# Model KO264 Micro-Cell Kit Assembly and Checkout Instructions

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# Model K0264 Micro-Cell Kit
# Assembly and Checkout

## 1. Introduction

This instruction guide provides assembly instructions for the Model K0264 Micro-Cell, which is normally shipped as a kit to be assembled by the user. It additionally includes a procedure for performing a Cyclic Voltammetry experiment so that correct operation can be verified. Although he procedure is written around use of the Model 384B-ED Console as the potentiostat, it could be readily adapted to use with any potentiostat able to provide the required sweep voltages. **Note:** *The standard Model 384B cannot be used unless the internal electrometer chip has been installed.*

Basically, the procedure consists of doing a cyclic voltammetry measurement of a known sample using a glassy-carbon electrode mounted on the K0264 Micro-Cell. Should you have any problems with the assembly or procedure, contact the factory authorized representative in your area for assistance.

## 2. Required Equipment and Chemicals

1. A scanning potentiostat able to make a cyclic scan from 0 to +1.2 V.

2. A digital plotter that is compatible with the scanning potentiostat. If you are controlling the cell from a Model 384B, use a RE0093 Digital Plotter.

3. An Eppendorf pipette (or similar device) for adding a 500 $\mu$l spike to a sample.

4. A squeeze bottle of distilled water.

5. A beaker for catching the rinse water when the cell electrodes are rinsed.

To perform the discussed experiment, you will also require the following chemicals.

1. 0.1*M* Tetrabutylammonium tetrafluroborate (Aldrich) in acetonitrile buffer prepared by disolving 15.45 *g* $TBA^+$ $BF^-_4$ in 500 ml freshly distilled reagent grade acetonitrile. *Molecular weight of Tetrabutylammonium tetrafluoroborate is 329.29*.

2. Stock solution of 20 m*M* Ferrocene prepared by disolving 372 mb Ferrocene (Aldrich) in 100 ml of above buffer. Molecular weight of Ferrocene is 186.04.

# 3. K0264 Assembly

The following assembly procedure is keyed to Figure 1, *MODEL K0264 MICRO-CELL*. Whenever possible, parts are identified by name and by item number as listed in the figure.

1. Place the black O-Ring into groove on under side of Cell Top (1).

2. Take the K0265 Reference Electrode (4) and insert it into the appropriate Bushing (3). Next slide a Ferrule (15), from the bottom of the electrode, up the electrode to the Bushing (3). Then screw the completed Reference Electrode Assembly into the Cell Top (1).

3. Take the K0266 Counter Electrode (6) and insert it into the appropriate Bushing (5). Ne& slide a Ferrule (15), from the bottom of the electrode, up the electrode to the Bushing (5). Then screw the completed Counter Electrode Assembly into the Cell Top (1).

4. Take the Optional Working Electrode (8) and insert it into an appropriate Bushing (7). NM slide a Fernile (15), from the bottom of the electrode, up the electrode to the bushing (7). Then screw the completed Working Electrode Assembly into the Cell Top (1).

5. Screw in the Sample Port Plug (9) or the optional Sample Port Bushing (10).

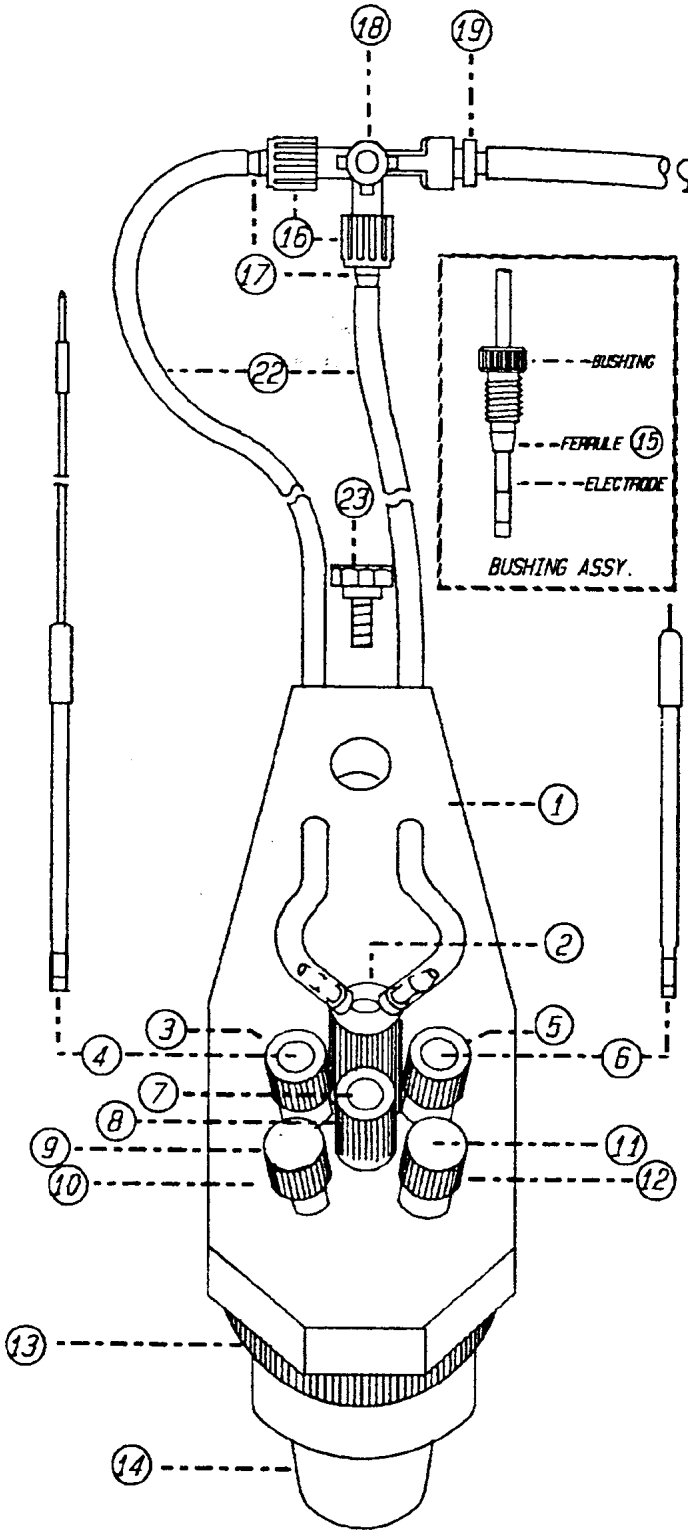
6. Screw in the Thermometer Port Plug (11) or the optional Thermometer Port Bushing (12).

7. Take the length of smaller diameter tubing (22) and cut it with a razor blade into two equal lengths. Then locate the K0268 Purge Tube Assembly (2) and connect one end of one of the two lengths of tubing to the black fitting of the Purge Tube Assembly and one end of the second length of tubing to the white fitting of the Purge Tube Assembly (2).

8. Screw the K0268 Purge Tube Assembly (2) into the Cell Top (1). Then route the free end of one of the lengths of tubing through one of the small holes located towards the end of the Cell Top and the free end of the other length of tubing through the other small hole. Extend both lengths of tubing to the rear as shown in Figure 1.

9. Stopcock Assembly: Locate the Stopcock and the small bag containing the parts require to complete the assembly. Then proceed as follows.

    a. Examine the Stopcock Assembly. Note that there are three ports. Two of the ports are the same. A Removable Luer Lock Ring is attached to these two ports. ***Unscrew the two removable Luer Lock Rings and discard*** **them.** The third port has a non- removable Luer Lock Ring, into which an insert has been placed. **Pug the insen from the non- removable Luer Lock Ring Then discard the insert which has just been removed**.




| PARTS LEGEND | | | |
|---|---|---|---|
| ITEM | DESCRIPTION | PART NO. | MATERIAL |
| 1 | CELL TOP | 219581 | POLYPROPYLENE |
| 2 | K0268 KIT-<br>PURGE TUBE ASSY.<br>FITTING<br>TUBING<br>FITTING<br>FITTING | <br><br>220254<br>230212<br>230213<br>230214 | <br><br>POLYPROPYLENE<br>TEFLON<br>POLYPROPYLENE<br>POLYPROPYLENE |
| 3 | BUSHING | 220196 | POLYPROPYLENE |
| 4 | K0265 KIT-<br>REF. ELECTRODE | 219995 | SODA LIME GLASS |
| 5 | BUSHING | 220196 | POLYPROPYLENE |
| 6 | K0266 KIT<br>COUNTER ELECTRODE | 220198 | SODA LIME GLASS |
| 7 | BUSHING | 220253 | POLYPROPYLENE |
| 8 | WORKING ELECTRODE-<br>MICRO-GOLD<br>MICRO-PLATINUM<br>MICRO-GC<br>MILLI-GOLD<br>MILLI-PLATINUM<br>MILLI-GC | <br>G0224<br>G0225<br>G0226<br>G0227<br>G0228<br>G0229 | SODA LIME GLASS |
| 9 | PLUG | 220325 | POLYPROPYLENE |
| 10 | TO TAKE SAMPLE PORT (OPTIONAL) | | |
| 11 | PLUG | 220262 | POLYPROPYLENE |
| 12 | TO TAKE THERMOMETER (OPTIONAL) | | |
| 13 | CAP | 220553 | POLYPROPYLENE |
| 14 | SPECIMEN CELL | 219600 | GLASS |
| 15 | FERRULE | 230259 | TEFLON |
| 16 | LUER LOCK RING | 231573 | POLYPROPYLENE |
| 17 | FITTING | 231574 | POLYPROPYLENE |
| 18 | STOPCOCK | 231572 | POLYCARBONATE |
| 19 | FITTING | 231575 | POLYPROPYLENE |
| 20 | TUBING | 231581 | POLYETHYLENE/ETHYL VINYL ACETATE SHELL |
| 21 | FITTING | 231576 | POLYETHYLENE |
| 22 | TUBING | 230197 | POLYETHYLENE/ETHYL VINYL ACETATE SHELL |
| 23 | KNOB | 230125 | THERMOPLASTIC |

**Figure 1. MODEL K0264 MICRO-CELL**

b. Locate the two removable Luer Lock Ring supplied in the plastic bag. These differ from the ones discarded in the previous step in that they have a square opening instead of a round opening. Then locate the two identical beveled Fittings (17) supplied in the plastic bag. *There is a third Fitting (19) as well, but if you look closely, you will see that it is different from the other two*. Take one of the Luer Lock Rings (16) and insert the beveled end of a Fitting (17) into it. It should be inserted into that end of the Luer Lock Ring that has the circular opening so that the two pieces will make a locked connection. *It may be necessary to slightly rotate the Fitting (17) so that the square flange on the Fitting (17) mates with the square opening on the Luer Lock Ring (16).*

   Repeat for the second Fitting (17) and Luer Lock Ring (16).

c. Screw these two just completed assemblies onto the two alike ports (one is the purge gas output, the other the blanketing gas output) of the Stopcock (18).

d. Locate Fitting (19) in the plastic bag. Looking at it, note that one end is tapered for insertion into tubing. The other end has an almost elipsoidal shape. Insert Fitting (19), elipsoidal end first, into the non-removable Luer Lock Ring of the Stopcock Assembly and rotate it clockwise to the locked position. Then rotate the non-removable Luer Lock Ring counterclockwise so that it secures the Fitting (19). This completes the Stopcock Assembly.

e. Take the larger size Tubing (20) and push it onto the end of Fitting (19). **Note:** Fit will be very tight. We suggest that you soak the tubing in acetone to soften it. Alternatively, heat it gently to soften it.

f. Insert Fitting (21) into the free end of Tubing (20). This fitting is optional. If the tubing coming from your purge/blanketing gas source is a different size, installation of an appropriate adapter will be necessary.

g. Connect the free end of the tubing coming from the Purge (white) port of the Purge Tube Assembly and connect it to one of the two alike ports of the completed Stopcock Assembly. Similarly take the free end of the tubing coming from the Blanketing (black) port of the Purge Tube Assembly and connect it to the other one of the two alike ports of the completed Stopcock Assembly.

10. Insert the Specimen Cup (14) into the Cap (1-3). *Note: The Cap (14) is also referred to as the **Retaining Ring**.*

11. Screw the Cap with Specimen Cup to the bottom of the Cell Top (1).

12. Screw the Locking Knob (23) into the hole at end of the Cell Top (1).

13. Mount the completed MicroCell Assembly onto an appropriate ring-stand rod and secure with the Locking Knob (23).

# 4. INSTALLATION

1. Set the Model 384B, the assembled Model K0264 MicroCell, and the RE0093 Digital Plotter on a suitable lab bench. Again, this procedure assumes a 384B, but can be adapted to other instruments.

2. Plug the Model 384B and the Plotter into a source of suitable ac power.

3. Using the cables provided, interconnect the system components.

   Two cables are supplied with the Model 384B Educational System. One of these interconnects the Model 384B with the Plotter. The other, which terminates in a pin jack and three color-coded alligator clips, interconnects the Model 384B with the cell assembly. The numbers of the cables are C0191 (Model 384B to Plotter) and C0175 (Model 384B to K0264 Micro-Cell Assembly).

   The cable between the Model 384B and the Plotter mates with the RS232 connector at the rear of the Model 384B and with the INPUT connector at the rear of the plotter.

   The Model 384B to cell-assembly cable mates with the CELL COMPARTMENT connector at the rear of the Model 384B. The alligator clips at the other end of the cable connect to the appropriate electrodes at the K0264 Micro-Cell. The white clip leads to the reference electrode, the green clip lead connects to the working electrode, and the red clip lead connects to the counter electrode. The black clip lead is a convenient source of ground for a Faraday shield, and can be left disconnected. However, if left unconnected, take care that it doesn't short against any of the other circuit elements.

4. Check that the disk supplied with the Model 384B is properly installed. *See Section 5.4F.*

5. Connect a source of deoxygenated nitrogen to the gas input port of the K0264 MicroCell *(see Figure 1).* As shown, the gas input is to the gas-valve input fitting (19). There are two outputs, one to the purge-gas line, the other to the blanketing-gas line. The valve can be set to **OFF** or to direct the gas to either the purge-gas or blanketing-gas line. The gas pressure must be adjustable up to a maximum of nominally 5 psi (34.5 kPa). In most applications the optimum pressure will be lower.

6. Install a pen in the clip on the plotter's penholder assembly. Before installing the pen, first remove the protective cap and rub the pen on a paper surface to establish the ink flow. Take care to save the protective cap. It should be pressed back onto the pen when The pen is not in use. The pen is installed by gently pushing the large center rim of the pen between the jaws of the pen holder.

7. Turn on the plotter and press the plotter's SNLALL button. Then install a sheet of 81/2 x 11 chart paper in the plotter. (The paper provided with the system can be used.) To do so, first lift the lever at each side of the platen. Then slip the paper between the pressure rollers and the platen. Position the paper so that the edge of the paper is aligned with the front edge of the plotter platen. When the paper is correctly positioned,

secure it by returning the levers to the down position. Pictures and additional information are provided in the Plotter Instruction Manual.

**Note:** If the plotter is equipped with the roll-feed option, users will probably find it more convenient to load a roll of paper and simply advance the paper by pressing the **Paper Advance key** whenever a fresh plotting surface is required. *Alternatively, setting Override 22 to Y will automatically advance the paper.*

8. Turn on the Model 394B Power switch (switch is at the rear and to the right, of the Model 384B). When this is done, the message **WAIT XX** (XX represents the two character code that designates the version of Read Only Memory firmware in the unit) is displayed. This message is followed by the message **MODEL 384B POLAROGRAPHIC ANALYZER YY.XX** (YY is the disk software version; XX is the firmware version). If these messages do not appear as described, the unit is malfunctioning and the user should contact the factory for advice.

# 5. Cell Preparation

1. Unscrew the Retaining Ring with Specimen Cup from the bottom of the Cell Top. Then rinse the electrodes using distilled water from a squeeze bottle. *Put a beaker beneath the K0264 MicroCell to catch the rinse water.*

2. Place 10 ml of the 0.1 *M* Tetrabutylammonium tetrafluoroborate (Aldrich) in acetonitrile buffer previously prepared into the specimen cup and screw the Retaining Ring with Specimen Cup to the bottom of the Cell Top.

# 6. Blank

An underlying assumption at this point is that the Model 384B power is on and that the display indication is **BLANK DPP.** This message should have been displayed for all but the first ten or fifteen seconds after powerup. If the displayed message is anything other than **BLANK DPP,** press the rear-panel **RESET** pushbutton. The desired message should appear within 10-15 seconds, and a 'beep' indicating that the unit is receptive to keyboard commands should occur in another two to five seconds.

Note: This is not to say that the intended technique for these initial checks is DPP. it is rather Cyclic Voltammetry.

Using the console panel's membrane keys, set the analysis parameters as follows.

Technique: CYCLIC VOLTAMMETRY
Purge time: 0
INITIAL POT'L: 0 V
FINAL POTL: + 1.0 V

Pulse Height: unused in cyclic voltammetry
Drop/Step Time: 0.250 s
Scan Increment: 10 mV
Sample Type: BLANK
Replications: 1
Sample #: 1
Date: 9-JUL-93
Cell Number: 0
Scan Rate (calculated value): 40 mV/s

The procedure for running the blank follows.

1. Set the K0264 MicroCell gas input Stopcock to the purge position, and allow purging (steady stream of nitrogen bubbling through the solution) to continue for at least four minutes. Than transfer the gas input Stopcock to the blanketing position.

2. Press **RUN.** The run will proceed and the point-by-point current/voltage values will be displayed but not plotted. On completion of the run, the curve will be automatically recorded on the internal disk. A tone will then sound to indicate that this operation is complete. The display will return to the status state.

3. Establish a fresh plotting surface at the plotter.

4. Press **PLAYBACK**. The playback data will be automatically scaled and should appear something like Figure 2, although wide variations are possible.

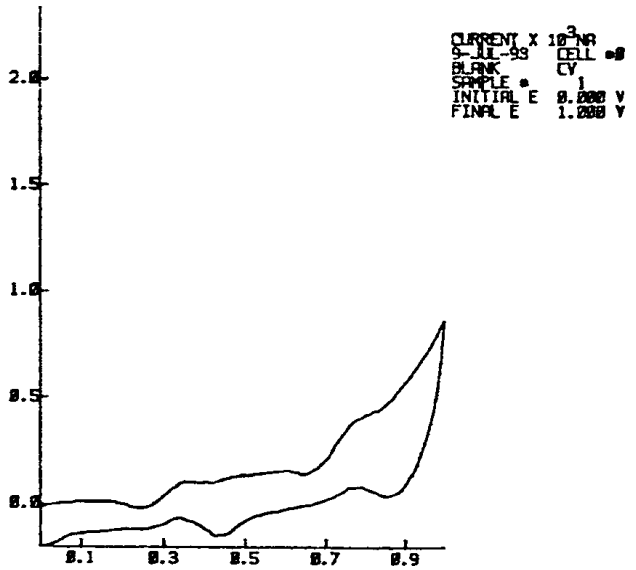



**Figure 2. PLAYBACK OF BLANK**

***Note**: In this example, Blank Subtract is set to NO. If you want to subtract your blank from your standard and sample, reset it to **YES**.*

# 7. STANDARD

1. Using an Eppendorf Pipette, spike the 10 ml of buffer with 500 $\mu$l of the 20 m*M* Ferrocene solution previously prepared. Assuming the premeasured volume of the blank solution is 10 mL there will be a 20:1 dilution of the 'spike', resulting in a solution containing Ferrocene at a concentration of 1.0 m*M.* This solution will serve as the *standard* for these checks.

2. Turn the Stopcock from **blanket** to **purge** to gently stir the solution with the purge gas. After approximately ten seconds, set back to the **blanket** position.

3. Press the **STANDARD** key. The displayed word **BLANK** will be replaced by **STANDARD # 1.**

4. Press RUN. Analysis of the standard will commence and go to completion. The resulting curve will not be plotted but will be automatically stored on the disk.

5. Replace the paper to obtain a. fresh plotting surface.

6. Press **PLAYBACK.** The data acquired during the preceding **run** will be played back, rescaled and complete with labeled axes. The peak representing Ferrocene should be clearly **visible** and the peak potential and current listed. Although the actual values may vary somewhat, by and large, the playback curve produced should be similar to that shown in Figure 3.

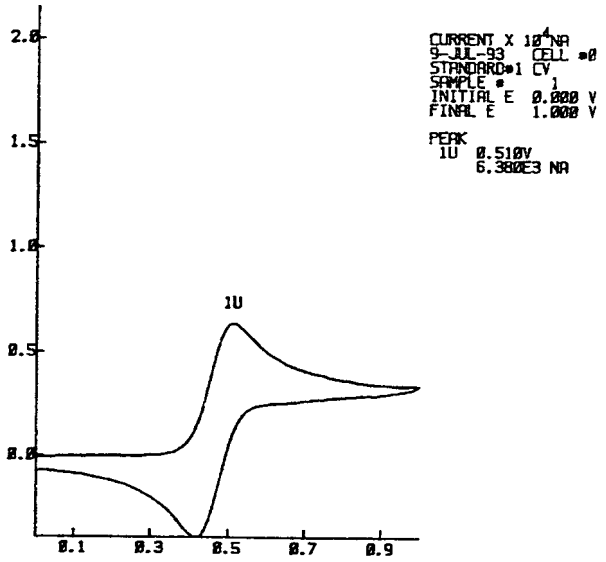



**Figure 3. PLAYBACK OF STANDARD Before Entering Concentration**

7. In order for the Model 384B to do concentration calculations based on the data taken from the standard run, concentration and peak potential information must be entered. This is done as follows:

   a. Press **PEAK POTENTLAL** and "**1**". Then key the potential listed for Peak 1 in the results listing for the playback of the standard. Value should be 0.510 V in this example.

   b. Press **PEAK CONCENTRATION** and "**1**". Then key **"186** and **"PPM".** *A concentration of 1 mM Ferrocene is equivalent to 186ppm.*

8. For the Model 384B to use the information just entered, it is necessary to run another playback. To do so, replace the paper to obtain a new plotting surface and press **PLAYBACK** . A new playback record will be produced. In this one, the specified concentration (186 ppm) will be listed for the peak, together with the potential and current. The playback should be similar to that depicted in Figure 4.

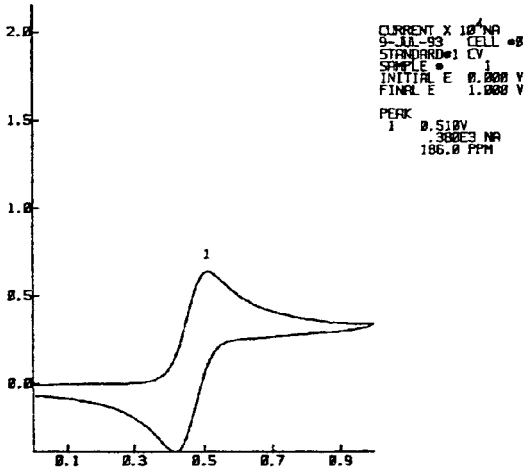



**Figure 4. PLAYBACK OF STANDARD After Entering Concentration**

# 8. SAMPLE

1. Spike the solution in the sample cup (again) with 500 $\mu$l of the previously prepared 20 m*M* Ferrocene solution. Then turn on the purge for 10 s to stir the sample.

2. Press the **SAMPLE** key. The displayed **STANDARD 1** message should be replaced by **SAMPLE 2.** In effect, the 'standard' has been converted to a convenient "sample".

3. Install a fresh sheet of paper in the plotter.

4. When ready, press **RUN.** The analysis of the 'sample' will proceed in much the same way as the run-time analyses of the blank and standard. The acquired data is not plotted but is automatically stored on disk and a beep sounds to indicate that the user can proceed to the next operation.

5. When ready, press **PLAYBACK.** The resultant playback recording of the sample data will be complete with the computed concentration (367.4 ppm in example). The recording should be similar to that depicted in Figure 5. Note that, at this concentration, a small impurity at 0.150 V is evident. This doesn't interfere with the Ferrocene analysis. The calculated concentration of our peak is 372 ppm with the actual value recorded from the analysis being 367.4 ppm.

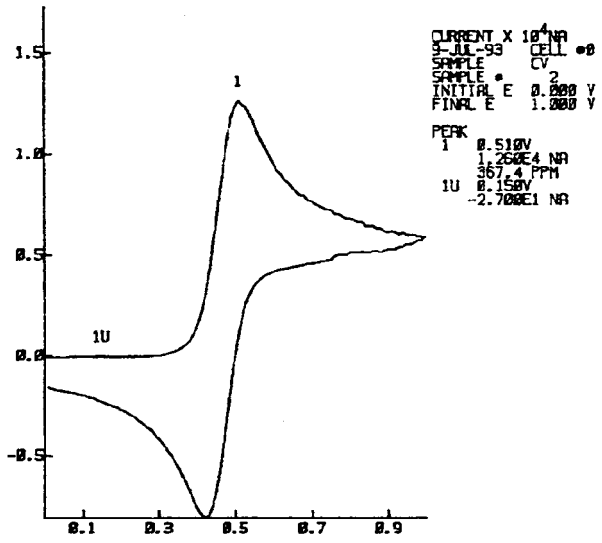



**Figure 5. PLAYBACK OF SAMPLE**

6. Replace the paper to provide a fresh plotting surface.

   When ready, press **LIST METHOD**. The recorder will proceed to generate a complete listing of the critical parameters for the just completed analysis. The final record should be similar to that shown in Figure 6.

```
                    MODEL 384B POLAROGRAPHIC ANALYZER
9-JUL-93      CELL NUMBER  0          SAMPLE _____________________
CV                                     ANALYTE _____________________
INITIAL E  0.000 V                     SUPP. ELEC. _________________
FINAL E    1.000 V                     SAMPLE PREP. ________________
PURGE      0 SECONDS                   _____________________________
STEP TIME 0.250 SECONDS                _____________________________
SCAN INCREMENT  10 MV
CYCLES     1
REPLICATIONS   1

STANDARD CURVE
BLANK SUBTRACTION: NO
TANGENT FIT: YES
PEAK LOCATION: YES
DERIVATIVE: NO
FORCE LINEAR FIT: NO

                    P P P P P P P P P P P P P P P P
OVERRIDE            Y N Y Y N Y Y N N Y N Y Y N Y N

PEAK    POT          STD 1        STD 2        STD 3
  1   0.510 V      186.0 PPM
```

**Figure 6. METHOD LISTING**

# 9. Final Comments

This completes the checkout procedure. If the indicated behavior was observed, the user can be reasonably confident that the cell is working properly.

Failure to obtain the indicated results could mean that one of the system components is malfunctioning. However, this may not necessarily be the case. The system is complex and experimental difficulties can be encountered in a number of different areas. Diagnosing a problem may not be simple or straightforward. Before assuming there is an equipment failure, users should review the instruction manuals in depth. Carefully check the installation for cabling or other errors. If all possibilities for the cause of an Initial Checks failure have been exhausted, contact the factory or the factory representative in your area for further assistance.