

# TRAMFLOC® 900 SERIES JAR TESTING PROCEDURE for METAL PRECIPITANTS

Precipitation is the chemical conversion of soluble substances (including metals) into insoluble particles. Coagulation and flocculation cause a chemical reaction that promotes the formation, agglomeration or clumping of such particles to facilitate their removal from solution.

The amount or dosage of a precipitant, coagulant and/or flocculant required to precipitate and remove metals in wastewater solutions is not only dependent on the concentration of such metals in solution, but also on several other factors. To optimize the dosage, the following parameters must be considered:

1. type of coagulants and flocculants applied
2. sequence of reagent addition

1. solution pH.
2. chemical used to adjust the pH (i.e. NaOH, lime,  $Mg(OH)_2$ ,  $Na_2CO_3$ ).
3. different types (and concentrations) of metals present in solution.
4. amount and types of chelants and complexing agents present in solution.
5. amount of residual oxidizers present in solution.

## JAR TESTING PROCEDURE

As indicated above, untreated process wastewaters may contain chemicals other than dissolved metals that will affect the treatment methodology. Therefore, the procedure which follows provides a starting point and adjustments may be required to achieve the desired results.

### RECOMMENDED EQUIPMENT:

1. pH meter with electrode to monitor pH.
2. ORP meter with electrode to monitor the reduction reaction.
3. 300 ml - 1000 ml Beakers, clear plastic or glass.
4. Magnetic Stirrer/ jar mixer or equivalent.
5. Syringes for adding chemical reagents.
6. Laboratory Type Filter.
7. Metals Test Kit, ICAP, or AA Spectrophotometer, etc.

### CHEMICAL REAGENTS:

1. Sodium-Hydroxide (Caustic-Soda) solution.
2. Sulfuric-Acid solution.
3. Tramfloc® 900 Series samples.
4. Tramfloc® 500, 700 and 800 Series coagulant solution(s) – see product bulletin for suggested dilution ratio.
5. Tramfloc® 100, 200, 300, 400 Series flocculant solution(s) – see product bulletin for suggested dilution ratio.

### PROCEDURE:

1. Pour a sample of untreated wastewater into a beaker (ex 1000 ml). While mixing, adjust the pH using caustic soda or sulfuric acid to the optimum pH for hydroxide precipitation of mixed metals, i.e. pH 9.0.
2. While mixing the sample, use a syringe to add the precipitant until the ORP value drops rapidly by 150mV (typically to -250 mV). If an ORP meter with electrode is not available, use several beakers and add different amounts of precipitant to each beaker.

Please note that, although the precipitating reactions appear to be instantaneous, a retention time of up to 15 minutes may be required to obtain a complete reaction.

3. Add 100 to 200 ppm (approx. 0.1-0.2mls neat reagent to 1000ml) of coagulant solution. Mix at high speed for 1 to 3 minutes. Turn off mixer and observe the coagulation (agglomeration) of the precipitated particles. Monitor the pH, as certain coagulants are acidic in nature and will alter the pH. If the particles appear to be coagulating but need assistance to accelerate their settling, a flocculant may be added to the solution followed by a slow mixing to allow for floc building. If the settling action is too slow or incomplete, redo the test using more of the coagulant solution

4. After several minutes, a sample of clear supernatant may be taken for metals test, or the entire contents of the beaker(s) may be filtered to remove solids, then the filtrate tested/analyzed.

Note: When the supernatant has a yellow or orange tint (similar to the color of the precipitant), that is an indication of overdosing the precipitant. If necessary, dilute the precipitant before adding it to the wastewater. Make note of the dilution ratio for determining the optimum full-scale dosage. Overdosing the precipitant can also cause a significant increase of colloidal particles and may interfere with the normal coagulation/flocculation reactions.

To project the dosage results from a jar test to full scale, the following information may be helpful:

1 drop = 0.05 ml

1 drop per liter = 50 mg/l (ppm)

### **PRECIPITANT CONTROL SYSTEM**

Precipitant control systems utilize an electrode for accurate measurement (in millivolts) of the reduction reaction of precipitants in wastewater. The electrode signal is monitored by the controller that controls the metering pump for dosing the required amount of precipitant. Systems are also available for pH adjustment under electrode control.

### **pH**

The solubility of metallic ions/particles is pH dependent. That is, dissolved heavy metal ions can be precipitated chemically by adjusting the pH of a wastewater stream. The pH is important because all metals have a pH at which their solubility is minimal. Although this pH differs for all metals, it generally lies between 7.5 and 11. Since most wastewaters contain a variety of metals, it is difficult choosing the optimum pH at which their solubility is minimal. See attached chart for optimum removal ranges.

Precipitants will simultaneously precipitate a variety of metals at any given pH within the above range. These precipitants, with low solubility, can achieve very high removal efficiencies.

When used as a "polishing" precipitant, the dosage of a precipitant can be lowered depending on the quantity of metals that are precipitated as hydroxides by pH adjustment. While a pH of 8.5 is normally recommended for the polishing effect -- the pH value will vary depending on the presence of chelating and/or complexing agents in the wastewater. A jar test procedure as described above can help to establish the optimum pH within the 7.5 to 11 range. For example, if nickel is the main metal of concern, adjust the final pH to 10 to fully optimize the effects of hydroxide precipitation.

For adjusting pH, sodium hydroxide is recommended. However, other common chemicals can be used -- such as soda ash and lime. For some applications (nickel complexes) magnesium hydroxide is an effective reagent.

### **RESIDUAL OXIDIZERS**

When a precipitant is added to a wastewater stream containing residual oxidizers along with dissolved metals, dual reactions occur. These reactions are: 1) the reduction of the oxidizers and 2) the precipitation of the metals. This, of course, increases the amount of precipitant required for total metals precipitation.

To optimize the usage of a precipitant, adding a reducer (i.e. sodium metabisulfite) before the precipitant addition will remove the oxidizers from the wastewater solution.

### **FLOCCULANTS/COAGULANTS**

Characteristically the insoluble particles formed by adding a precipitating reagent to a wastewater solution are very small and suspended in the solution (colloidal). The suspended stability of such particles is due to both their small size and to the electrical charge (usually negative) on their surface causing them to repel their neighboring particles.

To promote the removal of these suspended particles use chemical coagulation and/or flocculation. Adding coagulants to the wastewater creates a chemical reaction in which the repulsive electrical charges surrounding colloidal particles are neutralized, allowing the particles to stick together creating clumps or flocs (often called pinfloc). When required, flocculants with an anionic charge are commonly used to facilitate the agglomeration of the floc and its settling. The flocculants act like a net, bringing together a number of the now neutralized particles, allowing for separation. The aggregation of these particles into larger floc permits their separation from solution by sedimentation, flotation, filtration or straining.

Some precipitants contain cationic polymers that neutralize the precipitated particles. The cations (positive charges) from the polymer reduce or reverse the negative charges of the precipitate which, in turn, permits the coagulation and flocculation of the particles.

### **COMPLEXING AND CHELATING AGENTS**

Some process wastewaters include complexing and chelating agents, which bond to the metal ions making precipitation difficult, if not impossible, for many precipitating reagents.

There are a variety of different precipitants capable of breaking many of these bonding agents and thereby precipitating the metal ions without the addition of other chemicals. In some instances a combination of pH adjustments and varying reaction times may be required along with a precipitant and flocculants.

### **ODOR CONTROL**

By oxidation, some precipitants have a characteristic sulfide odor. This odor may be eliminated or minimized by following simple control procedures:

- a. Assure that the waste stream or sludge contains no oxidizers.
- b. Dose the precipitant below the wastewater surface to prevent surface air oxidation.
- c. Use below surface mechanical agitation for mixing. Do not use air agitation.
- d. Do not overdose. The Reagent Control System mentioned above will prevent overdosing.
- e. Maintain a wastewater pH of 7.0 or above.
- f. Utilize mechanical exhaust ventilation.
- g. Use closed top reaction/mixing tanks when possible.
- h. After wastewater clarification, any sulfide odor or color is a positive sign of overdose. If impractical to control dosing, excess may be easily removed with a minimal injection of peroxide or hypochlorite oxidant into the effluent.

Solubility of Metals VS pH

