# Cleaning and Analysis of Flight Hardware with an Emphasis in Particle Contamination

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### **Product Cleanliness Levels**

The aerospace industry standard for cleanliness is defined in IEST-STD-CC1246E, "Product Cleanliness Levels – Applications, Requirements, & Determination."

For particulates, this analysis comes down to a logarithmic equation that was modified to its current form by Ma. P.T., M.C. Fong, and A.L. Lee and published in 1989.<sup>1</sup> The size, quantity, and general particle shape are extrapolated to provide an overall area of obscuration into a single cleanliness level value.

This paper reviews the methodology of collecting the particle counts via optical microscopy and the impact of the setup on the actual counts. This analysis may then be applied to improving counting accuracy and system correlation.

<sup>1</sup> P.T. Ma, M.C. Fong, and A.L. Lee, "Surface Particle Obscuration and BRDF Predictions. Proceedings of the Conference on Scatter from Optical Components" 1165:381-391. J.C. Stover, Ed. The International Society for Optical Engineering (SPIE).



SDL Certificate of Analysis – Particle and NVR



### **MIL-STD-1246: Product Cleanliness Levels & Contamination Control**

Since the particle distribution is linear on a log – log<sup>2</sup> scale, particle size distribution may be denoted by the log-normal slope and an intercepting value. MIL-STD-1246B set the abscissa intercept (X coordinate) as the parameter to define the cleanliness level with a standard slope of -0.926.

This cleanliness level is interpreted as the largest particle within the distribution of the abscissa intercept.

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Log n = log n_0 + S Log^2L
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Where, n = \# \text{ particles } / \text{ ft}^2 \text{ larger than size L}

n_0 = \# \text{ particles } / \text{ ft}^2 \text{ larger } > 1 \ \mu\text{m}

L = Largest linear particle dimension (mm).

S= log-log<sup>2</sup> slope, defined as \frac{\log n_1 - \log n_2}{\log^2 L_1 - \log^2 L_2}
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<sup>2</sup> IESTsSTD-CC1246 Rev A & B– Product Cleanliness Levels – Applications, Requirements, and Determination



Research shows that naturally occurring particulate contamination follows a log-normal distribution with a geometric mean of neur one (1) micron particle. This distribution follows a straight line when plotted on a log x log<sup>2</sup> scale graph. The grid is derived from the log-normal (1) Gaussian distribution function which provides a close fit for real contamination data. The lines on the chart represent the maximum contamination permitted for each level on the plot point is the number of particles abave given size versus particle size. The curves can be expressed as  $\log n = 0.9260$  ( $\log^2 X_1 - \log^2 X_1$ ), where n is the number of particles, 0.9260 is the tangent of the angle. X is the particle size, and X<sub>1</sub> is the closeliness level.

## Surface Particle Obstruction & BRDF Predictions - P.T. Ma, M.C. Fong, & A.L. Lee

The relationship of particle aspect ratio and particle size was investigated by Ma, Fong, and Lee based on a study by Rabb<sup>3</sup>. This led to the proposal for a particle geometry model where particles may be characterized by a cylindrical hemispherical shape. Small particles will tend to be spherical, while larger particles are more ellipsoidal as denoted in the following equation.

```
A = D (L-D) +\pi D<sup>2</sup> / 4
A = (AR<sup>-1</sup> + AR<sup>-2</sup> (\pi /4 - 1)) L<sup>2</sup> Where aspect ratio, AR = L/D
```



With this information, Ma et al. incorporated particle geometry to develop their obscuration ratio by integrating the particle coverage area with respect to particle size. By defining the particle as cylindrically hemispherical from a natural order viewpoint, obscuration values are 32% to 19% lower than if the particle was assumed to be just spherical or cylindrical. Assuming that the particles are randomly orientated rather than in a prone position further reduces the obscuration value of a cylindrically hemispherical-sized particle calculation by 7%.

Log (%Obs) = -7.245 + 0.926 log <sup>2</sup> (Level)	Sphere	(17a)
-7.289 + 0.926 log <sup>2</sup> (Level)	Cylinder	(17b)
-7.364 + 0.926 log <sup>2</sup> (Level)	Cylindrically Hemispherical	(17c) <mark>Equation used at SDI</mark>
-7.394 + 0.926 log <sup>2</sup> (Level)	Cylindrically Hemispherical (Randomly Oriented)	(17d)
-8.140 + 0.926 log <sup>2</sup> (Level)	10:1 Fiber	(17e)

<sup>3</sup> J.H. Rabb's paper, "Particulate Contamination Effects on Solar Cell Performance" (Martin Marietta Aerospace Report MCR -86 -2005, January 1986)



## **IEST-STD-CC1246E – Product Cleanliness Levels**

MIL-STD-1246 went through five revisions before the Army commissioned IEST to adapt the standard for industry use in 1997. Five additional revisions have occurred as an IEST document, with the latest being revision E in 2013. The document provides methods for specifying product cleanliness levels with respect to particles and nonvolatile residue.

Percent area coverage (PAC) is calculated by using the Table B1 in Annex B. The "Times Conversion Factors" are based off statistical studies of particle shapes from the 1980s.

		IEST-STD-CC1246E Level (Maximum Particle Count)								
min (µm)	max (µm)	25	50	100	200	300	400	500	750	1000
5	15	19	141	1519						
15	25	2	17	186	2949					
25	50	1	6	67	1069	6433				
50	100	0	1	9	154	926	3583	10716		
100	250	0	0	1	15 (Note 3)	92	359	1073	8704	
250	500	0	0	0	0	2 (Note 3)	8 (Note 3)	25	205	983
500	750	0	0	0	0	0	0	1	7	33
750	1000	0	0	0	0	0	0	0	1	3
1000	1250	0	0	0	0	0	0	0	0	1

#### IEST-STD-CC1246 - Table 1 Particle Cleanliness Levels<sup>4</sup>

Note 1. Sampling areas other than 0.1 m<sup>2</sup> shall be calculated to the basis of 0.1 m<sup>2</sup>. Areas may be estimated if total area is considered by all parties in a multi-party agreement to be too difficult to measure within two significant figures. This condition shall be noted and low/high ranges shall be used. Parts with a total significant surface area less than 0.1 m<sup>2</sup> and which have had the entire critical surface area sampled shall be accepted on the basis of actual count. For small parts cleaned by a batch process, a quantity of cleaned items from that batch sufficient to make up 0.1 m<sup>2</sup> of surface area may be sampled to characterize that batch.

Note 2. Size bins that are blank in the table are not required to be counted.

Note 3. Under no circumstances is more than one particle larger than the level designation allowed per 0.1 m<sup>2</sup> of surface area.

Note 4. Sampling volumes other than 0.1 L shall be calculated to the basis of 0.1 L. Volumes may be estimated if the total volume is considered by parties in a multi-party agreement to be too difficult to measure within two significant figures. This condition shall be noted and low/high ranges shall be used. Fluids with a total significant volume less than 0.1 L and which have had the entire critical volume sampled shall be accepted on the basis of actual count.

Range of Consered Particle Sizes	Surface Concentration in Particles / 0.1 m <sup>2</sup>	Multiplcation Factor for bins not counted*	Times (x) Conversion Factor*	Equals effective % area coverage
5 -<15		8.15	4.1780E-08	
15 -<25		2.76	2.0880E-07	
25 -<50		6.95	6.2500E-07	
50 -<100			2.1920E-06	
100-<250			5.5160E-06	
250 -<500			6.4300E-06	
500 -<750			1.0130E-05	
750-<1000			1.5320E-05	
1000 -<1250			2.0590E-05	
	Sum	all values to obtain %	6 Obscuration =	

#### IEST-STD-CC1246 - Annex B, Table B1<sup>4</sup>

<sup>4</sup> IESTsSTD-CC1246 – Product Cleanliness Levels – Applications, Requirements, and Determination



# **Microscopy Correlation – Initial Tests between Olympus and Keyence**

SDL currently conducts IEST Standard CC1246E Cleanliness Microscopy Analysis of 100 to 500 cleanliness levels, with particle counts down to 0.3µ, using an Olympus BX51 digital microscope.

Calibration at 5X:	
Calibration at 50X:	
Total test time:	

0.53801 μm/pixel 0.05268 μm/pixel ~2.5 hours

#### Sample 1 Correlation: Thermal Strap 210104

Calibration at 80X: Total test time:

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0.962 μm/pixel ~30 minutes

- SDL Olympus BX51 Microscope and the Keyence 7000 correlate well from 6 to 200  $\mu m$
- SDL method is comprised of five 50X measurements (< 1% of the filter surface) and two 5X measurements (< 39% of the filter surface)
- Keyence trial was conducted at 80X and measured the entire filter area

	Olympu	is BX51:	Keyence VHX7000			
Magnification	5X	50X	100X	2500X		
Particle Size, $\mu$	10 to 550	0.3 to 10	5 to 550	0.3 to 10		
Calibration (um/pixel)	0.53801	0.05268	1.0330	0.0417		
Array Area, mm <sup>2</sup>	192.31	1.85	176.76	12.57		
Test Time (min):	150		30	30+		



#### Trial 1: Filter Membrane Correlation – Olympus & Keyence at 50X

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## **Microscopy Correlation (continued)**

#### Sample 2 Correlation: MISE 299-0072 200810

Calibration at 500X: 0.20

 $0.205 \,\mu\text{m/pixel}$ 

- Evaluated a 500X sample using a MISE sample to improve the resolution to the 0.3  $\mu m$  level
  - Sample area was 7.25mm<sup>2</sup> as compared to the fivesample area of 10.71 mm<sup>2</sup> of the Olympus measurement
- 80X Keyence sample matched well for data > 20  $\mu m$
- Olympus data set resulted in a higher at 0.3  $\mu m$  count, thus a steeper curve when compared to Keyence

In the spring of 2021, SDL purchased a Keyence VHX 7000 for use in optical inspection and particle microscopy. Projects with this system include:

Optical inspection of lenses

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- Microscopy Work Instruction 100X, 5μ analysis
- Microscopy analysis Cleaning optimization DOE
- Accessory Development filter holder, lens holder, light diffusor, and fallout plate holders



Trial 2: Filter Membrane Correlation – Olympus & Keyence at 500X

## **Particle Count Microscopy Optimization**

Key Microscopy settings include:

- 1) Filter Mounting: Optimizes visual scan area
- 2) Lighting: Full ring with transmitted lighting at 100% power
- 3) Magnification: 100X\*; Keyence 80X Bin sizing is locked in for VDA 19 automotive standards at  $5\mu$  for the smallest bin
- 4) Image Focus: Initial trials seemed to do better using Auto Focus; considering the new data, will switch to Depth Composition
- 5) Auto Area Data File: Set by engineer for optimal and standardized counting

#### Keyence VHX7000 Optics Parameters

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	20X	30X	40X	50X	80X	100X	500X	700X	1000X	1500X	2000X	2500X
Field of View (mm)	15	10	7.5	6	3.75	3	0.6	0.428	0.3	0.2	0.15	0.12
Pixel Size (µm)	5.2	3.5	2.6	2.1	1.3	1.04	0.208	0.149	0.104	0.069	0.052	0.0417
Repeatability (±µm)	15.6	10.5	7.8	6.3	3.9	3.12	0.624	0.447	0.312	0.207	0.156	0.1251
Max. Stitch Length (mm)	260	174	130	104	65	52.08	10.417	7.431	5.208	3.472	2.604	2.083

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#### Depth Composition and Auto Focus at Medium Range for select magnifications

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### **Image Focus**

### **Auto Focus:**

Finds the planar image focus by adjusting the z height for a given range and selecting the best focus that provides the sharpest contrast.





Keyence Example of Auto Focus

### **Depth Composition:**

Multiple pictures are taken at different Z heights. Each image is focused then combined to form a single image.











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### **Auto Area File**

Particle / filter membrane differentiation is based on a contrast value that may be set manually each time or pulled from a previous setting. Adjustments are made to the dark field setting such that only particles are highlighted. As this is subjective, the setting is saved for standardized use for future tests.



Keyence Extraction Area Settings



### **Product Cleanliness Levels**

A cleaning optimization test was conducted and evaluated for particle removal efficiency via microscopy. Each data point is the average of four samples for the various test conditions.



Sample Information	Blank	As Rec	Standard Process	Valtech SP2600	MicroCare Tergo
Total Count Avg $\geq 5~\mu m$ / 1,017 mm^2	26	165,899	105,775	121,737	35,651
Total Sample Obscuration (cm <sup>2</sup> ):	0.000	0.291	0.093	0.242	0.048
Percent Obscuration of Total Surface Area (%):	7.0731E-07	3.20E-03	1.02E-03	2.65E-03	5.25E-04
Derived Cleanliness Level $\geq$ 5 µm:	71	519	412	500	359

# **Cleaning Solution:** Valtech SP2600 aqueous detergent and MicroCare Tergo solvent

### Ultrasonics: Crest ultrasonics 40 and 132 kHz



Sample Information	Blank	As Rec	Standard Process	Valtech SP2600	MicroCare Tergo
Total Count Avg ≥ 5 μm / 178 mm^2	214	89,117	85,457	87,692	61,745
Total Sample Obscuration (cm <sup>2</sup> ):	0.000	0.197	0.072	0.085	0.055
Percent Obscuration of Total Surface Area (%):	6.1552E-08	3.2243E-05	1.1726E-05	1.3837E-05	9.0268E-06
Derived Cleanliness Level $\geq 5 \ \mu m$ :	116	481	391	404	370



Note: The "Standard" process for thermal straps was in reference to a general lab process. This is not the standard cleaning process for thermal straps.

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### **Product Cleanliness Levels Test Results**

Statistically significant difference was observed for the use of the test solvent.

Decoupling of the interaction of the test ultrasonics (Crest 40 / 132 kHz) and this process with SDL's current thermal strap cleaning process will help lead to process improvements.



### **Microscopy Accessories**

Based on a Keyence design, SDL has designed and manufactured several accessories for filter holding, light diffusion, fallout collection, and optics cleaning and inspection.



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### Conclusions

- Ma et al. obscuration equation and its incorporation of particle geometry, remains a critical calculation for component cleanliness
  - Incorporation of Ma et al. equation (17a-e), into IEST-STD-CC1246 for obscuration calculations is recommended
- Today's digital microscopes offer advantages that were not available 10 years ago, specifically the display resolution and the processing speed as well as greater functionality (head inclination, stage rotation, etc.)
- Depth composition is the best solution for image capture
- Use of a filter holder for repeated accurate positioning of samples is important
- Lighting / Auto Area settings are critical and need to be established for each different type of scope
  - Should be reviewed periodically to ensure that optimum particle counting is achieved
- Magnification is only part of the equation
  - Field of view of the Keyence VHX7000 is different from SDL's current Olympus scope which results in the need to use a higher magnification
  - Pixel resolution and repeatability should be factored into the procedure
- Although lower particle counts may seem advantageous, the overall impact with respect to obscuration may be negligible
  - Additional time that may be required at high magnification may not be warranted