

Design and Construction of a Reactor for *in Situ* Infrared Studies of Catalytic Reactions

Transmission infrared spectroscopy is a very useful technique for characterizing the structure of adsorbed species present on a catalyst under reaction conditions. Reactors for conducting such *in situ* studies have been described by a number of authors [e.g., Refs. (1-7)]. In the present note we wish to report the details for a very simple reactor which can be used to study catalytic reactions at moderate temperatures (<573 K) and elevated pressures. The reactor has a very low dead volume and a short optical path length, and is built in such a fashion that infrared windows and catalyst samples can be replaced quite easily.

An exploded view of the reactor is shown in Fig. 1 and more detailed views of the primary components are shown in Fig. 2. The reactor body is constructed of two stainless-steel flanges, 7.62 cm in diameter. Knife edges are cut into both flanges to permit the formation of a gas-tight seal when the reactor is assembled. Polycrystalline CaF_2 windows (Harshaw Chemical Co.), 25 mm wide and 7 mm thick, are placed into wells cut in each flange. The seal between each window and its corresponding flange is formed by Kalrez O-rings (Du Pont). The bottom flange has two passages, terminated by 1/16-in. Swagelok fittings, one for the inlet and one for the outlet of the gas flow. A third passage is used to introduce a 0.8-mm sheathed thermocouple. By soldering a 1.6-mm sleeve over the sheath, the thermocouple can be seated into the reactor using a 1/16-in. Swagelok fitting.

A pressed catalyst wafer, 20 mm wide and 0.25 mm thick, weighing 75 mg, is held in an aluminum sample holder. Grooves cut on the inside surfaces of the sample holder

assure an even distribution of gas flow around the catalyst disk and good gas mixing in the space surrounding the catalyst disk. A small notch located on one side of the holder permits insertion of the thermocouple and prevents rotation of the holder in the reactor. By placing the thermocouple in close proximity with the catalyst, an accurate measurement of the catalyst temperature can be obtained.

When the reactor is assembled, the CaF_2 windows press directly against the sample holder and act as walls for the cavity surrounding the catalyst disk. The gas volume

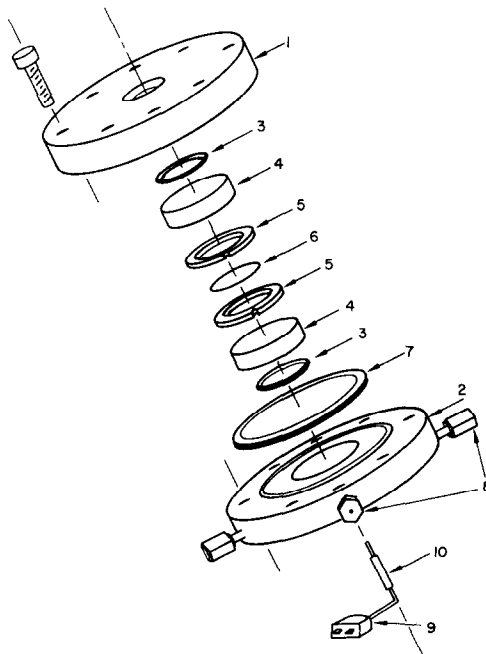


FIG. 1. Exploded view of the reactor: (1) top flange; (2) bottom flange; (3) Kalrez O-ring; (4) CaF_2 window; (5) sample holder; (6) catalyst disk; (7) copper gasket; (8) Swagelok fitting; (9) sheathed thermocouple; (10) sleeve attached to thermocouple sheath.

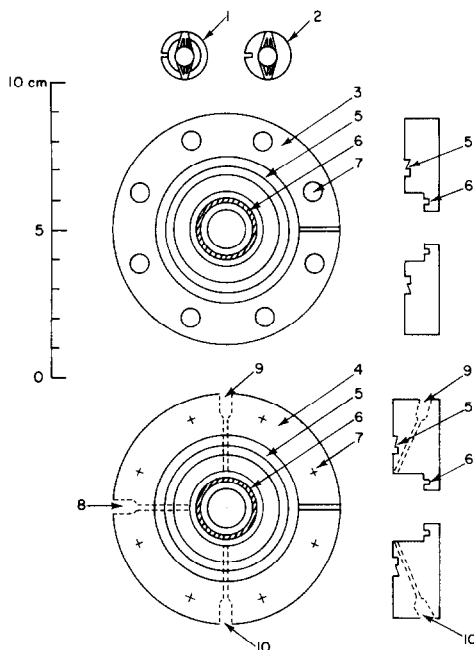


FIG. 2. Details of reactor and sample holder: (1) bottom half of sample holder; (2) top half of sample holder; (3) top flange; (4) bottom flange; (5) knife edge; (6) O-ring groove; (7) bolt holes; (8) thermocouple port; (9) gas inlet; (10) gas outlet.

of this cavity is 0.4 cm^3 and the optical path length through the cavity is 2.4 mm, including the thickness of the catalyst disk. Because the gas cavity volume is small, residence times of a few tenths of a second can be achieved with gas flow rates of several hundred cubic centimeters per minute at STP. This characteristic is particularly attractive for conducting transient response studies. The short optical path length through the reactor helps to minimize infrared absorption in the gas phase, and thereby facilitates the detection of spectral features due to adsorbed species.

The reactor is heated by two Heat-Sheet etched foil heating elements (Thermal Circuits, Inc.), each of which is capable of delivering 200 W. To distribute the heat evenly, a 0.32-cm-thick copper gasket is placed between each heating element and the circular face of the flange that it heats. The reactor temperature is maintained con-

stant to within $\pm 0.5 \text{ K}$ by a time-proportioning temperature controller (Omega Engineering, Inc.) which is connected to the heating elements and the thermocouple located inside the reactor.

The reactor described here has been used to study the hydrogenation of CO at temperatures up to 573 K and pressures as high as 2.4 MPa. No problems have been encountered with either extended operation or cycling of the temperature between 373 and 573 K. The upper limit on the operating temperature is 590 K and is set by the Kalrez O-rings. The upper limit on pressure is not as clearly established. Based on the manufacturer's literature (8), the CaF_2 windows described here can be used for pressures up to 7.6 MPa at a temperature of 298 K. The extent to which this limit should be reduced at higher temperatures is not specified. It should be noted that operation of the reactor at higher pressures could have been achieved by using thicker windows and altering the dimensions of the flanges accordingly.

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