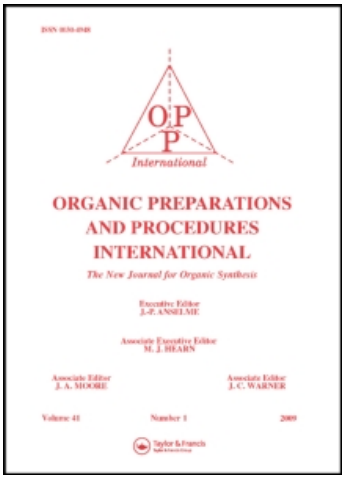

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A SIMPLE, INEXPENSIVE SYSTEM FOR PERFORMING HIGH PRESSURE REACTIONS

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kopf, Ber., 42, 617 (1909)].

5. Tautomerization of a nitrolic acid into a nitronic acid is apparently unknown.

6. P. A. S. Smith and J. E. Robertson, J. Am. Chem. Soc., 84, 1197 (1962).

7. P. A. S. Smith, "Open-chain Nitrogen Compounds", Vol. II, p. 431, W. A. Benjamin, Inc., New York, 1966.

A SIMPLE, INEXPENSIVE SYSTEM FOR PERFORMING HIGH PRESSURE REACTIONS

Submitted by (2/26/82) Philip DeShong,*[†] C. Michael Dicken,[†] Joseph J. Perez,[†] and Robert M. Shoff[††]

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A pressure system capable of maintaining reaction volumes at pressures of 1-8 Kbar was required because previously reported systems[1] have a variety of flaws:

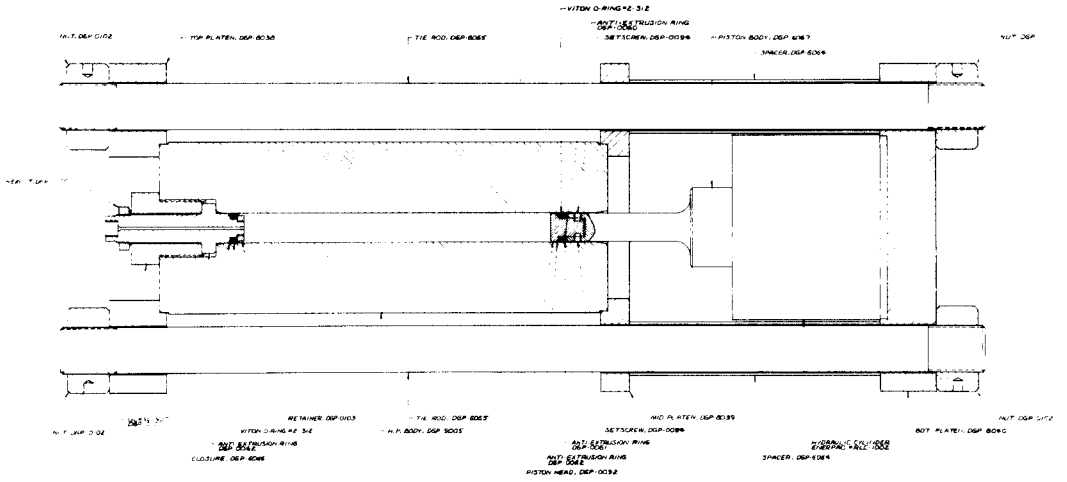
1. They required elaborate pumping apparatus for attaining the required pressure and are therefore, expensive.

2. Large reaction volumes could not be employed.

3. The system utilized a gas-pressurized reaction vessel, thus introducing safety considerations.

We now report a high pressure system which is mechanically simple, inexpensive to build, safe, and which can be used with

Figure 1. Schematic of High Pressure System



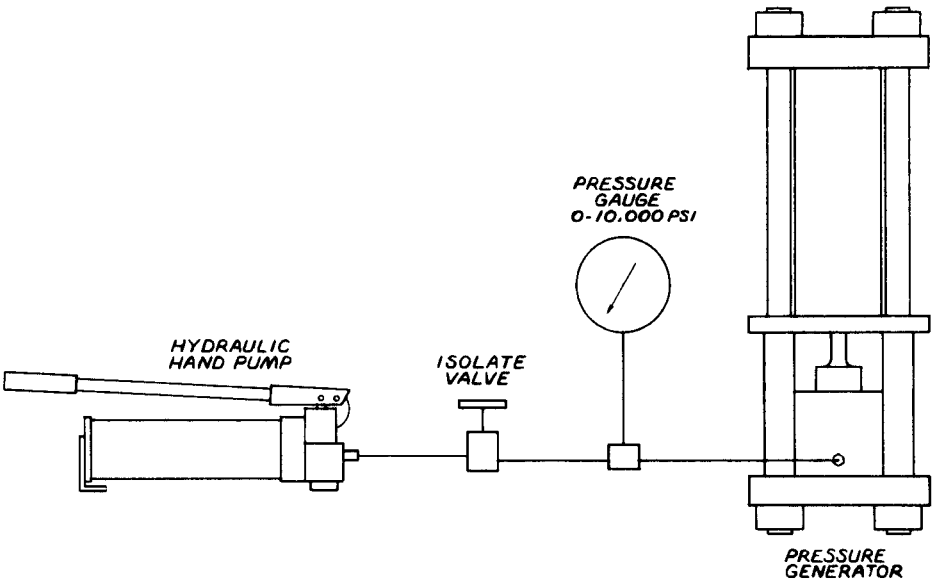
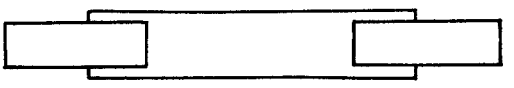


Figure 2. Reaction Vessel

glass plug heat shrinkable teflon tubing



synthetically significant volumes of reactants.

A schematic of the system is presented in Fig. 1 and consists of a reinforced, steel chamber which is pressurized by a hydraulically-driven piston using unsupported area seals. A smaller hydraulic device drives the piston (Castor oil is employed as the high pressure medium). Reaction pressures are measured indirectly by measuring the pressure generated in the smaller hydraulic device which has been calibrated. This indirect method alleviates the need for an expensive, ultra high pressure gauge.[2] An additional problem encountered concerned the separation of the chemical reactants from the pressurizing fluid. A variety of reaction containers were investigated such as metal, teflon, polymeric substances and were found to be deficient. The container does not have to withstand tremendous pressures since the surrounding environment is isobaric, but must be capable of contracting and expanding during the reaction. Finally the container has to be inert to an assortment of organic reagents. It was found that a simple container can be made from a length of heat-shrinkable Teflon tubing (Ace Glass Co.) of an appropriate diameter and length to accomodate the volume of reactants, plugged at each end with a small section of solid glass rod (Fig. 2). Care is taken to remove gas bubbles after the reaction mixture has been introduced. This system has never failed even under conditions as extreme as 75^{o}/8 Kbar/48 hrs.

REFERENCES

1. M. Tkacz and B. Baranowski, Rocz. Chem., 50, 2159 (1976); W. LeNoble, Prog. Phys. Org. Chem., 5, 207 (1967); F.

Fleischmann, E. Conze, D. Stranks and H. Kelm, Rev. Sci. Instrum., 45, 1427 (1974); R. Greiger and C. Eckert AIChE J., 16, 766 (1970). K. Brower, J. Am. Chem. Soc., 90, 5401 (1968); T. Moriyoshi; Bull. Chem. Soc. Japan, 44, 2582 (1971); H. Yamada, Chem. Lett., 747 (1972). A. Jost, Ber. Bunsenges. Phys. Chem., 79, 850 (1975); A. Yu, M. Waissbluth and R. Grieger, Rev. Sci. Instrum., 44, 1390 (1973); J. Jonas, ibid, 43, 643 (1972).

2. The apparatus can be obtained commercially from Leco Corporation, Tem-Pres Division, Bellefonte, PA 16823.