Classroom

In this section of *Resonance,* we invite readers to pose questions likely to be raised in a classroom situation. We may suggest strategies for dealing with them, or invite responses, or both. "Classroom" is equally a forum for raising broader issues and sharing personal experiences and viewpoints on matters related to teaching and learning science.

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An Easy, Rapid and Cost-effective Method of Microtitration

An economical and less time consuming microtitration method is presented. The method has been applied to two types of titration, viz., neutralization and complexometric. The results obtained by the new method are comparable to those obtained by conventional method using burette and pipette. It is proposed that this method can be implemented in high school and undergraduate classes as the quantities of chemicals consumed are far less and the experiment can be completed within the given laboratory period.

Introduction

The risk of drawing the analyte solution into the mouth while pipetting in conventional titrimetric methods is very high. This has given rise to alternative methods, such as the two-burette method [1] and titration by drop counting [2]. The method described here is another alternative one, where the apparatus of conventional titration are replaced by weighing bottle, syringes and a standard digital balance. The quantity of reagent used for titration is drastically reduced ($\sim$20 to 50 times as that required

in conventional titration), and the minimum volume of analyte solution used for acid-base or complexometric titration is as low as 0.2 ml. The method is risk-free and economical.

The Method

Acid-base titration: Strong acid HCl has been titrated against strong base NaOH. The density ρ_2 of sodium hydroxide solution is first determined with the help of a specific gravity bottle. A small known volume of hydrochloric acid solution (V_1) is taken in a weighing bottle with the help of a syringe. The weight W_1 of the acid solution is determined by a standard digital balance. A drop of phenolphthalein solution is added and the weight W_2 is taken. Now standard sodium hydroxide solution is added dropwise through a dropper with continuous shaking till the end point (colorless to pink) is reached and the final weight W_3 is noted. The weight of NaOH solution required is, $W_4 = W_3 - W_2$. A known volume of acid solution is added once again to the same solution in the weighing bottle, and the pink color disappears. This weight is noted down. Then NaOH solution is added dropwise till the pink color reappears. The weight of the resultant solution is determined. This process is repeated a few times. The weight of the titrated acid solution and the corresponding weight of NaOH solution required each time is determined (see *Table* 1).

Complexometric titration: The titration of Mg^{2+} by EDTA (disodium salt of ethylene diamine tetra-acetic acid) has been carried out by this method. The density ρ_2 of the EDTA solution of known strength is determined using a specific gravity bottle. A known volume of magnesium sulfate solution (V_1) is taken in a weighing bottle with the help of a syringe. Previously prepared mixture of water (20 ml), buffer of pH = 10 (0.5 ml) and Eriochrome Black T indicator (1 drop) is taken in a separate syringe (approximately 3.5 times V_1) and added to it. The weight of the above mixture W_1 is noted. EDTA solution is added through a syringe till the end point (red to blue) is reached and the total weight W_2 is noted. The weight of the EDTA solution

The quantity of reagent used for titration is drastically reduced (~20 to 50 times as that required in conventional titration), and the minimum volume of analyte solution used for acid-base or complexometric titration is as low as 0.2 ml. The method is risk-free and economical.

Indicator: Phenolphthalein, End point: colorless to pink					
Equation: NaOH + HCl = NaCl + H_2O					

No. of observation	1	2	3	4	5
Volume of HCl solution (V_1, ml)	0.2	0.4	0.6	0.8	1.0
Weight of HCl solution (W_1, g)	0.201	0.404	0.598	0.802	1.004
Weight of NaOH solution required for neutralization (W_4, g)	0.176	0.355	0.529	0.701	0.884
Volume of NaOH solution required for neutralization ($V_2 = W_4/\rho_2$), ml	0.175	0.354	0.527	0.698	0.880
Molarity of HCl solution $M_1 = (M_2 \times V_2)/V_1$	0.088	0.088	0.088	0.087	0.088

From *Figure* 1 the slope of the line m = 0.8778 = V_2/V_1

Therefore $M_1 = (0.877 \times 80.01)M = 0.088M$

Using conventional titration method where 10 ml of HCl solution is neutralized by 8.8 ml of NaOH solution to yield the end point, $M_1 = (0.1 \times 8.8)/10 = 0.088M$
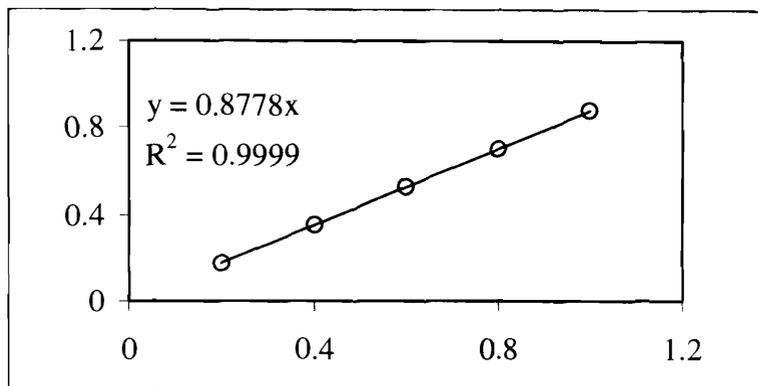
Table 1. Results of titration of HCl (unknown strength) with NaOH having strength $M_2 = 0.1M$ and density $\rho_2 = 1.004$ gcm^{-3}.

required is, $W_3 = W_2 - W_1$. The process is repeated four times with different volumes of magnesium sulfate solution ranging from 0.2 to 1 ml.

Results and Discussion

The method does not involve any conceptual deviation from the principle of strength calculation. For acid base titration the molarity of the unknown acid solution M_1 can be calculated from $M_1 = (M_2 \times V_2 \times n_1)/(n_2 \times V_1)$, where M_2 is the molarity of sodium hydroxide solution, V_2 is the volume of sodium hydroxide solution required for neutralization of $V1$ volume of HCl, and n_1, n_2 are the number of moles of HCl and NaOH reacted ($n_1 = n_2 = 1$). The volume of NaOH solution added is determined from measured weight W_4 and density ρ_2. The results of the experiment and values of $M1$ calculated for each observation is given in Table1. For graphical method of calculation of M_1, *Figure* 1 is drawn showing the plot of volume of acid versus volume of alkali, which is a straight line passing through the origin with

high correlation coefficient ($R^2 = 0.9999$). From the slope of the line and the molarity of known NaOH solution, strength of the acid solution M_1 is determined. Excellent agreement can be observed for M_1 values determined by the present microtitration method and the conventional method of titration (see *Table* 1). In order to estimate Mg^{2+} by complexometric titration, the volume (V_2) of titrant added is calculated from the weight (W_3) and measured value of density of the titrant solution (ρ_2). *Figure* 2 exhibits the plot of V_1 against V_2 representing an equation of the form $y = mx$. The value of the slope of the line ($m = V_2/V_1$) is utilized to estimate the strength of the analyte solution using the equation $M_1 = (m \times M_2 \times n_1)/n_2$, where M_2 represents the strength of titrant, n_1 and n_2 refer respectively to number of moles of Mg^{2+} and EDTA ($n_1 = n_2 = 1$). The graphical method for evaluating the strength of the analyte solution can be used
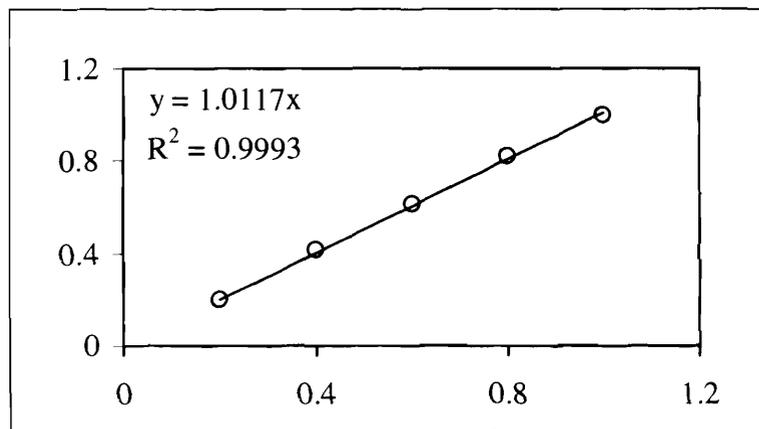
Figure 2. Plot between volume of Mg^{2+} solution (V_1) versus volume of EDTA solution ($V_2 = W_3/p_2$) of microtitration. The molarity of Mg^{2+} solution is given in Table 2.

Indicator: Eriochrome Black T, End point: red to blue

Equation: $Mg^{2+} + H_2Y^{2-} = MgY^{2-} + 2H^+$, where Y^{2-} refers to the anion of disodium salt of ethylene diamine tetra-acetic acid having structure as shown below

HOOCH$_2$C   CH$_2$COOH
    | |
    N-CH$_2$-CH$_2$-N
    | |
$^-$OOCH$_2$C   CH$_2$COO$^-$
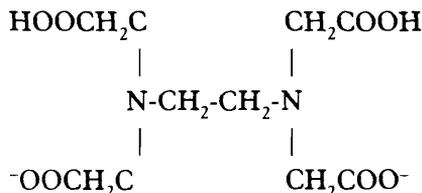
No. of observation	1	2	3	4	5
Volume of Mg^{2+} solution (V_1, ml)	0.2	0.4	0.6	0.8	1.0
Weight of Mg^{2+} solution + other reagents +indicator (W_1, g)	0.860	1.807	2.788	3.880	4.394
W_1 + weight of EDTA solution till end point (W_2, g)	1.057	2.220	3.400	4.702	5.399
Weight of EDTA solution required for titration ($W_3 = W_2 - W_1$), g	0.197	0.413	0.612	0.822	1.005
Volume of EDTA solution required for titration ($V_2 = W_3/\rho_2$), ml	0.196	0.411	0.610	0.819	1.001
Molarity of Mg^{2+} solution, $M_1 = (M_2 \times V_2)/V_1$	0.0098	0.0103	0.0102	0.0102	0.0100

From *Figure* 2 the slope of the line $m = 1.0117 = V_2/V_1$.

Therefore $M_1 = (1.0117 \times 0.01)M = 0.0101M$

Using conventional titration method where 5 ml of Mg^{2+} solution needed 5 ml of EDTA solution to yield the end point, $M_1 = (0.01 \times 5)/5 = 0.01M$

Table 2. Results of titration between epsom salt (MgSO$_4$, 7H$_2$O) solution (strength unknown) and EDTA solution having strength $M_2 = 0.01M$ and density $\rho2 = 1.0037$ gcm^{-3}.

only when the number of observations for different V_1 is three or more. For a single observation, the value of M_1 can be found out using the formula $M_1 = (M_2 \times V_2 \times n_1)/(n_2 \times V_1)$. This has been done in *Table 2*.

Titration by the method presented here has several advantages.

(a) The amounts of reagents used are small. Hence the cost involved is less.

(b) Use of burettes and pipettes is avoided. Thus the storage space required in the laboratory is saved and their breakage is avoided.

(c) The present method is superior to the drop count method2, because the volume of each drop in the latter may not be the same due to possibility of incorporation of small air bubble within the drop. Under such situation the experiment needs to be repeated.

(d) The method is easy and rapid compared to other methods.

(e) The accuracy is unaffected.

(f) It is adoptable to acid-base as well as complexometric titrations.

In conclusion, it is hoped that this method which offers so many advantages will be able to replace other methods in under-graduate and graduate laboratories for titrimetric analyses.

Suggested Reading

[1] Laboratory Manual of University of Wisconsin, La Crosse (cited in reference 2)

[2] Shrinivas L Kelkar, Dilip D Dhavale and Prabodh G Pol, Microscale Experiments in Chemistry The Need of the New Millenium 3. Micro-scale Inorganic Quantitative Analysis and New Methods of Titrations for Introduction at All Levels in Chemistry Laboratories, Resonance, Vol. 6, No 2, pp 14-22, 2001.

Please Note
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Page 43: line 4 from bottom (also highlighted on the page), "the group" should read as "a group"

Page 46: line 7 from top – (K,Φ) should read as (K,ϕ).

Page 51: line 14 from top – 'where A is a abelian' should read as 'where K is a abelian'

Page 52: line 4 from top – Q^{n+1} should read as Q^{n-1}.

Page 53: Author introduction – 'R Srinivasan was the former Director of Centre for Advanced Technology, Indore' should read as 'R Srinivasan was the Director of Inter-University Consortium for DAE Facility, Indore.