CROWN INDUSTRIAL SERVICES INC. **CLEANLINESS TESTING LABORATORY**

We test to ISO 16232

Component Cleanliness Testing:

'The good, the bad, and the ugly' regarding Extraction Validation aka Reduction Curves

In a previous article in our "Component Cleanliness Testing" series Jared Friedman explained the basics of Extraction Validation testing. If you are new to Extraction Validation testing aka Reduction Curves then please read Jared's article first.

- COMPONENT CLEANLINESS TESTING: EXTRACTION VALIDATION

So, today let's take a look at some examples of Extraction Validation Curves aka Reduction Curves. Some are "good" – meaning they achieve the target level of extraction efficiency and thereafter run in a "sideways moving channel" at a low Mass. Some are "bad" meaning they never achieve the target level of extraction efficiency. Some are "ugly" meaning they do something quite unexpected and out of the ordinary such as initially showing good/great reduction (perhaps even achieving target efficiency) followed by spiking up to higher Mass or Particle Count than the first extraction in the series. So let's take a look at "the good, the bad, and the ugly" regarding Extraction Validation (EV) aka Reduction Curves and see what can be learned.

Batch of ten parts tested	10 micron patch before - milligrams	10 micron patch after - milligrams	Total contamination - 10 micron patch - milligrams	Extraction Efficiency	Solvent Spray Passes over all surfaces (two sides) approx. solvent volume used	**Extraction Parameters repeated six times on same part**
1st Extraction B	70.77	73.44	2.67	88.12%	3 passes 1000ml	**Solvent Pressure Rinse**
2nd Extraction C	71.47	71.83	0.36	92.38%	6 passes 2000ml	3 times across all surfaces (two sides)
3rd Extraction D	70.72	70.97	0.25	100.00%	9 passes 3000ml	Ultra Solve Plus = solvent
4th Extraction E	71.14	71.14	0.00	100.00%	12 passes 4000ml	approx. 1000ml solvent used
5th Extraction F	71.56	71.56	0.00	100.00%	15 passes 5000ml	on part
6th Extraction G	71.04	71.04	0.00			

QF1185B

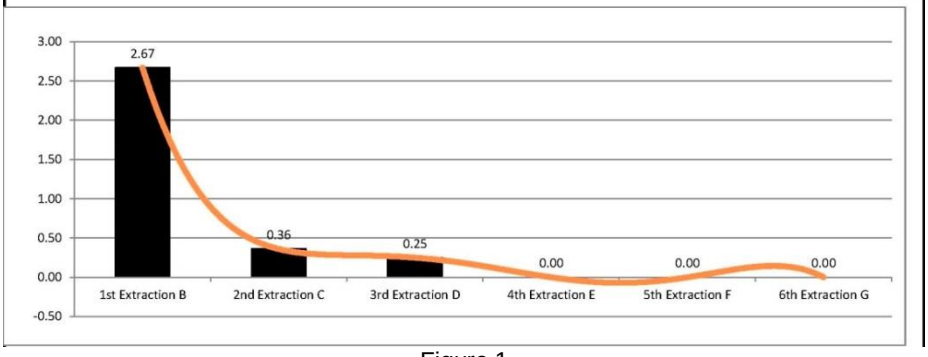

Figure 1

In Figure 1 (above) we see a "good" Extraction Validation test. Excellent reduction of contamination was achieved right away and reduction continued during extractions 2 and 3. This suggests that the tested extraction formula/recipe was well chosen based on a quick glance at the chart. The target of

90+% Extraction Efficiency or <10% remaining contamination on the part (or batch of parts) was achieved after two extractions and the fourth, fifth, and sixth extractions all showed no measurable mass of contamination. This is an example of what a "good" extraction validation curve based on Mass looks like. (Don't however expect to get repeated zeros – it doesn't happen that way every time – for a variety of reasons.)

ISO 16232 Size Class:	**Extraction 1** (standardized particle count on sample "J" 10um patch)	**Extraction 2** (standardized particle count on sample "K" 10um patch)	**Extraction Efficiency:** ($E^1/(E^1+E^2)$)
Total Population: 15um-600um (C-I)	149569.70	26981.10	84.72%

QF1185B

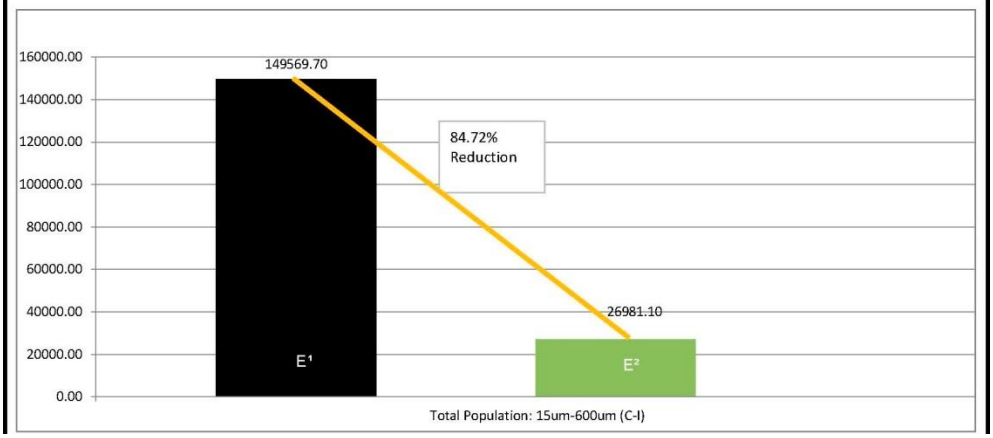

Figure 2

In Figure 2 (above) we see that the extraction formula/recipe derived from the testing done in Figure 1 produced 84.72% Particle Reduction when tested for Particle Count reduction rather than Mass reduction. The target in this case was 70+% Particle Reduction (per the OEM spec in this instance) so this EV was "good" for both Mass reduction and Particle Count reduction.

Let's move on to Figure 3 and take a look at another example – this time when the target isn't reached – when things technically went "bad."

This example in Figure 3 (next page) is technically "bad" because the target extraction efficiency of 90+% (based on Mass) was never reached even after 6 extractions of the same part or batch of parts. What you do see is that the first extraction in the series achieved the REAL maximum level of reduction and then the remaining 5 extractions fell into a "sideways moving channel" showing no further reduction actually occurring. If you decided maximum efficiency based on math alone with no connection to the reality of what is happening you might be inclined to set up your extraction procedure based on 15 passes of Pressure Rinsing with a cumulative volume of 8,000ml of extraction fluid because the math says that comes closest to the target of 90+% extraction efficiency or you might use a larger batch size so you start out with higher Mass of contamination or you might set up new extraction parameters then try again. While those are viable alternatives you may note by actually charting out your data that the cessation of reduction is visibly noted allowing the perceptive cleanliness testing technician to correctly discern that 3 passes of Pressure Rinsing with a cumulative volume of 1,600ml of extraction fluid used is adequate – which saves time and solvent on all future extractions – along with saving time presently doing another extraction validation.

Batch of five parts tested	10 micron patch before - milligrams	10 micron patch after - milligrams	Total contamination - 10 micron patch - milligrams	Extraction Efficiency	Solvent Spray Passes over all surfaces approx. solvent volume used	**Extraction Parameters repeated six times on same batch of parts**
1st Extraction K	67.27	67.51	0.24	77.42%	3 passes 1600ml	**Solvent Pressure Rinse**
2nd Extraction L	67.21	67.28	0.07	83.78%	6 passes 3200ml	3 times across all surfaces
3rd Extraction M	67.49	67.55	0.06	84.09%	9 passes 4800ml	Ultra Solve Plus = solvent
4th Extraction N	71.57	71.64	0.07	84.62%	12 passes 6400ml	approx. 1600ml solvent used
5th Extraction O	71.36	71.44	0.08	88.89%	15 passes 8000ml	Batch of five parts
6th Extraction P	71.38	71.45	0.06			

QF1185B

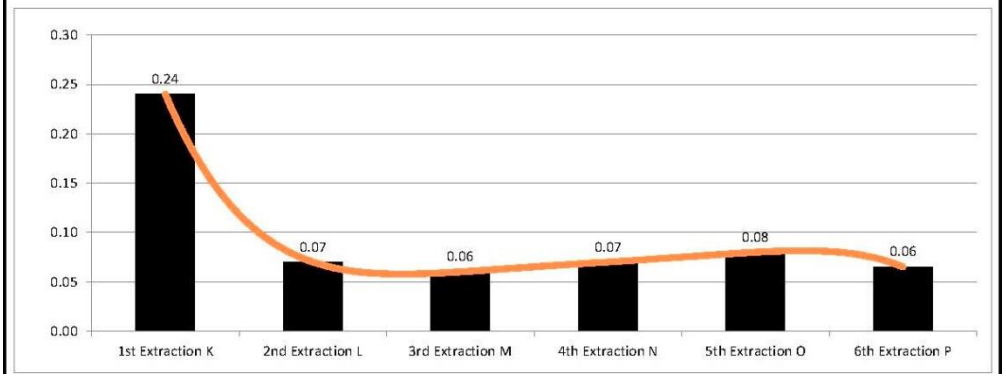

Figure 3

For those skeptical of our conclusion that adequate extraction was accomplished by extraction 1 in the example shown in Figure 3 (because the target % wasn't hit) let's look into the matter a bit further and perhaps alleviate your skepticism. Figure 3 is showing a more typical dispersion of values after reduction ceases – that is a range of values that move a wee bit up and a wee bit down but maintain a consistent range of values that form a "sideways moving channel" graph formation rather than showing further reduction. But alas that still doesn't mean it hit the target reduction % so how can we be sure it is adequate? The chart is telling you the truth – you got all the reduction you are going to get via extraction 1 but with a low Mass of contamination you can't get the math to agree you have achieved your objective (hit your target) unless you nearly miraculously get all zeros after reduction ceases. So how can the skeptic within you be satisfied when the math won't work out? How about doing a particle count reduction instead of a Mass Reduction? See Figure 4 below and watch the technically "bad" Extraction Validation turn good before your very eyes.

ISO 16232 Size Class:	**Extraction 1** (standardized particle count on sample "Q" 10um patch)	**Extraction 2** (standardized particle count on sample "R" 10um patch)	**Extraction Efficiency:** ($E^1/(E^1+E^2)$)
Total Population: 15um-600um (C-I)	3884.10	420.30	90.24%

QF1185B

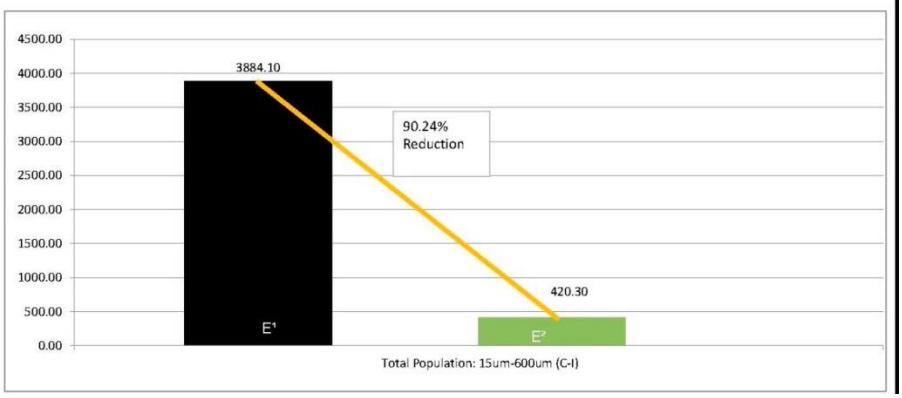

Figure 4

In Figure 4 (previous page) the particles in ISO 16232 size classes C-I were counted per an OEM specification and a 90.24% reduction was achieved. Extractions E1 and E2 were done based on what Figure 3 indicated – namely 3 passes over all surfaces with a total of 1,600ml of solvent used during the Pressure Rinse extraction. In this case the particle count reduction was actually greater than the mass reduction by a small margin though that isn't always the case. The results from Figure 4 validate that the first extraction in Figure 3 actually achieved adequate extraction efficiency.

So now it's time to look at some "ugly" results that do something quite unexpected. These types of results are something the cleanliness testing technician really doesn't want to see but they do occur so let's look at an example and see if we can learn something. (See Figure 5 below) On this "ugly" extraction validation you see that the target of 90+% extraction efficiency was "allegedly" (mathematically) achieved by the second extraction. You would have never known it would turn "ugly" if you stopped there. But then the fourth extraction had nearly as much Mass as extraction one and the fifth and sixth extractions each had three times the Mass of extraction one. The "ugliness" lead us to do an additional six extractions which themselves showed more "ugliness" – definitely not the neat and tidy reduction you want to see. So what could cause the "ugliness" in the fortunately rare examples of "ugly" extraction validations? Here's a short list (not all inclusive by any means) of some possible causes: part material incompatible with extraction fluid; inappropriate extraction fluid which is unable to quickly dissolve residual material from process fluids including Rust Preventative; material characteristic of being prone to shedding of particles ad infinitum (cast metals, powdered/sintered metals, etc.); surface treatment which flakes or sheds (plating, paint, etc.) or has potential to entrap then later release particles (shot blasting, etc.).

Functional Test Bed / Timed Flow Extraction

Tested Part	5 micron patch before - milligrams	5 micron patch after - milligrams	Total contamination - 5 micron patch - milligrams	Extraction Efficiency	5 minutes flow through cycle	Extraction Parameters repeated twelve times on same batch of parts
1st Ext B	102.49	102.80	0.31	70.45%	5 Minutes	3000ml Ultrasolve Plus
2nd Ext C	101.16	101.29	0.13	97.78%	10 Minutes	recirculated through "that part"
3rd Ext D	100.35	100.36	0.01	64.29%	15 minutes	and inline 5um filter.
4th Ext E	101.06	101.31	0.25	41.92%	20 minutes	Flowed for 5 minutes/cycle
5th Ext F	101.64	102.61	0.97	63.50%	25 Minutes	35-40psi compressed air input
6th Ext G	101.54	102.50	0.96	72.853%	30 Minutes	to pump
7th Ext N	101.77	102.75	0.98	99.724%	35 Minutes	
8th Ext O	100.10	100.11	0.01	88.943%	40 Minutes	Extractions 1-6 shared fluid
9th Ext P	100.86	101.31	0.45	82.637%	45 Minutes	Fluid drained thinking completed
10th Ext Q	101.59	102.38	0.79	84.030%	50 Minutes	Extractions 7-12 shared fluid
11th Ext R	101.41	102.25	0.84	92.105%	55 Minutes	
12th Ext S	101.70	102.15	0.45			

QF1185B

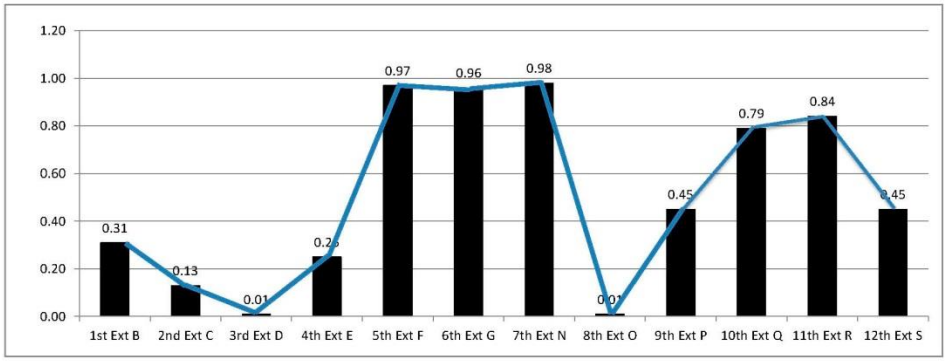

Figure 5

Once you have “ugly” extraction validation results you are faced with the challenge to figure out what happened so that an appropriate extraction method can then be developed. It can be fairly simple if the extraction fluid degrades the material being extracted or doesn’t quickly dissolve residual materials from process fluids – that “simply” requires an appropriate extraction fluid to be chosen. But some of the other potential causes of “ugly” extraction validation results can be a LOT more complicated to remedy IF they can be remedied at all in the near term. Persevere.

Oh for the perfect world wherein Extraction Validation Testing aka Reduction Curves always come out perfect and easy to understand – very clearly pointing out optimum extraction parameters. But alas the path is strewn with variables and the impact of multiple variables can occasionally leave you scratching your head and wondering what happened and why.

Please feel free to give me a call – we do a lot of ISO 16232 based testing for a wide array of customers here at the Crown Cleanliness Testing Laboratory in Jackson, Michigan USA. Give me a call when you have a question about cleanliness testing or need cleanliness testing done. We offer Standard Turnaround for scheduled cyclical testing and Expedited Turnaround when you need results ASAP. We also sell Lab kits and can train your personnel to do cleanliness testing if your customer insists you do the testing in-house.

Jack Griffes | Laboratory Supervisor | Crown Cleanliness Testing Laboratory
Crown Industrial Services Inc. | 2070 Brooklyn Rd. | Jackson, MI 49203 | USA
jgriffes@crownindservices.com | (517) 905-5328 Direct