EXPERIMENT VIII

DISSOCIATION OF WEAK ACIDS (S&G Expt. 17)

1. Purpose

Determination of the dissociation constants of acetic acid and monochloroacetic acid at 25 C, from conductivity measurements.

2. Pre-Lab preparation

Work out in advance the details of the standardization of acetic and monochloroacetic acid (see below). In particular, for each of the acid decide on the volume to use for the titration. Also, knowing that ≈ 50 ml are needed for a measurement, work out how (and how much) to prepare the required series of dilutions.

3. Safety

The chemicals used in this experiment can be corrosive; please wear your safety goggles at all times. A lab coat is recommended.

4. Standardization of solutions

Good results for this experiment depend critically on an accurate knowledge of the solution concentrations.

To prepare the solutions, use Ultra-pure water available from the Millipore™ apparatus found in the lab; regular distilled water may not give quite accurate results.

- *0.037 M KCl*. To check the calibration of the conductivity meter, you will have to prepare precisely by weight in a 100 ml volumetric flask your own 0.037 M KCl standard solution (conductivity 5.000 mS cm-1 at 25 C). For this purpose, dry KCl is kept in one of the desiccators in the lab.

- *Acid solutions*. You will also have to prepare your own acid stock solutions, 1) 250 ml of ≈ 0.1 M acetic acid by appropriate dilution of concentrated (so-called "glacial") acetic acid which is ≈ 17.2 M, and 2) 250 ml of ≈ 0.05 M monochloroacetic acid by weighing the appropriate amount of crystals of monochloroacetic acid; both glacial acetic acid and crystals of monochloroacetic acid are available in the lab. These prepared stock solutions must be standardized by titration against standard 0.1 M NaOH using phenolphtalein as end-point indicator. Then for each acid, a series of three other dilutions (1/4, 1/16, 1/64) are prepared by serial dilution as suggested in S&G (obtained by pipetting 25 ml of each previous solution into a 100 ml volumetric flask, which is filled to the mark with ultra-pure water).

5. Apparatus and measurements

Instead of the AC conductivity bridge described in S&G, a calibrated conductivity meter is used which reads directly the conductivity of the solution (instead of the resistance from which the conductivity would be deduced knowing the "cell constant").

HANNA© conductivity meter model HI 4321

This commercial instrument uses a 4-electrodes conductivity probe which, once calibrated, reports directly the conductivity of the solution being tested. Fig. 1 shows the functions available on the front panel of the instrument. The measuring cell is attached to a circulating bath thermostated at 25C.

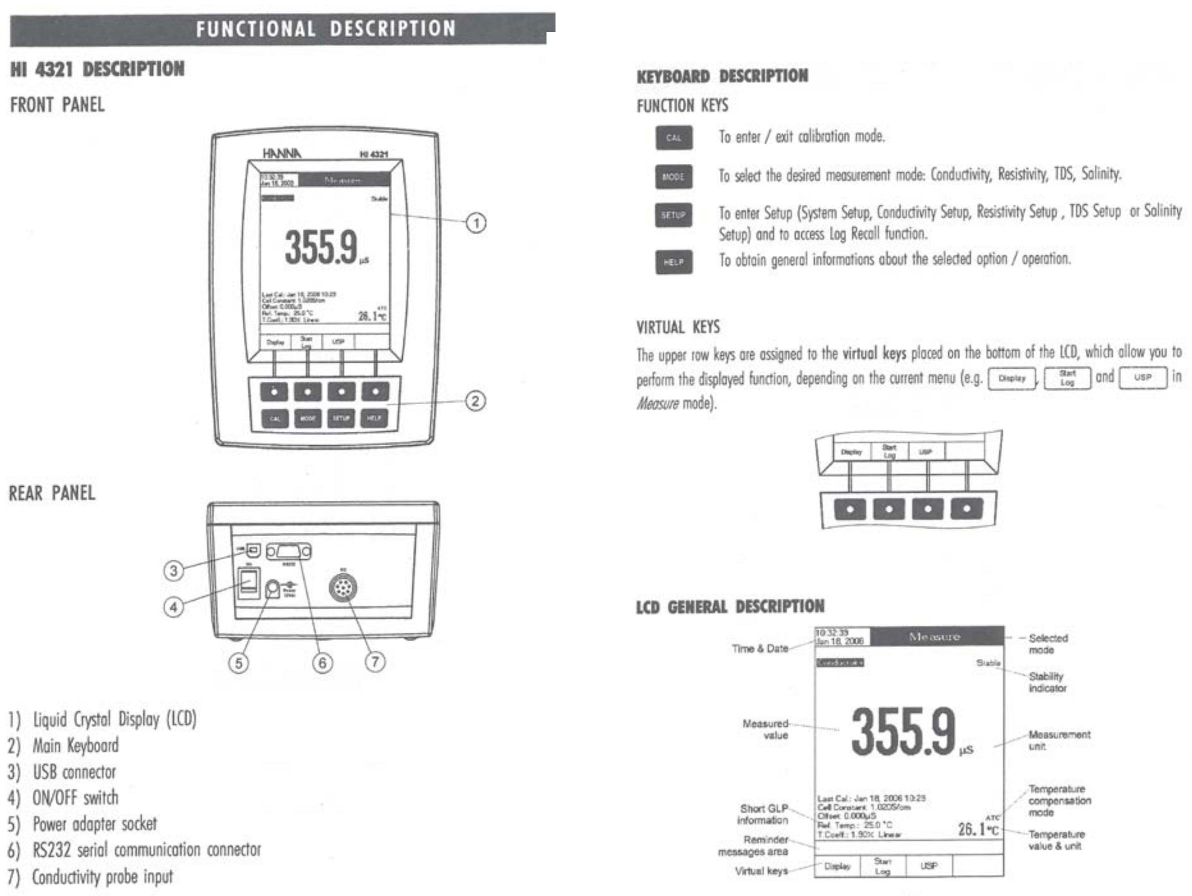

Figure 1. Front panel and keyboard function of HANNA HI 4321 conductivity meter.

- *Turn the instrument ON*; the instrument may go into an initialization routine for a few minutes.

- *Equilibrate the solutions to 25 C.* All solutions to be tested must be pre-equilibrated at 25 C; the flask containing the solution can be clamped in the temperature bath for at least 10 min before using it.
- *Check the calibration:* 1) Take the probe out of the storage solution, dry it with some tissue and let it stand in the air; the reading on the meter should be close to zero. If it not reading zero, go to the Cond. Calibration window, ie, CAL --> wait --> (the previous calibration may have to be erased) Accept to set the offset to zero --> ESC to go back to Measure window. The meter should now read zero. 2) Pour into the cell the 0.037 M KCl solution. Using a nalgene disposable pipette, rinse the probe with small amounts of your KCl solution, insert the probe (*important: the probe has to be immersed up to its black ring*), and verify that the temperature indicated by the meter is 25 C (± 0.5 C); if not, wait until the temperature is within this range before proceeding. 3) Go to CAL --> the instrument should recognize the 5.000 mS cm^{-1} standard solution --> Accept, then ESC back to the Measure window. The meter should read 5.000 mS cm^{-1}; the instrument is now calibrated. 4) Record the values of the cell constant and of the offset which are showing on the screen.
- *Check the conductivity* of the water used to prepare the solutions: 1) Discard the previous sample and rinse the cell with distilled water (DW), then dry the cell with some tissue. 2) Pour a sample of the water used to prepare the solutions into the cell, insert and tap gently the probe (to dislodge potential air bubbles), then take a reading within 10s; the reading will drift as the water takes up CO_2 from the air; the conductivity should be not higher than ~2.5 μS cm^{-1}.
- *Measurement.* Start measurements with the most dilute acid solution. 1) Discard the previous sample, rinse with DW then dry the cell with some tissue, 2) pour the solution into the cell (at least cell 3/4 full). 3) Before inserting the probe, rinse it with several small amounts of the solution using a nalgene pipette, 4) insert the probe, tap it gently, wait until the meter beeps and displays the "Stable" enunciator before recording the reading. You may want to obtain several readings; eventually record the minimum and the maximum values.
- *Storage of the conductivity probe.* Once all the measurements have been performed, rinse the probe abundantly with distilled water, then store the probe immersed in distilled water in the measuring cell; wrap a piece of *Parafilm* around the opening to prevent evaporation on the water.

6. Calculations and discussion

In the case of monochloroacetic acid, the relevant values for the equivalent conductance at infinite dilution are not provided in the text book and are to be obtained from other sources.

Follow the points of discussion raised in the text book. About the probe used in this experiment, what is(are) the reason(s) for using a 4 electrode arrangement? What other electrode (simpler) arrangement is possible in principle?

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Chem 366W report check list

A report will not be accepted without all the items of this list checked. If a checked item is found missing in the report, the report will be automatically down-graded.

Student Name:____

Report: Dissociation of Weak Acids by Conductivity

Title page.
Correct title of the experiment .... ☐
Student Name & student ID .... ☐
Partner name (*if applicable*)____
Date of performance of experiment .... ☐
Abstract.. ☐
Introduction and theory .... ☐
Experimental
Changes from text description mentioned (*if applicable*)..................................... ☐
Sample ID, ser no, stock solution …etc recorded (*if applicable*) .... ☐
Results
Results as Tables .... ☐
Graphs
Size, at least ½ page .... ☐
Axis labelled .... ☐
Axis labels have units... ☐
Axis scales are sensible .... ☐
Only significant figures .... ☐
Uncertainties quoted... ☐
Raw data provided (*electronic form, if applicable*) .... ☐
Calculations
Sample calculation provided.. ☐
Error analysis .... ☐
Sample error calculation provided .... ☐
Discussion
Comments on results.. ☐
Questions in text book and in manual answered .... ☐
Comparison with literature value(s) .... ☐
Conclusion.. ☐

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