

METHODS OF MEASURING EFFECTS OF FINE FILTRATION CONDITIONS ON
LUBRICANT DEFOAM RETENTION PROPERTIES

FIELD OF THE INVENTION

5 [0001] The field of the invention relates to methods of measuring lubricant defoam properties after simulated turbulent flow or fine filtration conditions. These methods are particularly suited to simulate field conditions of industrial gear oils.

BACKGROUND OF THE INVENTION

10 [0002] Foam in industrial gear oils (“IGO”) can cause problems in the gearbox, including, excessive wear, heat, and oxidation. Foam can contribute to wear because air can displace the lubricant between the gears and other metal components in the gearbox causing metal to metal contact to occur. The air in the foam can also cause an insulating barrier preventing efficient heat dissipation throughout the gearbox. Excessive heat can cause various components in the industrial gear lubricant to decompose and thereby reduce its effectiveness. Entrapped air can also cause excessive oxidation of the lubricant and lubricant components which also reduces their effectiveness.

15 [0003] There are several industry-accepted test methods for assessing IGO performance, including defoaming and filterability, under laboratory conditions. These industry-accepted test methods, however, do not always correlate well with, or predict, an IGO’s actual performance when used in the field, or “real world” applications. Thus, there is a need for a laboratory test method that better predicts an IGO’s actual field performance.

SUMMARY OF THE INVENTION

20 [0004] The disclosed test methods are better predictors of an IGO’s field performance. Accordingly, test methods for assessing lubricant performance, including defoaming and filterability, are disclosed. The methods include the utilization of a filtration test rig comprising a heated tank equipped with a temperature sensor and wherein the heated tank is fluidly connected to a circulating pump and a filter housing equipped with a filter having a pore size equal to or less than 25 microns. The heated tank is then filled with the lubricant to at least 50% capacity and the lubricant is heated to at least 80 °C. The lubricant is then circulated through the filtration test rig using the circulation pump so that the lubricant passes through the 25-micron (or less) filter at least 5,000 times. A sample of the filtered lubricant is then obtained and its defoam properties are measured using ASTM D892.

[0005] In some embodiments, the filter may have a pore size equal to or less than 10 microns. In some embodiments, the lubricant may comprise at least one polyacrylate antifoam and/or at least one silicon-containing antifoam.

5 [0006] In some embodiments, the lubricant is circulated through the filtration test rig at a flow rate of 1 to 15 liters per minute. In some embodiments, an aliquot of the lubricant may be tested using ASTM D892 prior to being added to the filtration test rig and/or prior to being circulated through the filtration housing to establish a baseline of defoam performance. These baseline results may then be used to compare to the results after the lubricant is circulated through the filtration housing. In yet other embodiments, the filtration
10 test rig may be flushed with an aliquot of the lubricant prior to circulating the lubricant through the filtration housing.

[0007] The heated tank may be heated with a jacket, a heat exchanger, and/or a direct immersion heater.

BRIEF DESCRIPTION OF THE FIGURES

15 [0008] FIG. 1 shows a diagram of a filtration test rig.

DETAILED DESCRIPTION OF THE INVENTION

[0009] Various preferred features and embodiments will be described below by way of non-limiting illustration.

20 [0010] Test methods for assessing lubricant performance, including defoaming and filterability, are disclosed. The methods include the utilization of a filtration test rig comprising a heated tank equipped with a temperature sensor and wherein the heated tank is fluidly connected to a circulating pump and a filter housing equipped with a filter having a pore size equal to or less than 25 microns.

Filtration Test Rig

25 [0011] The filtration test rig may be configured as shown in FIG. 1. The filtration test rig (10) may comprise a heated tank (12) equipped with a heat source and temperature sensor (not shown). The heated tank is filled with the lubricant to be tested to at least 50% capacity via a charge port (14) or other opening, which may be submerged. The lubricant may then be heated in the tank to at least 80 °C. The heated tank may be heated via any
30 means known to those ordinarily skilled in the art, including a jacket, a heat exchanger, and/or a direct immersion heater. The lubricant may also be lightly agitated with a paddle or impeller to facilitate heating, provided excessive air is not introduced into the lubricant, which could affect defoam test results.

[0012] Once the lubricant is at temperature, the lubricant is circulated from the heated tank (12) to a circulating pump (18) via a first transfer line (16). The lubricant passes through the circulating pump (18) to the filter housing (22) through a second transfer line (20). The filter housing is equipped with a filter having a pore size of 25 microns or less. After passing through the filter housing, the lubricant is circulated back to the heated tank (12) via a third transfer line (24). The tank inlet from the third transfer line (24) may be submerged. In some embodiments, various portions of the filtration test rig (tank (12), transfer lines, etc.) may be insulated to help maintain a constant temperature and avoid significant cooling of the test lubricant as it flows through the filtration test rig.

[0013] Suitable filtration test rigs include commercially available filtration test rigs or other test rigs, such as clutch test rigs, modified appropriately, for example rigs available from Siemens Aktiengesellschaft (Munich, Germany) or ZF Friedrichshafen AG (Friedrichshafen, Germany). The ZF filtration test rig may be based on either the ZF SSP180, the ZF GK1, or the ZF GK2 clutch test rigs that have been modified to be filtration test rigs. The inventors of the methods disclosed herein, however, have found the recommended testing procedures accompanying these rigs do not always correlate well or predict the defoam filtration performance of lubricants in the field. This can result in the approval of new lubricants using known test methods, that may not perform as well under real world or field conditions, resulting in field issues.

[0014] The filtration test methods disclosed herein, however, more accurately correlate with real world, or field conditions, and therefore are better tests for approving new lubricants than the known testing procedures recommended by test rig suppliers. Without limiting the disclosed technology to one theory of operation, it is believed some types of antifoams form droplets that are prone to shearing as they pass through the filter, resulting in smaller droplets of antifoam present throughout the lubricant. The smaller droplets may be less effective at reducing foam. It is believed the test methods disclosed herein may more accurately simulate these shearing effects occurring in the field.

[0015] The test methods disclosed herein include the steps of heating the lubricant to at least 80°C and circulating the lubricant through the filtration test rig filter at least 5,000 times. A sample of the filtered lubricant is then obtained and its defoam properties are measured using ASTM D892.

[0016] The filter in the filter housing may have a pore size of less than or equal to 25 microns. In some embodiments, the filter may have a pore size equal to or less than 10 microns (fine filtration). Exemplary filters are not overly limited and can include any

commercially available inline filter having the requisite pore size and that is compatible with the selected filter housing. HYDAC and MAHLE are well known suppliers of inline filters. In some embodiments, the lubricant may comprise at least one polyacrylate anti-foam and/or at least one silicon-containing antifoam. Fine filtration, or a filter having at least 10 microns may be used with lubricants comprising an acrylate antifoam. When using the silicon-containing antifoam, it is generally preferable to use a filter with a pore size of 25 microns.

[0017] In some embodiments, the lubricant is circulated through the filtration test rig at a flow rate of 1 to 15 liters per minute. In some embodiments, an aliquot of the lubricant may be tested using ASTM D892 prior to being added or circulated through the filter housing (and filter) to establish a baseline of defoam performance. These baseline results may then be used to compare to the results after the lubricant is circulated through the filtration housing. In yet other embodiments, the filtration test rig may be flushed with an aliquot of the lubricant prior to circulating the lubricant through the filtration housing.

[0018] The test methods disclosed herein include the steps of heating the lubricant to at least 80°C and circulating the lubricant through the filtration test rig filter at least 5,000 times, or cycles. Table 1 below shows some operating conditions of various commercially available test rigs that will result in 5,000 cycles. The level of filtration may be adjusted by changing the pore-size of the filter.

Table 1

Test Rig	Siemens Fil-tration	ZF SSP180	ZF GK1	ZF GK2
Filter model no (10 µm)	0030 D 010 ON (HYDAC)	PI 3105 PS10 (MAHLE)	PPI 3108 PS10 (MAHLE)	PPI 3108 PS10 (MAHLE)
Filter model no (25 µm)	-	PI 3105 PS25 (MAHLE)	PPI 3108 PS25 (MAHLE)	PPI 3108 PS25 (MAHLE)
Lubricant temp (°C)	80	80	80	80
Lubricant flow rate (L/min)	1	9	15	15
Lubricant volume in tank (L)	1.5	10	14	13
Turbulence level	Low	Low	High	Low
Heater type	Jacket	Immersion	Heat exchanger	Heat exchanger
Run time (5,000 cycles)	125 hours	144 hours	75 hours	72 hours

[0019] A sample of the filtered lubricant is then obtained and its defoam properties are measured using ASTM D892.

[0020] Accordingly, in some embodiments, the filtration test rig is a Siemens filtration test rig equipped with a 25 or 10-micron HYDAC filter. In the Siemens embodiments, the lubricant volume in the tank may be 1.5 liters and the flow rate may be 1 liter per minute. The lubricant is circulated through the Siemens test rig for 125 hours (or 5,000 cycles).
5 After 5,000 cycles, a sample of the filtered lubricant is obtained and the defoam properties are measured using ASTM D892.

[0021] In some embodiments, the filtration test rig is a ZF SSP180 clutch test rig equipped with a 25 or 10-micron MAHLE filter. In the ZF SSP180 embodiments, the lubricant volume in the tank may be 10 liters and the flow rate may be 9 liters per minute.
10 The lubricant is circulated through the ZF SSP180 test rig for 144 hours (or 5,000 cycles). After 5,000 cycles, a sample of the filtered lubricant is obtained and the defoam properties are measured using ASTM D892.

[0022] In some embodiments, the filtration test rig is a ZF GK1 or GK2 clutch test rig (collectively “GK”) equipped with a 25 or 10-micron MAHLE filter. The GK1 and GK2
15 test rigs are similar, except the GK1 test rig uses the test lubricant as a hydraulic oil for operating the clutch, whereas the GK2 has a separate circuit. The procedures disclosed herein, however, can be applied to both rigs. In the ZF GK1 embodiments, the lubricant volume in the tank may be 14 liters and the flow rate may be 15 liters per minute and a cycle time of 75 hours (or 5,000 cycles). The ZF GK2 rig is plumbed with a bypass line
20 with a valve from the filter housing to the heated tank to bypass (when the valve is open) the test head where the friction disks are housed to reduce the surface area of the rig. The bypass outlet into the tank is submerged in the lubricant. The lubricant may be subjected to low turbulence conditions (going through the bypass) or high turbulence conditions (going through the test head). In the ZF GK2 embodiments, the lubricant volume in the
25 tank may be 13 liters and the flow rate may be 15 liters per minute and a cycle time of 72 hours (or 5,000 cycles). After 5,000 cycles, a sample of the filtered lubricant is obtained and the defoam properties are measured using ASTM D892.

[0023] The test methods disclosed herein are useful for simulating the conditions a lubricant will experience in the field, which may be better understood with reference to
30 the following examples.

EXAMPLES

[0024] The defoam characteristics using an aquilot of commercially available lubricant samples (Examples 1 and 2) are tested in a laboratory setting. Examples 1 and 2 are different commercial lubricant products comprising the same base oil, but different

additive packages, including polyacrylate antifoams. First, a portion of the samples are tested prior to filtration using ASTM D892 – Standard Test Method for Foaming Characteristics of Lubricating Oils (hereinafter referred to as “D892”).

[0025] For the D892 test, the fluid is heated to 49 °C to remove any thermal history.

5 A portion of the fluid (190 mL) is then transferred to a transparent 1000 mL graduated cylinder and allowed to cool to 24 °C. As soon as the fluid reaches 24 °C, air is blown through the sample at a rate of 94 mL/min for 5 minutes. After 5 minutes, the air source is turned off and the volume of foam (mL) is recorded immediately. The sample is then allowed to settle for 10 minutes, and the volume of foam (mL) is recorded again. This is referred to as Sequence I in the ASTM D892-18 test.

[0026] Meanwhile a second portion of the fluid (180 mL) is transferred to a transparent 1000 mL graduated cylinder and heated to 93.5 °C. When the fluid has reached thermal equilibrium, air is blown through the sample at a rate of 94 mL/min for 5 minutes. After 5 minutes, the air source is turned off and the volume of foam (mL) is recorded immediately. The sample is then allowed to settle for 10 minutes, and the volume of foam (mL) is recorded again. This is referred to as Sequence II in the ASTM D892-18 test.

[0027] The same sample used in Sequence II is then allowed to cool back to 24 °C. As soon as the fluid reaches 24 °C, air is blown through the sample at a rate of 94 mL/min for 5 minutes. After 5 minutes, the air source is turned off and the volume of foam (mL) is recorded immediately. The sample is then allowed to settle for 10 minutes, and the volume of foam (mL) is recorded again. This is referred to in the ASTM D892-18 test as Sequence III.

[0028] A portion of the commercial lubricants were then charged to a Siemens filtration test rig to test their filterability using Flender’s Procedure of testing the filterability of oil/fluid used in Flender gear units (FFT 7300 _ Revsion 3, hereinafter “FFT 7300) and their foam characteristics were tested as specified in FFT 7300 using Flender ISO 12152 - Lubricants, industrial oils and related products — Determination of the foaming and air release properties of industrial gear oils using a spur gear test rig — Flender foam test procedure (hereinafter referred to as “Flender Foam”). All of the steps in the FFT 7300 procedure were followed. The Siemens filtration test rig has a sealed observation window equipped with a scale (in ml) on the front of the gearbox housing. For the Flender foam test, the gear motor is turned on for 300 (+/- 5) seconds, then stopped. The volume of foam is observed using the observation window and scale and recorded at various time intervals (may be up to 90 minutes). A volume of foam of 15 ml at 1 minute

is considered a failure. A volume of foam of 10 ml at 5 minutes is considered a failure. The laboratory results of the defoam tests for the commercial lubricants are shown in Table 2.

Table 2

D892 Pre filtration (ml)	Example 1	Example 2
D892 Sequence I*	0; 0	0; 0
D892 Sequence II*	0; 0	0; 0
D892 Sequence III*	0; 0	0; 0
Siemens Filtration (FFT 7300)	pass	pass
SOT ¹ . Total volume at 1 min (ml 15 max)	6	5
SOT. Air-oil dispersion at 5 min (ml 10 max)	6	4
Step 3 ² . Total volume at 1 min (ml 15 max)	8	7
Step 3. Air-oil dispersion at 5 min (ml 10 max)	6	4
Step 4 ³ . Total volume at 1 min (ml 15 max)	9	10
Step 4. Air-oil dispersion at 5 min (ml 10 max)	8	6

- 5 * - The data includes the volume of foam (mL) that was recorded immediately after the air source was turned off and after settling for 10 minutes in “immediate; settled” format.
 1 – SOT = Start of Test; unfiltered lubricant at 25 °C
 2 – Third test step of FFT 7300 (lubricant temp 80°C; run time 7.5 hours; total run time 22.5 hours)
 10 3 – Fourth test step of FFT 7300 (lubricant temp 80°C; add 1.5 ml distilled water to oil; run time 4.5 hours total run time is 27 hours)

[0029] As shown in Table 2 above, both commercial lubricants, Examples 1 and 2, do not exhibit any foaming tendencies in a laboratory setting using the D892 test prior to
 15 filtration and passed the Siemens filtration using the known FFT 7300 test method. However, Example 1 exhibited foaming tendencies in the field. Example 2 has no known foaming tendencies or other issues in the field.

[0030] Despite passing the known D892 and FF 7300 defoam tests in the laboratory, Example 1 lost its defoaming tendencies in the field. Four field samples of Example 1 were
 20 obtained (Field Drains 1 through 4) and subjected the D892 defoam test. The defoam properties of the lubricant were permanently affected by their use in real world applications in a manner that was not detectable or predictable using the known D892 and FFT 7300 laboratory tests.

[0031] A fresh sample of commercial lubricant, Example 1, was obtained and sub-
 25 jected to the filtration and D892 test method disclosed above using the Siemens Test Rig. Another aquilot was subjected to filtration in a Siemens test rig and the defoam properties of the aquilot was measured at 25°C as specified in the FFT 7300 test.

[0032] The D892 tests of the field samples and the laboratory samples using the known FFT 7300 test and the filtration and foam test method disclosed herein are shown in Table 3 below.

Table 3 – Example 1 – Inventive Method and Field Drain Correlation

Post Filtration Results (mL)	Inventive Method ¹	Field Drain 1 low turbulence; fine filtration	Field Drain 2 low turbulence; fine filtration	Field Drain 3 low turbulence; fine filtration	Field Drain 4 high turbulence; fine filtration
D892 Seq I*	0; 0	0; 0	10; 0	20; 20	510; 470
D892 Seq II*	700; 650	690; 600	580; 70	550; 410	650; 600
D892 Seq III*	30; 0	0; 0	0; 0	20; 20	600; 570
FFT 7300 (at 25°C)	Pass	-	-	Pass	

- 5 * - The data includes the volume of foam (mL) that was recorded immediately after the air source was turned off and after settling for 10 minutes in “immediate; settled” format.
1 – Inventive Method using Siemens test rig

[0033] As shown in Table 3 above, the test results for Example 1 using the inventive method disclosed herein is more closely correlated with the field drain samples than the known laboratory test methods as shown in the data for Example 1 in Table 2. Also, the modified Siemens filtration test rig (and FFT7300 test) alone is not a good predictor of field performance, however, when combined with D892 the modified Siemens filtration rig correlates well with field performance. Thus, the inventive methods disclosed herein are a better predictor of a lubricant’s field performance than the known laboratory tests methods prior to the present disclosure.

[0034] The same filtration and foam test method disclosed herein were also applied to other laboratory filtration test rigs. The defoam test results of Examples 1 and 2 using the disclosed methods in different tests rigs is shown in Table 4 below.

Table 4 – Test Rig to Test Rig Correlation

Inventive Method Test Results	Siemens Rig	ZF SSP180 Rig 1	ZF SSP180 Rig 2	ZF GK2
Example 1				
D892 Seq I*	0; 0	0; 0	0; 0	0; 0
D892 Seq II*	700; 650	550; 420	660; 420	700; 610
D892 Seq III*	30; 0	20; 20	0; 0	0; 0
Example 2				
D892 Seq I*	0; 0	0; 0	0; 0	0; 0
D892 Seq II*	280; 0	260; 0	280; 0	170; 0
D892 Seq III*	0; 0	0; 0	0; 0	0; 0

* - The data includes the volume of foam (mL) that was recorded immediately after the air source was turned off and after settling for 10 minutes in “immediate; settled” format.

** These results a copied from Table 3 above.

[0035] Example 1 showed poor performance in all the test rigs using the inventive methods disclosed herein, which also correlates with the defoam performance with Example 1 observed in the Field Drains as shown in Table 3. It is noted that the Siemens test rig results in Table 3 are copied into Table 4 for easy reference purposes.

[0036] There are no known problems with the defoam performance of Example 2 in the field. Example 2 also showed good performance in all the test rigs using the inventive methods disclosed herein.

[0037] The data in Table 4 shows that the inventive methods disclosed herein results in similar results for all test rig types. Further, the data in Table 4 correlates well with the observed performance of Examples 1 and 2 in the field. The data shows that Example 1 failed in the laboratory setting using the inventive test methods, which is what was observed in the field, but not what was observed using the known laboratory test methods prior to the present invention. Example 2 showed good performance using the inventive method and also has no known defoam issues in the field. It therefore follows that the inventive test methods disclosed herein are a good predictor of field performance of lubricant formulations, no matter what type of test rig is used.

[0038] Each of the documents referred to above is incorporated herein by reference, including any prior applications, whether or not specifically listed above, from which priority is claimed. The mention of any document is not an admission that such document qualifies as prior art or constitutes the general knowledge of the skilled person in any jurisdiction. Except in the Examples, or where otherwise explicitly indicated, all numerical quantities in this description specifying amounts of materials, reaction conditions, molecular weights, number of carbon atoms, and the like, are to be understood as modified by the word "about." It is to be understood that the upper and lower amount, range, and ratio limits set forth herein may be independently combined. Similarly, the ranges and amounts for each element of the invention can be used together with ranges or amounts for any of the other elements.

[0039] As used herein, the transitional term "comprising," which is synonymous with "including," "containing," or "characterized by," is inclusive or open-ended and does not exclude additional, un-recited elements or method steps. However, in each recitation of "comprising" herein, it is intended that the term also encompass, as alternative embodiments, the phrases "consisting essentially of" and "consisting of," where "consisting of" excludes any element or step not specified and "consisting essentially of" permits

the inclusion of additional un-recited elements or steps that do not materially affect the basic and novel characteristics of the composition or method under consideration.

[0040] While certain representative embodiments and details have been shown for the purpose of illustrating the subject invention, it will be apparent to those skilled in this art that various changes and modifications can be made therein without departing
5 from the scope of the subject invention. In this regard, the scope of the invention is to be limited only by the following claims.

What is claimed is:

1. A method of evaluating the defoam performance properties of a lubricant comprising:
 - a) using a filtration test rig, wherein the filtration test rig (10) comprises a heated tank (12) equipped with heat source and a temperature sensor and wherein the heated tank is fluidly connected to a circulating pump (18) and a filter housing (22) equipped with a filter having a pore size equal to or less than 25 microns;
 - b) filling the heated tank with the lubricant to at least 50% capacity;
 - c) heating the lubricant to at least 80 °C;
 - d) circulating the lubricant through the filtration test rig using the circulation pump so that the lubricant passes through the filter 5,000 times to obtain a filtered lubricant;
 - e) obtaining a sample of the filtered lubricant; and
 - f) measuring the defoam properties of the obtained sample of the filtered lubricant using ASTM D892 to obtain post filtration results.
2. The method of claim 1, wherein the filter has a pore size equal to or less than 10 microns.
3. The method of claim 1 or 2, wherein the lubricant comprises at least one polyacrylate antifoam.
4. The method of any one of claims 1 to 3, wherein the lubricant comprises at least one silicon-containing antifoam.
5. The method of any one of claims 1 to 4, wherein the lubricant is circulated through the filtration test rig at a flow rate of 1 to 15 liters per minute.
6. The method of any one of claims 1 to 5, wherein an aliquot of the lubricant is tested using ASTM D892 prior to the filling step b) or the circulation step d) to obtain pre filtration results and comparing the pre filtration results to the post filtration results of step f).
7. The method of any one of claims 1 to 6, wherein the filtration test rig is flushed with an aliquot of the lubricant prior to circulation step d).
8. The method of any one of claims 1 to 7, wherein the heated tank is heated with a jacket, a heat exchanger, and/or a direct immersion heater.