

Processing KODAK Motion Picture Films, Module 5

Chemical Recovery Procedures



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5 Chemical Recovery Procedures

INTRODUCTION

Recovery or reuse of processing chemicals should be given prime attention for four reasons:

- An effective chemical-recovery program can be a cost-saving operation for a processing laboratory.
- Silver recovery from fixes and wash waters is a source of revenue and conservation of a natural resource.
- Chemical recovery is highly acceptable ecologically.
- Laws may require reduced chemical discharge into sewers as a condition of continued operation.

DEVELOPER RECOVERY

Reconstitution of Developer Overflow

Recommendations are not provided for the reconstitution or recycling of ECN-2 developer solution. This practice is not recommended because the wide range of available camera negative and intermediate films create a complex matrix of seasoning products that may adversely effect films with a broad range of sensitivities.

ECP developer overflow can be reconstituted into a usable replenisher if the bromide and iodide levels are not greatly higher than the replenisher level. By addition of the depleted developer constituents, pH adjustment, and dilution to lower the bromide and iodide levels, the solution can be brought to replenisher specifications for Process ECP-2D.

Reconstitution Procedure

1. Collect the developer overflow in a holding tank (with minimum aeration) until a sufficient volume has been collected to make a replenisher. This volume of overflow should be about 80% of the volume of replenisher desired.
2. Analyze the overflow for pH, specific gravity, total alkalinity, Developing Agent CD-2, sodium bromide, and sodium sulfite. Use the *Analytical Procedures* in Module 3.
3. Compute the amount of dilution, and the amounts of makeup chemicals needed to make a replenisher. See sample calculations following.

EXAMPLE: Reconstitution Calculation

Produce 500 litres of reconstituted replenisher from Process ECP-2D developer overflow (SD-50Ra). The following data apply:

Constituent	Overflow Analysis	Replenisher Level	Amount Needed
KODAK Color Developing Agent, C-2	3.00 g/L	6.00 g/L	+ 3.00 g/L
Sodium Bromide (NaBr)	1.70 g/L	1.60 g/L	- 0.10 g/L
Sodium Sulfite (Na ₂ SO ₃)	4.10 g/L	4.50 g/L	+ 0.40 g/L

4. Dilute the overflow, and make the necessary chemical additions in the proper mixing order and with adequate agitation.
5. After all the additions have been made, analyze the reconstituted replenisher for pH, specific gravity, total alkalinity, Developing Agent CD-2, sodium bromide, and sodium sulfite.
6. Adjust pH to replenisher specification.
7. If the total alkalinity is below specification, add more sodium carbonate. If it is less than 10% higher than specification, do nothing. If the total alkalinity is more than 10% high, reanalyze the solution since a mistake has probably been made in the chemical additions.
8. Pump the reconstituted developer to the replenisher holding tank.

Step 1. Determine the amount of water dilution needed:

$$\begin{aligned} \text{a) Volume of overflow that can} &= \frac{(\text{volume of replenisher to be mixed}) (\text{g/L NaBr in replenisher})}{\text{be used} \quad (\text{g/L NaBr analyzed in overflow})} \\ &= \frac{(500 \text{ L of replenisher}) (1.60 \text{ g/L NaBr})}{(1.70 \text{ g/L NaBr})} \end{aligned}$$

$$\text{Volume of overflow} = 470 \text{ L}$$

$$\text{b) Volume of water to be added} = (\text{L of replenisher to be mixed}) - (\text{L of overflow to be used})$$

$$\text{Volume of water} = (500 \text{ L of replenisher}) - (470 \text{ L of overflow})$$

$$= 30 \text{ L}$$

Step 2. Determine the amount of CD-2 and Na₂SO₃ to be added:

$$\text{a) Amount of CD-2} = \frac{(\text{Amount of CD-2 needed to bring overflow to replenisher std}) (\text{Volume of overflow used})}{\text{to be added} \quad + (\text{Amount of CD-2 in std replenisher}) (\text{Volume of water added})}$$

$$\text{Amount of CD-2} = (3.0 \text{ g/L CD-2}) (470 \text{ L of overflow}) + (6.0 \text{ g/L CD-2}) (30 \text{ L of water})$$

$$\text{b) Amount of} \quad \frac{(\text{Amount of Na}_2\text{SO}_3 \text{ needed to bring overflow to replenisher std}) (\text{Volume of overflow used})}{\text{Na}_2\text{SO}_3 \text{ to be} \quad + (\text{Amount of Na}_2\text{SO}_3 \text{ in std replenisher}) (\text{Volume of water added})} \\ \text{added}$$

$$\text{Amount of} \quad = (0.4 \text{ g/L Na}_2\text{SO}_3) (470 \text{ g/L of overflow}) + (4.5 \text{ g/L Na}_2\text{SO}_3) (30 \text{ L of water}) \\ \text{Na}_2\text{SO}_3$$

$$\text{R h} = 323 \text{ grams}$$

Step 3. Determine the amount of sodium carbonate (Na₂CO₃) and KODAK Anti-Calcium, No. 4, to be added:

Note: Assume these two chemical levels in the developer overflow are correct. The amounts of these chemicals in the overflow are either already close to replenisher requirements or difficult to determine and without major sensitometric effect:

Use the following formula for these chemicals:

$$\text{Total amount of chemical} = \frac{(\text{Amount of chemical in replenisher std}) (\text{Volume of water added})}{\text{to be added}}$$

Recovery of Color Developing Agent

KODAK Color Developing Agent, CD2, can be recovered from spent Process ECP-2D Color Developer and Stop Bath. Recovery of the developing agent has both environmental and economic justification. The developing agent has the highest oxygen demand and the highest potential cost in Process ECP-2D Color Developer. Sufficient CD-2 is also found in the stop bath to make it economical to remove it from that solution. The developing agent can be removed from both spent solutions by means of absorbent resin technology*.

In principle, the color developer and stop bath are collected together or separately. The pH of the collected solution is adjusted to about 9.5. The solution is then pumped through an absorbent resin. Effective resins are Duolite, ES-862, ES-866, S-761 and Amberlite XAD-4†. The developing agent is removed from the resin with a sulfuric-sulfite solution. This acid solution contains the recovered CD-2 and is used to prepare a color developer replenisher.

Regeneration of Color Developer Using Ion-Exchange

It has been demonstrated by Burger‡ that the bromide ion can be removed from a spent color developer using a strong-base ion exchange resin such as Rohn and Haas IRA-400. The resulting developer can be reused and reconstituted as a replenisher. The resin is regenerated with 1.0 M sodium chloride followed by 0.5 M sodium bicarbonate.

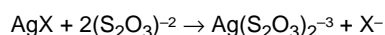
* J. L. Burger, H. E. Fowler, B. A. McPhee, J. E. Yager "Recovery of KODAK Color Developing Agent CD-2 from Process ECP-2A Color Developer and Stop Bath," *Journal of the SMPTE*, 94-648, June 1985.

† Rohn and Haas

‡ J. L. Burger and R. Mina, "An Alternative Ion-Exchange Regeneration System for Recovery of KODAK EKTAPRINT 2 Developer," *Journal of Applied Photographic Engineering*, Vol. 9, No. 2, April 1983.

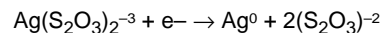
SILVER RECOVERY

In fixers, thiosulfate (hypo) is used to fix the image by converting the undeveloped, insoluble silver halide to a soluble complex. This soluble complex diffuses out of the emulsion into the fixer. Halides (X = bromide or chloride ion) also diffuse from the film.



As the fixer is used, it becomes a very complex mixture. In addition to its original components, a used fixer contains ferrocyanide, sodium sulfate, sodium bromide, sodium chloride, gelatin, complex silver salts, and varying amounts of practically all the chemicals used in processing. Therefore, any method of silver recovery has to make allowances for these interfering substances. Fortunately, silver is far removed in the electromotive series from any other metallic ion in the solution. Most silver recovery methods take advantage of this fact. If silver is continuously recovered, the fixer can be reused until the halides, ferrocyanide, etc, build up to concentrations high enough to retard fixing. Usually, enough fixer overflow is discarded to prevent this from occurring.

The most common silver recovery method from fixer is electrolytic. An electrolytic method is fundamentally an electroplating process in which the silver in the fixer is deposited on a set of stainless steel plates that serve as the cathode in an electrolytic cell. The primary cathode reaction is a reduction of complex silver into a silver metal.



The thiosulfate is released from the complex to react with other silver ions. The cathode is removed from the cell periodically to strip off the plated silver. This technique has the advantage of being clean, allowing reuse of the fixer, and yielding silver metal with a high degree of purity (90-99 percent).

The most effective method of recovery of silver from fixer wash waters or dilute fixers is by using ion-exchange resins.

Electrolytic Methods—Fixer

There are two basic types of electrolytic methods: batch and continuous. Either one can be employed whether the fixer is reused or not. If fixer is not reused, the cell current must be adjusted to very low levels as the silver is depleted. The silver level in fixer waste cannot be reduced much below 0.1 g/L in a practical system. At the 0.1 g/L concentration the cell degrades the fixer making it unsatisfactory for reuse.

Since reuse of fixer has economic as well as ecological advantages, the following discussion assumes fixer reuse.

Batch Method

The batch method requires a minimum of equipment, but is not as efficient as the continuous method. When a sufficient volume of used fixer is collected, the silver content in the fixer is reduced to 0.5 to 1.0 g/L by an electrolytic cell. Chemicals are then added to reconstitute the fixer for use as a replenisher.

Continuous Method

This method includes the electrolytic cell in the fixer recirculation system. The fixer is continuously drawn from the processing machine through the electrolytic cell. The silver content is reduced to 0.5 to 1.0 g/L by the cell, and the fixer is returned to the machine. To maintain the proper thiosulfate, sulfite, and pH levels, a suitable replenisher is added to the system at a convenient location. This is a very efficient method, but it requires more space and careful control of the current density (i.e., amperes per unit cathode area) of the cell. If current is applied when no silver is present or if too high a current density is applied for the amount of silver in the bath, a layer of silver sulfide forms on the cathode. This sulfide layer can prevent the recovered silver from adhering properly to the plate. Flaking of silver from the plate can in turn short out the electrodes. Fine particles of silver and/or silver sulfide generated by an improperly adjusted electrolytic cell can be a serious source of process dirt. The silver level in a fixer can be determined precisely by the latest versions of Analytical Methods *I208E* and *I209D** (Module 3) or, roughly, by means of Silver Estimating Test Books, or by a Colorimetric Silver Test Kit.

Electrolytic Cells

The electrolytic cells are available in two types; low-current-density units and high-current-density units. The latter type is recommended for motion picture laboratories because of its high capacity, making it more attractive for large-volume film processing,

Tailing

After silver has been reduced to 0.5 g/L by either the batch or continuous method, 80% of the fixer is used for reconstituting the replenisher and 20% is sent to tailing for further reduction in silver level. After tailing, the fixer is discarded. The electrolytic cell can be used to reduce the silver to 0.1 g/L during tailing. Further reduction to 1 mg g/L can be obtained by using metallic replacement, described below.

General Rules of Operating Electrolytic Cells

Although much has been written about electrolytic silver recovery, a brief review of the important factors required for efficient operation follows:

1. Agitation is probably the most important consideration in obtaining efficient plating without decomposing the fixer. Agitation prevents buildup of a boundary layer of desilvered solution at the cathode, which would prevent the silver-bearing solution from coming in contact with the cathode, thus preventing the silver from plating out. Also, with low agitation there will be localized areas where the current density will be too high with respect to the silver level, resulting in the formation of silver sulfide.
2. The amount of current passing through the fixer, usually stated in terms of amperes/ft² of plating surface or current density, also has an important bearing on the efficiency of the cell. Too little current results in less silver plating out within a reasonable time period. Too high a current density results in sulfiding, which decreases not only the efficiency of the cell, but also the purity of the recovered silver. Excessive current density adversely affects a seasoned fixer, causing dirt and unwanted sensitometric effects.
3. The silver concentration dictates the current density at which the cell can be operated. Failure to reduce current as the silver level decreases can cause sulfiding.
4. The type of fixer is a consideration. Generally ammonium fixers can tolerate higher current than sodium fixers and silver can be plated more efficiently. Alkaline fixers can be successfully desilvered, but generally require lower current densities than acid fixers.
5. During electrolysis, sulfite is consumed and the pH drops; if these parameters are not maintained, the fixer will tend to decompose.
6. Good filtration is necessary. Dirt present in the fixer can become occluded in the silver plate, and sometimes these particles become preferred plating sites. Sufficient silver could build up at these sites to bridge the cathode and anode, causing the cell to short out. Dirt in the fixer must also be removed to prevent it from adhering to the film.

* For more information, see KODAK Publication No. J-211, *Measuring Silver in Photographic Processing Facilities*.

Metallic Replacement Method—Fixer and Wash Waters

In this method, a metal (usually iron) reacts with the silver thiosulfate and goes into solution. The less active metal (silver) settles out as a solid. To bring the silver into contact with the iron, the fixer or bleach-fix is passed through a container filled with steel wool. The steel wool provides the source of iron to replace the silver.

The main advantages of this method are the very low initial cost, and the simplicity of installation; only a few simple plumbing connections are required. They are excellent means to recover silver from small amounts of fixers, especially for tailing operations.

The disadvantages, compared to the electrolytic method, are that the silver is recovered as a sludge, making it more difficult to determine the exact amount recovered, and the sludge requires more refining than the plate silver obtained from electrolytic methods. The cartridges cannot be reused, and require replacement when exhausted. The effluent from the cartridges contains high amounts of iron. This would be a problem in a locality with a sewer code that limits the iron concentration in the effluent. Kodak sells two types of metallic replacement cartridges, the KODAK Chemical Recovery Cartridge, Model II and Junior Model II.

Ion-Exchange Method—Fixer Wash Waters

The use of ion-exchange resins are effective in removing silver from dilute fixers such as fixer wash waters. In this method, the silver-bearing solution is passed through a column containing an ion-exchange resin. As the solution passes through the column, the silver is absorbed in the resin, reducing the silver to a level between 2 and 0.1 mg/L.

Either a strong-base resin such as IRA-458 or a weak-base resin IRA-67 from Rohm and Haas has been used. R. Mina describes the use of such resins.* The silver is absorbed on the resin and then removed with a high concentration of hypo. The silver is subsequently recovered using an electrolytic cell. P. R. Quinones describes an in-situ precipitation procedure using resin IRA-68.† This procedure consists of precipitating the silver in the resin instead of removing the silver from the resin with a regenerant such as hypo solution. The resin is used for many cycles and the absorbed silver is then recovered by incinerating the resin. The advantages of this procedure are the need for less equipment and supervision.

Chemical Precipitation

Precipitation can remove silver from silver-rich solutions, reducing it to very low levels. Properly applied, levels can be reduced to the low ppm (part-per-million) range. Until recently, precipitation has not been as widely used as a silver-recovery technique. Common precipitating agents classically have been alkali metal salts of sulfide (sodium sulfide, potassium sulfide, etc) which will form silver sulfide in solution; the silver sulfide is removed by filtration.

The lack of acceptance of the silver sulfide precipitation-filtration process can be attributed primarily to two factors:

- You must measure the solution silver concentration accurately prior to sulfide addition to prevent overdosing and toxic hydrogen sulfide gas from being discharged. Until recently, no easy, readily available, analytical technique existed to measure the silver concentration before treatment.
- The silver sulfide precipitate is difficult to filter, plugging filter media.

Sulfide desilvering is most effective in centralized facilities when used by trained personnel.

Other precipitating procedures generally involve converting the silver in solution to the metallic state by adding strong reducing compounds such as borohydrides. These techniques are best used by solution service companies or centralized treatment facilities staffed with technical professionals. There are serious safety considerations when handling chemicals like borohydrides.

The use of TMT (trimercapto-S-triazine) can also precipitate silver from silver-rich solutions. TMT produces

an insoluble silver compound that is more easily filtered than silver sulfide. For many processes, silver levels may be reliably and consistently reduced to an average of less than 1.5 ppm. Advantages of TMT include consistent low silver discharges and reduced cost. Commercial units are available for several applications. These include units designed for large photographic processing facilities.‡

Reverse Osmosis

This method uses high pressure to force the solution through a semipermeable membrane to separate larger molecules such as salts and organic compounds, from smaller molecules like water. Once the silver is separated from the wash water in this manner, the silver can be recovered by conventional means such as metallic replacement, electrolytic recovery or chemical precipitation. The method is capable of removing almost 90 percent of silver thiosulfate complexes from wash water.

Other chemicals, such as color couplers or ferricyanide can also be recovered by reverse osmosis. Since this process can remove almost all other chemicals, the treated wash water may be suitable for reuse in final washes. The method is useful when wash water treatment is required to reduce the amount of silver or other chemicals being discharged.

Silver Recovery Management

To aid in management of a silver recovery program, P. J. Mutter and S. J. Powell describe expected amounts of silver available from films and eleven common silver recoverable layouts indicating concentrations of silver in fixers, tailings and wash waters with various fixer replenisher rates.** This information can be valuable in helping a laboratory select the most economical procedure to recover silver from their processes.

* R. Mina, "Silver Recovery from Process ECP-2 Wash Waters by Ion-Exchange Methods," *Journal of the SMPTE*, 90:754-759, September 1981.

† P. R. Quinones, "In-Situ Precipitations as the Regeneration Step in Ion-Exchange for Silver Recovery," *Journal of the SMPTE*, 93:800-807, September 1984.

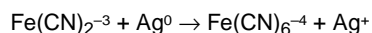
‡ For more information, see KODAK Publication No. J-208, *Introducing the Silver Management Series*.

** P. J. Mutter and S. J. Powell "Silver Recovery Management for Motion Picture Laboratories," *Journal of the SMPTE*, 93:808, September 1984.

BLEACH RECOVERY

Ferricyanide Bleach

Ferricyanide bleach is used to render the silver image removable from the film. During bleaching, the silver is converted to silver ions according to the following equation.



Since the bleach contains bromide ions, the silver ions in the emulsion are converted to silver bromide during bleaching. The silver bromide is removed in the fixing and washing steps that follow.

For economy, use ferrocyanide in place of ferricyanide for preparing the bleach solution and convert the ferrocyanide to ferricyanide by oxidation. Collect and regenerate bleach tank overflow for additional economy.

The most common method of bleach regeneration uses a persulfate oxidizing agent. This simple technique involves no significant investment and uses relatively safe and stable chemicals.

It is also possible to oxidize ferrocyanide to ferricyanide by electrolytic means, or by using ozone, for both preparation and regeneration of bleach solutions.

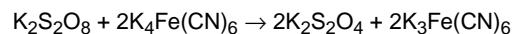
The Persulfate Method for Ferricyanide Bleach Recovery

The method below utilizes either sodium or potassium persulfate to convert the ferrocyanide to ferricyanide in solution. Ammonium persulfate* can also be used but requires substantial pH adjustments.

To minimize sludging from hard water, prepare bleach solutions using sequestering agents†, such as Calgon (Calgon Corporation) or Quadrafos (Rumford Chemical Company). These agents do not interfere with the regeneration using persulfate.

1. Raw Materials

The equation for the oxidation of ferrocyanide by persulfate follows:



The reaction has been written using potassium salts for all of the chemicals for simplicity. In practice, however, other compounds can be used, their choice depending upon cost, availability in specific localities, transportation costs, etc. Table 5-1 lists some of the available compounds, their chemical equivalent weights, and the amount of each required to produce 1 gram of potassium ferricyanide.

* Ammonium persulfate is not recommended when preparing a fresh ferricyanide bleach if the bleach will subsequently be regenerated in an electrolytic cell. The ammonium ions will interfere with the electrolytic rejuvenation of the bleach.

† A practical amount to add with moderately hard waters is 0.5 g/L.

Table 5-1 Ferricyanide Bleach Chemicals

Chemical	Formula	Chemical Equivalent Weight	Grams of Compound Required to Produce 1,000 Gram of $\text{K}_3\text{Fe(CN)}_6$
Ferricyanides:			
Sodium Ferricyanide Anhydrous	$\text{Na}_3\text{Fe(CN)}_6$	281	0.854
Sodium Ferricyanide Monohydrate	$\text{Na}_3\text{Fe(CN)}_6 \cdot \text{H}_2\text{O}$	299	0.901
Potassium Ferricyanide Anhydrous	$\text{K}_3\text{Fe(CN)}_6$	329	1.000
Ferrocyanides:			
Sodium Ferrocyanide Anhydrous	$\text{Na}_4\text{Fe(CN)}_6$	304	0.924
Sodium Ferrocyanide Decahydrate	$\text{Na}_4\text{Fe(CN)}_6 \cdot 10\text{H}_2\text{O}$	484	1.471
Potassium Ferrocyanide Anhydrous	$\text{K}_4\text{Fe(CN)}_6$	368	1.119
Potassium Ferrocyanide Trihydrate	$\text{K}_4\text{Fe(CN)}_6 \cdot 3\text{H}_2\text{O}$	422	1.283
Persulfates:			
Potassium Persulfate Anhydrous	$\text{K}_2\text{S}_2\text{O}_8$	135	0.410
Sodium Persulfate Anhydrous	$\text{Na}_2\text{S}_2\text{O}_8$	119	0.362
Ammonium Persulfate Anhydrous	$(\text{NH}_4)_2\text{S}_2\text{O}_8$	114	0.346

Sodium ferrocyanide decahydrate $\text{Na}_4\text{Fe}(\text{CN})_6 \cdot 10\text{H}_2\text{O}$, is commercially available under the name Yellow Prussiate of Soda. No caking has been noted during storage. A typical analysis of shipments received is shown in Table 5-2. It loses water readily during storage unless packaged in moisture-proof bags or other moisture-proof containers. If partially dehydrated material is used, its purity will be greater than 100 percent, in terms of the formula $\text{Na}_4\text{Fe}(\text{CN})_6 \cdot 10\text{H}_2\text{O}$.

Table 5-2 Typical Analysis of Sodium Ferrocyanide Decahydrate

Assay [$\text{Na}_4\text{Fe}(\text{CN})_6 \cdot 10\text{H}_2\text{O}$]	99.9%
Insoluble Matter	0.03%
Volatile Matter (water)	37.0%
Halides (as Cl)	0.015%

Potassium persulfate (peroxydisulfate) is available in 100-pound bags or 300-pound drums with polyethylene liners. It is not sufficiently hazardous for the Interstate Commerce Commission to require it to bear a yellow label for shipment. However, it is advisable to avoid storing this oxidizing agent adjacent to organic materials because, in case of fire, the heat would decompose the persulfate, liberating oxygen. It should not be mixed with other chemicals in the solid state. Mixtures of potassium persulfate and sodium bromide react slowly if stored in contact with each other. A typical analysis of a grade of potassium persulfate, which is commercially available and which meets the ANSI/ACS specifications, is shown in Table 5-3.

Table 5-3 Typical Analysis of Potassium Persulfate*

Assay [$\text{K}_2\text{S}_2\text{O}_8$]	100.5%
Halides (as Cl)	<0.006%
Fe	<0.005%
Heavy Metals	<0.005%

In the discussion that follows, it is assumed that sodium ferrocyanide decahydrate $\text{Na}_4\text{Fe}(\text{CN})_6 \cdot 10\text{H}_2\text{O}$, and anhydrous potassium persulfate $\text{K}_2\text{S}_2\text{O}_8$, are used as the raw materials for preparation of both fresh mixes and regeneration of the bleach solution. If other materials are used, the calculations given below can be modified accordingly, using the data in Table 5-1.

The quantities of the various ingredients required for a fresh mix and the desired composition of bleach are given in the formula section of each process specification module.

2. Procedure for Preparing a Fresh Ferricyanide Bleach Using Ferrocyanide and Persulfate

- a. Calculate the amount of sodium ferrocyanide decahydrate $\text{Na}_4\text{Fe}(\text{CN})_6 \cdot 10\text{H}_2\text{O}$, required to produce the formula amount of potassium ferricyanide for a fresh bleach:

$$\begin{aligned} \text{g Na}_4\text{Fe}(\text{CN})_6 \cdot 10\text{H}_2\text{O} &= \\ \frac{[\text{eq wt Na}_4\text{Fe}(\text{CN})_6 \cdot 10\text{H}_2\text{O}] [\text{g K}_3\text{Fe}(\text{CN})_6]}{[\text{eq wt K}_3\text{Fe}(\text{CN})_6]} &= \\ \frac{484 [\text{g, K}_3\text{Fe}(\text{CN})_6]}{329} &= \\ 1.47 [\text{g, K}_3\text{Fe}(\text{CN})_6] &= \end{aligned}$$

- b. Next calculate the amount of potassium persulfate required to produce the formula amount of potassium ferricyanide.

$$\begin{aligned} \text{g K}_2\text{S}_2\text{O}_8 &= \\ \frac{[\text{eq wt K}_2\text{S}_2\text{O}_8] [\text{g K}_3\text{Fe}(\text{CN})_6]}{[\text{eq wt K}_3\text{Fe}(\text{CN})_6]} &= \\ \frac{135 [\text{g, K}_3\text{Fe}(\text{CN})_6]}{329} &= \\ 0.41 [\text{g, K}_3\text{Fe}(\text{CN})_6] &= \end{aligned}$$

- c. The required amount of sodium bromide is given in the formula in each process specification module.
- d. In preparing the mix, place an amount of water equivalent to about 80 percent of the final volume in the mix tank. Add a sequestering agent if needed. Then add the calculated amounts of sodium ferrocyanide decahydrate and potassium persulfate and the necessary amount of sodium bromide, and stir the solution until all salts are dissolved. To avoid having an excess of persulfate in the oxidized bleach solution, add an excess of 2 g/L sodium ferrocyanide decahydrate. Dilute the solution to volume, and stir again. Adjust the pH by the addition of 2.5 N sodium hydroxide solution or 2.5 N Sulfuric acid.
- e. At a solution temperature of 70° to 80°F (21° to 26.7°C), the reaction is complete in about 1 hour. At the end of this period, analyze the solution for sodium bromide, potassium ferricyanide, sodium ferrocyanide, and pH. See Module 3 for the required *Analytical Procedures*. Adjust the solution to specification if necessary.

* Meets ANSI/ACI Standard PH4.303-1984.

3. Procedure for Regeneration of Ferricyanide Bleach Overflow Using Ferrocyanide and Persulfate

- Collect the bleach tank overflow in a holding tank until a sufficient volume has been collected to make a new bleach replenisher.
- Analyze the bleach overflow for potassium ferricyanide, sodium ferrocyanide, and sodium bromide using the methods in Module 3, *Analytical Procedures*.
- Transfer the solution to a regeneration tank.
- Calculate the amounts of the various constituents needed to rejuvenate this bleach overflow:

1) Sodium ferrocyanide decahydrate

g/L $\text{Na}_4\text{Fe}(\text{CN})_6 \cdot 10\text{H}_2\text{O}$ required =

$$\left[\frac{\text{g/L Na}_4\text{Fe}(\text{CN})_6 \cdot 10\text{H}_2\text{O} \text{ needed to bring the g/L K}_3\text{Fe}(\text{CN})_6 \text{ to replenisher level}}{\text{g/L Na}_4\text{Fe}(\text{CN})_6 \cdot 10\text{H}_2\text{O} \text{ in bleach overflow}} \right] -$$

+ [2 g/L excess]

$$= 1.47 [\text{K}_3\text{Fe}(\text{CN})_6 \text{ in replenisher}] - \text{K}_3\text{Fe}(\text{CN})_6 \text{ in overflow} -$$

+ [2 g/L excess]

2) Potassium persulfate

$$\text{g/L K}_2\text{S}_2\text{O}_8 \text{ required} = \frac{\text{g/L K}_2\text{S}_2\text{O}_8 \text{ needed to bring the g/L K}_3\text{Fe}(\text{CN})_6 \text{ to replenisher level}}{\text{g/L K}_3\text{Fe}(\text{CN})_6 \text{ in replenisher} - \text{K}_3\text{Fe}(\text{CN})_6 \text{ in overflow}}$$

$$= 0.410 [\text{K}_3\text{Fe}(\text{CN})_6 \text{ in replenisher} - \text{K}_3\text{Fe}(\text{CN})_6 \text{ in overflow}]$$

3) Sodium bromide

$$\text{g/L NaBr required} = \frac{\text{g/L NaBr in replenisher} - \text{g/L NaBr in overflow}}{\text{g/L NaBr in replenisher} - \text{g/L NaBr in overflow}}$$

- Add the calculated amounts of the various chemicals to the bleach overflow, and mix the solution thoroughly. (See example below.) As noted above, an excess of 2.0 g/L of sodium ferrocyanide decahydrate is added. This highly hydrated chemical increases the volume of the solution, amounting to about 0.5 mL for each gram of salt added. It may be necessary, therefore, to add more than the calculated amount to compensate for this dilution.
- After all additions have been made, adjust the pH to specification, and allow the reaction to proceed for 1 hour. At the end of this period, analyze the solution again for the critical constituents and pH, and readjust if necessary.
- Pump the regenerated bleach to the bleach replenisher holding tank.

EXAMPLE: Regeneration Calculation

Prepare a Process ECP-2D Ferricyanide Bleach Replenisher (SR-27R) from 300 litres of Ferricyanide Bleach Overflow (SR-27) using persulfate.

	$\text{K}_3\text{Fe}(\text{CN})_6$	$\text{Na}_4\text{Fe}(\text{CN})_6 \cdot 10\text{H}_2\text{O}$	NaBr
Desired in replenisher	49 g/L	2 g/L	26 g/L
Overflow analysis	30 g/L	10 g/L	15 g/L

Step 1. $\text{Na}_4\text{Fe}(\text{CN})_6 \cdot 10\text{H}_2\text{O}$ required
 $= 1.47[49 \text{ g/L K}_3\text{Fe}(\text{CN})_6 - 30 \text{ g/L K}_3\text{Fe}(\text{CN})_6] -$
 $[10 \text{ g/L Na}_4\text{Fe}(\text{CN})_6 \cdot 10\text{H}_2\text{O}] + [2 \text{ g/L}]$
 $= 19.9 \text{ g/L}$

Step 2. Total $\text{Na}_4\text{Fe}(\text{CN})_6 \cdot 10\text{H}_2\text{O}$ required for 300 litres
 $= 300 \text{ litres} \times 19.9 \text{ g/L} = 5979 \text{ g}$

Step 3. $\text{K}_2\text{S}_2\text{O}_8$ required
 $= 0.410[49 \text{ K}_3\text{Fe}(\text{CN})_6 - 30 \text{ g/L K}_3\text{Fe}(\text{CN})_6]$
 $= 7.79 \text{ g/L}$

Step 4. Total $\text{K}_2\text{S}_2\text{O}_8$ required for 300 litres
 $300 \text{ litres} \times 7.79 \text{ g/L} = 237 \text{ g}$

Step 5. NaBr required
 $= 26 \text{ g/L NaBr} - 15 \text{ g/L NaBr}$
 $= 11 \text{ g/L}$

Step 6. Total NaBr required for 300 litres
 $= 300 \text{ litres} \times 11 \text{ g/L}$
 $= 3300 \text{ g}$

4. Effect of Sulfate Buildup

As persulfate reacts with ferrocyanide, sulfate ions are produced. Repeated regenerations increase the sulfate concentration up to an equilibrium level that is dependent upon factors such as ferricyanide concentration, film rate through the bleach, replenisher rate, carry-in and carry-out rates, etc. In practice, no difficulty has been experienced in several production processes that have been regenerated repeatedly for months. If the sulfate level of the bleach goes above 50 g/L, it may be necessary to properly dispose of a small percentage of the bleach with each regeneration cycle. Maintaining bleach specific gravity within specification will assure the sulfate is at a satisfactory level.

5. Reaction Rate

Further information on factors influencing the reaction rate is given in published literature.*

* Hutchins, B. A. and West, L.E. "The Preparation or Regeneration of a Silver Bleach Solution by Oxidizing Ferrocyanide with Persulfate," *Journal of the SMPTE*, 66:12 76—768, December 1957.

The Ozone Method for Ferricyanide Bleach Recovery

The method described below utilizes ozone to convert ferrocyanide to ferricyanide in solution.

As mentioned in the preceding section, regeneration of a bleach with persulfate results in a buildup of sulfate ion which can slow the rate of bleaching. Using ozone for bleach regeneration does not cause the build-up of sulfate.

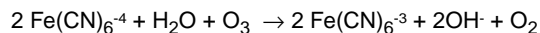
1. Toxicity of Ozone

Ozone gas is corrosive, unstable, and highly reactive. Exposure to ozone at levels higher than 0.1 ppm (part per million) of air can cause severe irritation to the respiratory system.

For environmental and safety reasons, take care to prevent significant amounts of ozone from escaping through the processing laboratory exhaust. The ozone generator should be adjusted carefully to avoid producing more ozone than is actually needed. A cascade system whereby excess ozone from the top of the regeneration tank is pumped into a second regeneration tank can minimize the amount of ozone exhausted. If necessary, escaping ozone can be destroyed with a catalytic filter in the exhaust line.

2. Chemicals

The equation for the oxidation of ferrocyanide by ozone follows.



The pH of the bleach increases as the reaction proceeds. Hence in order to keep the pH close to standard, it is necessary to add acid slowly and continually until regeneration is complete. Using hydrobromic acid* (HBr) furnishes both the bromide ion necessary for bleaching and the hydrogen ion needed to reduce the pH. In theory, there should be no excess bromide after regeneration, since one bromide ion is required for each ferrocyanide ion oxidized to ferricyanide. If in practice there is a slight buildup of bromide ion after repeated regenerations, a small amount of sulfuric acid can be used for pH adjustment without danger of high sulfate buildup. Likewise, small pH adjustments can be made with sodium hydroxide.

Table 5-1 lists some of the forms of ferrocyanide and a typical analysis of shipments received is shown in Table 5-3.

Hydrobromic acid, HBr, is available as 49% HBr in water and is sold in 4-kg containers. Hydrobromic acid is a corrosive liquid. Its properties are similar to those of hydrochloric (muriatic) acid, and it should be handled with the same techniques and precautions as hydrochloric acid.

* **DANGER!** Hydrobromic acid causes severe skin and eye burns. Harmful if inhaled. Do not get in eyes, on skin, or clothing. Avoid breathing vapor. Use with adequate ventilation. Wash thoroughly after handling.

In the discussion that follows, it is assumed that sodium ferrocyanide decahydrate, $\text{Na}_4\text{Fe}(\text{CN})_6 \cdot 10\text{H}_2\text{O}$, is used as the raw material for regeneration of the bleach solution. If other forms of ferrocyanide are used, the calculations given below can be modified accordingly, using the data in Table 5-1.

The quantities of the various ingredients required for a fresh mix and the desired composition of the bleach are given in each process specification module.

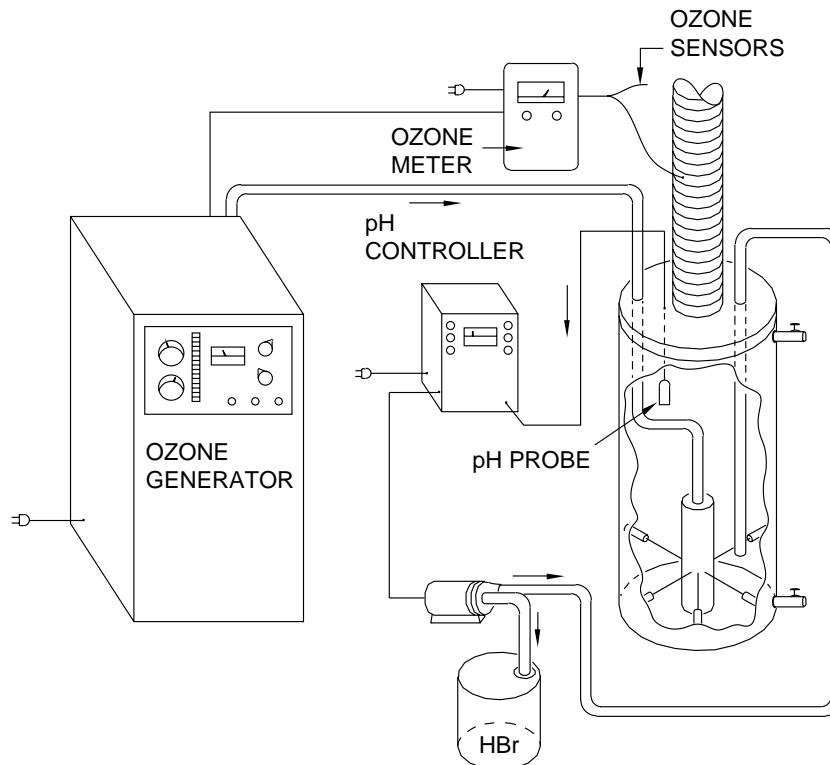
3. Equipment

Figure 5-3 is a schematic of an ozone regeneration system.

The basic equipment necessary for an ozone regeneration system includes:

- Ozone generator
- Automatic pH controller
- Acid pump
- Regeneration tank
- Ozone detector meter
- Sparging system
- Lid or exhaust hood for the tank
- Piping, wiring, etc.

Figure 5-1 Ozone Regeneration System Schematic



Electrolytic Method for Ferricyanide Bleach Recovery

An electrolytic cell can be used to convert ferrocyanide to ferricyanide as described by J. W. Kleppe and C. R. Nash*.

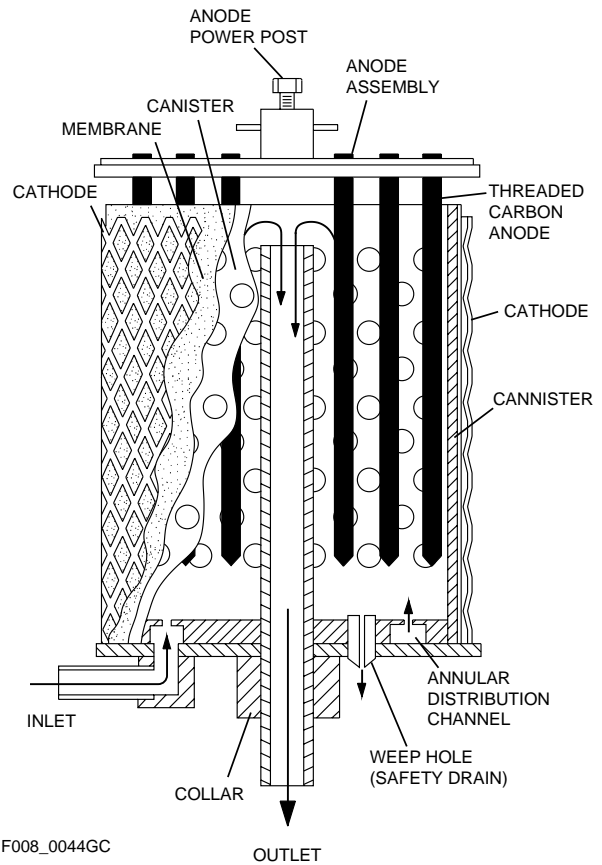
By applying an electric current to a used ferricyanide bleach, the ferrocyanide (formed by the bleaching action of the film) is converted to ferricyanide with the formation of hydroxide ions (OH^-) and hydrogen gas (H_2). The hydroxide ions generated in the bleach solution are neutralized with a solution of hydrobromic acid (HBr), to form bromide ions (Br^-) and water. The small amount of hydrogen gas is vented to the atmosphere. The advantage of this method is it does not produce sulfate which can inhibit bleaching.

The cell, commonly called the Nash cell, consists of:

- a perforated PVC canister
- a carbon anode assembly
- a membrane
- a cathode

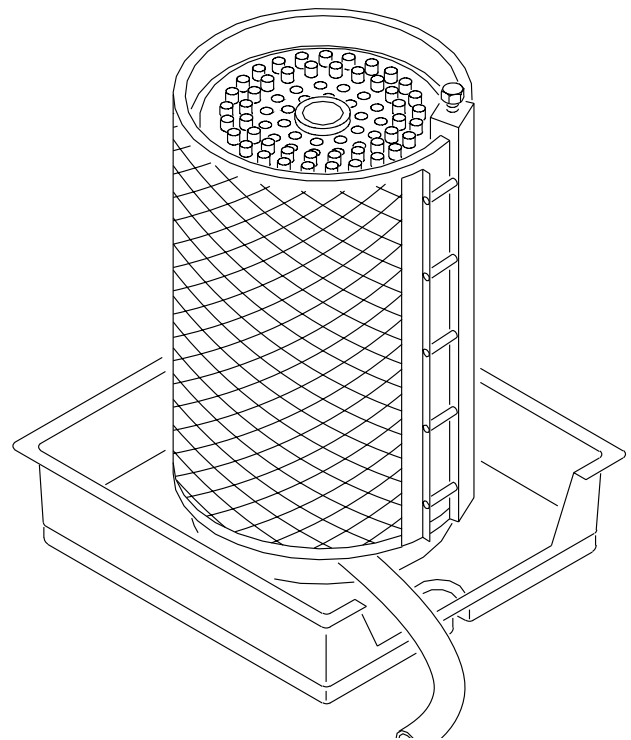
The canister is perforated over most of its surface for ease of solution passage. The model 3 anode assembly is made with three concentric rings of carbon welding rods which extend into the canister. The membrane is wrapped around the outside of the canister, and is sealed to it with an elastomer. The membrane is made of Celgard 2500 microporous polypropylene film or Dynel (a tightly woven cloth) that is resistant to alkaline solutions. The cathode is made of expanded 316 stainless steel sheeting, and is clamped around the membrane. Figure 5-2 is a cross-sectional drawing of the Nash cell and Figures 5-3, 5-4, and 5-5 show the cell assemblies. Exhausted ferricyanide bleach is pumped into the bottom of the canister, and rises upward past the anode rods until it overflows through a central weir pipe. The flow rate is maximized to ensure an ample supply of ferrocyanide ions near the anode rods to promote efficient conversion. A current density of 10 amperes/ ft^2 (1.1 A/ 100 cm^2) of anode surface is maintained within the cell. The voltage drop across the cell is approximately 7 volts DC.

Figure 5-2 A Cross-Sectional View of a Nash Cell



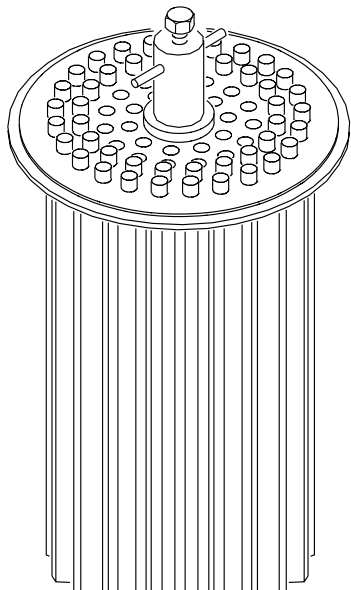
F008_0044GC

Figure 5-3 Nash Cell Canister and Cathode



* J. W. Kleppe and C. R. Nash, "A Simplified Electrolytic Method for Ferricyanide Bleach Regeneration," *Journal of the SMPTE*, **87**:168, April, 1978.

Figure 5-4 Nash Cell Anode Assembly (Model 3)



If a current of 60 to 80 A is applied to a production-size Nash cell having 11 ft² (1 m²) of participating anode surface, then 6 to 8 g/min of sodium ferricyanide monohydrate will be produced (or 10.3 to 13.8 g/min of sodium ferrocyanide decahydrate will be converted to ferricyanide).

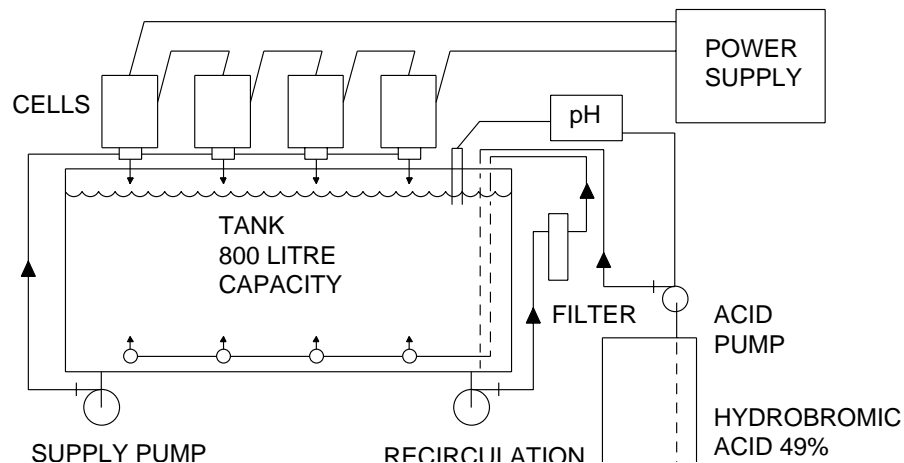
In a typical production set-up, four cells are placed above a tank as shown in Figure 5-5. Recirculation pumps maintain a flow of 10 L/min (2.2 gal/min) through each cell as well as providing filtration and tank agitation. In addition, a pH controller set at 8.2 is used to monitor the recirculated bleach and add hydrobromic acid (49% solution) to neutralize the hydroxide produced from the regeneration process. Hence, when a batch of bleach overflow has been regenerated to replenisher strength (in ferricyanide), the required amount of bromide is already included, and no pH adjustment is necessary. In fact, the pH is stable and buffering agents can be minimized. A four-cell installation can easily support a film processing laboratory that uses 1000 L (220 gal) of ferricyanide bleach each day.

Current Vendors for Nash Cells are:

Kobelcell Inc.
PO Box 206
Clifton, NJ 07011
973-772-4979

The Allen Products Company
180 Wampus Lane
Milford, CT 06460
203-874-2563

Figure 5-5 Typical Four-Cell Electrolytic Regeneration System

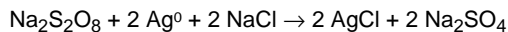


Ferricyanide Wash Treatment— Ion-Exchange Method

The loss of ferricyanide/ferrocyanide (hexacyanoferrates) contained in the ferricyanide bleach wash is an ecological and economical concern. D. Brugger* described the use of a weak-base ion-exchange resin to remove hexacyanoferrates from ferricyanide bleach washes. Brugger used Rohm and Haas Amberlite IRA-67. The resin was regenerated with 4 to 8% sodium hydroxide to elute the hexacyanoferrates. The resulting caustic solution contained about 50 g/L of hexacyanoferrate which is sufficient to allow their direct use in the preparation of a replenisher when regenerated. This technology offers a closed-loop ferricyanide system.

Persulfate Bleach Reconstitution

In the bleach, the silver is converted to silver halide according to the following simplified equation.



In the reaction, sodium persulfate and sodium chloride are consumed, and sodium sulfate is generated. The silver chloride formed in the film is removed by the fixer.

As the sodium sulfate level increases, it tends to slow down the bleaching rate. Sodium sulfate levels greater than 60 g/L in the replenisher could cause bleaching problems. The amount of sodium sulfate in the bleach will depend on the type of film being processed, leader-to-film ratio, carry-in rate, and the number of times the bleach has been reconstituted. Some of the bleach may have to be discarded during reconstitution after the bleach has been reconstituted a number of times. The amount of bleach to be discarded, if any, can be determined by measuring the specific gravity of the collected bleach overflow. Then, by using the equation shown, the amount of the bleach to be discarded can be calculated.

* D. Brugger, "Removal of Hexacyanoferrate from Selected Photographic Process Effluents by Ion-Exchange," 88:237, *Journal of the SMPTE*, April 1979.

Procedure

1. Collect the persulfate bleach overflow (V_c) in a holding tank until a sufficient quantity has been collected to make a new persulfate bleach replenisher (V_r). This volume will depend on the amount of film processed and the size of the holding and replenisher tanks.
2. Measure the specific gravity of the persulfate bleach overflow (K). If it is greater than 1.120, calculate the volume to be discarded (V_d) using the following equation.

$$V_d = V_c [(K - 1.120) / (K - 1.088)]$$

K = specific gravity of collected overflow.

If the specific gravity is less than 1.120, the entire collected overflow can be reconstituted. In a fully reconstituted system, usually 10% to 15% of the bleach may have to be discarded.

3. Analyze the persulfate bleach overflow for sodium persulfate and sodium chloride.
4. Transfer the solution to a mix tank.
5. Calculate the amounts of the various constituents needed to reconstitute the bleach:

a. Sodium Persulfate

g, $\text{Na}_2\text{S}_2\text{O}_8$ required =

$$V_r (\text{g/L Na}_2\text{S}_2\text{O}_8 \text{ in replenisher} - \text{g/L Na}_2\text{S}_2\text{O}_8 \text{ in overflow}) \\ + V_d (\text{g/L Na}_2\text{S}_2\text{O}_8 \text{ in replenisher})$$

b. Sodium Chloride

g, NaCl required =

$$V_r (\text{g/L NaCl in replenisher} - \text{g/L NaCl in overflow}) \\ + V_d (\text{g/L NaCl in replenisher})$$

c. Sodium Dihydrogen Phosphate

g, NaH_2PO_4 required =

$$V_r (10\% \text{ g/L NaH}_2\text{PO}_4 \text{ in replenisher}) \\ + V_d (\text{g/L NaH}_2\text{PO}_4 \text{ in replenisher})$$

d. Chlorine Scavenger

g, scavenger required* =

$$V_r (15\% \text{ g/L scavenger in replenisher}) \\ + V_d (\text{g/L scavenger in replenisher})$$

where:

V_r = Volume being reconstituted to replenisher

V_d = Volume discarded

* If Chlorine is detected in the bleach, additional chlorine scavenger should be added.

6. Add a volume of water equal to the volume of persulfate bleach that was discarded. Then add the calculated amount of the various chemicals and mix the solution. If after a number of reconstitutions a loss in volume occurs, the volume should be made up by adding fresh replenisher.
7. After all of the additions have been made, adjust the pH to 2.5 ± 0.2 by adding sodium hydroxide or phosphoric acid. Analyze for the critical constituents, pH, and specific gravity, and adjust if necessary. If the specific gravity is greater than 1.140, additional bleach must be discarded and fresh bleach added.
Pump the certified reconstituted bleach to the bleach replenisher holding tank.

Bleach Accelerator Reconstitution

The accelerator for persulfate bleach can also be reconstituted. During the bleach accelerator step of the process, sodium metabisulfite reacts with the KODAK Persulfate Bleach Accelerator PBA-1 to form the active accelerator species. This particular component is adsorbed to the metallic silver in the film as the film passes through the accelerator. This enables the persulfate to oxidize the silver to silver chloride in the persulfate bleach.

Sodium metabisulfite must be added to make up for the bisulfite that is oxidized, and also to react with the PBA-1 added to form the active accelerator species.

Procedure

1. Collect the accelerator tank's overflow in a holding tank until a sufficient volume has been collected to make a new accelerator replenisher. The volume will depend on the amount of film processed and the size of the holding and replenisher tanks.

Note: Because of the unpleasant odor of the accelerator, and to reduce chemical oxidation, it is advisable to have the overflow enter the bottom of the collection tank and to use a floating cover and lid.

2. Analyze the accelerator overflow for PBA-1 and sodium metabisulfite.
3. Transfer the solution to a mix tank.
4. Calculate the amounts of the various constituents needed.
 - a. PBA-1

g, PBA-1 required =

{(g/L, PBA-1 [from replenisher specification])

– (g/L PBA-1 in overflow)}(volume reconstituted)

- b. Sodium Metabisulfite (anhydrous) $\text{Na}_2\text{S}_2\text{O}_5$

g, $\text{Na}_2\text{S}_2\text{O}_5$ required =

{(g/L $\text{Na}_2\text{S}_2\text{O}_5$ [from replenisher specification])

– (g/L $\text{Na}_2\text{S}_2\text{O}_5$ in overflow)

+ 0.45(g/L PBA-1 [from replenisher specification])

– (g/L PBA-1 in overflow)}(volume reconstituted)

5. Add the calculated amounts of various chemicals and mix the solution.

Note: Because of the unpleasant odor of the accelerator, the mixing should be done under an exhaust hood. Avoid overmixing because of the possibility of oxidizing the accelerator agent, PBA-1.

If after a number of reconstitutions a loss in volume occurs, the volume loss should be made up by adding fresh replenisher.

6. After all the additions have been made, adjust the pH to the formula specification using either sodium hydroxide or sulfuric acid.
7. Analyze the solution for the critical constituents and pH; readjust if necessary.
8. Pump the reconstituted accelerator to the accelerator replenisher tank.

Process ECN-2 "UL" Bleach Reconstitution

Procedure

1. Collect the bleach overflow in a holding tank until a sufficient volume has been collected to make a replenisher.
2. Analyze the overflow for total iron, iron II and ammonium bromide.
3. If the iron II concentration is less than 0.5 g/L, proceed to the next step. If the level is greater than 0.5 g/L (an uncommon occurrence), aerate the overflow until the iron II level drops below 0.5 g/L.
4. Calculate the amounts of makeup chemicals needed to make a replenisher. See the sample calculations following.
5. Add the make-up chemicals to the collected overflow, allowing time for dissolution of each solid before the next chemical is added. If ferric nitrate solid is used, predissolve it in water before addition. Ferric nitrate is added after the acetic acid to prevent possible precipitation should the solution pH be too high. Acetic acid and ammonium hydroxide should be added below the solution surface to prevent noxious fumes from forming.

Mixing Order	
Constituent	Amount
Proxel GXL	0.03 mL/L*
KODAK Chelating Agent No. 1	Calc. Amt.
Ammonium hydroxide	9 mL/L
Ammonium bromide	Calc. Amt.
Glacial acetic acid	4.5 mL/L
Ferric nitrate	Calc. Amt.

* Adding 0.03 mL/L to a regenerated solution should maintain a replenisher level of 0.10 mL/L. If this level fails to control biological problems, it may be doubled without photographic effect.

6. Adjust pH to replenisher specification. Use nitric acid, sulfuric acid or ammonium hydroxide as necessary. Do not use sodium hydroxide or phosphoric acid.

7. Pump the reconstituted replenisher to the replenisher holding tank.

EXAMPLE: Calculation of Make-up Chemicals

A total of 500 litres of bleach overflow is available and was analyzed as follows.

Constituent	Overflow Level	Replenisher Level	Amount Needed
Fe II	< 0.5 g/L	< 0.5 g/L	No aeration
Total Iron	4.0 g/L	6.35 g/L	2.35 g/L
NH ₄ Br	32.5 g/L	51.6 g/L	19.1 g/L

Step 1. Determine the multiplication factor "F"

$$F = (\text{g/L Total Iron in replen.}) - (\text{g/L Total Iron in overflow})$$

$$F = 6.35 - 4.0 = 2.35$$

Step 2. Calculate the amount of ferric nitrate nonahydrate to add:

$$\begin{aligned} \text{a. Fe(NO}_3)_3 \cdot 9 \text{H}_2\text{O} &= (7.2) (F) (\text{volume of overflow}) \\ &= (7.2) (2.35) (500 \text{ L}) = 8460 \text{ g} \\ &\text{or} \end{aligned}$$

$$\begin{aligned} \text{b. Fe(NO}_3)_3, &= (6.54) (2.35) (500 \text{ L}) \\ 45\% \text{ solution} &= 7684.5 \text{ mL} \end{aligned}$$

Step 3. Calculate the amount of Chelating Agent, No. 1 to add:

$$\begin{aligned} \text{Chelating Agent,} &= (6.0) (F) (\text{volume of overflow}) \\ \text{No. 1} &= (6.0) (2.35) (500 \text{ L}) \\ &= 7050 \text{ g} \end{aligned}$$

Step 4. Calculate the amount of ammonium bromide to add:

$$\begin{aligned} \text{NH}_4\text{Br} &= [(\text{g/L NH}_4\text{Br in replenisher}) \\ &\quad - (\text{g/L NH}_4\text{Br in overflow})] \\ &\quad \times (\text{volume of overflow}) \\ &= [(51.6 \text{ g/L} - 32.5 \text{ g/L})] [500 \text{ L}] \\ &= 9550 \text{ g} \end{aligned}$$

TECHNICAL ASSISTANCE FROM KODAK

The above chemical recovery procedures are presented mainly as suggested outlines. Because of the individualities of processing laboratories and of changing technologies, it is further suggested that a processing laboratory contact their local Kodak Entertainment Imaging representative when that laboratory considers any of these recovery procedures. Your Kodak Entertainment Imaging representative will be pleased to assist you in obtaining current technology and assist you in implementing that technology.

MORE INFORMATION

For more information on motion picture products, call or write to the Entertainment Imaging office nearest you.

Or access Kodak's home page on the Internet, web site address—

<http://www.kodak.com/go/motion>

You may want to bookmark our location so you can find us more easily.

Processing KODAK Motion Picture Films, Module 5

Chemical Recovery Procedures

