Maintaining Laboratory Controls

Most finishers have a quality manual that matches the requirements of the top 20 percent of their customers. If, however, you are doing a lot of business with the “elite” (you know—the “rubber meets the road” guys or the “friendly skies” guys), you may have to include controls for your chemical control laboratory. Here are some suggestions that may help make your life a little easier.

Testing Documentation
Let’s start out by documenting the analytical testing procedures. Reference the published analytical methods that you’re using (document control). It doesn’t matter if you are using the product technical data sheet, Metal Finishing Guidebook or an analytical textbook. Depending on your source, you should include: Title, revision level, issue date, product name and page number. If you rewrite the procedure, it should include as a minimum: Sample size, chemical recipe of the procedure and the calculations. Calculations are usually written as Mls (milliliters of the tritrant) times N (the normality of the tritrant) or M (the molarity) times a factor. I know of examples where people have shorted the calculation by multiplying the normality and the factor together, using the result as the factor. I would not recommend using this shortcut. I might, for example, just for convenience, use either 1N or 0.71N sodium hydroxide (0.71N is used because the Mls of tritrant directly equals the percent of hydrochloric or nitric acid). Always remember: Say what you do and do what you say.

If you use an independent analytical laboratory for your finishing control, check to see that the lab has a basic quality system, with written policies in place. The written procedures should govern laboratory operations to ensure adequate and traceable calibration and certification of measurement and test equipment. The lab’s QA manual should be held to the same standards as yours, by including manual number, revision, sign-offs and dates. Equipment must be identified according to its calibration status.

Accountability
You and/or the independent lab must issue a traceable lab report, which should include:

- Processor name
- Shop name
- Report date
- Signature or identification of analyst
- Chemical solution name
- Tank number
- Date sampled (very important)

The report should list the allowable concentration range for each chemical and the concentration found. Include the units of measure (g/L, oz/gal) in the analysis results. Allow a column for recommended additions and comments. The report should be retained for a minimum of one year.
Solution analysis results should be recorded with permanent media (ink), in permanently bound binders (lab book) with numbered pages. Computer records can be changed in the blink of an eye, so if you use a computer for parts of the record-keeping, the report should lead back to an inked hard copy.

Achieving Consistent Results
The finishing solution chemistry information on the lab report needs to be copied to its “home” page. This page (see figure) lists the solution name, tank number, volume of the tank in gal/liter measured so many inches from the top of the tank. Be sure to consistently sample the tank at this same level to achieve repeatable results. Record the allowable concentration range for each chemical and other conditions maintained within specifications. Use a column for recommended additions and comments, such as the dates of new solution makeups and other control measures (e.g., carbon treatment). Often, the tank ingredients are maintained to an external specification (the customer’s). Results that are not within specification limits must be “flagged” or specially noted.

Frequently, a failure to meet external specifications will require that the process tank be taken out of service until the chemistry has been corrected and reanalyzed. Stop a moment and think about the gravity of this situation. The test failure is not limited to just analytical measurements. Salt spray testing is regularly part of the external specifications. Let’s see ... 24 hours for a chromate film to harden before testing, then a 96-hour test to—oh, no!—a failure. Everything you ran in this system (tank and/or chromate) since you ran the test samples, therefore, is in question and must be reported to the customer. You can run a whole lot of parts before the test is concluded. That is why it is so important to keep up the system as a whole. To steal a concept from Milton Weiner (former “Good Days and Bad Days” columnist for Metal Finishing), it’s best to make small daily additions. Should an out-of-specification situation occur in a process solution, the laboratory might document in writing to both QA and manufacturing leadership.

Putting Safety First
The personnel responsible for sample acquisition should be using a documented procedure for withdrawing chemical samples from the process solution tanks. Do they use a sampling tube to core-sample the solution, or do they mix up the contents of the bath first? Please be sure to put safety first.

Standard titration solutions should be labeled with the solution name, concentration, date of standardization and expiration date. If you make your own standards, they should be standardized after makeup. Depending on the customer specification, this standardization may have to be accurate to three decimal places. Commercially purchased standards with certification are not required to be restandardized. Include pH buffer solutions for traceability and change them out at a documented frequency (once a week, for example). Titration solutions should be maintained in an environment that guards and ensures the concentration stability. Depending on the solution type, you may use some of the following: Inverted air inlets, moisture traps or dark containers (for silver nitrate and other light-sensitive chemicals).

I know most of us hate to wash the glassware, but it must be semi-clean, free of spiderwebs and have less than a quarter-inch of dust. (For an audit, the dust thickness is best expressed in some obscure metric term, because no one will be able to do the conversion in their head ... but won’t admit it.) The pipettes, burettes and volumetric flasks used for the analysis should be class “A” to maintain the accuracy of the analytical testing. Get ride of any glassware that is not up to standard. If fluorides play a large part in the chemistry of the shop, it is acceptable to switch to plastic containers.

In conclusion, these are the very things you would do anyway to manage a major tool in your “quality toolbox.” Controlling the way things are done in the lab can have a significant impact on the quality in the shop.

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