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

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Abstract

In 2015 NIST released the latest batch of SRM2806 calibration fluid for optical particle counters. This fluid is identified as SRM2806b and has certified values that are considerably higher than those of the previous batches of this calibration fluid (SRM2806 and SRM2806a). The increase in count values can apparently be attributed to both an increase in the concentration of ISO MTD in the fluid and to a more accurate determination of the particle size distribution in the fluid during the certification process. When optical particle counters are calibrated with fluids with traceability to SRM2806b they show alarming shifts in threshold settings for the various particle sizes and when samples are subsequently tested, count data and cleanliness codes are significantly higher compared to historical data. This indicates that the more accurate certification accounts for a significant proportion of the count increases that are seen on the certificate for SRM 2806b. Although SRM2806b is more accurately certified than previous batches of calibration fluid its release is generating problems with optical particle count data that are not dissimilar to those that occurred in 1998 when ISO MTD replaced ACFTD as the source of the particulates in calibration fluids. This paper looks at raw count data from two secondary calibration fluids, one that is traceable to SRM2806a and the other to SRM2806b. The data is used to estimate the "certification error" between the two standards and to derive two sets of calibration points that are subsequently used to obtain comparative particle counts measurements on a number of routine samples. The paper also looks at the measures the ISO committee responsible for ISO11171 has proposed through a minor revision of ISO 11171 to manage the problems that result from the differences between these two standards.

Keywords:

ISO MTD, ISO 11171, ACFTD, SRM2806, Calibration

1 INTRODUCTION

In 1999 ISO replaced the ISO4402 calibration standard for optical particle counters with a completely new standard, ISO11171. This change occurred as a result of the introduction of ISO MTD (Medium Test Dust) as a replacement for ACFTD (Air Cleaner Fine Test Dust) in the fluid suspensions used in the calibration of optical particle counters. The new MTD based primary calibration fluid (SRM2806) was a superior calibration fluid as it was more suitably certified for optical particle counters and NIST traceable. The particles in the new material were certified in terms of their circular equivalent diameter rather than their longest chord length (see Figure 1). The use of calibration standards based on SRM2806 however generated different calibration data for optical particle counters than ACFTD based fluids and resulted in significantly higher contamination levels being measured on samples after an optical particle counter was calibrated with the new fluid.

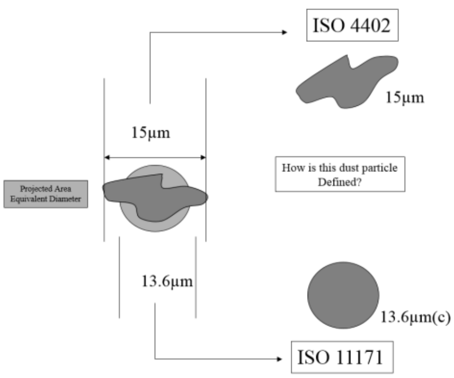

Figure 1: Comparison of how the size of a dust particle is obtained using ISO 11171 compared to ISO 4402.

This was a major concern for the fluid power industry and ISO addressed this issue by redefining the ISO4406 reporting standard. The revised standard officially introduced the three tier cleanliness code and specified the use of counts for particles greater than 4µm(c), 6µm(c) and 14µm(c) respectively to generate the code while retaining the cleanliness code table unchanged. These modifications minimized the changes that would be seen in both count data and cleanliness codes on oil analysis reports but meant that the previously reported sizes of 2µm, 5µm and 15µm would be replaced with 4µm(c), 6µm(c) and 14µm(c).

2 ISO 4406:1999

2.1 Standards SRM2806 and SRM2806a

There was much confusion surrounding the changes that were introduced in 1999 and a degree of resistance from oil labs to adopt the new standards but there is no doubt the changes eventually had a beneficial effect on the quality and reproducibility of count data produced by oil labs. It was just unfortunate that it took so long for the benefits to be fully realized. Since 1999 the industry has enjoyed a little over 13 years of consistency with respect to the optical particle calibration fluids based on the new MTD standard and during this time NIST released for sale two batches of certified primary calibration fluids SRM2806 and SR2806a. (See certification history in table 1 in the Tables section of this paper).

2.2 Standard SRM2806b

This consistency was interrupted in mid-2014 when NIST release the third batch of primary standard identified as SRM2806b. This standard had certified counts that were considerably higher than those of the previous two batches which had identical certification values. The higher counts of SRM2806b were due to firstly a higher concentration of test dust being used in the preparation of the primary fluid and secondly to a more accurate certification procedure. The increased accuracy of the certification was as a result of the technological advances in the measuring equipment used in the certification as well as the analysis of orders of magnitude of more particles and many more bottles of the fluid. The differences between the batches are shown in table 2.

Although the test dust content was increased in SRM2806b the new nominal MTD content of this fluid was not published by NIST. This "missing" information made it difficult to assess the relative contributions the higher concentration of test dust and the more accurate certification had made toward the higher counts for this new batch of fluid.

3 FINDING THE SOURCE OF ERROR

3.1 Comparing the Two Standards

In an attempt to quantify the relative contributions of these two variables to the higher counts, two secondary Conostan PartiStan^{TM} 2806 Secondary standards were acquired and used to calibrate a Klotz Multi-Channel Optical Particle Counting System and the data used to compare the two fluids. One of the standards (2806 Lot 36) was traceable to NIST SRM 2806a and the other (2806 Lot 10B) traceable to SRM 2806b. The certified counts for these two standards are compared for the 4µm(c), 6µm(c) and 14µm(c) counts in the table 3 and also compared individually to the Primary standard that they are traceable to in table 4 and table 5 respectively.

When the two secondary standards were used to separately calibrate the Klotz Particle Counting System two significantly different sets of calibration data were obtained and these differences are summarized for the 4µm(c), 6µm(c) and 14µm(c) settings in table 6.

While the calibration data derived from the certified values for the two fluid was significantly different, the raw count data from the 4096 channels of the counter for the two fluids was remarkably similar. This is clearly illustrated in the graphical representations of this data in the series of figures that follow (see Figures 2, 3 and 4).

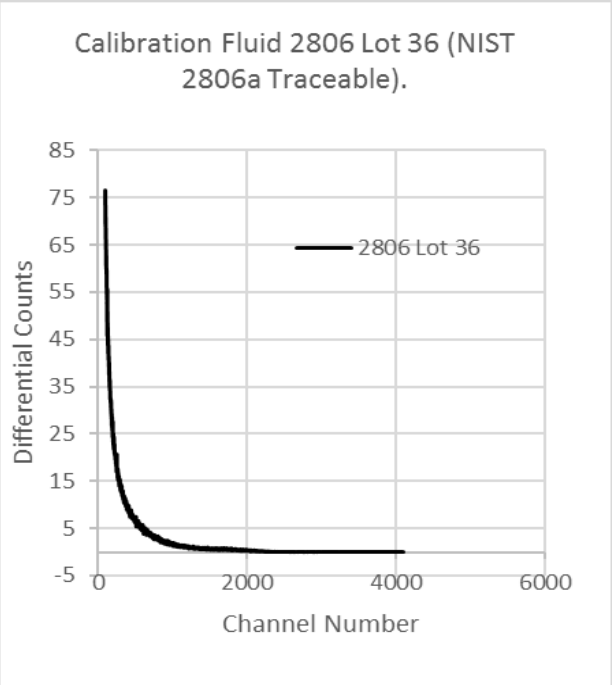

Figure 2: Differential Counts vs. Channel Number for calibration Fluid 2806 Lot 36.

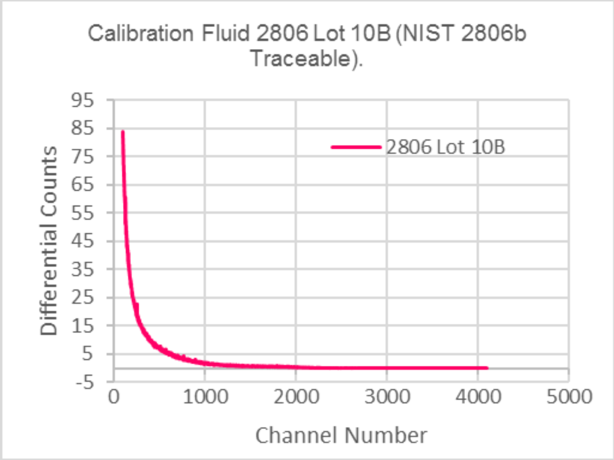

Figure 3: Differential Counts vs Channel Number for Calibration Fluid 2806 Lot 10B

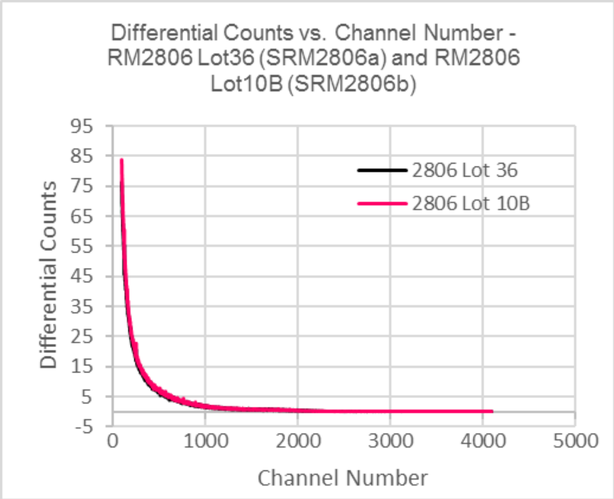

Figure 4: Superimposed data from the graphs in Figures 2 and 3.

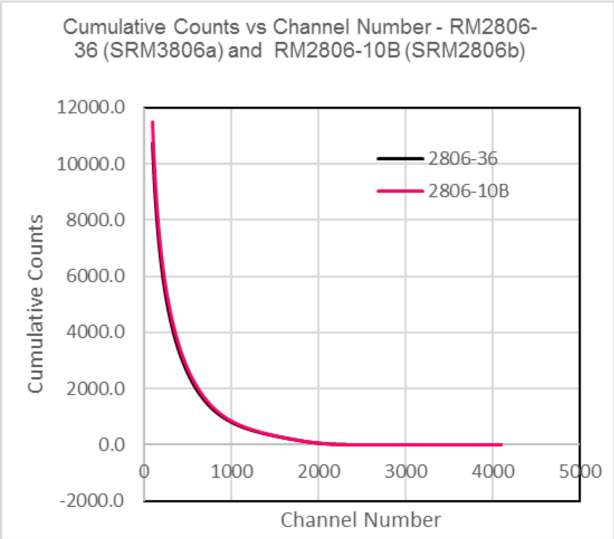

Figure 5: Superimposed Cumulative Count Data for 2806 lot 36 and 2806 Lot 10B.

The raw count data shown in these graphs represents the differential count data and this has to be converted into cumulative count data to determine calibration points as the values quoted on the certificates of calibration fluids are specified as cumulative counts. Figure 5 shows a graph of the superimposed cumulative count data for the two calibration fluids.

3.2 Determining Test Dust Concentration

The data displayed in figure 5 confirms that these two fluids are very similar and therefore likely to contain very similar concentrations of test dust.

As the concentration test dust in RM 2806 Lot 36 was stated on the certificate as a nominal 3.3 mg/L it was possible to use this information to estimate the test dust concentration of SRM2806 Lot 10B. This was done by treating the count data from 2806 Lot 10B as if it was from a routine sample and using the calibration data derived from SRM2806 Lot 36 to determine the counts for particles >4µm(c), >6µm(c) and >14µm(c) in the fluid. The results from this exercise yielded the counts that are summarized in table 7.

The 2806 Lot 10B fluid yielded slightly higher counts in all three of the sizes measured and were found to increase on average by a factor of 1.062. This increase would suggest the test dust concentration in this fluid is a nominal 3.5mg/L as determined by multiplying 3.3mg/L x 1.062.

As the certified values for secondary standard 2806 Lot 10B are very similar to the certified values for the primary standard SRM2806b (Table 5) it is reasonable to assume the nominal concentration of test dust in the primary fluid is also about 3.5mg/L. This equates to a 25% increase relative to the nominal 2.8mg/L of test dust used in the preparation of primary standard SRM2806a.

3.3 Estimates for Certification Error

With this data it was possible to estimate the relative contributions of the increase in test dust concentration and the previous certification “error” had toward the increased counts that are seen on the certificate for SRM 2806b. This data is shown in table 8.

Based on the above data it is reasonable to assume particle counts on samples will be seen to increase by at least 40% or more after an optical particle counter is calibrated with a calibration fluid traceable to NIST SRM2806b. To test this assumption a number of samples provided by WearCheck Canada Inc. were run through the Klotz Optical Particle counting system and particle count results determined by calculating counts based on the two sets of calibration data shown in table 6.

4 IMPACT ON REAL WORLD SAMPLES

4.1 Test Results

The samples came from a variety of components and represented a reasonably wide range in cleanliness levels and in all cases the difference between the two results was considerable. Two of the samples tested showed an increase large enough to move the cleanliness code number for the >4µm count up by 2 units. The test results for the samples is shown in Table 9 and the relative increases for all samples for all three particle sizes are shown as percentages in table 10.

The above results were not unexpected as particle size distributions profiles of “real samples” vary considerably and tend to be significantly different from the test dust particle size distribution in the calibration fluid. Figure 6 shows a graphical representation of the differences between particle size distributions of the 2806-lot 10B calibration fluid (red curve), a hydraulic sample (light blue curve) and a bearing sample (dark blue curve). These curves shown in figure 6 were determined using the data from the Klotz Multi-channel analyzer and represent the relationship between the Cumulative particle counts and Channel number.

Figure 7 shows an exploded view of the steep vertical section of the previous graph (figure 6) and includes two vertical lines that correspond to the channel numbers of the 4µm setting for the calibration data derived from calibration fluids 2806 Lot36 and 2806 Lot 10B respectively.

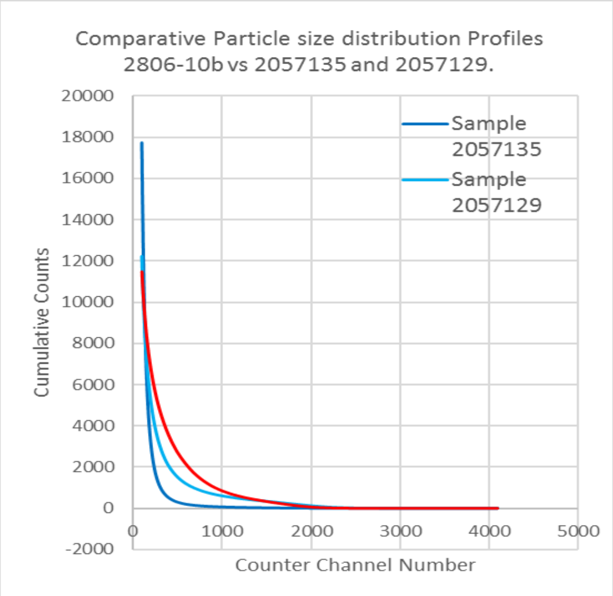

Figure 6: Comparative Particle Size distributions of three fluids.

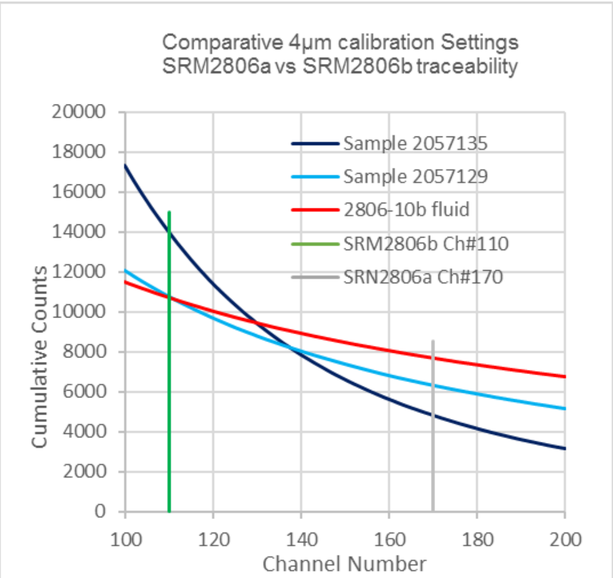

Figure 7: Exploded View of previous Graph (figure 6) - Channel Numbers 100 through 200.

Table 11 shows the 4µm count data and corresponding ISO4406 code that is derived from the intersections of vertical lines drawn from channel numbers 110 and 170 and the respective particle size distribution curves.

4.2 The Impact for Particle Count Results

The considerable increase in particle count values that occur when samples are tested on optical particle counters that have calibrations traceable to SRM2806b is a major concern as the higher counts will have an impact in hydraulics, lube and fuel applications on a global basis. Fluid samples will appear to be dirtier and filters will appear to be less efficient while it will be impossible to compare new ISO code and filter Beta Ratio data with historical data. This will have far reaching consequences as a vast number of technical specifications will need to be revised to accommodate the changing numbers.

These issues are just as significant as those that arose in 1998 when ISO MTD replaced ACFTD as the suspended material in calibration fluids for optical particle counters. At that time, as mentioned earlier, the problem was addressed by the revision of the ISO4406 reporting standard and the impact on industry was minimized.

5 PROPOSED SOLUTION

5.1 ISO 4406 Reporting Options

At present the ISO committee responsible for ISO11171 has, with the aid of round robin results from 15 participating laboratories, completed a thorough investigation of the performance of SRM2806b relative to SRM2806a. The outcome of this investigation has resulted in the committee agreeing to draft a minor revision to ISO11171 that will allow the impact of the use of the new batch of calibration fluid to be minimized. This draft is currently under review at ISO and still has to be issued as an FDIS for ballot purposes. If the final draft passes the ballot it should be published and become available later in the year.

As it stands the draft revision of the ISO11171 standard will allow micron (c) and micron (b) as legitimate reporting options to distinguish between results traceable to the NIST SRM2806a and NIST SRM2806b respectively. Reporting results as >4µm (b), >6 µm (b) and >14µm (b) will indicate NIST SRM2806b traceability, while using >4µm(c), >6 µm(c) and >14µm(c) will indicate NIST SRM2806a traceability.

The revision also and most importantly allows for the calculation of micron (b) sizes that would be equivalent to the old micron(c) sizes in order that the new calibration fluid can be used to calibrate an instrument using settings that will yield results that are consistent with a calibration that would historically be traceable to NIST SRM 2806a.

The work done by the committee in conjunction with participating laboratories was able to establish that there was a linear relationship between the micron(c) and micron(b) sizes and it is given by the formula:

$$\mathbf{d_c = 0.898 d_b} \quad (1)$$

In table 12 this relationship has been used to calculate the size of micron (b) particles that are equivalent to the micron(c) particles of 4 µm, 6 µm and 14 µm in diameter.

5.2 ISO 11171 Draft Revision

If the draft revision of ISO11171 is adopted a laboratory will be able to purchase calibration fluid that is traceable to NIST SRM2806b and then have two options with regard to how their particle counter is calibrated. The lab can either calibrate the instrument based on the 4, 6 and 14 counts specified on the certificate and report subsequent particle count results as micron(b) numbers or determine the threshold settings for micron(b) sizes that are equivalent the 4,6 and 14 micron(c) sizes and use these values to set up the particle counter. E.g. using the data in table 1.9, the lab would use the 4.45, 6.68 and 15.6 micron (b) particle thresholds to set up the 4, 6 and 14 micron channels on their particle counter. With this latter option the instrument calibration would be equivalent to a calibration with a fluid that is traceable to NIST 2806a and the lab would continue to report particle count results as micron(c) values as they have done in the past with no change in count data or ISO cleanliness codes occurring as a result of the use of the new calibration fluid.

5.3 Calibration of Larger Particles

Only 4 µm, 6 µm and 14 µm particle sizes have been discussed to this point as they are the only counts that are used to determine ISO 4406 cleanliness codes but particle counters are normally set up to measure particles >21 µm, >38 µm and >70 µm as well as these counts are needed if sample cleanliness is reported to the AS4059 standard. The 21 µm settings can be derived during calibration in the same way as the smaller sizes as the MTD based calibration fluids cover particles up to 30 µm in size and the mathematical relationship between micron(c) and micron (b) sizes applies over the full certification range. Settings for particles larger than 30 µm are typically determined independently using PSL (Polystyrene Latex Spheres) fluids and therefore unaffected by the MTD based calibration fluid. PSL based fluids are very different to MTD fluids as they contain spherical particles with a narrow particle size distribution and a separate fluid is needed for each calibration point. The particle distribution of a nominal 40 µm PSL fluid is shown in the graph in figure 8 below.

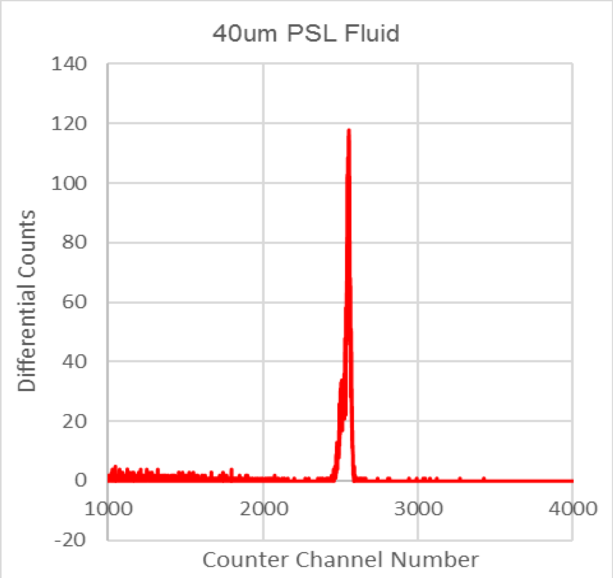

Figure 8: Particle Size distribution in nominal 40 µm PSL fluid.

6 SUMMARY

The approval and publication of the draft revision of ISO 11171 as quickly as possible is vital if the impact of SRM2806b is to be minimized. Secondary calibration fluids traceable to NIST SRM2806b are already in use and those that are traceable to NIST SRM2806a are in extremely short supply and will be exceeding their expiry date in the near future. The stock of SRM2806a primary standard was actually exhausted in 2010 and the expiry date for this fluid was extended a couple of time and then finalized as Dec 31, 2014 in the last revision of the SRM2806a certificate. Assuming availability of the fluid, Dec 31, 2014 would have been the last date for the valid calibration of a particle counter that could have subsequently been used to certify secondary calibration fluids for which traceable to NIST 2806a can be claimed. Allowing 6 months for the last NIST 2806a primary calibration to remain valid, the last batch of secondary calibration standard could have been produced and certified no later than June 30, 2015. If calibration fluid was produced on this date and allowing for a 2-year shelf life, it would expire on June 30, 2017 and no valid claim can be made for calibration traceability to NIST SRM2806a after this date.

What the revision of the ISO 11171 standard means, if it is adopted, is that when the particle count results on an oil analysis report are review by an end user in the near future it will be vitally important to note how the particle sizes are reported. The results reported as >4µm (b), >6µm (b) and >14µm (b) are going to be significantly higher than those reported as >4µm(c), >6µm(c) and >14µm(c) and it will be very likely that the cleanliness code applicable to the micron (b) data will indicate a more severe level of contamination in the sample than has historically been measured with micron(c) reporting.

Although micron (b) data will be a more accurate representation of the actual particulate contamination in fluid samples it is unclear how this data is going to be accepted and used in the future. In the meantime, the revision of ISO11171 if accepted, will buy some time to allow more thought to be given to how this complex problem is to be finally addressed.

7 ACKNOWLEDGMENTS

The author would like to thank the following three people who all provided valuable input for this paper. Barry Verdegan[1] for explaining how the ISO committee for ISO11171 are addressing the issues with respect to SRM 2806b and for sharing his comprehensive training material that covered the results of the extensive work that has been undertaken by the ISO committee and the laboratories that participated in the round robin testing. Robert Fletcher[2] for providing some insight into the certification process of SRM2806b and the reasons for the shift in the certified values and finally Terry Phillips[3] for accepting all the phone calls and sharing his considerable knowledge of the calibration fluids and the certification processes.

[1]Barry Verdegan Ph.D. Research Fellow. Cummins Filtration, [2]Robert A Fletcher – Physicist, National Institute of Standards and Technology, [3]Terry Phillips. – Project Manager Fluid Technologies Inc.

8 TABLES

SRM-Revision#	Certificate Date	Reason for Revision	Expiration Date
SRM2806-0	10-Dec-97	Original Certificate	
SRM2806-1	1-Mar-99	Revised uncertainties and change of >30µm values to information values	
SRM2806-2	9-Aug-00	Revision of expiration date.	
SRM2806-3	16-Nov-04	Decrease in expiration date due to instability.	17-Sep-04
SRM2806a-0	13-Oct-04	Original Certificate	
SRM2806a-1	29-Jan-07	Update of expiration date and editorial changes.	
SRM2806a-2	16-Dec-08	Extension of certification period.	
SRM2806a-3	30-May-13	Extension of certification period; editorial changes.	31-Dec-14
SRM2806b-0	12-Jun-14	Original Certificate	31-Dec-20

Table 1: Particle counting certification fluid history.

Fluid ID	SRM2806	SRM2806a	SRM2806b	
Nominal MTD Content	2.8mg/L	2.8mg/L	Not published	
Particle Size	Particles /ml	Particles /ml	Particles /ml	Percent Increase
4µm	6095	6095	10864	78.2
6µm	2395	2395	4210.2	75.8
14µm	170.4	170.4	389.26	128.4
30µm	8.568	8.568	19.698	129.9

Table 2: Comparison of 4, 6, 14 and 30µm certified particle counts for the three NIST primary standards.

Calibration Fluid ID	2806 lot 36	2806 Lot 10B	Percent
Particle Size	Particles /ml	Particles /ml	Change
4µm	7300.5	10665.2	46.1
6µm	2907.9	4432.9	52.4
14µm	209.8	362.1	72.6
Nominal MTD Content	3.3mg/L	Not Published	n/a

Table 3: Certified count comparison between Conostan PartiStan™ 2806 Secondary standards, Lots 36 and Lot 10B.

Calibration Fluid ID	SRM 2806a	2806 Lot 36	Percent
Particle Size	Particles /ml	Particles /ml	Change
4µm	6095	7300.5	19.8
6µm	2395	2907.9	21.4
14µm	170.4	209.8	23.1
Nominal MTD Content mg/l	2.8	3.3	17.9

Table 4: Certified Count comparison between Primary Standard SRM2806a and Secondary Standard 2806 lot36. (Note the higher concentration of test dust in the secondary standard)

Calibration Fluid ID	SRM 2806b	2806 Lot 10b	Percent
Particle Size	Particles /ml	Particles /ml	Difference
4µm	10864	10665.2	-1.8
6µm	4210.2	4432.9	5.3
14µm	389.26	362.1	-7.0
Nominal MTD Content mg/l	Not Specified	Not Specified	n/a

Table 5: Certified Count comparison between Primary Standard SRM2806b and Secondary Standard 2806 lot10B.

Particles	2806_lot 36 Calibration Data		2806_lot 10B Calibration Data	
Size	Channel No	mV	Channel No	mV
>4µm	170	415	110	268.6
>6µm	455	1110.8	325	793.5
>14µm	1682	4106.4	1458	3559.6

Table 6: Threshold settings comparison between calibrations using 2806 lot 36 and 2806 lot10B respectively.

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%

Table 7: Estimations of 4, 6 and 14µm particles present in SRM2806 Lot 10B based on calibration with SRM2806 Lot 36.

Particle Size	SRM2806a (2.8mg/l) Certified Counts	SRM2806b (3.5mg/l) Certified Counts	Overall Count Increase	Expected Counts 2.8mg/l x 1.25	Unexpected Increase	Change from "Certification Error"
>4µm	6095	10864	**78%**	7619	3245	**43%**
>6µm	2395	4210	**76%**	2994	1216	**41%**
>14µm	170.4	389.3	**128%**	213	176	**83%**

Table 8: Relative contribution of increased test dust concentration and certification “error” to the increase in counts.

Sample Number	2806 Cal	Count >4µm	Count >6µm	Count >14µm	Cleanliness Code	Component Sampled
(1)2806-10B	a	7702	3060	214	20/19/15	Medium Test Dust Suspension
	b	10710	4412	360	21/19/16	
(2) 2057382	a	384	114	12	16/14/11	Wind turbine Gearbox
	b	698	171	18	17/15/11	
(3) 2057613	a	618	179	19	16/15/11	Turbine Bearing.
	b	1085	277	32	17/15/12	
(4) 2057353	a	612	138	12	16/14/11	Hydraulic System – Construction.
	b	1250	230	20	17/15/11	
(5) 2057333	a	648	162	14	17/15/11	Wind turbine Hydraulic
	b	1186	262	24	17/15/12	
(6) 2057437	a	1198	166	8	17/15/10	Hydraulic System – Piston Pump.
	b	3606	325	13	19/16/11	
(7) 2057335	a	1548	456	53	18/16/13	Wind turbine Hydraulic
	b	2752	687	79	19/17/13	
(8) 2057135	a	4803	375	14	19/16/11	Turbine Bearing.
	b	14001	874	26	21/17/12	
(9) 2057129	a	6336	1726	261	20/18/15	Hydraulic System – Rubber Mill
	b	10783	2734	357	21/19/16	
(10) 2057440	a	14104	1255	46	21/17/13	Hydraulic System – Construction.
	b	32024	3018	74	22/19/13	

Table 9: Comparative Counts for sensor calibrations traceable to SRM2806a and SRM2806b respectively for a series of samples run at WearCheck Canada Inc.

Sample Number	% change a to b Particles > 4µm	% change a to b Particles > 6µm	% change a to b Particles >14µm
(1) 2806-10B	39%	44%	68%
(2) 2057382	82%	50%	50%
(3) 2057613	76%	54%	68%
(4) 2057353	104%	67%	67%
(5) 2057333	83%	62%	71%
(6) 2057437	201%	96%	63%
(7) 2057335	79%	51%	49%
(8) 2057135	192%	133%	86%
(9) 2057129	70%	58%	37%
(10) 2057440	127%	140%	61%

Table 10 – Percent change in count data when the sensor calibration was traceable to SRM2806b.

Fluid ID	2806- lot 10b		Sample 2057129		Sample 2057135	
	Count	4406 Code	Count	4406 Code	Count	4406 Code
Channel 170 count	7702	20	6336	20	4803	19
Channel 110 Count	10710	21	10783	21	14001	21
Change	39%	1	70%	1	192%	2

Table 11: 4µm count data and corresponding ISO4406 codes derived from the intersections of vertical lines drawn from channel numbers 110 and 170 and the respective particle size distribution curves.

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

Table 12: Micron(b) size equivalences to micron(c) particles derived using the relationship established in the proposed ISO 11171 revision.