

KODAK Q-LAB Process Monitoring Service

PROCESS CONTROL HANDBOOK • Z-6

Reversal Bath

This step prepares the film for the color-developer step. In the reversal bath, a chemical reversal agent is absorbed into the emulsion. *No chemical reaction takes place until the film enters the color developer.*

FUNCTION

The reversal agent enters the emulsion and prepares the unexposed, undeveloped silver halide for the chemical reversal that occurs in the color developer.

COMPONENTS

Reversal Agent:

KODAK Reversal Agent, RA-4 (stannous chloride)

This reversal agent is absorbed into the emulsion in the reversal bath. Then as film enters the color developer, the reversal agent is activated by the high pH of the color developer and chemically exposes the undeveloped silver halide. The chemical reaction that occurs in the color developer is—

 $AgX + stannous chloride \rightarrow Ag*X + stannic chloride$

AgX = silver halide

 Ag^*X = chemically exposed silver halide

The KODAK Reversal Agent Test Kit, Process E-6, enables you to measure the concentration of reversal agent in your Process E-6 reversal bath and detect changes in the concentration before they affect the film you process. For example, when the reversal-agent concentration is high, the process will drift blue; when the concentration is low, the process will drift yellow. When the reversal-agent concentration is extremely low, the color balance of upper-scale densities and D-max will drift green. Instructions for using this kit are given on page 9-3.

Preservative:

Para-aminophenol

Para-aminophenol acts as a preservative to protect the reversal agent from oxidation.

Buffer:

Acetic acid

Acetic acid helps maintain the pH of the reversal bath. It also helps control the activity of the reversal agent.

Fungicides:

Acetic acid

Additional fungicide

These fungicides help prevent biological growth (slime).

Sensitometric Effects of Reversal-Agent Concentration

The following tables list the effects on processed film of high and low reversal-agent concentration.

HIGH CONCENTRATION

Solution Component	Sensitometric Effect
KODAK Reversal Agent, RA-4	Color balance drifts blue

LOW CONCENTRATION

Solution Component	Sensitometric Effect
KODAK Reversal Agent,	Color balance drifts yellow
177-4	Color balance of upper-scale densities and D-max drifts green when concentration is extremely low



SPECIFICATIONS

Parameter	Aim	Tolerance	Acceptable Range	Plot Parameter
Time	2 minutes	±15 seconds	1 to 4 minutes	
Temperature	75 to 103°F (24 to 39.4°C)	_	_	
Replenishment Rate	100 mL/ft ² (1076 mL/m ²)	± 15 mL/ft ^{2*} (± 160 mL/m ²)*	_	Х
Specific Gravity				
Seasoned Tank Solution	1.005 at 80°F (27°C) 1.002 at 100.4°F (38°C)	± 0.003	_	x
Fresh Tank Solution†	1.004 at 80°F (27°C) 1.001 at 100.4°F (38°C)	± 0.003	_	
Replenisher	1.006 at 80°F (27°C) 1.003 at 100.4°F (38°C)	± 0.003	_	
Reversal-Agent Concentration				
Seasoned Tank Solution	1.20 g/L	± 0.2 g/L	_	X
Fresh Tank Solution†	1.20 g/L	± 0.2 g/L	_	
Replenisher	1.65 g/L	± 0.2 g/L		

^{*}For optimum performance, maintain your replenishment rate to within ± 5% of the specified aim (±15% tolerance allows for measurement "noise").

PREPARING A FRESH TANK SOLUTION

To prepare a fresh tank solution that will give results similar to those produced by a seasoned tank solution, follow the appropriate mixing instructions given below. (These instructions are different from those provided with the chemicals.) Following either procedure will help you obtain results closer to those produced at optimum process levels.

Note: These instructions are for mixing solutions from KODAK PROFESSIONAL Reversal Bath and Replenisher, Process E-6 and E-6AR (5-gallon flexible container).

From Mixed Replenisher Solution

For each litre of tank solution, mix 800 mL of replenisher with 200 mL of water.

From Concentrate

For each litre of tank solution, mix 40 mL of concentrate with 960 mL of water.

Note: When you prepare the solution in either of these ways, the specific gravity and the reversal-agent concentration will be within the tolerances for a fresh tank solution.

[†]See the mixing instructions at the right. Plot the specific gravity and the reversal-agent concentration of the new mix on Form Y-35. Note "NEW REVERSAL-BATH TANK MIX" on Form Y-33.

USING THE KODAK REVERSAL AGENT TEST KIT, PROCESS E-6

Use this kit to measure the concentration of reversal agent in your Process E-6 reversal bath. Read these instructions before you run tests so that you are familiar with the procedure. *Use only distilled water to dilute the reversal-bath sample as required in the procedure.* (Your results may be unreliable if you use other types of water [e.g., demineralized, deionized, tap, etc].)

- 1. Fill both viewing tubes to the top line with distilled water.
- 2. Place one tube in the left opening of the color comparator box. Use the other tube to prepare your sample.
- 3. Remove 0.1 mL of reversal-bath solution with the TenSette Pipet and add it to the second viewing tube.
- 4. Add 0.5 mL of Molybdate 3 Reagent to the viewing tube.
- 5. Cap the tube with a stopper and invert it 3 times. **Do not** shake the tube to mix the solution.
- 6. Place the tube in the right opening of the color comparator box.
- 7. After 5 minutes, compare the sample (right viewing hole) with the color disc (left viewing hole) to determine the reversal-agent concentration. Read the result in the scale window.
- 8. Calculate and plot your variation from aim on Form Y-35; see "Using Forms Y-34, Y-35, and Y-36" on page 4-13 for more information. Check the plots of reversal-agent concentration for outliers, shifts, trends, and cycling to determine the state of control. If you detect an out-of-control condition, refer to the diagnostic charts on pages 9-9 and 9-10.

 Discard the solution from both tubes and rinse the tubes and stoppers thoroughly with distilled water. Remove the tip from the pipet and discard it.

Using the KODAK Reversal Agent Test Kit to Monitor Reversal-Bath Uniformity

In addition to using the test kit to ensure that the reversal-agent concentration of your tank and replenisher solutions is on aim, you can use it to check solution uniformity. To check uniformity, measure the reversal-agent concentration of the solution at the bottom of the tank and at the top of the tank. The measurements should be the same or within 0.15 g/L for good solution uniformity.

The following conditions can cause differences in concentration at the top and the botton of unrecirculated tanks:

- 1. The replenisher input valve is at or close to the bottom of the tank, and carryover from the first wash enters the top of the tank.
- 2. The replenisher provided by in-line dilution or blender systems is not thoroughly mixed with water before it enters the tank.
- 3. Pre-mixed replenisher solution is overconcentrated or too dilute.
- 4. Pre-mixed replenisher solution is not thoroughly mixed. The replenisher concentrate and water in the solution can separate (or stratify). When replenisher solutions are thoroughly mixed, they will not stratify.
- 5. The fresh tank solution is not thoroughly mixed. The concentrate and water in the solution can separate (or stratify). When fresh tank solutions are thoroughly mixed, they will not stratify.

To determine if any of these conditions is affecting the uniformity of your tank solution, use the KODAK Reversal Agent Test Kit, Process E-6, to measure the reversal-agent concentration of a solution sample from the top of the tank and another sample from the bottom of the tank. Then subtract the concentration of the sample from the concentration of the sample from the bottom. If the difference is 0.15 g/L or less, *do not* take any corrective action. If the difference is 0.16 or more, follow the steps below.

- 1. Check that your replenisher is thoroughly mixed before it enters the tank (for all types of replenishment systems). If it is not thoroughly mixed, take corrective action (see pages 9-9 and 9-10).
- 2. To maintain solution uniformity, agitate the solution periodically with a mixing paddle or stirring rod, or recirculate the solution for approximately 5 to 10 minutes by turning on the recirculation pumps. You can agitate the solution manually or you can set the timer on your recirculation pumps so that they will automatically turn on at regular intervals for short periods during the day. Determine the frequency of agitation/recirculation by measuring the reversal-agent concentration at the top and the bottom of the tank at least once a week. The frequency of agitation/ recirculation will also depend on the amount of film you process (e.g., the less film you process, the more frequently you will have to agitate/recirculate the solution; the more film you process, the less frequently you will have to agitate/ recirculate the solution).

Note: Do not agitate or recirculate the solution continuously, or use agitation techniques that mix air into the solution.

Two examples of checking solution uniformity are given below.

Example 1

Measurement:

at the top of the tank = 1.15 g/Lat the bottom of the tank = 1.56 g/L

Difference = 1.56 - 1.15 = 0.41 g/L

The difference is greater than 0.15 g/L, so the operator checks the reversal-agent concentration of the replenisher.

Measurement:

at the top of the replenisher tank = 1.65 g/Lat the bottom of the replenisher tank = 1.62 g/L

Difference = 1.65 - 1.62 = 0.03 g/L

The difference is less than 0.15 g/L; therefore, the replenisher is mixed sufficiently.

The operator does not find any other problems, so he must agitate or recirculate the tank solution regularly to maintain solution uniformity. The operator sets the recirculation pumps on a timer; the pumps turn on for 30 minutes at start-up and for 10 minutes every 2 hours during the day.

After the solution is recirculated at the pre-set intervals for one day, the operator rechecks the reversal-agent concentration at the top and the bottom of the tank.

Measurement:

at the top of the tank = 1.27 g/Lat the bottom of the tank = 1.34 g/L

Difference = 1.34 - 1.27 = 0.07 g/L

The difference is less than 0.15 g/L; therefore, the tank solution is mixed sufficiently. The operator measures the reversal-agent concentration of the solution **at the top of the tank** daily for process control, and measures the concentration of the solution at the bottom of the tank once a week to ensure solution uniformity.

Example 2

Measurement:

at the top of the tank = 1.25 g/Lat the bottom of the tank = 2.01 g/L

Difference = 2.01 - 1.25 = 0.76 g/L

The difference is greater than 0.15 g/L, so the operator checks the reversal-agent concentration of the replenisher.

The replenisher is provided by an in-line dilution system. The operator takes a sample of the replenisher as it enters the tank. The appearance of the sample indicates that the concentrate and water are not thoroughly mixed. Measurements of the reversal-agent concentration confirm poor mixing (i.e., samples from the top and the bottom of the sample vessel are tested). The operator must solve the poor mixing problem (e.g., he must change the type of funnel, the point where the concentrate and water mix, etc).

After adjusting the replenishment system, the operator rechecks the reversal-agent concentration of the tank solution.

Measurement:

at the top of the tank = 1.26 g/Lat the bottom of the tank = 1.48 g/L

Difference = 1.48 - 1.26 = 0.22 g/L

The difference is greater than 0.15 g/L; therefore, the operator must agitate or recirculate the solution. The operator agitates the solution once per hour with a mixing paddle.

After a few days, the operator rechecks the reversal-agent concentration of the tank solution.

Measurement:

at the top of the tank = 1.24 g/Lat the bottom of the tank = 1.23 g/L

Difference = 1.24 - 1.23 = 0.01 g/L

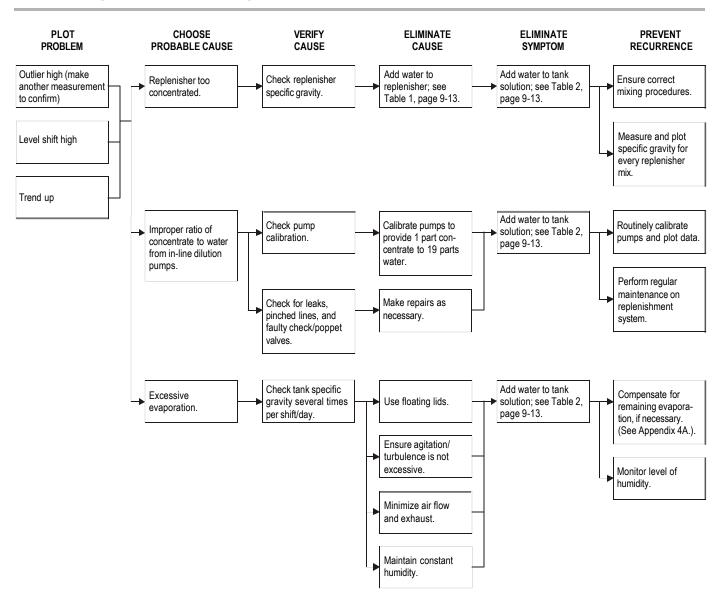
The difference is less than 0.15 g/L; therefore, the solution is thoroughly mixed. The operator measures the reversal-agent concentration of the solution **at the top of the tank** daily for process control, and measures the concentration of the solution at the bottom of the tank once a week to ensure solution uniformity.

DIAGNOSTIC CHARTS

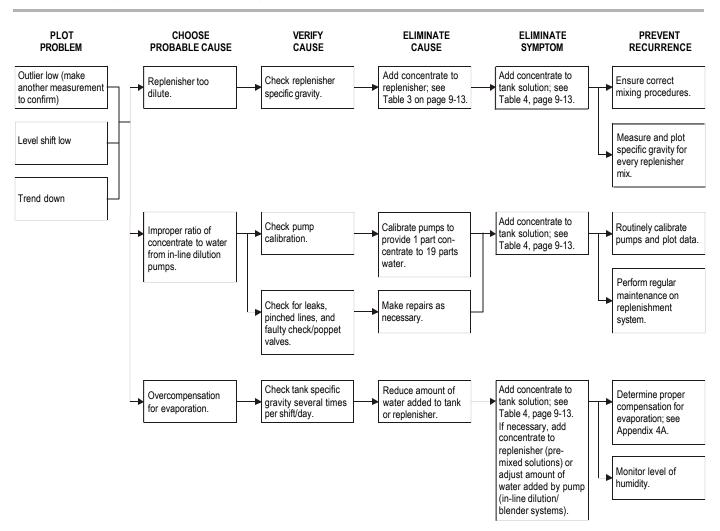
The diagrams on pages 9-5 to 9-11 provide you with a step-by-step approach to diagnosing process problems; they include the most common causes of problems. They are organized according to the appearance of your control plots for the key

parameters for reversal bath. The recommendations in the charts will help you correct outliers (data on or outside the tolerance lines), level shifts, trends, and cycling. For more information on evaluating control-chart plots, see *Process Control—A Better Way*, Section 1.

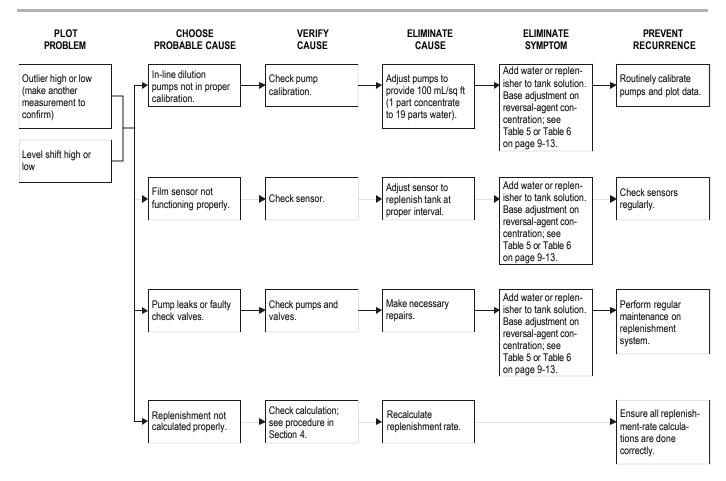
REVERSAL BATH—SPECIFIC GRAVITY— Outlier High, Level Shift High, Trend Up



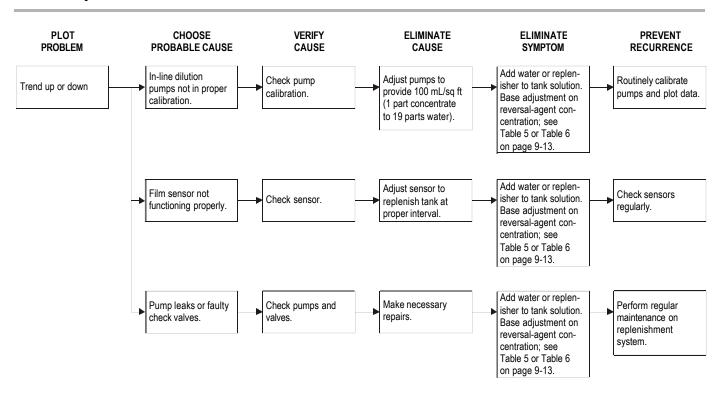
REVERSAL BATH—SPECIFIC GRAVITY— Outlier Low, Level Shift Low, Trend Down



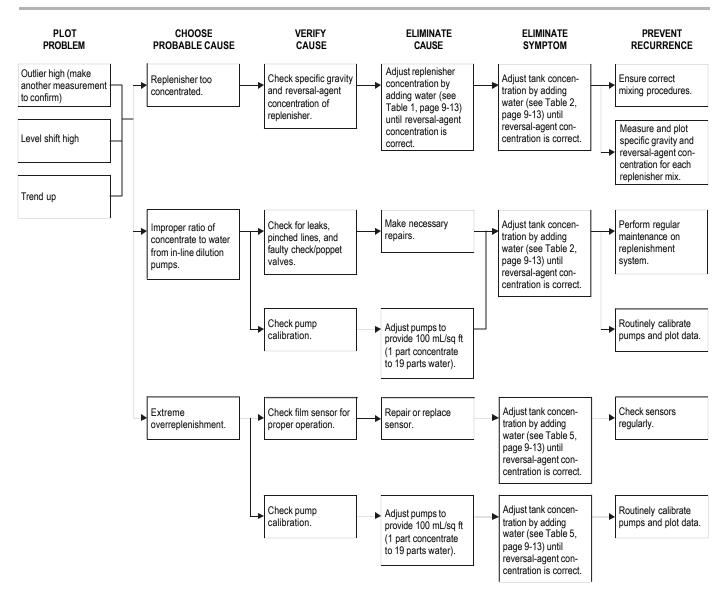
REVERSAL BATH—REPLENISHMENT RATE— Outlier High or Low, Level Shift High or Low



REVERSAL BATH—REPLENISHMENT RATE— Trend Up or Down

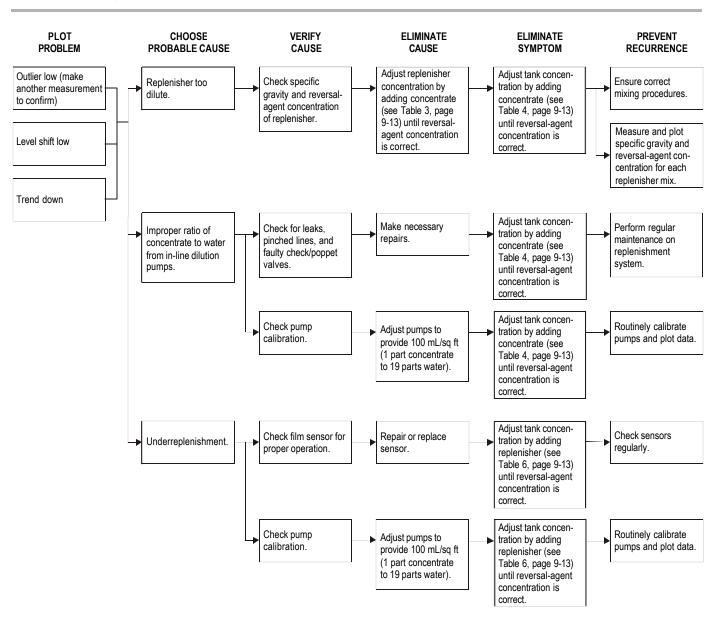


REVERSAL BATH—REVERSAL-AGENT CONCENTRATION—Outlier High, Level Shift High, Trend Up

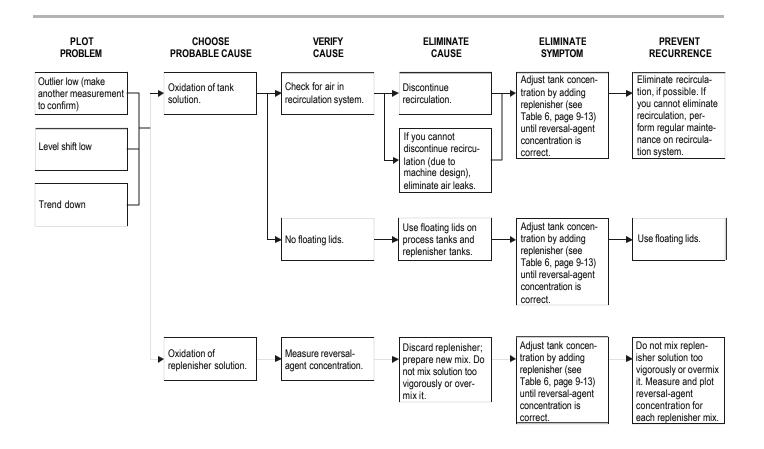


Note: If the reversal-agent concentration is still **high** after you have taken corrective action to eliminate all probable causes, you must use a more dilute replenisher; see the information on page 9-14.

REVERSAL BATH—REVERSAL-AGENT CONCENTRATION—Outlier Low, Level Shift Low, Trend Down



(Continued on next page)



Note: If the reversal-agent concentration is still **low** after you have taken corrective action to eliminate all probable causes, you must increase your replenishment rate; see the information on page 9-15.

REVERSAL BATH—Cycling

SPECIFIC GRAVITY

Look for a relationship between cycling and events that occur regularly:

- Start-up on Monday mornings
- Shift/operator changes
- Use of new replenisher mixes (check mixing procedures)
- · Intermittent use of air conditioning

Check that you are compensating for evaporation correctly (see the procedure given in Appendix 4A, "Compensating for Evaporation"). Make specific-gravity measurements every 2 to 4 hours for 2 or 3 days to determine the cause of cycling.

Make corrections or adjustments to eliminate the cause of cycling.

REPLENISHMENT RATE

Look for a relationship between cycling and events that occur regularly:

- Shift/operator changes
- Power-supply fluctuations in the lab during the day/week
- Fluctuations in machine utilization
 Make corrections or adjustments to eliminate the cause of cycling.

REVERSAL-AGENT CONCENTRATION

Look for a relationship between cycling and events that occur regularly:

- Shift/operator changes
- Measurements made by different operators
- Use of new replenisher mixes (check mixing procedures)
- Use of new tank mixes

Make corrections or adjustments to eliminate the cause of cycling.

TABLE 1
Addition of Water
to Correct for Overconcentration of
Replenisher Solution

Specific Gravity Measured at 80°F (27°C)	mL of Water per Litre of Replenisher Solution	Specific Gravity Measured at 100.4°F (38°C)	mL of Water per Litre of Replenisher Solution
1.006	0	1.003	0
1.007	100	1.004	100
1.008	282	1.005	182
1.009	250	1.006	250
1.010	308	1.007	308
1.011	357	1.008	357
1.012	400	1.009	400

TABLE 2
Addition of Water
to Correct for *Overconcentration* of
Seasoned Tank Solution

Specific Gravity Measured at 80°F (27°C)	mL of Water per Litre of Tank Solution	Specific Gravity Measured at 100.4°F (38°C)	mL of Water per Litre of Tank Solution
1.005	0	1.002	0
1.006	111	1.003	111
1.007	200	1.004	200
1.008	273	1.005	273
1.009	333	1.006	333
1.010	385	1.007	385
1.011	429	1.008	429

TABLE 3
Addition of Reversal-Bath Concentrate to Correct for *Underconcentration* of Replenisher Solution

Specific Gravity Measured at 80°F (27°C)	mL of Water per Litre of Replenisher Solution	Specific Gravity Measured at 100.4°F (38°C)	mL of Water per Litre of Replenisher Solution
1.006	0	1.003	0
1.005	8	1.002	8
1.004	15	1.001	15
1.003	22	1.000	22
1.002	29	0.999	29
1.001	37	0.998	37

TABLE 4
Addition of Reversal-Bath Concentrate
to Correct for *Underconcentration* of
Seasoned Tank Solution

Specific Gravity Measured at 80°F (27°C)	mL of Water per Litre of Tank Solution	Specific Gravity Measured at 100.4°F (38°C)	mL of Water per Litre of Tank Solution
1.005	0	1.002	0
1.004	7	1.001	7
1.003	15	1.000	15
1.002	22	0.999	22
1.001	29	0.998	29
1.000	36	0.997	36

TABLE 5
Addition of Water to Correct for
High Reversal-Agent Concentration
Due to Overreplenishment of
Seasoned Tank Solution

Reversal Agent Concentration (g/L)	mL of Water per Litre of Tank Solution
1.65	273
1.60	250
1.55	226
1.50	200
1.45	172
1.40	143
1.35	111
1.30	77
1.25	40

TABLE 6
Addition of Replenisher to Correct for Low Reversal-Agent Concentration in Seasoned Tank Solution

Reversal Agent Concentration (g/L)*	mL of Replenisher per Litre of Tank Solution
0.75	500
0.80	470
0.85	437
0.90	400
0.95	357
1.00	308
1.05	250
1.10	182
1.15	100

^{*} If the reversal-agent concentration is lower than 0.75, discard the tank solution and prepare a new reversal bath according to the mixing instructions on page 9-2.

High Reversal-Agent Concentration

Important: Use the information below in conjunction with the diagnostics on page 9-9.

If the reversal-agent concentration is still *high* after you have taken corrective action to eliminate all probable causes (see page 9-9), you must use a more dilute replenisher. For pre-mixed solutions, you must adjust the amount of concentrate and water; in blender or in-line dilution systems, decrease the amount of concentrate added by the concentrate pump and increase the amount of water added by the water pump.

Note: High reversal-agent concentration is not a common problem. Recheck all measurements and probable causes **before** you modify your replenisher or adjust your replenishment system.

Pre-Mixed Solutions: To dilute your replenisher, decrease the amount of concentrate and increase the amount of water **by the same amount.** To determine the adjustments, use this calculation:

$$\frac{\text{Reversal-agent}}{\text{Reversal-agent concentration}} \cdot \frac{\text{Amount of}}{\text{x}} \quad \frac{\text{Amount of concentrate}}{\text{in mix (mL/L)}} = \frac{\text{Amount of concentrate}}{\text{decrease and amount of water increase (mL/L)}}$$

For example, if your reversal-agent concentration is 1.5 g/L, the aim is 1.2 g/L, and the amount of concentrate in your mix is 50 mL/L, calculate the adjustments as follows:

$$\frac{1.5 \text{ g/L} - 1.2 \text{ g/L}}{1.5 \text{ g/L}} \times 50 \text{ mL/L} = 10 \text{ mL/L}$$

You should decrease the amount of concentrate **and** increase the amount of water by 10 mL/L:

Adjusted volume of concentrate in mix =
$$50 \text{ mL/L} - 10 \text{ mL/L} = 40 \text{ mL/L}$$

Adjusted volume of water in mix = $950 \text{ mL/L} + 10 \text{ mL/L} = 960 \text{ mL/L}$

In-Line Dilution or Blender Systems: To dilute your replenisher, decrease the amount of concentrate added by the concentrate pump and increase the amount of water added by the water pump **by the same amount.** To determine the adjustments, use this calculation:

Reversal-agent concentration - Aim		Amount of concentrate		Amount of concentrate
Reversal-agent concentration	Х	added by pump (mL/cycle)	=	decrease and amount of water increase (mL/cycle)

For example, if your reversal-agent concentration is $1.5\,\mathrm{g/L}$, the aim is $1.2\,\mathrm{g/L}$, and the amount of concentrate added per pump cycle is $5\,\mathrm{mL}$, calculate the adjustment as follows:

$$\frac{1.5 \text{ g/L} - 1.2 \text{ g/L}}{1.5 \text{ g/L}} \text{ x 5 mL/L} = 1 \text{ mL/cycle}$$

You should decrease the amount of concentrate added by the concentrate pump \boldsymbol{and} increase the amount of water added by the water pump by 1mL/cycle:

Adjusted volume of concentrate/pump cycle
$$= 5 \text{ mL} - 1 \text{ mL} = 4 \text{ mL}$$

Adjusted volume of water/pump cycle $= 95 \text{ mL} + 1 \text{ mL} = 96 \text{ mL}$

Low Reversal-Agent Concentration

Important: Use the information below in conjunction with the diagnostics on page 9-10.

If the reversal-agent concentration is still *low* after you have taken corrective action to eliminate all probable causes (see page 9-10), you must increase your replenishment rate. To determine the new replenishment rate, use the following calculation or the table below.

Note: Do not alter the ratio of concentrate to water; maintain the ratio at 1 to 19.

Reversal-Agent Concentration (g/L)	New Replenishment Rate in mL/sq ft and mL/sq M (concentrate/water)	
(3" /	mL/sq ft	mL/sq M
0.75	200 (10/190)	2153 (108/2045)
0.80	189 (9/180)	2034 (97/1937)
0.85	178 (9/169)	1915 (97/1818)
0.90	167 (8/159)	1797 (86/1711)
0.95	156 (8/148)	1678 (86/1592)
1.00	144 (7/137)	1549 (75/1474)
1.05	133 (7/126)	1313 (75/1356)
1.10	122 (6/116)	1431 (65/1248)
1.15	111 (6/105)	1194 (65/1129)
1.20	100 (5/95)	1076 (54/1022)

Note: Volumes are rounded to the nearest mL to simplify measurement.