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# Performing Particle Counting on Opaque and Heavily Contaminated Samples

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# What is the biggest issue with Particle Count Testing?

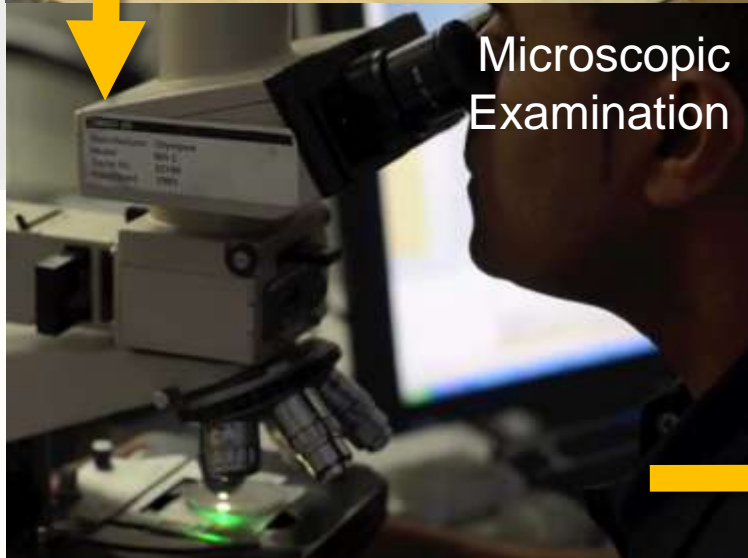
**Reproducibility of Results is Questionable!**

# Why?

Different Methods Employed by Commercial Laboratories each with their sources of variability;

- **ASTM F312 – Particle Debris Patch & Visual Count (Microscopic)**
  - Time consuming and expensive
  - Operator dependent
  - Precision of measurement is questionable
  
- **No Method – Pore Blockage**
  - Current technology is now obsolete
  - Fairly accurate for total particles, but high error for particle size distribution
  
- **ISO 4406:1987 – Optical Particle Count 5 $\mu$ m/15 $\mu$ m....maybe 2 $\mu$ m/5 $\mu$ m/15 $\mu$ m**
  - Discontinued method
  - Many instruments in the field may still be calibrated to this method
  - Coincidence - cannot count dark or opaque samples, or fluids with water > 0.03%
  - “Soft” particle effects from oxidative byproducts (varnish precursors, some additives)
  
- **ISO 4406:1999 – Optical Particle Count 4 $\mu$ m/6 $\mu$ m/14 $\mu$ m**
  - Coincidence - cannot count dark or opaque samples, or fluids with water > 0.03%
  - “Soft” particle effects from oxidative byproducts (varnish precursors, some additives)

Particle Debris Patch



## What Did we Study?

Diluted Samples (Automatic) vs. Undiluted Samples (Manual)

- Samples with a lot of **water contamination**
- “**Soft**” **Particle** Effects - Samples with oxidative byproducts (varnish)
- **Coincidence** Issues - Samples with a whole lot of particulate

Automatic Diluted Samples

- Testing **Repeatability**

# Hiac 8012 vs. CINRG CS-APC-2



**Undiluted**



**Diluted 1:1**



## Processing Samples

*Water Contamination - Laboratory Samples*



## Processing Samples

### *Water Contamination*

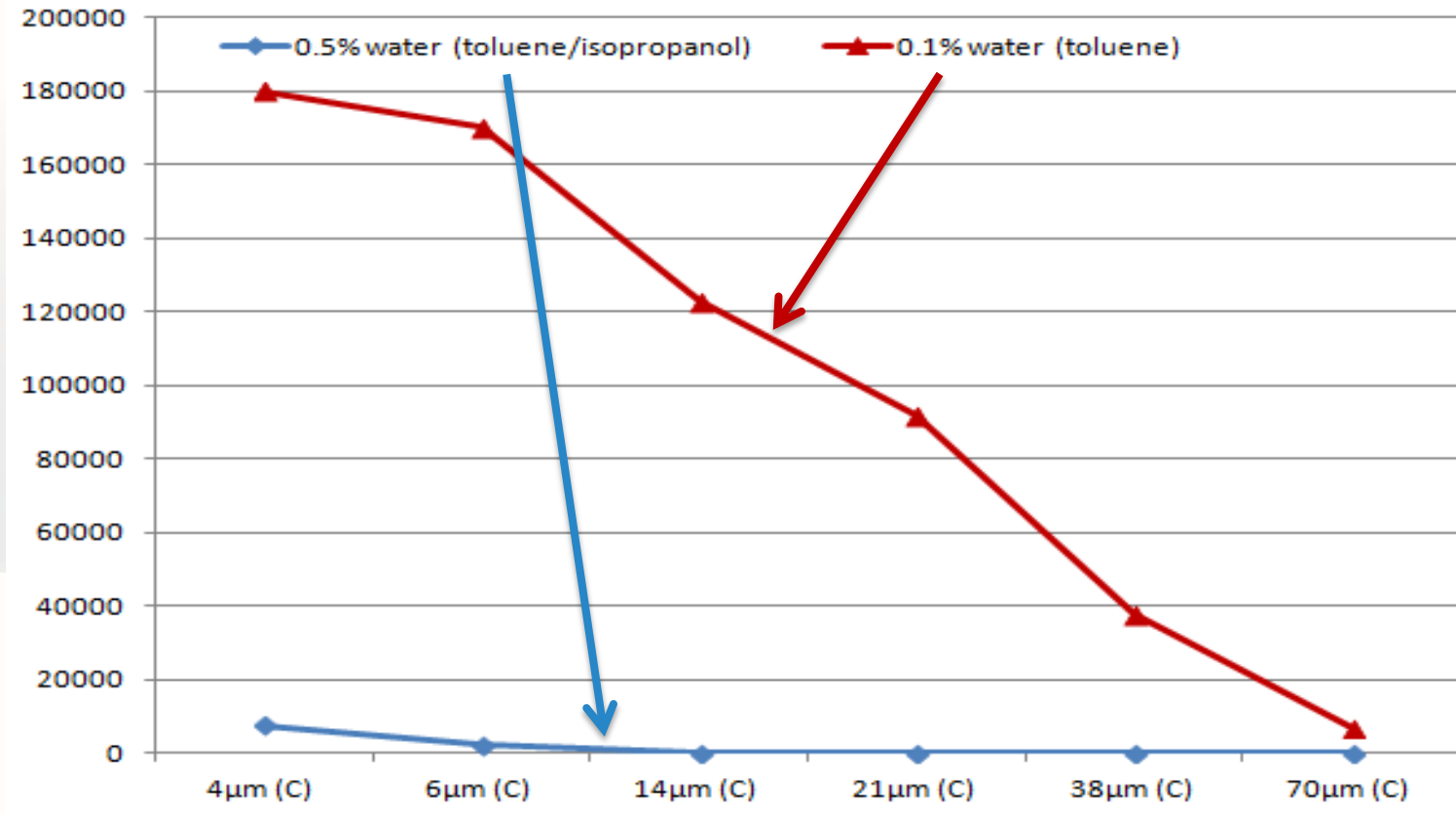
Sample ID	% Water	4µm (c)	6µm (c)	14µm (c)	21µm (c)	38µm (c)	70µm (c)	Cleanliness Code
RM8632-01T	0.1%	180022	170028	122925	91843	37729	6798	25/25/24

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



# Processing Samples

## Water Contamination



**Figure 5** – 0.1% water contamination using 100% toluene as a diluent vs. 0.5% water contamination using 75% toluene / 25% isopropanol as a diluent.

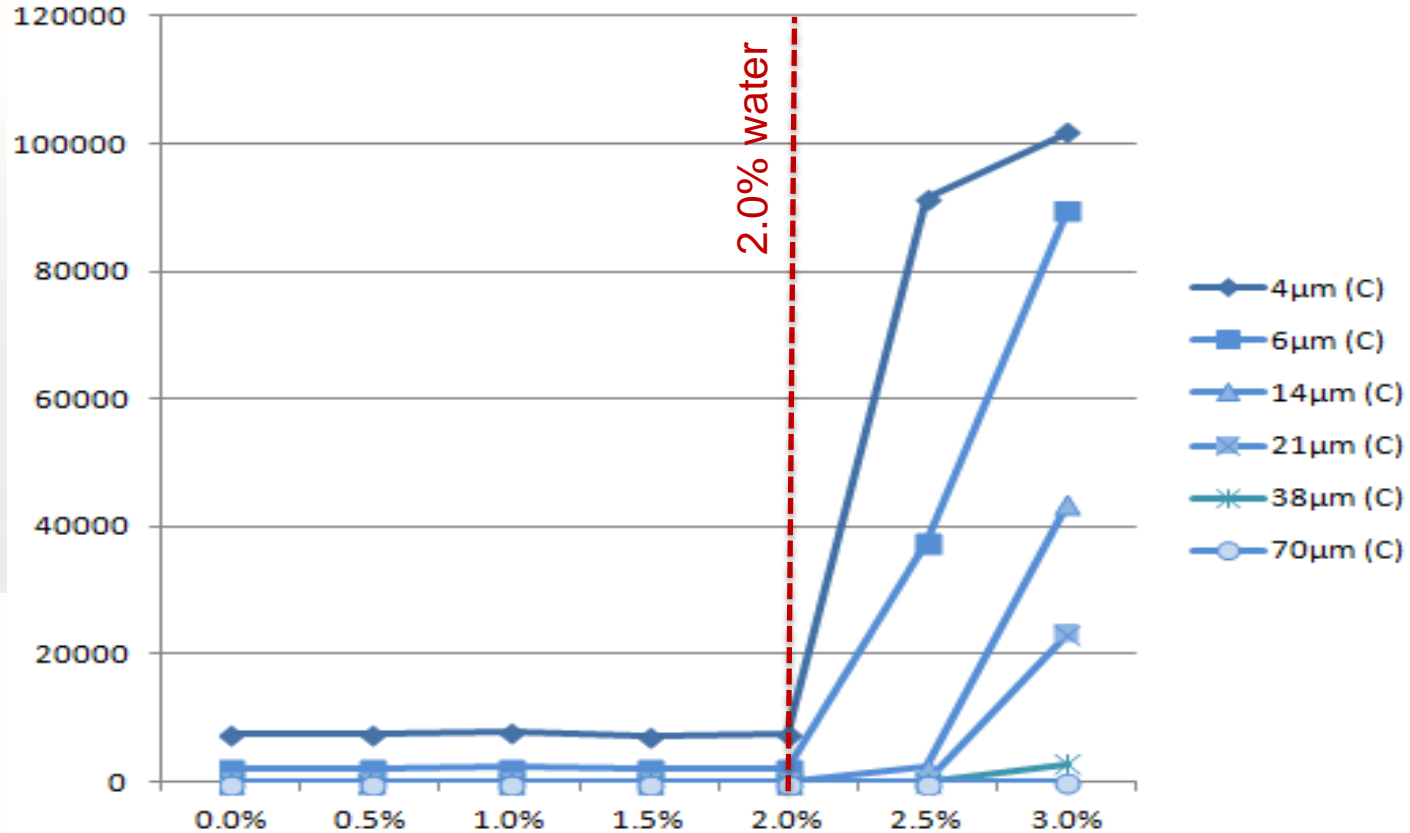
## Processing Samples

### *Water Contamination*

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

## Processing Samples *Water Contamination*



**Figure 4** – 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.

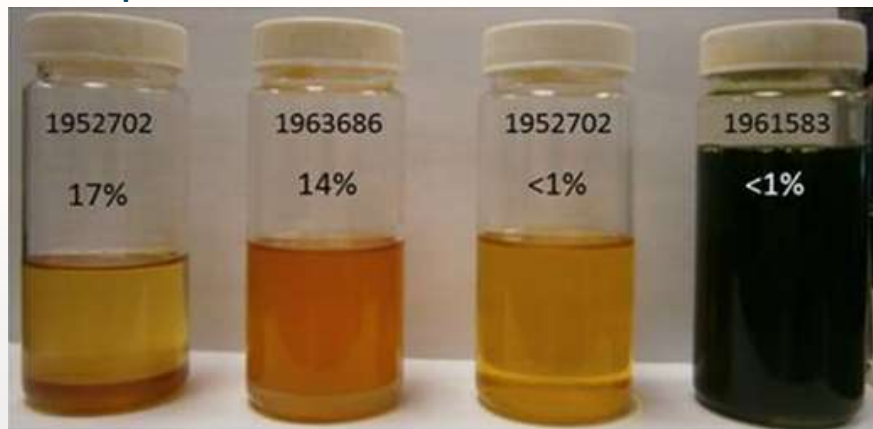


## **Processing Samples**

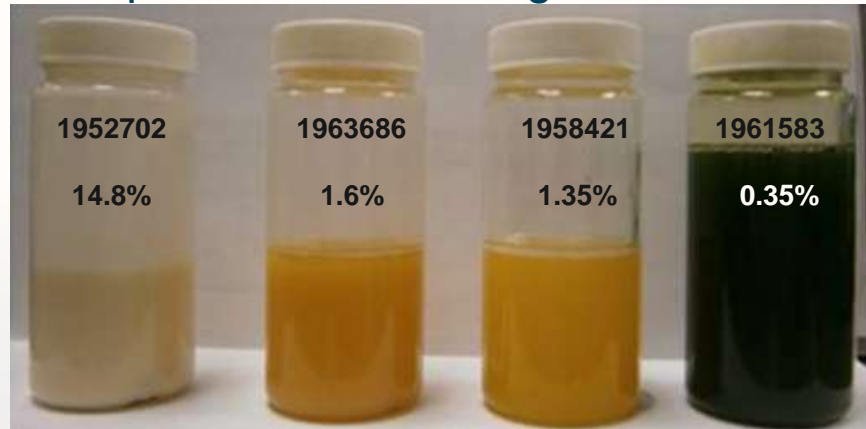
### *Water Contamination in Used Oil Samples*

## Processing Samples - Water Contamination in Used Oil Samples

Samples – As Received



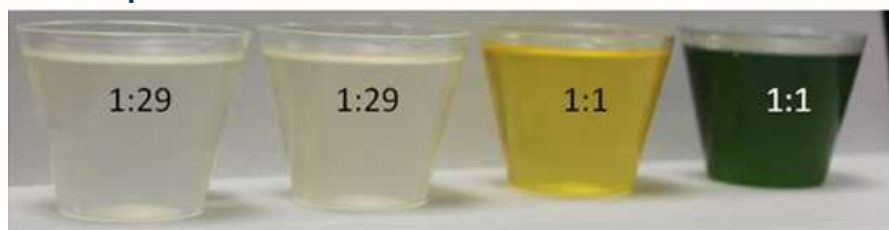
Samples – After Shaking



Samples – Before Dilution



Samples – After Dilution



01961583 & 01952702



## Processing Samples

### *Water Contamination*

**Table 3a** – Testing results from used oil samples with high concentrations of free and emulsified water on the **Hiac 8012** undiluted.

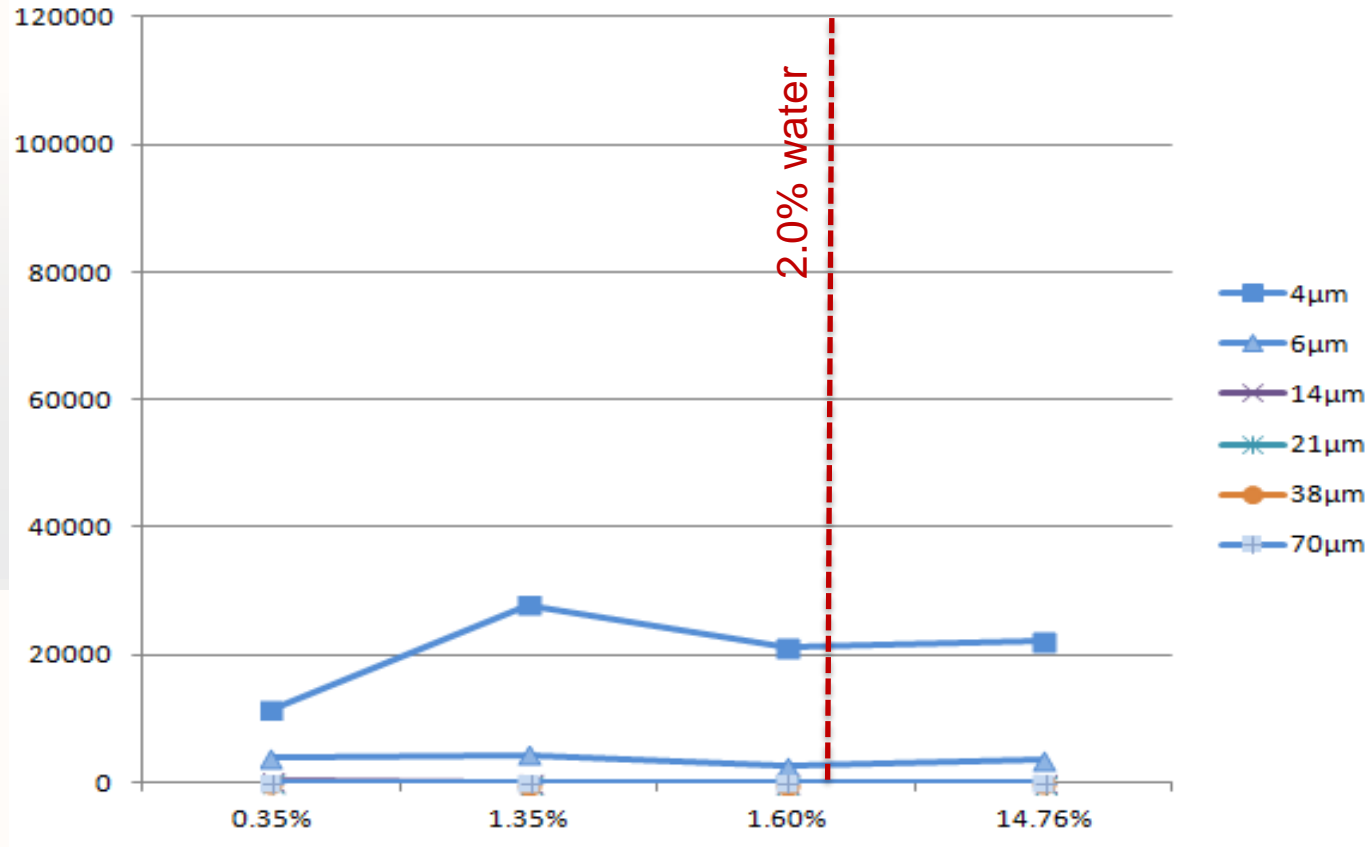
Sample ID	Free Water	% Water	Dilution Oil:Solvent	4µm (c)	6µm (c)	14µm (c)	21µm (c)	38µm (c)	70µm (c)	Cleanliness Code
1952702	17%	14.76%	none	0	0	0	0	0	0	0.7/0.7/0.7
1963686	14%	1.60%	none	0	0	0	0	0	0	0.7/0.7/0.7
1958421	<1%	1.35%	none	0	0	0	0	0	0	0.7/0.7/0.7
1961583	<1%	0.35%	none	0	0	0	0	0	0	0.7/0.7/0.7

**Table 3b** – Used oil samples with high concentrations of free and emulsified water on the **CINRG CS-APC-2** using 75% Toluene / 25% Isopropanol as a

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



## Processing Samples *Water Contamination*



**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.

A photograph of a laboratory rack containing several glass vials. The vials are arranged in two rows. The top row has three vials with white caps. The bottom row has three vials, with the middle one containing a yellowish liquid. A white label is attached to the front of the rack, and a small yellow tag with the number '2' is visible on the left side.

**Processing Samples**  
*Opaque Samples*

2 THU-12-Gear (3) 01738033-035

## Processing Samples

### *Opaque Samples (Castrol Optigear A 320)*

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

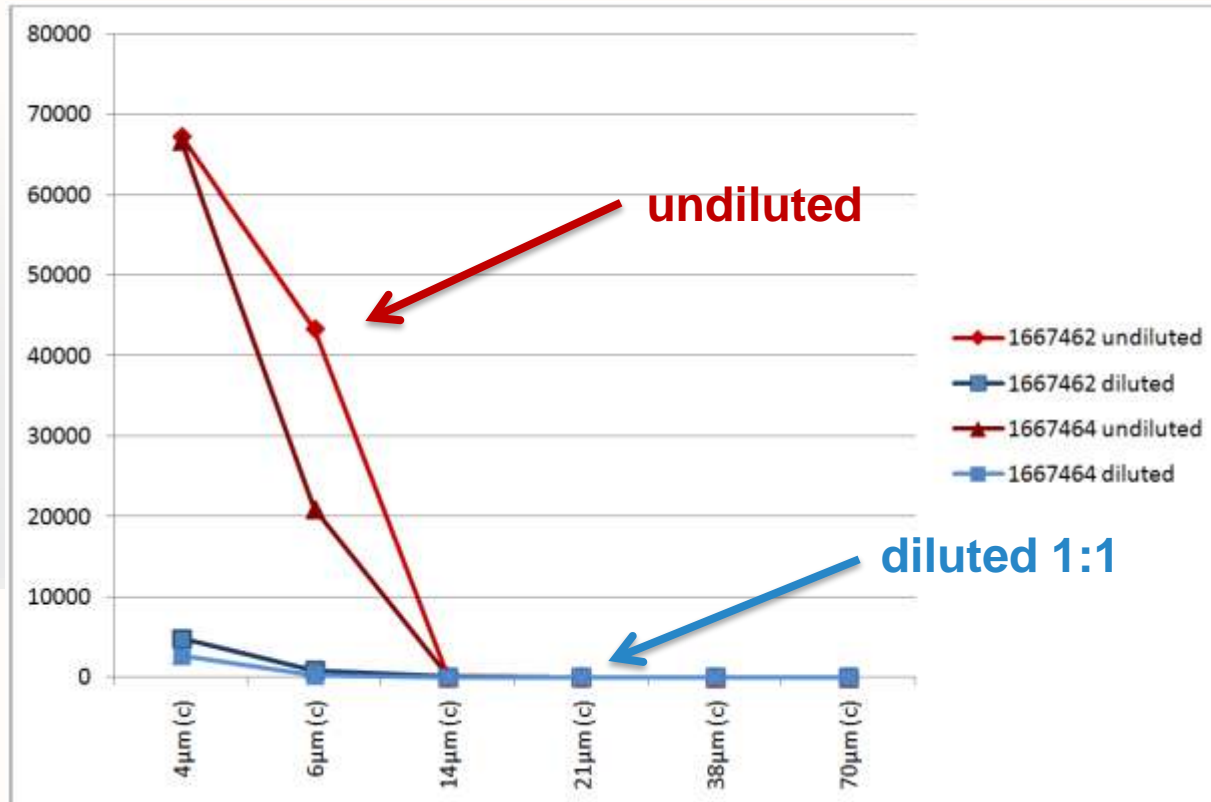
**Table 4** – Particle Testing results for both diluted and undiluted preparation for sample 01667462.

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

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

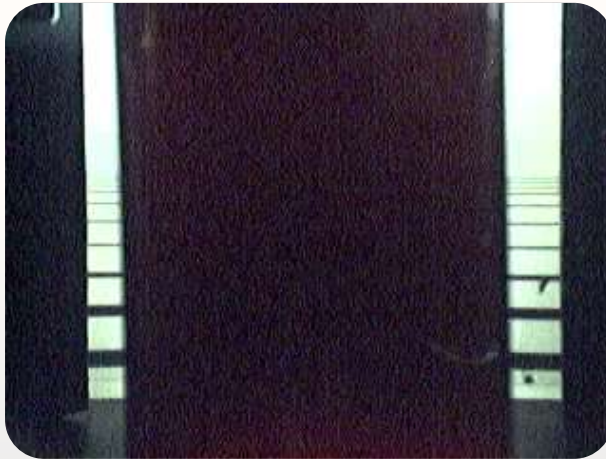
## Processing Samples

### *Opaque Samples (Castrol Optigear A 320)*



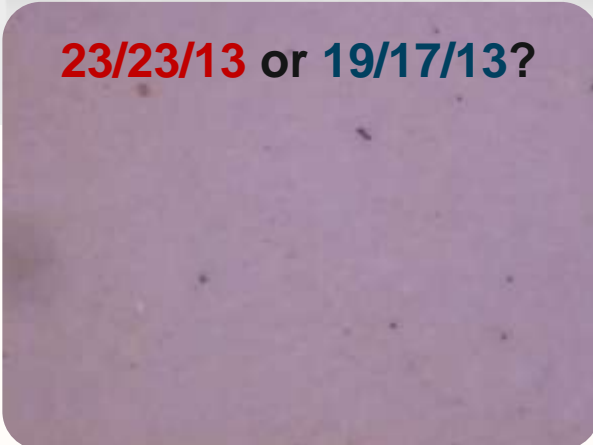
**Figure 8** – Comparison of undiluted particle counts on the Hiac Royco SDS particle counter versus diluted particle counts for Castrol Optigear Synthetic A ISO 320 on the CS-APC-2 particle counter.

## Sample 01667462



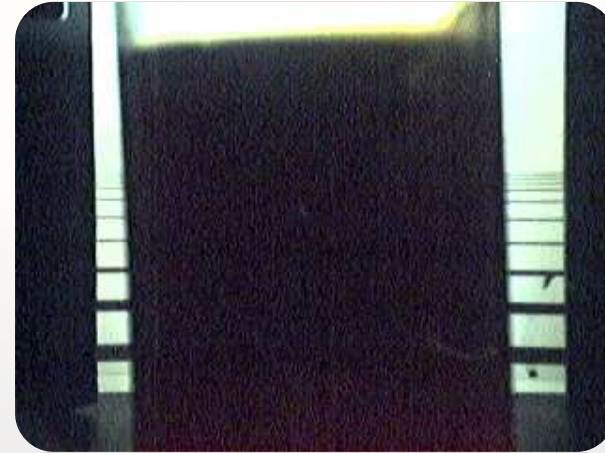
Color Image with Visibility Scale

**23/23/13** or **19/17/13?**



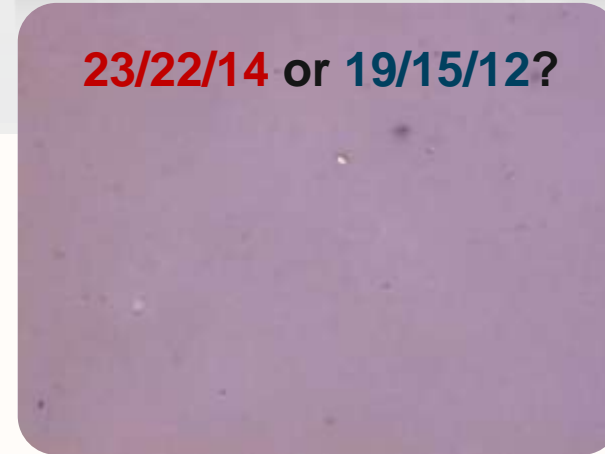
Particle Debris Patch @ 100X magnification

## Sample 01667464



Color Image with Visibility Scale

**23/22/14** or **19/15/12?**



Particle Debris Patch @ 100X magnification

**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 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

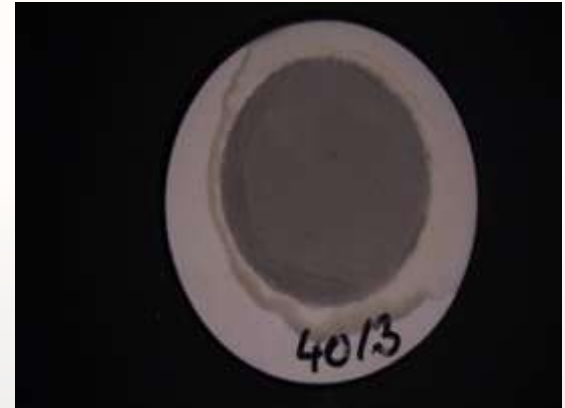
## MPC Images / Results



01964016 (MPC = 8)



01961018 (MPC = 157)



01964013 (MPC = 29)



01961020 (MPC = 73)



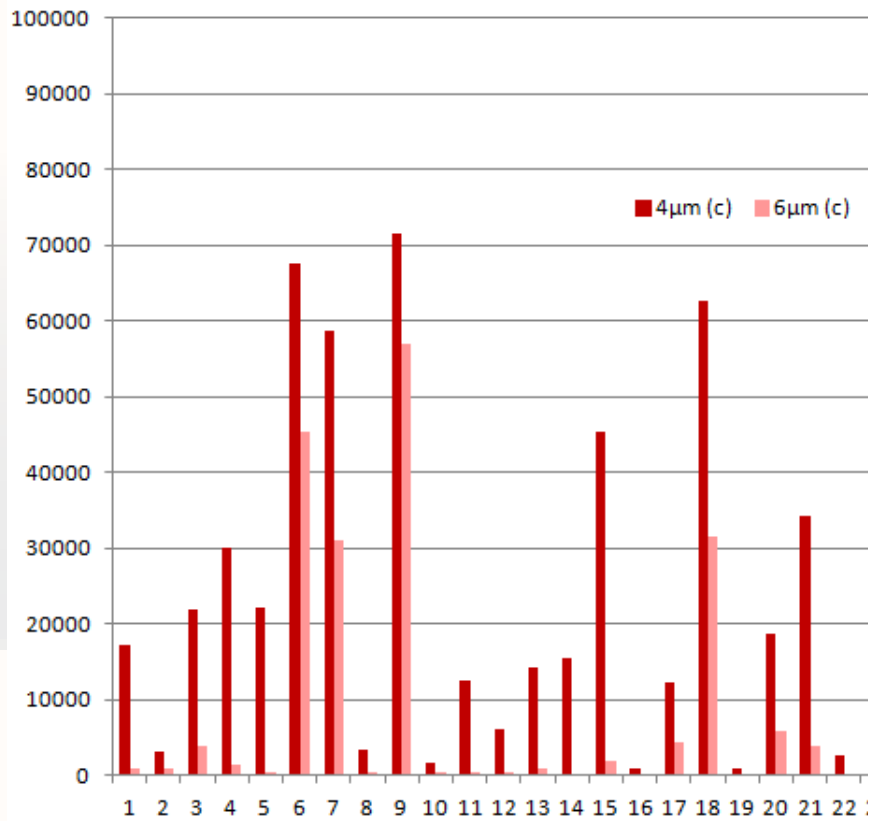
01961013 (MPC = 29)



01964012 (MPC = 53)

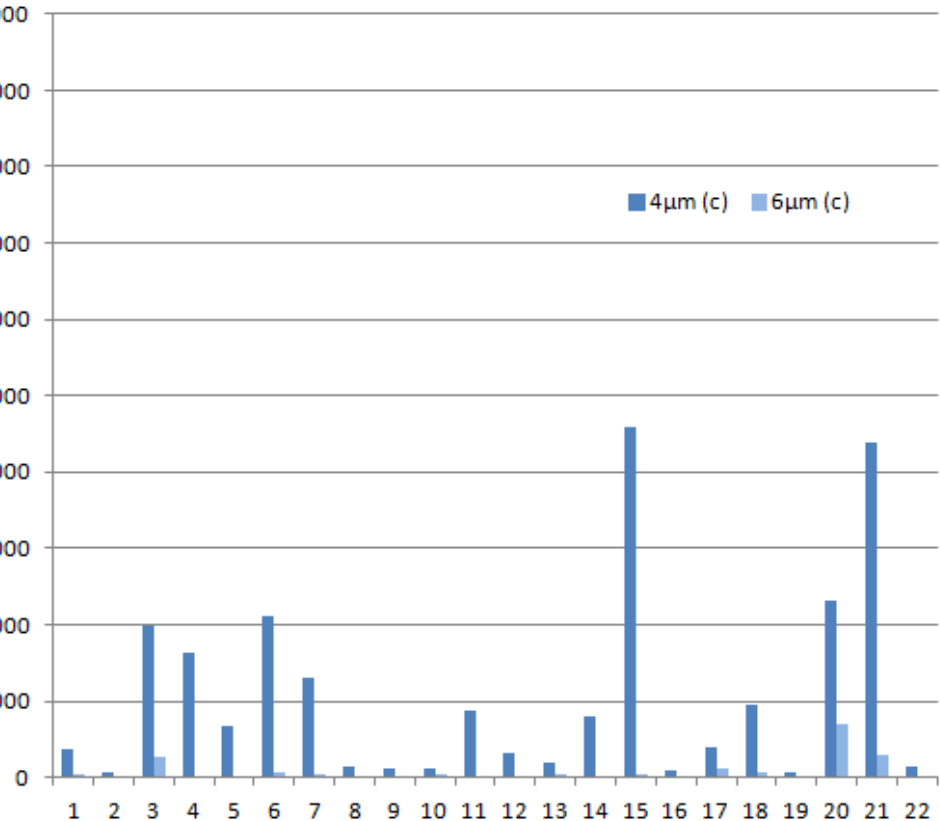
**Figure 10:** MPC patches for a variety of the gearbox samples tested

## HIAC 8012



**undiluted**

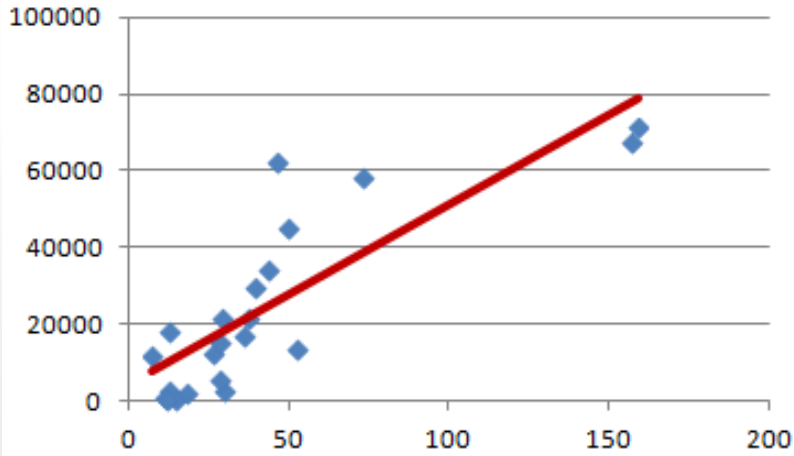
## CINRG CS-APC-2



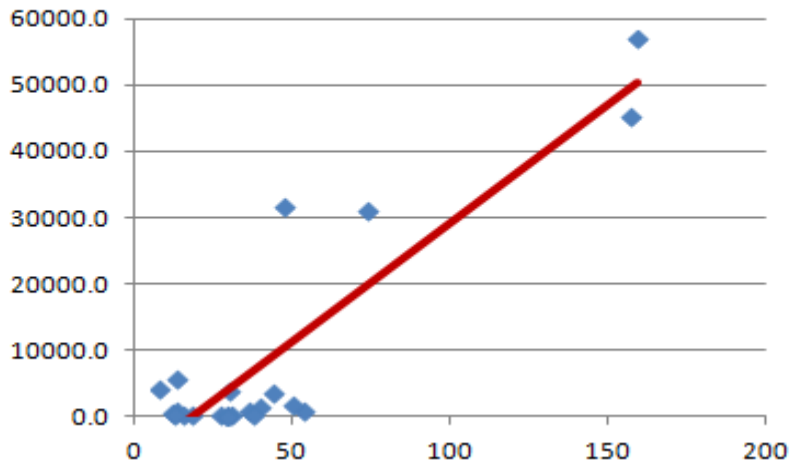
**diluted 1:1**

**HIAC 8012 undiluted**

**4 $\mu$ m**

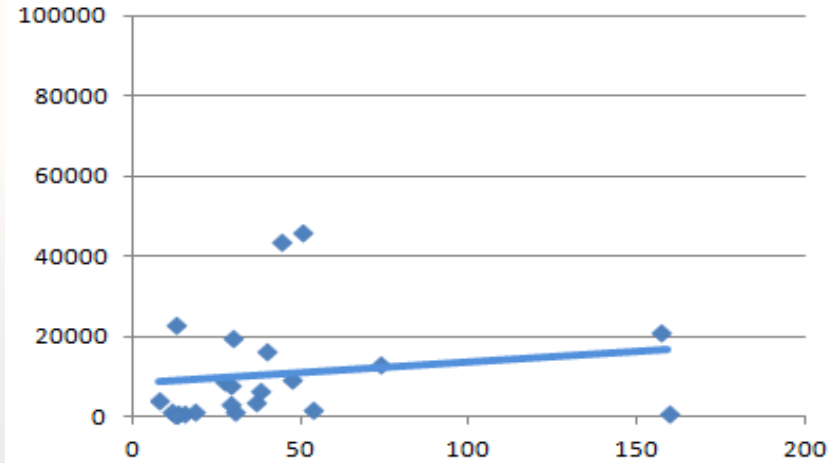


**6 $\mu$ m**

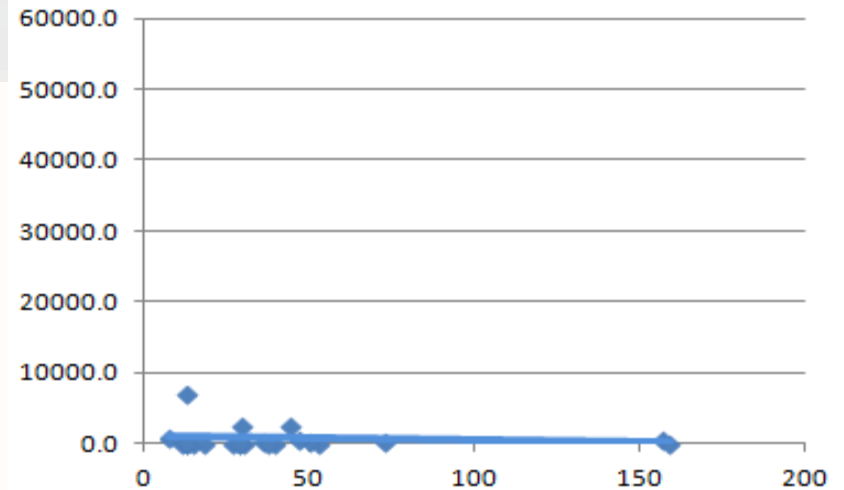


**CINRG CS-APC-2 diluted 1:1**

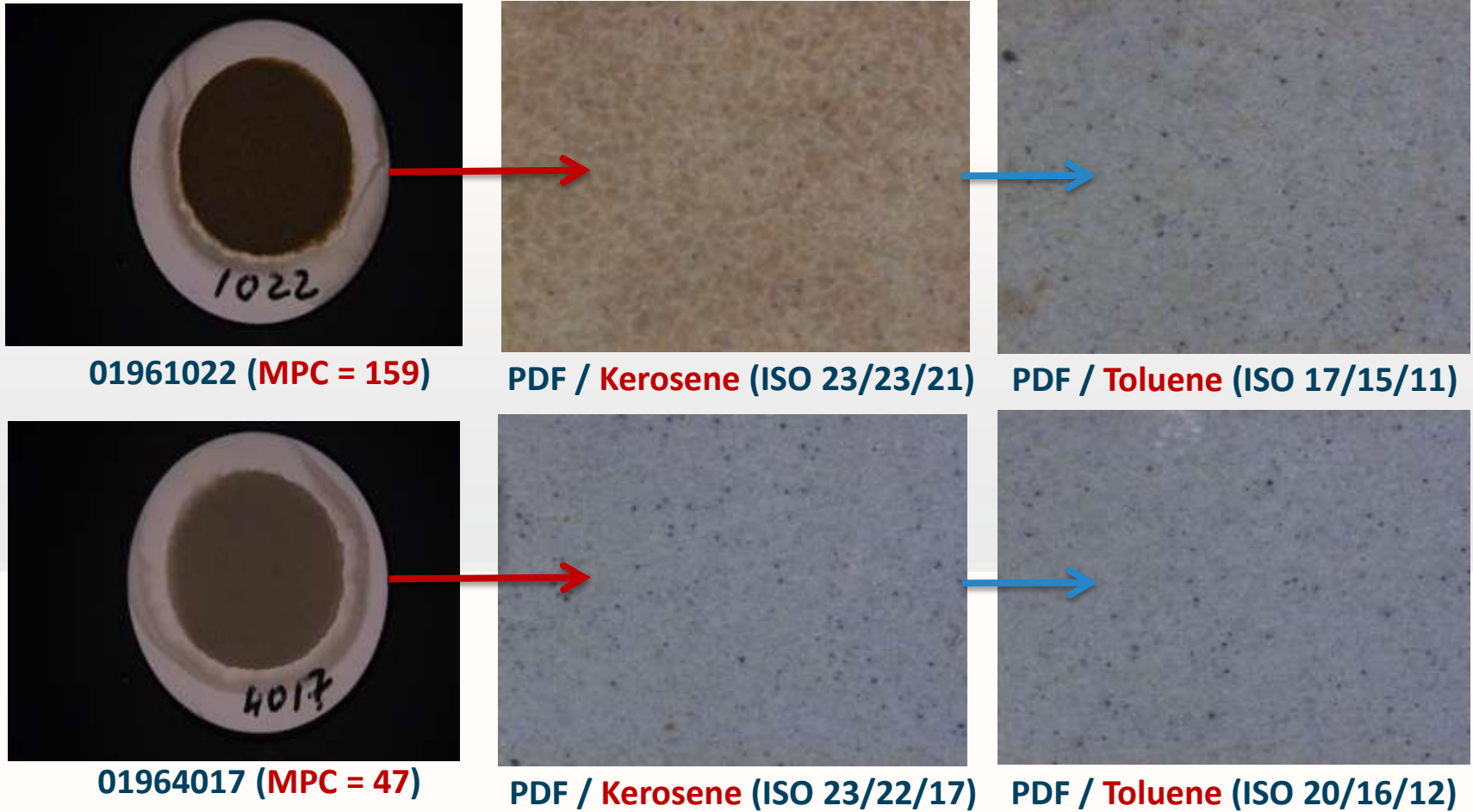
**4 $\mu$ m**



**6 $\mu$ m**

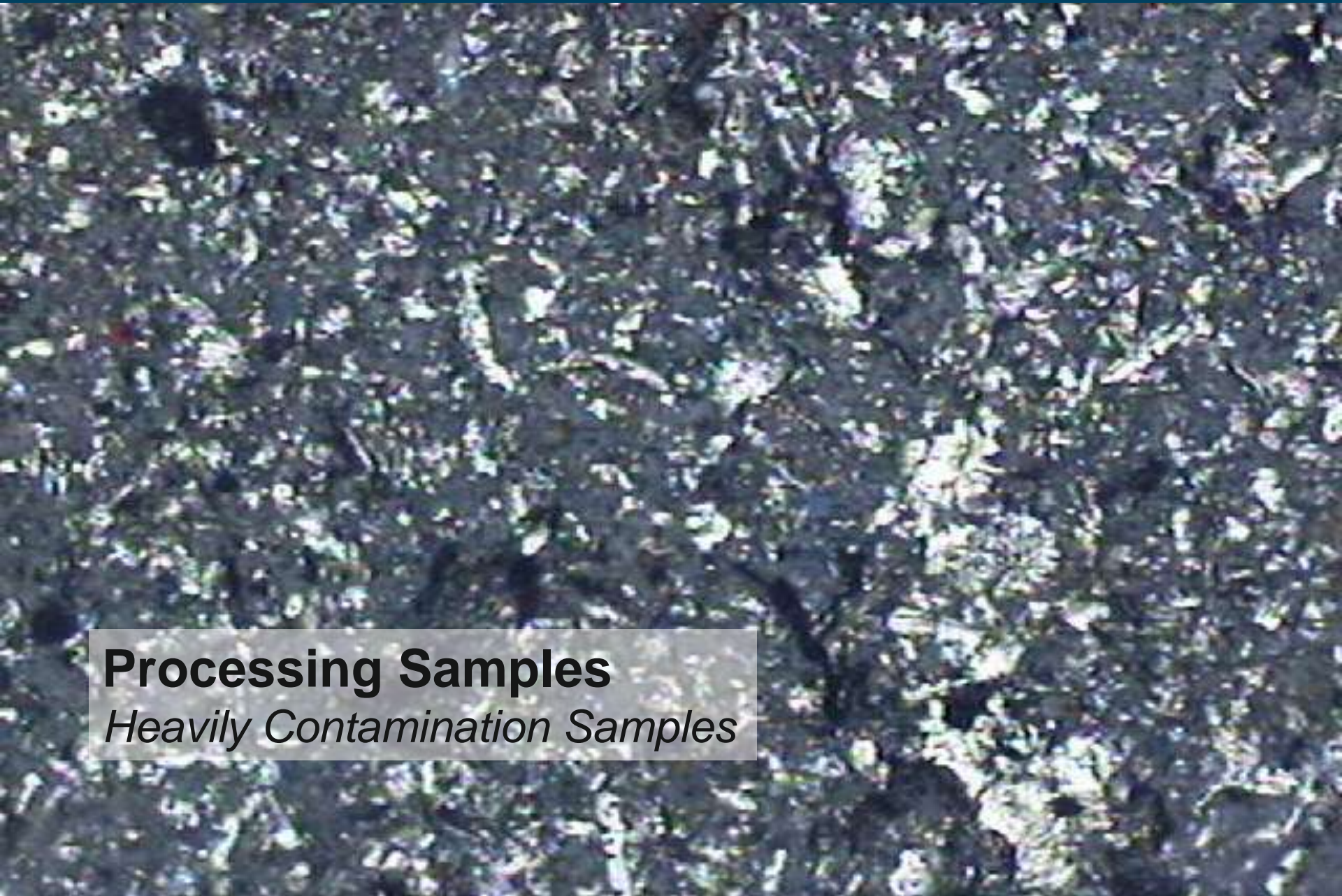


# Particle Debris Patches



**Figure 11:** Particle Debris patches for two of the gearbox samples tested comparing solvents.



A high-magnification microscopic image showing a dense, dark, and highly textured surface. The surface is covered with numerous small, irregular, light-colored particles and fibers, creating a complex, almost crystalline or fibrous structure. The overall appearance is that of a heavily contaminated or processed material.

**Processing Samples**  
*Heavily Contamination Samples*



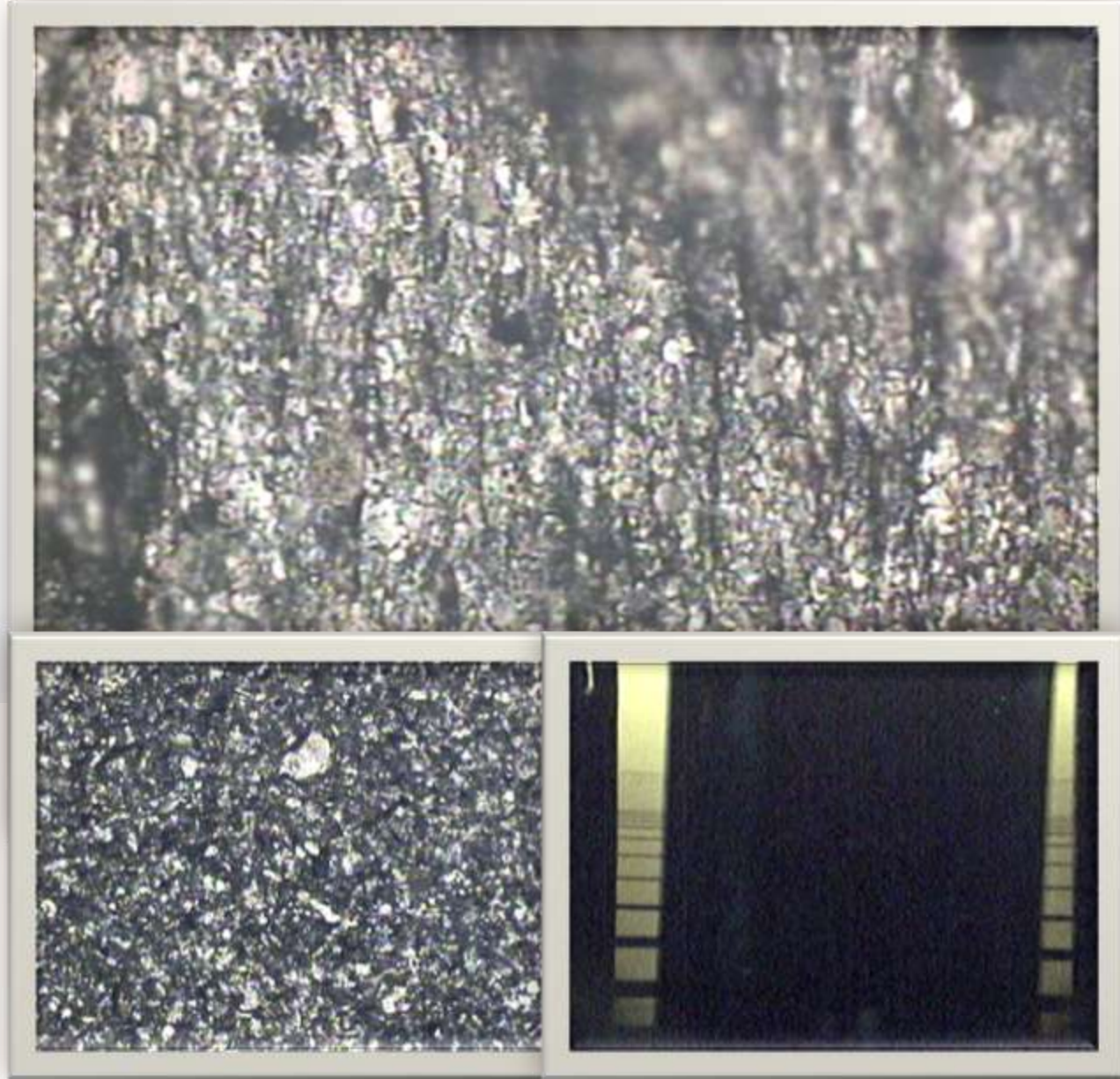
## Processing Samples

*Heavily Contaminated (Gross Particulate Concentration)*

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

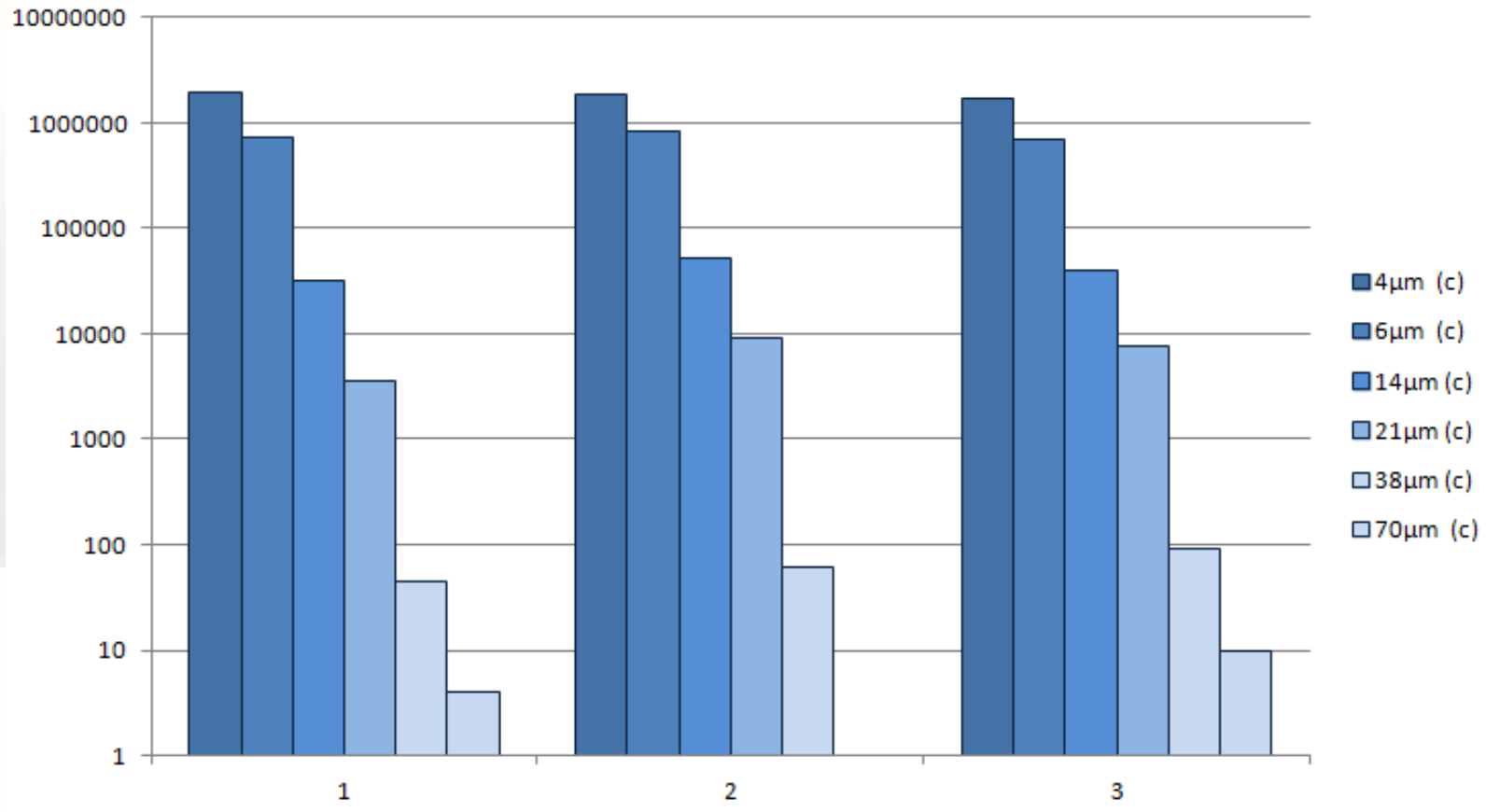
**Table 10** – *Particle Testing results for heavily contaminated gearbox samples at 1:30 dilution ratio.*

**Figure 17** – A ferrogram (top) 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). A particle debris patch (bottom left) and sample visual clarity image (bottom 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)



## Processing Samples

*Heavily Contaminated (Gross Particulate Concentration)*



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





**Processing Samples**  
*Repeatability*

## Repeatability

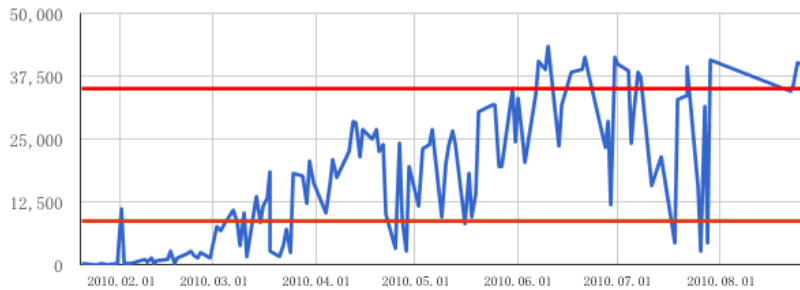
### HIAC 8012

Internal Standard 2010 n:149

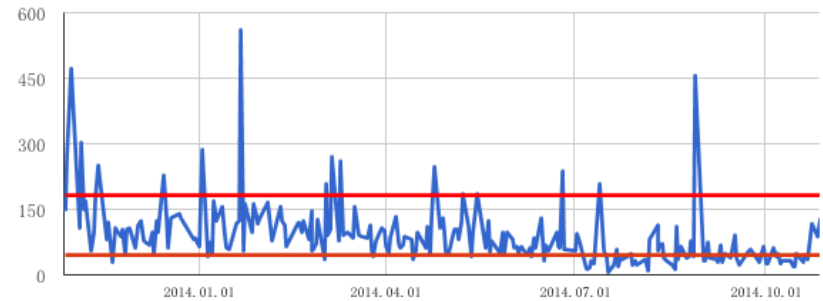
### CINRG CS-APC-2

Internal Standard 2014 n:247

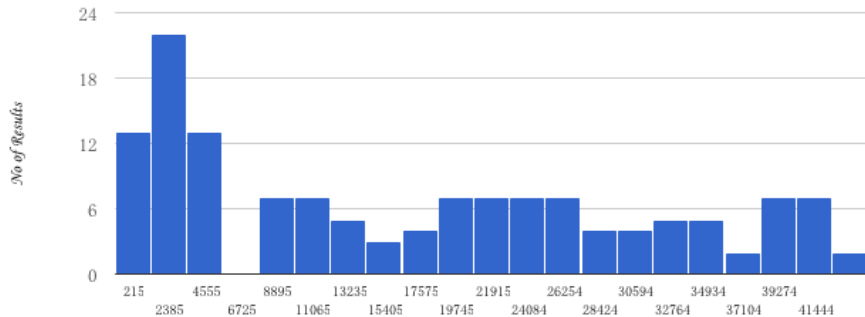
Trend for PC<sub>s</sub>M



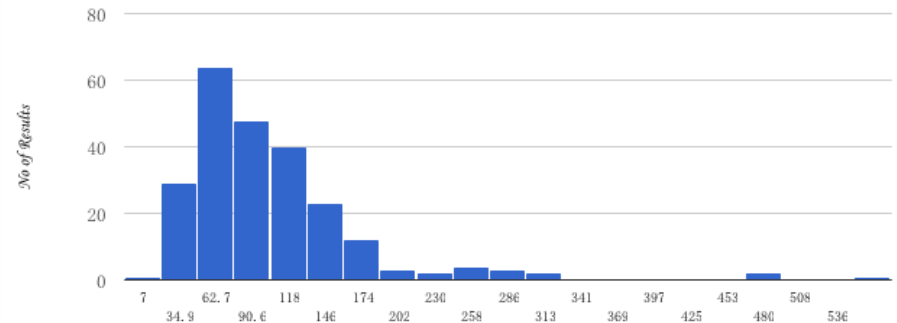
Trend for PC<sub>s</sub>M



Histogram of PC<sub>s</sub>M



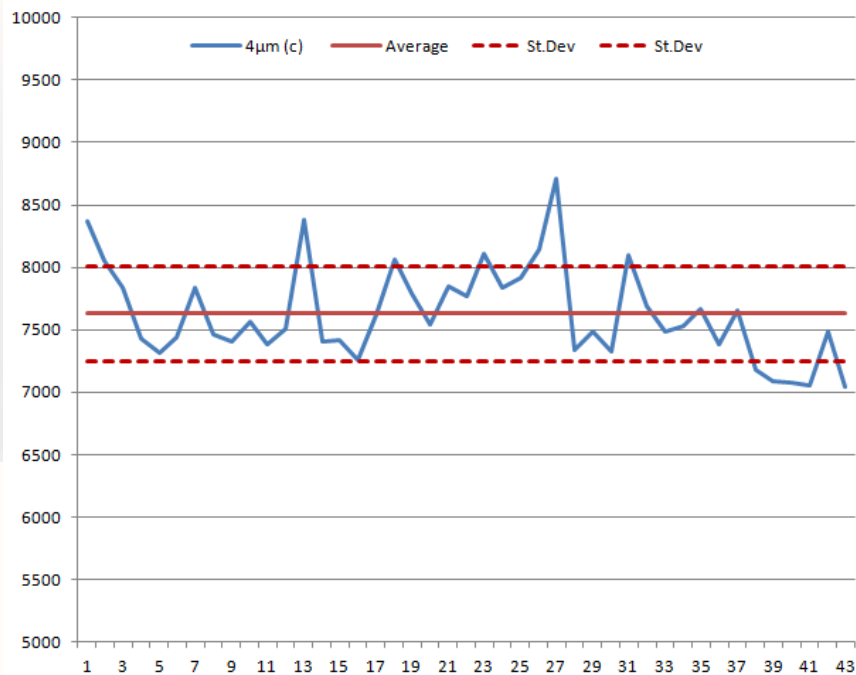
Histogram of PC<sub>s</sub>M



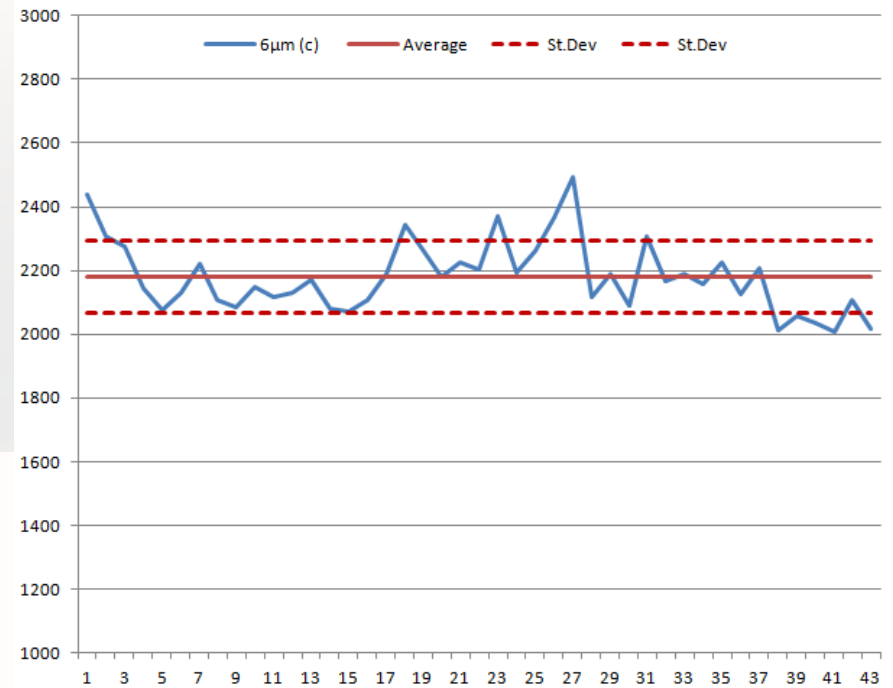
## Repeatability

**CINRG-CS-APC2** Process Control Standard (*over a 2-month period*)

### 4µm (c)



### 6µm (c)





## Future Study

Reproducibility of Particle Count Results using the  
CINRG CS-APC-2 Instrument

- 6 Instruments in 4 countries

**Thank-you!**