CROWN
INDUSTRIAL SERVICES INC.

CLEANLINESS TESTING LABORATORY

We test to ISO 16232

Component Cleanliness Testing:
Extraction Validation

Component cleanliness testing has become a necessity in the Automotive Industry to ensure the functionality as well as the longevity of the vehicle. Whether you are experienced in the field of component cleanliness testing, just beginning, or simply interested in learning more about the processes involved in determining if the cleanliness of a component is in compliance with its engineering standard, a primary factor to take into consideration is extraction validation.

Once the extraction method has been established (appropriate extraction fluid, mechanics by which the component is extracted) the next step is to validate one's extraction procedure. Depending on the OEM (original equipment manufacturer) standard, "extraction validation" may be referenced by various terminologies but the basic concept is the same: to exhibit the efficacy of one's extraction procedure. For example, ISO 16232 requires a 90+% reduction of contaminants over a maximum of six extractions conducted on the same part or batch of parts to be considered a valid extraction. The reduction of contaminants between extractions is calculated by analyzing either the mass of or total count of particulates obtained with each sample and dividing the results of the last sample attained by the sum of the results of all samples. For instance, it is established that a particular component will be extracted using a pressure rinse technique with a mixture of isoparaffins. An isoparaffin is a non-polar alkane with branch chained hydrocarbons that give off little to no odor. Often referred to as mineral spirits, isoparaffins are called out in many standards as being an appropriate solvent for extraction. The individual conducting the test decides to spray all surface areas of the part three times for the initial extraction using approximately 1000ml of solvent. Once the contamination has been filtered from the sample, the resulting mass is 1.15mg. Using the exact same parameters as the initial extraction (it is very important to document the procedure in detail for proper replication) the next sample collected from the same part is filtered and analyzed. This time the resulting mass is 0.64mg. The result of the last sample, in this case 0.64mg, is then divided by the sum of all extractions. 0.64mg plus 1.15mg yields a total of 1.79mg of contamination removed from the part after two extractions. 0.64mg divided by 1.79mg is 0.358. This indicates that 0.64mg is 35.8% of the total contamination removed during the extractions (1.79mg.) As 1.79mg is 100% of the contamination recovered, simply subtract 35.8% from 100% and it is evident a 64.2% percent reduction of contaminants occurred between the first two extractions. Since the 90+% reduction of contaminants did not occur it can be concluded that 3 passes over all surface areas of the part using approximately 1000ml is not a valid extraction. On the third extraction 0.11mg of contamination was removed from the part. After calculating the data (0.11/ 0.11+0.64+1.15,) it is found that a 90+% reduction of contaminants did occur. This would indicate that spraying all surface areas 6 times using approximately 2000ml of solvent is a valid extraction procedure. Do note that the six extractions should occur one after another and typically are conducted prior to the analyzation of the data. A graph of the data should show a declining trend in the amount of contamination removed by each extraction. This graph is also known as the Extraction Validation Curve. ISO 16232 does point out that the graph of the data may not always appear the way it should in particularized situations such as low contamination levels or difficulty removing contamination. (See Figure 1 from **ISO 16232-3:2007(E)** for example of Extraction Validation Curve.)

It is also important to take into consideration that, per ISO 16232, if a 90+% reduction of contaminants has not occurred after six extractions then the extraction parameters are not suitable and must be adjusted. Suppose if instead of using 1000ml over the course of three passes over all surfaces, only 100ml was used. After six extractions, 18 passes over all surfaces using a total 600ml of solvent failed to yield the target reduction indicating an adjustment to the parameters needs to be made. Spray time as it correlates to solvent volume usage seems to be the factor that would call for adjustment. This concept delves not only on the validity of one's extraction but also the efficiency of the extraction. Using 100ml of solvent with 3 passes over all surfaces is not as efficient as using 1000ml when the goal is to remove 90+% of the contamination in as few extractions as possible to validate an appropriate procedure. Consider if the same extraction parameters as the initial example were used but deionized water was substituted as the extraction fluid. In this case, a 90+% reduction of contaminants was never proven over the series of six extractions. Using isoparaffins as the extraction fluid completed the task in two extractions. Perhaps a 90+% reduction of contaminants was capable of being proven using deionized water but it took eight extractions to do it, indicating to have a validated extraction process using deionized water one would have to spray all surface areas 24 times using approximately 8000ml of solvent. This is not nearly as efficient of a procedure as using isoparaffins.

Of course, not all standards require a 90+% reduction of contaminants as does ISO 16232. Ford's ESBC3P-7W092-AA/BA only requires a 60+% reduction of contaminants by mass and is to be achieved with two extractions on the same part or batch of parts which required three passes over all surfaces using 1000ml of solvent in the initial 90+% reduction example given. Thus, depending on the standard which applies to the component being tested, it may be easier to validate the extraction. Ford's ESHL1P-7W092-BA requires a 70+% reduction of contaminants, however this must be proven strictly by particle count. Consequentially, this can be difficult to validate on the first attempt and may cause one to have to adjust the extraction parameters a number of times before attaining the goal. In an instance like the aforementioned, it may be prudent to conduct a six extraction "ISO 16232 style" mass based extraction efficiency or

validation test aiming for 90+% to get an idea of suitable parameters that would yield 70+% reduction of contaminants by particle count based on Ford's extraction validation formula which uses only two extractions.

When it comes to component cleanliness testing validating one's extraction is crucial to ensuring the proper removal of contaminants to determine whether or not the test results are accurate. Sure, one could randomly spray a part to be tested with solvent and remove a certain amount of contamination to get the sample to comply with the standard to which it is being tested but that doesn't necessarily mean the test was conducted appropriately. Establishing adequate extraction parameters as well as warranting the test is being conducted with the utmost efficiency will allow for realistic and repeatable results that deem the procedure valid. In the quest for true cleanliness it is not just about reporting something to be clean but being able to prove it.

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Please feel free to give us 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. Do not hesitate to contact us 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.

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Figure 1 from **ISO 16232-3:2007(E)**

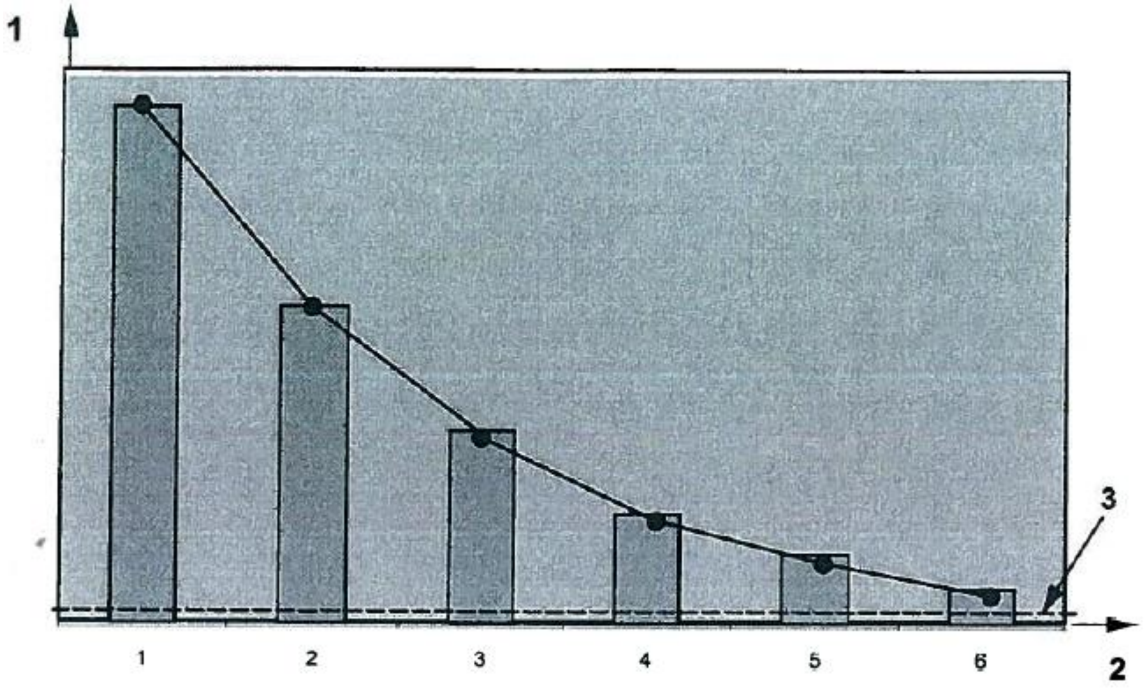

Figure 1 - Example of extraction curve (ISO 16232-3:2007(E))

Figure 1 shows an example where each of the six extractions in a row is showing a reduction in contamination.