

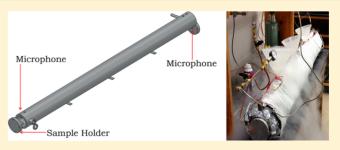
# Measuring the Speed of Sound through Gases Using Nitrocellulose

Karen Sinclair Molek,\* Karl A. Reyes, Brandon A. Burnette, and Jacob R. Stepherson

Department of Chemistry, University of West Florida, Pensacola, Florida 32514, United States

## **S** Supporting Information

**ABSTRACT:** Measuring the heat capacity ratios,  $\gamma$ , of gases either through adiabatic expansion or sound velocity is a wellestablished physical chemistry experiment. The most accurate experiments depend on an exact determination of sound origin, which necessitates the use of lasers or a wave generator, where time zero is based on an electrical trigger. Other experiments use loudspeakers as the sound source, which eliminates the ability to accurately measure time zero of sound generation. To date, experimental heat capacity ratio data have been reported for measurements at room temperature. We



have designed an apparatus to directly measure the speed of sound generated as a result of nitrocellulose ignition via two microphones. Our experimental design also provides the ability to measure the speed of sound at various temperatures and thus determine the heat capacity ratio as a function of temperature. When implemented in a junior-level physical chemistry laboratory course, students learned to use equipment with which they were unfamiliar, such as home-built ignition circuits, a vacuum pump, thermocouple temperature and vacuum gauges, gas cylinders, and an oscilloscope. Students used the data to determine the speed of sound and heat capacity ratio through nitrogen, carbon dioxide, atmospheric air, and argon gases both at 298 K and approximately 253 K. Error analyses of the experimental speed of sound and heat capacity ratio using percent error and propagation of error were performed to ensure a high level of accuracy and precision.

**KEYWORDS:** Upper-Division Undergraduate, Physical Chemistry, Hands-On Learning/Manipulatives, Kinetic-Molecular Theory, Laboratory Equipment/Apparatus, Laboratory Instruction

R ecent experiments noted by Baum et.  $al^1$  and Epstein et.  $al^2$  presented a modern twist on a standard undergraduate physical chemistry experiment measuring the speed of sound in a gas. Traditional experiments measured the nodal distance in a Kundt's tube. $^{3-5}$  The experiment described herein was designed with limited resources and resulted in accuracy comparable to modern experiments. The speed of sound in a medium can be determined by directly measuring the time it takes a sound wave to travel a known distance and is given by eq 1, where S is the speed of sound, D is the distance traveled, and t is the time it took the sound to travel the distance. The data collected can be used in eq 2 to determine the experimental heat-capacity ratio,  $\gamma$ , at a given temperature, T, using the molar mass of the gas, M, and the ideal gas constant, R. Our experiment measures the speed of sound directly through gases over a range of temperatures from 298 to 253 K. The calculated heat capacity ratio can be compared to known values to determine the  $accuracy^{6-8}$ 

$$S = \Delta D / \Delta t \tag{1}$$

$$\gamma = S^2 M / RT \tag{2}$$

In this experiment, the sound source is a small piece of nitrocellulose wrapped in a Nichrome ignition wire. Nitrocellulose is classified as a low grade explosive, which means that it will burn very quickly, but the pressure wave generated travels at or below the speed of sound. This is in contrast to high grade explosives, whose pressure wave travels faster than the speed of sound. The sound produced by the quick-burning nitrocellulose sample will travel at the speed of sound through the gas medium. The amount of time it takes the sound wave to propagate a known distance between two microphones placed on each end of the tube is used to calculate the speed of sound in the sample gas at the specific temperature. Monitoring both microphones with the oscilloscope gives an absolute time of sound origin and detection. The data collected gives similar accuracies to those obtained in previous undergraduate laboratory experiments.

## EXPERIMENTAL DESIGN

The speed of sound tube (Figure 1) is a 1.57 m long aluminum tube sealed on both ends with removable O-ring sealed caps. The tube, fittings, and end-caps were designed in-house and machined accordingly. A microphone (Mouser 665-POW1644LLWC50BR) and a thermocouple temperature probe (OMEGA K Thermocouple) are mounted on both ends of the tube. Microphone 1 is located approximately 2 cm from the location of nitrocellulose detonation and is vertically parallel to the sound origin. Microphone 2 is located at the opposite end of the tube on the end-cap flange. The distance between the two microphones,  $\Delta D$ , is 1.480 m  $\pm$  0.005 m. A

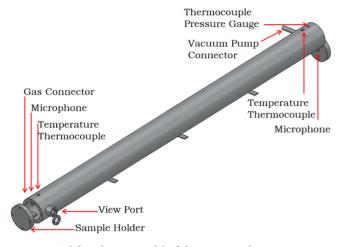


Figure 1. SolidWorks 3-D model of the vacuum tube.

gas inlet and a feedthrough for the electrical ignition system are on the front end of the tube, the same end as the sound origin, whereas a vacuum adaptor and vacuum thermocouple (TC) gauge are on the opposite end of the tube. The temperature thermocouple feedthroughs are located at both ends of the tube to accurately monitor the gas temperature inside the vacuum tube. A window is located on the ignition side of the tube to allow viewing of sample ignition and to provide a method through which laser ignition can be implemented for future experiments. All feedthroughs are sealed via O-ring connections. Detailed drawings and an inventory list of parts can be found in the Supporting Information.

The speed of sound tube is wrapped in pipe insulation and sits in a metal trough. The insulation allows experiments to be run at room temperature or below. For example, most cold temperature measurements were at approximately 253 K. Liquid nitrogen is used to cool the tube to approximately 253 K. When liquid nitrogen is poured into the pipe insulation the tube is cooled rapidly. A surrounding metal jacket serves as the trough, which collects the liquid nitrogen and cools the tube more quickly without wasting liquid nitrogen. Three additional temperature thermocouples are located at either end and in the middle of the tube. These are used to monitor the external vacuum tube and ensure consistent temperatures over the entire surface area of the tube.

The electrical ignition circuit, Figure 2, comprised of a 12 V DC power supply, nichrome ignition wire, a status LED, and a

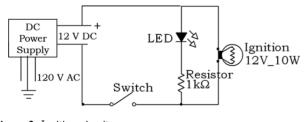


Figure 2. Ignition circuit.

push button switch, is used to ignite the nitrocellulose. A 12 V DC power supply sources 12 milliamps of current through the circuit and nichrome wire igniting the nitrocellulose. The microphones are connected to a Tektronix TDS 1002 oscilloscope via the two corresponding microphone boxes, which are powered by a 9 V battery. The microphone boxes simply convert the microphone cables to BNC cables, which

are connected to the oscilloscope. The oscilloscope is used to record and analyze the time difference in the signals from both microphones.

The first sound wave detected using microphones 1 and 2 are the initial and final waves, as shown in Figures 3 and 4 where

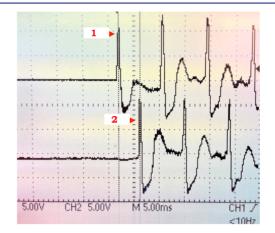


Figure 3. Speed of sound in air where the top and bottom traces (noted as trace 1 and 2 in red) are measuring the sound waves at microphones 1 and 2, respectively.

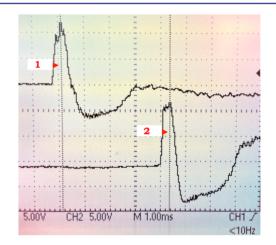


Figure 4. Expanded view of Figure 3, where cursors 1 and 2 correspond to microphones 1 and 2, respectively.

the top and bottom traces correspond to microphones and cursors 1 and 2, respectively. The view is expanded in Figure 4 whereby the cursors are used to determine the time lapsed in sound detection between the two microphones with a 20  $\mu$ s time resolution.

## EXPERIMENTAL PROCEDURE

Approximately 0.1 to 0.2 g of presynthesized nitrocellulose<sup>9</sup> wrapped with nichrome wire is attached to the electrical ignition leads inside the speed of sound tube. The nitrocellulose is then placed on a sample holder mounted to the flange opposite the vacuum pump. The flange must be secured onto the tube in order to maintain vacuum. The tube is evacuated using the attached vacuum pump until the pressure is roughly  $1.0 \times 10^{-4}$  Torr as per the vacuum thermocouple (TC) gauge. The pump is switched off and the tube is filled with the desired gas until the pressure is slightly above atmospheric pressure. This pump/fill cycle is repeated three times to ensure only the sample gas is present inside the tube. On the final fill cycle, a

## Table 1. Comparative Speed of Sound Results

Gas	Average <sup><math>a</math></sup> T, K (SD)	Average <sup><math>a</math></sup> Experimental S, m/s (SD)	Average Accepted S, <sup>b</sup> m/s	Error, %
Argon	254.1 (0.5)	297.3 (4.6)	297.3	0.0
	297.2 (0.8)	321.7 (5.0)	321.1	0.2
Atmospheric Air	255.6 (2.4)	316.2 (7.4)	320.9	1.5
	296.6 (1.1)	349.1 (4.3)	345.8	0.9
Carbon Dioxide	255.7 (1.3)	253.4 (3.8)	251.5	0.8
	298.1 (3.7)	268.3 (4.8)	269.4	0.4
Nitrogen	255.6 (2.3)	327.4 (5.4)	327.3	0.0
	297.1 (0.6)	354.1 (3.8)	351.3	0.8

<sup>*a*</sup>Data from 12 students in CHM 3741L. <sup>*b*</sup>Accepted speeds from literature heat capacity ratios (see refs 7–9) at ~298 and 253 K were calculated for the corresponding measured temperatures.

Table 2. Exp	perimental S	Speed o	f Sound	and Hea	it Capacity	<sup>v</sup> Ratio,	γ, ]	Results
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Gas	Average <sup><math>a</math></sup> T, K (SD)	Average <sup><math>a</math></sup> Experimental S, m/s (SD)	Average <sup><i>a</i></sup> Experimental Ratio, $\gamma$ (SD)	Theoretical Ratio, $\gamma^b$	Error, <sup>c</sup> %
Argon	254.1 (0.5)	297.3 (1.6)	1.672 (0.012)	1.671	0.03
	297.2 (0.8)	321.7 (1.8)	1.674 (0.013)	1.667	0.40
Atmospheric Air	255.6 (2.4)	316.2 (1.7)	1.368 (0.011)	1.403	3.40
	296.6 (1.1)	349.1 (2.0)	1.433 (0.012)	1.400	1.60
Carbon Dioxide	255.7 (1.3)	253.4 (1.2)	1.334 (0.009)	1.310	1.50
	298.1 (3.7)	268.3 (1.3)	1.278 (0.009)	1.289	0.80
Nitrogen	255.6 (2.3)	327.4 (1.8)	1.402 (0.011)	1.402	0.02
	297.1 (0.6)	354.1 (2.1)	1.422 (0.012)	1.400	1.60

<sup>*a*</sup>Data from 12 students in CHM 3741L. <sup>*b*</sup>Theoretical heat capacity ratios for cold temperatures were calculated using literature  $C_{p,m}$  and  $C_{v,m}$  data (see refs 7–9) at 253 K. <sup>*c*</sup>Errors determined via propagation of error analysis.

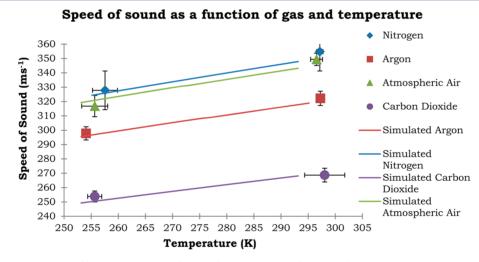


Figure 5. Experimental and simulated/theoretical speed of sound for each gas as a function of temperature.

pressure relief valve connected to the gas manifold is used to release the excess gas and return the tube to atmospheric pressure. Temperature thermocouples, located at either end and inside the tube, are used to record gas temperature prior to each experiment. For cold temperature experiments, cooled gas is run through a coil of copper tubing cooled with liquid nitrogen. The cooler gas allows the tube to maintain a stable temperature thereby reducing systematic errors arising from the introduction of room temperature gas into a 253 K tube. The external temperature of the tube is recorded using the three temperature thermocouples located at either end and in the middle of the tube. For experiments below ambient temperature, liquid nitrogen must be poured over the tube and the temperature must be allowed to equilibrate until the desired temperature of approximately 253 K is reached. The nitrocellulose is ignited to generate sound, which is measured

between the microphone and oscilloscope. The data are recorded and analyzed as described in the Supporting Information and results sections, respectively.

## HAZARDS

Nitrocellulose is a low grade explosive thus it must be kept away from fire or sparks. Nitrocellulose can ignite due to electrostatic discharge. Do not stockpile more than 0.5 g of dry nitrocellulose in one container and do not store it for more than 2 weeks. Nitrocellulose should be stored in a container with a slightly open lid in a vacuum desiccator to minimize damage should it combust. Nitrocellulose can be stored wet for up to one month, with approximately 25% by weight ethanol/ water in an antistatic bag to prevent evaporation of the wetting agent, and the moisture level should be checked weekly. Compressed gas cylinders contain gases at extremely high

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pressures; ensure they are chained to the wall or lab desk at all times. Students must wear safety goggles at all times. Electronic components do not function below 243 K; thus, care should be taken not to cool the tube below that temperature.

## RESULTS AND DISCUSSION

The measured sound times from microphones 1 and 2 are used in combination with the distance the sound traveled, 1.480 m  $\pm$ 0.005 m, to directly calculate the speed of sound, *S*, via eq 1. Equation 2 is used to determine the experimental heat capacity ratio,  $\gamma$ , for each gas as a function of temperature change. The results of 12 students enrolled in Physical Chemistry Laboratory (CHM 3741L) are shown in Tables 1 and 2. Standard deviations were calculated for all measurements. Propagation of error analyses were used to determine more accurate errors associated with the experimental speeds and heat capacity ratios. Both the experimental speed and heat capacity ratios were compared to theoretical values to determine accuracy.<sup>6–8</sup> The standard deviations are shown in Table 1 and propagation of errors are shown in Table 2.

These data are comparable to those obtained in previous undergraduate laboratory experiments.<sup>1,2</sup> The experimental accuracy could be attributed in part to the 1.480 m distance between the two microphones. This length provides a nominal travel time of approximately 4 ms for sound to propagate through atmospheric air. This time eliminates overlap of the sound waves measured via the two microphones. These data are near the accuracy of the more complex spherical resonator apparatus.<sup>10</sup> To the authors' knowledge, these are the first undergraduate laboratory experiments to experimentally show the effect of temperature on the speed of sound in a gas.

According to eq 2, the speed should decrease with a decrease in gas temperature or an increase in molar mass. Figure 5 shows the experimental data in good agreement with the theoretical calculated speeds for each gas over the corresponding temperature range. The dependence on molar mass is a direct consequence of kinetic molecular theory. The effect of temperature on the speed of sound through molecules is directly proportional to the average speed of the molecules, which is proportional to the square root of the temperature and inversely proportional to the square root of the molecules' mass, as described previously by Baum et al.<sup>1</sup>

## FURTHER WORK

Additional experiments will be run as a function of gas composition. Knowing the partial pressure of each gas provides a direct determination of the corresponding mole fraction. The speed of sound, and thereby the heat capacity ratio, can be experimentally measured as a function of mole fraction and gas temperature.<sup>1</sup> The change in the speed of sound and heat capacity ratio of the original gas would allow students to determine the gas composition. Additional modifications could be made by decreasing the length of the tube, thereby reducing both space requirements and costs, though this is not recommended due to the potential decrease in experimental accuracy. Experiments at additional temperatures would be useful. A method published by A. I. Bennett<sup>11</sup> will be used to heat the tube above room temperature via convective heating to provide a third temperature measurement for each gas. Caution should be taken when heating the gas and tube, though, due to nitrocellulose's explosive characteristics. Lower temperatures were also explored but were not possible due to the breakdown

of electronics below -30 °C. Heating tape combined with dry ice will be explored in an effort to maintain better temperature control of the cold tube. Current work is underway to integrate a home-written LabVIEW program via an IEEE-488 interface between the oscilloscope and PC as the data collection method. This will allow easier manipulation of data and potentially reduce the precision error associated with using cursors.

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Students found this experiment to be challenging yet rewarding. Most students had never used an oscilloscope, vacuum pump, or gas cylinder and most had never seen a home-built electrical circuit. The experiment is very robust, which provides students the opportunities to problem-solve without the fear of breaking equipment. As previously noted, this experiment is performed in a junior-level Physical Chemistry lab course, and thus, students are given minimal procedural information, as described in the Supporting Information section. User manuals are provided, along with some basic instructions on connecting the electronics and using the apparatus. It generally takes students a couple of hours to become familiar with the equipment, properly connect all wires, and measure a sound wave on the oscilloscope. Once the experiment is properly set up, two trials of each gas can be measured in approximately 30 min. The students were required to measure two trials of either argon and nitrogen or carbon dioxide and atmospheric air at cold (between 263 and 253 K) and room temperatures. In the future, students will be required to run two additional trials of two gases with varying percent composition at the previously specified temperatures.

## ASSOCIATED CONTENT

#### Supporting Information

Student handouts, including postlab questions; procedural details; hazards; experimental setup instructions for both the oscilloscope and sample; information regarding using the oscilloscope for data analysis; CAD drawings of apparatus and inventory list for parts. This material is available via the Internet at http://pubs.acs.org.

#### AUTHOR INFORMATION

#### Corresponding Author

\*E-mail: ksmolek@uwf.edu.

#### Notes

The authors declare no competing financial interest.

## ACKNOWLEDGMENTS

The authors acknowledge the following offices, departments, and colleagues: The University of West Florida Office of Undergraduate Research (OUR) for the two project awards used to build the apparatus; Michael T. Huggins, Interim Dean of the College of Arts and Sciences at the University of West Florida for the generous donation of Chemistry Department funds used to build the apparatus as well as the useful suggestions regarding cooling the apparatus; and Leo ter Haar, Director of the School of Science and Engineering, for donating a retired oscilloscope.

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