

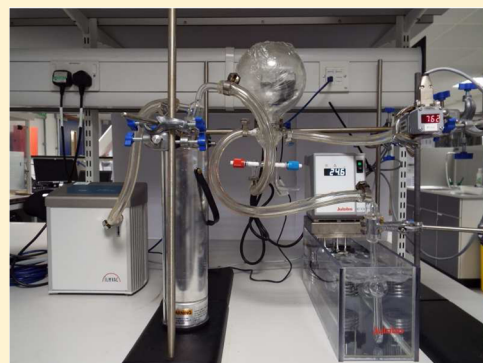
Measuring Vapor Pressure with an Isoteniscope: A Hands-On Introduction to Thermodynamic Concepts

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S Supporting Information

ABSTRACT: Characterization of the vapor pressure of a volatile liquid or azeotropic mixture, and its fluid phase diagram, can be achieved with an isoteniscope and an industrial grade digital pressure sensor using the experimental method reported in this study. We describe vapor-pressure measurements of acetone and *n*-hexane and their azeotrope, and how the data can be used to calculate thermodynamic properties of the test liquids, such as the molar heat of vaporization. This hands-on experience allows students to appreciate important thermodynamic concepts such as phase equilibrium, preparing them for more advanced studies of the subject.



KEYWORDS: First-Year Undergraduate/General, Chemical Engineering, Laboratory Instruction, Hands-On Learning/Manipulatives, Laboratory Equipment/Apparatus, Thermodynamics, Phases/Phase Transitions/Diagrams

INTRODUCTION

A key question in the design and operation of many processes involving fluids is “What is the state of the fluid?” Among the first things taught to undergraduate students of chemical engineering embarking on the study of thermodynamics is the need to continually ask themselves this question as part of their analysis of whatever process they may be considering. In asking this question, we recognize the importance of understanding where and when within the process the fluid will be present as liquid, as vapor, or as a mixture of (saturated) vapor and liquid in equilibrium with each other (VLE). Examples of important industrial processes in which this knowledge is vital include the following: chemical separation processes such as distillation; heat engines and refrigeration cycles that exploit the phase change as part of their function; cryogenic processes; fluid transport in pipelines due to the difference in the flow characteristics of single-phase and two-phase (or multiphase) flow.

The answer to the question “what is the state of the fluid?” is obtained from knowledge of the fluid phase diagram, in which the position of the phase boundary between the liquid and vapor phases is mapped out. This phase boundary is commonly known as the vapor-pressure (or saturation) curve. A typical example of a fluid phase diagram (plotted in the usual pressure, p , and temperature, T , space) is given in Figure 1. Clearly, the class of experiments through which such diagrams are determined is of fundamental importance.

The accepted standard test method for investigating the relationship between vapor pressure and temperature is detailed in ASTM standard D-2879-10, which was first published in

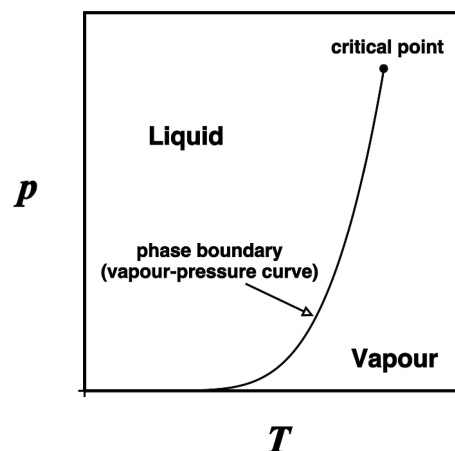


Figure 1. A typical p – T fluid-phase diagram, illustrating the liquid and vapor regions, and the vapor-pressure curve.

1970 and recently revised in 2010.¹ The ASTM standard describes the use of an isoteniscope to measure the vapor pressure of a pure liquid or liquid mixtures against an inert gas at a known pressure. The valid pressure range is between 133 Pa (1.0 Torr) and 101.3 kPa (760 Torr), and the temperature range is between 293 and 748 K.¹

The most common demonstration used for education purposes to date is that of Koubek,^{2,3} who employed a U-

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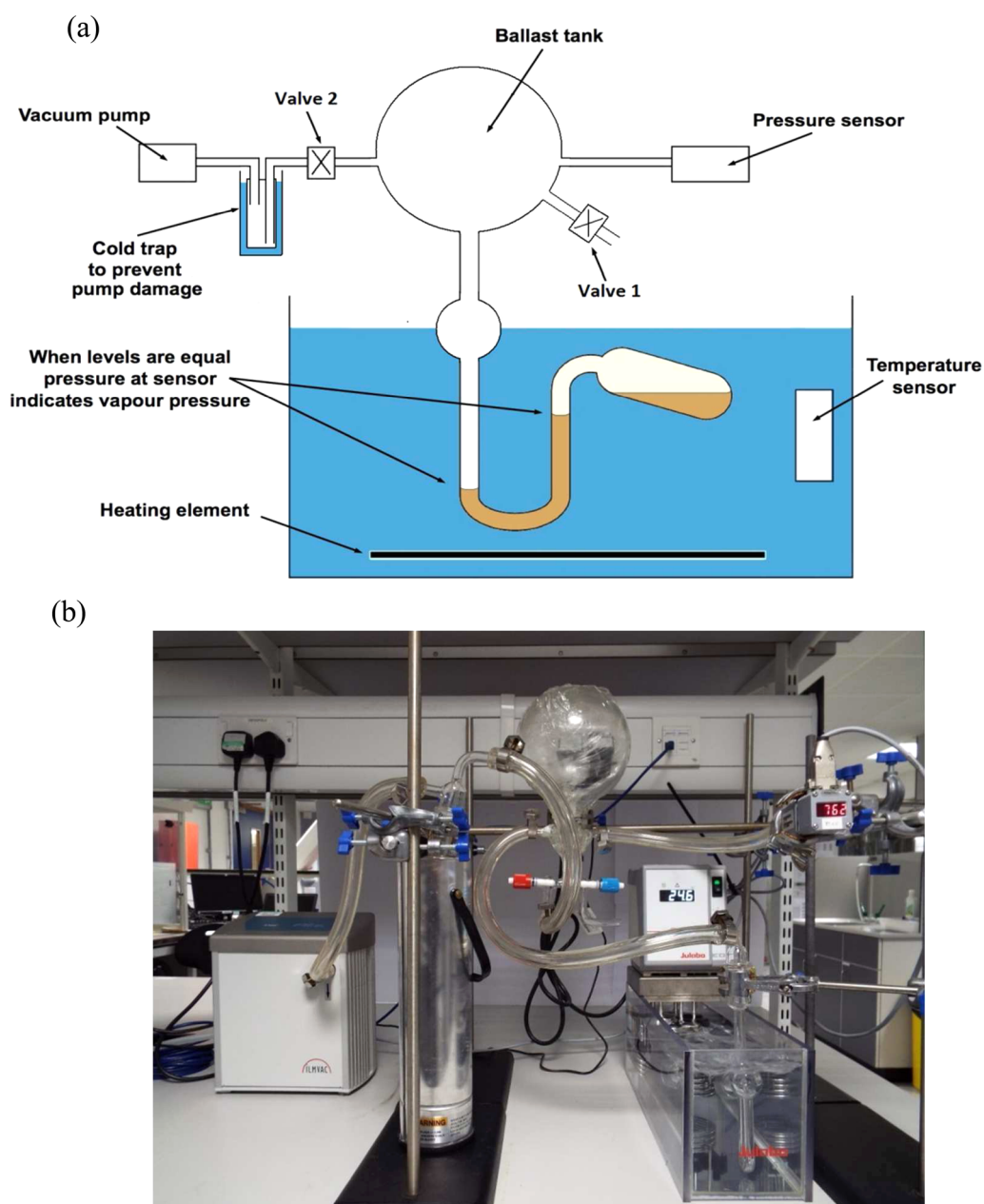


Figure 2. (a) Schematic of the experimental setup, and (b) photo of actual setup.

tube manometer to measure the vapor pressure of benzene with a static method, mainly to measure vapor pressure and to illustrate Raoult's Law. More recently, Pearson⁴ reported a simple demonstration using water and aqueous salt solutions. In this experiment, comparison is made between deionized water and a salt solution in closed containers. The differences in the degree of condensation within the two containers highlights the differences in vapor pressure.⁴ An inexpensive method for vapor-pressure measurement has been proposed recently by DeMuro et al.⁵ With the use of this method, in which a bubble is trapped in a graduated microtube prepared from a small pipet, the vapor pressures and enthalpies and entropies of vaporization of different organic solvents have been determined at different temperatures and were found to agree well with literature.

The use of an isoteniscope as a tool for measuring vapor pressure has been proposed before by Smith and Menzies,⁶ and

included in a teaching context by Steinbach⁷ and Sternberg.⁸ Dannhauser⁹ proposed an experimental validation of the third law of thermodynamics for two different reactions, involving the isoteniscope as a tool for characterization of vapor pressure. An experimental setup on which most of the modern experiments are based was first proposed by Van Hecke.¹⁰ Van Hecke used a complex isolation manifold involving four different valves to ensure that vapor and liquid phases are in equilibrium. Garland, Nibler, and Shoemaker proposed using an isoteniscope for chemistry teaching purposes,¹¹ while Halpern and McBane reported a much simpler and more modern design using a pressure transducer which required regular calibration.¹²

Vapor pressure measurements using an isoteniscope have been used widely in teaching undergraduates experimental physical chemistry. Over the years, methods have been refined to reduce complexity and enhance precision of the measurements. The experiment presented in this article builds on

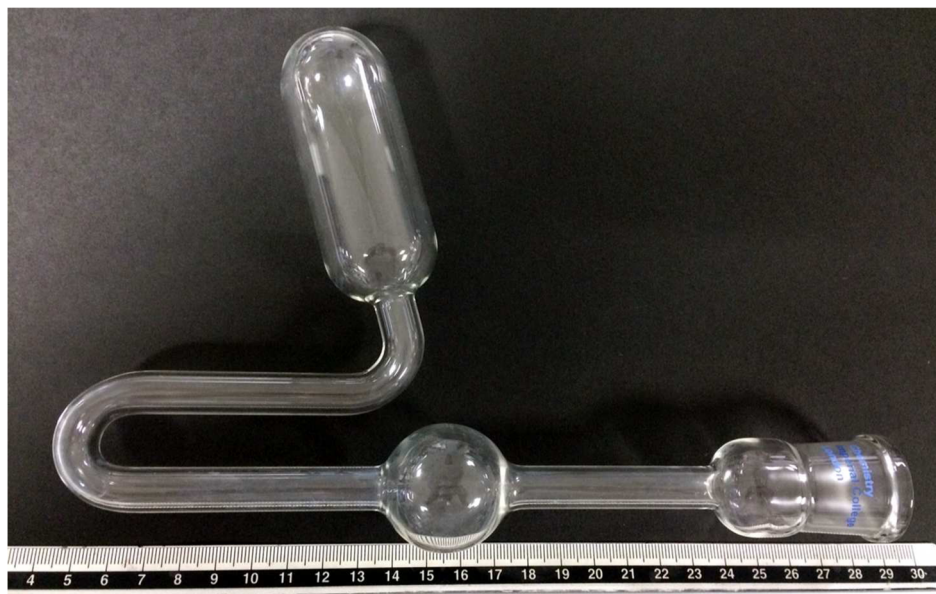


Figure 3. Photograph of the isoteniscope (ruler units are in centimeters).

previous work by updating the design of the isoteniscope assembly and using an industrial grade absolute piezo transducer, which combines a MEMS (microelectromechanical)-based high precision piezo sensor with a metal sealed SS316 diaphragm and integrated electronics, and a digital pressure display. Our intent was to increase simplicity and robustness while maintaining the inherent elegance and accuracy of the classic technique. The combination of the high precision pressure transducer with a digital display means that students do not have to calibrate the sensor before each experiment, which saves time and avoids any calibration errors.

This experiment was specifically developed for first-year undergraduate students in chemical engineering to obtain a “hands-on” appreciation of the VLE phenomenon by fusing traditional experimental techniques with modern laboratory technology. As laboratory-based teaching occupies significantly less space in a chemical-engineering curriculum than in a comparable chemistry program, we wanted to retain as many practical elements as possible to train essential fine-motor and manual handling skills while streamlining the technical procedure.

A further goal was to show how information about the phase boundary can be used to determine thermodynamic properties essential for engineering design calculations. These include vapor pressures of the test fluids determined using the Clausius–Clapeyron equation.¹³ This equation can be conveniently written in integrated form as

$$\ln\left(\frac{p_{\text{sat}(2)}}{p_{\text{sat}(1)}}\right) = \frac{\Delta H_{\text{m}}^{\text{vap}}}{R} \left(\frac{1}{T_{\text{sat}(1)}} - \frac{1}{T_{\text{sat}(2)}} \right) \quad (1)$$

where p_{sat} and T_{sat} are the pressure and temperature at saturation, subscripts (1) and (2) refer to the corresponding pressure/temperature pair at conditions (1) and (2), R is the universal gas constant, and $\Delta H_{\text{m}}^{\text{vap}}$ is the molar heat of vaporization. Note that this equation can be easily extended to ideal and nonideal binary mixtures, where $\Delta H_{\text{m}}^{\text{vap}}$ is taken to be the average molar heat of vaporization of the mixture, weighted according to the mixture composition.¹⁰

■ MATERIALS

Chemicals

Reagent-grade acetone (CAS number: 67-64-1) and *n*-hexane (CAS number: 110-54-3) were obtained from VWR International Ltd. Lutterworth, U.K. and used without further purification.

Equipment

As shown in Figure 2a, the major components of the experimental setup include an isoteniscope (GPE Scientific Ltd., Leighton Buzzard, U.K.), a ballast tank (GPE Scientific Ltd., Leighton Buzzard, U.K.), a diaphragm vacuum pump (ILMVAC GmbH, Ilmenau, Germany), a piezo pressure transducer with digital pressure display (MKS Instruments, Inc., Andover, MA), two valves (GPE Scientific Ltd., Leighton Buzzard, U.K.), a glass cold trap (GPE Scientific Ltd., Leighton Buzzard, U.K.), and a water bath (Julabo UK Ltd., Peterborough, U.K.).

The isoteniscope itself (Figure 3) was designed at Imperial College London, and custom-made by GPE Scientific Ltd. according to the technical drawing, which is provided in the Supporting Information to enable easy reproduction of the instrument. The selection of the vacuum pump depends on the physical properties of the liquid/liquid mixture to be investigated. For our purposes, the diaphragm vacuum pump, model number 412421, proved to be adequate. It can generate pressures less than 60 mbar (as per DIN 28432), and is suitable for the temperature range of 10–60 °C. The piezo transducer, series 902B, provides absolute pressure measurement with a measurement range of 0.1–1000 Torr. The sensor has an accuracy of $\pm 1\%$ of reading, a reproducibility of $\pm 0.3\%$ and is supplied precalibrated by the manufacturer. This instrument is typically used in industries requiring high precision pressure measurements, e.g., semiconductor manufacturing, thin film coating, and aerospace, and does not require calibration by the end user. This saves time and allows students to focus on the main purpose of the experiment within a 3 h lab session, which is generating VLE data. Calibration of instruments is taught in a separate experiment in our laboratory and is out of scope of this

article. The sensor is very robust and has proven itself since 2011 without any operational issues. A Julabo ED-5MB open heating batch circulator with transparent tank completes the assembly. It can readily control temperature in the range of 20–100 °C with set-point deviation of ± 0.03 °C. All experiment components were installed as received.

The isoteniscope (Figure 3) is submerged in a water bath that can be heated in a controlled manner. It is connected with the ballast tank, which is opened to the atmosphere through valve 1 and connected to the vacuum pump through valve 2. The pressure sensor measures the pressure inside the ballast tank. When the liquid levels in the U-tube are horizontally balanced, the pressures on both sides are equal, and hence, the pressure indicated by the pressure sensor is the same as in the bulb of the isoteniscope. A cold trap (water–ice mixture in a Dewar flask) is in place between valve 2 and the vacuum pump to prevent any vapor of the test liquid entering the pump. Figure 2b displays the complete experimental assembly on the laboratory bench. As can be seen, it is reasonably compact and from experience we can also claim that it is considerably robust: In almost five years of operation and handling by 120 first year students each year, so far only one isoteniscope had to be physically repaired because of glass breakage.

The question of “why use an isoteniscope when one can measure the vapor pressure using the direct method?” may spring to the mind of the interested reader. The direct measurement of vapor pressure in a simple flask containing the test liquid is undoubtedly more convenient. However, the accuracy of such a method depends on the simultaneous temperature control of the vapor and liquid phases. Hence, the entire equipment that contains both phases has to be at the same temperature, so that a thermal equilibrium can be established. In a simple flask, the section which leads to the pressure sensor often lacks this temperature control because of its exposure to room temperature, which makes it difficult for the two phases to reach thermal equilibrium. As a result, this causes a systematic error in the measurement of vapor pressure. The use of an isoteniscope ensures that both vapor and liquid phases are always subject to the same temperature, as bulb and manometer section are submerged simultaneously. In addition, the user of an isoteniscope can easily see whether the vapor and liquid phases are at equilibrium by checking the liquid level in the manometer section, which is one of the key learning experiences we want to pass on to the students.

METHOD

The bulb of the isoteniscope is filled with the test liquid to approximately three-quarter height, so that no liquid flows into the U-tube when the isoteniscope is placed vertically. The isoteniscope is then connected to the ballast tank which is at atmospheric pressure (valves 1 and 2 are closed), before it is placed in the water bath, which is set to 25 °C.

Valve 2 is opened slowly to allow the vacuum pump to evacuate the air from the system. As the pressure inside the system decreases below the vapor pressure of the test liquid at 25 °C, the test liquid in the bulb of the isoteniscope starts boiling. The vacuum is maintained for another 5 min in order to remove as much air from the system as possible. Valve 2 is then closed and the vacuum pump is switched off. The isoteniscope is tilted slightly so that some of the test liquid flows into the U-tube. Once the bubbling liquid fills up the U-tube, the U-tube is placed vertically.

Valve 1 is opened slowly to allow air to enter the ballast tank in order to increase its internal pressure. Boiling ceases and the liquid level on the ballast tank side of the U-tube decreases slowly until it is level with the bulb-side liquid level. Valve 1 is then closed. The pressure indicated by the pressure sensor is then recorded, as is the temperature of the water bath.

The water bath temperature is increased by 5 °C and valve 1 is opened gradually to maintain the balanced liquid levels in the U-tube. Once the temperature of the water bath stabilizes and the liquid levels in the U-tube are at equal height, valve 1 is closed. The pressure indicated by the pressure sensor is recorded with the temperature of the water bath. This procedure is iterated until the highest target temperature is reached; then, the entire experiment is repeated to check for data consistency.

Step-by-step equipment operating instructions are provided in the notes to instructors and students, which are included in the Supporting Information.

The selection of acetone as the test fluid was made with some care. An important criterion was that even students with limited experimental skills should be able to perform the experiment safely and within a reasonable time period, while producing verifiably accurate results. The nontoxic nature of acetone minimizes the risk when conducting the experiment in case of poor manual handling skills. Moreover, its relatively high volatility ensures that students obtain reliable results within a reasonable time frame, while working at a temperature range (25–50 °C) that is well below temperatures that would involve the danger of burns and scalds; for example, acetone is more volatile than other safe-to-handle liquids such as acetonitrile, ethanol, and octane. A lesser, though not insignificant, consideration was that acetone represents a reasonably economical choice of test fluid.

Students find that the most difficult part of the procedure is the adjustment of the liquid levels in the isoteniscope by either venting the vacuumed ballast tank to atmosphere or to the vacuum pump. When the pressure inside the ballast tank increases too quickly, some of the liquid in the U-tube of the isoteniscope flows back into the bulb together with the mixture of air and vapor of test liquid in the ballast tank. It is then necessary to repeat the removal of air by vacuum, as described earlier in this section. The necessary degree of care in manual handling and applying fine motor skills in response to visual observation is an important learning experience.

HAZARD AND SAFETY PRECAUTIONS

Although acetone and *n*-hexane are chosen for their nontoxic nature, they are still hazardous when inhaled or ingested. In addition, they are irritants to skin and eyes. Therefore, personal protective equipment (PPE) including lab coat, safety goggles and disposable gloves should be worn during the experiment in order to prevent direct contact with the test fluids. Both acetone and *n*-hexane are highly flammable and, hence, must be handled and stored away from sources of heat and ignition.

Another hazard is the possible implosion of glassware caused by the vacuum created by the pump. It is important to tape the heavy-walled glassware in cling film to contain potential projectiles in case of implosion. Before setting up the experimental system, it is necessary to inspect the glassware for scratches, cracks and chips. The experiment should be conducted in a well-ventilated area.

Risk assessments for the experiment and the use of hazardous chemicals are also included in the Supporting Information.

RESULTS AND DISCUSSION

Vapor-Pressure Measurements for Acetone

To illustrate the effectiveness of the experiment, and the level of accuracy that may be expected, we report the results of 20 randomly chosen experiments carried out by first-year undergraduate students, each within an allotted time of 3 h, using acetone. The measured vapor pressures are presented in Figure 4 together, for comparison, with a curve obtained with the

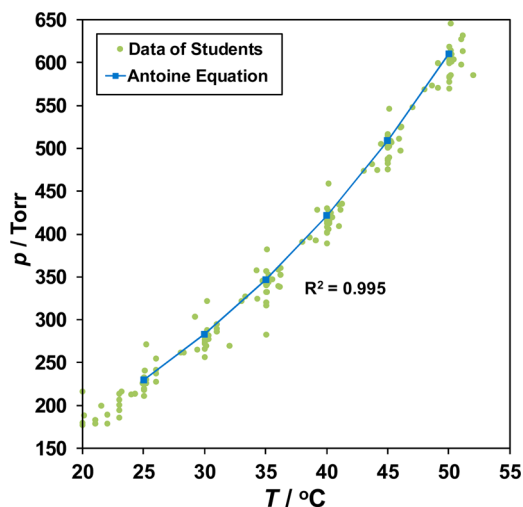


Figure 4. Vapor pressures of acetone as measured by undergraduate students together, for comparison, with the vapor-pressure curve generated using the Antoine equation (see Table 1).

Antoine equation^{14,15} (see also Table 1), representing the accepted true vapor pressure curve. Even though the students were relatively inexperienced in performing experiments, the data points fit closely to the Antoine curve (the coefficient of determination, $R^2 = 0.995$). This demonstrates both the efficacy of the experiment and the reproducibility of the measurements.

Implementation in Teaching

Undergraduate students studying Chemical Engineering at Imperial College typically carry out the vapor-pressure experiment in the second term of their first year; every year, over 120 such students conduct the experiment, in pairs, in one 3 h laboratory session. For logistical reasons, many of the students perform the experiment before they have been introduced to the Gibbs function or the concept of chemical potential in their Thermodynamics lecture course, whereby they are also unfamiliar with the Clausius–Clapeyron equation and its application for the calculation of molar heats of vaporization. Accordingly, they are supplied with a laboratory script designed on the basis of the students' background knowledge of the subject area to bridge the knowledge gap between experimental objectives and existing understanding. The laboratory script provides a brief introduction to the key concepts of phase equilibrium. The only prerequisites are that

the students understand that the heat, Q , related to a constant-pressure process is given by the enthalpy change, and that for a reversible process the Second Law provides that $\Delta S = Q/T$, where S is the entropy, and T is the (absolute) temperature. The prerequisite is typically taught in Key Stage-5 of the U.K. school education system or equivalent levels of secondary education elsewhere. We find that students across a wide range of ability comfortably complete the experiment within the allotted time, producing good-quality measurements (as illustrated in Figure 4); this is a testament to the successful design and operational simplicity of the experiment. The course module in which this experiment is taught usually receives well over two-thirds student satisfaction. From student and demonstrator feedback, we know that students find the experiment to be a positive and rewarding learning experience.

Extension 1: Vapor Pressure Measurements for Other Volatile Liquids and Binary Liquid Mixtures

This vapor-pressure experiment can be easily extended to binary liquid mixtures. Although we have not included such an extension in our undergraduate laboratory exercise, for illustration we have employed our apparatus to study the azeotrope of acetone/*n*-hexane, which occurs at a molar composition of 0.64/0.36.

Before considering the binary mixture, we first examine the other constituent pure component, *n*-hexane. In Figure 5, we

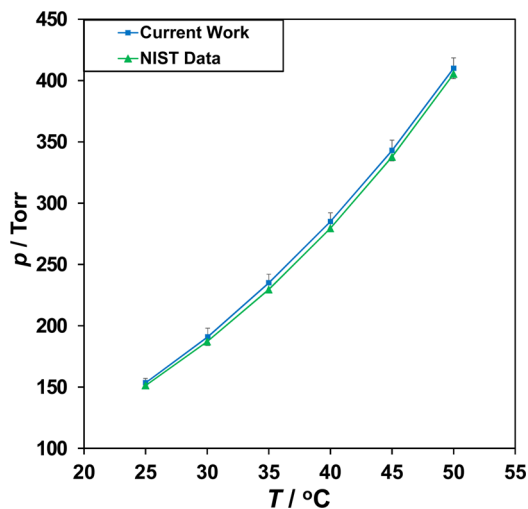


Figure 5. Vapor pressures of *n*-hexane (connected filled-squares) measured using our teaching-laboratory apparatus. Note that the error bars in the horizontal direction are smaller than the size of the symbols. Also shown (for comparison) are accepted vapor pressures (continuous curve) from the NIST Chemistry Web Book.¹⁸

show the measured vapor pressures of *n*-hexane, again (for comparison) with reference to accepted values; in this case, we take pseudo-experimental values obtained from the NIST Chemistry Web Book, which are conveniently available for *n*-hexane. Similar to the case of acetone, the data measured using

Table 1. Coefficients in the Antoine Equation:^a $\log_{10}(p_{\text{sat}}) = A - \frac{B}{T+C}$

Substance	Temperature Range/K	A	B	C	Reference
Acetone	259.16–507.60	4.42448	1312.253	−32.445	NIST database, using data from Ambrose et al. (1974) ¹⁶
<i>n</i> -Hexane	286.18–342.69	4.00266	1171.53	−48.784	Williamham et al. (1945) (taken from NIST database) ¹⁷

^aNote: p_{sat} in bar and T in K.

our teaching laboratory experimental setup is in close agreement with the accepted values. For validating the robustness of data, actual values of the vapor pressure obtained from NIST, in addition to its Antoine parameter fit, were also compared with the vapor pressure values measured at all relevant temperatures. The standard deviation between the values was found to be less or equal to ± 3 Torr.

Although acetone is more volatile than *n*-hexane, the molar heats of vaporization for both test liquids are close to identical, both are 31 kJ mol^{-1} . This is calculated based on the Clausius–Clapeyron equation by plotting $\ln p$ against $1/T$ (i.e., constructing a van't Hoff plot as shown in Figure 6) and then multiplying the gradient of the best-fit line with the gas constant. They are identical to literature values.¹¹

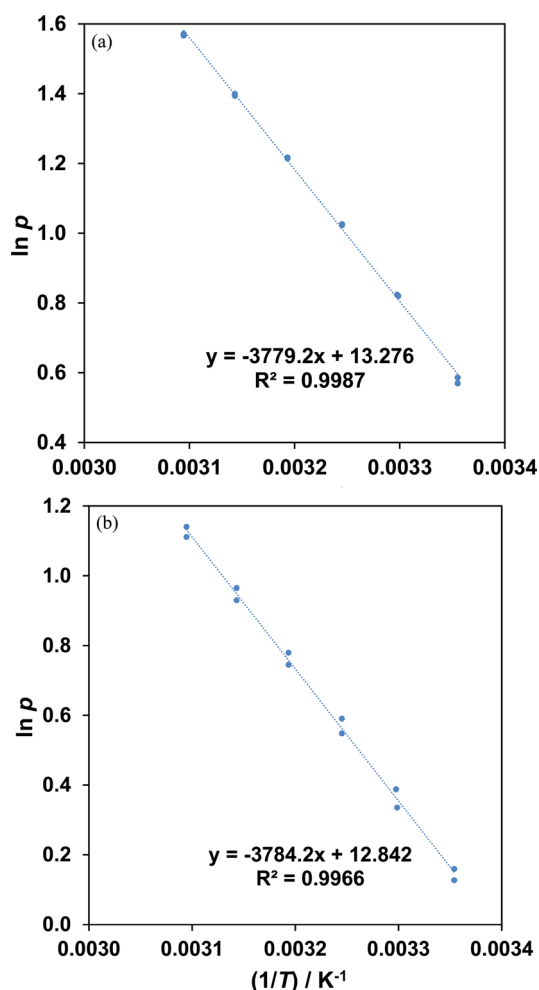


Figure 6. van't Hoff plots for (a) acetone and (b) *n*-hexane.

Through the van't Hoff plots, advanced students could also determine the molar entropy of vaporization (ΔS_m^{vap}) for the test compound from the y -intercept of the best-fit line, which is equal to $\Delta S_m^{\text{vap}}/R$. The molar entropies of vaporization for acetone and *n*-hexane are 110 and 107 $\text{J mol}^{-1} \text{ K}^{-1}$, respectively.

In Figure 7, we present the measured vapor pressures of an acetone/*n*-hexane binary mixture at the azeotropic molar composition (0.64/0.36), and compare these with reference data for an ideal mixture.

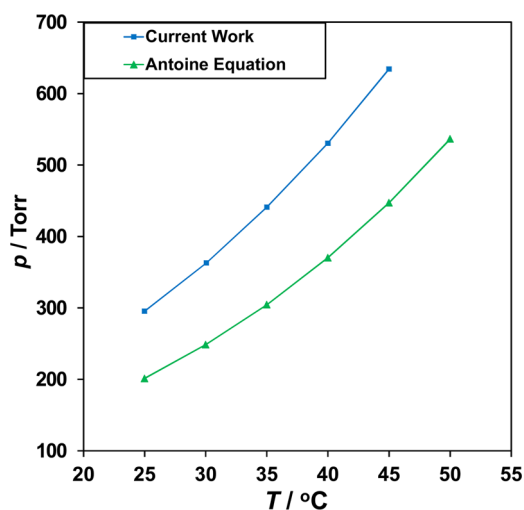


Figure 7. Vapor pressures of the acetone/*n*-hexane azeotrope *n*-hexane (connected filled-squares) measured using our teaching-laboratory apparatus. Note that the error bars in both horizontal and vertical directions are smaller than the size of the symbols. Also shown (for comparison) are calculated vapor pressures (continuous curve) obtained using the Antoine equations relating to the pure compounds (see Table 1).

As is evident from Figure 7, the measured vapor pressures of this azeotrope deviate significantly from the calculated values that would correspond to ideal mixture behavior. This positive nonideality means that the molecules of these two liquids have lower affinity with one another than with other molecules of their own species, making the liquid mixture more volatile than either liquid in its pure form. The calculated average molar heat of vaporization (30 kJ mol^{-1}) is similar to the value for the pure compounds, as expected.

Extension 2: Calculation of Activity Coefficients for Nonideal Mixtures

This experiment can also be used to exemplify the concept of the activity coefficient, as students can calculate the activity coefficients of the two components in the azeotrope to account for the nonideal behavior of a mixture.

Assuming nonideal behavior in the liquid phase, the equations are

$$p_a = \gamma_a \cdot x_a \cdot p_a^* \quad (2)$$

$$p_b = \gamma_b \cdot x_b \cdot p_b^* \quad (3)$$

$$p_{\text{mixture}} = p_a + p_b = \gamma_a \cdot x_a \cdot p_a^* + \gamma_b \cdot x_b \cdot p_b^* \quad (4)$$

Here, p_i is the partial pressure of component i in the mixture, p_i^* is the vapor pressure of pure component i , γ_i is the activity coefficient of component i , and x_i is its mole fraction in the liquid phase. Indices a , b , and mixture denote compounds a , b , and their mixture, respectively.

In the vapor phase, we assume ideal behavior depending on the mole fraction y as follows:

$$p_a = y_a \cdot p_{\text{mixture}} \quad (5)$$

$$p_b = y_b \cdot p_{\text{mixture}} \quad (6)$$

In an azeotropic mixture, the mole fraction of a compound is the same in both phases:

$$x_a = y_a \quad (7)$$

$$x_b = y_b \quad (8)$$

By rearranging the equations, the activity coefficient is simply the ratio of the vapor pressure of the mixture and the vapor pressure of the pure component at a particular temperature:

$$\gamma_a = \frac{p_{\text{mixture}}}{p_a^*} = \frac{p_{\text{mixture}}}{10^{A_a - [B_a/(T+C_a)]}} \quad (9)$$

$$\gamma_b = \frac{p_{\text{mixture}}}{p_b^*} = \frac{p_{\text{mixture}}}{10^{A_b - [B_b/(T+C_b)]}} \quad (10)$$

At any temperature, nonideal behavior of an azeotrope can now be accounted for, and the vapor pressure can be predicted using the Antoine equations for the pure compounds in combination with eq 4:

$$p_{\text{mixture}} = \gamma_a \cdot x_a \cdot 10^{A_a - [B_a/(T+C_a)]} + \gamma_b \cdot x_b \cdot 10^{A_b - [B_b/(T+C_b)]} \quad (11)$$

For the acetone/*n*-hexane system, the activity coefficients were found to be $\gamma_a = 1.25$, and $\gamma_b = 1.89$.

As a final comment, we note that as an additional learning opportunity, one may draw students' attention to the similarity in the mathematical forms of the Clausius–Clapeyron equation and the Antoine equation, which has been exploited in our current work to represent accepted values of the pure-component vapor pressures. Indeed, one can consider the Antoine equation to be a simple modification of the Clausius–Clapeyron equation,¹⁵ albeit that the parameters employed are generally empirical. It is important that the Antoine equation is not used outside its stated temperature range of validity; failing to observe this restriction can lead to absurd results due to the empirical nature of the equation. This could form the basis of an interesting discussion for more experienced students. However, for inexperienced first-year students such as those in our own teaching laboratory, we recommend the more focused, simpler analysis based purely on the Clausius–Clapeyron equation, in which the constituent quantities retain clear, physical meaning.

CONCLUSION AND OUTLOOK

The use of an isoteniscope represents a convenient, simple, and effective “hands-on” experiment in which the vapor pressure of a fluid may be measured. Students can easily perform the experiment twice for one test fluid in 3 h. The vapor-pressure measurements are highly reproducible and, for the test fluids investigated here (acetone and *n*-hexane), seen to be in good agreement with literature values. Data obtained in this experiment can be used to calculate the molar heat of vaporization based on the Clausius–Clapeyron equation, again yielding values that compare favorably with literature values. The experiment can be extended further to study binary liquid mixtures, as illustrated for the case of acetone/*n*-hexane, to introduce the concept of azeotropes and ideal/nonideal binary mixtures to students.

ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available on the ACS Publications website at DOI: 10.1021/acs.jchemed.5b00990.

Notes to students and instructors (PDF, DOCX)

Detailed technical drawing of the isoteniscope (PDF)

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Notes

The authors declare no competing financial interest.

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