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The experiments described in these materials are potentially hazardous and require a high level of safety training, special facilities and equipment, and supervision by appropriate individuals. You bear the sole responsibility, liability, and risk for the implementation of such safety procedures and measures. MIT shall have no responsibility, liability, or risk for the content or implementation of any of the material presented.

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Appendix 1

OPERATION OF THE ACCUMET® PORTABLE AP61 PH METER.

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The Electrode

The **Accumet®** combination electrode is responsive over a pH range of 0 to 14. It consists of a measuring electrode, a reference electrode, and an automatic temperature compensated (ATC) unit that are built into a single housing. The measuring electrode is located in the center of the probe and the Ag/AgCl reference electrode and the ceramic junction are in the outer annular compartment. The outer space of the housing is filled with 4M KCl that is saturated with AgCl.

- **The plastic cot (encap seal) must be placed over the probe tip when the electrode is not in use.** This seal keeps the membrane moist and ready for use.
- **A slight scratch on the glass bulb can render the electrode useless. Exercise care when handling the electrode.**
- Wet the cotton inside the plastic cot with **pH 4 buffer**.
- **Never store the electrode in distilled water!**
- **The fill hole, located on the cap ring, must be closed.**

Figure removed due to copyright reasons.

Fig. 1. The schematics of an ATC Combination Electrode. However, the electrode that is connected to the pHmeter that you are working with is a 13-650-AP50. It is a pH/ATC single-junction model, with a polypropylene body (built-in protection) that encases a glass pH membrane in its tip.

OPERATION:

Before you start the standardization and pH measurements, please read the following useful hints:

- **Rotate the fill hole to OPEN.**
- **To obtain accurate readings, immerse the pH sensing bulb and the reference junction on the electrode completely. For accurate temperature**

- compensation always immerse the electrode at least 2" into sample (the internal temperature sensor is located 1 inch above the probe end).
- Rinse the electrode with distilled water between samples. Wipe the electrode with a KimWipe.



Fig. 2. The portable AP62 pH meter

Key pad:

- **Mode:** If your meter is not in pH mode, press this button until the meter in this mode.
- **Std:** initiates standardization for the current measurements mode.
- **Setup:** Clears existing buffers, selects measurement resolution (x.x or x.xx). Press to scroll through.
- **Enter:** press to accept an option displayed in **setup**.
- **Auto:** eliminates the need for the operator to subjectively judge electrode stability and terminate measurements. Also, **auto** locks the measurements onto the screen when stable.

- **On/off:** Press to turn meter on or off. When turned on the meter resumes all operations in the same mode it was when it was turned off.

Standardization:

Allow the electrode to soak in a buffer for 10 minutes prior to standardization!

1. The meter must be in pH mode. (Press **Mode** until the display indicates pH mode.)
2. Immerse the rinsed electrode into buffer pH=4. Stir moderately if possible.
3. Press **Std**. The prompt **press std to standardize** will flash.
4. Press **Std** to initiate standardization. The meter will automatically recognize the buffer used and display the value on the screen. Standardize will flash until the buffer is accepted and the meter returns to the **Measure** screen.
5. Repeat steps 2-4 with the second buffer, pH=10.
6. When the meter accepts the second buffer, it will briefly display the efficiency (as percent slope) of the electrode. If percent slope is outside the range of 90-102, the meter will display **ELECTRODE ERROR**. Re-do the standardization.

Measurement:

1. Set up the titration apparatus as shown in figure 3.
2. Check that the meter is in pH mode. In a 450-500-mL beaker, while stirring with a 1" magnetic bar, dissolve the unknown carboxylic acid in 150-200 mL of distilled water.
3. Immerse the electrode into the sample solution.
4. When the reading is stable, **STABLE** will appear under the measurement. Write down the starting volume of NaOH. At the beginning, add 0.5X mL from the 0.1XXX M solution from the buret. When the difference between two consecutive pH readings is about 0.3, decrease the volume added to about 0.2X mL. When the titration enters the steep part of the curve, add only 0.1X mL increments. As the pH change between additions decrease, the added volume may be again increased to 0.2X mL and 0.5X mL. When you reach pH=11.4 stop the titration.
5. Determine the $V_{\text{equivalent}}$ and K_a (pK_a) by calculating the second derivative of titration curve or/and from preparing a Gran's plot. Details regarding these procedures will be provided during the UACA lecture #2.

NOTE: pH meters may be checked out for the day from the stockroom.



Fig. 3. Titration apparatus: 400-500-mL beaker, 1" stir bar, 25-mL buret, magnetic stirrer, cork, electrode connected to Accumet AP61 pH meter.