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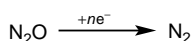
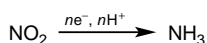
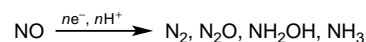
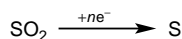
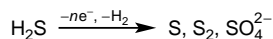
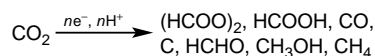
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# Laboratory Experiments on Electrochemical Remediation of the Environment. Part 5: Indirect H<sub>2</sub>S Remediation†

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Electrochemical remediation methods require an ion-conducting medium to perform their function of oxidizing or reducing polluting species. We have reported experiments to demonstrate the use of electrochemical techniques to clean an oil-water emulsion (1), indirectly oxidize pollutants (2), visualize the effects involved in the electrokinetic remediation of soils (3), and clean up a dye-containing solution by electroflotation–electrofloculation–electrocoagulation (4). However, since gaseous mixtures are nonconducting, they normally must first be absorbed (usually in aqueous solutions) to be treated (5). This can be accomplished either by using an absorption medium inside an electrochemical cell (*inner-cell process*) or by absorbing the gas first and then transferring the absorption medium into the electrochemical cell for treatment (*outer-cell process*). Also, in such methods the polluting species can either undergo electron transfer on an electrode surface (*direct electrolysis*), or electrons can be shuttled to or from the electrode by an electron carrier or *mediator* (*indirect electrolysis*) (6–8). Some examples of gases treated electrochemically are shown below (6, 9). The equations are not balanced, and only products containing the main element are shown.



Indirect electrolysis is used when a species either is non-electroactive or cannot efficiently adsorb onto an electrode to undergo direct electron transfer. The diffusion of a low-concentration species (e.g., dissolved pollutant) toward the electrode is often the rate-controlling step in an electrochemical process. In these cases, the process can be facilitated by the use of a mediator that can react homogeneously in solution with the target species and be regenerated later. Several methods of electrolysis, which have been proposed for the treatment of H<sub>2</sub>S, are shown in Figure 1.

†This paper is dedicated to Wayne E. Wentworth (University of Houston) on the occasion of his 70th birthday.

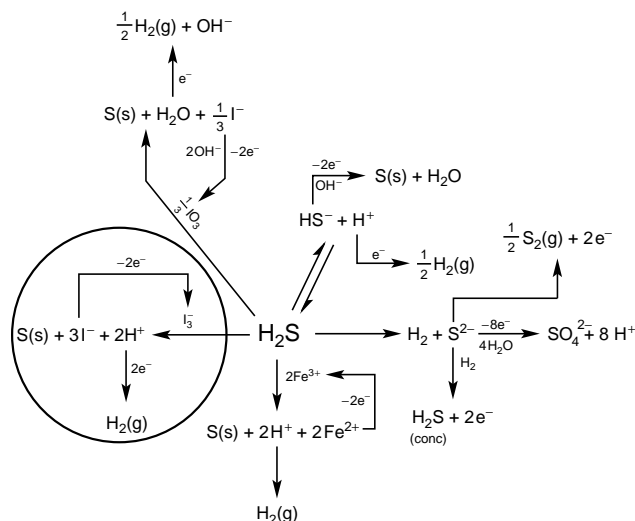
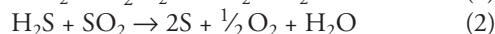
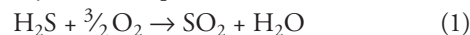


Figure 1. Electrochemical treatments of H<sub>2</sub>S.

In the following experiment, students taking general chemistry, environmental chemistry, or electrochemistry will learn the concept of indirect electrolysis, its application in environmental remediation schemes, the role of a mediator, and the application of redox chemistry concepts by performing an experiment that involves visual changes in every step.

One way in which the electrochemical treatment of H<sub>2</sub>S can be accomplished is shown here. Hydrogen sulfide is a well-known pollutant produced in considerable amounts from sulfate reduction in organic-rich (anaerobic) environments, heavy-oil desulfurization processes, oil recovery operations, coal gasification/liquefaction processes, etc. (10, 11). It is frequently treated by absorption in basic solutions of different amines (12) and by the Claus process:



In the first process, the scrubbing liquor can be regenerated, providing a concentrated stream of H<sub>2</sub>S that requires further treatment, whereas the Claus process presents the following disadvantages:

1. Hydrogen is essentially wasted, since H<sub>2</sub>O is produced from it.
2. The high temperatures and catalysts required do not offer flexible adjustment to varying concentrations of H<sub>2</sub>S.
3. A pretreatment for the separation of companion hydrocarbons and H<sub>2</sub> is required.
4. A posttreatment is required because the Claus process converts only 90–98% of the initial H<sub>2</sub>S content (9).

As shown in Figure 1, many pollutant-treatment schemes involving the electrochemical route have been studied (6, 9). In this experiment,  $\text{H}_2\text{S}$  will be produced and then absorbed, with simultaneous reaction whereby sulfide ions are oxidized by a chemical oxidant (mediator) to elemental sulfur. Then, elemental hydrogen will be produced at the cathode of an electrolytic cell and the oxidant will be simultaneously regenerated at the anode. The net result is rather uncommon: the decomposition of a pollutant ( $\text{H}_2\text{S}$ ) into its components in their pure, useful elemental forms. See the circled portion in Figure 1 (9, 13, 14).

## Experimental Procedure

Prepare with stirring a stock solution 0.5 M in KI and 0.1 M in  $\text{I}_2$  in 0.1 M HCl. This will be called the scrubbing solution. Pour 5 mL of this solution into a 10-mL beaker. Under a well-ventilated hood bubble through it  $\text{H}_2\text{S}$  gas, produced by conventional methods (e.g., by adding HCl to ZnS or pyrite). Alternatively, add slowly 5 to 7 drops of a concentrated sulfide solution (ammonium sulfide works well); this simulates the presence of  $\text{H}_2\text{S}$  in the scrubbing solution.

A striking change in color will occur within a few minutes owing to the oxidation of  $\text{S}^{2-}$  ions by  $\text{I}_2$  (or better, by the  $\text{I}_3^-$  ions), producing a suspension of yellow elemental sulfur and colorless iodide ions. Filter this suspension through a very fine filter paper and collect the filtrate in a 10-mL beaker. This will serve as the electrochemical cell for this experiment. The resulting filtrate will be electrolyzed with a 9-V battery or any electronic direct power source. Partially dip two electrodes (e.g., platinum foil, wire or mesh, lead rods or wires, graphite rods) into the solution. Do not let them touch each other! With alligator clips, connect one electrode to the positive terminal and the other to the negative terminal of the power source.

Turn the power on. If a regulated power source is used, maintain a voltage between 1 and 3 V. If graphite is used, maintain the voltage as low as possible (just enough to see a color change, as described below). Otherwise the graphite may disintegrate into pieces owing to the production of  $\text{O}_2$ , which diffuses into its basal planes, causing their rupture. Also, if a chlorine-like smell is noticed, decrease the applied potential because chloride ions from the HCl present in the solution can be oxidized to produce chlorine gas. After a short time, a dramatic color change (back to dark brown) is noted in the vicinity of the anode as a result of the re-oxidation of the  $\text{I}^-$  ions to  $\text{I}_2$  or  $\text{I}_3^-$ . Highly pure hydrogen is simultaneously produced at the cathode, which can be collected and tested in the traditional manner. The iodine solution is now ready to be reutilized for sulfide oxidation. This makes this experiment even more appealing from an environmental perspective. (We have performed at least 20 of these cycles with the same solution).

A similar method that can be used in the lab involves the absorption and oxidation of  $\text{H}_2\text{S}$  by a  $\text{FeCl}_3$  solution, with its concomitant regeneration (15). The end result is the same as that described above.

## Hazards

$\text{H}_2\text{S}$  is a very poisonous gas. It binds to hemoglobin where dioxygen should be bound, preventing dioxygen's uptake and

transport. Fortunately, this gas has a strong foul smell that warns of its presence. Prepare and use  $\text{H}_2\text{S}$  under the hood. It can lead to intoxication (and in extreme cases, death). Sulfide solutions must be handled with the same precautions.

## Summary

A microscale gas remediation system can be easily set up by using an indirect electrochemical oxidation step with an  $\text{I}_2/\text{I}_3^-$  solution, which is regenerated and reused. The polluting gas treated is  $\text{H}_2\text{S}$  (or a sulfide solution), and the end products are pure elemental sulfur and high-purity hydrogen. The net result is the decomposition of a hazardous gas into its components in their pure, commercially valuable elemental forms.

## Acknowledgments

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## Supplemental Material

Further information, problems, and detailed instructions for students are available in this issue of *JCE Online*.

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