

# Apparatus for the study of reaction kinetics of gas—liquid systems under pressure

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Received 2 November 1983

Accepted for publication 12 March 1985

The apparatus for kinetic study of gas—liquid reactions at high pressures and temperatures is described.

Описан прибор для кинетического изучения газо-жидкостных реакций при высоких давлениях и температурах.

Measuring of gas consumption at constant pressure provides an advantageous system for the study of the course of many gas—liquid reactions, e.g. hydrogenation, oxidation, alkylation, carboxylation, etc. For this purpose various automatic apparatuses, mostly working at lower pressure, were constructed [1—3]. In practice, however, kinetic data free from mass and heat transfer at the conditions of the technological process, i.e. at high temperatures and high pressures, are needed. In cases, when during a chemical reaction the increased production of secondary gas products is limited, it is desirable to make use of the principle of gas consumption measurement also at the above-mentioned conditions [4—6].

In this paper a pressure apparatus for both the kinetic study of gas—liquid reactions and the rapid catalyst testing at high pressures (0.1—10.0 MPa or more) and high temperatures (up to 250 °C) is described. The schematic diagram of the apparatus is shown in Fig. 1. The major parts including tubes and fittings are made from stainless steel. The reacting gas is supplied from a high pressure cylinder (1) into the measuring and reacting system. Pressure in the system is measured by a precise Bourdon-tube pressure gauge (2). The reaction vessel (3) immersed in a thermostatically controlled oil bath (4) with intense shaking using a vibrator (5) is connected to the gas supply with a flexible metal capillary (7). Small changes in pressure inside the vessel due to the chemical reaction produce pressure differences between the two chambers in the supply system which are separated by a conductive fluid in U-tube manometer (8). These cause in the manometric system (8) an interruption of the electric circuit (max. 24 V) between the manometric contact (9) and the fluid, the consequence of which is an automatic

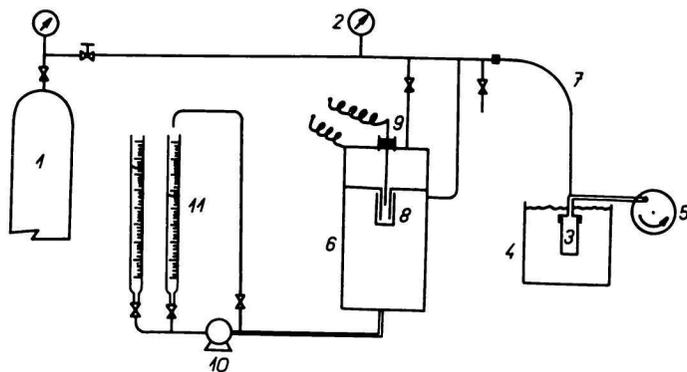


Fig. 1. Schematic diagram of the apparatus.

switching off the pressure pump (10) by a relay system. The unreactive fluid with a low viscosity and volatility is pumped from a glass burette (11) to the lower chamber of the supply system (6), until the pressure decrease is compensated, i.e. the relay operates on the closing of the manometric contact.

The U-tube manometer (8), depicted in detail in Fig. 2, separating two chambers (12, 13) of the supply system (6) consists of a 70–90 mm long tube (14) with an inner diameter of 6–8 mm placed in a reservoir (15) with inner diameter of 18–25 mm fixed on a plate between the chambers. The manometric contact (16) insulated from the metal body of the supply system by a Teflon seal (17) is placed in the centre of the tube (14) which is inside covered with an insulated layer to prevent a false contact with the tube wall.

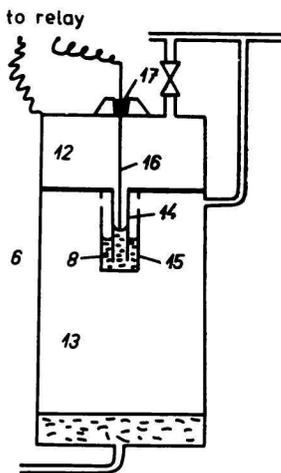


Fig. 2. Manometric system.

Before each experiment the apparatus is flushed with the reacting gas. Then, 15—20 cm<sup>3</sup> of the fluid is pumped to the thermostated supply system (6). Under the applied pressure and temperature of the supply system, a certain solubility of the reacting gas in the fluid exists and therefore calibration to zero consumption is done following the gas consumption in the absence of the reacting component inside the reactor. The standard apparatus has a 50 cm<sup>3</sup> stainless steel reactor and an approximately 350 cm<sup>3</sup> supply system. The catalyst is separately weighed on a Teflon foil or enclosed in a sealed tube placed inside the reactor and after heating of the reactor in a thermostated oil bath (5—7 min) the catalyst is mixed shaking the reactor using a vibrator. Measuring the volume of the pumped fluid under vigorous agitation in time intervals one can estimate the rate of gas consumption at constant pressure. The fastest recorded gas absorption is mostly dependent upon the capacity of the pressure pump.

The described apparatus has proved to be reliable in thousands of runs, mostly in oxidation, hydrogenation, and alkylation reactions. It was also used for the measurement of the solubility of different gases in organic solvents under pressure [7] and the evaluation of gases during a chemical reaction. The simplicity of operation makes the apparatus suitable for automation.

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Translated by M. Hronec