Particle Counting Methodologies

Introduction

Fluid cleanliness is an important indicator of system condition that, when used as part of a wholistic fluid monitoring program, can prolong component life and warn of eminent failure. In this context, lubricant cleanliness is not a general term, but a specific, quantitative value that describes the distribution and size of particles present in the fluid (for more on cleanliness reporting refer to ISO 4406). Since the most damaging particle in lubricants are microscopic, measuring fluid cleanliness requires special equipment and careful sample handling.

Counts of particles in lubricants were initially performed by microscopy. However, particle counting technologies have advanced and fluid cleanliness evaluations (particle counts) can now be obtained from nearly all commercial oil analysis laboratories and from equipment on-site at many facilities. The variety of test methods available and impact of lubricant formulation on results make it important to understand the strengths and weaknesses of the selected particle counting methodology before acting on fluid cleanliness information.

Optical Particle Counting Technologies

Light Extinction Particle Counters

Optical particle counters are by far the most common instruments in use and there are two main types. Light extinction (LE) and direct imaging. Of the two variants, light extinction particle counters are prevalent technology. Commonly referred to as laser particle counters, these counters work on a simple principle that relates a voltage change to particle size. Light passes through a narrow stream of sample onto a photocell. When a particle blocks the light, photoreceptors experience a voltage change proportional to the size of the particle. The magnitude of the voltage change provides information on the size of the particle, but not the shape (Figure 1). Particles counted by light extinction are sized by an equivalent diameter method. The size of each particle is reported as the diameter of a spherical particle that would cause the recorded voltage to drop [1].

Light extinction counters are widely available, relatively inexpensive, and repeatable. As a result, they are the most widely used particle counting technology. However, as light extinction particle counters record each voltage drop as particulate, anything in the lubricant that does not have the same index of refraction as the bulk fluid is reported as particulate. This leads to a tendency for these types of counters to over report particles – counting water, air, and additives (especially foam inhibitors) as particles.

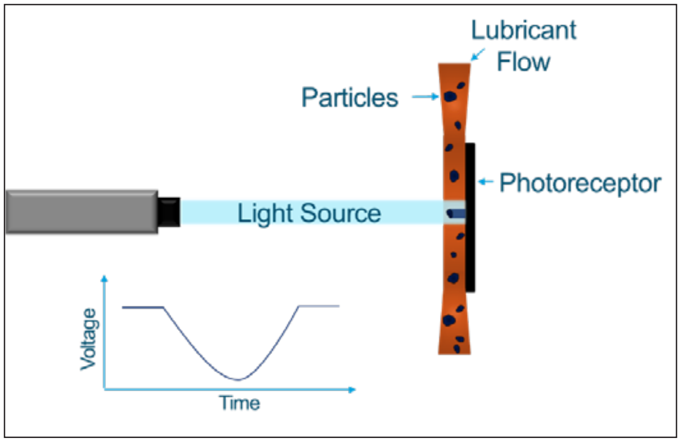

Figure 1: *Automatic particle counting under the light extinction principle. A particle enters the light path and blocks a portion of the light reaching the photocell resulting in a drop in voltage. The change in voltage is proportional to the size of the particle.[2]*

Direct Imaging Particle Counters

Like light extinction particle counters, direct imaging counters pass light through a stream of fluid. However, rather than a voltage change, direct imaging counters capture an image of the particle. The particle is sized by both maximum chord and equivalent diameter (Figure 2).

For particles that are large enough, the image is then analyzed by advanced computer algorithms that further evaluate the particle aspect ratio, perimeter length, and circularity [3] to classify particles as fatigue, sliding, and cutting wear; the system is also able to differentiate water, air bubbles, fibers, and other nonmetallic contaminants for particles greater than 20 microns [4]. However, water, air, and additives between 4 and 20 microns are reported as particles.

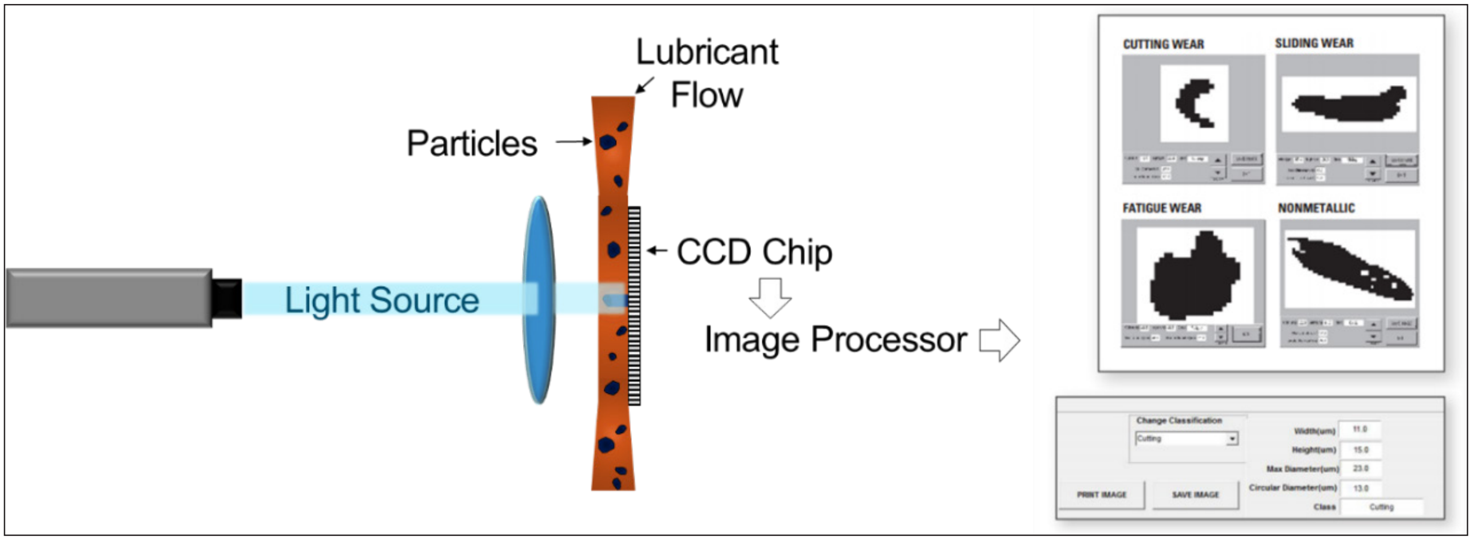

Figure 2: *Schematic image of the LaserNet Fines direct imaging particle counter. As fluid passes through the viewing area, a laser is pulsed, and an image of the particles are captured on a CCD sensor. An artificial neural network analyzes the pixels and classifies the particles according to size and type.*

Additive Impacts on Optically Detected Lubricant Cleanliness

It has been well documented that additives, particularly foam inhibitors (FI), in finished lubricants interfere with the ability of optical particle counters to accurately report the particulate contamination of new lubricants [5], [6], [7]. It is accepted in the industry that additive induced particle counts are not real contaminants as they are intentionally added to the lubricant and will not cause wear damage. However, since most optically based particle counters cannot tell the difference between hard and soft particles, users face a frustrating situation when trying to achieve cleanliness targets without impacting lubricant performance (See [8] for more on the risks associated with cleaning finished lubricants).

Mitigating Additive Impacts on Optically Detected Fluid Cleanliness

Sample Dilution

Efforts have been made to identify methods to mitigate the impact of additives on detected lubricant cleanliness. ASTM D7647 [9] and ASTM D7596 Appendix X1 [3], detail a sample dilution process designed to minimize the impacts of water and other "soft" particles. This technique can eliminate a sizable portion of the additive interference. Figure 3 illustrates the impact of dilution on particle counts of a group II base oil with and without FI. Without utilizing dilution methods, addition of the FI increases the particle count of the sample significantly, after dilution, the sample returns to its initial cleanliness. However, dilution is not always effective, and the additional sample preparation provides a chance to introduce contamination.

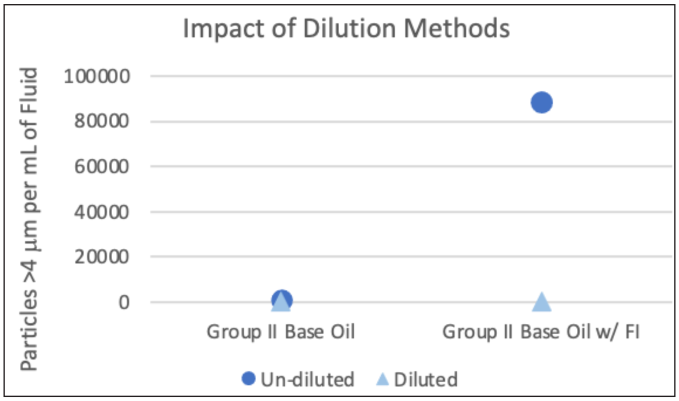

Figure 3: *impact of ASTM D7647 dilution on particle counts of a Group II Base Oil with and without FI. On this freshly blended sample, dilution nearly completely mitigates the impact of additives on the optically detected cleanliness. However, studies have shown that the efficacy of dilution can decrease over time.*

Particle Count Stability

Experience has shown that additive induced particle counts can significantly increase over time, as dispersed additives agglomerate and become 4-microns or larger. For example, in a 2021 study, several bulk samples were carefully stored to minimize external contamination and counted with a direct imaging (DI), particle counter weekly for 6 months. A statistical analysis of the results was performed to determine if there were significant changes in particle counts over time.

- Both diluted and undiluted particle counts for the neat Group II Base Oil control sample remained relatively stable and did not increase over the course of the study (Figure 4).
- The undiluted counts of a second sample (Figure 5), a Group II Base Oil with a moderate FI treatment, increased over the course of the study; the diluted samples did not. This indicates that although FI induced particle counts were growing, the dilution method retained its efficacy throughout the life of the study.
- The last sample, an ISO 68 Gear Oil (Figure 6), showed statistically significant growth in both the undiluted and diluted particle counts. The growth of particle counts by both methods, when coupled with the stable control sample, indicates that additive interference grew significantly and overcame the dilution method's ability to mitigate its impact.

This study provides only a glimpse into potential particle count variation over time. The shelf life of finished lubricants can commonly reach ten times the length of the study (5 years) and factors such as storage temperature impact the rate of additive agglomeration. In real world scenarios, it is difficult, if not impossible, to know the efficacy of dilution methods on optically detected particle counts for a particular sample.

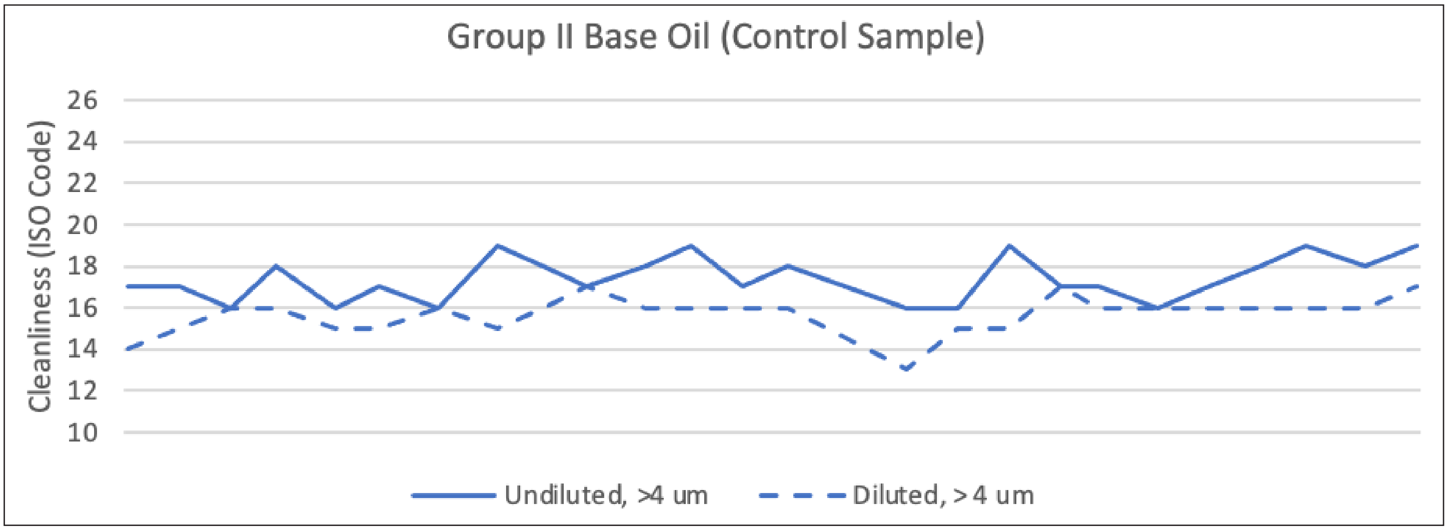

Figure 4: *Comparison of DI particle counts over a 6-month period measured with and without dilution for a Group II Base Oil. Both diluted and undiluted particle counts remain stable; particles were not introduced and did not significantly agglomerate over the course of the test.*

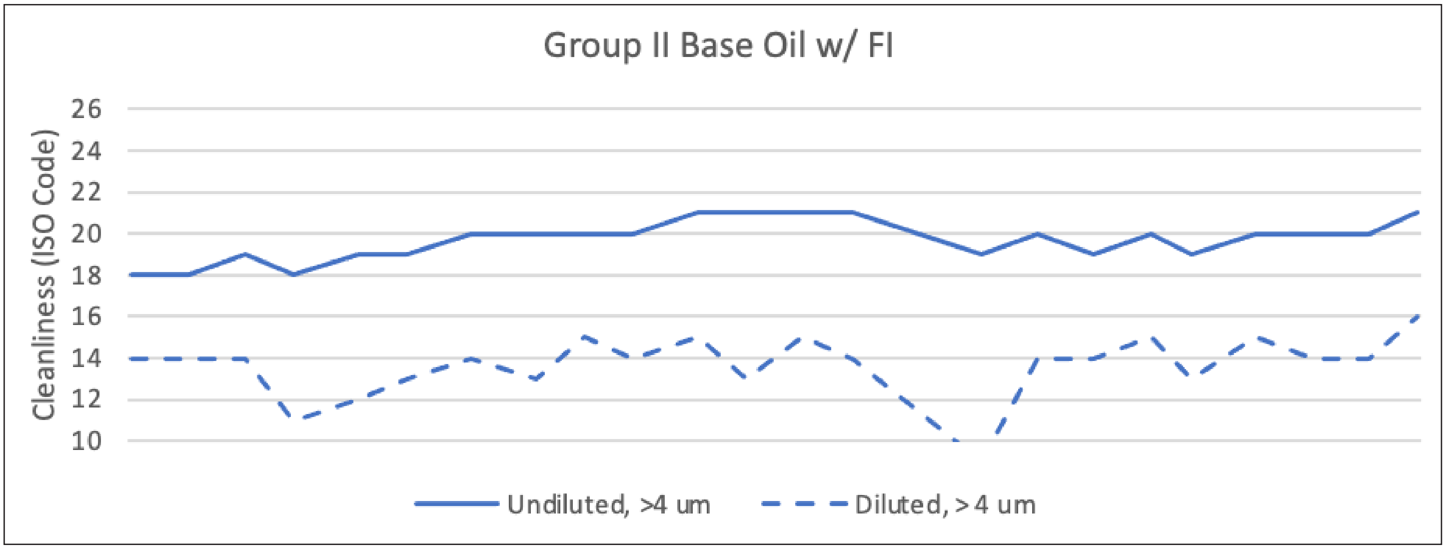

Figure 5: *Comparison of DI particle counts over a 6-month period measured with and without dilution for a Group II Base Oil w/ Foam Inhibitor. Undiluted particle counts increased significantly over the course of the study, but dilution was effective at mitigating the impact of the FI (diluted particle counts did not significantly increase).*

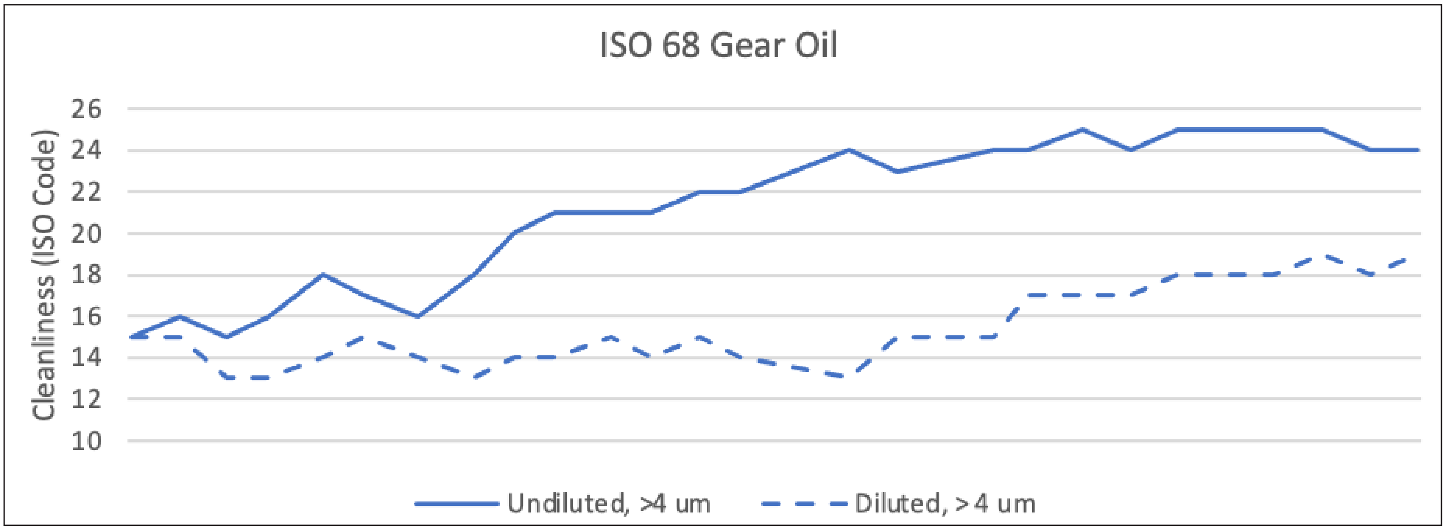

Figure 6: *Comparison of DI particle counts over a 6-month period measured with and without dilution for an ISO 68 Gear Oil. Both undiluted and diluted particle counts experienced a statistically significant increase over the 6-month study. Initially, diluted counts were stable indicating continued dilution efficacy; however, after approximately 3-months the dilution method was no longer able to fully mask the particle agglomeration.*

Alternative Particle Counting Techniques

Microscope Particle Counts

With this technique a known volume of lubricant sample is carefully filtered through a membrane. The membrane is placed in a filter holder and examined by a technician at multiple magnifications to determine the number of particles/mL in several size ranges. The counting portion of the process can be automated by image analysis. In this case, a digital image of the magnified membrane is captured, and the software counts and reports the particles.

Whether the count is performed manually or by image analysis, the lab must check the count for validity. If the validation is not successful, the count is repeated. If the validation succeeds, the count is reported. See ISO 4407 for more detail on the validation process.

Microscope particle counts are often considered to be the most accurate as counts obtained from this method are less influenced by “soft” particles such as water, air, and lubricant additives. However, counting particles in this manner is time consuming, requires experienced personnel, and is now rarely utilized outside of research activities.

Patch Comparison Tests

Patch comparisons are not quantitative tests but rather a qualitative visual comparison. A sample is filtered through a membrane and dried. A microscopic image of the membrane is captured and compared to a collection of reference patch images created from fluids with a "known" cleanliness (Figure 7).

Patch comparison tests do not provide quantitative results on their own, but when evaluated by an experienced operator provide a quick screening tool to indicate if further testing is warranted.

Pore Blockage

Pore blockage particle counters are typically used for in-service lubricants and can be performed on-site or by a lab. The sample lubricant is directed through a fine mesh in which the particles will accumulate which increases the differential pressure across the mesh. The particle count distribution is estimated by extrapolation from the rate of pressure increase and mesh size. Pore blockage particle counts are relatively uncommon.

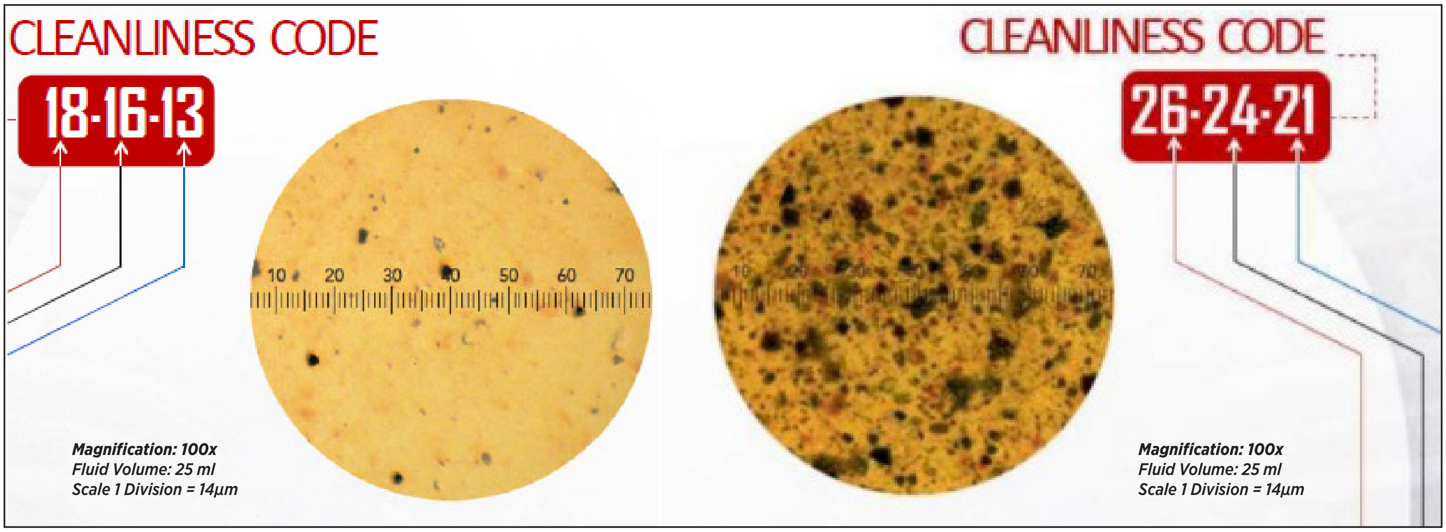

Figure 7: *Source: Example of patch comparison images from Oil Filtration Services Fluid Cleanliness Comparison Guide [10]*

Method and Laboratory Variations

Given all the challenges associated with collecting and analyzing a sample, it is not hard to imagine that particle count results are well known to be somewhat inconsistent. Published test methods commonly indicate repeatability approaching +/- 40% of the particle count; in the field it is often more common for samples to vary in cleanliness by an ISO 4406 code or more.

Figures 8-10 compare test results for samples taken from common bulk fluids and tested at several sites. Figure 8 illustrates the potential variability between particle counting methods and equipment, even when counts are performed at the same lab by the same technician. At the greater than 4 µm level, for example, employing dilution to mask the contribution of soft particles resulted in a reduction of up to an ISO Code. Utilizing a particle counter from a second manufacturer resulted in an additional reduction of up to three additional ISO Codes.

Cleanliness can also vary significantly when performed by different labs utilizing the same technology and method. Figure 9 compares the ISO cleanliness codes for five samples counted using the same technique (Light Extinction w/ Dilution) at three labs. There are several things to note. First, reported cleanliness varies by up to two ISO Codes for samples that were taken from the same bulk fluid and tested using the same method. Second, none of the three labs was uniformly higher or lower than the others; the variation appears to be the result of test variation rather than lab capabilities. Lastly, these results are comparable to the results from Direct Imaging Counter 1 (DI-1) with and without dilution. The similarity in DI and LE counter results is not surprising. Most direct imaging counters only discriminate between hard and soft particles that are greater than 20 µm in size. Since these samples were all new products, there are few wear or other large particles, minimizing the impact of direct imaging technology on detected cleanliness.

As optical methods are heavily influenced by additive interference, microscope particle counts (ISO 4407) provide a good measure of solid contaminates in a sample. Since the counts measure only objects captured on a filter patch (0.8 µm), they are minimally impacted by additives or other soft particles. The light extinction and direct imaging results previously discussed are presented in Figure 10 along with results for microscope particle counts for the same samples. Direct Imaging Counter-2 (DI-2) appears to be the most effective at mitigating the impact of soft particles on the detected cleanliness and does not vary significantly from the contour of the microscope counts. However, the remaining particle counts deviate from the microscope counts by as much as 8 ISO Codes at the >4 µm level. It is also important to note that the magnitude of the disparity between the microscope and optical particle counts correlates well to the amount of additive in the respective products. The base oil and turbine oil, for example, have the least variance and are formulated with 0 - < 2 % additive, while the heavy-duty engine oils can be formulated with more than 20% additive and have the largest disparity.

The amount of variation that exists in cleanliness measurements can be frustrating for many users. Determining which methodology is "right" can be very subjective and requires a balance between turnaround time, cost, and purpose. For most users, particle count analysis will provide the most benefit when results are taken in a consistent manner from a consistent location, analyzed using the same technique at the same laboratory (for more on trend analysis, see ASTM D7669 [11].

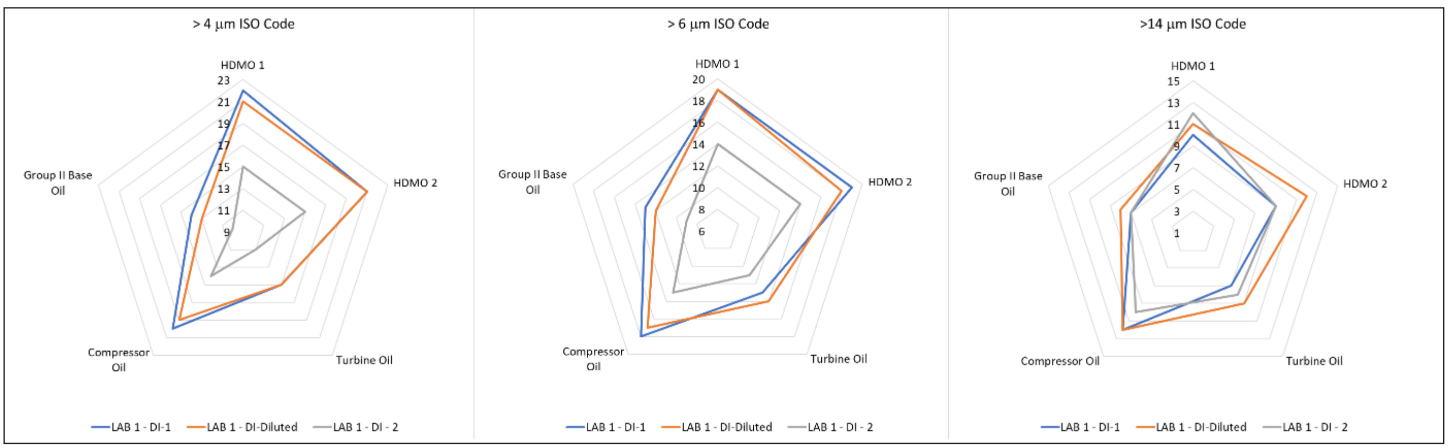

Figure 8: *Comparison of lubricant cleanliness results for five lubricants using direct imaging particle counters from two manufacturers, DI-1, and DI-2. Analyses with the DI-1 particle counter were also performed using sample dilution to mask the contribution of soft particles. DI-2 reported cleanliness was significantly better for >4 and >6 µm sizes, than that reported by DI-1.*

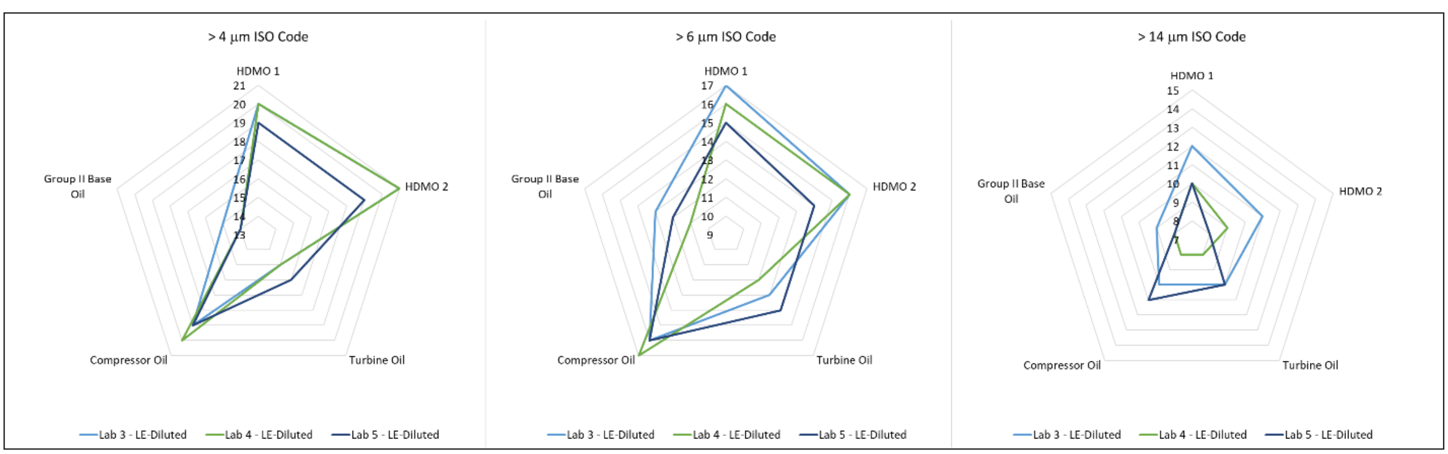

Figure 9: *Comparison of lubricant cleanliness measurements of five lubricants using light extinction particle counters with dilution (ASTM D7647). Cleanliness measured with the same technique varied by up to two ISO Codes across the three labs.*

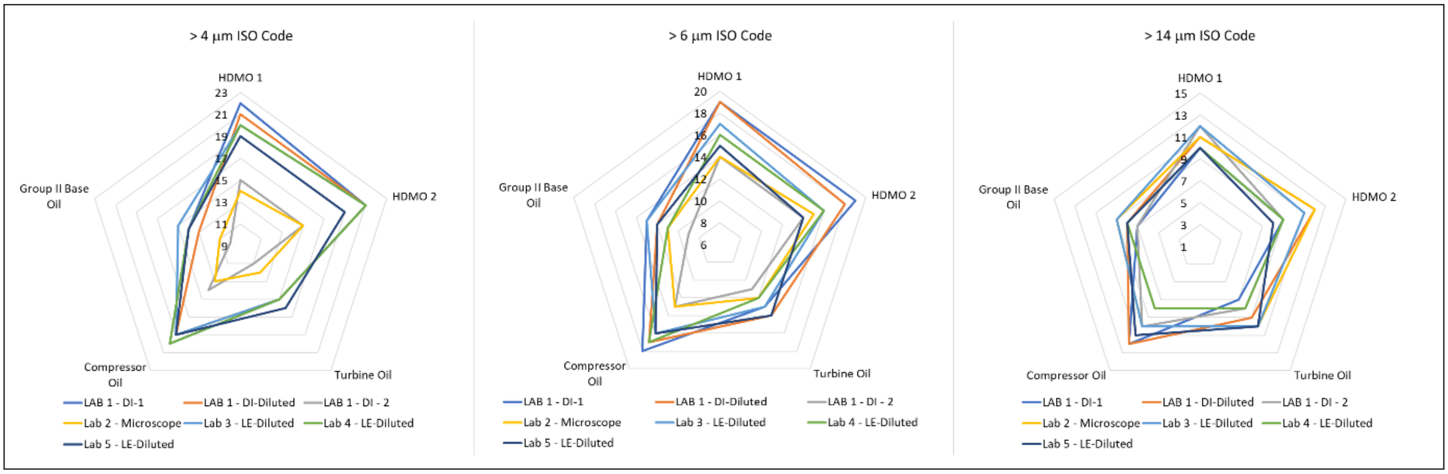

Figure 10: *Comparison of lubricant cleanliness measurements to the lubricant cleanliness as measured by microscopic examination (ISO 4407). Microscope particle counts are most resistant to additive interference and are generally considered the most reliable measure of hard particle contamination. Cleanliness as measured by DI-2 was consistent with counts by microscope indicating that amongst optical counters, they were least impacted by additive interference.*

Note: ISO 4406 specifies that microscope particle counts be reported in the size ranges > 5 µm and > 15 µm. In the plots above the > 5 µm ISO Code for the microscope counts is presented on the > 4 and > 6 µm plots, while the >15 µm code is presented on the >14 µm plot.

Implications

It is becoming common knowledge that clean oil is a critical step in the process to maximize equipment life and many users now filter oil prior to use. It has been said that we cannot improve what we cannot measure. While that is the case for lubricant cleanliness, measuring it is not always straight forward.

There are standards for particle counting and instrument calibration, but it is common to get different cleanliness results for the same sample from different labs, different particle counting instruments, or even the same lab and equipment. There are many contributing factors to particle count variation, including sample collection, sample preparation, and test method. Even lubricant formulation can play a role, as some technologies and/or instruments are more sensitive to additive interference.

Care must be taken to ensure that cleanliness results are well understood prior to taking corrective action. Filtration is the most common action taken to achieve, or restore, lubricant cleanliness. However, some additives can be removed by filtration, and this can negatively impact its performance in the application. Moreover, when the risk of contamination from unnecessary handling of the lubricant is added to the equation, filtration is a potentially expensive proposition that should not be undertaken lightly.

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[2] Noria Corporation, "The Low-Down on Particle Counters," *Practicing Oil Analysis Magazine*, Jul-2002.

[3] J. E. Tucker, J. Reintjes, M. D. Duncan, T. L. McClelland, L. L. Tankersley, A. Schultz, C. Lu, P. L. Howard, T. Sebok, C. Holloway, and S. Fockler, "LaserNet Fines Optical Oil Debris Monitor," in *Joint Oil Analysis Program International Condition Monitoring Conference*, 1998.

[4] ASTM International, "ASTM D7596-14 Standard Test Method for Automatic Particle Counting and Particle Shape Classification of Oils Using a Direct Imaging Integrated Tester," West Conshohocken, PA, 2014.

[5] P. W. Michael, T. S. Wanke, M. a. McCambridge, S. Tung, B. Kinker, M. Woydt, and S. W. Dean, "Additive and Base Oil Effects in Automatic Particle Counters," *J. ASTM Int.*, vol. 4, no. 4, 2007.

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[8] Chevron ISOCLENA Certified Lubricants, "Technical Bulletin: Impacts of Filtration on New Lubricant Performance," Richmond, CA, 2021.

[9] ASTM International, "ASTM D7647-10 Standard Test Method for Automatic Particle Counting of Lubricating and Hydraulic Fluids Using Dilution Techniques to Eliminate the Contribution of Water and Interfering Soft Particles by Light Extinction," West Conshohocken, PA, 2018.

[10] Rocky Mountain Filtration Solutions, "Fluid Cleanliness Comparison Guide," Commerce City, CO

[11] ASTM International, "ASTM D7669-15 Standard Guide for Practical Lubricant Condition Data Trend Analysis," West Conshohocken, PA, 2015.