

TECHNIQUE OF VOLUMETRIC ANALYSIS

Following are the various steps that need to be observed carefully so as to achieve reasonably correct and reproducible results in the volumetric titrations :

- 1) Conical flasks are considered to be the most suitable vessels meant for volumetric titrations because the mixing can be performed quite rapidly, easily and safely by gently swirling the contents of the flask during the titration,
- 2) Beakers are not usually preferred, but in case they are to be used in volumetric analysis, following *two* provisions may have to be made for stirring :
 - use of a magnetic stirrer with a magnetic guide for the solution, and
 - use of a stirring rod,
- 3) The titration container or vessel must always be kept polished so as to view the end point vividly,
- 4) The solution under titration is normally viewed against a white background *e.g.*, white tile or white paper,
- 5) When the end point is being approached it is always advisable to have the drops of titrant split. It can be accomplished by opening the stopcock of the burette in such a manner that only a fraction of a drop flows out and remains adhered to the tip of the burette. Touch of the liquid against the inside of the flask and wash it down into the main bulk of the liquid with a fine jet of DW (from a wash-bottle),
- 6) In a situation, where the colour-change at the end-point is rather gradual and not abrupt, it is always useful to have a comparison-solution readily available.

Example : Methyl orange offers a gradual end-point. Hence, two flasks containing the same volume of solution having approximately the same composition as the liquid being titrated may be prepared; first, slightly acidic—Red solution, second, slightly basic—Yellow solution.
- 7) In fact, these carefully-prepared comparison solutions would ultimately help in deciding the colour change thereby confirming the actual end-point without any controversy, whatsoever,
- 8) All titrations must be carried out in triplicate and the results of two concurrent readings (*i.e.*, whose difference falls within 0.05 ml-based on a 20 ml titration) may be taken into consideration,
- 9) Remainder solution in the burette, after titrations have been performed must be rejected and should not be put back to the stock-bottle for obvious reasons of contamination. The burette in operation is then washed thoroughly with DW and allowed to drain by placing it up-side down on a burette stand.