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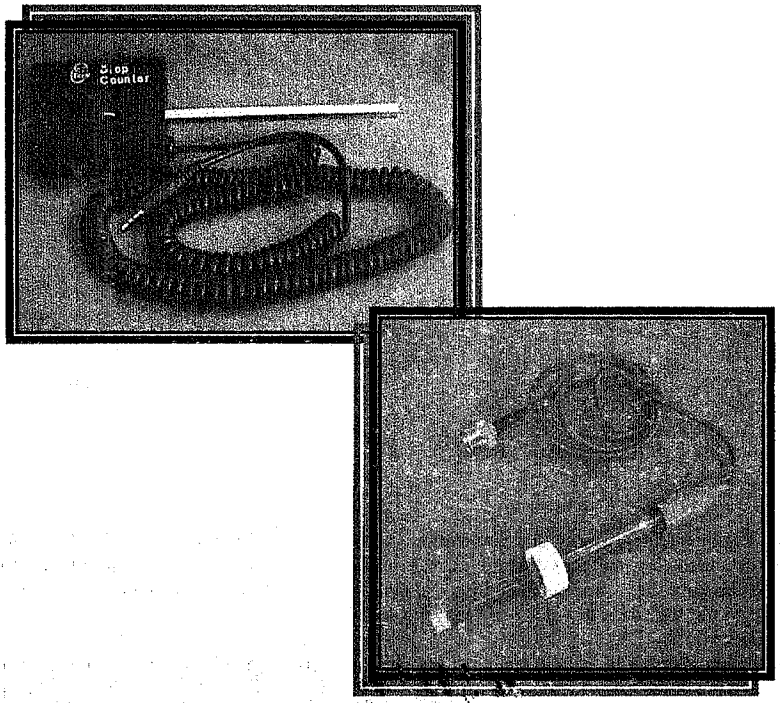
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Titration Techniques with LabWorks



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INTRODUCTION

The LabWorks™ interface can be used to perform a potentiometric acid/base titration and obtain a pH titration curve. This curve can be used to determine the equivalence point of an acid/base titration. Potentiometric titrations measure pH with respect to the volume of titrant added. It is more accurate than a titration that uses a visual indicator because the equivalence point can be found graphically.

DESCRIPTION

The SCI Technologies Drop Counter and the pH electrode are used in conjunction with one of the LabWorks™ interface models to obtain a pH titration curve of pH versus volume.

Drop Counter (model C0015)

The drop counter counts the number of drops of a conductive solution that passes between the adjustable electrodes in the center of the sensor body. Drops of solution with conductivity at least as high as tap water can be accurately counted.

pH Electrode (model A0079)

The pH electrode is a rugged Standard electrode with a BNC connector and epoxy body. It will measure pH in the range of 0 to 12, or 0 to 14 depending on the electrode model.

SPECIFICATIONS

Drop Counter

The drop counter is packaged in a black Delrin® housing with a 5/8 inch hole to allow for passage of drops. Platinum wire is used for the two electrodes to reduce corrosion and extend the life of

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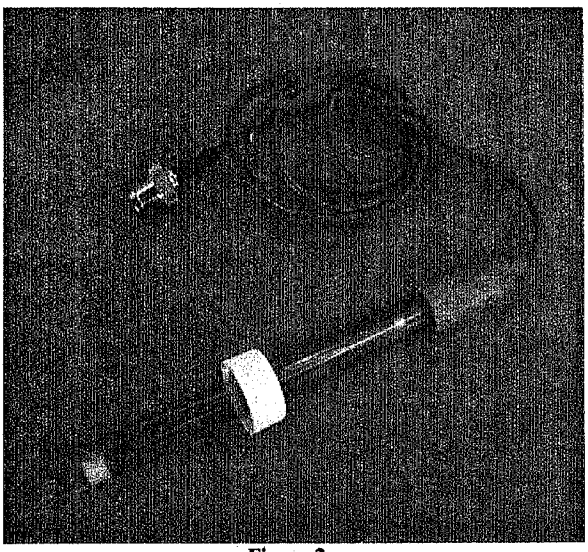


Figure 2
pH Electrode

CONNECTIONS

Drop Counter

Insert the 1/8" stereo plug on the end of the sensor cable into the corresponding jack on the interface, labeled Counter on the front panel of the LabWorks™ and LabWorks II® interfaces and CNT on the front panel of the LabWorks II®-100 interface.

pH Electrode

Connect the BNC plug on the end of the pH electrode cable to the BNC jack on the interface, labeled pH/mV 1 on the front panel of the LabWorks™ and LabWorks II® interfaces and pH/mV on the LabWorks II®-100 interface. Make sure to turn the BNC connector ¼ turn clockwise after lining up the notches in the plug with the bumps on the interface jack. This will lock the connector and ensure a tight connection.

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a stable reading, see the technical specifications section (page 20) for more information.

5. Enter the pH of the buffer solution in the box with the blinking cursor and hit the "enter" key to lock the millivolt reading.

6. If the pH reading has been entered correctly, click on the "OK" button or hit "enter" again to write the calibration information to computer memory (Figure 4)

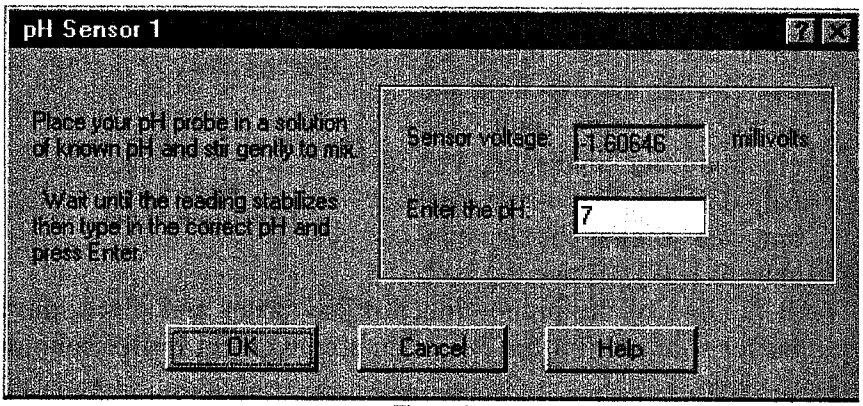


Figure 4
pH Calibration Window

The LabWorks software will use this calibration information to convert the analog signal from the pH electrode to units of pH prior to being displayed.

See the usage section (page 7) for programming examples.

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Calibration information is stored in the interface memory (DOS and Macintosh) or in the computer memory (Windows) and will remain until a new calibration is performed. If the interface, computer, or sensor is changed, a new calibration will have to be performed. Calibrations will also have to be performed periodically due to changes in parameters for the components used in the sensor and interface.

Alternate Calibration for the pH electrode

The pH electrode is a voltage-producing sensor. At room temperature (25°C), the pH probe changes its output voltage by 59 millivolts for each unit change of solution pH. The single point calibration routine described above assumes a temperature of approximately 25°C. For solutions that are not close to room temperature or if a two-point calibration is desired, use the mVProbe calibration routine. Calibration steps are the same as the steps mentioned previously, except the mVProbe calibration routine corresponding to the input used is chosen instead of the pH calibration routine. Two buffer solutions with known pH will also be needed. Experiments will have to be written to reflect the use of the mVProbe calibration. See the Usage section for programming examples.

USAGE

Using LabWorks to perform a potentiometric titration has the distinct advantage of providing a graphical indicator of the equivalence point of the acid/base titration. The key to using LabWorks for performing titrations is obtaining an accurate representation of the volume of titrant added. Three different methods of measuring volume can be used: a user input titration, where the volume of titrant added is entered at a prompt; a timed titration, where the flow rate of a burette is determined as volume per unit time; or a drop count titration, where the volume of each drop of titrant is determined. The last two methods require

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7. Repeat Steps 4-6 at least two more times.

8. Average your readings and determine the flow rate in ml per second.

Experiment for determining volume based on flow rate

Windows software using a LabWorks™, LabWorks II®, or LabWorks II®-100 interface

```
1  If SwitchW = Released (fast reading) then GoTo line 1
2  Start Timer1 counting in seconds
3  Read Timer1; store in Variable A; Graph on None
4  If SwitchW = Released (fast reading) then GoTo line 3
5  Stop
```

DOS or Macintosh software using a LabWorks™ or LabWorks II® interface

```
1  PRINT "This experiment will help you to determine the flow
   rate for your burette." ON LINE 1
2  PRINT "First, fill your burette with titrant." ON LINE 3
3  PRINT "Second, adjust the flow of the burette to 1-2 drops
   per second." ON LINE 5
4  PRINT "Third, press Switch W when the burette level reaches
   an easily" ON LINE 7
5  PRINT "readable mark. This will start the timer." ON LINE 8
6  PRINT "Finally, when the level reaches another easily" ON
   LINE 10
7  PRINT "readable mark, press Switch W again. This stops"
   ON LINE 11
8  PRINT "the timer and displays the value." ON LINE 12
9  PRINT "Elapsed Time (seconds)" ON LINE 15
10 PRINT "Press the 'S' key to stop." ON LINE 24
11 IF INPUT FROM LastKey = 115.000000 GOTO 22
12 IF INPUT FROM SwW = Released GOTO 11
13 DELAY 500 millisec
14 START Timer1 FOR sec
15 PRINT INPUT FROM Timer1 ON LINE 16
16 IF INPUT FROM LastKey = 115.000000 GOTO 22
```


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5. Check to see that the LabWorks™ Counter is counting by looking at the Pulses LED or by looking at the display of counts on screen.

6. Once your setup is complete, and LabWorks™ appears to be counting the droplets correctly, you are ready to determine the drop volume. Press Switch W on the front of the LabWorks™ or LabWorks II® interface when you are ready to begin and the titrant level in your burette reaches an easily readable mark. This will reset the counter. LabWorks II®-100 users must use the software "button" located on the Acquire screen. This switch functions identically to the hardware switch on the LabWorks™ and LabWorks II® interfaces.

7. When the level has decreased by either 2 or 3 ml, press Switch W again. The number of drops counted will be displayed. Write the elapsed drop count and the volume of titrant used in a notebook.

8. Repeat steps 7 and 8 at least two more times.

9. Average your readings and determine the volume of titrant per drop. You may use larger volumes to more accurately determine the volume, or average more trials.

Experiment for determining volume based on drop count

Windows software using a LabWorks™, LabWorks II®, or LabWorks II®-100 interface

```
1  If SwitchW = Released (fast reading) then GoTo line 1
2  Reset counter to zero
3  Read Counter; store in Variable A; Graph on None
4  If SwitchW = Released (fast reading) then GoTo line 3
5  Stop
```

DOS or Macintosh software using a LabWorks™ or LabWorks II® interface

```
1  RESET COUNTER
```


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recording the values read them from the last cell of the spreadsheet view. It gives more decimal places than the text display.

User-Input Titration

For a good titration curve, you will want at least 30 data points. Follow these steps to perform a user-input titration.

1. Clean, rinse, and fill your burette with the titrant and record its concentration.

2. Place the beaker with the solution you wish to titrate under the tip of the burette. Place a calibrated pH electrode into the solution.

3. Begin stirring the solution with a glass rod and leave the pH electrode stationary if stirring by hand. If you have an electric stirring device, arrange the sensors so that they do not interfere with it. Remember that you must continue to stir the solution throughout the titration.

4. Run a program written for performing a user-input titration (see below).

5. Allow 1 ml of titrant to flow into the solution from the burette. You may use smaller additions of titrant, but there may be little change in pH at the start of the titration (depending on the acidity or basicity of the solution and titrant).

6. Stir the solution for a few seconds, and enter the ml of titrant added to the solution at the software prompt.

7. Repeat steps 5-6 until the pH has changed by 1 pH unit or until the solution begins to change color due to an indicator (if used). If your solution is basic and your titrant is acidic, the pH will increase. If the reverse is true, it will decrease.

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This experiment will store the user-input volumes and the pH electrode readings in a spreadsheet file. In addition, a run-time graph and text format will also be displayed. The total volume of titrant added from the start of the experiment must be entered when prompted. The display mode chosen (Figure 6) determines which values will be displayed on the screen for the DOS and Macintosh software.

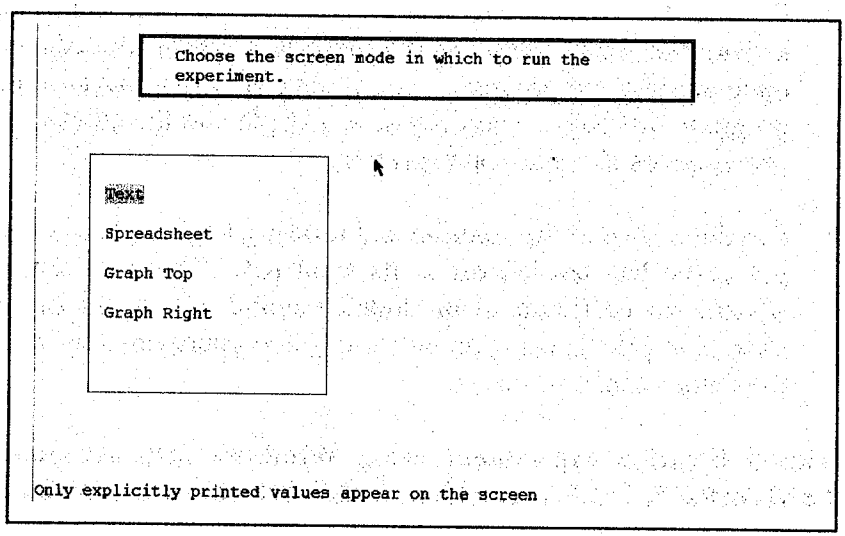


Figure 6
DOS Screen Mode Window

Timed Titration

In this procedure, it is important to maintain a constant level of titrant. You may find it easiest to fill the burette with a funnel or pipette, but it is important not to spill the titrant when filling, as it will most likely end up in the titration solution.

1. At this point, you should have already determined the flow rate for your burette, and the burette should still be dripping into a waste beaker.

2. Place your titration solution to the side of the waste beaker and insert the calibrated pH electrode into the solution. If you have an electric stirring device, arrange the probe so that it

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```
6  SEND INPUT FROM pH1 TO COL-B
7  PRINT INPUT FROM COL-A ON LINE 18
8  PRINT INPUT FROM COL-B ON LINE 21
9  SEND INPUT FROM COL-A TO GraphX (0 TO 300)
10 SEND INPUT FROM COL-B TO GraphY (0 TO 14
11 IF INPUT FROM LastKey = 115.000000 GOTO 14
12 DELAY 500 millisec
13 GOTO 5
14 STOP
```

This experiment will store the timer readings and the pH electrode readings in a spreadsheet file. In addition, a run-time graph and text format will also be displayed. This titration is automated and no user entry is required. The display mode chosen (Figure 6) determines which values will be displayed on the screen for the DOS and Macintosh software.

Drop Count Titration

In this procedure, it is important that the drops from the tip of the burette make contact with both electrodes in the through-hole of the Drop Counter.

1. At this point, you should have already determined the droplet volume. Prepare for the titration by stopping the flow of drops from the burette.

2. Place your titration solution under the burette and insert the calibrated pH electrode into the solution. If you have an electric stirring device, arrange the sensor so that it does not interfere with it. If stirring by hand, stir the solution with a glass rod, leaving the pH electrode stationary. Remember that you must continue to stir the solution throughout the titration.

3. Run a program written for a drop count titration (see below).

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```
9  SEND INPUT FROM COL-A TO GraphX (0 TO 600)
10 SEND INPUT FROM COL-B TO GraphY (0 TO 14)
11 IF INPUT FROM LastKey = 115.000000 GOTO 14
12 DELAY 500 millisec
13 GOTO 5
14 STOP
```

This experiment will store the drop counter readings and the pH electrode readings in a spreadsheet file. In addition, a run-time graph and text format will also be displayed. This titration is automated, and no user entry is required. The display mode chosen (Figure 6) determines which values will be displayed on the screen for the DOS and Macintosh software.

The experiments referenced for all three titration types can also be modified to include temperature. The reactions that occur during an acid/base titration are either exothermic or endothermic, and a properly calibrated temperature sensor can be used to determine the equivalence point of the titration thermometrically.

Figure 7 shows a typical acid/base titration curve. A strong base (NaOH) was titrated with a strong acid (HCl). The equivalence point is the point at which the pH versus volume curve is the steepest. The spreadsheet makes it easy to find the equivalence point of a titration curve. Since the equivalence point is the steepest point of the curve, taking the derivative of the curve will make the equivalence point readily apparent. The derivative curve can be plotted on the same graph as the titration curve. Spreadsheet formulas can also be used to convert the elapsed time (timed titration), or the drop count (drop count titration) to volume corresponding to the calibration information obtained using the calibration experiments.

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They are resistant to acids and mineral acids. The Delrin® housing offers good resistance to weak acids and bases, as well as organic solvents. Strong acids and bases outside the pH range of 4-9 will attack the housing and care should be taken to avoid spilling or splashing strong acid or base titrants on the housing. If you do get titrant on the housing, wipe it off as quickly as possible to avoid damage.

pH Electrode

SCI Technologies uses two sources for its pH electrodes: Omega® and Analytical Sensors, Inc.

Omega® pH electrode

The Omega® pH electrode is an epoxy body, rugged, general purpose, gel-filled, combination electrode. Insertion length is 110 mm, and probe diameter is 12 mm. pH measurements in the range of 0-12 can be measured. (Solutions with pH greater than 12 require a special type of glass in the electrode. Contact SCI for more information.) The reference type is Ag/AgCl with a dual-pin ceramic junction, and the probe impedance at 25°C is 80 Megohms. The reference is sealed and cannot be replaced. A three-foot cable with a BNC connector is used to connect the probe to the LabWorks™ interfaces.

Analytical Sensors, Inc. pH electrode

The Analytical Sensors pH electrode is a plastic body, rugged, general purpose, gel-filled, combination electrode. Overall probe length is 150 mm, and probe diameter is 12 mm. pH measurements in the range of 0 to 14 can be measured. The reference type is Ag/AgCl with a dual pin fiber junction This electrode has a replaceable reference junction (reference junction material is supplied). When the pH readings become slow and readings drift, the user can install a new reference junction. This will immediately restore electrode performance. To replace the

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and become unstable. High humidity environments will also cause this problem. Probes should also be placed in the solution so as not to interfere with proper mixing (especially when performing a titration). The solution being measured should be stirred gently to avoid causing turbulence. If the probe is in a dead spot or a spot that is too turbulent (causing air bubbles), then erroneous readings will occur. A clean probe is also essential to obtaining proper pH measurements. Even in clean solutions, probes can become coated with contaminants. If you suspect you have a fouled electrode (unstable or drifting readings), lightly scrub the bulb at the end of the electrode with warm soapy water. The glass bulb is VERY fragile, so care must be taken to prevent damage. Rinse the probe with distilled water after cleaning and return to soaker solution for at least an hour before using. If you still do not get stable readings, it is possible that the circuitry inside the interface is causing the error. Contaminants, such as dust or humidity are the biggest causes. If you suspect the interface is causing the erroneous readings, unplug it, pour a small amount of isopropyl alcohol into the BNC connector, and let the interface sit so that the alcohol drains into the enclosure. This will help to clean any contaminants and the evaporation of the alcohol will aid in drying. **MAKE SURE THE INTERFACE IS UNPLUGGED FROM AC POWER BEFORE CLEANING WITH ALCOHOL.** Allow the interface to sit unused for at least 24 hours after cleaning. Care should also be taken to avoid getting the alcohol on the interface front panel and enclosure. If you do get alcohol on the interface, gently blot with a paper towel so as not to smear the paint on the front panel.

Finally, the proximity to AC power devices and fluorescent lighting can affect the signal in the cable of the pH electrode and cause unstable readings. Keep the cable as far away from AC power lines as possible. If the cable crosses an AC power line, make sure it crosses at a 90° angle. If you have to run the cable parallel to AC lines at a close distance, or have to use the electrode in close proximity to fluorescent lighting, you may have to run the cable through a conduit to prevent AC noise from giving you unstable readings.

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- Digital outputs
  - 2 TTL controlled transistor collector outputs
    5V switched on or off
- 8 TTL control outputs

LabWorks II®-100 Interface

- Analog Inputs
  - 3 auto-ranging current inputs
    ±2500µA, 1.22µA resolution
    ±250µA, 0.122µA resolution
    ±25µA, 0.0122µA resolution
    ±2.5µA, 1.22nA resolution
    ±0.25µA, 0.122nA resolution
  - 3 auto-ranging voltage inputs
    ±25V, 12.2mV resolution
    ±2.5V, 1.22mV resolution
    ±250mV, 0.122mV resolution
    ±25mV, 12.2µV resolution
    ±0.25mV, 1.22µV resolution
  - 1 pH/mV input
    ±2500mV, 1.22mV (0.02 pH) resolution
- Digital inputs
  - 3 TTL compatible digital inputs
  - 2 photogate inputs
    5V-power output and input for TTL level signals
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    5V-power output and input for TTL level pulses up to 20 MHz rate
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