TOTAL MICROSCALE ANALYTICAL CHEMISTRY: PRECISION DATA IN VOLUMETRIC TITRATIONS

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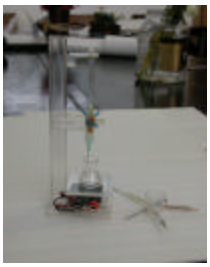
Microscale laboratory has been widely used in General Chemistry mainly in Synthetic Chemistry (inorganic and organic chemistry). Analytical Chemistry approaches just concern to titrimetric determinations with acid-base indicators using 5 mL pipets as burets to teach semi quantitative analysis aspects. Teaching Analytical Chemistry requires to focus in quality aspects such as accuracy and precision. This latter is the most affected when microscale conditions are assayed: "to microtechniques, macroerrors". However if good experimental practices are observed these errors can be controlled and minimized to yield an adequate and good experimental teaching experience.

In this work we show that results obtained with low cost equipment with locally materials are equivalent to those obtained in macroscale conventional conditions respect to precision parameters assayed. Volumetric titrations results are shown monitored by colored chemical indicators or instrumentally (micropotentiometry or conductimetry).

Experimental results

Figure below shows the 1 mL buret used to determined the equivalent point volume when titrating aliquots of 0.01 mol/L HCl with normalized 0.01M NaOH using phenolphthalein as indicator.

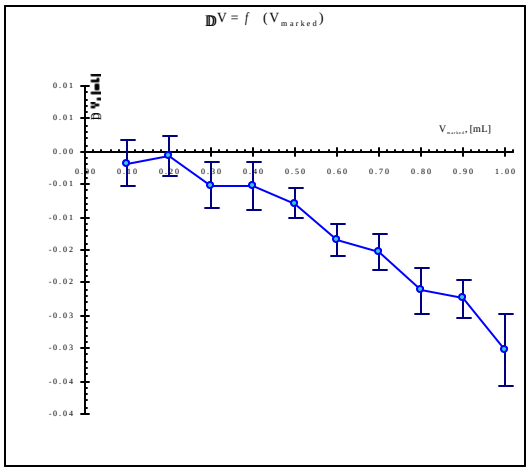


V_{eq},: mean, standard deviation and variation coefficient per cent, (s.d./x)100: VCP are determined.

Results obtained are shown bellow compared with those obtained for two commercial 25 and 10 mL burets[1]:

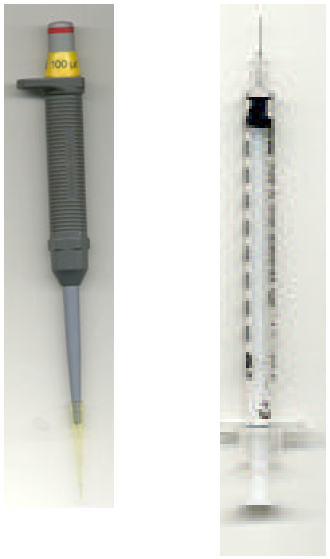
Buret	mean	s.d.	VCP
25 mL			
$V_{eq.}$(mL)	14.72	0.3858	**2.62**
10 mL			
$V_{eq.}$(mL)	5.897	0.1140	**1.94**
1 mL			
$V_{eq.}$(mL)	0.561	0.0058	**1.03**

Additionally a calibration curve was performed to measure the real volume poured by 1 mL microburet (by weighting the poured water in analytical balance) respect to marked volume in the buret body : $\Delta V = (V_{marked}-V_{measured})$ versus the marked volume.
Figure in the next page shows that ΔV is around ± 0.00791 mL for 0.5 mL as equivalent volume (1.6%). For a 50 mL buret a V = 30 mL shows a 0.1% deviation[2].



A redox titration of 0.5 mL 0.1N sodium tiosulphate with $KMnO_4$ 0.1 N was additionally performed . The average V_{eq} found was 0.61 mL± 0.01 (n=17, VCP = 1.99%). Visual indication was used.

In all cases above, insulin syringes were used to deliver 0.5 mL samples to titrate. In order to know the precision associated to these syringes used as pipetting devices, measurements of the weight of water delivered with a 1 mL insuline syringe were performed and compared with those obtained with a commercial automatic pipet. The figure below show the pipets assayed.



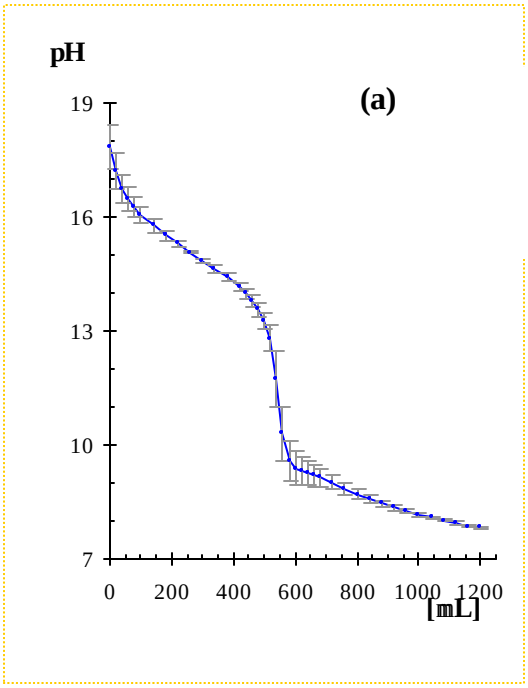
(A) **(B)**

For n= 30 next results were obtained:

	(A)	(B)
mean	**0.0990 mL**	**0.0980 mL**
s.d.	**0.0011**	**0.0008**
VCP	**1.16%**	**0.84%**

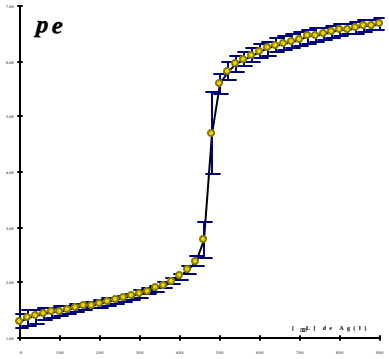
End point monitoring potentiometrically were performed with low cost microelectrodes: W° or PANI as pH sensors in aqueous[3,4] and non aqueous solvents, C° for redox and complexometric titrations and Ag° for halide titrations[5].

Next figure shows typical average potentiometric titrations: (a) pH non aqueous solution titration of 1 mL of 0.2M 2-6 lutidine with 0.4 M methane sulphonic acid with a PANI micro pH sensor in acetonitrile as solvent (n=5):



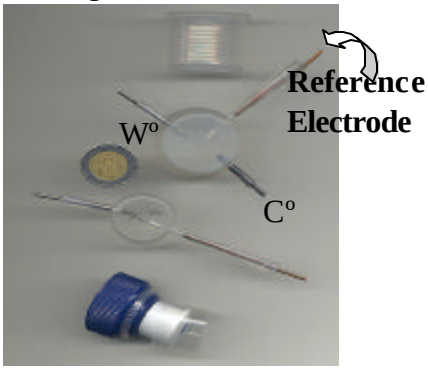
and (b) 0.5 mL of 0.1 M NaCl with 0.1 M $AgNO_3$ with a silver rod microsensor with no salt bridge separation (n=10):

(b)

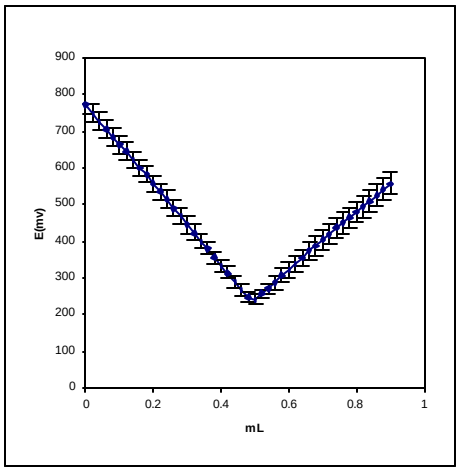


note: ***pe*** = E(mV)/60 (mV)

Next figure shows the microsensors used



Finally a conductimetric experimental set up design in our laboratory is used to determine the equivalence point in acid-base titrations. C and W rods use for potentiometry can be used as conductors connected to a oscillographic interphase to measure the conductivity of the titrated solution. Figures below show the experimental set up used and a typical 0.1M HCl-0.1 M NaOH conductimetric titration average plot (n=10).



Conclusions

Precision in microscale conditions is suitable enough to ensure a good experimental teaching in volumetric analysis.

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