

Demonstrating Concepts in Catalysis, Renewable Energy, and Chemical Safety with the Catalytic Oxidation of Hydrogen

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Cite This: *J. Chem. Educ.* 2021, 98, 2036–2041



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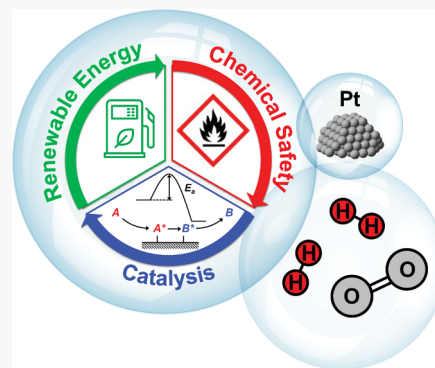
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Supporting Information

ABSTRACT: A versatile and portable apparatus was developed to demonstrate exciting visual displays of catalytic phenomena that introduce basic concepts in catalysis, renewable energy, and chemical safety, in order to pique scientific curiosity in a variety of audiences including middle and high school students, undergraduate and graduate students, and the general public. The demonstration uses the platinum-catalyzed oxidation of hydrogen by oxygen as a model reaction to illustrate concepts in thermodynamics, reaction kinetics, and electrochemistry. The apparatus was designed to contain inherent safety features and the versatility to adopt several configurations to perform a wide range of experiments in classroom settings, informal science education activities, and public outreach events. Soap bubbles are used to confine small, controlled volumes ($<35\text{ cm}^3$) of hydrogen/oxygen gas mixtures and probe the reactivity of bulk and nanoparticle forms of platinum to illustrate how high metal surface areas increase catalytic reaction rates. In parallel, a hydrogen/oxygen proton-exchange fuel cell is used to demonstrate how chemical energy released from the exergonic hydrogen oxidation reaction can be converted into electricity by using a platinum-containing electrocatalyst. With adjustments of the operating configuration and the experimental conditions of the apparatus, the demonstrations can be tailored toward target demographics of varying scientific proficiency to emphasize specific learning objectives for topics in reaction chemistry and engineering. Potential hazards and important safety precautions are addressed.

KEYWORDS: General Public, High School/Introductory Chemistry, First-Year Undergraduate/General, Demonstrations, Public Understanding/Outreach, Hands-On Learning/Manipulatives, Misconceptions/Discrepant Events, Catalysis, Electrochemistry, Kinetics



Catalysts are vital to society and contribute significantly to value creation; they are responsible for nearly 90% of all chemical product manufacture and up to 20% of the economic activities in industrialized countries.¹ A catalyst accelerates the rate of a chemical reaction without itself being consumed, by facilitating the adsorption and reaction of molecules at active sites that provide access to lower energy pathways to transform reactants into products that would otherwise be inaccessible in uncatalyzed or thermal routes.² This process is illustrated by the reaction coordinate diagram in Figure 1 for the case of hydrogen oxidation to produce water ($2\text{H}_2 + \text{O}_2 \rightarrow 2\text{H}_2\text{O}$, $\Delta G_r^\circ = -237\text{ kJ mol}^{-1}$).

When students and the general public are introduced to catalysis, it is often presented to them in the context of a specific application like renewable energy, environmental remediation, or sustainable manufacturing, before introducing the fundamental concepts of catalysis science. While such discussions of practical importance are useful for emphasizing the impact of catalysis and may be relatable to the audience, they can lead to misconceptions regarding the thermodynamic and kinetic implications of catalysis^{3–6} unless some of the basic underlying principles are also addressed. In certain cases, visual demonstrations can be useful supplements to traditional

lecture-based instruction by providing connections between theoretical concepts and observables that enable prediction, clarify discrepant events, aid in knowledge retention, and promote scientific curiosity through entertaining displays.^{7–9} For example, exploding gas balloons containing mixtures of either H_2 or CH_4 with O_2 are among the most popular demonstrations in introductory chemistry courses to illustrate topics such as stoichiometry, activation energy, and reaction enthalpy.^{10–13} Although dramatic and loud, these exploding balloons can pose significant safety risks due to the large volume of gas used ($\sim 3,000\text{--}14,000\text{ cm}^3$), the presence of an open flame required to ignite the gas mixture, and significant sound pressure levels ($>128\text{ dB}^{11}$) that are generated indoors. Demonstration variants that use eggshells¹⁴ or plastic bottles¹⁵ to limit volumes of H_2/O_2 mixtures to $\sim 60\text{--}1,000\text{ cm}^3$ have

Received: February 23, 2021

Revised: April 22, 2021

Published: May 17, 2021



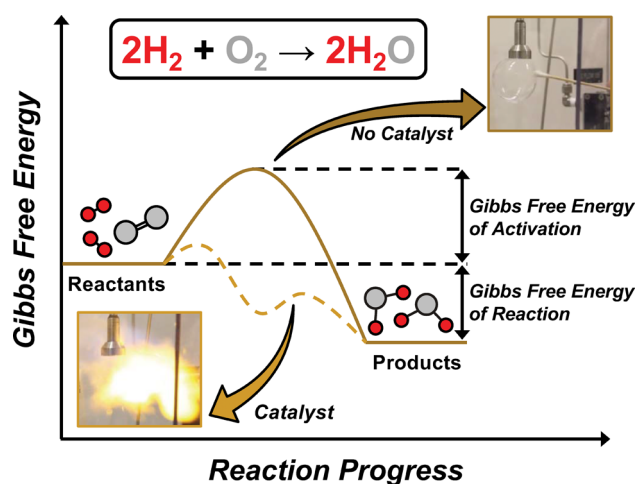


Figure 1. Reaction coordinate diagram to illustrate the uncatalyzed and catalyzed reaction pathways for the oxidation of H_2 .

been shown to reduce peak sound pressure levels when performed behind protective barriers at distances >2 m, but in certain cases may still exceed the Occupational Health and Safety Administration's (OSHA) impulsive noise limit of 140 dB.¹⁶ Furthermore, only a narrow range of scientific concepts can typically be demonstrated by these experiments.

The demonstration apparatus described herein uses the catalytic oxidation of H_2 as a model reaction to link visual displays of catalytic phenomena with fundamental concepts in catalysis, electrochemistry, thermodynamics, and renewable energy while promoting safety in the practice of chemistry and engineering. The unit is lightweight and portable and was designed with safety features, while containing the versatility to provide several different experimental configurations for the demonstrator. H_2 and O_2 lecture bottles equipped with pressure regulators, check valves, flame arrestors, and flow meters are used to supply the reacting gases at the desired partial pressures in a carefully controlled manner. Soap bubbles are used to contain H_2/O_2 gas mixtures in a similar fashion to an exploding gas balloon but in smaller, limited volumes (<35 cm^3). The energy released from the exploding H_2/O_2 gas bubble upon reaction with a Pt catalyst is equivalent to approximately one-half of the energy released from detonation of a single consumer firecracker with a sound level of 89 ± 5 dB at a distance of 3 m. In subsequent experiments, H_2 and O_2 are supplied to a proton-exchange membrane (PEM) fuel cell to produce electricity by using a Pt electrocatalyst. Over the course of the past several years, we have safely and successfully performed these demonstrations with the apparatus before hundreds of spectators at Purdue University during course lectures, research seminars, public events on campus, and various informal science education activities for K–12 students sponsored by Purdue's Women in Engineering Program.

EXPERIMENTS

Description of Demonstration Apparatus

The catalytic demonstration apparatus consists of two small, compressed gas cylinders (lecture bottles, Flinn Scientific, $\geq 99.99\%$) of H_2 and O_2 , each equipped with independent flow control and safety devices including isolation valves, pressure regulators, flame arrestors, check valves, and rotameters; a nozzle to mix the exiting reactant gases; and an educational H_2

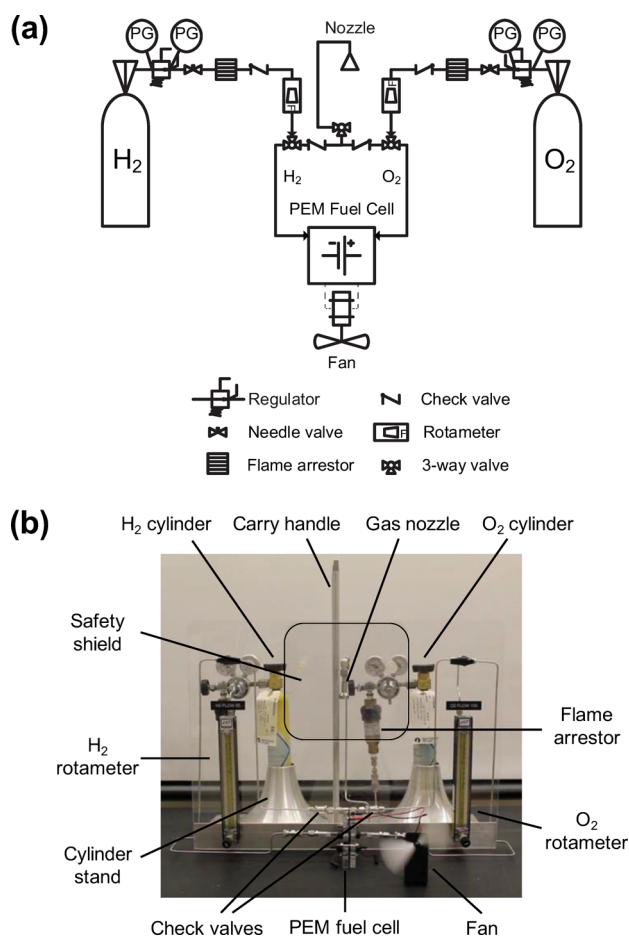


Figure 2. (a) Schematic and (b) photo of the catalytic demonstration apparatus.

PEM fuel cell kit (H-TEC, F107) as shown in Figure 2. The apparatus is fixed to an aluminum base plate equipped with an aluminum carry handle and impact-resistant plastic safety shields that make it lightweight, portable, and easily viewable. These features allow the apparatus to operate safely in several different configurations under a wide range of experimental conditions to demonstrate various scientific and engineering concepts as listed in Table 1. Additionally, an itemized price list of all parts and equipment needed to construct the complete demonstration apparatus can be found in Table S.1 (Supporting Information Section 1).

Experimental Procedure: H_2/O_2 Gas Bubble

A video demonstrating the use of the apparatus in each operating configuration can be viewed at the link provided in Section 2 of the Supporting Information. Additionally, a complete set of instructions for the demonstrator to perform these experiments and transport the apparatus can be found in Section 3 of the Supporting Information. In the first configuration, pure H_2 and O_2 are supplied from lecture bottles at low delivery pressures (<15 psig) to the gas exit nozzle by means of independent 3-way isolation valves as shown in Figure 2a. The desired gas composition is achieved by adjusting the individual gas flow rates with needle valves on the respective rotameters. A H_2/O_2 gas bubble is formed by pouring a small amount of soap solution into a dish and touching the surface of solution to the gas exit nozzle while the gases are flowing. Once the bubble fills to a diameter of ~ 3 –4

Table 1. Experimental Capabilities for the Catalytic Demonstration Apparatus

Configuration	Experimental Conditions	Concepts Demonstrated
Gas bubble	Blank (no catalyst)	Activation energy, reaction kinetics
	Bulk metal (Pt ribbon)	Active surface area, molecular binding sites
	Pt catalyst	Activation energy, reaction kinetics, role of catalyst, Gibbs free energy of reaction
	Vary P_{H_2} , P_{O_2} with Pt catalyst	Stoichiometry, upper/lower explosivity limits (UEL/LEL), chemical safety
Fuel cell	Electrical device (fan or light)	Electrochemistry, role of electrocatalyst, green energy
	Vary P_{H_2} , P_{O_2} with multimeter	Fuel cell thermodynamics, electrochemical device performance
Electrolyzer ^a + fuel cell	H ₂ O splitting and H ₂ oxidation in series	Energy storage and conversion, renewable energy

^aOptional add-on accessory that can be purchased to provide alternative source of H₂ and O₂ directly from H₂O.

cm ($\sim 15\text{--}35\text{ cm}^3$), the 3-way valves are closed to stop the flow of H₂ and O₂. It may be possible to generate larger gas bubbles, but they are less stable and tend to burst before the demonstration can be completed. First, a clean, cotton-tipped application swab ($\geq 15\text{ cm}$) is used to burst the gas bubble and show that no observable reaction occurs due to the large activation barrier for the thermal (i.e., uncatalyzed) pathway (Figure 1). Next, the same sequence is repeated, but by using a pair of tweezers or forceps to hold a small ($\sim 1.2\text{ cm}^2$) piece of bulk Pt metal (e.g., ribbon, wire) and burst the H₂/O₂ gas bubble to illustrate that no observable reaction occurs due to the slow kinetics as a result of the low metal surface area and dearth of favorable molecular binding sites for H₂ and O₂. Last, the sequence is repeated, but the tip of a clean application swab is coated with a small amount ($\sim 5\text{ mg}$, $\sim 1,250\text{ cm}^2$) of Pt black powder (Sigma-Aldrich, $\geq 99.95\%$) catalyst and touched to the surface of the gas bubble causing it to detonate with a loud bang and a bright flash.

Experimental Procedure: H₂/O₂ PEM Fuel Cell

In the second configuration, the H₂ and O₂ supply lines from the 3-way valves are connected to the PEM fuel cell anode and cathode, respectively. Depending on the fuel cell used, the polymer electrolyte membrane separating the anode and cathode may need to be activated in deionized H₂O before use in order to hydrate the ionic groups on the proton-conducting polymer surface and facilitate proton transport through hydrogen bonding.^{17,18} Tap water contains ions and other dissolved species that can irreversibly bind to the polymer electrolyte and inhibit device performance. The demonstrator may note to the audience that the solid polymer (rather than liquid H₂O) functions as the electrolyte in a PEM fuel cell to conduct protons from the anode to the cathode.

A demonstration device such as a small fan, light, or multimeter is connected across the fuel cell electrodes at the appropriate terminals. The 3-way isolation valves for the H₂ and O₂ supply are opened to direct the gases to the fuel cell and observe the immediate response in the demonstration device (e.g., fan, light, multimeter) as electricity flows within the circuit. As with the H₂/O₂ gas bubbles, the desired gas composition is achieved by adjusting the flow rates with the individual rotameters. The 3-way valves are then closed to stop

the flow of gases. When shutting down the apparatus at the conclusion of the demonstration or for transport, the H₂ and O₂ lecture bottle supply valves should be closed and the system slowly depressurized by allowing the small amount of residual gas to be consumed in the fuel cell.

HAZARDS AND SAFETY PRECAUTIONS

H₂ is extremely flammable across a wide range of concentrations (4–75 vol % in air) and can ignite with very low energy input. Additionally, oxygen-enriched atmospheres ($>23.5\text{ vol \% O}_2$) are highly reactive and can increase the risk of ignition and fire, even in the presence of low volatility oils and greases. The U.S. Chemical Safety and Hazard Investigation Board (CSB),¹⁹ the National Fire Protection Association (NFPA),²⁰ and the American Chemical Society Division of Chemical Education^{21,22} have put forth guidelines and recommended best practices regarding educational demonstrations involving the use of flammable and reactive chemicals. These recommendations specifically address the need to conduct a comprehensive hazard review and risk assessment of the demonstration, implement strict safety protocols including the use of PPE and safety barriers, minimize the quantities of flammable and hazardous chemicals, and follow rehearsed written procedures. For the present demonstration, the Hazard Analysis and Pre-Startup Safety Review (Supporting Information Section 4) identified the use of flammable (flash point $<22\text{ }^\circ\text{C}$) and oxidizing gases, the handling of compressed gas cylinders ($>2\text{ bar}$), and the potential for energy release from desired and undesired reactions as the primary risks to be mitigated through engineering and administrative controls along with proper PPE.

To address these hazards, the demonstration apparatus was constructed from appropriate materials, and passive safety devices such as flame arrestors, redundant check valves (i.e., back-flow prevention), and impact-resistant shields were installed. Lecture bottles are used to minimize the volume of stored H₂ and O₂ gas while allowing the composition of the gas mixtures to be adjusted for various experiments. Unlike the traditional exploding gas balloons that consume large volumes of flammable gas, require an open flame to ignite, and generate extremely loud sound levels, the soap bubbles used in the present apparatus limit the volume of explosive gas mixture to $\sim 35\text{ cm}^3$ or less. Approximately 270 J of energy is released upon detonating a gas bubble of this size with a 2:1 volume ratio of H₂:O₂ compared to an estimated 530 J of energy released by a single consumer firecracker containing 50 mg of flash powder^{23,24} (see Supporting Information Section 4). Thus, the explosion is loud enough to startle and entertain spectators but not so loud as to risk damage to their hearing.^{11,14–16}

Before using the apparatus, the instructor should first verify the absence of leaks by (i) opening the H₂ and O₂ cylinder valves and using the gas regulators to pressurize the system ($\sim 30\text{--}50\text{ psig}$), (ii) closing the cylinder valves and monitoring the pressures on the respective gauges, (iii) applying a small amount of soap solution to the connections and monitoring the formation of bubbles, and then (iv) slowly depressurizing the system by opening the 3-way isolation valve immediately upstream of the gas exit nozzle to vent the residual gas (Figure 2a). Prior to performing the demonstration in front of a live audience, the instructor should thoroughly rehearse each experiment by using the instructions provided (see Supporting

Information Section 3). Verify that the area is well-ventilated with a fire extinguisher nearby in the event of an emergency. The demonstration apparatus should be placed at least 3 m away from the audience in a location that does not block the path of egress. Recommended PPE for the instructor consists of safety glasses, a fire-retardant lab coat, gloves, long pants or a dress, and closed-toe shoes.

DISCUSSION

For members of the general public and K–12 students with limited chemistry background, the pedagogical goals of these demonstrations are primarily to (i) generate excitement in science, (ii) raise public awareness on the importance of catalysis, and (iii) engage the audience in the topics being discussed while introducing some basic principles in reaction chemistry, kinetics, and energy storage and conversion. The demonstrations serve as live visuals to accompany slideshow presentations at research seminars and scientific outreach events for middle and high school students, where these concepts are subsequently incorporated into hands-on activities involving the catalytic decomposition of hydrogen peroxide.²⁵ Spectators are able to actively participate in the demonstrations by making predictions and observations, thinking critically about relationships between evidence and explanations, and asking questions to address misconceptions and erroneous beliefs.^{3–6,8,9}

For example, even though metal surfaces can catalyze a variety of reactions, bulk noble metals (e.g., Pt, Pd, Au) are often relatively inert compared to their nanoparticle forms, which is why they are used in jewelry. Many audience members predict that a metallic Pt ribbon will catalyze the oxidation of H_2 and are puzzled when no observable reaction occurs, yet they observe that the reaction occurs rapidly and dramatically as evidenced by the exploding gas bubble upon contact with a Pt black catalyst of identical composition as the metallic ribbon. We often invite those in the audience wearing Pt jewelry to test it for further evidence, but most are reluctant to do so! As illustrated by the reaction coordinate diagram in Figure 1, even though the thermodynamics of the uncatalyzed hydrogen oxidation reaction are exergonic ($\Delta G_r^\circ < 0$), the kinetics at ambient temperature are extremely slow due to the large Gibbs free energy of activation. The Pt black catalyst provides a lower energy pathway to increase the reaction rate by decreasing the Gibbs free energy of activation without affecting the reaction thermodynamics. While bulk metal forms of Pt (e.g., ribbon, wire, mesh) also catalyze hydrogen oxidation,^{26–29} their specific surface areas (i.e., number of exposed metal atoms per unit mass) are much lower those associated with finely divided Pt powders and nanoparticles, resulting in lower reaction rates (per mass). By comparison, the Pt black catalyst ($\sim 25 \text{ m}^2 \text{ g}^{-1}$) used for this demonstration has $\sim 1,000\times$ greater surface area (per mass) than the Pt ribbon ($\sim 2.5 \times 10^{-2} \text{ m}^2 \text{ g}^{-1}$), thus greatly increasing the total number of adsorption–surface reaction–desorption events per unit mass per time. An application of this catalytic reaction of historical significance was the development of self-lighting mantles for gas lamps, ovens, and matchless pocket lighters. A small amount of Pt black catalyst was incorporated into these mantles to raise the temperature of coal or town gas to its ignition point by rapidly combining H_2 in the gas with air to generate heat.^{30,31} Heated Pt filaments were also added to the mantles to improve their reliability by maintaining the temperature of the H_2 -containing gas as the Pt black catalyst

would sinter and lose active surface area with continued use.^{31,32}

The exploding gas bubble in the present demonstration represents a combination of several phenomena. First, the high surface area Pt black powder facilitates the adsorption of H_2 and O_2 and rapidly raises the local temperature at the gas–solid interface as a result of the exothermic enthalpies of adsorption^{33,34} and reaction ($\Delta H_r^\circ = -286 \text{ kJ mol}^{-1}$). Next, the temperature at the interface increases above the auto-ignition temperature of H_2 ($\sim 560^\circ \text{C}$) and ignites the contacting gas layer to generate a small volume of high temperature free radicals within the combustion zone. Last, this flame kernel initiates a detonation wave that propagates into the adjacent unburned gas mixture by thermal and free radical diffusion, leading to accelerated reactions and eventually to explosion.^{35,36} As a result, the experiment demonstrates how the higher metal surface areas and favorable molecular binding sites associated with nanoparticle forms of Pt increase catalytic H_2 oxidation rates to initiate this rapid reaction cascade compared to the bulk metal as illustrated in Figure 3a. The chemical energy released from this exergonic

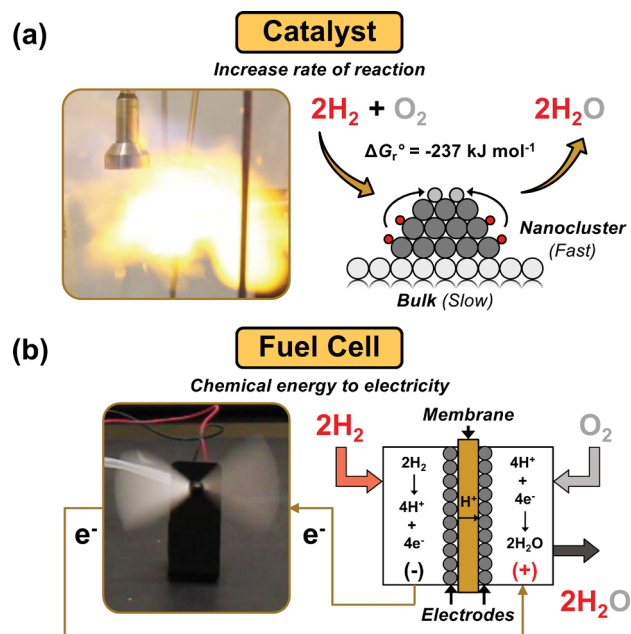


Figure 3. Use of the catalytic oxidation of H_2 to illustrate (a) the role of small, active catalytic domains in accelerating the rate of reaction, and (b) power generation by electrocatalysts in fuel cell devices.

reaction can also be converted into usable electricity with H_2O as the only byproduct by using a Pt-containing electrocatalyst in a PEM fuel cell device as shown in Figure 3b. Furthermore, adding an educational H_2O electrolyzer kit to the demonstration apparatus for on-demand generation of H_2 and O_2 allows the audience to see how H_2 can function as an energy storage medium for electrical grid balancing by using surplus renewable energy from wind and photovoltaic sources to drive the electrolysis reaction.^{37–40}

The catalytic demonstration apparatus is also a useful supplement for undergraduate and graduate courses in chemistry and chemical engineering to illustrate key concepts in reaction kinetics, electrochemistry, and chemical safety. For these advanced audiences, the demonstration objectives can be tailored to (i) provide intellectual stimulation by connecting

fundamental principles with observable phenomena, (ii) challenge assumptions or mistaken beliefs that can become entrenched through traditional lecture instruction, and (iii) develop proper safety practices in the handling of hazardous materials and operation of laboratory and chemical process equipment. For instance, students can be asked to consider how the Pt-catalyzed reactions in the gas bubble and the fuel cell behave differently even though the same reactants, catalytic surfaces, and conditions are present in both systems shown in Figure 3. Unlike the gas phase chemical reaction with H_2 and O_2 where charge transfer occurs between the neighboring adsorbates that react on the Pt surface, the electrochemical reaction involves charge transfer between individual adsorbates and the Pt electrodes.^{41,42} Electrons liberated at the Pt anode flow through the closed circuit with a current density (i.e., amperes per unit area) that is proportional to the active interfacial surface area and is analogous to the observed reaction rate in the heterogeneous catalyst system (Figure 3b). Fuel cell thermodynamic and device performance measurements can be conducted by connecting a multimeter across the terminals for the anode and cathode and, then, varying the H_2 and O_2 partial pressures (P_{H_2} , P_{O_2}) to measure changes in the fuel cell response. The concepts of electrical work (W_{el} , J mol^{-1}) and fuel cell efficiency (η) can be demonstrated³⁸ by first measuring the voltage across the cell according to $W_{\text{el}} = -nEF$, where E is the voltage (V), n is the number of electrons transferred (moles), and F is Faraday's constant (96,485 C mol^{-1}). Then, by dividing W_{el} for the electrochemical reaction from the heat released by the enthalpy of reaction (ΔH_r), the fuel cell efficiency can be calculated according to $\eta = \left| \frac{W_{\text{el}}}{\Delta H_r} \right|$. Additionally, the rate of hydrogen consumption (r , moles s^{-1}) can be calculated^{40–42} by measuring the current (i , C s^{-1}) produced according to $r = \frac{i}{nF}$.

In parallel to these demonstrations, the apparatus can be used to introduce students to important chemical safety practices by tracing flow paths with process flow diagrams and laboratory equipment, discussing the workings of process safety devices, examining the lower and upper explosivity limits (LEL/UEL) for H_2/O_2 mixtures, and performing energy release calculations during safety reviews.

CONCLUSIONS

A lightweight, portable demonstration apparatus is presented for performing a range of experiments to introduce basic principles in catalysis, electrochemistry, thermodynamics, and renewable energy with the aim of promoting scientific curiosity among different types of audiences with varying levels of chemistry proficiency. The unit is designed with multiple safeguards to enable safe and reliable operation according to guidelines and best practices established by the U.S. Chemical Safety and Hazard Investigation Board (CSB), the National Fire Protection Association (NFPA), and the American Chemical Society Division of Chemical Education for conducting educational demonstrations with flammable and reactive chemicals. The Pt-catalyzed oxidation of H_2 is used as a model reaction to provide connections between fundamental concepts in reaction chemistry with entertaining visual displays that can be used as a supplement to classroom instruction, an educational tool at public outreach events, and an introductory component at broader informal science education programs that includes hands-on activities in catalysis and renewable

energy. We have safely and effectively performed these demonstrations in front of hundreds of spectators on numerous occasions at Purdue University and received positive feedback from those in attendance.

ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available at <https://pubs.acs.org/doi/10.1021/acs.jchemed.1c00188>.

Itemized price list, link to demonstration video, demonstration instructions, hazard analysis and prestart-up safety review, and additional notes for the demonstrator (PDF)

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Notes

The authors declare no competing financial interest.

ACKNOWLEDGMENTS

We acknowledge financial support from the National Science Foundation CAREER program under Award 1552517-CBET, the National Science Foundation under Cooperative Agreement EEC-1647722 (an Engineering Research Center for the Innovative and Strategic Transformation of Alkane Resources), and the Purdue University Student Grant Program for Community Service/Service Learning Projects to purchase the materials and supplies to fabricate the apparatus and offer multiple demonstrations. We thank Casey Jones (Purdue University) for assistance with the demonstration and for helpful technical discussions.

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