CONSTRUCTION OF MINIMAL INSTRUMENTATION MICRO-ELECTROCHEMICAL METHODS FOR KARL FISCHER WATER TITRATION

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Abstract

Low-cost and local acquisition micro-scale equipment has been built in order to obtain Karl Fischer Reagent titre, therefore, to quantify in many samples such as low-moisture content foods, organic solvents, and pharmaceutical drugs.
Visual, Photometric, biamperometric and coulometric methods has been used for monitoring water titration and construct calibration plots.
Biamperometry with non-linear adjustment and photometric typical plots are presented to show the dependence on water during the titration.

Micro-Biamperometry

A constant potential was imposed to measure the current resultant by the predominant species during the titration. Figure 1. Electric circuit for biamperometry equipment is presented.
A titre of (5.12 ± 0.64) mg of Water per mL of titrant has been obtained.

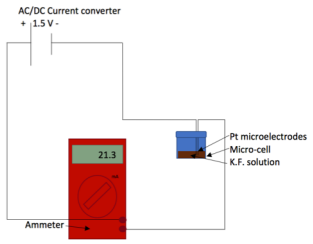

Figure 1. Electric circuit diagram for micro-scale equipment.

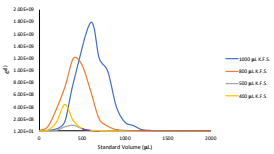

Figure 2. Typical plots of non-linear adjustment biamperometry

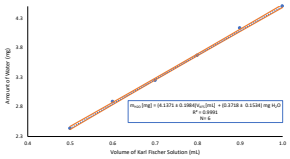

Figure 3. Calibration Plot for biamperometry method

Mico-Photometry

An incised blue light on the reaction cell was used to indirectly measure absorbance produced by an excess of iodine during the titration. The Figure 4. shows a photometry micro-equipment diagram.
A titre of (7.52 ± 4.26) mg of Water per mL of titrant has been obtained.

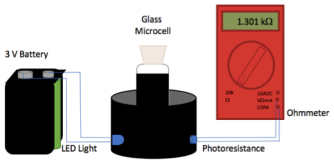

Figure 4. Diagram for phot-ometric detection equipment.

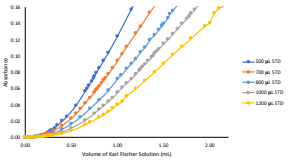

Figure 5. Typical plots of photometry with different aliquots of water standard solution.

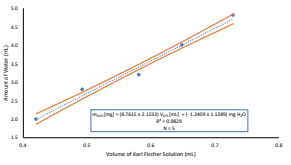

Figure 6. Calibration Plot for photometric detection

Visual Micro-Titration

A solution who equivalency point was prepared to compare with cell titration and measure end point volume of titration and then a calibration plot was gained.
A titre of (4.51 ± 0.35) mg of Water per mL of titrant has been obtained.

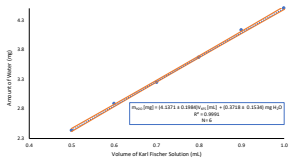

Figure 7. Calibration Plot for Visual detection

Micro-Voltammetry

Voltammograms was realized with a Minimal Instrumentation Micro-Polarograph, a supporting electrolyte of NH_4NO_3 0.5 $molL^{-1}$ in MetOH was used to analyze Karl Fischer Solution.

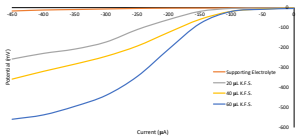

Figure 8. Cathodic Voltammograms of Karl Fischer Solution (K.F.S.) with different aliquots

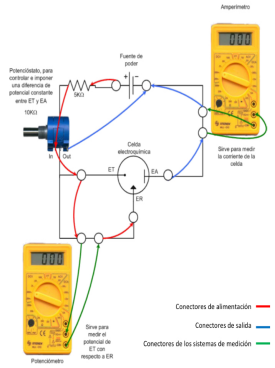

Figure 9. Minimal Instrumentation Micro-Polarograph (MIMP) electric circuit diagram.

Methods Comparison

Table 1. Statistical comparison

Methods	Titre of K.F.S. (mg H_2O/ mL)	$F_{Experimental}$	$F_{Theorical}$	¿Different?
Biamperometry	5.12	1.88	2.87	NO
Photometry	7.52			NO
Visual	4.51			NO
Theoretical	5.00			NO

The Table 1. shows the comparison of micro-scale methods. According to the value of an F obtained by Analysis of Variance with $\alpha = 0.05$, the proposed methods are statistically equivalent to the theoretical value; therefore, micro-scale methods has been demonstrated that are viable for doing analysis.

Table 2. Volumes comparison

Methods	Volume of K.F.S. (mL)	Water Standard (mg/g)	Total Volume (mL)
Conventional	80 - 150	10	180-250
Biamperometry	0.4 - 0.9	4	1.5-3.0
Visual	0.5 - 1.0		1.1-2.2
Photometry	1.6 - 2.5		2.1-3.4

Table 2. Shows the reduction of volumes of reagent and total volume spent during each titration into a conventional method with micro-scale ones.

Membraneless Micro-Coulometric Photodetection of Water

Preliminary experiments of Micro-scale Karl Fischer coulometric titration with photodetection are presented.

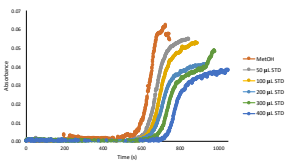

Figure 10. Typical plots of coulometric titration with coupled photodetection.

Conclusion

It is the possibility to realize Karl Fischer water titration through micro-scale methods and depending on the sample, will be the use of a certain method.

References
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