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## Hands-on Management

### Quality Control for Aluminum Dyes

Quality surface finishing is very dependent on today's modern organic chemicals. Many of the enhancements in finishing are a direct result of improvements in the organic chemicals used by our industry. Does anybody else remember cars in their prime just rusting away before your eyes? I sure do. Organics used in finishing range from plating bath brighteners and surfactants to dye baths used to color anodized aluminum. Modern black dyes used for coloring anodized aluminum can perform equivalent to older dyes, with half the coating thickness. "That's half the anodize time and power typically used throughout the industry!"\*

I would like to share some of the controls used for aluminum dyes. Quality control for the dye baths comes in three levels:

- First level—monitoring temperature, pH and "eyeballing" the concentration.
- Second level—monitoring temperature, pH and using a spectrophotometer to measure the dye-stuff concentration.
- Third level—monitoring temperature, pH and using a spectrophotometer to measure the dye-stuff concentration and the percentage of active dye in the dye bath (coloring efficiency).<sup>1</sup>

Operational temperature of the dye baths has a wide range, but for the most consistent results, you should strive to be within a few degrees of optimum. Control of the pH of the solution is very important because it affects the solubility (amount of dye-stuff dissolved in the bath) of the dye. Generally speaking, less dye can be held in solution as the dye bath becomes more acidic (lower pH—watch out for drag-in). So, even if the dye bath contains the ideal concentration of dye in a low-pH bath, only a

part of the total dye might be in solution and ready to work. In this case, the anodized parts come out lighter in color for the same thickness of coating and time in the dye bath. Different hues or colors are also possible at the wrong pH, because some dyes are made from two or more dyes with different solubilities. If only one of the available dye concentrations changes, the color must change as well. You can save a lot of needless detective work by using a well-maintained pH meter as many times as needed on a daily basis. The change in our response to the different levels for temperature and pH is largely determined by our need for high-quality work.

A "first-level" examination of the dye bath concentration uses your color judgement to "measure" the apparent concentration of dye. Prepare several dye bath standards by using a micro-balance to weigh out a few standards above and below the gram-per-liter concentration you want to maintain. When using a red dye at two grams-per-liter, for example, weigh out samples of 1.5, 1.75, 2.0, 2.25 and 2.5 grams. Dissolve each sample in its own 1,000 mL (1 L) volumetric flask, shake well, adjust the pH and then pipette a 5-mL sample of each into its own 1000 mL volumetric flask. Stronger black dyes may need to be diluted on the order of 2 mL per 1,000 mL. These flasks form the range of standards needed for comparison to an unknown bath. Adds to the dye bath can now be calculated (*e.g.*, the best match of the unknown to the standards is 1.75 grams-per-liter, and the ideal is 2 grams per liter—just multiply 0.25 grams by the number of liters in the bath, which equals the number of grams to add to the bath). For long-term storage, fill a smaller but standard-size bottle with each solution. These

standards will last quite a while, but not forever. Change them as needed (at least once or twice a year).

A spectrophotometer replaces the eyeball to measure the concentration of the dye bath in level two examinations. The sample standards can be prepared using the same methods as level one, but fewer standards are needed because the line generated is straight. Now, to make the transmittance curve (actually a straight line) for the dye in question, dial the spectrophotometer to the wavelength for the dye in question and plot the readings (two points minimum) over the concentration range on graph paper. This will give you the *apparent* concentration of dye in the bath. What is the apparent concentration vs. the active concentration? Not all of the dye in an aged bath is actively capable of becoming incorporated into the anodized aluminum. Anions—such as sulfates, metals (especially aluminum) and molds—can make a percent of the dye inactive. Lab work instructions should look something like the forms\* on the next page.

The third level of quality testing measures the difference between the apparent concentration and the percentage of active dye (the difference is coloring efficiency) in the dye bath. Start with two pieces of 5000-series aluminum (very easy to anodize) the same size and/or known area anodized to a thickness of about 0.7 mil. Holding conditions the same for both, place one panel in a standardized dye bath (we'll use a black dye at 10 g/L for this example) and the other in the unknown working dye bath. By extracting the dye from the panels, we can find active dye concentration. A solution of 12 g/L sodium hydroxide (NaOH extracts the dye) and 7.5 g/L of tartaric acid

\*M. Jozefowicz, Clariant Corp., Charlotte, NC

(protects the dye's hydrogen bonds, read color) is used to wash the panels three times, and the washes are combined in a 1,000-mL volumetric flask. The spectrophotometer is used to measure the percentage differences between the two samples.

Quality and economics are the driving forces for level-three testing. Light fastness is adversely affected if the working dye bath's coloring

efficiency drops below 80 percent of the freshly prepared bath values.

Economics is a double-edged sword—on one hand, you want the dye bath to last forever and on the other, you start to waste the dye replenishment dollars. If an aged bath at 10 g/L has only 2 g/L of dye active, then an addition of 8 g/L may only bring the bath to an activity of 4 g/L, wasting the other 4 g/L of dye

replenishment. All of the push and pull of quality vs. economics has created the “rule of thumb.” Simply put, the rule of thumb says you should drop/decant the dye bath when it reaches a 60-percent coloring efficiency.

The attractive and highly functional aluminum coatings that are produced today are synonymous with *quality*,<sup>1</sup> which will lead to even higher demand for products in the future. P&SF

#### Lab Work Instruction

##### Procedure for Generating a Curve to Determine the Nominal Dye Concentration

**1.0 Purpose:** This procedure can be used to establish the transmittance curve needed in order to test the concentration of the dye baths.

**2.0 Scope:** The determination of dye bath concentration, measured in g/L, is used to maintain consistent quality.

**3.0 Responsibilities:**

**4.0 Special Definitions:**

**5.0 Work Instruction Steps:**

Reagents required:	Dye-stuff DI water
Equipment required:	Micro-balance Spectrophotometer Lab glassware 5 mL pipette 1,000-mL volumetric flask

**6.0 Procedure:** To prepare the transmittance curve for the dye in question—Prepare two or more standard dye solutions at appropriate concentrations and pHs using a microbalance and 1000 mL volumetric flasks, *e.g.*, (in their normal ranges) blacks: 2–10 g/L; other colors: 0–5 g/L. Pipette 5 mL of each solution and dilute to 1,000 mL (1 L) in volumetric flasks. Be certain that the spectrophotometer has been warmed for at least 10 min. Standardize the spectrophotometer at the standard wavelength (different for each dye) per the instruction manual. Rinse and fill the cuvettes with each of the dilutions. Measure and plot the transmittance for each with the original concentration in g/L on the “X” axis. This now becomes the standard curve. Be sure to note on the curve the name of the dye and wavelength used on the curve.

#### Lab Work Instruction

##### Procedure for Determining Dye Concentration

**1.0 Purpose:** This procedure can be used to test the concentration of dye baths. In essence, the nominal concentration relates to the amount of dye-stuff dissolved. It does *not* relate to the effectiveness of that dye.

**2.0 Scope:** The determination of dye bath concentration, measured in g/L, is used to maintain consistent quality.

**3.0 Responsibilities:**

**4.0 Special Definitions:**

**5.0 Work Instruction Steps:**

Reagents required:	Dye-stuff DI water
Equipment required:	Spectrophotometer Lab glassware 5 mL pipette 1000 mL volumetric flask Transmittance curve for the dye in question.

**6.0 Procedure:** Pipette 5 mL of the dye solution into a 1,000-mL volumetric flask and dilute to the mark with DI water. Mix well. Turn on the spectrophotometer and follow the directions of the instruction manual. Set the wavelength for the dye to be measured. Zero the spectrophotometer by rinsing a cuvette and filling it with DI water. Tissue-dry the cuvette and insert it into the sample holder. Push zero to set the baseline. Take a reading by rinsing and filling the cuvette with the dye solution to be tested. Tissue-dry the cuvette and insert it into the sample holder. Press the read button. Read off the transmittance and determine concentration by cross-referencing the reading to the standard transmittance curve.