



Environment

I N F O R M A T I O N F R O M K O D A K

Recovering Silver from Photographic Processing Solutions



THE PHOTOGRAPHIC PROCESS

Silver halide crystals are a major component of photographic film or paper. The chemistry of silver halide image capture and the subsequent chemical changes that take place during processing represent the most efficient form of high-quality image capture known today. Although electronics are changing the frontier of imaging science, they will probably never replace all silver halide products, but will instead complement the imaging technology.

As silver halide films and papers undergo the photographic processing steps, key changes take place in the form and location of the silver. In color processes virtually 100% of the silver comes out of the film or paper and remains in one or several of the processing solutions. With black and white processes the silver is split between remaining in the processed image and migrating into a photographic processing solution.

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Kodak's health, safety, and environmental publications are available to help you manage your photographic processing operations in a safe, environmentally sound and cost-effective manner. This publication is part of a series of publications on silver management designed to help you optimize silver recovery. It will help you understand available silver-recovery techniques.



Silver is recovered from the photographic processing solutions for two reasons—economic and regulatory (compliance). The table below presents an overview of the three most common types of silver recovery.

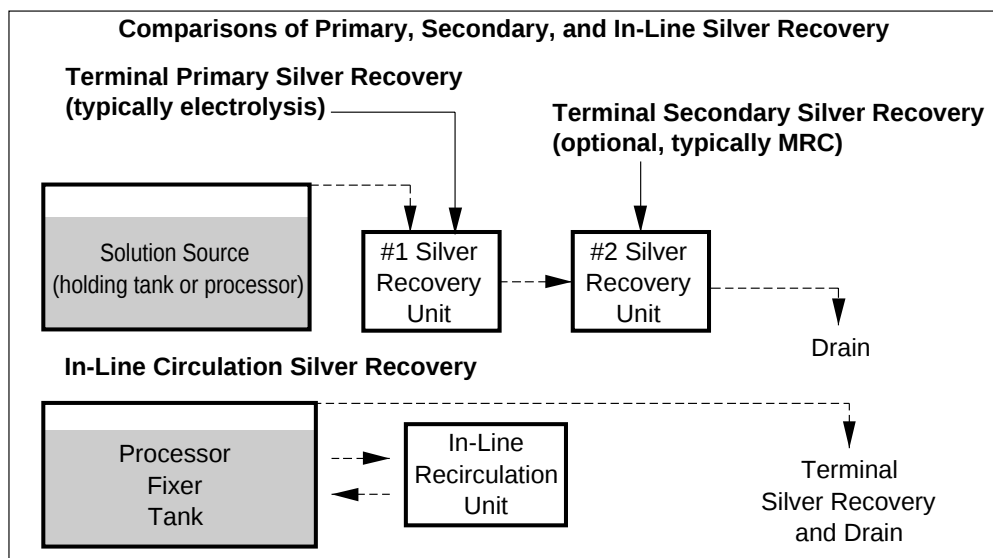
The technique you choose and the method you use depends on a number of factors including:

- Whether you are recovering the silver for economic reasons or to comply with stringent discharge codes
- The volume of solution to be treated
- Your budget

Comparison of Silver-Recovery Techniques

	Electrolysis	Metallic Replacement Cartridges	Precipitation
Recovery Efficiency	> 90%	> 95%	> 99%
Advantages	Can produce > 90% pure silver	Initial costs are relatively inexpensive; if used for compliance; ongoing costs can be high.	Consistent low silver concentration. Easy to monitor performance.
Disadvantages	Relatively high final silver concentration; may require secondary recovery	Difficult to know when to replace; discharges iron; limited by some sewer codes. In some cases, not consistent.	Not available for all processes
Applications	All photographic processing facilities except very small facilities	All photographic processing facilities	Very small and large facilities

The diagram below details the differences between terminal primary silver recovery, terminal secondary silver recovery, and in-line recirculation silver recovery.



Silver Concentrations in Various Overflow Solutions

The amount of silver found in overflow photographic processing solutions varies extensively from solution to solution. The table at the right shows some of shows the ranges that are found in common processing cycles.

Solution	KODAK Process	Silver Concentration (mg/L)
Bleaches (Washless Process)	C-41 Minilab	5 – 200
Bleach-Fix	RA-4, NR	6,000 – 10,000
	RA-4, NT	3,000 – 4,000
Fixer	Black-and-White	3,000 – 7,000
	E-6, Tank 1	5,000 – 12,000
	E-6, Tank 2	1,000 – 3,000
	C-41	5,000 – 12,000
	RA-4, separate bleach and fixer	2,000 – 10,000
Low Flow Washes	RA-4, Tank 1 Low Flow	1,000 – 3,000
Stabilizers (Washless Process)	C-41/RA-4 Minilab	100 – 1,000

ELECTROLYSIS

Electrolytic silver recovery is, in many cases, the most cost-effective and efficient way to remove silver from silver-rich photographic processing solutions. However, silver concentrations out of the electrolytic unit usually are in the range of 200 – 800 mg/L and some type of secondary or *tailing* recovery technique is necessary in order to meet stringent regulatory discharge limits. Trying to “push” most electrolytic equipment to treat solutions lower than about 200 mg/L:

- Is usually not energy or time efficient
- May generate noxious by-products
- Requires larger-than-normal cell size (high cost)

You can treat almost all fixers, bleach-fixes, low flow washes, and combinations by electrolysis.

TYPES OF UNITS

There are three basic types of electrolysis cells:

- Fixed cathode
- Rotating cathode (or some well-designed fluid pump through cells)
- In-line recirculation (either fixed or rotating cathode cells with the installation of special electronic controllers)

Fixed cathode units generally are used for low-iron solutions such as Process C-41 or black-and-white fixers. Rotating cathode cells are common for solutions that contain a high level of iron chelate such as color paper bleach-fixes. The iron in these solutions produces an electrochemical reaction that competes with the reduction of silver at the cathode; increased fluid movement across the cathode is necessary to produce a higher plating efficiency to offset this competition and provide an adequate desilvering rate. The disadvantage of rotating cathode units is that they contain more parts, which adds to their cost and maintenance.

In-line electrolytic fixer recirculation or “closed-loop fixer recirculation,” is common in certain black-and-white and Process C-41 color applications. Either kind of electrolytic cell will work. The solution is continually desilvered and returned to the processor tank. The fixer tank is maintained at a much lower silver level than “normal” (0.5–1.0 g/L versus as much as 6.0 g/L or higher). The result is a reduced amount of silver being carried into the wash tank in the next processor section.

Therefore, you don’t need to be concerned with silver concentration in the wash discharge. The replenishment rate of the fixer may also be reduced by as much as 50%.

A colorimetric silver test is an excellent tool used to adjust and monitor in-line electrolytic recirculation equipment to ensure optimum performance. Too much silver being removed by the unit may result in solution sulfiding and deposits on the film or paper; too little silver being removed defeats the purpose of the equipment.

SELECTING THE PROPER UNIT

Two of the major factors that determine the correct electrolytic unit for a photographic processing facility to use are the type of solution(s) being treated and the daily volume requiring treatment. If you are going to treat large quantities of bleach-fix, low-flow wash following bleach-fix tanks, or E-6 fixer, you should use a rotating cathode unit or “pump-through” unit specially designed for this application. If you are going to treat only low iron solutions such as black-and-white or Process C-41 fixers, you can use a simpler (and usually less expensive) fixed cathode unit. Always pay careful attention to the manufacturer’s advertised rate of silver removal. Although occasionally optimistic, these specifications represent the best approximation of how much silver can be removed per unit time and therefore are key in determining the size of cell necessary to meet a facility’s needs. The examples on the right provide a method of calculation to size an electrolytic unit to your operation.

Sizing an Electrolytic Silver Recovery (ESR) Unit

YOU NEED TO KNOW:

NATURE OF EFFLUENT	
If Effluent is:	Then B =
• Black-and-White Fixer	4
• C-41 or E-6 Fixer	3
• Bleach-Fix + Low Flow Wash, C-41, and/or E-6	2
• Bleach-Fix alone	1.5

A = Gallons per day of silver-rich solution.

B = Nature of the Solution (chart at left).

C = Silver Content (grams/litre) in the Solution.

D = Time available (daily, in hours) to run ESR.

$$\text{Determine the Required Amperage (amps)} = \left(\frac{A \times 3.785 \times C}{B \times D} \right)$$

$$\text{Determine the Cathode Area Required (ft}^2\text{)} = \left(\frac{\text{Amps} \times B}{40} \right)$$

EXAMPLE #1

A consumer lab produces 100 gallons of combined Process RA-4, C-41, and E-6 effluent on a busy day. Average silver content is approximately 3 g/L with a second-shift operation.

A	B	C	D	Required Amps	Required Cathode Area
100 gal	2 (chart)	3 g/L	16 hours	35.5 amps	1.8 ft ²

$$\text{Required amps} = \frac{100 \times 3.785 \times 3}{2 \times 16} = 3.55 \text{ amps}$$

$$\text{Required Cathode Area} = \frac{35.5 \times 2}{40} = 1.8 \text{ ft}^2$$

EXAMPLE #2

A radiography group produces 20 gallons of black-and-white fixer at 4.5 g/L; work time is only 8 hours per day.

A	B	C	D	Required Amps	Required Cathode Area
20 gal	4 (chart)	4.5 g/L	8 hours	10.6 amps	1.1 ft ²

$$\text{Required Amps} = \frac{20 \times 3.785 \times 4.5}{4 \times 8} = 10.6 \text{ Amps}$$

$$\text{Required Cathode Area} = \frac{10.6 \times 4}{40} = 1.1 \text{ ft}^2$$

GENERAL TIPS FOR STARTING UP AN ELECTROLYTIC UNIT

It is very important that you follow the installation / operation manual that comes with your new unit. Install the electrolytic unit in a well-ventilated area with enough room around it to allow someone to service it. The electricity to the unit must be sufficient to provide the voltage / current requirements specified for the unit. Follow the current and time recommendations suggested in the manual for your type of solution or mixtures. Keep a log detailing critical parameters (voltage, current, electrolysis time, preventive-maintenance service, general comments) to allow a process re-creation in the event troubleshooting becomes necessary. Use the corrective actions shown in the troubleshooting table to optimize your process.

REMOVING SILVER FROM THE CATHODE

The correct initial unit set-up is essential to good long-term performance, the quality of silver flake produced, and the ease of removing that flake from the cathode (sometimes referred to as “mining the cathode”). Some troubleshooting guides are included in table at the right. Generally, current adjustment is the best tool. Surface preparation is key to easy removal of the silver flake from the cathode. We recommend mildly polishing the surface with a Scotch-Brite or similar pad prior to beginning electrolysis. In some cases, where good flake is being formed at the correct deposition rate but is still difficult to remove from the cathode, a very thin coating of automotive wax prior to electrolysis will help. Consult your equipment manufacturer for their advice in this area. Developments in electronics and equipment design over the past decade have significantly reduced problems in this area.

TROUBLESHOOTING AND TIPS FOR BETTER OPERATION

The table below provides a guide to troubleshooting various problems that can occur with electrolytic silver recovery equipment.

Proper pH is important for optimum performance in electrolytic desilvering; this is

especially true for iron-containing solutions. Increasing pH:

- Reduces the tendency for iron to interfere with reduction silver electroplating.
- Makes the cathode reaction for silver plating energetically more favorable.

Electrolysis Troubleshooting

Problem	Probable Cause	Corrective Action
Poor plating—low silver plating or lost silver	• Current too high • Improper plating time • Improper cathode surface preparation	• Adjust current or plating time • Scour w/Scotch-Brite-like pad prior to electrolysis
Sulfiding	• Current too high for silver concentration • Plating time too long • Agitation too low	• Adjust current or plating time
Recovered silver not pure	• Current too high, resulting in sulfiding • Current is not appropriate for the type of solution being desilvered	• Adjust current or plating time • Match current needed with the type of solution being desilvered
Nodules on the cathode or sell shorts out	• Accumulated dirt in the bath plates or becomes occluded in the silver plate	• Make sure used fixer fixer is adequately filtered prior to electrolysis
Silver sulfide formation (silver plating falls off or silver collects in the bottom of the cell)	• Low sulfide in solution • Plating current too high or unit run too long	• To maintain plating efficiency from most fixers, the concentration of sodium sulfite should be 8 – 10 g/L when the silver level is 1 – 5 g/L • Adjust current or time
Solution not desilvered to proper level	• Low pH • Low current or insufficient time • Electrical problems, bad connection	• Adjust pH • Adjust current or time • Check connections, consult operation manual
Very dark plate	• Initial silver concentration of solution is too low • Plating current too high or unit running too long	• Adjust current • Adjust current or plating time

Always exercise care when working with corrosive chemicals (use proper eye protection, adequate ventilation, gloves, etc.)

Generally, the higher the pH, the better. In practice pH should not be increased above 7.8 – 8.0 due to the evolution of ammonia that takes place in ammoniumbased fixers and bleach-fixes. You can use a strong base, such as sodium (or ammonium) hydroxide or sodium (or potassium) carbonate, to adjust the pH upward. The carbonates are the safest to work with; the hydroxides are usually the least expensive. You can also use pH adjustment chemicals sold by many swimming pool supply houses. Some examples are given in table on the right.

Always exercise care when working with these corrosive chemicals; use proper eye protection, adequate ventilation, gloves, etc.

You can perform the adjustment satisfactorily using most brands of pH measurement strips. Two types of strips that perform particularly well are:

- colorPHast Indicator Strips pH 0 - 14, by EM Science, Gibbstown, New Jersey.
- Baker pHIX, Universal pH Sticks, range 2.0 – 9.0.

Obviously, a pH meter works better than pH adjustment papers, but requires significant effort to calibrate. Consult the video, *Using KODAK Control Tools—How to Measure pH* (CAT No. 129 5914), for additional information on pH measurement and adjustment with meters.

If your facility plans to reuse the solution being electrolytically desilvered or is experiencing difficulty with the quality of silver flake formed, you may wish to add

additional sodium sulfite to the batch prior to electrolysis. A rule-of-thumb is to add the equivalent of 1 g / L of additional sulfite for every g / L of silver to be removed from the solution. This will help to prevent the solution from being degraded and the cathode “burned.” Never add sulfite to Process RA-4 Bleach-Fix, since it significantly reduces the electrolysis efficiency.

Recommendations for Adjusting the pH of Iron-Containing Solutions

Solution	mL of Solution per Litre of Bleach-Fix Overflow*
28% Ammonium hydroxide	10
10N Sodium hydroxide	13
45% Potassium hydroxide	14
10% Sodium carbonate	200
10% Potassium carbonate	300
EKTACOLOR RA Developer Tank Overflow	900

* Approximate mL required to raise EKTACOLOR RA-4 Bleach-Fix overflow pH to 7.5 – 8.0. Use a pH meter or pH strip to determine exact amount required for your facility's overflow.

METALLIC REPLACEMENT CARTRIDGES (MRCs)

A metallic replacement cartridge contains metallic iron in a form that photographic solutions can pass through in a controlled manner to allow the silver complexes in the solution to react with the iron. The silver is reduced to its metallic form and stays in the cartridge while the iron is oxidized and passes into solution. There are many forms of iron that can be used in MRCs. Some include:

- Steel wool
- Iron filings
- Steel screen wire
- Iron filings on a rigid support

Most photographic solutions can be desilvered by the proper use of a MRC or MRC system. Some exceptions include:

- Black-and-white reversal bleach
- Fixer preceded directly by ferricyanide bleach
- Seasoned ferric-ammonium EDTA bleach
- Fixers with a highly alkaline pH

Call Kodak Environmental Services for information on how to desilver these specific cases.

Some of the pro’s and con’s of MRC use are shown in the following table.

Pro’s and Con’s of MRC Desilvering

Pro’s	Con’s
• If used properly, can be very efficient for low silver compliance. • MRCs are relatively inexpensive and work reasonably well with a broad range of solution types. • The metallic replacement process is a simple technology not requiring a high degree of sophistication to perform. • It is easy to monitor canister breakthrough, especially with a colorimetric silver test. • Breakthrough can be estimated by keeping records of time in service and volume throughout.	• Although capable of good efficiency, a less than optimum performance is common. • Spent canisters are more costly to refine than silver recovered by other techniques. • MRCs are dependent upon contact time with steel media and therefore require good control of solution flow rate. • MRCs frequently require a “break-in” period and work best when used continually with no breaks in service. • Monitoring required to detect breakthrough. • MRCs discharge iron to the effluent.

CHOOSING THE PROPER UNIT

The single biggest factor influencing the performance of MRCs is the “residence” or “dwell time” of the solution in the cartridge. In actual practice, the cartridges are usually used in pairs for performance safety. You can control solution flow either by gravity feed through a flow-restricting orifice or by a metering pump. Almost always, metering pump systems do a better job. Always follow the flow rate recommendations provided by the manufacturer of your MRC. If you must, use a longer residence time rather than a shorter one. Although this may dissolve the steel slightly faster than necessary and reduce the ultimate capacity of the cartridge, too little residence time can lead to high silver discharges from the system.

USEFUL LIFE OF MRCs

Each MRC has a fixed amount of steel in it. As this is consumed by the desilvering process the MRC will eventually decrease in its capacity to remove silver from the solutions. MRCs are normally used in pairs (series)*, preferably with a sampling port placed between individual units. Samples are taken from the port and silver concentration measured by some convenient technique such as estimating paper, copper strips or a colorimetric method. (See KODAK Publication J-211, *Measuring Silver in Photographic Processing Facilities*, for additional information on measuring silver.) When either of these tools detect silver—the colorimetric method is much more accurate and reproducible—you should replace the cartridges.

* A few customers have used 3 MRCs in series for added “insurance.”

Generally, you remove the first MRC, move #2 into the #1 position, and place a new unit in the #2 position. If strict silver compliance is necessary, you can replace both cartridges at the same time.

You can use Metallic Replacement Cartridges as a primary (and only) system or as a secondary or tailing system following electrolysis. A common misconception is that cartridges will work much longer before replacement when they are used for the tailing operation (i.e., due to less silver removal). This is true, but the natural corrosivity to steel of many photographic processing solutions consumes steel wool even when silver is not being removed. Always monitor the system to detect breakthrough, and replace the cartridges on a regular basis.

ADJUSTMENT OF pH

You must monitor (and sometimes control) the pH of the solution passing through the system. To operate most efficiently, the pH of the solution needs to be acidic to encourage the steel wool to dissolve. Most photographic fixers and bleach-fixes have an acceptable pH range. Ideally, the pH should be between 5.5 and 6.5. If the pH is below 5.0, the steel wool will be catalyzed to dissolve too rapidly and the capacity of the cartridge could be reduced significantly. If the pH is

basic (above 7), the steel wool dissolution reaction will be slow and optimum silver removal may not take place.

You can adjust the pH upward or downward with acetic acid or pH adjustment chemicals found at swimming pool supply stores. Again, pH papers are sufficient to monitor this adjustment.

PRE-CONDITIONING MRCs

MRCs work best when they are pre-conditioned by allowing to stand for several hours with a mildly acidic solution such as fixer or bleach-fix in them. This allows the steel wool to

begin to “etch” or become chemically activated for silver recovery. At the very least, fill a new MRC with water to begin to chemically prepare the steel surface and minimize the potential to “channel” or form pathways that do not contact the steel wool once it is placed in service.

You will obtain the best performance and longest life by continuous use with approximately the same solution composition / mixture. Intermittent or infrequent use of MRCs allows the steel wool to oxidize or rust. Once this process has begun, it is very difficult to predict the useful life of the cartridge.

MRC Troubleshooting

Problem	Probable Cause	Corrective Action
Cartridge becomes exhausted too quickly	- Flow rate too high - MRC too small for application - pH of solution too low	- Adjust flow rate - Use larger MRC - Raise the pH
Loss of recovery efficiency - Lower silver yield than expected - High silver concentration out of MRC - Solution discharged is untreated	- Solution pH too high - Flow rate too high - Cartridge not "preconditioned" - Solution flow pattern causes steel wool erosion to form a channel; solution is able to pass untreated. (This is a possible result of low volume or intermittent usage.) - Flow has become plugged allowing MRC by-pass	- Lower the pH - Adjust flow rate - Soak with solution prior to installing - Pre-condition MRCs before installation. Use a mildly acidic solution, fixer, bleach-fix, or at the very least, water - Look for obstructions to flow; may be as simple as a "crimped tube"
Plugged drain lines	Precipitation of iron hydroxide	Use an acid-type drain cleaner when changing MRC

PRECIPITATION

Chemical precipitation, or the addition of a chemical to the photographic processing solution to cause an insoluble silver salt to form, has not been popular in the past except at large centralized waste-treaters. Over the past several years, Kodak has developed a new precipitation technology based on Tri-sodium 2,4,6-Trimercapto-s-triazine, more commonly called (for obvious reasons) TMT. TMT precipitation technology was initially optimized for use as secondary treatment following electrolysis at large photographic processing facilities. The advantages of the technique versus traditional secondary treatment with MRCs are:

- More consistent low silver results
- Lower cost over the long-term
- Reduced refining cost of the resulting silver sludge
- Less labor required

Large-scale secondary recovery can be very simple. However, you should follow certain procedures. You must use the proper TMT dosage for the silver level of the mixture under treatment. Consistently mix the same solutions (day after day) and use a colorimetric silver test to determine the correct dosing level. Typically, the silver concentration of the solution mixture before treatment should not be below 300 mg/L. Choose the proper electrolysis conditions during primary treatment. If you use the proper TMT dosage level and do not obtain acceptable final silver concentrations, consult the following table.

Several manufacturers now make customized equipment for automated secondary precipitation treatment with TMT.

In 1995, Kodak introduced a semi-automatic unit designed to serve as the primary silver-recovery unit for smaller Process C-41, Process RA-4, and Process E-6 photographic processing facilities. This unit has significantly changed the way silver is recovered at many small photographic processing facilities. Some of the features of the unit include:

- More consistent low silver results
- Less labor (almost fully-automatic operation)
- Lower refining cost of the resulting silver sludge
- Less floor space required
- Easy-to-monitor performance

Large-Scale Secondary Precipitation Troubleshooting

Concern	Probable Cause	Corrective Action
Final silver concentration too high	• Improper dosing	• Determine initial silver concentration of mixture and adjust dose • Try to treat as consistent a mixture of overflow solutions as possible
	• Insufficient settling time	• Optimize using a controlled study
	• Silver concentration before treatment less than 300 mg/L	• Adjust electrolysis conditions with primary equipment
	• Settled sludge volume exceeds stand-pipe height. (You may see excessive yellow solid getting to final filter.)	• Adjust stand-pipe height
	• Line purge step has been skipped or shortened	• Lengthen purge line
	• Improper filter cartridge or improper installation	• Check filter type and installation

The operation of the semi-automatic unit makes silver recovery as simple as it can get; however, you must keep an eye on several very basic items. Like large-scale TMT recovery, proper dosing level is important with the semi-automatic unit. The Part B flocculant must be mixed properly and within mix age to ensure the formation of large "scrambled egg-like" particles that easily separate from the liquid phase of the treated solution. To guarantee easy solution flow through the unit, you must rinse the system according to the maintenance schedule in the operator's manual and you must occasionally clean the particle screen in the unit's hold tank. Although these items may seem cumbersome, proper routine maintenance on the unit is by far the least time-consuming of any type of functional silver-recovery equipment today.

Semi-Continuous Precipitation Troubleshooting

Concern	Probable Cause	Corrective Action
Final silver concentration too high	- Improper Part A dosing - Part B is either not mixed properly or is very old. (Another clue is lack of formation of "large particles" in the reactor tubing prior to the filter.)	- Determine initial silver concentration of mixture and adjust Part A dose. - Always treat solutions in "as replenished" proportions. If you periodically dump your stabilizer tanks, try to blend this solution gradually with the other overflows. - Refill the Part B reagent tank with a fresh mix of Part B.
Short filter life	Equipment not forming large particles (i.e., a lot of "dust" instead of clumps).	Check Part A & Part B delivery rates. Check initial silver concentration to verify at least 1 g/L concentration. Modify procedure for stabilizer dump to increase silver concentration, if applicable.
Slow flow-through system	- Tubing is beginning to plug - The screen on the pump suction line in the collection tank is plugged - Pumps need calibrating or check valves require cleaning	- Rinse system according to User's Guide - Clean screen per User's Guide - Check flow rate of each pump using the calibration procedure in Appendix C of the User's Guide; remove and clean the check valve.
Semi-automatic unit will not run, although plugged in and turned on	- Solution level in collection tank is low - Low level in Part A tank; not indicated by status light - Low level in Part B tank; not indicated by status light - High system pressure; not indicated by status light	- This is part of the normal operation; you do not need to take any corrective action. - Check solution level in Part A tank and refill, if necessary. Replace indicator bulb, if necessary. - Check solution level in Part B tank and refill, if necessary. Replace indicator bulb, if necessary. - Check sludge level in filter. Replace, if full. Replace indicator light bulb, if necessary.



MORE INFORMATION

If you have environmental or safety questions about Kodak products or services, contact Kodak Environmental Services at 1-716-477-3194, between 8 a.m. and 5 p.m. (Eastern time) or visit KES on-line at www.kodak.com/go/kes.

Kodak also maintains a 24-hour health hotline to answer questions about the safe handling of photographic chemicals. If you need health-related information about Kodak products, call 1-716-722-5151.

For questions concerning the safe transportation of Kodak products, call Kodak Transportation Services at 1-716-722-2400.

Additional information is available on the Kodak website and through the U.S.A./Canada faxback systems.

The products and services described in this publication may not be available in all countries. In countries other than the U.S., contact your local Kodak representative, or your usual supplier of Kodak products.

The following publications are available from Kodak Customer Service or from dealers who sell Kodak products.

- J-210 *Sources of Silver in Photographic Processing Facilities*
- J-211 *Measuring Silver in Photographic Processing Facilities*
- J-212 *The Technology of Silver Recovery for Photographic Processing Facilities*
- J-213 *Refining Silver Recovered from Photographic Processing Facilities*
- J-214 *The Regulation of Silver in Photographic Processing Facilities*
- J-216 *The Fate and Effects of Silver in the Environment*
- J-217 *Using Code of Management Practice to Manage Silver in Photographic Processing Facilities*

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