

5900 Schooner Street – Belleville, MI 48111 – USA TEL (800) 422-2558 – FAX (734) 665-9099 www.**Eberbachlabtools.com**

E1000.00 Electro Analysis Apparatus 115/230 Vac, 50/60 Hz

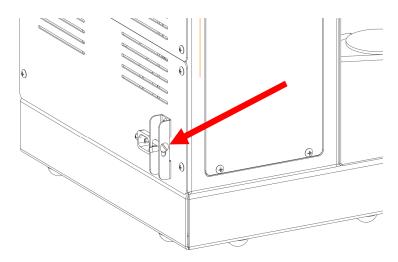
USE AND CARE FOR CATALOG NUMBER:

- E1000.00 Electro Analysis Apparatus
- 115 /230Vac, 50/60 Hz

SETTING UP APPARATUS

LOCATION: Establish location for apparatus on a table or wall. For wall mounting remove the rear panel and use the two holes provided in each side panel. Bolts to be employed depend on type of wall construction.

POWER SOURCE: Source of electrical power may be 115 volt or 230 volt, 50/60 HZ capable of delivering 550 watts. Apparatus is factory set for 115 volt, 50/60 HZ. *For* 230V, remove switch post, flip the switch located on the rear of the unit.



<u>POWER SWITCH</u>: To power apparatus, place Power Switch in "on" position. With Power Switch on, the indicator will glow. If indicator fails to glow, check: (a) Source of power, (b) A.C. 5 ampere fuse on panel, and (C) Indicator bulb.

MOUNTING STIRRER ROD: Place a stirrer rod into "hollow spindle" where it is held by concealed spring clips. Its height can be adjusted by slipping it up or down.

MOUNTING ELECTRODES: After selecting electrodes, mount them in electrode holders by depressing end of holder and placing the stem of the electrode in jaws. The electrode holder may be moved laterally to position electrodes in relation to stirrer rod. Care should be taken to position Electrodes concentric to each other to insure uniformity of electrodeposition. If a rotating electrode is used, Rotating Anode Holder No. 1050 is required; it mounts in place of the stirring rod. When the rotating anode is employed, use the right electrode holder for the stationary electrode. The polarity of the rotating electrode is the same as that of the left electrode holder. This is selected by the polarity switch. When removing stationary electrodes, it is only necessary to depress the ends of the holders, and the electrodes will drop into receptacle chosen to transport them.

ADJUSTMENT OF BEAKER SUPPORT: The beaker supports are adjustable up and down, and they will swing either to right or left. They are provided with stops for positioning. To adjust height of beaker support, loosen the thumbscrew in the collar just below control panel and slide shelf to desired height. It may be necessary to use a coin or a screw driver. Shelf may also be adjusted to swing right or left. To adjust it to swing right hold shelf in working position. With the above-mentioned thumbscrew loose, rotate the collar counterclockwise until it stops and tighten thumbscrew. To adjust shelf to swing left, hold the shelf in working position. With the thumbscrew loose, rotate the collar clockwise until it stops and tighten the thumb screw. The swinging beaker shelf facilitates easy placement and removal of beakers without disturbing electrodes or stirrer.

STIRRERS: Switch marked "Stirrer Switch" on instrument Panel controls stirrer motor. Stirrers rotate at a constant speed of 620 rpm., when operating from 60 HZ source.

CURRENT CONTROL: With the apparatus set up and ready for use, the direct current to each set of electrodes is adjusted by a potentiometer controlling a Darlington power transistor from 0 to 10 amperes. Rotate the Potentiometer Clockwise to increase current and Counter-Clockwise to decrease current. A polarity reversing switch changes polarity of the electrodes. The switch is wired so that the switch position and the direction of deflection of the voltmeters indicates the positive electrode. Each spindle is provided with ammeter reading 0 to 10, voltmeter reading 10-0-10, polarity reversing switch, and potentiometer for current control. All of these controls are located on the panel directly above their respective spindles. To turn off direct current to either spindle, use the center position of the polarity reversing switch. Allow sufficient time for the temperature of the system to stabilize, and then adjust for desired current. Within the control capabilities of the circuit, the current through the cell is dependent on the potentiometer adjustment. The control circuit in the Analyzer maintains a constant current through the cell. The voltage across the electrodes required to attain a particular current is a function of the following:

- 1. Resistance of the Electrolyte
- 2. Surface area of the electrodes
- 3. Geometry of the spacing of the electrodes.

CARE OF THE APPARATUS

<u>FUSES</u>: A 20 ampere fuse protects the D.C. circuit, and a 5 ampere fuse guards the A.C. circuit. To replace either fuse, remove fuse holder with a coin or screw driver and replace cartridge with fuse of similar size and identical capacity.

<u>LUBRICATION</u>: The motor bearings are grease packed ball bearings and require no lubrication. The stirrer spindle bearings are of the oil-less bronze type and under normal usage do not require any lubrication.

<u>CLEANING AND LONG LIFE</u>: The apparatus is constructed chiefly of enameled sheet steel and stainless steel. Provision has been made to keep fumes from the inside. If the stainless steel panel and front pieces are kept clean by wiping with damp cloth regularly when they become splashed or subject to acid fumes, they will retain their high finish many years. Once stained, however, they are difficult to clean.

<u>BELT TENSION</u>: The position of the motor should be adjusted so the belt tension is very loose. Proper belt tension can be checked by squeezing the belt between the motor pulley and the two spindle pulleys to a position where the slack is removed, (belt is in straight line between pulleys) and at this point, the belt should be free of the motor pulley groove.

INTRODUCTION TO METHODS

The following information is a product of research carried on at Battelle Memorial institute, Columbus, Ohio under the sponsorship of Eberbach Corporation. It is intended to serve as an introduction to the use of electrolytic methods in quantitative analysis.

Electroanalysis, as used in this text, is a process of plating an element on an electrode which is weighed before and after deposition. Elements are plated on the cathode as metals or, in some cases, on the anode as hydrated oxides. Most electrolyses are made in dilute mineral acid solutions. Some elements require special electrolytes such as ammonium hydroxide, cyanides, and organic acids.

Although a number of elements are deposited under ordinary conditions, it is possible to effect specific determinations in many systems with a minimum of chemical preparation. Metals may be re-plated to effect additional purification. When the ratio of electrode weight to deposited-metal weight is unfavorable, the metal is dissolved from the electrode and determined by chemical or other procedures. In some instances, electroanalysis is used to advantage in the removal of interfering ions.

Copper and lead as metals or in alloys such as brasses, bronzes, etc., are particularly well suited to electroanalytical techniques. This discussion will be confined to these two elements. However, numerous references to the literature are included, covering other elements and special electroanalysis procedures.

GENERAL CONDITIONS FOR PLATING, COPPER AND LEAD

Electroanalysis provides a convenient method for the simultaneous or individual determination of copper and lead. Copper may be electrolytically separated from lead, cadmium, zinc, and other metals having an electrode potential near, or more positive than, copper(Table 1). In some cases, it is necessary to separate copper and/or lead from interfering ions. For instance, copper may be precipitated as the sulfide or thiocyanate⁽²⁾ and then be determined electrolytically after appropriate chemical treatment.

TABLE 1. ELECTROMOTIVE SERIES

Element	E _o , Volts
Li	-3.02
K	-2.92
Ва	-2.90
Sr	-2.89
Ca	-2.87
Na	-2.71
Mg	-2.34
Al	-1.67
Mn	-1.05
Zn	-0.76
Cr	-0.71
Fe	-0.44
Cd	-0.40
Co	-0.28
Ni	-0.25
Sn	-0.14
Pb	-0.13
Н	0.00
Sb	0.15
As	0.24
Bi	0.32
Cu	0.34
Ag	0.80
Hg	0.80
Au	1.42

In the electroanalysis of copper, the ideal electrolyte would consist of 3 per cent H₂SO₄ and 2 per cent (or less) of HNO₃. The nitric acid serves as a depolarizing agent to prevent the discharge of hydrogen at the cathode, and also assists in producing a more dense and coherent copper plate. The concentration of nitric acid should be kept low because of its tendency to dissolve the plated copper or retard its electrodeposition⁽²³⁾.

For the highest efficiency, the volume of electrolyte should be just sufficient to cover the electrodes. Many analysts insist that when plating copper, the temperature of the electrolyte should not be permitted to rise above 40°C. When more than one ampere of current is supplied to the cell, the electrolyte should be stirred to prevent the polarization due to ion starvation at the electrode surface.

When plating lead alone as the dioxide on the anode, the nitric acid concentration of the electrolyte should be high. Fifteen to thirty per cent (v/v) yields the best results⁽¹⁰⁾. The addition of a few drops (0.25 ml.) of H_2SO_4 produces a more dense and adherent "lead dioxide plate"^(10, 18). The lead solution should be heated nearly to boiling at the start of the electrolysis. It may be allowed to cool during the plating process; in fact, better recovery is attained when the last few milligrams plate from a cool solution.

Large amounts of copper, ten grams or more, can readily be plated in electroanalysis. However, a sample containing more than one gram is seldom justified. Samples to be analyzed for lead should contain 0.05 to 0.150 gram of the metal. Larger amounts of lead may produce a plate that is loosely adherent to the anode, so that washing the plate may result in mechanical loss of the lead dioxide.

Stirring the solution during electrolysis markedly increases the rate of deposition^(9,23). Stirring is especially important in the deposition of lead because only the ions in contact with the anode are oxidized to the tetravalent state. The tetravalent ion then reacts with water to produce the dioxide (PbO₂⁻⁻), which is held to the anode by electrical attraction (anaphoresis)^(3,4,9).

INTERFERENCES IN THE DETERMINATION OF COPPER

THE NOBLE METALS

Gold, mercury, and silver will deposit with copper unless they are removed. Silver may be removed as the chloride. It is important then to remove the residual chloride by fuming the sample with sulfuric acid.

SELENIUM, TELLURIUM, ARSENIC, AND LEAD

Selenium and tellurium will interfere by plating with the copper unless they are oxidized and kept in the hexavalent state during electrolysis. An ammoniacal solution of the sample is boiled with a gram of potassium persulfate which will oxidize both selenium and tellurium. The solution can then be made acid and electrolyzed as usual. The addition of another portion of persulfate after start of the electrolysis may be necessary to keep the tellurium oxidized. When lead is not being determined at the same time as the copper, a strong oxidizing condition to prevent interference from tellurium can be maintained the addition of ten milligrams of manganese nitrate. The electrically generated permanganate will effectively prevent the interference also of arsenic and antimony⁽⁸⁾. Note that because selenium has a lower decomposition potential than copper, it must first be oxidized with persulfate as above and the manganese nitrate added before the electrolyzing is begun.

TIN, TUNGSTEN, BISMUTH, AND MOLYBDENUM

Tin may be removed from a copper sample by precipitating it as the metastannic acid in nitric acid. If the sample is dissolved in fuming nitric acid, the metastannic acid formed will be granular, easily filtered and washed, and will occlude little, if any, copper⁽²⁶⁾. Tungsten as the trioxide will also separate from solution with this treatment.

Bismuth will deposit along with the copper. It may then be dissolved from the cathode with the copper and determined separately. Molybdenum also plates with copper to produce a dark-colored plate. When a very small quantity (6 mg.) is present, the addition of a milligram of sodium chloride will prevent its deposition (26, 19)

IRON, CHROMIUM AND MANGANESE

Iron, chromium, or manganese may interfere in the copper determination because copper is appreciably soluble in solutions of salts of metals having two states of oxidation. More than 0.05 gram of iron in the sample⁽²⁶⁾ will result in incomplete recovery of the copper. The separation of copper from iron may be accomplished by plating the copper from an electrolyte containing phosphate or fluoride. The latter materials sequester the iron so that it will not affect the copper deposit. This is an especially valuable technique when the copper content of steel is to be determined^(17, 22). Iron is reduced to the ferrous state at the cathode and then can convert nitric to nitrous acid. If this occurs, the copper already deposited is rapidly redissolved because of the ease with which it is attacked by nitrous acid. Manganese and chromium in amounts less than 0.05 gram do not interfere with the quantitative removal of the copper⁽²⁶⁾. When more than 0.05 gram of these metals is present, two ammonia separations are required to separate them from the copper.

THE HALOGENS

Chlorides, bromides, and iodides must be removed by fuming the sample with sulfuric acid. Even milligram amounts of the halides may cause the copper plate to be spongy or loosely crystalline. Although a drop of O.1 \underline{N} HCl is often added to a nitrate electrolyte⁽²⁾, it is undesirable and unnecessary in the mixed nitrate-sulfate solution (26).

NITROUS ACID

It is imperative that all nitrogen oxides be boiled from the electrolyte before attempting to plate copper. Copper will not deposit completely from a solution containing nitrous acid and any deposited copper may redissolve at once. Urea or sulfamic acid may be added to combine with any nitrous acid that may be produced during electrolysis. Sulfamic acid is the preferred reagent. The addition of urea may produce erroneous results because of the possible occlusion of organic matter in the copper plate⁽²⁶⁾.*

INTERFERENCES IN THE DETERMITION OF LEAD

Silver, bismuth, manganese, antimony, arsenic, mercury, tin, chromate, chlorine, and phosphate all interfere with the deposition of lead on the anode as dioxide^(IO). If significant amounts of these are present, they must be removed by appropriate chemical treatment before lead can be plated as the dioxide. Silver, bismuth, and manganese will co-deposit as oxides on the anode with lead. Chromate, arsenic, antimony, tin, phosphate, and chlorine either hinder the deposition of the lead dioxide or contaminate the deposit. Mercury will cause part of the lead to co-deposit on the cathode as a lead amalgam⁽¹⁸⁾.

*Any sample containing organic matter must be treated by controlled ignition at low temperatures, or by a "wet ashing process" to destroy the organic materials.

ANALYSIS FOR BRASS** FOR COPPER AND LEAD

Many electrolytic procedures are described in the literature for the analysis of brass, bronze, and other copper-containing alloys. The following method is intended to illustrate the techniques used for the assay of copper and lead in a typical brass.

Wash the sample in ether or benzene to remove any traces of oil or grease. Place 1-gram sample in a 150-ml. beaker with a watch-glass cover. Add 15 ml. of nitric acid*** slowly to prevent too violent a reaction. Permit the solution to evaporate at a temperature somewhat below the boiling point until the solution volume is about 5 ml. (Never evaporate to dryness.) Slow evaporation (about 1 hour) will insure precipitation of the tin as metastannic acid. Dilute the solution to 50 ml. and heat for half an hour to ensure solution of all soluble salts. Add some paper pulp to assist in collecting the metastannic acid, and filter (No. 42 Whatman or equivalent) the solution into a 250-ml. beaker. Wash with 1 per cent nitric acid. Reserve filtrate.

To recover copper and lead that may have been occluded by the metastannic acid, use either of the two following procedures:⁽²⁴⁾

A. Transfer the filter paper and contents to the original beaker and add 15 ml. of H₂SO₄ and 25 ml. of HNO₃. Heat until all organic matter is decomposed (H₂SO₄ fumes). Transfer solution to a 400-ml. beaker and dilute to 250 ml. Make the solution alkaline with 25 per cent NaOH to dissolve the metastannic acid. Add 20 ml. of 25 per cent Na₂S and digest the solution on the steam bath until the supernatant liquid is clear. Cool the solution to room temperature and filter through a fine textured paper, washing with 2 per cent Na₂S. Dissolve the residue in a few milliliters of 1:1 HNO₃ and add it to the filtrate reserved above.

**Per cent composition: Cu 55.0 to 90.0, Sn 0.0 to 5.0, Pb 0.0 to 5.0, P 0.0 to 0.1, As 0.0 to 0.1, Sb 0.0 to 0.1, Fe under 0.25, Ni under 0.25, Zn remainder.

***Solution of the sample in fuming nitric acid and subsequent boiling after diluting to 50 ml. is said to yield a metastannic acid that is crystalline, easily filtered, and will hold up little, if any copper or lead⁽²⁶⁾.

B. Place filter paper containing the metastannic acid in the original beaker and add 20 ml. of HNO₃ and 15 ml. of HClO₄. Heat to copious white fumes to destroy organic matter.* Cool beaker and wash cover glass and sides of beaker, then add 15 to 25 ml. of HBr. Heat to copious fumes to volatilize tin, arsenic, and antimony. If the solution is not clear, repeat the treatment with HBr. Evaporate the solution nearly to dryness, dissolve the residue in a few ml. of 1:1 HNO₃, and add it to the original filtrate.

Heat the combined filtrate containing the copper and lead almost to boiling and place it on the electroanalyzer. Add sufficient water to cover the electrodes. Add five drops of sulfuric acid and electrolyze with a current of 1.0 to 2.0 amperes for half an hour. Increase current to 4.0 amperes, and add six ml. of H₂SO₄ to the electrolyte.

Ten minutes after the solution has lost its blue color, some water is added to raise the level of the solution on the stem of the cathode. If no copper deposits on the freshly exposed surface during the next ten minutes, the copper may be assumed to have been quantitatively plated from the solution.

With the current on, the beaker containing the electrolyte is lowered rapidly away from the electrodes and replaced with a beaker of water. After the electrodes are rinsed thoroughly with water, they are rinsed with acetone and dried at 100° C. for 20 to 30 minutes and then weighed.

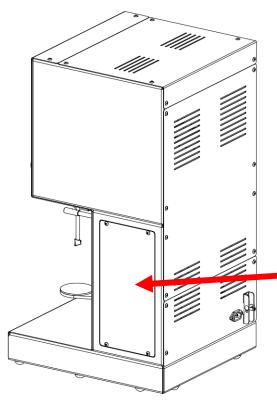
The conversion factor commonly used for the lead as dioxide is 0.8640⁽¹⁸⁾, instead of the theoretical 0.8660. The deposit nearly always contains a slight excess of oxygen or combined water.

*It may be necessary to add additional HNO₃.

Transistor Replacement

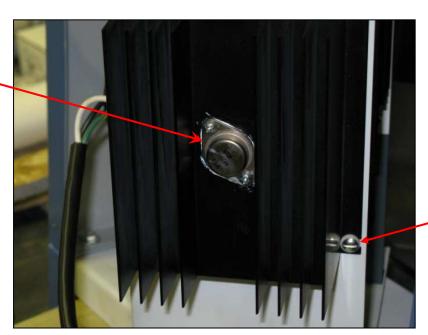
Remove the side panel from the unit.

Remove the 4 panel screws from the side of the machine.



Remove four heat sink screws.

Transistor



Heat Sink Screws

Remove the two transistor screws and pull the transformer out of the socket.

Examine the new transistor and make sure the legs are straight and not damaged on the transistor when replacing it.

Put silicone heat sink compound on the transistor with finger. Apply a smooth even coat across the surface of the transistor, but do not apply too much.

Install the new transistor and be careful not to bend the legs and make sure to line up the screw holes with the socket holes on the back end. Use a finger behind the socket to hold the socket in place.

Tighten the screws in firmly, but do not over tighten.

Clean off any access compound around the transistor once fastened down.

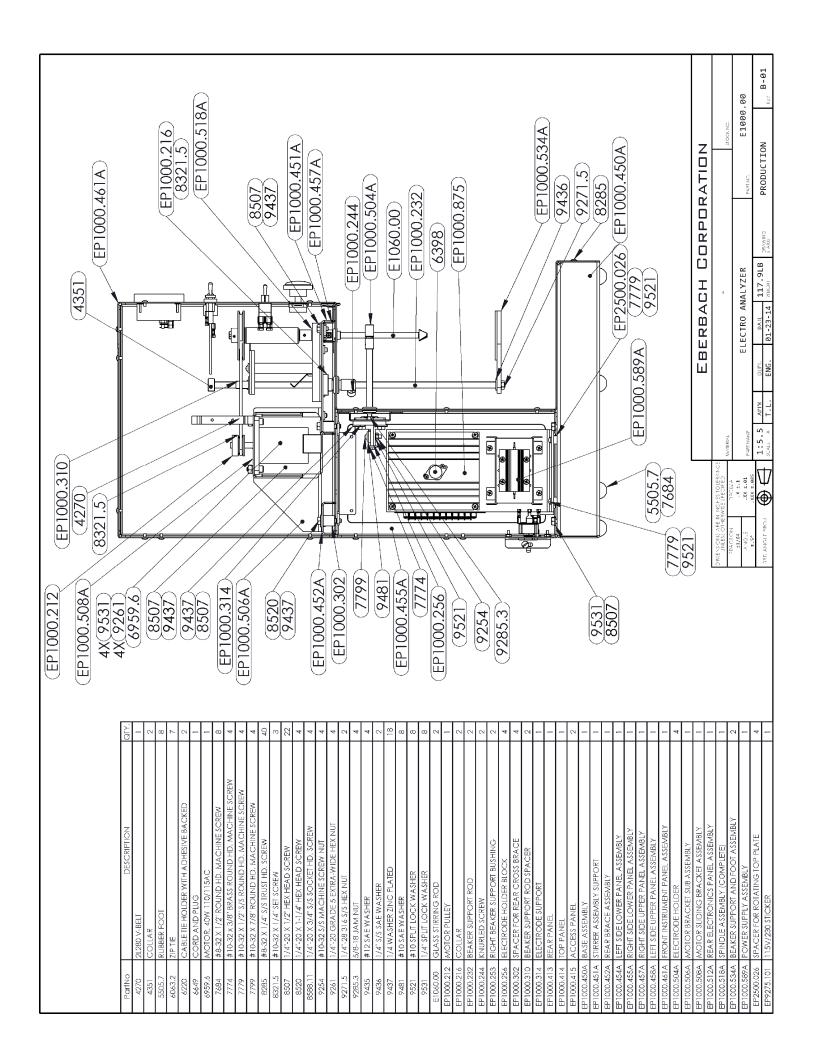
Reattach the heat sink to the power supply assembly.

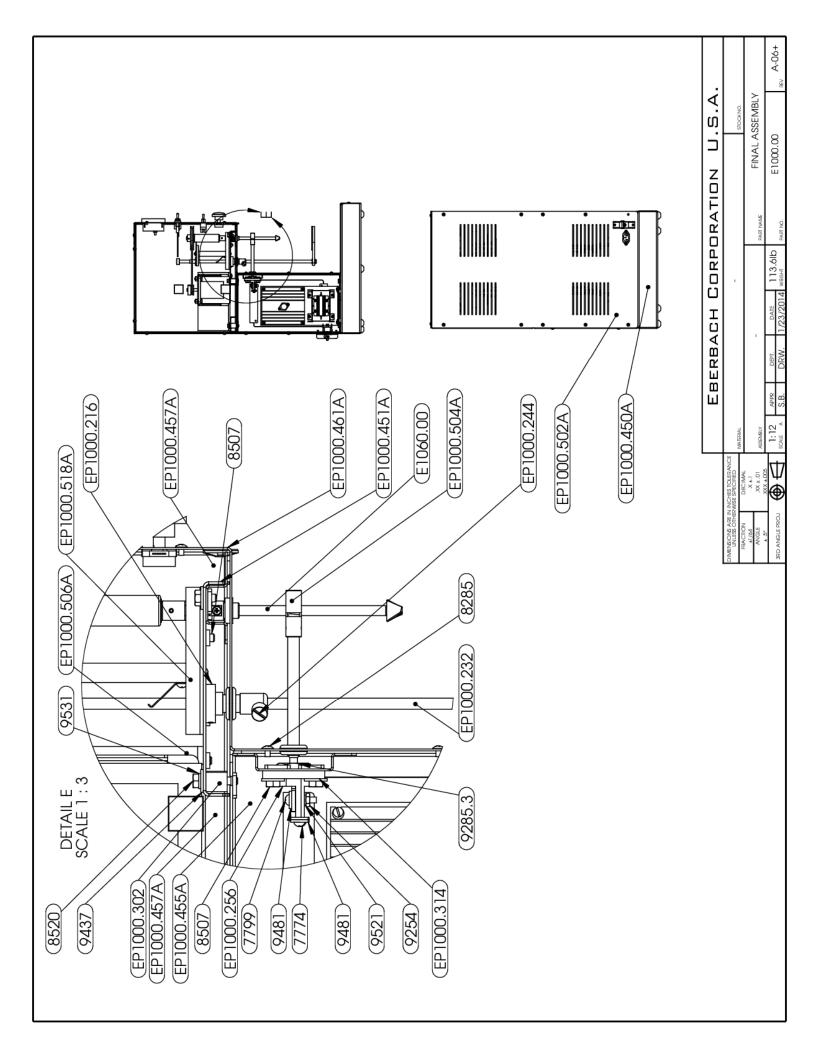
Fasten the side panel onto the unit.

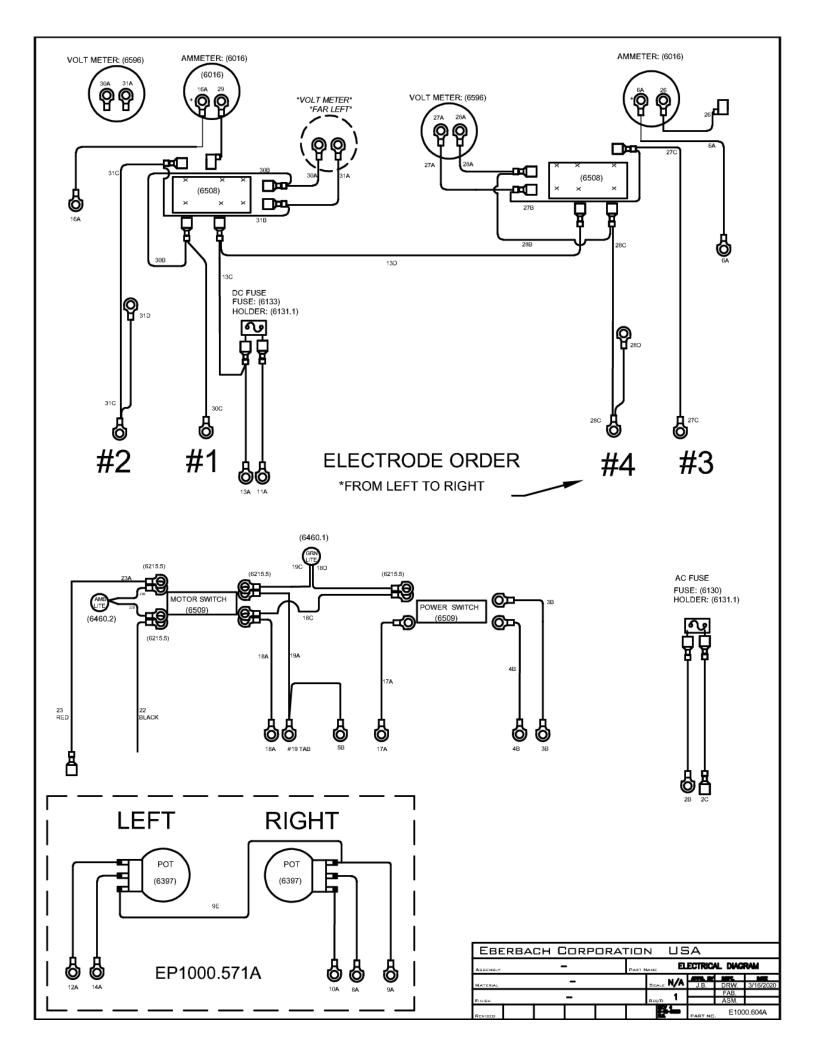
REPLACEMENT PARTS LIST FOR E1000.00

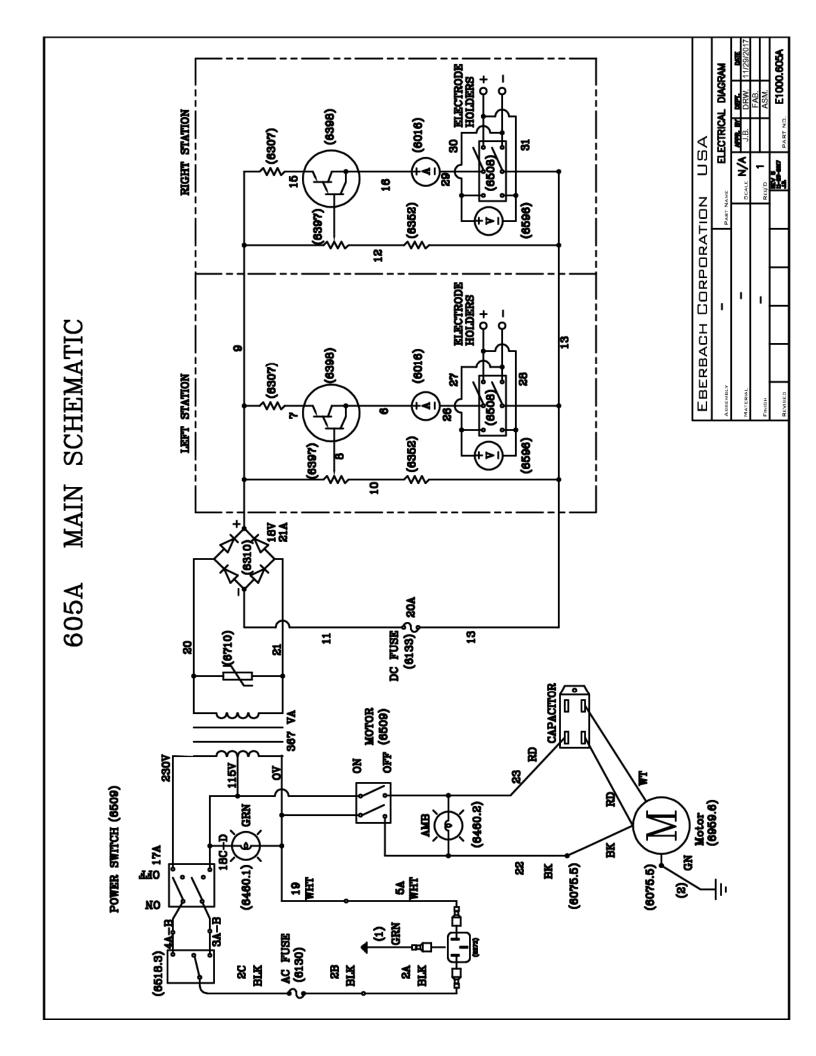
Part #	DESCRIPTION	QTY.
4270	V BELT	1
4351	COLLAR	2
5505.7	RUBBER FOOT	8
6063.2	ZIP TIE	7
6220	CABLE TIE HOLDER WITH ADHESIVE BACKED	2
6649	CORD AND PLUG	1
6959.6	MOTOR, 40W 110/115AC	1
7684	#8-32 X 1/2" ROUND HD. MACHINE SCREW	8
7774	#10-32 x 3/8" BRASS ROUND HD. MACHINE SCREW	4
7779	#10-32 X 1/2" S/S ROUND HD. MACHINE SCREW	4
7799	#10-32 X 7/8" ROUND HD. MACHINE SCREW	4
8285	#8-32 X 1/4" S/S TRUST HD. SCREW	40
8321.5	#10-32 X 1/4" SET SCREW	3
8507	1/4"-20 X 1/2" HEX HEAD SCREW	22
8520	1/4"-20 X 1-1/4" HEX HEAD SCREW	4
8588.11	1/4"-20 X 3/4" S/S SOCKET HD. SCREW	4
9254	#10-32 S/S MACHINE SCREW NUT	4
9261	1/4"-20 GRADE 5 EXTRA-WIDE HEX NUT	4
9271.5	1/4"-28 316 S/S HEX NUT	2
9285.3	5/8-18 JAM NUT	4
9435	#12 SAE WASHER	4
9436	1/4" S/S SAE WASHER	2
9437	1/4 WASHER ZINC PLATED	18
9481	#10 SAE WASHER	8
9521	#10 SPLIT LOCK WASHER	8
9531	1/4" SPLIT LOCK WASHER	8
E1060.00	GLASS STIRRING ROD	2
EP1000.212	MOTOR PULLEY	1
EP1000.216	COLLAR	2
EP1000.232	BEAKER SUPPORT ROD	2

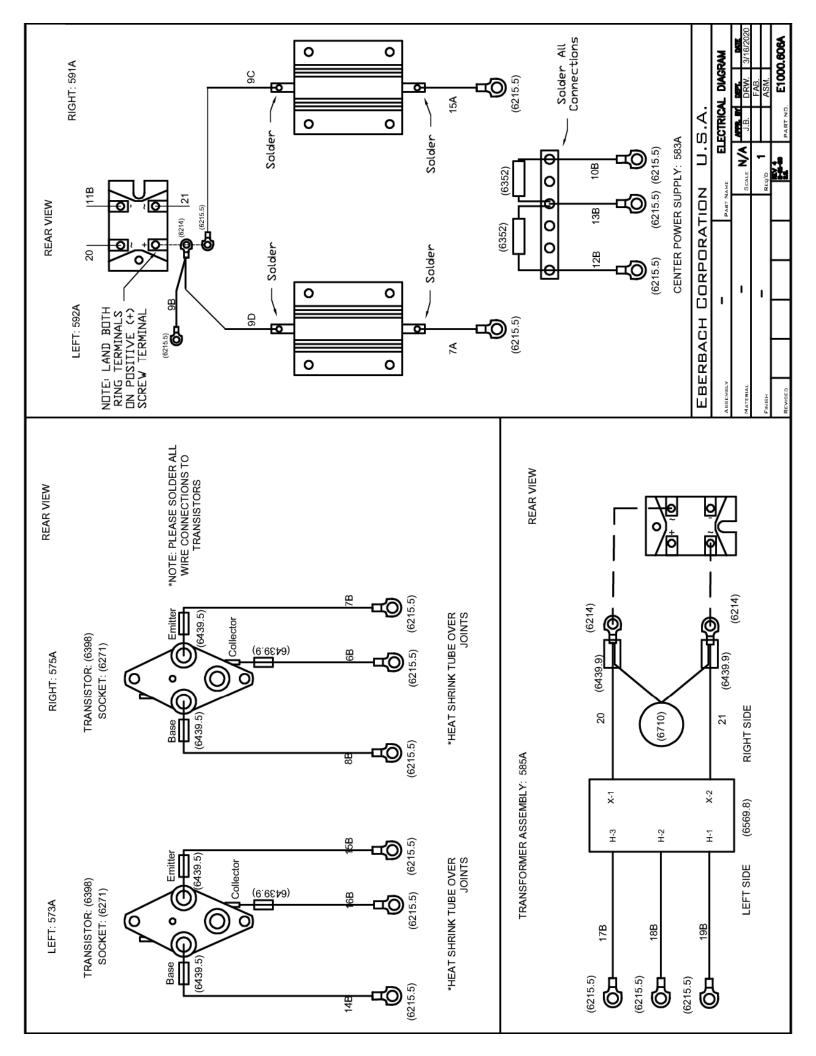
EP1000.244	KNURLED SCREW	2
EP1000.253	RIGHT BEAKER SUPPORT BUSHING	2
EP1000.256	ELECTRODE HOLDER BLOCK	4
EP1000.302	SPACER FOR REAR CROSS BRACE	4
EP1000.310	BEAKER SUPPORT ROD SPACER	2
EP1000.314	ELECTRODE SUPPORT	1
EP1000.413	REAR PANEL	1
EP1000.414	TOP PANEL	1
EP1000.415	ACCESS PANEL	2
EP1000.450A	BASE ASSEMBLY	1
EP1000.451A	STIRRER ASSEMBLY SUPPORT	1
EP1000.452A	REAR BRACE ASSEMBLY	1
EP1000.454A	LEFT SIDE LOWER PANEL ASSEMBLY	1
EP1000.455A	RIGHT SIDE LOWER PANEL ASSEMBLY	1
EP1000.457A	RIGHT SIDE UPPER PANEL ASSEMBLY	1
EP1000.458A	LEFT SIDE UPPER PANEL ASSEMBLY	1
EP1000.461A	FRONT INSTRUMENT PANEL ASSEMBLY	1
EP1000.504A	ELECTRODE HOLDER	4
EP1000.506A	MOTOR BRACKET SUB ASSEMBLY	1
EP1000.508A	MOTOR SLIDING BRACKET ASSEMBLY	1
EP1000.512A	REAR ELECTRONICS PANEL ASSEMBLY	1
EP1000.518A	SPINDLE ASSEMBLY (COMPLETE)	1
EP1000.534A	BEAKER SUPPORT AND FOOT ASSEMBLY	2
EP1000.589A	POWER SUPPLY ASSEMBLY	1
EP2500.026	SPACER FOR ROTATING TOP PLATE	4
EP9275.101	115V/230 STICKER	1

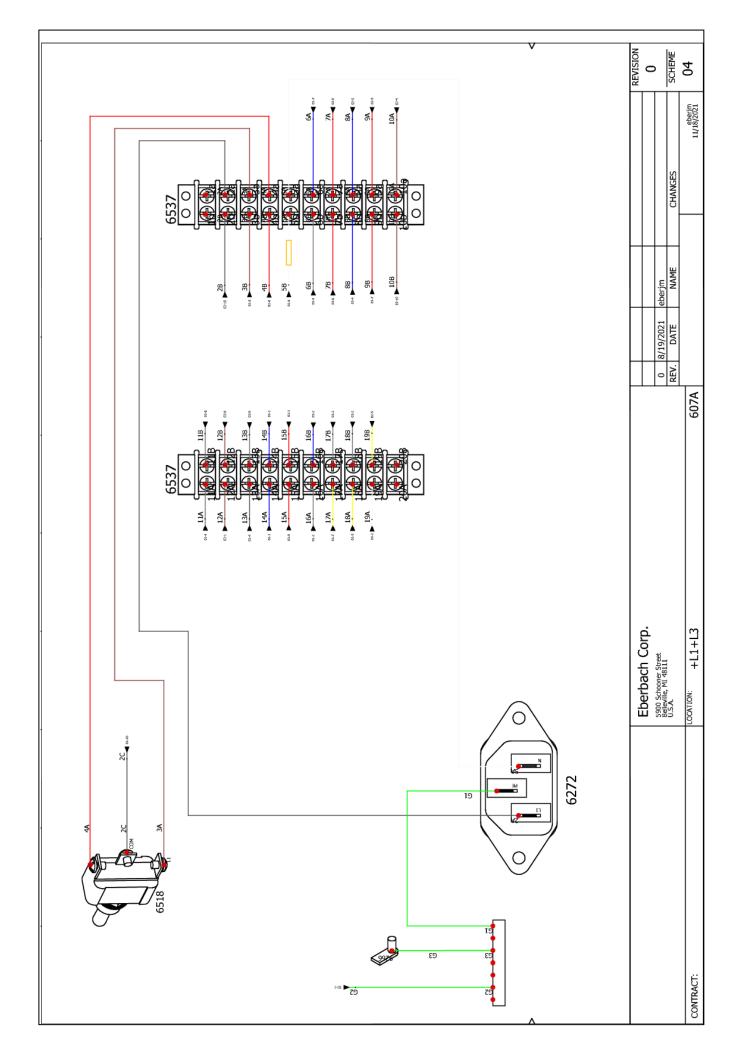












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