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Additional Procedures & Tips For Analysis & Control

Process baths from surface preparation to plating and post treatments require specific analysis and control. Most of the procedures incorporate typical steps with which we are quite familiar. These include titrations, pH measurement, and Hull Cell evaluations. The information obtained is very important to quickly determine the particular bath's status when compared to the desired optimum. Two very important benefits stand out. These are accuracy of the analyses and the acceptable short time for turnaround. Anything more that can be done would certainly be helpful.

Are there additional testing procedures that would help round out the analysis picture? Yield more substantive information? Provide data that traditional procedures might miss or generalize? Yes, there are. If so, are they cumbersome, time consuming, require expensive instrumentation, or need specially trained lab personnel? No, they do not. Let us review some helpful procedures that will certainly facilitate and round-out the valuable data obtained from traditional analysis.

Soak Cleaners

The universal analysis procedure is the alkalinity titration. It provides data generalizing the concentrations of all the alkaline components (caustic, silicate, phosphates, soda ash, etc.). There is no distinction or focus on surfactants, wetting agents, complexors, chelates, or other complimentary additives in the formulation or product used. Data interpretation compares the actual analysis to desired concentration range. Additions of the particular cleaner blend are made on this basis. The product added contains a proportionally blended mix of all the raw material components. It's been acknowledged that this procedure reconstitutes and adjusts the cleaner with sufficient levels of the total mix. This maintains or corrects for desired cleaning effect. We keep adding the cleaner on this basis until such additions have little or no positive contribution toward the cleaning process. When this occurs, the cleaner bath is normally dumped and replaced with a fresh make up. A valuable commodity is the old or spent cleaner bath—or specifically what's in it—in addition to an appropriate analysis. It affords very useful data that will supplement the titration analysis. The next time your cleaner is ready to be dumped, take a sample of it and consider the following quick, informative analysis procedures.

Determine the product concentration of the spent cleaner sample and if required, adjust the concentration to optimum.

Specific Gravity

At a fixed solution temperature (*e.g.*, 80-100°F [27-38°C]), accurately weigh 100 mL of the cleaner bath. Or, measure specific gravity by using the appropriate range hydrometer at specified or calibrated temperature. Record the specific gravity. Next, repeat the specific gravity measurement for the freshly prepared cleaner bath (that has not yet processed any parts). Its concentration by titration should be equivalent to the old bath.

As the cleaner bath ages, the introduction of, and contamination with, emulsified oils, grease, and dissolved metals, combine to steadily increase the specific gravity. The practical service life of the cleaner can thus be determined by comparison to specific gravity of the previously spent bath, in addition to concentration by titration. This gives the operation or line maintenance economic benefits.

- Cleaner bath dumps can be scheduled to avoid production shutdowns or bottlenecks.
- Wasteful and needless cleaner additions are avoided, because the determined contamination level cancels any benefits to extending cleaner bath life.
- Tracking specific gravity data may help to justify modifying the cleaner formulation, or switching to a different, better performing, longer lasting cleaner product formulation.
- Tracking specific gravity data may also help to justify installing mechanical oil separators, filters, or membrane technology.

Emulsification

This test is especially useful for emulsifying cleaners. The following procedure will determine the amount of oils and grease actually emulsified in the working cleaner, by splitting them. At operating temperature, carefully add 50 mL of 50 percent v/v sulfuric acid to 50 mL of the cleaner. Avoid solution spattering. Be certain standard lab safety procedures are followed and wear the proper protective clothing. After the acid has been added, mix the solution thoroughly for 10–15 min. Then, transfer the solution to a 100-Ml graduate cylinder. If necessary, adjust the volume to 100 mL with clean water. Let the solution stand until the oil separation or split gives a stable measurement. Calculate emulsified oils and grease:

(mL of oil & grease X 2) / 100 X 100 = % emulsified

Determine the emulsified oils and grease in the spent or ready to dump cleaner. This will provide a practical contamination value. It may be used in conjunction with specific gravity to track cleaner service life and predict anticipated dump cycle. Repeat the emulsion test for the freshly prepared cleaner (no parts processed), at concentration equivalent to the spent cleaner. You may find some of the wetters and surfactants to have oiled out (the value should be a small percentage). Use this as a correction factor when determining emulsification percentage of the aging cleaner. I have found that most emulsifying-type soak cleaners saturate at 5–15 percent oils & grease. Combination soak/electrocleaners also benefit from this test. A true electrocleaner bath should, at worst, hold less than five percent emulsified oils and grease. Higher levels indicate strong consideration for changing the soak cleaner or its operating parameters, improve rinsing between soak and electrocleaner, or other changes, such as precleaning parts off line. The benefits of this test are the same as given for specific gravity measurement.

Performance Test—Soak Cleaner

If you have already sampled the cleaner bath for analysis, save a portion of it for an additional, quick performance test. Use a panel of the predominant base metal being processed in the bath (*e.g.,* steel, brass, copper). Clean the panel confirming absence of water breaks. Steel panels are great, because the protective zinc coating is stripped in hydrochloric acid. Wipe off smut, and rinse to confirm a clean, water break surface. This test will determine if soils are depositing on the panel. It will also indicate if booster adds of the cleaner are required, in conjunction with the titration analysis.

1. Using a pair of tongs so you do not have to physically touch the panel, immerse it in the working cleaner, maintained at the line operating temperature and time.
2. Immerse the panel in clean running water for 30 sec.
3. Inspect the panel for any water breaks (30–60 sec). If water breaks occur, process another fresh panel, to confirm this condition. Otherwise, continue to step #4.
4. Immerse the panel in five percent hydrochloric or sulfuric acid for 15 sec.
5. Repeat step #2.
6. Inspect the panel for any water breaks (30–60 sec).

The absence of water breaks will indicate the cleaner retains sufficient levels of the additive system (not just alkalinity by titration), preventing redeposition of oils & grease. Water breaks will confirm an addition of the cleaner concentrate is needed. The requirement may coincide with the titration analysis. Make the addition to the cleaner bath sample and repeat the performance test. Absence of water breaks will confirm the practical need for the addition of cleaner concentrate to the bath on line. If water breaks continue, evaluate the correction by adding up to 25 percent of the initial charge to a new bath. If more than 25 percent is required, the economics may actually justify dumping the bath in favor of a new make up. Titration of the cleaner may indicate no addition is required, or not to the extent by comparison to the performance test.

We now have three simple, yet accurate and quick tests that can supplement the titration procedure. I would like to squeeze in one more procedure, but it does include an instrumental method. This is atomic absorption (AA). I include it only because many jobshops and captive installations have AA, commonly to support the waste treatment system. If you have ready access to an AA unit, make use of it for another analysis procedure.

Metallic Contaminants

The particular base metals processed in the cleaner bath, (or types of plating baths down the line) confirm the particular analyses required. For example, steel parts may be zinc plated and chromated. Analyze a sample of the spent cleaner for iron, zinc, and chromium. The levels of these metals in the old cleaner indicate their complimentary levels for dumping it (in conjunction with oils, grease, etc.). Analyze for and confirm less than 0.1 ppm of iron, zinc, and chromium in the new cleaner (no parts as yet processed in it). Now, track levels of the specific metallic contaminants in the aging cleaner. Rising concentrations of the metallic contaminants in the working cleaner are another indicator of its practical service life on line. A benefit of this test is determining the appropriateness of the cleaner formulation. Modifying the cleaner, by including a hexavalent chromium reducer, may result in extending the service life of the cleaner. For those equipped with an AA, you have a fourth quick, dependable analysis, supplementing the titration procedure.

Next month we'll focus on analysis and performance testing for electrocleaners and acid dips. *P&SF*