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# More on Analysis & Control: Additional Procedures & Tips

## Electrocleaners

The most common analysis procedure is the alkalinity titration. Neutralization to a specific endpoint is a good determination of the product or formula mixture, related to the levels of the alkaline components. This method does not provide data with respect to any of the other very important ingredients in the product formulation. Inhibitors, complexors, descalers, reducing agents, wetters, and surfactants are some of these special additives. Any of them, by themselves, or in combination with others, may be critical to success of the intended electrocleaning application. Maintenance and booster additions of the electrocleaner blend, based on titration analysis, are intended to restore optimum levels of all the working components. In some electrocleaning steps, the conditioning requirements, or presence of contaminants, may consume or deplete additives at different rates. Based on these considerations, we offer additional tests, intended to supplement the general alkalinity titration.

## Specific Gravity

At a fixed solution temperature (e.g. 80 - 100 degF, 27 - 38 degC), accurately weigh 100 milliliters of the cleaner bath. Or, measure the specific gravity by using the appropriate range hydrometer at the specified or calibrated temperature. Record the specific gravity. Next, repeat the specific gravity measurement for the old, ready-to-dump bath, as well as the freshly prepared electrocleaner bath (which has not yet processed any parts).

As the electrocleaner bath ages through usage, the introduction of and contamination with dissolved metals, oils & grease, combine to steadily increase the bath's specific gravity. The practical service life of the electrocleaner can thereby be determined or predicted by comparison to specific gravity of the previously spent bath, in addition to concentration by titration.

## Performance Test

Why not run a Hull cell on the electrocleaner solution? It's easy, quick, and provides useful information. I have found it to be a good method to verify problems, by duplicating them. This in turn helps to determine the appropriate corrective action.

### *Method:*

A square or rectangular working cell ( steel, stainless) is recommended. A standard hull cell (porcelain) is also good. Set up to conduct the evaluation with cathode and anode in a parallel position to one another. Cut if necessary, a measured section of anode and cathode. Use this to calculate the appropriate current density (anodic, cathodic) for the test. Clean and prepare anode and cathode, confirming cleanliness by water break free or equivalent condition.

1. Heat the electrocleaner to and maintain at the preferred temperature.
2. Insert the cathode into its position in the cell. Connect to negative clip. (More than 90% of electrocleaning treatments are anodic).
3. Insert the anode into its position in the cell. Connect to positive clip.
4. Commence test. Adjust rectification. Run for time equivalent to process cycle or longer if this is necessary to identify or troubleshoot a problem.
5. At the end of the test cycle, remove the anode first. Rinse thoroughly.
6. Observe the surface of the anode for any imperfections, such as water breaks, darkness, discoloration, pits, or etching.
7. Remove the cathode. Rinse thoroughly. Observe its surface. Remember that dissolved metals in the electrocleaner solution will plate out, usually as a smut film, on the cathode.

Some of the conditions you may detect could be due to the following problems, for which corrective measures are suggested. Examples for different metals are given.

- Water breaks. Excess of oils and grease. Improve detergency of the electrocleaner or the preceding soak cleaner.
- Discoloration. Brown film or soft, sludgy material on steel? It could be the electrocleaner is lacking in reserve alkalinity. Solution pH at the steel surface drops during the anodic treatment. Insufficient replenishment of alkalinity (caustic) results in precipitated iron hydroxide film formation. Dipping the panel or part in acid, dissolves the film, revealing an etch of the base metal. Adjust the electrocleaner concentration or switch to a higher caustic-containing formulation. Or, the correction may be to lower the current density to where it should be.
- Darkness. On brass? One condition is low electrocleaner concentration. Another is the insufficient ratio in the concentrate blend of caustic to inhibitors. Usually the surface has been dezincified, forming copper patches. Adjust electrocleaner concentration or change to a properly formulated concentrate with respect to caustic, silicate, and borax.
- Etching. One source of etching is low electrocleaner concentration. Adjust to appropriate level of the concentrate in the working solution. Another problem may be grease etch. The soak cleaner

does not completely remove the oil or grease, leaving a stain or blemish. This shields the electrocleaning action, resulting in etched spots. Improve soak cleaning or improve detergency of the combination soak and electrocleaner.

- Pits. Zinc parts may exhibit pits, sometimes denoted by a white color. Either the alkalinity is low or the ratio of caustic to silicate inhibitor is not correct. Adjust product concentration or change to a better formulated mix.
- Corrosive pits. Brown, period-shaped dots covering all or part of the surface of steel? This can be a nagging problem, especially in double cleaning cycles. In this case, it's due to dragging chloride from the first acid into the second electrocleaner (or when cycling from acid back to electrocleaner). It also occurs in standard cycles, if the water source is heavy in chlorides. While electrolyzing the part, chlorides are attracted by opposite charge to it. Undergoing oxidation, they form chlorine bubbles. These moieties reside on the part surface, etching and corroding it. Eliminating the source of chlorides (changing acid type) is one solution. Modifying the electrocleaner is another. Caustic and silicate are effective inhibitors. Adjust the concentration of the electrocleaner or switch to one with the inhibitor included to control this type of corrosive etching.

## Hexavalent Chromium Test

There are several ways to contaminate the electrocleaner with hexavalent chromium. Continually stripping rack tips of plated chrome and chromates are typical problems, as is the anodic stripping of chrome off plating rejects. Less common, but also a problem, is the carryover of chrome solution inside damaged rack splines and insulation. As little as 20 PPM of hexavalent chromium will passivate steel, brass, and copper, during anodic treatment. Plating blisters usually result from this problem. Hexavalent chromium also immobilizes surfactants, reducing the cleaning action.

A quick, reliable spot test can be used to test for hexavalent chromium in PPM levels. Prepare a one percent stock solution of 1,10 phenanthroline (diphenylhydrazine, available from local lab suppliers) in an alcohol/water mix. Dip some of the electrocleaner solution on a white paper (filter paper is fine). Next, place a few drops of the indicator on the wetted paper. The immediate formation of red color is a confirmation of hexavalent chromium.

Hexavalent chromium level in the electrocleaner can be high enough to turn the solution color yellow. Electrocleaners that effectively control hexavalent chromium contain reducing agents, such as sugars, that change the hexavalent chromium to its insoluble trivalent species. Separate, over-the-side additions of these reducing agents can also be made. The yellow problem reverts to a positive green-colored solution.

It's not enough to perform the analysis, any of which described can be easily included with the titration. The operating parameters are very important. Aside from concentration, be certain temperature and time are in the correct respective ranges. Rinsing should be thorough before the electrocleaner and after exiting it. Problems with any of these steps will result in plating hazes, poor adhesion of deposits, and possibly unwanted production downtime.

Next month we'll check into additional controls for the acid dip. *P&SF*