

**LABORATORY EXPERIMENTS ON THE ELECTROCHEMICAL REMEDIATION OF THE ENVIRONMENT. PART 7. Microscale Production of Ozone.**

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Ozone, a powerful oxidizing/disinfecting agent, is produced electrochemically in the undergraduate laboratory with simple equipment and under very mild conditions. Tests are given to characterize it, to observe its action in simulated environmental applications, and to measure its rate of production.

<b>Keywords</b>	
<b>General Categories:</b>	<b>Specific Categories</b>
Environmental Chemistry	Microscale
Laboratory Instruction	Electrochemistry
	Gases
	Aqueous Solution Chemistry

# **Laboratory Experiments on the Electrochemical Remediation of the Environment. Part 7. Microscale Production of Ozone.**

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## **BACKGROUND**

Ozone is one of the most powerful oxidants and disinfectants. It has been used in Europe in drinking water treatment for over a century. Additional uses and advantages as an environmentally clean oxidant/disinfectant in water treatment and in several new processes in

chemical and pulp industries have increased its popularity (1-10). (See Tables 1 and 2 in the Lab Documentation).

Challenges encountered when using ozone include its hazards, cost, short decomposition time and low solubility (6, 9, 11). (See Table 3 in the Lab Documentation).

The main techniques used for the production of ozone (for more details, see Table 4 in the Lab Documentation) are:

A. *Corona discharge* (silent electric discharge process) (6, 11)



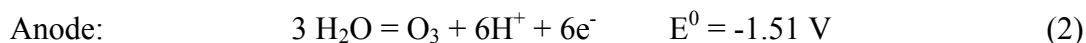
B. *UV irradiation* (8)

Ozone produced by UV-irradiation of air has a concentration in the range of a few thousand parts per million. This method is used for the production of small amounts of ozone.

C. *Electrochemical process* (2-4, 6, 9, 12-20).

Due to the high capital costs coupled with low ozone concentrations involved in the methods of production just described, the electrochemical route has been sought as an alternative.

(Advantages and challenges in using the electrolytic approach are summarized in Table 5 of the Lab Documentation).  $\text{PbO}_2$  grown on Ti or Pt substrates is the material of choice for electrode fabrication due to the simplicity of preparation and good efficiency. The reactions involved in the electrolytic production of ozone are (6):



Note that dioxygen is also evolved at the anode.

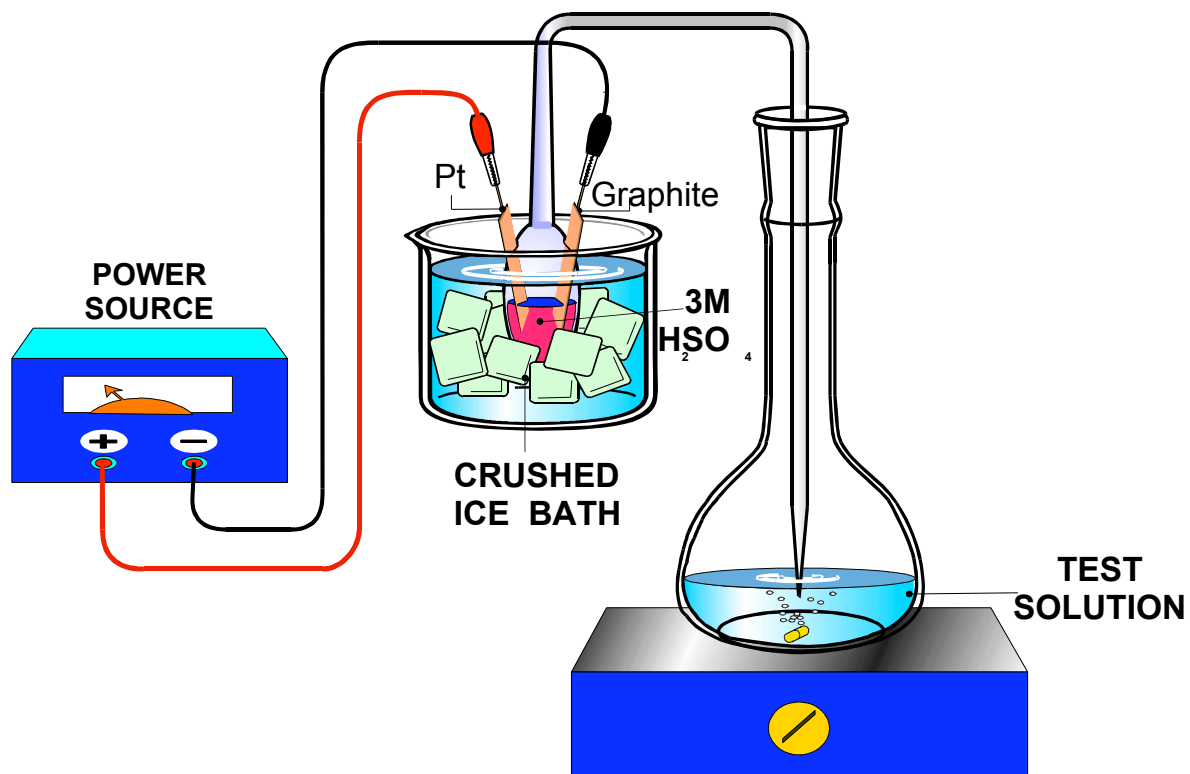
In light of these considerations and data, and since ozone destroys (oxidizes) organic materials whether they be rubber products that we don't wish to damage, organic contaminants in drinking water that we do wish to destroy, organic food colors, etc. it is clear that it would be important for students to be able to prepare and test ozone in a simple and safe way. Since the use of the traditional techniques (A and B) in a teaching laboratory is limited by equipment cost and/or safety requirements, we sought the electrochemical route to develop an experiment to produce and test ozone with rather simple equipment and materials. Here we describe an inexpensive and reliable method that generates microscale quantities of ozone in the steady-flow device so that ozone can be tested or used in a variety of experiments designed by the user. The device can be used to deliver ozone on demand (i.e., unplug the power source and you turn off the ozone; plug it back in and ozone is produced again). It is suitable for students taking General, Inorganic or Introductory Environmental Chemistry laboratories.

## EXPERIMENTAL AND RESULTS

### *Fabrication of the electrochemical cell for ozone production*

A cell is made with a plastic transfer pipet (thin stem Beral pipet) as the container, 3 M H<sub>2</sub>SO<sub>4</sub> as the electrolyte, a graphite rod from a pencil (or mechanical pencil) as the cathode, and a platinum wire as the anode. The power source is a 9V battery or a suitable DC power source. An ice bath is required to prevent softening of the plastic pipet (it may even melt) due to the high current passing through the system, and to increase the ozone yield. See Figure 1. With this, one can typically generate 7–8 mL gas/min. The ozone produced can easily be detected by its odor and by direct contact with a moistened starch-KI indicator paper, which immediately becomes dark. If the reaction is to be done for more than a few minutes, the use of a fume hood is necessary.

*Figure 1. Electrochemical cell schematics and set up.*



### *Ozone quantitation*

The rate and amount of ozone produced are analyzed by bubbling it through a solution containing I<sup>-</sup> ions. This produces I<sub>3</sub><sup>-</sup> ions, which then react with a standardized solution of S<sub>2</sub>O<sub>3</sub><sup>2-</sup> to a blue starch-KI indicator endpoint. The preparation of the solutions is given in the Lab Documentation.

### RESULTS AND DISCUSSION

### *Determination of the rate of ozone production*

A volumetric flask containing KI, H<sub>2</sub>SO<sub>4</sub> and a few drops of aqueous starch solution is used for ozone collection. A moistened starch-iodide indicator paper placed at the mouth of the flask serves to establish that ozone loss due to diffusion is not occurring. A buret containing Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> solution is positioned above the mouth of the flask. As the ozone is generated, it bubbles through the KI(aq) to produce KI<sub>3</sub>(aq), which is blue in the presence of starch. The reaction is:

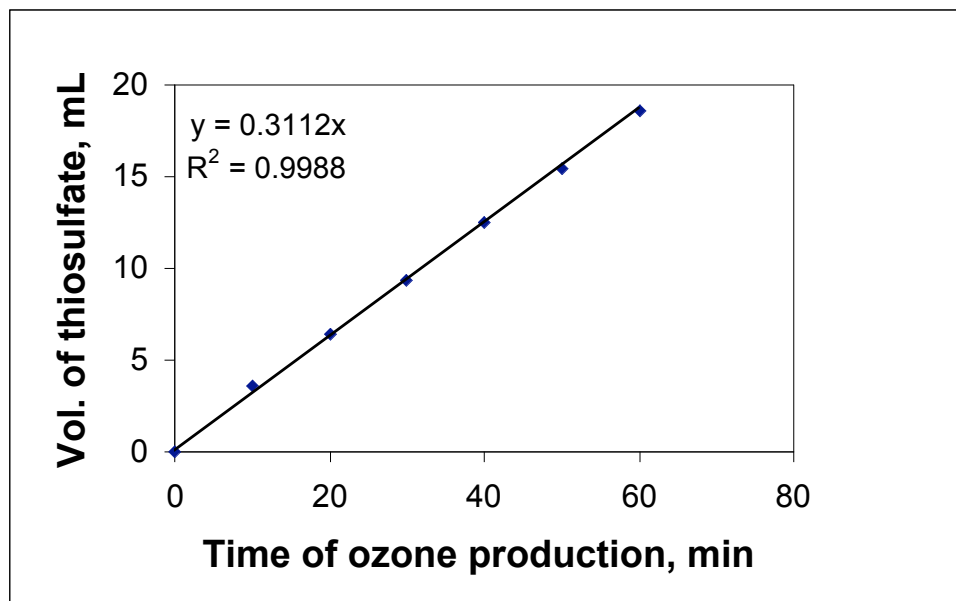


The titration with Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> solution can be conducted as the ozone is being generated with a new endpoint established as a function of time. Thus, the volumes of Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> solution required to attain the endpoints as a function of time for a typical ozone generator are given in Figure 2. The slope gives the rate of Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> solution added to be 0.311 mL/minute. Given the concentration of Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> solution (5.06 x 10<sup>-3</sup> M), the rate of Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> added in terms of moles is 1.57 x 10<sup>-6</sup> mol Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>/min. The ionic reaction between KI<sub>3</sub> and Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> is:



Thus, the rate of KI<sub>3</sub> production is determined to be 7.85 x 10<sup>-7</sup> mol KI<sub>3</sub>/min. Given the reaction between ozone and KI to produce KI<sub>3</sub> (above), the rate of ozone production is determined to be 7.85 x 10<sup>-7</sup> mol O<sub>3</sub>/min (785 nanomole/min). The rate of all gases produced was determined to be 7.6 mL/min that corresponds to 3.1 x 10<sup>-4</sup> mol/min at 300 K and 1 atm. Thus, ozone constitutes approximately 0.25% (or 2500 ppm) of the gases produced.

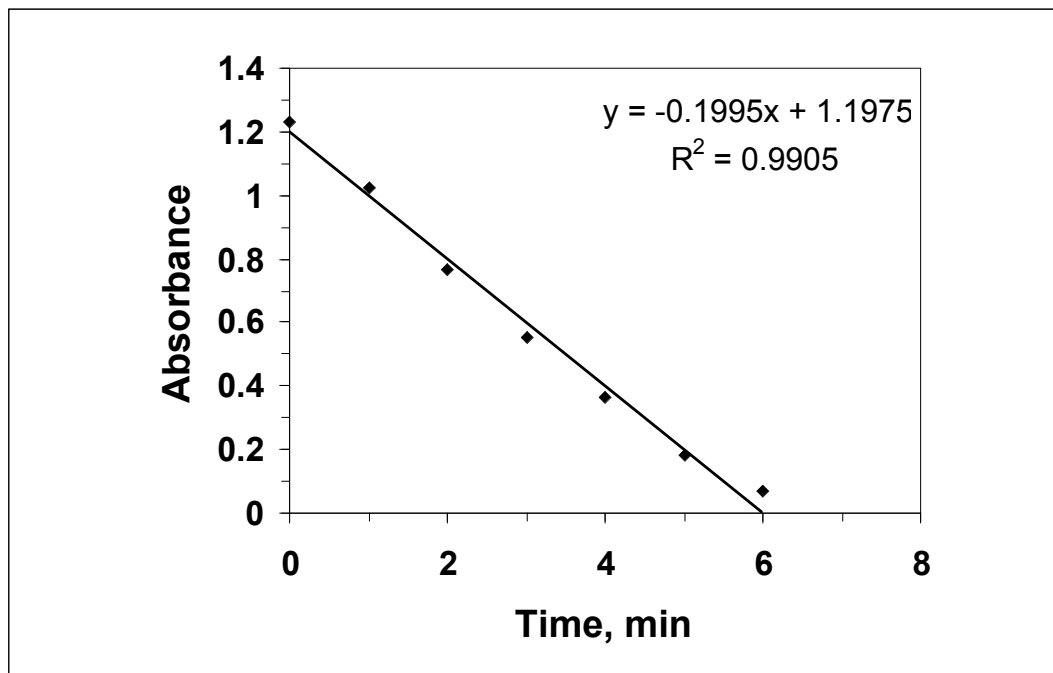
Figure 2. Rate of ozone production.



**Reaction between Blue Food Coloring and Ozone.** To observe the effect of ozone on some dyes, a solution of blue food coloring can be reacted with ozone.. The absorbance is measured at  $\lambda_{\max} = 630 \text{ nm}$  with a UV-VIS spectrophotometer. The sample is then returned to the flask for continued reaction. Typical results are shown in Figure 3.

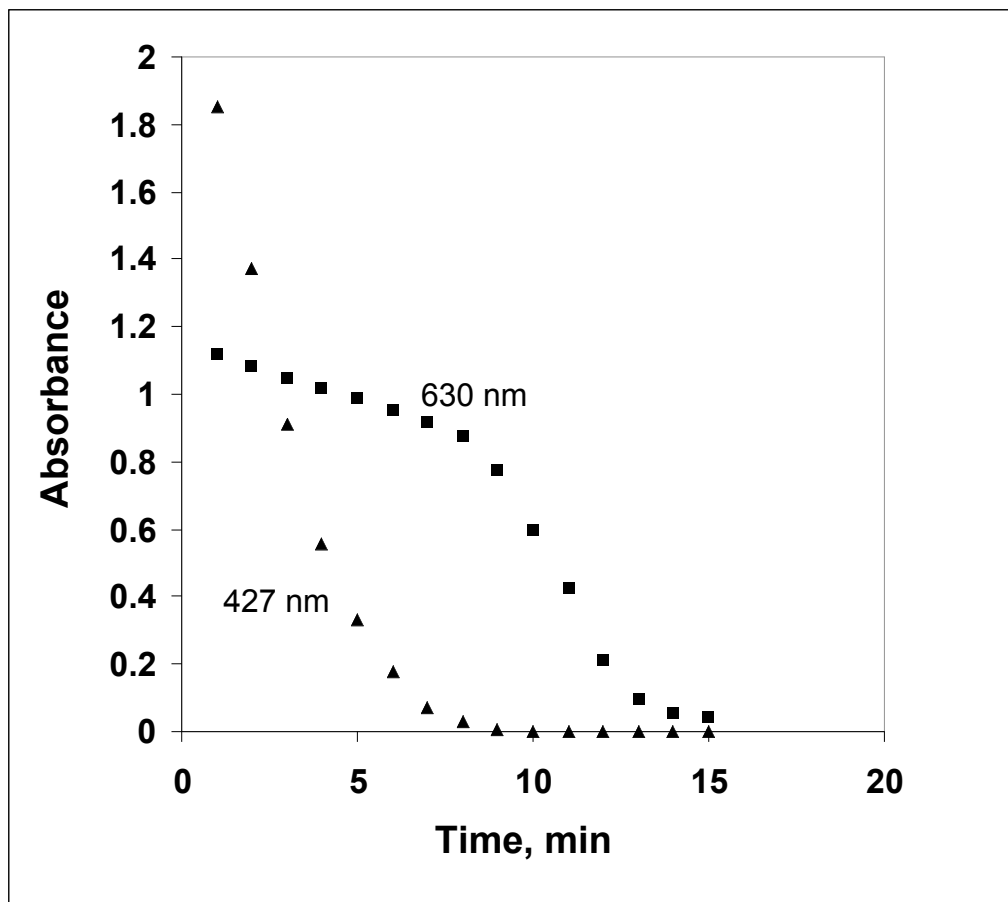
Figure 3. Absorbance decrease of blue food coloring with ozone.





**Reaction between Green Food Coloring and Ozone.** Green food coloring is a mixture of blue and yellow. The yellow color is far less reactive towards ozone than blue, so that solutions of green food coloring turn yellow then colorless. In an experiment analogous to the one described above for blue food coloring, one can monitor the absorbance of green food coloring at its two absorption maxima wavelengths, 630 nm and 427 nm. Typical results are given in Figure 4.

*Figure 4. Absorbance decrease of green food coloring with ozone.*



### *HAZARDS*

Ozone is explosive when concentrated, and its highly oxidizing power promotes strong interaction (frequently leading to degradation) with surrounding materials. Its oxidizing action is non-selective. The electrolyte solution (3 M sulfuric acid) is corrosive and should be handled with care. Eye protection must be used. The acid solutions resulting from the experiments for ozone production may be neutralized and discarded down the drain. The rest of the solutions can be discarded according to local regulations.

## CONCLUSIONS

We present a series of experiments for the production and testing of ozone in a laboratory session with simple equipment and materials.

## ACKNOWLEDGEMENTS

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