JOURNAL OF CATALYSIS 4, 625–627 (1965)

NOTES

# A Reliable Noncontaminating Gas Recirculation Pump*

A reliable, maintenance-free, noncontaminating gas recirculation pump was developed for use in recirculation reactor systems.

Recirculation systems have been stated by Temkin (*1*, *2*) as being a superior way to perform kinetic studies with solid catalysts. However, wider use of these systems has been prevented by the unavailability of a truly satisfactory pump.

There are no commercial pumps that satisfy the criteria of providing a high flow rate and a moderate pressure differential while being inert, vacuum-tight, and noncontaminating. Several designs have been mentioned in the literature (*3–6*), but none was completely satisfactory: some used metal parts exposed to reactants, some required lubricating agents, and others employed glass pistons inside glass cylinders which tended to either wear excessively or develop insufficient pressure differential. A design that comes close to being satisfactory is that of Artyukh and Nikolenko (*7*), who used a Teflon piston with Teflon rings inside a glass cylinder. A magnetic drive raised the piston, the return stroke being provided by gravity. This design was satisfactory except for the different speed of the two strokes and the low differential pressure necessitated by the gravity return stroke.

The basic ideas of Artyukh and Nikolenko were incorporated into the current pump except that a design was used in which both piston strokes were driven by alternately energizing the solenoids shown in Fig. 1. The piston is constructed of Teflon with an internal cavity which contains an iron-nickel alloy core (Armco 48NI) chosen for its magnetic properties. The cylinder is made from a 220-mm section of a 1½-inch ID precision-bore Pyrex tubing. A series of Teflon O-rings provides the seal between the piston and cylinder. Surrounding the glass cylinder is a 1⅞-inch brass tube upon which the solenoids

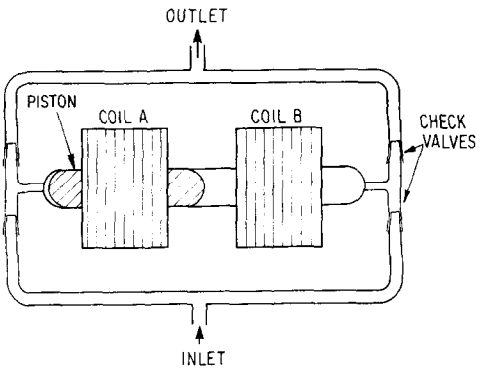


FIG. 1. Schematic drawing of pump.

are wound. A small air stream (<10 l/min) flows through the narrow annulus between the cylinder and the brass tube to provide temperature control. Because of the high thermal expansion of Teflon relative to glass, this temperature control adjusts the force of the O-rings against the cylinder wall, hence adjusting the seal between the piston and the cylinder. This provides a reliable method of achieving satisfactory pump performance unaffected by solenoid current.

The solenoid and cylinder assembly is enclosed in a steel shell fitted with a water-cooling coil to dissipate the heat from the solenoids. Steel or alloy sections on either side of the solenoids provide an effective magnetic path maintaining a high magnetic

* This work was supported in part by NSF Grant GP-2305.

flux through the alloy core. The 4500-turn solenoids are alternately energized with direct current by means of a Microswitch operating off an adjustable speed cam. The check valves are constructed of 6 mm × 2 mm quartz discs hand-polished to the end of lengths of 6-mm capillary tubing. This simple valve provides an amazing seal, the total leakage for the four valves being less than 3% of the pump output at 100 Torr pressure differential.

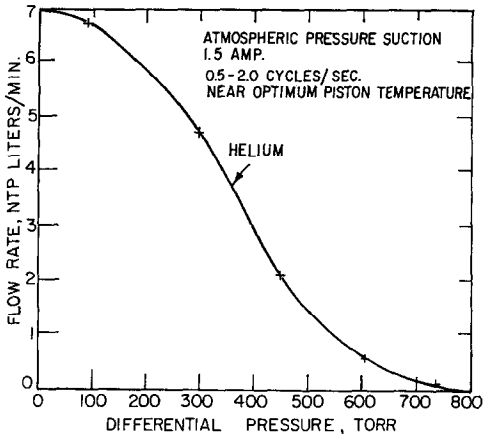


FIG. 2. Pump characteristic curve.

The pump characteristic curves, Fig. 2, indicate that the pump delivers more than ample pressure differential. A pump constructed from this design has been used for kinetic studies and has provided maintenance-free operation, except for several Microswitch changes, over more than 500 hr of operation without loss of capacity. Not only is the pump maintenance-free, but it is also quite versatile. It is capable of operating under pressure or vacuum conditions and, with adjustment of piston rings, can be operated up to the degradation temperature of Teflon.

ACKNOWLEDGMENT

Many thanks to Dr. R. J. Madix for his valuable contributions in the early stages of this work.

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*Received June 17, 1965*

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## Oxidation of $Mn^{II}$ Acetate by Alkylperoxy Radicals

The catalytic effect of heavy metal ions such as those of Mn and Co often depends on their efficiency in generating free radical species from hydroperoxides, both in the lower and in the higher valency stage. The present communication deals with a case where both stages are relatively inert towards hydroperoxide. It is shown that $Mn^{II}$ acetate is readily oxidized directly by alkylperoxy radicals to form $Mn^{III}$ acetate. This direct oxidation has been postulated [e.g., ref. (*1*)] but not, to our knowledge, unequivocally demonstrated.

Cyanoisopropyl radicals (R·) were gen-