THE PREPARATION AND STANDARDIZATION OF A 0.1M HCl SOLUTION

The purpose of this experiment is to prepare and standardize a 0.1 M solution of hydrochloric acid, 0.1 M HCl. Once standardized, this hydrochloric acid will be saved and used in additional experiments as directed by your instructor.

The simplest method of calculating the volume of a more concentrated solution needed to prepare a diluted solution is by using the solution dilution equation:

$$\text{Vol. (conc.)} \times \text{M (conc.)} = \text{Vol. (dil.)} \times \text{M (dil.)}$$

For example, if you instructor asked you to prepare 250 ml of 0.2 M hydrochloric acid from concentrated hydrochloric acid, 12 M HCl, then you would measure 4.2 ml of 12 M HCl and dilute to 250 ml. Note the following calculation:

$$\text{Vol. (conc.)} \times \text{M (conc.)} = \text{Vol. (dil.)} \times \text{M (dil.)}$$
$$x \text{ ml HCl} \times 12 \text{ M HCl} = 250 \text{ ml} \times 0.2 \text{ M}$$
$$12\,x = 250 \times 0.2 = 50$$
$$x = 4.2 \text{ ml}$$

Once the approximate solution is prepared, it must be standardized. While there are a number of methods and reagents available to standardize an acid, we are going to employ a redox reaction utilizing potassium iodate. Potassium iodate is an ideal substance for standardization as it may be obtained in a pure form, does not decompose readily, and may be used directly without any additional purification steps.

The chemistry of the iodate - iodide in-hydrogen ion is unique in acid-base chemistry as it is a redox reaction, not an acid - base reaction. Note the overall equation:

$$IO_3^-(aq) + 6H^+(aq) + 5I^-(aq) = 3I_2(s) + 3H_2O(R)$$

As the iodate ion reacts with the iodide ion, it consumed hydrogen ions. By measuring the amount of iodate ion needed to consume all the hydrogen ion in a solution, we can determine the number of moles of hydrogen ion reacted and since we measured the volume of the acid solution, the number of moles per liter or concentration.

Here's how it works. In an acid solution, the iodine in the iodate ion is reduced from a +5 oxidation state to 0 by gaining electrons.

$$2IO_3^-(aq) + 12H^+(aq) + 10e^- = I_2(s) + 6H_2O(R) \qquad E^0 = 1.229 \text{ volts}$$

Simultaneously, the iodine in the iodide ion gains an electron as it is oxidized from -1 to 0.

$$I_2(aq) + 2e^- = 2I^-(aq) \qquad E^0 = 0.621 \text{ volts}$$

But this process requires hydrogen, six moles for each mole of iodate reacted. It is this hydrogen that we are going to determine as it combines with the oxygen in the iodate ion forming water.

However, the elemental iodine produced by the reaction forms a yellow-brown solution which will obscure the orange end point of the reaction. The bromocresol green acid-base indicator changes from blue to orange when all the iodate is consumed and the pH drops. The counter measure is to add sodium thiosulfate. Sodium thiosulfate reacts with the elemental iodine liberated oxidizing it back to the iodide ion, while it is being converted to the dithionite ion, $S_4O_6^{2-}$. The plus in the technique is that the increased iodide ion concentration helps speed up the reaction. Note the reaction below:

$$2S_2O_3^{2-}(aq) + I_2(aq) \rightarrow S_4O_6^{2-}(aq) + 2I^-(aq)$$

Now let's look at the math. In an experiment similar to your experiment, a student reacted 1.00 ml of 0.0267 M KIO_3 with 0.84 ml of the approximate 0.2 M HCl solution prepared above. Given that information, lets calculate the actual concentration of diluted HCl solution.

How many millimoles, mmol, of potassium iodate were reacted with the HCl? Since the number of millimoles, mmol, equals volume the of solution in milliliters x molarity of the solution, then the number on millimoles of KIO_3 = 1.00 ml x 0.0267 M = 0.0267 mmol KIO_3.

$$(1 \text{ mole})\ IO_3^-(aq) + 6\ (\text{moles})\ H^+(aq) + 5I^-(aq) = 3I_2(s) + 3H_2O(l)$$

Next, we know from the balanced equation above that 6 moles of hydrogen ion, $H^+(aq)$, reacts with one mole of iodate, $IO_3^-(aq)$. Therefore, the number of millimoles of hydrogen ion reacted is six times the number of millimoles of iodate, 0.0277 mmol, or 6 x 0.0267 = 0.16 mmole of $H^+(aq)$.

$$0.0267 \text{ mmol } IO_3^- \text{ x } \frac{6 \text{ moles } H^+}{1 \text{ mol } IO_3^-} = 0.16 \text{ mmol } H^+$$

Since we found from the titration that the 0.16 mmol of HCl had a volume of 0.84 ml, the concentration of the hydrochloride acid must be 0.19 M. Where

$$\text{molarity of a solution, } \frac{\text{mole}}{\text{liter}} = \frac{\text{number of moles}}{\text{volume in liters}} = \frac{0.16 \text{ mmol}}{0.84 \text{ ml}} = 0.19 \text{ M}$$

Note: we are using millimoles and milliliter rather than moles and liters to simply our calculation.

Materials:

hydrochloric acid solution	acid/base indicator solution	1 M $Na_2S_2O_3$
graduated cylinder	small beaker or plastic cup	storage bottle
1 ml hypodermic syringe	2- 1 ml graduated pipets	1-ml glass pipet
tip extender for the syringe	0.0133 M KIO_3	pipet pump
1- thin stem pipet	3 M KI	

Procedure:

1. Prepare a 0.1 M hydrochloric acid solution, 0.1 M HCl by diluting the more concentrated solution provided by your instructor. First, you must calculate the volume of the more concentrated solution needed to prepare 50-ml of a 0.1 M HCl solution.

   Once the required volume of a more concentrated hydrochloric acid is determined, measure that volume in a graduated cylinder and add water until the total volume equals 50 milliliters. Mix the solution by pouring the solution back and forth between the graduated cylinder and a beaker a few times to insure that the solution is mixed throughly. Store the solution in a stoppered flask or bottle for further use.

2. If necessary, attach a tip extender to the hypodermic syringe. It is important to rinse and prepare the syringe as directed by your instructor. Record the initial volume of the solution in the syringe to the **nearest 0.01 ml** in the space provided on your data table. Partly fill a polyethylene transfer pipet with bromocresol green/methyl orange solution, the acid-base indicator in this experiment.

4. Add exactly 1.00 ml of 0.0133 M potassium iodate solution, KIO_3, with a 1.00 ml pipet to a small beaker or plastic cup along with1 drop of 3 M potassium iodide solution, KI, 2 drops of 1 M sodium thiosulfate solution, $Na_2S_2O_3$, and one drop of bromocresol green/methyl orange indicator.

5. Carefully add 0.10 M HCl dropwise with constant with swirling until one drop of acid just turns the solution orange. Always wait between drops near the end point as the reaction slows. You may inadvertently add too much acid and miss the endpoint. The solution will turn from blue to orange at the endpoint. When you are satisfied that the titration is complete, record the final volume of the solution in the syringe to the **nearest 0.01 ml** in the space provided on your data table .

5. Refill the syringe with their respective solutions, rinse the beaker throughly with deionized or distilled water, and repeat the experiment as time permits.

6. When you are satisfied with your data, throughly rinse all glassware and the syringes, and the syringe and return them to their storage area. Discard the pipet containing the bromcresol green indicator. Save the standardized hydrochloric acid solution for future experiments if directed to do so by your instructor.

Questions and Calculations:

Q1. (a) For each trial, calculate the number of millimoles of potassium iodate reacted from the volume of KIO_3 add to the beaker and its concentration.

(b) For each trial, determine the number of moles of hydrogen ion consumed from the moles of iodate reacted and the mole ratio in the equation.

(c) For each trial, find the concentration of the hydrochloric acid prepared from the volume of acid titrated and the number of moles of iodate consumed.

(d) Calculate the average concentration of your freshly prepared hydrochloric acid solution.

Q2. If 50 ml of 0.2 M nitric acid is diluted to 250 ml, what would be the resulting concentration of the nitric acid solution?

Q3. (a) 0.75 ml of a unknown nitric solution reacts stoichiometrically with 5.00 ml of 0.02 M potassium iodate solution containing the necessary amount of KI. What is the molarity of the nitric acid solution?

(b) If 0.75 ml of a sulfuric acid solution were used instead of nitric acid, what would be the concentration of the unknown sulfuric solution?

DATA:

	Trial #1	Trial #2	Trial #3	Trial #4
Initial volume of HCl sol'n	ml.	ml.	ml.	ml.
Final volume of HCl sol'n	ml.	ml.	ml.	ml.
Volume of HCl reacted	ml.	ml.	ml.	ml.
Volume of 0.0133 M KIO_3	1.00 ml	1.00 ml	1.00 ml	1.00 ml
Millimoles of KIO_3 reacted	mmol	mmol	mmol	mmol
Millimoles of HCl reacted	mol	mol	mol	mol
Concentration of the HCl solution, in moles/liter	M.	M.	M.	M.
Average concentration of the hydrochloric acid solution				M.

NOTES FOR TEACHERS

1. Preparation of the solutions:
   0.01333 KIO_3 - Dissolve 2.853 grams of reagent KIO_3 in hot distilled or DI water, cool, and dilute to 1-liter.
   3 M KI - Dissolve 125 grams of KI in warm water, cool, and dilute to 250 ml.
   1 M $Na_2S_2O_3$ - Boil 250 ml of distilled or DI for a few minutes to remove dissolved CO_2. Dissolve 87 grams of $Na_2S_2O_3$ in 200 ml of the boiling water, cool, and dilute to 250 ml.
   Bromocresol green/methyl orange indicator - Dissolve 1 gram of the sodium salt of bromocresol green and 0.20 gram of the sodium salt of methyl orange in 100 ml of distilled or DI water.

2. The advantage of using potassium iodate to standardize an acid is that the reagent can be purchased in pure form and the solutions are stable for considerable periods of time. See J. Chem. Ed, **26**, p. 588 (November, 1949) for a discussion of the procedure. Moreover, potassium iodate can be used directly from the bottle without any special preparation.

3. The purpose of boiling the distilled water is to remove dissolved carbon dioxide and raise the pH of the solution. Thiosulfate reacts with hydrogen ions in water producing colloidal sulfur reducing its self life.

4. Any acid base indicator which will yield its acid color at a pH of 3.5 is suitable in this experiment. However, the bromocresol green/methyl orange mixture gives a very sharp blue to orange change at the end point of the titration.

Sample data:

	Trial #1	Trial #2	Trial #3	Trial #4
Initial volume of HCl sol'n	0.00 ml.	0.00 ml.	0.00ml.	0.00 ml.
Final volume of HCl sol'n	0.80 ml.	0.79 ml.	0.80 ml	0.80 ml.
Volume of HCl reacted	0.80 ml.	0.79 ml.	0.80 ml.	0.80 ml.
Volume of 0.0133 M KIO_3	1.00 ml	1.00 ml	1.00 ml	1.00 ml
Millimoles of KIO_3 reacted	0.0133 mmol	0.0133 mmol	0.0133 mmol	0.0133 mmol
Millimoles of HCl reacted	0.080 mol	0.079 mol	0.080 mol	0.080 mol
Concentration of the HCl solution, in moles/liter	0.10 M.	0.10 M.	0.10 M.	0.10 M.
Average concentration of the hydrochloric acid solution				0.10 M.