 | 62 | 32 |
| LV3 | 63 | 128 |
| LV4 | 64 | 640 |
| LV5 | 65 | 1280 |
| SPIRAL | 70 | 105 |

| SPINDLE | CODE | SMC |
|---|---|---|
| T-A | 91 | 20 |
| T-B | 92 | 40 |
| T-C | 93 | 100 |
| T-D | 94 | 200 |
| T-E | 95 | 500 |
| T-F | 96 | 1000 |
| ULA | 00 | 0.64 |
| DIN-ULA | 85 | 1.22 |
| TSEL-DIN-81 | 81 | 3.7 |
| SSA-DIN-82 | 82 | 3.75 |
| SSA-DIN-83 | 83 | 12.09 |
| ULA-DIN-85 | 85 | 1.22 |
| ULA-DIN-86 | 86 | 3.65 |
| ULA-DIN-87 | 87 | 12.13 |
| SC4-14 | 14 | 125 |
| SC4-15 | 15 | 50 |
| SC4-16 | 16 | 128 |
| SC4-18 | 18 | 3.2 |
| SC4-21 | 21 | 5 |
| SC4-25 | 25 | 512 |
| SC4-27 | 27 | 25 |
| SC4-28 | 28 | 50 |
| SC4-29 | 29 | 100 |
| SC4-31 | 31 | 32 |
| SC4-34 | 34 | 64 |
| SC4-37 | 37 | 25 |

**Table C-2** lists the model codes and spring torque constants for each viscometer model.

Table C-2

| VISCOMETER MODEL | TORQUE CONSTANT TK | MODEL CODE ON DV-E SCREEN |
|---|---|---|
| LVDV-E | 0.09373 | LV |
| RVDV-E | 1 | RV |
| HADV-E | 2 | HA |
| HBDV-E | 8 | HB |

| SPECIAL ORDER TORQUE SPRINGS | | |
|---|---|---|
| VISCOMETER MODEL | TORQUE CONSTANT TK | MODEL CODE ON DV-E SCREEN |
| 2.5xLVDV-E | 0.2343 | 2.5LV |
| 5xLVDV-E | 0.4686 | 5LV |
| 1/4 RVDV-E | 0.25 | 1/4RV |
| 1/2 RVDV-E | 0.5 | 1/2RV |
| 2xHADV-E | 4 | 2HA |
| 2.5xHADV-E | 5 | 2.5HA |
| 2xHBDV-E | 16 | 2HB |
| 2.5xHBDV-E | 20 | 2.5HB |
| 5xHBDV-E | 40 | 5HB |

## Appendix D - Calibration Procedures

The accuracy of the **DV-E** is verified using viscosity standard fluids which are available from Brookfield Engineering Laboratories or your local Brookfield agent. Viscosity standards are Newtonian, and therefore, have the same viscosity regardless of spindle speed (or shear rate). Viscosity standards, calibrated at 25°C, are shown in **Table D-1**.

**Container size:** For Viscosity Standards < 30,000 cP, use a 600 ml Low Form Griffin Beaker having a working volume of 500 ml.

For Viscosity Standards ≥ 30,000 cP, use the fluid container.

Inside Diameter: 3.25"(8.25cm)
Height: 4.75"(12.1cm)

Note: Container may be larger, but may not be smaller.

**Temperature:** As stated on the fluid standard label: (+/-) 0.1°C

**Conditions:** The **DV-E** should be set according to the operating instructions. The water bath should be stabilized at test temperature. Viscometers with the letters "**LV**" or "**RV**" in the model designation should have the guard leg attached.

| Normal 25°C Standard Fluids Viscosity (cP) | Viscosity (cP) | High Temperature Standard Fluids for use with Thermosel Accessory |
|---|---|---|
| 5 | 5,000 | HT-30,000 |
| 10 | 12,500 | HT-60,000 |
| 50 | 30,000 | HT-1000,000 |
| 100 | 60,000 | Calibrated at three viscosity/temperatures 25°C, 93.3°C, 149°C |
| 500 | 100,000 | *Refer to Brookfield catalog for more information.* |
| 1,000 | | |

Table D-1(Silicone Oils)

### Brookfield Viscosity Standard Fluid - General Information

We recommend that Brookfield Viscosity Standard Fluids be replaced on an annual basis, one year from date of initial use. These fluids are pure silicone and are not subject to change over time. However, exposure to outside contaminants through normal use requires replacement on an annual basis. Contamination may occur by the introduction of solvent, standard of different viscosity or other foreign material.

Viscosity Standard Fluids may be stored under normal laboratory conditions. Disposal should be in accordance with state, local and federal regulations as specified on the material safety data sheet.

Brookfield Engineering Laboratories does not recertify Viscosity Standard Fluids. We will issue duplicate copies of the Certificate of Calibration for any fluid within two years of the purchase date.

Brookfield Viscosity Standard Fluids are reusable provided they are not contaminated. Normal

practice for usage in a 600 ml beaker is to return the material from the beaker back into the bottle. When using smaller volumes in accessories such as Small Sample Adapter, UL Adapter, Thermosel or Spiral Adapter, the fluid is normally discarded.

**Calibration Procedure for LV(#1-4) and RV,HA,HB(#2-7) Brookfield spindles:**

1) Place the viscosity standard fluid (in the proper container) into the water bath.

2) Lower the **DV-E** into measurement position (with guard leg if **LV** or **RV** series viscometer is used).

3) Attach the spindle to the viscometer. If you are using a disk shaped spindle, avoid trapping air bubbles beneath the disk by first immersing the spindle at an angle, and then connecting it to the viscometer.

4) The viscosity standard fluid, together with the spindle and guard leg (if supplied), should be immersed in the bath for a **minimum** of 1 hour, stirring the fluid periodically, prior to taking measurements.

5) After 1 hour, check the temperature of the viscosity standard fluid with an accurate thermometer. Fluid must be within $\pm 0.1^{\circ}C$ of the specified temperature, normally $25^{\circ}C$. Allow longer soak time if required to come to test temperature.

6) If the fluid is at test temperature, measure the viscosity and record the viscometer reading.

**Note:** The spindle must rotate at least five (5) times before readings are taken.

7) The viscosity reading should equal the **cP** value on the viscosity fluid standard to within the combined accuracies of the viscometer and the standard (as discussed in the section entitled, **Interpretation of Calibration Test Results**).

**Calibration Procedure for a Small Sample Adapter**

When a Small Sample Adapter is used, the water jacket is connected to the water bath and the water is stabilized at the proper temperature:

1) Put the proper amount of viscosity standard fluid into the sample chamber. The amount varies with each spindle/chamber combination. (Refer to the Small Sample Adapter instruction manual.)

2) Place the sample chamber into the water jacket.

3) Put the spindle into the test fluid and attach the extension link, coupling nut and free hanging spindle (or directly attach the solid shaft spindle) to the **DV-E**.

4) Allow 30 minutes for the viscosity standard, sample chamber and spindle to reach test temperature.

5) Measure the viscosity and record the viscometer reading.

**Note:** The spindle must rotate at least five (5) times before a viscosity reading is taken.

**Calibration Procedure for a Thermosel System**

A two-step process is recommended for the Thermosel.

A) Evaluate the calibration of the Viscometer alone according to the procedure outlined in **this** section, entitled **Calibration Procedure for LV (#1-4) and RV,HA,HB (#2-7) Brookfield spindles.**

B) Evaluate the Viscometer with the Thermosel according to the procedure decribed below.

When a Thermosel is used, the controller stabilizes the Thermo Container at the test temperature.

1) Install the tube end cap and put the proper amount of HT viscosity standard fluid into the HT-2 or HT-2DB sample chamber. The amount varies with the spindle used. (Refer to the Thermosel instruction manual).

2) Place the sample chamber into the Thermo Container.

3) Put the spindle into the test fluid and attach the extension link, coupling nut and free hanging spindle (or directly attach the solid shaft spindle) to the **DV-E**.

4) Allow 30 minutes for the viscosity standard, sample chamber and spindle to reach test temperature.

5) Measure the viscosity and record the viscometer reading.

**Note:** The spindle must rotate at least five (5) times before a viscosity reading is taken.

**Calibration Procedure for UL Adapter**

When a UL Adapter is used, the water bath should be stabilized at the proper temperature:

1) Install the tube end cap and put the proper amount of viscosity standard fluid into the UL Tube. (Refer to the UL Adapter instruction manual).

2) Attach the spindle (with extension link and coupling nut) onto the **DV-E**.

3) Attach the tube to the mounting channel.

4) Lower the tube into the water bath reservoir, or if using the ULA-40Y water jacket, connect the inlet/outlets to the bath external circulating pump.

5) Allow 30 minutes for the viscosity standard, sample chamber and spindle to reach test temperature.

6) Measure the viscosity and record the viscometer reading.

**Note:** The spindle must rotate at least five (5) times before a viscosity reading is taken.

**Calibration Procedure for DIN Adapter Accessory**

When a DIN Adapter is used, the water bath should be stabilized at the proper temperature:

1) Put the proper amount of viscosity standard fluid into the UL Tube. (Refer to the DAA instruction manual).

2) Attach the spindle (with extension link and coupling nut) onto the **DV-E**.

3) Attach the tube to the mounting channel.

4) Lower the tube into the water bath reservoir, or if using the ULA-40Y water jacket, connect the inlet/outlets to the bath external circulating pump.

5) Allow 30 minutes for the viscosity standard, sample chamber and spindle to reach test temperature.

6) Measure the viscosity and record the viscometer reading.

> **Note:** The spindle must rotate at least five (5) times before a viscosity reading is taken.

**Calibration Procedure for Spiral Adapter**

1) Place the viscosity standard fluid (in the proper container) into the water bath.

2) Attach the spindle to the viscometer. Attach chamber (SA-1Y) and clamp to the viscometer.

3) Lower the **DV-E** into measurement position. Operate the viscometer at 50 or 60 RPM until the chamber is fully flooded.

4) The viscosity standard fluid, together with the spindle, should be immersed in the bath for a minimum of 1 hour, stirring the fluid periodically (operate at 50 or 60 RPM periodically), prior to taking measurements.

5) After 1 hour, check the temperature of the viscosity standard fluid with an accurate thermometer.

6) If the fluid is at test temperature (+/- 0.1°**C** of the specified temperature, normally 25°**C**), measure the viscosity and record the viscometer reading.

> **Note:** The spindle must rotate at least five (5) times for one minute, whichever is greater before readings are taken.

7) The viscosity reading should equal the **cP** value on the viscosity fluid standard to within the combined accuracies of the viscometer and the standard (as discussed in the section entitled, **Interpretation of Calibration Test Results**).

**Interpretation of Calibration Test Results:**

When verifying the calibration of the **DV-E**, the instrument and viscosity standard fluid error must be combined to calculate the total allowable error.

The **DV-E** is accurate to (+/-) 1% of any full scale spindle/speed viscosity range.

Brookfield Viscosity Standards Fluids are accurate to (+/-) 1% of their stated value.

**EXAMPLE:** Calculate the acceptable range of viscosity using RV**DV-E** with RV-3 Spindle at 2 RPM; Brookfield Standard Fluid 12,500 with a viscosity of 12,257 **cP** at 25°C:

1) Calculate full scale viscosity range using the equation:

$$\text{Full Scale Viscosity Range } [cP] = TK * SMC * \frac{10{,}000}{RPM}$$

Where: **TK** = 1.0 from **Table C2**
**SMC** = 10 from **Table C1**

$$\text{Full Scale Viscosity Range} = \frac{1 * 10 * 10{,}000}{2} = \mathbf{50{,}000\ cP}$$

The viscosity is accurate to (+/-) 500 **cP** (which is 1% of 50,000)

2) The viscosity standard fluid is 12,257 **cP**. Its accuracy is (+/-)1% of 12,257 or (+/-)122.57 **cP**.

3) Total allowable error is (122.57 + 500) **cP** = (+/-) 622.57 **cP**.

4) Therefore, any viscosity reading between 11,634.4 and 12,879.6 **cP** indicates that the viscometer is operating correctly. Any reading outside these limits may indicate a viscometer problem. Contact the Brookfield technical sales department or your local Brookfield dealer/distributor with test results to determine the nature of the problem.

**The Brookfield Guardleg**

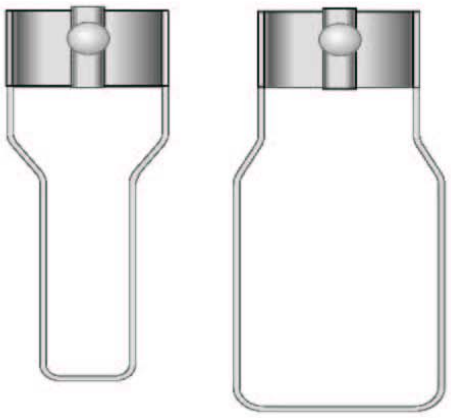
The *guard leg* was originally designed to protect the spindle during use. The first applications of the Brookfield Viscometer included hand held operation while measuring fluids in a 55 gallon drum. It is clear that under those conditions the potential for damage to the spindle was great.

The current guard leg is a band of metal in the shape of the letter U with a bracket at the top that attaches to the pivot cup of a Brookfield Viscometer/Rheometer. A guard leg is supplied with all LV and RV series instruments, but not with the HA or HB series. It's shape is designed to accommodate the spindles of the appropriate spindle set; therefore, the RV guard leg is wider than the LV due to the large diameter of the RV #1 (optional) spindle. They are not interchangeable.

The calibration of the Brookfield Viscometer/Rheometer is determined using a 600 ml Low Form Griffin Beaker. The calibration of LV and RV series instruments includes the guard leg. The beaker wall (for HA/HB instruments) or the guard leg (for LV/RV instruments) define what is called the "outer boundary" of the measurement. The spindle factors for the LV, RV, and HA/HB spindles were developed with the above boundary conditions. The spindle *factors* are used to convert the instrument torque (expressed as the dial reading or %Torque value) into centipoise. Theoretically, if measurements are made with different boundary conditions, e.g., without the guard leg or in a container other than 600 ml beaker, then the spindle factors found on the Factor Finder cannot be used to accurately calculate an absolute viscosity. Changing the boundary conditions does not change the viscosity of the fluid, but it does change how the instrument torque is converted to centipoise. Without changing the spindle factor to suit the new boundary conditions, the calculation from instrument torque to viscosity will be incorrect.

Practically speaking, the guard leg has the greatest effect when used with the #1 & #2 spindles of the LV and RV spindle sets. Any other LV (#3 & #4) or RV (#3 - #7) spindle can be used in a 600 ml beaker with or without the guard leg to produce correct results. The HA and HB series Viscometers/ Rheometers are not supplied with guard legs in order to reduce the potential problems when measuring high viscosity materials. HA/HB spindles #3 through #7 are identical to those spindle numbers in the RV spindle set. The HA/HB #1 & #2 have slightly different dimensions than the corresponding RV spindles. This dimensional difference allows the factors between the RV and HA/ HB #1&#2 spindles to follow the same ratios as the instrument torque even though the boundary conditions are different.

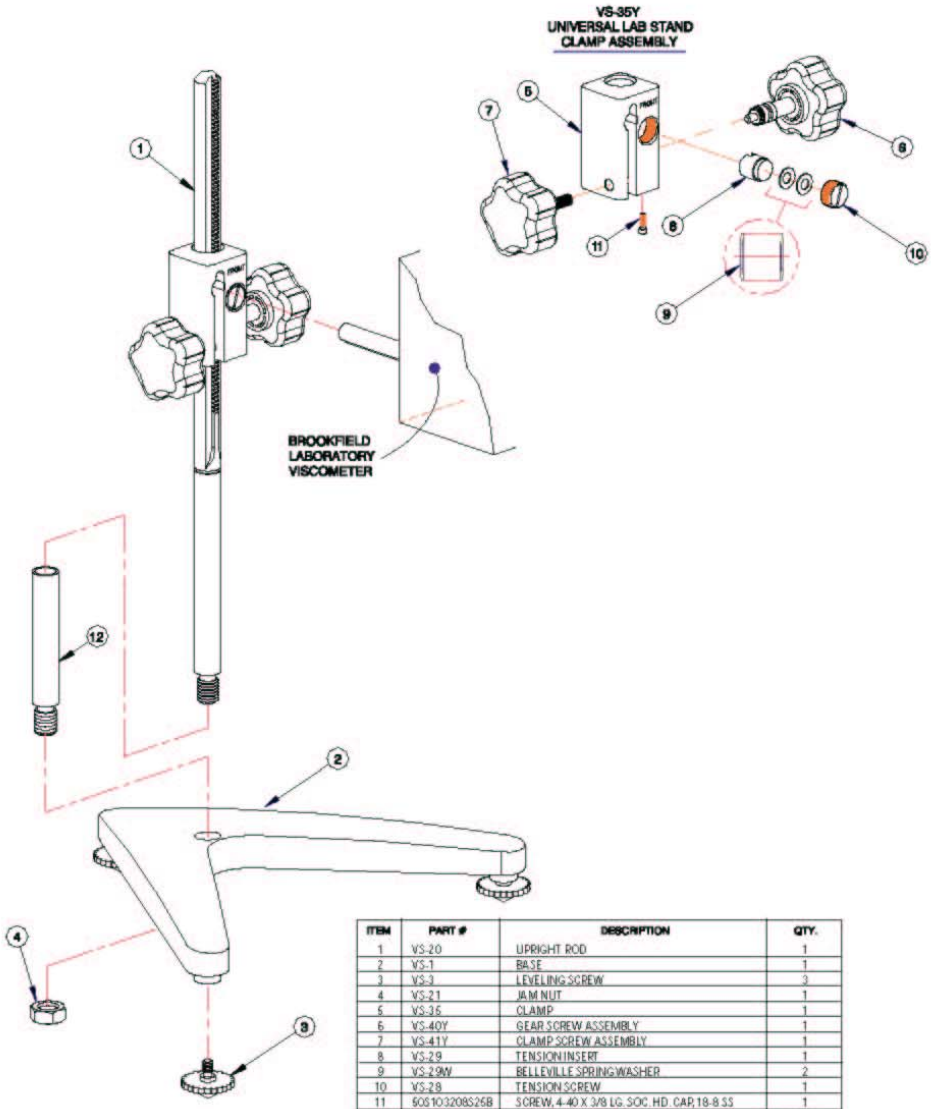
The recommended procedures of using a 600 ml beaker and the guard leg are difficult for some customers to follow. The guard leg is one more item to clean. In some applications the 500 ml of test fluid required to immerse the spindles in a 600 ml beaker is not available. In practice, a smaller vessel may be used and the guard leg is removed. The Brookfield Viscometer/Rheometer will produce an accurate and repeatable torque reading under any measurement circumstance. However, the conversion of this torque reading to centipoise will only be correct if the factor used was developed for those specific conditions. Brookfield has outlined a method for recalibrating a Brookfield Viscometer/Rheometer to any measurement circumstance in "More Solutions to Sticky Problems", Section 3.3.10. It is important to note that for many viscometer users the true viscosity is not as important as a repeatable day to day value. This repeatable value can be obtained without any special effort for any measurement circumstance. But, it should be known that this type of torque reading will not convert into a correct centipoise value when using a Brookfield factor if the boundary conditions are not those specified by Brookfield.



LV Guardleg RV Guardleg

The guard leg is a part of the calibration check of the Brookfield LV and RV series Viscometer/ Rheometer. Our customers should be aware of its existence, its purpose and the effect that it may have on data. With this knowledge, the viscometer user may make modifications to the recommended method of operation to suit their needs.

## Appendix E - Model A Laboratory Stand with Parts Identification




| ITEM | PART # | DESCRIPTION | QTY. |
|---|---|---|---|
| 1 | VS-20 | UPRIGHT ROD | 1 |
| 2 | VS-1 | BASE | 1 |
| 3 | VS-3 | LEVELING SCREW | 3 |
| 4 | VS-21 | JAM NUT | 1 |
| 5 | VS-35 | CLAMP | 1 |
| 6 | VS-40Y | GEAR SCREW ASSEMBLY | 1 |
| 7 | VS-41Y | CLAMP SCREW ASSEMBLY | 1 |
| 8 | VS-29 | TENSION INSERT | 1 |
| 9 | VS-29W | BELLEVILLE SPRING WASHER | 2 |
| 10 | VS-28 | TENSION SCREW | 1 |
| 11 | 6QS10320BS25B | SCREW, 4-40 X 3/8 LG. SOC. HD. CAP, 18-8 SS | 1 |
| 12 | BLM-4E | ROD EXTENSION-4" LONG | OPTIONAL |
| | BLM-4E2 | RODE EXTENSION - 8" LONG | OPTIONAL |

Figure E-1

## Unpacking

Check carefully to see that all the components are received with no concealed damage.

| | |
|---|---|
| 1 base | 1 jam nut |
| 3 leveling screws | 1 clamp assembly |
| 1 upright rod | |

Remove the three (3) leveling screws from the base and discard the packing material. Remove the jam nut from the upright rod.

## Assembly (Refer to Figure E1)

Screw the leveling screws into the base. Insert the threaded end of the upright rod into the hole in the top of the base and attach the jam nut to the rod on the underside of the base. With the rod gear rack facing forward (toward the "V" in the base), gently tighten the jam nut.

## Viscometer Mounting

The VS-35Y clamp assembly should be positioned so that the word 'front' is facing the operator. This will ensure the cut-away slot of the clamp assembly will align properly with the machined key ridge of the viscometer handle. Insert the viscometer rod into the cut-away hole of the clamp assembly. Adjust the instrument level until the bubble is centered within the target and tighten the clamp screw, VS-26Y.

**The small clamp adjusting screw (Figure E1) on the front of the clamp assembly should be loosened or tightened as necessary to provide smooth height adjustment and adequate support for the Viscometer.**

Center the Viscometer relative to the stand base and retighten the jam nut as required. Referring to the Viscometer bubble level, adjust the leveling screws until the instrument is level.

## Operation

Rotate the Gear Screw to raise or lower the viscometer.

## Appendix F - Fault Diagnosis and Troubleshooting

Listed are some of the more common problems that you may encounter while using your DV-E Viscometer. Review these items *before* you contact Brookfield.

### Spindle Does Not Rotate

- ❑ Make sure the viscometer is plugged in.
- ❑ Check the voltage rating on your viscometer (115V, 220V): it must match the wall voltage.
- ❑ Make sure the power switch is in the ON position.
- ❑ Make sure the speed set knob is set properly and securely at the desired speed.

### Spindle Wobbles When Rotating or Looks Bent

- ❑ Make sure the spindle is tightened securely to the viscometer coupling.
- ❑ Check the straightness of all other spindles; replace them if bent.
- ❑ Inspect viscometer coupling and spindle coupling mating areas and threads for dirt: clean threads on spindle coupling with a 3/56 left-hand tap.
- ❑ Inspect threads for wear; if the threads are worn, the unit needs service (see **Appendix G**).
- ❑ Check to see if spindles rotate eccentrically or wobble. There is an allowable runout for 1/32-inch in each direction (1/16-inch total) when measured from the bottom of the spindle rotating in air.
- ❑ Check to see if the viscometer coupling is bent; if so, the unit is in need of service.

If the pointer sticks and/or does not rest at zero, the unit is need of service. See **Appendix G** for details on how to return your viscometer.

### Inaccurate Readings

- ❑ Verify spindle, speed and model selection
- ❑ Verify test parameters: temperature, container, volume, method. Refer to:
  - "More Solutions to Sticky Problems"; Section II.2a — Considerations for Making Measurements
  - Dial Viscometer Operating Manual; Appendix B — Viscosity Ranges
  - Dial Viscometer Operating Manual; Appendix C — Variables in Viscosity Measurement

❑ Perform a calibration check. Follow the instructions in **Appendix D**.

- Verify tolerances are calculated correctly.
- Verify calibration check procedures were followed exactly

If the unit is found to be out of tolerance, the unit may be in need of service. See **Appendix H** for details on how to return your viscometer.

# Appendix G - Warranty Repair and Service

## Warranty

Brookfield Viscometers are guaranteed for one year from date of purchase against defects in materials and workmanship. The Viscometer must be returned to **Brookfield Engineering Laboratories, Inc.** or the Brookfield dealer from whom it was purchased for no charge warranty evaluation service. Transportation is at the purchaser's expense. The Viscometer should be shipped in its carrying case together with all spindles originally provided with the instrument as shown below.

- ❑ Remove and return all spindles (properly packed for shipping).
- ❑ Clean excess testing material off the instrument.
- ❑ Include MSDS sheets for all materials tested with this instrument.
- ❑ Protect the pointer shaft with a shipping cap as shown in Figure G-1.
- ❑ Pack the instrument in its original case. Cases are available for immediate shipment from Brookfield. If the case is not available, take care to wrap the instrument with enough material to support it. Avoid using foam peanuts or shredded paper.
- ❑ DO NOT send the laboratory stand unless there is a problem with the upright rod, clamp or base. If there is a problem with the stand, remove the upright rod from the base and individually wrap each item to avoid contact with the instrument. Do not put lab stand in viscometer carrying case.
- ❑ Fill out the Viscometer Information Sheet (included with the information packet you received on purchase) with as much information as possible to help expedite your service. If you do not have this form, please include a memo indicating the type of problem you are experiencing or the service you need performed. Please also include a purchase order number for us to bill against.
- ❑ Mark the outside of the shipping box with handling instructions, for example: "Handle with Care" or "Fragile - Delicate Instrument".

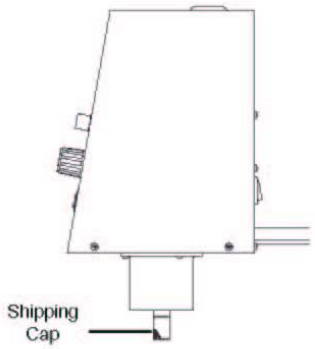



Figure G-1

For repair or service in the **United States** return to:

Brookfield Engineering Laboratories, Inc.
11 Commerce Boulevard
Middleboro, MA 02346 U.S.A.

Telephone: (508) 946-6200 FAX: (508) 946-6262
email: service@brookfieldengineering.com
www.brookfieldengineering.com

For repair or service outside the United States consult Brookfield Engineering Laboratories, Inc. or the dealer from whom you purchased the instrument.

For repair or service in the **United Kingdom** return to:

Brookfield Viscometers Limited
1 Whitehall Estate
Flex Meadow
Pinnacles West
Harlow, Essex CM19 5TJ, United Kingdom

Telephone: (44) 27/945 1774 FAX: (44) 27/945 1775
email: service@brookfield.co.uk
www.brookfield.co.uk

For repair or service in **Germany** return to:

Brookfield Engineering Laboratories Vertriebs GmbH
Hauptstrasse 18
D-73547 Lorch, Germany

Telephone: (49) 7172/927100 FAX: (49) 7172/927105
email: info@brookfield-gmbh.de
www.brookfield-gmbh.de

***On-site service at your facility is also available from Brookfield. Please contact our Service Department for details.***