2.5) INSTRUCTOR'S GUIDE TO THE CHEMISTRY OF FLUE GAS DESULFURIZATION

Overview: Apply a small amount of low-viscosity silicone oil to make sure that all the syringes' plungers slide easily. This facilitates collection and transfer of gases and improves the accuracy of gas absorption readings. Pressurized silicone spray is not recommended because it may have hydrocarbons that will damage the rubber plunger over time.

Part A (*Gas Preparation*) should never be attempted by high-school students, even at the AP level. Parts B-D are less dangerous but require one-on-one supervision during the gas transfer step due to the risk of gas release. To minimize risk further, you may even choose to carry out all of the gas transfers yourself and involve the students only in the testing portions of Parts C-D and slurry preparation in Part D. If you choose to allow the students to carry out the gas transfers directly, you should prepare all the sulfur dioxide syringes the day before so you can supervise them more effectively. Parts D-3 and E are the least dangerous because they only involve aqueous sulfur dioxide, but safety goggles must be worn and skin contact must be avoided because aqueous sulfur dioxide is corrosive.

For Parts C-D, use a reliable source of sodium hydroxide that has not had time to absorb water from the air. This will help ensure a sharp distinction between your control wells and your SO_2-treated wells.

The quickest means of preparing oxygen for Part E is to do it in a syringe (using the same technique as in Part A). Add about 1 gram potassium iodide to about 20 mL of 3% hydrogen peroxide (available in drugstores). These chemicals are relatively safe; therefore, you can direct the students to do it. In the absence of a catalyst (in this case Fe^{3+}), the oxidation of sulfite can easily take over a week, even when the solution is being exposed to pure oxygen.

Sample Results for Lab 3-10 Part C:

Treatment	pH (color)	$KMnO_4$ (color)
Water + SO_2	Red (acidic)	Clear (reducing)
NaOH solution + SO_2	Blue (basic)	Green, then brown (reducing)
Pure water (control)	Green (neutral)	Purple (no rxn)
Pure NaOH solution (control)	Blue (basic)	Purple (no rxn)

Sample Results for Lab 3-10 Parts D-1 & D-2:

Scrubber reactant	NaOH	Na_2CO_3	Lime	NaCl	Na_2SO_4
Gas volume absorbed (mL)	52-11= 41	53-20 = 33	53-22 = 31	50-31 =19	52-37=15
pH (color) based on gas	N/A*	Orange (acidic)	Orange (acidic)	Red (acidic)	Red (acidic)
$KMnO_4$ (color) based on gas	N/A*	Purple (no rxn)	Purple (no rxn)	Clear (reducing)	Clear (reducing)

*This step could not be applied because not enough gas remained available for testing. This result is expected.

Sample Results for Lab 3-10 Part D-3:

Scrubber reactant		NaOH	Na_2CO_3	Lime	NaCl	Na_2SO_4
pH (color) based on solid	Before reaction	Blue (basic)	Blue (basic)	N/A◊	Green (neutral)	Blue (basic)
	After reaction	Blue (basic)	Blue (basic)	N/A◊	Orange (acidic)	Orange (acidic)
$KMnO_4$ (color) based on solid	Before reaction	Purple (no rxn)	Purple (no rxn)	N/A◊	Purple (no rxn)	Purple (no rxn)
	After reaction	Brown (reducing)	Brown (reducing)	N/A◊	Purple (no rxn)	Purple (no rxn)

◊This step could not be applied because the dark color of the garden lime did not allow us to discern the colorimetric tests.

Sample Results for Lab 3-10 Part E:

Treatment		O_2	Air (w/exchange)	Air (no exchange)
pH (color)	Day 0	Red (acidic)		
	Final	Red (acidic)	Red (acidic)	Red (acidic)
$KMnO_4$ (color)	Day 0	Clear (reducing)		
	Day 1	Purple (no rxn)	Clear (reducing)	Clear (reducing)
	Day 2	Purple (no rxn)	Light brown (reducing)	Clear (reducing)
	Day 3	Purple (no rxn)	Purple/brown (reducing)	Clear (reducing)
	Day 4	Purple (no rxn)	Purple (no rxn)	Clear (reducing)

Answers to questions:

1) The water becomes acidic due to the production of sulfurous acid.

2) The permanganate ion was reduced to form either the manganous (II) ion (which is clear) or manganese (IV) dioxide (which is brown).

3) The pH was probably not significantly affected because of the high concentration of hydroxide that neutralized the sulfurous acid.

4) The permanganate ion was reduced to form either manganese (IV) dioxide (which is brown) or the manganate (VI) ion (which is green).

5) The sodium hydroxide, calcium carbonate, and garden lime reacted with the sulfur dioxide because the dissolved solid product caused the permanganate to change color. The sodium chloride and sodium sulfate did not react because there was no change in color.

6) The syringe with sodium hydroxide absorbed the most gas. This resulted in the production of sodium sulfite.

7) There was much less net loss of gas in the other syringes. This did not always mean that no reaction took place. It could also mean that the sulfur dioxide was replaced by another gas.

8) If the gas in the syringe did not reduce the permanganate it was not sulfur dioxide. The new gas product in this case is carbon dioxide. This replacement takes place with any slurry containing carbonate ions. Even though carbon dioxide will not reduce permanganate, it will lower the pH of the water through the production of carbonic acid.

9) The pure oxygen is the fastest oxidizer because it provides the highest concentration of this reagent.

10) The "stale" air is the least effective oxidizer because it has the most limited amount of oxygen.

11) Oxidation does not "neutralize" the water because it converts the sulfurous acid into sulfuric acid.

12) Sulfites are known to release sulfur dioxide when they are wet, particularly if the water is acidic (see Part A).

Logistics: High school students should not carry out Part A, but you may consider having the students simulate this procedure using white vinegar and sodium bicarbonate (as substitutes for HCl and sodium bisulfite). Sulfur dioxide to be used by high school students should be prepared ahead of time. Parts B and C can be accomplished in one 40-minute period. Part D requires at least two 40-minute periods. Part E requires daily testing in addition to one 40-minute period. The daily testing should take no more than five minutes. You may choose to opt out of Part D-3 (the solid testing) or E (oxidation) if you do not have enough periods to spare. To cover all these labs you need at least 12 syringes.

Degree of Difficulty: 4—This lab provides a deep understanding of how smokestack scrubbers work. Even though a deep knowledge of chemistry is not necessary (note the lack of mole conversions), students must have good lab skills. Rehearsal of all procedures by the instructor is mandatory.

Product Guidelines: Most of these items for gas collection can be found in a chemistry lab. A complete gas collection kit and manual also can be purchased online from Educational Innovations. This kit also includes a low-viscosity silicone oil syringe lubricant. Silicon lubricants packed in aerosol spray cans are not recommended for syringes because they may contain hydrocarbons that corrode the plunger. The only place I could find a reasonably priced non-aerosol, low viscosity silicon lubricant to replace the supply that came from the kit was from www.9X6lubes.com. Since 9X6 is a *sexual* lubricant that comes in a blue bottle, it may be wise for you to keep it hidden from your students if you are teaching high school.

Materials: For all procedures: a 1-liter plastic beaker with 500 mL 0.25-0.5M NaOH solution (for cleanup purposes only). For Procedure A: forceps; two 60-mL syringes with screw-on caps; aquarium tubing; sodium bisulfite; 100 mL of 6M HCl; and a plastic vial cap that easily fits into the syringe. For Procedure B: two 60-mL syringes; aquarium tubing; a piece of strawberry or radish that fits in the syringe. For Procedure C: four 60-mL syringes; aquarium tubing; incubation wells; and colorimetric testing reagents (potassium permanganate solution and broad-range pH indicator); 1M NaOH solution; and distilled water. For Procedure D: eight 60-mL syringes; aquarium tubing; distilled water; sodium hydroxide; garden lime; sodium carbonate; and sodium chloride; four 50-mL beakers; a drying oven; incubation wells; and colorimetric testing reagents. For Procedure E: six 60-mL syringes; aquarium tubing; distilled water; 40 mL of oxygen; one drop of 0.1M iron (III) nitrate solution; incubation wells; and colorimetric testing reagents (see Procedure C).