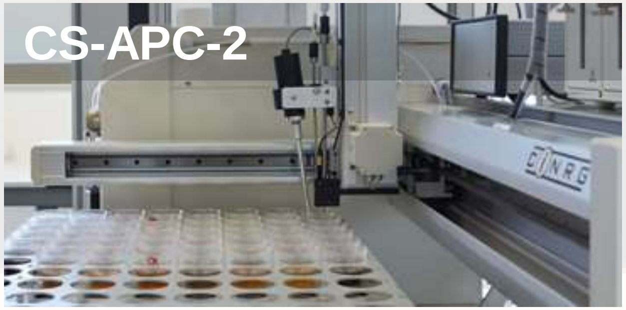
CINRG Systems Inc.

Is NIST SRM2806b Responsible for the Sudden Increase in Particle Counts you have Seen on your Oil Analysis Reports?

A. Geach, B. Quesnel

Don't just automate, innovate.

CINRG Systems Inc. offers a range of flexible laboratory solutions. Our latest product offerings are a fully automated auto-diluting particle counter and a robotic Houillon viscometer automation system that was developed in partnership with WearCheck International.



The Authors

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Alistair has been in the oil analysis industry for 20 years, formerly with SetPoint Technologies in Africa. Alistair's unique skills in chemistry, physics and engineering have helped him in his career of laboratory automation and instrument development.

STLE OMA I Certified
ILMA MLA I, MLT I, LLA 1 Certified

Bill Quesnel, President

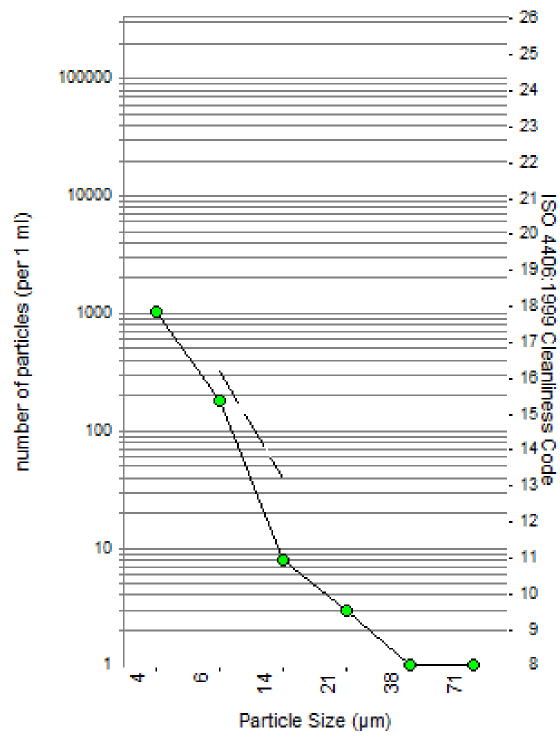
Bill Quesnel has been in the oil analysis industry for 24 years. Bill is president and former laboratory manager for WearCheck in Toronto, Ontario and graduated from the University of Waterloo in pre-med with minors in Biology, Chemistry and Computer Science.

STLE OMA I, OMA II Certified
ILMA MLA I/II/III, MLT I/II, LLA 1 Certified

What is the issue?

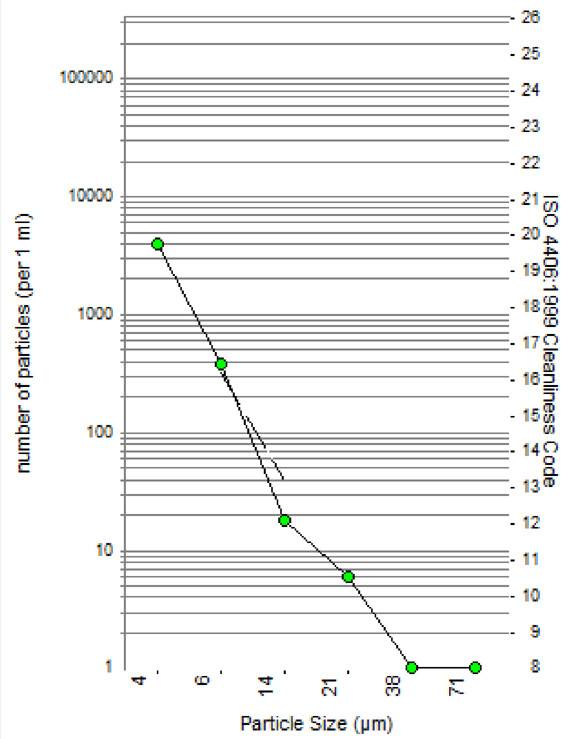
- Same Oil Sample
- Results are different because they are based on two different ISO Particle Count calibrations
- SRM2806a vs. SRM2806b

ISO 4406:1999 17/15/10



$0.00

ISO 4406:1999 19/16/11



2 x tech x 3 hrs
\+ Oil Filter

$700.00 x 133

= $93,100.00

Ref: G. Tapp – GE Wind

Particle Count (PC) ISO 4406:1999 (ISO 11171)

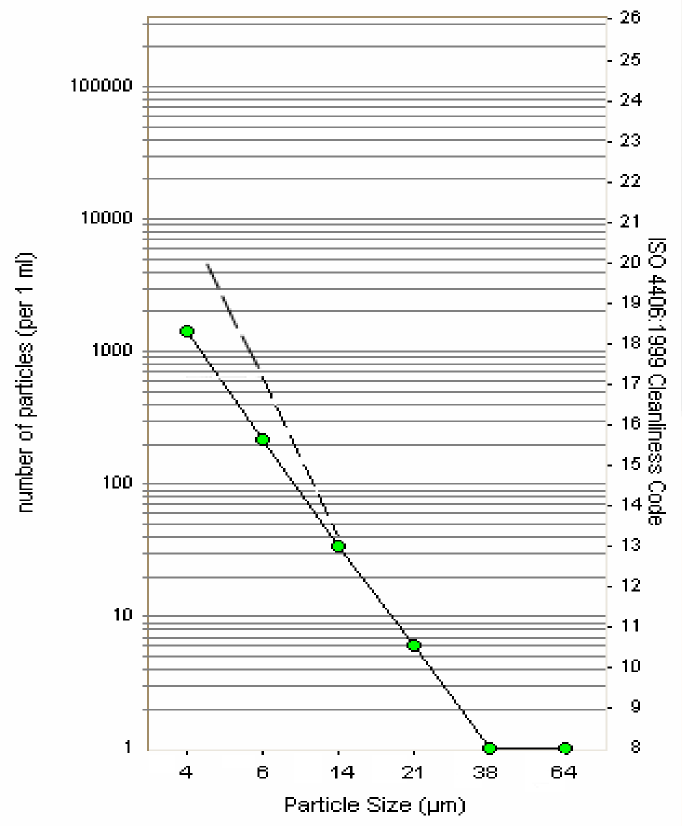
Particle count measurements are taken on typically 10 mL of oil with the results averaged to 1 mL. Prior to counting, the particles in the oil sample must be homogenized which can be accomplished in a combination of ways including shaking, sonication, de-gassing, etc. Most important is that sample preparation 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

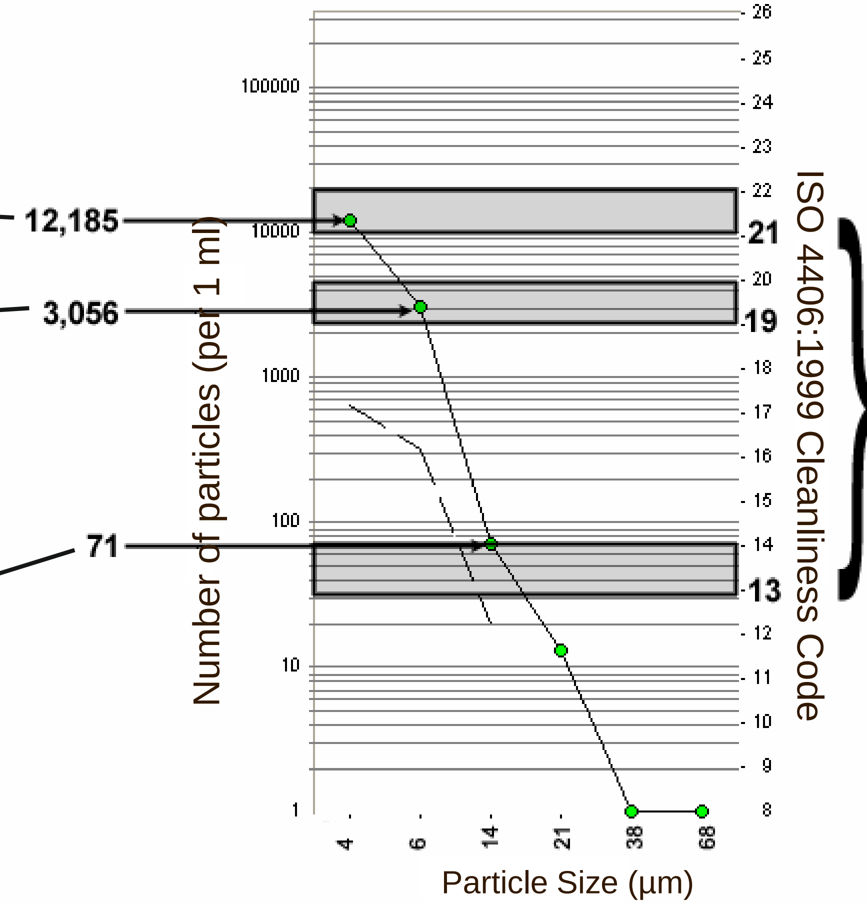


How is Oil Cleanliness Measured?

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.

ISO 4406 Cleanliness Code

ISO Code	More than	Up to
25	160,000	320,000
24	80,000	160,000
23	40,000	80,000
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	4	10
9	2.5	4
8	1.25	2.5



21/19/13

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

19/13

ISO 4406:1987 report as Number of particles (Np) >5µm over the Np >15µm

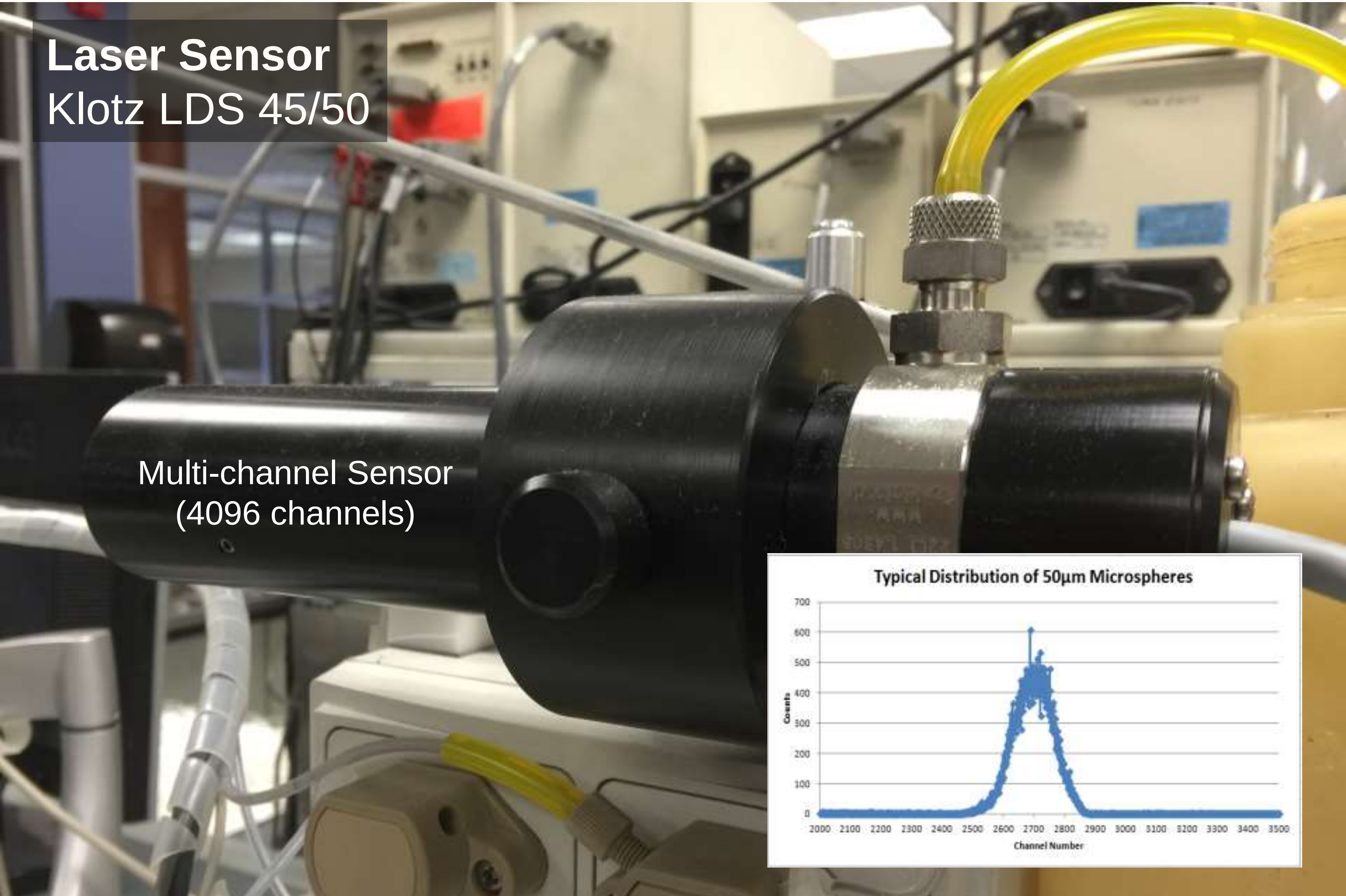
Ref: WearCheck

APC
Calibration Fluid History

Material	ISO Standard	Certificate Date	Reason for Revision	Expiration Date
ACFTD	4402:1991	1960 – 1999	AC Fine Test Dust (ACFTD) no longer commercially available	
ISO MTD SRM2806-0	11171:1999	10-Dec-97	ISO Medium Test Dust (MTD) - NIST Traceable Standard - Original Certificate	
ISO MTD SRM2806-1	11171:1999	1-Mar-99	Revised uncertainties and change of >30µm values to information values	
ISO MTD SRM2806-2	11171:1999	9-Aug-00	Revision of expiration date.	
ISO MTD SRM2806-3	11171:1999	16-Nov-04	Decrease in expiration date due to instability.	17-Sep-04
ISO MTD SRM2806a-0	11171:1999	13-Oct-04	Original Certificate	
ISO MTD SRM2806a-1	11171:1999	29-Jan-07	Update of expiration date and editorial changes.	
ISO MTD SRM2806a-2	11171:1999	16-Dec-08	Extension of certification period.	
ISO MTD SRM2806a-3	11171:2010	30-May-13	Extension of certification period; editorial changes.	31-Dec-14
ISO MTD SRM2806b-0	11171:2016?	12-Jun-14	Original Certificate	31-Dec-20

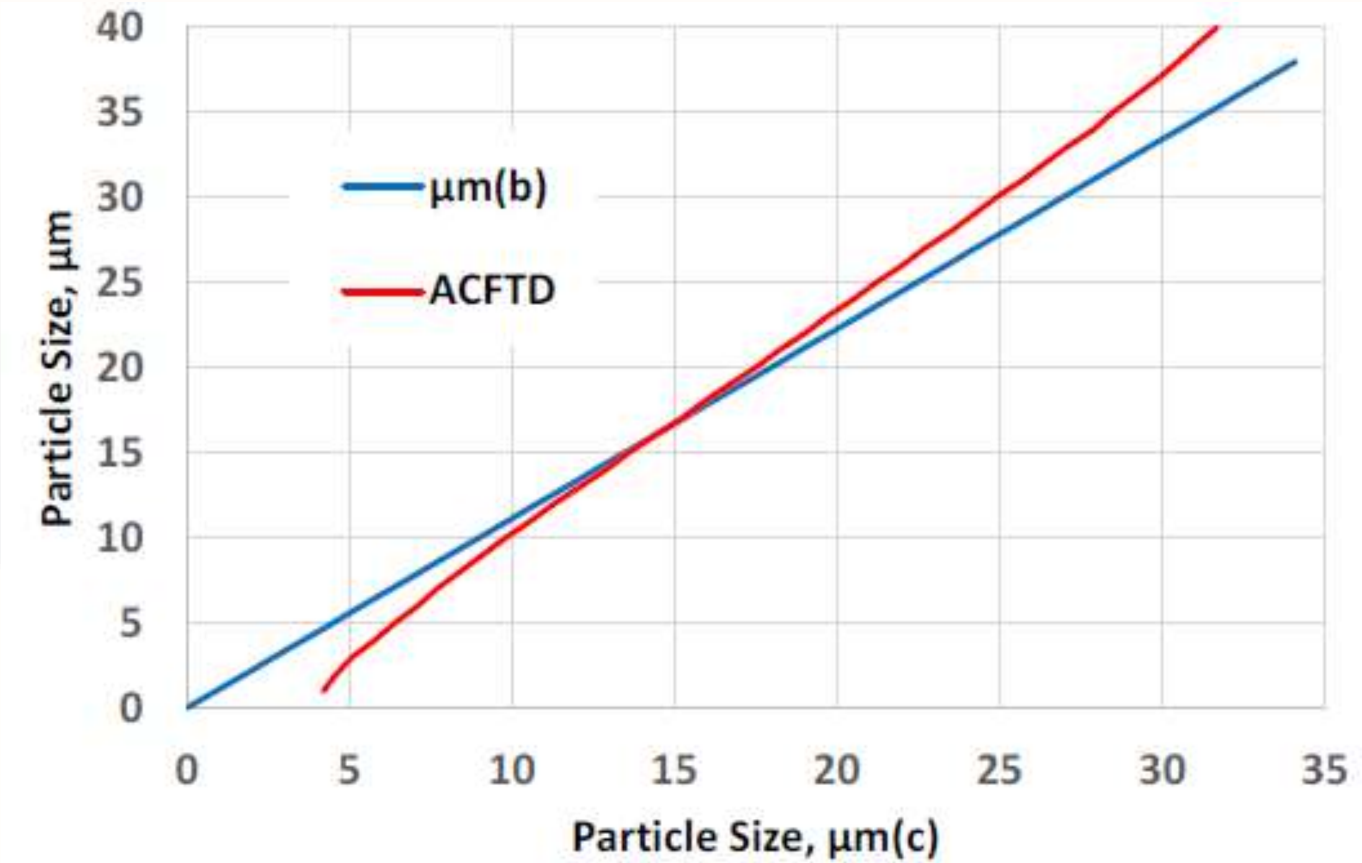
NIST2806b **Particle Count Increase**

NIST2806b **Particle Count Increase**



1998 - Effect of ISO MTD replacing ACFTD

- Counts < 10µm increased
- Counts > 10µm decreased



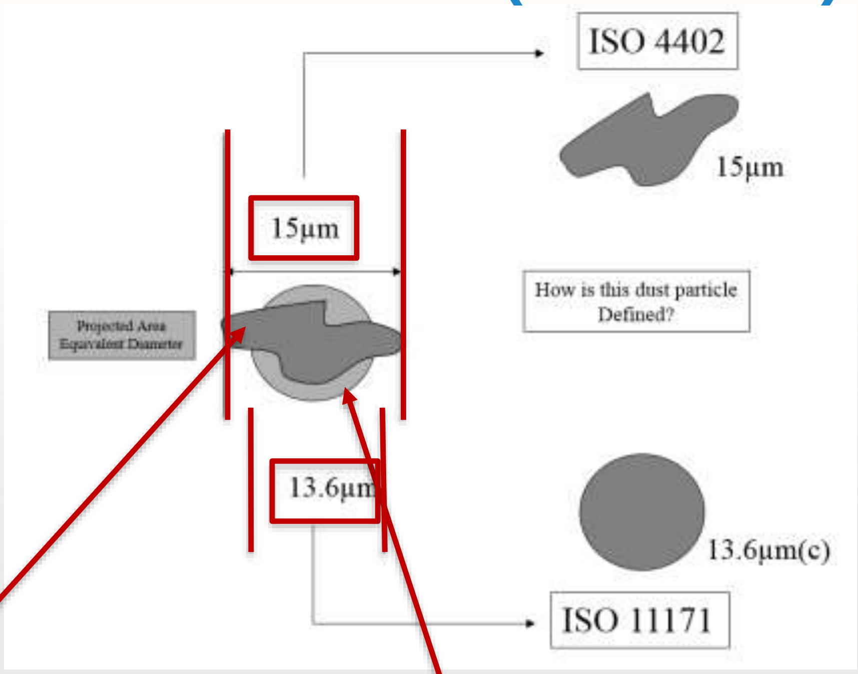
Ref: Barry Verdegan, Cummins Filtration

ISO 4406:1987 (ISO 4402) ⟶ ISO 4406:1999 (ISO 11171)

ACFTD (Air Cleaner Fine Test Dust)

Replaced By

ISO MTD (Medium Test Dust)

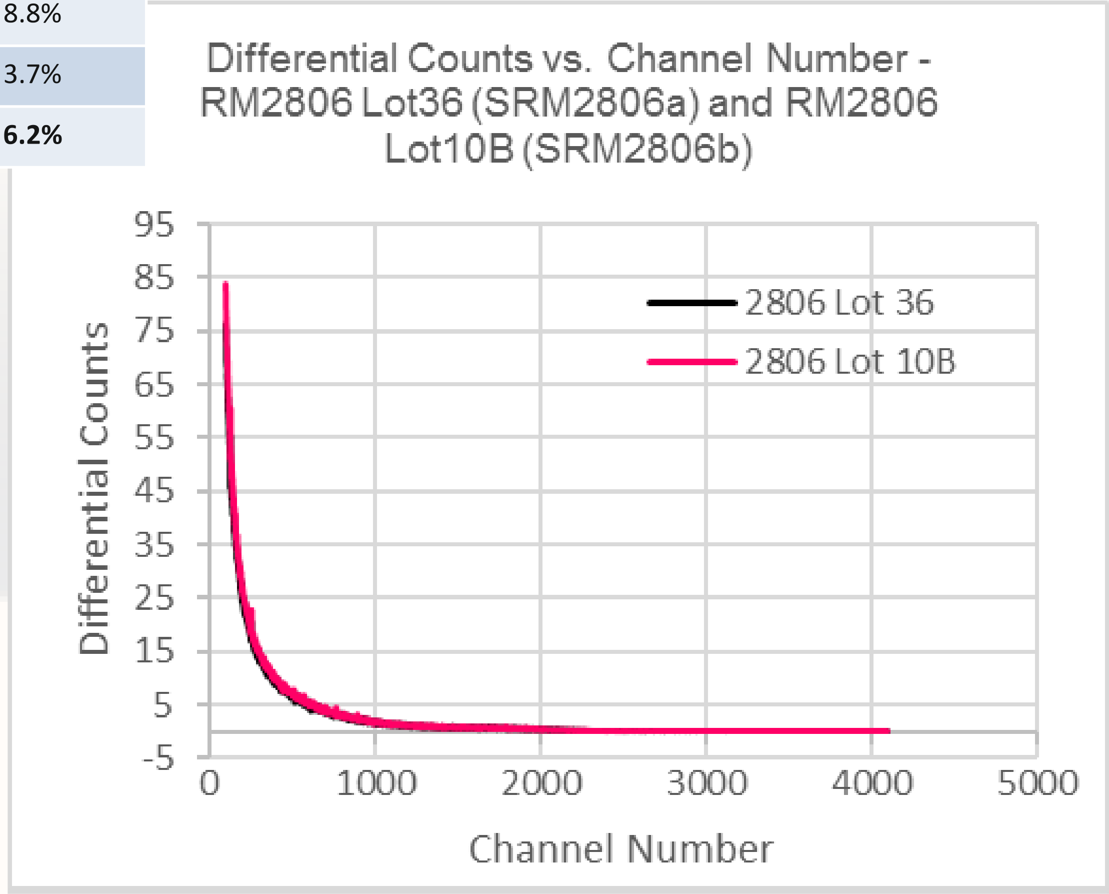


Original Sizes	Replaced By
2µm	4µm(c)
5µm	6µm(c)
15µm	14µm(c)

NIST2806b **Particle Count Increase**

SIZE	LOT 36	LOT 10B	CHANGE
> 4µm	7295	7734	6.0%
> 6µm	2906	3161	8.8%
> 14µm	210	217	3.7%
		Average Change =	**6.2%**

- Raw counts from SRM2806a & b "remarkably" similar
- Derived dust concentration 25% higher than SRM2806 (2.8 mg/L -> 3.5 mg/L)
- Only 6% higher than RM2806a Lot 36 (3.3 mg/L -> 3.5 mg/L)



Determine the concentration of 2806 Lot 10B

Size µm(c)	Mean Particle Concentration (>Particles/mL) 2806 Lot 36	2806 Lot 10B
>4	7300.5	10665.2
>5	4385.6	6615.8
>6	2907.9	4432.9
>7	1939.9	3065.7
>8	1273.8	2146.0
>9	851.2	1523.5
>10	599.8	1096.1
>11	445.7	796.1
>12	361.6	594.0
>13	304.3	461.5
>14	209.8	362.1
Conc.	3.3mg/L	?

Calibrate APC with 2806 Lot 36
Then run 2806 Lot 10B

SIZE	LOT 36	LOT 10B	CHANGE
> 4µm	7294.8	7734.1	6.0%
> 6µm	2905.9	3160.7	8.8%
> 14µm	209.6	217.4	3.7%
	Average Change =		6.2%

Conc 2806 Lot 10B = 3.3 mg/L * 1.062 = 3.50 mg/L

Calibrate APC with 2806 Lot 10B
Then run 2806 Lot 36

SIZE	LOT 10B	LOT 36	CHANGE
> 4µm	10713.2	10050.9	6.6%
> 6µm	4436.4	4184.4	6.0%
> 14µm	362.0	348.2	4.0%
	Average Change =		5.5%

Conc 2806 Lot 10B = 3.3 mg/L * 1.055 = 3.48 mg/L

Estimate of Certification "Error"

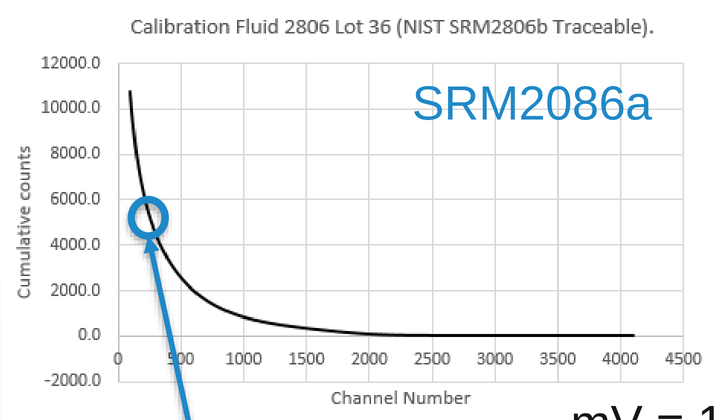
Relative contribution of increased test dust concentration and certification "error" to the increase in counts.

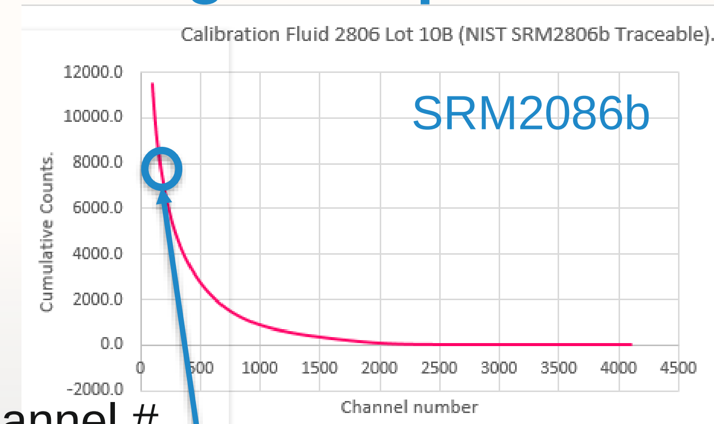
Particle Size	SRM2806a (3.3mg/l) Certified Counts	SRM2806b (3.5mg/l) Certified Counts	Overall Count Increase	Expected Counts 3.3mg/l x 1.062	Unexpected Increase	Change from "Certification Error"
>4µm	7300.5	10864	49%	7753.1	3110.9	**40%**
>6µm	2907.9	4210	45%	3088.2	1121.8	**36%**
>14µm	209.8	389.3	86%	222.8	166.5	**75%**

>4µm = 7300 + 6.2% = 7753(from 10864) = 3110 / 7753 = 40%

NOTE: The "error" is with SRM2806a not with SRM2806b

Determination of Channel / mV settings for 4µm



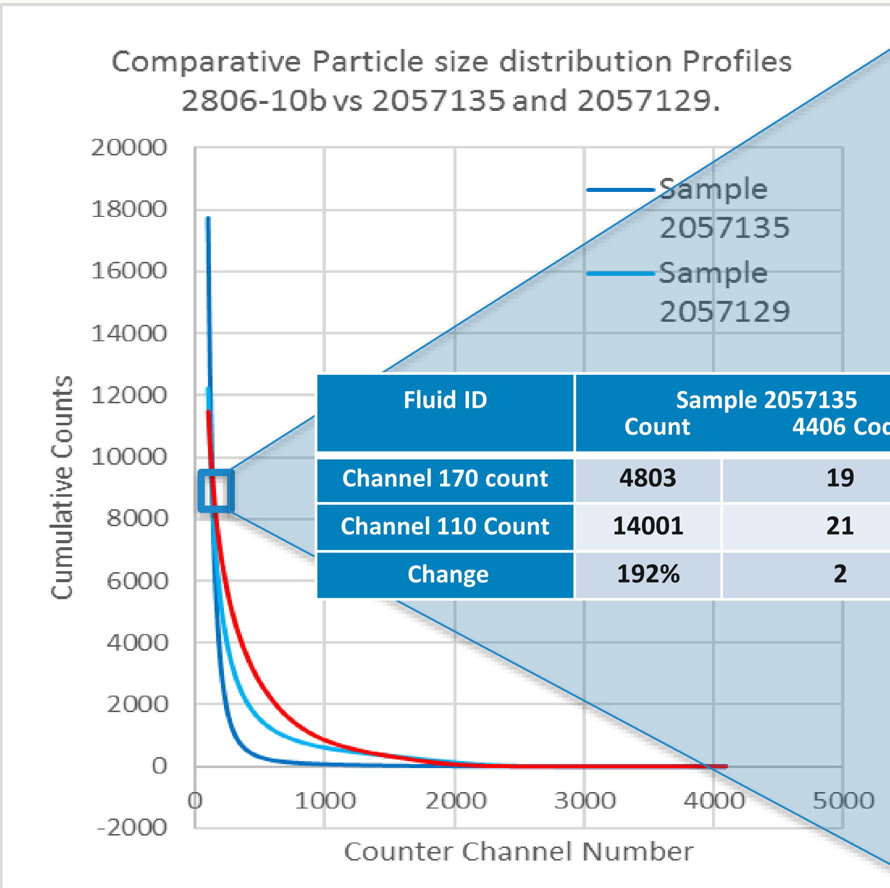


$$mV = \frac{10{,}000mV \times Channel\ \#}{4{,}096}$$

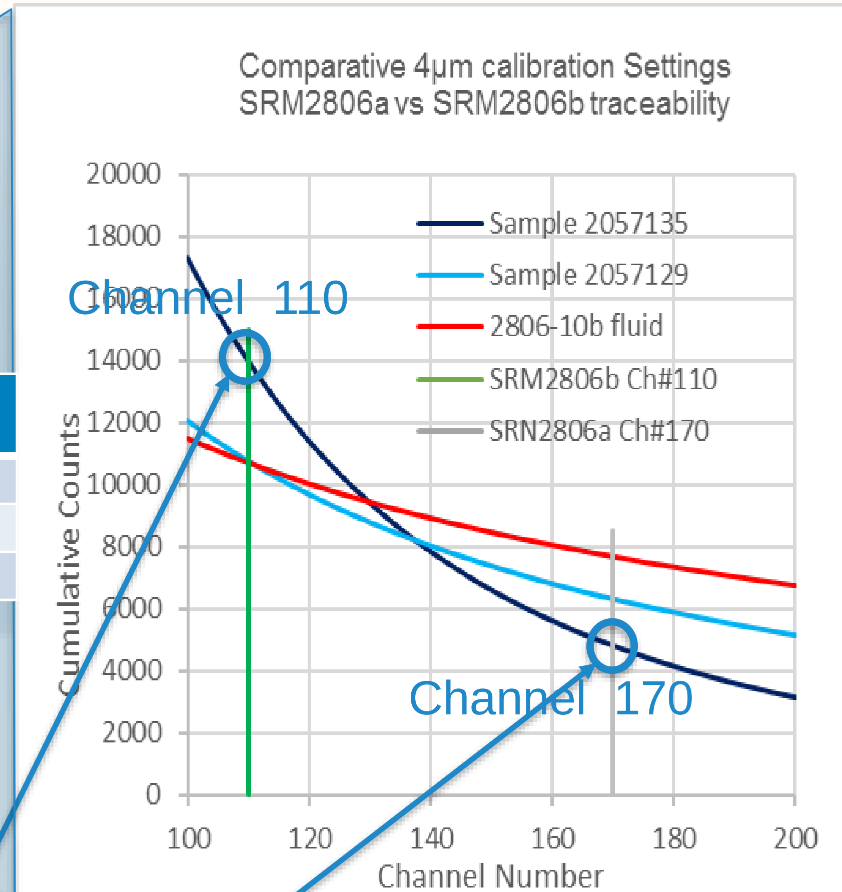
	13716.2	1			
		:			
	7495.7	164			
	7461.6	165			
	7427.0	166			
	7392.5	167			
	7360.2	168			
	7327.3	169			
7300.5	**7294.8**	**170**	**4**	**415.0**	**mV**
	7262.7	171			
	7230.9	172			
	7199.1	173			
	7167.7	174			
	7136.0	175			
		:			
	0.0	4096			

	14786.3	1			
		:			
	11160.9	104			
	11085.0	105			
	11007.3	106			
	10931.6	107			
	10857.4	108			
	10785.2	109			
10665.2	**10713.2**	**110**	**4**	**268.6**	**mV**
	10641.6	111			
	10571.9	112			
	10503.9	113			
	10434.3	114			
	10368.4	115			
		:			
	0.0	4096			

How will smaller particles be affected (4, 6, 14, 21µm)?



Fluid ID	Sample 2057135 Count	4406 Code
Channel 170 count	4803	19
Channel 110 Count	14001	21
Change	192%	2



4µm = 14,001 vs. 4,803

Comparative Counts for

Sensor Calibrations traceable to SRM2806a and SRM2806b

Sample Number	2806 Cal	Count >4µm	Count >6µm	Count >14µm	Cleanliness Code	Component Sampled
2057337	a	447	156	15	16/14/11	Hydraulic System
	b	702	226	25	17/15/12	
2057341	a	321	121	12	16/14/11	Hydraulic System
	b	496	170	20	16/15/12	
2057382	a	384	114	12	16/14/11	Wind Turbine Gearbox
	b	698	171	18	17/15/11	
2057613	a	618	179	19	16/15/11	Gas Turbine
	b	1085	277	32	17/15/12	
2057380	a	355	86	10	16/14/10	Wind Turbine Gearbox
	b	728	137	15	17/14/11	
2057353	a	312	138	12	16/14/11	Excavator Hydraulics
	b	1250	230	20	17/15/11	
2057333	a	648	162	14	17/15/11	Hydraulic System
	b	1186	262	24	17/15/12	
2057384	a	686	187	16	17/15/11	Wind Turbine Gearbox
	b	1252	294	27	17/15/12	
2057437	a	1198	166	8	17/15/10	Hydraulic System
	b	3606	325	13	19/16/11	
2057390	a	1256	218	9	17/15/10	Wind Turbine Gearbox
	b	2475	419	15	18/16/11	
2057335	a	1548	456	53	18/16/13	Hydraulic System
	b	2752	687	79	19/17/13	
2057135	a	4803	375	14	19/16/11	Steam Turbine Bearing
	b	14001	874	26	21/17/12	
2057129	a	6336	1726	261	20/18/15	Hydraulic System
	b	10783	2734	357	21/19/16	
2057440	a	14104	1255	46	21/17/13	Excavator Hydraulics
	b	32024	3018	74	22/19/13	

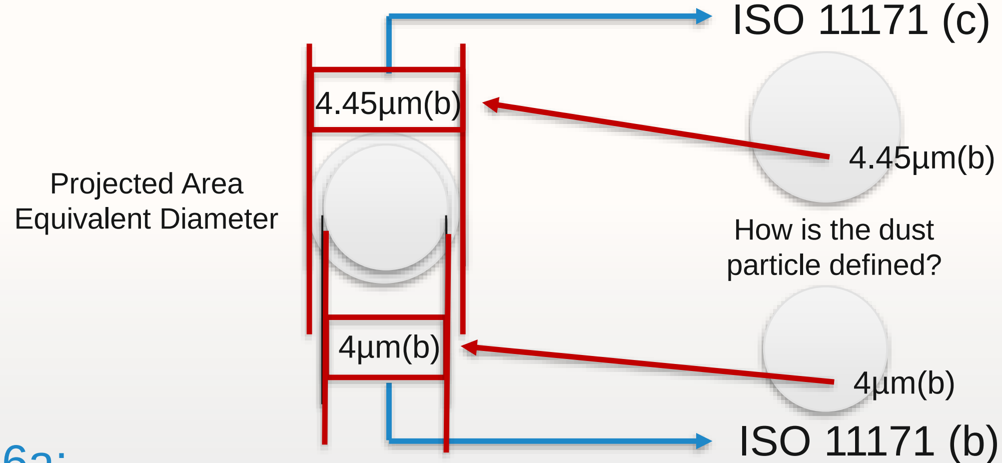
Increase
0-25%
>25%
>50%
>100%

Increase
0 ISO
1 ISO 1
1 ISO 2
2 ISO

Ref: WearCheck Canada Inc.

What is the solution?

$$d_c = 0.898 d_b$$



- Draft revision to ISO1171
- *Ability to report to SRM2806a: 4µm(c), 6µm(c), 14µm(c) using 4.45µm(b), 6.68µm(b), 15.6µm(b)
- Ability to report to SRM2806b: 4µm(b), 6µm(b), 14µm(b)

*Size Equivalence	
Micron(c)	Micron(b)
4µm(c)	4.45 µm(b)
6µm(c)	6.68 µm(b)
14µm(c)	15.6 µm(b)

NOTE: Relationship determined using round robin results from 15 laboratories using secondary samples from 7 different sources in 4 countries.
- FDIS ballot of 11171 will be out soon.
- ISO TC131/SC6 will meet to vote in next few months.

Comparative Counts for

Sensor Calibrations traceable to SRM2806a and using FDIS11171(c)

Sample Number	2806 Cal	Count >4µm	Count >6µm	Count >14µm	Cleanliness Code	Component Sampled
2057337	a	447	156	15	16/14/11	Hydraulic System
	c	503	175	17	16/15/11	
2057341	a	321	121	12	16/14/11	Hydraulic System
	c	361	135	14	16/14/11	
2057382	a	384	114	12	16/14/11	Wind Turbine Gearbox
	c	445	128	14	16/14/11	
2057613	a	618	179	19	16/15/11	Gas Turbine
	c	719	205	22	17/15/12	
2057380	a	355	86	10	16/14/10	Wind Turbine Gearbox
	c	428	98	12	16/14/11	
2057353	a	312	138	12	16/14/11	Excavator Hydraulics
	c	737	163	14	17/15/11	
2057333	a	648	162	14	17/15/11	Hydraulic System
	c	763	187	17	17/15/11	
2057384	a	686	187	16	17/15/11	Wind Turbine Gearbox
	c	805	214	19	17/15/11	
2057437	a	1198	166	8	17/15/10	Hydraulic System
	c	1567	200	9	18/15/10	
2057390	a	1256	218	9	17/15/10	Wind Turbine Gearbox
	c	1518	266	10	18/15/10	
2057335	a	1548	456	53	18/16/13	Hydraulic System
	c	1802	516	60	18/16/13	
2057135	a	4803	375	14	19/16/11	Steam Turbine Bearing
	c	6485	481	17	20/16/11	
2057129	a	6336	1726	261	20/18/15	Hydraulic System
	c	7338	1981	290	20/18/15	
2057440	a	14104	1255	46	21/17/13	Excavator Hydraulics
	c	17997	1629	53	21/18/13	

Increase
0-25%
>25%
>50%
>100%

Increase
0 ISO
1 ISO 1
1 ISO 2
2 ISO

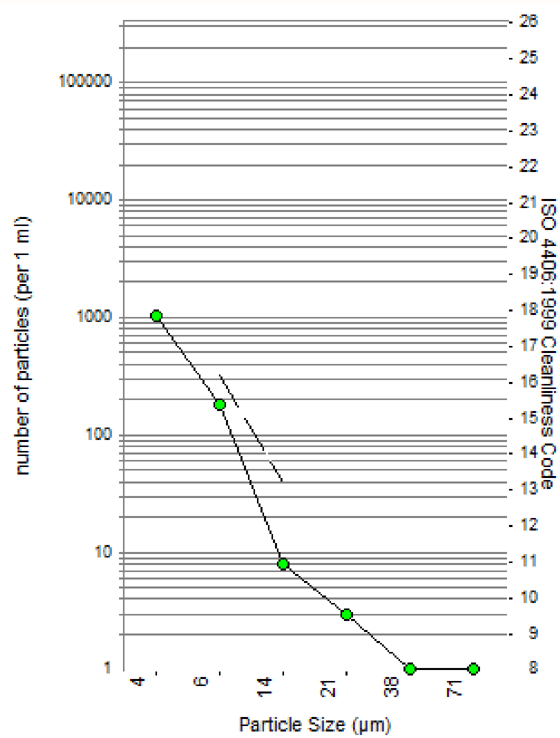
Ref: WearCheck Canada Inc.

NIST2806b **Particle Count Increase**

What is the issue?

- Same Oil Sample
- Results are only slightly different because of revisions to ISO 11171
- SRM2806b using size modification to report as 4µm(c),6µm(c),14µm(c)

ISO 4406:1999 17/15/10



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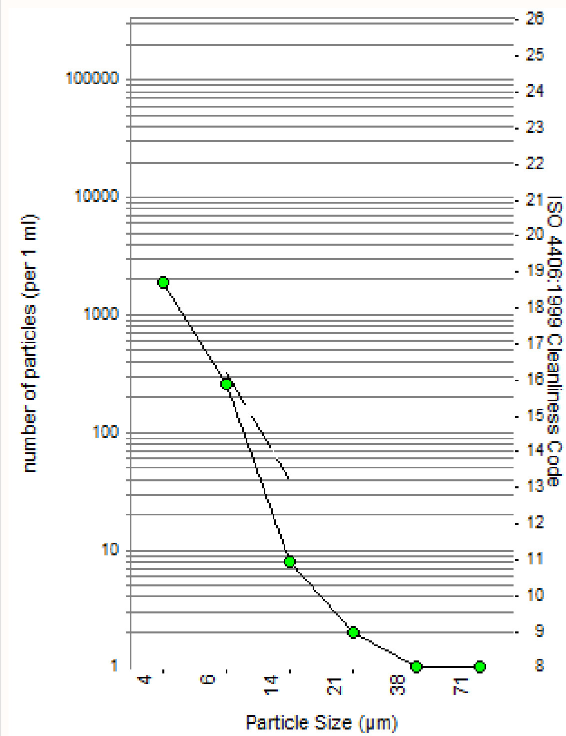
ISO 4406:1999 18/15/10



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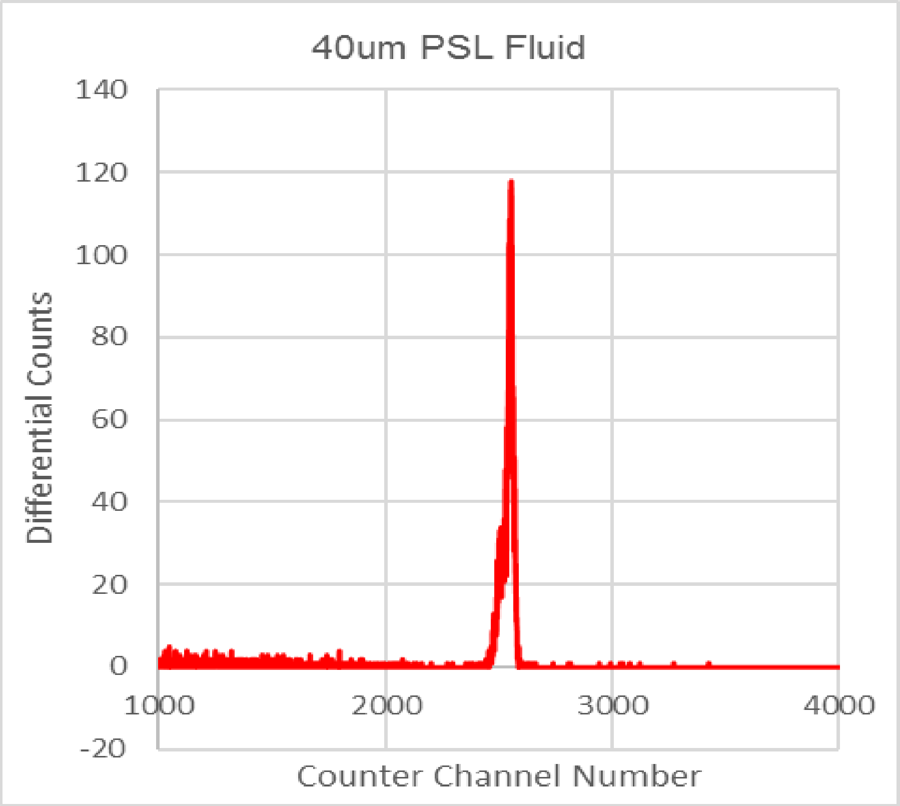
Will larger particle sizes be affected (38 & 71µm)?

- Larger particles calibrated using PSL fluid.
- Unaffected by MTD based calibration fluid.
- No change for large particles.



Summary

- 1998 Discontinuation of ACFTD leads to change in calibration method ISO 4402 -> ISO 11171
- Due to change in accuracy of certification standards particle sizes are redefined: 2/5/15 -> 4/6/14
- 2016 New ISO MTD SRM2806b, no availability of SRM2806a may lead to a new ISO11171:2016 revision
- Due to further improvement in accuracy we may see particle sizes redefined again (*but only for calibration purposes*).
- Ability to report to either standard;
  4µm(c), 6µm(c) 14µm(c)
  or
  4µm(b), 6µm(b) 14µm(b)
- No change to larger particle calibration. Still using PSL fluid.
- If you want to get involved contact your national representative to ISO TC131/SC6. Advise them of your concerns and support the FDIS.

CINRG Systems Inc.

Innovation in Automation

For Commercial Oil Analysis Laboratories