Formulation and Evaluation of Chemistries for Cleaning Applications

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In today's accelerated regulatory environment, new cleaning chemistries and formulations are introduced to the market at a sometimes-alarming rate. As such, companies face the common challenge of adequate and cost effective performance evaluation. The purpose of this paper is to introduce methods by which those who use chemicals can increase their selection efficiency, minimizing the time and effort to do screenings, and maximize the quality of the results of those screenings. The session will provide insight on future regulatory trends and keys to ensuring compliance. Simple, practical methods will be presented by which anyone can effectively conduct performance screenings for making concrete decisions about alternative cleaning agent selection.

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Introduction

Welcome to 2001. Over 12 years after the mad dash to replace chemicals brought on by Montreal Protocol, cleaning processes for manufacturing, repair, and maintenance have evolved significantly. Over 99% of companies have fully made the transition from ozone-depleting compounds. A few still remain, especially government facilities and government contractors, who use chemicals from stockpiles due to inability to locate adequate replacements to clean critical parts. This population, however, is quickly declining as a function of both reasonable replacements being identified and the added pressure of dwindling stockpiles

Today's regulatory environment is constantly changing, becoming more and more restrictive with each passing day. In addition, the first generation of cleaning chemistry replacements offered far less than "drop-in" replacements. Performance was not always equal and each chemical came with a host of new baggage like VOCs, toxicity, waste concerns, etc. There is a strong desire to reduce this baggage, coupled with consistent advances in cleaning chemistry from the multitude of vendors offering so-called "ideal" solutions to all of cleaning's problems. A good analogy would be to say that there is no "Microsoft" when it comes to cleaning chemicals. This definitely has its pros and cons, and depending on whom you ask, opinions vary widely. One of its primary effects, however, is to add both end-user confusion in choosing a reliable product and significantly increasing the number of candidate solutions one must evaluate.

This confusion and frustration is brought to an even higher level when faced with the challenge of quickly, fairly, and effectively testing potential replacements. The quality and frequency with which this testing is performed is critical. It has a strong and direct impact on ensuring that the cleaning process produces both top quality results that maximize revenues and a safety profile that minimizes resource expenditures.

The purpose of this paper is to introduce methods by which those who use cleaning agents can increase their selection efficiency, minimize the time and effort to do screenings, and maximize the quality of the results of those screenings. This can occur by following a diligent process of both carefully researching potential solutions first and then a scientific approach to the screening process.

The Scientific Method

Any evaluation of new candidates is technically an experiment and as such, should be done by employing good science. Critical to ensuring that any experiment is performed properly, the scientific method is a rigorous process that employs four simple steps:

1. Observation
2. Hypothesis
3. Prediction
4. Testing

Notice that testing comes last. This is because without doing the right work prior to testing, those tests essentially become meaningless, as the experimenter may not know what the tests are really saying. A good example of this would be the performance seen by using a replacement solvent at a higher temperature. Did the solvent work because it was a better solvent or because the heat was higher? Would the old solvent have worked just as well at this temperature? Was the real problem not the chemical at all, but some mechanical deficiency?

To many, this process may just seem like common sense, and for those experienced in the process, it is. Understanding each step and its place in the solution process, however, is key to ensuring that solid and reliable results are obtained each time the process is initiated. Lets discuss each of the steps in the scientific method and relate them to evaluating new chemistries.

Observation: Good scientists are observant and notice everything about a given situation. For example in a given cleaning situation, it is important to recognize and record everything that may influence the process, of which, chemistry is just one element. Other important elements include mechanical action (spraying, pressure, wiping, etc), temperature, equipment function, soil load and type, cleaning time, etc. The key to the observation step is to feel confident that the chemistry is what needs to be replaced prior to embarking on a process to do so. If the key reason for replacement is not performance related (i.e.: toxicity, treatability, etc.), this step might seem unnecessary, however, knowing all of this information will be critical when determining the performance of a new compound.

An example of an observation would be that particles are being left behind from a wipe cleaning application. This in effect becomes the problem that needs to be solved. The next step in the scientific method describes how to handle that observation.

Hypothesis: This is a tentative explanation for what is being observed, not an actual observation. It is a good chance for the experimenter's past experiences with similar problems to assist in making the most accurate solution to the observation requiring action. It is important to propose as many hypotheses as possible and be sure that both deductive reasoning and experimentation can test them.

Two good example of hypotheses could be drawn from this given wipe-cleaning situation where particulate soil is being registered in cleaning verifications. One hypothesis would be that the cleaning agent is not doing an effective job of removing the particulates. Another would be that these particles are being shed from the wipe substrate. Both hypotheses can be tested through proper experimental design.

Prediction: This is the point where the experimenter uses deductive reasoning to test the hypothesis he has proposed. In other words, one "predicts" that specific results will be seen if something is tested. For example, one might predict that if the wipe substrate were causing the particulates, then using a non-linting wipe would correct the problem. If the problem lies in the cleaning agent, however, one would predict that a better cleaning agent will be the solution.

Testing: This is the final step in the scientific method where all of the due diligence put forth in prior steps comes together to determine if a solution can be found. The most important key to testing is to run "controlled" experiments. Experimenters must contrast experimental groups with a "control" group, which represents the current application being replaced. The two groups must be treated exactly equal except for the one variable being tested. For example, when testing a new wipe substrate, it is key to ensure that the exact same chemistry is used and that the temperature, pressure, and number of wipe cycles are identical in both tests. The only difference can be the substrate

Also, when running experiments, it is critical to do replication of tests. Every test should be replicated at least 3 times to ensure that the results are true and minimize the occurrence of anomalies that would not show up in real life cleaning.

Finally, the researcher must be able to extract quantitative data from the tests. Just saying that Prototype B works better than Prototype A will not do. Unless the degree of improvement can be measured in some fashion, the justification for making a switch could be questionable. Much of this can be resolved just by using a set of predetermined standards. Then, results can be compared to standards that represent "pass or fail" results.

That concludes the discussion of following the scientific method when evaluating new options for cleaning. Though general and possibly very simplistic in nature, it represents a proven reliable method of doing the right research to obtain sustainable and credible results. It is now prudent to move into a more targeted discussion of steps to follow when working with cleaning chemistries and evaluating new ones for use in various applications.

The following steps 1-3 would be considered things to be done in between the *prediction* and *testing* steps of the scientific method. By performing these steps properly, the researcher can intelligently narrow the candidates that should be tested as well as significantly raising the likelihood that a suitable and sustainable replacement will be identified.

Step 1: Set the priorities

It is a rare case that new chemistries are brought on site for no particular reason. It is key that the reason new compounds are being sought be set as priority number one. From there, the remaining priorities need to be set and ordered by importance. Be sure that every issue that can accompany a chemical is accounted for and noted as a "need" or a "want." For example, ozone compatibility is a "need" for 99.9% of all cleaning applications.

Other needs would be non-carcinogenicity and non-flammability. Low odor or recyclability may actually just be "wants" as these issues can be managed and still produce quality parts. In most cases, chemical cost, though often touted as a "need," is in reality, just a "want." Used properly, most good chemicals incoming costs can be positively balanced against the benefits they offer. It is important to realize that in any situation, you always get what you pay for.

Step 2: Do your homework

It's a common and not necessarily wrong practice that frequently occurs in today's cleaning applications. Without spending a significant level of time researching all of the critical issues associated with specific chemistries, these chemistries are brought into facilities, quickly and haphazardly evaluated, and later, found to have some flaw that makes them significantly unattractive. From that point on, resources are put into efforts to identify the next replacement chemistry. These resources are, to a large extent, wasted because had the process been done right the first time, those resources could be spent more productively elsewhere. In addition, it can easily become an endless cycle as one chemistry after another is evaluated without maximum efficiency.

To prevent this from occurring, it is essential that all relevant qualities of a proposed solution be fully researched. Make a list of key questions to be answered; especially those that will impact the specific process being upgraded. Be sure to know which criteria <u>must</u> versus the ones that <u>should</u> be met. The following list provides a good starting point for a list of critical areas of knowledge to which answers should be provided before moving forward:

1. Physical properties of the chemistry
    a. Flashpoint
    b. Odor
    c. Vapor Pressure
    d. % Solids
    e. Level of Impurities
2. Necessary equipment to maximize worker safety
3. Proper equipment and operating conditions needed to maximize performance
4. Incompatibilities
5. References of current customers
6. Technical service agreement

Of course there may be other key attributes that are needed depending on the application. A final hint is that if vendors are unwilling or unable to provide important data on products, those products should be avoided. There is no point risking a significant loss of time and resources because negative properties of the chemical were discovered too late and because of lack of due diligence on the part of the vendor or customer. Finally, the old adage remains that anything that looks too good to be true probably is.

Step 3: Ensure smooth production

In many cases, the stated reason for misguided decisions is that there was not enough time to do a thorough evaluation. Production needed to begin immediately and the old chemistry needed to be removed or money/opportunity would be lost. While this is certainly an easily understandable scenario, the money and opportunity gained in the short term rarely outweighs that which is lost in the long term. Most of this urgency is caused by two thing that act together or alone:

1. Someone waited until the last minute to evaluate replacements - No explanation on remedies needed here.
2. Any worthwhile testing is performed on the actual production line rather than a "proving area" due to the lack of effective and correlatable test methods. Many are all too familiar with the feeling of "it doesn't work right until it works on the real thing."

It therefore becomes essential to complete the following steps before actually disturbing production:

1. Begin researching replacements immediately
2. Set up a method for gauging performance that correlates with production, but does not require shutdown for testing; in other words, a reliable "lab test."
3. Use that method to narrow candidates down to one or two before testing on the production line.

Step 4: The methods

Evaluating the performance of different chemicals can be a very ambiguous and misleading science if not performed correctly and with the right perspective on the data. This is exceedingly true in the case of solvent versus aqueous chemistries. In most cases, solvents work primarily by dissolving soils into them. Aqueous chemistries, however, generally need some form of mechanical action in order to effectively activate their cleaning powers. Just to name a few, wetting, emulsification, dispersion, and suspension can only be fully optimized with some sort of mixing and impingement.

Since almost all cleaning systems work using a variable mixture of chemical and mechanical factors, it is critical to isolate each factor and note its impact on cleaning. By doing so, it will be possible to theorize what ideal combination of potential variables will be needed to do the most effective job of cleaning. For example, if very delicate parts are being cleaned, then strong mechanical force is impossible. As such, pure chemical factors take a higher seat in the order of importance. A solvent may be the ideal choice. On the other hand, where parts are rugged, but flammability and vapors may be an issue, pure chemical effects are less needed and can be replaced by aqueous chemistries coupled with stronger impingement.

In lab-scale evaluations, there are three key steps in testing: sample preparation, testing, and results analysis. Each step is equally critical and defects in any one can result in erroneous evaluations.

Sample preparation is the first step in conducting any lab scale evaluation. How one prepares samples for testing is just as critical as how the tests themselves are run. Any error or improper variability in sample preparation will ultimately lead to invalid test results. Any improvements or shortcomings exhibited by tests can then potentially be traced to variability in sample prep rather than strengths or weaknesses of the solutions.

For each of the tests in this paper, sample preparation is identical except that the size of the test coupon varies by test and will be noted in the procedure. In general test coupons are prepared by placing a known and repeatable weight of "soil" (ie: grease, oil) onto the coupon. The test coupon should be made from a material equal or similar to that which will be used in production. It is critical that the coupon material not be significantly different (ie: metal vs. plastic vs. glass). This is because each material has a specific critical surface tension that can have a direct impact on the ease with which soils are removed. A final note on sample preparation is to ensure that both the environment in which samples are prepared and the length of time before they are tested stays constant.

To accomplish the task of properly evaluating chemistries, three basic and variable "lab tests" can be constructed. Each test can be set up to minimize and maximize both chemical and mechanical factors in cleaning. When used together, the combined data can be extremely telling as to how cleaning will be best accomplished in a production scale environment. Figure 1 shows a graphic representation of the stress that each test can place on both mechanical and chemical properties. This gives the experimenter an idea what test(s) to use and in what manner in order to most accurately evaluate solutions, as they would be used in production.

The following discusses these three tests and how each can be custom tailored to give data that corresponds to varying blends of chemical and mechanical factors. Always remember to follow safe procedures based on the chemicals in use. Medium to high vapor pressure solvents should be used in a hood or well-ventilated area.

Test #1: Immersion – This is a test based primarily on chemical factors and incorporates varying levels of mechanical factors. The immersion test is an ideal test for pure solvency and showing the effects that chemicals have in both static and agitated conditions under liquid. Temperature can be varied from ambient through super-heated. Agitation can be varied from zero agitation to intense agitation. With this test, the effects of pure solvency can be explored as well as how that solvency can be enhanced with agitation. It also shows how a good cleaning agent can remove and emulsify or split soils given the proper conditions. On a one to one basis, this is a good test for mimicking what happens in a vapor degreaser or agitated/non-agitated dip tank.

Appendix A shows a diagram of the system as well as the items needed in order to make it function properly. It is important to remember to run all prototypes at the same soil weight, time interval, temperature, and RPM so as not to void results with a second variable.

Test #2: Impingement – This test now incorporates the impingement factor which can be correlated to any spraying or pouring action in a cleaning process. This is an ideal way to look at comparing a solvent versus and aqueous agent. It allows a cleaning agent to exhibit its qualities as a solvent and its qualities for wetting, soil rollup, emulsification, and dispersion all in one tunable step.

The temperature of the solution in the system can be varied, but always check the temperature at the point of impingement as heat exchange will occur in the downspout. Also, the height and angle of impingement will have a significant impact on cleaning. The sharper the angle of impact, and the higher the liquid drop, the easier cleaning will occur. Try to adjust parameters to most closely mimic what parts will actually see.

Appendix B shows a diagram of the system as well as the items needed in order to make it function properly. Again, it is important to remember to run all prototypes at the same soil weight, time interval, temperature, and height/angle of impingement so as not to void results with a second variable.

Test #3: Wiping – This test has the best potential for looking at purely mechanical effects coupled with the increased efficiency brought in by the right solvent. Not only is it the ideal test to use when evaluating wipes, but it also gives an outstanding indication of a soil's ability to be removed by purely mechanical forces.

Variables include the quality and type of wipe substrate, which can make a huge impact on the efficiency of the product. The obvious one is the cleaning agent on the wipe, however, level of cleaning agent, pressure, and number of cycles can all have a huge impact on performance.

Appendix C shows a diagram of the system as well as the items needed in order to make it function properly. As always, it is important to remember to run all prototypes with the same soil weight, number of cycles, substrate, and pressure so as not to void results with a second variable.

Proper analysis of the data collected in testing is critical to ensure that the results are presented in a manner that shows exactly how well or poorly each cleaning agent performed under the conditions of the test. Although there are a number of methods by which analysis can occur (i.e. particle counting, Millipore test, non-volatile residue, weight difference, contact angle, etc.) the key is to be consistent with analysis method.

Remember that these tests are not the actual cleaning system to be used in real life. As such, they should not be expected to give production quality results and should therefore not be analyzed with the same scrutiny. Rather, the experimenter needs to set a "standard" that represents what is currently being done in real life cleaning. If that means that the test leaves a certain level of soil behind, that's OK. In fact, it is critical to calibrate each test such that the control fails to fully clean to some extent. Otherwise, it will be impossible to determine which prototypes actually performed better. Some may even argue that this does not hold when looking for equal performance. Unfortunately, this is when the case holds most strongly. For example, if the control system fully cleans a coupon in two cycles or ten minutes, but the test runs for 4 cycles or twenty minutes, the end analysis is the same for a system that cleans worse but in three cycles or fifteen minutes. Be careful!

Finally, it is important to note that these methods are for the most part, merely correlative in nature. Aside from the wipe test, they do not represent exact mimics of real-life cleaning situations. These tests need to be properly calibrated such that either the results will directly correlate to improvements or shortcomings in the actual cleaning system or, more likely, simply give the experimenter a solid idea of the level of improvement likely to be seen in real life. Also, realize that the tests also do not account for other very critical factors in cleaning systems such as corrosion, foaming, recyclability, etc. These are critical factors for which preliminary data should be available from manufacturers that can be verified in house once cleaning results warrant further qualification.

Step 4: Do the math

How much money will implementing this system actually save? Are the benefits worth the cost? Many conventional wisdoms state that this is something that should be considered prior to testing. Unfortunately in many cleaning cases, nothing could be more misleading. It is very easy to put together a list of figures on a new system, compare them to current ones, and quickly come to the conclusion that the new system will be more expensive.

When looking at replacement options, it is crucial to evaluate what they offer from a global perspective. The term global means the company as a whole and any parties it affects such as the environment, surrounding community, and its customers. Each has an impact, direct or indirect, on the company's short term and long term profitability. Good examples of global factors not apparent in just and initial number comparison are reduced energy needed to clean, less waste, higher throughput, lower reject rate, improved part quality, and lower customer complaints.

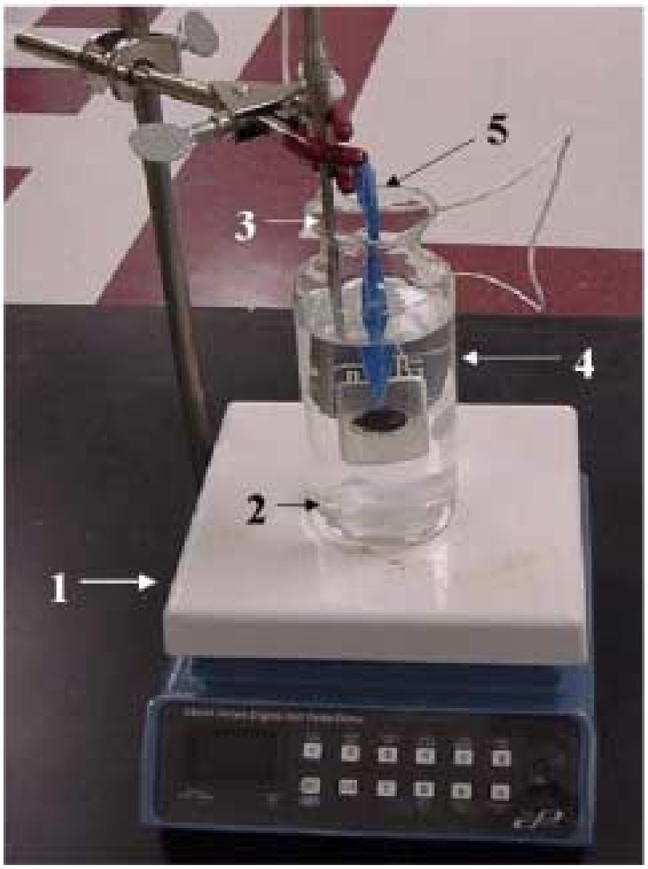
The bottom line is that its essential to do a thorough front and back end evaluation on the economic pros and cons of any potential replacements. The upfront analysis can do a good job of determining if a proposed system is within the ballpark of what may or may not be acceptable. From there, it is imperative to consider both the performance improvements and the global benefits that will lead to lower overall costs.

Conclusion

In conclusion, it is safe to say that when evaluating potential replacements for cleaning systems, having a simple method for screening is highly valuable to the overall process. No method can be to simple, however, without detracting from the quality of the data it provides. It therefore becomes necessary to identify and implement methods which simply and speed the qualification process and do so in a high quality manner.

The methods presented in this paper can be very powerful when used alone to identify trends and improvements in cleaning agents. When used and analyzed properly and together, however, they wield an enormous power to effectively separate and sort any and all cleaning agents as to their level performance alone and with varied degrees and types of mechanical synergy.

Appendix A – Immersion Test



Procedure

1. Prepare a 1” x 2” test coupon of appropriate composition by placing a known quantity of soil (about 0.5g) in the center of the coupon. When applying soil, it is sometimes useful to draw a repeatable area on the coupon by tracing a coin and placing the soil within that circle.
2. Place test solution at appropriate concentration in test vessel and heat to desired temperature. When using solvents, exercise caution by venting vapors and removing any sources of spark.
3. Set stir bar to spin at desired RPM. Obviously, more spin means more mechanical action.
4. Set the timer for the desired test time interval.
5. Clip the prepared test coupon in the hemostat and place into the test solution
6. When time interval is up, remove the coupon
7. Rinse coupon in clean solvent or water and allow to dry
8. Analyze residue by preferred method

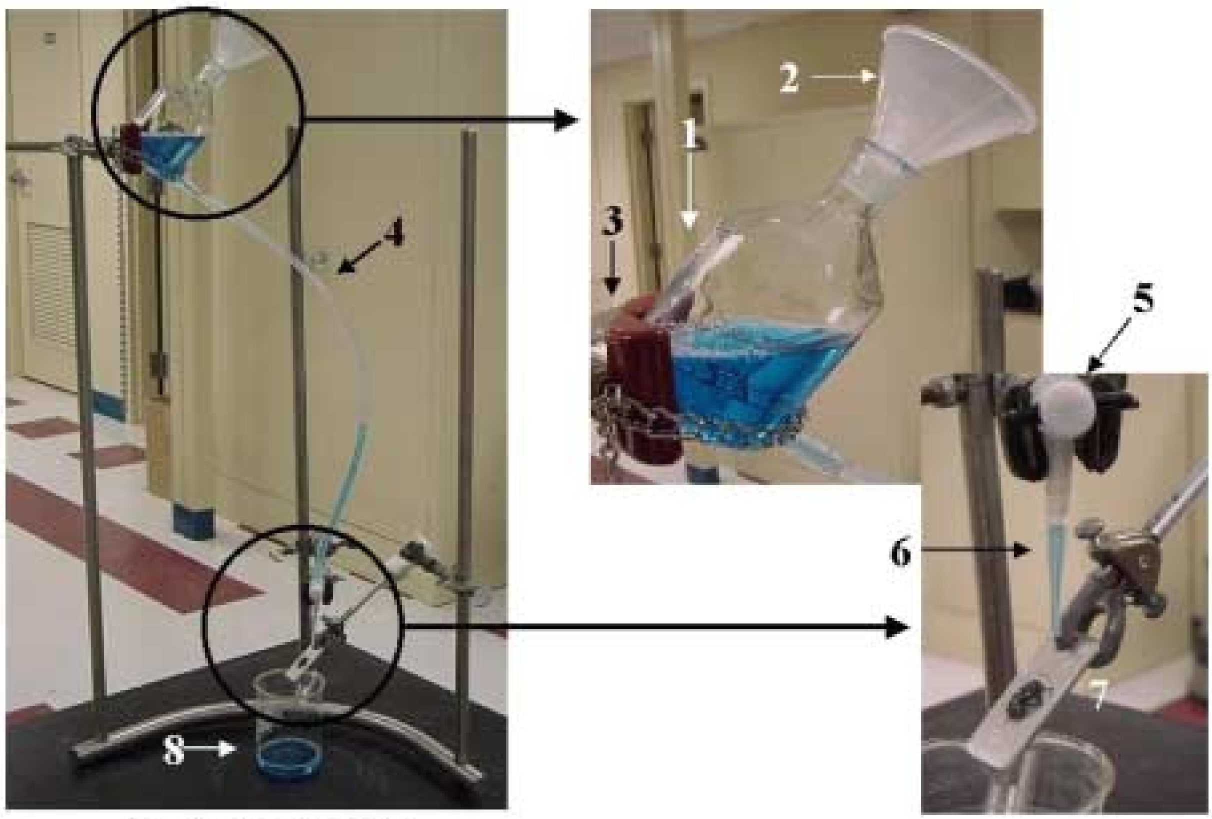
Materials

1. Stir Plate
2. Magnetic Stir Bar
3. Temperature Probe
4. 500ml Fleaker
5. Sample Assembly
6. “Soiled” Aluminum 1100 Coupon
7. Hemostat

* - Materials available from Lab Supply Companies

- Cole Parmer – (800) 323-4340
- VWR – (800) 932-5000

Appendix B – Impingement Test



Impingement Rig

Procedure

1. Prepare a 1” x 2” test coupon of appropriate composition by placing a known quantity of soil (about 0.5g) in the center of the coupon. When applying soil, it is sometimes useful to draw a repeatable area on the coupon by tracing a coin and placing the soil within that circle.
2. Place test solution at appropriate concentration in aspirator bottle at desired temperature. When using solvents, exercise caution by venting vapors and removing any sources of spark.
3. Ensure flow outlet is in proper position
4. Clip the prepared test coupon into place
5. Open stopcock to start test
6. When time interval is up, remove the coupon
7. Rinse coupon in clean solvent or water and allow to dry
8. Analyze residue by preferred method

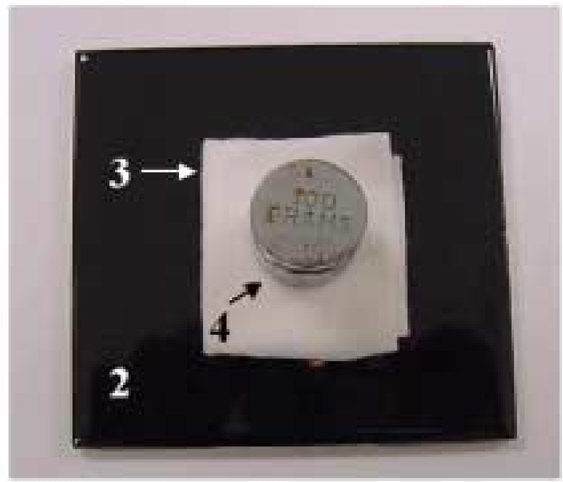
Materials

1. 300 ml Aspirator Bottle
2. Funnel
3. Beaker Clamp
4. Teflon Tubing
5. Stopcock
6. Disposable Pipet Tip
7. “Soiled” Aluminum 1100 Coupon Assembly
8. Catch Beaker

* - Materials available from Lab Supply Companies
  - Cole Parmer – (800) 323-4340
  - VWR – (800) 932-5000

Appendix C – Wipe Test

Soil on White Tile



Test Assembly on Black Tile

Procedure

1. Prepare a 6” x 6” test coupon of appropriate composition by placing a known quantity of soil (about 0.5g) in the center of the coupon. When applying soil, it is sometimes useful to draw a repeatable area on the coupon by tracing a coin and placing the soil within that circle.
2. Prepare substrate by folding and wetting with appropriate level of cleaning agent (about 5-6g). When using solvents, exercise caution by venting vapors and removing any sources of spark.
3. Place wetted substrate on soil with standard weight on top
4. With no additional pressure downward, move the substrate forward and backward to the edges of the tile 3 full cycles.
5. Remove substrate, refold to a clean surface, and repeat scrub.
6. Repeat cycle enough times to calibrate standard to a level below absolute clean
7. After test, rinse coupon in clean solvent or water and allow to dry
8. Analyze residue by preferred method

Materials

1. White Ceramic Tile
2. Black Ceramic Tile
3. Folded Wipe Substrate
4. Standard Weight

* - Materials available from Lab Supply Companies
- Cole Parmer – (800) 323-4340
- VWR – (800) 932-5000

* - Gardner Abrasion Tester
- Byk-Gardner – (800) 343-7721