ISOCLEAN® CERTIFIED LUBRICANTS

Chevron

TECHNICAL BULLETIN

ISOCLEAN® CERTIFIED LUBRICANTS TESTING AND MEASURING LUBRICANT CLEANLINESS

Introduction

The marketplace is aware that particulate contamination can lead to increased wear, degradation of lubricant and premature component failure. The cost of premature wear and failure is significant compared to the cost of a proactive/preventative maintenance program. Particulate contamination may come from a variety of sources such as; environmental (dust), maintenance induced (lubricant top-off), water ingression (vapors or leaks), wear generated debris (from operation), and improper lubricant storage/handling (lack of air-management). As more companies work to resolve the contaminant issue the demand for filtering lubricants and tools to manage ingression points are increasing. To improve any issue, a form of measurement is needed. Thus, the premise of this paper is to discuss how particulate contamination is measured and create awareness around some of the challenges.

How is Particulate Contamination Measured Today?

Studies suggest particles ranging in size from 2 to 20 micron cause 80-90% of abnormal wear in lubricated equipment [1]. Therefore, the lubricant industry has by in large moved to the ISO 4406 standard for measuring lubricant cleanliness, which reports fluid contamination at three sizes equal to or greater than: 4 µm (micron), 6 µm and 14 µm and greater in a 3-digit code such as XX/YY/ZZ. Each number represents a contaminate level code for the correlating particle size on a scale and is written such as 20/17/13. The code correlates to a table showing how many of that size particle are represented in a 1 milliliter sample of fluid. It is important to highlight an increase in just 1 ISO Code means up to double the number of contaminants, thus as the code moves higher on the scale, the amount of contamination in the sample becomes more significant. In the event an entity only reports a 2-digit code, that means the first level of contaminate reporting, the size 4 µm has fallen off and only the later two measurements are reported (size 6 and 14 µm).

ISO 4406:99 Code Chart

- **XX = total number of particles > 4 µm**
- **YY = total number of particles > 6 µm**
- **ZZ = total number of particles > 14 µm**

	Particles/ml	ISO Code
> 4 µ	9,721	20
> 6 µ	1,254	17
> 10 µ	326	
> 14 µ	73	13
> 21 µ	12	
> 38 µ	5	
> 70 µ	0	
> 100 µ	0	

More than (p/ml)	Up to and including (po/ml)	ISO Code
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
1,300	2,500	18
640	1,300	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

ASTM/ISO Standards [2,3]

Next, there are various types of particle counters in the marketplace used to evaluate a lubricant's cleanliness level. ASTM International and the International Standards Organization (ISO) develop and publish technical test standards for these instruments which are noted herein.

- **Estimated Particle Count by Patch comparison**
  - *A trained technician utilizes a microscope to evaluate the sample against the Industry Fluid Cleanliness Comparison Guide to provide a reported cleanliness level*
- **Particle Count by Optical Microscope**
  - *ISO 4407 Fluid contamination — Determination of particulate contamination by the counting method using an optical microscope*
- **In-Line Laser**
  - *ASTM D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products*
  - *ISO 11171 Hydraulic fluid power—Calibration of automatic particle counters for liquids*
- **Laser Counters (e.g. Portable Lasers)**
  - *ASTM D6786 Particle Count in Mineral Insulating Oil Using Automatic Optical Particle Counters*
  - *ASTM D7647: Automatic Particle Counting of Lubricating and Hydraulic Fluids Using Dilution Techniques to Eliminate the Contribution of Water and Interfering Soft Particles by Light Extinction.*
  - *ISO 11500 Determination of the particulate contamination level of a liquid sample by automatic particle counting using the light-extinction principle - Second Edition*
  - *ISO 11171 Hydraulic fluid power—Calibration of automatic particle counters for liquids*
- **Pore Blockage**
  - *ISO 21018:3 Hydraulic fluid power—Monitoring the level of particulate contamination of the fluid—Part 3: Use of the filter blockage technique*
- **Direct Imaging Particle Counters (e.g. Laser Net Fines)**
  - *ASTM D7596 Standard test method for automatic particle counting and particle shape classification of oils using a Direct Imaging Integrated Tester*

Testing for Lubricant Cleanliness vs. Contamination

Selecting the right tool for the right job makes life easier and that principle holds true when it comes to testing for lubricant cleanliness or contamination. The chart below provides a generalized overview of the technology's effectiveness in measuring cleanliness using a stop light approach (green = good, yellow = ok and red = not good). Since there is no one perfect particle counting tool available, the Chevron ISOCLEAN® Certified Lubricants uses multiple tools to measure cleanliness; namely the patch, in-line laser and laser net fines. This repetitive testing enables more effective results to certify the new lubricant and ISO 4406 Cleanliness Level for the end use customer.

Testing a new lubricant cleanliness level vs. an in-service lubricant should require different instrumentation as the data needed and the technology effectiveness vary.

For example, the ASTM Journal reported three different particle counters were compared, in which the two laser particle counters using the light-blockage principle were found to produce phantom counts from additives, whereas the direct imaging laser particle counter was less susceptible [6]. Therefore, to ensure accurate and meaningful measurement, what you aim to measure impacts the tool and testing selection.

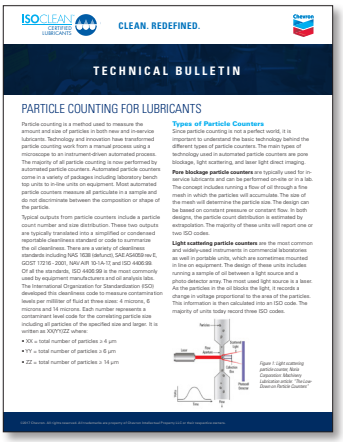
Particle Counting Methods and Effectiveness

Counter Technology	Approx. Cost	Measure Multiple Products	Air/Water/ Additive Counting Errors	Accuracy/ Responsibility	Count Small Particles	Ease of use	Particle Classification
ISOCLEAN® Patch test	$2,700	🟢	🟢	🔴	🟡	🟢	🟡
Optical Microscope	$50,000	🟢	🟢	🟢🟡	🟢	🔴	🟢
ISOCLEAN® In-line laser	$2,000	🔴	🔴	🟢	🟢	🟢	🔴
Portable laser	$11,000	🟢	🔴	🟢	🟢	🟢	🔴
Pore blockage	$25,000	🟢	🟢	🟡	🔴	🟡	🔴
ISOCLEAN® Laser Net Fines	$50,000	🟢	🟡	🟢	🟢	🟢	🟢

Mitigating False Positives: Additive Induced Particle Contamination, Soft Particles, Ghost Particles

When testing a new lubricant, it is important to note "Certain ingredients in a new lubricant sample will give positive readings in some of the particle counting instruments, making new oil seem to contain particulate contamination even though the lubricant has not been used [4]." This phenomenon is known by several names, additive induced particle contamination, phantom particles, ghost particles or soft particles. The bottom line is this: depending on the final lubricants formulation, base oil and additive package and particle count testing methodology, the results will vary. [5] Therefore, it is critical to collaborate with the lubricant formulator when measuring or setting a new lubricant cleanliness level. There are standardized procedures labs may use to mitigate the soft particles from inflating particle count readings. Having a lubricant provider who understands the new lubricants' filterability regarding filter size, flow rate, temperature and maximum number of passes is critical. Chevron has seen important additives stripped out on both small and larger micron filters. Knowing the right way to filter, utilizing the appropriate measurement procedures, provides control on variables otherwise largely unknown.

ISOCLEAN® Certified Lubricants–An Industry Disruptor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TECHNICAL BULLETIN

PARTICLE COUNTING FOR LUBRICANTS

Particle counting is a method used to measure the amount and size of particles in both new and in-service lubricants. Technology and innovation have transformed particle counting work from a manual process using a microscope to an instrument-driven automated process. The majority of all particle counting is now performed by automated particle counters. Automated particle counters come in a variety of packages including laboratory bench top units to in-line units on equipment. Most automated particle counters measure all particulate in a sample and do not discriminate between the composition or shape of the particle.

Typical outputs from particle counters include a particle count number and size distribution. These two outputs are typically translated into a simplified or condensed reportable cleanliness standard or code to summarize the oil cleanliness. There are a variety of cleanliness standards including NAS 1638 (defunct), SAE AS4059 rev E, GOST 17216 - 2001, NAV AIR 10-1A-17, and ISO 4406:99. Of all the standards, ISO 4406:99 is the most commonly used by equipment manufacturers and oil analysis labs. The International Organization for Standardization (ISO) developed this cleanliness code to measure contamination levels per milliliter of fluid at three sizes: 4 microns, 6 microns and 14 microns. Each number represents a contaminant level code for the correlating particle size including all particles of the specified size and larger. It is written as XX/YY/ZZ where:

- XX = total number of particles ≥ 4 µm
- YY = total number of particles ≥ 6 µm
- ZZ = total number of particles ≥ 14 µm

Types of Particle Counters

Since particle counting is not a perfect world, it is important to understand the basic technology behind the different types of particle counters. The main types of technology used in automated particle counters are pore blockage, light scattering, and laser light direct imaging.

Pore blockage particle counters are typically used for in-service lubricants and can be performed on-site or in a lab. The concept includes running a flow of oil through a fine mesh in which the particles will accumulate. The size of the mesh will determine the particle size. The design can be based on constant pressure or constant flow. In both designs, the particle count distribution is estimated by extrapolation. The majority of these units will report one or two ISO codes.

Light scattering particle counters are the most common and widely-used instruments in commercial laboratories as well in portable units, which are sometimes mounted in line on equipment. The design of these units includes running a sample of oil between a light source and a photo detector array. The most used light source is a laser. As the particles in the oil blocks the light, it records a change in voltage proportional to the area of the particles. This information is then calculated into an ISO code. The majority of units today record three ISO codes.

Figure 1: Light scattering illustration from "The Lubrication Machinery Lubrication article "The Low-Down on Particle Counters"

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Chevron created the ISOCLEAN® Certified Lubricants program to meet the marketplace need for ultra-clean new lubricant. We know how time consuming and difficult it can be to safely filter new oil. This is why Chevron ISOCLEAN® Certified Lubricants Marketers have strict guidelines to ensure no harm comes to the final formulated product. Prior to a product being released as an ISOCLEAN® Certified Lubricants, a significant amount of work takes place to understand the formulation and appropriate methods to filter the oil, such as temperature, filter size, beta ratio, passes, etc. This is to ensure the lubricant can be cleaned without harming the final formulation or stripping out additives. This lengthy process in testing, re-testing, validation and program execution procedures is what allows Chevron to stand behind their products as Certified Clean at delivery. The variables have been controlled to the highest level to make sure new clean lubricants can be delivered at the requested ISO 4406 level.

The ISOCLEAN® Certified Lubricants laboratories that perform the ISO 4406 testing have strict protocols in place to ensure the lubricant is clean and to minimize counts of ghost particles. Round robin testing is also performed to ensure the repeatability of ISOCLEAN® Certified Lubricants' Laboratories; for published results, please read the Chevron Technical Bulletin "Particle Counting for Lubricants," which shows their success with repeatable results.

ISOCLEAN® Certified Lubricants Test Results


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CERTIFICATE *of* ANALYSIS

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This original Certificate of Analysis verifies that the product delivered with this certificate meets the ISO Cleanliness standards as indicated below.

Chevron Rando HD68 - ISOCLEAN Certified
ISO Cleanliness Code: 18/16/13

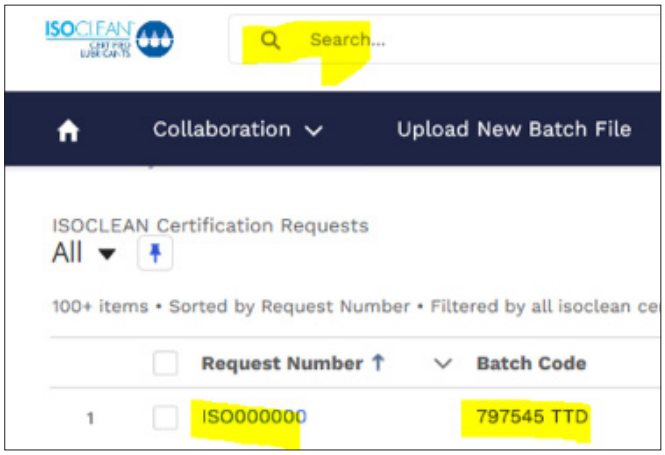
Date: 11/1/2014
Product Code: 254614
Volume Certified: 330 gallons
ISOCLEAN Certificate # 24831CHV1101141030

ABC Marketer
Orlando, FL
Joe ISOCLEAN - Technician

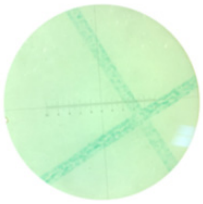
The Chevron ISOCLEAN® Certified Lubricants Program provides a certificate of analysis validating the new lubricants cleanliness meets the reported level. This should provide confidence to the end user in the lubricant cleanliness at the time of delivery. In the event there is concern or a desire to test the cleanliness level, we'd encourage working with the Chevron ISOCLEAN Certified Lubricants distributor. First, the distributor will keep a retain on hand and we also encourage end users to take a retain at the time of delivery off the transport vehicle. If end users desire to see the laboratory test, their lubricant supplier may provide them upon request or at the time of delivery.

However, some still choose to validate results on their own. Which is fine as long as there is an understanding that results may vary. Several factors may cause a variation in reported cleanliness results. It may be due to different lab instrumentation, a change in sample preparation, equipment calibration/operation or from design limitations, coincidence error or coincidence loss. Or, in some cases this may happen due to self-contamination when taking a new sample. These issues are all explained in our Technical Bulletin titled "Particle Counting for Lubricants," which was mentioned above. Another good resource is an Efficient Plants article titled, "Do We Really Know What We Are Measuring" [7]. The premise is about the risk of not understanding the different particle counting tools or counting methods. The article also indicates that inaccurate and unreliable particle counting can hinder a site's ability to make suitable decisions and incur significant costs in the process. In the event you experience a variance in particle count results and want more info, these are some simple steps to follow:

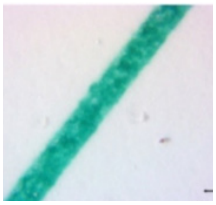
1) Gather the original laboratory paperwork from your ISOCLEAN Certified Lubricants Distributor. The Chevron ISOCLEAN Certified Lubricants system retains this information. It can be found by the ISO Certification Request number or Batch number as depicted below:



2) Request the patch test results from the ISOCLEAN Certified Lubricants Distributor. Evaluate the fluid comparison guide against your patch and the targeted ISO 4406 specification. When in doubt, the ISOCLEAN Certified Lubricants Distributor has a Patch Test Kit on hand and may be able to come out and perform a patch test on site.



Retain Patch



Laboratory Patch

Industry Fluid Comparison Patch

3) Evaluate how the sample taken for testing was pulled: for example, was a dirty towel used to wipe the nozzle, was the appropriate sample pulling procedure used, etc., because unintended self-contamination may occur and it's critical to be thorough.

4) If there is still doubt, ask the ISOCLEAN Certified Lubricants Distributor to provide the retain sample along with a new sample pulled while you and a Chevron representative are present. The local Chevron Business Consultant can complete a lubricant Technical Service Request and send in the appropriate sample volume for evaluation and re-certification.

In closing, it is critical to have the conversation with the end user about the ISOCLEAN Certified Lubricants process, testing and results. This instills confidence and makes trouble-shooting together easier in the event something has gone awry.

References:

(1) Simmons, R., *"It's All About Size,"* Practicing Oil Analysis Magazine, Noria, Tulsa, OK, January 2007)

(2) American Society for Testing Materials (ASTM), https://www.astm.org/Standard/standards-and-publications.html

(3) International Organization for Standardization (ISO,) https://www.iso.org/home.html

(4) Sander et al. (2009) *"The Effects of Lubricant Ingredients on New Hydraulic Oil Cleanliness,"* Journal of ASTM International, 6(1), pp 1–9).

(5) Paul W. Michael, Thomas S. Wanke, and Michael A. McCambridge, *"Additive and Base Oil Effects in Automatic Particle Counters,"* Journal of ASTM International, Vol. 4, No. 4 Paper ID JAI100941

(6) Michael et al. (2007) "*Additive and Base Oil Effects in Automatic Particle Counters,"* Journal of ASTM International, Vol. 4, No. 4. Paper ID JAI100941.

(7) Kathy, *"Do We Really Know What We Are Measuring,"* Efficient Plant, May 1, 2007. https://www.efficientplantmag.com/2007/05/do-we-really-know-what-we-are-measuring/

For further details, please contact your Chevron Representative.

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1-866-354-4476