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Visualizing Competing Charge-Transfer Reactions with Electrochemiluminescence

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ABSTRACT: While electrochemistry is a pervasive concept in chemistry and general science education, being introduced as early as middle school, accessible hands-on experiments to understand fundamental concepts are lacking. One such fundamental concept is that in many electrochemical applications (batteries and electrolyzers), several reactants compete for the charge in a sort of sparring-for-sparks. Here, we present an activity, suitable even for children, to demonstrate this cardinal idea. Our goal is for students to visualize concurrent charge transfer reactions by taking advantage of electrochemiluminescence (ECL). We demonstrate that ECL can be observed through the coreduction of persulfate and $[Ru(bpy)_3]^{2+}$, which displays a high ECL emission efficiency. By simply tuning the electrode potential, the electrochemical experiment is then tuned to favor both the ECL reaction as well as the bubble-forming hydrogen evolution reaction. We present an experimental guide for students and instructors, demonstrate several accessible options for materials that can be used to carry out these experiments, and provide extensions for the activity to make it suitable for several age levels, focusing on middle school and above.

KEYWORDS: *Elementary/Middle School Science, Introductory Chemistry, Analytical Chemistry, Laboratory Instructions, Hands-on Learning, Aqueous Solution Chemistry, Bioanalytical Chemistry, Electrochemistry*

Electrochemistry is one of the oldest disciplines in chemistry, with the founding of the field at the turn of the 19th century by Volta through his construction of the Voltaic pile.[1,2] Since the early days of Volta, electrochemistry has expanded to a field with applications from biology to batteries, and everything in between. Fundamental electrochemical developments, such as Volta's, paved the way for applications of societal importance, including the lithium-ion battery[3] and the glucometer.[4] Despite the advances of the field, students' views of electrochemistry are often myopic, and often ending with the writing of cell diagrams in general chemistry.[5] This is unfortunate as this field and its crucial concepts are not only easily applicable to real world problems, but they are easily displayed in quite striking visual demonstrations.[6−8] Recently, there has been work done by our group and others to update and reinvigorate the chemical education literature with new activities and teaching methods to illustrate to students the exciting developments of electrochemistry[2,9−20] as well as to meet the need for the next generation of scientists with formal electrochemical training.[21−25]

Electrochemiluminescence (ECL) is a visually stunning technique where reactions at an electrode surface give way to visible light production.[26] In general, two species are oxidized or reduced at an electrode surface where a secondary reaction between the two activated molecules follows to promote an excited state. As this excited state relaxes, light may be observed. While these reactions can be complex, the light-emitting nature of these systems presents an exciting system for electrochemical demonstrations and activities. Others first outlined a short demonstration in 2001 and laid the groundwork by detailing the production of light from the TPrA-$[Ru(bpy)_3]^{2+}$ system.[27] Now, over 20 years later, there are just a few more publications in the science education literature, to our knowledge, extending these systems to the classroom. In 2013 a demonstration using the H_2O_2-luminol system was shown.[6] Also, more recently, an undergraduate laboratory experiment for the detection of trace H_2O_2 using the luminol luminophore was published.[28] These systems present visually appealing demonstrations which are sure to captivate their target audience of undergraduate students.

While our group has had a longstanding interest in advancing ECL,[29−32] we have recently been interested in the question: If an electron is sitting at the surface of an electrode, what solution species will take the electron, and what factors dictate to whom the electron will go?[33−35] This very fundamental question, which we illuminate here, is at the core of almost any energy storage and conversion device and electrochemical biosensor. In this article, we present a set of

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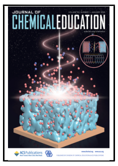

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hands-on experiments for the visualization of competitive charge-transfer reactions, through ECL reactions, for students as young as middle school. Additionally, we provide lesson extensions for the proposed experimentation to be used at the undergraduate level. The persulfate anion-$[Ru(bpy)_3]^{2+}$ system was used, leading to an extremely high ECL emission intensity to allow for an easy visualization of the electrochemical process without a completely dark environment. With a potentiostat, voltage modifier, or batteries, students will perform ECL experiments to both qualitatively and quantitatively determine the effect of the applied potential on interfering reactions. These reactions include the hydrogen evolution reaction (HER), which can be covisualized along with the ECL reaction due to the bubble formation process. Also, students will be exposed to data analysis through the use of powerful and exciting freeware to examine the effects of competing reactions (applied voltage) on the intesnisty of light. This freeware shows students just how low the activation barrier for scientific inquiry is. Finally, fundamental concepts of electrochemistry will be reinforced through these experiments because students will be able to visually identify the working electrode within an electrochemical cell, understand that different reactions occur at the working and counter/reference electrodes, and understand that more than one reaction can take place at each of these electrodes.

■ HAZARDS

Proper protective eyewear and gloves should be worn when performing all experiments. Students should be informed of the danger of contacting and ingesting any chemicals and there should be a discussion on proper safety practices before any experimentation. All solutions used for the outlined experiments should be prepared by the instructor in a fume hood before use by students such that the students are not exposed to concentrated acetonitrile, as it is highly flammable, harmful if swallowed in inhaled, and can cause serious eye irritation, or solid potassium persulfate, as it can cause serious eye irritation, may cause respiratory irritation if inhaled, and is harmful to aquatic life. The hazards associated with these chemicals are minimized when diluted and dissolved in solution, respectively. All other chemicals have no associated hazards. Students should be warned that $[Ru(bpy)_3]^{2+}$ is a strong dye and can stain clothes, skin, and surfaces if it comes in contact. All experiments should be performed in a well-ventilated area away from sparks/open flames/hot surfaces as the solution contains flammable components.

■ MATERIALS AND METHODS

Materials and Instrumentation

Potassium persulfate (99%) was purchased from Sigma-Aldrich. Acetonitrile (99.9%) and potassium chloride (99%) were purchased from Fisher Chemical. Tris(bipyridine)-ruthenium(II) chloride hexahydrate ($[Ru(bpy)_3][Cl_2]\cdot 6H_2O$, 98%) (in this paper we will denote as $[Ru(bpy)_3]^{2+}$) was purchased from Acros Organics and was kept under nitrogen in a desiccator. Ultrapure water (>18.20 MΩ·cm, Thermo Scientific) was used for all experiments. All chemicals were used without further purification steps.

A CHI 290 Potentiostat (CH Instruments, Austin, TX) was used for amperometry and cyclic voltammetry. A WavePico Potentiostat (Pine Research, Durham, NC) was used for instructor/student guide experiments. A USB Step Up/Down Power Supply Regulator and batteries (1.5 and 9 V) were purchased from Amazon.com.

For all experiments, a piece of pencil graphite (d = 0.7 mm) was used as a working electrode and a graphite rod (d = 6 mm) was used as a quasi-reference/counter electrode (QRCE) (Amazon.com). Copper tape was used on both electrodes to facilitate connection to electrode leads (Amazon.com). In all electrochemical experiments the electrodes were rinsed with water and dried with a KimTech wipe. A list of materials and the suggested vendors can be found in the Table S1.

Determining the Effect of Applied Potential

For all experiments, a 10 mL solution containing 10 mM $[Ru(bpy)_3]^{2+}$, 50 mM $K_2S_2O_8$, and 100 mM KCl in 60/40 Milli-Q water/acetonitrile was prepared in a 20 mL. The prepared solution was ultrasonicated for at least 15 min before use. If no sonicator is available, increasing the acetonitrile/water ratio (to increase acetonitrile concentration) can promote solubility of the reagents, at the cost of increased organic solvent content. In addition, the prepared solutions can be shaken gently over a 15 min time period. Solutions were prepared analytically by the authors (BRL and LEK) and with common lab equipment by undergrads, with no difference noted in dissolution.

All experiments performed with a potentiostat were performed in a well-grounded Faraday cage. For the amperometric experiments, a 0.0167 s sample interval was chosen and the potential was held statically for 10 s. During the voltammetry, a potential window of −1.4 to −3.6 V (vs graphite rod QRCE) and a scan rate of 50 mV·s^{-1} was used. For the USB voltage modifier, the chosen potential was adjusted with the buttons on the device. After the desired potential was reached, the device was turned off for a few (*ca.* 5−7) seconds before turning the device back on for 10 s. for image acquisition. Connection of the USB voltage modifier to the electrodes was done with commercially available electrical wire and alligator clips [Amazon.com]. In the experiments with batteries, the applied potential was determined from the voltage listed on the batteries. Connection to the electrode leads were done similarly to the voltage modifier, where the pencil graphite (0.7 mm) working electrode was connected to the negative terminal and the graphite rod counter electrode was connected to the positive terminal of the battery/batteries.

Light Intensity Analysis

All image acquisition was performed with a cellular phone (iPhone 15 Pro Max, Apple Inc.) which was kept in the same position between experiments. In each experiment the phone was placed so that the working electrode in the vial was in focus, and a video was recorded throughout all experiments. A screenshots of the video were taken 2 s after the potential was applied. Image analysis was performed using either ImageJ or the Carolina RGB colorimeter application (available for free on both iPhone and Android). For analysis done with ImageJ, a horizontal line was drawn across the area in the image where the vial was placed and the intensity was plotted. The average intensity across the solution area was used as the light intensity.[36] For analysis performed with the colorimeter app, only the Red Intensity was considered.

■ RESULTS AND DISCUSSION

Electrochemiluminescence (ECL) is an excited-state electrochemical technique that is relatively unique since photons are produced without excitation light. Instead, photons are emitted

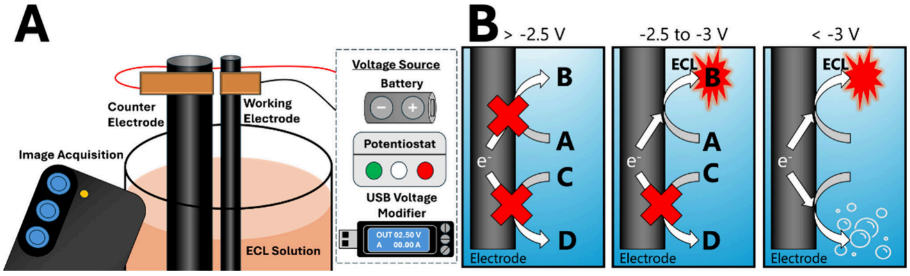

Figure 1. (A) Experimental Schematic including the working electrode, quasi-reference counter electrode (QRCE), and different voltage sources. The ECL solution is contained in a 20 mL scintillation vial. Image acquisition is conducted through a cell phone. The working electrode is composed of a small graphite rod and the quasi reference counter electrode (labeled "Counter Electrode") is composed of a larger graphite rod. The different voltage sources used are a battery, a potentiostat, and a USB voltage Modifier. (B) Simplified schematic to describe the ECL and HER reactions at various potential ranges.

as the result of charge-transfer reactions at a conductive interface. In general, there are two molecules that are necessary: a luminophore and coreactant (in some cases, the co-reactant can be the same luminophore). For ECL to occur, the luminophore and coreactant get oxidized or reduced simultaneously at the surface of an electrode. The often-radical coreactant then acts as a strong oxidizing or reducing agent against the oxidized or reduced luminophore. The oxidation or reduction of the reacted luminiphore by the activated coreactant then forms an excited state in the luminophore. Finally, a portion of the population of excited state luminophore molecules can undergo radiative relaxation to emit a photon. This leads to the beautiful glowing observed in ECL experiments. Our effort in this manuscript is not to elucidate intricate mechanistic details of ECL but rather reinforce competing charge transfer reactions.

Common coreactants include tri-n-propyl amine (TPrA), benzoyl peroxide, sodium oxalate, and the focus of this activity: the persulfate anion. While many types of molecules and low-dimensional materials can act as luminophores, tris(bipyridyl) ruthenium complexes are the most popular due to their high ECL efficiency and solubility in aqueous solutions.

In this activity $[Ru(bpy)_3]^{2+}$ was selected as the luminophore of choice. A solution containing 10 mM $[Ru(bpy)_3]^{2+}$, 50 mM $K_2S_2O_8$, and 100 mM KCl in 40/60 (v/v) acetonitrile−water was used for all experiments. In the electrochemical cell, a pencil graphite rod (0.7 mm) working and a graphite rod (d = 6 mm) counter electrode were connected to the applied voltage source (Figure 1A). Both rods can be obtained from a mechanical pencil. While a graphite rod is used for the counter electrode in these experiments, several pieces of pencil graphite taped together could also be used so long as the area of the counter electrode is bigger than that or the working electrode. This is important to ensure the reaction at the working electrode is rate limiting and, thus, the reaction being measured.

Students should be given an explanation of this electrochemical cell via direct instruction or a simple diagram before moving on to the hands-on experiments. We have provided an example of an electrochemical cell diagram in the instructor's guide (Supporting Information, Section 2, Figure S1). Briefly, students can be told that the working electrode is where the desired electron-transfer reaction takes place and the potential, or energy which is given to the system to make certain reaction happen, is applied. The counter electrode drives an opposite reaction to complete the electrical circuit. This explanation can be expanded upon if students are unfamiliar with electrons (the part of an atom that carries a negative charge), electron transfer reactions (were an electron goes from one place to another), or circuits/electricity in general. An important questions for students to consider after getting this introduction: do they expect to see glowing and bubble formation at both electrodes, or just the working electrode or just the counter electrode? Why?

The observations from the experiments below should help solidify these questions. Figure 1B outlines the information middle school students were given about the ECL processes. While students could grasp the full ECL mechanism if offered extensive lecture time, the key ideas to be conveyed to the students before the experiments are as follows: In the solution (or, as we explained: "the liquid which contains the chemicals needed for these reactions"), there exists a chemical A which gives the solution it is red color. As we apply a potential, or we give energy to our working electrode, we can cause A to get an electron which will convert it to B, a species which has a lot of excess energy or is in an "excited state", which causes light to be given off. There is a certain energy we must apply to make this reaction take place which we will figure out by applying many different potentials. There also exists a chemical C in solution which will be converted to D at a specific potential. This reaction will be obvious because D is a gas and a visible bubble will be given off.

While the above-described explanation is ample for the purpose of this activity; students, being heterogeneous in curiosity often question concepts at greater depths (to the joy of the instructor(s)!). Below is an appropriate *"high-school level"* description of the processes that occur in the solution.

We propose to tell the students: "Both the luminophore and coreactant gain an electron at the surface of the electrode (eqs 1 and 2). The newly formed coreactant (the sulfate radical) then takes an electron from a luminophore molecule that has not been reduced, or chemically oxidizes it. Then there is both +1 and +3 oxidation states of the luminophore present. The state with an extra electron can give its electron to the molecule more deficient, a process known as radical annihilation. If the energy of annihilation is sufficiently high, an excited state (eq 4) forms. This excited state has extra energy, which it releases in the form of light (eq 5)."[37] For chemistry instructors, it's important to note that the tris(bipyridine)ruthenium(II) reduction occurs at the bipyridine ligand.

$$[Ru(bpy)_3]^{2+} + e^- \rightarrow [Ru(bpy)_3]^{+} \qquad (1)$$

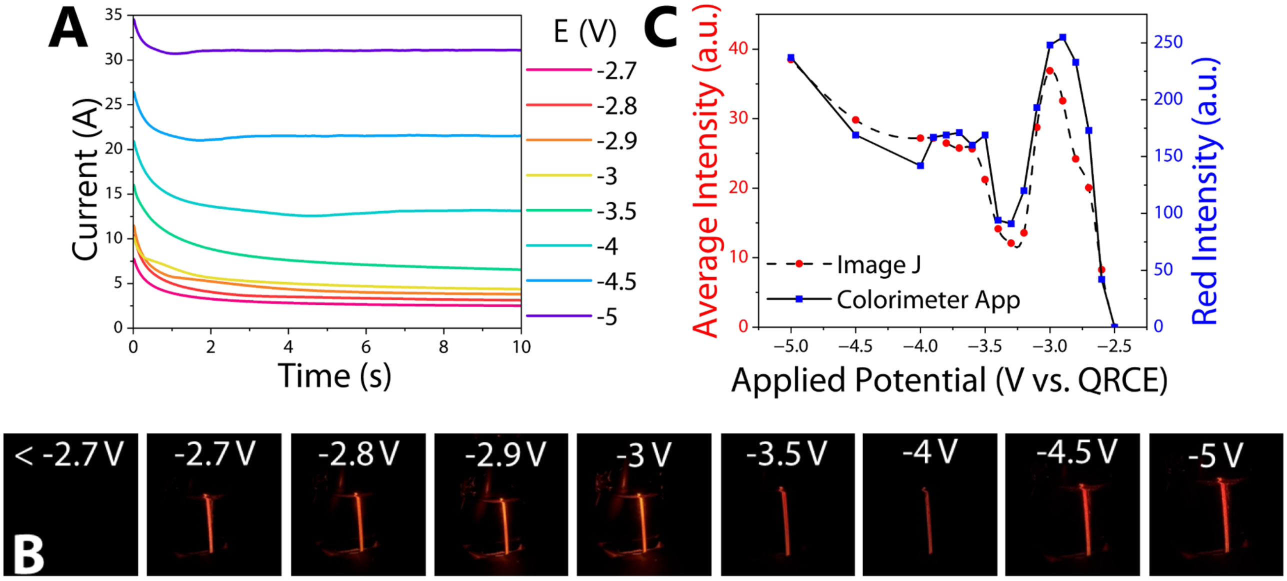

Figure 2. (A) Amperometric *i-t* curves of selected applied potentials overlaid (−2.7, −2.8, −2.9, −3, −3.5, −4, −4.5, and −5 V vs graphite rod QRCE). Amperometry was taken in 10 mM $[Ru(bpy)_3]^{2+}$, 50 mM $K_2S_2O_8$, and 100 mM KCl in 40/60 (v/v) acetonitrile−water with a pencil graphite rod (0.7 mm) working electrode and a graphite rod counter electrode for 10 s. (B) Photographs taken during the amperometry plotted in (A). (C) Light intensity plotted versus applied potential. Intensity determined with ImageJ image processing software (red dots and dashed line) and a free smartphone colorimeter app [Carolina RGB Colorimeter] (blue dots and solid line) at each applied potential are overlaid.

$$[S_2O_8]^{2-} + e^- \rightarrow [SO_4]^{\bullet -} + [SO_4]^{2-} \tag{2}$$

$$[SO_4]^{\bullet -} + [Ru(bpy)_3]^{2+} \rightarrow [Ru(bpy)_3]^{3+} + [SO_4]^{2-} \tag{3}$$

$$[Ru(bpy)_3]^{+} + [Ru(bpy)_3]^{3+} \rightarrow [Ru(bpy)_3]^{2+*} + [Ru(bpy)_3]^{2+} \tag{4}$$

$$[Ru(bpy)_3]^{2+*} \rightarrow [Ru(bpy)_3]^{2+} + h\nu \tag{5}$$

A more complete description of this reaction mechanism is provided in the instructor guide in the Supporting Information Section 2 (Equations S1−S7) as informational to the readership. In addition, a more detailed description of the competing reactions (Equation S8) and a table of chemicals used with their respective SDSs (Table S2) are provided in the instructor's guide in Supporting Information Section 2.

For the purposes of this activity, we will first describe the set of experiments which can be performed with a potentiostat, where amperograms can be recorded. Here amperometry was performed in solution from −2.5 to −5 V over 10 s. The amperograms were recorded, and a select set of amperograms are presented in Figure 2A. The potentials chosen were based on observations during voltammetry in the prepared solution (Figure S2), where ECL was only visible at potentials more negative than −2.8 V and bubble formation was seen at potentials more negative than −3.2 V. The full set of amperograms taken in these experiments can be found in Figure S3. The measured current, which can be explained to students as the rate of electrons going to species in solution, increases when more negative potentials are applied. During the amperometric experiments, photographs were taken with a smartphone (select photographs in Figure 2B and all photographs used in Figure S4). These images were then analyzed with either ImageJ software on a computer (the standard for image analysis) or with the Carolina RGB Colorimeter smartphone app (a free app available on both iPhone and Android). The trends exhibited by these traces will remain constant between brands of cameras; however, the actual intensity values will vary. The intensity determined with both methods is plotted in Figure 2C. From the overlay of both methods, it is apparent that the analysis performed with only a smartphone aligns well with that done on a computer with analysis software, demonstrating that the intensity analysis can be accomplished with nothing more than a smartphone. While this demonstrates the accessibility of the image analysis, the use of a potentiostat is likely prohibitive in most classroom settings. Regardless, a guide which can be given to students in the instance that these experiments are accessible is provided in the Supporting Information Section 6 (Figures S5−S13, Table S3).

To make these experiments more accessible to the general classroom, a USB voltage modifier can be purchased ($18 from Amazon at time of purchase in Fall 2024) to get a large range of potential values without the need for a potentiostat. To set up the cell, the working electrode (pencil graphite) can be connected to the negative terminal of the modifier and the counter electrode (graphite rod) can be connected to the positive terminal via electrical wire. When the intensity (determined with the colorimeter app on a smartphone) is plotted versus applied potential, where the voltage modifier is the applied potential source, (Figure 3A) the same trend can be observed (Figure S15 shows the two plots overlaid). This trend reflects an initial intensity of zero as potentials less than −2.5 V are applied. An initial increase in ECL intensity as potentials more negative than −2.5 V are applied. After potentials sufficient to drive HER as well as the ECL reaction are applied (>3 V) the observed ECL intensity initially goes down as the light-emitting reaction starts to compete with the bubble forming reaction. The ECL intensity then begins to increase again with more negative potential. This observation may seem counter-intuitive and is the opportunity for the instructor to point out the violent nature of bubbles dislodging from a surface (a bit like releasing a beach ball from below the

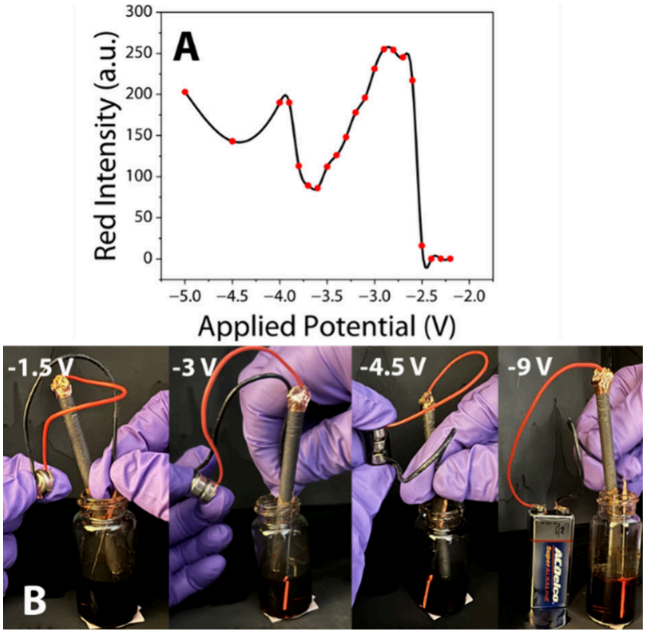

Figure 3. (A) Light intensity plotted versus applied potential. Intensity was determined with a free smartphone colorimeter app [Carolina RGB Colorimeter] at each potential applied with a USB voltage modifier. (B) Photographs of ECL experiments performed with various combinations of commercially available batteries. Images are labeled with the applied potential as determined by the listed voltages on the batteries.

surface of a pool or the ocean). Bubbles dislodging enhances mass transport, increasing the overall reaction and light intensity. All photographs used for the intensity analysis presented can be seen in Figure S14. Additionally, these experiments can be performed by simply using batteries as the applied potential source (Figure 3B). To accomplish this, electrical wire can be used to connect the working electrode and the negative terminal of the battery and the counter electrode to the positive terminal of the battery. It should be noted that the electrodes can be placed anywhere in solution such that they are submerged without being in contact with one another so long as they are connected to the appropriate leads. The applied potential can be determined by the output voltage listed on the batteries. It may be beneficial to check the output voltage of the batteries with a voltmeter, if possible, to ensure the batteries have not depleted over time. The use of batteries makes these experiments more obvious. When batteries are stacked, more energy can be supplied to the system, and more bubbles/light intensity can be observed. However, the limited options for output voltage on commercial batteries (typically 1.2 and 1.5 V) limit the potentials possible (i.e., −1.2, −1.5, −2.4, −2.7, −3, etc.).

The experiments outlined above, regardless of the potential source used, present an exciting hands-on activity for students as young as middle school. While the experiments are run, students should be encouraged to record their observations. Even without access to image analysis (i.e., colorimeter app and photography with a smartphone), students should be able to observe that there exists some potential range where there are no reactions occurring (i.e., no bubbles or ECL), there is only ECL with no bubble formation, or there is visible ECL as well as bubble formation. This could be observed visually or plotted via intensity vs potential. After the observations are made, a discussion around the results should help solidify the concepts of (1) the function of the working and counter electrodes and (2) energy required to drive a specific reaction (and how applied potential affects the observed reaction) and (3) the possibility of more than one reaction occurring simultaneously and how these may affect one another.

Students should be asked whether they saw light coming from both the working and counter electrode. Why did they only see light at the working electrode? To show that the counter electrode is performing a function (and is not just simply a rod in solution), sodium oxalate (50 mM) can be added to the solution. Sodium oxalate is a species which is oxidized to form a radical species which can lead to ECL. As such, the counter electrode, which is positively poised in these reactions, should then be able to lead to observed ECL signal from the oxidative oxalate ECL reaction rather than the reductive persulfate ECL reaction (Figure 4). When −5 V is applied to the system with the USB voltage modifier, light emission at the counter electrode can be seen. However, the intensity at the counter electrode is much lower than that at the working and requires a dark space for the observation of the emitted light. This is due to the lower ECL efficiency of the sodium oxalate-induced ECL reaction.[38,39] From these experiments, students should see that once a species which can be oxidized is added to solution, glowing can then be seen at the counter electrode. Because opposite potentials occur at both electrodes, one will have the oxidative oxalate ECL reaction occurring, and one will exhibit the reductive persulfate ECL reaction.[31] However, the light intensity seen is different than the light intensity seen at the working electrode, and as such two different reactions are occurring at each electrode. A schematic of the concurrent ECL reactions at the working and counter electrodes can be found in Figure S16.

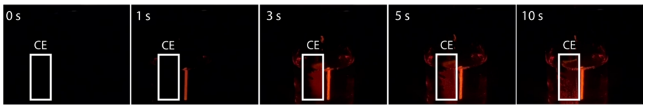

Figure 4. −5 V applied to solution containing 10 mM $[Ru(bpy)_3]^{2+}$, 50 mM $K_2S_2O_8$, 50 mM $Na_2C_2O_4$ and 100 mM KCl in 40/60 (v/v) acetonitrile−water with a pencil graphite rod (0.7 mm) working electrode and a graphite rod counter electrode for 10 s with USB Voltage modifier. The counter electrode area is shown in the white box. Over time, light can be observed at the counter electrode.

CONCLUSIONS

Electrochemiluminescence (ECL) reactions pose a very exciting opportunity for visually striking experiments for students. Here, we provide a visually compelling ECL reaction which can be accomplished with just batteries, pencil graphite, and electrical wire with an ECL efficiency high enough to allow for observation of emitted light without the need of a completely dark environment. Additionally, the experiments proposed not only give insight into ECL and experimental observations of light emission, but also allow for introduction to HER and bubble formation. Through the proposed experiments students will be able to make observations which enforce 3 critical concepts: (1) the working electrode and counter electrode perform different reactions in an electrochemical cell to make these experiments possible, (2) potential is a form of energy which can drive reactions and different potentials can drive different reactions and (3) more than one reaction can occur at one time at one applied potential. It is our hope that the experiments proposed herein will present an exciting opportunity for young students to gain access to beautiful hands-on electrochemical experimentation and complex chemistry concepts.

ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available at https://pubs.acs.org/doi/10.1021/acs.jchemed.4c00778.

> List of materials, instructor's guide, example voltammetry on ECL solution, amperometric i-t curves at all potentials, all images taken during amperometry used for analysis, student guide, all images used for analysis with USB voltage modifier, overlaid intensity vs potential plots, and schematic of concurrent ECL reactions (PDF)

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Author Contributions

[†]BRL and LEK contributed equally to this project.

Notes

The authors declare the following competing financial interest(s): Jeffrey E. Dick and Brady R. Layman have filed 2 provisional patents over this technology.

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