Experiment 2: Effectiveness of SO₂ Scrubbers CH3600: Environmental Chemistry, Plymouth State University

Adapted from Schilling AL, Leber PA, Yoder CH. "Exploration of SO₂ Scrubbers: An Environmental Chemistry Project." *J Chem Ed* **86**(2):225 (2009)

Background information provided by Eubanks, et. al., Chemistry in Context, 6th edition, McGraw-Hill, 2008.

Introduction:

Energy production is a major issue in the United States and around the world. In the United States, power generated from coal-fired power supplies nearly 50% of the US power needs—with a majority of these plants located in the northeast and near-midwest.

Generation of power by burning coal has several advantages: coal is relatively inexpensive, quite plentiful and has a high energy content, supplying 20-30 kJ/g of coal burned. However, the environmental impact of burning coal is significant. Due to its complex composition (approximated $C_{135}H_{96}O_9NS$), high temperatures, and incomplete oxidation, combustion of coal results in several undesirable by-products: particulate matter (PM), nitrogen oxides (NO_x) and SO₂.

Once in the atmosphere, these air pollutants have serious ramifications, and research shows that the acidity of precipitation has greatly increased in regions relying heavily on coal for power production. The SO_2 (and NO_x) gases leaving the smoke stacks at power generation stations react with the water in the atmosphere to create "acid rain":

$$\begin{split} & \text{SO}_{2(g)} + \text{H}_2\text{O}_{(l)} \twoheadrightarrow \text{H}_2\text{SO}_{3(aq)} \text{ (sulphurous acid)} \\ & \text{SO}_{2(g)} + \text{H}_2\text{O}_{(l)} + \frac{1}{2}\text{O}_{2(g)} \twoheadrightarrow \text{H}_2\text{SO}_{4(aq)} \text{ (sulphuric acid)} \\ & \text{4NO}_{2(g)} + 2\text{H}_2\text{O}_{(l)} + \text{O}_{2(g)} \twoheadrightarrow \text{4HNO}_{3(aq)} \text{ (nitric acid)} \end{split}$$

The addition of these acids to the atmosphere, lakes, streams, and soils can have a dramatic impact on the health of the ecosystem.

To address the issue of acid rain, the Clean Air Act Amendments of 1990 established the Acid Rain Program at the EPA (http://www.epa.gov/airmarkets/progsregs/arp/index.html) and made reduction of NO_x and SO_2 emissions a national priority. The program's efforts to reduce SO_2 emissions has been quite successful, utilizing three methods: (1) switching to low-sulfur content coal; (2) removing the sulfur from the coal prior to burning; and (3) scrubbing stack gases to remove the SO_2 before the gases are released to the atmosphere. In this scrubbing process, stack gases are passed through a slurry of CaCO₃ and the following reaction occurs:

 $2SO_{2(g)} + O_{2(g)} + 2CaCO_{3(s)} \rightarrow 2CaSO_{4(s)} + 2CO_{2(g)}$

This process is highly efficient at removing the SO_2 , but unfortunately is very costly, with estimates ranging from \$400-600/ton of SO_2 removed.

In this lab, you will test different materials for their effectiveness of scrubbing to remove SO₂ from "simulated" stack gases. The effectiveness of the scrubbers will be analyzed using a pH meter.

Equipment:

- 3 side-arm test tube with stoppers
- 3 pieces of glass tubing
- 4 pieces of Tygon tubing
- 3 rubber stoppers with holes
- 3 ring stands with clamps
- Bunsen burner
- 100 mL beaker

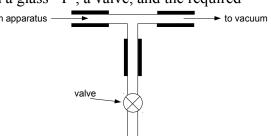
- pH meter
- Distilled water
- ~2 grams sulfur
- Materials for "scrubbing" $0.3 \text{ g CaCO}_{3(s)}$ 25 mL 1 M Na₂CO_{3(s)} 1 g KOH pellets 25 mL 1M KOH_(aq)

- Safety Considerations:
 - Dust from sulfur, calcium carbonate, and calcium sulfate can be irritating to the upper respiratory tract. Avoid breathing any dust by performing all operations in a fume hood.
 - KOH and Na₂CO₃ are highly corrosive, strong bases and should be handled with caution; direct contact with skin and eyes should be avoided.
 - As always, appropriate eye protection should be worn at all times.
 - Take caution when adjusting rubber tubing and stoppers connected to the glass tubing, as it can ٠ break easily and create sharp edges.

Procedure:

General Setup

- 1. Construct a vacuum valve as shown in the figure from a glass "T", a valve, and the required tubing. Leave the valve in the "open" position. from apparatus -
- 2. Connect the "vacuum" side to a vacuum aspirator in the hood



Part 1: Measuring the increase in acidity of rain water by reaction with $SO_{2(g)}$

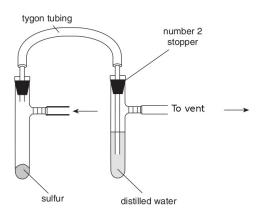


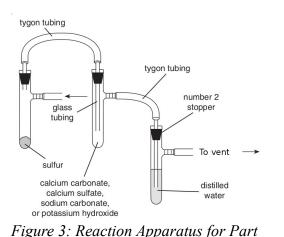
Figure 2: Reaction apparatus for Part 1

Figure 1: Vacuum valve

- 1. Using 2 side arm test-tubes, 2 glass pieces of glass tubing and two pieces of tubing, assemble the reaction apparatus, as shown in Figure 1.
- 2. Using two clamps, attach the test tubes to two ring stands.
- 3. Attach the "vent" tube to the vacuum valve.
- 4. Fill a 100 mL beaker with distilled water.
- 5. Record the pH of the distilled water with a pH meter.
- 6. Add 25 mL of the distilled water to the second (right) test tube. Be sure that the glass tubing is fully submerged in the water.
- 7. To the first test tube (left), add 0.1 g of sulfur.

- 8. Connect everything, turn on the vacuum aspirator, and slowly adjust the vacuum valve until a steady stream of air bubbles is seen in the distilled water. *Be careful not to let it bubble too vigorously!*
- 9. Use a Bunsen burner to gently heat the sulfur in the test tube.
- 10. Heat for ~1-2 minutes. Record observations and the exact time of heating in your lab notebook.
- 11. Extinguish Bunsen burner, open the vacuum valve, and turn off the vacuum aspirator.
- 12. Use a pH meter to record the pH of the water.
- 13. Dispose of the water down the sink. Dispose of the sulfur in Tube #1 in a trash can (this may require scraping with a long spatula). Rinse both test tubes thoroughly with distilled water.

Part 2: Exploration of different reagents as potential SO₂ scrubbers



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1. Adjust the reaction apparatus to contain three side arm test tubes (Figure 2).

- 2. Add 0.1 g sulfur to Test Tube #1
- 3. Fill a 100 mL beaker with distilled water.
- 4. Record the pH of the distilled water with a pH meter.
- 5. Add 25 mL distilled water to Test Tube #3. Be sure that the glass tubing is fully submerged in the water.
- 6. To Test Tube #2 add 1.0 g calcium carbonate and 25 mL distilled water. (Note: with stirring and agitation, the solution will become cloudy, but the calcium carbonate will not dissolve).
- 7. Attach the apparatus to ring stands.
- 8. Attach the "vent" tube to the vacuum valve.
- 9. Connect everything, turn on the vacuum aspirator, and slowly adjust the vacuum valve until a steady stream of air bubbles is seen in the distilled water. *Be careful not to let it bubble too vigorously!* Allow air to blow through for ~2 minutes to agitate the calcium carbonate slurry.
- 10. Use a Bunsen burner to gently heat the sulfur in the test tube.
- 11. Heat for ~1-2 minutes. Record observations and the exact time of heating in your lab notebook.
- 12. Extinguish Bunsen burner, open the vacuum valve, and turn off the vacuum aspirator.
- 13. Use a pH meter to record the pH of the water in Test Tube #3.
- 14. Dispose of the calcium carbonate slurry and the water in Tube #3 down the sink. Dispose of the sulfur in Tube #1 in a trash can (this may require scraping with a long spatula). Rinse the test tubes thoroughly with distilled water.
- 15. Repeat Steps 2-14 three times, using 1 M Na₂CO₃, KOH(s), and 1 M KOH instead of calcium carbonate.

<u>Analysis</u>

The following should be done in your notebook. You may choose to do this after the lab, but your lab will not be signed off, and your duplicate pages will not be accepted, until it is completed. This analysis should also appear in your formal lab report.

	pH of Distilled water		$H_{3}O^{+}$ Conc. ([H ₃ O ⁺])		Change in	
Reagent	Before	After	Before	After	pН	$[H_3O^+]$
H ₂ O(1)						
CaCO ₃ (slurry)						
1 M Na ₂ CO ₃ (aq)						
KOH(s)						
1 M KOH(aq)						

1. Complete the following data table in your lab notebook:

- 2. Calculate the theoretical number of moles of sulfate that could be produced from 0.1 g of sulfur, if 100% of the sulfur is converted to SO_2 , and 100% of this is absorbed and converted to H_2SO_4 .
- 3. Assuming that the change in concentration of hydronium ion ([H₃O⁺]) is only from the reaction: H₂SO₄ (aq) + H₂O \rightarrow HSO₄⁻ (aq) + H₃O⁺ (aq)

Calculate the moles of SO₂ absorbed in the distilled water in each experiment.

Conclusions

Answers to these questions must be included as part of your Conclusions in your written lab report. Include them in your Conclusions narrative, not as numbered list of questions and answers.

- 1. Determine which scrubbing agent is the most effective by calculating the percent of SO_2 (by moles) absorbed by the scrubbing agents using your answers from questions 2-4 above.
- 2. Speculate as to why CaCO₃ is the scrubber of choice for industry.