REB, The Book
Example 11.5.1 Solution

A rate expression for liquid-phase reaction (1) is needed for the design of a new batch reactor process. Preliminary studies suggest that a temperature in the range of 65 to 90 °C will be needed. At lower temperatures the reaction proceeds too slowly, and at higher temperatures undesired side reactions begin to occur. At a temperature of ca. 80 °C, it takes approximately 30 minutes for the reaction to go to completion. The preliminary experiments suggest that the reaction is irreversible and that the rate is not affected by the concentration of the product, Z. It is expected that the reactant, A, will be available at concentrations between 0.8 and 1.2 M. A perfectly mixed, 1 L BSTR is available for kinetics experiments. The reactor has a sampling port that allows the concentration of A to be measured while the reaction is taking place. Design a set of experiments to generate kinetics data that can be used to develop a rate expression.

$$A \rightarrow Z \tag{1}$$

Experimental Design

Three reactor inputs will be adjusted in the experiments: the temperature, T, the initial concentration of A, $C_{A,0}$, and the reaction time, t_{meas}. The temperature levels will be 65, 73, 82, and 90 °C; the initial concentration levels will be 0.5, 1.0, and 1.5 M; the reaction time levels will be 5, 10, 15, 20, 25, and 30 min. All possible combinations of these levels will be studied giving a total of 72 experimental data points.

It will only be necessary to perform 12 experiments to generate those 72 data points. After setting the temperature and the initial concentration of A for an experiment, the response can be measured at all six reaction times during that experiment. There are 12 possible combinations of the temperature levels and the initial concentration levels as shown in Table 1. Each experiment in the table will generate 6 data points, resulting in a total of 72 data points.

Table 1. Experimental Design

Experiment	Temperature (°C)	Initial A Concentration (M)
1	65	0.5
2	65	1
3	65	1.5
4	73	0.5
5	73	1
6	73	1.5
7	82	0.5
8	82	1
9	82	1.5
10	90	0.5
11	90	1
12	90	1.5

Discussion

The range of temperatures where the rate expression may be used only spans 25 °C, so four evenly spaced levels were deemed sufficient. The assignment narrative notes that at 80°C the reaction goes to completion in 30 min, so six reaction time levels, spaced at 5 min intervals were selected. At the highest temperature, 90 °C, the reaction will likely go to completion in less than 30 minutes, so the last few data points may not yield useful data points. Noting that during the course of the experiments the concentration of A will decrease with reaction time, only three initial concentration levels were chosen. The full range of measured responses should span the range of interest from 1.2 M to 0 M. The narrative notes that Z does not affect the rate, so the initial concentration of Z is zero in all experiments. Similarly, it was assumed that because the reaction is liquid-phase, pressure would not affect the rate significantly, so all experiments are to be performed at the same pressure of 1 atm. A full factorial design was specified noting that it would only require 12 experiments that each yield 6 data points.