Titration with low volume of solutions

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Titration is the simplest experiment which is taught in school to measure concentration of a solution using another solution of known concentration and volume. Traditionally, the experiment is carried out by taking a solution of known concentration in a burette and a specific amount of solution of unknown concentration in a conical flask, using a pipette.

For example, if there is an alkali solution in the conical flask and an acid is poured into the flask a reaction is initiated. The alkali with the indicator has a particular colour. As the acid is poured into the flask, the alkali starts getting neutralized by the acid and its pH value begins to drop. When the alkali is just neutralized by acid, the solution in the flask changes colour, indicating complete neutralization of alkali; this is referred to as the end point.

The standard method to do the experiments involves taking up to 10 ml of alkali in a pipette and another 10 ml of acid in a burette. In a typical class strength of 40 students, if all are required to do the experiment, around 500 ml of acid and 500 ml of alkali is required (accounting for some wastage and some repetitions). Of course at school level the acids and alkali used are not concentrated which implies that one only needs few tens of ml of concentrated acid (or alkali) for a single class experiment.

But whatever be the amount, the acids, alkali or salts have to be disposed off appropriately and purchasing of raw materials is a financial load to the teaching institutes. So, is it possible to reduce the quantity of raw materials (consumables) by a factor of 10 or 100?

Yes. Teachers have developed micro-chemistry where instead of ml of chemical only a few tens of μl is used, thereby reducing the usage of chemicals by a factor of 100. However, micro pipettes used in such micro-chemistry experiments can be costly and the tips used cannot be reused without contaminating the subsequent experiments. Thus these costly micro pipettes are very appropriate for research level experiments. But for school level experiments one may, as is shown below, use syringes.

At HBCSE we have developed titration techniques using 1 ml syringes that are available very easily and in-expensively at medical stores. The reduction in use of chemicals is not a factor of 100 but a factor of 10. However, the cost cutting in using a 1 ml syringe in comparison to a pipette or burette is a factor of 100, at the least. A typical cost of glass pipette and burette is Rs 200 -400, micro-pipette is Rs. 2100 (cost of tips not included) whereas, a 1 or 2 ml syringe can cost in the range of 4 to 13 rupees (glass syringes cost is on the higher side). Only caution is that the accompanying needles have to be appropriately disposed off. Several techniques are available to dispose them.

A test experiment was carried out to test the viability of titration using 1 ml glass syringes. When concentrated solutions are to be used, plastic syringes should

be used with caution as they may react with the solution. It is up to the teacher to decide when to use the plastic syringes.

A standard experiment was carried out between strong acid and strong base.

Aim:

Determination of the concentration (strength) of a given sodium hydroxide solution by titrating it against a standard solution of diluted Hydrochloric acid.

Theory:

In the titration of a strong acid with a strong base, the amounts of acid and base become chemically equivalent at the end point and the chemical reaction is called a neutralization reaction. Near the end point there is a sudden change in the pH of the solution. If after end point even a small amount of base/acid is added the solution would become slightly alkaline or acidic respectively. In the titration between Hydrochloric acid (strong acid) and sodium hydroxide (strong base), the following reaction takes place:

$$NaOH + HCl \rightarrow NaCl + H_2O$$

In this titration phenolphthalein (HPh) is used as an indicator.

Two or three drops of phenolphthalein was added to the conical flask

Material required:

A) For Preparation of solution: Sodium Hydroxide pellets, conc. Hydrochloric acid, distilled water, measuring cylinders (10 ml to 1000 ml), weighing pan balance, stirrer, Beaker (small, large).
B) For Titration Method: Burette (25ml), Pipette (10 ml), Burette stand, conical flask, Funnel, Measuring cylinder (10 ml), Phenolphthalein (indicator), Syringe (1ml, 5ml, 10ml).

Procedure:

A) How the solution of NaOH & solution of HCl was prepared.

To make 0.1M NaOH solution, 4.0 g of NaOH (pellets/flakes) were dissolved in distilled water and then additional water was added to make the final solution of $1l$ quantity. The solution was prepared in a $1l$ standard flask. 10 ml of this solution was then transferred to a conical flask for the titration.

To make 0.19 M HCl solution, 16.0 ml of concentrated (37%) HCl was measured in a pipette and diluted in distilled water. Additional water was added to make the final solution as 1 l in quantity (in a standard flask of 1 l). 25 ml of this HCL solution was transferred to a burette.

$V_1 \times 12 = 1000 \times 0.19$ ➔ $V_1 = 190/12 = 16$ ml of concentrated HCl to be mixed in water so that the final volume is 1000 ml with molarity of 0.19 M.

B) Titration of Sodium Hydroxide and Hydrochloric Acid Solution:

Method 1.

i) Clean the burette thoroughly, wash it with distilled water and finally rinse it with 0.19M HCl solution. (Always rinse the burette with the solution, which is to be taken in it). Clamp the burette vertically in a burette stand.

(ii) Fill HCl solution into the burette through a funnel above the zero mark. (As per titration procedure, known concentration solution is always taken into the burette)

(iii) Remove the air trapped, if any, from the nozzle of the burette by running the solution forcefully through the burette's nozzle.

(iv) Remove the funnel before noting the initial reading on the burette. Also, while noting the reading, see that no drop of the liquid is hanging at the nozzle of the burette.

(v) Note the initial reading by keeping the eye exactly at the lower meniscus of the solution.

(vi) Pipette out 10 mL of sodium hydroxide solution in a clean and dry conical flask. Always use clean and dry pipette which is rinse with the liquid to be measured before pipetting out the liquid. (same procedure is also doing by 10 ml measuring flask.)

(vii) Add 2-3 drops of phenolphthalein indicator to the conical flask, the solution turns pink. Titrate the sodium hydroxide solution with acid till the pink colour of the solution becomes colourless.

(viii) Read the lower meniscus of the solution in the burette again and record it as final reading. ***(Always read the lower meniscus for all transparent solutions and upper meniscus for coloured solutions)***

(ix) Repeat the procedure until three concordant readings are obtained.

Method 1 : Titration by standard method

	Trial 1	**Trial 2**	**Trial 3**
Volume of NaOH solution titrated	10 ml	10 ml	10 ml
Volume of HCl solution added	4.2 ml	4.1 ml	4.2 ml

Method 2 : Titration of sodium hydroxide and hydrochloric acid solution by 1 ml syringe method (least count of 1 ml syringe is 0.01 ml):

The same titration is being tried out with 1 ml syringes. The rinsing and cleaning process of syringe is same as that of burette or pipette.

Image of syringes used, can be seen at the end of this article.

i) Take two 1ml syringes(plastic) which are clean and dry.

ii) Fill hydrochloric acid solution into the first syringe up to the 1 ml mark. (As per titration procedure, known conc. solution is taken into the first syringe)

iii) Remove air bubbles, if any.

iv) Transfer 1 ml of sodium hydroxide solution into clean and dry small beaker, using another 1 ml syringe. (Instead of small beaker use transparent small plastic round container)

v) Add one drop of phenolphthalein indicator to the small beaker, and ensure that the solution turns pink. If required another drop of phenolphthalein should be added.

Titrate the sodium hydroxide solution with acid (drop by drop) till pink colour of solution changes to colorless. Note the reading on the syringe and determine the volume required to reach the end point.

vi) Repeat the procedure until three concordant readings are obtained.

Result of the experiment carried out.

Method 2 : Titration by Injection syringe(1 ml) method:

	Trial 1	**Trial 2**	**Trial 3**
Volume of NaOH solution titrated	1 ml	1 ml	1 ml
Volume of HCl solution added	0.41 ml	0.4 ml	0.41 ml

Result

From the two data tables, it is very clear that the experiment can be carried out using 10 times smaller quantity of acid and base solutions.

Pipette and burettes are made of thin glass walls and hence vulnerable to breakage by school children, whereas the possibilities of breakage of syringes is smaller owing to the design of the syringes.