Measurement Tools for Coagulation and Flocculation

Measuring Aluminum and Iron

When iron or aluminum chemicals are used as coagulants, the metal should be measured in the raw water, filter influent and filter effluent. The iron or aluminum concentration in the filter effluent should be no more than and preferably less than, the raw water and filter influent concentrations. For most water the FerroVer® 3 Iron Reagent (1, 10 Phenanthroline method) for total iron is appropriate for iron and the AluVer 3® Aluminum Reagent (Aluminon method) is appropriate for aluminum. For low level iron use the FerroZine™ Iron Reagent and for low level aluminum the Eriochrome Cyanide R (ECR) method (ECR may not be used with DR900's). When measuring aluminum, fluoride interferes (and vise versa). All aluminum measurements must be corrected for fluoride interference. Once the fluoride is measured, use the fluoride interference correction chart in the method. The correction charts for the AluVer 3 and the ECR method are different. Care must be taken to use the correct chart.

Use the SPADNS 2 (arsenic-free) or fluoride electrode to measure fluoride. Fluoride must be measured regardless of whether or not the utility fluoridates. Fluoride exists naturally in every water source on earth – ground or surface. Natural fluoride concentration may range from 0.1 to over 10 mg/l.

Iron and Aluminum Reagents				Instrument*
Test	Reagent	Range – mg/l	Cat. No.	
Iron (total)	FerroVer PP	0.02 - 3.00	2105769	C, S, PC
	FerroVer AV	0.02 - 3.00	2507025	C, S, PC
Iron	FerroZine	0.009 - 1.400	230166	C**, S
Aluminum	AluVer 3	0.008 - 0.800	2242000	C, S, PC
Aluminum	ECR	0.002 - 0.250	2603700	S
* PC – Pocket Colorimeter C – colorimeter S – spectrophotometer **DR900				

Figure 17: Reagents for Iron and Aluminum Tests

The Jar Test

The jar test is the most basic test for control of coagulation/flocculation/filtration and is completed with a multiple stirrer such as the Phipps Bird. It would seem a test and an apparatus so simple would have existed for many years. Yet, at least in the water industry, the multiple place stirrers can be traced to as recently as about 1920. An early attempt to conduct the equivalent of today's jar test but using a single glass dish is recorded just a few years earlier.

Figure 18: Jar test apparatus
Phipps Bird 6-Place Programmable Multiple Stirrer with 1-liter round glass beakers. Hach Company stock photo

The jar test can be performed with round jars, square jars, ½ L jars, 1 L jars, 2 L Wagner Jars or for that matter, mayonnaise jars.

- Features the customer should look for are a back panel, typically black to view the water in the jars and a white or lighted base.
- You may encounter Hach brand multiple stirrers that used ½ liter jars. The product was discontinued several years ago.
- The Phipps Bird has a lighted base under the jars.
- When using the lighted base, the light should be left off except when observing the floc formation or settling process. When the lights are on, the base will generate heat sufficient to create convection currents. Changing the temperature of the water during coagulation and flocculation will lead to non-representative floc formation and the convection currents will interfere with settling.

Wagner™ Jar – p/n 41170-00, 2-liter square plastic floc jar for the jar test. The Wagner Jar has a tap near the bottom of the jar to facilitate withdrawal of a sample for further testing of pH, turbidity, alkalinity, streaming current, zeta potential, etc. Photo by author.

Figure 19: Wagner™ Jar

The jar test is as much art as it is science. A different coagulant dose is added to each of the 4 or 6 jars. A short period of rapid mixing (for coagulation) and then a longer period of slow mixing (flocculation) occur. Last, a no-stirring quiescent period permits settling. Chemicals for pH adjustment, coagulant aids; ballasting substances (carbon, clay, etc.) also may be added to the jars. It is important to vary only one parameter at a time!

During stirring and the quiescent periods the operator or lab tech will observe the jar for floc formation and settling rate and use this information to then make chemical dose changes to the process. Each plant operator and chemist (or university professor, engineer, chemical sales person, etc) is very sensitive about their particular technique so

one should tread carefully in suggesting any variation in their technique. Users will be adamant about use of a square vs. round jar, big jar or little jar, this rapid mix period vs. another, the slow stir speed, etc. They will be absolutely sure their combination of art and science is THE way to do it.

The jar test is an attempt to simulate in a one or two liter jar what is going on in a basin 20'X30'X15' containing 67,000 gallons. The jar test is also an attempt to simulate with little 1"x2" paddle stirrers and jars the mixing energy with a train of huge paddles extending the entire length of a 40 foot long flocculation chamber and maybe 15 feet in diameter.

It is as much an art as a science because operators have to learn to interpret "when my little jar looks this way, my big basin will look this way." The more measurements are made; the better the operator or lab person can interpret the jar test results – based more on measurement (science) and less on art. This is important because filter performance is directly affected by how well the floc forms, settles and withstands shearing effects during mixing and filtration. Apparatus to enhance the jar test include a wide array of other Hach products:

- Measure pH with the HQd series or SensIon series pH meter and probe – One must measure pH especially with aluminum or iron salts (aluminum sulfate, liquid alum, ferric chloride, ferric sulfate).
  - Coagulants have an optimal pH range in which they should be used.
  - Aluminum sulfate or liquid alum work well from a pH of about 5.5 (optimum color removal) to the low 7's.
  - Iron compounds – ferric sulfate and ferric chloride – operate well over a much wider range of pH well into the high 8's.
  - Monitor the endpoint of the alkalinity titration with pH measurement, see below.
- Measure alkalinity with the Digital Titrator® and associated reagents. Use of the metallic salts as coagulants consumes alkalinity.
  - As a rule of thumb, one must have (numerically) ½ the alkalinity of the amount of alum or ferric sulfate coagulant dose needed. If a dose of 20 mg/l of alum is needed, then the alkalinity must be at least 10 mg/l. For ferric chloride, it's nearly 1:1. That is, for a dose of 30 mg/l ferric chloride, at least 30 mg/l of alkalinity must be available.
  - Customers should be encouraged to monitor the alkalinity titration with pH measurement rather than trying to observe the color changes. Whether using methyl orange or bromcresol green/methyl red indicators, it is difficult for many if not most people to see the subtle color changes.
- Measure turbidity with a lab or portable turbidimeter (2100P, 2100Q, 2100N or 2100AN). Measure the turbidity at the beginning and the turbidity of the supernatant at the end of the settling period. Filter a portion of the supernatant through medium speed filter paper and again measure the turbidity.
- Both a large (1-10ml) and small (0.1-1.0 ml) TenSette® Pipet – Use the TenSette to:
  - Prepare standard jar test solutions such that each ml of stock solution added to a jar of sample to be tested results in a concentration of 10 mg/l. Add the number

of grams or milliliters specified to 300 ml of dilution water. Mix and dilute up to one liter (1000 ml) for the stock solution.

For dry alum or iron coagulants:		
Size of sample for jar test	Milligrams of dry alum or iron coagulants for each 1 liter of stock solution	Concentration resulting when 1 ml of stock solution is added to the water to be tested
0.5 liter jars	5,000 mg (5 g)	10 mg/l
1.0 liter jars	10,000 mg (10 g)	
2.0 liter jars	20,000 mg (20 g)	

For liquid alum or liquid ferric chloride			
Size of sample for jar test	ml of liquid alum (assuming a 48% solution) to prepare 1 liter of stock solution	ml of ferric chloride (assuming a 40% solution) to prepare 1 liter of stock solution	Concentration resulting when 1 ml of stock solution is added to the water to be tested
0.5 liter jars	7.8 ml	8.9 ml	10 mg/l
1.0 liter jars	15.6 ml	17.8 ml	
2.0 liter jars	31.2 ml	35.7 ml	

Figure 20: Preparation of stock jar test solutions

- Use the TenSette pipet to dose each of the jars with the appropriate coagulant/coagulant aid dose.
  - Use the 1-10 TenSette pipet for 10 mg/l increments or
  - Use the 0.1-1.0 TenSette pipet for 1 mg/l increments.
  - Realistically 1 mg/l increments are about all the resolution one can achieve with the jar test.
- Use to withdraw aliquot of supernatant
  - For testing turbidity and for a filtration test
  - Alkalinity measurement

- Plastic funnels and medium speed filter paper. Filtering supernatant through medium speed filter paper is a surprisingly good simulation of what can be achieved with filtration in the plant's filters. Measure turbidity before filtration to determine effectiveness of settling and then after filtration to estimate how well the sample will hold up (floc tough enough to withstand the shearing forces) during filtration.

Figure 21: Six-place assembly for filtering samples after a jar test. Photo by author.

When a treatment plant uses liquid alum, or other liquid coagulant, coagulant aids or filter aids, the products can vary in percent of active component from manufacturer to manufacturer and in some cases from lot to lot. The percent concentration must be known before one can calculate how to make a standard solution (as above) for these liquid products.

Equipment and Apparatus for the Jar Test		
Cat. No.	Description	Use
Multiple Stirrer, choose one of the following		
2631700	Phipps Bird 6-Place Programmable Multiple Stirrer supplied with 6 1-liter round glass beakers,	Multiple stirrer for jar test
2703800	6-place nonprogrammable w/o beakers	Multiple stirrer for jar test
2704000	4-place programmable w/o beakers	Multiple stirrer for jar test
2703900	4-place nonprogrammable w/o beakers	Multiple stirrer for jar test
4117000	Wagner Jar	2-liter square plastic floc jar
50083	Glass Beaker, round, 1 liter, pk/6	Jar test w round jars
pH Meter, choose one of the following or better		
pH	SensION+ 3w/ pH Combination Electrode	Measure pH/ alkalinity end point
8505900	HQ11d pH meter w/ gel-filled combination pH electrode, buffers and probe stand	Measure pH/ alkalinity end point
Digital Titrator, cartridges and indicators		
2270900	Universal Digital Titrator Kit w/ manual, 100 ml graduated cylinder, 125 and 250 ml Erlenmeyer flasks	Alkalinity test
1438801	0.1600 N H_2SO_4 Titration Cartridge	Low range alkalinity test
1438901	1.600 N H_2SO_4 Titration Cartridge	High range alkalinity test
94299	Phenolphthalein PP, pk/100	Indicator for p-alkalinity test
94399	Bromcresol Green Methyl Red PP pk/100	Indicator for total alkalinity test
2271900	Reagent Set for Alkalinity – includes titration cartridges and indicators above.	
Other Instruments and Apparatus		
2100Q01 4700000 4700100	2100QPortable Turbidimeter OR 2100N Laboratory Turbidimeter OR 2100AN Laboratory Turbidimeter	Test clarity of supernatant and filtrate from jar test
19700-01	TenSette Pipet, 0.1-1.0 in	Jar test chemical dosing
2185696	Pipet tips, 0.1-1.0	
1970010	TenSette Pipet, 1.0-10.0 in	Jar test chemical dosing, transfer supernatant for further testing
2199796	Pipet tips, 1.0-10.0	
108368	Funnel, each	Filtration testing of the supernatant
69257	Filters, pleated	Filtration testing of the supernatant

Figure 22: Equipment and apparatus for the jar test

Zeta Potential

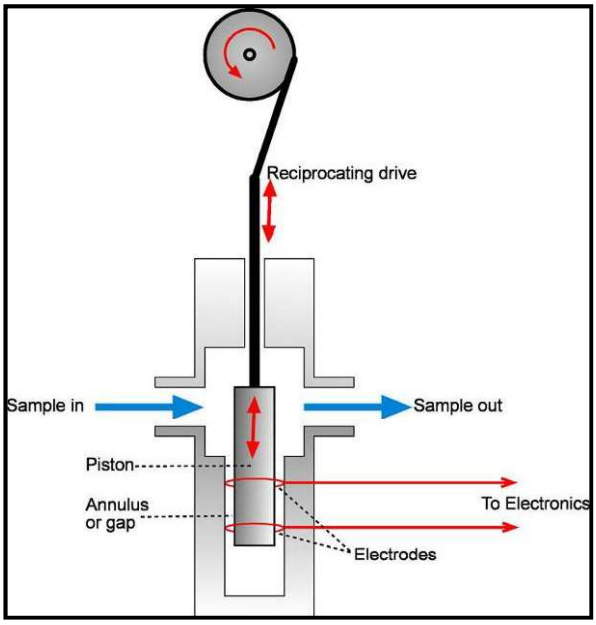
Zeta potential is a test to quantify the charge on colloids in the water to be treated. Ideally one would like to be able to monitor the zeta potential of the raw water and use with feed-forward control to set the coagulant dosage. In practice it is nearly universally used for feed-back control. That is, after coagulant addition a sample can be immediately taken to determine the charge neutralization and then that information used to adjust the coagulant dose. Zeta potential of zero is theoretically ideal. In practice most utilities will have a slightly negative zeta potential after coagulation. A positive zeta potential indicates a likely overfeed of coagulant. There are several drawbacks to use of zeta potential.

- It is a laboratory, grab sample tool.
- Instruments for measuring zeta potential are relatively expensive, typically in excess of $15,000.
- While they are not complicated tools, learning to interpret the data from a zeta meter is often time consuming.
- There is not a clear cut procedure for how to interpret zeta potential measurements and apply them to the process. Every treatment plant is different and each water source is different.
- Learning what zeta potential is ideal for a particular treatment plant and water involves repeated testing and observation. A good place to start is with the jar test. If a treatment plant has learned to interpret the jar test, then the zeta potential of the dosage selected during the jar test can be measured. A sample is also taken from the application point of the coagulant in the process immediately after rapid mixing. If the plant sample has a different zeta potential than the jar, the coagulant feed can be adjusted to match the zeta potential of the jar test.
- After further observation of the process quality, additional minor adjustments can be tried. Again the process should be observed and measured. The results are used to refine judgments made both in the process and in interpretation of the jar test.
- This trial and error process carried out over time in a disciplined manner will result in a better optimized chemical feed. While a jar test may indicate a coagulant dose to the nearest 2-3 mg/l, using zeta potential can refine that judgment to within tenths of an mg/l of coagulant. The time invested is well spent as savings in several areas of the treatment process will result. Properly applied, the return on investment can easily be less than a year.
- The bottom line is few utilities use measurement of zeta potential. Cost, complexity, lack of understanding of the principle, and lack of the desire for disciplined study have limited the use of this very valuable tool.

Streaming Current

Streaming current is an on-line measurement of how well charge neutralization has occurred. It is not the same as zeta potential but can provide much the same level of information for process control. It has both drawbacks and advantages over zeta potential measurement.

Figure 23: Schematic diagram of a streaming surrent sensor (Edney)



The SCM is based on the effect where the walls of the capillaries through which the colloidal material flows quickly gain a coating of particles and take on the surface charge characteristics of these particles. The SC sensor consists of a piston and a close-ended chamber. A narrow gap, an annulus 200-500um wide, exists between the piston and the walls of the chamber. The piston is driven up and down at a fixed frequency, typically 4-5 strokes per second, forcing sample water in and out of the chamber through the annulus.

As the piston and chamber surfaces are coated with charged particles, the water flowing rapidly up and down through the annulus results in displacement of the counter-ions. The SC signal measured by electrodes in the annulus is proportional to the water velocity and therefore alternates in time with the piston. This signal is typically in the range of 0.05uA to 5uA depending on the particular conditions. Measurement of SC in a closed chamber has several advantages compared to measurement directly in a flowing stream:

- **The closed end is electrically isolated and removes problems caused by large potentials in the process stream from other sources.**
- **The signal is alternating at the frequency of the piston. This allows it to be separated from external noise and offset caused by electrode drift and dissymmetry.**
- **Practical aspects of instrumentation, such as that the closed chamber can be shielded from electromagnetic interference and cleaned easily. (Edney)**

- Streaming current is an on-line measurement providing continuous feedback
- Optimally, one would use both zeta potential and streaming current measurement.
- pH of the process must be controlled for effective coagulation and flocculation. If pH is not controlled, it will be difficult to achieve benefit of a streaming current meter.
- Streaming current is strongly influenced by salinity, conductivity and pH variations. If the pH, conductivity, or salinity of the water to be treated is highly variable, streaming current measurements may have limited value or will be problematic.
- On the other hand, if streaming current has worked well for a period of time and suddenly seems to not correlate well, that is a signal of a significant change in water quality that should be investigated and understood.
- Streaming current requires much less effort to learn to use than a zeta potential measurement.
- Streaming current meters are less expensive than zeta meters.
- One of the greatest challenges of streaming current application is locating the right point of measurement. The sample must be as close as possible to the point of application of the coagulant but after it is well mixed. Often the ideal point is not accessible.

Figure 24: Accufloc Streaming Current Monitor.

Clarification

Clarifiers (sedimentation basins) provide a quiescent, low velocity (typically <0.5 ft/sec) area where the solid/floc mixture can settle from solution. A variety of designs are in use – square, rectangular and round. Many are fitted with proprietary modifications to improve settling performance. Detention time in a clarifier is typically between 30 and 45 minutes.

Some will be described below. Characteristics common to all will be:

- A means to introduce the flocculated water uniformly to the clarifier
- A means to collect and discharge accumulated solids
- A means to collect clear effluent and transport to the filtration step
- A means to ensure complete treatment and minimize or prevent short circuiting

Perhaps the simplest designs one will encounter is a rectangular basin with sloped floor equipped with manual or automatic means of solids collection and withdrawal.

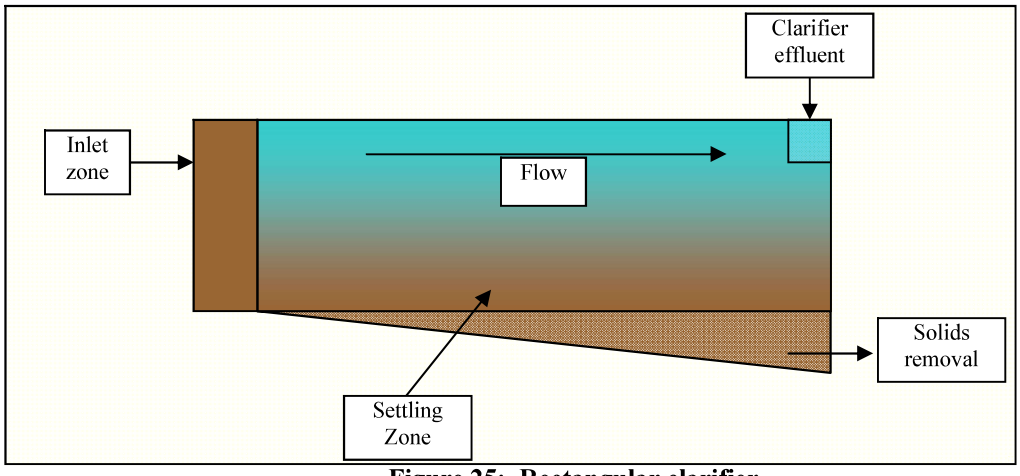


Figure 25: Rectangular clarifier