

A simple device for loading gases in high-pressure experiments*

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ABSTRACT

We have developed a conceptually and operationally simple device for loading gases into capsules for experimentation at high pressures, such as in cold-seal bombs and piston-cylinder apparatus. For example, we have successfully loaded Ar, Xe, Kr, N₂, CO₂, and other gases, together with silicate solids, in noble-metal capsules.

This device is a small block, which is easily machined from metal or plastic, that uses vacuum to evacuate the air from the capsule, loads gas into the capsule, and holds the capsule for crimping prior to welding. The gas pressure and the precise amount of gas in the capsule are easily controlled, and it is possible to load as much as 100-bars pressure in capsules with 0.2-mm-thick walls.

The main advantage of this system is that precise quantities of very pure gases can be loaded with minimal pollution by water or other contaminants in the atmosphere, because the ambient atmosphere is purged from the capsule during the loading process. Some substances, e.g., Xe, are obtainable only in the gaseous state and are difficult to load using other techniques. For CO₂, this system eliminates the need for using silver oxalate, which adds Ag to the system and is impossible to dry adequately. Our system is preferable for nearly all volatile components, and it is easily adaptable to mixtures of two or more gases.

INTRODUCTION

Recently, we completed a study of the solubility of Ar in silicate liquids at high pressures (White et al., 1989), using a special glove box to load liquid Ar into the capsules. We used a similar procedure for N₂ (Boettcher et al., 1987). We extended these studies to other volatile components, but we have now developed a technique for loading these components as *gases*. The resulting process provides a simple, inexpensive, and effective way to load almost any gas or mixture of gases with virtually no contamination by the atmosphere or by other elements. For example, CO₂ can be loaded as a very pure gas rather than using silver oxalate, which cannot be adequately dried and which releases Ag under the experimental conditions.

THE APPARATUS

The device has several functions. (1) It connects the capsule to a vacuum line to evacuate most of the air from

the capsule and the sample. (2) It connects the capsule to the source of pressurized gas. (3) It crimps the capsule so that the gas does not escape after the capsule is removed and before it is welded.

The basic design is shown in Figure 1. We make our blocks of unhardened 4340 steel, but plastic or other materials would work for most applications. The design in Figure 1 is for a 2-mm outside diameter capsule, but it can be adapted for other sizes. A schematic of the entire system is shown in Figure 2. This system is easily modified for loading measured quantities of mixtures of gases. The desired amount of gas in the capsule is obtained by adjusting the pressure on the gas regulator(s).

OPERATION

Preparation of the capsule begins with proper annealing of the tubing to ensure that it is sufficiently malleable to avoid damage during the sealing and crimping processes. For Pt, we recommend 1400 °C for 1 h. After the capsule is (triple) welded at one end, the sample is loaded into this end and packed tightly with a steel plunger.

The capsule is inserted into components *B* and *E* from the top, with the welded end down. The outer surface of

* Institute of Geophysics and Planetary Physics Contribution Number 3239.

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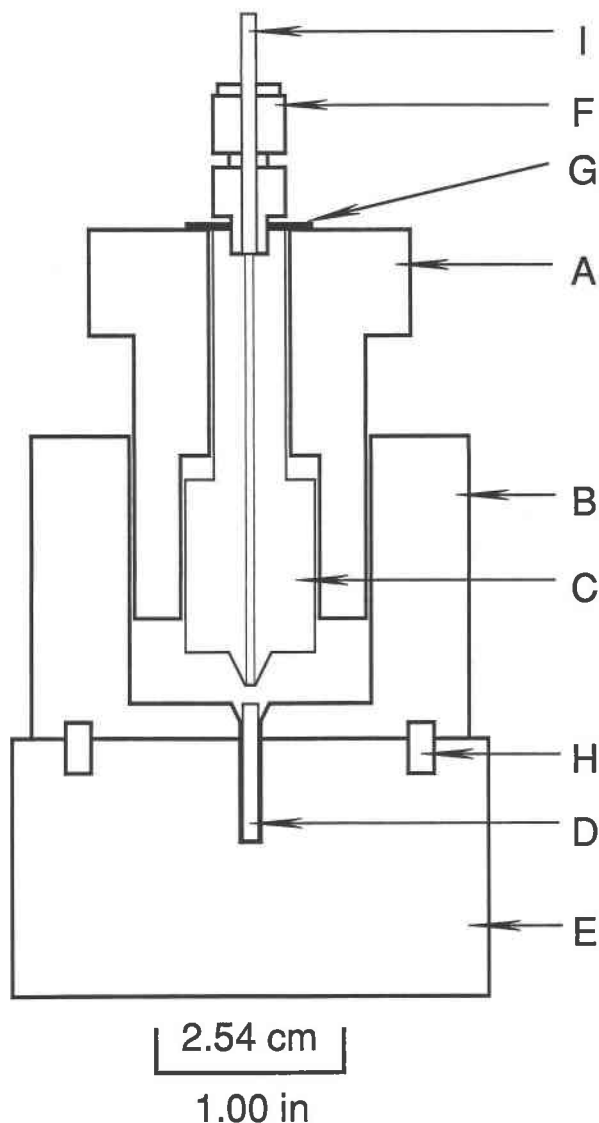


Fig. 1. Gas-loading device. *A*, *B*, *C*, and *E* are steel cylinders. The outside of *A* and the inside of *B* are threaded and screwed together. The Swagelock fitting *F* is screwed into a fitting, which is screwed into *C*. *D* is the sample capsule. *H* is one of two alignment pins. *G* is a loosely fitting steel washer that holds *A* and *C* together. *I* is the line for connection to the vacuum pump and for gas loading.

A and the inner surface of *B* are machined with at least 8 threads per centimeter to provide accurate travel and alignment of the cone into the opening of the capsule. Parts *B* and *E* are held together by hand or fastened with

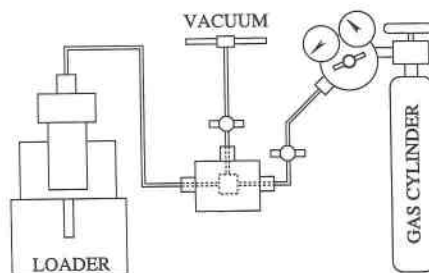


Fig. 2. Schematic arrangement of gas-loading system.

internal screws during sealing. *A* rotates independently of *C* to avoid twisting the capsule during insertion of the cone into the capsule. When screwing *A* into *B*, fingertight force is sufficient to obtain the necessary seal between the capsule and *C*. At this point, vacuum is initiated on line *I*, held for ~ 2 min, shut off, and the gas is introduced. We repeat this process several times to purge the ambient atmosphere from the chamber and capsule.

In the final stage, *B* and *E* are pulled apart, which exposes the lower part of the capsule, enabling component *B* to be placed on the vise. The gas remains on until the capsule is crimped in the vise. The jaws of the vise are made with rounded edges and a small surface area to avoid damage and excessive loss of Pt or other material. After the capsule is crimped, the gas is shut off and the chamber is evacuated. Finally, *A* is twisted partially out to remove the cone from the capsule. With the capsule in the vise, *B* is pulled upward and off to expose the top of the capsule, which is then crimped with pliers and cut along the top to create an ideal surface for welding. It is unnecessary to use water or other coolant during welding. Once welded, the capsule is removed from the vise and reweighed, along with the "cut" portion of the capsule to determine the amount of gas. The capsule is now ready for experimentation. With some practice, the entire loading operation takes less than 5 min.

This system is advantageous in that precise quantities of a very pure gas or a mixture of gases can be loaded into metal capsules without contamination by the ambient atmosphere.

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MANUSCRIPT RECEIVED MAY 22, 1989

MANUSCRIPT ACCEPTED JULY 26, 1989