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Foam Inhibitor Effects on Optically-Detected Fluid Cleanliness

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Foam Inhibitor Effects on Optically-Detected Fluid Cleanliness

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Abstract

Foam inhibitors (FI)s are included in lubricant formulations to reduce foam tendency and stability. However, light-based automatic particle counters record some FI droplets as contaminants. In order to meet stringent cleanliness goals, the effects of the FI dispersion on ISO 4406 particle counts must be minimized. It has been shown previously that particle counts resulting from FIs can be reduced by filtration, but it is unclear if smaller FI droplets are also removed and how this may affect lubricant foaming. To perform this research, three test stations were constructed. We assembled a lubricant blending station that provides control over blending parameters such as, temperature, impeller speed, and container size. We obtained and repaired a nonoperational Koehler Dual Bath Foaming Characteristics Apparatus and developed replacement/alternative components for the apparatus that allowed us to conduct ASTM D892 foaming characterizations. Finally, we designed and constructed a filter station that circulates, filters, and particle counts fluids. The station includes an inline particle counter, adjustable flow rate, and accepts fluid viscosities ranging from 15 to 250 cSt. A base oil and foam inhibitor are circulated through the apparatus; fluid samples are taken as filtration progresses and then tested for foaming in accordance with ASTM D892. For each case, we characterize the effect of the inhibitor on fluid cleanliness and the effect of filter passes on particle counts and foam tendency/stability. These effects are investigated across two filter sizes and four additive chemistries. The results provide information about the relationships between additive chemistry, filter media, optically-detected oil cleanliness, and FI effectiveness.

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List of Symbols

f	Force of Friction
μ	Coefficient of Friction
F_N	Normal Force
w	Wear
$f_{cn}()$	Function of ()
v	Kinematic Viscosity
H	Surface Hardness
l	Length
t	Time

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1 Introduction

1.1 Background

1.1.1 Tribology

Tribology is the study of friction, wear, and lubrication between surfaces in relative motion and affects modern life to a much greater extent than is generally realized. Wear is the major cause of material waste and loss of mechanical performance. Furthermore, friction is responsible for much of the energy dissipation recognized as mechanical inefficiency [1]. Lubrication, on the other hand, provides an effective means of controlling both friction and wear. As an example of tribology's impact on modern life, it is estimated that fully one third of the world's energy consumption is used to overcome friction [2]. This friction, and the resulting wear, cost the U.S. 200 billion dollars annually due to necessary material replacements and increased fuel consumption; even small reductions in friction and wear can mean considerable savings [3].

Understanding the complex mechanical, chemical, and metallurgical relationships between friction, wear, and lubricants allows tribologists to optimize moving interfaces and lubricants [4]. In most mechanical systems, it is desirable to minimize both friction and wear, but when designing an automotive break system, for example, it is desirable to maximize friction while minimizing wear. In other instances, low friction and high wear or high friction with high wear may be desirable.

1.1.2 Friction

Friction is the resistance to sliding between two contacting bodies. It is a force acting tangentially to the interface opposite the direction of motion. The magnitude of the frictional force is proportional to the normal force on the surfaces and is often given as:

$$f = \mu F_N \tag{1-1}$$

where f is the frictional force, F_N is the force normal to the mating surfaces, and μ is the coefficient of friction. The coefficient of friction is a complex function of material, surface texture, and operating conditions [5]. Coefficients of friction are determined experimentally for mating surface and are reported as dynamic, bodies already moving, and static, when no relative motion yet exists. The force required to overcome friction in static conditions is greater than the force of friction once motion has begun.

1.1.3 Wear

Wear is the loss of material from a solid body due to mechanical and chemical processes [6]. Chemical wear results from electrochemical reactions that cause surface oxidation and/or corrosion. Mechanical wear refers to the processes by which solid body interactions remove surface material. Although, chemical and mechanical wear are synergistic processes [2], mechanical wear is generally predominant.

It is suspected that wear, w, is a function of applied load F_N, sliding speed v, coefficient of friction μ, hardness of the softer surface H, time t, and the size of the contact area as represented by a characteristic length l [6], such that:

$$w = f_{cn}(F_N, v, \mu, H, l, t) \tag{1-2}$$

or in dimensionless form:

$$w = f_{cn}(\frac{F_N}{l^2 H}, \frac{vt}{l}, \mu) \tag{1-3}$$

However, no clear correlation between frictional coefficient and wear rate has been identified. Archard's model is typically utilized to model wear. Under, Archard's model the total volume of wear is described as proportional to the load, hardness and sliding distance [7].

From a practical standpoint, wear is viewed as a continuum from mild to severe. Mild wear generally results in a surface that has been smoothed appearing polished. Severe wear, however, results in a surface that is roughened and appears damaged. The process of wear is ongoing and must be controlled. Typically the wear process starts with a newly manufactured surface. New surfaces have a high rate of mild wear during the running-in period and transition to a nearly flat rate of wearing during its useful life. As the component nears the end of its useful life, the wear increases sharply until failure. The most common wear forms are briefly introduced below.

1.1.3.1 Adhesive Wear

Adhesive wear describes the direct transfer of material between surfaces in contact. It occurs when high loads, temperatures, and/or pressures cause asperities on mating surfaces in relative motion to adhere to each other then shear apart. Surfaces that have undergone adhesive wear are often left relatively smooth with a smeared appearance. Normal running-in is a form of mild adhesive wear, while smearing and seizing result from severe adhesion. Adhesive wear is not common in equipment operating within normal design constraints [6].

1.1.3.2 Fatigue Wear

Fatigue wear occurs under cyclical loading. The stress cycles that accompany cyclical loading result in microcracks on the surface of a solid body. Continuing the stress cycles causes the fractures to migrate into the material eventually leading to microspalling. Microspalling leaves surface edges that are cold worked by the cyclical loading, thereby accelerating the process. As fatigue progresses the microcracks grow, propagate, and intersect within the material resulting in visible surface wear [8].

Fatigue wear may be accelerated by contaminant particles, improper handling, or manufacturing errors that result in surface damage. Surface dents are often encircled by raised berms that act as stress concentrators and/or allow surface to surface contact thereby accelerating the onset of spalling. Fatigue wear results in surfaces that appear dimpled and is the predominant failure mode in rolling contacts (gears, bearings, etc.).

1.1.3.3 Abrasive Wear

Abrasive wear is generally accepted to be the most common form of wear in lubricated machinery and occurs when particle contamination and/or surface asperities scratch or cut mating surfaces. There are two methods of abrasive wear, two-body abrasion and three-body abrasion.

Two-body abrasion describes damage that results when asperities on one surface directly damage a mating surface. As two surfaces slide against each other, parts of the softer surface are cut, ground into, or sanded away by the harder surface. Two-body abrasion occurs when the lubricant film thickness is not sufficient to separate the surfaces or when other wear mechanisms result in excessive surface roughness.

Three-body abrasion is wear resulting from a relatively hard contaminant (particle of dirt or wear debris) that becomes trapped between two sliding surfaces. The contaminant then cuts or gouges one, or both, of the surfaces, creating parallel furrows in the direction of motion. Contaminants that are close in size to either mechanical clearances or lubricating film thickness result in contact and three-body abrasion.

1.1.3.4 Erosion

Erosion is wear resulting from the flow of hard particles in a fluid. As particles impact a surface, small amounts of material are continuously removed. Erosive wear is a function of impact frequency, particle characteristics, and impact angle. This type of wear is consequently affected by heightened levels of particulate contamination; when the concentration of particles is not excessive, erosion is unlikely to occur.

1.1.3.5 Cavitation

Cavitation is a process that results from excess air entrainment or excessive pressure differentials. When vapor bubbles are allowed to form within low pressure regions of a fluid, at a pump inlet for example, and then pass to a relatively high pressure area, they implode. The implosions are often powerful enough to damage nearby surfaces. Hydraulic pumps and high speed impellers/propellers are particularly susceptible to cavitation. Unlike the other forms of mechanical wear discussed here, no metal-to-metal contact is needed to cause cavitation [8].

Multiple wear mechanisms are normally present in any given system. Adhesion and fatigue, for example, release hardened wear particles, which leads to three-body abrasive wear. The introduction of properly formulated lubricants and filtration can minimize many wear modes.

1.1.4 Lubricants

Lubricants perform several important functions in mechanical systems. They are employed to minimize friction, corrosion, and noise/vibration transmission. They transfer heat and power and carry away contaminants. Properly applied, these fluids form a hydraulic film between surfaces in relative motion. If the film is of sufficient thickness, surface separation occurs thus reducing friction, surface fatigue, heat generation, and wear. Furthermore, the movement of lubricants through mechanical systems has multiple benefits. Movement of hydraulic fluids transfers power via pistons and actuators, and lubricants pumped through a system aid in maintaining film thickness. Also, as a result of the flow, heat and contaminates are transported away from the mechanical.

Most lubricants and hydraulic oils are formulated to be hydrophobic and to form protective bonds with surfaces, protecting the surface from atmospheric and moisture exposure. Capillary forces draw fluid into clearances, creating seals and damping vibrations and/or sound. It is desirable then for the fluid to have several physical characteristics. It should be chemically, thermally and hydraulically stable. It must remain liquid over a wide range of temperatures, and freely release entrapped air. It should also be contaminant free, viscous and demulsible.

The performance of a lubricated contact is largely determined by the viscosity; viscosity strongly influences lubricant film thickness [9]. Too thin a film allows surface asperities to come into contact while too thick a film introduces hydrodynamic drag. Additionally, pumping difficulty increases with viscosity, complicating system design. It is generally desirable to specify the least viscous fluid that results in complete surface separation.

Lubricants and hydraulic fluids are formulated to meet the specific and varying demands of many applications. Formulations begin with one or more mineral or synthetic base oils. Base oils may be derived from several sources including crude oil, natural gas, and plants or animals (Figure 1-1). The source of the base oil will determine many of the formulated fluids final properties; plant and animal based fluids, for instance, enable the creation of biodegradable or environmentally friendly fluids [10].

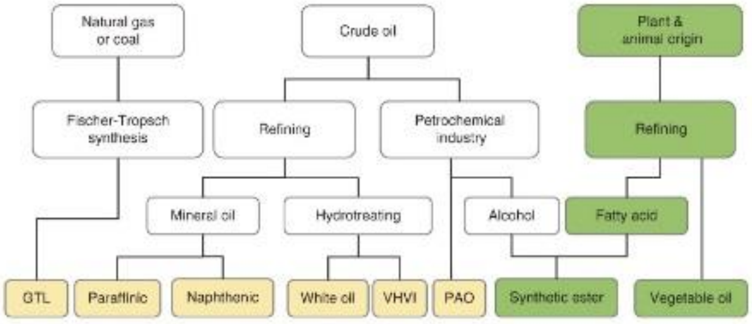


Figure 1-1: Base oil origins and types [10]

1.1.5 Additives

Lubricating oils are formulated to meet the demanding specifications of numerous applications. Base oils are fortified with chemical additives to achieve each application's specific combination of performance requirements. Performance properties commonly modified with additives include, but are not limited to, viscosity, lubricity, chemical stability, and contamination control.

Additives may be dissolved or suspended in the fluid and typically vary from 0.1% to 30% of the total oil volume. They may enhance desirable properties, suppress undesirable properties (i.e. viscosity index improvers), or impart new, desirable, properties (extreme pressure additives). In this thesis, we focus on foam inhibitors which will be described in more detail later. Some typical combinations of additives included in common lubricant formulations are summarized in Table 1-1.

Table 1-1: Common additives included in lubricant formulations, by application [11].

Application	Common Additives	Volume of Oil (%)
Engines	Antioxidants, corrosion inhibitors, detergents/dispersants, anti-wear, foam inhibitors, alkalinity improvers	10 – 30
Steam Turbines, Compressors	Antioxidants, corrosion inhibitors, demulsifiers, foam inhibitors	0.5 – 5
Gears (Spiral, bevel, or hypoid)	Anti-wear, antioxidants, foam inhibitors, corrosion inhibitors (sometimes), extreme pressure	1 – 10
Gears (Worm)	Extreme pressure, antioxidants, corrosion inhibitors, fatty acids	3 – 10
Hydraulic Fluids	Antioxidants, anti-wear, foam inhibitors, corrosion inhibitors, pour-point depressants, viscosity index improvers	2 – 10

1.2 Fluid Cleanliness

When discussing lubricants, cleanliness is not a general term but a specific quantitative value that describes the distribution and size of particles present in the fluid. There are four primary sources of solid particulate contamination in lubricant systems: contaminated new fluid, built-in contamination, ingressed, and internally-generated contamination [12].

System contamination can result in catastrophic failure, degradation failure, or intermittent failure. As a contaminated fluid interacts with surfaces, contaminants plough and cut fragments from component surfaces. Catastrophic failures result when particles causes complete failure of the machine, i.e. when large particles or debris injected into a clearance cause the entire machine to seize. Intermittent failure describes a phenomenon whereby the system momentarily fails to operate correctly, a valve failing to seat due to contamination that is washed away on the next cycle for example. Degradation failure describes the general wearing out of components that results from contaminates that are just large enough to bridge the fluid film in components. Degradation may lead to leakages in the system, decreased efficiency, and eventually catastrophic failure. Contaminant induced abrasive wear and surface fatigue accounts for almost 90% of degradation failures [12].

In order to ensure system performance, built-in contamination must be minimized. Built-in contaminants, particularly welding slag and metal shavings/chips from fabrication cause catastrophic failure within the first minutes of machine operation [12], [13]. Built-in contamination is reduced through system flushing. The effectiveness of flushing, however, depends on the filters used, temperature, viscosity, velocity, and turbulence of the flushing fluid; unless high velocities and turbulence are attained, much of the contamination will not be dislodged [12].

Contaminants are often introduced to the system when new oil is added. Lubricants are generally refined and blended under acceptably clean conditions. However, the fluid travels through many hoses, pipes and tanks before it is stored in drums or bulk tanks, all of which contribute particulate matter. When the fluid is properly stored, the main contaminants will be metal, silica, and fiber particles [12]. Multiple studies have found that new oil cleanliness often exceeds component recommendations by an order of magnitude, or more [12]–[14] .

Ingressed contamination refers to particulates that enter the lubricant from its surroundings. Common sources of ingressed particles are: missing or inoperable breathers, access panels or reservoir caps that are not properly sealed, faulty seals, and when the system is opened or disassembled for maintenance. Proper maintenance procedures and care when accessing the system can greatly reduce this source of contamination.

Corrosion, abrasive (two and three body), erosive, cavitation, fatigue, and adhesion wear all lead to an increase of particulates in the lubricant. This is internally generated contamination and is particularly troublesome. The particles generated by this wear are often work hardened and may have sharp or jagged edges. This type of contamination causes an increase in the rate of wear and thereby increases the rate of contamination and if left uncontrolled can quickly lead to component failure. Fortunately, in a properly operating system there is little internally generated particulate after the run in period [6].

Fluid cleanliness is an important indicator of system condition [15]. Monitoring programs for particles in the lubricant can be used in conjunction with basic Statistical Process Control to identify when component failure is imminent [15]–[17]. Repairs can then be scheduled before catastrophic failure, thereby minimizing productivity interruptions and machine downtime.

Particle counts, a count of the particles per mL of fluid, may be performed by automatic particle counters or through microscopy [18]. ISO 4407 [19] describes the process through which manual particle counts are obtained by microscopy. Automatic particle counter standards are presented in ISO 11500 [20].

1.2.1 Manual Particle Counting

Manual particle counts in lubricating fluids are conducted by microscopy. Particles deposited on a membrane are counted either manually or by image analysis [21]. Transmitted or incident lighting may be utilized [19].

The overall process for manual counting particles is straightforward. A known volume of fluid is vacuum filtered through a membrane. The membrane is placed in a membrane filter holder and examined by a technician, at various magnifications, to determine a particle size distribution. Conversely, the count may be automated through image analysis . A digital image of the magnified membrane is captured and software counts and reports the particles. In both cases, the count must then be checked for validity by ensuring that the particle count increases with particle size and checking for sudden increases or decreases in particle count as specified in ISO 4406 Figure A.1 [18], [19]. If the count is not validated the process is repeated. If the validation succeeds, the count is reported in accordance with ISO 4406.

Counting particles in this manner is time consuming and requires experienced personnel. As a result, manual particle counts are expensive and are usually avoided [22].

1.2.2 Automatic Particle Counters

Automatic counters are the most common method used to quantify the contamination level of hydraulic fluids. Automatic particle counters are reliable, fast, and acceptably accurate. These counters work on a simple principle that relates a voltage change to particle size. Light is passed through a narrow stream of the sample fluid. When a particle blocks the light, photoreceptors experience a voltage change proportional to the size of the particle.

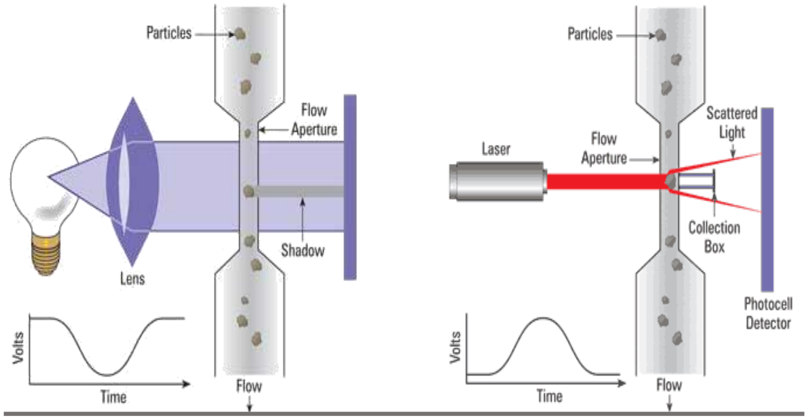


Figure 1-2: Automatic particle counting under the light extinction (left) and scattering (right) principles. A particle enters the light path and either blocks or scatters light. The change in lighting causes a photo a change in voltage at the photoreceptor. The change in voltage is proportional to the size of the particle. [25]

Common particle counters utilize either light extinction or scattering to measure a particles reflected light. Extinction particle counters illuminate a photoreceptor and measure the voltage drop caused by a particle's shadow as it passes through the viewing region, and is the only of the two methods suitable for counting particles larger than 2.0 μm in liquids [23], [24]. Particle counters that measure a particles size through the principle of light scattering, direct a laser through the fluid sample. As particles pass through the viewing area light scattered by the particle is detected by photoreceptors located either behind a light collection block, or at 90 degrees to the laser path (Figure 1-2) [22]–[25]. In both cases, particles are sized according to an equivalent diameter. Particles are counted under the size range in which a perfectly spherical droplet would have resulted in an equivalent voltage drop [24].

Both the light extinction and scattering methods of operation can result in over reporting of particles. Entrained air has been shown to cause an increase in particle counts [22]. Overly dark samples or samples containing soot can produce unreliable results and additives in oil have been shown to cause an increase in counts reported by these counters [13], [22], [26].

Advances in automatic particle counters, however, have resulted in systems that are able to classify particles and reject counts from liquids and gasses that would otherwise be reported as particles. LaserNet Fines is a system developed by the US Naval Research Laboratory, Lockheed Martin Tactical Defense Systems, and Predict/DLI [27]. As fluid passes through the viewing area, a laser is pulsed and an image of the particles are captured on a charge-coupled device (CCD) sensor (Figure 1-3) [27]. An artificial neural network analyzes the pixels captured by the CCD and determines the particles maximum diameter and surface area[13], [27]. For particles larger than 20 μm, the system also determines the particle aspect ratio, perimeter length, and circularity [27]. The particles are then classified into fatigue, sliding, and cutting wear using a mathematical algorithm; the system is also able to differentiate water and air bubbles, fibers, and other nonmetallic contaminants [13], [27]. At this time, LaserNet Fines is relatively costly and has not achieved widespread implementation.

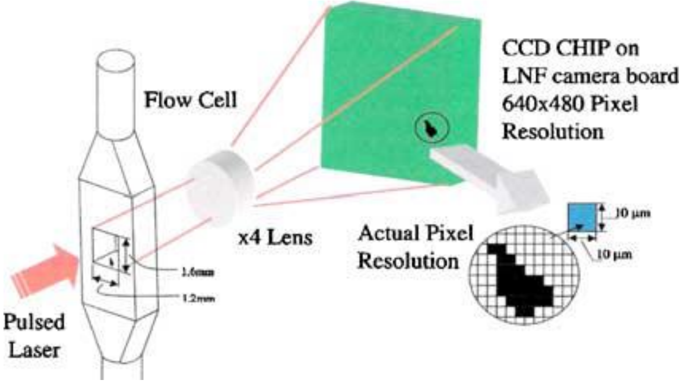


Figure 1-3: Schematic image of the LaserNet Fines particle counter. As fluid passes through the viewing area, a laser is pulsed and an image of the particles are captured on a CCD sensor. An artificial neural network analyzes the pixels and classifies the particles according to size and type. [13]

1.2.3 ISO 4406

ISO 4406 standardizes the reporting of particle count data; other particle count reporting standards include SAE AS4059 [23] and NAS 1638 [24]. Under ISO 4406, counts of particles greater than or equal to 4 µm, 6 µm, and 14 µm are classified by a scale that ranges from 0 to > 28. Each increase in code represents up to a doubling of the number of particles, with zero representing a particle count of between 0 and .01 particles per milliliter, and > 28 representing a particle count of more than 2,500,000 particles per milliliter (Table 1-2).

The three scales are then reported as a sequence of three codes separated by slashes. For example, 18/16/13 indicates that a sample contained between 1,300 and 2,500 particles greater than or equal to 4 µm, 320 to 640 particle of at least 6 µm, and 40 to 80 particles of at least 14 µm per milliliter of fluid. When less than 20 particles are counted, the statistical significance of the count is diminished and the results are reported with the ≥ symbol; this is especially problematic below ISO 8 [18]. A code of 12/9/≥6 indicates 20 to 40 particles per milliliter were 4 µm or greater, 2.5 to 5 particles per milliliter were 6 µm or greater, and that although 0.32 to 0.64 particles per milliliter of 14 µm or greater were recorded, less than 20 particles of 14 µm or larger were counted and the actual count may be higher.

Table 1-2: ISO 4406 Particle count codes. Particles are reported in the form a/b/c, where a, b, and c correspond to the ISO code for counts larger than 4 µm, 6 µm, and 14µm respectively. [18]

More than	Up to and including	ISO Code
2,500000		>28
1,300,000	2,500000	28
640,000	1,300,000	27
320,000	640,000	26
160,000	320,000	25
80,000	160,000	24
40,000	80,000	23
20,000	40,000	22
10,000	20,000	21
5,000	10,000	20
2,500	5,000	19
1300	2,500	18
640	1300	17
320	640	16
160	320	15
80	160	14
40	80	13
20	40	12
10	20	11
5	10	10
2.5	5	9
1.3	2.5	8
0.64	1.3	7
0.32	0.64	6
0.16	0.32	5
0.08	0.16	4
0.04	0.08	3
0.02	0.04	2
0.01	0.02	1
0.00	0.01	0

As it is common for manufacturers to describe minimum lubricant cleanliness levels using ISO 4406, the standard also specifies a method to indicate that a count at a given level is not required. When there is no requirement for a given size, two hyphens take the place of the code; 18/--/-- indicates that there is no restriction on particles 6 µm or larger, and that the lubricant should have no more than 2500 4 μm particles per milliliter. Similarly, a minimum cleanliness of --/13/11 relates that there is no restriction on particles smaller than 6 μm.

1.3 Foam and Foam Inhibitors

1.3.1 Fluid Aeration and Foaming

Foam occurs when bubbles in the fluid become enlarged, rise to the fluid surface, and remain stable. Bubbles may be formed by equipment design. Splash lubrication can result in significant entrained air. Liquid streams entrain air as they enter fluid surfaces, for example, return lines above the reservoir level and/or relief valves. Cavitation and system leaks, especially at the pump intake, can result in fluid aeration as well.

Liquid aeration is distinct from, but related to, foaming. Aeration or air entrainment describes small bubbles trapped in the bulk fluid. The rate of bubble rise is determined by Stoke's Law which indicates that the rate of ascent is a function of fluid viscosity and bubble size [28]. Additionally, bubbles that enter the reservoir prior to rupture, may rise to the surface or be drawn back into the pump as entrained air. In order to minimize fluid aeration it is important that the fluid reservoir be large enough to provide enough resident time for the bubbles to escape the fluid.

Lubricant aeration and foaming can result in increased system noise, poor component response, cavitation, and fluid degradation. Cavitation results when the pressure in a system drops below the saturation pressure of a gas dissolved in the fluid. The gas then forms bubbles, small gas pockets entrained in the fluid, which continue through the system. If bubbles enter a region of high pressure they often rupture violently resulting in component damage. As the bubbles are adiabatically compressed, temperature at the surface of the bubble rises sharply, speeding fluid degradation processes such as oxidation [29]. The increases in fluid compressibility that results from entrained air leads to poor power transmission and a spongy response. Foaming may result in an overflow if the volume of foam exceeds reservoir capacity, or, in extreme cases, may lower the lubricant level sufficiently to result in pump starvation, cavitation, and severe air entrainment.

The causes of foaming are poorly understood, however, it is clear that there must be a source of air in order to form bubbles in the fluid and a surfactant to stabilize the bubbles once they rise to the surface [28]. It is known that pure fluids do not foam and that the presence of a small amount of surfactant is required to enable foam formation [28], [29]. In the case of unblended hydrocarbon fluids, mixed isomers act as the surfactant [29]. Additionally, additives designed to enhance other lubricant performance characteristics can also increase foaming [28]–[30].

1.3.2 ASTM D892

ASTM D892 provides a means of evaluating the foaming characteristics of lubricating oils [31]. Air is passed through a diffusing stone into a sample of oil for five minutes at controlled temperature. After five minutes have elapsed, the volume of foam is recorded in milliliters as the oils foaming tendency. The oil is then allowed to rest for ten minutes. At the conclusion of ten minutes the remaining volume of foam is recorded as the foam stability. As it is common for the oils to have zero foam stability, the time required to reach zero foam, in seconds, is routinely reported as foam stability.

The equipment and process for carrying out the test is prescribed in the standard. The apparatus consist of a 1000 mL graduated cylinder, two baths capable of maintainin 24 ± 0.5 °C and 93.5 ± 0.5 °C respectively, a method of supplying clean dry air to a metal or stone diffuser at 94 ± 5 mL/min, and a method for measuring the volume of air supplied over the five minute test period.

Cleaning processes for the equipment are proposed in the standard and it is noted that failure to fully clean the apparatus between tests will seriously interfere with future results. The standard suggests that the cylinder be rinsed with heptane, cleaned with a laboratory grade detergent, rinsed in distilled water, rinsed with acetone, and then air dried. Furthermore, it notes that several rinsings with distilled water and acetone may be required to fully clean the cylinder. It is proposed that toluene and then heptane be flushed back-and-forth through the diffusing stone a minimum of five times. The stone should then be air dried. It is important that the stone not be touched by hands. Although these cleaning methods are not required, it is noted that results are sensitive to contamination. Under all circumstances, cleaning should result in removal of all prior test material.

A sequence of three tests is required to fully evaluate the foam characteristics of an oil. The first trial, Sequence I, is carried out at 24 ± 0.5 °C. A second sample, Sequence II, is tested at 93.5 ± 0.5 °C. For the third test, Sequence III, the second sample is retested at 24 ± 0.5 °C. A more detailed description of the three tests can be found in Sections 3.3.1.1 through 3.3.1.3.

The standard provides for two common variations to the test procedures outlined above, Option A and alternative procedure IP 146. Option A describes procedures for samples that have been stored for two weeks or more. Under Option A, 500 mL of the fluid to be tested is measured into a solvent cleaned 1000 mL high-speed blender. The fluid is then blended at maximum speed for one minute. After mixing, the sample is allowed to rest until all entrained bubbles have dissipated.

Alternative IP 146 allows the tests to be conducted without measuring the volume of air passed through the system as long as the air source is correctly calibrated and closely monitored. Although the alternative is specified as part of the standard, the standard warns that without capturing and measuring the volume of air passed through the system there is no way to ensure the system is air tight and requires that results obtained by the alternative procedure be identified by ASTM D892 – IP 146.

ASTM conducted a cooperative laboratory program to determine repeatability and reproducibility guidelines [31]. The resulting expectations for repeatability and reproducibility are presented in Figure 1-4 and Figure 1-5 respectively. The solid lines in the figures represent values for the foaming tendency in Sequence I, II, and III and the foam stability for Sequence I and II; Sequence III stability was found to have more variability and is indicated by the dashed lines. The figures indicate that successive results from a single technician conducting characterizations according to standard, utilizing identical test material and identical test equipment, should exceed the values in Figure 1-4 for only one case in twenty [31]. Likewise, two technicians working in independent laboratories on identical material should produce results that exceed the values in Figure 1-5 for only one in twenty cases [31]. The cooperative study was unable to determine repeatability of the tests under option A and because there is no accepted reference material, the bias of the process remains undetermined [31].

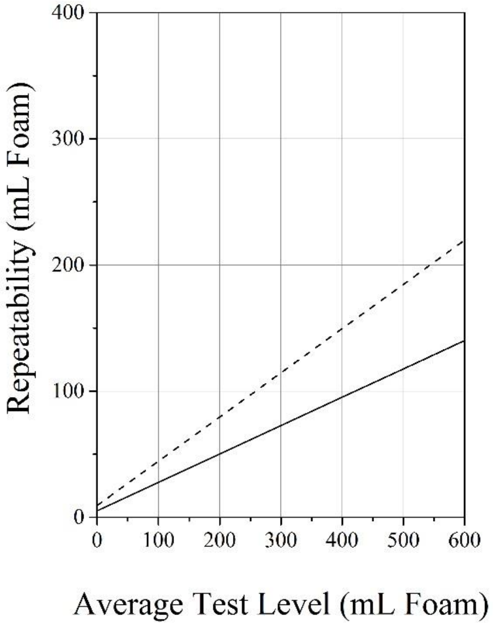


Figure 1-4: Maximum expected variation between ASTM D892 characterizations performed on identical test material by the same technician utilizing identical test equipment. The solid line represents values for the foaming tendency in Sequence I, II, and III and the foam stability for Sequence I and II; Sequence III stability is indicated by the dashed line [31]

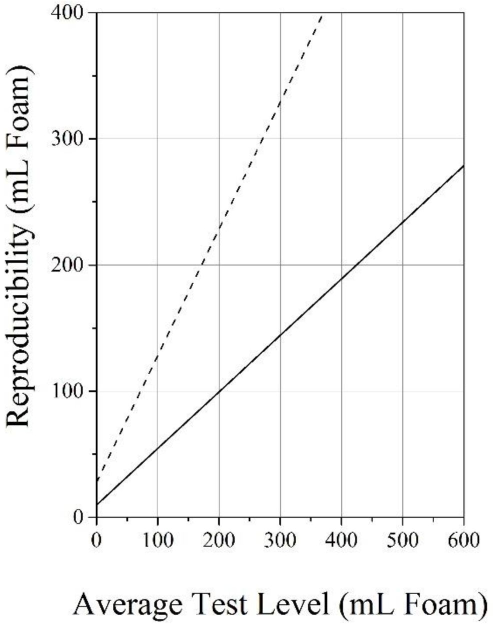


Figure 1-5: Maximum expected variation between ASTM D892 characterizations performed on identical test materials by different technicians in different laboratories. The solid line represents values for the foaming tendency in Sequence I, II, and III and the foam stability for Sequence I and II; Sequence III stability is indicated by the dashed line [31]

1.3.3 Foam Inhibitors

Foam Inhibitors (FIs, antifoams) are included in lubricant formulations to control foaming tendency and persistence. Although the exact method of foam destruction is not fully understood, it is generally accepted that there are two primary methods of foam destruction [28], [29], [32]. Foam is unstable by nature and seeks to return to its lowest energy liquid state. The return to the liquid state is accelerated when foam inhibitors spread along the bubble surface and displace the foam's stabilizing surfactant film thinning the bubble wall and leading to rupture [28], [29], [33]. The other primary method of foam destruction is through local weakening of bubble walls. Small droplets of non-spreading low surface tension inhibitors form small micelles on the surface of the bubble. As the bubble spreads the low surface tension creates a weak spot on the surface of the bubble and causes the wall of the bubble to fail (Figure 1-6) [29].

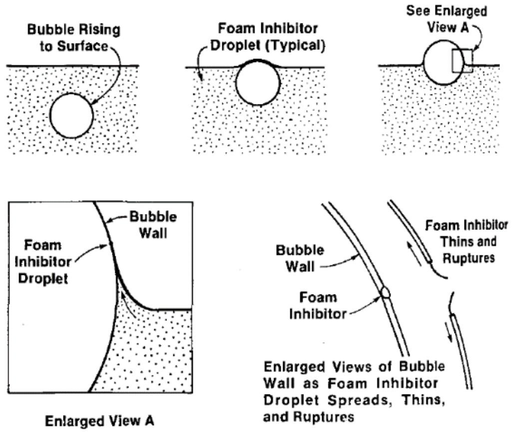


Figure 1-6: Foam inhibitor droplets dispersed within a fluid coalesce on a bubble wall, weaken the surface, and cause bubble rupture [28].

Effective foam inhibition requires chemicals with specific characteristics. They must have a surface tension lower than the base fluid. They must be insoluble in the base oil. Additionally, they must be dispersible into fine droplets within the base oil. Many materials meet these requirements and may be employed as foam inhibitors in lubricants. Silicones and acrylic copolymers are the most common foam inhibitors in petroleum based fluids, and have been so for some time. The foam inhibitors included in our study include perfluoropolyether, polyacrylate, and organosiloxane compounds.

1.4 Filtration, Foam Inhibitors, and Fluid Cleanliness

1.4.1 Filtrations Effect on FIs

In 1987 Chevron received reports that transmission fluid which had met ASTM foam performance specifications, failed foam tests after 50 hours of operation; T.V. Friesen conducted an investigation into the matter. Field tests were conducted, comparing fresh fluid to fluid from several John Deere tractors. All of the used fluids failed to meet foaming specifications (Table 1-3) [28]. These findings led to further tests which revealed a relationship between filtration and foam performance. First, it was found that tractor hydraulic fluid circulated through a 25 μm paper filter for up to 93 hours successfully met foam specifications. However the addition of a 7 μm synthetic filter resulted in a sharp increase in foaming and failure to meet specifications after only 24 hours of filtration [28]. Subsequent tests with various filters found the loss in foam performance was accentuated by decreasing filter pore size and synthetic filters (Table 1-4). Finally, it was determined that the increase in foaming was not fluid specific; three widely used fluids were tested

Table 1-3: ASTM D892 Foam Test Results, John Deere Tractor Field Test, Friesen 1987 [28].

Tractor Model		4240	4240	4040	2840	4440	Foam Spec
Hours on Oil	Fresh	407	152	298	301	252	25/0
Volume of Foam, (mL)		After Blowing / After 30s Settling					
Sequence I	0/0	0/0	50/0	10/0	0/0	30/0	25/0
Sequence II	30/0	150/0	180/0	270/0	130/0	290/0	50/0
Sequence III	0/0	0/0	10/0	20/0	0/0	40/0	25/0

under identical conditions. All of the fluids showed dramatic increases in foaming tendency and foam stability (Table 1-5).

Tests indicated that the filters were removing the foam inhibitor from the fluid. A fluid with 50 ppm silicone antifoam was filtered for 121 hours with a 7 µm synthetic filter.

Table 1-4: Foam test results, transmission-hydraulic fluid filtered through various filters for 24 hour hours at 57 ºC and 9.5 L/min, Friesen 1987 [28].

Filter	Sequence	Tendency (mL)	Settling	
			Sec to Break	mL @ 10 min
Fresh Fluid Before Filtering	I	0	0	0
	II	20	15	0
No Filter	I	Nil	0	Nil
	II	40	67	0
10 µm paper	I	170	>600	25
	II	550	212	0
2 µm paper	I	330	>600	35
	II	580	225	0
7 µm synthetic	I	430	>600	135
	II	510	>600	220
3 µm synthetic	I	450	>600	175
	II	730	>600	270

The filter was found to contain 4400 ppm silicone, confirming that the filter was removing FI from the fluid. Further experiments indicated that the concentrated silicone FI was not due to adsorption but that the FI was preferentially wetting the filter media [28].

Efforts to over-treat the fluid in hopes that enough silicone would remain in the fluid, post filtering, to meet foaming specifications proved unsuccessful. Two fluids, one with 10 ppm silicone and the other with 3000 ppm silicone, were filtered with a 7 µm synthetic filter for 24 hours. Both fluids initially met foaming specifications, as identified in Table 1-3, before filtration. After filtration, neither fluid met the specification for foam tendency (Table 1-6). It was concluded that in-use fluids should not be expected to meet new fluid foam specifications and that specifications should be written that anticipate the loss of performance from filtration [28].

Table 1-5: Comparison of the effects of filtration time on foaming tendency and foam stability for three widely used transmission-hydraulic fluids, Friesen 1987 [28].

Fluid	Hours Filtered	Sequence	Tendency (mL)	Settling	
				Sec to Break	mL @ 10 min
Fluid A	0.2	I II	Nil 10	0 9	0 0
	48	I II	480 65	395 30	0 0
	72	I II	470 55	463 36	Nil 0
Fluid B	0.2	I II	Nil 70	0 55	0 0
	48	I II	260 670	>600 >600	20 300
	72	I II	260 720	>600 >600	85 355
Fluid C	0.2	I II	20 110	70 52	Nil 0
	48	I II	560 720	>600 532	295 0
	72	I II	680 740	>600 >600	340 115

Table 1-6: Foam characterization results (pre and post filtration) for transmission-hydraulic fluid charged with 10 ppm silicone and for the same fluid charged with 3000 ppm silicone, Friesen 1987 [28].

10 ppm of 6,000 cSt Silicone			
Hours Filtered	Sequence	After Blowing (mL)	Vol at 600 Sec (mL)
0	I II	0 20	0 0
24	I II	430 510	135 220
3000 ppm 60,000 cSt Silicone			
Hours Filtered	Sequence	After Blowing (mL)	Vol at 600 Sec (mL)
0	I II	15 15	0 0
24	I II	505 130	0 0

1.4.2 Additive Effects on Lubricant Cleanliness

It has been shown that some additive packages can increase particles reported by optical particle counters [13], [14], [26]. A 1996 investigation into procedures to deliver clean lubricants to end users found that the inclusion of a specific additive package resulted in increased particle counts [14]. This finding lead to additional studies into the matter; surface active additives such as foam inhibitors, detergents and anti-wear agents have been found to be the primary source of this increase with FIs causing the most dramatic effects.

Table 1-7: Particle count results for Group I and Group III base oils before additive introduction, 15 min after introduction, and after thirty minutes of filtration, Michael et al. 2007 [13].

Additive	Base Oil at Start of Test 100 °F	Filter Off 15 Min After Additive Addition	After 30 Min of Filtration
	Group I, ISO Code		
50 TBN calcium sulfonate	15/13/<7	13/11/9	13/12/9
400 TBN calcium sulfonate	15/13/10	15/14/11	13/9/<7
Succinimide	13/11/8	16/14/11	16/14/11
Zinc dithiophosphate	16/15/12	17/15/11	17/15/12
Boronated succinimide	16/14/11	17/15/11	17/15/11
Polyacrylate	14/12/8	16/12/10	17/14/12
Polydimethylsiloxane	15/13/9	>28/22/21	>28/22/16
Diesel engine additive package	17/14/<7	22/19/14	20/16/7
	Group III, ISO Code		
50 TBN calcium sulfonate	15/13/10	15/13/10	15/14/10
400 TBN calcium sulfonate	13/12/11	15/13/10	15/13/10
Succinimide	15/13/10	15/14/10	15/13/10
Zinc dithiophosphate	15/14/10	15/14/10	14/12/9
Boronated succinimide	13/12/7	14/12/10	14/13/11
Polyacrylate	12/10/<7	15/12/11	15/12/11
Polydimethylsiloxane	15/12/10	>28/24/21	21/19/12
Diesel engine additive package	18/14/<7	22/19/15	21/16/7

In 2007, Paul Michael and co-workers conducted a study into the effects of base oil solubility on optical particle counts and found no clear relationship between base oil group and the effects of additives on particle counts. In Michael et al.'s study, a base oil was circulated through a test rig at a rate of one fluid change per minute (i.e. a 5 gal reservoir run at 5 gpm). Additives were introduced into the reservoir and allowed to mix via circulation. After 30 min of mixing, the fluid was filtered for 30 minutes or until reaching ISO --/15/12, whichever came first. Through the entire process, particle counts were obtained from a MP Filtri LPA-2 in-line particle counter. Samples were taken for offline analysis as well. As can be seen in Table 1-7, Michael et al. did not observe any correlation between the base oil group and the magnitude of the observed particle count increase. He did, however, note that the largest increases in particle count came from the polydimethylsiloxane (PDMS) foam inhibitor and diesel engine additive package, and that they responded less predictably to filtration than the remaining additives [13]. A 2009 study confirmed Michael et al.'s findings and noted that non-silicone based antifoams also caused a large increase in observed particle counts [26].

Table 1-8: Comparison of particle count results for Group I base oil plus Detergent- Inhibitor (DI) package for three instruments, Michael et al. 1997 [13]

Description	LPA-2	8000 A	LNF
Group I Base Oil @ 120 ºF	13/9/<8	15/11/9	13/11/<10
Addition of DI Package	22/20/13	23/21/16	23/21/15
	22/20/16	23/21/16	23/21/15
Begin Filtration	20/15/10	21/15/19	17/14/10
	19/14/9	21/14/<8	16/13/<10
	18/13/8	20/14/9	15/13/10

Michael et al. also utilized a Detergent-Inhibitor (DI) package containing 6 ppm silicone to compare additive effects on different particle counters. A group I base oil was circulated through his test rig, a DI package was added to the reservoir and allowed to mix before being filtered. Particle counts were obtained by the MP Filtri LPA-2 online counter and offline by two desktop particle counters, a HIAC 8000A and a Spectro LaserNet Fines. The DI package was found to increase counts for all additives substantially, but there was less agreement in the post filtering counts (Table 1-9) [13]. LaserNet Fines detected many hollow spherical particles that the LaserNet Fines artificial neural network classified as water droplets; it is believed that these are droplets of silicone antifoam [13].

Table 1-9: ASTM D892 foam test results for DI package in Group I and III base oils pre and post filtration. Michael et al. 2007 [13].

		Group I Before/After		Group III Before/After	
Seq.	Temp.	Tendency (mL)	Collapse Time Seconds	Tendency (mL)	Collapse Time Seconds
I	24.0 ºC	0/0	0/0	0/10	0/0
II	93.5 ºC	20/30	11/16	20/40	10/18
III	24.0 ºC	0/0	0/0	10/20	12/31

Michael et al. concluded that silicone antifoam agent is the most likely source of additive induced particle counts and performed two analyses to determine possible effects of filtering on foam inhibitor performance. Silicone inhibitors must be dispersed into the fluid in small droplets in order to function [13], [28], [29] . The reduction in particle counts post filtration likely came from removal of FI droplets. A comparison of pre and post filtering foam performance indicated a slight increase in foaming tendency and foam stability (Table 1-9). Additionally, Michael et al. found through an inductively coupled plasma (ICP) analysis that the change in silicone content was 1 ppm or less and calculated that 90% of the FI remained in the fluid at particle sizes smaller than 4 μm [13].

1.5 Research Objective

We believe that the most immediate method of controlling the effects of foam inhibiting additives on new fluid cleanliness to be fluid filtration. Technological solutions such as LaserNet Fines are currently available, however their relative expense prevents wide spread use. Furthermore, while advanced mixing methods may promote a dispersion with a maximum size that is invisible to ISO 4406, the mechanics of liquid-liquid dispersions are quite complex, and scaling liquid-liquid mixing experiments is notoriously difficult [34]. Lastly, filtration for cleanliness is already a common industry practice.

While it is known that FIs degrade fluid cleanliness ratings and that prolonged filtration degrades FI performance, the rate at which foam performance deteriorates has not been studied. This investigation seeks to understand the effects of filtration as commonly seen in lubricant manufacturing and to deepen the understanding of the effects of FI chemistry on additive induced particle counts. Our goal is to identify and characterize the interaction of foam inhibitors, optical particle counters and filters over the first ten filter passes. We seek to understand the effect of filtering on FI induced particle counts and quantify the resulting changes in FI performance as characterized by ASTM standard D892. Furthermore, we will evaluate these effects across multiple additive chemistries and filter pore sizes.

This first chapter serves to provide the required background information and a clarification of the scope and purpose of this work. Chapter 2 will detail the test equipment and materials utilized in this study. We will present our research processes in Chapter 3. A discussion of our experimental results follows, in Chapter 4, while Chapter 5 presents our conclusions from this work and recommendations for future studies.

2 Research Equipment

2.1 Fluids

2.1.1 Base Oil, Chevron 220R

The American Petroleum Institute (API) has categorized base oils into five groups [36]. The first three groups are refined from petroleum crude oil. Group IV base oils are full synthetic (polyalphaolefin) oils. Group V is for all oils not included in Groups I through IV. Previous research has indicated that base oil group has little effect on additive induced particle counts [13].

Chevron 220R, a Group II neutral oil, was selected as the base oil for this study. It is a globally available premium base oil with high oxidation stability, low volatility, and low sulfur. It is commonly used as the base for engine, process, and industrial oil blends. Table 2-1 presents readily available proprieties of 220R.

Table 2-1: Typical properties of Chevron 220R Neutral Oil [35]

API Base Oil Category	Group II
Appearance	Bright and Clear
Color	L0.5
API Gravity, deg.	32.1
Density, lb./gal	7.202
Density, kg/l	.865
Specific Gravity @ 60 ^{0}F/60 ^{0}F	.865
Viscosity @ 40 ^{0}C, cSt	41.2
Viscosity @ 100 ^{0}C cSt	6.4
Viscosity @ 100 ^{0}F SUS	212
Viscosity Index	104
CCS @ -20 ^{0}C, cP	2800
CCS @ -25 ^{0}C, cP	5600
CCS @ -30 ^{0}C, cP	N/A
Pour Point, ^{0}C	-15
Flash point, COC, ^{0}C	230
Volatility, wt. % distilled at 700 ^{0}F/ 371 ^{0}C	N/A
Evaporative Loss, Noack wt. %	10
Water, ppm	<50
Sulfur, ppm	<6
Saturates, HPLC wt. %	>99
Aromatics, HPLC wt. %	<1

2.1.2 Foam Inhibitors

2.1.2.1 FI-S1

Silicone 1000, subsequently referred to as FI-S1, is the identifier given to a polydimethylsiloxane (PDMS) foam inhibitor comprised of Dow Corning 1000 Silicone in a hydrocarbon solvent. The solution is approximately 1 wt. % silicone. PDMS has been long been used as an effective FI and has been shown to have a large effect on ISO 4406 particle counts [13], [26]. Treatment rates are generally such that the silicone delivered is 1 to 10 ppm [37], or 0.01 to 0.1 wt. %, though higher treatment rates are not uncommon [38]

2.1.2.2 FI-A

Table 2-2: Available properties of FI-A

Appearance	Clear/ Colorless to light yellow liquid
Refractive Index, nD25 @ 25 ^{0}C	1.4380 – 1.4405
Non-volatiles, % (2 hrs. @ 105 ^{0}C)	38-42
Viscosity @ 38 ^{0}C, SUS	150 – 330
Viscosity @ 25 ^{0}C cP	30 – 80
Specific Gravity @ 60 ^{0}F	.84 – .86
Color APHA max	150
Flash Point, (Pensky Martin, Closed Cup) ^{0}F (^{0}C)	144 (60)
Solvent	Hydrocarbon
Solubility in Water	Negligible

Acrylate A (FI-A) is silicone-free general use foam inhibitor for non-aqueous fluids. It is a 40% solution of acrylic copolymer in a petroleum solvent. Treatment rates range from 50 – 500 ppm (20 – 200 ppm active polymer), but rates of 200-250 ppm (80 – 100 ppm polymer) are most common. Readily available properties of FI-A are summarized in Table 2-2.

2.1.2.3 FI-MO

Modified Organosilicone A, subsequently referred to as FI-MO, is a blend of silicone compounds (organosiloxanes) dispersed in synthetic hydrocarbons. It is primarily included in non-aqueous grinding fluids, cutting oils, hydraulic oils, and gear oils. Typical treatment rates range from 0.05 – 0.10 percent by weight. Few properties of this additive are readily available (Table 2-4).

Table 2-4: Available properties of Modified Organosilicone A.

Appearance	Clear colorless liquid
Boiling point, ^{0}C	260
Flash Point, ^{0}C	> 118
Specific Gravity	.82
Solubility in Other Solvents	Insolvent
Viscosity @ 25^{0}C, mPas	< 50
Viscosity @ 40^{0}C, mm^{2}/s	<25
Density, @ 20 ^{0}C lb./gal	6.8

2.1.2.4 FI-E

Experimental FI A, FI-E subsequently, is an experimental foam inhibitor that is neither silicon nor acrylate based. FI-E exhibits a low pour point yet remains stable at high temperatures, making it appropriate for application across a wide temperature range. Additionally, its viscosity is within a range that enables it to be readily re-dispersed into the fluid after resting. Treatment rates range from 5 ppm to 1 wt. %. Table 2-3 summarizes the properties of FI-E.

Table 2-3: Available properties of Experimental FI A.

Average Molecular Weight (amu)	3700
Viscosity @ 20 ^{0}C, cSt	250
Viscosity @ 40 ^{0}C cSt	80
Viscosity @ 100 ^{0}F SUS	10
Viscosity Index	108
Pour Point, ^{0}C	-35
Evaporative Loss, @ 120 ^{0}C, 22 hr. wt. %	-
Evaporative Loss, @ 149 ^{0}C, 22 hr. wt. %	2
Evaporative Loss, @ 204 ^{0}C, 22 hr. wt. %	15
Surface Tension, @ 20 ^{0}C dyne/cm	22
Density, @ 20 ^{0}C g/cm^{3}	1.9

2.2 Blending Station

A lubricant blending station that provides control over blending parameters such as temperature, impeller speed, and container size was assembled (Figure 2-1). A magnetic stirrer/hot plate allows fluids to be heated and stirred via magnetic stir bars. The station is also equipped with a 1/15 hp adjustable speed motor, enabling mixing speeds of up to 2000 rpm. The motor is coupled to a 3 in 4-blade rotary impeller by a 3/8 in stainless steel shaft. We are able to mix up to 2 gallons of fluid with impeller to container ratios, an important factor in liquid-liquid mixing [39], of 1.3 to 2.67.

Figure 2-1: Fluid blending station utilized in this work, capable of heating and blending fluids in batches of up to 2 gallons.

2.3 Foam Station

We obtained and repaired a nonoperational Koehler Dual Bath Foaming Characteristics Apparatus (Figure 2-2). The apparatus is composed of two liquid baths, a hot tank, and room temperature (cold) tank. The hot tank contains two resistive heating elements, while the cold tank is equipped with a single heating element and a two pass cooling coil. Each tank contains an agitator to minimize temperature variations within the bath. An over-temperature controller allows the system to operate at temperatures above 100 ºC, four flow meters deliver air to up to four testing cylinders, and a wet test gas meter records the volume of air passed through the cylinders.

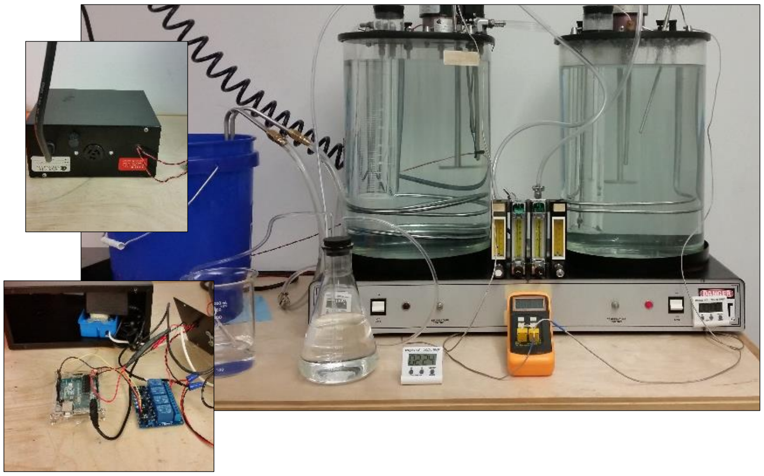


Figure 2-2: (Central) Koehler Dual Bath Foaming characteristics Apparatus after repairs and with replacement accessories (Central). Cooling loop controls (Bottom Left). Rear of control box after installation of cooling loop controls. (Top left)

Upon receipt of the unit, one tank agitator was not operational and the unit was unable to regulate the temperature of either tank. We were unable to obtain original equipment (OE) replacements for the cooling loop components or the wet gas meter. The OE cooling loop controller and related components are unavailable and while equivalent commercial wet gas meters are available, they were found to be cost prohibitive.

The unit was returned to operating condition. First, the system was disassembled and all electrical connections were cleaned. The agitator motor was found to have seized bearings and was replaced. It was then determined that the over-temperature controller (OTC) was malfunctioning. This controller is not required for operation of the unit when performing ASTM D892 trials (Section 1.3.2), so it was bypassed. After bypassing the OTC and reassembling the unit, it was able to maintain the temperature of the hot bath as required.

At this point, the system was capable of maintaining the temperature of the cold bath only while the hot tank was not in use. Use of the hot bath resulted in the temperature of the cold bath exceeding allowable values. Replacement components for the cooling loop were designed and constructed (see Section 2.3.1). The unit was then able to regulate the temperatures of both tanks as required by ASTM D892.

ASMT D892 requires specifies collection of the air volume delivered to each cylinder. Additionally, the standard describes one possible method of obtaining the air volume without utilizing a wet gas test meter. We implemented a similar solution (Section 2.3.2) and completed repair of the foam test station. In its current condition the foam test station enables compliance with all ASTM D892 Standards.

2.3.1 Cooling Loop

Operation of the partially repaired foam test station resulted in a steady state temperature of approximately 25 °C. This indicated that the cooling loop would be required to meet operational guidelines. The temperature rise of the tank was slow. The maximum temperature of the cold tank was not achieved until the hot tank had been operational for approximately 6 hours. This indicated that the rate of heat transfer was slow and could be easily controlled with a low flow water/ice-bath cooling loop.

The implemented system contains four primary components: a controller, pump, and two coils. The fluid is pumped through the cooling coils in the cold tank to a larger set of coils in an ice bath (Figure 2-3). The cooling coils in the cold tank transfer heat from the tank to the cooling fluid (water). The water in turn transfers its heat to the ice bath. A 1gpm pump circulates the fluid. The tank temperature is monitored by a 10 kΩ thermistor and controlled by an Arduino UNO micro controller.

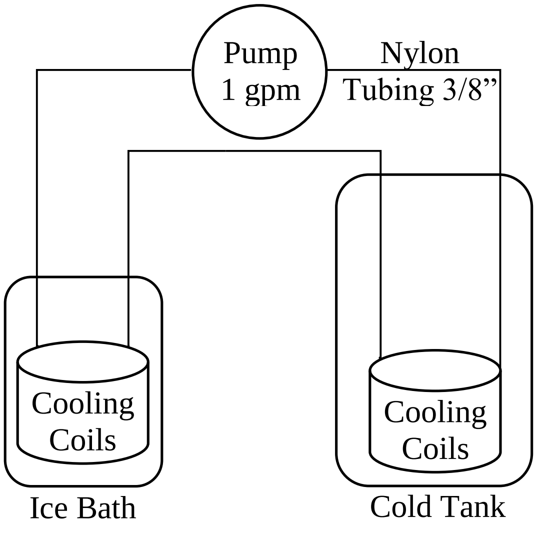


Figure 2-3: Cooling loop visualization. Water is circulated though the foam test station cold tank and ice bath, maintain the temperature of the bath to 23.5 ± 0.5 °C.

A simple control system was implemented. The temperature of the tank is monitored by the controller. When the temperature of the tank reaches 24.3 ºC, the controller activates the pump. Water circulates through the system until the temperature of the tank reaches 23.9 ºC, the controller then turns off the cooling pump. The cooling loop and implemented controls successfully maintain the maximum temperature of the cold tank, while the heating element and OE controls maintain the minimum temperature range.

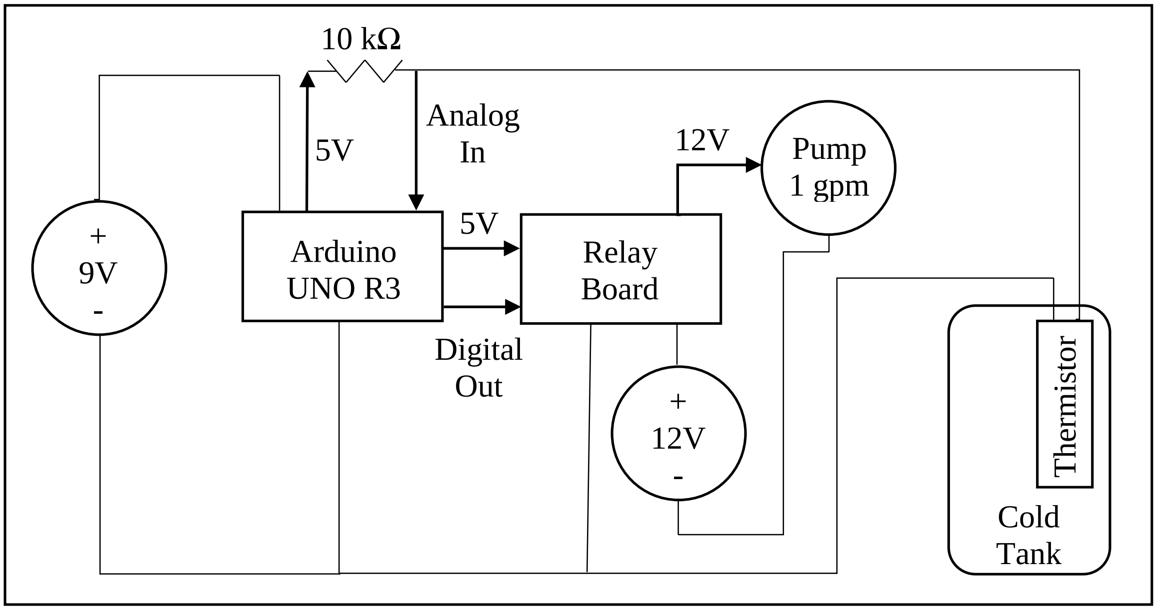


Figure 2-4: Cooling loop schematic. A microcontroller monitors the temperature of a thermistor submerged in the room temperature foam test bath. When the temperature exceeds the set point, a signal activates the 1 gpm pump through a relay board. When the temperature then reaches the lower set point, the pump is deactivated.

2.3.2 Air Volume Collection Device

ASTM D892 (Section 6.3, Note 8), describes a device for collecting the volume of air passed through a test cylinder. A 1 L cylinder full of water is inverted into a tall beaker also filled with water. Metered air then flows through the test cylinder and into the inner cylinder, displacing water. The volume of air is measured by equalizing the water levels inside and outside of the cylinder [31].

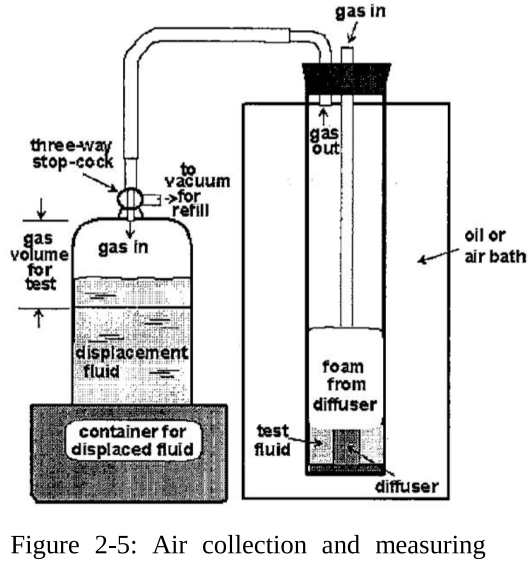


Figure 2-5: Air collection and measuring device utilized in inter-laboratory study on process repeatability as described in ASTM D892 Section X.2.2.1 [31].

A similar method was utilized in the inter-laboratory study used to determine the standards repeatability [31]. The device employed by the inter-laboratory study (Figure 2-5) consisted of a large container connected to the test cylinder and inverted into a container filled with fluid. A vacuum is applied to the large container, filling it with fluid. After the container was filled with fluid, the vacuum is disconnected. During the test air flows into the large container and displaces fluid. The water level is then equalized to measure the volume of collected air.

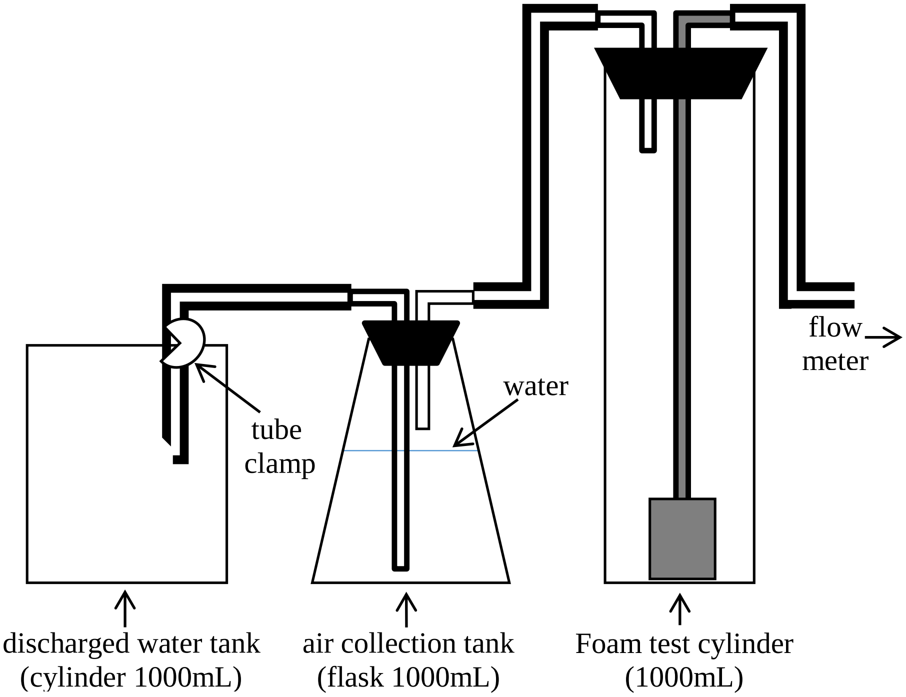


Figure 2-6: Air collection device used to conduct ASTM D892 foam characterizations. A syphon is established between the air collection tank and discharged water tank. Air flowing through the foam test cylinder then displaces water from the air collection tank into the discharged water tank. After equalizing the pressure in the two vessels by leveling the fluid and closing the tube clamp, the volume of displaced fluid is equal to the volume of air passed through the foam test cylinder.

The apparatus designed and implemented here (Figure 2-6) works on similar principles as those described in the standard. The implemented apparatus utilizes a 1000 mL Erlenmeyer flask and 1000 mL short form graduated cylinder. The Erlenmeyer flask is filled with water, a two hole stopper fitted with glass tubing is inserted into the flask and a syphon is established on one of the outlets. The inlet of the stopper is connected to the foam test cylinder. When all lines are connected, the syphon will cease to flow. The outlet the syphon tube is then placed in the graduated cylinder. When air flows through the test cylinder, the water is again able to passes through the syphon. At the conclusion of the test, the water level of the flask and cylinder are equalized. The volume of air passed through the system is then known by the volume of collected water.

2.4 Filter Station

2.4.1 *Design Requirements*

Characterizing the effects of filtration on ISO 4406 cleanliness and FI performance required a test station that could circulate, filter and, particle count fluids. Several design requirements were determined for the filter test station. In order to minimize fluid usage, it was preferred that the rig operate on as little fluid as possible while still allowing collection of five to ten 500 mL samples. Maximizing testing flexibility dictated that the filter rig accept a wide range of fluid viscosities, offer variable flow rates (up to one fluid change per minute), and operate at temperatures ranging from room temperature (~20 ºC) to 60 ºC. Additionally, the filter rig should utilize a specific set of filters (Donaldson DT Synteq filters) and a common inline particle counter in order to match known industry practices. Finally, in order to ease construction and maximize our ability to modify the design, we prefer ½ stainless steel tubing and compression fittings [40] be employed to plumb the system.

Figure 2-7: Donaldson HPK02 filter housing is compatible with DT Synteq filters, has a maximum working pressure of 2000 psi and a burst pressure of 4500 psi. It accepts flow rates up to 20 gpm [41] .

Figure 2-8: HIAC ROC inline particle counters are commonly utilized in process monitoring and operate under a wide range of conditions. [42]

The required filters, Donaldson DT Synteq, are only compatible with select filter housings. As there are few differences in the compatible housings, we selected a Donaldson HPK02 housing (Figure 2-7) [41]. The inline particle counter selected for this application, a HIAC ROC-01, is known to be commonly utilized, required the least amount of lead time, is compatible with a wide range of fluids, viscosities, pressures, and temperatures (Figure 2-8) [42]–[44].

2.4.2 *Design Process*

2.4.2.1 *Reservoir*

When determining the reservoir size there were two primary considerations, the volume of fluid required to operate the system without causing fluid aeration and the total volume of fluid that would be removed from the system by sampling. The selected filter housing has a fluid capacity of approximately 0.25 gal, and during the design process, it was predicted that the maximum length of tubing in the rig would be 8 ft. corresponding to a fluid volume of 0.16 gal. The fluid required to draw 10 500 mL samples is ~1.3 gal. The sum of these, ~ 1.7 gal, gives the total amount of fluid lost from the reservoir during general filter rig operation and sampling.

In order to prevent fluid aeration in the reservoir, it is necessary to ensure that the return line is submerged and/or that the reservoir is large enough to allow any air bubbles to rise to the surface. The volume of the fluid remaining in the reservoir after allowing for operation and sampling must then result in a fluid depth sufficient to submerge the fluid return line (Figure 2-9). While we have some control over the height of the return line (via design), the fluid height is largely a function of reservoir geometry. We selected a 2.125-gallon capacity cylindrical reservoir with an internal diameter of 8 in (Figure 2-10) which, when operating at 2 gallons, results in a maximum attainable fluid height of 6 in. The original return height of the selected reservoir was 16 in., through-bore tube fittings enabled plumbing the return line to a height of 5 in (Figure 2-10). With the maximum volume established, the desired flow rate of one fluid pass per minute becomes two gallons per minute.

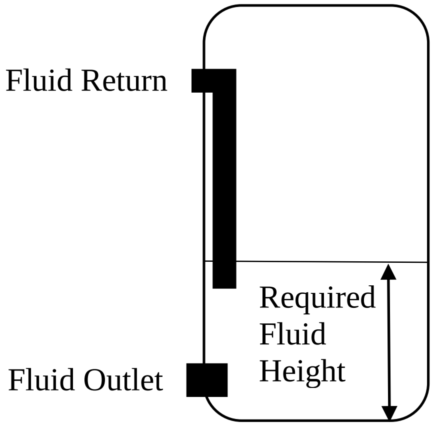


Figure 2-9: A minimum fluid height must be maintained to ensure that the return line remains submerged during operation; failure to maintain oil return submersion may result in fluid aeration.

Figure 2-10: 2.125 gal. capacity oil reservoir with 6 in internal diameter provides sufficient capacity to extract ten 500 mL samples (left). The return line was plumbed to ensure return flow remains submerged (right)

The reservoir is equipped with two threaded fittings that will allow the installation of temperature monitoring and control devices. The in-line particle counter currently monitors the temperature.

2.4.2.2 Motor, Pump, Motor Controller

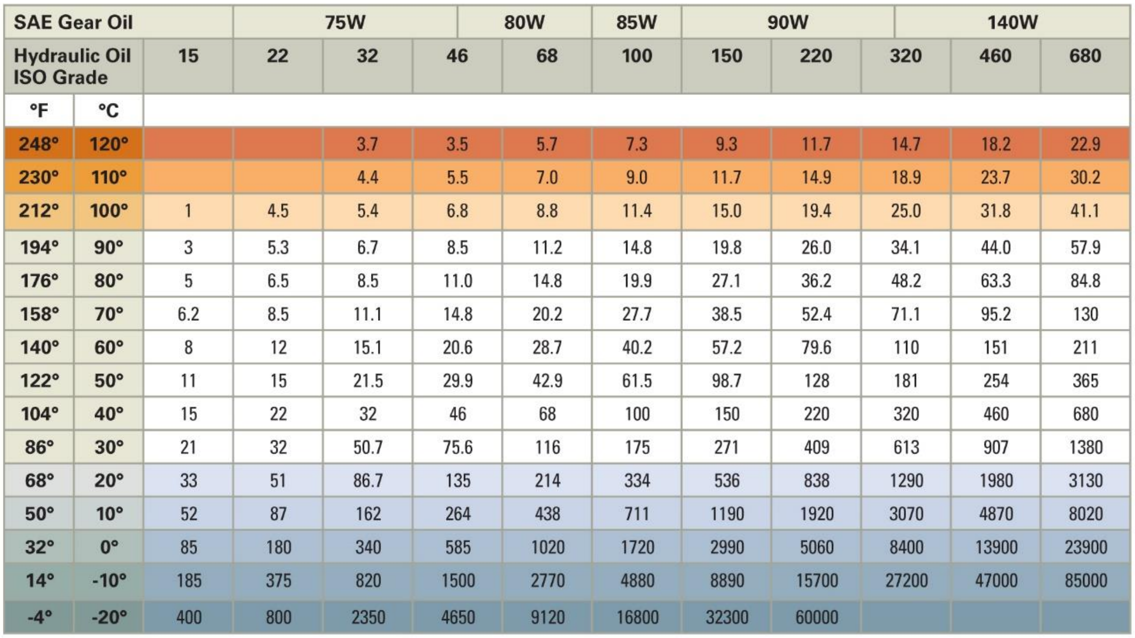
The required filters, inline particle counter, and flow rate place restrictions on many of the system's remaining design choices. The particle counter should be placed just before the return lines, discharge directly into the reservoir [43], and requires a minimum inlet pressure of 20 psi [43]. An adjustable backpressure regulator is included in the system to maintain adequate pressure for the particle counter (Figure 2-11) [45]; however, the filter will cause the majority of pressure loss in the system.

Figure 2-11: An adjustable back pressure regulator maintains pressures required for particle counter operation [45]

Donaldson provides pressure loss vs flow charts to determine the expected pressure drop for its filter housings and filters. The charts are based on an ISO 32 fluid with 32 cSt viscosity, specific gravity of 0.9, and temperature of 150 °F [46]. After finding the expected pressure drop (ΔP), the actual expected pressure drop for non-standard operating fluids or conditions is given by:

$$\Delta P_{actual} = \Delta P_{graph}\left[\frac{Actual\ Viscosity\ (cSt)}{32\ (cSt)}\right]\left[\frac{Actual\ Specific\ Gravity}{.9}\right] \qquad (2\text{-}1)$$

While the temperature of the fluid does not appear in the correction factor, it is accounted for when calculating the actual fluid viscosity. For example, ISO 32 hydraulic operating at 40 °C has a viscosity of 32 cSt, however if the fluid will be operating at 20 °C the viscosity can be expected to have increased to 86.7 cSt (Figure 2-12). At a flow rate of 2 gpm the graph pressure loss for the filter (Figure 2-13) and housing (Figure 2-14) are expected to be <1 psi and 5 psi respectively. Considering the lowest expected operational temperature (20 °C) and highest expected fluid grade (ISO 320) we find an expected maximum viscosity of 1290 cSt. Assuming a maximum specific gravity that is negligibly different from unity, we find an overall pressure drop across the housing of ~270 psi. However, the HPK02 filter housing indicates a filter collapse pressure of 150 psi. In order to accommodate this requirement and include a 1.25 factor of safety against filter collapse, we find that the maximum acceptable viscosity at 2 gpm to be ~570 cSt.



SAE Gear Oil			75W		80W		85W	90W		140W		
Hydraulic Oil ISO Grade		15	22	32	46	68	100	150	220	320	460	680
°F	°C											
248°	120°			3.7	3.5	5.7	7.3	9.3	11.7	14.7	18.2	22.9
230°	110°			4.4	5.5	7.0	9.0	11.7	14.9	18.9	23.7	30.2
212°	100°	1	4.5	5.4	6.8	8.8	11.4	15.0	19.4	25.0	31.8	41.1
194°	90°	3	5.3	6.7	8.5	11.2	14.8	19.8	26.0	34.1	44.0	57.9
176°	80°	5	6.5	8.5	11.0	14.8	19.9	27.1	36.2	48.2	63.3	84.8
158°	70°	6.2	8.5	11.1	14.8	20.2	27.7	38.5	52.4	71.1	95.2	130
140°	60°	8	12	15.1	20.6	28.7	40.2	57.2	79.6	110	151	211
122°	50°	11	15	21.5	29.9	42.9	61.5	98.7	128	181	254	365
104°	40°	15	22	32	46	68	100	150	220	320	460	680
86°	30°	21	32	50.7	75.6	116	175	271	409	613	907	1380
68°	20°	33	51	86.7	135	214	334	536	838	1290	1980	3130
50°	10°	52	87	162	264	438	711	1190	1920	3070	4870	8020
32°	0°	85	180	340	585	1020	1720	2990	5060	8400	13900	23900
14°	-10°	185	375	820	1500	2770	4880	8890	15700	27200	47000	85000
-4°	-20°	400	800	2350	4650	9120	16800	32300	60000			

Figure 2-12: Kinematic viscosity, ISO Grade, and SAE Weight vs Temperature [46].

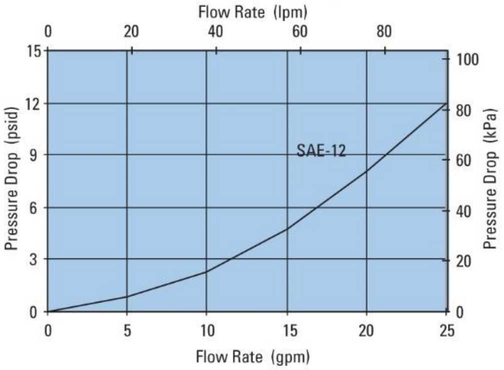


Figure 2-13: Pressure drop vs Flow rate for Donaldson HPK02 filter housing with SAE-12 fittings at the inlet and outlet [41].

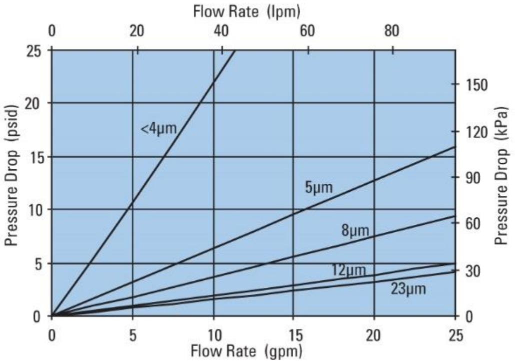


Figure 2-14: Pressure drop vs Flow rate for various pore size DT Synteq filters [41].

The remaining system pressure drop was then calculated utilizing the equivalent length method [47] and a very preliminary sketch of the hydraulic circuit. The maximum minor and major expected losses were calculated to be ~120 psi with a factor of safety of 1.5 to account for component changes as the design progressed. The sum of the major and minor losses, the filter and housing losses, and minimum pressure at the particle counter results in an expected pump head pressure of <300 psi.

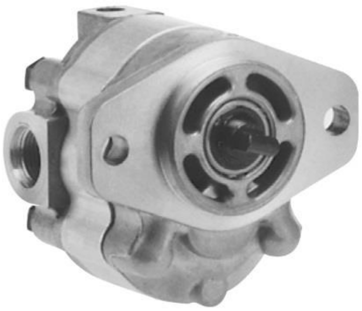


Figure 2-15: Parker D09 SAE-AA flanged gear pump has a displacement of 0.21 in3/rev, maximum operating speed of 3600 RPM, and is rated for fluids ranging from ~ 15cSt to 250 cSt [48].

After determining the expected pressure and flow rate, the pump head and motor were selected. We obtained a positive displacement hydraulic gear pump [48]. The pump displaces 0.21 in^3 fluid per revolution and has a maximum operating speed of 3600 rpm. This results in a nominal flow rate of 3.63 gpm. The pump is compatible with fluid viscosities from ~ 15 cSt to 240 cSt. This range is increased by operating the pump at reduced speed, giving high viscosity fluids sufficient time to enter the pump cavities. In order to expand the pumps compatibility with viscous

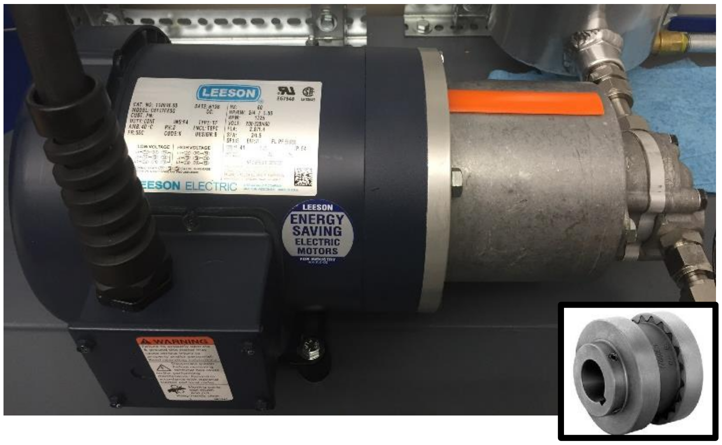


Figure 2-16: A ¾ hp 3-phase 208V motor powers a 0.21 ci/rev gear pump. The motor is coupled to the pump by a flexible coupling (insert) housed inside an aluminum bell housing. The flexible coupling allows for parallel and angular misalignments while absorbing both linear and torsional vibrations and shocks [51].

fluids, we chose the displacement such that we would obtain our desired maximum flow at approximately 60% of the pumps limiting speed. While this will allow us to pump fluids above the pumps viscosity rating, it will further limit the systems maximum viscosity.

The motor and motor control were chosen next. A flow rate of 2 gpm, maximum pump head pressure of 300 psi, and expected minimum motor efficiency of 0.75 or greater, leads to a required power input of 0.5 hp [49]. In order to operate at or near 60% of the pumps maximum speed, we choose a motor with a no load speed of 1800 RPM and reduce our maximum flow rate to 1.65 gpm. Furthermore, our desire to have full control over the motors speed made it desirable that the motor operate on three-phase AC power. We choose a ¾ hp 3-phase 208V totally enclosed fan cooled (TEFC), motor [50]. The NEMA 56C face motor is coupled to the SAE-AA flanged hydraulic pump through an aluminum bell housing and flexible coupling [51]. Finally, we employ a 3-phase variable frequency drive to control the motors operation (Figure 2-17) [52].

Figure 2-17: A GS1-20P2 variable frequency drive provides control and automation of motor speed and start-up [52].

2.4.2.3 *Electrical Components*

The selection of electrical components was relatively straightforward. The particle counter requires a 9-33V DC, 150 mA power supply [53]; the selected supply delivers 12V at up to 2.5A providing flexibility to add additional 12V devices, such as a temperature controller, as required. The power supply draws 120V 0.7A, we selected a 1A fuse block. For the motor circuit, we selected 4A slow blow fuses as recommended in the Cooper Bussman Motor Circuit Protection Tables [54]. All switches were selected to be compatible with up to 30A of current, the point at which the buildings circuit breakers offer protection.

Figure 2-18: A DC power supply provides 12V, 2.5A power to the particle counter [53].

2.4.3 Design Details

The filter station constructed for this project (Figure 2-19, Table 2-5) successfully meets the design requirements previously detailed. The flow rate is controllable from <0.1 gpm to 1.68 gpm. When fully charged with fluid, it allows collection of ten 500 mL samples; when fewer samples are required, the system will operate on as little as 0.75 gal. of fluid. It accepts fluid viscosities from 15 cSt to 250 cSt or more and can operate at temperatures from room temperature (~20 °C) to 60 °C, providing an effective fluid range of ISO 15 through 680 hydraulic oils. A model of the system (Figure 2-21) confirmed that with these viscosities, we remain within operating pressures. Pressures predicted by the model match known pressures for a given flow rate and fluid viscosity, match those experienced during station operation. Expanding the model to higher viscosities shows that pressures at the pump head and across the filter housing remain below 300 and 150 psi respectively.

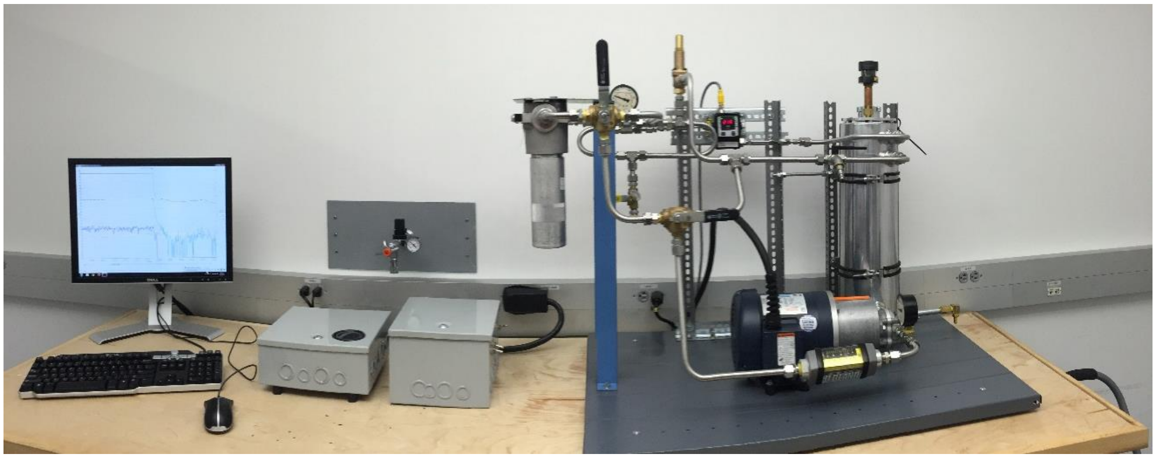


Figure 2-19: The completed filter test station operates at flow rates of <1 to 1.68 gpm, is compatible with temperatures from ~20 to 60 °C, viscosities from 15 to 250 cSt, accepts DT Synteq filters and is equipped with an ISO 11500 compliant particle counter.

Table 2-5: Filter rig schematic and list of primary components of the filter test station.

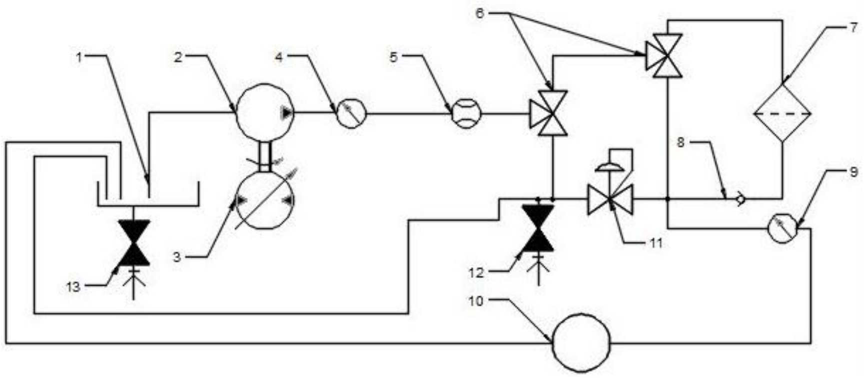
<table>
<tr><td colspan="6">

Figure 2-20: Filter rig schematic.</td></tr>
<tr><td>1</td><td>2.125 gal. reservoir</td><td>6</td><td>3-way balve valve</td><td>11</td><td>Adjustable back pressure regualtor, 0-100 psi</td></tr>
<tr><td>2</td><td>0.21 in^3/rev gear pump</td><td>7</td><td>Filter</td><td>12</td><td>Sample point</td></tr>
<tr><td>3</td><td>Variable speed motor, 40 – 1800 rpm</td><td>8</td><td>Check valve</td><td>13</td><td>Reservoir drain</td></tr>
<tr><td>4</td><td>Pressure gauge, 0-600 psi</td><td>9</td><td>Pressure gauge, 0-200 psi</td><td></td><td></td></tr>
<tr><td>5</td><td>Flow meter, 0-5 gpm</td><td>10</td><td>In-line particle counter</td><td></td><td></td></tr>
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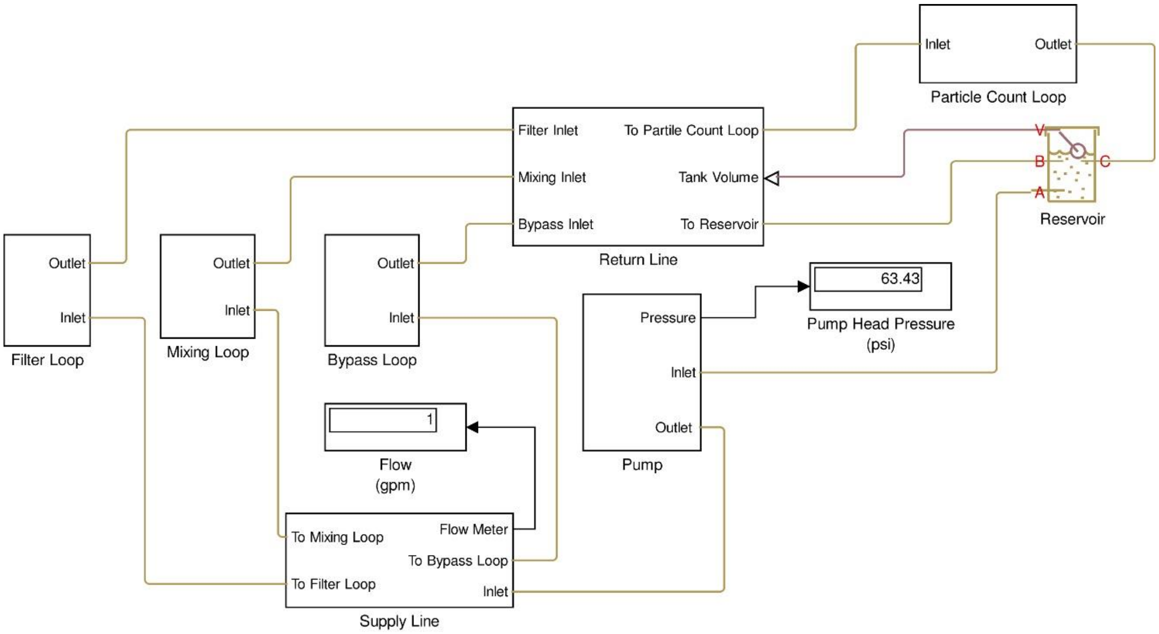


Figure 2-21: Model of the filter station utilized to ensure pump head and filter pressures remained below maximum predicted values for viscosities up to 250 cSt.

The station requires 208V 3-phase power for the motor and 120V single-phase power for the particle counter power supply (Figure 2-22). 120V building power is connected to a 1A fuse block, a single pole single throw switch, and power supply, in sequence. 12V power is then delivered to the particle counter from the power supply. 208V 3-phase power for the motor passes through a 4A 3-pole fuse block and is fed through a three-pole single throw switch and into the motor controller. The motor controller varies the output voltage and frequency of the 208V 3-phase wall power according to user input enabling control over the motor [55]. The control is initially programed by PC, via a Modbus ASCII cable. Then, a common port and three 0-5V inputs on the controller provide user control of the motor (Figure 2-24). The common port and three digital inputs are paired with three single pole single through switches to provide on/off capabilities, 3

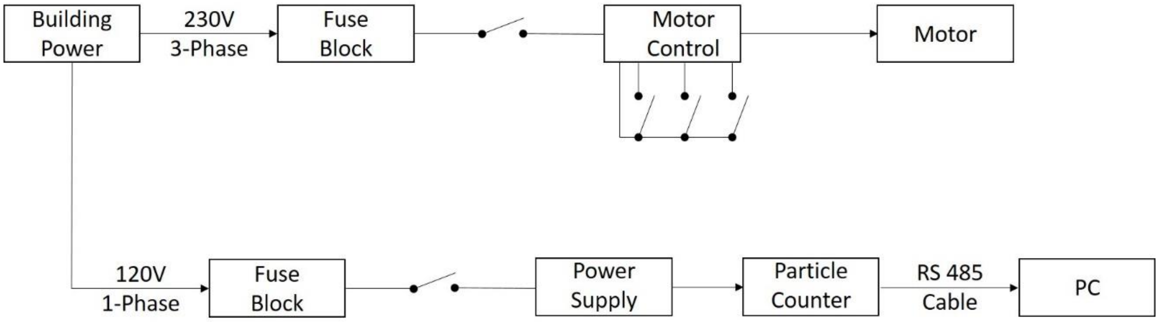


Figure 2-22: Filter test station electrical system overview.

preset motor speeds, and the ability to override the preset speeds. When the presets are overridden, the motor is controlled from the controller keypad.

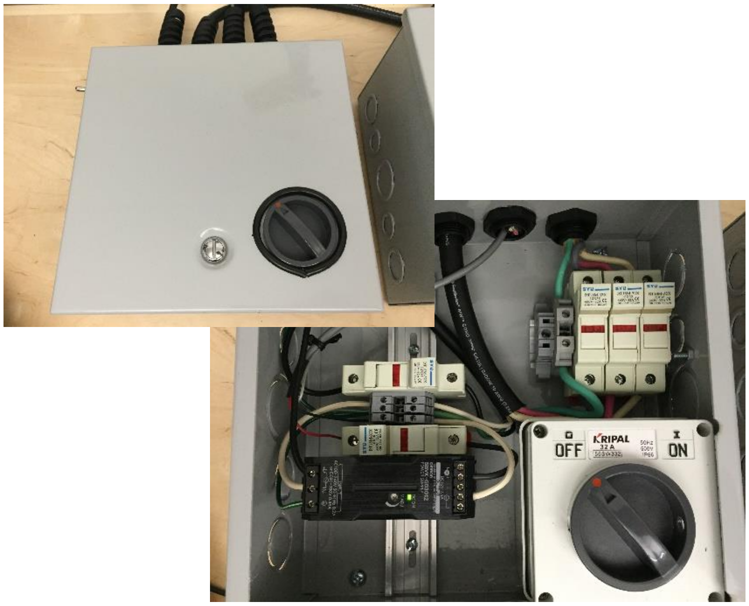


Figure 2-23: All fuse blocks, power switches, and the DC power supply are housed in a junction box.

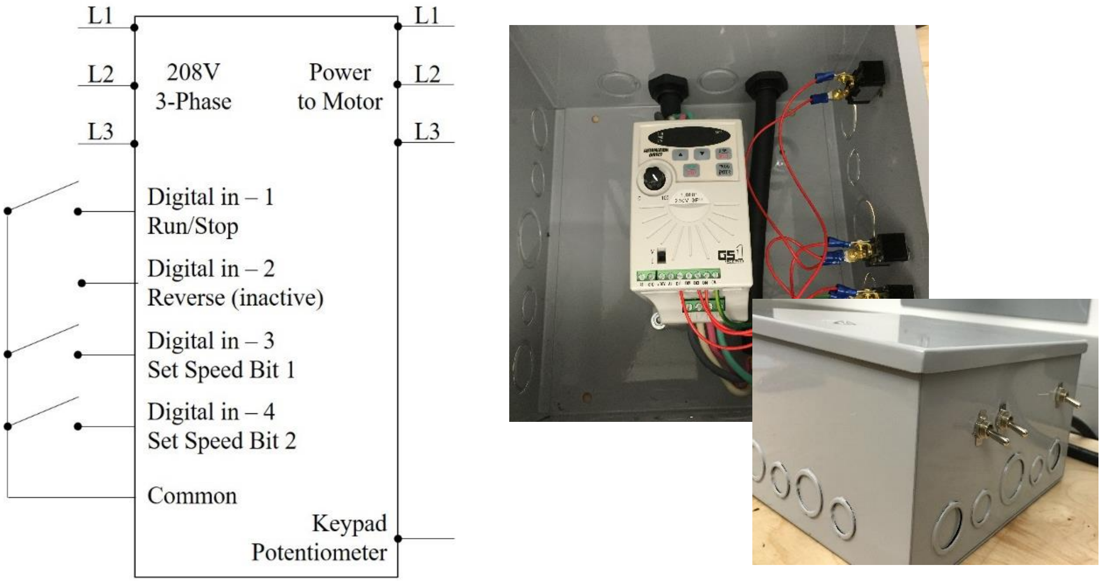


Figure 2-24: Motor control wiring schematic (left). Motor controller housing, and control switches (right).

3 Study Methodology

Our primary study seeks to enhance the understanding of the effects of FIs on ISO 4406 cleanliness, the effect of filtration on FI induced particle counts and the resulting changes in FI performance, we investigate and compare these phenomena for four additives and two filters. We test each FI by recording the increase in base oil particle count from addition of the inhibitor. We then quantify the change in particle count and foam performance over10 filter passes through a 12 μm β1000 filter. This process is repeated with a 2 μm β1000 filter. Prior to beginning this study, test station validation was required. Details for all procedures follow.

3.1 Fluid Blending

Fluids blended as described in Section 3.1.1 were used to validate the foam test station. Section 3.1.2 details the blending method for experiments involving filtration. The treatment rates detailed in Table 3-1 are considered 100% treatment rates for our study. Future work may investigate the effects of treatment rate based on this starting point (see Section 5.3.1)

Table 3-1: Inhibitors and treat rates investigated.

Inhibitor Solution	Solution Treat Rate (wt.%)	Type of Inhibitor	Percent Inhibitor in Solution	Inhibitor in Base Oil
FI-S1	0.14%	Polydimethylsiloxane	~1.1%	15 ppm
FI-A	0.025%	Polyacrylate	40%	100 ppm
FI-MO	0.05%	Organosiloxane	<10%	<50 ppm
FI-E	0.001%		100%	10 ppm

3.1.1 General Fluid Blending Process

The base oil-FI blending process employed here consist of three steps, preheating the base oil, injecting the additive, and thoroughly mixing the fluids. A container of base oil is placed on the hot plate. A mixing apparatus is inserted into the fluid; if a magnetic stir bar is chosen, it is placed in the bottom of the container. If impeller mixing is chosen, the impeller and motor is positioned such that the impeller is approximately one-third of the fluid height, from the bottom of the container. Agitation then begins; the speed is selected such that it creates a visible vortex that does not extend more than 1.5 in. below the fluid surface or reach the mixing apparatus. The temperature of the hot plate is increased and the base oil is heated to 135 º F (~57 ºC). Having achieved mixing temperature, FI is injected by pipette below the surface of the base oil as near the mixing apparatus as possible. Blending is completed by allowing the mixture to mix for 30 minutes.

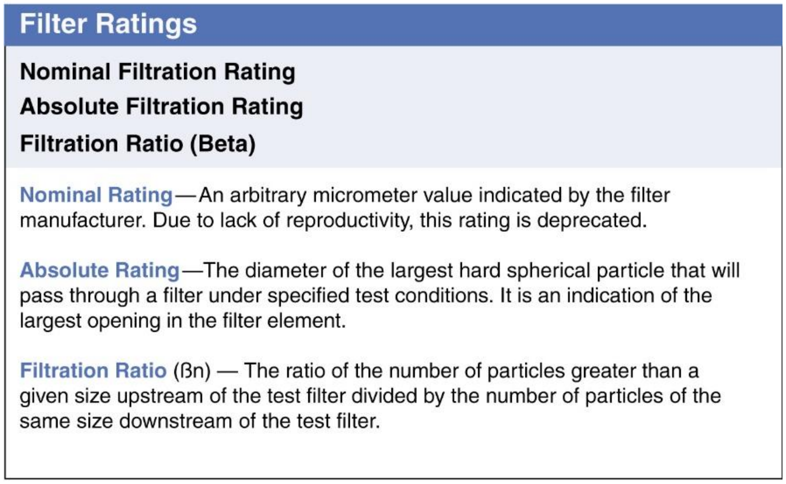
3.1.2 Blending for Filtration Testing

Our investigations are limited to filter pore sizes on various commercially available FI products, and as such, the primary metrics of interest are changes resulting from filtration. It is known that antifoam dispersion characteristics, average droplet size for example, have a strong influence on FI performance. It is further acknowledged that mixing temperature strongly influences dispersion characteristics. However, as this study seeks only to characterize changes in performance we determine that optimizing the dispersion is unnecessary and focus on blending the fluids in the most repeatable manner available.

Inhibitors are blended with the base oil by circulation through the filter station. Each additive is injected into the reservoir with the base oil circulating at one gallon per minute. The fluid is introduced below the surface by pipette at the center of the reservoir diameter. The fluids are then mixed by turbulence in the tubing, valves, and pump. Each batch is allowed to circulate until the fluid in the reservoir has circulated through the system 30 times; for example, a two-gallon batch will mix for one hour at 1 gpm. However, the filter station validation tests were allowed to mix by circulation for a least 30 min. with no maximum time constraint. Table 3-1 details the treatment rates for the tested foam inhibitors.

3.2 Filtration

3.2.1 Filters



Filter Ratings

Nominal Filtration Rating
Absolute Filtration Rating
Filtration Ratio (Beta)

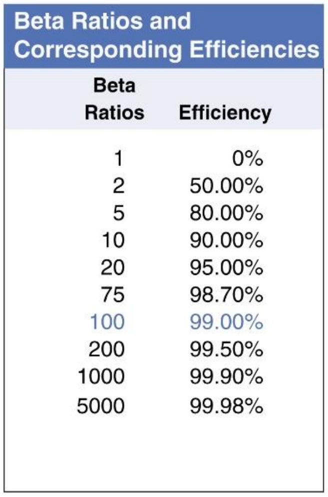
Nominal Rating—An arbitrary micrometer value indicated by the filter manufacturer. Due to lack of reproductivity, this rating is deprecated.

Absolute Rating—The diameter of the largest hard spherical particle that will pass through a filter under specified test conditions. It is an indication of the largest opening in the filter element.

Filtration Ratio (ßn) — The ratio of the number of particles greater than a given size upstream of the test filter divided by the number of particles of the same size downstream of the test filter.

Figure 3-1: Summary and explanation of filter rating methods [12].

Filters elements trap contaminants by forcing fluid flow through a porous media. Common filter media types are cellulose, wire mesh, or synthetic (glass). Filter effectiveness is rated nominally, absolutely, or with a beta rating (Figure 3-1). Nominal ratings are assigned by manufactures with no standard for reproducibility; cellulose and wire mesh filter elements are typically rated nominally at the particle size for which they achieve 50% efficiency [56]. Absolute ratings are more systematic in nature and represent the largest particle that will pass through the filter, under test conditions [12]. ISO 4572 standardizes beta ratings and describe the ratio of upstream to downstream particles at a given size [12], [56], [57]; a filter that removes 99.00% of particles ≥ 12 μm, for example, is described as a 12 μm β100 filter. Figure 3-2 lists common beta ratings and their associated efficiencies. Synthetic filters are typically rated either absolutely or with a beta rating. Finally, filters are classified as low or high-collapse. Low collapse filters typically have crush ratings of 450 psi or less while high-collapse filters are commonly rated for up to 3000 psi [58]. Three filter elements were selected for this study. All are 8 in. synthetic media, low-collapse, β1000 filters. The filters selected and utilized for all studies here are Donaldson DT Synteq filters with 12, 5 and 2 μm β1000 ratings [59] (Table 3-2).



Beta Ratios and Corresponding Efficiencies

Beta Ratios	Efficiency
1	0%
2	50.00%
5	80.00%
10	90.00%
20	95.00%
75	98.70%
100	99.00%
200	99.50%
1000	99.90%
5000	99.98%

Figure 3-2: Common beta ratios and corresponding efficiencies [12].

Table 3-2: Filters used to study the effects of filtration on FI induced particle counts and FI performance.

Pore Size	Beta	Donaldson Part #
12 μm	1000	P566202 [60]
5 μm	1000	P566200 [61]
2 μm	1000	P566199 [62]

3.2.2 Test Procedure

A standardized procedure was established for conducting filtration, mixing, and filtering tests. The reservoir is charged with 2 gallons of base oil. The base oil is allowed to circulate until all air is removed from the system. The flow rate is then set to 1 gpm and the particle counter is activated; following particle counter activation, raw particle counts and ISO 4406 codes are recorded at 10 s intervals. If, at this time, the particle count is not 17/--/-- or better, the base oil is filtered until reaching 17/--/-- or better. With the flow rate at 1 gpm, the particle counter active, and a base cleanliness reached, FI is injected into the reservoir and allowed to mix for one hour. After one hour of mixing, a 500 mL sample of fluid is drawn and filtration is initiated. Further 500 mL samples are then drawn after 2.5, 5, 7.5, and 10 filter passes.

3.2.2.1 Sampling procedure

Sampling procedures were established in order to ensure consistency and minimize the possibility of contamination. When collecting a sample, a small amount (~100 mL) of fluid is collected from the system in a sample bottle. The bottle is capped and shaken; the small amount of fluid is discarded and 500 mL of fluid are collected in the prepared bottle. Each sample is labeled with the date, base oil, additive, filter size, and sample number.

3.2.2.2 Flushing Procedure

At the conclusion of each test, the system is thoroughly drained, the filter is removed, and the system is charged with 2 gal of base oil. The oil is then circulated through the system at 1.6 gpm (1800 rpm) for a minimum of one hour to flush the system. Following the fluid circulation, the system is thoroughly drained, and a new filter is installed.

3.2.3 Particle Counts

Raw particle counts and the corresponding ISO 4406 codes (Section 1.2.3) are obtained at ~10 s intervals during the testing process. At point (a) in Figure 3-3, FI is injected into the fluid reservoir. Filtration begins at point (b). At the conclusion of the test, particle counts are examined to determine the magnitude of ISO code increase following FI addition (Magnitude A, Figure 3-3), change in ISO code between 0 and 10 filter passes (Magnitude B, Figure 3-3), and number of filter passes to reach an ISO cleanliness goal of 18/16/13 ((d), the difference between points c and b, Figure 3-3) . In each case, ISO codes and particle counts are based on seven point averages.

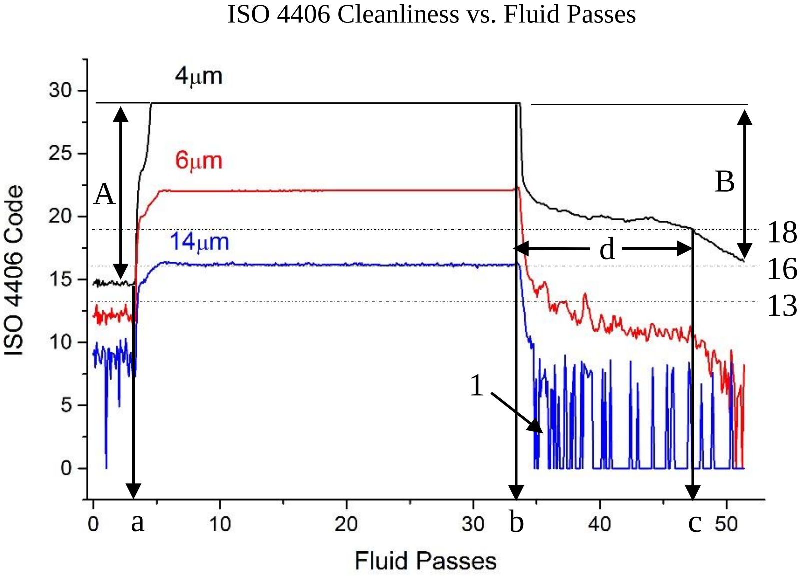


Figure 3-3: Representative ISO Code vs. Fluid Pass plot. FI is introduced to the fluid reservoir at (a). (A) illustrates the magnitude of the increase in ISO code following FI introduction. Filtration begins at (b), and at (c) the ISO cleanliness code has reached 18/16/13 or better. (d) is the number of passes required to reach our cleanliness goal. (B) represents the decrease in ISO code resulting from filtration. ISO Codes resulting from counts of less than 20 particles are statistically unreliable [18], and manifest as noise (1).

If ISO codes are based on a raw counts of less than 20 particles, the statistical significance of the code is diminished. ISO 4406 notes that this is especially problematic below ISO 8 [18]. The particle counter employed here internally regulates flow at 50 to 500 mL/min [44]. With a sample time of ~10 s, we have a resulting fluid volume of 8.3 to 83 mL per count. A count of 20 particles would result in a cleanliness of .24 to 2.4 particles/mL, ISO 4406 codes of 5 and 8 respectively. This leads to the expectation that codes reported by the particle counter maintain statistical reliability at ISO 9 or above. However, usage has shown that there is significant noise in the data once the ISO Code falls below 10 (Figure 3-3, C). We thus report all codes of 9 or smaller as <10.

3.3 Foam Characterizations

All foam characterizations are conducted in accordance with ASTM D892 utilizing the foam test station detailed in Section 2.3. Each trial is conducted in triplicate. The mean value of the three trials and the standard error associated with them are then reported. An example is shown in Figure 3-5. The reproducibility of each set of tests is evaluated by plotting the trial average against the maximum variation in individual results and comparing them to the reproducibility guidelines in ASTM D892 [31]. Figure 3-4 provides an example of a reproducibility plot. Details for the three test sequences described in ASTM D892 follow.

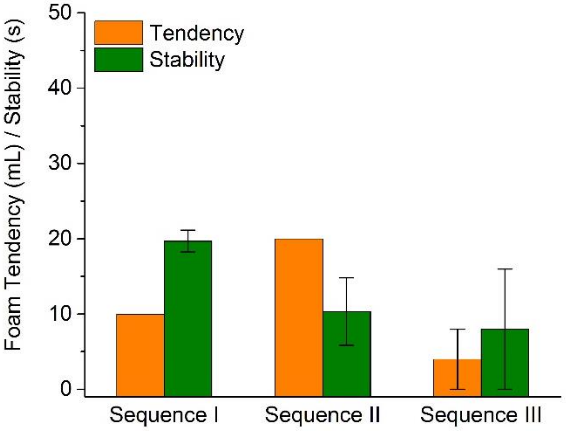


Figure 3-5: Foam tendency in (mL) and Stability in (s) by ASTM D892 Sequence for Chevron 220R base oil with 0.14% FI-S1 foam inhibitor. The volume of foam resulting from 5 min of airation is reported as foam tendency. Stability is represented here, by the time required for the samples to reach 0 foam following five minutes of airation. Error bars are representative of the standard error for three measurements made by different individuals.

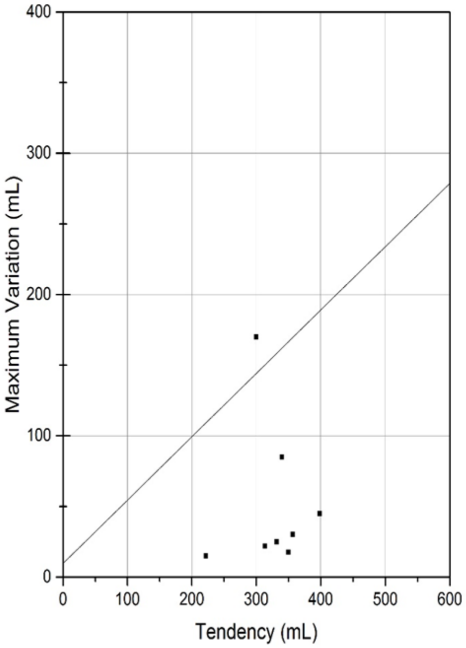


Figure 3-4: Reproducibility plot for eight series of three foam characterizations. The points are the difference between the maximum and minimum measurements obtained by three people for the same test material is plotted against the mean of the three results. The diagonal line on the lots represents the maximum expected variation as determined by ASTM D892. Results that fall above the diagonal line on the plot are outside of reproducibility guidelines.

3.3.1 ASTM D892 Sequences I-III Procedures

3.3.1.1 Sequence I

A 200 mL sample of fluid is decanted, heated to 49 ± 3 °C, and allowed to cool to 24 ± 3 °C, without mechanical stirring or shaking [31]. 190 ± 5 mL of the sample is then transferred to the 1000 mL test cylinder. The cylinder is immersed in the 24 ± 0.5 °C bath to at least the 900 mL mark. Once the oil has reached the bath temperature, the diffusing stone and air delivery tube, with air source disconnected, are placed in the cylinder. The diffusing stone is allowed to soak for five minutes. The air source is then connected to the air delivery tube; the flow rate of the air source is adjusted to 94 ± 5 mL/min. Clean dry air is forced through the diffuser for 5 min ± 3 s, as timed from the first appearance of bubbles rising from the diffuser's surface. After passing air through the stone for 5 min ± 3 s, the air source is disconnected and the volume of foam between the surface of the oil and the top of the foam is recorded as foaming tendency. The cylinder is allowed to rest for 10 min ± 10 s and the volume, in mL, of any remaining foam is recorded as foam stability. If the volume of foam reaches zero before 10 min. have elapsed, the time required for the foam to dissipate is recorded in seconds. The total volume of the air passed through the system should be 470 ± 25 mL.

3.3.1.2 Sequence II

A separate 180 ± 5 mL sample of fluid is decanted into a clean 1000 mL test cylinder. The cylinder is immersed in the 93.5 ± 0.5 °C bath to at least the 900 mL mark and allowed to come to temperature. A clean diffusing stone and air delivery tube, with air source disconnected, are placed in the cylinder. The diffusing stone is allowed to soak for five minutes and the air source is then connected to the air delivery tube. The flow rate of the air source is adjusted to 94 ± 5 mL/min and clean dry air is again forced through the diffuser for 5 min ± 3 s, as timed from the first appearance of bubbles rising from the diffuser's surface. After passing air through the stone for 5 min ± 3 s, the air source is disconnected and the volume of foam between the surface of the oil and the top of the foam is recorded as foaming tendency. The cylinder is allowed to rest for 10 min ± 10 s and the volume, in mL, of any remaining foam is recorded as foam stability. The total volume of the air passed through the system should be 470 ± 25 mL.

3.3.1.3 Sequence III

The Sequence II sample is retested for Sequence II. Any foam remaining in the cylinder following Sequence II is collapsed and the air delivery tube and diffusing stone are removed. The sample and cylinder are then allowed to cool at room temperature until reaching a temperature below 43.5 °C. The cylinder is then immersed in the 24 ± 0.5 °C bath to at least the 900 mL mark and allowed to continue cooling until reaching the bath temperature. A clean diffusing stone and air delivery tube, with air source disconnected, are placed in the cylinder. The diffusing stone is allowed to soak for five minutes and the air source is connected to the air delivery tube. The flow rate of the air source is adjusted to 94 ± 5 mL/min and clean dry air is again forced through the diffuser for 5 min ± 3 s, as timed from the first appearance of bubbles rising from the diffuser's surface. After passing air through the stone, the air source is disconnected and the volume of foam between the surface of the oil and the top of the foam is recorded as foaming tendency. The cylinder is allowed to rest for 10 min ± 10 s and the volume, in mL, of any remaining foam is recorded as foam stability. The total volume of the air passed through the system should be 470 ± 25 mL.

3.3.1.4 Cleaning Methods

As discussed in Section 1.3.2, foam characterizations are sensitive to contamination from cleaning processes. In order to minimize the possibility of contamination from cleaning procedures, the cleaning process was standardized to the cleaning process described in ASTM D892 Helpful Hints X1.1.7.1 [31]. Glassware is cleaned in sequence with heptane, detergent, water, and acetone. It is then dried with clean, dry, pressurized air. The diffusing stones are cleaned by flushing 5 times in sequence with heptane and acetone in turn. They are then dried with clean dry pressurized air.

3.3.2 Representative Results 0-10, Filter Passes

ASTM D892 Sequence I is employed to characterize foaming tendency for the experiments investigating the effects of FI addition and filtration over 10 filter passes. Results from the Sequence I characterizations are then compared to Caterpillar Inc. TO-4 fluid foaming specifications [63]. Under TO-4 specifications, Sequence I tendency and stability must not exceed 25 mL and 0 mL of foam respectively. Because all ASTM D892 stability volumes recorded during these experiments are negligible or nil, foam stability is characterized by time to reach zero foam, in seconds and a foam tendency of < 25 mL indicates TO-4 compliance.

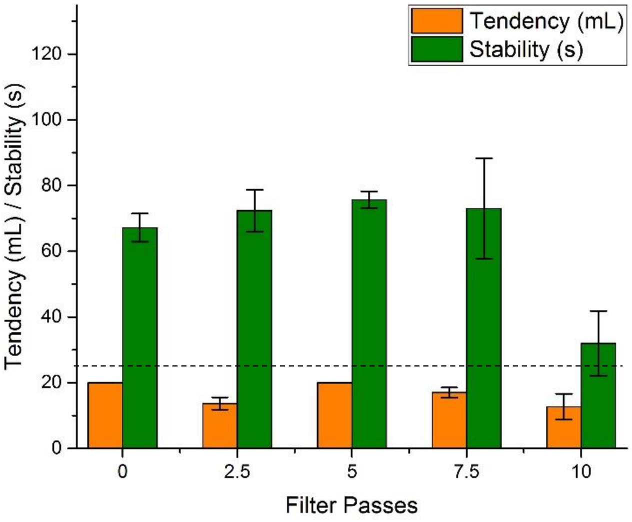


Figure 3-6: Representative plot of foam characterizations results for 0 – 10 filter passes. Sequence I test measurements for 220R w/ 0.14 wt. % FI-S1 filtered with a 12 micron β1000 filter. All stability volumes negligible or nil; stability reported here as time to reach zero foam. Dashed line represents 25 mL of tendency volume and, since all recorded stability volumes are 0, the remaining metric for TO-4 compliance.

4 Results and Discussion

4.1 Design Validation

4.1.1 Filter Station

Prior to beginning our new work, the filter station was validated by reproducing previously published results. Specifically, trials were conducted for FI-S1 (polydimethylsiloxane) and FI-A (polyacrylate) with a 12 μm β1000 filter. Results from these trials were compared to published results From Michael et al. for polydimethylsiloxane (PDMS) and polyacrylate [13].

Figure 4-1 contains the 4, 6, and 14 μm ISO codes as a function of time for 2 gallons of 220R. FI-S1, 0.14%, was introduced to the reservoir and allowed to mix. The mixture was then filtered. Figure 4-2 is two previously published plots of 4, 6, and 14 μm particle counts as a function of time, for Group III (left) and Group I (right) base oils with a Diesel engine oil additive package that contains a PDMS foam inhibitor. The inhibitor was added to circulating base oil, allowed to circulate, and was then filtered. Qualitatively, we find that plots of the ISO 4406 codes for FI-S1 agree in shape with the plots published by Michael et al.

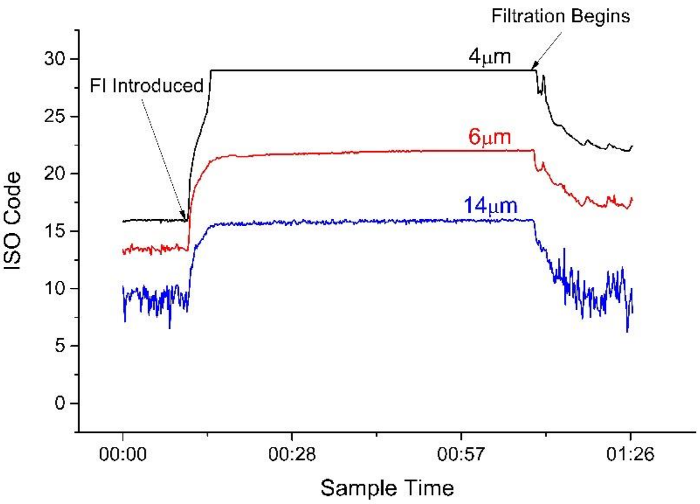


Figure 4-1: Cleanliness vs. time profile for 0.14 wt.% FI-S1 in 220R base oil. The sharp increase and decrease in particle counts following FI addition and filtration respectively match qualitatively with previously published contamination-time profiles for a silicone inhibitor containing additive package (Figure 4-2)

Table 4-1 summarizes our findings and previously published results for changes in ISO codes resulting from polyacrylate and PDMS inhibitors added to base oils. We find that the results obtained during our validation trials are consistent with those obtained by Michael et al. A quantitative comparison of the results shows that the increase in particle count magnitudes, with exception of the 6μm counts, match to within one ISO code. We find this to be an acceptable variance, given experimental differences, i.e. particle counters and additive formulators. The post filtration numbers do not agree; however, we believe this is explained by filtration parameters. Michael et al. employed a 3 μm β200 filter, while we employed a

12μm β1000 filter. Additionally, variations in filter media are known to effect the rate of inhibitor removal [28].

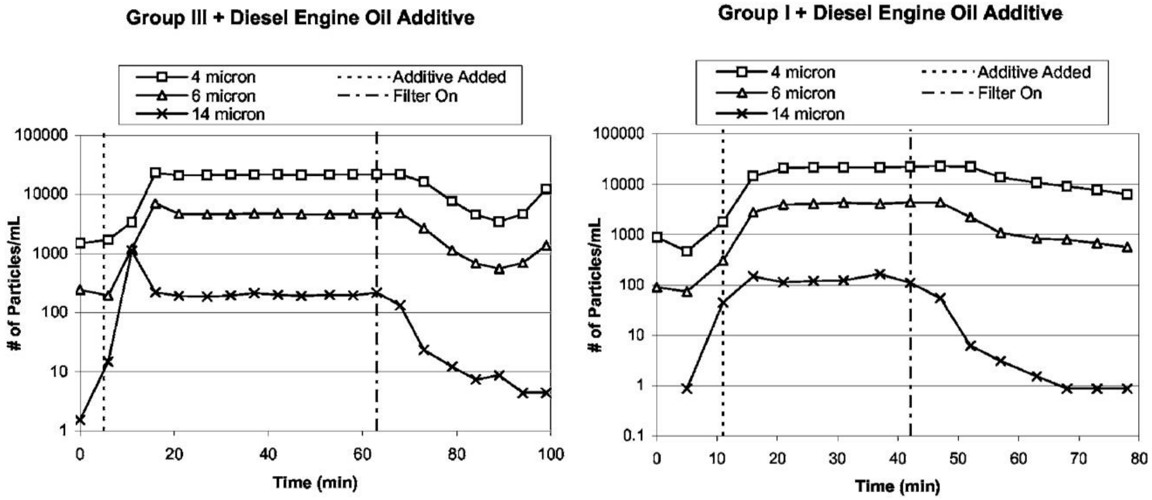


Figure 4-2: Contamination vs. time profiles for diesel engine oil additive package that includes a silicone antifoam in Group II and III base oils [13]. The sharp rise in particle counts and post filtration decrease match qualitatively with results obtained during filter station validation experiments (Figure 4-1).

Table 4-1: Particle count changes during filter station validation experiments as compared to previously published results (Michael et al., 2007)

Additive	Average Value, Michael et al. 2007 [13]	
	Change in ISO Code 15 Passes after FI addition	Change in ISO Code After 30 Filtration Passes
Polyacrylate	3/1/4	1/1/1
Polydimethylsiloxane	14/11/12	0/-3/-7
Additive	Results from Validation Experiments	
FI-A (polyacrylate)	2/5/5	-1/-3/-10
FI-S1 (polydimethylsiloxane)	13/9/12	-5/-3/-11

4.1.2 Foam Station

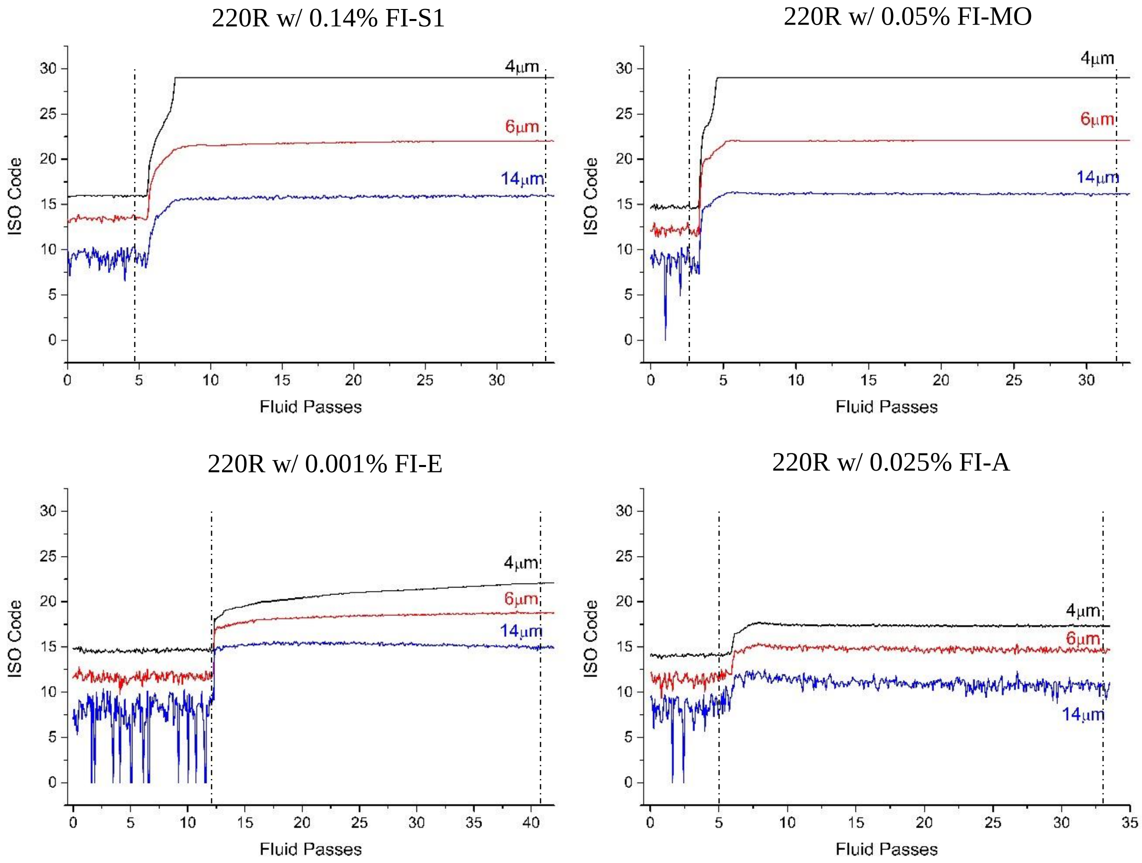
As there are no accepted reference materials, the foam test station was validated by: 1) Ensuring that each component meets the requirements detailed in ASTM D892 and 2) Showing a decrease in foaming performance for filtered fluids. Each component utilized in the testing process complies with ASTM D892. We have encountered some difficulty consistently meeting reproducibility guidelines, and obtaining reliable foam test results remains one of our largest challenges. However, we were successfully able to measure a decrease in foam performance following fluid filtration (see Section 4.6).

4.2 Particle Counts Resulting from FI Introduction

In this section, and the next two, we investigate the particle counts resulting from FI inclusion on fluid cleanliness and the subsequent ability of filtration to remove the induced particle counts.

Figure 4-3 contains four representative plots from tests to determine the effect of FIs on fluid cleanliness. The plots show ISO 4406 codes for each particle size as a function of time. In each case, an inhibitor was added to 2 gallons of circulating oil and allowed to mix for 30 passes. The introduction of foam inhibitors to circulating base oil results in a dramatic increase in particle counts for all investigated additives. For both silicone based FIs, FI-S1 and FI-MO, the 4 μm particle counts following FI addition exceeded 2.5 million particles/ mL, the maximum detectable by the HIAC ROC inline particle counter, resulting in the highest ISO code prescribed by ISO 4406, >28. Figure 4-4 shows the ISO codes for the fluids before the addition of the FIs and after mixing. The difference in the height of these bars is the magnitude of the change in ISO codes resulting from inclusion of the inhibitor, and is displayed in Figure 4-5. We note that the change in ISO code is most pronounced for the silicone based inhibitors, FI-S1 and FI-MO, followed by FI-E and FI-A and that this trend holds across particle sizes. Additionally, for each additive, the increase in counts is greater for smaller particle sizes.

Table 4-2 summarizes the ISO codes for the fluids before and after adding foam inhibitors. While the increase in counts resulting from the FI-E is not as extreme as the silicon inhibitors, it does result in the fluid failing to meet cleanliness standards of 18/16/13. Although all fluids successfully met the cleanliness standard before FI addition, only the FI-A blend continued to meet specifications afterwards.



220R w/ 0.14% FI-S1

220R w/ 0.05% FI-MO

220R w/ 0.001% FI-E

220R w/ 0.025% FI-A

Figure 4-3: Representative fluid cleanliness profiles following the Introduction of foam inhibitors to circulating base oil. FI is introduced to the reservoir at the first dashed line. At the second line, the FI and Base oil have mixed by circulation for 30 fluid passes.

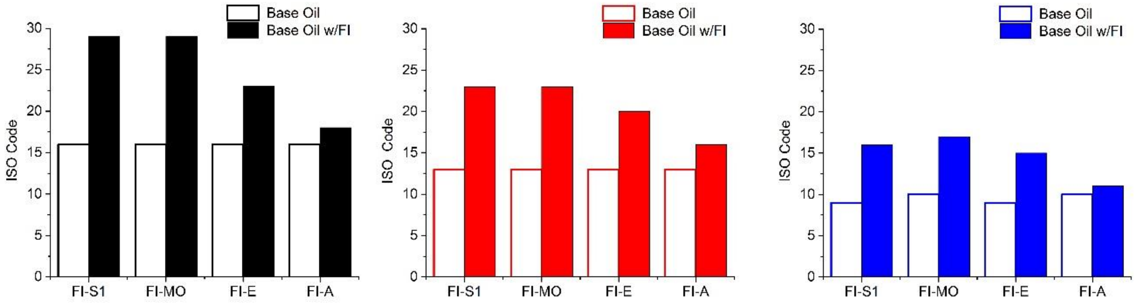


Figure 4-4: 4 μm (left) 6 μm (center) 14 μm (right) ISO codes for 220R base oil before and after FI introduction. The addition of FI to the base oil results in a large increase in particle counts as measured by a HIAC ROC inline particle counter. This effect is most pronounced for the silicone based inhibitors. This trend hold across all ISO 4406 size classifications.

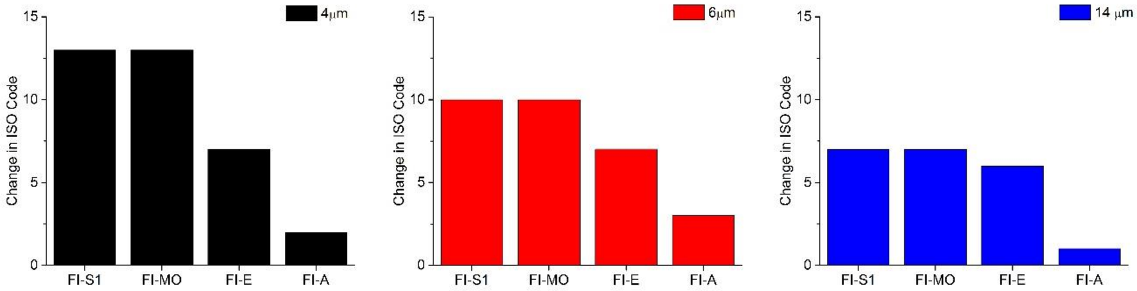


Figure 4-5: Increase in ISO code following FI introduction by particle size classification. The largest increase in particle counts resulted from the addition if the silicone based inhibitors, FI-MO and FI-S1. FI-E caused the next largest increase in particle count with FI-A resulting in the least reduction in fluid cleanliness rating. This trend holds for all three particle size classifications, 4 μm (left), 6 μm (center), and 14 μm (right).

Table 4-2: Fluid cleanliness for Chevron 220R before and after introducing foam inhibitors.

Additive (Treat Rate)	Base Oil ISO Code Before FI Introduction	ISO Code 30 Fluid Passes After FI Addition
FI-S1 (0.14%)	16/13/<10	>28/23/16
FI-MO (0.05%)	16/13/10	>28/23/17
FI-E (0.001%)	16/13/<10	23/20/15
FI-A (0.025%)	16/13/10	18/16/11

4.3 Effects of Extended Filtration on Fluid Cleanliness

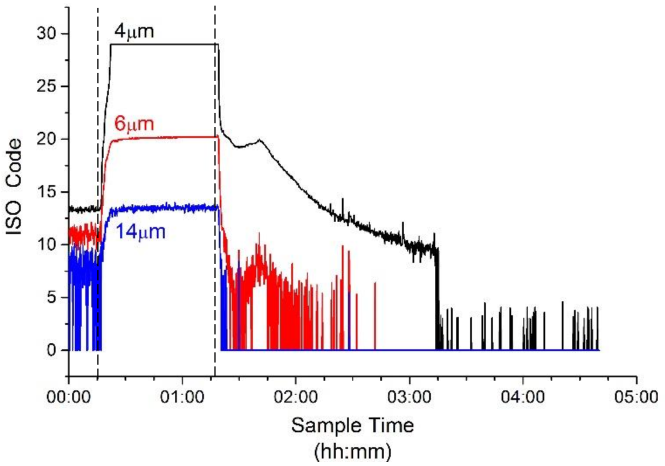


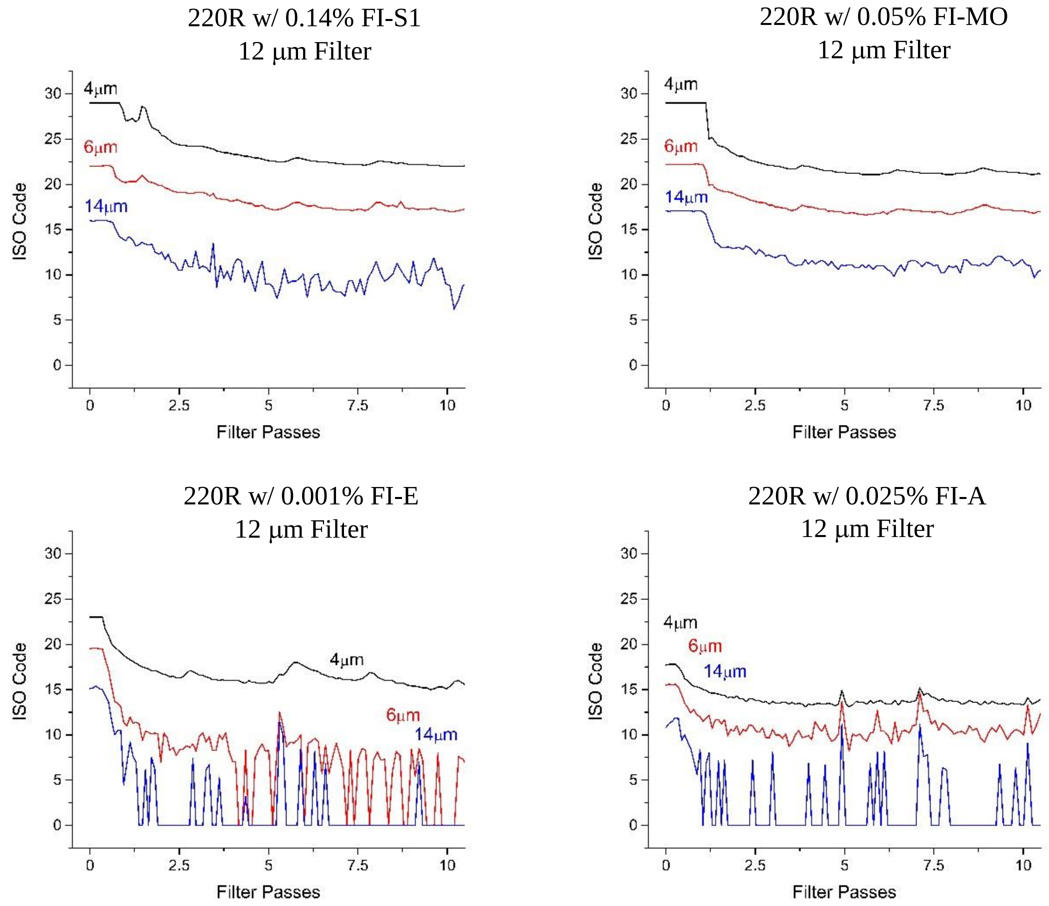
Figure 4-6: Fluid cleanliness profile for the addition and filtration of 0.14% FI-S1 in 220R. FI-S1 was added to the reservoir at the first dashed line (00:16). Filtration was initiated at the second line (01:16) and allowed to continue until the particle counts were insignificant, (04:40).

Figure 4-6 is the fluid cleanliness profile for one of the experiment conducted to ensure all FI induced particle counts could be eliminated by filtration. For this trial, 2 gallons of 220R was added to the reservoir and allowed to circulate at 1 gpm. At 00:16 0.14% FI-S1 was injected into the reservoir. The fluids were allowed to mix for 1 hour and, at 01:16, filtration was initiated. The particle counts drop rapidly. After 30 minutes of filtration, the fluid has reached the target cleanliness level of 18/16/13. After one hour of filtration, the 6 and 14 μm particle counts are detected as 0. After 1 hour 45 minutes of filtration, the 4 μm particle counts are negligible. These types of trials indicate that desired cleanliness levels can be achieved with persistent filtration. However, this would add significant time to fluid production and it has been shown that extended filtration results in poor foam performance and an inability to meet foaming standards [28].

4.4 Effect of Filtration on Fluid Cleanliness (0 – 10 Filter Passes)

It is common industry practice to filter production fluids to meet cleanliness standards. As it is known that filtration decreases lubricant performance [28], production filtration cycles are generally limited to 10 or less filter passes. Here we investigate the likely effects of this practice on fluid particle counts.

We again charged the reservoir with 2 gallons of base oil and introduced FIs as previously described. The fluid was then filtered. Particle counts and ISO codes were recorded at ~10s intervals throughout the test. Representative plots of fluid cleanliness vs. time profiles are presented in Figure 4-7; ISO codes for fluids undergoing filtration are plotted against filter passes. Filtration of the 220R with foam inhibitors results in a significant decrease in particle counts. Additionally, from these plots we see that the majority of the decrease occurs during the first 2.5 filter passes. The plots presented here resulted from experiments with 12-micron filters; 2-micron filtration plots exhibit the same trends. Results for each filter size and a comparison of those results follow in Sections 4.4.1 through 4.4.3



220R w/ 0.14% FI-S1
12 μm Filter

220R w/ 0.05% FI-MO
12 μm Filter

220R w/ 0.001% FI-E
12 μm Filter

220R w/ 0.025% FI-A
12 μm Filter

Figure 4-7: Representative fluid cleanliness profiles for FI blends filtered, for 10 passes, with a 12 μm β1000 filter at 1 gpm. Filtration begins at 0 filter passes and continues throughout the plots.

4.4.1 12 μm Filtration Results

As discussed in Section 4.2, FIs were introduced to circulating base oil and allowed to mix. After mixing, the fluids were filtered for 10 passes, with a 12 μm β1000 synthetic media filter. Figure 4-8 shows the ISO codes for the fluids before and after filtration. The difference in the height of these bars is the magnitude of the change in ISO codes resulting from filtration of the fluids, and is displayed in Figure 4-9. From these plots, we see that filtration results in a significant decrease in FI induced particle counts. Table 4-3 summarizes the ISO codes of the fluids before and following filtration. We see in Figure 4-8 and Table 4-3 that FI-E responded the most strongly to filtration, dropping below ISO 10 for both 6 and 14 μm particle counts. Table 4-4 lists the filter passes required for each fluid to meet the cleanliness standard of 18/16/13. While the FI-S1 and FI-MO experienced significant changes in ISO code across particle sizes, both failed to achieve the cleanliness standard. FI-E achieved the standard after only 2 filter passes while FI-A met the standard prior to beginning filtration.

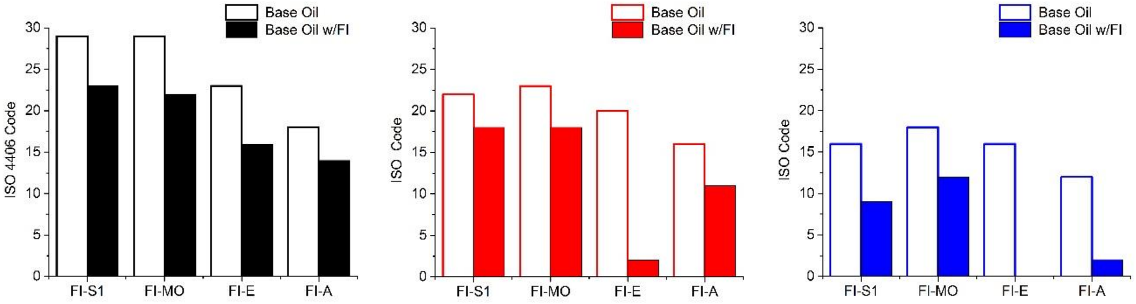


Figure 4-8: 4 μm (left) 6 μm (center) 14 μm (right) ISO codes for 220R-FI blends before and after filtration for 10 passes, with a 12 μm β1000 filter at 1 gpm.

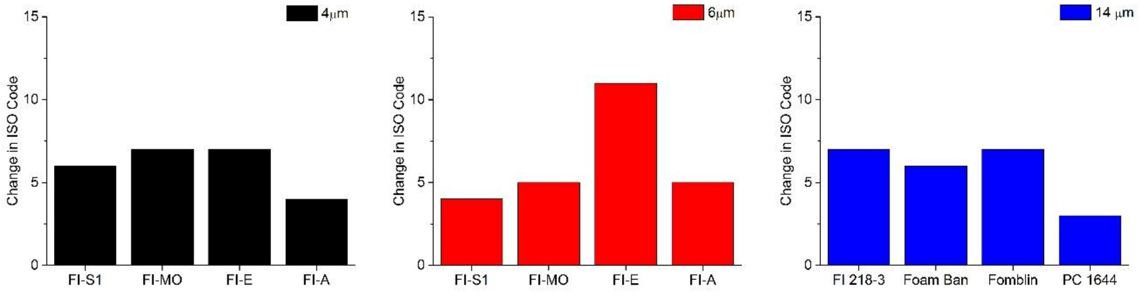


Figure 4-9: Decrease in ISO code following FI filtration for 10 passes, with a 12 μm β1000 filter at 1 gpm. Results are plotted by ISO size, 4 μm (left), 6 μm (center), and 14 μm (right). ISO codes below 10 are not considered statistically reliable, and as such are considered as <10 in calculations. This has the effect of limiting the possible magnitude of ISO code change for fluids that achieve an ISO of <10.

Table 4-3: Fluid cleanliness for 220R – FI blends before and after filtration for 10 passes, with a 12 μm β1000 filter at 1 gpm.

Fluid	ISO Code Before Filtration	ISO Code After 10 Filter Passes
220R w/ FI-S1 (0.14%)	>28/22/16	23/18/<10
220R w/ FI-MO (0.05%)	>28/23/18	22/18/12
220R w/ FI-E (0.001%)	23/20/16	16<10<10
220R w/ FI-A (0.025%)	18/16/12	14/11/2

Table 4-4: 12 μm β1000 filter passes required for 220R-FI blends to achieve 18/16/13 ISO cleanliness. FI-S1 and FI-MO Did Not Reach (DNR) the cleanliness goal. 0 indicates a fluid that met specifications before beginning filtration.

Fluid	Filter Passes Required to Reach 18/16/13
220R w/ FI-S1 (0.14%)	DNR
220R w/ FI-MO (0.05%)	DNR
220R w/ FI-E (0.001%)	2
220R w/ FI-A (0.025%)	0

4.4.2 2 μm Filtration Results

FIs were again introduced to circulating base oil and allowed to mix. After mixing, the fluids were filtered for 10 passes, with a 2 μm β1000 synthetic media filter. Figure 4-10 shows the ISO codes for the fluids before and after filtration. The difference in the height of these bars is the magnitude of the change in ISO codes resulting from filtration of the fluids, and is displayed in Figure 4-11. It is clear from these plots that filtration for 10 passes, with a 2 μm β1000 synthetic media filter, results in a significant decrease in particle counts. The pre and post filtering ISO codes for these experiments are presented in Table 4-5. It can be seen from the table and figures that FI-E again experiences the most dramatic change in particle counts. Furthermore, it required only a single filter pass to meet the 18/16/13 cleanliness standard. Table 4-6 summarizes the passes required for each blend to meet the cleanliness goal. We see that FI-S1 and FI-MO still fail to achieve cleanliness standards due to excessive 4 μm counts, and FI-A continues to meet specifications without filtering.

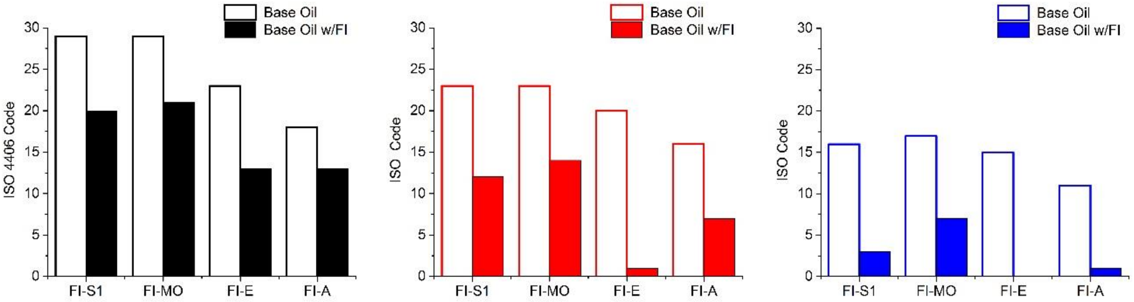


Figure 4-10: 4 μm (left) 6 μm (center) 14 μm (right) ISO codes for 220R-FI blends before and after filtration for 10 passes, with a 2 μm β1000 filter at 1 gpm.

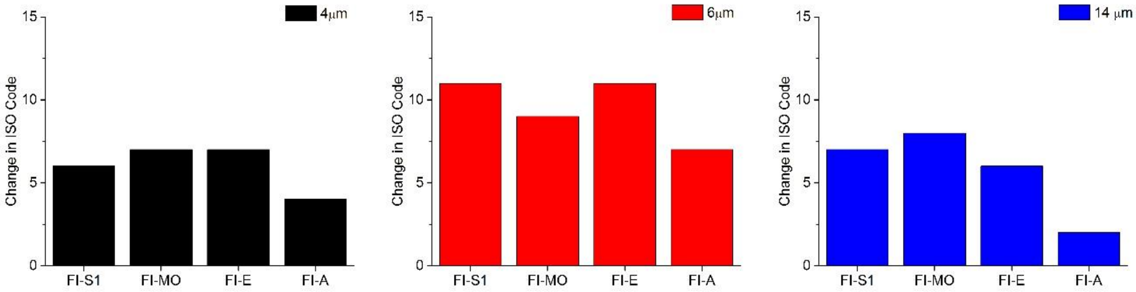


Figure 4-11: Decrease in ISO code following FI filtration for 10 passes, with a 2 μm β1000 filter at 1 gpm. Results are plotted by ISO size, 4 μm (left), 6 μm (center), and 14 μm (right). ISO codes below 10 are not considered statistically reliable, and as such are considered as <10 in calculations. This has the effect of limiting the possible magnitude of ISO code change for fluids that achieve an ISO cleanliness <10.

Table 4-5: Fluid cleanliness for 220R – FI blends before and after filtration for 10 passes, with a 2 μm β1000 filter at 1 gpm.

Fluid	ISO Code Before Filtration	ISO Code After 10 Filter Passes
220R w/ FI-S1 (0.14%)	>28/23/16	20/12/<10
220R w/ FI-MO (0.05%)	>28/23/17	21/14/<10
220R w/ FI-E (0.001%)	23/20/15	13<10<10
220R w/ FI-A (0.025%)	18/16/11	13<10<10

Table 4-6: 2 μm β1000 filter passes required for 220R-FI blends to achieve 18/16/13 ISO cleanliness. FI-S1 and FI-MO Did Not Reach (DNR) the cleanliness goal. 0 indicates a fluid that met specifications before beginning filtration.

Fluid	Filter Passes Required to Reach 18/16/13
220R w/ FI-S1 (0.14%)	DNR
220R w/ FI-MO (0.05%)	DNR
220R w/ FI-E (0.001%)	1
220R w/ FI-A (0.025%)	0

4.4.3 *Effect of Filter Pore Size on Fluid Cleanliness*

Here we compare the effect of filter sizes for the results presented in Sections 4.4.1 and 4.4.2. Table 4-7 summarizes the changes in ISO code for filtration at 2 and 12-microns; this data is also presented in Figure 4-10. It indicates that filtration with smaller pore filters resulted in greater overall decreases in ISO code. We see from the left plot that this is the case for all four additives. This trend holds across sizes for FI-S1, FI-MO, and FI-A. However, the center and right plots show that the 2-micron filter resulted in a smaller decrease for FI-E at particle sizes of 6 and 14 μm. The difference in achieved ISO code change for the 2 and 12-micron filters reflects the effect of the decreased pore size. This difference is presented in Figure 4-13. We see again that the increase holds across additives, except FI-E, and additionally note that the magnitude of the increase is most significant at the 6-micron particle size. It could be expected that there would be no significant change in the magnitude of ISO code change at 14 microns, since the particles in that size range exceed the beta rating of both filters. We see, in Figure 4-13, that that there is no more significant effect on ISO code at 2-microns than at 12-microns for FI-S1, FI-E, and FI-A. Filtration of FI-MO at 2 micron however resulted in removal of three more ISO codes than flirtation at 12 microns, a larger change than at 4 microns.

Table 4-7: Change in fluid cleanliness for 220R – FI blends after filtration for 10 passes, with 2 and 12 μm β1000 filters at 1 gpm.

Fluid	Change in ISO Code 12 μm Filter	Change in ISO Code 2 μm Filter
220R w/ FI-S1 (0.14%)	6/4/7	9/11/7
220R w/ FI-MO (0.05%)	7/5/6	8/9/9
220R w/ FI-E (0.001%)	7/11/7	10/11/6
220R w/ FI-A (0.025%)	4/5/3	5/7/2

Table 4-8: Summary of 12 and 2 μm β1000 filter passes required for 220R-FI blends to achieve 18/16/13 ISO cleanliness. FI-S1 and FI-MO Did Not Reach (DNR) the cleanliness goal. 0 indicates a fluid that met specifications before beginning filtration.

Fluid	Filter Passes Required 12 μm	Filter Passes Required 2 μm
220R w/ FI-S1 (0.14%)	DNR	DNR
220R w/ FI-MO (0.05%)	DNR	DNR
220R w/ FI-E (0.001%)	2	1
220R w/ FI-A (0.025%)	0	0

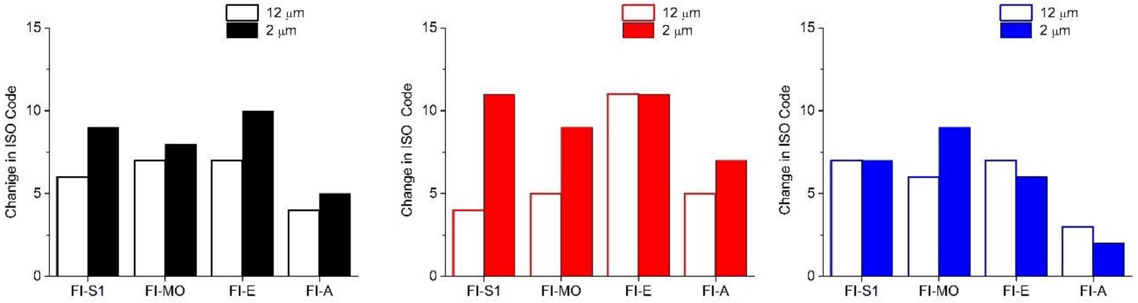


Figure 4-12: Change in 4 μm (left) 6 μm (center) 14 μm (right) ISO codes for 220R-FI blends after filtration for 10 passes, with 2 and 12 μm β1000 filters at 1 gpm.

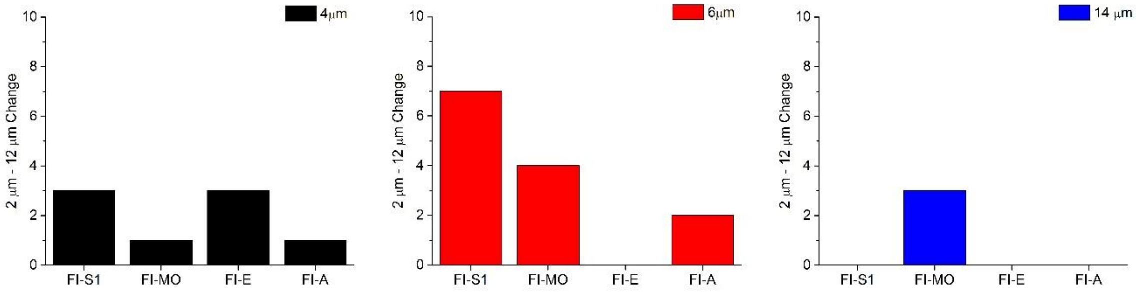


Figure 4-13: Effect of filter pore size on the magnitude of ISO code decrease from filtration. The difference in the magnitude of change for filtration with a 2 μm filter and 12 μm filter are presented by additive and particle size, 4 μm (left), 6 μm (center), and 14 μm (right).

4.5 Baseline Foam Characterizations

A production fluid must conform to fluid specifications provided by equipment manufacturers or oil certification standards such as those published by the American Petroleum Institute (API). Several standards are outlined in API 1509; generally, they allow for 10 mL of tendency and set a limit on time to meet zero volume stability [36]. As the treatment rates investigated here are more commonly found in hydraulic fluids we compare foam requirements to those put forth in Caterpillar TO-4, which calls for < 25 mL of foam tendency and 0 mL foam stability as defined by ASTM D892.

When conducting filtration trials as previously described, samples of each fluid were collected prior to beginning filtration. Foam characterizations were performed on these samples in order to provide a measure of baseline foam performance (Table 4-9). Additionally, many tests were conducted on 220R base oil, the results of which are also included in Table 4-9. The tendencies and stabilities presented for the FI blends are the mean result of 6 measurements taken for 2 samples by 3 people. The tendency and stability of the 220R are the mean values of 24 measurements taken by 3 people on 6 samples. In all cases, the indicated accuracies are represented by the standard error of the data set.

Table 4-9: Baseline ASTM D892 Sequence I foam characterizations for 220R base oil and four 220R-FI blends. Both the untreated base oil and the base oil-FI-E blend fail to meet TO-4 tendency specifications. 220R values represented by the mean and standard error of 24 measurements performed by 3 people. All other values are the result of 6 measurements by three people.

Fluid	Sequence I Tendency (mL of Foam)	Sequence I Stability (s to 0 mL of Foam)
220R	207 ± 19	204 ± 20
220R w/ FI-S1 (0.14%)	20 ± 0	70 ± 4
220R w/ FI-MO (0.05%)	8 ± 2	43 ± 14
220R w/ FI-E (0.001%)	156 ± 37	126 ± 14
220R w/ FI-A (0.025%)	16 ± 2	27 ± 6

Figure 4-14 displays the results tabulated in Table 4-8. The left plot displays the foam tendency of the 220R-blends, solid orange bars, in comparison to untreated 220R, orange outline bar. The right plot compares the stability, in terms of time to zero foam, results in the same manner utilizing green bars, and outlines. In all cases investigated, addition of the foam inhibitor improved foaming performance. The base oil alone does not meet TO-4 specifications for tendency. The addition of FI-MO, FI-A, and FI-S1 resulted in tendency reductions of 96%, 92%, and 90% respectively, allowing all three of these blends to meet TO-4 tendency specifications (<25 mL Foam). FI-E reduced foam volume by 25%, however still did not comply with TO-4 specifications. The FIs also provided significant improvements in foam stability performance. Although the base oil met the TO-4 specification of 0 mL of foam, the time to zero foam was reduced 86%, 79%, 66%, and 38% by FI-A, FI-MO, FI-S1, and FI-E, respectively.

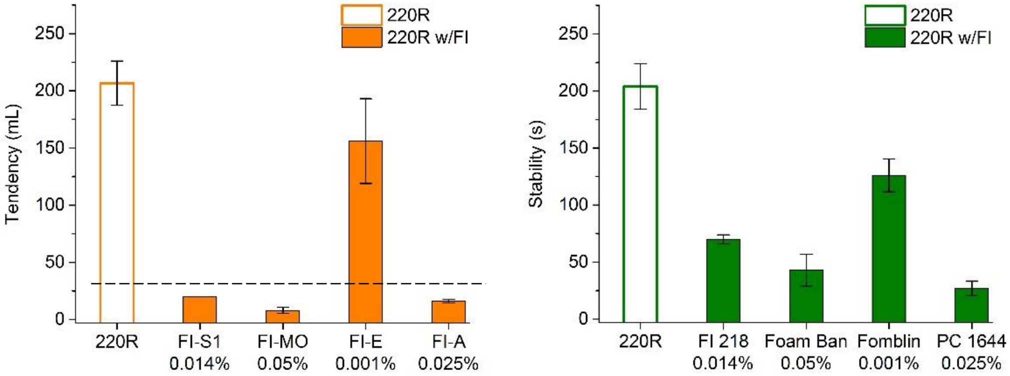


Figure 4-14: Baseline ASTM D892 Sequence I foam characterizations for unfiltered 220R base oil and four 220R-FI blends. As expected, all additives markedly improve both tendency (left) and stability (right) performance. FI-E has the least effect on foam performance and fails to meet the TO-4 tendency standard (<25 mL foam).

4.6 Effect of Extended Filtration on Foam Tendency

We ran trials to verify the ability of the foam station to detect changes in foam performance resulting from filtration. The fluid reservoir was charged with 2 gallons of 220R base oil. FIs were introduced to the reservoir and allowed to mix via circulation. After mixing, filtration was initiated. Samples were collected at 0, 7.5, 15, 25, 35, 50, 70, 90, and 110 filter passes.

Results for 220R w/ 0.14% FI-S1 are presented in Figure 4-15. Initial filtration, up to 35 passes, seems to have little effect on foam tendency. However, beginning at 50 filter passes, foaming tendency increase with filter passes. This effect is present across ASTM D892 sequences, but is least pronounced in Sequence II results.

Foam stability results, in terms of time to zero foam, also increase with filtration. Increases in time to zero foam for Sequence I can be seen beginning at 7.5 filter passes and continuing through 110 passes. As indicated by the near constant height of the teal bars in the right plot, Sequence II stability shows little change due to filtration. Sequence III results remain relatively stable until 35 filter passes and then begin to increase sharply at 50 passes.

These results indicate that prolonged filtration results in a marked decrease in foaming performance.

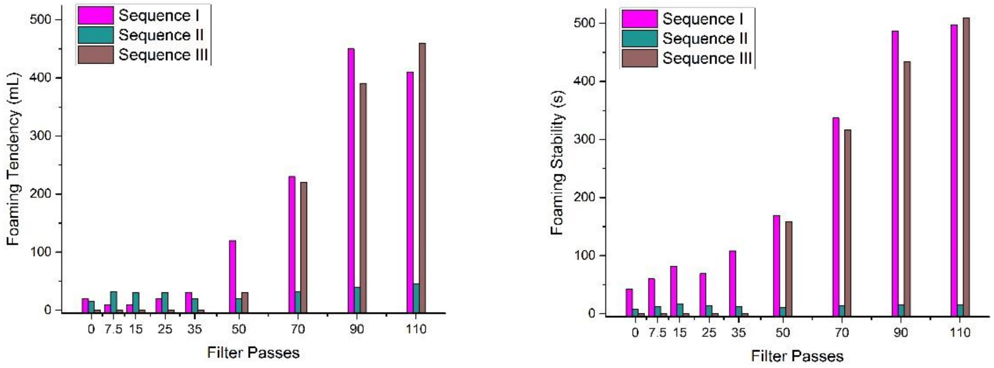


Figure 4-15: ASTM D892 Sequence I-III foam characterizations for 220R base oil w 0.14% FI-S1 filtered with a 12 μm β1000 filter. While initial filter passes appear to have little effect on foaming performance, both tendency and stability performance show a strong decrease in performance as filter passes increase.

4.7 Effect of Filtration on Foam Tendency (0 – 10 Filter Passes)

Filtration trials were conducted for each inhibitor and filter size. An inhibitor was added to 2 gallons of circulating oil, allowed to mix for 30 passes, and filtered. During each trial samples were drawn at 0, 2.5, 5, 7.5, and 10 filter passes. Foam performance for these samples was then characterized in accordance with ASTM D892 Sequence I and by recording the time to zero foam stability; stability foam volume was 0 mL for all samples. Table 4-10 and Table 4-11 tabulate the results of the tendency and time to zero foam measurements. We see that even limiting filtration of the fluids to 10 passes results in increased average foam volumes and time to zero foam though increases are not always statistically significant due to large variations in the data. We see as well that the increase is more pronounced for the 2-micron filter.

Stability results are less conclusive. All foam stability volumes continue to be 0 mL complying, with TO-4 stability standards. However, a slight tendency towards a greater time to zero foam seems to follow filtration.

Table 4-10: Mean ASTM D892 Sequence I foam tendencies for 220R-FI blends filtered with 12 μm / 2 μm β1000 filters.

Fluid	Sequence I Tendency (mL Foam) 12 μm Filter / 2 μm Filter				
Filter Passes	0	2.5	5	7.5	10
220R w/ FI-S1 (0.14%)	20 / 20	14 / 20	20 / 27	17 / 20	13 / 35
220R w/ FI-MO (0.05%)	12 / 3	10 / 7	10 / 11	8 / 12	8 / 9
220R w/ FI-E (0.001%)	90 / 222	217 / 298	227 / 340	257 / 357	253 / 332
220R w/ FI-A (0.025%)	18 / 13	73 / 273	70 / 140	93 / 210	83 / 217

Table 4-11: Mean ASTM D892 Sequence I foam stabilities for 220R-FI blends filtered with 12 μm (left) and 2 μm (right) β1000 filters.

Fluid	Sequence I Stability (Time (s) to 0 Foam) 12 μm Filter / 2 μm Filter				
Filter Passes	0	2.5	5	7.5	10
220R w/ FI-S1 (0.14%)	67 / 77	72 / 114	76 / 152	73 / 95	32 / 148
220R w/ FI-MO (0.05%)	62 / 22	40 / 25	32 / 51	46 / 57	36 / 49
220R w/ FI-E (0.001%)	120 / 151	144 / 218	162 / 206	181 / 210	181 / 207
220R w/ FI-A (0.025%)	21 / 32	55 / 158	59 / 96	73 / 138	65 / 127

Table 4-12: 12 and 2 μm β1000 filter passes that maintain TO-4 compliance (<25 mL tendency volume and 0 mL stability volume). 220R-FI blends were filtered for 10 passes. A value of 0 (FI-A) indicates that the fluid complied with TO-4 before filtration, but failed after 2.5 passes. FI-E Did Not Reach (DNR) the tendency goal prior to filtration.

Fluid	Filter Passes 12 μm Filter / 2 μm Filter
220R w/ FI-S1 (0.14%)	10 / 2.5
220R w/ FI-MO (0.05%)	10 / 10
220R w/ FI-E (0.001%)	DNR / DNR
220R w/ FI-A (0.025%)	0 / 0

We compiled the maximum number of filter passes retained TO-4 compliance, <25 mL foam tendency / 0 mL foam stability, for each fluid and filter size. These results are presented in Table 4-12 and indicate that filtering 220R-FI blends results in a decreased ability to meet TO-4. FI-MO showed the greatest resistance to filtering and complies with TO-4 specifications after 10 filter passes with either filter. FI-S1 continues to meet foaming specifications through 10 filter passes with the 12-micron filter. However, it fails to comply beginning at 5 filter passes with the 2-micron filter. FI-A fails to meet the standard after any filtration and FI-E does not meet it before filtration.

Results for each additive are presented in the following sections. These results include plots of the foam tendency and stability functions of filter passes and filter. The height of the orange bars represents foam tendency. Each tendency plot contains a dashed line at 25 mL of foam; a bar height below the dashed line indicates compliance with TO-4 requirements. Green bars indicate foam stability, in terms of time to zero foam. In these plots, the height of the bar corresponds to the mean value of three measurements performed by three people and the error bars represent the standard error for each set of measurements.

4.7.1 FI-S1

Filtration with the 2-micron filter showed a larger effect on both tendency and stability than the 12-micron filter. After 10 passes through the 12 micron filter, FI-S1 continues to comply with TO-4 specifications (Figure 4-16). However, under 2-micron filtration it fails to comply beginning at 5 passes. Time to zero foam shows a slight increase for FI-S1 over 10 filter passes at 2-microns, however seems unaffected by filtration at 12-microns (Figure 4-17).

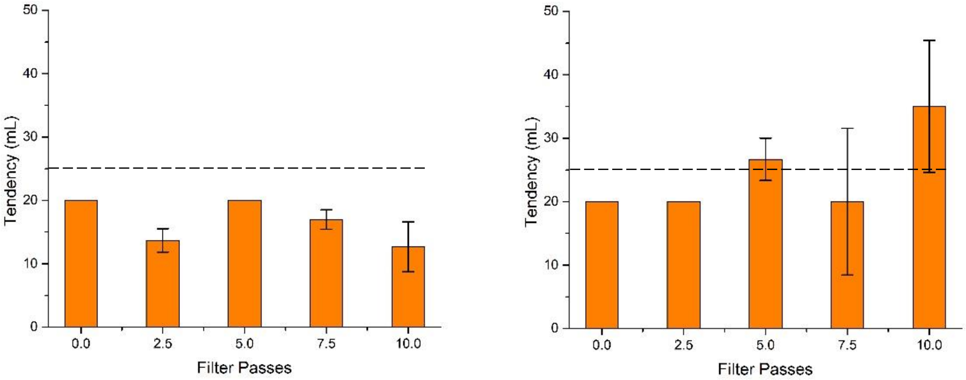


Figure 4-16: ASTM D892 Sequence I foam tendency for 220R w/ 0.14% FI-S1 (0 - 10 Filter Passes). Filtered with 12 μm (left) and 2 μm (right) β1000 filters. The dashed line at 25 mL of Foam indicates the maximum volume allowed under TO-4. When error bars are not visible, the results of the three measurements were in complete agreement.

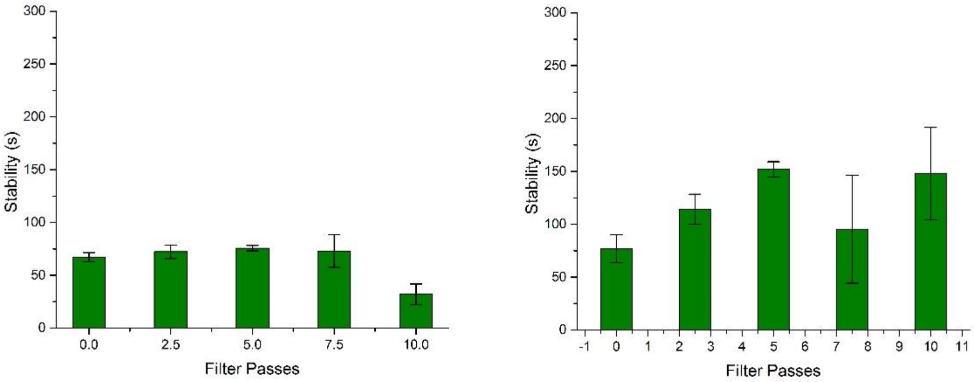


Figure 4-17: ASTM D892 Sequence I foam stability for 220R w/ 0.14% FI-S1 (0 - 10 Filter Passes). Filtered with 12 μm (left) and 2 μm (right) β1000 filters.

4.7.2 FI-MO

FI-MO showed the least deterioration in performance following filtration and continued to comply with specifications after 10 passes with either filter (Figure 4-18). Additionally, filtration with either filter showed no discernable effect on time to zero foam (Figure 4-19).

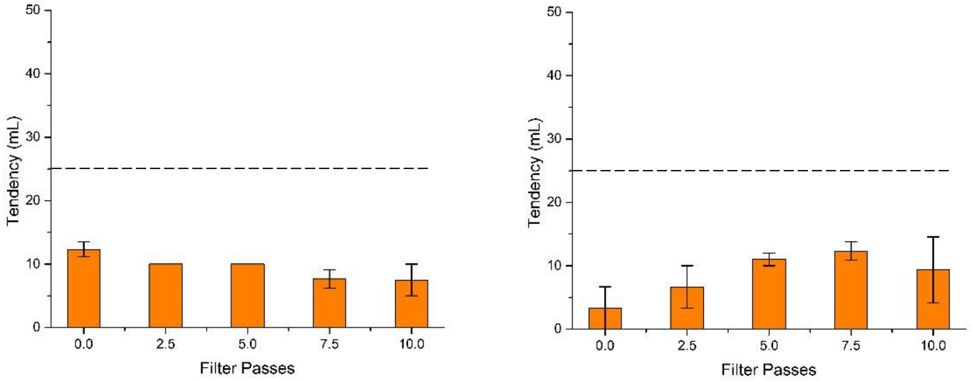


Figure 4-18: ASTM D892 Sequence I foam tendency for 220R w/ 0.05% FI-MO (0 - 10 filter passes). Filtered with 12 μm (left) and 2 μm (right) β1000 filters. The dashed line at 25 mL of Foam indicates the maximum volume allowed under TO-4.When error bars are not visible, the results of the three measurements were in complete agreement.

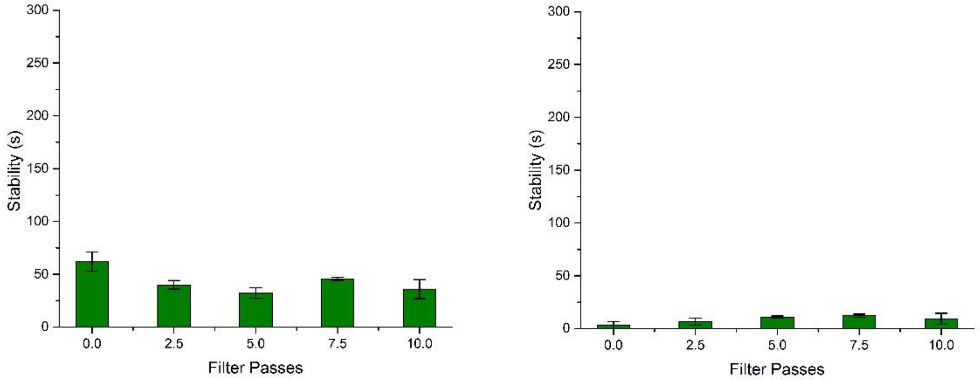


Figure 4-19: ASTM D892 Sequence I foam stability for 220R w/ 0.05% FI-MO (0 - 10 filter passes). Filtered with 12 μm (left) and 2 μm (right) β1000 filters.

4.7.3 FI-E

FI-E shows a significant increase in foaming tendency and time to zero foam following filtration with either filter. The effect is more pronounced for the smaller pore size filter. Measured tendency volumes do not indicate a correlation to filter passes. A large initial change in performance, from 0 to 2.5 passes, can be seen. However, subsequent results do not continue this trend. Stability results for the fluid filtered at 12-microns seem to indicate an increased time to zero foam for increasing filter passes. However, the 2-micron results show the same trend as the tendency values. FI-E failed to comply with TO-4 tendency specifications prior to filtration.

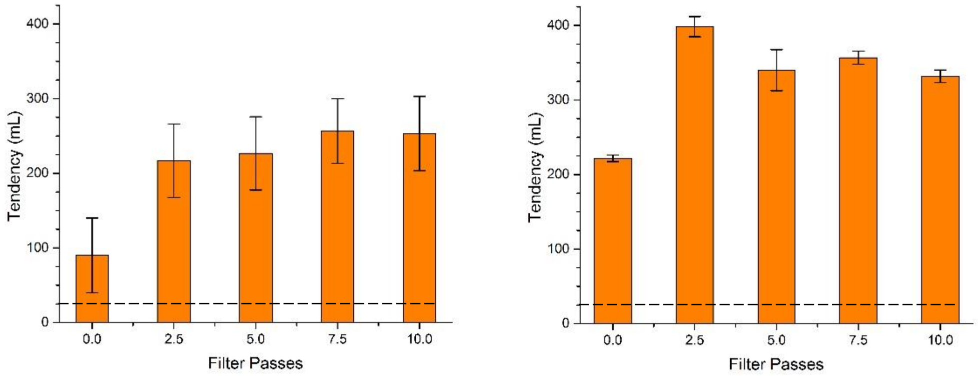


Figure 4-20: ASTM D892 Sequence I foam tendency for 220R w/ 0.001% FI-E (0 - 10 filter passes). Filtered with 12 μm (left) and 2 μm (right) β1000 filters. The dashed line at 25 mL of Foam indicates the maximum volume allowed under TO-4.

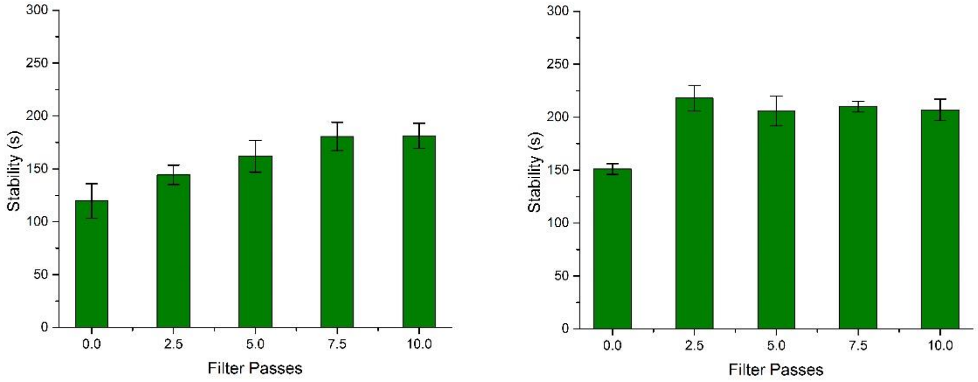


Figure 4-21: ASTM D892 Sequence I foam stability for 220R w/ 0.001% FI-E (0 - 10 filter passes). Filtered with 12 μm (left) and 2 μm (right) β1000 filters.

4.7.4 FI-A

For FI-A, filtering with either filter causes a sharp increase in foam tendency and in foam stability (Figure 4-22, Figure 4-23). An initial decrease in performance occurs between 0 and 2.5 filter passes, similar to results for FI-E. The decreased performance does not appear to worsen as filtration continues. The effect continues to be greater for smaller pore size and present, although less clearly, in time to zero foam stability results. Unlike the FI-E however, FI-A initially complies with TO-4 tendency, but fails at 2.5 passes and beyond. The change in time to zero foam appears much more pronounced for FI-A than for any of the other additives investigated here.

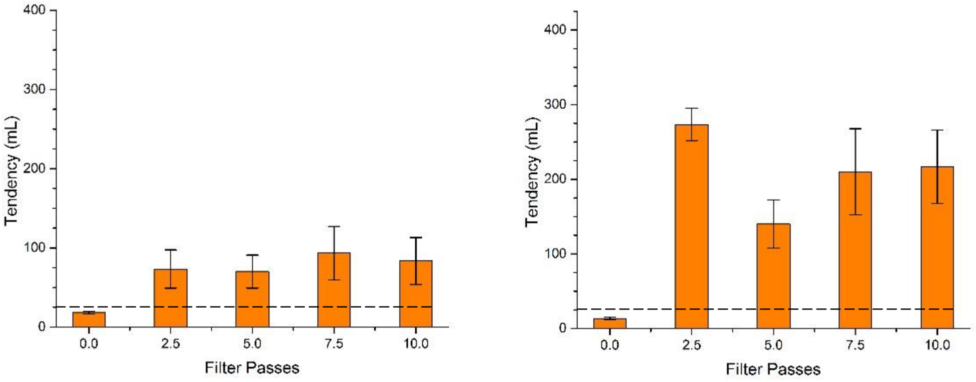


Figure 4-22: ASTM D892 Sequence I foam tendency for 220R w/ 0.025% FI-A (0 - 10 filter passes). Filtered with 12 μm (left) and 2 μm (right) β1000 filters. The dashed line at 25 mL of Foam indicates the maximum volume allowed under TO-4.

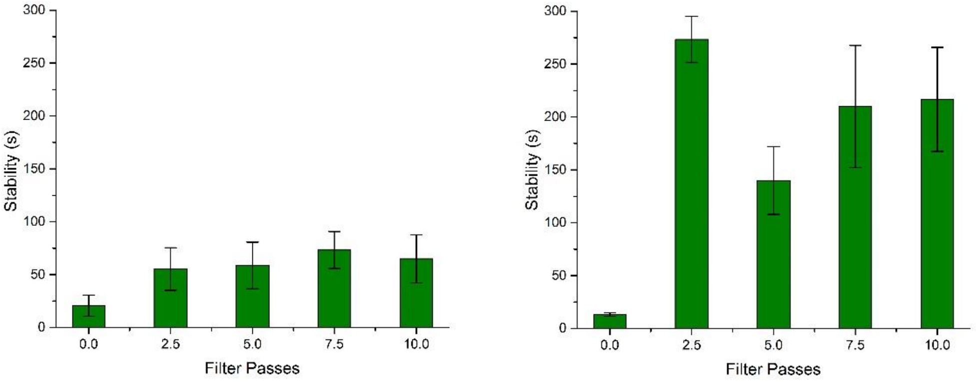


Figure 4-23: ASTM D892 Sequence I foam stability for 220R w/ 0.025% FI-A (0 - 10 filter passes). Filtered with 12 μm (left) and 2 μm (right) β1000 filters.

5 Conclusion and Recommendations

5.1 Research Summary

The goal of this project was to identify and characterize the interaction of foam inhibitors, optical particle counters and filters over the first ten filter passes. We sought to understand the effect of filtering on FI induced particle counts and quantify the resulting changes in FI performance as characterized by ASTM standard D892. While it was already known that FIs degrade fluid cleanliness ratings and that prolonged filtration degrades FI performance, the rate at which foam performance deteriorates had not previously been studied. This investigation sought to understand the effects of filtration as commonly seen in lubricant manufacturing and to deepen the understanding of the effects of FI chemistry on additive induced particle counts.

Three test stations were constructed for this work. We assembled a lubricant blending station that provides control over blending parameters such as, temperature, impeller speed, and container size. We obtained and repaired a nonoperational Koehler Dual Bath Foaming Characteristics Apparatus and developed replacement/alternative components for the apparatus that allowed us to conduct ASTM D892 foaming characterizations. Finally, we designed and constructed a filter station that circulates, filters, and particle counts fluids. The station allows the flow rate to be set from <0.1 gpm to 1.68 gpm. When fully charged with fluid, it allows collection of ten 500 mL samples; when fewer samples are required, the system will operate on as little as 0.75 gal. of fluid. It accepts fluid viscosities from 15 cSt to 250 cSt or more and will operate at temperatures from room temperature (~20 °C) to 60 °C, providing an effective fluid range of ISO 15 through 680 hydraulic oils.

Cleanliness profiles and pre/post filtering particle counts performed on the equipment constructed here match sufficiently with findings in [13]. We also found that we were able to measure the increase in foaming tendency from extended filtration matching expectation based on findings in [28]. We concluded that the validation experiments were completed successfully and proceeded to new work.

We first examined the increase in particle counts resulting from the addition of FIs to 220R base oil. For each inhibitor, 2 gallons of 220R base oil was added to the reservoir and allowed to circulate. The inhibitor was then introduced to the reservoir and allowed to mix for 30 passes. Particle counts and ISO codes were recorded at ~10 s intervals during the entire process. The results of these trials show that, for every additive investigated in this study, introduction of the inhibitor to circulating base oil results in a dramatic increase in particle counts. The silicone based inhibitors resulted in the largest particle count increase with the 4 μm particle counts following FI addition exceeding 2.5 million particles/ mL, the maximum detectable by the HIAC ROC inline particle counter and resulted in the highest ISO code prescribed by ISO 4406, >28. We note that the increase in particle counts is greater for smaller particle sizes and that only FI-A retained compliance with the 18/16/13 cleanliness standard following FI addition.

After verifying the increase in particle counts resulting from inhibitor introduction, we again charged the reservoir with 2 gallons of base oil and introduced FIs as previously described. We filtered each fluid for an extended amount of time. ISO codes and particle counts were again collected at ~10 s intervals throughout the tests. We verified that these particle counts could be eliminated through persistent filtration and, furthermore, that nearly any desired cleanliness could be obtained via persistent filtration. For FI-S1, the filtration time required to reach the 18/16/13 cleanliness goal was approximately 30 minutes and within 2 hours all particle counts are far below cleanliness standards.

Based on the fact that it is common industry practice to filter production fluids and that production filtration cycles are generally limited to 10 passes or less, we investigated the effects of filtration up to 10 filter passes. We conducted testing within 10 filter passes to identify our ability to regain cleanliness standards after the addition of FIs.

We again charged the reservoir with 2 gallons of base oil and introduced FIs as previously described. The fluid was then filtered for 10 passes. We continued to record particle counts and ISO codes at ~10 s intervals. Additionally, samples were collected from the test station at 0, 2.5, 5, 7.5, and 10 filter passes. This process was completed for both 12 and 2-micron filters. We found that filtration of 220R with foam inhibitors results in a significant decrease in particle counts. Additionally, we found that the majority of the decrease occurs during the first 2.5 filter passes. It was determined that the 2-micron filter resulted in a greater change in ISO code and that FI-E responded the most strongly to filtration, dropping below ISO 10 for both 6 and 14 μm particle counts with either filter. In each experiment 220R met cleanliness goals prior to FI addition and filtration greatly improved fluid cleanliness. However, neither FI-S1 nor FI-MO reached the 18/16/13 cleanliness goal within 10 filter passes. FI-A met cleanliness goals prior to filtration. FI-E returned to 18/16/13 or better after 2 and 1 filter passes through the 12 and 2-micron filters, respectively.

The unfiltered (0 filter passes) samples collected from the filtration tests were analyzed to provide a baseline measure of FI performance. ASTM D892 Sequence I foam characterizations were performed on the unfiltered 220R-FI blends and compared to results for untreated 220R. The performance of the blends and untreated oil were also examined to determine compliance with Caterpillar TO-4 requirements, which call for < 25 mL of foam tendency and 0 mL foam stability as defined by ASTM D892. All blends and the untreated 220R complied with the zero volume stability requirement. However, untreated 220R fails to comply with TO-4 tendency requirements. Each FI investigated decreased foaming tendency significantly and resulted in compliance for FI-S1, FI-MO, and FI-A treated base oil. Only FI-E treated oil still failed to comply with TO-4.

We then examined the ability of the fluids to meet foaming specifications following filtration. The samples collected during the experiments to determine the ability to regain cleanliness standards within 10 filter passes were measured for Sequence I foaming tendency and stability in terms of time to zero foam. Prior to filtration, three of the 220R-FI blends met TO-4 specifications, however only one blend was able to meet the cleanliness specification of 18/16/13. We found that even limiting filtration of the fluids to 10 passes resulted in increased foam volumes and time to zero foam for all additives, except FI-MO. It was also found that the effect was more pronounced for the 2-micron filter. For all additives, excluding FI-MO, filtration decreased our ability to meet TO-4 standards. We find that FI-A meets TO-4 prior to filtration but fails after 2.5 filter passes. FI-E fails to comply prior to filtration and foam volume further increases with filtration. FI-MO complies through 10 filter passes with either filter. FI-S1 complies for up to 10 passes with the 12-micron filter but begins to fail after 2.5 filter passes at 2-microns.

Overall, we find that FI induced particle counts can be eliminated through sufficient filtration, but that filtration at the levels required to reach cleanliness goals is not a reasonable solution. The time and expense associated with extended filtration make it economically unrealistic and the decrease in FI performance results in unacceptable foam performance. When the addition of the inhibitor resulted in failure to meet cleanliness goals (FI-S1, FI-MO, FI-E), we were unable to simultaneously regain fluid cleanliness standards and maintain foaming characteristic compliance.

5.2 Recommendations for Improving Test Equipment and Processes

5.2.1 Blending Station

While not necessary for this investigation, modifications to the blending station will increase the ability to obtain further results and expand this work. The current blending station allows us to mix lubricant blends in house. However, it does not provide sufficient control over blending parameters to allow for experimental investigations. An investigation into the effects of blending parameters on requires the ability to ensure that all parameters, other then the parameter under investigation, remain constant.

The distance from the mixing impeller, the floor of the mixing vessel is an important factor in determining the time to reach a stable dispersion. A pressure knob on a vertical pole currently provides continuously adjustable control over the impellers mixing height. Ensuring that the distance from the impeller to the floor of the mixing vessel remains consistent, is readily accomplished by either permanently affixing the vertical support at a given height, or modifying the adjusting mechanism such that it has multiple, fixed, positions.

Shear rate in the fluid is primary factor in determining important characteristics of a liquid-liquid dispersion, maximum and average drop size for example. Impeller speed is one of many factors that influences the shear rate in the fluid. In the current blending station, mixing speed is controlled by controlled by a rotary knob on the motor. The knob allows the technician to select a speed from 1 to 10. Selection of a different mixing head or the addition of a feedback sensor and controller, such as a rotary encoder and programmable logic controller, would provide control over blending speed.

Finally, as temperature strongly influences viscosity and surface tension, it is also an important factor in determining dispersion characteristics. The temperature of the fluid is currently controlled manually. The person blending the fluid monitors a thermometer in the fluid and adjusts the temperature of the hot plate surface. The addition of a temperature probe compatible with the hot plate in use would allow the hot plate controller to motor the temperature of the fluid and adjust the temperature of the plate automatically. This would alleviate variations in final dispersions that result from viscosity and surface tension differences.

5.2.2 Foam Station

Obtaining reproducible foam characterizations proved to be a challenge. The air flow to the cylinders is currently regulated by two analog flow meters. Although the collected air volumes remain consistent with ASTM standards, we are not confident the air is supplied at a consistent rate throughout the test. Additionally, the thermocouple thermometers utilized to measure the temperature of the baths have been seen to vary from each other by up to 2 °C. The accuracy of these meters is somewhat suspect. It is recommended that the air lines on the test station be plumbed to ensure they are leak free, that the flow meters be calibrated to ensure they are providing a consistent flow of air, and that the thermocouples are either replaced or transitioned to liquid filled thermometers. It is believed that these changes, when coupled with the recommendations put forth in process improvements for foam testing, will increase our ability to obtain consistently reproducible results.

5.2.3 Foam Tests

The error in in our foam measurements is greater than desired. As multiple individuals were conducting foam testing, the three measurements were often taken up to one week apart. ASTM D892 notes that FI performance degrades and recommends performing Option A (vigorous blending before testing) on samples that have rested for 2 weeks or more. All of our tests were completed within this timeframe. However, in an effort to tighten the error on some foam results we re-tested the five samples collected for 12 micron filtered FI-A. As these samples had rested for more than two weeks, they were treated in the same manner as the samples tested from the round robin study conducted by ASTM, i.e. they were slowly upended ten times to redistribute the FI in the sample. The resulting measurements are plotted side by side with the original results in Figure 5-1. As can be seen by the difference in the height of the bars, the subsequent retest resulted in significantly larger foam measurements. The

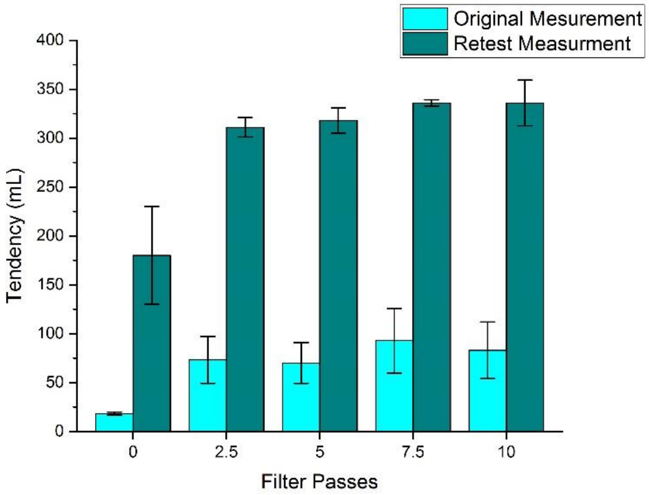


Figure 5-1: Foam tendency vs filter passes for 220R w/ 0.025 % FI -A The light colored bars represent the results from testing carried out within one week of filtration. The darker bars were tested ~7 weeks after filtration but were gently agitated. The deterioration of the FI is clearly observed in the height difference of the bars.

error bars however, represent a much smaller percentage as can be seen by the size of the error bars relative to the overall foam height. At this time, it is suspected that turning the bottles slowly as described in the ASTM round robin study will result in less variation between tests. It is also suspected that conducting foam characterizations for each sample on the same day will further minimize variation in results. Preliminary trials are underway to determine if this is the case.

5.2.4 Filter Station

5.2.4.1 Temperature Controls

Heating the test fluids will reduce their viscosities and increase their ability to release entrained air. It is well known that many properties of oils, such as viscosity and surface tension, are strongly dependent on temperature and that these properties play an important factor in determining liquid-liquid dispersion characteristics. Dispersion characteristics then strongly influence FI performance. Installation of an in reservoir heating element and basic temperature controller will increase confidence in results and allow further investigation into the effects of temperature on particle counts incurred during mixing, while still allowing the filter rig to standardize other mixing parameters. Gaining control over this parameter is an important next step.

5.2.4.2 Station Plumbing

The initial iteration of the filter station was equipped with pressure relief valves at two locations (Figure 5-2). These two valves provided a measure of safety, protecting system components from damage and users from potential injury. However, the system suffered from an intermittent condition where the particle counts of a circulating fluid would increase indefinitely.

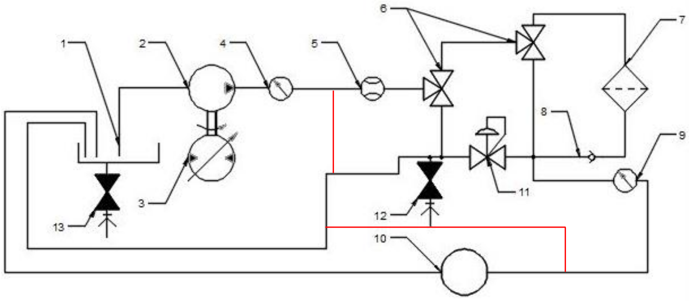


Figure 5-2: Location of removed pressure relief valves (in red) on filter rig schematic.

Systematic testing of the system revealed that this was not the result of air-leaks or cavitation at the pump, finding instead, that it resulted from a failure to successfully blead all air from the system. Although the system was carefully purged of air and allowed to rest overnight before each test, the problem persisted. It was found that the "dead legs" on the lines leading to the valves hindered our ability to successfully blead all air from the lines, and resulted in the intermittent problem with increase particle counts. These valves have been removed, but have reduced the safety and durability of the system.

Re-plumbing the station will allow us to place the pressure relief valves in positions that do not result in "dead legs" and result in entrained air. Furthermore, the station should be reconstructed so that it is arranged vertically, thereby allowing bleed valves to be strategically placed near the top of the station. During this process, a set of valves should be placed in the system so that compressed air can be utilized to fully flush the system. These relatively simple modifications will result in a more robust and user-friendly test station.

5.2.4.3 Particle Counts

It has been shown that there are variations between particle counts obtained by different particle counters [13], [26]. Including a second inline particle counter in the filter station and/or verify counts with a desktop counter would give further statistical significance to our findings.

5.2.5 Filtration Tests

Production fluids are filtered for up to 10 filter passes while retaining necessary performance characteristics. It is possible that the interactions of the other additives present in fully formulated lubricants limit the effects of FIs on particle counts and the effects of filtering on FI performance. We must eliminate the possibility that the differences in performance we observed are not related to variations between blending in our lab and in production.

Production blends are created by stirring heated fluids with impellers or paddles, often in baffled tanks. Also, experimental blends created by manufactures match production blends sufficiently well, and that these blends are created by stirring heated fluids in un baffled tanks. We recommend that this study be repeated with heated fluids blended in the filter station and with fluids blended externally. A comparison of the results from these two sets of results will allow us to determine if our experiments, that utilize circulation mixing, accurately describe the effects of FIs on production fluid cleanliness. If it is found that the results are significantly different from each other, further investigation of mixing parameters should be performed.

5.3 Areas for further study

5.3.1 Blending Parameters

It is known that manufactures are able to filter fluid to a cleanliness of 18/16/13 without degrading FI performance to below foaming standards. Our inability to replicate this practice is likely due to differences in mixing. It is suspected that the most significant mixing disparity is fluid temperature. Typically, fluid blending occurs at temperatures between 55 and 65 ºC, in our filtration trials mixing occurs at room temperature of ~25 ºC.

Foam inhibitors and base oils are liquid-liquid dispersions or solutions in which two or more insoluble fluids exist in distinct phases. The base oil exists as the continuous phase and the FI as a dispersed phase. Liquid-liquid dispersions are described by their dispersed phase concentration (volume fraction), coalescence rate mean drop size, and drop size distribution. The characteristics of the liquid-liquid dispersion is determined by system agitation. Flows created by an agitator are responsible for the breakup, coalescence and suspension of drops within the system. Turbulence and areas of high shear near the impeller causes drop breakup and leads to dispersion. Coalescence, on the other hand, results in areas of laminar flow when drops of the dispersion are allowed to remain in contact for extended periods of time.

Drop sizes are dependent on flow, shear, turbulence, and dispersion time as well as on physical and interfacial system properties such as viscosity and surface tensions. In the case of stirred vessels, mean drop size and drop size distribution depend on the placement and operational speed of the agitator [64]. High speeds lead to fine, stable, emulsions, while inadequate speed often results in phase separation [34]. Coalescence and dispersion are both dependent on the fluid dynamics created by the agitation [65].

Optical particle counters show an increase in particle counts as droplets of FI pass through the viewing area. Additionally, foam inhibitors are known to perform best in a range of dispersion sizes. Some inhibitors, PDMS, for example, may even lead to foam stabilization if they form too fine a dispersion. This indicates that the mixing conditions of the FI and base oil will strongly influence the performance of the inhibitor. If the dispersion can be created such that the maximum or even average drop size is below 4 microns, the effects on fluid cleanliness will be minimized.

An investigation into the effects of blending parameters on the particle counts resulting from FI addition to base oils, the ability to filter these particle counts from the fluid, and the ability of the fluid to retain its foaming performance may provide important insight. This may in turn lead to the ability to predict viable combinations of treatment rate, blending, and filtering that results in clean fluids that conform to foaming standards.

5.3.2 Filtering Parameters

It has been previously shown that FI removal by filtration occurs at a greater rate for synthetic filters than for cellulose filters [28]. It was further noted that the primary method of silicone FI removal appeared to be preferential wetting of the filter media. The method of removal for additives other has not been investigated. Furthermore, as filter media has advanced the primary method of removal may have evolved. An investigation into the effect of filter media and the primary method of FI removal may allow insights that would allow a treatment / filtration scheme to be devised that allows both FI effectiveness and fluid cleanliness.

5.3.3 Treatment Rates

Although additive manufactures provide a recommend treatment range, specific treatment rates are determined through trial and error. Treatment rates should be such that the fluid meets foam performance standards following sufficient filtration to meet cleanliness goals. Our results seem to indicate that the treatment rates here may need adjustment. FI-E, for example, fails to meet TO-4 specifications prior to filtration. This could indicate an insufficient treatment rate of dispersion size, while FI-MO continues to meet specifications at all filtration levels, which may indicate overtreatment. Results for FI-A and FI-S1 are less clear. FI-A meets both foam and cleanliness standards without filtration. Reducing the treatment rate for FI-A may continue to allow foaming compliance while reducing the effects on cleanliness. FI-S1 allows some level of filtration before failing to meet foaming specifications but fails to meet cleanliness standards. A reduction in the treat rate for FI-S1 may reduce FI induced particle counts to a level that can be addressed with filtration without compromising TO-4 compliance.

Excessive treatment rates result in unnecessary expenses, while insufficient treatment rates decrease performance. We suggest an investigation into the effects of treatment rate on the particle counts resulting from FI addition to base oils, the ability to filter these particle counts from the fluid, and the ability of the fluid to retain its foaming performance. This may in turn lead to the ability to predict viable combinations of treatment rate, blending, and filtering that results in clean fluids that conform to foaming standards.

5.4 Final Thoughts

The inclusion of FIs in lubricant formulations is a necessity; however, it also reduces the ability of manufacturers to meet cleanliness goals as measured by optical particle counters. The results here indicate that an increase in particle counts follows the addition of foam inhibitors and that the largest increase in particle counts results from silicone-based inhibitors. Additionally, our results indicate a more significant decrease in performance as filter pore size decreases. We further note that the effects on inhibitor performance occur before filtration has enable compliance with cleanliness goals. Further investigation is required to determine a balance of treat rate, blending, and filtration that provides the ability to meet both cleanliness and foaming standards.

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