MICROSCALE ANALYTICAL CHEMISTRY EXPERIMENTAL TEACHING WITH LOCALLY PRODUCED LOW- COST INSTRUMENTATION.

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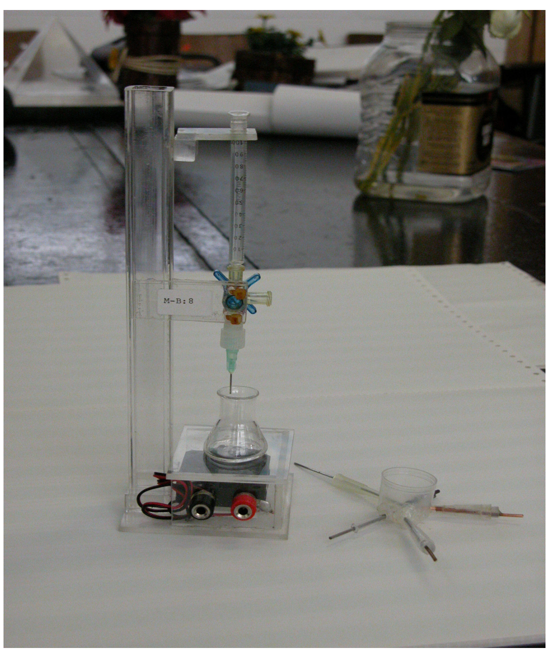
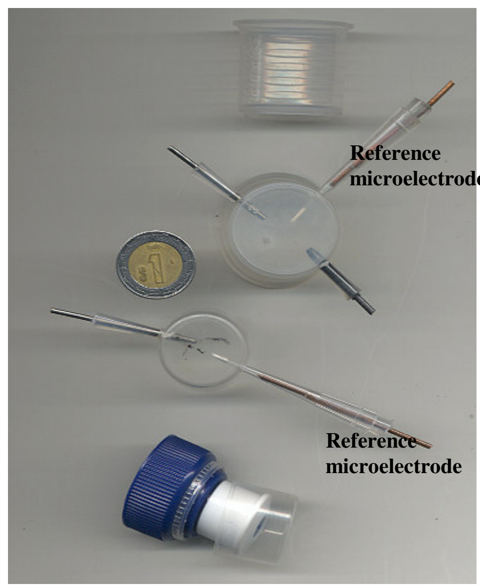
Microscale laboratory has been widely used in General Chemistry mainly in Synthetic Chemistry (inorganic and organic chemistry). Analytical Chemistry approaches just concern to titrimetric determinations with acid-base indicators using 5 mL pipets as burets to teach semi quantitative analysis aspects. Instrumental measurements play a very important role in teaching analytical methodologies. Chemical Instrumentation has been developed mainly for basic or applied research so it is difficult to access to such devices in teaching laboratories. In our laboratory we have developed low cost equipment with locally materials to performe potentiometric, conductimetric, electrochemical and photocolorimetric measurements to teach Instrumental Analytical Chemistry (1-4).

Experimental results

Next figure shows the microburet made with 1 mL insuline syringe connected to a 3 way key both hold in a strong plastic support coupled with a micromagnetic stirrer. A multiple microelectrode cell is shown as well:

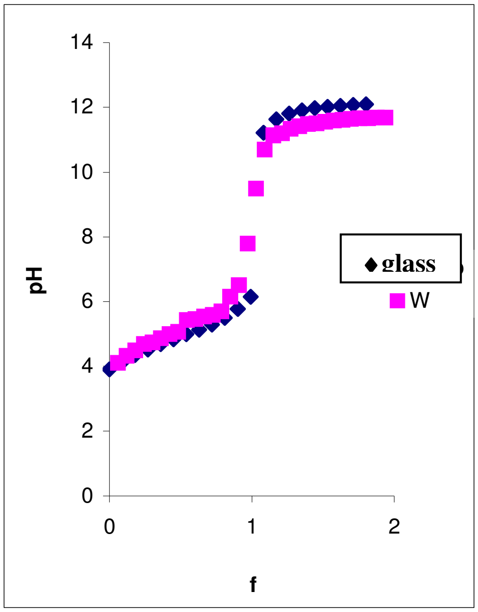


e built with W, C, Ag wires
introduced and fixed into plastic automated pipettes tips. W° wire is used as pH sensor, C rod is useful to perform redox measurements and Ag wire is used for monitoring halide titrations. Microreference electrode is made in a similar way: a Cu rod is introduced in a plastic tip that contains water or a Cu(II) solution. A piece of cotton is used as liquid junction separation. The figure below shows a typical three microelectrodes cell arrangement. Samples of only 500 μL are required. **A 5 mL plastic vessel is used to hold the electrodes:**



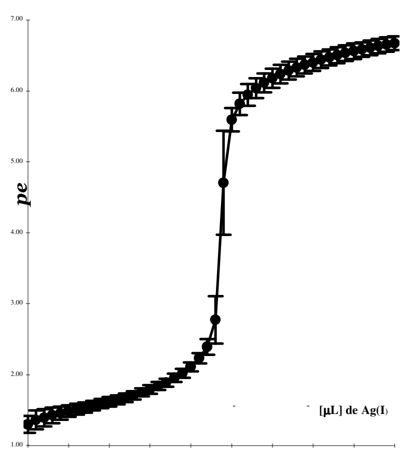
Potentiometric titrations.

The pH determination is made calibrating the potential measured between the W wire and the Cu reference microelectrode with pH=4 and 7 buffer solutions. A low cost voltmeter is used. The arranged is shown in the next page with a typical potassium hydrogen phtalate 0.1 mol/L solution with 0.1 mol/L NaOH titrant. The titration plot obtained with a glass commercial combined electrode is also shown to compare macro and microscale titrations. A 25 mL conventional buret was used for the purpose.

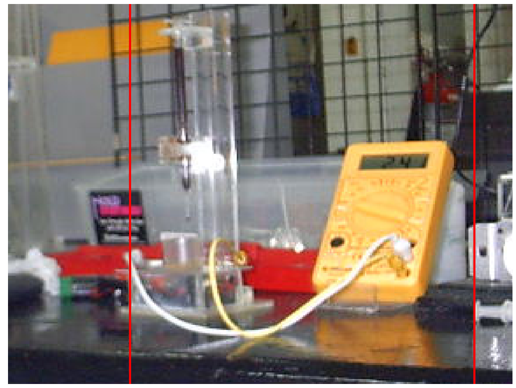


where **f** is equal to the quotient ($v_{added}/v_{equivalence}$) for each case.

Next figure shows an average titration plot of 500 µL of 0.1 mol/L NaCl standar solution with silver nitrate *without salt bridge.*



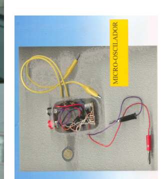
We have performed redox titrations with C microelectrode using iodine and dichromate as titrants and EDTA in complexometic titrations as well. Next picture shows the complete experimental set up for a yodometric titration.



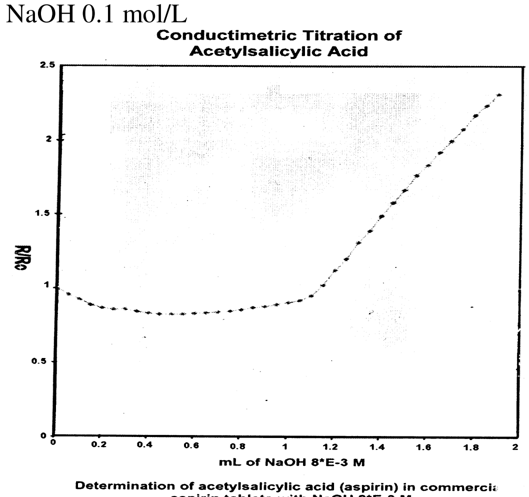
Equivalent results to conventional scale redox titrations are obtained.

Conductimetric titrations

In order to obtained alternate perturbation to two carbon rods used as conductimetric electrodes an electronic interphase designed and built in our laboratory was connected to the same low cost voltmeter used in potentiometry. The pictures below shows an inner view of the interphase and a final experimental set up showing the little blue cage containing the interphase connected to cell and to voltmeter:



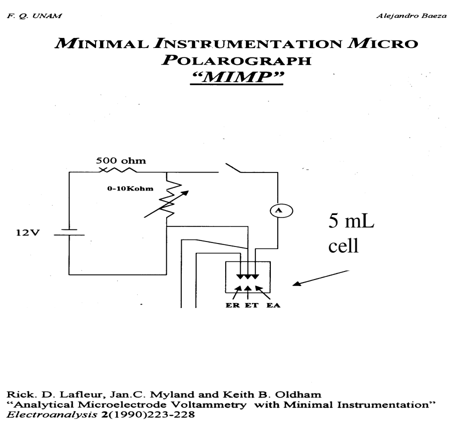
Next plot shows a typical titration of aspirin with NaOH 0.1 mol/L

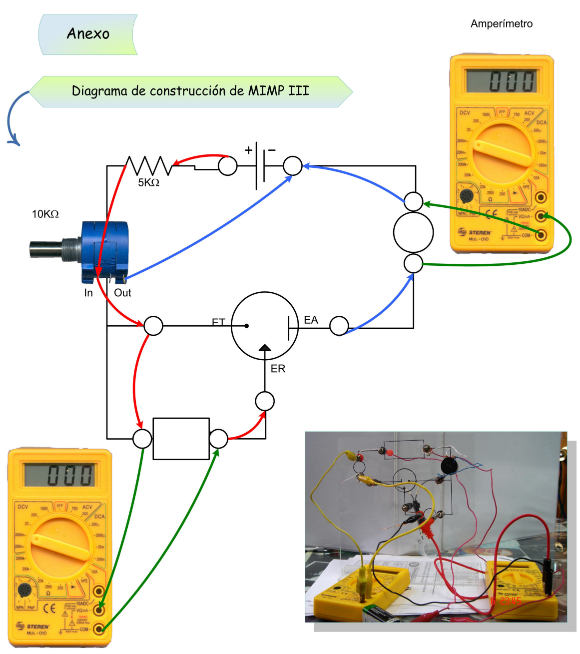


Some other samples have been analyzed successfully with these interphase-voltmeter set up: water quality, tobacco alkaloid non-aqueous titrations, electrolytic dissociation studies.

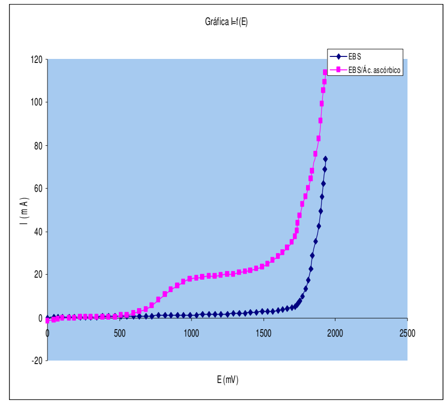
Electrochemical experiments

To perform cronoamperometry with a E imposed we have designed a *Minimal Instrumentation Micro Polarograph* (*MIMP*) which permits use a low DC voltage to be imposed over a microcarbon disc electrode, microcarbon rod as auxiliary electrode and the microreference electrode describe above in non stirring conditions After 1 minute the electrolysis current is measured with a low cost amperometer. Next picture shows the circuit employed:

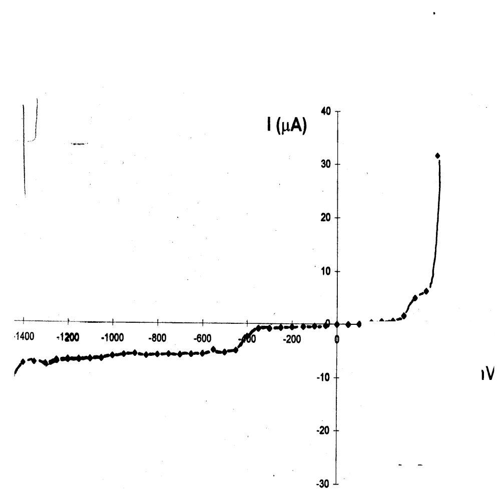




Next figure shows a typical I¨/E plot of the electro-oxidation of a 10 mF ascorbic acid in electrolyte buffer solution, EBS:



DC Polarography es performed using a dropping mercury electrode. Next figure shows the polarographic detection of a 1 mM Pb(II) solution:



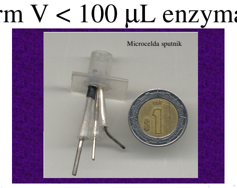
We have used the *MIMP* in determinations of nalidixic acid (antibiotic), vitamins, nitroderivatives an quinones in non aqueous solvents, H_2O_2 produced in glucose oxidase, (GOD) glucose analysis in wines and human serum, Ni and Cd in recycling batteries processes. Next picture shows a complete set up with a cover MIMP and three microelectrodes cell:

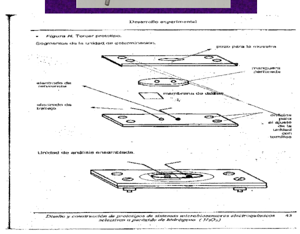
Additionally we have built 1 mL microcoulometric cell to perform acid-base and redox titrations at controlled current

Microbiosensors

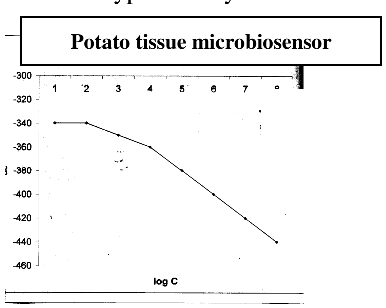
Plastic micropipets tips are used to form a microchamber with W or C transducers to hold enzymatic crude extracts from soybeam flour (urease), potato fresh tissue (peroxidase) and cucumber skin juice (ascorbate dehydrogenase). Next picture shows the micropotentiometric arranged used:

Microcilinders and flat microcells have been used to perform V < 100 µL enzymatic assays:



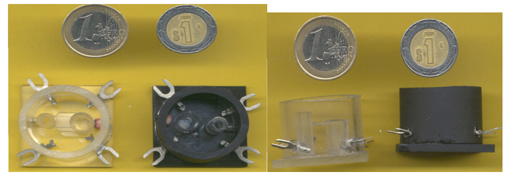


Next figure show a typical enzymatic calibration plot:

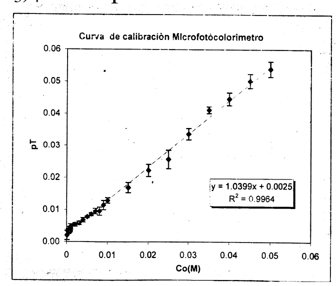


Microphotocolorimetry

Next picture show the *Minimal Instrumentation Micro Colorimeter (MIMC)* that shows: microlamp, filter solution tube, sample´s container (300µL), microphotoresistor detector and connections for 1.5 V and the same multimeter to measure the resistance detector:



We have used this *MIMC* to determine proteins, hemoglobine, Cu, Ni, glucose (by GOD), acetylsalicylic acid and chlorophylls successfully. Next figure shows a typical absorbance, pT- concentración , calibration plot for $Cu(NH_3)_4^{2+}$ complex:



Conclusions

Locally produced low cost equipment for teaching Instrumental Analysis is possible.

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