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Note

Simple pulse micro-reactor combined with a gas chromatograph*

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Micro-reactors combined with a gas chromatograph are extensively employed[1,2] in catalytic research for catalyst evaluation, studies of kinetics and mechanisms using labelled compounds, poisoning, adsorption and studies of the initial surface state of solid catalysts. Several designs for micro-reactor are available[2]. In order to connect a micro-reactor to a gas chromatograph, certain modifications to the conventional gas chromatographic (GC) unit have to be made. Further, it is necessary to heat the long connecting tube between the micro-reactor and the analytical GC column in order to prevent condensation of the reactants and products. Both problems can be overcome by fitting the micro-reactor directly in the oven of the chromatograph.

In this paper, a simple pulse micro-reactor, suitable for fitting in the oven of a conventional dual-column gas chromatograph with a flame-ionization detector (FID), without making any modifications to the GC unit is described. The apparatus is shown in Fig. 1. It consists of a rotameter for measuring the carrier gas flow-rate,

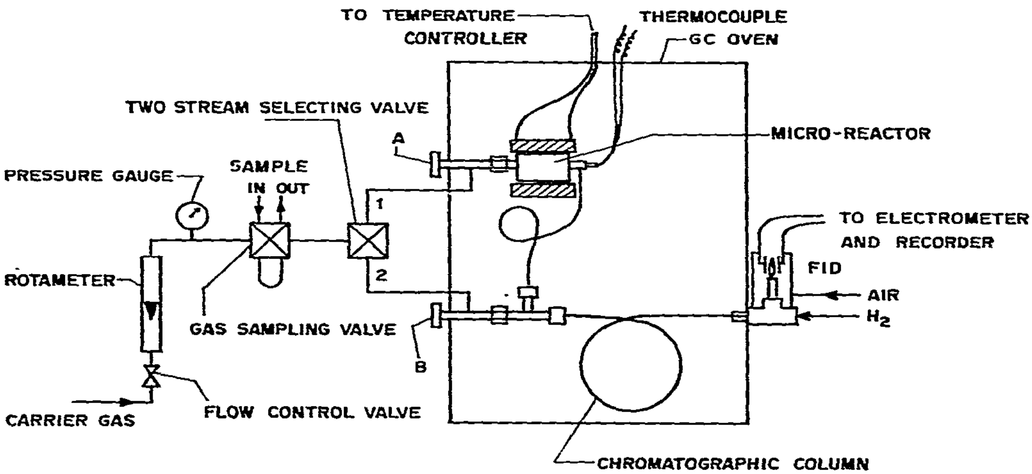

Fig. 1. Schematic diagram of micro-reactor combined with a gas chromatograph.

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a pressure gauge, a gas sampling valve for introducing gaseous samples or reactants, a two-stream selecting valve for diverting the flow of carrier gas to position (1) or (2) and a conventional dual-column gas chromatograph with injector ports (A and B) for injecting liquid samples or reactants, a gradientless air oven in which the micro-reactor and chromatographic column are connected in series and an FID.

The detailed design of micro-reactor is shown in Fig. 2. The reactor and the furnace are made of stainless steel. About 0.1–0.8-cm^3 catalyst particles can be charged into the reactor. The reactor temperature can be varied from the minimum (the oven temperature required for the effective separation of a reaction mixture on the chromatographic column) to a maximum of *ca.* 500°. The catalyst temperature is measured with a chromel–alumel thermocouple. As the furnace is well insulated, its temperature has a negligible influence on the oven temperature. The capillary tube connection between the micro-reactor and the chromatographic column acts as a heat exchanger and the gas entering the column attains the temperature of the oven. With high-temperature operation of the micro-reactor, the length of the connecting capillary tubing can be increased in order to obtain the required cooling of the reactor effluent gases before they enter the chromatographic column. The micro-reactor has

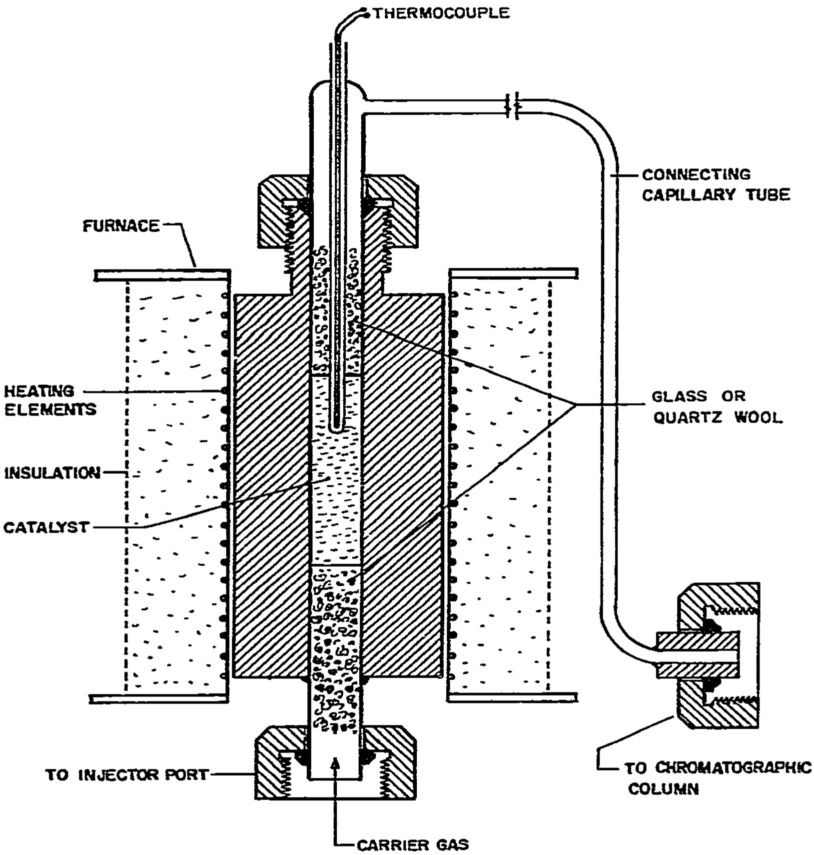

Fig. 2. Details of micro-reactor.

a thick wall (*ca.* 10 mm), which helps to achieve a uniform temperature throughout the catalyst bed and also to ensure a high heat capacity of the reactor, which overcomes any small heat changes that might occur in the reactor owing to highly exothermic or endothermic reactions. In fact, the heat changes involved in the pulse micro-reactor due to reaction are negligibly small as the extent of reaction involved is very small.

When the flow of carrier gas is switched to position (1), the pulse of reactant (or reactants) is carried through the reactor (where the reaction occurs on the catalyst surface) by a stream of carrier gas, and the reaction products are conducted directly into the chromatographic column. The micro-reactor can be by-passed by switching the flow of carrier gas to position (2) for the calibration required for quantitative analysis or for any other analysis when the micro-reactor is not in use. Thus, any other routine analysis can be carried out by replacing the column by another one without disturbing the micro-reactor assembly.

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