CINRG Systems Inc.

Innovation in Automation

OilDoc 2023 – Particle Counting Workshop

Don't just automate, innovate.

Welcome to CINRG Systems Inc. Our focus is the delivery of global laboratory solutions. Our philosophy is centered on the automation and integration of laboratory testing and information systems. We develop systems that easily integrate multiple operations. For we believe that the partnering of global clients develops solutions of greater value. If you are interested in how a CINRG system can improve your laboratory please contact us.

Our latest product offerings are a fully automated sample dilution system an auto-diluting automated particle counter (available both in a large and small format instrument) and a robotic Houillon viscometer automation system.

Alistair Geach

CTO

Bill Quesnel has been in the oil analysis industry for 30 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. Bill is CLS, OMA II, MLA III, MLT II, LLA I certified.

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Kannan Viswanathan

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Kannan comes with 10 years of combined engineering experience in automobile and Oil & Gas industry. Product development, Production operations and Testing & Field services are his key expertise. Kannan is a Masters graduate from University of Waterloo in Mechanical & Mechatronics Engineering.

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CS-APC-2 Automated Auto-Diluting Particle Counter

CS-APC-22M Mini-Automated Auto-Diluting Particle Counter

Applications

Solvent Performance
Diesel Engines
FRF / Water Glycol
Metalworking Fluids
NAS 1638 & AS4059 Testing

ASTM D7647

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

Method covers

- Standards
- Terminology
- Significance of Method
- Interferences
- Procedure
- Reporting

This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.

Designation: D7647 – 10 (Reapproved 2018)

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]

This standard is issued under the fixed designation D7647; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of particle concentration and particle size distribution in new and in-service oils used for lubrication and hydraulic purposes.

1.2 Particles considered are in the range from 4 µm $_{(c)}$ to 200 µm $_{(c)}$ with the upper limit being dependent on the specific automatic particle counter being used.

NOTE 1—For the purpose of this test method, water droplets not masked by the diluent procedure are detected as particles, and agglomerated particles are detected and reported as a single larger particle.

NOTE 2—The subscript$_{(c)}$ is used to denote that the apparatus has been calibrated in accordance with ISO 11171. This subscript$_{(c)}$ strictly only applies to particles up to 50 µm.

1.8 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.9 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

Investigation into the suitability of the dilution solvents allowed for ASTM D7647.

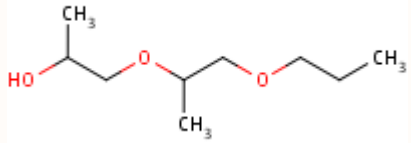
Solvents Investigated

1. Toluene/IPA 75/25%
2. Ethylene Glycol Butyl Ether (Chemically similiar to Dipropylene glycol n propyl ether).
3. Varsol (Stoddard Solvent).
4. Kerosine

Lamp oil is a liquid petroleum product that is designed to burn cleanly in brass and glass oil lamps, torches, and lanterns. In the same family as kerosene. It has been further processed and refined so that it doesn't produce as much harmful smoke, soot, and other pollutants.

Stoddard solvent is a widely used synthetic, organic solvent that comes from the refining of crude oil. It is a petroleum mixture made from distilled alkanes, cycloalkanes (naphthenes), and aromatic compounds. It also goes by other names such as Varsol 1, TexSolve S and others.

Dipropylene glycol monpropyl ether (DPnP) is a colorless liquid with an ether-like odor that evaporates slowly. It is used as a solvent and as a coalescent for water-borne latex coatings.

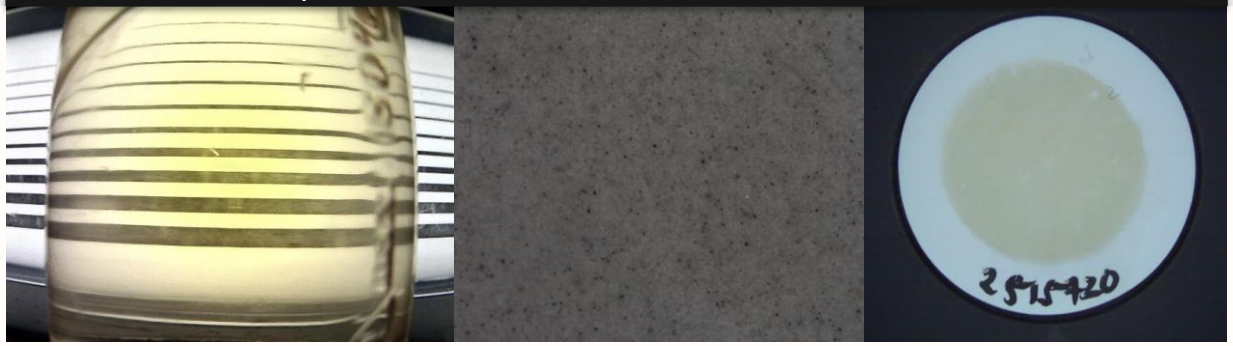


Results Disparity even with low levels of insoluble

Sample No 02515720
Component Thrust Bearing
Fluid Petro Canada TurboFlo R&O 46
MPC 15

02515720	>4µm(c)	>6µm(c)	>14µm(c)	>21µm(c)	>38µm(c)	>70µm(c)	ISO CODE
Undiluted	1657.1	191.5	15.3	6.6	1.7	0.6	18/15/11
Diluted 1:1 Toluene/IPA	1477.3	211.6	17.6	5.3	0.3	0	18/15/11
Diluted 1:1 Varsol	2115.6	216.1	15.8	6.6	0.1	0.1	18/15/11
Diluted 1:1 EGBE	1600.2	227.8	22.3	8.4	1	0.3	18/15/12
Diluted 1:1 Kerosine	1986.6	200.3	14.5	4.8	0.6	0	18/15/11

Visual Clarity | PrtFilter 18/15/11 | MPC 15



Sample No 02521133
Component Turbine
Fluid Castrol Perfecto XPG 32
MPC 23

02515720	>4µm(c)	>6µm(c)	>14µm(c)	>21µm(c)	>38µm(c)	>70µm(c)	ISO CODE
Undiluted	7312.8	2250.5	150.4	40.1	3	0.1	**20/18/14**
Diluted 1:1 Toluene/IPA	290.2	88	6.1	1.7	0.2	0	15/14/10
Diluted 1:1 Varsol	7009.2	1909.1	86.3	17.9	0.6	0	**20/18/14**
Diluted 1:1 EGBE	394.1	99.6	6.1	2.1	0.4	0	16/14/10
Diluted 1:1 Kerosine.	7495.4	2063.6	85.3	16.8	0.5	0	**20/18/14**

Visual Clarity | PrtFilter 15/14/10 | MPC 23

Particle Debris Patches with solvent treatment

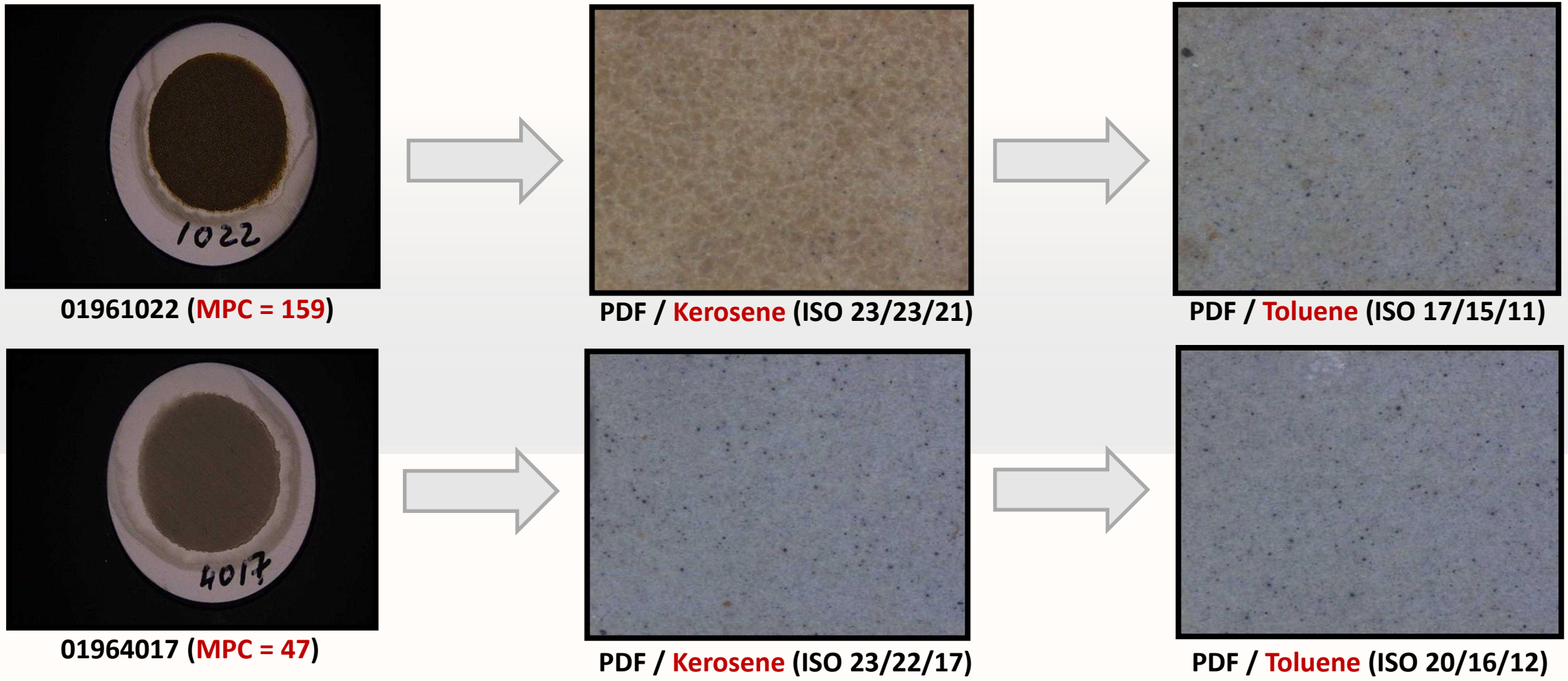


Figure 11: Particle Debris patches for two of the gearbox samples tested comparing solvents.

Particle Count
Diluted vs. Undiluted

Solvent Performance

Water Contaminated Samples

Processing Samples

Water Contamination

Table 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 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

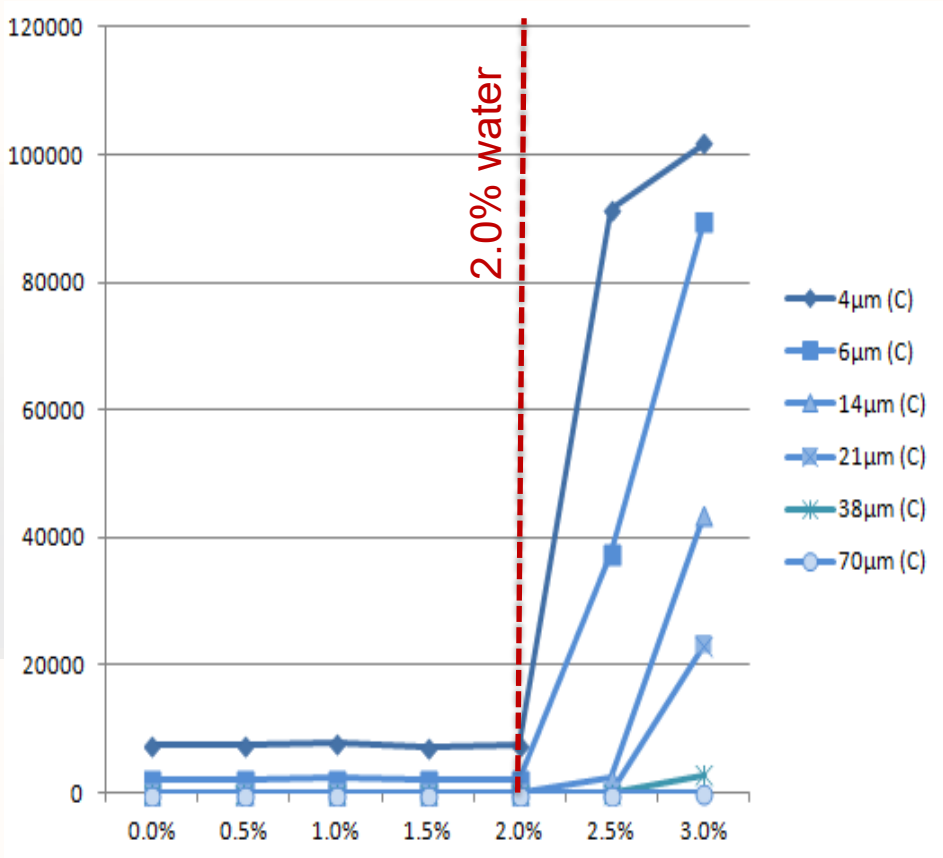


Figure 4 – Individual micron channel results from trial of 0.5% to 3.0% water in MIL-H-5606 oil using 75% Toluene / 25% Iso-propanol as a solvent.

Processing Samples

Water Contamination

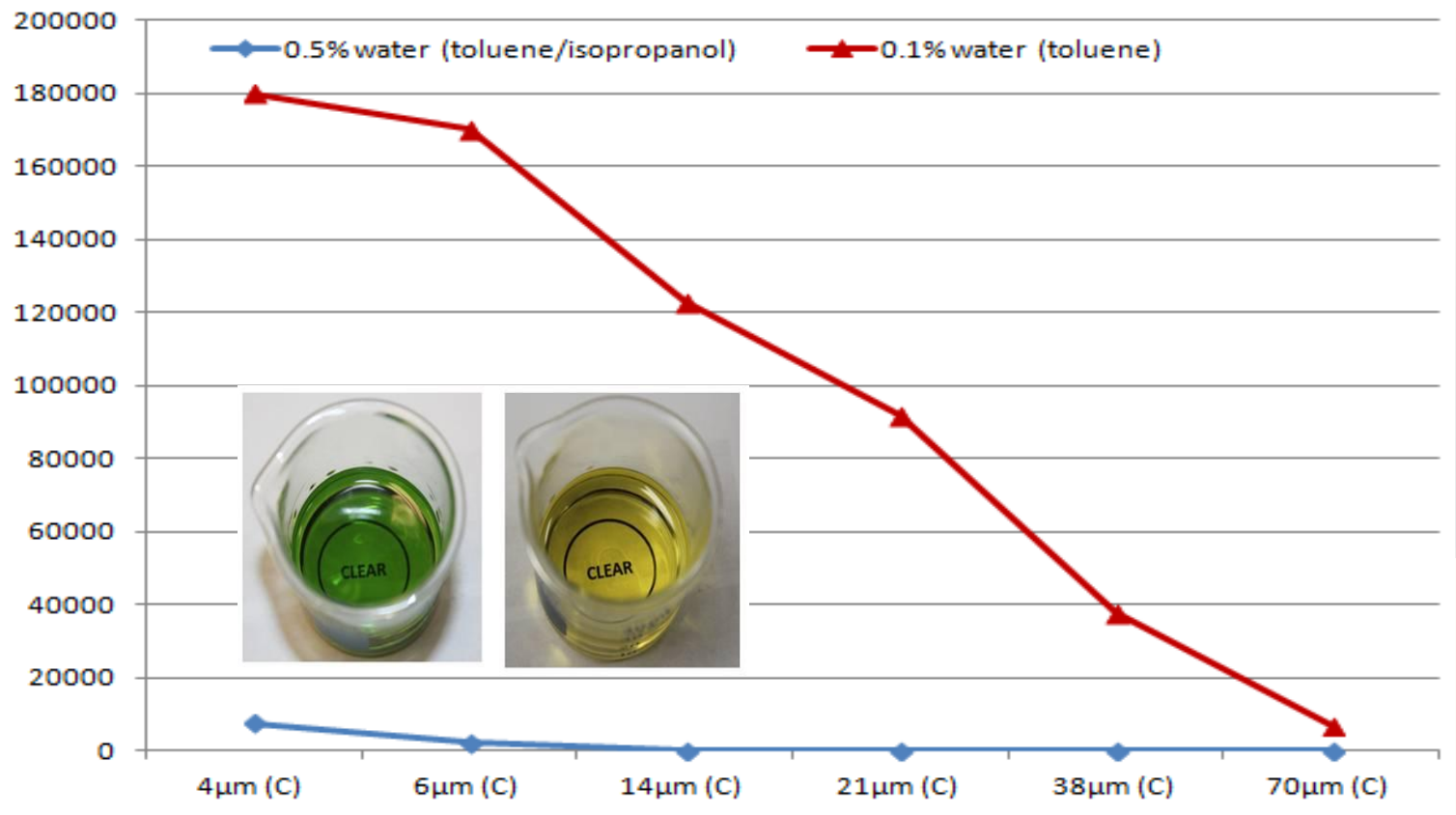
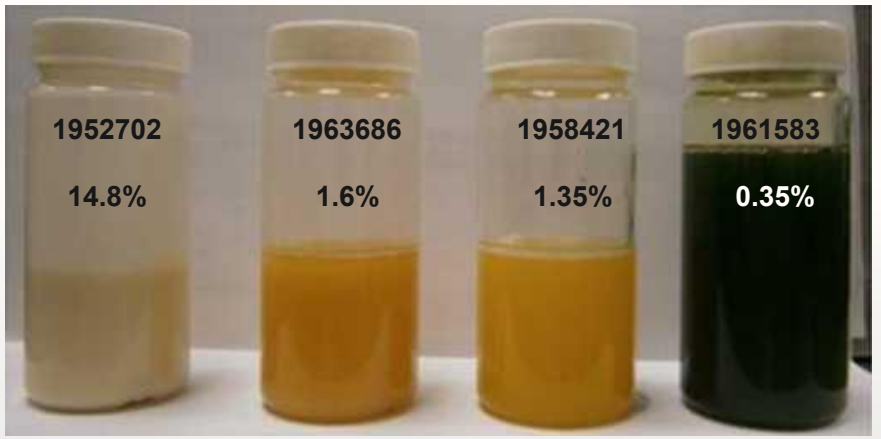


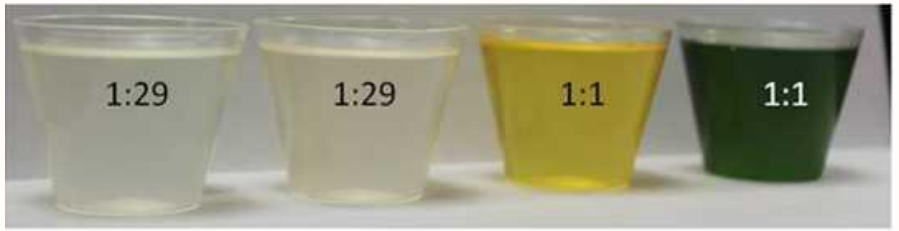
Figure 5 – 0.1% water contamination using 100% toluene as a diluent vs. 0.5% water contamination using 75% toluene / 25% isopropanol as a diluent.

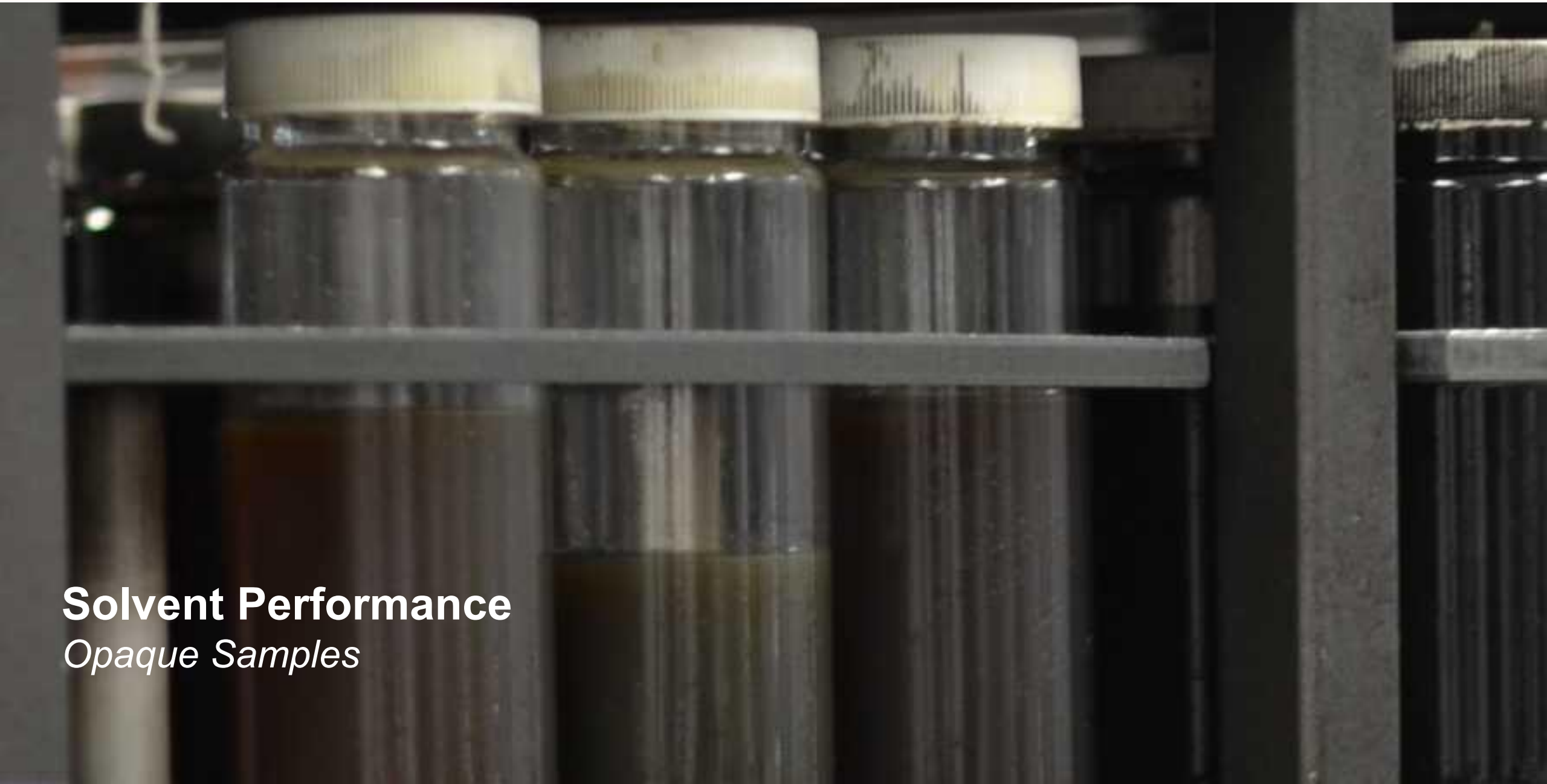
Samples – After Shaking



Samples – Before Dilution

Samples – After Dilution





Solvent Performance

Opaque Samples

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)

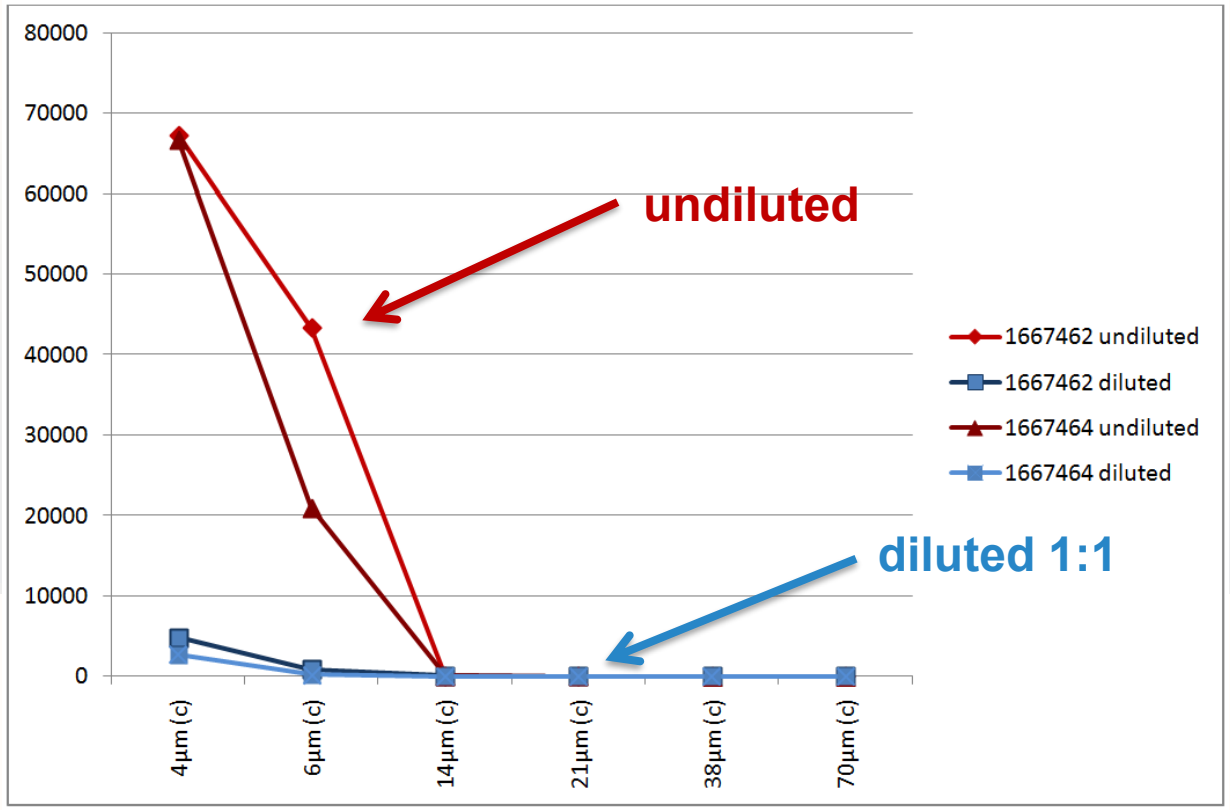
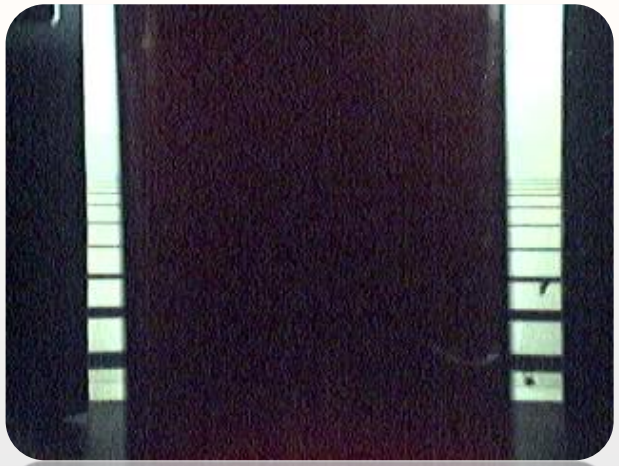
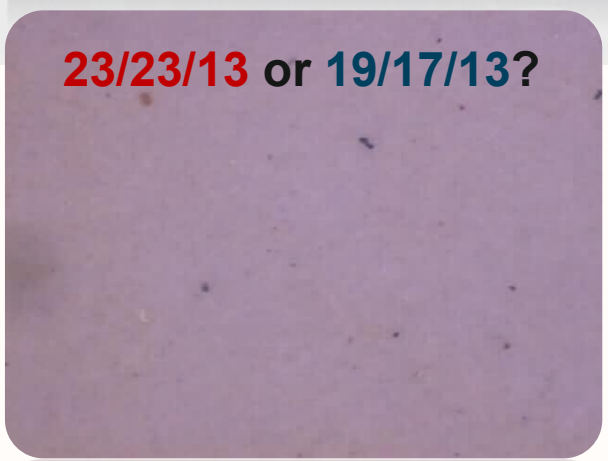


Figure 8 – Comparison of undiluted particle counts on the Hiac Royco SDS particle counter versus diluted particle counts for Castrol Optigear Synthetic A ISO 320 on the CS-APC-2 particle counter.

Sample 01667462



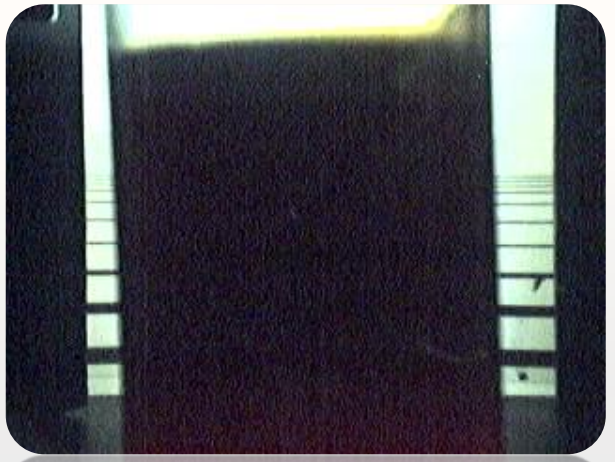
Color Image with Visibility Scale



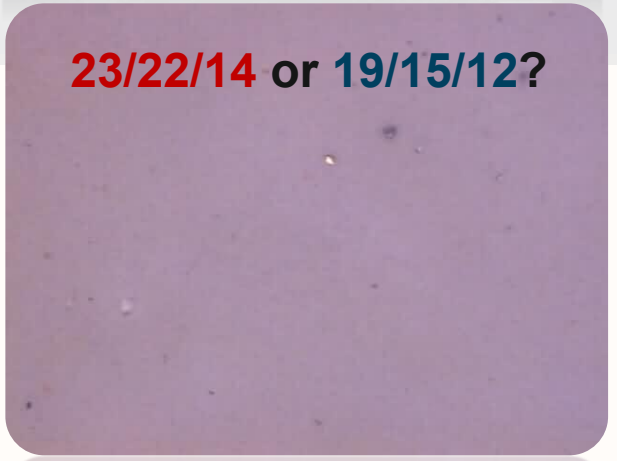
23/23/13 or 19/17/13?

Particle Debris Patch @ 100X magnification

Sample 01667464



Color Image with Visibility Scale



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

Particle Debris Patch @ 100X magnification

MPC Images / Results

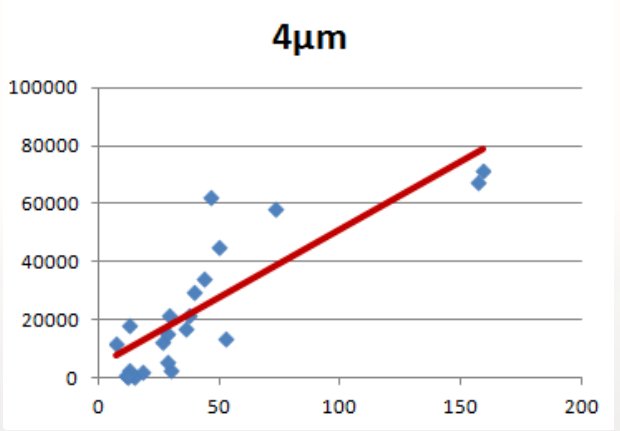
01964016 (MPC = 8)

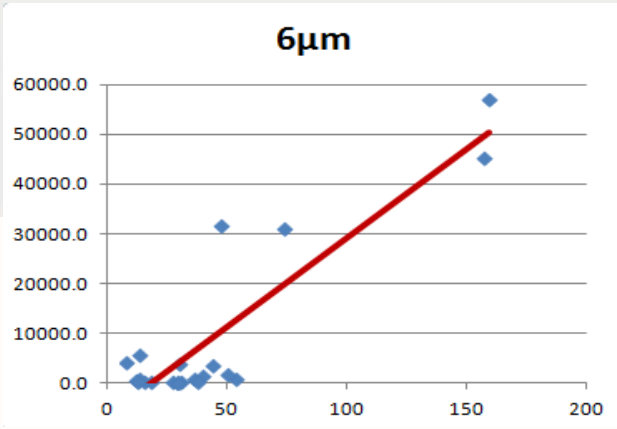
01961018 (MPC = 157)

01961020 (MPC = 73)

01961013 (MPC = 29)

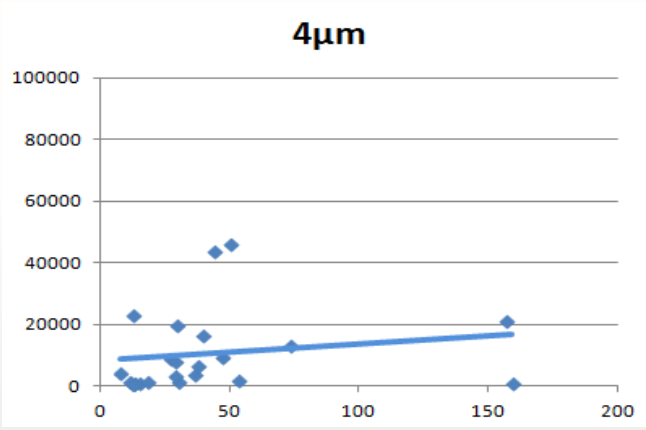
HIAC 8012

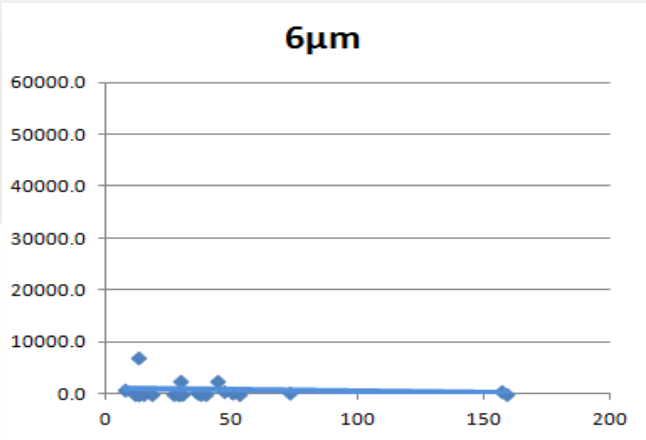




undiluted

CINRG CS-APC-2





diluted 1:1

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

Applications

Processing Diesel Engine Samples with Soot

Procedure Measurement of Samples with Soot

Sample Type

Diesel engine oil samples with < 1.5% soot

Materials

- Dilution Solvent - 75% Toluene / 25% Isopropanol (mix and let settle for 12 hours)
- Sample Diluent - Filtered White Oil/API Group II Base Oil (filtered to ISO 13/12/9 or better)

Procedure

1. Set stirring speed to 6 and stirring time to 1 minute.
2. Run sample on FT-IR for soot % measurement (if less than 1.5% - LDS30/30 sensor, or less than 1.0% - LDS 45/50 sensor then process on CINRG SDS).
3. Dilute sample with filtered white oil [sample:oil] [1:49].
4. Add 15 ml of diluted sample to sample cup [sample:solvent] [1:1].
5. Run samples using standard processing.

Klotz 30/30 Sensor

(Klotz Factory Calibration - similar to but not traceable to NIST SRM 2806b)

Coincidence Limit = 120,000 particles/ml.

Dilution	SAMPLE ID	Fe	SOOT %	4µm(c)	6µm(c)	14µm(c)	21µm(c)	38µm(c)	70µm(c)	ISO 4406	APC_2 Soot
0.51	1_100	11	0 (<0.05)	14881	641	8.3	1.8	0.1	0.1	21/17/10	>0.0005
0.51	3_100	31	0.2	14950	780	35	7.3	0.5	0.1	21/17/12	0.002
0.5	4_100	29	0.3	5768	358	9.7	1.7	0.1	0	20/16/10	0.003
0.5	5_100	54	0.4	5720	501	10.1	1.9	0.3	0.1	20/16/11	0.004
0.5	6_100	46	0.5	47552	3346	27.8	4.7	0.8	0.1	23/19/12	0.005
0.51	7_100	100	0.6	35461	2309	30.7	3.1	0.1	0	22/18/12	0.006
0.5	8_100	22	0.7	11033	823	23	5.3	0.9	0	21/17/12	0.007
0.5	9_100	44	0.8	27732	729	4.5	0.8	0.1	0	22/17/9	0.008
0.5	10_100	42	0.9	25478	889	8.3	2.8	0.5	0	22/17/10	0.009
0.5	11_100	83	1	64965	2067	8.1	2.6	1.2	0.8	23/18/10	0.01
0.5	12_100	78	1.1	70931	1669	34.1	5.2	0.3	0	23/18/12	0.011
0.51	13_100	28	1.2	3943	126	10.1	2.1	0.4	0	19/14/11	0.012
0.5	14_100	26	1.3	31678	228	11.1	2.5	0.4	0.1	22/15/11	0.013
0.5	15_100	118	1.4	53440	1170	24.4	5.4	0.7	0	23/17/12	0.014

NOTE: Actual dilution is approximately100:1

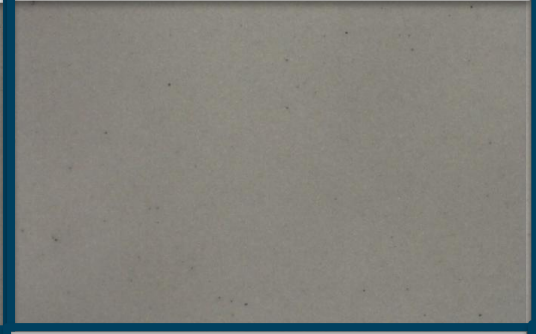
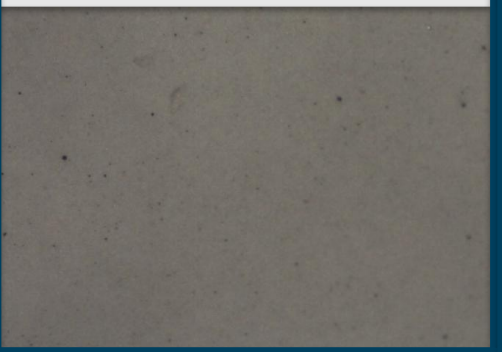
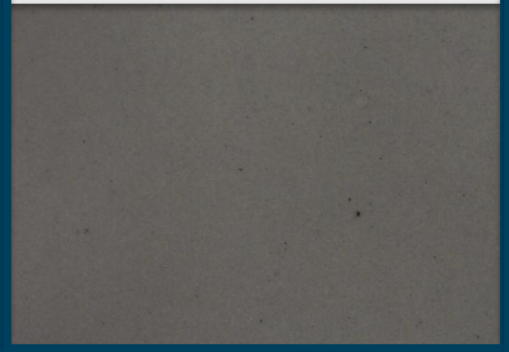
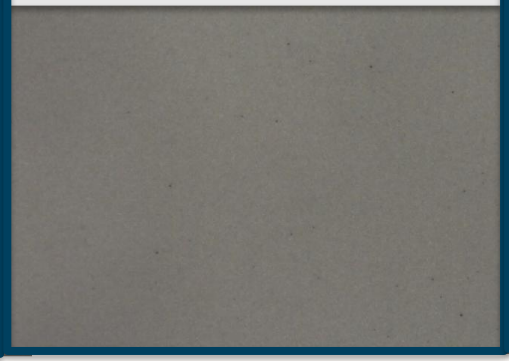
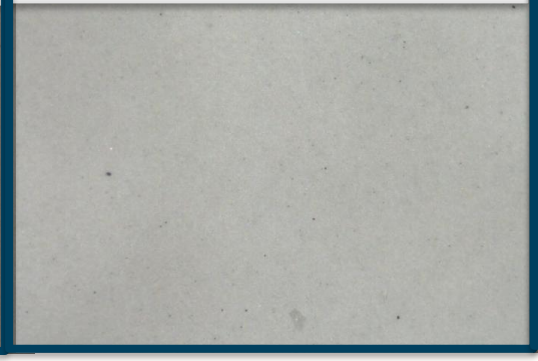
Klotz 45/50 Sensor

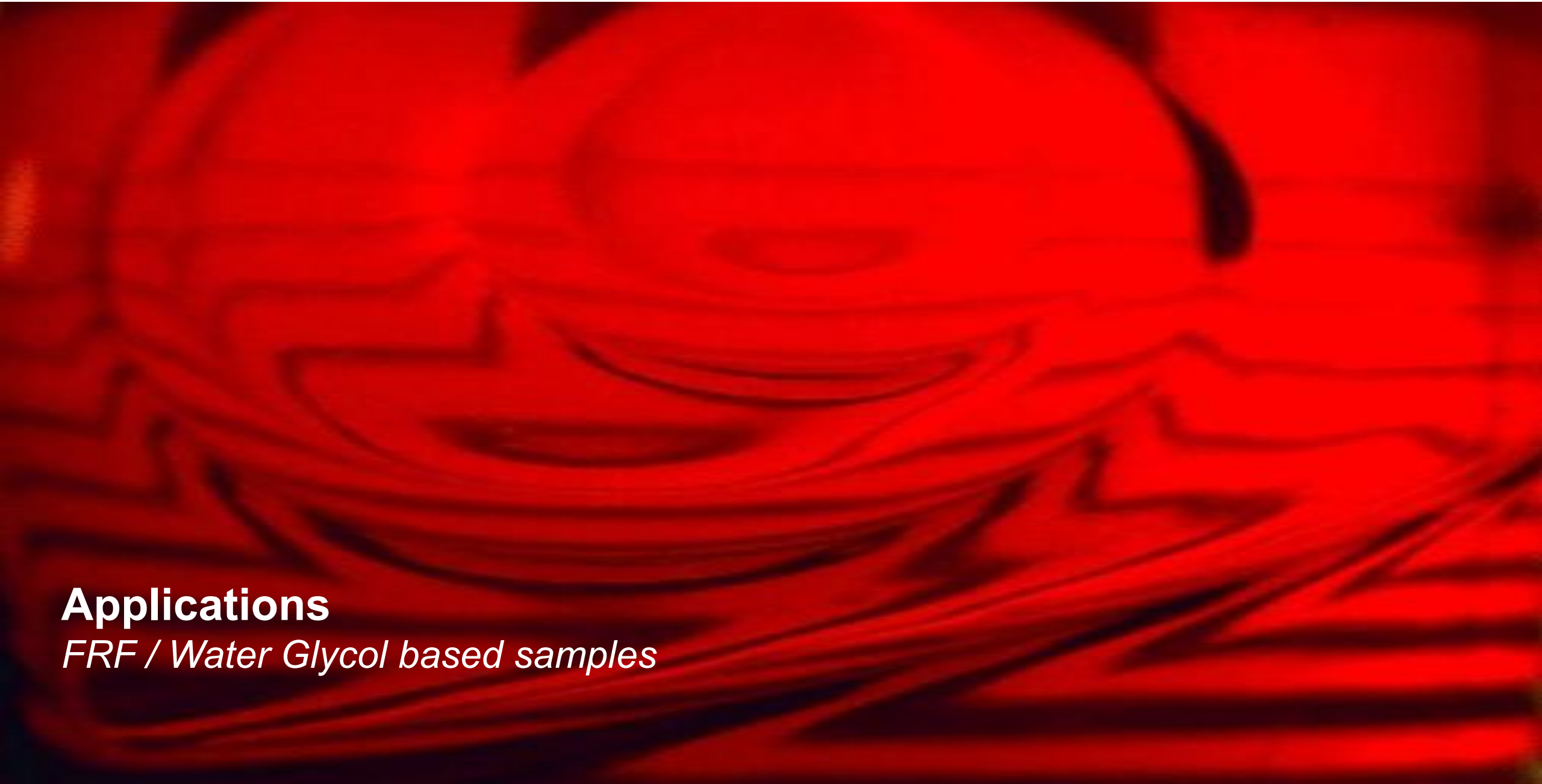
(CINRG Calibration with Conostan 2806 secondary Calibration fluid traceable to NIST SRM 2806b)

Coincidence Limit = 25000 particles/ml.

Dilution	SAMPLE ID	Fe	% SOOT	4µm(c)	6µm(c)	14µm(c)	21µm(c)	38µm(c)	70µm(c)	ISO 4406	APC_2 Soot
0.51	1_100	11	**0 (<0.05)**	19759	813	12.7	3.5	0.4	0	**21/17/11**	>0.0005
0.51	3_100	31	**0.2**	24212	922	42.8	10	0.5	0	**22/17/13**	0.002
0.51	4_100	29	**0.3**	7252	578	10.8	1.4	0	0	**20/16/11**	0.003
0.5	4a_100	54	**0.4**	8618	450	10.4	2.4	0.3	0	**20/16/11**	0.004
0.52	6_100	46	**0.5**	47428	3931	38.5	6.5	0.9	0.6	**23/19/12**	0.005
0.51	7_100	100	**0.6**	43832	2690	42.1	4.4	0.4	0.1	**23/19/13**	0.006
0.51	8_100	22	**0.7**	19102	1079	30.2	7.3	0.8	0.1	**21/17/12**	0.007
0.51	9_100	44	**0.8**	39612	966	7.7	1.7	0.7	0.7	**22/17/10**	0.008
0.5	10_100	42	**0.9**	35671	1166	8.8	2	0	0	**22/17/10**	0.009
0.5	11_100	83	**1**	0	0	0	0	0	0	**-/-/-**	0.01
0.5	12_100	78	**1.1**	0	0	0	0	0	0	**-/-/-**	0.011
0.5	13_100	28	**1.2**	0	0	0	0	0	0	**-/-/-**	0.012
System stopped	14_100	26	**1.3**								0.013

NOTE: Actual dilution is approximately100:1

ISO 21/17/10 0.1%	ISO 21/17/12 0.2%	ISO 20/16/10 0.3%	ISO 20/16/11 0.4%	ISO 23/19/12 0.5%
				
ISO 22/18/12 0.6%	ISO 21/17/12 0.7%	ISO 22/17/9 0.8%	ISO 22/17/10 0.9%	ISO 23/18/10 1.0%
				
ISO 23/18/12 1.1%	ISO 19/14/11 1.2%	ISO 22/15/11 1.3%	ISO 23/17/12 1.4%	ISO 22/19/13 reference
				



Applications

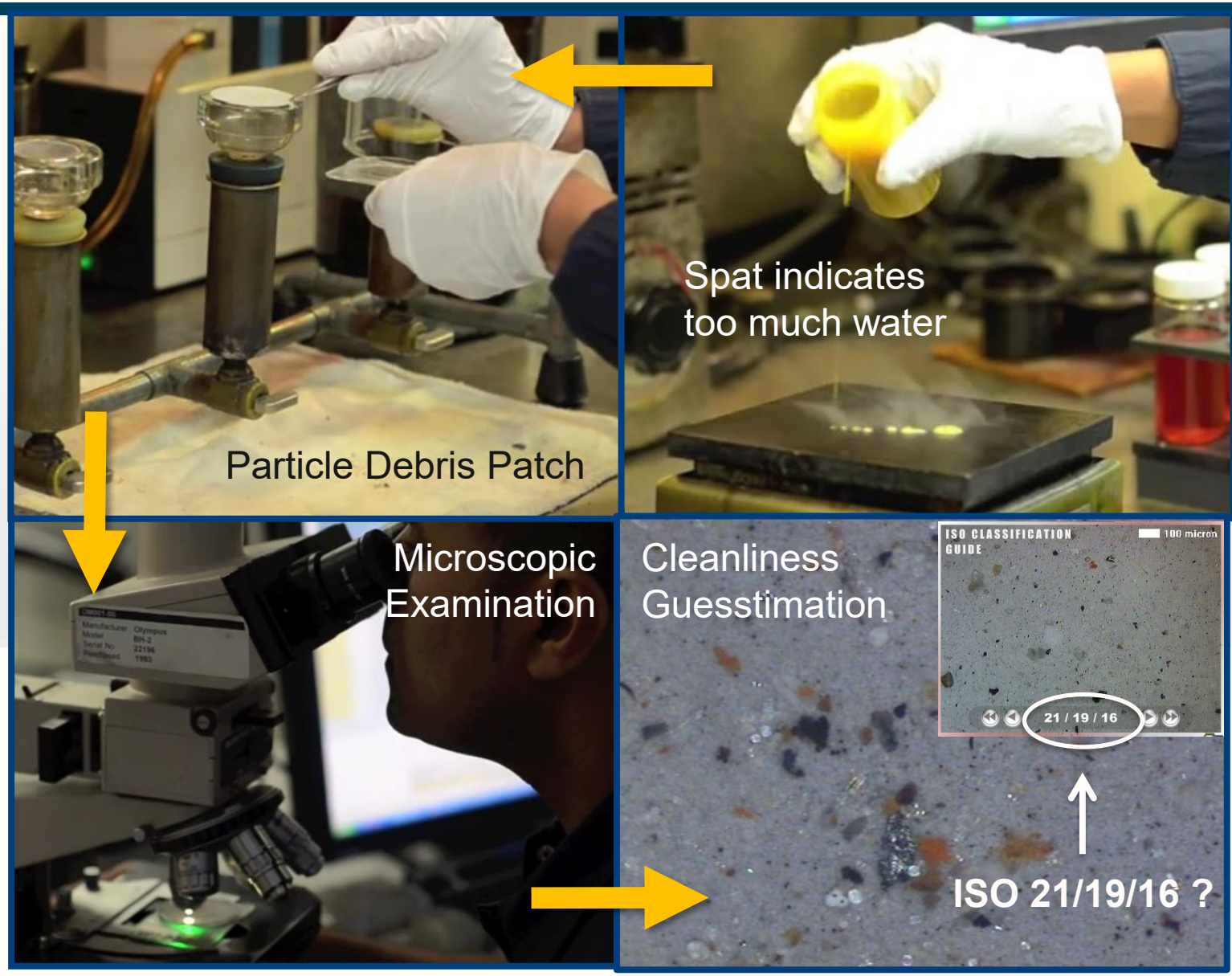
FRF / Water Glycol based samples

Procedure Prior to ASTM D7647

Sample Type
FRF – Fire-Resistant Fluids

Materials A
- Millipore 0.8µm filter patch

Procedure A
1. Spat indicates >0.1% water.
2. Filter 10 ml through a Millipore patch.
3. Dry, then examine under a microscope
4. Compare the patch to a set of ISO 4406 Classification images and select the closest ISO xx/yy/zz match.
5. Particle count size values are mean of the ISO class selected.



Procedure Measurement of FRF / Water Glycol Fluids

Sample Type

FRF – Fire-Resistant Fluids (i.e. Houghton Houghto-Safe 620) typically have between 35% and 45% water

Materials A

- Dilution Solvent - 40% Heptane / 60% Isopropanol (filter through 0.45µm Durapore filter, HVLP)

Procedure A

1. Add 2 ml of diluted sample to sample cup [sample:solvent] [1:15].
2. Run samples using standard processing.

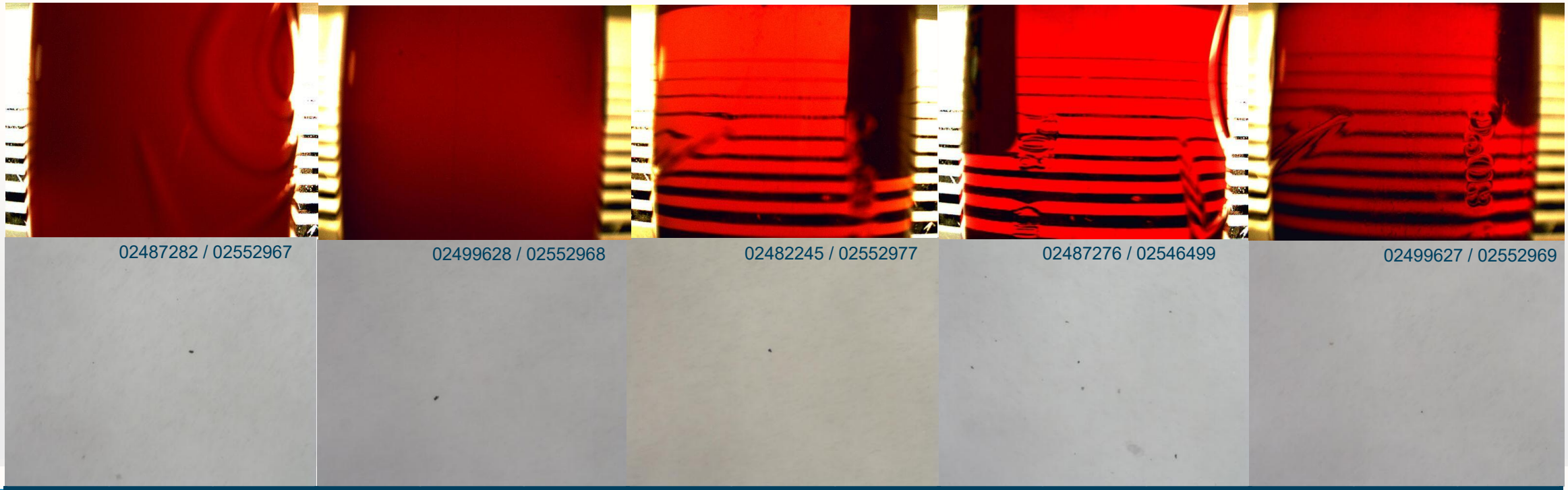
Materials B

- Dilution Solvent - Ethylene glycol butyl ether (2-Butoxyethanol) or DPnP (let settle for 12 hours)

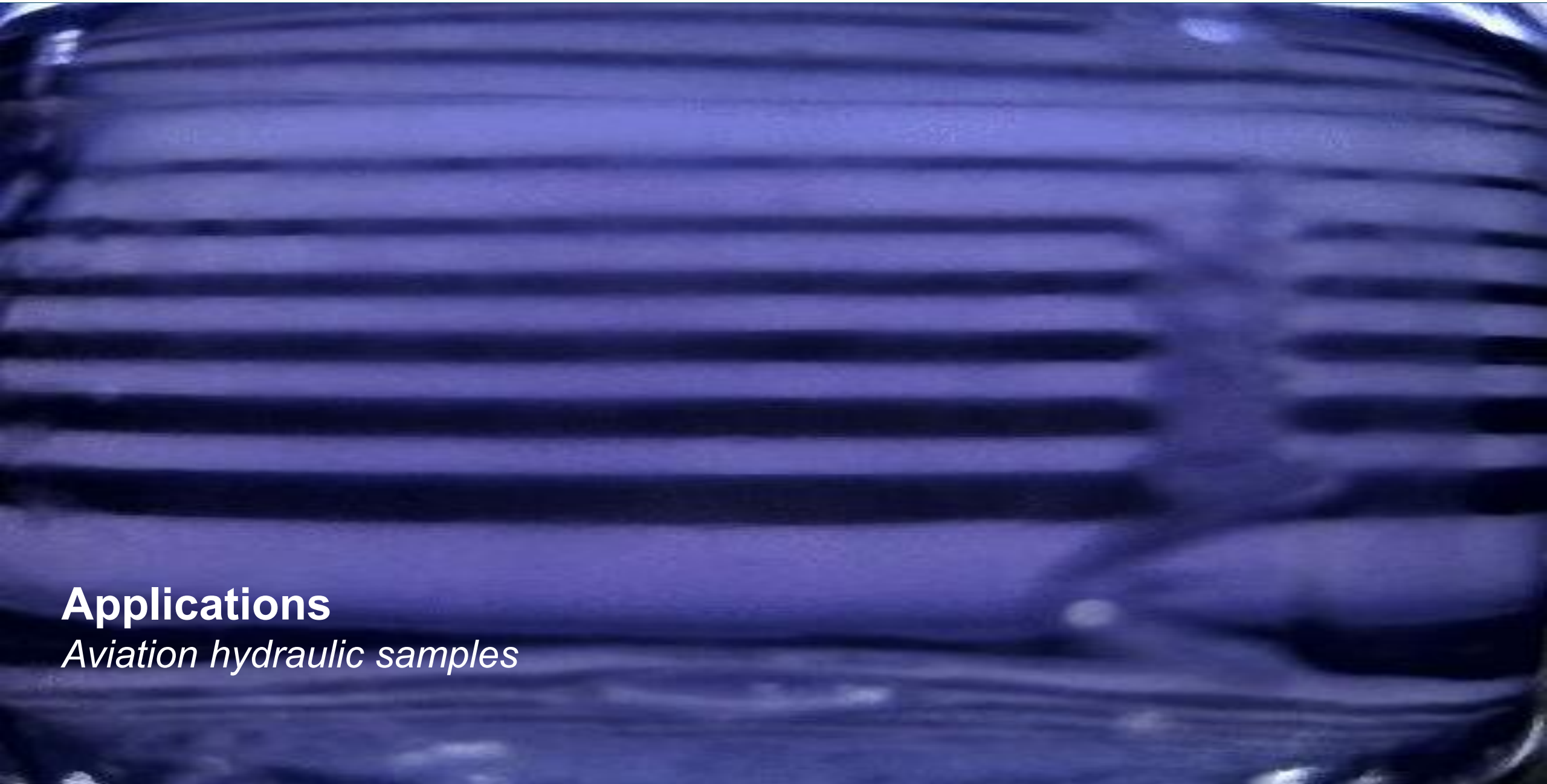
Procedure B

1. Set stir speed to 6, using a minimum mixing time of 1 minute.
2. Add 5 ml of sample to sample cup [sample:solvent] [1:5].
3. Run samples using standard processing.
4. If sample fails, reprocess adding only 1ml of sample to sample cup [sample:solvent] [1:29].

Historical Particle Counts Effects between Heptane/Iso vs. EGBE



Size	Visual	Hep/Iso	EGBE	Visual	Hep/Iso	EGBE	Visual	Hep/Iso	EGBE	Visual	Hep/Iso	EGBE	Visual	Hep/Iso	EGBE
>4µm	120	2568	1086	120	2069	181	120	29951	111	240	1186	2772	120	3381	358
>6µm	60	786	185	60	538	75	60	8486	57	120	296	504	60	830	75
>14µm	7	70	13	7	30	11	7	546	18	15	31	22	7	30	10
>21µm	0	28	4	0	8	4	0	87	13	2	20	11	0	8	3
>38µm	0	0	0	0	2	2	0	2	3	0	4	3	0	4	0
>71µm	0	0	0	0	2	2	0	0	1	0	0	1	0	2	0
ISO 4406	14/13/10	19/17/13	17/15/11	15/14/11	18/16/12	15/13/11	14/13/10	22/20/16	14/13/11	15/14/11	17/15/12	19/16/12	14/13/10	19/17/12	16/13/10



Applications

Aviation hydraulic samples

ISO 4406:1999

ISO code number	Number of particles per ml	
	More than	Up to and including
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	5	10
09	2.5	5
08	1.3	2.5
07	0.64	1.3

- Based on ISO11171 Calibration
- MTD Calibration Fluid
- Light Extinction Based Method
- Cumulative Count
- >4, >6, >14, >21, >38, >70 micron

NAS 1638:1987

Size range		5–15 µm	15–25 µm	25–50 µm	50–100 µm	>100 µm
NAS classes (based on maximum contamination limits, particles per 100ml)	00	125	22	4	1	0
	0	250	44	8	2	0
	1	500	89	16	3	1
	2	1,000	178	32	6	1
	3	2,000	356	63	11	2
	4	4,000	712	126	22	4
	5	8,000	1,425	253	45	8
	6	16,000	2,850	506	90	16
	7	32,000	5,700	1,012	180	32
	8	64,000	11,400	2,025	360	64
	9	128,000	22,800	4,050	720	128
	10	256,000	45,600	8,100	1,440	256
	11	512,000	91,000	16,200	2,880	512
	12	1,024,000	182,400	32,400	5,760	1,024

- Based on ISO4402 Calibration
- ACFTD Calibration Fluid
- Optical Microscope Based Method
- Differential Count
- 5-15, 15-25, 25-50, 50-100, >100 micron

SAE AS4059 (Differential)

Contamination Levels	(1)	5, incl. to 15, incl. µm	15, excl. to 25, incl. µm	25, excl. to 50, incl. µm	50, excl. to 100, incl. µm	>100 µm
	(2)	6, incl. to 14, incl. µm(c)	14, excl. to 21, incl. µm(c)	21,excl. to 38, incl. µm(c)	38, excl. to 70, incl. µm(c)	>70 µm(c)
00		125	22	4	1	0
0		250	44	8	2	0
1		500	89	16	3	1
2		1000	178	32	6	1
3		2000	356	63	11	2
4		4000	712	126	22	4
5		8000	1425	253	45	8
6		16 000	2850	506	90	16
7		32 000	5700	1012	180	32
8		64 000	11 400	2025	360	64
9		128 000	22 800	4050	720	128
10		256 000	45 600	8100	1440	256
11		512 000	91 200	16 200	2880	512
12		1 024 000	182 400	32 400	5760	1024

(1) Size range, microscope particle counts, based on longest dimension as measured per AS598 or ISO 4407.
(2) Size range, APC calibrated per ISO 11171 or an optical or electron microscope with image analysis software, based on projected area equivalent diameter.
(3) Contamination classes and particle count limits are identical to NAS 1638.

- Based on ISO11171 Calibration
- MTD Calibration Fluid
- Light Extinction or Optical Microscope Method
- Differential Count
- 6-14, 14-21, 21-38, 38-70, >70 micron
- Result is a “Contamination Class”
- EQUIVALENT TO NAS 1638

SAE AS4059 (Cumulative)

Contamination Levels	(1)	>1 µm	>5 µm	>15 µm	>25 µm	>50 µm	>100 µm
	(2)	>4 µm(c)	>6 µm(c)	>14 µm(c)	>21 µm(c)	>38 µm(c)	>70 µm(c)
000		195	76	14	3	1	0
00		390	152	27	5	1	0
0		780	304	54	10	2	0
1		1560	609	109	20	4	1
2		3120	1217	217	39	7	1
3		6250	2432	432	76	13	2
4		12 500	4864	864	152	26	4
5		25 000	9 731	1731	306	53	8
6		50 000	19 462	3462	612	106	16
7		100 000	38 924	6924	1224	212	32
8		200 000	77 849	13 849	2449	424	64
9		400 000	155 698	27 698	4898	848	128
10		800 000	311 396	55 396	9796	1696	256
11		1 600 000	622 792	110 792	19 592	3392	512
12		3 200 000	1 245 584	221 584	39 184	6784	1024

(1) Size Range, Optical Microscope, based on longest dimension as measured per AS598 or ISO 4407.
(2) Size Range, APC Calibrated per ISO 11171 or an optical or electron microscope with image analysis software, based on projected area equivalent diameter.

- Based on ISO11171 Calibration
- MTD Calibration Fluid
- Light Extinction or Optical Microscope Method
- Cumulative Count
- >4, >6, >14, >21, >38, >70 micron
- Result is a “Contamination Code”
- MORE EQUIVALENT TO ISO4406:1999

ISO/DIS 4406 BS 5540/4 codes	NAS 1638
13 / 11 / 08	2
14 / 12 / 09	3
15 / 13 / 10	4
16 / 14 / 09	
16 / 14 / 11	5
17 / 15 / 09	
17 / 15 / 10	
17 / 15 / 12	6
18 / 16 / 10	
18 / 16 / 11	
18 / 16 / 13	7
19 / 17 / 11	
19 / 17 / 14	8
20 / 18 / 12	
20 / 18 / 13	
20 / 18 / 15	9
21 / 19 / 13	
21 / 19 / 16	10
22 / 20 / 13	
22 / 20 / 17	11
23 / 21 / 14	
23 / 21 / 18	12
24 / 22 / 15	
25 / 23 / 17	

Size range in µm	5, incl. to 15, incl. µm	15, excl. to 25, incl. µm	25, excl. to 50, incl. µm	50, excl. to 100, incl. µm	>100 µm
Particle count per 100 mL	114 000	9200	3920	172	52
Contamination Level	9	8	9	7	8
Contamination Code	AS4059 Contamination Code [9/8/9/7/8]				
AS4059 Contamination Class	AS4059 Class 9				
NAS 1638 Contamination Class	NAS 1638 Class 9				

Size range in µm	>4 µm(c)	>6 µm(c)	>14 µm(c)	>21 µm(c)	>38 µm(c)	>70 µm(c)
Particle count per 100 mL	382 000	127 000	13 300	4140	224	52
Contamination Level	9	9	8	9	8	8
Contamination Code	AS4059 Contamination Code cpc[9/9/8/9/8/8]					
AS4059 Contamination Class	AS4059 cpc Class 9					
ISO 4406 Cleanliness Code	ISO 4406 19/17/14					

Procedure Measurement of Samples for AS4059 (equivalent to NAS 1638)

Sample Type

Aviation hydraulic fluids max ISO 46 (i.e. Skydrol LD-4, 500B-4)

Materials

- No materials required

Procedure

1. Set stirring speed to 6 and stirring time to 1 minute.
2. Run sample **neat** by adding 28 to 30 ml of sample to the sample cup.
3. Run samples using standard processing.

Class	Maximum Particles/100mL in Specified Size Range (µm)				
	5-15	15-25	25-50	50-100	>100
00	125	22	4	1	0
0	250	44	8	2	0
1	500	89	16	3	1
2	1,000	178	32	6	1
3	2,000	356	63	11	2
4	4,000	712	126	22	4
5	8,000	1,425	253	45	8
6	16,000	2,850	506	90	16
7	32,000	5,700	1,012	180	32
8	64,000	11,400	2,025	360	64
9	128,000	22,800	4,050	720	128
10	256,000	45,600	8,100	1,440	256
11	512,000	91,200	16,200	2,880	512
12	1,024,000	182,400	32,400	5,760	1,024

NOTE:

- Removes issues with particle counts considering APC counts are 3 x 5 ml instead on 1 x 100ml.
- Running the sample neat keeps more of the larger particles (>14µm) in suspension.
- Specify up to 3 decimal places for results in software settings.

CINRG CS-APC Output File (Example)

Sample_Number:02333147
Time_Stamp:1/24/2020 8:26:20 AM
ISO_4406:20/18/15
IVL:PASS
ASTM7647:PASS
4059_Dif_Code:10/9/10/7/1
4059_Cum_Code:10/10/9/10/7/000
Undiluted_Fraction:0.42
um_Sizes:4,6,14,21,38,70
um_Cumulative_Counts_Per_ml:6091.8,1809.6,205.4,54.5,1.6,0.0
um_Cumulative_Counts_Per_100ml:609177,180964,20539,5452,159,0
Solvent_um_Sizes:4,6,14,21,38,70
Solvent_um_Cumulative_Counts_Per_ml:16.4,4.4,0.5,0.2,0.0,0.0

IVL – Internal variance limits (customizable) – i.e. 5%

ASTM7647 – Hard-coded variance limits per method

SAE AS 4059 Contamination Code

1 ml & 100 ml cumulative counts

AS4059 / NAS 1638 Calculation

Sample_Number:02333147
Time_Stamp:1/24/2020 8:26:20 AM
ISO_4406:20/18/15
IVL:PASS
ASTM7647:PASS
4059_Dif_Code:10/9/10/7/1
4059_Cum_Code:10/10/9/10/7/000
Undiluted_Fraction:0.42
um_Sizes:4,6,14,21,38,70
um_Cumulative_Counts_Per_ml:6091.8,1809.6,205.4,54.5,1.6,0.0
um_Cumulative_Counts_Per_100ml:609177,180964,20539,5452,159,0
Solvent_um_Sizes:4,6,14,21,38,70
Solvent_um_Cumulative_Counts_Per_ml:16.4,4.4,0.5,0.2,0.0,0.0

Size	cum (100ml)	sum-cum	diff (100ml)
4µ	609177	207114	402063
6µ	180964	26150	154814
14µ	20539	5611	14928
21µ	5452	159	5293
38µ	159	0	159
70µ	0	---	0
Code	**10/10/9/10/7/000**	Class	**10**

Operation

Using PCS and Verification Fluids
Internal Variance Limits

Primary & Secondary Calibration Fluids for Optical Particle Counters

MIL-H-5606 Fluid

ACFTD *or* MTD

APC Calibration Fluid

Primary Calibration Fluid

- Sold by NIST
- Production contracted
- Tested by external parties
- Very expensive
- 3 x 400ml – US$4742

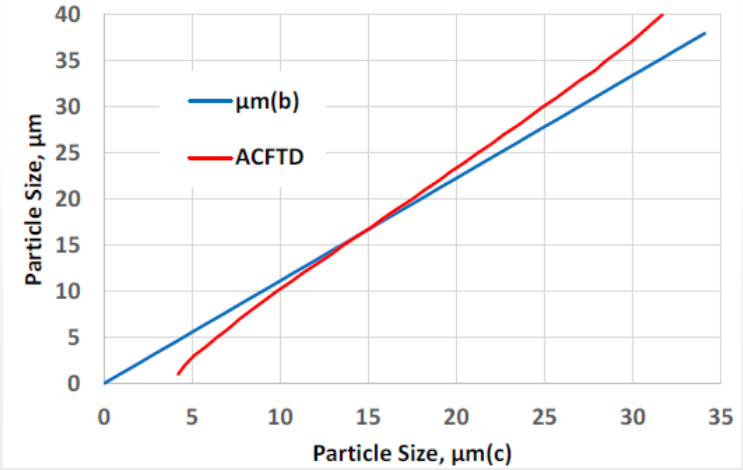
Secondary Calibration Fluid

- Prepared/sold by certified companies
- Traceability to primary NIST certification
- Less expensive
- 3 x 400ml – US$1155

APC Calibration Fluids History

Material	ISO Standard	Certificate Date	Reason for Revision	Expiration Date	Test Dust	Cert Method	NIST Traceable?
ACFTD	4402:1991	1960 – 1999	AC Fine Test Dust (ACFTD) no longer commercially available		ACFTD	FP/EM	No
ISO MTD SRM2806-0	11171:1999	10-Dec-97	ISO Medium Test Dust (MTD) - NIST Traceable Standard - Original Certificate		MTD	FP/EM/IPSW	Yes
ISO MTD SRM2806-1	11171:1999	1-Mar-99	Revised uncertainties and change of >30µm values to information values		MTD	FP/EM/IPSW	Yes
ISO MTD SRM2806-2	11171:1999	9-Aug-00	Revision of expiration date.		MTD	FP/EM/IPSW	Yes
ISO MTD SRM2806-3	11171:1999	16-Nov-04	Decrease in expiration date due to instability.	17-Sep-04	MTD	FP/EM/IPSW	Yes
ISO MTD SRM2806a-0	11171:1999	13-Oct-04	Original Certificate		MTD	FP/EM/IPSW	Yes
ISO MTD SRM2806a-1	11171:1999	29-Jan-07	Update of expiration date and editorial changes.		MTD	FP/EM/IPSW	Yes
ISO MTD SRM2806a-2	11171:1999	16-Dec-08	Extension of certification period.		MTD	FP/EM/IPSW	Yes
ISO MTD SRM2806a-3	11171:2010	30-May-13	Extension of certification period; editorial changes.	31-Dec-14	MTD	FP/EM/IPSW	Yes
ISO MTD SRM2806b-0	11171:2016	12-Jun-14	Original Certificate	31-Dec-20	MTD	FP/EM/IPSW	Yes
ISO MTD SRM2806d-0	11171:2020	1-Dec-20	Latest certification		MTD	ILS	Yes

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

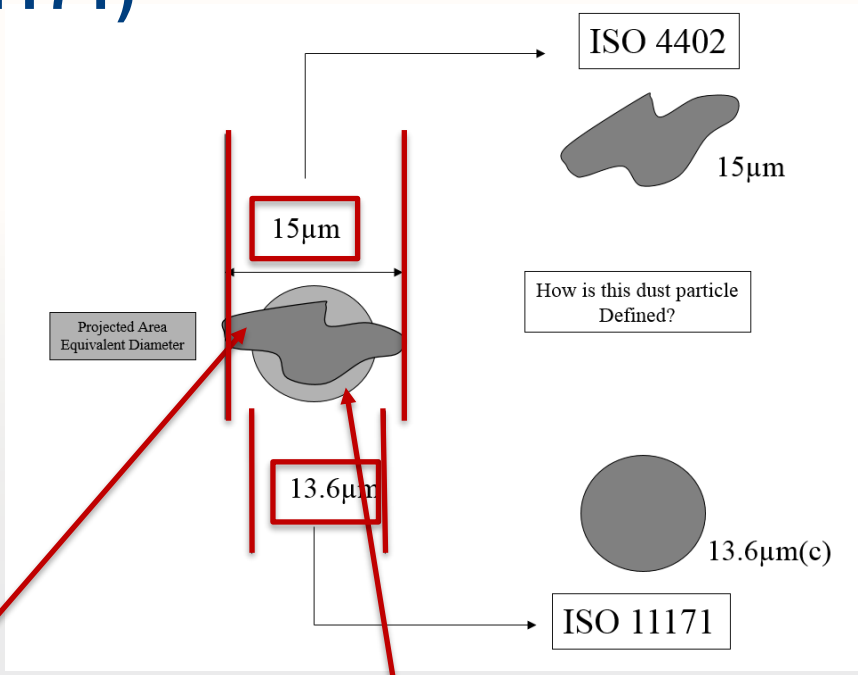


ACFTD	MTD
Not NIST traceable	NIST traceable
Particle size based on longest chord length	Particle size is circular area of cross section
ISO 4402 based on (2), 5, 15 µm	ISO 11171 based on 4, 6, 14 µm

ACFTD (Air Cleaner Fine Test Dust)

Replaced By

ISO MTD (Medium Test Dust)



Original Sizes
(2µm)
5µm
15µm

Replaced By
4µm(c)
6µm(c)
14µm(c)

Ref: Barry Verdegan, Cummins Filtration

Revision of ISO 4406:1999 – Method of Coding the level of Contamination by Solid Particles

ISO 4406 Cleanliness Codes

MORE THAN	UP TO AND INCLUDING	ISO CODE
(p/ml)	(p/ml)	
80,000	160,000	24
40,000	80,000	23
20,000	40,000	22
10,000	20,000	21
5,000	10,000	20
2,500	5,000	19
1,300	2,500	18
640	1,300	17
320	640	16
160	320	15
80	160	14
40	80	13
20	40	12
10	20	11
5	10	10
2.5	5	9
1.3	2.5	8

Results on fluid from APC with ACFTD Calibration

Particle Size (ACFTD)	Counts /ml	4406 Range Code
2µm	11200	21
5µm	4319	19
15µm	410	16

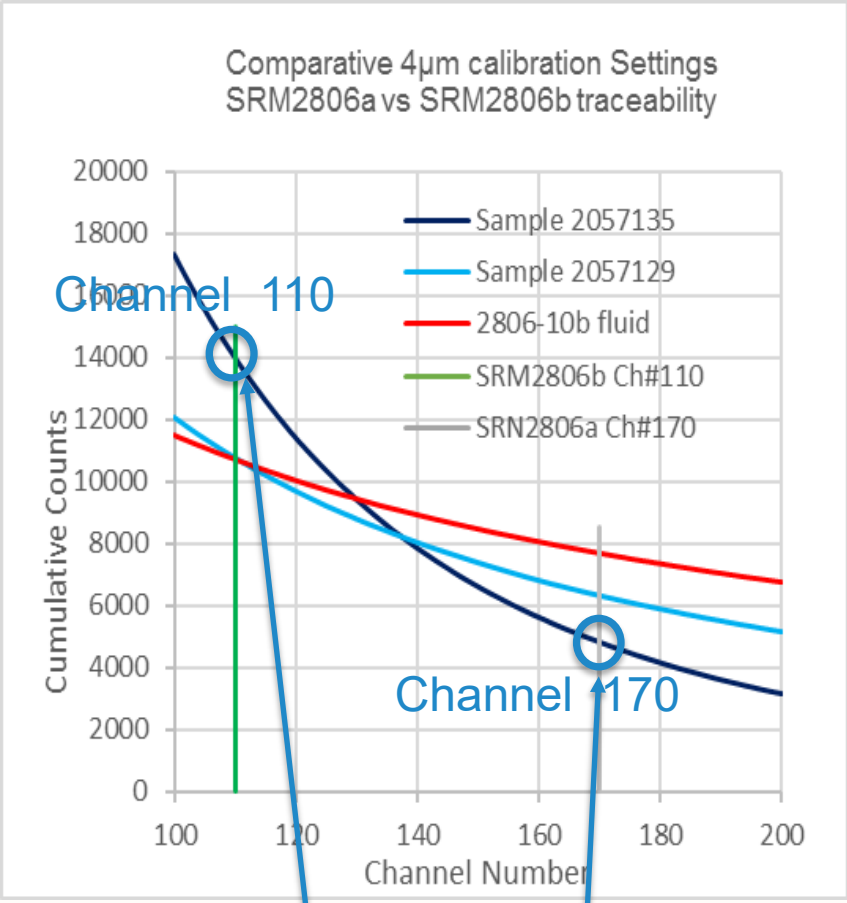
19/16
or
21/19/16
Code based on 2,5 and 15 µm counts/ml

Results on same fluid from APC with MTD Calibration

Particle Size (MTD)	Counts/ml	Range Code
2µm(c)	22340	22
4µm(c)	10842	21
5µm(c)	6681	20
6µm(c)	4210	19
14µm(c)	389	16
15µm(c)	300	15

21/19/16
Code based on 4,6 and 14 µm (c) counts/ml

SRM 2806b – What Happened?



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%

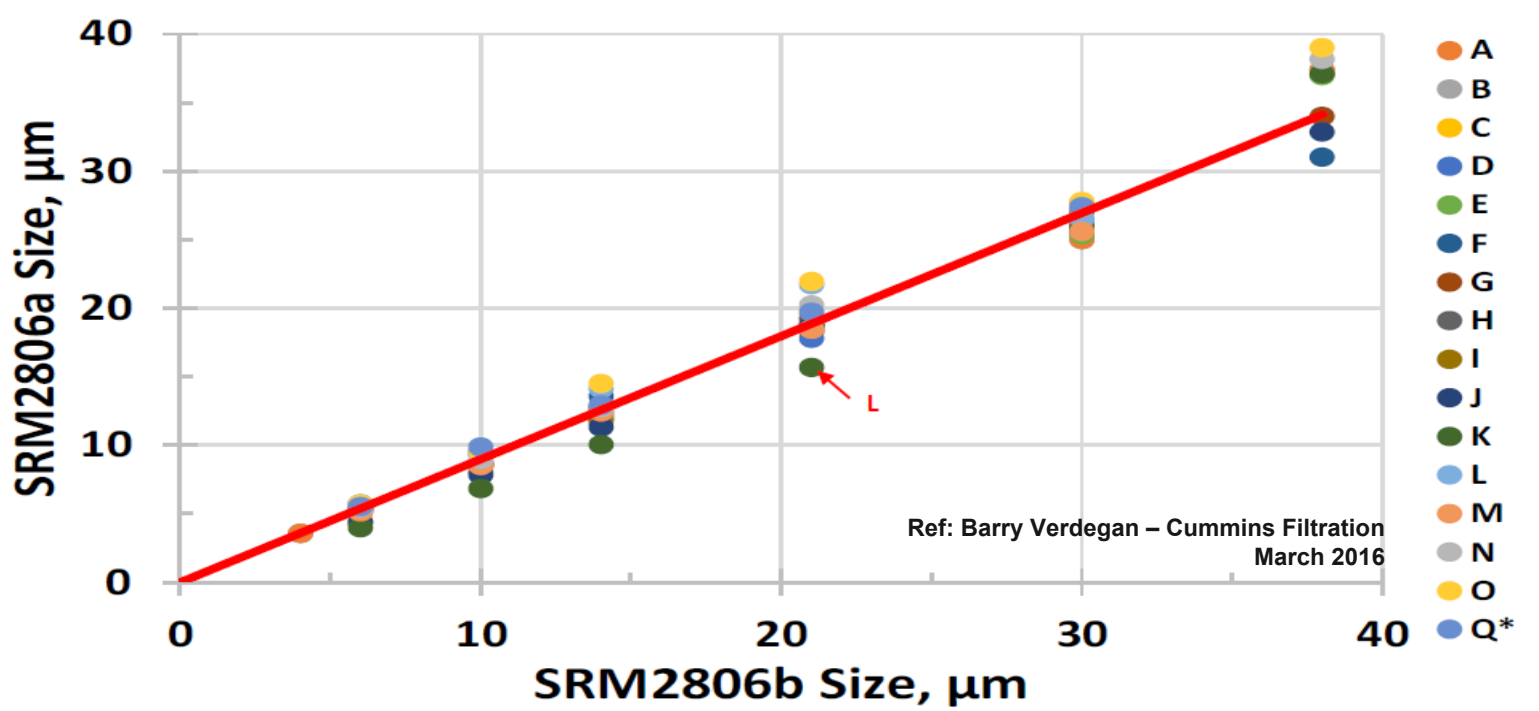
NOTE: The "error" is with SRM2806a not with SRM2806b

SRM 2806b – What is the Solution?

Calibration (b) to (c) Conversion Factor

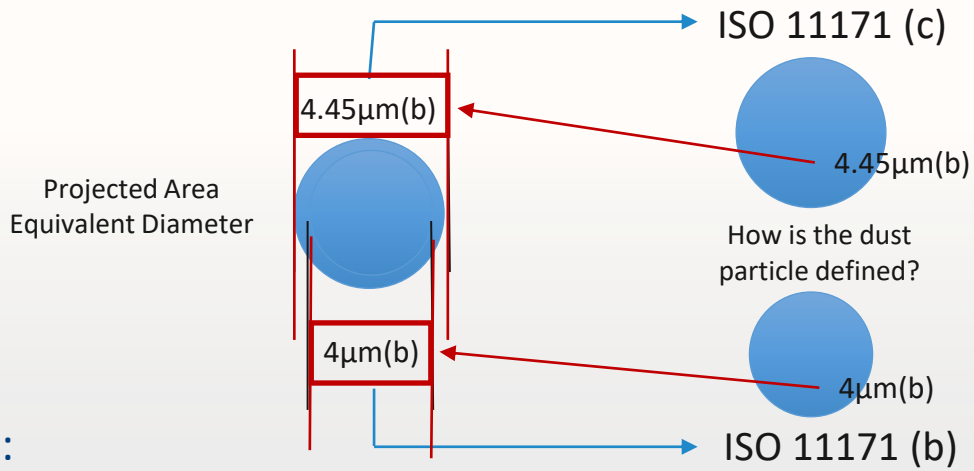
Lab	A	B	C	D	E	F	G	H	I	J	K	L	M	N	O	P	Q	Mean	s
m	0.886	0.875	0.927	0.844	0.891	0.882	0.904	0.873	0.895	0.880	0.880	0.938	0.859	0.948	0.987	---	0.924	**0.898**	**0.037**
R^2	0.978	0.999	0.994	0.998	0.985	0.985	0.996	0.997	0.997	0.997	0.973	0.980	0.998	0.995	0.990	---	0.994	**1.0**	**0.01**

- Round-robin with 15 labs in 4 countries (secondary samples from 7 sources)
- Linear relationship from 0-38µm
- Beyond 38µm can use latex spheres for calibration.



SRM 2806b – What is the Solution?

$$d_c = 0.898 d_b$$



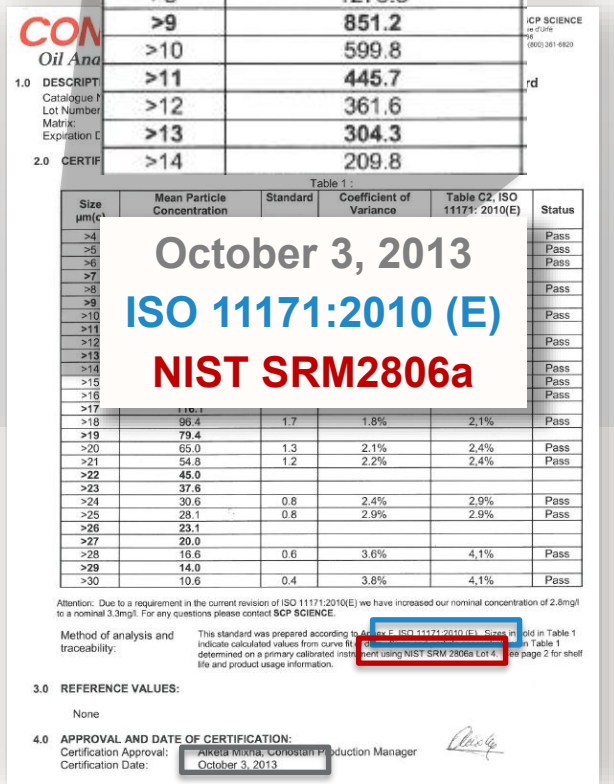
- Draft revision to ISO1171
- *Ability to report to SRM2806a: 4µm(c), 6µm(c), 14µm(c) using 4.45µm(b), 6.68µm(b), 15.6µm(b)
- Ability to report to SRM2806b: 4µm(b), 6µm(b), 14µm(b)

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.

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

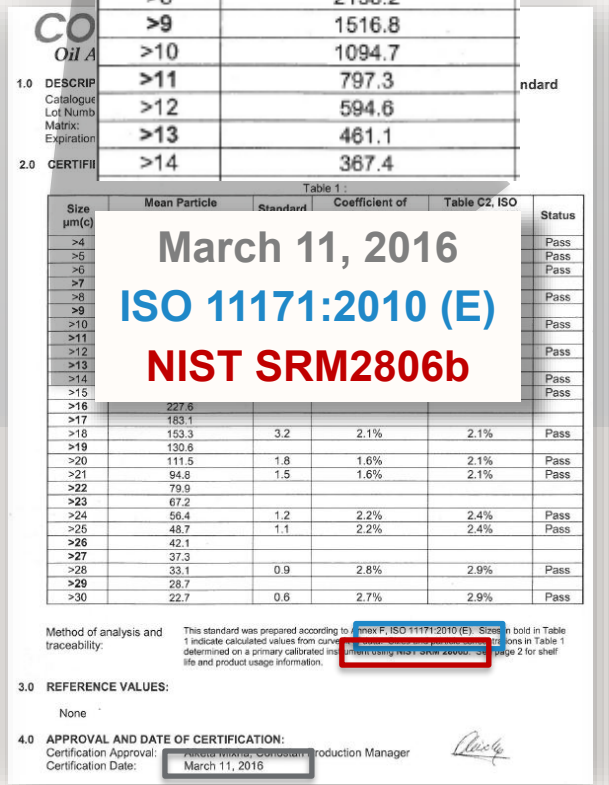
Secondary Calibration Fluid - RM2806a / RM2806b / RM2806d

Size µm(c)	Mean Particle Concentration (>Particles/mL)
>4	7300.5
>5	4385.6
>6	2907.9
>7	**1939.9**
>8	1273.8
>9	**851.2**
>10	599.8
>11	**445.7**
>12	361.6
>13	**304.3**
>14	209.8



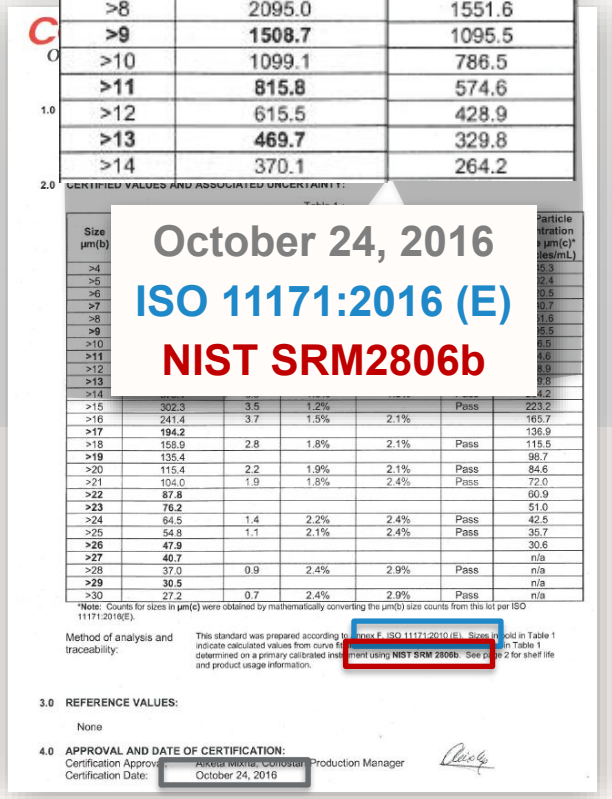
October 3, 2013
ISO 11171:2010 (E)
NIST SRM2806a

Size µm(c)	Mean Particle Concentration (>Particles/mL)
>4	10850.8
>5	6792.3
>6	4500.5
>7	3073.4
>8	2138.2
>9	1516.8
>10	1094.7
>11	797.3
>12	594.6
>13	461.1
>14	367.4



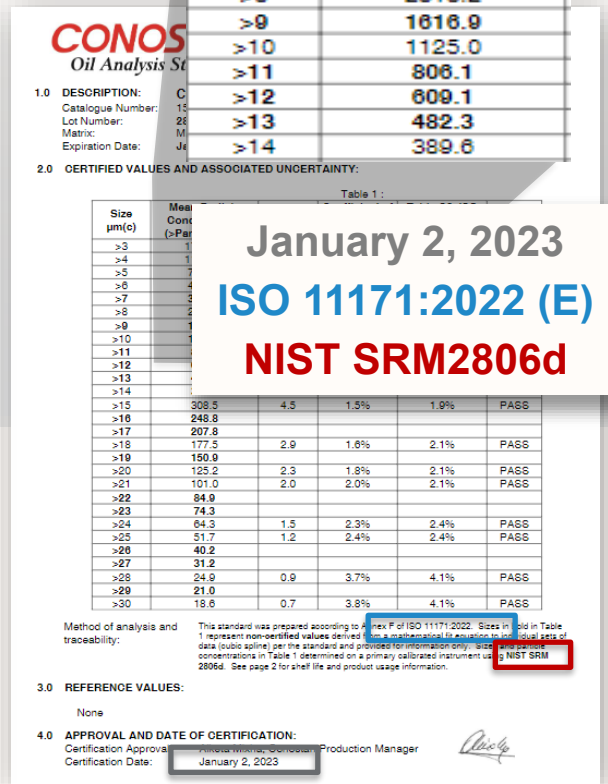
March 11, 2016
ISO 11171:2010 (E)
NIST SRM2806b

Size µm(b)	Mean Particle Concentration (>Particles/mL)	Mean Particle Concentration For size µm(c)* (>Particles/mL)
>4	10196.6	8245.3
>5	6472.3	5102.4
>6	4289.1	3320.5
>7	**2597.1**	2240.7
>8	2095.0	1551.6
>9	**1508.7**	1095.5
>10	1099.1	786.5
>11	**815.8**	574.6
>12	615.5	428.9
>13	**469.7**	329.8
>14	370.1	264.2



October 24, 2016
ISO 11171:2016 (E)
NIST SRM2806b

Size µm(c)	Mean Particle Concentration (>Particles/mL)
>3	17974.4
>4	11138.6
>5	7927.8
>6	4865.6
>7	3295.4
>8	2313.2
>9	1616.9
>10	1125.0
>11	806.1
>12	609.1
>13	482.3
>14	389.6



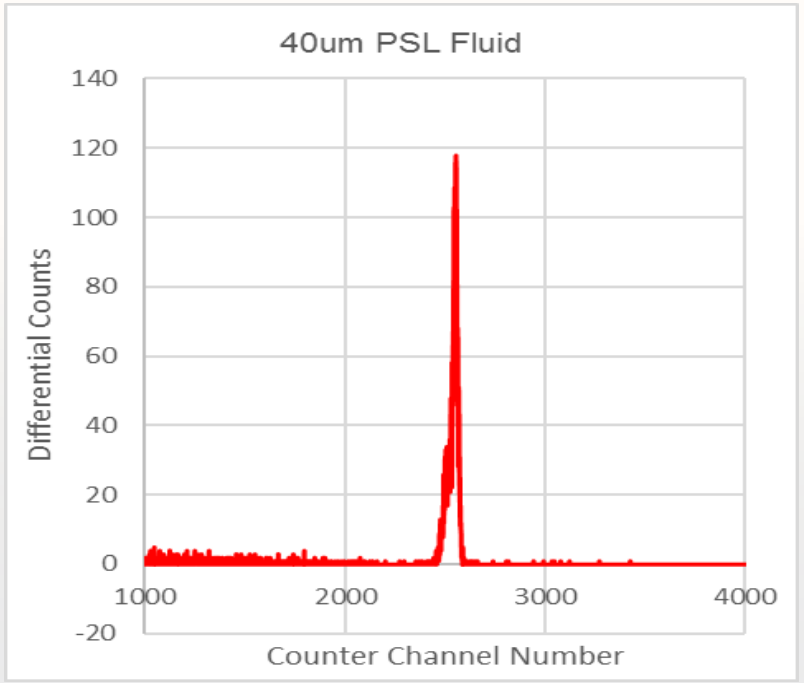
January 2, 2023
ISO 11171:2022 (E)
NIST SRM2806d

SRM2806 – Significant Changes

Standard	Date	Significant Changes
SRM 2806	1997	Discontinuation leads to change in calibration method (ISO 4402 -> ISO 11171). First traceable MTD based calibration fluid. Due to change in accuracy particle sizes are reported as 4(c), 6(c), 14(c)µm
SRM 2806a	2013	Counts reports as 4(c), 6(c), 14(c)µm
SRM 2806b	2014	Counts increased dramatically (approx. +40%). An inter-laboratory study was conducted and a calculate was determined to correlate the (c) and (b) fluids ($d_c = 0.898d_b$). Leads to ISO 11171:2016 revision. Counts can be reported as either (b) or (c) <– using calculation
SRM 2806d	2022	ISO 11171:2020 revision. Counts reported as (c) (the (b) reporting has now been eliminated). Fluid now certified using Inter-Laboratory Study (ILS). **Committee sets the ISO code as the priority!**

Calibrating 38 & 70µm

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



CINStan Calibration Kit

The CINRG CINStan Calibration Fluids Kit is suitable for a single calibration of a CS-APC-2 or CS-APC-22M instrument. The kit includes;

- CINStan (CONOSTAN) Super Clean Fluid (400 ml)
- CINStan PSL40 SCF Blend - 40µm monospheres in SCF (250 ml)
- CINStan PSL70 SCF Blend - 70µm monospheres in SCF (250 ml)
- CINStan PSL100 SCF Blend - 100µm monospheres in SCF (250 ml)
- PartiStan 2806 Calibration Fluid (400 ml)

Calibrating 38 & 70µm

Changes to ISO 11171:2022 allow for the use of coarse test dust to produce secondary calibration standards

- Must meet ISO 12103-1 specification
- Any size test dust may be used
  - A1 – Ultrafine Test Dust (UTD) 0-10µm
  - A2 – Fine Test Dust (FTD) 0-80µm
  - A3 – Medium Test Dust (MTD) 0-80µm
  - A4 – Coarse Test Dust (CTD) 0-180µm

Standards made with coarse test dust

- Include 38µm & 70µm particle sizes
- Increased counts (3 mg/L into MIL-H-5606 fluid)
- Longer shelf life than PSL (2 years vs. 3 months)
- No preparation required
- Lower cost ($60 for 3.5kg of test dust!)

ISO 12103-1, A3 MEDIUM TEST DUST

ISO 12103-1, A3 MEDIUM TEST DUST is used in many applications such as filter efficiency, fuel filter testing, hydraulic filter testing, etc. Click on the following link to see a list of standards and specifications that use ISO 12103-1, A3 Medium Test Dust. Filtration Standards & Specifications.

Click on the following link for chemical composition listed on the material safety data sheet. SDS

ISO 12103-1 ARIZONA TEST DUST CONTAMINANTS A3 MEDIUM GRADES

Iso Test Dust Particle Size Distributions by Volume %

Size Micrometer	*ISO 12103-1, A3 Medium Test Dust % Less Than*
0.97	2.0 – 2.4
1.38	3.8 – 4.4
2.75	10.3 – 11.1
5.50	22.1 – 23.2
11.00	42.3 – 43.6
22.00	62.5 – 64.5
44.00	82.0 – 83.5
88.00	94.7 – 96.0
124.50	97.2 – 98.6
176.00	99.0 – 100

Available in 1 Gallon Jars (3.5kg Net Weight)

Setting IVL

Internal Variance Limits (IVL)
Maximum allowable differences (expressed as percentages) in particle counts between runs

$$Dq = \frac{X(max) - X(min)}{X(ave)}$$

NOTE:

- IVL settings are initially set to the values stated in ISO 11171 and ASTM D7647
- Can be changed in the software, however, can only be set lower.

ISO 11171:2020 – Maximum Allowable IVL

If the mean number of particles counted is		Use these Values for the maximum allowable Dq for an individual sample
Greater than or equal to	but less than	
10000		11.0
5000	10000	11.3
2000	5000	11.9
1000	2000	13.4
500	1000	15.6
200	500	19.3
100	200	27.5

D7647 – 10 (2018)

TABLE A2.1 Maximum Allowable Percent Differences in Particle Counts Between Runs

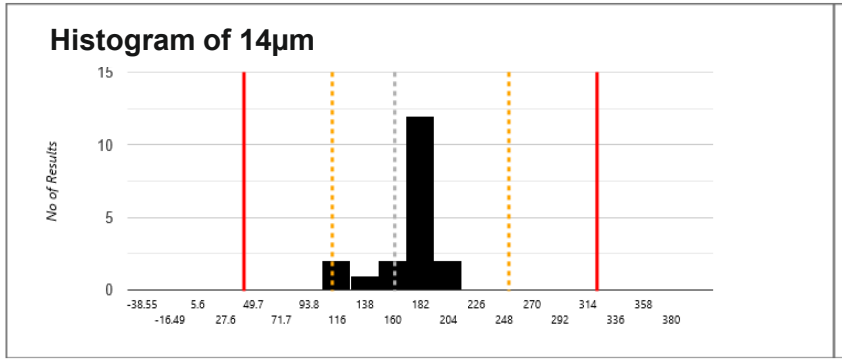
Average Number of Total Particles Counted per Run, $\bar{C}$	Maximum Allowable Percent Difference
$10\,000 \leq \bar{C} < \infty$	11.0
$5000 \leq \bar{C} < 10\,000$	11.3
$2000 \leq \bar{C} < 5000$	11.9
$1000 \leq \bar{C} < 2000$	13.4
$500 \leq \bar{C} < 1000$	15.6
$200 \leq \bar{C} < 500$	19.3
$100 \leq \bar{C} < 200$	27.5
$50 \leq \bar{C} < 100$	37.4
$20 \leq \bar{C} < 50$	51.8

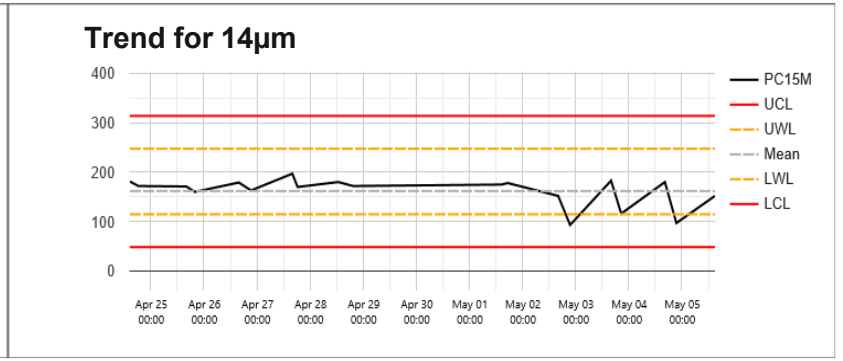
Process Control Standards (PCS)

- PCS should be made with test dust (increased counts)
- PCS at beginning of run, PCS at end of run
- Name sample PCS{…} automatically apply the control limits to the run
- Set UCL/LCL for PCS based on ASTM D7647 repeatability specification.

Process Control Statistics – CS-APC-2 Particle Counter PCS

	>4µm(c) 2µm	>6µm(c) 5µm	>14µm(c) 15µm
Cert Val	5,239	2,274	182
+/- Precision %	30.0	30.0	73.0
X̄ (Mean)	5,819	2,469	163
σ (Std Dev)	474	274	28.9
UCL	6,810	2,957	315
UWL	6,025	2,615	248
LWL	4,453	1,933	116
LCL	3,667	1,592	49.1
n	19	19	19
Conforming	19	19	19
Outside Limit	0	0	0
Compliance	100.0	100.0	100.0





ASTM D7647-10(2018) Precision Statement

TABLE 1 Temporary Precision with 95 % Confidence

Parameter	Units	Repeatability 2.77 × std dev	Reproducibility 2.77 × std dev	Data Range Low	Data Range High
Counts ≥4 µm (c)	counts/mL	30 %	113 %	150	110 000
Counts ≥6 µm (c)	counts/mL	30 %	76 %	60	60 000
Counts ≥14 µm (c)	counts/mL	73 %	135 %	6	22 000
ISO ≥4 µm (c)	code value	<1	1.7	14	24
ISO ≥6 µm (c)	code value	<1	1.2	13	22
ISO ≥14 µm (c)	code value	1.5	2	10	18

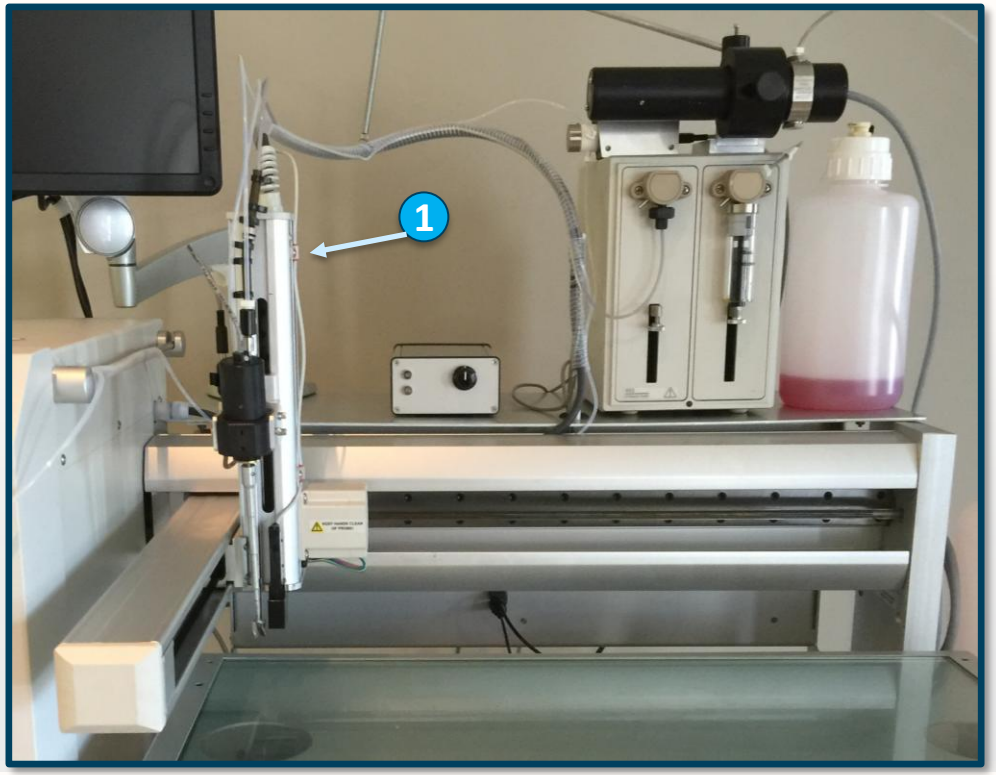
Maintenance

- Maintenance Tips
- Flanging Kit for System Tubing
- Calibration Tips

Possible Flow Restriction Sites
In-line Filter

1. **In-line 200µm filter**
   - Remove the filter
   - Open the filter at the o-ring
   - Remove the fine metal mesh screen and soak in toluene for 30 minutes
   - Rinse the screen with your system solvent, and reinstall

2. **Replacement Filter**
   - Filter can be purchased from any RCA hobby shop.
   - GreatPlanes.com P/N GPMQ4150



NOTE: *Refer to page 42 of the CS-APC-2 System User Manual.*

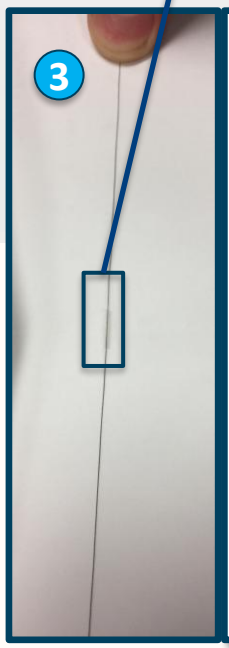
Possible Flow Restriction Sites
Particle Count Sensor

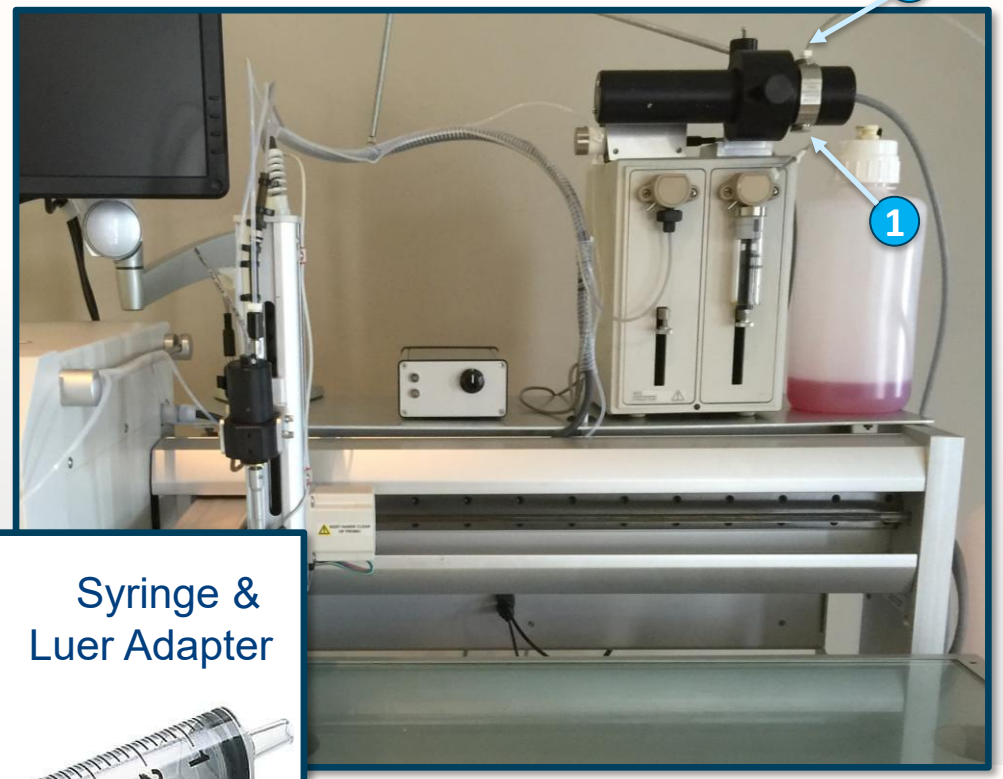
1 Sensor Inlet 2 Sensor Outlet

- Disconnect the sensor inlet/outlet lines (tubing).
- Use an ultra-fine lab brush (3) to remove any solids present in the sensor cell.
- Connect a syringe assembly filled with CitraJet (see 4) to the sensor outlet (2) NOTE: heated CitraJet works better.
- Place a waste beaker below the inlet side of the sensor (1)
- Push solvent into the sensor and let sit for 30 minutes to break-up any varnish/fibres
- Flush the CitraJet back and forth thru the sensor.
- Re-assemble the sensor tubing, and test.

4 Syringe Assembly

- Ultra-fine lab brush (100mm x 2mm)
- Clear disposable syringe
- Luer adpater (1/4-28)
- CitraJet is supplied by ALCONOX and comes in 1L plastic containers.





NOTE: *Refer to page 43 of the CS-APC-2 System User Manual.*

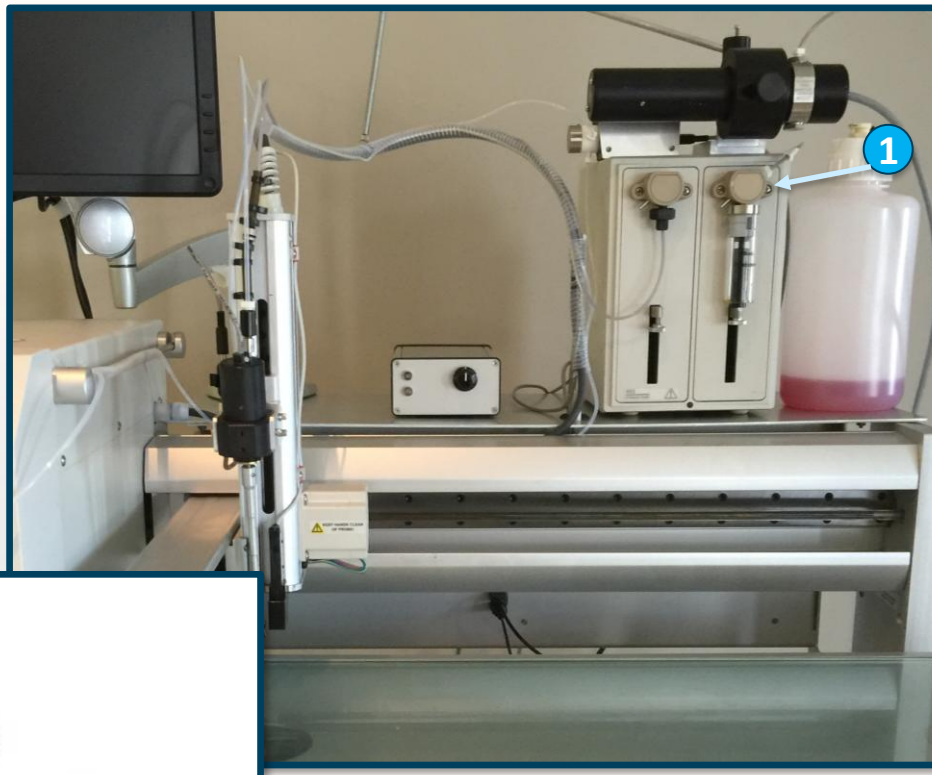
Possible Flow Restriction Sites
Syringe Valve

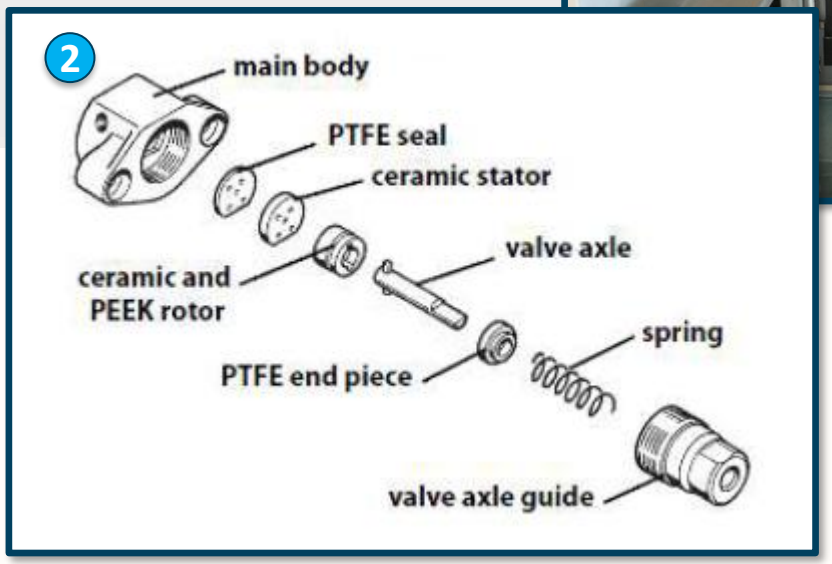
1 Syringe Valve

- Remove the syringe valve by unfastening the two screws holding the valve to the syringe pump cover.
- Disassemble the syringe and clean the internal components. If there is varnish build-up then these components may need to be soaked in Toluene for 1-2 hours.
- Reassemble the valve and install onto the syringe pump.
- Test the system.

2 Syringe Valve Assembly

- Refer to page 45 of the CS-APC-2 System User Manual.





NOTE: *Refer to page 45 of the CS-APC-2 System User Manual.*

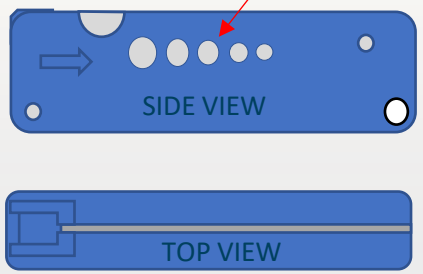
APC Flanging Tool and Tubing Kit

KIT CONTENTS

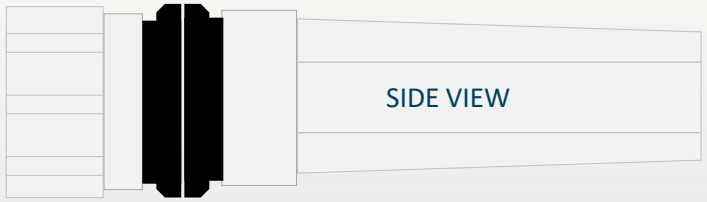
1. Easy-Flange Kit – Low Pressure.
2. PTFE tubing – 1/8” OD x 0.085” ID (25ft).
3. IDEX Tube Nuts and washers (18).
4. Viton Tube 1/4” OD 1/8” ID – 4cm (2).
5. Tube Clamps for 1/4“ tube (6).

Items from flanging kit needed to cutting tubing and creating a flange on 1/8” Od x 0.085” ID PTFE tubing.

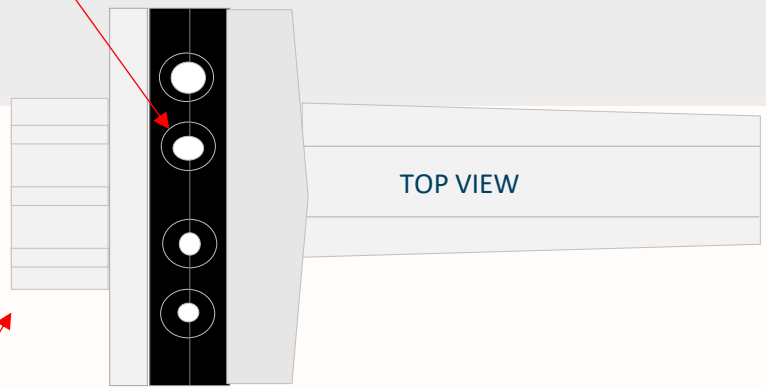
Use these holes are for 1/8” OD x 0.085” ID PTFE tube



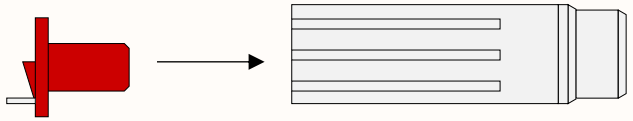
Tube Cutter



Tubing Vice



Flange
forming Insert

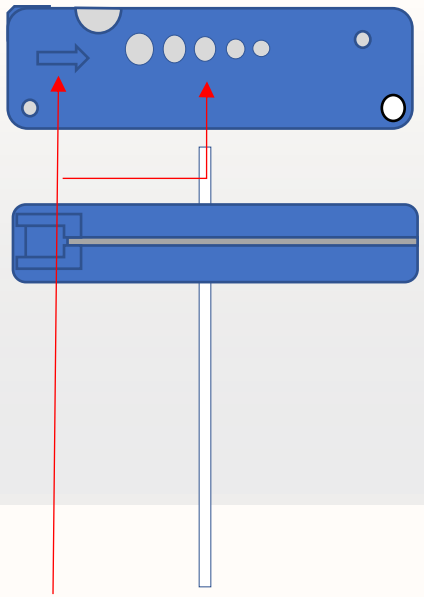


Insert Handle

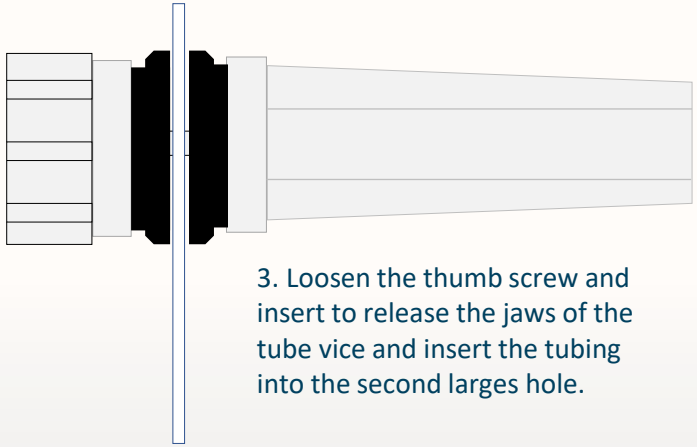
Insert with Metal Pin is used for
1/8” OD x 0.085” ID PTFE tubing

Thumb Screw

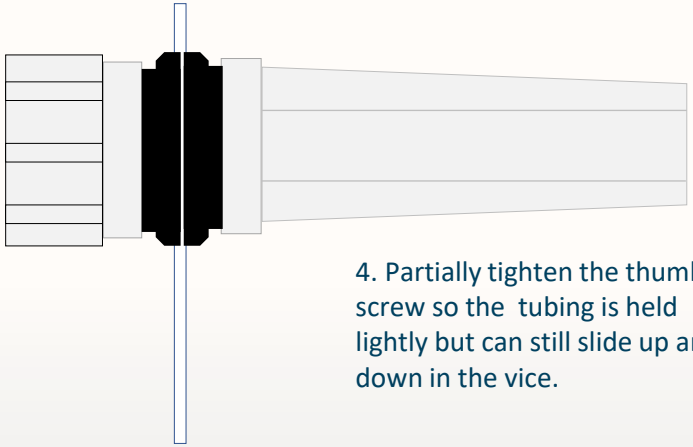
Flange Forming Process



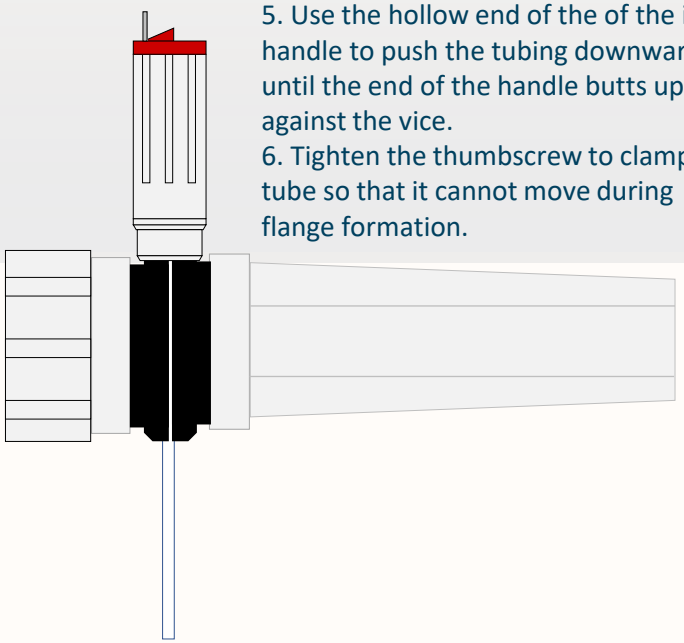
1. With the cutting blade raised, inset 1/8 tube into third hole from the side marked with the arrow.
2. Press the blade down to create a square cut on the end of the tubing



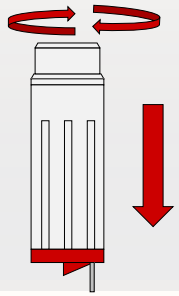
3. Loosen the thumb screw and insert to release the jaws of the tube vice and insert the tubing into the second larges hole.



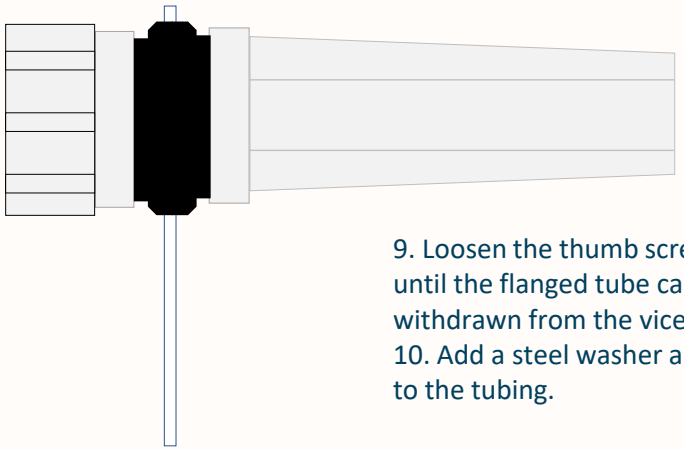
4. Partially tighten the thumb screw so the tubing is held lightly but can still slide up and down in the vice.



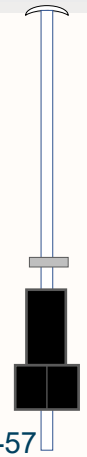
5. Use the hollow end of the of the inset handle to push the tubing downwards until the end of the handle butts up against the vice.
6. Tighten the thumbscrew to clamp the tube so that it cannot move during flange formation.



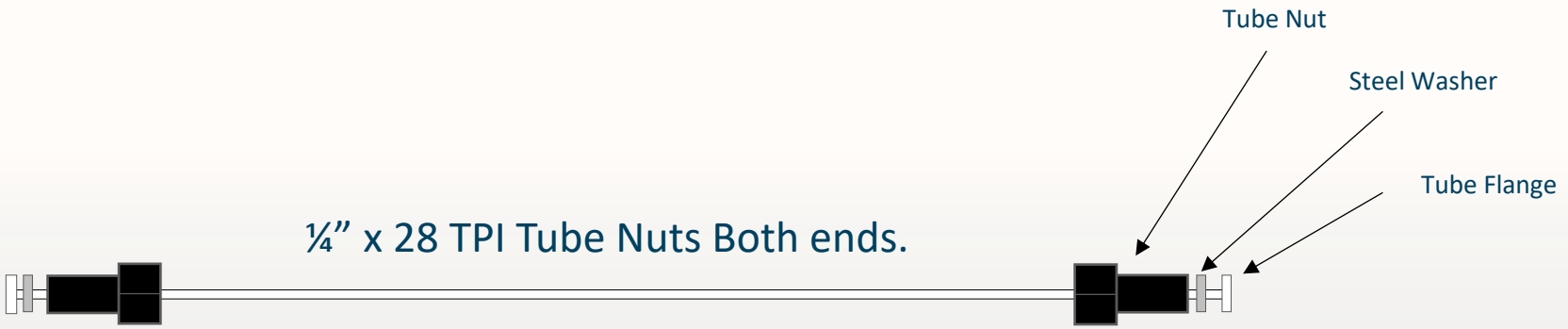
7. Insert the metal pin of the flange forming insert into the inside diameter of the tubing.
8. While pushing firmly down on the flange forming tool, rotate the handle several times about the pin to create the flange.



9. Loosen the thumb screw of the vice until the flanged tube can be freely withdrawn from the vice.
10. Add a steel washer and a tube nut to the tubing.



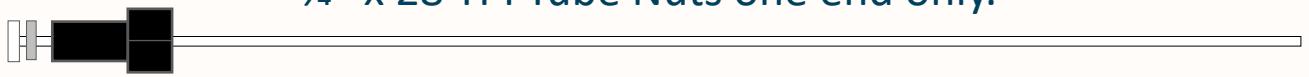
Delivery Tubes for CINRG CS-APC-2.



Delivery Tube Description	Length “L”	Quantity
Sample Syringe Valve to laser Sensor Inlet.	7.1cm (71mm)	1
Solvent Syringe Vale Port to 24V Solenoid Valve Inlet (Bottom Port).	128cm	1
Front 24V Solenoid Valve Port to Sampling head (Fine jet Solvent Needle).	92cm	1
Back 24V Solenoid Valve Port to sampling Head Block.	130cm	1
Cup Calibration Tube.	90cm	1

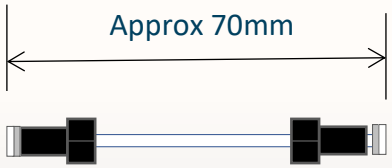
Delivery Tubes for CINRG CS-APC-2.

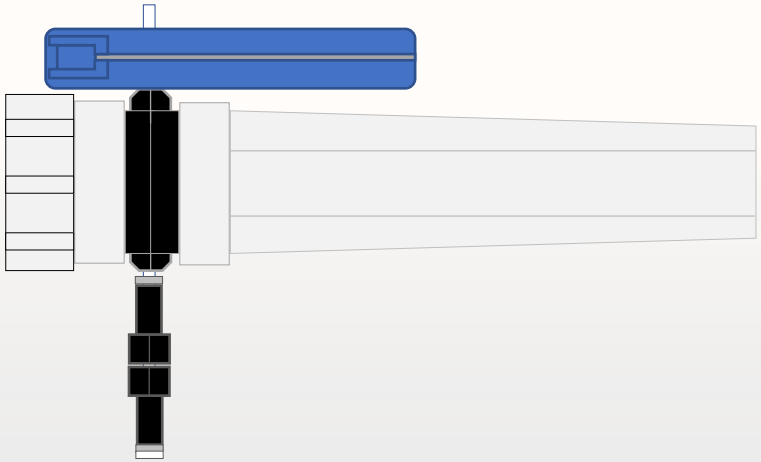
¼” x 28 TPI Tube Nuts one end only.

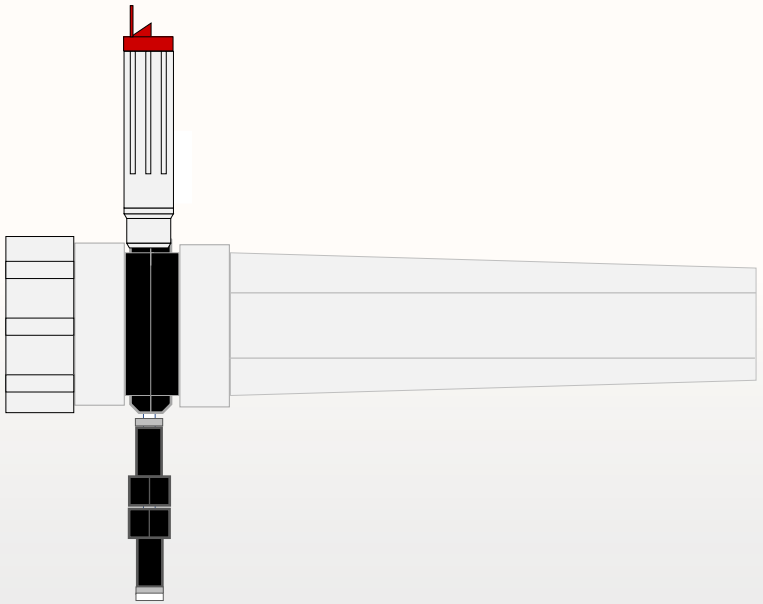


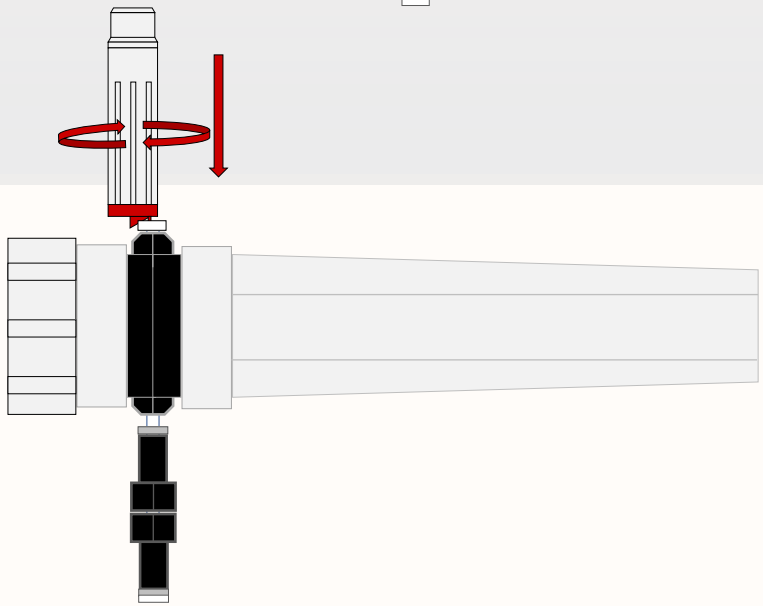
Delivery Tube Description	Length “L”	Quantity
Sample needle to inline filter.	7cm	1
Sample Syringe to in-line filter.	100cm	1
Solvent Syringe to solvent reservoir.	122cm*	1
Laser Sensor Outlet to waste collection receiver.	122cm*	1
Sensor Calibration Tubing.	75cm	2
Cup Calibration Tube - Solvent syringe to sample bottle (Oil Reservoir).	65cm	1

* These tubes may need to be longer if the solvent reservoir and waste receiver are further from the APC_2.



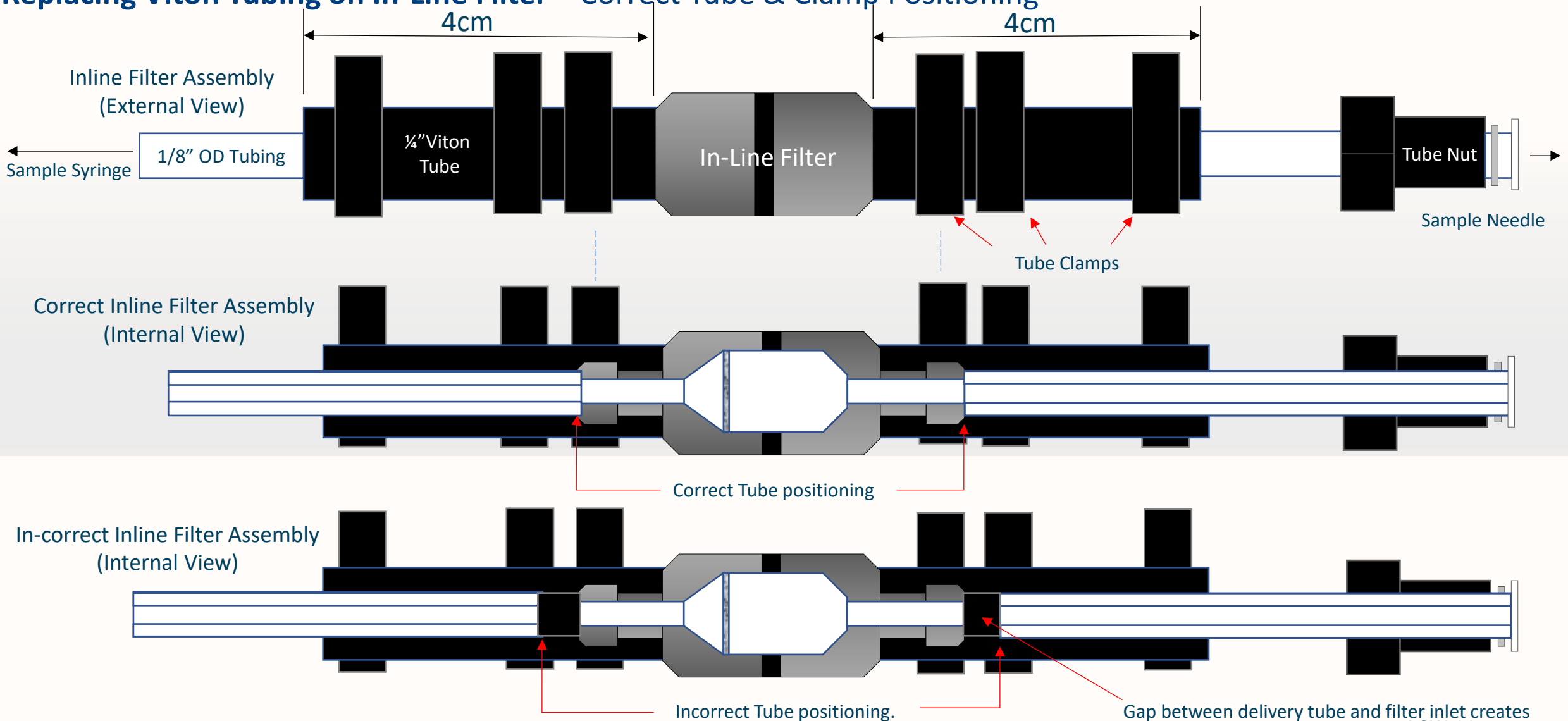






1. Cut an approximately 10cm length of tubing using the tube cutter.
2. Create a flange on one end. Insert a washer and a tube nut followed by a second tube nut and washer so the tube nuts are positioned “Back-to- Back” on the tube.
3. Insert the tube into the tubing vice as far as it will go and after clamping it firmly, insert the tube cutter onto the tube and slide it down as far as it will go before cutting the tube (Diagram B).
4. Loosen the vice thumb screw a little to free up the tube and use the hollow end of the flanging tool handle to push the tubing down to the required height (Diagram B).
5. Retighten the thumb screw to prevent the tube from moving and create the flange on the other end of the tube to complete the tubing (Diagram C).
6. This process will create a delivery tube od approximately 70mm in length with a flange, washer and tubing nut on both ends.

Replacing Viton Tubing on In-Line Filter – Correct Tube & Clamp Positioning



CINRG Systems Inc.

Innovation in Automation

OilDoc 2023 – Particle Counting Workshop