LEACHING

OBJECTIVE

Experimentally determine the extraction yield for a single stage leaching operation and study the effect of temperature on the leaching of a solid impregnated with a soluble salt, both overall yield and extraction rate.

REFERENCES


EQUIPMENT

1. 1-3 gallon white bucket
2. Stop watch
3. 1-large funnel
4. 15-screw capped test tubes
5. Specific ion meter and chloride specific ion electrode
6. Paper filters with twist lock ties or string
7. Beakers
8. 10 and 20 mL repeater pipets
9. Top loading Balance
10. 10 and 20 mL transfer pipets
11. 1000 mL graduated cylinder
12. 100 mL graduated cylinder
13. Magnetic stirrer and stir bars
CHEMICALS/MATERIALS

1. Deionized water.
2. Prepared sample of NaCl adsorbed on vermiculite.
3. Sodium chloride.
4. Potassium nitrate buffer

EXPERIMENTAL PROCEDURE

A diagram and description of the experimental apparatus is found in Attachment A. Familiarize yourself with the piping network of the leaching apparatus prior to class.

It will be necessary to generate a standard curve of chloride ion (Cl\(^-\)) concentration vs electrode potential of the standards. A **new standard curve should be constructed on each day that measurements of Cl\(^-\) are made.** The standard curve should include chloride ion concentrations from 10 to 10,000 mg/L. See the analysis procedure for details.

This experiment consists of two parts. In part one a determination of the total amount of soluble sample extracted with a known amount of solvent is made using three different analysis procedures. In part two a determination of the effect of temperature on the leaching rate of a soluble salt is made. This part includes two leaching tests, one at ambient temperature and one at an elevated temperature. Part one and the ambient temperature leaching test of part two should be done the first lab period.

**Part 1: Single stage leaching operation.**

1. Close the drain valves on the solvent (20) and receiving vessel (5).
2. Fill the solvent vessel (20) with 6 liters of deionized water.
3. Transfer 20.0 gm of sample (use a top loading balance and record weight) into a leaching filter (coffee filter). Tie the top of the filter with string or a twist lock (whichever is provided) and put it into the terylene leaching bag provided with the solid-liquid extraction unit. **Note:** The weight of the filter paper (include twist tie or string) and the weight of the sample will be needed for the calculations.
4. Tie up the terylene leaching bag with the string provided which is attached to the funnel found in the extractor vessel (17).
5. Set the pump rate to 40% (F1). **Note:** At a pump rate of 40%, with the 6 liters of water in the solvent vessel, there should be enough water in the system to leach the sample for 30 to 40 minutes.
6. Open valve V6. Close valve V1 and V5. Turn V7 so the arrow points to the left. **Note:** This allows the effluent to flow into the receiving vessel (5).

7. Close all other valves.

8. Plug the unit into a power source. Energize the control panel by pressing button S1.

9. Remove the lid from the extractor vessel (17). Place the sample bag into the extractor vessel. Replace the lid on the extractor vessel.

10. Start the pump by pressing button S2. Pump the solvent vessel empty, transferring the solvent through the extractor vessel into the receiving vessel. The solid in the extractor vessel will be leached for 30 to 40 minutes. Air bubbles enter the extractor vessel when the solvent vessel is empty, but only for a short time. Record the temperature of the solvent in the extractor vessel at the beginning, middle and end of the test.

11. While waiting for the extraction to finish, the chloride ion standards should be made up and the standard curve run using the chloride specific ion electrode. See “Analysis Procedures” for more detail.

12. During this time a determination of the moisture content of the vermiculite/salt sample should also be made. Use the analytical balance and record the tare weight of two 250 mL beakers. Add approximately 10 gm of sample to each beaker and record the actual weight. Place the samples in the oven. Include a tag with your lab section and group number written on it to identify your samples. During the next lab period remove the samples and let them cool in a desiccator. When the samples are cool, weigh them and record their weight.

13. When the leaching is finished, turn off the pump, button S2, and if the unit will not be used again during the lab period, button S1.

14. Open valve V1, which allows the solvent remaining in the extractor vessel to flow into the receiving vessel.

15. When the extractor vessel is drained, remove the terylene bag containing the sample from the vessel. Remove the sample from the terylene bag and place it in a small aluminum pie tin. Place it in an oven at 105°C to dry. Include a tag with your lab section and group number written on it. During the next lab period, remove it from the oven and place it in a desiccator. When the sample is cool, weigh it. From the data gathered a determination of the amount of soluble salt extracted can be made. This is the first of three ways used to determine the amount of soluble salt extracted from the substrate.
16. Measure and record the volume of effluent in the receiving vessel using a 1000 mL graduated cylinder, saving the effluent in the 3-gallon white plastic bucket provided.

17. The second of three ways to determine the amount of soluble salt extracted from the substrate is an evaporation method. Mix the sample in the 3-gallon bucket (from step 16) with a stirring rod and transfer a 100 mL sample, in triplicate, to tared 250 mL beakers (use an analytical balance). Place the beakers in an oven at 105°C. Evaporate and dry the samples. During the next lab period, remove the samples from the oven, place them in a desiccator to cool. Record the weight of each beaker and sample when cool. From the data gathered and the volume of solvent used in the extraction, a determination of the amount of soluble salt extracted can be made.

18. The third of three ways to determine the amount of soluble salt extracted from the substrate is a specific ion electrode method. Mix the sample in the 3-gallon bucket (from step 16) with a stirring rod. Transfer 3-10 mL samples, using a 10 mL transfer pipet, to 50 mL beakers. These samples are analyzed using the chloride specific ion electrode. The procedure is found under “Analysis Procedures, Samples.” From the concentration of chloride ion in the receiving vessel and the volume of solvent used in the extraction, a determination of the amount of soluble salt extracted can be made.

19. Clean the extractor and receiving vessels using the procedures described in Attachment A.

Part 2: Effect of temperature and volume of leachate on the leaching of a soluble salt (ambient temperature).

1. Close the drain valves on the solvent (20) and receiving vessel (5).

2. Fill the solvent vessel (20) with 6 liters of deionized water. Use a procedure that will allow you to calibrate the solvent vessel sight glass — you will use the scale to measure the volumetric flow rate.

3. Transfer 20.0 gm of sample (use a top loading balance and record weight) into a leaching filter (coffee filter). Tie the top of the filter with string or a twist lock (whichever is provided) and put it into the terylene leaching bag provided with the solid-liquid extraction unit.

4. Tie up the terylene leaching bag with the string provided which is attached to the funnel found in the extractor vessel (17). Do not place the sample bag into the extractor vessel at this time.
5. Set the pump rate to 40% (F1). **Note:** At a pump rate of 40%, with 6 liters of solvent, there should be enough water in the system to last 30 to 40 minutes.

6. Open valve V6. Close valve V1 and V5. Turn V7 so the arrow points to the left. **Note:** This allows the effluent to flow into the receiving vessel (5).

7. Close all other valves.

8. Plug the unit into a power source. Energize the control panel by pressing button S1.

9. Start the pump by pressing S2. Fill the extractor vessel to the black line with solvent. Stop the pump by pressing S2.

10. Remove the lid from the extractor vessel (17). Place the terylene sample bag into the extractor vessel, replace the lid and restart the pump S2. Start the timer when liquid begins to flow from the sample valve, V4.

11. Once the effluent starts to overflow from the extractor vessel, start sampling the effluent (almost fill a test tube — 15 mL minimum) through sampling valve V4 discarding the first few mL. Sample at 30 sec intervals during the first minute, every minute during the next 4 minutes, and then every 5 minutes until the solvent vessel is pumped dry (this will be evident by observing bubbles entering the bottom of the extractor vessel). Record the temperature of the solvent in the extractor vessel at the beginning, middle and end of the test. Measure the volumetric flow rate using the scale on the solvent vessel sight glass and a stopwatch. Remeasure it from time to time. As samples are collected, analyze them using the specific ion electrode. See “Analysis Procedures, Samples” for details.

The flow rate can also be measured by opening the drain valve on the receiving vessel and timing the collection of a measured volume of liquid. Make sure the receiving vessel has been drained of excess liquid and the sampling valve V4 is closed during this process. Which is the more accurate method?

12. Turn off the pump, switch S2, and if the unit will not be used again during the lab period, switch S1.

13. Open Valve V1, which allows the effluent remaining in the extractor vessel to flow into the receiving vessel. When the extractor vessel is drained, remove the terylene bag containing the sample. Remove the sample from the bag and place it in a pan to dry following the procedure described in step 15 of “Part 1: Single Stage Leaching Operation.”
14. Clean the extractor and receiving vessels using the procedures described in Attachment A.

Part 2: Effect of temperature and volume of leachate on the leaching of a soluble salt (elevated temperature).

1. Close the drain valves on the solvent (20) and receiving vessel (5).

2. Fill the solvent vessel (20) with 6 liters of deionized water.

3. Plug the unit into a power source. Energize the control panel by pressing button S1. Turn on switch S3. Rotate dial R1 to the maximum position of 10.

4. Allow the solvent vessel to heat up to approximately 80°C (60 to 90 min) and then turn R1 to position 5.

   **NOTE:** Turn off the heaters, Switch S3, when the water level decreases to approximately 1/2 inch in the solvent vessel sight glass.

5. Transfer 20.0 gm of sample (use a top loading balance and record weight) into the leaching filter (coffee filter). Tie the top of the filter with string or a twist lock (whichever is provided) and put it into the terylene leaching bag provided with the solid-liquid extraction unit.

6. Tie up the terylene leaching bag with the string provided which is attached to the funnel found in the extractor vessel (17). Do not place the sample bag into the extractor vessel at this time.

7. Set the pump rate to 40% (F1). **Note:** At a pump rate of 40%, with the solvent vessel filled, there should be enough water in the system to last 30 to 40 minutes.

8. Open valve V6. Close valve V1 and V5. Turn V7 so the arrow points to the left. **Note:** This allows the effluent to flow into the receiving vessel (5).

9. Close all other valves.

10. Turn on the pump by pressing button S2. Allow the pump to fill the extractor vessel to the black line with the solvent. Stop the pump by pressing S2.

11. Remove the lid from the extractor vessel (17). Place the sample bag into the extractor and restart the pump by pressing S2. Start the timer when liquid begins to flow from the sample valve, V4.
12. Once the effluent starts to overflow from the extractor vessel, start sampling the effluent (almost fill a test tube - 15 mL minimum) through sampling valve V4 discarding the first few mL. Sample at 30 sec intervals during the first minute, every minute during the next 4 minutes, and then every 5 minutes until the solvent vessel is pumped dry (this will be evident by observing bubbles entering the bottom of the extractor vessel). Record the temperature of the solvent in the extractor vessel right after sampling begins, midway through the test, and immediately after the last sample has been collected. Measure the volumetric flow rate using the scale on the solvent vessel sight glass and a stopwatch. Remeasure it from time to time. As samples are collected, analyze them using the specific ion electrode. See “Analysis Procedures, Samples” for details.

**Note:** Turn off the heaters, switch S3, when the water level decreases to approximately 1/2 inch in the solvent vessel sight glass.

The flow rate can also be measured by opening the drain valve on the receiving vessel and timing the collection of a measured volume of liquid. Make sure the receiving vessel has been drained of excess liquid and the sampling valve V4 is closed during this process. Which is the more accurate method?

13. Turn off the pump, switch S2, and if the unit will not be used again during the lab period, switch S1.

14. Open Valve V1, which allows the effluent remaining in the extractor vessel to flow into the receiving vessel. When the extractor vessel is drained, remove the terylene bag containing the sample. Remove the sample from the bag and place it in a pan to dry following the procedure described in step 15 of “Part 1: Single Stage Leaching Operation.”

15. Clean the extractor and receiving vessels using the procedures describe in Attachment A.

**ANALYSIS PROCEDURES**

**Samples**

Transfer a 10 ml sample, using a 10 mL transfer pipet, from the white bucket or from each test tube to a 50 mL beaker. Dilute the 10 mL sample in the 50 mL beaker with 10 mL of deionized water. Add 20 mL of ISAB (KNO₃ buffer) to the sample. Immerse the electrode in the sample with gentle stirring and record the potentials. The concentration found from the standard curve will have to be multiplied by 2.0 to give the actual concentration in the sample.
Analysis of chloride using a chloride specific ion electrode

Standards

1. Make up a 10.0 gm/L (10,000 mg/L) chloride ion stock standard by transferring 1.644 gm of NaCl to a 100 mL volumetric flask, dissolving in deionized H₂O and diluting to the mark.

2. Make up 1000, 100, and 10 mg/L chloride ion standards by serial dilution to 100 mL. Also prepare a 40 mg/L chloride ion standard.

3. Pipet 20 mL of each standard to a 50 mL beaker containing a magnetic stirring bar. Add 20 mL of the KNO₃ solution (the Ionic Strength Adjustment Buffer (ISAB)) to each standard.

4. The Corning Model 455 meter will be used to make the measurements. Place the lowest standard (10 mg/L) on the magnetic stirrer with gentle stirring. Remove the rubber inserts from the breather holes (top side of electrode) on the double junction reference electrode. Insert the electrodes into the liquid and follow the procedure found on the last page to set up the meter for taking mV (potential) measurements. Record the potential.

5. Remove the electrode and rinse with a small volume of deionized water. Immerse the electrode sequentially in the next higher buffered standards (40, 100, 1000, 10000 mg/L) and record the potentials.

6. Construct a standard curve by plotting the log of the chloride ion concentration in mg/L vs the potential in mV. Four-cycle semilog paper can also be used with concentration in mg/L on the log axis and mV on the linear axis.

7. If samples are not run immediately after the standards, immerse the electrode in deionized water until the samples are ready to be run.

8. Remove the electrode from the last standard and immerse the electrode in each buffered sample with gentle stirring. See “Samples” above for sampling procedure. Record the potential. The sample should be at room temperature before the sample is analyzed. The concentration can be determined from the standard curve.

9. Before leaving the lab make sure the breather holes (top side of electrode) in the double junction electrode are plugged.
SAFETY NOTES

To avoid electrical shock be sure the floor is dry around the extraction apparatus before plugging it into a power source.

WASTE DISPOSAL PROCEDURES

The waste from this experiment can be put into the sink drain for disposal.

01/12
SOLID LIQUID EXTRACTION UNIT
SOLID-LIQUID EXTRACTION APPARATUS

The equipment is fitted into a welded steel framework constructed from square-section and rectangular-section members and supported on adjustable feet. Two 5 liter cylindrical stainless steel vessels, the receiving vessel (5) and the supply vessel (20), contain the solvent used in the extractions. The vessels are fitted with filler plugs (6 and 19), sight glasses (2), thermometers (T1 and T3) and drain cocks (V2 and V9). The left-hand vessel (5) is the boiler for the distillation column and the right-hand vessel (20) contains the solvent supply for the extractions. Both vessels are fitted with electrical heaters.

A meter pump (4) provides a controllable known solvent flow to the extractor vessel (17) by either of two possible routes, one to the top of the vessel through valve V5 and the other to the bottom of the vessel through valve V6. Delivery from the pump is set by a continuously variable adjustment F1, the position of which is indicated as a percentage of maximum flow (5 mL/sec). The flow adjustment knob can be locked by withdrawing it slightly.

The extractor vessel (17) is a cylindrical glass tube with stainless steel end plates, sealed to the glass with molded PTFE gaskets and clamped together with bolt-through flanges fitted around the glass. A removable lid (16) allows access to the interior for insertion of the sample contained in a terylene fabric bag (21). Solvent entering the top of the extractor is directed to the axis of the vessel by a funnel fitted below the solvent inlets. The funnel is removed with the sample after an extraction.

Solvent leaves the extractor vessel either from the top or the bottom, depending on whether the valve (V1) is closed or open respectively, and passes to the two-way valve (V7) whose position determines whether the solvent passes to the boiler (5) or back to the supply vessel (20). In the latter case a closed circulation circuit is formed, the former an open circuit.

The supply vessel (20) and the high points of the solvent pipe work are connected to a breather tube system (11) and (14) to allow the liquid levels to be determined by gravity.

The glass distillation column (7) contains four sieve plate type trays and is mounted vertically above the boiler (5). A glass reflux divider (8) with a thermometer (9) in the vapor stream is mounted on the column and clamped to it by means of bolt-through flanges. The stainless steel condenser (10) is fitted above the reflux divider in the same way, joints being sealed in the usual way with PTFE gaskets. Cooling water circulation for the jacket of the condenser is provided via the pipes (12) with a hose nozzle fitting at the rear. Condensate is taken off from the reflux divider via the control valve (C1) which determines the reflux ratio.

Solvent samples can be taken at a number of points around the system. Samples from the supply vessel (20) and the boiler (5) are taken with the drain cocks (V2) and (V9) fitted to the vessel bases. Solvent pumped to the extractor vessel (17) is sampled via (V8) and solvent leaving the extractor vessel via (V4). Pure solvent from the distillation column can be sampled via the cock (V3).

The extractor vessel, the distillation column and the solvent-carrying pipe work are lagged to reduce heat losses.

The control panel (13) for the heaters and pump takes the form of a system diagram which shows clearly how each control relates to the system. Appropriately placed illuminating push buttons switch the power for pump and heaters. Two thyristor regulators control the power supplied to the heaters. The electrical module (15) houses these components and wiring.
transformer (18) will be fitted if the electrical supply is 100/130V, 50 or 60 Hz.

PROCEDURE FOR CLEANING THE SOLID-LIQUID EXTRACTION APPARATUS

After each run the extractor and receiving vessels should be cleaned using the procedure outlined below. These procedures should insure there is no carryover of sample from one run to the next.

1. Rinse out the extractor vessel by filling it twice with tap water (fill it above the discharge line so water will overflow and rinse out the discharge line), open valve \( V1 \) and let the remainder of the water drain into the receiving vessel. After each rinse, open the valve on the bottom of the receiving vessel and drain the receiving vessel. The third rinse should be done with deionized water in the same manner as the first two rinses.

2. After rinsing the extractor vessel three times, close the drain valve on the receiving vessel and fill it with tap water. Drain the receiving vessel. Close the valve and add 1 to 2 liters of deionized water. Drain the deionized water out of the vessel and it should be ready for the next run.

Rinse the terylene bags two or three times in tap water in the sink and allow to dry.

Instructions For Use of The Corning Model 455 Specific Ion Meter

1. MV should be shown at the top of the display. If not press the mode button until it does.
2. Place the electrodes in the sample.
3. Press the read button. The mv reading of the sample will be shown. The reading will drift for a short period of time. At low concentrations it may drift for a longer period of time. In this case, take the reading after 4 to 5 minutes. When the reading is stable, press read to freeze the display. Record the reading.
4. Remove electrodes from the sample, rinse the electrodes with deionized water and place them in a new sample.
5. Press the read button to get the new reading as in item 3 above. Record the reading.
6. Continue.

NOTE: Always keep the electrodes in deionized water when not in use.
Department of Chemical Engineering  
Stockroom Checkout slip

Leaching  
ChE 4211

Name: ___________________________  
(Date: ___________________________  
(print name)

Lab No.: Lab 1  Tuesday  12:00 - 4:50 PM  
Lab 2: Thursday  12:00 - 4:50 PM  
Lab No.: Lab 3  Tuesday and Thursday morning (9:30 - 11:50 AM)  
(circle one)

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