Particle Counting of Heavily Contaminated Oil Samples

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1. Summary

With an increase in education in the industry regarding the direct effect of particulate levels in oil on MTBF (mean time between failures) reliability personnel are requesting particle count analysis on an ever increasing range of lubricated machinery. In effectively managed oil analysis and lubrication programs oil cleanliness has become one of the predominant metrics for tracking the health of lubricated machines. This increase in both awareness and demand for particle count testing has put pressure on commercial oil analysis laboratories to improve their current particle counting methodologies in terms of both automation and the ability to perform accurate particle counts on oils that are either opaque, heavily contaminated, contain water or contain "soft particles" such as varnish or certain oil additives.

Currently available instruments for particle counting using optical sensors are not capable of measuring opaque oils, heavily contaminated samples (more than 10,000 particles at a given size range) or samples that contain more than a few hundred ppm (>0.01%) water. Additionally these instruments produce falsely high counts when analyzing oils that contain varnish or certain oil additives (friction modifiers, antifoam agents are some examples) [1]. Samples that cannot be analyzed using the particle counting instrument require additional laboratory processing, typically involving the production of a Millipore patch and subsequent manual or automated counting of the particles present on the patch to produce an oil cleanliness code. In the authors laboratory samples requiring additional processing were increasing each year leading to more laboratory costs and poorer laboratory turn-around times. These issues could be mitigated by diluting oil samples using the solvent outlined in 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 [1]. A major impediment to any commercial oil laboratory, however, was that currently available automated particle counters are not designed to automatically dilute oil samples to take advantage of the benefits of ASTM D7647-10. The labor involved in pre-diluting samples and subsequent back-end processing to determine and normalize the particulate present in the dilution solvent from the sample results is a non-starter for most laboratories.

One of the authors, Alistair Geach, has a long career of designing and developing automated instrumentation for commercial oil analysis laboratories, and had long thought of how to automate particle counting. The introduction of sample dilution to particle counting in the method ASTM D7647-10 solved some of the more significant issues regarding automation of particle counting. The first instrument was developed in-house for use at Wearcheck Canada Inc. and was put into production in this laboratory in 2011. The initial system (CS-APC-1) was based on the Hiac 8000A particle counter and Gilson's 215 liquid handler and 402 syringe pump. The instrument worked extremely well in the laboratory and the expected improvement in the quality of count data that automation and the new method (ASTM D7647-10) provided was immediately evident. There was also a significant impact on the laboratory's productivity through the elimination of a labor intensive test. The first instrument required sample to be added to sample cups via pipette and within a short time the laboratory staff were complaining of RSI (Repetitive Strain injury) and this led to a further productivity improvement with the integration of an ultrasonic level sensor into the system for automatic sample volume measurements and automated sample dilution.

1. Introduction

The CINRG CS-APC-2 particle counting system (Figure 1) is a fully automated system that meets the requirements of ASTM D7647-10 in which oil samples are automatically diluted with solvent prior to testing in order to eliminated interferences from" soft" particles such as water, varnish and suspended liquid additives.

The CINRG CS-APC-2 automatic auto-diluting particle counter system combines equipment from several leading equipment manufacturers with some innovative technology and sophisticated software that was developed by CINRG Systems for use in a number of laboratories of a global oil analysis laboratory group. The system has a high degree of flexibility and can be customized to a large extent to suit local laboratory processing requirements.

In conjunction with a local laboratory belonging to this global oil analysis laboratory group a number of heavily contaminated oil samples that normally could not be processed by an optical particle counter were tested on the CS-APC-2. For this paper samples with high water contamination, opaque samples, samples containing "soft" particles and heavily contaminated samples were tested to compare the results from the CS-APC-2 versus the Hiac 8012 particle counter (Figure 2).

Figure 1: The CINRG CS-APC-2 fully automated auto-diluting particle counting instrument with Klotz LDS Sensor, Klotz USB counter, Baumer UNKC sample level sensor, Gilson 215 liquid handler and CINRG Systems controller.

Figure 2: The Hiac 8012 liquid particle counter with 8000A controller, HRLD Sensor and Syringe Driven Sampler (SDS).

2. Water in Oil

For the water test a series of samples were prepared from a fluid containing 1mg/L of UFTD (Ultra Fine Test Dust) in MIL-H-5606 hydraulic fluid. Samples were spiked with increasing amounts of water in 0.5% increments up to a maximum of 3.0%. The results are shown in Table 1.

Samples were vigorously shaken, placed in an ultrasonic bath for 10 seconds and approximately 15ml immediately poured into the sample cups for testing. Samples were diluted with solvent (75% Toluene / 25% Isopropanol) to yield a final sample volume of 30ml and stirred for 30 seconds before testing.

For comparison purposes a sample of 0.1% water in MIL-H-5606 oil was prepared and counted using the CINRG CS-APC-2 using 100% Toluene as the diluents and the results are shown in Table 2.

Particle count results from the CINRG CS-APC-2 particle counter remained within repeatability limits for the samples prepared with 0.5% up to and including the sample prepared with 2.0% water. Using only a 1:1 dilution a sharp increase in the amount of particles counted was observed at 4, 6, 14 and 21 microns when water was >= 2.5% (Figures 3 and 4).

Table 1: Testing results from trial of water in MIL-H-5606 oil using 75% Toluene/25% Isopropanol as a solvent.

Sample ID	% Water	4µm (c)	6µm (c)	14µm (c)	21µm (c)	38µm (c)	70µm (c)	ISO Code
RM8632-00	0.0%	7424	2144	20	3	0	0	20/18/11
RM8632-05	0.5%	7415	2153	15	2	0	0	20/18/11
RM8632-10	1.0%	7852	2269	14	3	0	0	20/18/11
RM8632-15	1.5%	7098	2078	11	2	1	0	20/18/11
RM8632_20	2.0%	7484	2215	13	2	0	0	20/18/11
RM8632_25	2.5%	91341	37472	2512	183	1	0	24/22/19
RM8632_30	3.0%	101760	89620	43566	23261	2816	9	24/24/23

Table 2: Testing results from trial of 0.1% water in MIL-H-5606 oil using 100% Toluene as a solvent.

Sample ID	% Water	4µm (c)	6µm (c)	14µm (c)	21µm (c)	38µm (c)	70µm (c)	ISO Code
RM8632-01T	0.1%	180022	170028	122925	91843	37729	6798	25/25/24

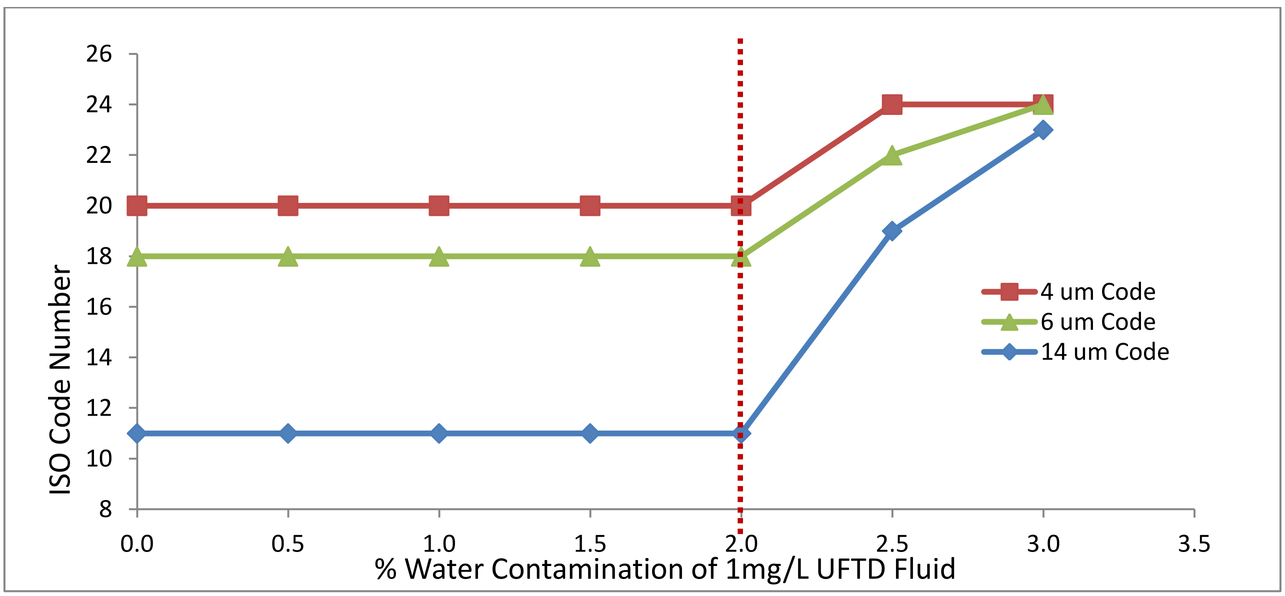


Figure 3: *ISO Code results from trial of 0.5% to 3.0% water in MIL-H-5606 oil using 75% Toluene / 25% Isopropanol as a solvent*.

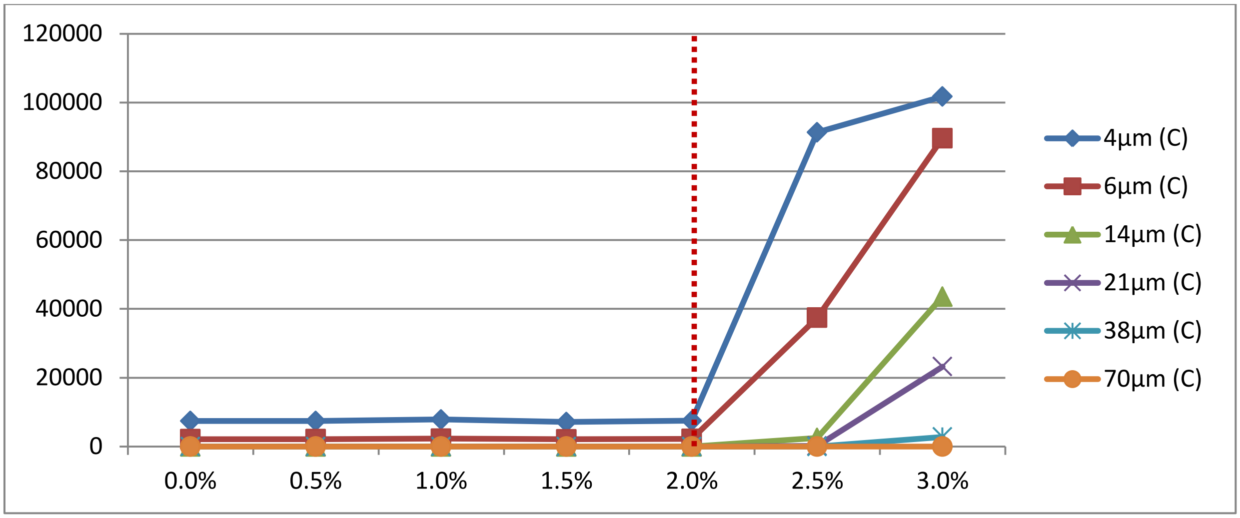


Figure 4: *Individual micron channel results from trial of 0.5% to 3.0% water in MIL-H-5606 oil using 75% Toluene / 25% Isopropanol as a solvent*.

Next to test the effectiveness of using 75% Toluene / 25% Isopropanol as a solvent on actual used oil samples a number of routine hydraulic oil samples with high water concentration were selected from the WearCheck sample database. The samples, shown in Figure 5 below, varied from <1% free water up to 17% free water and after shaking from 0.35% emulsified water up to 14.76% emulsified water. The samples were run on both the Hiac 8012 particle counter and the CINRG CS-APC-2 particle counter. The samples, when run neat on the Hiac 8012 particle counter, resulted in a baseline error and produced zero (0) counts across all channels and a resulting ISO Cleanliness code of 0.7/0.7/0.7. The samples were diluted using the 75% Toluene / 25% Isopropanol solvent (see Figure 6 and Figure 7 below) at 1:1 dilution for <2.0% water, and at a 1:29 dilution for high water levels and run on the CINRG CS-APC-2 particle counter, and the results are outlined in Table 3 below. All the samples run on the CINRG particle counter produced cleanliness codes that were in line with historical sample results of the machines that were sampled.

Table 3: Testing results from used oil samples with high concentrations of free and emulsified water using 75% Toluene / 25% Isopropanol as a solvent.

Sample ID	Free Water	% Water	Dilution Oil:Solvent	4µm (c)	6µm (c)	14µm (c)	21µm (c)	38µm (c)	70µm (c)	Cleanliness Code
1952702	17%	14.76%	1:29	22241	3499	88	12	2	0	22/19/14
1963686	14%	1.60%	1:29	21186	2499	26	2	0	0	22/18/12
1958421	<1%	1.35%	1:1	27918	4380	78	7	0	0	22/19/13
1961583	<1%	0.35%	1:1	11497	3977	344	69	2	0	21/19/16

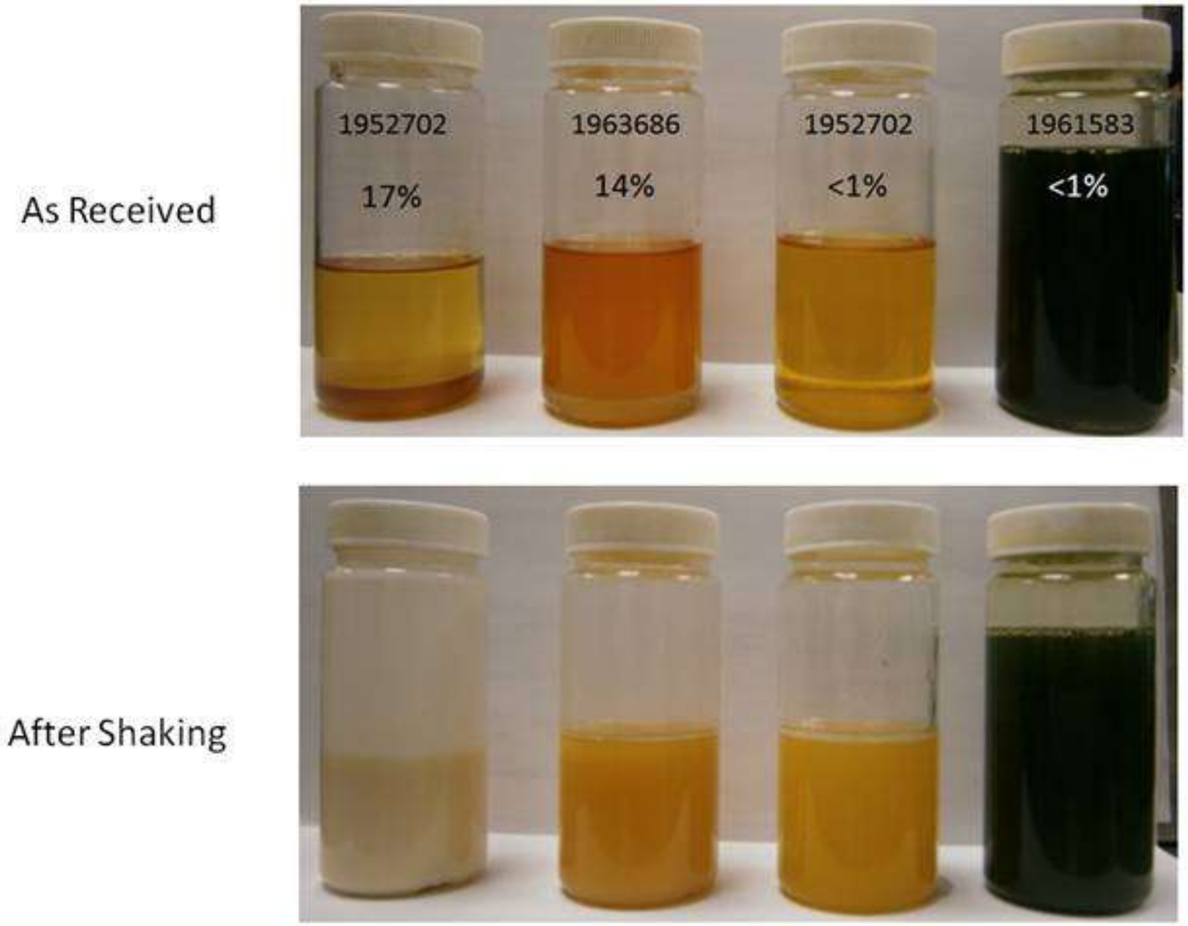


Figure 5: *Routine oil samples from the WearCheck laboratory containing high concentrations of free and emulsified water as received and after shaking*.

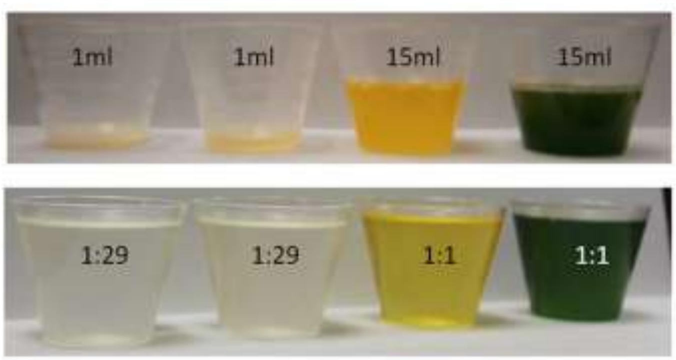


Figure 6: *Routine oil samples from the WearCheck laboratory containing high concentrations of free and emulsified water before and after dilution*.

Figure 7: *The last two samples (01961583 and 01952702) after being transferred from the sample cup to glass beakers and placed on top of a white piece of paper on which the word "CLEAR" is printed to demonstrate the effect of dilution on sample clarity*.

3. Opaque Oil Samples

Several opaque used oil samples of Castrol Optigear Synthetic A ISO 320 from wind turbine gearboxes were selected to be tested undiluted on the Hiac 8012 particle counter versus diluted on the CINRG CS-APC-2 instrument. Initially the cause of the opaqueness was not apparent and we suspected that degradation of the unique additive package was the cause. Figure 8 (below) shows the color image of the samples and the resulting particle debris patches.

Samples were tested on a Hiac 8012 particle counter unit (undiluted), and then tested using the CINRG CS-APC-2 unit with the standard 1:1 dilution. The results for both instruments are outlined in Tables 4 and 5.

Table 4: Particle Testing results for both diluted and undiluted preparation for sample 01667462.

Count Data	4µm (c)	6µm (c)	14µm (c)	21µm (c)	38µm (c)	70µm (c)	Cleanliness Code
Undiluted Sample	67307	43281	46	4	0	0	23/23/13
Diluted Sample(1:1)	4833	811	57	6	1	0	19/17/13

Table 5: Particle Testing results for both diluted and undiluted preparation for sample 01667464.

Count Data	4µm (c)	6µm (c)	14µm (c)	21µm (c)	38µm (c)	70µm (c)	Cleanliness Code
Undiluted Sample	66792	20984	97	15	1	0	23/22/14
Diluted Sample(1:1)	2730	228	21	5	2	0	19/15/12

The results indicate that there were "soft" particle effects on the counts at 4 and 6 microns for the undiluted samples tested on the Hiac 8012 particle counter (Figure 8). The counts from the CINRG CS-APC-2 particle counter showed much lower 4 and 6 micron counts. Particle debris patches for the two samples were prepared to verify the particle counts. The particle debris patches show very few particles and are in line with the ISO cleanliness codes from the diluted samples tested on the CINRG CS-APC-2 particle counter.

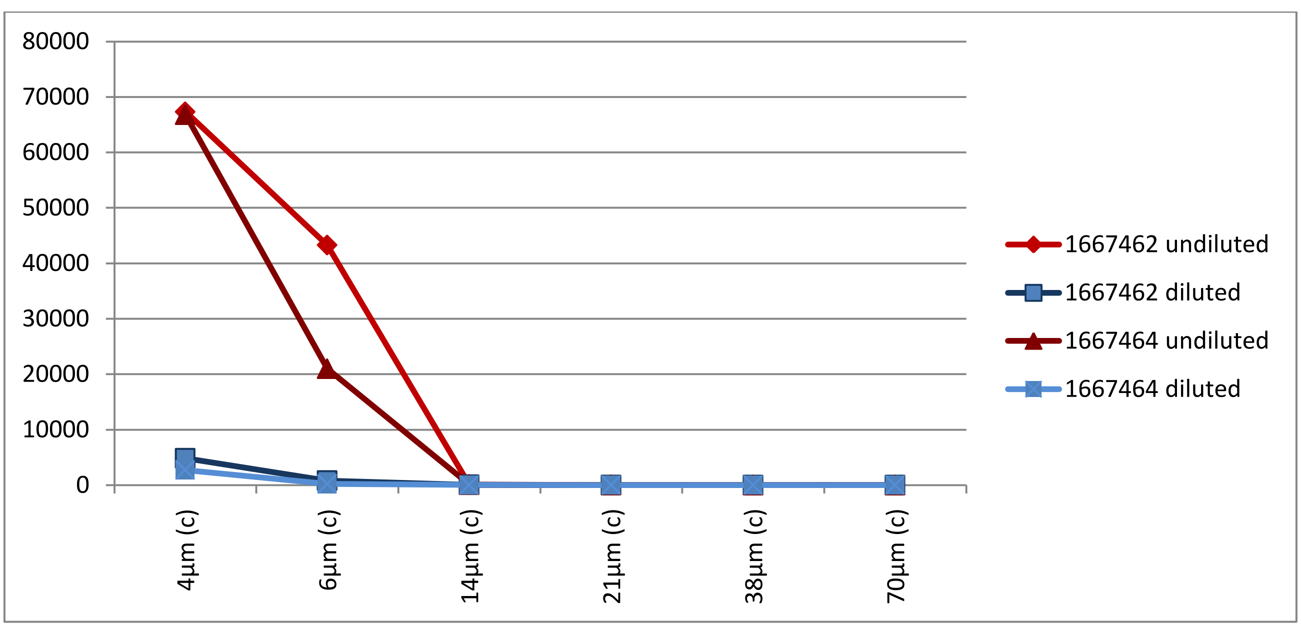


Figure 8: *Comparison of undiluted particle counts on the Hiac 8012 particle counter (red lines) versus diluted particle counts for Castrol Optigear Synthetic A ISO 320 on the CS-APC-2 particle counter (blue lines).*

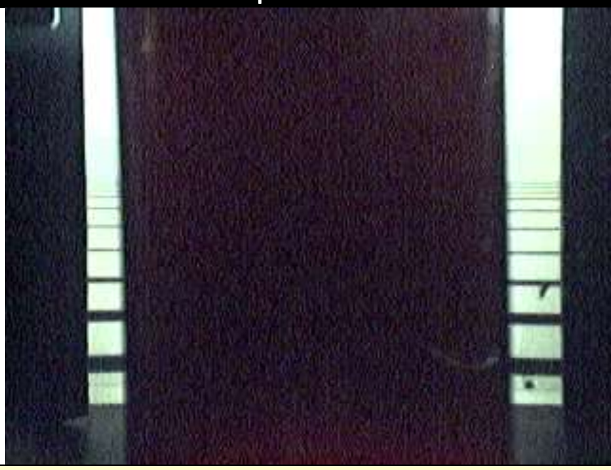
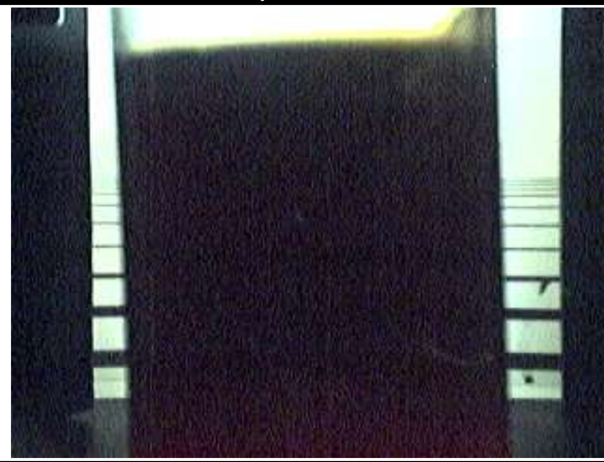
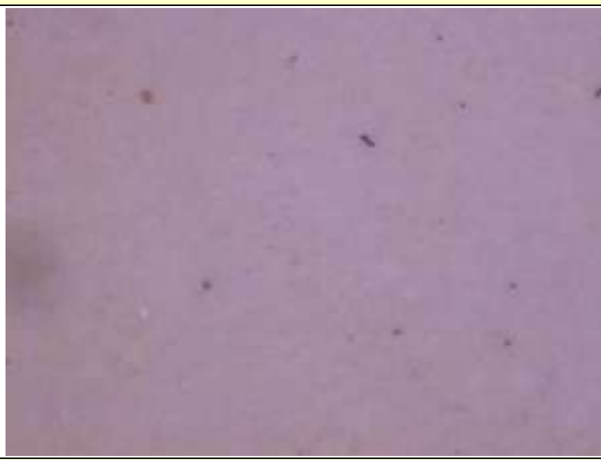
Sample 01667462	Sample 01667464
	
Color Image with Visibility Scale	Color Image with Visibility Scale
	
Particle Debris Patch @ 100X magnification	**Particle Debris Patch @ 100X magnification**

Figure 9: *Color images of the samples 01667462 and 01667464 with the resulting particle debris patches.*

Encouraged by the results in our limited study of wind turbine gearbox used oil samples, and to answer a lingering question about the nature of the "soft" particles present in these samples, we undertook a more thorough study using 22 oil samples from wind turbine gearboxes. The samples were run undiluted on the Hiac 8012 particle counter and diluted 1:1 with 75% Toluene / 25% Isopropanol as a solvent on the CINRG CS-APC-2 particle counter. Additionally the Membrane Patch Colorimetery (MPC) test was performed on each sample as per ASTM D7843-12 to determine if oxidation byproducts (varnish precursors) were the cause of the "soft" particle effects on the 4 and 6 micron counts on the undiluted samples (see Figure 10 for examples of a low MPC and high MPC patches from the testing conducted).

The data showed that undiluted samples run on the Hiac 8012 particle counter showed a significant increase in both the 4 and 6 micron counts with increasing MPC result indicating that the results were indeed being affected by the "soft" particles effect of the oxidation byproducts (varnish precursors) present in the oil (Figure 11). The diluted samples run on the CINRG CS-APC-2 instrument showed only a minor effect on the 4 micron particle count by these "soft" particles, and the 6 micron particle count showed no effect "soft" particles effect whatsoever (Figure 12).

Table 6: Particle count data from the Hiac 8012 particle counter on wind turbine gearbox used oil samples using Castrol Optigear Synthetic A 320 gear oil (undiluted). NOTE: Coinc. Ftr is the calculated coincidence factor given the 4 micron count.

Sample ID	Coinc. Ftr	MPC	4µm (c)	6µm (c)	14µm (c)	21µm (c)	38µm (c)	70µm (c)	ISO Code
01961012	1.72	36	17227	904.0	93.0	32.0	7.6	1.2	21/17/14
01961013	0.30	13	3028	861.0	73.6	20.0	5.2	0.6	19/17/13
01961014	2.18	29	21808	3888.0	332.0	93.0	17.2	0.0	22/19/16
01961015	3.00	39	30036	1376.4	96.2	30.4	4.6	0.2	22/18/14
01961016	2.22	37	22214	272.2	19.4	5.8	1.2	0.2	22/15/11
01961018	6.76	157	67642	45383.0	6910.0	1691.0	178.0	17.9	23/23/20
01961020	5.87	73	58733	31098.0	3029.0	720.0	75.4	4.2	23/22/19
01961021	0.33	30	3256	347.4	26.4	7.2	1.0	0.2	19/16/12
01961022	7.16	159	71570	56962.0	14343.0	3532.0	302.0	20.0	23/23/21
01964007	0.15	11	1539	443.0	71.6	32.6	7.8	1.0	18/16/13
01964010	1.25	27	12546	436.0	68.2	26.4	7.4	2.6	21/16/11
01964011	0.61	29	6118	274.0	29.4	11.8	4.8	2.6	20/15/12
01964012	1.42	53	14187	813.0	50.4	16.4	3.6	1.2	21/17/13
01964013	1.55	29	15496	118.4	15.6	6.4	1.2	0.4	21/14/11
01964014	4.53	50	45253	1757.0	39.6	12.6	2.4	0.4	23/18/12
01964015	0.09	15	902	237.0	37.0	15.0	2.6	0.2	17/15/12
01964016	1.22	8	12178	4340.8	364.4	94.6	13.8	1.2	21/19/16
01964017	6.26	47	62550	31578.0	700.0	34.2	6.8	1.8	23/22/17
01964018	0.08	12	776	185.4	29.2	14.4	3.8	1.2	17/15/12
01964019	1.86	13	18575	5784.0	371.8	116.6	24.4	4.2	21/20/16
01964020	3.43	44	34329	3735.0	208.0	71.0	12.8	2.4	22/19/15
01964022	0.26	18	2626	251.0	37.8	16.6	5.0	1.4	19/15/12

Table 7: Particle count data from the CINRG CS-APC-2 particle counter on wind turbine gearbox used oil samples using Castrol Optigear Synthetic A 320 gear oil (samples diluted 1:1 with 75% Toluene / 25% Isopropanol as a solvent). NOTE: Coinc. Ftr is the calculated coincidence factor given the 4 micron count.

Sample ID	Coinc. Ftr.	MPC	4µm (c)	6µm (c)	14µm (c)	21µm (c)	38µm (c)	70µm (c)	ISO Code
01961012	0.07	36	3679	510.0	36.0	10.0	1.0	0.0	19/16/12
01961013	0.02	13	758	132	16	7	2	2	17/14/11
01961014	0.40	29	19802	2648.0	118.0	22.0	1.0	0.0	21/19/14
01961015	0.33	39	16318	223.0	9.0	3.0	1.0	0.0	21/15/10
01961016	0.13	37	6736	81.0	9.0	5.0	2.0	1.0	20/14/10
01961018	0.42	157	21026	722.0	24.0	4.0	0.0	0.0	22/17/12
01961020	0.26	73	13041	516.0	30.0	7.0	0.0	0.0	21/16/12
01961021	0.03	30	1348	142.0	13.0	3.0	1.0	0.0	18/14/11
01961022	0.02	159	1095	172.0	18.0	7.0	4.0	2.0	17/15/11
01964007	0.03	11	1283	290.0	24.0	7.0	0.0	0.0	17/15/12
01964010	0.17	27	8697	265.0	27.0	8.0	0.0	0.0	20/15/12
01964011	0.06	29	3230	179.0	13.0	4.0	0.0	0.0	19/15/11

01964012	0.04	53	1840	299.0	16.0	5.0	0.0	0.0	18/15/11
01964013	0.16	29	7995	114.0	10.0	4.0	2.0	0.0	20/14/10
01964014	0.92	50	45816	327.0	20.0	5.0	0.0	0.0	23/16/11
01964015	0.02	15	1019	227.0	18.0	5.0	0.0	0.0	17/15/11
01964016	0.08	8	4054	1110.0	88.0	18.0	1.0	0.0	19/17/14
01964017	0.19	47	9400	586.0	38.0	9.0	0.0	0.0	20/16/12
01964018	0.01	12	574	122.0	14.0	5.0	0.0	0.0	16/14/11
01964019	0.46	13	23027	7079.0	709.0	196.0	12.0	0.0	22/20/17
01964020	0.88	44	43785	2823.0	74.0	17.0	1.0	0.0	23/19/13
01964022	0.03	18	1368	206.0	20.0	7.0	1.0	0.0	18/15/11

Figure 10: MPC patches for samples 01964016 (MPC = 8) and 01961018 (MPC = 157).

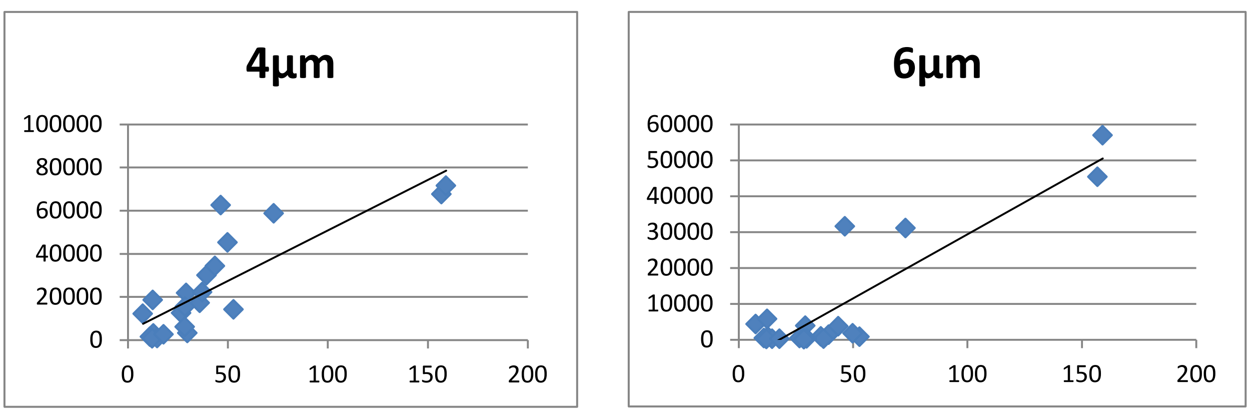
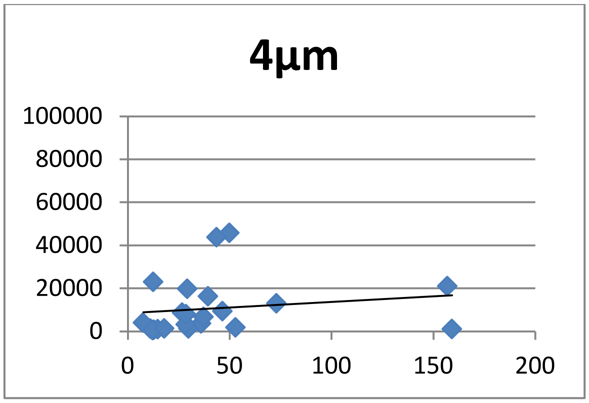


Figure 11: 4µm and 6µm particle count data from the Hiac 8012 particle counter correlated against MPC results.



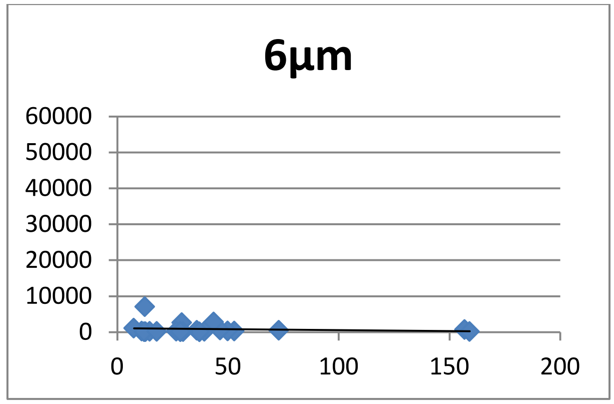


Figure 12: 4µm and 6µm particle count data from the CINRG CS-APC-2 particle counter correlated against MPC results.

4. Heavily Contaminated Samples

A customer of the new gearbox was concerned about the amount of visible metal debris in the oil, so the manufacturer was monitoring the gearbox using oil analysis. The manufacturer stated that the visible debris in the oil is the result of the gears not having been properly ground prior to installation and stated that this is normal for small gearboxes. The manufacturer wanted to determine if the amount of ferrous debris decreased with subsequent oil changes, and so was interested in the quantity and size of the metal particles in the oil. The samples used in this study were taken over a 5 month period in which the oil was not changed in the gearbox. Samples were diluted using a positive displacement pipette (1 ml of oil for a 1:30 dilution ratio) and run on the CS-APC-2 using the manual dilution mode for very heavily contaminated oil samples.

The trend of the samples over a 5 month period was excellent considering the high amount of visible metal present in the oil. The ISO code trend ranged only from 28/27/22 to 28/27/23 for the three samples. At a dilution level of 1:30 the Klotz sensor can count 750,000 particles per channel before coincidence occurs allowing the CS-APC-2 to produce trendable counts on samples with an ISO code as high as 28/27/23.

Table 10: Particle Testing results for heavily contaminated gearbox samples at 1:30 dilution ratio.

Count Data	4µm (c)	6µm (c)	14µm (c)	21µm (c)	38µm (c)	70µm (c)	Cleanliness Code
Current	1906022	732558	31188	3536	44	4	28/27/22
Historical 1	1864264	846612	53158	9111	60	0	28/27/23
Historical 2	1731806	683214	40629	7763	93	10	28/27/23

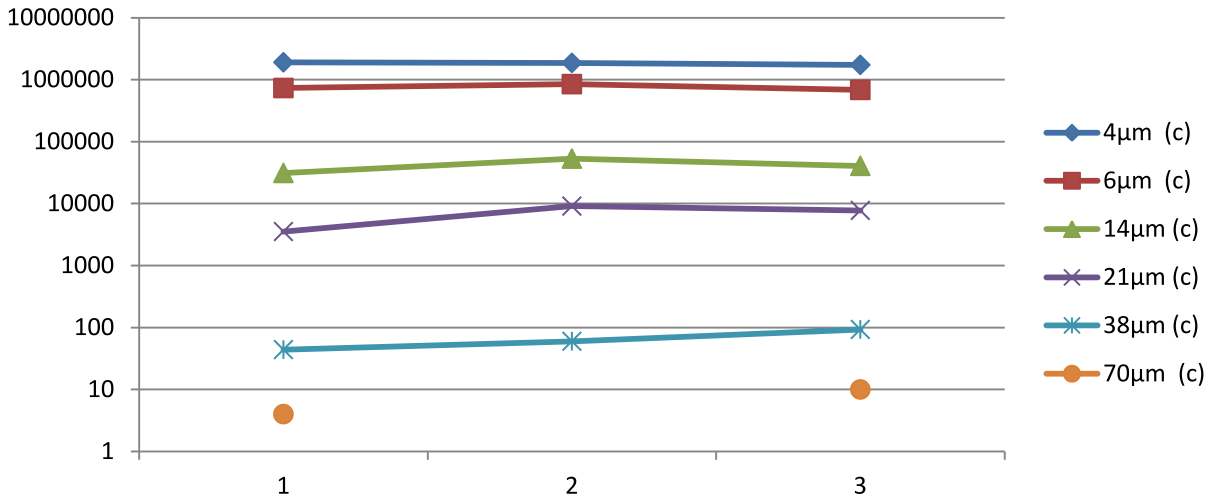


Figure 15: *Trend of 3 samples over a 5 month period from a heavily contaminated gearbox*.

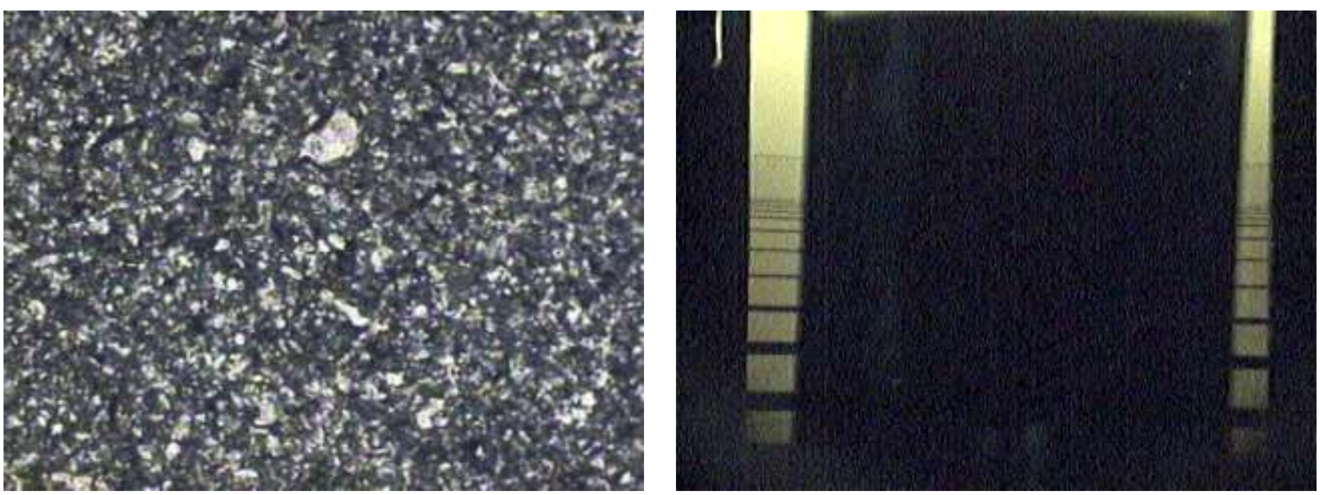


Figure 17**:** *A particle debris patch (left) and sample visual clarity image (right) of the most current gearbox sample illustrating the high quantity of metal present in the oil (particle debris patch 100x magnification, reflected white light).*

5. Conclusion

A number of routine oil samples tested by commercial oil analysis laboratories are considered to contain too much water, visible debris/metal or are too dark (or opaque) to be tested by a conventional optical particle counter instrument. Samples with water contamination or that are opaque when processed using a conventional particle counter result in erroneously high particle counts. Samples that are too heavily contaminated saturate the particle counter instrument resulting in a failed count, or produce poor results due to high levels of coincidence.

This presents an issue as customers of filtered industrial equipment require particle count data and more specifically ISO Cleanliness Codes in order to determine the maintenance activity to be carried out on a specific component. When an optical particle count cannot be performed, the laboratory must resort to producing a particle debris patch and manually assess the particle counts by reviewing the patch using a microscope. The results of this manual analysis of the patch are typically not in-line with the historical particle count results, and as a result are not trendable and generally provide for a sub-standard quality of analysis.

Processing of heavily contaminated oil samples using an automatic auto-diluting instrument provides accurate particle count results for samples that are opaque, heavily contaminated with debris and/or metal or contain high levels of emulsified water. For samples that contain interfering additives and/or high amounts of oxidation byproducts (i.e. varnish precursors) the dilution improves the solubility of the additives and oxidation byproducts in the resulting solution eliminating the effect of "soft" particles to produce accurate particle counts. Normally it would not be possible to perform particle counts on oil samples heavily contaminated with debris and/or metal, however with the use of a manual dilution mode using a positive displacement pipette to achieve a high but accurate dilution ratio, we were able to produce particle counts on samples with very high ISO Cleanliness Codes that are both accurate and trendable.

Additionally, as a result of the ability to perform particle counting on heavily contaminated samples, the oil analysis laboratory involved in the test was able to reduce the number of particle debris patches requiring manual particle count by 88% through the introduction of the CINRG CS-APC-2 instrument.

References:

1. ASTM Standard D7647, 2010, "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", ASTM International, West Conshohocken, PA, 2010, DOI: 10.1520/D7647-10, www.astm.org.