

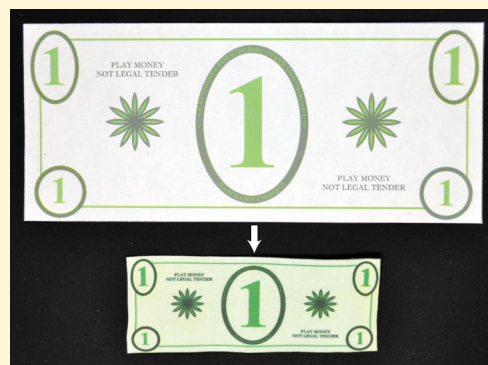
How To Shrink Paper Money: A Macroscopic Demonstration of the Malaprade Reaction

E. Brandon Strong,[†] Brittany A. Lore,[†] Emily R. Christensen,[†] Nathaniel W. Martinez,[†] and Andres W. Martinez^{*,†}

[†]Department of Biological Sciences and ^{*}Department of Chemistry & Biochemistry, California Polytechnic State University, San Luis Obispo, California 93401, United States

S Supporting Information

ABSTRACT: Shrinking paper money is a captivating and memorable demonstration of chemistry in action that can be carried out using minimal equipment and reagents. Paper bills can be shrunk down to ~25% of their initial surface area by treating them with an aqueous solution of sodium periodate. The cellulose in the paper bill is oxidized to dialdehyde cellulose via the Malaprade reaction, and the resulting change in size of the paper money provides macroscopic evidence of the reaction. The chemical change that occurs in the cellulose can be confirmed by a Tollen's test.



KEYWORDS: General Public, Elementary/Middle-School Science, High School/Introductory Chemistry, First-Year Undergraduate/General, Second-Year Undergraduate, Demonstrations, Hands-On-Learning/Manipulatives, Alcohols, Aldehydes/Ketones, Oxidation/Reduction

INTRODUCTION

Paper money is arguably one of the most recognizable objects in the world. Magicians, artists, and scientists have all capitalized on the popularity of banknotes by using them as props to capture the attention of the public. Two popular chemistry demonstrations involving paper currency are the burning money demonstration, wherein a bill is soaked in a solution of alcohol and water and then lit on fire (the alcohol burns but the bill remains intact), and extracting analytes, such as drugs of abuse, from banknotes for analysis via mass spectrometry.^{1–3} Herein, we describe a new chemistry demonstration involving paper money where the bill itself participates as a reagent in a chemical reaction. The reaction leads to macroscopic changes in the bill—the bill shrinks—in addition to molecular changes—alcohol groups from the cellulose of the bill are oxidized to aldehyde groups via the Malaprade reaction (Figure 1).

The Malaprade reaction was first reported in 1928 and is an oxidative cleavage of vicinal diols by periodate to yield two aldehydes.^{4–6} When performed on cellulose, the reaction produces dialdehyde cellulose (Figure 1 and Supporting Information Figure S1).^{7,8} The Malaprade reaction has been used extensively to chemically modify cellulose by incorporating aldehyde functional groups.^{8–11} The aldehyde groups can be further derivatized to other functional groups or used to covalently bind molecules of interest to cellulose, often via imine formation or reductive amination.^{12,13}

In 1937, Jackson and Hudson reported that a piece of filter paper shrank to 25% of its original surface area upon reacting with periodic acid.¹⁴ The shrinkage was later attributed to the reorganization of the dialdehyde cellulose chains into nonlinear conformations that lead to buckling and shrinking of the dialdehyde cellulose fibers (Figure S2).¹⁰ Our research group recently characterized the Malaprade reaction as a method for shrinking paper, and we found that, in addition to producing striking results, the reaction could be performed easily and reliably in aqueous solution and with minimal equipment and workup.^{15,16} For these reasons, we have now developed this reaction as a chemistry demonstration.

DEMONSTRATION

The demonstration involves three steps: (1) preparing a solution of sodium periodate in water, (2) immersing a piece of paper in the periodate solution to shrink it, and (3) washing and drying the miniaturized paper. As far as we know, the miniaturization process is irreversible, so we do not recommend attempting this demonstration with any objects of value.

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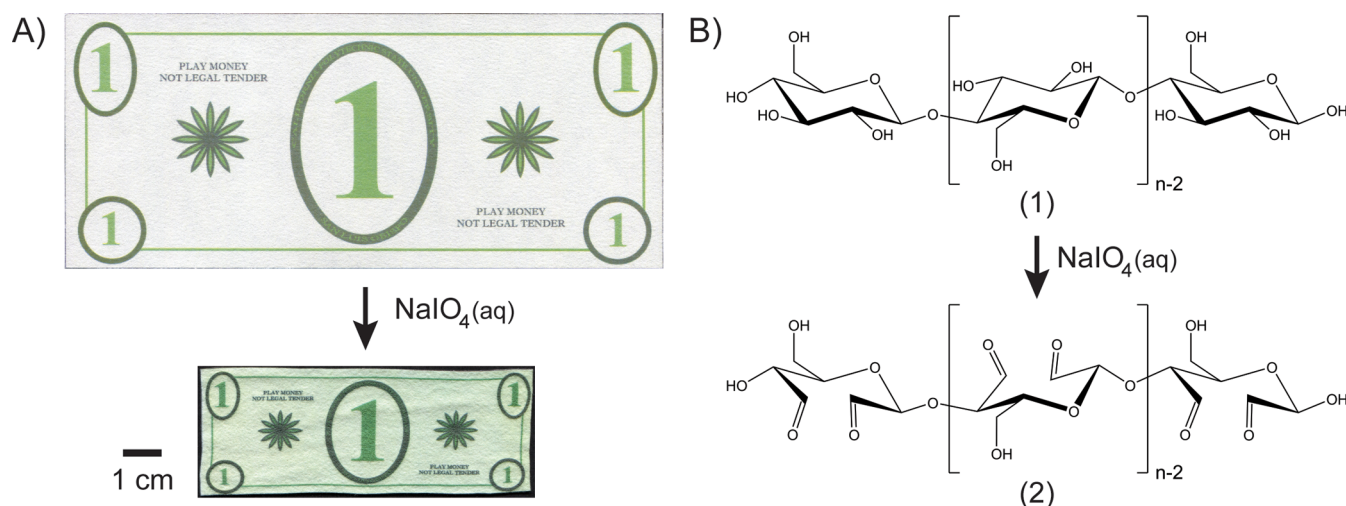


Figure 1. Shrinking paper money via the Malaprade reaction. (A) Images of play money before and after reacting with 0.5 M sodium periodate for 48 h. (B) Reaction of cellulose (1) with sodium periodate to generate 2,3-dialdehyde cellulose (2).

Materials and Equipment

The demonstration requires sodium periodate (sodium metaperiodate, NaIO_4), water, a reaction vessel (e.g., a 15 or 50 mL plastic centrifuge tube), a balance, and paper money. To perform the reaction at 55 °C also requires a large test tube (e.g., 25 mm $\times$ 200 mm), a ring stand, a clamp, a thermometer, and a water bath (e.g., a hot plate and 400 mL beaker). A fume hood is recommended for performing the reaction at temperatures above 55 °C. The Tollen's test requires two small test tubes (e.g., 13 $\times$ 100 mm) and approximately 6 mL of fresh Tollen's reagent.^{17–20}

For this work, we shrunk play money (15.6 cm $\times$ 6.6 cm, Figure S3) and periodic tables (10.0 cm $\times$ 7.3 cm, Figure S4) that were printed using a color laser printer (Samsung CLP-680ND) on Whatman No. 1 chromatography paper, 20 lb. bond paper (i.e., standard printer paper), and 24 lb. 25% cotton resume paper. The printed sheets of paper were baked for 5 min in an oven set to 195 °C to further anneal the toner to the paper.

How To Shrink Paper Money

For the simplest version of the demonstration, prepare 12 mL of a 0.5 M sodium periodate solution as follows: weigh out 1.28 g of sodium periodate, transfer it to a 15 mL centrifuge tube, add water to the 12 mL mark on the tube, and cap and shake the tube until the sodium periodate is dissolved. A 0.5 M sodium periodate solution is nearly saturated at 20 °C. If a small amount of solid sodium periodate remains in the tube after preparing the solution, it will have no negative impact on the experiment. The periodate solution can also be prepared well in advance of the demonstration. We have successfully used periodate solutions over 48 h after they were first prepared. Roll up a paper bill and insert it into the centrifuge tube so that the bill is completely submerged in the periodate solution. Allow the reaction to proceed for 12–48 h; most of the miniaturization will take place within the first 24 h (Figure 2). The tube may be shaken gently every 6–12 h to maintain an even distribution of the periodate. Once the miniaturization is complete, decant the periodate solution into an appropriate waste container. Wash the miniaturized bill with water three times to remove any remaining periodate. Finally, blot the bill with paper towels and dry it overnight. The bill can be dried

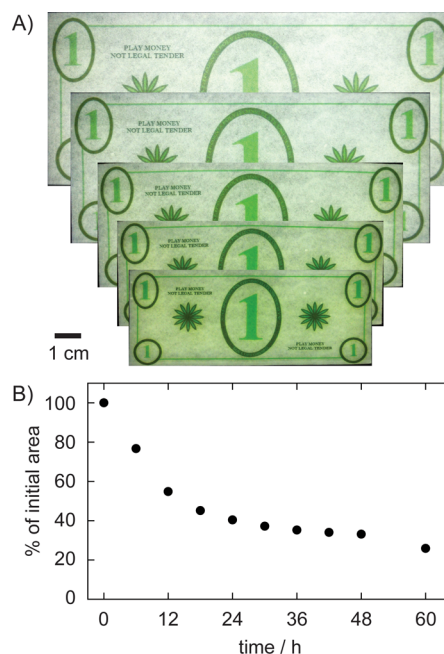


Figure 2. Characterization of paper money miniaturization. (A) Time-lapse images of the miniaturization of a bill printed on chromatography paper as it reacts with sodium periodate. Images were taken every 12 h from 0 h (top image) to 48 h (bottom image). (B) Plot of the relative surface area of a bill printed on chromatography paper versus time as it reacts ($n = 1$). The bill was removed from the periodate solution after 48 h. The data point at 60 h is for the bill after it was washed and dried.

pressed between the pages of a book—a heavy chemistry textbook works particularly well—so that it will remain flat. A video of the procedure is included in [Supporting Information Video S1](#).

To achieve maximum miniaturization of the bill, the reaction should be carried out in a 50 mL centrifuge tube using 40 mL of 0.5 M sodium periodate, prepared using 4.28 g of solid sodium periodate. And, to significantly increase the speed of the demonstration, the reaction can be carried out in a water bath at higher temperatures (e.g., 55 °C) using 1 M sodium periodate (8.56 g of sodium periodate dissolved in 40 mL of

water, Figure S5). A 1 M concentration of sodium periodate can only be achieved at higher temperatures as the solubility of the salt increases. Temperatures above 55 °C lead to even faster reactions, but aqueous solutions of sodium periodate are known to decompose above 55 °C and produce iodine.^{21,22} Therefore, reactions at elevated temperatures should be conducted in a fume hood, especially if the temperature is above 55 °C.

Tollen's Test

To verify that the paper participated in a chemical reaction, a Tollen's test can be performed on the miniaturized bill to demonstrate the presence of aldehyde functional groups (Figure 3). To do so, simply apply a drop of fresh Tollen's

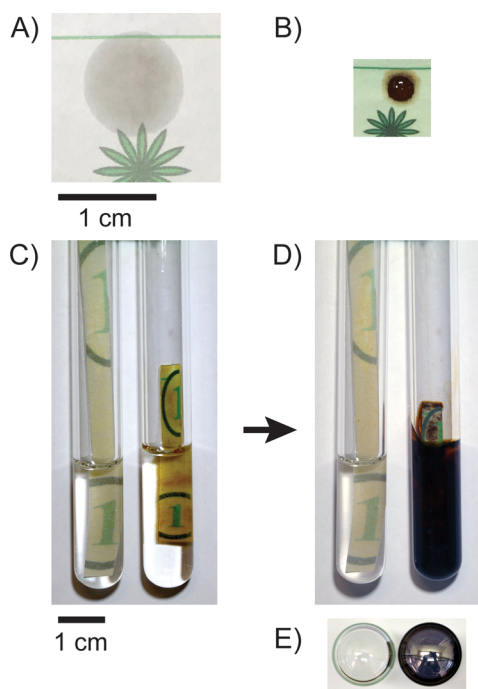


Figure 3. Tollen's test for the presence of aldehyde functional groups on miniaturized paper. (A) Photograph of a normal bill 30 s after applying Tollen's reagent. No reaction is observed. (B) Photograph of a miniaturized bill 30 s after applying Tollen's reagent. The formation of the dark brown color indicates a positive test for aldehydes. (C) Photograph of a normal bill (left) and a miniaturized bill (right) immediately after placing them in test tubes containing Tollen's reagent. (D) Photograph of the same tubes shown in panel C after 2 min. (E) Photograph of the bottom of the tubes in panel D after the Tollen's test, which shows the "silver mirror" in the tube that contained the miniaturized bill and confirms the presence of aldehyde groups.

reagent onto the surface of a miniaturized bill. A brown spot will develop within seconds wherever the Tollen's reagent is deposited and continue to get darker over time. A control test can be performed on a normal bill, and no color will develop (Note: as the Tollen's reagent dries out, it may eventually form a light brown spot on the control bill). Alternatively, a silver mirror test can be performed in a small test tube by immersing a piece of miniaturized paper in approximately 3 mL of Tollen's reagent. Silver will deposit on the walls of the test tube within minutes. Placing the tube in a warm water bath can accelerate the reaction. The Tollen's reagent, both the used and any remaining unused solution, should be disposed of

within 30 min of preparation to avoid any chance of forming silver nitride, a highly explosive substance (please see Hazards for further details).^{20,23} A video of the Tollen's test is included in Supporting Information Video S3.

HAZARDS

Proper personal protective equipment should be worn while performing this demonstration, including safety goggles, nitrile gloves, closed-toe shoes, and a laboratory coat. Solid sodium periodate must be handled with care as it is combustible and a strong oxidizer and may cause skin, eye, and respiratory irritation.^{24,25} Waste periodate solution from the demonstration should be collected in an appropriate waste container and disposed of in accordance with federal, state, and local regulations.²⁵ Prior to disposal, the waste solution may be treated with ethylene glycol to decompose any remaining periodate.²⁶ Alternatively, waste periodate solutions can be treated with a solution of sodium bisulfate followed by sodium carbonate.²⁷

If the reaction is heated, additional precautions must be taken with the water bath and heated periodate solution to prevent burns. A fume hood should be used if solutions of sodium periodate are heated above 55 °C because they could liberate iodine gas, which can cause eye and respiratory irritation.^{21,28} After completing the reaction at elevated temperatures, periodate solutions should be removed from the water bath and diluted with an equal volume of water at room temperature. The diluted solutions can then be disposed of following one of the aforementioned procedures.

The Tollen's reagent must be prepared fresh and handled with care as it contains silver nitrate, which is corrosive and toxic and will stain skin; ammonium hydroxide, which is corrosive, toxic, and a respiratory irritant; and potassium hydroxide, which is also corrosive.^{18,20} Tollen's reagent, both used and unused solutions, should be disposed of within 30 min of preparation.²⁰ Most references recommend diluting the Tollen's reagent waste ~20:1 with water and then either flushing it down the sink or precipitating the silver with chloride, though you should make sure your method of disposal is in accordance with state and local regulations.^{18–20,23}

RESULTS AND DISCUSSION

In introductory chemistry courses, students are often taught to look for four signs that a chemical reaction is occurring: formation of precipitates, formation of bubbles (gas evolution), temperature changes, and color changes. This demonstration provides a unique example of a fifth sign: the macroscopic change in size of a piece of paper upon reacting with periodate. Paper bills printed on chromatography paper were miniaturized to 33% of their original surface area upon reacting with 12 mL of 0.5 M sodium periodate for 48 h. The thickness of the bills increased as they were miniaturized, and they became more rigid, but the mass of the bills did not change significantly. To achieve maximum miniaturization of paper money, a larger volume of periodate solution must be used.^{15,16} In 40 mL of 0.5 M sodium periodate, bills shrank to 25% of their original surface area in 48 h (Figure 2). Using a larger tube for the miniaturization reaction also made it easier to flatten the bill after the reaction was complete since it was not rolled up as tightly. The disadvantages of using the larger volume of periodate solution are that it consumes more

sodium periodate and generates more waste. We did not explore the use of potassium periodate for this demonstration; however, potassium periodate has a much lower solubility in water than sodium periodate and will likely produce different results.

One limitation of this demonstration is that the miniaturization takes place over several hours. Therefore, it would work best with an audience that meets regularly, such as students in a class. In this case, the audience can track the progress of the reaction over time as the bill shrinks—students could measure and weigh the bill before and after the reaction, and they could mark the height of the bill on the tube at various time intervals over the course of the reaction. For shorter demonstrations, where the audience is only present for a few minutes, samples of bills before and after shrinking, as well as a time-lapse video of the shrinking process could be shown (see [Supporting Information Video S2](#)). The Tollen's test could also be performed as part of shorter demonstrations.

To overcome the limitation of the long reaction times, we explored performing the demonstration at higher temperatures by heating the periodate solution in a water bath (Figure S5). As could be expected, the rate of the reaction increased dramatically at higher temperatures.^{21,22} The solubility of sodium periodate also increases at elevated temperatures, so 1 M sodium periodate was used. By heating the reaction to 55 °C, it was possible to shrink paper by an appreciable amount (to 75% of its initial surface area) in 10 min, while full miniaturization (to 25% of its initial surface area) was achieved after 60 min. Temperatures above 55 °C increase the reaction rate even further. For example, at 65 °C, the bill shrank to 50% of its original surface area in 15 min. However, solutions of periodate are known to decompose at temperatures above 55 °C and produce iodine.¹⁹ The iodine will most likely stay in solution, but it is possible that some iodine gas could be generated. Therefore, for temperatures above 55 °C, we recommend performing the reaction in a fume hood. An additional limitation of performing the demonstration at elevated temperatures is that dialdehyde cellulose dissolves in hot water,²⁹ so the paper must be monitored more closely since it can begin to degrade when left in the heated periodate solution too long. At room temperature, degradation of the paper was less of a concern and was only observed if the paper was left in the periodate solution for extended periods of time (>5 days). Despite the inconveniences of performing the reaction at elevated temperatures, it could be useful as a demonstration of the effect of temperature on reaction rates.

An alternative method for shrinking paper money was described by Christine Hermann in 1997.³⁰ This process involves multiple cycles of soaking a bill in liquid ammonia and then drying it. Through this process, dollar bills shrank to ~55% of their initial surface area.³¹ While shrinking paper with ammonia can be achieved relatively quickly, the increased precautions that must be taken to work with liquid ammonia limit the venues where this experiment can be performed. Furthermore, to the best of our knowledge, the chemistry behind why a dollar bill shrinks after exposure to liquid ammonia is not well understood.^{32,33} Shrinking paper with periodate leads to greater miniaturization, can be performed on the benchtop, and illustrates well-known chemical reactions (Figures S1 and S2).

The Tollen's test on the miniaturized bills served to confirm that a chemical change occurred in the paper when it was miniaturized (Figure 3). The Tollen's test is used traditionally

in organic chemistry to detect the presence of aldehyde functional groups.^{18–20} On the basis of the chemical structure of cellulose and the mechanism of the Malaprade reaction, aldehyde groups should be abundant in miniaturized paper (dialdehyde cellulose) but not present in untreated paper (Figures 1 and S1). Tollen's reagent was observed to react rapidly with miniaturized paper and did not react with the original paper, thus confirming that the chemical structure of cellulose changed during miniaturization.

Many variations of this demonstration can be implemented given that the sodium periodate solution can be used to shrink any cellulose-based material including paper or cloth. Chromatography paper, bond paper (standard printer paper), and cotton resume paper all shrunk significantly when exposed to sodium periodate (Figure 4A). Chromatography paper

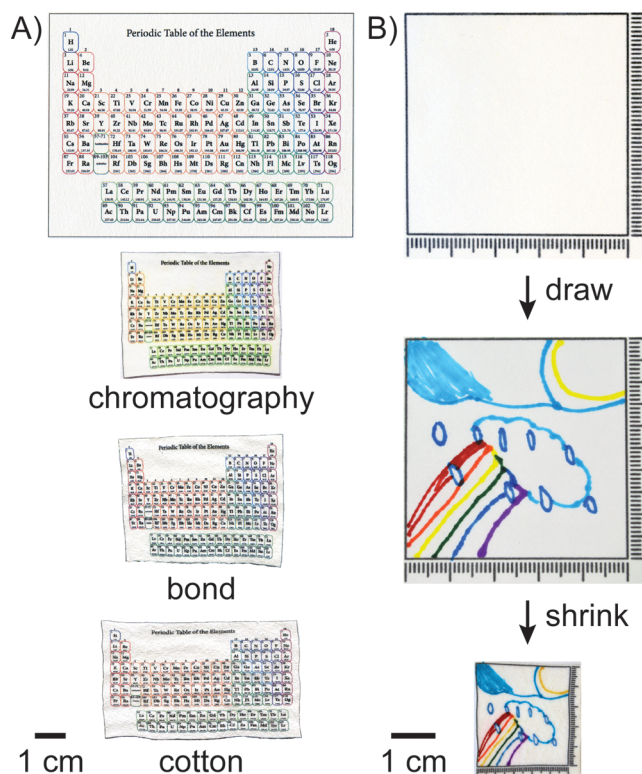


Figure 4. Examples of variations of the demonstration by shrinking periodic tables and art. (A) Periodic tables printed onto chromatography paper, 20 lb. bond paper (printer paper) and 25% cotton resume paper were all miniaturized. The top image shows the original periodic table. (B) Miniaturization of art. The drawing was made by a first grade student using permanent markers. All of the examples were miniaturized in 0.5 M sodium periodate for 48 h.

shrank the most, down to 25% of its original surface area; followed by printer paper, down to 31% of its original surface area; and resume paper, down to 34% of its original surface area. For images printed using a laser printer, we obtained the best results when we baked the printed paper in an oven prior to miniaturization to further anneal the toner to the surface of the paper. When the baking step was omitted, the toner was observed to flake off the paper (Figure S5). We did not explore the use of inkjet-printed images because inkjet printers often use water-soluble inks. In another variation of the demonstration, the audience could draw their own pictures and then shrink them, which is similar to the experience of using Shrinky

Dinks or other thermoplastic shrink films (Figure 4B). Drawings on paper made with pen, pencil, colored pencil, crayon, and permanent marker are all amenable to the shrinking process, but we found that permanent marker worked best because the markings did not flake off the paper the way pencil and crayon markings sometimes did.

CONCLUSIONS

This demonstration inspires wonder in children and adults alike. It could be used in a high school or general chemistry class to illustrate the progress of a chemical reaction or the effect of temperature on the rate of a reaction. It could be used to introduce the Malaprade reaction, as well as the mechanism for hemiacetal formation, in an organic chemistry course. Or, it could be used to awe a more general audience with the power of chemistry. Our society's fascination with miniaturization is evident from movies like "The Incredible Shrinking Man," "Fantastic Voyage," "Honey, I Shrunk the Kids," and more recently, "Ant-Man" and "Downsizing." This demonstration draws on this fascination, and unlike the movies, the effects are real and lasting.

ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available on the ACS Publications website at DOI: 10.1021/acs.jchemed.8b00951.

Supplemental figures (PDF)

Video S1 showing the procedure (AVI)

Video S2 showing time lapse of bill shrinking (AVI)

Video S3 showing Tollen's test (AVI)

AUTHOR INFORMATION

Corresponding Author

*E-mail: awmartin@calpoly.edu.

ORCID

Emily R. Christensen: 0000-0002-9990-9382

Andres W. Martinez: 0000-0002-2696-9165

Notes

The authors declare no competing financial interest.

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