ChE laboratory

DEMONSTRATING CATALYTIC REACTOR STABILITY

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THE PURPOSE OF THIS communication is to bring to the attention of the chemical engineering community an example of a catalytic reaction that among other things, can be used to demonstrate reactor stability. The demonstration uses very simple and inexpensive equipment, is easy to assemble (taking less than an hour), and is highly portable.

The reaction itself is the catalytic oxidation of ammonia. As an example of catalysis, this reaction is well known in various forms in the literature on chemical demonstrations [1, 2]. However, the published accounts of it fail to appreciate not only that the reaction is controlled by film heat transfer, but also that the experiment can be used for a demonstration of catalytic reactor stability.

The apparatus used in the demonstration consists of:

1 200 ml erlenmeyer flask
1 pair of tongs
3 5-cm lengths of glass rod (3 mm diameter)
1 bottle of concentrated ammonium hydroxide solution
1 propane torch or bunsen burner
Several 30-cm lengths of copper wire of varying diameters (AWG Nos. 12, 18, 24; see Diameters in Table 1).

We shall first illustrate the phenomenon of control of reaction by heat transfer across the gas film. First, add about a 1-cm depth of ammonium hydroxide solution to the bottom of the flask. Then, using the No. 18 copper wire, fashion a helix of about 3 or 4 turns 1½ cm in diameter at one end of the wire, and wind a turn or two around a glass rod such that the helix can hang inside the flask within ½ cm of the surface of the ammonia solution. A schematic diagram of this configuration is shown in Figure 1. Grasping the wire and glass rod with the tongs, heat the copper helix in the torch flame to incandescence (yellow-orange). Then quickly place the wire in the flask.

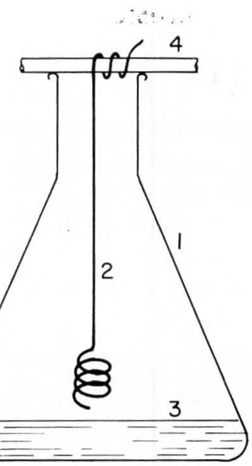

FIGURE 1. Diagram of the Apparatus.
Legend: 1 — erlenmeyer flask (200 ml), 2 — copper wire, 3 — concentrated ammonium hydroxide solution, 4 — glass rod.

The wire will continue to glow for some time (perhaps 10 to 15 min. or until the ammonia coming off the solution becomes too weak to support a reasonable rate of oxidation).

The experiment may be repeated using the No. 24 copper wire. The wire will reach yellow heat and melt. With No. 12 wire, however, the temperature will slowly decline during which the yellow-orange glow turns dull red and is finally extinguished. These various observations demonstrate the control of a catalytic reaction by heat transfer across the gas film.

Using the No. 18 copper wire, we can now illustrate the phenomenon of reactor stability. With the helix glowing in the flask, withdraw it fairly abruptly above the mouth of the flask. The glow should start to fade to a bright red. Immediately replace the wire in the flask. The glow should slowly return to the original yellow-orange colour. If the coil is once again withdrawn, but this time allowed to fade to dark red, and replaced in the flask again, the glow will not return. Rather, the glow will continue to be extinguished. This illustrates the phenomenon of reactor stability. Similar observations can be made with the No. 24 wire, except that the response time is much smaller. Also, some practice may be required to avoid melting the wire. With the No. 12 wire, a demonstration of stability is not possible, since the glow gradually declines after insertion in the flask. Perturbing the temperature by removing the wire from the flask simply hastens the decline. Typical stability results are offered in Table 1.

Some variation in these observations will occur depending upon the precise geometry of the copper coils, the proximity to the ammonia solution, and the concentration of the solution. The observations in Table 1 refer to fresh solution.

By now, an observer will have noticed a number of local changes in temperature on the surface of the copper coils. These take the form of the occasional flickering of the colour of the surface, sometimes in response to movement of the coil, at other times apparently in response to convection within the flask. Without pursuing the exact cause of these fluctuations, one may observe that the temperature colour changes are fairly large, and that they occur swiftly across the surface of the catalyst wire, despite its thermal inertia. This observation illustrates that transient temperatures are easily produced in catalyst particles with exothermic reactions.

Finally, questions that should be asked of students watching this demonstration include:

- What observations make it clear that the (pseudo) steady state reaction is heat transfer rather than mass transfer controlled?
- Estimate from the colour of the wire (or its melting point) a temperature difference across the gas film.
- Why does the thinner No. 24 wire become hotter than the thicker No. 18 wire? In your answer include the effects of radiation as well as conduction along the wire.
- Is conduction responsible for the gradual loss of glow in No. 12 wire? An experimental answer may be easiest here by cutting the helix off the No. 12 wire sample, and holding it by a thin wire (say No. 22 copper), heating to incandescence, and placing it in the flask.
- Explain by means of a diagram the return of the wire to its former temperature after withdrawal from and replacement in the flask. □

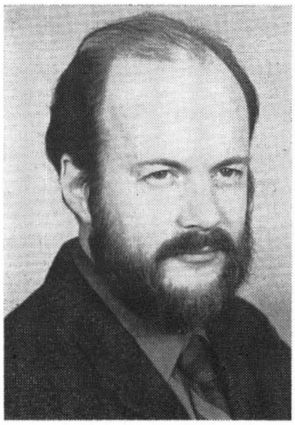

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TABLE 1
Typical Stability Observations

Wire gauge No. (AWG)	Diameter (in.)	Diameter (cm.)	Observations
12	0.081	0.21	Bright glow on entering flask gradually fades to extinction. Stability demonstration not possible.
18	0.040	0.10	Glow readily stabilizes at bright yellow-orange; when small temperature perturbation is introduced, glow slowly recovers to original colour; large perturbation of temperature extinguishes glow.
24	0.020	0.051	Glow can increase to yellow heat, melting wire. Observations similar to No. 18 wire, but with more rapid responses.

REFERENCES

1. G. Fowles, *Lecture Experiments in Chemistry*, 6th Ed., p. 178, G. Bell and Sons Ltd., London 1963.
2. N. Nechamkin and J. J. McClarnon, p. 169, 1960 Reprint Edition of "Tested Demonstrations", *J. Chem, Educ.*, H. N. Alyea, (editor).

ChE book reviews

Introduction to Control Systems
By D. K. Anand
Pergamon Press, Inc. 1974
Reviewed by Doug Wilde, Stanford University

This text is interchangeable with many other books on elementary linear control theory, all of which are good for electrical and mechanical engineers, and none of which deal with chemical engineering systems. There are books much more suitable for courses taught exclusively to chemical engineering students. Still other books would be better for a general control course mixing chemical with mechanical and electrical engineering students. This book would be appropriate only for a general control course not catering to chemical engineering students at all, and it is little different from earlier texts written for the same purpose. □