

Pulp & Paper Division

ECA 2000 P OPERATIONS MANUAL

**Electrokinetic Charge Analyzer
Laboratory Model**

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SAFETY PRECAUTIONS

BEFORE ATTEMPTING TO UNPACK, SET UP, OR OPERATE THIS INSTRUMENT, PLEASE READ THIS ENTIRE MANUAL.

MAKE CERTAIN THE UNIT IS DISCONNECTED FROM THE POWER SOURCE BEFORE ATTEMPTING TO SERVICE OR REMOVE ANY COMPONENT.

FAILURE TO FOLLOW THESE PRECAUTIONS COULD RESULT IN PERSONAL INJURY OR DAMAGE TO THE EQUIPMENT.

WARRANTY INFORMATION

Warranty: Chemtrac[®] Systems, Inc. warrants its product to be free of defects in material and workmanship for a period of one (1) year from date of shipment to the original Customer. Upon receipt of written notice from the Customer, Chemtrac[®] Systems, Inc. shall repair or replace (at the discretion of Chemtrac[®] Systems, Inc.) the defective equipment or components. Chemtrac[®] Systems, Inc. assumes no responsibility for equipment damage or failure caused by:

- A. Improper installation, operation, or maintenance of the equipment.
- B. Abnormal wear and tear on moving parts caused by some processes.
- C. Acts of nature (i.e. lightning, flooding, etc.)

This warranty represents the exclusive remedy of damage or failure of the equipment. Under no circumstances shall Chemtrac[®] Systems, Inc. be liable for any special, incidental, or consequential damage, such as loss of production, profits or product quality. The warranty cannot be guaranteed if the Customer fails to service and maintain the equipment in accordance with Chemtrac[®] System's written instructions and policies, as stated in the Operations Manual.

1.0 ELECTROKINETIC CHARGE ANALYSIS

1.1 INTRODUCTION

Although the streaming current principle is not completely understood, the Electrokinetic Charge Analyzer (ECA) can provide valuable data for colloidal systems. The ECA's output is the **STREAMING CURRENT VALUE (SCV)**, a term which is commonly interchanged with "charge" or "charge measurement." It is assumed that a streaming current is generated by electrically charged particles in the sample that are momentarily attached to the cylinder walls within the probe. A reciprocating piston causes fluid motion, which shears the loosely bound counter-ions away from the particles attached to the cylinder walls. Electrodes in the cylinder measure this current generated by the sheared counter-ions. The signal is electronically processed and the output is the SCV.

The loosely bound counter-ions, mentioned above, are a component of the "double layer." This concept is described in Section 1.3. Both streaming current and zeta potential are based upon the double layer principle. Therefore, the streaming current value produced by the ECA is comparable to the well-known zeta potential.

1.2 BACKGROUND

As originally developed, the streaming potential measurement utilized a capillary passage or porous plug of the material of concern, through which the bulk fluid can be forced by an applied pressure. Counter-ions in the diffuse layer surrounding the material then migrate with the fluid, creating an electrical potential or current, either of which can be measured. If the current is measured, it is termed the "streaming current." The ECA 2000P measures the streaming current without the need for a capillary passage or porous plug. Rather, the laboratory unit employs a piston and probe assembly, which is completely described in the following sections.

1.3 STREAMING CURRENT AND THE DOUBLE LAYER

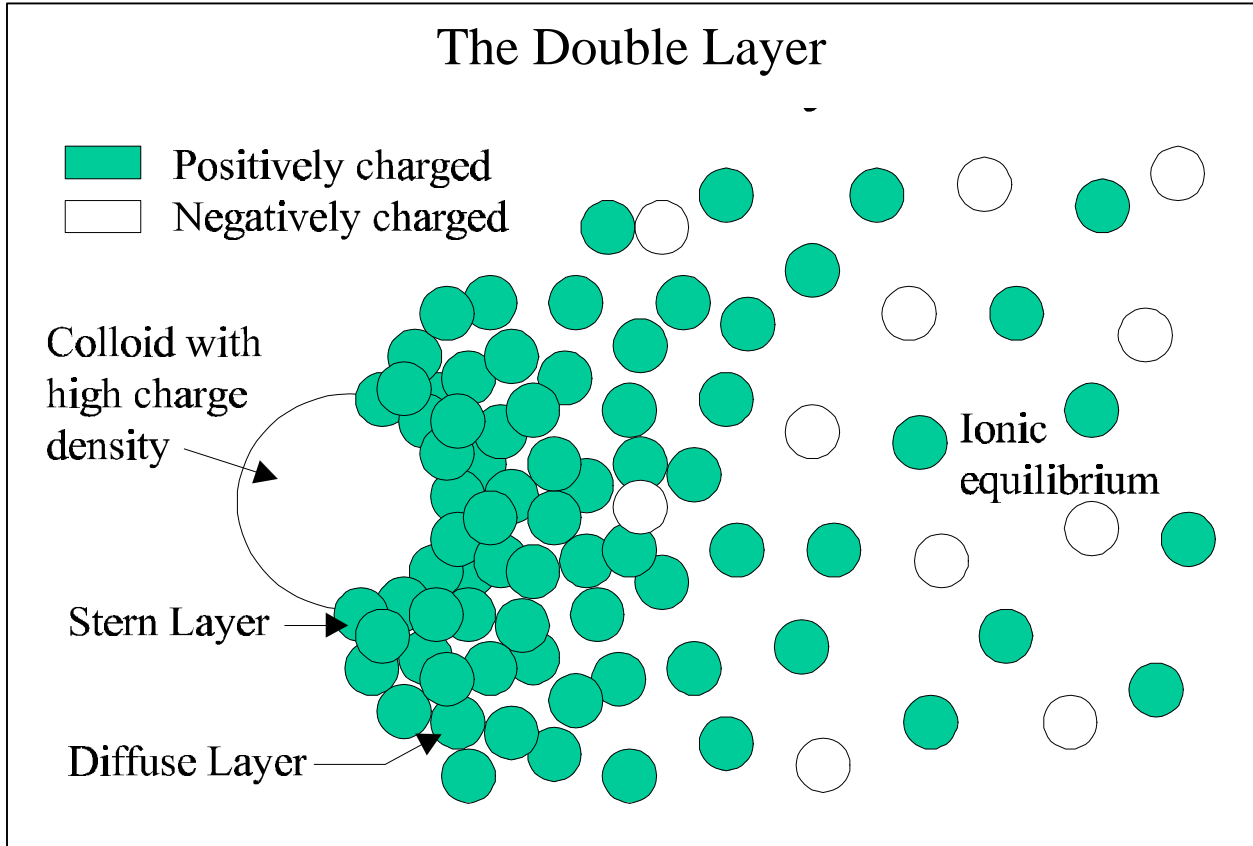
Surface chemistry and physics are important to papermaking chemistry, refining behavior, foaming and water treatment. One branch of surface chemistry is colloidal chemistry, which is the study of a stable combination of particles that are dissolved or suspended in a second substance. Two important aspects of colloidal chemistry are the particle sizes (on the order of microns) and the net charge of these particles. Because of their small size, surface area plays a major role in their behavior, along with the charge density associated with each particle.

Most materials involved in papermaking have charge surfaces. Namely, fibers, fines and fillers carry a net negative charge, as well as other papermaking additives. Some additives are cationic (positively charged) in nature and, therefore, are attracted to the anionic components. The cationic particles are tightly bound to the surface of the anionic colloid, thus forming the Stern layer. These cationic particles are also known as “counter-ions.” The electrostatic forces at this layer are strong enough to prevent displacement of the counter-ions by shear forces. Outside of the Stern layer is a grouping of counter-ions known as the Diffuse layer, hence the term “double layer.” This is depicted in Figure 1. The ions in the Diffuse layer are further apart and can be displaced when a shear force is applied. The ECA effects the shearing of counter-ions, and then measures the current generated by the movement of these cationic particles. This is how the streaming current value is measured by the ECA. The shearing of the counter-ions occurs between the Stern layer and Diffuse layer, known as the slip-plane.

As previously mentioned, the measurement of zeta potential is also based upon the double layer theory. The zeta potential is valid at the slip-plane. Essentially, the zeta potential is measured between the slip-plane and the bulk solution that is in ionic equilibrium. Whether streaming current or zeta potential is being measured, addition of an oppositely charged electrolyte will cause the slip-plane to contract towards the particle surface. In papermaking, the particle surface is negative, such as a fiber, and a cationic electrolyte would be added. As the slip-plane contracts, the streaming current (and zeta potential) will become less negative. Eventually, the isoelectric point will be reached, which is defined as a streaming current value (or zeta potential) equal to zero. At the isoelectric point, the solution is considered neutral (no net charge.) If additional

electrolyte is added, the system will become net cationic. Adding electrolyte to the sample is detailed in Section 4.5.

FIGURE 1



2.0 COMPONENTS

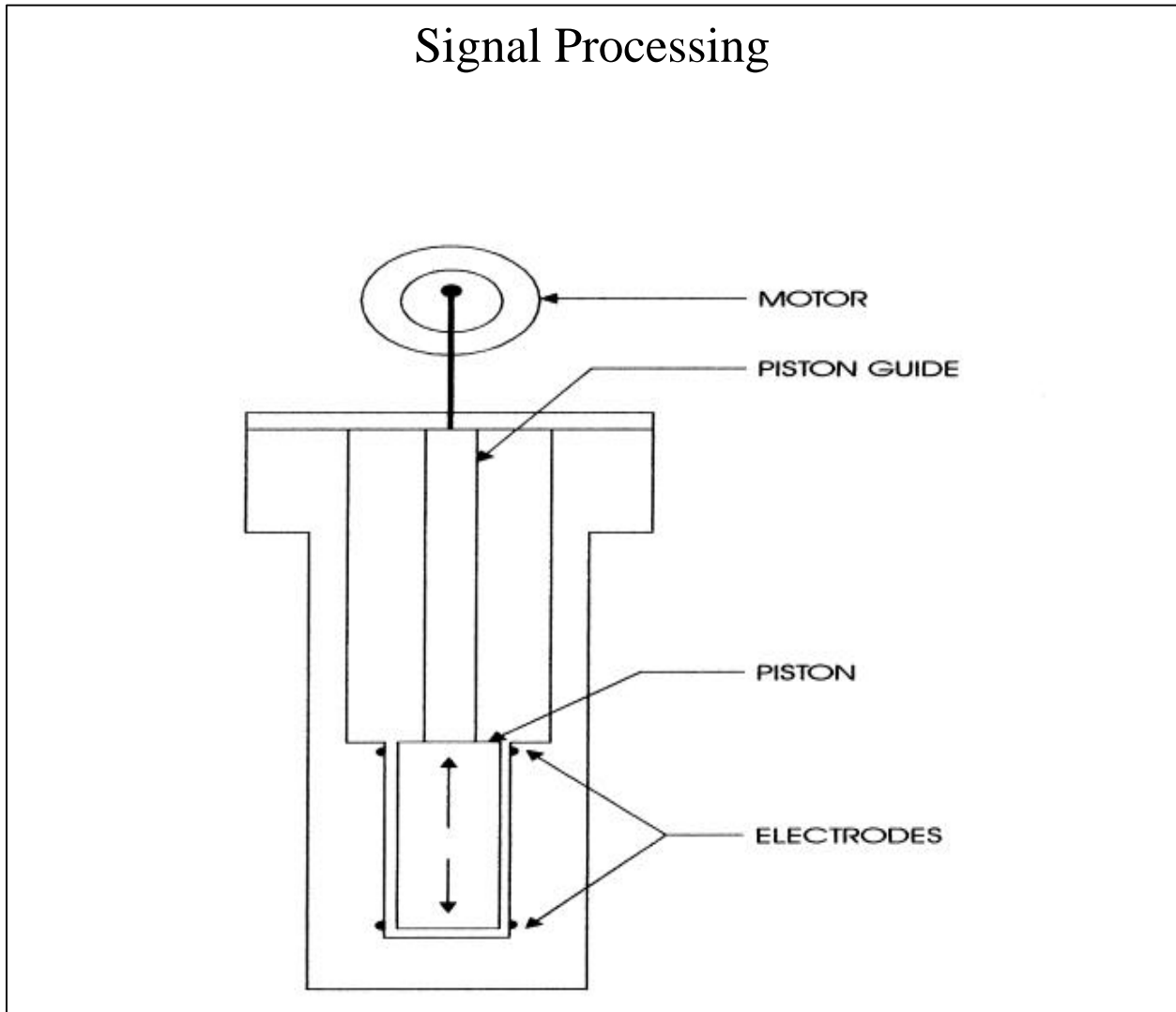
The ECA consists of a probe assembly and a signal processor. The probe assembly is comprised of the piston and sensor, which houses the electrodes that measure the streaming current. The signal processor is the circuit board inside the ECA.

2.1 PROBE ASSEMBLY

A simplified picture of the probe assembly is presented in Figure 2. The sample, which contains the colloids to be characterized, flows into the cylinder (via side holes that are not shown.) Inside the cylinder is a small piston that reciprocates vertically. Above the piston, the piston rod is attached to an eccentric point on a crank driven by a motor. The piston's velocity is thus sinusoidal in nature.

The piston moves up and down inside a cylinder that is closed at the bottom. The space between this piston and the cylinder is called the annulus, which is critical to the streaming current device. The annulus has the shape of a thin, cylindrical "shell," which contains sample fluid and particles. The clearance between the piston and cylinder walls is several thousandths of an inch (in metric units, on the order of hundreds of microns.) As the piston reciprocates, it acts as a pump: when moving downward, it forces sample out of the cylinder, upward through the annulus. Note that the annular clearance is large enough to allow most particles in a typical sample to pass in or out, but small enough to cause significant fluid velocity within this space. After the piston reaches its downward position, it proceeds upward, pulling fluid downward into the increasing volume below this piston. All directions are thus reversed in this stage of the piston's travel. The constantly reciprocating piston ensures continuous renewal of sample, such that changes in particle characteristics can be monitored.

FIGURE 2



It may then be assumed that the surfaces of both the piston and cylinder become adsorbed with particles from the sample. (There are other ways of viewing this phenomenon, but this assumption provides a workable explanation.) These particles, namely the anionic species, adhere to the piston and cylinder walls by Van der Waal's forces. As fluid motion is induced by the reciprocating piston, the counter-ions of the double layer are sheared away from the colloidal particle.

Figure 2 shows where the electrodes are positioned in the ECA's sensor. These are two metal rings inserted in upper and lower areas of the cylinder. If the piston is moving downward (and, therefore, the fluid motion is upwards), and the attached particles are negatively charged (and,

therefore, the outer charged layer is positive), then according to convention the electrical current is traveling from the lower to the upper electrode.

It should be clear that the STREAMING CURRENT VALUE is dependent on the fluid direction and velocity. (Of course, the fluid velocity is dependent upon the size of the annulus.) The resulting signal is an alternating current, which can be separated easily from constant background effects. The magnitude of the charge density determines the magnitude of the streaming current at any point, and thus determines the amplitude (peak height) of the alternating current signal.

2.2 SIGNAL PROCESSING

The processing of this alternating current is the other important aspect of an ECA. The current generated in the sensor is on the order of 10-12 microamps, which is extremely low, and a sensitive amplifier must be used. In order to measure the signal, the amplifier must be low impedance because, in its absence, the circuit would be completed by conductance back through the liquid. Essentially, the amplifier must have a lower conductance than the sample in order to complete the circuit and measure the current. Hence, conductivity of the sample is limited to approximately 7,000 μS .

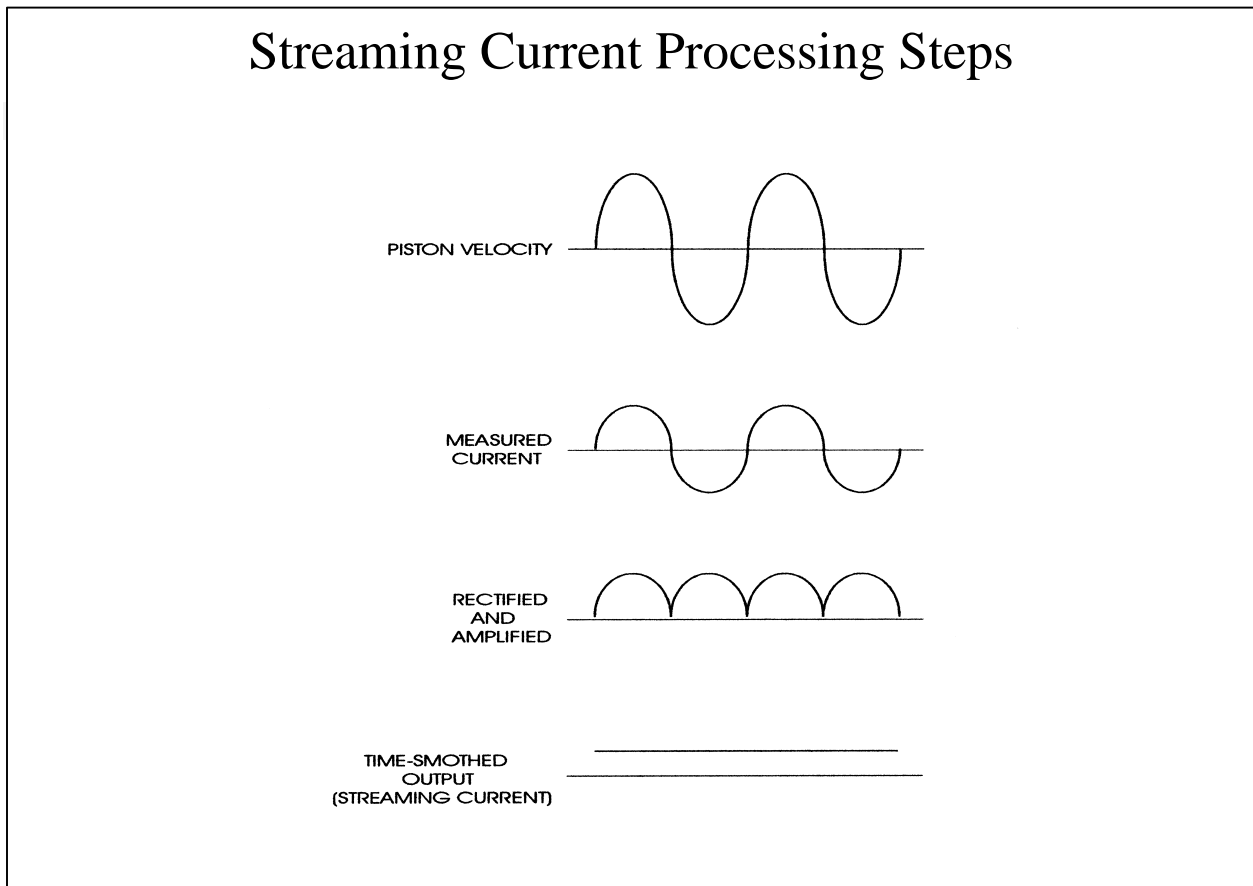
Figure 3 shows the stages involved in processing the streaming current signal. As indicated, the current generated at the sensor is directly proportional to the piston velocity, provided that the particle characteristics are not changing over this time frame. The circuitry must change this signal into a constant, readable value that is proportional to the amplitude (height) of the original sensor output. This is accomplished by rectifying the entire signal. The signal is simply changed to a positive value whenever it is negative. Finally, the signal is filtered, or “time-smoothed,” to give a continuous and consistent reading.

The value of this “streaming current” is in relative units. The instrument is not calibrated to be numerically equal to the actual current or charge density that exists in the sensor, primarily because the calibration would be sensitive to small differences in the physical dimension of the annulus. Another reason the instrument is not calibrated to display a “discrete charge value” is that the instrument is sensitive to differences in charge distributions that vary from simple systems

to complex systems. However, the unit includes a sensitivity adjustment (HI or LOW) that amplifies the original signal. Also, the ECA has a gain switch that allows the output to be varied by a factor of up to 20 times the primary signal. The instrument also provides a zero adjust which moves the reading up or down in value by a selected amount. Reasons that the zero adjust might be needed are discussed later.

Figure 3 also suggests how the processing of an alternating current signal from the sensor serves to eliminate any extraneous currents. Suppose the entire signal is moved upward a certain amount due to an interfering current which is constant in value. When the signal is rectified, the positive peaks remain at a higher level, but the negative peaks are at a lower level when they are “flipped” to the positive side. After this rectified signal is smoothed, the overall result is to eliminate the interference, since half of the peaks were too high and half were too low.

FIGURE 3



3.0 OPERATION

3.1 CONTROLS

There are three controls on the ECA 2000 P (Fig. 4):

- CONDUCTIVITY Switch
- GAIN Potentiometer
- ZERO Potentiometer

A brief description follows:

3.1.1 Conductivity Switch

The purpose of the CONDUCTIVITY Switch is to increase the sensitivity. Samples with conductivity greater than 1000 micromhos should be measured with the conductivity switch on high. The low range should work well for all other samples.

3.1.2 Gain Potentiometer

The GAIN pot may be adjusted to make the instrument more or less sensitive. The pot has a working range of ten turns. Clockwise rotation increases the GAIN and counterclockwise rotation decreases the GAIN. Start at the lowest setting and increase GAIN as needed. Figure 5 shows the relationship between the 10-turn potentiometer and the amplification factor. The GAIN pot allows the user to increase the resolution for small changes in the charge measurement or to amplify the *differences* in charge between two or more samples.

3.1.3 Zero Potentiometer

The ZERO pot allows the user to offset the display to obtain a desired starting point. The ZERO is only active when the toggle switch is flipped to the IN position and the yellow light is on. If the GAIN is changed the unit must be Re-ZERO-ed for that

GAIN setting. The ZERO may be used to offset a value that is off scale. The range for the display is -10 to +10.

FIGURE 4- RELATIONSHIP BETWEEN 10-TURN POTENTIOMETER & AMPLIFICATION FACTOR

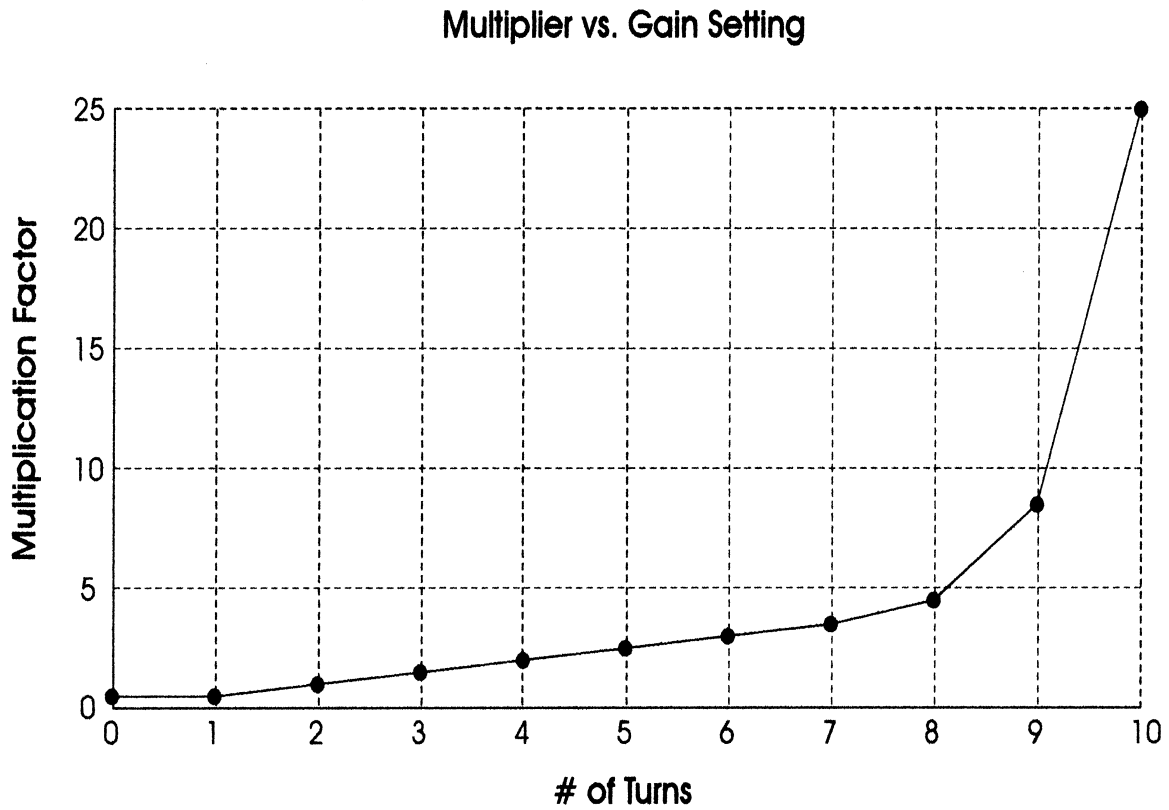


FIGURE 5 – ECAP 2000

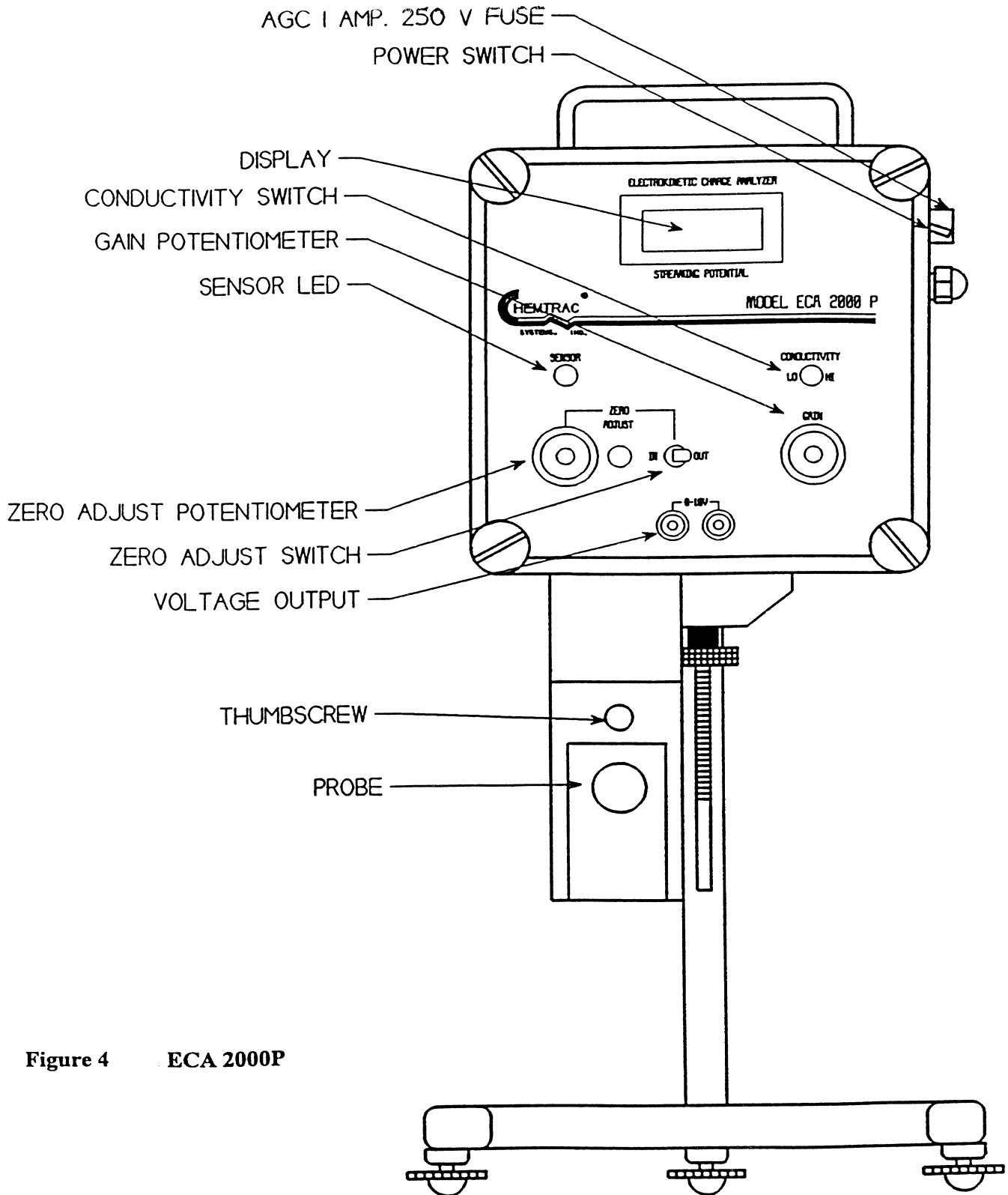


Figure 4 ECA 2000P

3.2 SENSOR AND PISTON

The sensor and piston are critical components of the ECA 2000P that require care and regular cleaning. For operator convenience, a spare set is provided with each unit.

3.2.1 Removal

1. Turn the ECA “Off.”
2. Loosen the thumbscrew on the lower portion of the sensor.
3. Secure the ECA with one hand and with the other, pull down on the lower portion of the sensor to disconnect at the banana plugs.
4. To remove the piston, unscrew by hand or, if necessary, with a flat blade screwdriver. **NOTE:** Hand-tight is sufficient to secure the piston.

3.2.2 Insertion

1. Turn the ECA “Off.”
2. Screw the piston on by hand, taking care not to damage the threads.
NOTE: Hand-tight is sufficient to secure the piston.
3. Position the sensor so that the thumbscrew is facing forward.
4. Secure the ECA with one hand, and then insert the sensor by aligning the banana plugs. The sensor will rest “flush” against the main body of the ECA so that the banana plugs are no longer visible. This will ensure a good electrical connection and prevent fouling of the connections.
5. Tighten the thumbscrew to secure sensor.

3.2.3 Standard Cleaning Procedure

With some particles, cleaning is easily accomplished by rinsing with tap water. Others (especially cationic particles) are tenacious and must be removed with fairly vigorous cleaning. The following cleaning procedure will assure that the ECA is providing accurate, reproducible measurements.

1. Remove the sensor and piston according to the procedure outlined above. Remove the plug and O-ring from the bottom of the sensor.
2. Scrub the surface of the piston and cylinder, (both inside and outside) with a stiff nylon test tube brush and a mild abrasive cleanser (e.g. Comet). Use room temperature tap water.

NOTE: Avoid water contact with the banana plugs.

3. Rinse all of the surfaces thoroughly with tap water, ensuring that they are free of soap film and any residue. Carefully inspect the inner surface of the cylinder to ensure that no visible particulate matter is left on the surfaces.
4. Completely dry the sensor, piston and plug with compressed instrument air or by allowing the components to air-dry in the laboratory.
5. Return the plug and O-ring to the base of the sensor. Omitting the O-ring will cause variations in the SCV.

Note: Distilled or de-ionized water may be used. However, the water's low conductivity will magnify the SCV. Thus, the operator must ensure that all surfaces are completely dry before use.

4.0 PRACTICAL CHARGE MEASUREMENT

This section provides the operator of the ECA 2000P with practical guidelines for obtaining reliable data. It is the responsibility of the operator to determine the appropriate application for charge measurement, consistently follow the procedures described in this manual, and then determine the meaning of the data.

4.1 REPEATABILITY

Repeatability is the key to success of any scientific analysis. The ECA uses the principles of surface and colloidal chemistry. Namely, Van der Waal's forces and the double layer principle are critical to the ECA's accuracy and repeatability. Thus, it is crucial that the ECA's sensor and piston are clean prior to each measurement. If residual molecules (with or without charge) contaminate the probe assembly, the STREAMING CURRENT VALUE will not be reliable. Any residual molecules, including distilled water molecules, can significantly affect the SCV. Therefore, follow the "STANDARD CLEANING PROCEDURE" outlined in Section 3.2.3 before each charge measurement is performed.

4.2 SAMPLE PREPARATION

After obtaining the desired pulp sample, one must consider the consistency of the slurry before proceeding with charge analysis. If the sample is less than 0.1% consistency, as is whitewater, then no preparation is necessary.

However, if the sample is at headbox consistency or greater, action must be taken to prevent fibers from accumulating in the annulus. There are two available methods (described in Subsections 4.2.1 and 4.2.2) that are left to the discretion of the operator. Results will vary between the two methods, so be consistent or note which method is used for each sample.

Regardless of the method used, the volume collected is important. For each charge measurement, a volume of 350 ml is ideal. Half will be used for CONDITIONING, as explained in Section 4.3. The remaining 175 ml will be used for the actual charge measurement. This is the recommended volume, especially when a titration is performed to determine charge demand (see Section 4.5.) However, if the sample volume is limited, the operator may employ the sampling cups that are provided with each unit. These small cups mount into either side of the sensor, allowing analysis of a 20 ml sample.

4.2.1 The Filtration Method

This is the recommended method for obtaining a sample. Simply pour the pulp slurry through an 80 or 100 mesh screen to remove the fibers. The filtrate will contain small (relative to the fibers) charged particles, as well as soluble charge. At the same time, the sample will have a suitable consistency for the ECA's probe assembly. Although charged fibers are removed from the sample, their contributing surface area is significantly less than the surface area of the fines, filler, and other small species in the system, not to mention the soluble charge. Thus, the charge density of these smaller components and, consequently, the "charge contribution", is significantly less than that of the removed fibers.

If the operator chooses to target soluble charge in the system, filter paper may be used to remove a majority of the solids in the pulp slurry. The type of filter paper used is left to the discretion of the operator.

Note: Consistent methodology is critical to obtaining reliable data. DO NOT change the filtering media without taking note.

4.2.2 The Dilution Method

A less commonly practiced method is to dilute pulp samples with distilled or de-ionized water to a consistency less than 0.1%. Recall that the SCV is affected by the low conductivity of distilled or de-ionized water. This may or may not be desirable.

However, if the fiber content of the sample is critical to the charge analysis, the operator may consider employing the dilution method.

4.3 CONDITIONING

Chemtrac recommends that the sensor and piston be conditioned before the actual charge measurement is performed. Conditioning will replace foreign molecules with ones that are similar to the sample being tested. Additionally, one may consider that conditioning will “coat” the surfaces of the sensor and piston with the sample to be tested.

Pour 175 ml of the conditioning sample into a 250 ml beaker. With the magnetic stirrer, position the beaker so that the fluid level rises to the center of the side holes on the sensor. (Avoid a fluid level that is above the set screw on the front of the sensor.) With proper mixing of the sample, allow the SCV to stabilize. This will take 30 to 180 seconds.

4.4 CHARGE MEASUREMENT

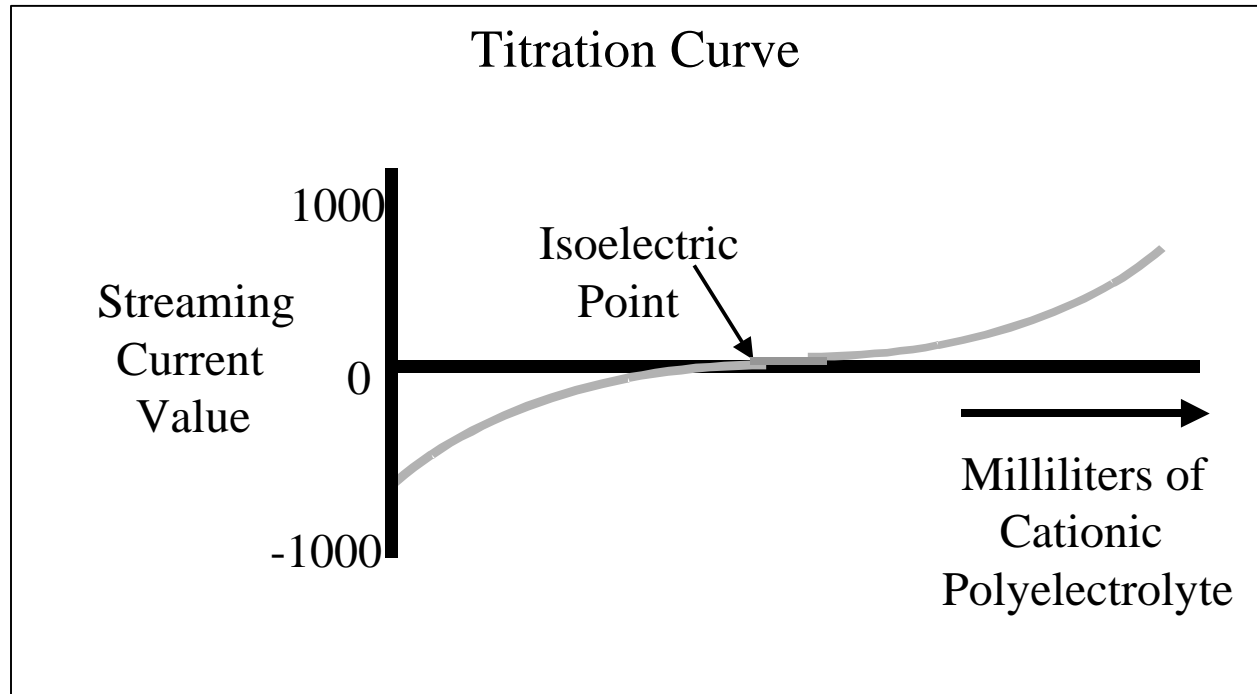
Remove the conditioning sample, and replace with the true sample. Follow the same steps as stated in the previous paragraph. After the SCV has stabilized, record this value, as well as the SENSITIVITY, GAIN SETTING and ZERO OFFSET. Do not discard this sample if a CHARGE TITRATION is to be performed (see Section 4.5.) Otherwise, discard the sample and clean the probe assembly according to the STANDARD CLEANING PROCEDURE in Section 3.2.3.

4.5 CHARGE DEMAND (TITRATION)

To determine the charge demand of a sample, a polyelectrolyte of opposite charge is added until the isoelectric point is achieved. A polyelectrolyte is a standardized polymer with known charge density, also referred to as “titrant.” As previously explained, the slipplane contracts towards the colloidal surfaces until there are no more counter-ions that are strongly bonded to each colloid. Thus, the Stern Layer is nonexistent and the STREAMING CURRENT VALUE goes to zero. The CHARGE DEMAND refers to the amount of titrant, in milliliters, that is required to bring a

sample to the isoelectric point. This is demonstrated in Figure 4, where a net anionic solution is dosed with cationic polyelectrolyte until the isoelectric point is achieved and then surpassed.

FIGURE 4



Note that in Figure 4, the SCV tangentially approaches zero. This is critical to achieving a valid titration, and is achieved by an appropriate addition rate of polyelectrolyte. (Of course, proper mixing of the sample during titrant addition is also crucial.) A slow addition rate ensures that each molecule is given the opportunity to adsorb onto a colloid and stabilize itself to achieve its lowest energy level. A hasty titration will cause the SCV to overshoot the isoelectric point, giving a false CHARGE DEMAND. This “overshoot” is depicted in Figure 4 but is not the normal procedure for a titration.

Since charge demand quantifies the number of charges in a volume of furnish, its value may be expressed in mL of titrant consumed by the sample to reach the isoelectric point. The polyelectrolyte’s charge density is known as “titrant normality” with units of equivalents/L. With this information, the CHARGE DEMAND (volume of titrant consumed) may be converted from mL to milliequivalents per liter $\left(\frac{\text{meq}}{\text{L}}\right)$:

$$\text{CHARGE DEMAND} \left(\frac{\text{meq}}{\text{L}} \right) = \frac{\text{Titrant Consumed (mL)} * \text{Titrant Normality} \left(\frac{\text{eq}}{\text{L}} \right) * 1000 \left(\frac{\text{meq}}{\text{eq}} \right)}{\text{Sample Volume (mL)}}$$

Converting the CHARGE DEMAND from ml to meq/L is especially useful if titrants with different normalities are used, or if different sample volumes are required.

The operator has a choice as to which polyelectrolyte, or titrant, to use. Chemtrac highly recommends NALCO™ products, namely:

Part Number	Description	Price	Notes
S5430-75	Dadmac, .001N, 1 liter	\$100.00	Cationic
S5430-77	Dadmac, .001N, 4 liters	\$250.00	Cationic
S5433-75	Dadmac, .005N, 1 liter	\$100.00	Cationic
S5432-75	PVSK, .001N, 1 liter	\$100.00	Anionic
S5432-77	PVSK, .001N, 4 liters	\$250.00	Anionic

These polymer can be ordered from Chemtrac Systems, or directly from Nalco at lower prices. The Nalco Order Desk in the United States can be reached at 708-496-7000.

Alternatively, the operator may choose to use a process additive. In doing so, a specific application may be analyzed or optimized. The limitation is that the data will not be universally applicable.

4.6 APPLICATIONS

Effective utilization of the ECA 2000P is the responsibility of the operator. Ingenuity and creativity are required. Charge analysis can be utilized on the following systems:

1. Wet-end approach system:
 - a. Pulp from the pulp mill, DIP or hydropulpers,
 - b. At the headbox, wire pit, machine chest, or blend chest,
 - c. On the broke system,

- d. Before and after chemical addition anywhere in the approach system, or
- e. Process water.
2. Brown stock washer,
3. Deinking plant,
4. Sludge dewatering, or
5. Any other colloidal system where proper charge balance would improve product quality, increase production, or optimize chemical addition.

4.6.1 Charge Measurement

Charge measurement indicates relative changes from an initial starting point. If the system experiences a charge-related change, the SCV will change. If the sample goes more cationic, the SCV will become more positive. Practical examples include an increase in cationic starch addition or a decrease in anionic trash. If the sample goes more anionic, the SCV will become more negative. Practical examples include a decrease in cationic starch addition, an increase in anionic trash, or an increase in filler addition. The instrument will not indicate what caused the charge to change. The instrument will indicate the direction and relative magnitude of the change. Check the obvious reasons for a change in charge first, such as a chemical pump that has been turned off or improperly adjusted, or a fluctuation in recycled material or broke entering the system.

As the operator studies the system over time, criteria can be established for a specific machine and optimum operating characteristics. This information can be correlated to a range of values that indicate when the sample has the correct charge balance. Because the ECA gives results quickly, it can be used to identify potential machine upsets quicker than traditional methods.

4.6.2 Charge Demand

CHARGE DEMAND is typically used to determine an appropriate chemical dosage at any given time, although other applications exist. The SCV and the CHARGE DEMAND do not necessarily correlate. For example, two different samples with the

same SCV may have significantly different CHARGE DEMAND values. By the same token, samples with different SCV's may have the same CHARGE DEMAND.

Initially, this approach allows experimentation on a bench scale without risking machine upsets. As more information and confidence are gained, the operator can begin to make chemical feed adjustments based upon the CHARGE DEMAND at any given time. Significant chemical reduction has been reported by this simple implementation of charge analysis. With more experience, chemical reduction may give way to increased productivity or product quality since first pass retention, drainage, and formation are all affected by the SCV and CHARGE DEMAND of the wet-end approach system.

5.0 LABORATORY STAND

5.1 UNPACKING

A portable laboratory stand is standard equipment with the ECA 2000P (Fig. 6) and includes:

1 Base	1 Jam Nut
3 Leveling Screws	1 Clamp Assembly
1 Upright rod	

Remove the three (3) leveling screws from the base and discard the packing material. Remove the jam nut from the upright rod.

5.2 ASSEMBLY

Screw the leveling screws into the base. Insert the threaded end of the upright rod into the hole in the top of the base and attach the jam nut to the rod on the underside of the base. With the rod gear rack facing forward (toward the “V” in the base), gently tighten the jam nut. When using the rod extension, screw the threaded end of the upright rod into the extension, then insert the threaded end of the rod extension into the base.

5.3 ECA MOUNTING

1. Slide the mounting bar (Fig. 6, Item 10) into the ECA mounting block (Fig. 6, Item 11). Make sure the ridge on the mounting bar is pointing downward. Lock the mounting bar in place with the 10-32 thumbscrew (Fig. 6, Item 13).
2. Slide the mounting bar into the stand clamp (Fig. 6, Item 5) and tighten clamp screw assembly.
3. Adjust the tightness of the gear screw (Fig. 6, Item 7) with set screw (Fig. 6, Item 6). If the ECA slides down because of its own weight, the set screw should be tightened.

FIGURE 6 – DIMENSIONAL DRAWING OF ECA LABORATORY STAND

