Preparation and Use of a Room-Temperature Catalytic Converter

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Air pollution is an environmental issue that captures the attention of students and offers the opportunity to teach fundamental chemical and instrumental concepts in an interesting and meaningful way. Articles in this *Journal* demonstrate that gas chromatography (*1*) and infrared spectroscopy (*2–4*) can be used to analyze polluted gas samples for contaminants while teaching fundamental chemical concepts.

An example of an undergraduate laboratory concerning pollution abatement using heterogeneous catalysis (the catalytic oxidation of sulfur dioxide by activated carbon) has also been published (*5*). Automotive air pollution has been a particular concern for the last quarter of a century, and the catalytic converter has had a significant impact on the reduction of emission levels during this time. Since 1975, all cars in this country have been equipped with catalytic converters and U.S. emission levels of hydrocarbons, carbon monoxide, and nitrogen oxides have diminished by 98, 96, and 90% respectively (*6*). Considering the environmental impact of catalytic converters, the function of catalysis in these devices is likely to be a topic of great interest to students.

Catalytic converters are not effective while cold and it takes two to three minutes for the average catalytic converter to reach operating temperatures. Because of this, much of a car's emissions occurs during this time. As environmental restrictions increase, one way that automobile manufacturers can reduce emissions is to reduce emissions during this start-up time. Thus, there has been a significant effort lately to prepare catalytic converters that will work at room temperature. A particularly interesting and easy-to-prepare catalyst system was reported by the group of M. Haruta (*7*). The Au/Fe_2O_3 catalyst system reported by that group is capable of converting CO into CO_2 at very low temperatures.

Herein, we present an experiment based on the catalyst prepared by Haruta et. al. The student prepares a Au/Fe_2O_3 catalyst and then passes a CO/air mixture over the catalyst and observes the conversion of CO to CO_2 using infrared spectroscopy. The chemicals are inexpensive and the flow cell is easily prepared from readily available supplies. A durable and inexpensive gas-phase infrared cell has been described separately (*4*). The students learn about methods of solid-state inorganic synthesis, infrared spectroscopy, and catalysis and gain a better understanding of a catalytic converter. The laboratory was written for a sophomore- or junior-level inorganic or environmental laboratory class but can be modified for an introductory class by preparing some of the materials for student use ahead of time.

Experiments and Results

Reagents and Equipment

The $HAuCl_4 \cdot 3H_2O$ and $Fe(NO_3)_3 \cdot 9H_2O$ required for the preparation of the catalyst are available through Aldrich. Because of the large Fe/Au ratio in the catalyst, 1 g of gold salt is enough to prepare at least six batches of the catalyst. Lecture bottles of CO are available through Matheson Gas Products. The plastic syringes, three-way stopcocks, and syringe needles can be purchased from Fisher and the syringe-to-needle adapters from Aldrich. The gas-phase IR cell is easily prepared from materials that can be purchased from a hardware store and is described separately (*4*). The IR investigations were performed using a Mattson Genesis IR spectrophotometer. One spectrometer per 24 students is preferable.

Experimental Procedure

Owing to time constraints on the first day, the instructor should prepare stock solutions of all three reagents, 0.055 M $HAuCl_4 \cdot 3H_2O$, 0.11M $Fe(NO_3)_3 \cdot 9H_2O$, and 0.2 M Na_2CO_3. The catalyst is prepared by addition of a mixture of 10 mL of the Au(III) solution and 90 mL of the Fe(III) solution to 300 mL of the Na_2CO_3 solution at about 70 °C. Heating for one hour results in the homogeneous coprecipitation of the hydroxides of iron and gold. The coprecipitate is then washed with distilled water to remove excess carbonate and chloride ions. After vacuum filtration, the sample is dried for

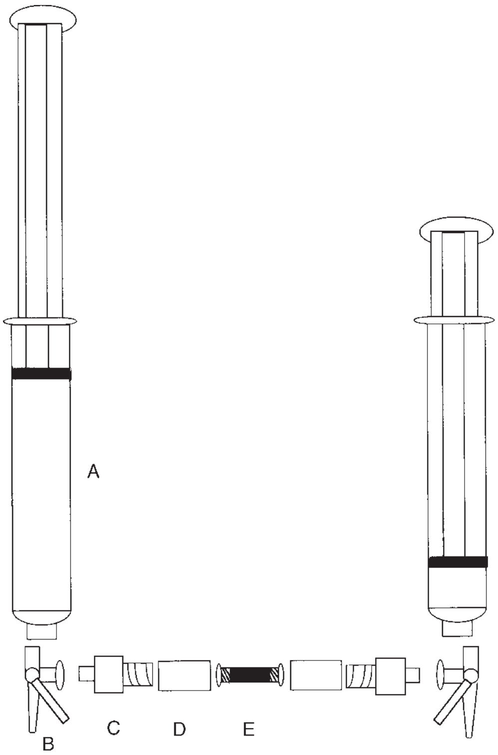

Figure 1. Apparatus designed to pass the CO/air mixture through the catalyst flow cell: (A) 20-mL plastic syringe; (B) 3-way plastic syringe stopcock; (C) needle to hose adapter; (D) rubber tubing; (E) catalyst packed into glass tubing.

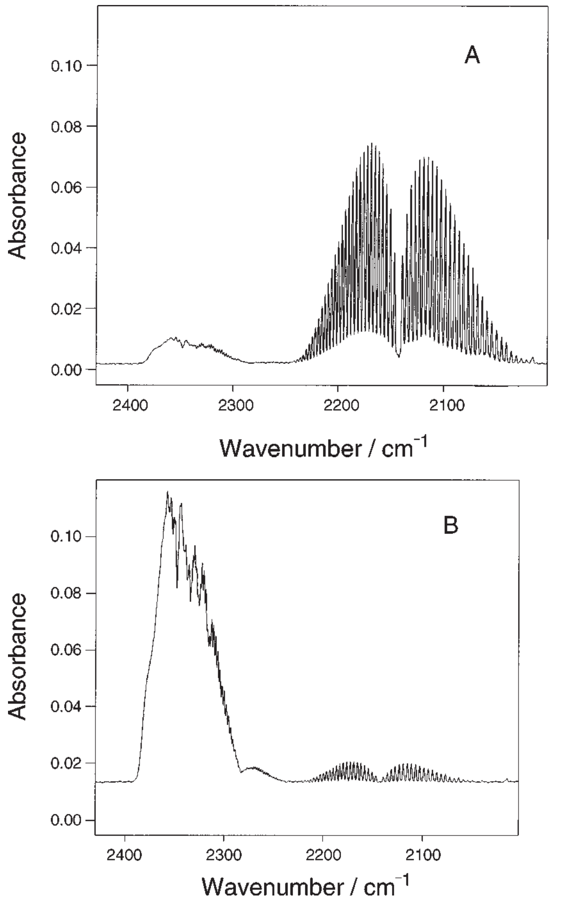

Figure 2. Infrared spectra of CO/air mixture (A) before and (B) after two passes through the catalyst. Note that the bands due to CO (centered at 2143 cm^{-1}) decrease and the bands due to CO_2 increase.

1 h at 120 °C and then calcined for 4 h at 400 °C. During calcination, the metallic hydroxides are dehydrated to oxides, and the gold oxide is decomposed further to metallic gold and oxygen. The catalyst itself is a mixture of iron(III) oxide impregnated with nanometer-sized gold particles (*7*).

The catalyst is packed into a small glass tube and held in place using glass wool plugs at both ends. This catalyst flow cell is then attached between two plastic syringes so that a 4% CO/air (v/v) mixture can be pushed back and forth through the catalyst (Fig. 1). The CO/air mixture can be prepared by injecting via a syringe 20 cm^3 of pure CO from a lecture bottle into a 500-mL round bottom flask covered with a septum.

The gas sample is then transferred into a gas-phase IR cell and its spectrum is compared to that of a sample that has not been passed through the catalyst. Typical results are shown in Figure 2. Note that the sample that has not been passed through the catalyst does have some CO_2 present owing to background levels of CO_2 in air. After passage of the CO/air mixture through the catalyst tube just twice, note that substantial conversion has occurred. Both the reduction of the CO bands and the increase of the CO_2 bands are observed, clearly demonstrating the catalysis of the CO oxidation reaction.

If time permits, the students can choose to do an additional experiment. For example, they could first predict what might happen if no O_2 is present in the gas mixture and then perform the same test on a CO/N_2 mixture. Though the catalytic oxidation requires oxygen, they will find substantial conversion due to adsorbed oxygen. This is an opportunity to discuss the role of adsorbed species in heterogeneous catalysis.

The experiment as written is designed for two laboratory periods, each of at least three hours' duration. Providing students with prepared catalyst tubes can shorten the lab to a single period. In fact, an inorganic laboratory class can prepare and test the catalyst tubes, which can then also be used in lower-division courses.

Hazards

Although CO is a hazardous substance, the risk of this experiment for students is small if the students are presented with a 4% CO-in-air mixture as suggested. In this way, the small amounts of CO used by students offer a nearly negligible safety risk, simply requiring disposal in a hood. The lecture bottle of CO handled by the instructor poses a more significant health hazard and should be used and stored with the appropriate precautions. The hydrogen tetrachloroaurate ($HAuCl_4$) is corrosive and an irritant to the skin, mucous membranes, and eyes. Care should be taken to avoid contact during the preparation of the solution.

^{W}Supplemental Material

The complete description of this experiment, with an equipment list, prelab and postlab questions, and notes for the instructor, is available in this issue of *JCE Online*.

Acknowledgments

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