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Laboratory Experiment

Feasibility of Using a Multicompartment Electrochemical Cell for Determination of Solubility Product Constants of Silver Salts from Potentiometric Measurements: A Directed Study in the Undergraduate Physical Chemistry Laboratory

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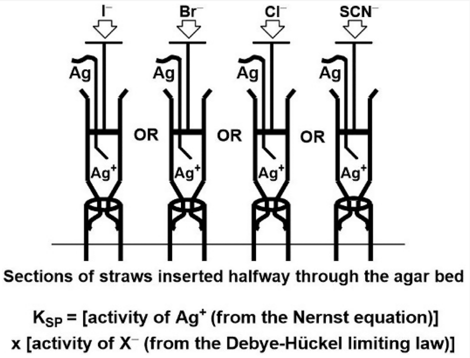
ABSTRACT: The use of a multicompartment electrochemical cell for the potentiometric determination of solubility product constants of aqueous I^-, Br^-, SCN^-, and Cl^- salts of silver is presented. An electrochemical cell presented in this study contained multiple half-cells made of disposable drinking straws, a silver wire electrode housed in a plastic syringe, and a reference electrode. An agreement between the solubility product constant values of AgI, AgBr, AgSCN, and AgCl obtained by the students (7.84×10^{-17}, 4.34×10^{-13}, 9.01×10^{-13}, and 1.44×10^{-10}) and the literature values (8.52×10^{-17}, 5.35×10^{-13}, 1.03×10^{-12}, and 1.77×10^{-10}, respectively) underscored the feasibility of using the cell for the determination of solubility product constants.



KEYWORDS: *Upper-Division Undergraduate, Analytical Chemistry, Physical Chemistry, Hands-On Learning/Manipulatives, Electrochemistry, Potentiometry*

INTRODUCTION

Solubility equilibria of sparingly soluble ionic compounds are an important aspect of undergraduate chemistry curriculum. Solubility rules broadly predict the solubilities of ionic compounds in water as 'soluble' or 'insoluble'. However, solubility product constants of insoluble or sparingly soluble ionic compounds cover a range of many orders of magnitude.[1] The knowledge of the solubilities of the ionic compounds is crucially important in both qualitative and quantitative chemical analysis.[2] The solubility product constant determines the amount of an ionic compound that has dissolved in water. For instance, an equilibrium between the solid and the ions formed by a sparingly soluble salt such as silver iodide is presented in form of a net ionic equation (eq 1) as

$$\mathrm{AgI\ (s)} \rightleftharpoons \mathrm{Ag^+\ (aq)} + \mathrm{I^-\ (aq)} \tag{1}$$

The thermodynamic equilibrium between the undissolved solid and the ions in an aqueous medium is presented (eq 2) as

$$K_{SP} = a_{Ag^+} \times a_{I^-} \tag{2}$$

In this equation, K_{SP}, a_{Ag^+}, and a_{I^-} represent the solubility product constant of AgI and the *activities* of aqueous Ag^+ and I^- ions, respectively. One way of quantifying the activity of silver ions in an aqueous medium is assembling an electrochemical cell and monitoring the electromotive force (*emf*) of the cell. An electrochemical cell can be assembled as shown in the following cell notation:

$$\mathrm{Ag\ (s)|Ag^+\ (aq),\ \ I^-\ (aq)||sat.\ \ KCl\ (aq)|SCE}$$

In this notation, Ag (s) represents a silver wire dipped in a solution containing Ag^+ (aq) and I^- (aq), the first vertical line represents a phase boundary, the double vertical lines represent a salt bridge connecting two half−cell compartments, SCE represents a KCl−saturated calomel reference electrode, and the second vertical line represents a contact between the reference electrode and sat. KCl (aq).

The electromotive force (*emf*) of this cell is (eq 3)

$$E_{\mathrm{cell}} = E_{\mathrm{Ag^+}} - E_{\mathrm{reference}} \tag{3}$$

Using the Nernst equation for $E_{\mathrm{Ag^+}}$, eq 4 is obtained:

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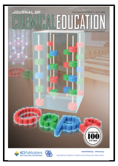

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$$E_{\mathrm{cell}} = \left[E_{\mathrm{Ag}^+}{}^{0} - \frac{RT}{nF}\ln\frac{1}{a_{\mathrm{Ag}^+}}\right] - E_{\mathrm{reference}} \tag{4}$$

Inserting the values[2] of $E_{\mathrm{Ag}^+}{}^{0}$ and $E_{\mathrm{reference}}$ as 0.799 and 0.244 V (both versus the standard hydrogen electrode), respectively, and $\frac{RT}{nF}$ (at 25.0 °C temperature and for the transfer of 1 mol of electrons) as 0.0257 V, we obtain eq 5:

$$E_{\mathrm{cell}} = \left[0.799 - 0.0257\ln\frac{1}{a_{\mathrm{Ag}^+}}\right] - 0.244 \tag{5}$$

Eq 5 rearranges to an exponential form (eq 6):

$$a_{\mathrm{Ag}^+} = e^{E_{\mathrm{cell}} - 0.555/0.0257} \text{ (at 25.0 °C)} \tag{6}$$

Assembling an electrochemical cell as presented in the cell notation and measuring the cell *emf* on a digital voltmeter facilitate the quantification of a_{Ag^+}.

For an MX type of ionic compound (such as AgI), the ionic strength of a solution equals its molality. For a dilute aqueous solution, the molality of a solution is approximately equal to its molarity. Applying the Debye−Hückel limiting law, the mean activity coefficient $\gamma_\pm$ and the activity of the I^- (aq), i.e., a_{I^-} can be quantified from eqs 7 and 8, respectively:[3,4]

$$\log \gamma_\pm = -|z_+ z_-| A\sqrt{(\textit{ionic strength})} \tag{7}$$

$$a_{\mathrm{I}^-} = (\textit{molality})\gamma_\pm \tag{8}$$

In eq 7, z_+, z_-, and A represent the charge numbers on the ions (+1 for cation and − 1 for anions in this work), and a constant factor equal to 0.509 at 25.0 °C, respectively. Using eq 2, the solubility product constant can then be determined from the activities of Ag^+ (aq) and I^- (aq).

Indeed, various reports highlight the potentiometric measurements of solubility products of silver salts and their applicability as an undergraduate laboratory experiment.[5−12] Typically a potentiometric cell includes a silver wire that monitors the activity of Ag^+ (aq). The indicator and the reference electrodes are placed in two beakers separated by a salt bridge. This potentiometric cell requires reassembling of the silver wire half-cell and cleaning of the salt bridge before performing the measurement of a different silver salt or Ag^+ (aq) concentration. Also, agar−electrolyte salt bridges easily dehydrate, and cleaning and refilling of agar−electrolyte mixture in a U-shaped glass tube can be difficult.

In this article, we present the feasibility of using a multicompartment potentiometric cell. The cell consists of an agar−electrolyte bed serving as a salt bridge and pieces of disposable drinking straws inserted in the bed serving as miniature half-cell compartments (Figure 1). A silver wire was housed in a disposable plastic syringe serving as a reservoir for an analyte solution. A syringe containing desired analyte was moved from compartment to compartment to facilitate the multiple *emf* measurements. Easy maneuvering of syringe from well to well enables the prompt replication of the *emf* measurements trials. The multicompartment cell presented in this article was developed by student authors (H.O. and K.S.) as supervised undergraduate research projects, and its feasible application as an electrochemical experiment was tested by undergraduate students as an experiment in the physical chemistry laboratory curriculum.

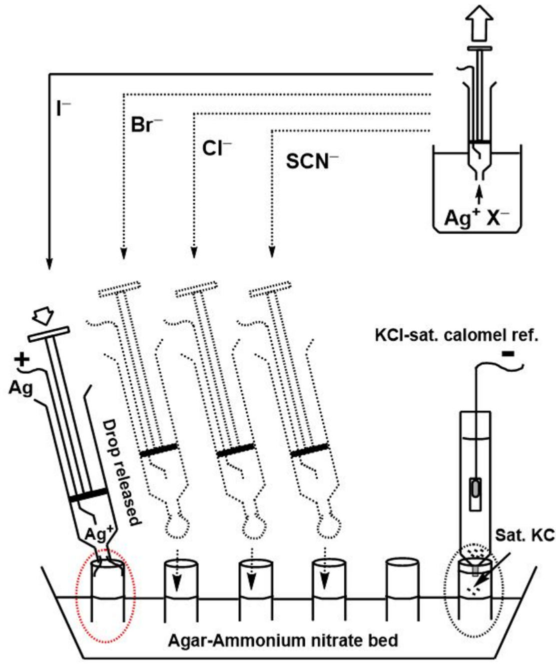

Figure 1. Schematic diagram of the multicompartment potentiometric cell for the *emf* measurement. Four syringes represent four successive potentiometric measurements for four silver salts. Prior to each measurement, the syringe was thoroughly rinsed and loaded with the desired analyte solution.

■ PEDAGOGICAL LEARNING OBJECTIVES

- Setup an electrochemical cell for the potentiometric measurements
- Perform potentiometric measurements for the aqueous solutions of I^-, Br^-, Cl^-, and SCN^- salts of silver
- Apply the Nernst equation to determine the activity of cations in an aqueous medium
- Apply the Debye−Hückel limiting law to determine the activities of anions in an aqueous medium

■ EXPERIMENTAL SECTION

Materials

Silver wire (0.8 mm diameter), KCl-saturated calomel (or Ag/AgCl) reference electrode, 12 mm diameter disposable drinking straws (e.g., smoothie straws), rectangular glass food storage container (about 12 cm × 7 cm at the base), a 3-mL plastic syringe (barrel and plunger), digital multimeter, and jumper leads.

Reagents and Chemicals

Agar, ammonium nitrate, sodium or potassium halides and thiocyanate, and silver nitrate.

Preparation of 'Salt Bridge' Bed

Approximately 8 g of ammonium nitrate and 2.5 g of agar were placed in a 150-mL beaker and deionized water was added to reach the 100-mL mark. The mixture was constantly stirred and heated until it boiled. The mixture was allowed to cool for about two minutes. About 95 mL of the mixture was poured in a rectangular glass food storage container and completely cooled to form a firm agar bed. A firm agar bed formed in about 35 min. The depth of the agar bed was about 1.5 cm.

Preparation of Silver Electrode

Silver wire was spot welded to a copper wire for the connection to the digital millivolt meter. A hole was drilled through the seal of the plunger, and the silver wire was inserted vertically through the seal. About 8 mm length of wire protruded from the tip of the seal. An epoxy was used to firmly hold the silver wire and prevent the leakage through the seal. Silver wire was polished with a sandpaper (grit number 240) and thoroughly cleaned with deionized water prior to each set of potentiometric measurements.

Preparation of Analyte Solutions

All solutions were freshly prepared in deionized water. Sodium or potassium salts of halides or thiocyanate were used to prepare the analyte solutions. Salts were dried in an oven (120 °C) for 18 h and cooled in a desiccator prior to weighing on an analytical balance. In a typical set of experiments, 0.050 M stock solution of a salt was prepared. Five drops of aqueous 0.010 M silver nitrate solution were added to the salts before diluting to the 100-mL mark. Mixtures were shaken for 10 min to equilibrate the solid silver salt with the corresponding ions. Diluted salt solutions were prepared in appropriate volumetric flasks by adding deionized water.

Setting up the Electrochemical Cell and *emf* Measurements

A straw was cut in several 2-cm-long sections. These sections served as half-cell compartments (illustrated as red and black dashed circles in Figure 1). A section was inserted halfway through in the agar bed to form a miniature well. Three drops of ammonium nitrate−agar mixture were added to the well and the layer allowed to cool. This additional layer of agar helped prevent the leakage of the contents of the well. A few drops of saturated KCl were added to the well, and the reference electrode was gently inserted in the solution. The reference electrode was connected to the negative terminal (black lead) of the digital multimeter. Another section of the straw was inserted in the agar, three drops of ammonium nitrate−agar mixture were added, and the additional layer allowed to cool. The new well formed by this section of straw served as a half-cell for the desired analyte of silver salt. Illustrative steps for assembling the cell and a picture of a few components of the cell are presented in Figures S1 and S2 in the Supporting Information section 1, respectively.

Measurement of *emf* for Different X^-

The iodide analyte solution was pulled in the syringe to rinse and the process was repeated three times. The syringe was loaded with approximately 2 mL of the iodide analyte ensuring that the silver wire is in contact with the solution. The silver electrode was connected to the positive terminal (red lead) of the digital multimeter. A few drops of iodide analyte solution were released from the syringe to partially fill the well. Gentle pressure was applied to barely release a new drop to form a contact with the iodide solution in the well and the syringe was steadily held during measurements. The internal connection to the reference electrode was established through the agar bed. The *emf* was recorded after about 60 s. The syringe was steadily held during this period to maintain the contact with the solution in the well. The rest period helped minimize the fluctuations in the *emf*. If the value continued to fluctuate excessively, the *emf* was recorded after a longer rest period. A clamp can also be used to hold the syringe in place. After completing desired sets of trials of the iodide, a new set of trials was performed for other analytes. As explained earlier, for each trial, a new section of straw was inserted in agar to form a new half-cell. The syringe was thoroughly rinsed prior to loading with the new analyte solution. The agar bed area available in the container was large enough to accommodate at least 12 wells serving as half-cells, in addition to the common well serving as a reference electrode cell compartment. For each set of trials, the temperature of the analyte solution placed in the well was recorded and the factor $\frac{RT}{nF}$ (eqs 4−6) was determined.

■ HAZARDS

Approved safety glasses and gloves must be worn while performing the experiment. Safety data sheets must be referred prior to handling reagents and material. Saturated calomel electrode contains mercury in the enclosed form. Mercury is acutely toxic. Alternatively, Ag/AgCl reference electrode can also be used. Pointed objects such as electrodes must be handled with appropriate care. Labeled waste containers must be used to collect the waste chemicals. Agar can be dissolved in warm water and collected in a waste container.

■ RESULTS

Solubility Product Constants of AgX

Experimental data and results obtained by the student authors for the solubility product constants for I^-, Br^-, Cl^-, and SCN^- are presented in Tables 1 and 2.

Table 1. Typical Set of Experimental Data and Results for Solubility Product Constants of AgX[a]

Analyte (X^-)	Experimentally measured and calculated properties	Experimental K_{SP}	Literature[1] K_{SP}
I^- (aq)	measured *emf* = −0.298 V vs SCE activity of Ag^+ (aq) (a_{Ag^+}) = 2.16×10^{-15} (eq 6)	8.32×10^{-17}	8.52×10^{-17}
Br^- (aq)	measured *emf* = −0.0777 V vs SCE activity of Ag^+ (aq) (a_{Ag^+}) = 1.33×10^{-11} (eq 6)	5.12×10^{-13}	5.35×10^{-13}
Cl^- (aq)	measured *emf* = 0.0615 V vs SCE activity of Ag^+ (aq) (a_{Ag^+}) = 3.28×10^{-9} (eq 6)	1.26×10^{-10}	1.77×10^{-10}
SCN^- (aq)	measured *emf* = −0.0607 V vs SCE activity of Ag^+ (aq) (a_{Ag^+}) = 2.60×10^{-11} (eq 6)	1.00×10^{-12}	1.03×10^{-12}

[a] Common experimental properties: analyte concentration = 0.0500 molal, ionic strength = 0.0500 molal (being MX type electrolyte), mean activity coefficient ($\gamma_\pm$) = 0.770 (eq 7), activity of X^- (aq) (a_{X^-}) = 0.0385 (eq 8), temperature = 20.0 °C (293.15 K), $\frac{RT}{nF}$ = 0.02526 V.

Measurement of *emf* for Varying Initial Molalities of Cl^- (aq)

In an independent set of experiments, student authors measured the *emf* of the half-cell containing different concentrations of Cl^- (aq). Experimental data and results for the solubility product constants for this set of experiments are presented in Table 2.

Experimental Results Obtained by Undergraduate Physical Chemistry Laboratory Class

Students from the Physical Chemistry 2 laboratory class tested the multicompartment cell presented in this article. Four students performed the trials of the experiment. They prepared reagents, set up the cell, independently performed the *emf*

Table 2. Typical Set of Experimental Data and Results for Solubility Product Constants of AgCl for Varying Initial Concentrations of Cl^- (aq)[a]

Initial molality of Cl^-	Experimentally measured and calculated properties	Experimental K_{SP}
0.0500 *m*	measured *emf* = 0.0613 V vs SCE activity of Ag^+ (aq) (a_{Ag^+}) = 3.47×10^{-9} (eq 6) mean activity coefficient ($\gamma_\pm$) = 0.770 (eq 7) activity of X^- (aq) (a_{X^-}) = 0.0385 (eq 8)	1.34×10^{-10}
0.0250 *m*	measured *emf* = 0.0800 V vs SCE activity of Ag^+ (aq) (a_{Ag^+}) = 7.26×10^{-9} (eq 6) mean activity coefficient ($\gamma_\pm$) = 0.831 (eq 7) activity of X^- (aq) (a_{X^-}) = 0.0208 (eq 8)	1.51×10^{-10}
0.0125 *m*	measured *emf* = 0.0917 V vs SCE activity of Ag^+ (aq) (a_{Ag^+}) = 1.15×10^{-8} (eq 6) mean activity coefficient ($\gamma_\pm$) = 0.877 (eq 7) activity of X^- (aq) (a_{X^-}) = 0.0110 (eq 8)	1.27×10^{-10}

[a] Common experimental properties: analyte Cl^-(aq), temperature = 21.0 °C (294.15 K), $\frac{RT}{nF}$ = 0.02535 V.

measurements, and completed the calculations in a 3-h laboratory session. A summary of the results on the experimentally determined solubility product constants is presented in Table 3.

In a supplementary set of experiments, students measured the *emf* of the half-cell containing different initial concentrations of Br^- (aq). A summary of the results for this set of experiments is presented in Table 4.

DISCUSSION

Quantitative Features

The quantities of solubility product constants for the silver salts of iodide, bromide, chloride, and thiocyanate (Table 1) determined by the student authors (8.32×10^{-17}, 5.12×10^{-13}, 1.26×10^{-10}, and 1.00×10^{-12}) were consistent with the literature values (8.52×10^{-17}, 5.35×10^{-13}, 1.77×10^{-10}, and 1.03×10^{-12}, respectively). The measured *emf* of the cell (Table 2) increased (0.0613 V, 0.0800 V, and 0.0917 V) with the decreased molality of chloride (0.0500 *m*, 0.0250 *m*, and 0.0125 *m*, respectively). The increasing *emf* accounted for an increasing activity of Ag^+, compensated the decreasing activity of Cl^- (aq), and maintained a relatively stable value of the solubility product constant of AgCl. The solubility product constants (Table 3) for the silver salts of iodide, bromide, chloride, and thiocyanate (as 7.84×10^{-17}, 4.34×10^{-13}, 1.44×10^{-10}, and 9.01×10^{-13}, respectively) determined by the Physical Chemistry 2 laboratory class were also consistent with the literature values. Relatively small coefficient of variation (<10%), with the exception of I^- (aq), indicated moderate dispersion in the measurement of *emf*. The solubility product constants obtained by the students (Table 4) for varying initial concentrations of Br^- (aq) (5.82×10^{-13}, 4.22×10^{-13}, 4.79×10^{-13}) agreed as well with the literature value of 5.35×10^{-13} for AgBr.

Table 4. Typical Set of Results for Solubility Product Constants of AgBr for Varying Initial Concentrations of Br^- (aq)

Initial molality of Br^- (aq)	Experimental K_{SP}
0.100 *m*	5.82×10^{-13}
0.050 *m*	4.22×10^{-13}
0.0250 *m*	4.79×10^{-13}

Practical Variations and Errors

The desired mass of a salt was dissolved in deionized water and diluted to a certain volume and we regarded the solution concentration as ‘molality’. The *emf* value monitored by the digital voltmeter fluctuated within about ±10%. For instance, a ±10% variation in measured *emf* of − 0.0607 V resulted in the variation in the solubility product constant of AgSCN from 7.86×10^{-13} to 1.27×10^{-12} (or ${}_pK_{SP}$ varied from 12.105 to 11.896). We used the Debye−Hückel limiting law[3] to determine the mean activity coefficients of the anions. The Debye−Hückel limiting law is more consistent with the experimental results in dilute electrolytes. Mean activity coefficient values tend to deviate from the experimental values at relatively higher concentrations.[13] The Debye−Hückel limiting law contains the factor *A*, which is calculated to be 0.509 at 25.0 °C. Deviations in temperature of the electrolyte quantitatively alter the value of this factor. Literature values indicate about 1% drop in the factor determined[1] at 20.0 °C. Likewise, variations in the temperature affect the reference electrode potential[2] and the quantitative relation (eq 5) between E_{cell} and a_{Ag^+}. Other studies[14,15] indicate quantitative deviations in the solubility equilibrium of silver chloride resulting from the formation of competing $AgCl_2^-$ (aq) ions. As indicated on the assay label, potassium chloride contained 0.002% and 0.01% iodide and bromide, respectively. These insignificant activities of interfering halides did not have any measurable effect on the quantitative assessment of chloride and the solubility product constant of silver chloride. Similarly, the distance between the reference electrode and analyte half-cells did not have any measurable effect on the *emf*.

Salient Features of the Multicompartment Cell

- Multiple compartments for different analytes or varying analyte concentrations can be created by simply inserting a new piece of straw in the agar−electrolyte bed.
- Miniature form of the cell saves the volumes (<3 mL) of analytes.
- Cell compartments and the agar−electrolyte bed are disposable.

Table 3. Typical Sets of Results for Solubility Product Constants of AgX

Analyte (0.0500 *m*)	Trial 1	Trial 2	Trial 3	Trial 4	Average K_{SP}	Coefficient of variation in K_{SP} (%) (*n* = 4)
I^- (aq)	8.32×10^{-17}	7.69×10^{-17}	6.06×10^{-17}	9.29×10^{-17}	7.84×10^{-17}	17.3
Br^- (aq)	4.92×10^{-13}	4.15×10^{-13}	4.07×10^{-13}	4.20×10^{-13}	4.34×10^{-13}	9.10
Cl^- (aq)	1.40×10^{-10}	1.50×10^{-10}	1.43×10^{-10}	1.44×10^{-10}	1.44×10^{-10}	2.82
SCN^- (aq)	8.96×10^{-13}	8.75×10^{-13}	8.96×10^{-13}	9.35×10^{-13}	9.01×10^{-13}	2.75

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- Easy maneuver of syringe from well to well enables prompt replication of trials of *emf* measurements.

Post-Laboratory Exercise

The post-laboratory exercise (see Supporting Information section 1) included numerical problems based on the quantitative relations between the measured *emf* and the activities of the ions. Four students who performed the trials of the experiment participated in the post-laboratory exercise. All students correctly solved the first numerical problem. In the second exercise, steps for the numerical calculation of *emf* were explained to students. In the third exercise, all students correctly determined the ion expected to precipitate out first form the mixture of halides.

■ CONCLUSIONS

As the Le Chatelier's principle indicates, when the concentration of the *common ion* is increased, the solubility equilibrium tends to minimize the change by shifting the equilibrium (as presented in eq 1) to the left. As a result, the solubility of the original salt can be expected to decrease.[3,16] Using ammonium nitrate (which has no ion in common with ions in the equilibrium) as a salt for the salt bridge helped eliminate the common ion effect on the solubility equilibrium. On the other hand, the *diverse ions* increase the solubility of silver salts.[17] Relatively high concentration of ammonium nitrate (1 M) in the agar bed can raise the ionic strength of silver salt solution in contact with the silver electrode. However, the silver electrode housed in a syringe helped minimize the effect of diverse ions (NH_4^+ and NO_3^-) leaking from the agar bed. Furthermore, a slight pressure applied to the syringe released a drop of electrolyte in the well and prevented the flow of (diverse) ions backward to the surface of the silver electrode.

Preparing the reagents, setting up the cell, performing potentiometric measurements, determining the solubility product constants, and completing the post-laboratory exercise can be accomplished in a 3-h laboratory session. The learning objectives of ionic activities, solubility equilibrium, the Nernst equation, and the Debye−Hückel limiting law are in line with the undergraduate physical chemistry and quantitative analysis course curriculum.[3,18] Undergraduate research allows students to integrate and reinforce chemistry knowledge from their formal course work.[19] Research experience gained by student authors (H.O. and K.S.) in developing and testing the cell presented in this work supplemented their formal course work. The regularity in the experimental results presented in Tables 1−4 underscores the practicality of using the multicompartment cell for monitoring the solubility product constants in an undergraduate laboratory. Determination of solubility product constant by potentiometric method has been known to students and educators for a long time. The potentiometric method presented in this article does not offer a new technique for determination of solubility product constant, rather it offers new practical learning experience with an adaptable use of a multicompartment electrochemical cell.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available at https://pubs.acs.org/doi/10.1021/acs.jchemed.2c01251.

Illustrative figures, instructions for students, and post−laboratory exercise questions (PDF, DOCX)

Notes for instructors, CAS numbers for chemicals and reagents, and key to the post−laboratory exercise questions (PDF, DOCX)

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Notes

The authors declare no competing financial interest.

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