VERSION [1.1]

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PROCESSING CONTAMINATED SAMPLES

CINRG CS-APC-2 WHITE PAPER

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PROCESSING CONTAMINATED SAMPLES

INTRODUCTION

The CINRG CS-APC-2 particle counting system is a fully automated system that meets the requirements of ASTM D7647-10 in which oil samples are 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 white paper samples with high water contamination, opaque samples and heavily contaminated samples were tested to compare the results from the CS-APC-2 versus other particle counters.

CINRG CS-APC-2 FULLY AUTOMATIC AUTO-DILUTING PARTICLE COUNTER

A. 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%.

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% Iso-propanol) 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 CS-APC-2 using 100% Toluene as the diluents.

A. RESULTS

Particle count results from the CS-APC-2 for remained within repeatability limits for the samples prepared with 0.5% up to and including the sample prepared with 2.0% water. At 2.5% a sharp increased in the amount of particles counted was observed at 4, 6, 14 and 21 microns.

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

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

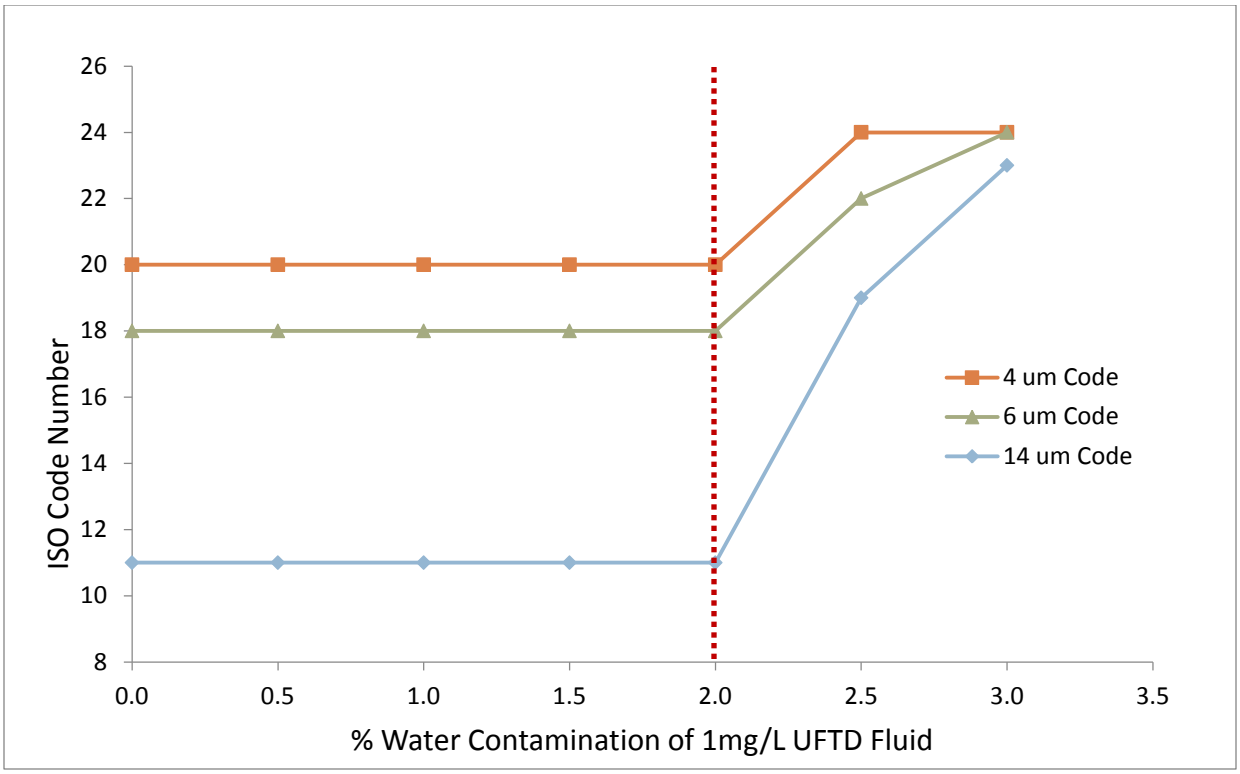


Figure A.1 – *ISO Code results from trial of 0.5% to 3.0% water in MIL-H-5606 oil using 75% Toluene / 25% Iso-propanol as a solvent*.

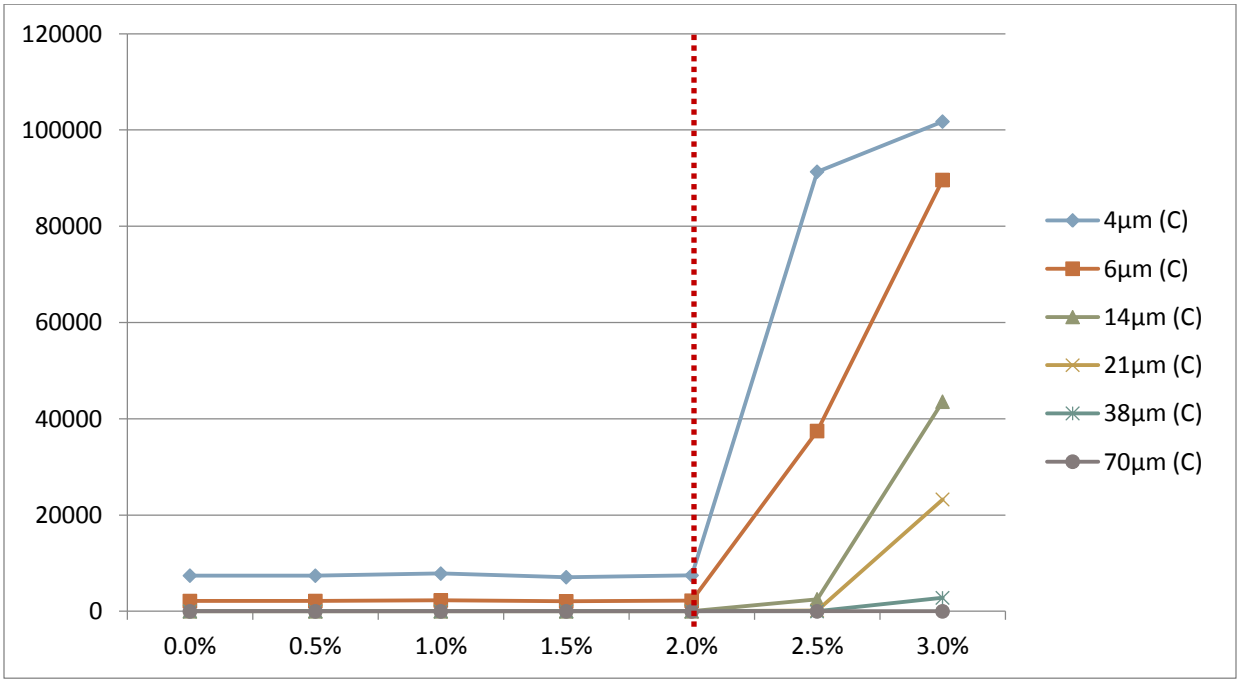


Figure A.2 – *Individual micron channel results from trial of 0.5% to 3.0% water in MIL-H-5606 oil using 75% Toluene / 25% Iso-propanol as a solvent*.

B. OPAQUE OIL SAMPLES

Several opaque samples of Castrol Optigear Synthetic A ISO 320 from wind turbine gearboxes were selected to be tested undiluted on a conventional particle counter versus diluted on the CS-APC-2 instrument. The opaqueness of the samples is due to poor solubilization of the additives. Figure B.1 (below) shows the color image of the samples and the resulting particle debris patches.

Samples were tested on a Hiac Royco SDS particle counter unit (undiluted), and then tested using the CINRG CS-APC-2 unit with the standard 1:1 dilution.

B. RESULTS

Table B.1 – 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 B.2 – 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

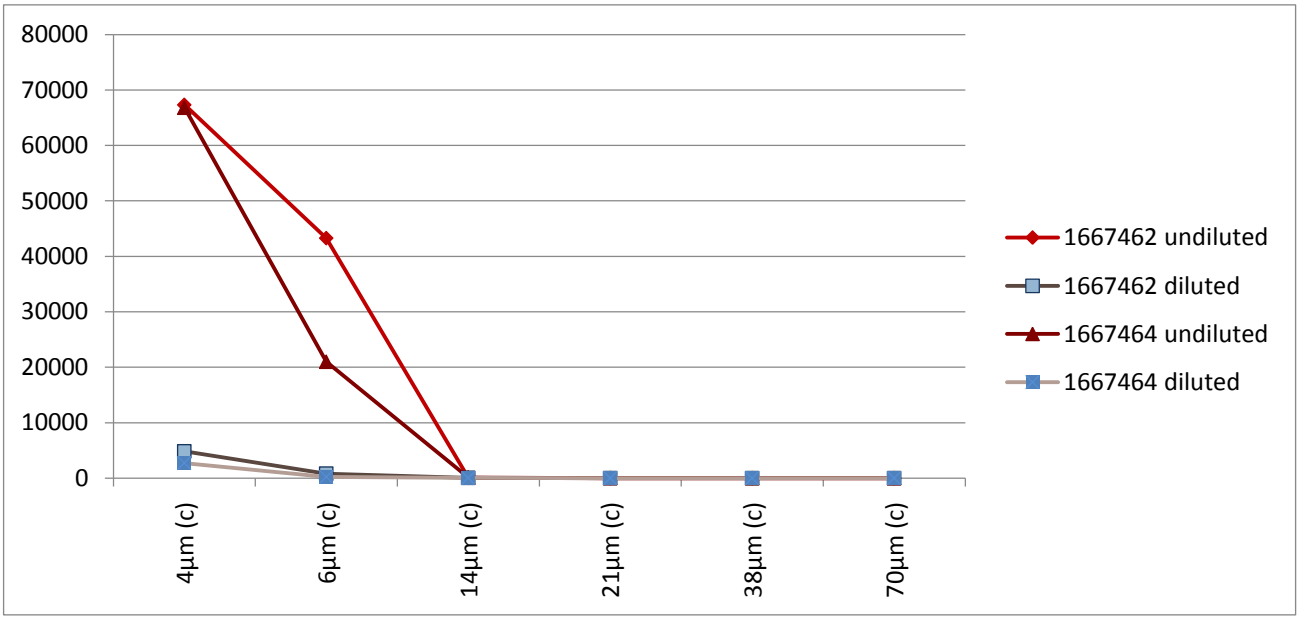


Figure B.1 – *Comparison of undiluted particle counts on the Hiac Royco SDS particle counter versus diluted particle counts for Castrol Optigear Synthetic A ISO 320 on the CS-APC-2 particle counter.*

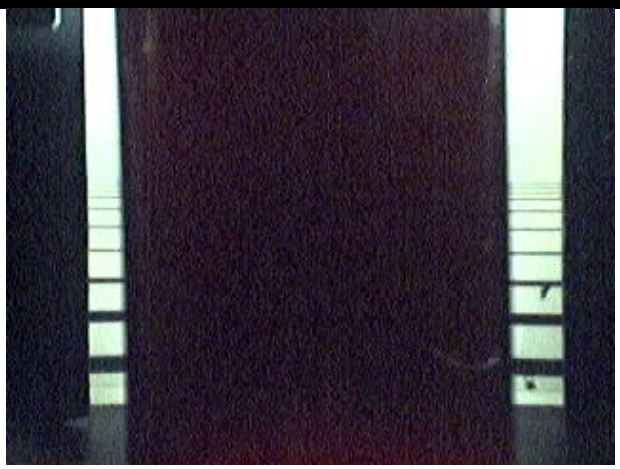
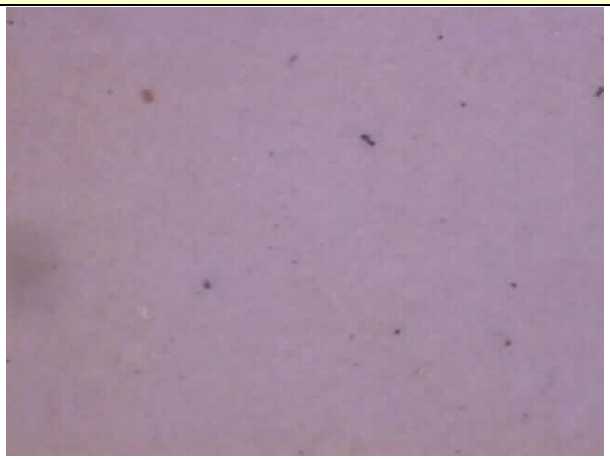
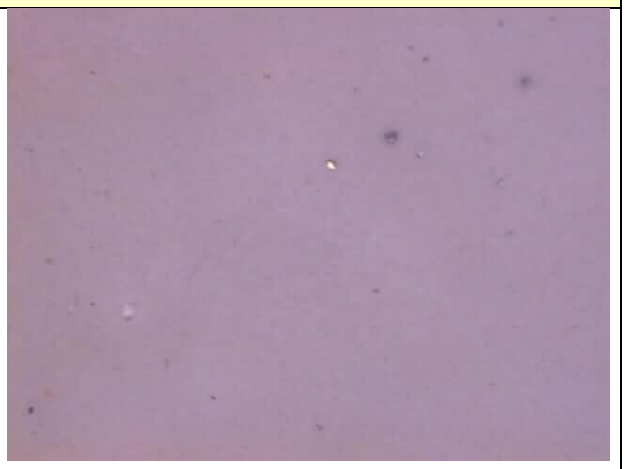
Sample 01667462	Sample 01667464
	
Color Image with Visibility Scale	Color Image with Visibility Scale
	
Particle Debris Patch @ 100X magnification	**Particle Debris Patch @ 100X magnification**

Figure B.1 - *Color images of the samples 01667462 and 01667464 with the resulting particle debris patches.*

C. HEAVILY CONTAMINATED SAMPLES

The customer of the new gearbox was concerned about the amount of visible metal debris in the oil, so the manufacturer has been monitoring the gearbox with oil samples. The manufacturer states that the visible debris is the result of the gears not having been ground prior to installation and states that this is normal for small gearboxes. The manufacturer wants to determine if the amount of ferrous debris decreases 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.

C. RESULTS

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 of 28/27/23.

Table C.1 – 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

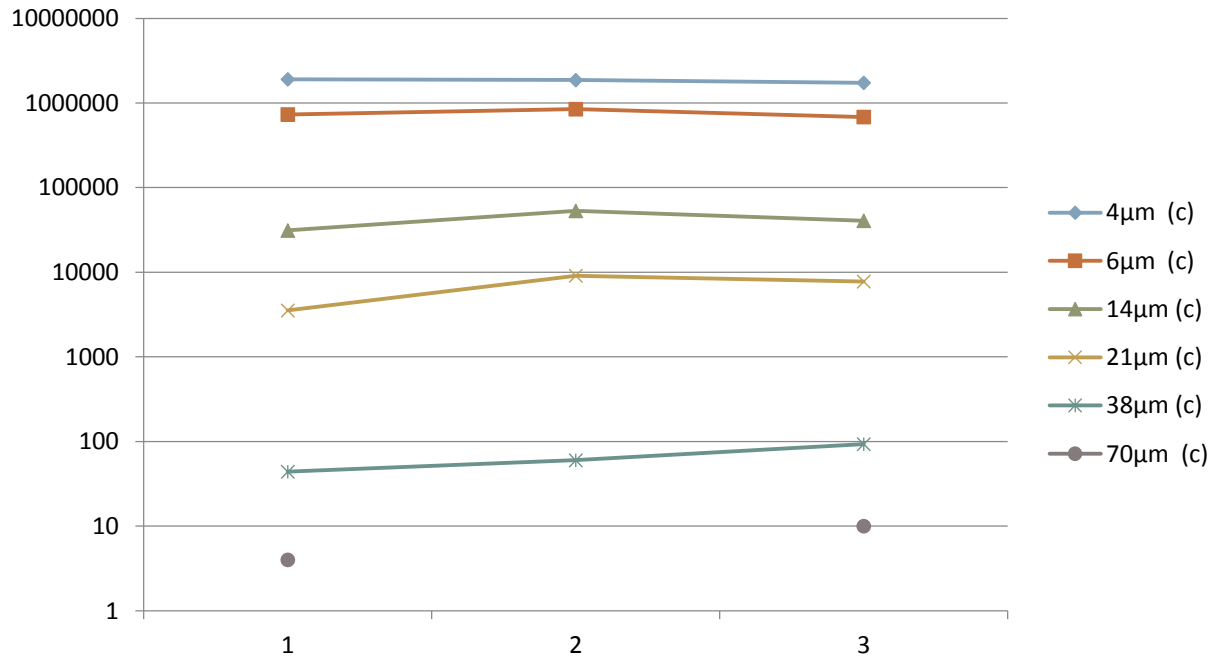


Figure C.1 – *Trend of 3 samples over a 5 month period from a heavily contaminated gearbox*.

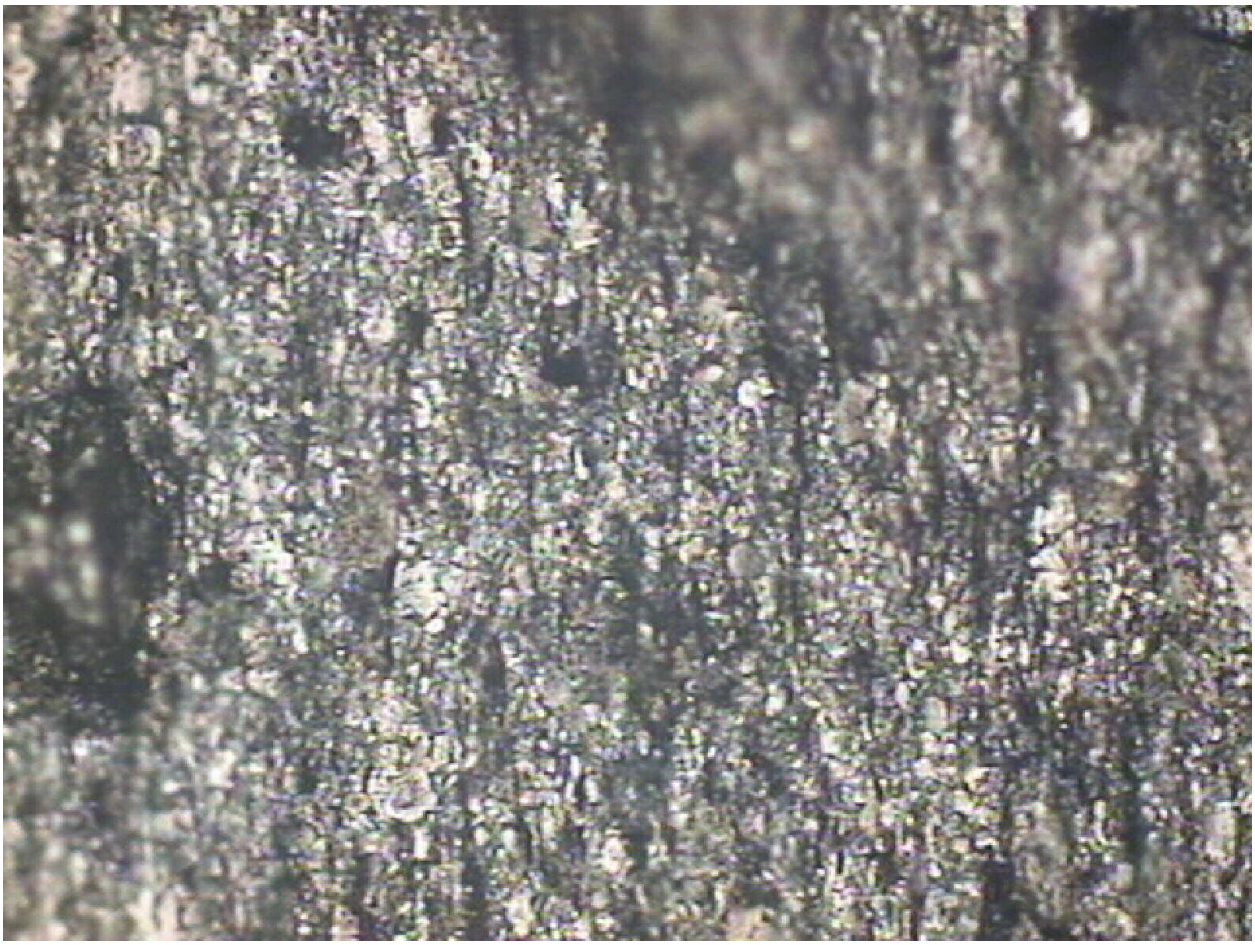


Figure C.2 – *A ferrogram of the most current gearbox sample illustrating the high quantity of metal present in the oil (200x magnification, bichromatic light, sample prepared with 1:10 dilution).*

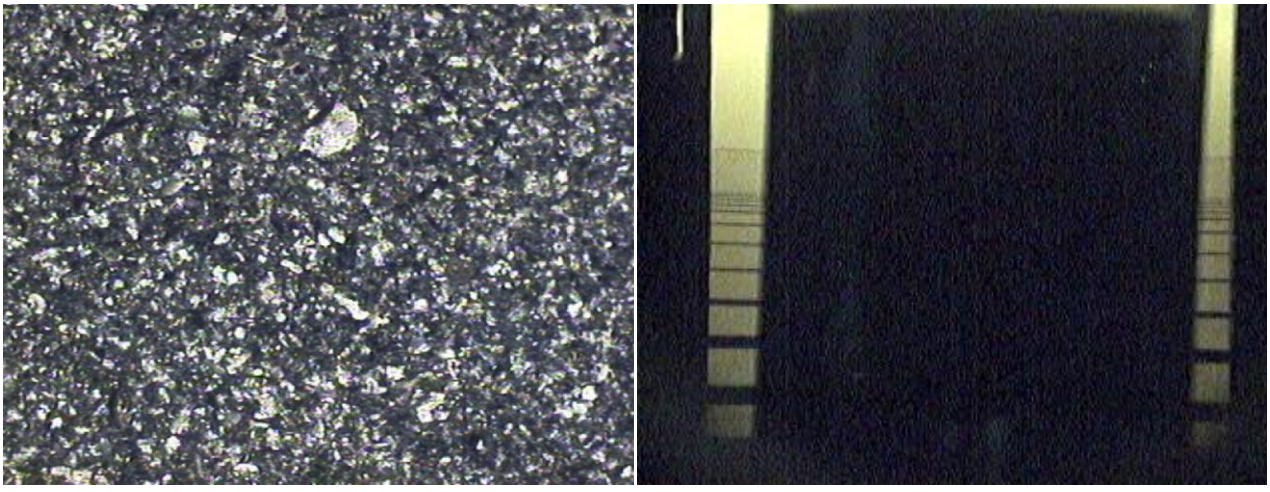


Figure C.3 – *A particle debris patch (left) and sample visual clarity image (right) of the most current gearbox sample illustrating the high quantity of metal present in the oil (particle debris patch 100x magnification, reflected white light).*

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, are not usually trendable and generally provide for a sub-standard quality of analysis.

Processing of heavily contaminated oil samples using the automatic auto-diluting CINRG CS-APC-2 instrument provides accurate particle count results for samples that are opaque, are heavily contaminated with debris/metal or contain as much as 2.0% water contamination. In the case of opaque samples, the dilution improves the solubility of the additives in the resulting solution eliminating the effect of “soft” particles. With respect to oil samples heavily contaminated with debris/metal it would not normally have been possible to perform particle counts on these samples, however with the CS-APC-2 using the manual dilution mode (1ml of sample dispensed with a positive displacement pipette) to achieve a high but accurate dilution ratio, we were able to produce accurate particle counts. The result is trendable and meaningful particle count data.

The oil analysis laboratory involved in the test was able to reduce the number of particle debris patches produced in the laboratory by 88% by introducing the CS-APC-2 instrument. Those samples are now routinely tested on the CS-APC-2 instrument.

FURTHER READING

CINRG CS-APC-2 Tech Data – http://www.cinrg.com/docs/CINRG-CS-APC-2.pdf

CINRG CS-APC-2-Overview – http://www.cinrg.com/docs/CINRG-CS-APC-2-WHITE-PAPER.pdf

CONTACT INFO

For more information the CINRG CS-APC-2 particle counter contact Bill Quesnel at;

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