In this chapter we introduced three electrochemical methods of analysis: potentiometry, coulometry, and voltammetry. In potentiometry we measure the potential of an indicator electrode without allowing any significant current to pass through the electrochemical cell. In principle we can use the Nernst equation to calculate the analyte’s activity—junction potentials, however, require that we standardize the electrode.

There are two broad classes of potentiometric electrodes: metallic electrodes and membrane electrodes. The potential of a metallic electrode is the result of a redox reaction at the electrode’s surface. An electrode of the first kind responds to the concentration of its cation in solution; thus, the potential of a Ag wire is determined by the activity of Ag⁺ in solution. If another species is in equilibrium with the metal ion, the electrode’s potential also responds to the concentration of that species. For example, the potential of a Ag wire in a solution of Cl⁻ responds to the concentration of Cl⁻ because the relative concentrations of Ag⁺ and Cl⁻ are fixed by the solubility product for AgCl. We call this an electrode of the second kind.

The potential of a membrane electrode is determined by a difference in the composition of the solution on each side of the membrane. Electrodes using a glass membrane respond to ions that bind to negatively charged sites on the membrane’s surface. A pH electrode is one example of a glass membrane electrode. Other kinds of membrane electrodes include those using insoluble crystalline solids or liquid ion-exchangers incorporated into a hydrophobic membrane. The F⁻ ion-selective electrode, which uses a single crystal of LaF₃ as the ion-selective membrane, is an example of a solid-state electrode. The Cu²⁺ ion-selective electrode, in which the chelating di-(n-decyl)phosphate is immobilized in a PVC membrane, is an example of a liquid-based ion-selective electrode.

Potentiometric electrodes can be designed to respond to molecules by using a chemical reaction that produces an ion whose concentration can be determined using a traditional ion-selective electrode. A gas-sensing electrode, for example, include a gas permeable membrane that isolates the ion-selective electrode from the gas. When the gas diffuses across the membrane it alters the composition of the inner solution, which is monitored with an ion-selective electrode. An enzyme electrodes operate in the same way.

Coulometric methods are based on Faraday’s law that the total charge or current passed during an electrolysis is proportional to the amount of reactants and products in the redox reaction. If the electrolysis is 100% efficient—that is, that only the analyte is oxidized or reduced—then we can use the total charge or current to determine the amount of analyte in a sample. In controlled-potential coulometry we apply a constant potential and measure the resulting current as a function of time. In controlled-current coulometry the current is held constant and we measure the time required to completely oxidize or reduce the analyte.

In voltammetry we measure the current in an electrochemical cell as a function of the applied potential. There are several different voltammetric methods that differ in terms of the type of working electrode, how we apply the potential, and whether we include convection (stirring) as a means for transporting of material to the working electrode.

Polarography is a voltammetric technique that uses a mercury electrode and an unstirred solution. Normal polarography uses a dropping mercury electrode, or a static mercury drop electrode, and a linear potential scan. Other forms of polarography include normal pulse polarography, differential pulse polarography, staircase polarography, and square-wave polarography, all of which use a series of potential pulses.

In hydrodynamic voltammetry the solution is stirred using either a magnetic stir bar or by rotating the electrode. Because the solution is stirred a dropping mercury electrode can not be used; instead we use a solid electrode. Both linear potential scans and potential pulses can be applied.

In stripping voltammetry the analyte is first deposited on the electrode, usually as the result of an oxidation or reduction reaction. The potential is then scanned, either linearly or by using potential pulses, in a direction that removes the analyte by a reduction or oxidation reaction.

Amperometry is a voltammetric method in which we apply a constant potential to the electrode and measure the resulting current. Amperometry is most often used in the construction of chemical sensors for the quantitative analysis of single analytes. One important example is the Clark O₂ electrode, which responds to the concentration of dissolved O₂ in solutions such as blood and water.

1. **11E.1 Key Terms**
2. **11E.2 Problems**
3. **11E.3 Solutions to Practice Exercises**
   3.1. Practice Exercise 11.1
   3.2. Practice Exercise 11.2
   3.3. Practice Exercise 11.3
   3.4. Practice Exercise 11.4
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11E.1 Key Terms

- amalgam
- amperometry
- anode
- anodic current
- asymmetry potential
- auxiliary electrode
- cathode
- cathodic current
- charging current
- controlled-current coulometry
- controlled-potential coulometry
- convection
- coulometric titrations
- coulometry
- counter electrode
- current efficiency
- cyclic voltammetry
- diffusion
- diffusion layer
- dropping mercury electrode
- electrical double layer
- electrochemically irreversible
- electrochemically reversible
- electrode of the first kind
- electrode of the second kind
- electrochemistry
- electrogravimetry
- enzyme electrodes
- faradaic current
- Faraday's law
- galvanostat
- gas-sensing electrode
- glass electrode
- hanging mercury drop electrode
- hydrodynamic voltammetry
- indicator electrode
- ionophore
- ion selective electrode
- junction potential
- limiting current
- liquid-based ion-selective electrode
- mass transport
- mediator
- membrane potential
- mercury film electrode
- migration
- nonfaradaic current
- Ohm's law
- overpotential
- peak current
- polarography
- potentiometer
- potentiostat
- pulse polarography
- redox electrode
- reference electrode
- residual current
- salt bridge
- saturated calomel electrode
- selectivity coefficient
- silver/silver chloride electrode
- solid-state ion-selective electrodes
- standard hydrogen electrode
- static mercury drop electrode
- stripping voltammetry
- total ionic strength adjustment buffer
- voltammetry
- voltammogram
- working electrode

11E.2 Problems

1. Identify the anode and cathode for the following electrochemical cells, and write the oxidation or reduction reaction at each electrode.
   a. Pt $|$ FeCl$_2$(aq,0.015),FeCl$_3$(aq,0.045) $|$ AgNO$_3$(aq,0.1) $|$ Ag
   b. Ag $|$ AgBr(s),NaBr(aq,1.0) $|$ CdCl$_2$(aq,0.05) $|$ Cd
   c. Pb $|$ PbSO$_4$(s),H$_2$SO$_4$(aq,1.5) $|$ H$_2$SO$_4$(aq,2.0),PbSO$_4$(s) $|$ PbO$_2$

2. Calculate the potential for the electrochemical cells in problem 1. The values in parentheses are the activities of the associated species.

3. Calculate the activity of KI, $x$, in the following electrochemical cell if the potential is +0.294 V
   Ag $|$ AgCl(s),NaCl(aq,0.1) $|$ KI(aq,$x$),I$_2$(s) $|$ Pt

4. What reaction prevents us from using Zn as an electrode of the first kind in acidic solutions? Which other electrodes of the first kind would you expect to behave in the same manner as Zn when immersed in an acidic solution?

5. Creager and colleagues designed a salicylate ion-selective electrode using a PVC membrane impregnated with tetraalkylammonium salicylate. To determine the ion-selective electrode’s selectivity coefficient for benzoate, they prepared a set of salicylate calibration standards in which the concentration of benzoate was held constant at 0.10 M. Using the following data, determine the value of the selectivity coefficient.

<table>
<thead>
<tr>
<th>salicylate (M)</th>
<th>potential (mV)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.0</td>
<td>20.2</td>
</tr>
<tr>
<td>1.0×10$^{-1}$</td>
<td>73.5</td>
</tr>
<tr>
<td>1.0×10$^{-2}$</td>
<td>126</td>
</tr>
<tr>
<td>1.0×10$^{-3}$</td>
<td>168</td>
</tr>
<tr>
<td>1.0×10$^{-4}$</td>
<td>182</td>
</tr>
<tr>
<td>1.0×10$^{-5}$</td>
<td>182</td>
</tr>
<tr>
<td>1.0×10$^{-6}$</td>
<td>177</td>
</tr>
</tbody>
</table>

What is the maximum acceptable concentration of benzoate if you plan to use this ion-selective electrode to analyze samples containing as little as 10$^{-5}$ M salicylate with an accuracy of better than 1%?

6. Watanabe and co-workers described a new membrane electrode for the determination of cocaine, a weak base alkaloid with a $pK_a$ of 8.64. The electrode’s response for a fixed concentration of cocaine is independent of pH in the range of 1–8, but decreases sharply above a pH of 8. Offer an explanation for this pH dependency.

7. Figure 11.20 shows a schematic diagram for an enzyme electrode that responds to urea by using a gas-sensing NH$_3$ electrode to measure the amount of ammonia released following the enzyme’s reaction with urea. In turn, the NH$_3$ electrode uses a pH electrode to monitor the change in pH due to the ammonia. The response of the urea electrode is given by equation 11.14. Beginning with equation 11.11, which gives the potential of a pH electrode, show that equation 11.14 for the urea electrode is correct.

8. Explain why the response of an NH$_3$-based urea electrode (Figure 11.20 and equation 11.14) is different from the response of a urea electrode in which the enzyme is coated on the glass membrane of a pH electrode (Figure 11.21 and equation 11.15).
9. A potentiometric electrode for HCN uses a gas-permeable membrane, a buffered internal solution of 0.01 M KAg(CN)₂, and a Ag₂S ISE electrode that is immersed in the internal solution. Consider the equilibrium reactions taking place within the internal solution, and derive an equation relating the electrode’s potential to the concentration of HCN in the sample.

10. Mifflin and associates described a membrane electrode for the quantitative analysis of penicillin in which the enzyme penicillinase is immobilized in a polyacrylamide gel coated on the glass membrane of a pH electrode. The following data were collected using a set of penicillin standards.

<table>
<thead>
<tr>
<th>[penicillin] (M)</th>
<th>potential (mV)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.0×10⁻²</td>
<td>220</td>
</tr>
<tr>
<td>2.0×10⁻³</td>
<td>204</td>
</tr>
<tr>
<td>1.0×10⁻³</td>
<td>190</td>
</tr>
<tr>
<td>2.0×10⁻⁴</td>
<td>153</td>
</tr>
<tr>
<td>1.0×10⁻⁴</td>
<td>135</td>
</tr>
<tr>
<td>1.0×10⁻⁵</td>
<td>96</td>
</tr>
<tr>
<td>1.0×10⁻⁶</td>
<td>80</td>
</tr>
</tbody>
</table>

(a) Over what range of concentrations is there a linear response?
(b) What is calibration curve’s equation for this concentration range?
(c) What is the concentration of penicillin in a sample that yields a potential of 142 mV?

11. An ion-selective electrode can be placed in a flow cell into which we inject samples or standards. As the analyte passes through the cell, a potential spike is recorded instead of a steady-state potential. The concentration of K⁺ in serum has been determined in this fashion using standards prepared in a matrix of 0.014 M NaCl.

<table>
<thead>
<tr>
<th>[K⁺] (mM)</th>
<th>potential (arb. units)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.1</td>
<td>25.5</td>
</tr>
<tr>
<td>0.2</td>
<td>37.2</td>
</tr>
<tr>
<td>0.4</td>
<td>50.8</td>
</tr>
<tr>
<td>0.6</td>
<td>58.7</td>
</tr>
<tr>
<td>0.8</td>
<td>64.0</td>
</tr>
<tr>
<td>1.0</td>
<td>66.8</td>
</tr>
</tbody>
</table>

A 1.00-mL sample of serum is diluted to volume in a 10-mL volumetric flask and analyzed, giving a potential of 51.1 (arbitrary units). Report the concentration of K⁺ in the sample of serum.

12. Wang and Taha described an interesting application of potentiometry, which they call batch injection. As shown in Figure 11.56, an ionselective electrode is placed in an inverted position in a large volume tank, and a fixed volume of a sample or a standard solution is injected toward the electrode’s surface using a micropipet. The response of the electrode is a spike in potential that is proportional to the analyte’s concentration. The following data were collected using a pH electrode and a set of pH standards.

<table>
<thead>
<tr>
<th>pH</th>
<th>potential (mV)</th>
</tr>
</thead>
<tbody>
<tr>
<td>2.0</td>
<td>+300</td>
</tr>
<tr>
<td>3.0</td>
<td>+240</td>
</tr>
<tr>
<td>4.0</td>
<td>+168</td>
</tr>
<tr>
<td>5.0</td>
<td>+81</td>
</tr>
<tr>
<td>6.0</td>
<td>+35</td>
</tr>
<tr>
<td>8.0</td>
<td>−92</td>
</tr>
<tr>
<td>9.0</td>
<td>−168</td>
</tr>
<tr>
<td>10.0</td>
<td>−235</td>
</tr>
<tr>
<td>11.0</td>
<td>−279</td>
</tr>
</tbody>
</table>

Determine the pH of the following samples given the recorded peak potentials: tomato juice, 167 mV; tap water, −27 mV; coffee, 122 mV.
13. The concentration of NO$_3^-$ in a water sample is determined by a one-point standard addition using a NO$_3^-$ ion-selective electrode. A 25.00-mL sample is placed in a beaker and a potential of 0.102 V is measured. A 1.00-mL aliquot of a 200.0-mg/L standard solution of NO$_3^-$ is added, after which the potential is 0.089 V. Report the mg NO$_3^-$/L in the water sample.

14. In 1977, when I was an undergraduate student at Knox College, my lab partner and I did an experiment to determine the concentration of fluoride in tap water and the amount of fluoride in toothpaste. The data in this problem comes from my lab notebook.

(a) To analyze tap water, we took three 25.0-mL samples and added 25.0 mL of TISAB to each. We measured the potential of each solution using a F$^-$ ISE and an SCE reference electrode. Next, we made five 1.00-mL additions of a standard solution of 100.0 ppm F$^-$ to each sample, and measure the potential after each addition.

<table>
<thead>
<tr>
<th>mL of standard added</th>
<th>sample 1</th>
<th>sample 2</th>
<th>sample 3</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.00</td>
<td>–79</td>
<td>–82</td>
<td>–81</td>
</tr>
<tr>
<td>1.00</td>
<td>–119</td>
<td>–119</td>
<td>–118</td>
</tr>
<tr>
<td>2.00</td>
<td>–133</td>
<td>–133</td>
<td>–133</td>
</tr>
<tr>
<td>3.00</td>
<td>–142</td>
<td>–142</td>
<td>–142</td>
</tr>
<tr>
<td>4.00</td>
<td>–149</td>
<td>–148</td>
<td>–148</td>
</tr>
<tr>
<td>5.00</td>
<td>–154</td>
<td>–153</td>
<td>–153</td>
</tr>
</tbody>
</table>

Report the parts-per-million of F$^-$ in the tap water.

(b) To analyze the toothpaste, we measured 0.3619 g into a 100-mL volumetric flask, added 50.0 mL of TISAB, and diluted to volume with distilled water. After ensuring that the sample was thoroughly mixed, we transferred three 20.0-mL portions into separate beakers and measured the potential of each using a F$^-$ ISE and an SCE reference electrode. Next, we made five 1.00-mL additions of a standard solution of 100.0 ppm F$^-$ to each sample, and measured the potential after each addition.

<table>
<thead>
<tr>
<th>mL of standard added</th>
<th>sample 1</th>
<th>sample 2</th>
<th>sample 3</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.00</td>
<td>–55</td>
<td>–54</td>
<td>–55</td>
</tr>
<tr>
<td>1.00</td>
<td>–82</td>
<td>–82</td>
<td>–83</td>
</tr>
<tr>
<td>2.00</td>
<td>–94</td>
<td>–94</td>
<td>–94</td>
</tr>
<tr>
<td>3.00</td>
<td>–102</td>
<td>–103</td>
<td>–102</td>
</tr>
<tr>
<td>4.00</td>
<td>–108</td>
<td>–108</td>
<td>–109</td>
</tr>
<tr>
<td>5.00</td>
<td>–112</td>
<td>–112</td>
<td>–113</td>
</tr>
</tbody>
</table>

Report the parts-per-million F$^-$ in the toothpaste.

15. You are responsible for determining the amount of KI in iodized salt and decide to use an I$^-$ ion-selective electrode. Describe how you would perform this analysis using external standards and using the method of standard additions.

16. Explain why each of the following decreases the analysis time for controlled-potential coulometry: a larger surface area for the working electrode, a smaller volume of solution, and a faster stirring rate.

17. The purity of a sample of picric acid, C$_6$H$_3$N$_3$O$_7$, is determined by controlled-potential coulometry, converting the picric acid to triaminophenol, C$_6$H$_9$N$_3$O.

\[
\begin{align*}
\text{O}_2\text{N} & + 18\text{H}_3\text{O}^+ + 18e^- & \rightarrow & \text{H}_2\text{N} & \text{NH}_2 \rightarrow & +24\text{H}_2\text{O}
\end{align*}
\]
A 0.2917-g sample of picric acid is placed in a 1000-mL volumetric flask and diluted to volume. A 10.00-mL portion of this solution is transferred to a coulometric cell and sufficient water added so that the Pt cathode is immersed. The exhaustive electrolysis of the sample requires 21.67 C of charge. Report the purity of the picric acid.

18. The concentration of H₂S in the drainage from an abandoned mine can be determined by a coulometric titration using KI as a mediator and I₃⁻ as the titrant.

$$\text{H}_2\text{S}(aq) + I_3^-(aq) + 2\text{H}_2\text{O}(l) \rightleftharpoons 2\text{H}_3\text{O}^+(aq) + 3\text{I}^-(aq) + \text{S(s)}$$

A 50.00-mL sample of water is placed in a coulometric cell, along with an excess of KI and a small amount of starch as an indicator. Electrolysis is carried out at a constant current of 84.6 mA, requiring 386 s to reach the starch end point. Report the concentration of H₂S in the sample in parts-per-million.

19. One method for the determination of H₃AsO₃ is a coulometric titration using I₃⁻ as a titrant. The relevant standard-state reactions and potentials are summarized here.

$$\text{H}_3\text{AsO}_4(aq) + 2\text{H}_3\text{O}^+(aq) + 2e^- \rightleftharpoons \text{H}_3\text{AsO}_3(aq) + 3\text{H}_2\text{O}(l) \quad E^0 = +0.559 \text{ V}$$

$$\text{I}_3^-(aq) + 2e^- \rightleftharpoons 3\text{I}^-(aq) \quad E^0 = +0.536 \text{ V}$$

Explain why the coulometric titration must be carried out in a neutral solution (pH = 7) instead of in a strongly acidic solution (pH < 0).

20. The production of adiponitrile, NC(CH₂)₄CN, from acrylonitrile, CH₂=CHCN, is an important industrial process. A 0.594-g sample of acrylonitrile is placed in a 1-L volumetric flask and diluted to volume. An exhaustive controlled-potential electrolysis of a 1.00-mL portion of the diluted acrylonitrile requires 1.080 C of charge. What is the value of n for the reduction of acrylonitrile to adiponitrile?

21. The linear-potential scan hydrodynamic voltammogram for a mixture of Fe²⁺ and Fe³⁺ is shown in Figure 11.57, where iₐ and iₖ are the anodic and cathodic limiting currents.

(a) Show that the potential is given by

$$E = E^0_{\text{Fe}^{3+}/\text{Fe}^{2+}} - 0.05916\log(K_{\text{Fe}^{3+}/\text{Fe}^{2+}}) - 0.05916\log((i - i_\text{a})/(i_\text{k} - i))$$

(b) What is the potential when i = 0 for a solution that is 0.1 mM Fe³⁺ and 0.05 mM Fe²⁺? You may assume that $K_{\text{Fe}^{3+}/\text{Fe}^{2+}} \approx K_{\text{Fe}^{3+}/\text{Fe}^{2+}}$.

![Figure 11.57 Linear-scan hydrodynamic voltammogram for a mixture of Fe²⁺ and Fe³⁺. See Problem 11.21 for more details.](image)

22. The amount of sulfur in aromatic monomers can be determined by differential pulse polarography. Standard solutions are prepared for analysis by dissolving 1.000 mL of the purified monomer in 25.00 mL of an electrolytic solvent, adding a known amount of S, deaerating, and measuring the peak current. The following results were obtained for a set of calibration standards.

<table>
<thead>
<tr>
<th>µg S added</th>
<th>peak current (µA)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>0.14</td>
</tr>
<tr>
<td>28</td>
<td>0.70</td>
</tr>
<tr>
<td>56</td>
<td>1.23</td>
</tr>
<tr>
<td>112</td>
<td>2.41</td>
</tr>
<tr>
<td>168</td>
<td>3.42</td>
</tr>
</tbody>
</table>

Analysis of a 1.000-mL sample, treated in the same manner as the standards, gives a peak current of 1.77 µA. Report the mg S/mL in the sample.

23. The purity of a sample of K₃Fe(CN)₆ was determined using linear-potential scan hydrodynamic voltammetry at a glassy carbon electrode. The following data were obtained for a set of external calibration standards.

<table>
<thead>
<tr>
<th>[K₃Fe(CN)₆] (mM)</th>
<th>limiting current (µA)</th>
</tr>
</thead>
<tbody>
<tr>
<td>2.0</td>
<td>127</td>
</tr>
<tr>
<td>4.0</td>
<td>252</td>
</tr>
<tr>
<td>6.0</td>
<td>376</td>
</tr>
<tr>
<td>8.0</td>
<td>500</td>
</tr>
<tr>
<td>10.0</td>
<td>624</td>
</tr>
</tbody>
</table>

A sample of impure K₃Fe(CN)₆ was prepared for analysis by diluting a 0.246-g portion to volume in a 100-mL volumetric flask. The limiting current for the sample was found to be 444 µA. Report the purity of this sample of K₃Fe(CN)₆.
24. One method for determining whether an individual has recently fired a gun is to look for traces of antimony in the residue collected from the individual’s hands. Anodic stripping voltammetry at a mercury film electrode is ideally suited for this analysis. In a typical analysis a sample is collected from a suspect with a cotton-tipped swab wetted with 5% v/v HNO₃. After returning to the lab, the swab is placed in a vial containing 5.0 mL of 4 M HCl that is 0.02 M in hydrazine sulfate. After allowing the swab to soak overnight, a 4.0-mL portion of the solution is transferred to an electrochemical cell along with 100 mL of 0.01 M HgCl₂. After depositing the thin film of mercury and the antimony, the stripping step gives a peak current of 0.38 μA. After adding a standard addition of 100 μL of 5.00x10⁻² ppb Sb, the peak current increases to 1.14 μA. How many nanograms of Sb were collected from the suspect’s hand?

25. Zinc can be used as an internal standard in the analysis of thallium by differential pulse polarography. A standard solution containing 5.00 x 10⁻⁵ M Zn²⁺ and 2.50 x 10⁻⁵ M Ti⁺ gave peak currents of 5.71 μA and 3.19 μA, respectively. An 8.713-g sample of an alloy known to be free of zinc was dissolved in acid, transferred to a 500-mL volumetric flask, and diluted to volume. A 25.0-mL portion of this solution was mixed with 25.0 mL of a 5.00 x 10⁻⁴ M solution of Zn²⁺. Analysis of this solution gave a peak current for Zn²⁺ of 12.3 μA, and for Ti⁺ of 20.2 μA. Report the %w/w Ti in the alloy.

26. Differential pulse voltammetry at a carbon working electrode can be used to determine the concentrations of ascorbic acid and caffeine in drug formulations. In a typical analysis a 0.9183-g tablet is crushed and ground into a fine powder. A 0.5630-g sample of this powder is transferred to a 100-mL volumetric flask, brought into solution, and diluted to volume. A 0.500-mL portion is then transferred to a voltammetric cell containing 20.00 mL of a suitable supporting electrolyte. The resulting voltammogram gives peak currents of 1.40 μA and 3.88 μA for ascorbic acid and caffeine, respectively. A 0.500-mL aliquot of a standard solution containing 250.0 ppm ascorbic acid and 200.0 ppm caffeine is then added. A voltammogram of this solution gives peak currents of 2.80 μA and 8.02 μA for ascorbic acid and caffeine, respectively. Report the milligrams of ascorbic acid and milligrams of caffeine in the tablet.

27. Ratana-ophas and co-workers described a stripping analysis method for determining the amount of tin in canned fruit juices. Standards containing 50.0 ppb Sn⁴⁺, 100.0 ppb Sn⁴⁺, and 150.0 ppb Sn⁴⁺ were analyzed giving peak currents (arbitrary units) of 83.0, 171.6, and 260.2, respectively. A 2.00-mL sample of lychee juice was mixed with 20.00 mL of 1:1 HCl/HNO₃. A 0.500-mL portion of this mixture was added to 10 mL of 6 M HCl and the volume adjusted to 30.00 mL. Analysis of this diluted sample gave a signal of 128.2 (arbitrary units). Report the parts-per-million Sn⁴⁺ in the original sample of lychee juice.

28. Sittampalam and Wilson described the preparation and use of an amperometric sensor for glucose. The sensor is calibrated by measuring the steady-state current when it is immersed in standard solutions of glucose. A typical set of calibration data is shown here.

<table>
<thead>
<tr>
<th>glucose (mg/100 mL)</th>
<th>current (arb. units)</th>
</tr>
</thead>
<tbody>
<tr>
<td>2.0</td>
<td>17.2</td>
</tr>
<tr>
<td>4.0</td>
<td>32.9</td>
</tr>
<tr>
<td>6.0</td>
<td>52.1</td>
</tr>
<tr>
<td>8.0</td>
<td>68.0</td>
</tr>
<tr>
<td>10.0</td>
<td>85.8</td>
</tr>
</tbody>
</table>

A 2.00-mL sample is diluted to 10 mL in a volumetric flask and a steady-state current of 23.6 (arbitrary units) is measured. What is the concentration of glucose in the sample in mg/100 mL?

29. Differential pulse polarography can be used to determine the concentrations of lead, thallium, and indium in a mixture. Because the peaks for lead and thallium, and for thallium and indium overlap, a simultaneous analysis is necessary. Peak currents (arbitrary units) at −0.385 V, −0.455 V, and −0.557 V were measured for a single standard solution, and for a sample, giving the results shown in the following table.

<table>
<thead>
<tr>
<th>standards</th>
<th>peak currents (arb. units) at analyte</th>
<th>-0.385 V</th>
<th>-0.455 V</th>
<th>-0.557 V</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pb²⁺</td>
<td>1.0</td>
<td>26.1</td>
<td>2.9</td>
<td>0</td>
</tr>
<tr>
<td>Ti⁺</td>
<td>2.0</td>
<td>7.8</td>
<td>23.5</td>
<td>3.2</td>
</tr>
<tr>
<td>In³⁺</td>
<td>0.4</td>
<td>0</td>
<td>0</td>
<td>22.9</td>
</tr>
</tbody>
</table>

Report the μg/mL of Pb²⁺, Ti⁺ and In³⁺ in the sample.

30. Abass and co-workers developed an amperometric biosensor for NH₄⁺ that uses the enzyme glutamate dehydrogenase to catalyze the following reaction

\[ 2\text{-oxoglutarate}(aq) + \text{NH}_4^+(aq) + \text{NADH}(aq) \rightarrow \text{glutamate}(aq) + \text{NAD}^+(aq) + \text{H}_2\text{O}(l) \]

where NADH is the reduced form of nicotinamide adenine dinucleotide. The biosensor actually responds to the concentration of NADH, however, the rate of the reaction depends on the concentration of NH₄⁺. If the initial concentrations of 2-oxoglutarate and NADH are the same for all samples and standards, then the signal is proportional to the concentration of NH₄⁺. As shown in the following table, the sensitivity of the method is dependent on pH.

<table>
<thead>
<tr>
<th>pH</th>
<th>sensitivity (nA μM⁻¹)</th>
</tr>
</thead>
<tbody>
<tr>
<td>6.2</td>
<td>1.67 x 10³</td>
</tr>
<tr>
<td>6.75</td>
<td>5.00x10³</td>
</tr>
<tr>
<td>7.3</td>
<td>9.33 x 10³</td>
</tr>
</tbody>
</table>
Two possible explanations for the effect of pH on the sensitivity of this analysis are the acid–base chemistry of NH₄⁺, and the acid–base chemistry of the enzyme. Given that the pKₐ for NH₄⁺ is 9.244, explain the source of this pH-dependent sensitivity.

31. The speciation scheme for trace metals shown in Table 11.12 divides them into seven operationally defined groups by collecting and analyzing two samples following each of four treatments—a total of eight samples and eight measurements. After removing insoluble particulates by filtration (treatment 1), the solution is analyzed for the concentration of ASV labile metals and for the total concentration of metals. A portion of the filtered solution is passed through an ion-exchange column (treatment 2), and the concentrations of ASV metal and of total metal are determined. A second portion of the filtered solution is irradiated with UV light (treatment 3), and the concentrations of ASV metal and of total metal are measured. Finally, a third portion of the filtered solution is irradiated with UV light and passed through an ion-exchange column (treatment 4), and the concentrations of ASV labile metal and of total metal again are determined. The groups that are included in each measurement are summarized in the following table.

<table>
<thead>
<tr>
<th>treatment</th>
<th>groups removed by treatment</th>
<th>groups contributing to ASV-labile metals</th>
<th>groups contributing to total metals</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>none</td>
<td>I, II, III</td>
<td>I, II, III, IV, V, VI, VII</td>
</tr>
<tr>
<td>2</td>
<td>I, IV, V</td>
<td>II, III</td>
<td>II, III, VI, VII</td>
</tr>
<tr>
<td>3</td>
<td>none</td>
<td>I, II, III, IV, VI</td>
<td>I, II, III, IV, V, VI, VII</td>
</tr>
<tr>
<td>4</td>
<td>I, II, IV, V, VI</td>
<td>III</td>
<td>III, VII</td>
</tr>
</tbody>
</table>

(a) Explain how you can use these eight measurements to determine the concentration of metals present in each of the seven groups identified in Table 11.12.

(b) Batley and Florence report the following results for the speciation of cadmium, lead, and copper in a sample of seawater.

<table>
<thead>
<tr>
<th>measurement (treatment: ASV-labile or total)</th>
<th>ppm Cu²⁺</th>
<th>ppm Pb²⁺</th>
<th>ppm Cu²⁺</th>
</tr>
</thead>
<tbody>
<tr>
<td>1: ASV-labile</td>
<td>0.24</td>
<td>0.39</td>
<td>0.26</td>
</tr>
<tr>
<td>1: total</td>
<td>0.28</td>
<td>0.50</td>
<td>0.40</td>
</tr>
<tr>
<td>2: ASV-labile</td>
<td>0.21</td>
<td>0.33</td>
<td>0.17</td>
</tr>
<tr>
<td>2: total</td>
<td>0.26</td>
<td>0.43</td>
<td>0.24</td>
</tr>
<tr>
<td>3: ASV-labile</td>
<td>0.26</td>
<td>0.37</td>
<td>0.33</td>
</tr>
<tr>
<td>3: total</td>
<td>0.28</td>
<td>0.50</td>
<td>0.43</td>
</tr>
<tr>
<td>4: ASV-labile</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
</tr>
<tr>
<td>4: total</td>
<td>0.02</td>
<td>0.12</td>
<td>0.10</td>
</tr>
</tbody>
</table>

Determine the speciation of each metal in this sample of seawater.

32. The concentration of Cu²⁺ in seawater may be determined by anodic stripping voltammetry at a hanging mercury drop electrode after first releasing any copper bound to organic matter. To a 20.00-mL sample of seawater is added 1 mL of 0.05 M HNO₃ and 1 mL of 0.1% H₂O₂. The sample is irradiated with UV light for 8 hr and then diluted to volume in a 25-mL volumetric flask. Deposition of Cu²⁺ takes place at -0.3 V versus an SCE for 10 min, producing a peak current of 26.1 (arbitrary units). A second 20.00-mL sample of the seawater is treated identically, except that 1 mL of a 5.00 μM solution of Cu²⁺ is added, producing a peak current of 38.4 (arbitrary units). Report the concentration Cu²⁺ in the seawater in mg/L.

33. Thioamide drugs can be determined by cathodic stripping analysis. Deposition occurs at +0.05 V versus an SCE. During the stripping step the potential is scanned cathodically, and a stripping peak is observed at -0.52 V. In a typical application a 2.00-mL sample of urine was mixed with 2.00 mL of a pH 4.78 buffer. Following a 2.00 min deposition, a peak current of 0.562 μA was measured. A 0.10-mL addition of a 5.00 μM solution of the drug was added to the same solution. A peak current of 0.837 mA was recorded using the same deposition and stripping conditions. Report the drug’s molar concentration in the urine sample.

34. The concentration of vanadium (V) in seawater can be determined by adsorptive stripping voltammetry after forming a complex with catechol. The catechol-V(V) complex is deposited on a hanging mercury drop electrode at a potential of -0.1 V versus a Ag/AgCl reference electrode. A cathodic potential scan gives a stripping peak that is proportional to the concentration of V(V). The following standard additions were used to analyze a sample of seawater.

<table>
<thead>
<tr>
<th>[V(V)]std (M)</th>
<th>peak current (nA)</th>
</tr>
</thead>
<tbody>
<tr>
<td>2.0×10⁻⁸</td>
<td>24</td>
</tr>
</tbody>
</table>
Determine the molar concentration of V (V) in the sample of sea water, assuming that the standard additions result in a negligible change in the sample’s volume.

35. The standard-state reduction potential for Cu²⁺ to Cu is +0.342 V versus the SHE. Given that Cu²⁺ forms a very stable complex with the ligand EDTA, do you expect the standard-state reduction potential for Cu(EDTA)²⁻ to be greater than +0.342 V, less than +0.342 V, or equal to +0.342 V? Explain your reasoning.

36. The polarographic half-wave potentials (versus the SCE) for Pb²⁺ and Ti³⁺ in 1 M HCl are, respectively, −0.44 V and −0.45 V. In an electrolyte of 1 M NaOH, however, the half-wave potentials are −0.76 V for Pb²⁺ and −0.48 V for Ti³⁺. Why does the change in electrolyte have such a significant effect on the half-wave potential for Pb²⁺, but not on the half-wave potential for Ti³⁺?

37. The following data for the reduction of Pb²⁺ was collected by normal-pulse polarography.

<table>
<thead>
<tr>
<th>potential (V vs. SCE)</th>
<th>current (µA)</th>
</tr>
</thead>
<tbody>
<tr>
<td>−0.345</td>
<td>0.16</td>
</tr>
<tr>
<td>−0.370</td>
<td>0.98</td>
</tr>
<tr>
<td>−0.383</td>
<td>2.05</td>
</tr>
<tr>
<td>−0.393</td>
<td>3.13</td>
</tr>
<tr>
<td>−0.409</td>
<td>4.62</td>
</tr>
<tr>
<td>−0.420</td>
<td>5.16</td>
</tr>
</tbody>
</table>

The limiting current was 5.67 µA. Verify that the reduction reaction is reversible, and determine values for n and E₁⁄₂. The half-wave potentials for the normal-pulse polarograms of Pb²⁺ in the presence of several different concentrations of OH⁻ are shown in the following table.

<table>
<thead>
<tr>
<th>[OH⁻]⁻¹ (M)</th>
<th>E₁⁄₂ (V vs. SCE)</th>
<th>[OH⁻]⁻¹ (M)</th>
<th>E₁⁄₂ (V vs. SCE)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.050</td>
<td>−0.646</td>
<td>0.150</td>
<td>−0.689</td>
</tr>
<tr>
<td>0.100</td>
<td>−0.673</td>
<td>0.300</td>
<td>−0.715</td>
</tr>
</tbody>
</table>

Determine the stoichiometry of the Pb-hydroxide complex and its formation constant.

38. In 1977, when I was an undergraduate student at Knox College, my lab partner and I did an experiment to study the voltammetric behavior of Cd²⁺ (in 0.1 M KNO₃) and Ni²⁺ (in 0.2 M KNO₃) at a dropping mercury electrode. The data in this problem comes from my lab notebook. All potentials are relative to an SCE reference electrode.

<table>
<thead>
<tr>
<th>potential for Cd²⁺ (V)</th>
<th>current (µA)</th>
</tr>
</thead>
<tbody>
<tr>
<td>−0.60</td>
<td>4.5</td>
</tr>
<tr>
<td>−0.58</td>
<td>3.4</td>
</tr>
<tr>
<td>−0.56</td>
<td>2.1</td>
</tr>
<tr>
<td>−0.54</td>
<td>0.6</td>
</tr>
<tr>
<td>−0.52</td>
<td>0.2</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>potential for Ni²⁺ (V)</th>
<th>current (µA)</th>
</tr>
</thead>
<tbody>
<tr>
<td>−1.09</td>
<td>1.90</td>
</tr>
<tr>
<td>−1.05</td>
<td>1.75</td>
</tr>
<tr>
<td>−1.03</td>
<td>1.50</td>
</tr>
<tr>
<td>−1.02</td>
<td>1.25</td>
</tr>
<tr>
<td>−1.01</td>
<td>1.00</td>
</tr>
</tbody>
</table>

The limiting currents for Cd²⁺ was 4.8 µA and that for Ni²⁺ was 2.0 µA. Evaluate the electrochemical reversibility for each metal ion and comment on your results.

39. Baldwin and co-workers report the following data from a cyclic voltammetry study of the electrochemical behavior of p-phenylenediamine in a pH 7 buffer. All potentials are measured relative to an SCE.

http://chemwiki.ucdavis.edu/Analytical_Chemistry/Analytical_Chemistry_2.0/11_Electrochemical_Methods/11E_Summary_and_Problems
<table>
<thead>
<tr>
<th>scan rate (mV/s)</th>
<th>( E_{p,a} (V) )</th>
<th>( E_{p,c} (V) )</th>
<th>( i_{p,a} (mA) )</th>
<th>( i_{p,c} (mA) )</th>
</tr>
</thead>
<tbody>
<tr>
<td>2</td>
<td>0.148</td>
<td>0.104</td>
<td>0.34</td>
<td>0.30</td>
</tr>
<tr>
<td>5</td>
<td>0.149</td>
<td>0.098</td>
<td>0.56</td>
<td>0.53</td>
</tr>
<tr>
<td>10</td>
<td>0.152</td>
<td>0.095</td>
<td>1.00</td>
<td>0.94</td>
</tr>
<tr>
<td>20</td>
<td>0.161</td>
<td>0.095</td>
<td>1.44</td>
<td>1.44</td>
</tr>
<tr>
<td>50</td>
<td>0.167</td>
<td>0.082</td>
<td>2.12</td>
<td>1.81</td>
</tr>
<tr>
<td>100</td>
<td>0.180</td>
<td>0.063</td>
<td>2.50</td>
<td>2.19</td>
</tr>
</tbody>
</table>

The initial scan is toward more positive potentials, leading to the oxidation reaction shown here.

\[
\text{NH}_2 - \text{NH}_2 \quad \text{+ 2H}^+ \quad \text{+ 2e}^- 
\]

Use this data to show that the reaction is electrochemically irreversible. A reaction may show electrochemical irreversibility because of slow electron transfer kinetics or because the product of the oxidation reaction participates in a chemical reaction that produces an nonelectroactive species. Based on the data in this problem, what is the likely source of \( p \)-phenylenediamine’s electrochemical irreversibility?

### 11E.3 Solutions to Practice Exercises

#### Practice Exercise 11.1

The oxidation of \( \text{H}_2 \) to \( \text{H}^+ \) occurs at the anode

\[
\text{H}_2(g) f \rightleftharpoons 2\text{H}^+(aq) + 2e^-
\]

and the reduction of \( \text{Cu}^{2+} \) to \( \text{Cu} \) occurs at the cathode.

\[
\text{Cu}^{2+}(aq) + 2e^- \rightleftharpoons f\text{Cu}(s)
\]

The overall cell reaction, therefore, is

\[
\text{Cu}^{2+}(aq) + \text{H}_2(g) f \rightleftharpoons 2\text{H}^+(aq) + \text{Cu}(s)
\]

Click [here](#) to return to the chapter.

#### Practice Exercise 11.2

Making appropriate substitutions into equation 11.3 and solving for \( \text{E}_{\text{cell}} \) gives its value as

\[
\text{E}_{\text{cell}} = (E^0_{\text{Cu}^{2+}/\text{Cu}} - (0.05916 / 2)\log(1 / a_{\text{Cu}^{2+}})) - (E^0_{\text{H}^+/\text{H}_2} - (0.05916 / 2)\log(f_{\text{H}_2} / (a_{\text{H}^+}))^2)
\]

\[
= (0.3419 \text{ V} - (0.05916 / 2)\log(1 / 0.0500)) - (0.0000 - (0.05916 / 2)\log(0.500 / (1.00)^2))
\]

\[
= +0.2531 \text{ V}
\]

Click [here](#) to return to the chapter.

#### Practice Exercise 11.3

Making appropriate substitutions into equation 11.3

\[
+0.257 \text{ V} = (0.3419 \text{ V} - (0.05916 / 2)\log(1 / a_{\text{Cu}^{2+}})) - (0.0000 - (0.05916 / 2)\log(1.00 / (1.00)^2))
\]

and solving for \( a_{\text{Cu}^{2+}} \) gives its activity as \( 1.35 \times 10^{-3} \).

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#### Practice Exercise 11.4

When using a saturated calomel electrode, the potential of the electrochemical cell is

\[
\text{E}_{\text{cell}} = E_{UO_2^{2+}/U^{4+}} - E_{\text{SCE}}
\]

Substituting in known values

\[
-0.0190 \text{ V} = E_{UO_2^{2+}/U^{4+}} - 0.2444 \text{ V}
\]

and solving for \( E_{UO_2^{2+}/U^{4+}} \) gives its value as \( +0.2254 \text{ V} \). The potential relative to the \( \text{Ag}/\text{AgCl} \) electrode is

\[
\text{E}_{\text{cell}} = E_{UO_2^{2+}/U^{4+}} - E_{\text{Ag}/\text{AgCl}} = 0.2254 \text{ V} - 0.197 \text{ V} = +0.028 \text{ V}
\]

and the potential relative to the standard hydrogen electrode is

\[
\text{E}_{\text{cell}} = E_{UO_2^{2+}/U^{4+}} - E_{\text{SHE}} = 0.2254 \text{ V} - 0.0000 \text{ V} = +0.2254 \text{ V}
\]
Practice Exercise 11.5
The larger the value of $K_{A1}$, the more serious the interference. Larger values for $K_{A1}$ correspond to more positive (less negative) values for $\log K_{A1}$; thus, $\Gamma$, with a $K_{A1}$ of $6.3 \times 10^{-2}$, is the most serious of these interferences.

To find the activity of $\Gamma^-$ that gives a potential equivalent to a $NO_2^-$ activity of $2.75 \times 10^{-4}$, we note that

$$a_{NO_2^-} = K_{A1} \times a_{\Gamma^-}$$

Making appropriate substitutions

$$2.75 \times 10^{-4} = (6.3 \times 10^{-2}) \times a_{\Gamma^-}$$

and solving for $a_{\Gamma^-}$ gives its activity as $4.4 \times 10^{-3}$.

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Practice Exercise 11.6
In the presence of $OH^-$, the cell potential is

$$E_{cell} = K - 0.05916\log\left(a_{NO_2^-} + K_{NO_2^-/OH^-} \times a_{OH^-}\right)$$

To achieve an error of less than 10%, the term $K_{NO_2^-/OH^-} \times a_{OH^-}$ must be less than 1% of $a_{NO_2^-}$; thus

$$K_{NO_2^-/OH^-} \times a_{OH^-} \leq 0.10 \times a_{NO_2^-}$$

$$630 \times a_{OH^-} \leq 0.10 \times (2.2 \times 10^{-4})$$

Solving for $a_{OH^-}$ gives its maximum allowable activity as $3.5 \times 10^{-8}$, which corresponds to a pH of less than 6.54.

The electrode does have a lower pH limit. Nitrite is the conjugate weak base of HNO$_2$, a species to which the ISE does not respond. As shown by the ladder diagram in Figure 11.58, at a pH of 4.15 approximately 10% of nitrite is present as HNO$_2$. A minimum pH of 4.5 is the usual recommendation when using a nitrite ISE. This corresponds to an NO$_2^-$/HNO$_2$ ratio of

$$\text{pH} = pK_a + \log\left([NO_2^-] / [HNO_2]\right)$$

$$4.5 = 3.15 + \log\left([NO_2^-] / [HNO_2]\right)$$

$$[NO_2^-] / [HNO_2] \approx 22$$

Thus, at a pH of 4.5 approximately 96% of nitrite is present as NO$_2^-$. 

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Practice Exercise 11.7
The reduction of Cu$^{2+}$ to Cu requires two electrons per mole of Cu ($n = 2$). Using equation 11.25, we calculate the moles and the grams of Cu in the portion of sample being analyzed.

$$NCu = \frac{Q}{nF} = \frac{16.11 \text{ C}}{(2 \text{ mol } e^- / \text{ mol Cu}) \times (96487 \text{ C} / \text{ mole}^-)} = 8.348 \times 10^{-5} \text{ mol Cu}$$

$$8.348 \times 10^{-5} \text{ mol Cu} \times (63.55 \text{ g Cu} / \text{ mol Cu}) = 5.301 \times 10^{-3} \text{ g Cu}$$

This is the Cu from a 10.00 mL portion of a 500.0 mL sample; thus, the %/w/w copper in the original sample of brass is

$$5.301 \times 10^{-3} \text{ g Cu} \times (500.0 \text{ mL} / 10.00 \text{ mL}) / 0.442 \text{ g sample} \times 100 = 60.00\% \text{ w/w Cu}$$

For lead, we follow the same process; thus

$$N_{Pb} = \frac{Q}{nF} = 0.422 \text{ C} / ((2 \text{ mol } e^- / \text{ mol Pb}) \times (96487 \text{ C} / \text{ mole}^-)) = 2.19 \times 10^{-6} \text{ mol Pb}$$

$$2.19 \times 10^{-6} \text{ mol Pb} \times (207.2 \text{ g Pb} / \text{ mol Pb}) = 4.53 \times 10^{-4} \text{ g Pb}$$

$$(4.53 \times 10^{-4} \text{ g Pb} \times (500.0 \text{ mL} / 10.00 \text{ mL}) / 0.442 \text{ g sample} \times 100 = 5.12\% \text{ w/w Pb}$$
Practice Exercise 11.8
For anodic stripping voltammetry, the peak current, $i_p$, is a linear function of the analyte’s concentration

$$i_p = KC_{Cu}$$

where $K$ is a constant that accounts for experimental parameters such as the electrode’s area, the diffusion coefficient for Cu$^{2+}$, the deposition time, and the rate of stirring. For the analysis of the sample before the standard addition we know that the current is

$$i_p = 0.886 \ A = KC_{Cu}$$

and after the standard addition the current is

$$i_p = 2.52 \ \mu A = K(C_{Cu} \times (50.00 \ mL / 50.005 \ mL) + (10.00 \ mg \ Cu / L) \times (0.005 \ mL / 50.005 \ mL))$$

where 50.005 mL is the total volume after adding the 5.00 μL spike. Solving each equation for $K$ and combining leaves us with the following equation.

$$0.886 \ \mu A / C_{Cu} = K = 2.52 \ A / (C_{Cu} \times (50.00 \ mL / 50.005 \ mL) + (10.00 \ mg \ Cu / L) \times (0.005 \ mL / 50.005 \ mL))$$

Solving this equation for $C_{Cu}$ gives its value as $5.42 \times 10^{-4} \ mg \ Cu^{2+}/L$, or 0.542 μg Cu$^{2+}$/L.

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Practice Exercise 11.9
From the three half-wave potentials we have a $\Delta E_{1/2}$ of −0.280 V for 0.115 M en and a $\Delta E_{1/2}$ of −0.308 V for 0.231 M en. Using equation 11.51 we write the following two equations.

$$-0.280 = -(0.05916 / 2)\log \beta_{p} - (0.05916/2)\log(0.115)$$
$$-0.308 = -(0.05916 / 2)\log \beta_{p} - (0.05916/2)\log(0.231)$$

To solve for the value of $p$, we first subtract the second equation from the first equation

$$0.028 = -(0.05916/2)\log(0.115) - (-(0.05916/2)\log(0.231))$$

which eliminates the term with $\beta_{p}$. Next we solve this equation for $p$

$$0.028 = (2.778 \times 10^{-3})p - (1.882 \times 10^{-2})p$$
$$0.028 = (8.96 \times 10^{-3})p$$

obtaining a value of 3.1, or $p = 3$. Thus, the complex is Cd(en)$_3$. To find the formation complex, $\beta_3$, we return to equation 11.51, using our value for $p$. Using the data for an en concentration of 0.115 M

$$-0.280 = -(0.05916 / 2)\log \beta_{3} - ((0.05916 \times 3) / 2)\log(0.115)$$
$$-0.363 = -(0.05916 / 2)\log \beta_{3}$$

gives a value for $\beta_3$ of $1.93 \times 10^{12}$. Using the data for an en concentration of 0.231 M gives a value of $2.10 \times 10^{12}$.

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References

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