Buffering Capacity Determination of EASTMAN Color Films, Process ECP-2
Accelerator Bath
ECP-0020-01

INTRODUCTION
The buffering capacity of an Eastman Color Films, Process ECP-2 accelerator bath is determined by adjusting a 100.0 mL aliquot of sample to pH 3.500 (± 0.005) using 1.0N sulfuric acid and then manually titrating to pH 6.000 (± 0.005) using standardized 1.00N sodium hydroxide solution. The volume of sodium hydroxide used is related to the amount of glacial acetic acid in the sample (buffering capacity) by a linear regression equation.

Repeatability and reproducibility studies were performed for both “fresh” and “seasoned” solutions. The linear regression equation generated from individual “seasoned” solutions were different from each other and significantly different from those generated from “fresh” solutions, due to the differing amounts of seasoning products contributing to the buffering capacity. For this reason, and since it is most representative of data generated by multiple analysts, the linear regression equation generated from the reproducibility study of a “fresh” accelerator bath solution was chosen for inclusion in this method.

Use of this method requires handling potentially hazardous chemicals. Consult the Material Safety Data Sheet for each chemical before use. MSDS’s are available from your chemical supplier.

PRECISION AND BIAS

Repeatability Standard Deviation (1s_r) & 95 Percent Confidence Estimate (not including bias)
Repeatability standard deviation is an estimate of the variability one trained analyst should be able to obtain under favorable conditions (analyzing a sample, with one instrument, within one day).

The 95 percent confidence estimate (calculated using the repeatability standard deviation) around a single test will include the mean value 95 percent of the time.

To obtain the repeatability data, the analyst who developed this method performed five (5) replicates on each of the following solutions:

1. A “fresh” accelerator bath prepared with components at their respective aim “working tank” concentrations, minus the glacial acetic acid component.
2. The same “fresh” solution as in number 1, above, reanalyzed after making standard additions of 3.0 mL/L, 7.0 mL/L and 10.0 mL/L glacial acetic acid.
3. A “seasoned” accelerator bath analyzed, as received.
4. The same “seasoned” solution as in number 3, above, reanalyzed after making standard additions of 3.0 mL/L, 7.0 mL/L and 10.0 mL/L glacial acetic acid.

“Fresh” tank solutions
Based on 20 determinations by a single analyst, the Standard Error Estimate (Sy.x) for an individual determination of a “fresh” ECP-2 accelerator bath is 0.08 mL/L glacial acetic acid, and the 95 percent confidence at the midpoint of the line is ± 0.18 mL/L glacial acetic acid.

“Seasoned” tank solutions
Based on 20 determinations by a single analyst, the Standard Error Estimate (Sy.x) for an individual determination of a “seasoned” ECP-2 accelerator bath is 0.03 mL/L glacial acetic acid, and the 95 percent confidence at the midpoint of the line is ± 0.07 mL/L glacial acetic acid.

Reproducibility
Customer Standard Deviation, 1s_c & 95 Percent Confidence Estimate (not including bias)
The reproducibility or customer standard deviation (1s_c) is an estimate of the variability a customer could expect when submitting a sample to any Photoprocessing Quality Services laboratory, where any trained analyst could test the sample using any instrument on any day.

The 95 percent confidence estimate (calculated using the customer standard deviation) around a single test result will include the mean value 95 percent of the time.

Four analysts analyzed eight “fresh” accelerator bath samples, on two different days. Duplicate analyses were performed on each sample, on each of the two days. These samples were prepared identically to the “fresh” solutions described in the repeatability section above.

Two analysts analyzed eight “seasoned” accelerator bath samples, on two different days. Duplicate analyses were performed on each sample, on each of the two days. These samples were prepared identically to the “seasoned” solutions described in the repeatability section.

“Fresh” tank solutions
Based on 64 determinations by four analysts, the Standard Error Estimate (Sy.x) for an individual determination of a “fresh” ECP-2 accelerator bath is 0.06 mL/L glacial acetic acid, and the 95 percent confidence at the midpoint of the line is ± 0.13 mL/L glacial acetic acid.

Based on chemical theory and measurement of a “fresh” tank solution prepared at aim concentrations, this method is believed to provide an accurate measure of the buffering capacity of an ECP-2 accelerator bath.

<table>
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<th>Process</th>
<th>ECP-2</th>
<th>ECP-2D</th>
<th>VNF-1/LC</th>
<th>RVNP</th>
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“Seasoned” tank solutions
Based on 31 determinations by two analysts, the Standard Error Estimate (Sy.x) for an individual determination of a “seasoned” ECP-2 accelerator bath is 0.04 mL/L glacial acetic acid, and the 95 percent confidence at the midpoint of the line is ± 0.09 mL/L glacial acetic acid.

Based on chemical theory and measurement of a “seasoned” tank solution, this method is believed to provide an accurate measure of the buffering capacity of ECP-2 accelerator bath.

APPARATUS
All volumetric glassware should meet all “Class A” specifications, as defined by American Society for Testing and Materials (ASTM) Standards E 287, E 288, and E 969, unless otherwise stated.

- 100-mL pipette
- 25-mL burette
- Beakers, 250-mL
- Dual channel pH meter, e.g. ORION EA 940 or equivalent.
- pH electrode, e.g. Corning Rugged Bulb pH electrode 476024 or equivalent
- Reference electrode, e.g. Corning 476002, reference, ceramic junction, calomel or equivalent
- Magnetic stirrer and magnetic stir bar

REAGENTS
Use ACS Reagent Grade reagents unless otherwise specified.

- pH 4 phthalate calibrating buffer (prepare from reagent grade chemicals, or purchase from vendor)
- pH 7 equimolar phosphate calibrating buffer (prepare from reagent grade chemicals, or purchase from vendor)
- pH 3.63 tartrate low pH control buffer (prepare from reagent grade chemicals, or purchase from vendor)
- 1.0 N Sulfuric Acid (H₂SO₄)
- 1.00 N (± 0.02 N) Sodium Hydroxide (NaOH), standardized to 4 decimal places
- Water, Type I Reagent – This method was developed, and the resulting statistical data were obtained using reagent water equivalent to purer than Type I Grade, as defined in ASTM Standard D 1193. Other grades of water, e.g., reverse osmosis (RO), demineralized, or distilled water, may give equivalent results, but the effects of water quality on method performance have not been studied.

PROCEDURE
Preparation of the meter
1. Follow method ULM-191-2 (or any subsequent pH method) for making pH measurements below pH 7.
   a. Adjust the temperature of the buffers.
   b. Calibrate the meter with the pH 4 and pH 7 buffers.
   c. Check the electrode calibration with pH 3.63 tartrate low control buffer.

Titration of Sample
1. Pipette (wipe the pipette before leveling) 100.0 mL of sample into a 250 mL beaker containing a magnetic stir bar.
2. Place beaker on magnetic stirrer
3. Immerse the electrode assembly in the sample solution and stir without splashing.
4. Adjust the pH of the sample to approximately pH 3.5, using 1.0 N sulfuric acid.
5. Add, from a pipette or burette, 1.00 N sodium hydroxide to attain a pH 3.500 (3.495 - 3.505). This volume does not have to be measured.

Caution
Stir the solution rapidly without splashing. Do not rinse the sides of the beaker with reagent water because dilution will affect the results.
6. Using a 25-mL burette, titrate the sample to pH 6.000 (5.995 - 6.005) with 1.00 N sodium hydroxide. Record the volume of titrant used.
   Note: If the titration exceeds pH 6.000, discard the sample and repeat the analysis.
7. Remove the sample and rinse the electrode assembly with reagent water. If rinsing does not completely remove sample deposits, wipe the assembly with a cleansing tissue and re-rinse. Replace the assembly in pH 7 buffer for storage.

Calculation
Buffering Capacity (mL/L glacial acetic acid) = m(x) + b

Where:

- m = Slope of the regression line
  \[
  \frac{[\text{mL/L glacial acetic acid}]}{\text{mL 1.00 N NaOH}}
  \]
- x = Volume of titrant consumed (mL 1.00 N NaOH)
- b = Intercept of regression line (mL/L glacial acetic acid)

Example
Buffering Capacity = 0.648 (7.47) – 0.311
= 4.53 mL/L glacial acetic acid