LA-950 Repeatability Study

Summary
Study on LA-950 gage-to-gage variation was performed with 4 different samples on 2 random selected units of LA-950 (S/N EVD and HOO). The samples were Whitehouse Standard PS-202 (3-30μm), Duke BCR-66 (1-10μm), a customer's samples A (median 0.7) and B (median 1.2). Variations between sampling-to-sampling, instrument-to-instrument are all below 10%.

Analytical test method

Due to different nature of each sample, we developed different method for each sample as followings.

1. Whitehouse Standard PS-202
Add de-ionized water to the LA-950 flow system, and then activate circulation to level (5) and agitation to level (2). Add the whole bottle of PS-202 into the flow system. Apply 4-5 minutes of internal sonication at level 7 and then take 3 consecutive measurements.

2. BCR-66
Use de-ionized water with 0.1% Sodium Pyrophosphate (NaPP) as dispersant and add about 0.1wt% of BCR-66 in it. Leave BCR-66 solution in water bath with 40 watts of ultra-sonication for about 10 minutes. Add dispersant into LA-950 flow system, and then activate circulation to level (3) and agitation to level (3). Add more BCR solution into the LA-950 flow system if necessary to reach laser transmittance level of 88-90%. Then take 3 consecutive measurements. Repeat the procedure with two additional samplings, each with 3 consecutive measurements.

3. Customer samples
Use de-ionized water with 0.1% Darvan C as dispersant and add about 0.1wt% of customer sample in it. Then put dispersant into LA-950 flow system and activate circulation to level (3) and agitation to level (1). Transfer enough pre-dispersed sample into the LA-950 flow system to reach laser transmittance level of 85-88%. Take 3 consecutive measurements. Repeat the procedure with two additional samplings, each with 3 consecutive measurements.

Example data Summary

One-way ANOVA analysis of the sample measurement results is shown in the Appendix. The size of the y axis shows the spec limits for each sample at its 10%, 50% and 90% percentile level. The mean particle size and standard deviation of each sample measured by the two LA-950 analyzers are shown in each graph. The Student t pair test of the two instruments is shown on the right side of each graph with the circle centered at the mean particle size and the radius of the circle represents 95% confidence level. No overlapping of the two circles indicates that with 95% confidence the particle sizes measured by the two units are different.

To summarize the results of all four samples:

PS-202: Statistically (by Student t test) there are significant differences in particle size measured by the two units at all three percentile level (10%, 50% and 90%). However, all the measurements fall well within the sample and tool spec limits (the tolerance level).
BCR 66: Statistically (by Student t test) there is no significant difference in particle size measured by the two units at all three percentile level (10%, 50% and 90%). Also, all the measurements fall within the sample and tool spec limits (the tolerance level).
Samples #1 and #2: Statistically (by Student t test) there are significant differences in particle size measured by the two units at 10% and 50% percentile level but no difference at 90% level. However, all the measurements fall well within the sample and tool spec limits (the tolerance level).

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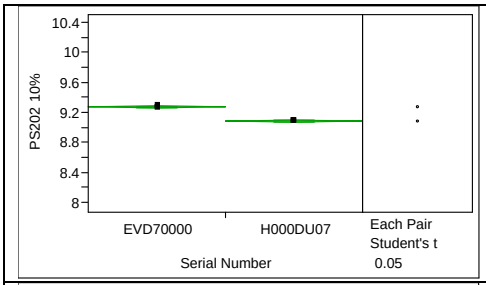
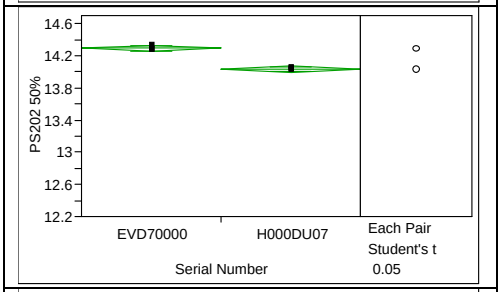
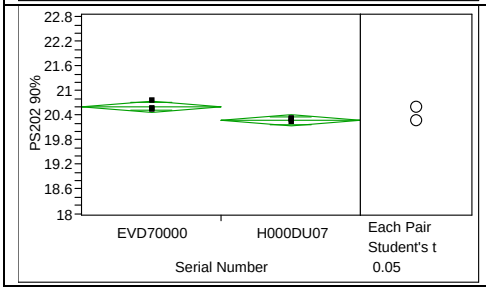
Appendix 1. Analysis of Variance (ANOVA)

Oneway Analysis of PS-202 By Serial Number





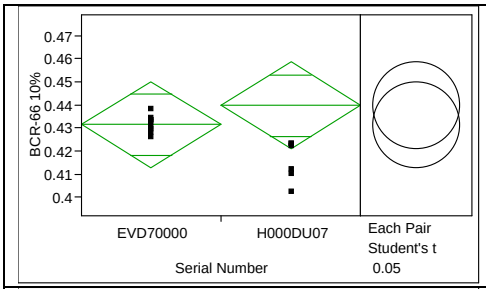


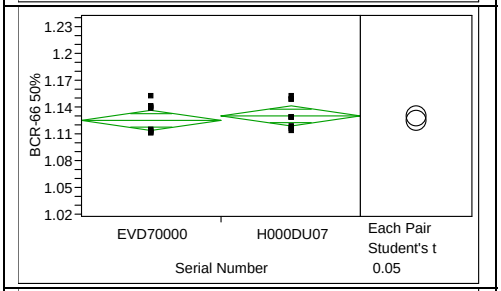
Summary of Fit	10%	50%	90%
Rsquare	0.9953	0.9745	0.8563
Adj Rsquare	0.9941	0.9681	0.8203
Root Mean Square Error	0.0079	0.0255	0.0844
Mean of Response	9.1762	14.1618	20.4358
Observations	6	6	6

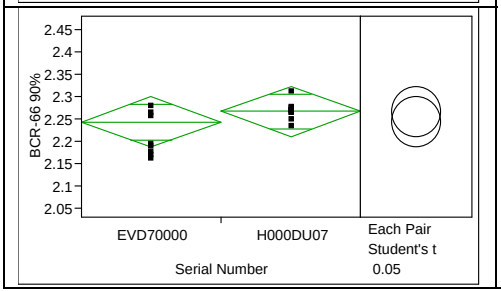
t Test	10%	50%	90%
Difference	-0.1863	-0.2577	-0.3363
Std Err Dif	0.0064	0.0208	0.0689
Upper CL Dif	-0.1685	-0.1998	-0.1450
Lower CL Dif	-0.2041	-0.3155	-0.5276
Confidence	0.95	0.95	0.95
t Ratio	-29.06	-12.36	-4.88
DF	4	4	4
Prob > |t|	<.0001	0.0002	0.0082

Tool Difference	10%	50%	90%
EVD70000 Mean	9.2693	14.2907	20.6040
H000DU07 Mean	9.0830	14.0330	20.2677
Grand Mean	9.1762	14.1618	20.4358
Difference of Tools	0.1863	0.2577	0.3363
% Difference	2.03%	1.82%	1.65%
Tolerance Level	±10%		

Oneway Analysis of BCR-66 By Serial Number





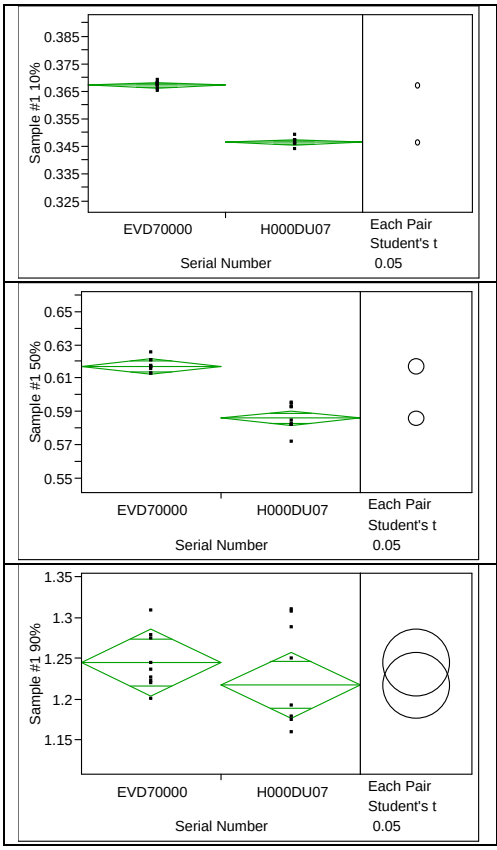


Summary of Fit	10%	50%	90%
Rsquare	0.02698	0.03032	0.02330
Adj Rsquare	-0.03384	-0.03029	-0.03775
Root Mean Square Error	0.02654	0.01566	0.07936
Mean of Response	0.4356	1.1276	2.2553
Observations	18	18	18

t Test	10%	50%	90%
Difference	0.00833	0.00522	0.02311
Std Err Dif	0.01251	0.00738	0.03741
Upper CL Dif	0.03486	0.02088	0.1024
Lower CL Dif	-0.01819	-0.01043	-0.05619
Confidence	0.95	0.95	0.95
t Ratio	0.6660	0.7072	0.6178
DF	16	16	16
Prob > |t|	0.5149	0.4896	0.5454

Tool Difference	10%	50%	90%
EVD70000	0.4314	1.1250	2.2438
H000DU07	0.4398	1.1302	2.2669
Grand Mean	0.4356	1.1276	2.2553
Difference of Tools	-0.00833	-0.00522	-0.02311
% Difference	-1.91%	-0.46%	-1.02%
Tolerance Level	±10%		

Oneway Analysis of Sample #1 By Serial Number

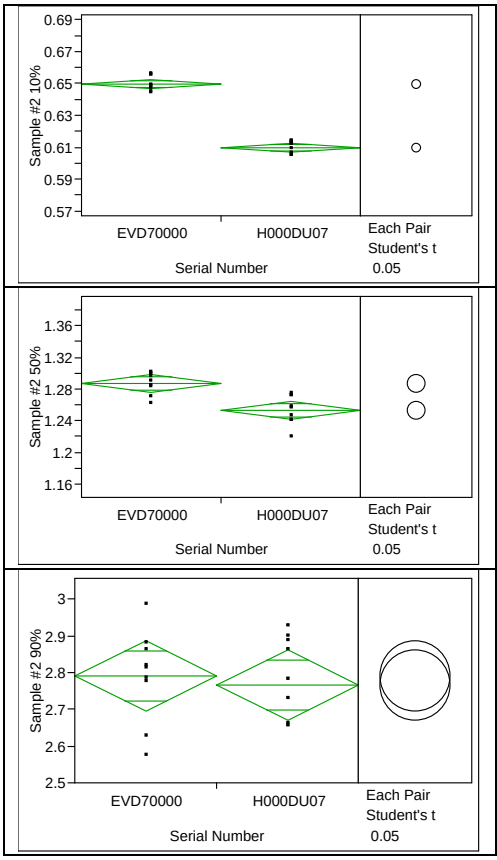


Summary of Fit	10%	50%	90%
Rsquare	0.9869	0.8710	0.0616
Adj Rsquare	0.9861	0.8630	0.0030
Root Mean Square Error	0.0013	0.0064	0.0575
Mean of Response	0.3568	0.6014	1.2309
Observations	18	18	18

t Test	10%	50%	90%
Difference	-0.0208	-0.0312	-0.0278
Std Err Dif	0.0006	0.0030	0.0271
Upper CL Dif	-0.0195	-0.0249	0.0297
Lower CL Dif	-0.0221	-0.0376	-0.0852
Confidence	0.95	0.95	0.95
t Ratio	-34.73	-10.39	-1.025
DF	16	16	16
Prob > |t|	<.0001	<.0001	0.3207

Tool Difference	10%	50%	90%
EVD70000	0.3672	0.6170	1.2448
H000DU07	0.3464	0.5858	1.2170
Grand Mean	0.3568	0.6014	1.2309
Difference of Tools	0.0208	0.0312	0.0278
% Difference	5.82%	5.19%	2.26%
Tolerance Level	±10%		

Oneway Analysis of Sample #2 By Serial Number



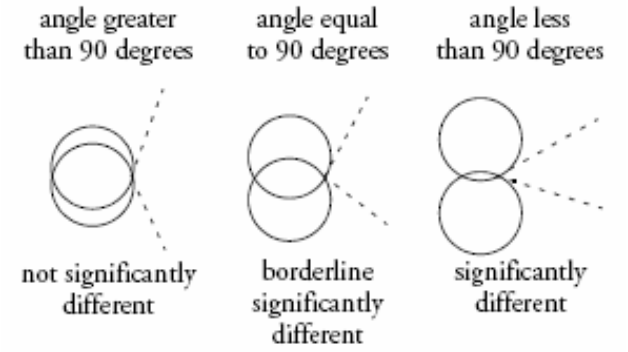
Summary of Fit	10%	50%	90%
Rsquare	0.9631	0.5585	0.00951
Adj Rsquare	0.9608	0.5309	-0.0524
Root Mean Square Error	0.00413	0.01608	0.1353
Mean of Response	0.6298	1.2699	2.7781
Observations	18	18	18

t Test	10%	50%	90%
Difference	-0.03978	-0.03411	-0.025
Std Err Dif	0.00195	0.00758	0.06379
Upper CL Dif	-0.03565	-0.01804	0.11022
Lower CL Dif	-0.0439	-0.05018	-0.16022
Confidence	0.95	0.95	0.95
t Ratio	-20.43	-4.50	-0.3919
DF	16	16	16
Prob > |t|	<.0001	0.0004	0.7003

Tool Difference	10%	50%	90%
EVD70000	0.6497	1.2870	2.7906
H000DU07	0.6099	1.2529	2.7656
Grand Mean	0.6298	1.2699	2.7781
Difference of Tools	0.0398	0.0341	0.0250
% Difference	6.32%	2.69%	0.90%
Tolerance Level	±10%		

Appendix 2. Quick Review of Analysis of Variance (ANOVA)

Take “Sample #2 90%” anova diagram for example, it shows the particle size distribution (@90%) of two levels (instruments EVD70000 and H00DU07) – the black dots. The horizontal width of the diamond is proportional to sample size in each instrument while the vertical width represents the 95% confident interval. Since there is same number of sampling points on each tool, there is no difference between the two diamonds. The middle line across the diamond represents the arithmetic mean of the particle sizes on each tool. ANOVA is to test whether the group means are significantly different at certain confidence level (usually 95%) by examining the ratio between mean squared sum of treatment (differences between group means and grand mean) and mean squared sum of errors (differences between group samples and group means)



The diameters of the circles are the same as the vertical widths of the diamonds, i.e., the 95% confidence intervals. As shown above, whether the two instruments are significantly different depends on the separation of the two circles, which is measured by the angle formed by the tangent lines at the place where the two circles intersect. If the angle is greater than 90 degrees, the two instruments have no significant difference; If the angle is less than 90 degrees, the two instruments are significantly different.