

A KINETICS EXPERIMENT

For the Unit Operations Laboratory

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The topic of kinetics, because it deals with change in molecular structure (as opposed to mere physical change), is, strictly speaking, not a subset of the term “unit operations.” Nevertheless, many schools include a kinetics experiment in what is nominally called a unit operations laboratory (UOL) course. This paper describes a kinetics experiment that was recently added to the senior UOL course at Clemson. It deals with selection of the reaction, the design and operation of the apparatus, incorporation of appropriate safety equipment, and analysis of results.

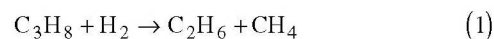
Once the decision to add a kinetics/reactor design experiment had been made, the first issue to be resolved was whether or not to purchase a complete “off the shelf” experiment from a vendor (*e.g.*, Armfield or Hampden), or to design/build our own. The latter path was chosen for several reasons. One was that this strategy would provide an excellent learning opportunity for the group of undergraduate students who played a major role in the construction/debugging of the apparatus and in the determination of feasible operating conditions. This aspect will be described in a separate paper.^[1]

Another reason for deciding to design our own experiment was that commercially available experiments use liquid-phase reactions (*e.g.*, saponification), whereas a heterogeneously catalyzed gas-phase reaction system was felt to offer several advantages, one of which would be greater variety regarding potential assignments since, with minor modification, the same apparatus could be used for many combinations of catalyst and reactants, often with major differences in apparent kinetics. Other advantages would be that such a system affords more accurate flowrate control/determination (through the use of mass-flow controllers) and more accurate composition measurements (through the use of a gas chromatograph equipped with a flame ionization detector). Furthermore, designing the experiments and conducting data analysis could be varied to fit the backgrounds of the students (and the temperament of the instructor). For example, the rate data could be fit to a simple “power law” expression or to a more complex Langmuir-Hinshelwood model that provides additional

insight into what is actually occurring during the reaction process.^[2] Finally, during the roughly eight months a year when the senior UOL course is not being taught, the apparatus would be available as a versatile platform for senior or graduate student research projects.

CHOICE OF REACTION/CATALYST

After considering several reactions, propane hydrogenolysis over an alumina-supported platinum (Pt/ γ -Al₂O₃) catalyst was chosen for the experiment. Under the conditions used, the reaction can be considered effectively irreversible and ethane hydrogenolysis, a possible complicating secondary reaction, occurs to a negligible extent. Data analysis is also made easier by the small number of species involved and by the fact that the simple stoichiometry results in no change in the total number of moles (shown in Eq. 1).



In the experiment, the catalyst (in a sense) merely serves as a “means to an end,” *i.e.*, students are not asked to study the catalyst *per se*. In designing the experiment, however, the choice of catalyst was important because the catalyst greatly influences the reaction rate, and thus, operational parameters such as reactor size, temperature, pressure, and flow-

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rate. An additional consideration was that the catalyst should experience minimal deactivation over the course of a given group's experiment (typically, three 3-hour periods) so that the determination of kinetic parameters would be straightforward. Combining both literature^[3,4] information and our "in-house" experience^[5,6] with this reaction over a variety of catalysts resulted in the selection of a commercial 0.6 wt.% Pt on γ -Al₂O₃ catalyst (PHF-5) obtained from Cyanamid.

EXPERIMENTAL APPARATUS

Figure 1 is a schematic showing the major features of the apparatus. The four main sections are

- The reactor and furnace/temperature controller
- The feed gas system and flow controllers
- The combustible gas detector/alarm and emergency gas shut-off system
- The computer-controlled gas chromatograph

The reactor consists of a 66-cm long stainless steel tube (15.9 mm OD, 13.6 mm ID) connected at each end to a Swagelok tee. Within the reactor, roughly 1.5 grams of 14 to 30 mesh (0.6 to 1.4 mm) catalyst particles are positioned at the midpoint, *i.e.*, roughly 30 cm from the inlet. The feed preheating zone between the reactor inlet and the catalyst bed is filled with 1.5-mm glass beads. These beads also serve, along with small pieces of Pyrex wool, to position the catalyst near the axial midpoint of the "wraparound" 1.3 kW Lindberg Blue M tube furnace. Due to low conversions and

the small size of the catalyst bed, the catalyst temperature is approximately uniform and is measured using a 3.2 mm OD type-K thermocouple that is coaxial with the reactor and that has its tip positioned in the center of the catalyst bed. Readings from the reactor thermocouple are obtained using an Omega digital thermometer. Another type-K thermocouple is used to measure the furnace temperature, *i.e.*, in the region between the outside of the reactor and the inner surface of the furnace. This thermocouple is connected to a Barber-Colman temperature controller. In the reactor exit line there is a pressure gauge and a Tescom back-pressure regulator.

The feed-gas system consists of

- Pressure regulators and high-pressure cylinders for the three gases used (instrument-grade propane, high-purity hydrogen, and high-purity helium)
- Normally closed solenoid valves for the hydrogen and propane lines that are energized (open) during normal operation and de-energized (closed) when the apparatus is not in use or when elevated levels of combustible gases are detected
- Three calibrated Brooks Model 5850E mass-flow controllers connected to a Brooks Model 0154 digital flow readout

After being combined, the three streams may be either routed to the reactor inlet or to a bypass line (for feed composition determination).

Due to the flammability of the reagents used in this experi-

ment, a combustible gas detector with accompanying alarm system (RKI Instruments, Inc.) is used to detect process leakage of hydrocarbon reactants and reaction products. The concentration of gaseous hydrocarbons is detected by a fixed-mount, continuous-monitoring detector head that displays the current concentration of combustible gases and transmits this information electrically (4-20 mA signal) to a multichannel gas monitor. The gas monitor is programmed to sound an alarm if hazardous levels of combustible gas are detected and to simultaneously de-energize (close) the solenoid valves connected to the propane and hydrogen pressure regulators.

Gas analysis is achieved using a Varian CP-3380 gas chromatograph equipped with a Valco 6-port gas-sampling valve actuated using a Valco 3-way solenoid valve manifold, and a flame ionization detector that uses hydrogen and compressed air. Separation is achieved using helium carrier gas flow through a 213-mm by 3.2-mm stainless steel column packed with 80/100-mesh poropak

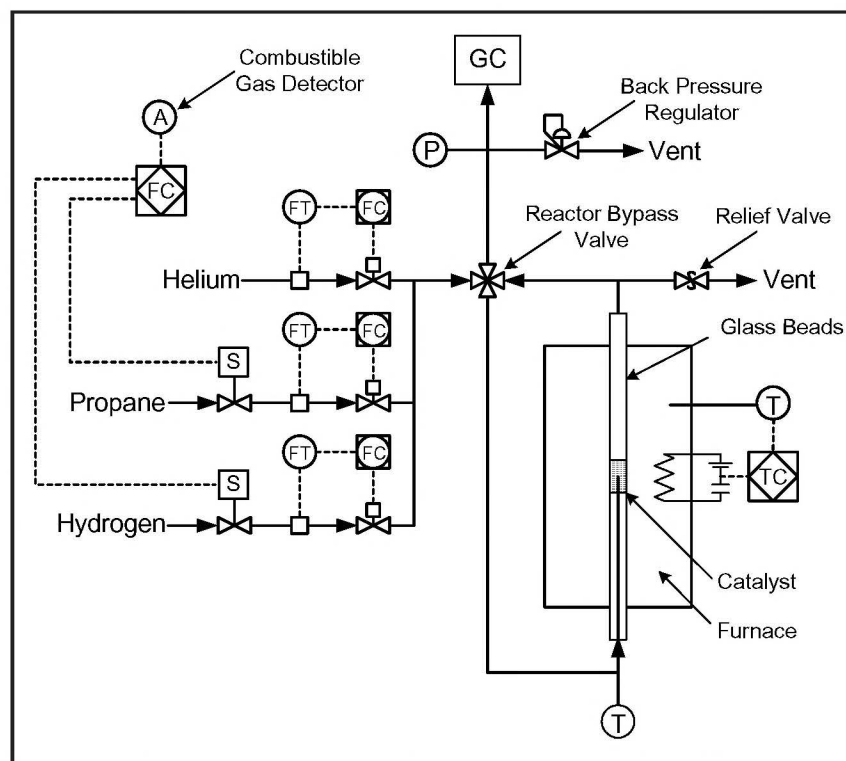


Figure 1. Schematic for kinetics experiment apparatus.

Q and maintained at 170°C. This gives well-separated peaks for the three hydrocarbons within an elution time cycle of only 2 minutes. A software package (CP-3800) obtained from Varian is used with a computer to operate the GC, perform data logging/peak area determination, etc. Exit streams from both the reactor and the GC are vented through tubing to the outside.

EXPERIMENTAL PROCEDURE

Before giving a general description of the procedure used, a few comments on how UOL is conducted at Clemson should be mentioned to provide proper context. The first is that, in contrast to many other laboratory courses that involve what is often called a “cookbook” approach, here each lab group (which consists of three or four students) writes a preliminary (pre-experiment) report as well as a final report. Prior to writing the preliminary report, each group is given a lecture on the topic, a brief “walk-through” of the apparatus, an assignment sheet outlining the objectives, and a handout that provides guidance regarding the use of the software and the safe operation of selected items of equipment, *e.g.*, the GC, flow and temperature controllers, and the combustible gas detector. The last of these handouts is felt to be necessary because of the complexity of the reactor system.

Once the group has received information about the experiment, they are required to submit the preliminary report, which contains

- A schematic and an experimental plan, *i.e.*, a fairly detailed listing of operational steps and safety issues
- Data tables
- Sample calculations
- Literature review

This report is then read, graded, and corrected by the supervising faculty member and returned to the group. A group normally begins actual experimentation the next lab period after the graded preliminary report has been returned.

Depending on the background of the students, the instructor can assign students to develop a classical or a factorial design of experiments. Traditional methods would involve students evaluating one variable at a time (*e.g.*, all variables are held constant during a set of experiments, except for the variable being evaluated). Greater sampling efficiency and complexity of data analysis are achieved, however, by having students use the statistics-based strategy known as *design of experiments* to develop a factorial design that will enable them to quantify each parameter in the selected reaction model. For example, the combined power law/Arrhenius law model contains four parameters that need evaluating (α , β , E , k_0), while a Langmuir-Hinshelwood model incorporating the effect of temperature has a total of five parameters (a , E , k_0 , ΔH_p , K_A). It should also be noted that using factorial designs often necessitates the use of nonlinear least-squares methods to obtain optimal values of ki-

netic parameters; hence, more sophisticated mathematical software programs may be required to complete data analysis. Discussions in this paper focus on traditional experiment designs and we would direct the reader to the literature for a detailed discussion of *design of experiments*.^[7]

The first steps in the experimental procedure are to turn on the combustible-gas-detector system, start flow of a mixture of helium (160 sccm) and hydrogen (160 sccm) to the reactor, set the reactor pressure to 5 psig (135 kPa), and adjust the setpoint of the temperature controller to obtain a catalyst bed temperature in the 460 to 495°C range. Once a temperature in this range is obtained, reduction of the catalyst is continued for roughly 1 hour; then the reactor temperature is lowered to the desired value for the first propane hydrogenolysis run, typically in the 310 to 340°C range. During this time, GC operation is initiated by setting flows of helium carrier to the column and both hydrogen and air to the flame ionization detector (FID). Next, propane is added to the reactor feed stream and the flowrates of the three components (C_3H_8 , H_2 , He) are set to the desired values. Hydrogen is fed in considerable excess (H_2/C_3H_8 molar ratio ≥ 6) in order to minimize deactivation due to coking. A typical feed mixture for a run might consist of 20 sccm C_3H_8 , 160 sccm H_2 , and 160 sccm He, corresponding to roughly 6 mole % C_3H_8 , 47% H_2 , and 47% He.

For the conditions associated with the sets of runs described below, propane conversions are generally in the 2 to 10% range; thus, the reactor can be approximated as being a “differential reactor.” Additionally, the selected flowrates, reactor temperature, and catalyst particle size ensure that the reactor pressure is axially uniform and are similar to conditions for which literature sources state that mass transfer effects did not distort the intrinsic kinetics.^[5,6] As will be discussed later, these approximations greatly simplify data analysis, leading to the determination of kinetic parameters.

When students are asked to determine power law kinetic parameters, the first set of runs will commonly focus on determining the propane reaction order (α) value. To collect data for these calculations, exit gas GC peak-area values are recorded for a range, *e.g.*, 10 to 30 sccm, of propane feed rates (and thus propane concentrations, C_p , or partial pressures, P_p), at constant reactor temperature and pressure. At the same time, the hydrogen concentration (or partial pressure) is held virtually constant by adjusting the helium flow such that total flow remains constant.

The aforementioned use of a great excess of H_2 , as well as differential reactor operation, facilitates isolating the effect of C_p on the rate of consumption of C_3H_8 , $-r_p$. For a given set of conditions, successive runs (typically two to four) are made to confirm that the data are reasonably reproducible.

In the second set of runs, data for the determination of the hydrogen reaction order (β) are acquired by varying the hy-

hydrogen feed rate (and concentration, C_{H_2}) at the same reactor temperature, pressure, and total flow, while using a constant feed C_p value. Occasionally, a second method for varying C_{H_2} is used, namely, varying the reactor pressure at a given H_2 feed rate, to obtain data for comparison with that of the first method.

The third set of runs examines the temperature dependence of the rate, specifically the activation energy, E , and the pre-exponential (frequency) factor, k_o , that appear in the Arrhenius expression for the specific reaction rate, k (*i.e.*, $k = k_o e^{-(E/RT)}$). In this part of the experiment, the pressure and feed composition are usually held constant, while the reactor temperature is varied in increments of 5 to 7 °C over an appreciable range, *e.g.*, from 310 to 340 °C.

The experiment as described takes two to three 3-hour periods that occur several days apart. Thus, it is important that the system be shut down after the first period and restarted for the second period in such a way that the catalyst's activity is unchanged. Shutdown is accomplished by first stopping propane flow while continuing to feed hydrogen and helium for at least fifteen minutes at the last temperature used, then lowering the reactor temperature set point to 0 °C. This purges the reactor of any adsorbed propane. During this interval, power to the GC/computer is cut off and flows of H_2 , He, and air to the GC are discontinued. Finally, power is cut off to the furnace, flow controllers, etc.

In order to achieve some diversity over a semester, the assignment (and thus the associated procedure) is often modified. In one such variant, the third set of runs is not devoted to determining the temperature dependence, but is used to study how well the reaction-rate expression developed from differential reactor operation at a constant temperature predicts integral packed-bed reactor behavior. In this case, the third set of runs involves conditions that give appreciably higher propane conversions, *e.g.*, 20 to 40%. Another variation involves students conducting experiments to determine parameters for a Langmuir-Hinshelwood model of the reaction process. A more detailed discussion of these experiments is provided in the data analysis section of this paper.

DATA ANALYSIS

As mentioned earlier, a group's preliminary report must address not only the procedure, but also the specific calculations and data analysis needed. The latter, in addition to being necessary for composing the final report, "shape" the experimental strategy by identifying the means by which the effect of a given variable can be isolated from that of others. The first portion of this section will describe data analysis for the simplified case of "power law kinetics." Later, a description is given outlining how this approach can be extended to deal with Langmuir-Hinshelwood rate equations. Before detailing how the power law kinetic parameters (α , β , E , k_o) are obtained, some background information will be provided.

Combining the rearranged propane balance for a packed-bed reactor with a power law approximation for the rate expressions gives

$$\frac{dF_p}{dW} = \frac{F_{po} dX_p}{dW} = -r_p = k_o e^{-\frac{E}{RT}} C_p^\alpha C_{H_2}^\beta \quad (2)$$

where F_p is the propane molar flow rate, W is the weight of catalyst, F_{po} is the propane molar feed rate, X_p is the propane fractional conversion, R is the gas constant, and T is the reactor absolute temperature.

For differential reactor operation, Eq. (2) can be approximated as a much simpler finite difference equation

$$\frac{F_{po} X_p}{W} = -r_p = k_o e^{-\frac{E}{RT}} \bar{C}_p^\alpha \bar{C}_{H_2}^\beta \quad (3)$$

where $\bar{T}$ is the virtually uniform temperature of the entire catalyst bed, and $\bar{C}_p$ and $\bar{C}_{H_2}$ are average concentration values for the respective species, which, for the very low conversions used, differ only slightly from either the feed or exit values. Before going further with the illustration of how Eq. (3) was used, two clarifying comments should be made. The first is that, although the use of low X_p values makes data analysis easier, it also introduces considerable relative uncertainty into the determination of X_p by the conventional method, *i.e.*, comparing the inlet and outlet GC peak areas for propane. To avoid this problem, a more accurate method for converting GC data to X_p values was used. This involved using the exit gas GC peak areas and FID response factors for C_3H_8 , C_2H_6 , and CH_4 , along with a carbon atom balance. The second comment regarding Eq. (3) is that, over the modest ranges of temperature and pressure studied, the irreversible power law expression is a reasonable approximation for the "true" rate equation.

Taking logarithms of both sides of Eq. (3) gives the "linearized" equation

$$\ln(-r_p) = \ln(k_o) - \frac{E}{RT} + \alpha \ln(\bar{C}_p) + \beta \ln(\bar{C}_{H_2}) \quad (4)$$

One option for evaluating the power law kinetic parameters, α , β , E , and k_o , is to conduct a series of experiments in which the rate is found for various combinations of $\bar{T}$, $\bar{C}_p$, and $\bar{C}_{H_2}$, and then use nonlinear least-squares software to extract the values from the entire data set. An optimal data set can be collected using experimental conditions obtained from a factorial design (or other design of experiments approach) that has been optimized for the variables of interest. This approach, while nominally viable and easy to implement, is, for several reasons, less desirable than the more structured approach associated with the experimental procedure described earlier. The first reason is that the inelegant (and, often, less reliable) "collective least squares" regression approach does not require the students to form their experimental plan in a logical fashion, *e.g.*, devise a sequence of experi-

mental (and computational) steps. A second reason is that it does not provide an opportunity to apply model development techniques that are central topics in most kinetics texts.^[2]

The data analysis strategy actually used by many groups is one that is a logical follow-up to the experimental procedure. It will now be illustrated for the simple power-law case using data/results taken from a representative UOL report. For the first set of runs, where $\bar{C}_p$ was the only independent variable, Eq. (4) simplifies to

$$\ln(-r_p) = \alpha \ln(\bar{C}_p) + C_1 \quad (5)$$

where C_1 is a constant. The value of α is found as the slope of a plot such as that seen in Figure 2. In the particular case shown, a value of 1.3 was found for α , which is above the 0.8 to 1.0 range found in the literature^[3-6] for this reaction over Pt/Al₂O₃. Over the past two years, other UOL groups have reported values ranging from 1.2 to 1.4. A representative sample of power-law kinetic parameters obtained by undergraduate students during the past two years is shown in Table 1. For the report whose results are being used as an example, a value of about -1.5 for β was found from a similar plot of $\ln(-r_p)$ versus $\ln(\bar{C}_{H_2})$ using data from the second set of runs, where $\bar{C}_{H_2}$ was the only independent variable. This value is within the -1.3 to -3 range reported in the literature and clearly shows the expected inhibiting effect of hydrogen adsorption. Other UOL groups reported β values ranging from -0.6 to -1.7.

Once the separate orders of reaction are known, the k_o and E values are found by first calculating specific reaction rate (k) values for each of the temperatures used in the third set of runs, where all concentrations and flow rates were held constant, and then constructing an "Arrhenius plot" such as Figure 3. The calculation of k is accomplished using

$$k = k_o e^{\frac{-E}{RT}} = \frac{-r_p}{\bar{C}_p^\alpha \bar{C}_{H_2}^\beta} \quad (6)$$

from which it can be seen that the slope of a linear plot of $\ln(k)$ versus $\bar{T}^{-1}$ equals $-E/R$ and the intercept as $T \rightarrow \infty$ equals $\ln(k_o)$. The results of Figure 3 correspond to $E \approx 164$ kJ/mole (39 kcal/mole) and $k_o \approx 8.5 \times 10^9$ moles^{1.2}/(cm^{0.6} · g catalyst · min). This activation energy is slightly lower than the 188 to 208 kJ/mole range reported by other investigators.^[3-6] As shown in Table 1, other UOL groups found E values ranging from a clearly too-low value of 93 kJ/mole to a reasonable value of 194 kJ/mole. It should also be noted that the same catalyst sample was used for all studies having data reported in Table 1.

If one wishes to use a Langmuir-Hinshelwood model, one of numerous possibilities is a model proposed by Leclercq, *et al.*,^[3] that assumes that the rate-controlling step is surface reaction between $C_3H_{x,ads}$ and either gaseous H_2 or associatively

adsorbed H_2 to form C_2H_y and CH_z , which are rapidly hydrogenated to (and desorbed as) C_2H_6 and CH_4 . Single site adsorption (with $C_3H_{x,ads}$ being the predominant surface species) is assumed. The rate expression in this case is

$$-r_p = k \frac{K_1 \bar{C}_p \bar{C}_{H_2}}{\bar{C}_{H_2}^a + K_1 \bar{C}_p} \quad (7)$$

where k is the pseudo surface reaction rate constant, K_1 is the propane equilibrium adsorption constant, and $a = 4 - x/2$. Note that if $\bar{C}_{H_2}^a \gg K_1 \bar{C}_p$, this simplifies to a power-law equation, i.e., $-r_p \approx k \bar{C}_p \bar{C}_{H_2}^{1-a}$. For the experimental results discussed above, where a value of -1.5 for β ($=1-a$) was found, the value for "a" would be 2.5. If accurate, this would imply that, on average, propane loses five H atoms upon adsorption and that the most probable values for y and z are 3 and 2, respectively. The proposed mechanism leading to this rate equation is summarized by the following elementary steps where "site" refers to an unoccupied surface site, "ads" implies adsorbed, and $y + z = x + 2 = 10 - 2a$:

- $C_3H_8 + \text{site} \rightleftharpoons (C_3H_{8-2a})_{ads} + aH_2$: fast, with equilibrium constant K_1
- $(C_3H_{8-2a})_{ads} + H_2 \rightarrow (C_2H_y)_{ads} + (CH_z)_{ads}$: slow, rate-controlling
- $(C_2H_y)_{ads} + (CH_z)_{ads} + H_2 \rightarrow C_2H_6 + CH_4$: fast

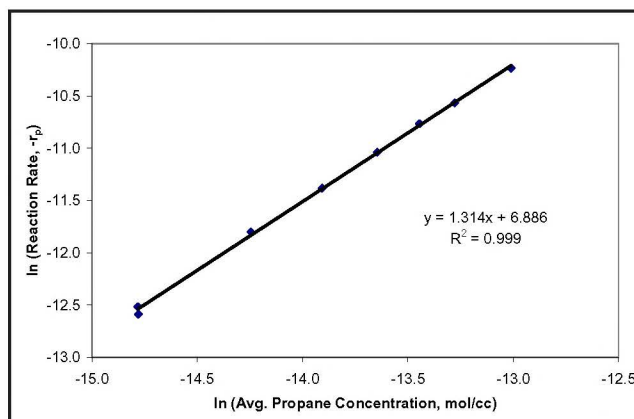


Figure 2. Determination of propane reaction order (α).

TABLE 1
Representative Power-Law Kinetic Parameters
Calculated from Experimental Data Collected
by Undergraduate Students

Experiment Date	E (kJ/mol)	α	β
9-23-02	185	1.3	-1.7
10-14-02	194	1.3	-1.5
11-11-02	164	1.2	-1.4
12-3-02	157	1.4	-1.2
4-1-03	164	1.3	-1.5
11-3-03	145	1.4	-0.6
12-3-03	93	1.3	-0.8

The specific rate equation shown above can be rearranged to give

$$\frac{\bar{C}_p \bar{C}_{H_2}}{-r_p} = \frac{\bar{C}_{H_2}^a}{k K_1} + \frac{\bar{C}_p}{k} \quad (8)$$

which, for a given assumed value of “a”, should give a straight line when $-\bar{r}_p^{-1} \bar{C}_p \bar{C}_{H_2}$ is plotted versus $\bar{C}_{H_2}^a$ for data taken at constant $\bar{C}_p$ and temperature. The best-fitting value for “a” can be found by varying $\bar{C}_{H_2}^a$ over the maximum practicable range and examining the resulting least squares correlation coefficient (R^2) values. Next, the k value can be determined from the intercept, *i.e.*, $k^{-1} \bar{C}_p$, and then the K_1 value can be found from the slope, *i.e.*, $(k K_1)^{-1}$. An alternative (or supplemental) method is to use data taken at constant $\bar{C}_{H_2}$ and temperature, with $\bar{C}_p$ intentionally varied. In that case (for a given assumed “a” value), the k and K_1 values can be found from the intercept $(k \bar{C}_{H_2})^{-1}$ and slope $(k K_1)^{-1} \bar{C}_{H_2}^{a-1}$ of a least squares fit of $-\bar{r}_p^{-1}$ versus $\bar{C}_p^{-1}$.

Assignments involving the use of Langmuir-Hinshelwood kinetics could range in difficulty from a case similar to the one just illustrated (where parameter evaluation is for a single given mechanism with the assumed rate-controlling step specified) to a challenging case in which the best-fitting mechanism/controlling step must be determined from a variety of proposed explanations/hypotheses. Students could also be asked to assume a specific Langmuir-Hinshelwood model and collect data to determine the activation energy, E , and frequency factor, k_o , for the reaction as well as a heat of adsorption, $-\Delta H_p$, for propane using an integrated form of van't Hoff's expression for the equilibrium adsorption constant,

$$K_1, \text{ i.e., } K_1 = K_A e^{-\Delta H_p / RT}$$

where K_A and ΔH_p are independent of temperature over the range studied.

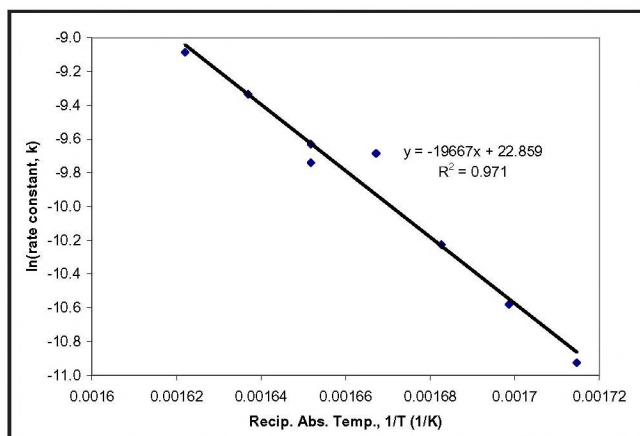


Figure 3. Arrhenius plot for determining frequency factor and activation energy for the Pt-catalyzed hydrogenolysis of propane.

CLOSING REMARKS

In this paper we have attempted to not only describe the apparatus and procedure used for our recently implemented kinetics experiment, but also to provide a rationale for its design and a sampling of results. The experiment offers students the opportunity to devise a workable plan for accomplishing a relatively challenging assignment (which can include developing a factorial design of experiments) and then to observe firsthand the effect of important variables (space time, feed composition, temperature) on the rate of a catalyzed reaction. In carrying out the experiment, students become familiar with up-to-date instrumentation and, in writing the final report, they employ a variety of numerical methods to obtain/analyze their results.

The primary advantage of the described kinetics experiment is its flexibility. The assignments can be kept simple and straightforward (*e.g.*, use classical methods to determine reaction order and activation energy values for a power-law model) or students can be challenged to develop a factorial design to efficiently determine all kinetic parameters for a Langmuir-Hinshelwood model that uses an Arrhenius law expression to describe variations in rate with temperature. Further, minimal changes to the reactor system would be required to have students examine other catalysts or gas-phase reactions (*e.g.*, hydrogenation of propene).

ACKNOWLEDGMENTS

The authors would like to thank Matt Rogers and Bill Coburn for their help in coupling the computer-controlled GC and reactor portions of the system, and Jay McAliley for assistance with drawing the process schematic. We also thank Norton Cater for arranging a financial gift from the Instrument Society of America and the National Science Foundation (CAREER-9985022) for support of this upgrade to our UO lab.

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