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Article

Coulometric Titration Experimental Practice for Undergraduate Laboratories: A 1960s Twist to a Modern Coulometer

Rafael S. Santos, Thiago R. L. C. da Paixão, and Gabriel N. Meloni*

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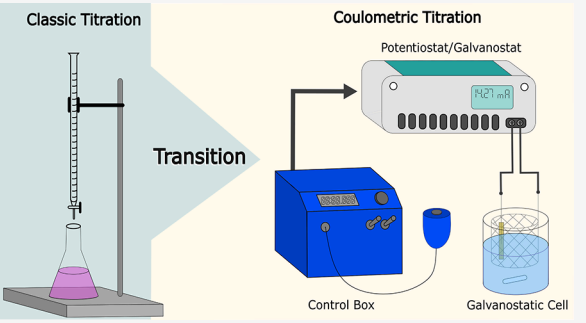
ABSTRACT: When teaching quantitative analytical chemistry to undergraduate students, the transitions from classical analytical methods to instrumental ones can be tough as most (if not all) of the visual/sensorial aspects of the analyses (solution color and volume and mass changes) are lost in the instrumental interface. We have observed that the loss of these aspects can have a significant impact on a student's ability to understand instrumental methods. Coulometric titrations offer a seamless transition path between classical and instrumental methods, offering several parallels between them, especially if visual indicators are used for determining the titration endpoint, and could be used in undergraduate laboratories to facilitate this transition. Unfortunately, modern instrumentation used for coulometric titrations does not offer a hands-on experience for the user, widening the gap between this technique and classical methods and limiting the use to bridge both. Here, we report on the fabrication of a simple and affordable instrumentation that brings back the hands-on interface of a 1960s coulometer to a modern potentiostat/galvanostat and its application to an undergraduate teaching laboratory for the coulometric titration of ascorbic acid using iodine and starch solution as a visual indicator. Molecular absorption spectra are used to quantify the student's increased accuracy in identifying the titration endpoint closer to the equivalency point with successive titrations, demonstrating important didactic aspects of this experimental practice and granting it a place in most chemistry undergraduate curriculums.



KEYWORDS: *Coulometric titration, Undergraduate laboratories, Coulometer, Molecular absorption spectra, Instrumentation*

INTRODUCTION

Coulometric titrations are one of the cornerstone techniques in electroanalytical chemistry and are among the few primary electroanalytical techniques, together with electrogravimetry, which do not require calibration plots or standardization of the titrant solution with a primary standard, as the titrant is electrogenerated in situ.[1] Although not often explored as a standalone technique, it is still found on automatic titration devices; one such example is the coulometric Karl Fischer apparatus, widely used in research and industry.[2]

In a teaching setting, coulometric titration is an important technique that bridges volumetric titrations to instrumental techniques and can be used for a wide range of analytes, including redox and neutralization titrations.[3−6] Exploring coulometric titrations using a visual indication of the endpoint point provides an easy transition for students from classical methods into instrumental techniques. With an understanding of the relation between the electric charge passed in a system (*quantity of electricity* in Faraday's own words)[7] and the amount of substance reduced or oxidized in an electrolyte bath (Faraday's law of electrolyses),[7] the students can correlate electrical currents and titration times (electrical charge) of the coulometric titrations with the volume of titrant added in a volumetric titration.

At the University of São Paulo, in the 2024 curriculum, we teach coulometric titrations to second-year undergraduate chemistry students following volumetric titrations. After extensive experimental sessions of neutralization, precipitation, complexation, and redox volumetric titrations, the students perform a constant current coulometric titration of ascorbic acid (AA) with iodine,[8] following the reaction

$$C_6H_8O_6 + I_2 \rightleftharpoons 2I^- + C_6H_6O_6 + 2H^+ \quad (1)$$

where the iodine is electrogenerated at a platinum electrode from potassium iodide (see eq 2), which is added in large excess to the AA solution.

$$2I^- \rightleftharpoons I_2 + 2e^- \quad (2)$$

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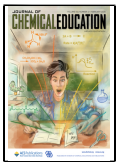

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The endpoint of this titration is determined by the first hint of a blue color in solution from the complex of starch with iodine, added as an indicator, with the excess of iodine in solution after all AA is titrated.

The equipment used in this practice is a "seasoned" 1965 E211 Metrohm coulometer. There is a visual and sensory aspect to using this equipment for teaching. The ability to start and stop the current flow with the press of a remote button (Figure 1A) that can be brought closer to the solution allows

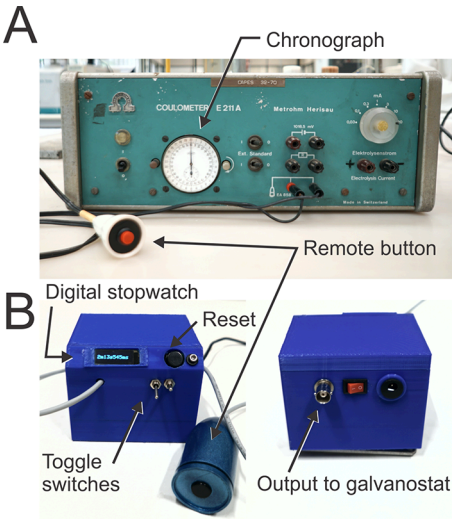

Figure 1. Comparison between the interface of the (A) 1960s Metrohm coulometer and (B) the developed control box for a modern potentiostat/galvanostat. The pictures highlight the important controls in both interfaces, such as the time measuring unit (chronograph and digital stopwatch), the remote button to switch the application of current on and off, the toggle switches for current selection, and the BNC output to control a modern galvanostat.

the students to keep close track of the solution color, stopping the titration at any suspected color change and deciding if the endpoint was reached or not. This control of the titration progress while keeping a close eye on solution changes is very relatable to controlling the buret valve in a volumetric titration while stirring the solution in the Erlenmeyer flask. Reading the analog chronograph (Figure 1A) is equivalent to reading the buret volume. These simple parallels help the students transition to instrumental methods and are of great didactic value.

Unfortunately, modern coulometers or potentiostats/galvanostats, which can also be used for coulometric titrations, do not offer a hands-on experience. The students must interact with the equipment using a software interface, and at least for Metrohm Autolab potentiostats/galvanostats, the student must select how long the current is applied prior to the experiment, requiring the student to select a long time and stop the experiment when the endpoint is perceived. The titration time must then be read from a potential vs time plot. There are no readily available keyboard shortcuts to start and stop the application of the titrating current, and the student must stop and start the current with the mouse cursor, having to look at the computer screen to do so, taking the student's attention from the experiment. There is also no option to pause the current application; therefore, in case the student misidentified the endpoint, the experiment must be restarted from the beginning and the times for both experiments added. Although feasible, using a modern galvanostat resulted in a more complex, less interactive, and less enjoyable experience, as perceived by surveyed students (Section SI-1 of the Supporting Information).

We imagine that very few analog coulometers are still working; hence, this experimental practice has been removed from most laboratory practices. After several repairs of our E211 Metro coulometer and to keep this experimental practice in our curriculum for years to come, we developed a simple and affordable way to control a modern potentiostat/galvanostat (in our case, Autolab 128N), using an Arduino Uno microcontroller and 3D printed parts, giving the student the same hands-on interface of the 1960s equipment, while retaining the precision and accuracy of the commercial instrument. The use of microcontrollers, particularly Arduino, is already well-established in the chemistry teaching setting due to their versatility, accessibility, and low cost, making them ideal for designing instruments tailored to educational laboratories.[9−12] The presented instrumentation (Figure 1B) allows titration currents to be selected with toggle switches and provides a remote button to start and stop the application of the current and a digital stopwatch that follows the current application time, all with a simple user interface. We believe that the didactic benefits of such an interface outweigh the apparent silliness of bringing back instrumental features of the 1960s to a modern machine.

Here, we report on the fabrication of a controlling box for any modern potentiostat/galvanostat that allows for external communication and its application to a real undergraduate experimental practice at the University of São Paulo. During the experimental practice, we explore the difference between the titration endpoint and the equivalency point, using molecular absorbance spectra data to quantify the experimental error and highlight the student's improved accuracy with successive titrations.

MATERIALS AND METHODS

Chemicals

All of the reagents used were of analytical grade. Potassium iodide and sodium sulfate were obtained from Merck (US). Ascorbic acid, soluble starch, and sodium acetate were obtained from Sigma-Aldrich (US). A 1 mL volume of a 1% starch indicator solution was used in the experiments. As the electrolyte solution, 70 mL of acetate buffer solution (pH 4.7) was added to the coulometric cell (Figure 2). The cathodic compartment was filled with a 0.5 mol L^{-1} sodium sulfate solution. A standard solution of 0.0981 mol L^{-1} AA in acetic acid/acetate buffer was used as the sample for all experiments. All of the solutions were prepared on the day of the experiment with deionized water.

Spectrophotometric Measurements

Spectrophotometric measurements were made using an Agilent 8453 UV−visible spectrophotometer. After the student identified the titration endpoint, 2 mL of titration solution was taken from the coulometric cell. A 1 cm optical path quartz cuvette was used, and absorbance spectra between 300 and 700 nm were recorded. After the measurements, the aliquots were quantitatively transferred back to the electrochemical cell.

Control Box

Selection of the titration current value and control of its applications, as well as recording the titration time, were

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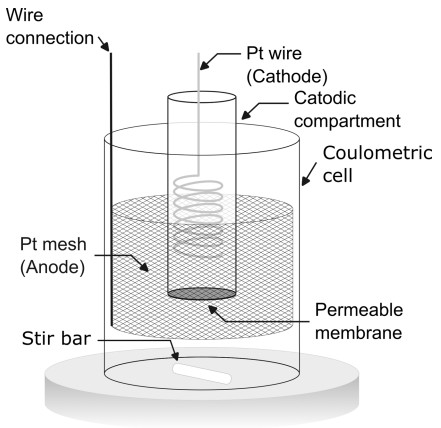

Figure 2. Representation of the coulometric titration cell showing the electrodes and principle components. The electrolyte solution, acetate buffer, is placed inside the coulometric cell, covering the entire platinum mesh, to which aliquots of 0.0981 mol L^{-1} of AA were added to be titrated. The cathodic compartment is filled with 0.5 mol L^{-1} Na_2SO_4 solution.

performed by a custom-made control box using an Arduino Uno microcontroller and a few discrete electronic components. The box was housed in a 3D printed enclosure. A detailed description of the controlling circuit, its components, and testing can be found in Section SI-2 of the Supporting Information.

Coulometric Titration

A Metrohm Autolab 128N potentiostat/galvanostat was used in galvanostatic mode to control the current flow in the titration cell. The coulometric titrations were performed at a constant current of 14.25 mA. A platinum wire coil was used as the cathode and a platinum mesh as the anode (Figure 2). The cathode was separated from the anode and placed in a reservoir (cathodic compartment) containing 0.5 mol L^{-1} Na_2SO_4 solution, connected to the coulometric cell by an ion permeable membrane (battery separator).[8,13] The solution in the coulometric cell, 70 mL of acetate buffer, was continuously stirred using a magnetic stir bar, and aliquots of 0.0981 mol L^{-1} AA were added to be titrated.

Evaluating Undergraduate Students' Feedback on the Proposed Control Box

To evaluate the impact and feedback of the proposed control box for teaching, a questionnaire consisting of four main questions was administered to three volunteer undergraduate students after conducting the coulometric titration experiment described above under two conditions: using only the AutoLab 128N instrument and using the AutoLab 128N instrument with the control box. Both the questionnaire provided and the data and answers distribution are presented in the Supporting Information (SI-1). These results are used to prove the statements regarding the developed control box and its applications.

Controlling a Modern Potentiostat/Galvanostat

Even though there are several alternatives for the affordable fabrication of potentiostats, which could be converted to work as a galvanostats,[14−16] at least in the scope of the Brazilian university landscape, there are plenty of commercial potentiostats that are used for teaching, with a particular disposition of ones manufactured by Metrohm Autolab. Instead of manufacturing an entire coulometer, we fabricated only a control box to allow students to interface with existing equipment in a more hands-on and didactic way. The control circuit is simpler than an entire potentiostat and hence can be replicated by a wider audience. It should be noted that a simple galvanostat for coulometric titrations could be made with a constant current power supply circuit, and this could be an alternative if instrumental resources are scarce. However, this would be at the expense of instrumental precision, which is needed for analytical applications of coulometry, as this is a primary method. Most commercial potentiostats have an analog input and output that allows for external control of the applied currents or potential in the electrochemical cell; hence, we believe that our approach can be extended to most equipment in use for electrochemistry teaching, and it is not restricted to Metrohm Autolab equipment. A precise reading of the titration current is key for a precise calculation of concentrations, and this can be done with a digital ammeter, in series, between one of the electrodes and the galvanostat. In our case, the display in the front panel of the Autolab 128N was used for reading the titration current.

With the Arduino microcontroller, the Autolab 128N galvanostatic current was controlled by supplying a voltage to the Autolab's analog input port, which corresponds to a current value. Three current levels could be selected by using toggle switches in the control box (Figure 1B), resulting in 14.25, 22.70, and 28.91 mA current values. These values were generated by a simple voltage divider (R-2R resistor ladder). A detailed explanation of how to externally control the Autolab 128N, in galvanostatic mode, and the control circuit, including the R-2R resistor ladder, can be found in Section SI-2. The application of the titration current in the coulometric cell by the galvanostat was turned on and off using the remote button (Figure 1B), which switches the control box output potential (also Figure 1B) between the value selected by the toggle switches and ground, with the latter stopping the application of the current. Pressing the remote button also starts and stops the digital stopwatch, which can be reset by pressing the reset button (Figure 1B). Details of the code running in the microcontroller can be found in Section SI-3.

RESULTS

Validation of the Controlling Circuit Using Electronic Components

The operation of the galvanostat through the control box was first tested by using electronic components instead of a real titration cell. Similarly to potentiostats, which are often validated by performing polarization curves to resistors,[14] we measured the voltage drop across resistors of known values under the different titration currents. The voltage was used to backcalculate the test resistor value, validating the circuit accuracy (Table S2). For teaching purposes, we also used a light-emitting diode (LED) as the electrochemical cell analogue, with the brightness of the LED at the different titration currents working as a proxy for the current values (Figure S3). This resource can help students (literally) visualize the titration currents; we can be a powerful teaching aid for electrochemistry. These results can be found in Section SI-4.

Coulometric Titration of Ascorbic Acid with Iodine

As part of our second-year undergraduate chemistry laboratories, we introduce experimental techniques with the constant current coulometric titration of AA with iodine (eq

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1), electrogenerated from iodide (eq 2).[8] For such, 50 μL aliquots of unknown (to the students) AA solution (0.0981 mol L^{-1}) are titrated using starch solution as a visual indicator of the endpoint, which reacts with iodine in excess (after the endpoint), forming a purple-brown color. The titration is sequentially repeated several times, with a new AA aliquot being added to the same electrochemical cell (containing previously titrated solution) once the student identifies the endpoint and the stopwatch is reset. The addition of a new AA aliquot consumes the excess iodine, complexed with starch, removing any color from the solution, which will be ready for a new titration.

Repeating titrations in the same electrolyte solution provides a means for the students to contrast the color of the identified endpoint between each successive titration, allowing the discussion during the practice about visual identification of the titration endpoint. Often the identification of the endpoint of the first titration happens well past the equivalency point, with the solution having a very strong purple-brown color. With some guided questioning from the demonstrators, and a reminder that the endpoint should be the point where the *first hint of color appears in the solution*, the student's identification of the endpoint progresses closer to the equivalency point with each titration, as we will discuss further.

Another interesting aspect to discuss with the students is that each titration can be treated as an individual experiment, with the AA concentration calculated for each one, or all titrations can be treated as a single experiment, summing all volumes of AA solution added to the cell and all of the titration times (or charge flown). As can be seen in Table 1, where real data from a student is portrayed for the titration of the AA solution at a constant current of 14.25 mA, by treating each titration as an individual experiment, there is an apparent increase in accuracy. However, there is no clear trend in the error values. Using the same electrolyte solution for all titrations, the excess iodine generated in a previous experiment reacts with the subsequent aliquot of AA added, decreasing the amount of AA to be titrated. Suppose we account for the previous titrations' errors (iodine excess) to calculate the actual amount of AA in the electrolyte solution at each experiment. In that case, the calculated relative error ("*Corrected Error*" in Table 1) and the increased accuracy between experiments are clear. More importantly, we can see that the previously smallest titration error (Titration 3) is not the most accurate titration, that being the last experiment.

Table 1. Titration Data from a Real Experiment Performed by an Undergrad Student[a]

Titration	Time/s	[AA]/mol L^{-1}	Error/%	Corrected Error/%
1	76.774	0.113	15.6	23.2
2	58.781	0.0868	11.5	13.1
3	65.221	0.0963	1.81	9.45
4	63.144	0.0933	4.93	5.49

[a]The titrations were performed at a constant current of 14.25 mA.

By treating all titrations as a single experiment, the error of every preceding titration is accounted for by the following one, by the consumption of the excess I_2 by the AA added. Hence, if we consider that the identification of the endpoint is the least precise aspect of the experiment, the AA quantification error results only from the last titration error, which is the one where the students can more accurately identify the end point, as seen in Table 1. Other errors are still affecting the precision of the measurement, such as the error in the aliquot volume, which compounds with successive titrations, but these are in a different order of magnitude than the errors calculated. It should also be noted that this is an approximation useful for teaching, and for analytical applications, all errors should be accounted for. Only the student's titration data are portrayed in the table, but multiple titrations were performed in the same cell with the data from all titrations found in Table S4 (Section SI-5). These include 2 titrations performed by the demonstrator to test the setup and 2 titrations performed by the student, which were flagged as outliers due to instrumental error as the remote button got stuck and the titration could not be stopped immediately. When all titrations are considered, including the outliers, an AA concentration of 0.0988 mol L^{-1} is calculated, representing a relative error of only 0.6%, much smaller than any individual titration error (see Table 1). This finding highlights the accuracy of coulometric titrations and serves as a great discussion point with the students, where quantification error and replication of an analysis can be explored.

Titration Accuracy

The improvement in the student's ability to accurately recognize the titration endpoint, reported by the corrected error in Table 1, can also be tracked by coupling the experiment with a spectrophotometer, measuring the solution absorbance spectrum at each identified endpoint and quantifying the amount of iodine present in solution (titration error). The absorbance spectra of each of the titrations' endpoints in Table 1 can be seen in Figure 3. From the absorbance data, we can observe that the iodine−starch complex presents a distinct color in the visible spectrum, with a broad absorption band, with a maximum of around 530 nm (purple-brown color; see Figure 3), enabling its use as a visual indicator. The solution also absorbs in the ultraviolet region, with a more pronounced absorbance band than in the visible region, with a maximum of 352 nm, likely originating from I_3^-,[17] formed within the iodine−starch complex, as there is no I_3^- free in solution due to the excess of starch. Both absorption maxima are marked in Figure 3 with dashed lines. Large titration errors result in a large excess of iodine−starch complex in the endpoint, resulting in larger absorbance values. As expected, the tendency of the absorbance values between titrations follows the corrected relative errors in Table 1.

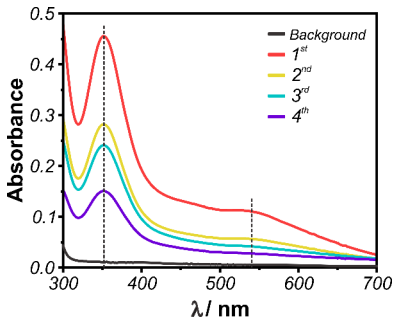

Figure 3. Absorbance spectra recorded at a blank solution (background) and at each of the titration endpoints identified by the students (in Table 1). Dashed lines mark the maximum absorbance in the ultraviolet region (352 nm) and visible region (530 nm).

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Qualitatively, the absorbance data at 530 nm mirror the student and demonstrator perception of increased accuracy in detecting the endpoint, with the color of the solution at the endpoint getting progressively fainter (lower absorbance value) with every experiment. At any titration using visual indicators, the endpoint should be recognized at the instance of the smallest perceptible change in solution color. Looking at the absorbance data at 530 nm between a background spectrum (no iodine in solution, absorbance of 0.00138) and titration 4 (absorbance of 0.0285) and the relation between absorbance and light intensity described by Beer–Lambert's law,[18] we can see that the student identified the endpoint when the solution changed in color intensity by only approximately 6%. This very minute change in color intensity is barely perceptible by the eye, setting this endpoint very close to the equivalency point.

We can use the absorbance band at 352 nm, not detectable by the human eye but with a more intense absorbance signal, to calculate how far the detected endpoints are from the equivalency point, where there should be no I_2 in solution to complex with the starch indicator. To determine the iodine concentration in each endpoint, the coulometer was used to generate known quantities of I_2 from I^- in a solution containing the same composition of the titration solution, except AA. The absorbance spectra for each I_2 concentration can be seen in Figure 4A, with the plot of absorbance value at 352 nm vs I_2 concentration in Figure 4B. From the linear fit, described by Beer–Lambert's law,[18] the molar absorption coefficient of the iodine–starch complex could be calculated, and this can be an elegant alternative for calculating such a property in teaching laboratories, which could be further explored. Here, as the optical path was identical between all experiments, we used Figure 4B as a calibration plot to calculate the iodine concentration at each endpoint, quantifying the titration error in a number of moles (Table 2). With this information and the titration current (14.25 mA), these concentrations can be converted to seconds, representing the time passed between the equivalency point and the student's identification of the end point (Table 2).

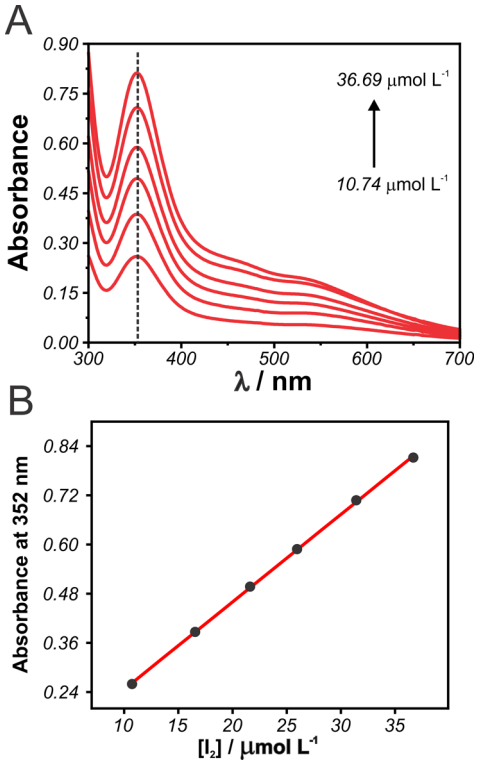

Figure 4. (A) Absorbance spectra recorded for different iodine concentrations, with the iodine being electrogenerated galvanostatically at a constant current of 14.25 mA. The resulting concentrations are 10.74, 16.55, 21.62, 25.97, 31.34, and 36.69 μ mol L^{-1}. (B) Plot of the absorbance intensity at 352 nm (marked by a dashed line in panel A) against the concentration of iodine generated in solution. The linear fit of the points in panel B can be used to calculate the molar absorption coefficient for the iodine–starch complex.

Table 2. Number of Moles of the Iodine and Time Associated with Passing the Equivalency Point in Each Coulometric Titration Made by the Student[a]

Titration	Iodine/μmol	Time/s
1	1.41	19.08
2	0.831	11.25
3	0.695	9.414
4	0.394	5.335

[a]The mole values were obtained using the absorbance values in Figure 3 and the calibration curve in Figure 4B.

It is interesting to note that, at the most accurate titration by the student, where a change of only approximately 6% in color was perceived, the endpoint was recognized 5 s after the equivalency point. The error in titration 4 represents a molar excess of only 0.394 μmol of the iodide in the coulometric cell. Following the stoichiometry of eq 1, this represents the same molar error for AA. A tangent, but worth discussion here, is the ability to visually detect a concentration of only 5.52 μmol L^{-1} of the iodine in solution (accounting for the coulometric cell volume and all aliquots added in titration 4), highlighting the sensitivity of visual methods. This point can be raised to the students, fomenting a discussion about visual and instrumental methods.

Expressing the titration error, the difference between equivalency and endpoint, in time is a powerful tool to discuss measurement error, precision, and accuracy with the students. Time is a much more tangible quantity than moles and helps the students understand error, as a quantity, more easily. The conversion from time back to moles, using Faraday's law of electrolyses, not only helps the students to cement the fundamental aspects of coulometry but also helps them to contrast the large temporal error with the small molar error, allowing discussion of important analytical concepts such as sensitivity and limit of detection, tying back to the discussion of the visual indicator sensitivity discussed above.

The 352 nm absorbance data allows the discussion of other endpoint identification methods besides visual, as the students can quickly observe that, even at the faintest endpoint (titration 4 in Figure 3), there is a considerable absorbance signal in the ultraviolet region that could be used instrumentally to track the titration progress. This is a key aspect to help further the student's transition from visual methods to instrumental methods, as even without the formal knowledge at this stage in the course of Beer–Lambert's law, they recognize that information about the solution composition can be acquired by an instrument and used to track the titration progress. This concept can greatly facilitate a student's understanding of other endpoint identification methods that are less tangible than color changes, such as biamperometry.[19]

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CONCLUSION

We report on the development of a control box based on an Arduino Uno microcontroller and affordable electronic components (bill of materials in Section SI-6) for controlling modern potentiostats/galvanostats, in a similar manner to a 1960s coulometer. The control box bridges the intuitive and hands-on analog interface of the old equipment with the modern reliability and capabilities of a new galvanostat. Although modern equipment can perform the same experiments described here, a modern, software-based interface is not always the most applicable for teaching, as it removes the student's focus from the reaction vessel (titration cell), steering it toward a computer screen. The analog interface allows a hands-on approach, which we believe has great didactic value. Using our proposed setup, we perform a titration experiment with chemistry undergraduate students of ascorbic acid with iodine, employing a visual indicator of the endpoint, a starch solution. By repeating successive titrations in the same coulometric cell, without changing the electrolyte solution, we could discuss the importance of replicating experiments multiple times and the impact of replication on the result accuracy.

We used absorbance measurements to track and gauge the students' accuracy in identifying the endpoint over time, showing a drastic improvement with successive titration. With the absorbance data, we could quantify each titration error in excess moles of titrant and convert it to time and analyte concentration, exploring Faraday's law of electrolysis, which is a more tangible quantity for the students. By bringing back the analog controls, we managed to steer the student's attention to the titration cell, giving us ample opportunities to discuss important analytical topics. We believe that coulometric titration experiments are extremely valuable in teaching analytical chemistry and, more specifically, electrochemistry, and we hope that our instrumentation approach and the discussions proposed here will help bring this practice back to undergraduate classes.

ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available at https://pubs.acs.org/doi/10.1021/acs.jchemed.4c01154.

A description and detailed information of the controlling circuit, the Arduino sketch running on the microcontroller, the test of the controlling circuit using electronic components as analogues of the coulometric cell, all the ascorbic acid titration data for the student experiment, and a bill of materials (PDF)

AUTHOR INFORMATION

Corresponding Author

Gabriel N. Meloni − *Institute of Chemistry, Department of Fundamental Chemistry, University of São Paulo, 05508-000 São Paulo, SP, Brazil;* orcid.org/0000-0002-2950-2464; Email: gabriel.meloni@usp.br

Authors

Rafael S. Santos − *Institute of Chemistry, Department of Fundamental Chemistry, University of São Paulo, 05508-000 São Paulo, SP, Brazil*

Thiago R. L. C. da Paixão − *Institute of Chemistry, Department of Fundamental Chemistry, University of São Paulo, 05508-000 São Paulo, SP, Brazil;* orcid.org/0000-0003-0375-4513

Complete contact information is available at: https://pubs.acs.org/10.1021/acs.jchemed.4c01154

Notes

The authors declare no competing financial interest.

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