Ghost Particles

Particle Counting Methods & Impact on ISO Codes

STLE Workshop – November 2023

Ghost Particles

Particle Counting Methods & Impact on ISO Codes

The **Authors**

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CINRG manufactures particle counting, and robotic instrumentation for the oil analysis industry.

HF Sinclair / Petro-Canada Lubricants

A manufacturer of world-class lubricants, specialty fluids and greases for over 30 years.

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An oil analysis laboratory and service provider for 57 years. The leader in oil analysis.

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Fluid Cleanliness and Ghost Particles

WHAT

Soft (or Ghost) particles are non-abrasive “particles” that are detected in the oil and are related to insoluble oxidative by-products and/or additives present in the lubricant

WHY

Soft (or Ghost) particles can cause laser light-scattering in optical particle counting instruments that lead to erroneously high ISO cleanliness codes

WHY

Optical Particle Analyzers cannot distinguish between these soft (ghost) particles and true abrasive contaminants like fibers, dust and dirt

HOW

By using the dilution method for particle counting (ASTM D7647) and the proper solvent, particle count results are more reproducible and repeatable

CINRG WEAR CHECK

PETRO-CANADA LUBRICANTS
AN HF SINCLAIR BRAND

Oil Filtration

Life Extension Tables – ISO Cleanliness Code & Moisture (ppm)

Clean & Dry Oil

↓

Increases MTBF

ISO Cleanliness

↓

Particle Counting

Machine life-extension table

Ref: Reliable Plant

MOISTURE	NEW MOISTURE LEVEL PPM (%)				
	1000 (0.1%)	500 (0.05%)	250 (0.025%)	100 (0.01%)	50 (0.005%)
5000	2.3x	3.3x	4.8x	7.8x	11.2x
2500	1.6x	2.3x	3.3x	5.4x	7.8x
1000		1.4x	2.0x	3.3x	4.8x
500			1.4x	2.3x	3.3x
250				1.5x	2.3x
100					1.4x

Ref: SKF / OSU

Ref: Lubrigard

CINRG WEAR CHECK

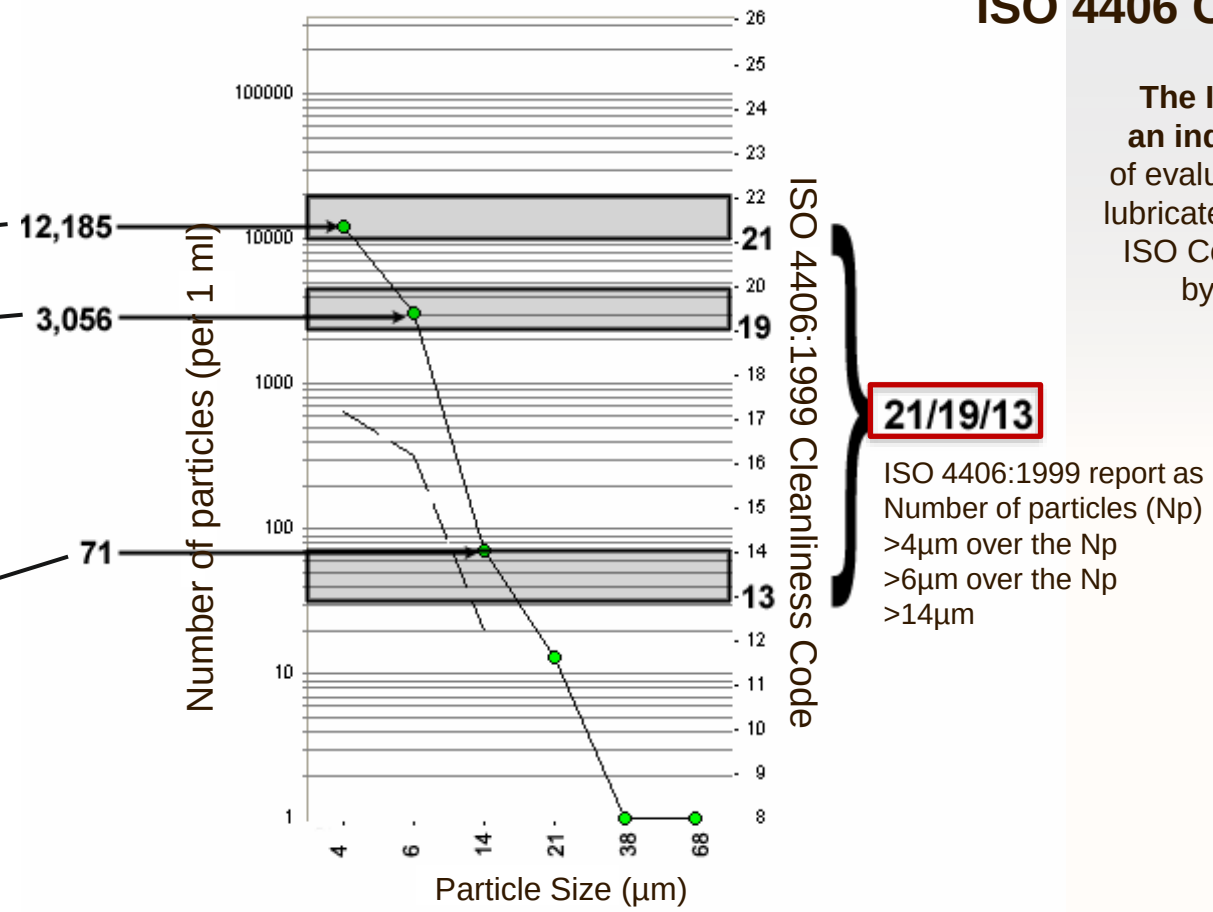
PETRO-CANADA LUBRICANTS

AN HF SINCLAIR BRAND

How is Oil Cleanliness Measured?

ISO 4406:1999 Classification

ISO code number	Number of particles per ml	
	More than	Up to and including
22	20,000	40,000
21	10,000	20,000
20	5,000	10,000
19	2,500	5,000
18	1,300	2,500
17	640	1,300
16	320	640
15	160	320
14	80	160
13	40	80
12	20	40
11	10	20
10	5	10
09	2.5	5
08	1.3	2.5
07	0.64	1.3



ISO 4406 Cleanliness Code

The ISO Cleanliness Code is an industry accepted method of evaluating the cleanliness of a lubricated component. When the ISO Code indicates an increase by more than one ISO code steps need to be taken to investigate the cause.

21/19/13

ISO 4406:1999 report as
Number of particles (Np)
>4µm over the Np
>6µm over the Np
>14µm

Ref: WearCheck

Particle Counting Instrumentation

On-site / In-Line / Laboratory

Entek Contam-Alert

On-Site Flow Decay

MP Filtri ICM

In-line Optical

MP Filtri LPA2

On-site Optical

Hiac Royco SDS

Laboratory Optical

CINRG CS-APC-2

Laboratory Automated Optical

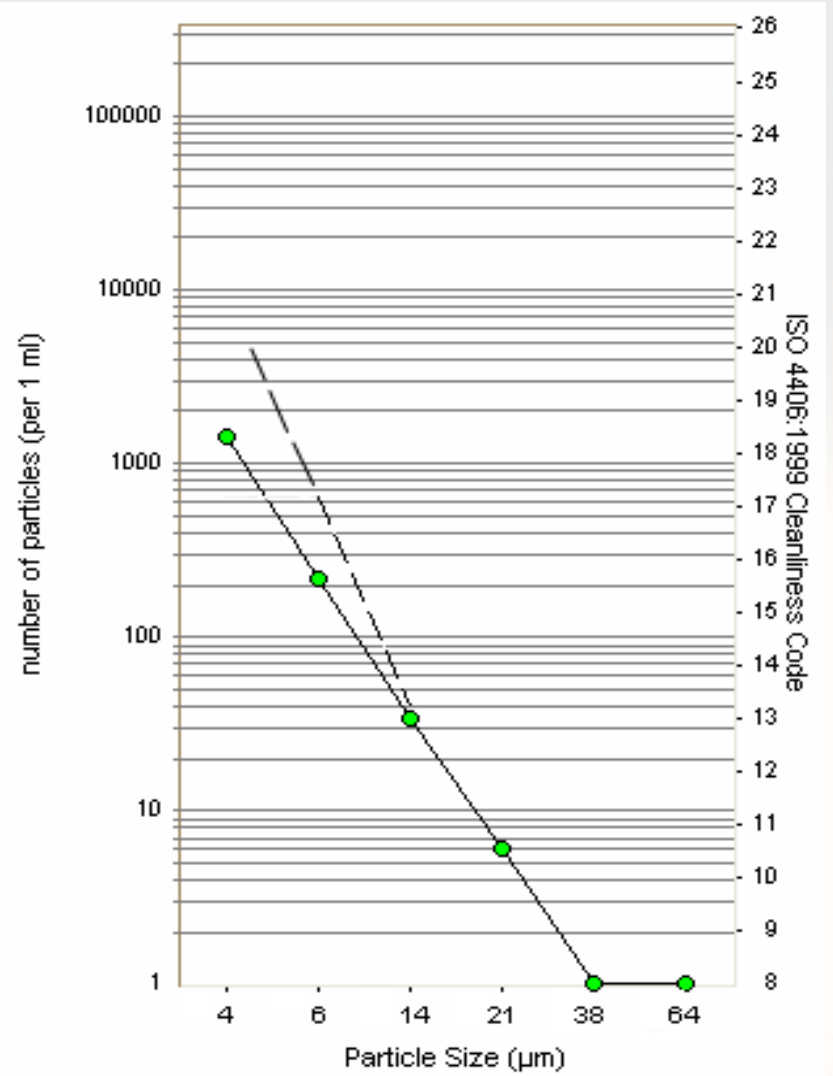
Particle Count Method

ASTM D7647

Ref: WearCheck

Particle count measurements are taken on typically 10 mL of oil with the results averaged to 1 mL. Prior to counting, the particles in the oil sample must be homogenized which can be accomplished in a combination of ways including shaking, sonication, de-gassing, etc. Most important is that sample preparation be carried out consistently. Once prepared the sample is loaded into a syringe and the contents of the oil are driven through the laser sensor at a controlled flow rate. The sensor "counts" the number of particles at the different size ranges for the duration of the test.

- Verify effectiveness of filtration
- Detect process contamination



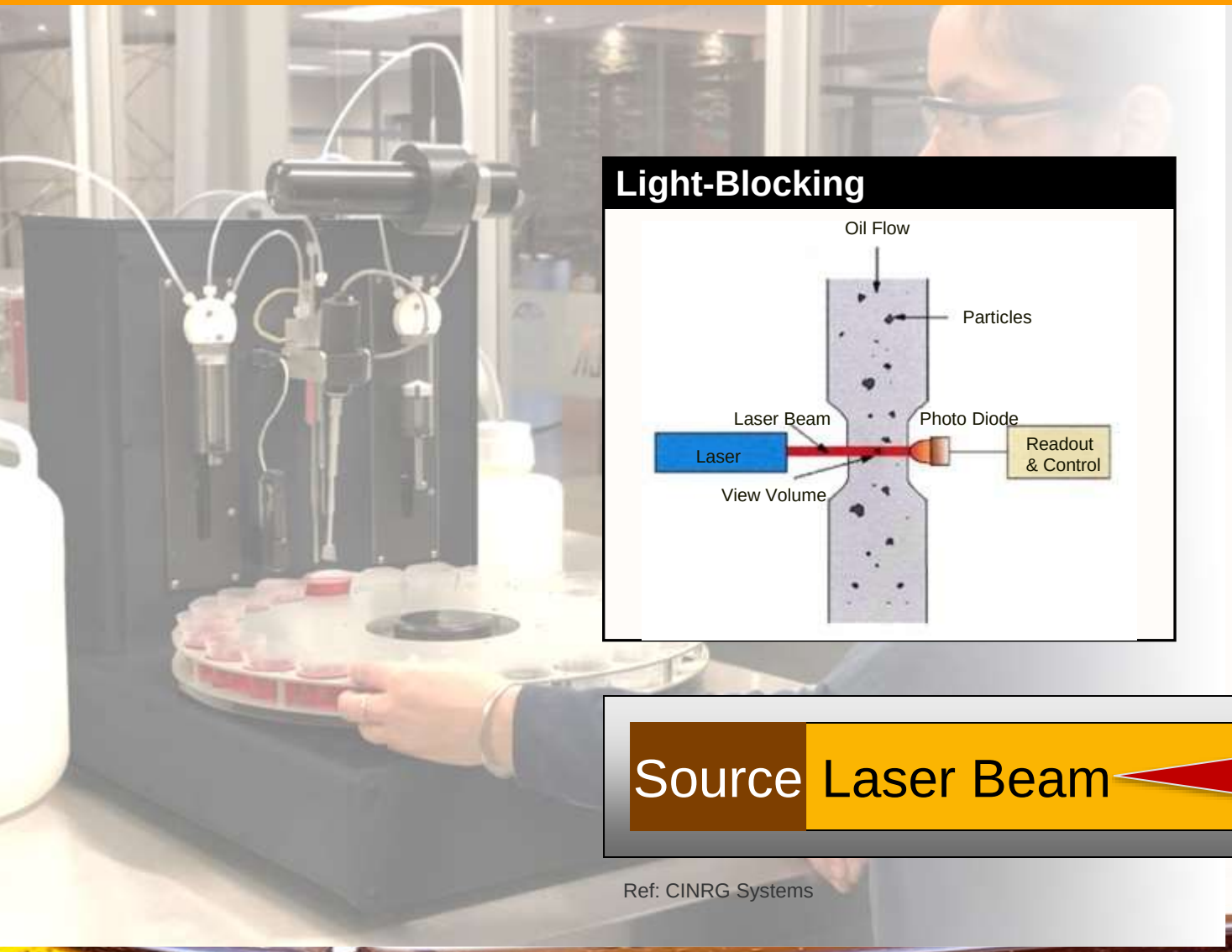
Example

Breather filters and improved oil filtration have brought the cleanliness of this system down from 20/18/16 to 18/15/13 *(sample is from a large hydraulic reservoir using Esso Nuto H 68).*

Test	Target	Current	3 months ago	6 months ago
>4µm	5,000	1,865	3,465	8,432
>6µm	1,300	254	868	2217
>14µm	160	46	187	402
ISO 4406	19/17/14	18/15/13	19/17/15	20/18/16

Ref: WearCheck

How an Optical Particle Counter Works



Light-Blocking

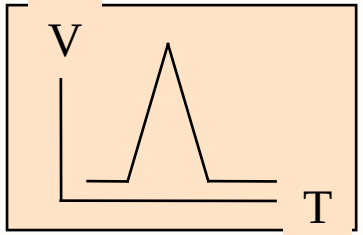
Debris Particles

Oil Stream

Particle Size Data

>4µm : 4,324
>6µm : 1,245
>14µm : 345

Voltage Pulse Counter



(Detector Response)

Source Laser Beam Detector

"Light Blockage"

Ref: CINRG Systems

CINRG WEAR CHECK PETRO-CANADA LUBRICANTS AN HF SINCLAIR BRAND

How an Optical Particle Counter Works

"Light Scattering" Interferences by

Water & Soft Particles

- **Color-bodies**
- **Varnish precursors**
- **Insoluble products**

Ref: CINRG Systems

CINRG WEAR CHECK PETRO-CANADA LUBRICANTS AN HF SINCLAIR BRAND

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.

Why ASTM D7647?

Appropriate solvent will eliminate interference from “soft particles”.

- Water (up to 2% can be effectively masked by a mixed solvent of 25% IPA/ 75% Toluene).
- Additives – Anti-foaming additives (Polydimethylsiloxane), specialized EP additives.
- Oxidative byproducts / color-bodies (varnish precursors).

This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.

Designation: D7647 – 10 (Reapproved 2018)

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[1]

This standard is issued under the fixed designation D7647; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method
concentration and parti
service oils used for lub

1.2 Particles consider
200 µm $_{(c)}$ with the uppe
automatic particle count

NOTE 1—For the purpose
by the diluent procedure a
particles are detected and re

NOTE 2—The subscript$_{(c)}$
calibrated in accordance wi
applies to particles up to 50 µm.

Diluent	Is this a water-masking diluent?
Stoddard solvent, also called Type 1 mineral spirits or white spirits	no
kerosene	no
lamp oil*	no
25 % 2-isopropanol / 75 % toluene	yes
dipropylene glycol n-propyl ether*	yes

2. Referenced Documents

Investigation into the suitability of the dilution solvents allowed for ASTM D7647.

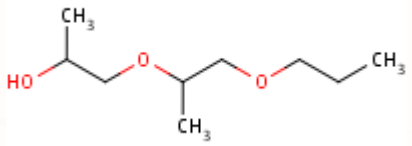
Solvents Investigated

1. Toluene/IPA 75/25%
2. Ethylene Glycol Butyl Ether (EGBE)
3. Dowanol (DPnP)
4. Kerosene
5. Varsol (Stoddard Solvent).
6. Kerosene / DPnP 67/33%

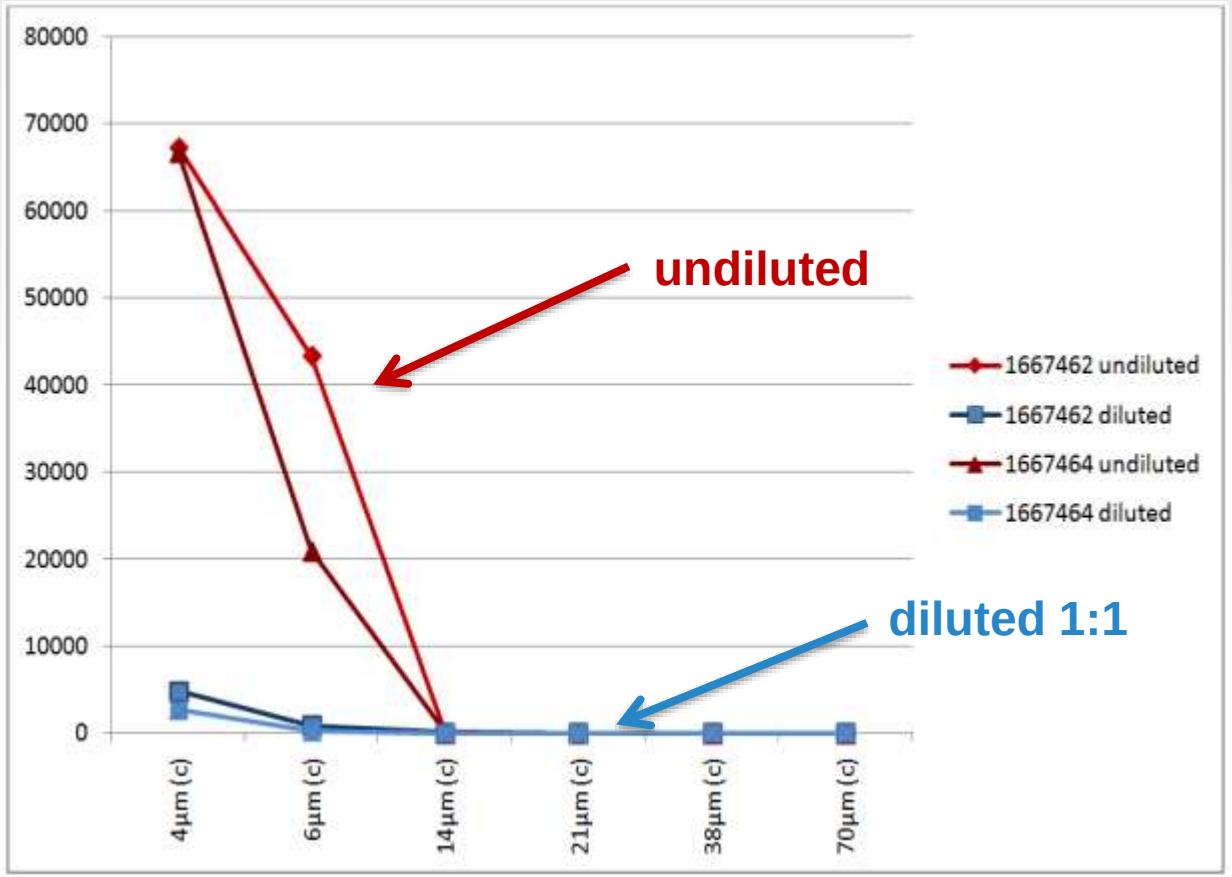
Lamp oil / Kerosene is a liquid petroleum product that is designed to burn cleanly in brass and glass oil lamps, torches, and lanterns. In the same family as kerosene. It has been further processed and refined so that it doesn't produce as much harmful smoke, soot, and other pollutants.

Stoddard solvent is a widely used synthetic, organic solvent that comes from the refining of crude oil. It is a petroleum mixture made from distilled alkanes, cycloalkanes (naphthenes), and aromatic compounds. It also goes by other names such as Varsol 1, TexSolve S and others.

Dipropylene glycol monpropyl ether (DPnP) is a colorless liquid with an ether-like odor that evaporates slowly. It is used as a solvent and as a coalescent for water-borne latex coatings.



Insolubles & Particle Counts



Ref: CINRG Systems

01961022 (MPC = 159)

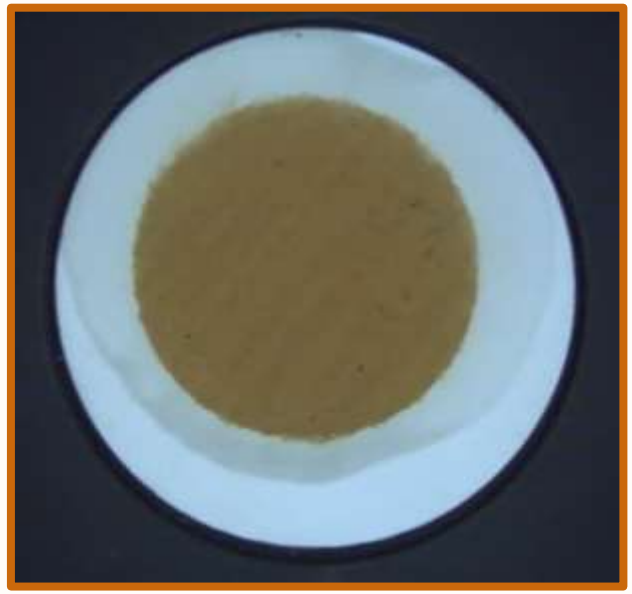
PDF / Kerosene (ISO 23/23/21)

PDF / Toluene (ISO 17/15/11)

(Used) Mobil DTE 846

Insolubles -> 23/23/19 -> 13/12/10

MPC (ΔE) = 60



Ref: WearCheck (02530825)

Solvent	U Frac	ISO Code (Ave)
UNDILUTED	1.00	23/23/19
Butyl Glycol	0.48	14/12/9
	0.31	15/13/11
75 Toluene 25 IPA	0.46	14/13/10
	0.31	14/13/10
Dowanol(DPnB)	0.49	14/13/11
	0.32	13/12/10
Kerosene	0.46	24/24/18
	0.29	25/23/16
Varsol	0.49	24/24/19
	0.28	25/24/18
90 Toluene 10 IPA	0.52	15/13/10
	0.30	13/12/9
67 Kerosene 33 DPnB	0.49	17/16/13
	0.32	14/13/10

Ref: CINRG Systems

Results Disparity even with low levels of insoluble

Sample No 02515720
Component Thrust Bearing
Fluid Petro Canada TurboFlo R&O 46
MPC 15

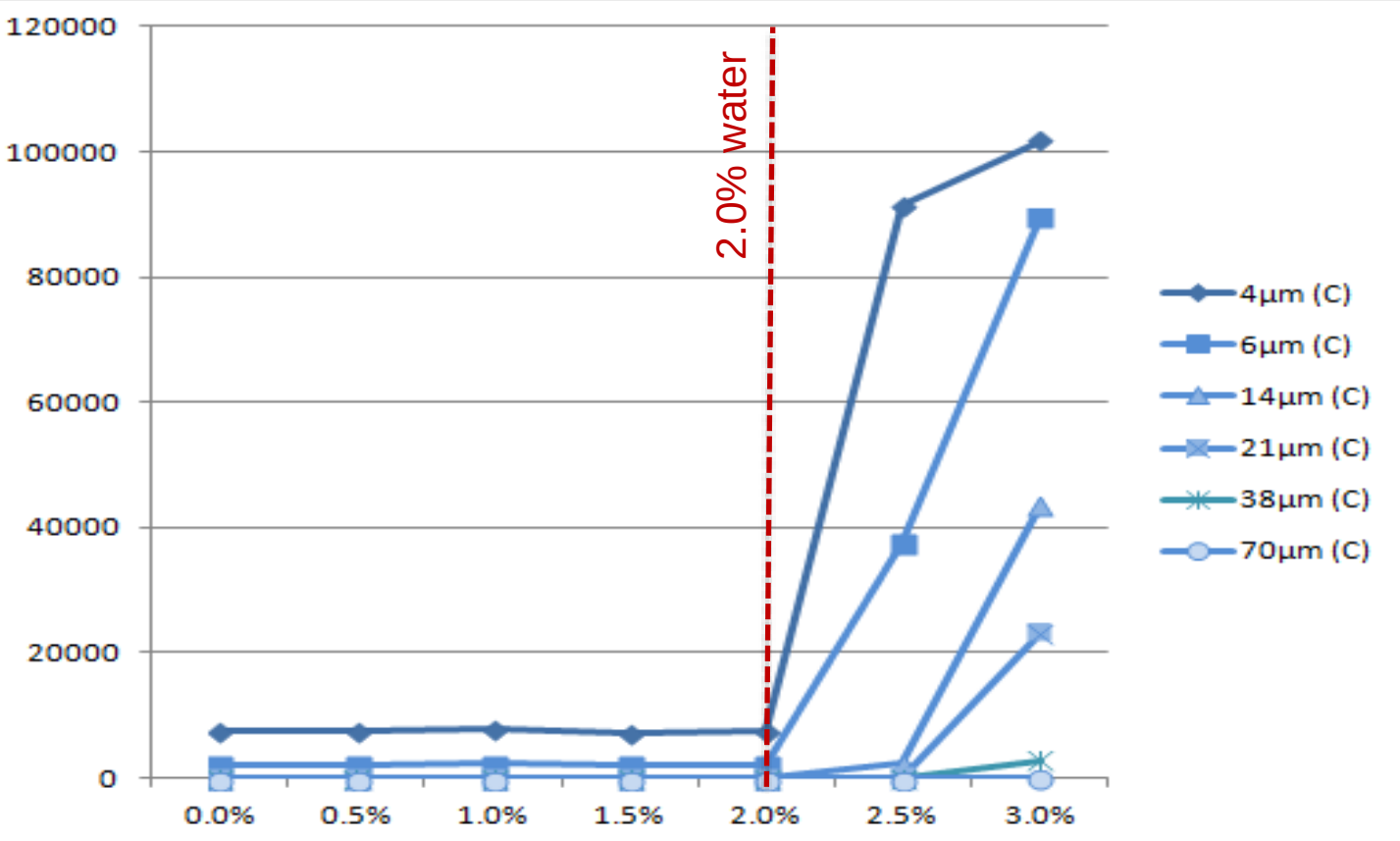
02515720	>4µm(c)	>6µm(c)	>14µm(c)	>21µm(c)	>38µm(c)	>70µm(c)	ISO CODE
Undiluted	1657.1	191.5	15.3	6.6	1.7	0.6	18/15/11
Diluted 1:1 Toluene/IPA	1477.3	211.6	17.6	5.3	0.3	0	18/15/11
Diluted 1:1 Varsol	2115.6	216.1	15.8	6.6	0.1	0.1	18/15/11
Diluted 1:1 EGBE	1600.2	227.8	22.3	8.4	1	0.3	18/15/12
Diluted 1:1 Kerosene	1986.6	200.3	14.5	4.8	0.6	0	18/15/11

Sample No 02521133
Component Turbine
Fluid Castrol Perfecto XPG 32
MPC 23

02515720	>4µm(c)	>6µm(c)	>14µm(c)	>21µm(c)	>38µm(c)	>70µm(c)	ISO CODE
Undiluted	7312.8	2250.5	150.4	40.1	3	0.1	**20/18/14**
Diluted 1:1 Toluene/IPA	290.2	88	6.1	1.7	0.2	0	**15/14/10**
Diluted 1:1 Varsol	7009.2	1909.1	86.3	17.9	0.6	0	**20/18/14**
Diluted 1:1 EGBE	394.1	99.6	6.1	2.1	0.4	0	**16/14/10**
Diluted 1:1 Kerosene.	7495.4	2063.6	85.3	16.8	0.5	0	**20/18/14**

Ref: CINRG Systems

Water Contamination & Particle Counts



Ref: CINRG Systems

Samples as Received

Mixed with masking solvent

Ref: CINRG Systems

CINRG WEAR CHECK PETRO-CANADA LUBRICANTS AN HF SINCLAIR BRAND

(Spiked) Medium Test Dust (MTD)

Water -> 22/22/22 -> 20/19/17

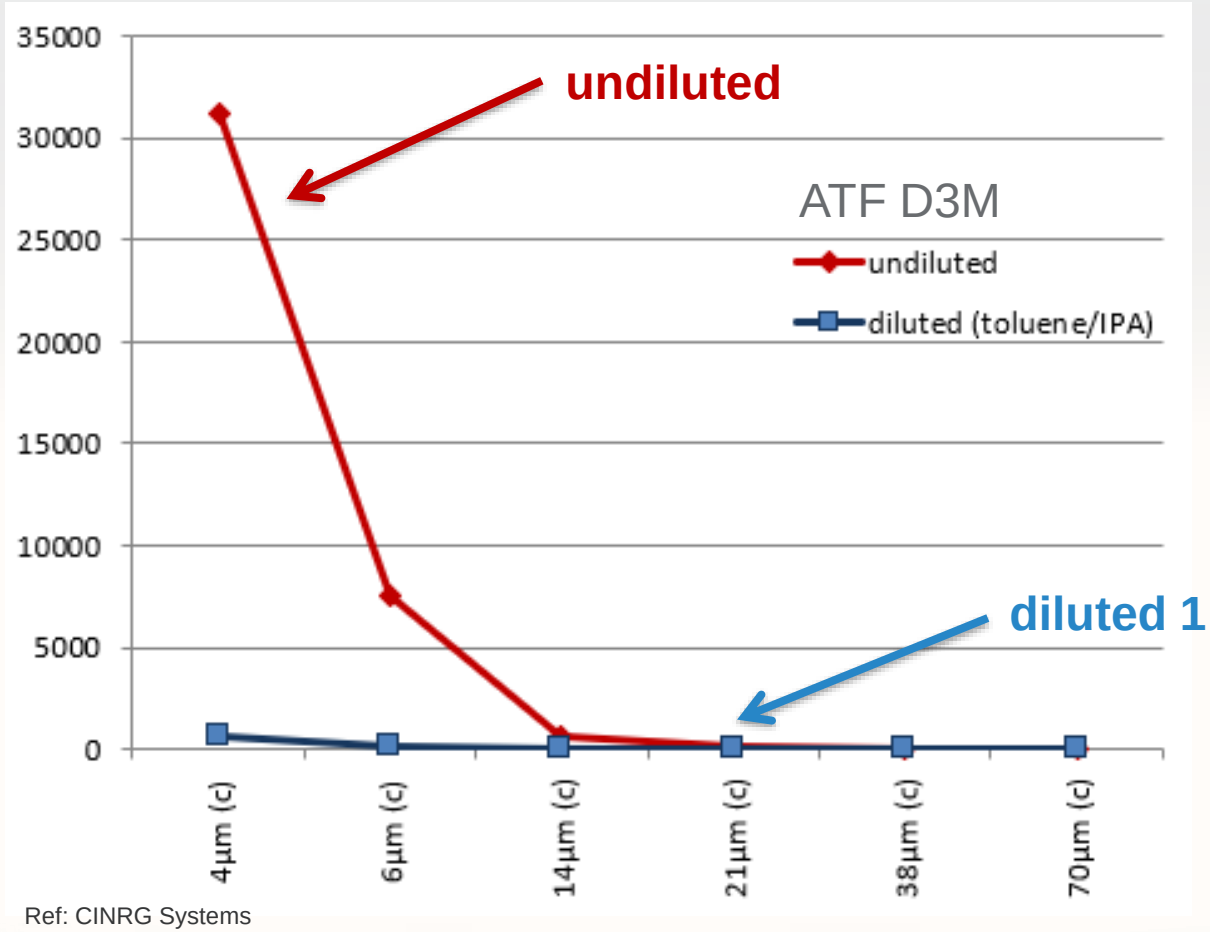
H_2O = 1% *or* 2%

Solvent	U Fraction	ISO Code (Ave)
UNDILUTED	1.00	21/19/16
UNDILUTED (1% H2O)	1.00	22/22/22
Butyl Glycol	0.44	21/20/16
1% H2O	0.31	21/20/16
Butyl Glycol	0.43	21/20/16
2% H2O	0.32	21/20/16
75 Toluene 25 IPA	0.44	21/19/16
1% H2O	0.34	21/19/15
75 Toluene 25 IPA	0.43	21/19/16
	0.30	21/19/15
Dowanol(DPnB)	0.43	21/19/16
1% H2O	0.28	21/19/16
Dowanol(DPnB)	0.49	21/19/17
2% H2O	0.28	20/19/17
Kerosene	0.46	25/25/24
1% H2O	0.31	25/25/25
Varsol	0.47	25/24/24
90 Toluene 10 IPA	0.41	22/21/21
1% H2O	0.27	20/19/16
	0.46	24/24/23
	0.27	22/21/21
67 Kerosene 33 DPnB	0.51	22/21/21
1% H2O	0.28	21/20/18

Ref: CINRG Systems

CINRG WEAR CHECK PETRO-CANADA LUBRICANTS AN HF SINCLAIR BRAND

Oil Additives & Particle Counts

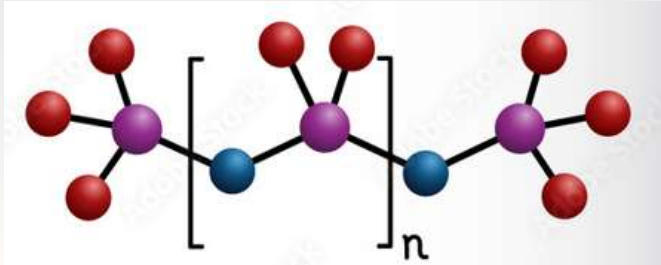


Ref: CINRG Systems

Polydimethylsiloxane

PDMS

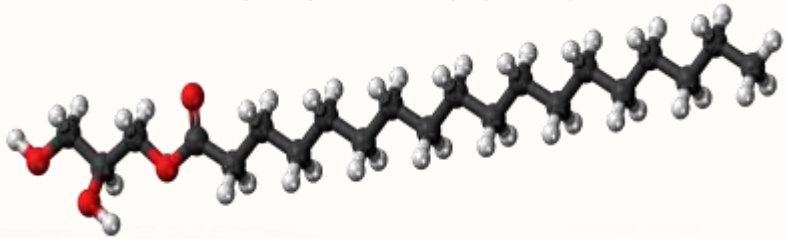
Anti-foaming Agent



Glycerol monooleate

GMO

Friction Modifier



Base Oil & Additives Effect on ISO Cleanliness Code

Conclusion

This study revealed that the **Antifoam**, in this case polydimethylsiloxane (PDMS) was the primary reason of the increased particle counts in the fresh fluid and higher baseline particle counts were to be expected.

Components	ISO Cleanliness Code
Commercial ATF	22/20/16 to 23/21/17
Base Oils	19/15/10
Friction Modifiers	20/17/12
Friction Modifiers + **Antifoam**	**22/20/15**
Dispersants + **Antifoam**	**21/19/14**
Dispersants + Viscosity Index Improver + **Antifoam**	**21/19/14**
Detergents	20/17/11
Dispersant + Friction Modifier + Viscosity Index Improver	20/17/11
Detergent + Friction Modifier + Dispersant + Viscosity Index Improver	20/17/12
Detergent + Friction Modifier + Dispersant + Viscosity Index Improver + **Antifoam**	**21/19/14**

Ref: HF Sinclair / Petro-Canada Lubricants

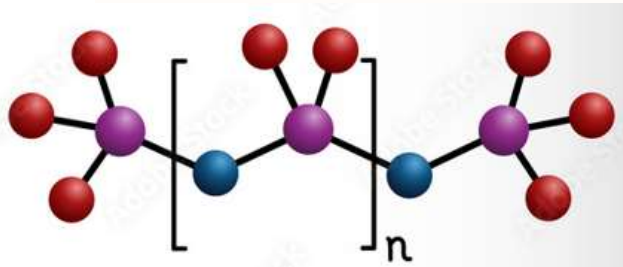
1- Paul W. Michael, Thomas S. Wanke, and Michael A. McCambridge; Additive and Base Oil Effects in Automatic Particle Counters - Journal of ASTM International, Vol. 4, No. 4 Paper ID JAI100941

(New) Petro-Canada ATF D3M

Additives -> 22/20/16 -> 16/14/11

Polydimethylsiloxane

PDMS



Solvent	U Frac	ISO Code (Ave)
UNDILUTED	1.00	22/20/16
Butyl Glycol	0.48	20/17/14
	0.30	19/17/13
75 Toluene 25 IPA	0.43	16/14/10
	0.30	16/14/11
Dowanol(DPnB)	0.46	19/17/14
	0.27	19/17/13
Kerosene	0.44	17/15/11
	0.29	17/15/11
Varsol	0.45	17/15/12
	0.30	17/15/11
90 Toluene 10 IPA	0.47	16/14/11
	0.31	16/14/10
67 Kerosene 33 DPnB	0.46	17/15/11
	0.29	17/15/12

Ref: CINRG Systems

(New) Mobil SHC 320

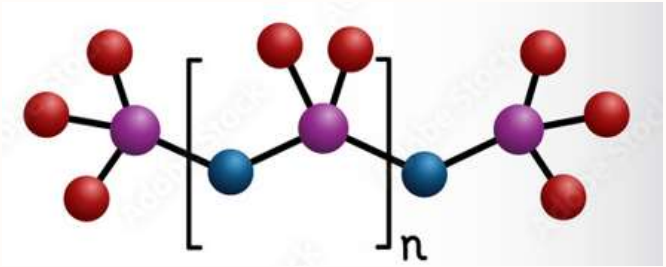
Base Oil/Additives -> 21/17/11 -> 15/13/8

Polyalphaolefin

PAO

Polydimethylsiloxane

PDMS



Solvent	U Frac	ISO Code (Ave)
	1.00	-/-/-
Butyl Glycol	0.50	-/-/-
	0.33	-/-/-
75 Toluene 25 IPA	**0.51**	**20/16/10**
	0.33	**15/13/8**
Dowanol(DPnB)		-/-/-
		-/-/-
Kerosene	**0.48**	**21/17/11**
	0.33	**16/14/10**
Varsol	**0.49**	**21/17/12**
	0.32	**16/14/11**
90 Toluene 10 IPA	**0.49**	**19/16/10**
	0.33	**15/13/10**
67 Kerosene 33 DPnB	**0.51**	**21/17/12**
	0.33	**16/14/11**

Ref: CINRG Systems

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(New) Irving Synthetic Compressor Oil

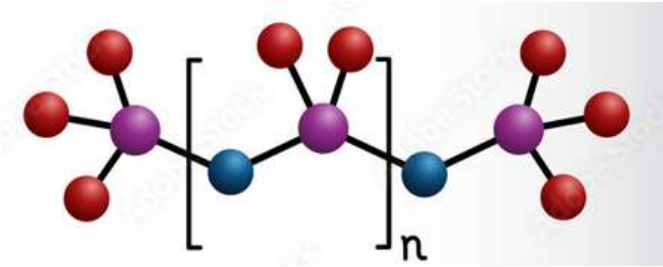
Base Oil/Additives -> 25/24/19 -> 18/16/11

Polyalphaolefin

PAO

Polydimethylsiloxane

PDMS

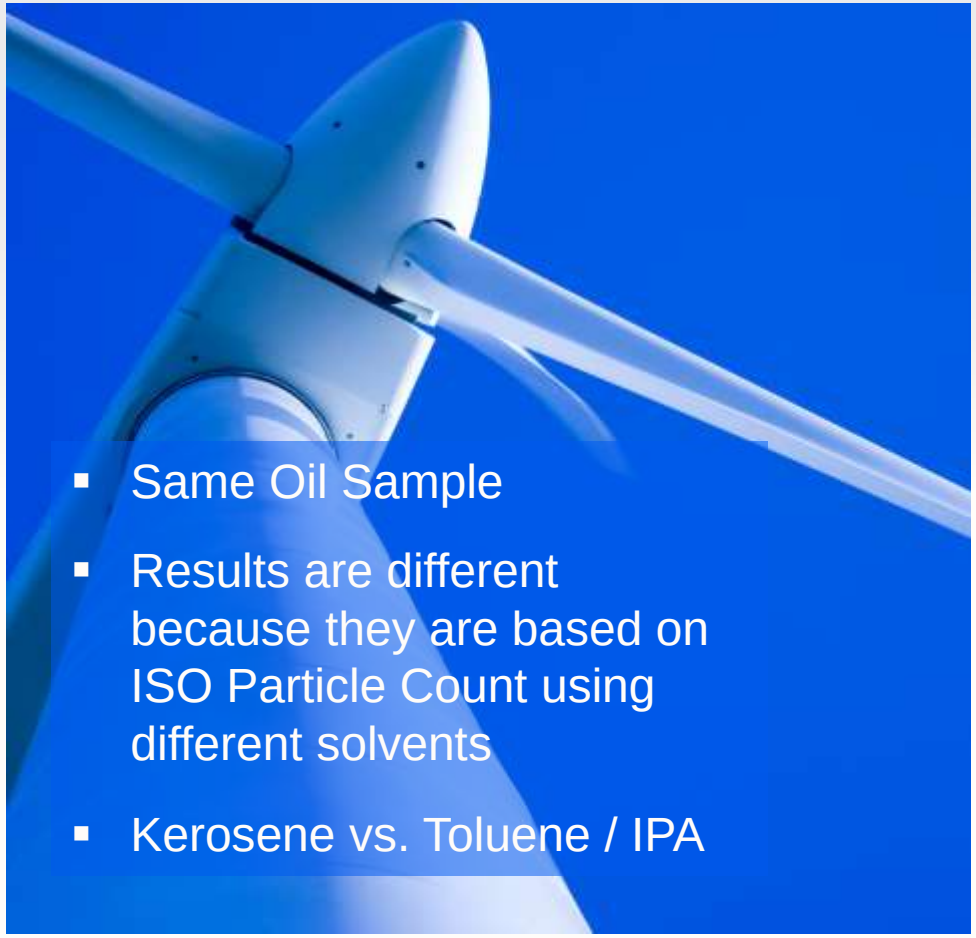


Solvent	U Frac	ISO Code (Ave)
UNDILUTED	1.00	**18/16/12**
Butyl Glycol	0.49	19/16/12
	0.32	**25/24/19**
75 Toluene 25 IPA	0.49	18/16/12
	0.29	18/16/12
Dowanol(DPnB)	0.50	18/16/11
	0.30	18/16/12
Kerosene	**0.49**	**19/16/12**
	0.30	**19/17/13**
Varsol	**0.48**	**19/17/13**
	0.28	**19/17/13**
90 Toluene 10 IPA	0.44	18/16/11
	0.30	18/16/11
67 Kerosene 33 DPnB	0.48	18/16/12
	0.28	18/16/12

Ref: CINRG Systems

CINRG WEAR CHECK PETRO-CANADA LUBRICANTS AN HF SINCLAIR BRAND

What is the issue?

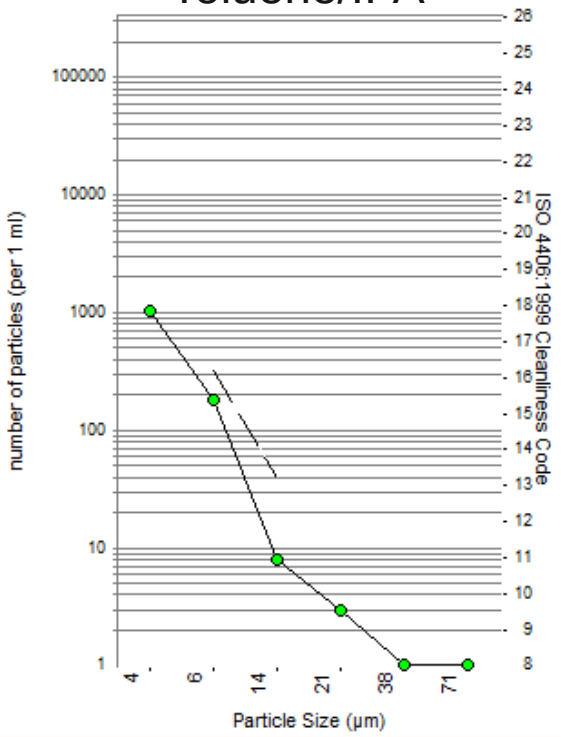


- Same Oil Sample
- Results are different because they are based on ISO Particle Count using different solvents
- Kerosene vs. Toluene / IPA

Ref: CINRG Systems

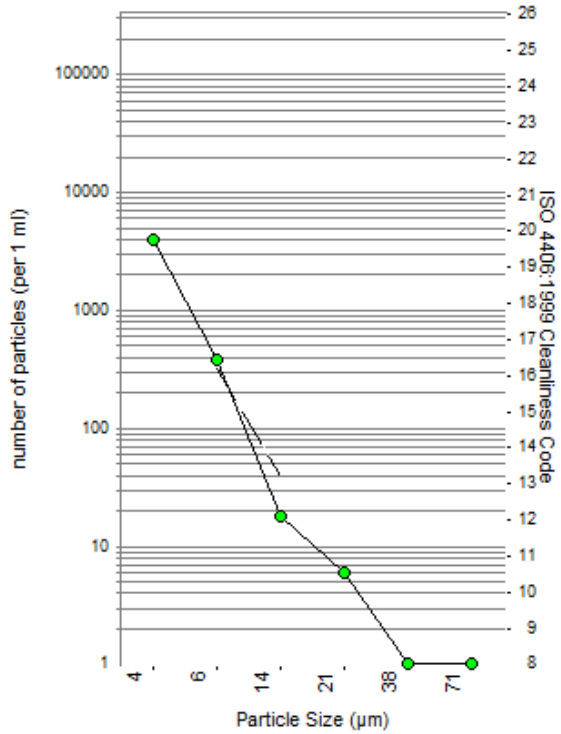
1- Alistair Geach, William A. Quesnel; Particle Counting of Heavily Contaminated Oil Samples – OilDoc Conference, Rosenheim Germany, January 2015

ISO 4406:1999 **17/15/10**
Toluene/IPA



$0.00

ISO 4406:1999 **19/16/11**
Kerosene



2 x tech x 3 hrs
+ Oil Filter

$700.00 x 133
= $93,100.00

Ref: G. Tapp – GE Wind

Filtration Effects on Oil Additives

Conclusion

This study reveled that the **Antifoam** and **Detergents** could be stripped out from the fluid with filtration. These additives are essential for the adequate performance of the fluid and care should be taken when selection filter pore size.

Sample Description Test Results	ATF A production batch	ATF A production batch after 3µm filtration*	ATF A production batch after 1µm filtration*
ISO Cleanliness Code	22/21/17	19/17/10	18/17/12
Silicon, ppm	6.7	4.4	2.0
Boron, ppm	86	82	81
Calcium, ppm	64	56	57
Phosphorus, ppm	203	194	193
Sulfur, ppm	1282	1246	1240

* Membrane filters were use in this evaluation – single pass

NOTE: Filtration was performed using a laboratory filtration system

Ref: HF Sinclair / Petro-Canada Lubricants

Conclusions & Recommendations

- Water & “Soft” Particles increase the apparent ISO Cleanliness Code (soft particles include insoluble oxidation by-products and some oil additives)
- The dilution method for particle counting (ASTM D7647) mitigates or eliminates the effect of water and “soft” particles
- Not all solvents used for dilution have the same masking effect. 75% Toluene / 25% Isopropanol (IPA) is the most effective solvent mix for water and “soft” particles
- Ultra-fine filtration can lead to the removal of some oil additives (notably anti-foaming agents and detergency additives)
- Before taking action based on your oil samples ISO Cleanliness results, ensure that your laboratory is using the appropriate particle counting method

Questions?