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Publication Date

1966-07-01

UCRL-16994

University of California

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Sub. to Rev.
Sci. Instr.

UCRL-16994

UNIVERSITY OF CALIFORNIA
Lawrence Radiation Laboratory
Berkeley, California
AEC Contract No. W-7405-eng-48

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July 1966

DETERMINATION OF MASS OF LARGE GAS VOLUMES BY DIRECT WEIGHING

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ABSTRACT

The usual method of accurately measuring the mass of a large (50 - 100 l.) gas sample involves the inconvenient handling of large volumes of gas at controlled temperature and pressure. The design and construction of a small high-pressure gas vessel (made possible by the recent development of strong, low-density alloys) is described. The full vessel was light enough to be accurately weighed on a conventional laboratory balance, and hence the mass of gas (high-purity argon, krypton) was determined directly and simply. Cryogenic pumping was used to return the gas to the weighing vessel after an experiment. Accessory gas-handling equipment is also described.

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INTRODUCTION

In recent measurements of the specific heat of solid argon and krypton at low temperatures¹ it was necessary to determine the mass of the sample to better than 0.1%. The usual method of measuring the mass of such a sample is to measure the pressure, volume and temperature of the gas at room temperature and at around one atmosphere. One then applies the ideal gas law, the departures therefrom usually being negligible. However, in the above experiments, large solid samples of from 50 c.c. to 60 c.c. were used, so that the room-temperature volume of some 50 l. would be awkward to thermostat and occupy much valuable equipment space. Hence it was decided to put the gas in a small pressure vessel, and weigh it directly on a laboratory balance, for which the accuracy is well within ± 10 mg. in 80 g. This previously impractical approach is now possible because of the recent development of lightweight high strength alloys. As is later described, this method also lent itself to convenient handling and storage of the high-purity gases.

DESIGN OF PRESSURE VESSEL

To examine the factors involved in the design of the pressure vessel, let us consider the simplest case of a spherical vessel of radius r , with thin walls of thickness t , of density ρ and ultimate tensile strength Y . For a working pressure p and safety factor s , the (optimistic) thin-wall pressure formula gives,²

$$p = 2 tY/rs.$$

By Boyle's Law,

$$p(4/3)\pi r^3 = p_0 v_0,$$

where v_0 is the volume of the gas at a pressure p_0 (usually atmospheric). Then the weight W of the vessel is

$$W = 4\pi r^2 t \rho = (3/2) p_0 v_0 S (\rho/Y).$$

Hence the weight is independent of the pressure of the gas contained. A cylindrical vessel is much easier to manufacture than a spherical one. However, when the thick-wall pressure formula below is applied (instead of the thin-wall formula above) to a practical cylinder with ends, the design problem for the minimisation of weight becomes somewhat of an art.² A (conservative) safety factor of 5 was used, and the vessel designed for 50 c.c. internal volume. The usual thick-wall formula² for the yield pressure p_{\max} of an elastic cylinder of inside radius r_i and outside radius r_o , and of yield tensile stress σ is

$$\sigma = p_{\max} (r_i^2 + r_o^2) / (r_o^2 - r_i^2).$$

The yield stress σ for the ends was computed using the formula² for a uniformly stressed plate of radius r_i , of thickness t , held at the edges:

$$\sigma = 3 p_{\max} r_i^2 / 4 t^2$$

A material with a low ρ/Y is precipitation-hardened beryllium copper. Because of its high density, for the amount of gas used the vessel walls would have been too thin for safe machining.

Steel has a low ρ/Y , and indeed a suitable vessel is available commercially,³ but it has the disadvantage of becoming brittle under the low temperature conditions (70°K) to be described later. So the final choice was the aluminium alloy 7075-T6, of tensile strength 5,300 atm. and density 2.8 g/c.c.⁴ The weighing vessel (see Fig. 1) was machined

from a block of 7075-T6 aluminium alloy, which cannot be welded nor soldered. The vessel was closed with a screwed brass plug. Because the vessel had to be cooled and evacuated at liquid nitrogen temperatures (when reclaiming the gas from the calorimeter after an experiment), it was necessary to devise a seal to be both vacuum- and pressure-tight at room and low temperatures. The following materials formed a vacuum seal in the screw-threads between room temperature and 77°K, but leaked under pressure: (a) epoxy casting resin ("Stycast" 2850 GT: Emerson and Cuming), (b) 80% aluminium-filled epoxy resin ("F": Devcon), (c) silicone rubber (Silastic No. 732 RTV: Dow Corning). A successful seal was made by winding two layers of PTFE tape (plumber's "Teflon Dope") onto the pipe thread: this was leak-tight after thermal cycling at 140 atm. pressure of helium gas and under vacuum at both room and low temperatures. A helium mass spectrometer leak detector was used for all tests. At 245 atm. water pressure, the outside diameter of the cylinder (measured at the middle of the cylinder) had expanded 0.14% above the zero-pressure value. The completed cylinder was anodised to prevent changes in its weight due to oxidation.

GAS HANDLING SYSTEM AND USE

A simplified diagram of the complete equipment is shown in Fig. 2. The weighing vessel was connected to the rest of the apparatus by stainless steel tubing (type 321, 1/8" o.d. x .020" wall) hard-soldered into the brass plug, a light-weight valve and a connector. A small aluminium flange (not shown) was attached to the tubing with "Stycast" 2850 GT epoxy resin to make a vacuum-tight seal to a dewar surrounding the weighing vessel. The entire apparatus was evacuated and filled with "Research Grade" gas of better than 99.99% purity, from the supplier's

cylinder. A small diffusion pump evacuated the system considerably faster than did a mechanical pump. This operation was repeated several times until the percentage of remaining air was estimated to be substantially less than the percentage impurities in the gas as supplied. The weighing vessel was then filled with an amount of gas sufficient to fill the calorimeter (as estimated from the pressure gauge and the approximate volume of the weighing vessel). The full weighing vessel was then removed from the apparatus, its valve leak-tested, and weighed on a standard laboratory beam-balance of 2 kg. capacity. It was returned to the apparatus, the connection-space purged of air as above, and its contents slowly condensed into the cold calorimeter as liquid. Argon and krypton are difficult substances to condense into a calorimeter: because the liquid range from the triple point to one atmosphere pressure is only 3.5°K for argon, and 4.0°K for krypton, it is very easy to block the calorimeter filling-tube. The filling pressure is limited to less than 2 atm. by the strength of the fragile calorimeter. Hence it is convenient to have an automatic pressure regulator to allow the gas to condense at reasonable rates at pressures from 0 to 2 atm. The pressure regulator was a "National" 100 atm. to 4 atm. two-stage gas regulator valve, with the final stage simply modified to deliver at below atmospheric pressure. This regulator is more convenient and simpler than the cartesian diver device, employing mercury, which is sometimes used as a gas flow regulator. When all the gas had been condensed into the calorimeter, the temperature was lowered to freeze the liquid. The resulting pressure was low enough that the amount of gas left in the capillary tubing, etc., of the system was negligible. After the specific heat measurements were finished, the weighing vessel was surrounded with a

bath of liquid nitrogen at reduced pressure (not shown in diagram). The calorimeter was then slowly warmed up and the gas distilled over into the weighing vessel (by-passing the reducing valve). A glass-covered electrical heating tape, wound round the 1/8" stainless steel tube, was useful (particularly for krypton) in preventing blockages at this point. When all the gas had been condensed over as solid, the amount of gas remaining was again negligible in quantity. The pressure vessel valves were then closed, and the vessel left overnight to warm up to room temperature, where a check weighing could be made. Alternatively, the gas could be released through the pressure reducing valve into large metal containers (not shown) at a couple of atmospheres for storage. A spectrographic analysis* of the gases after the experiment showed that the impurities introduced by the gas-handling system were less than 100 p.p.m.: most of this was water, probably absorbed on metal surfaces. The evacuated pressure vessel, with valve, connector and aluminium vacuum flange, was readily weighed to within ± 0.005 g. in 1600 g. (For illustration, the actual weights are rounded off.) With an approximately 2 gram-mole sample of argon, the pressure vessel weighed 1680 ± 0.005 g. Hence the sample weighed 80 ± 0.01 g. well within the 0.1% accuracy required. The krypton sample weighed some 180 g. The weighing vessel was not quite optimal for these quantities of gas, and could have been somewhat lighter.

* I am grateful to Dr. Amos Newton, of the Lawrence Radiation Laboratory, Berkeley, for performing the analysis.

ACKNOWLEDGEMENTS

I wish to thank Mr. Gerd Behrsing for his excellent mechanical designing and development of the seal of the weighing vessel, and Professor Norman E. Phillips, in whose laboratory this work was done. This paper was written during a University of Colorado Faculty Research Fellowship.

This work was performed under the auspices of the United States Atomic Energy Commission.

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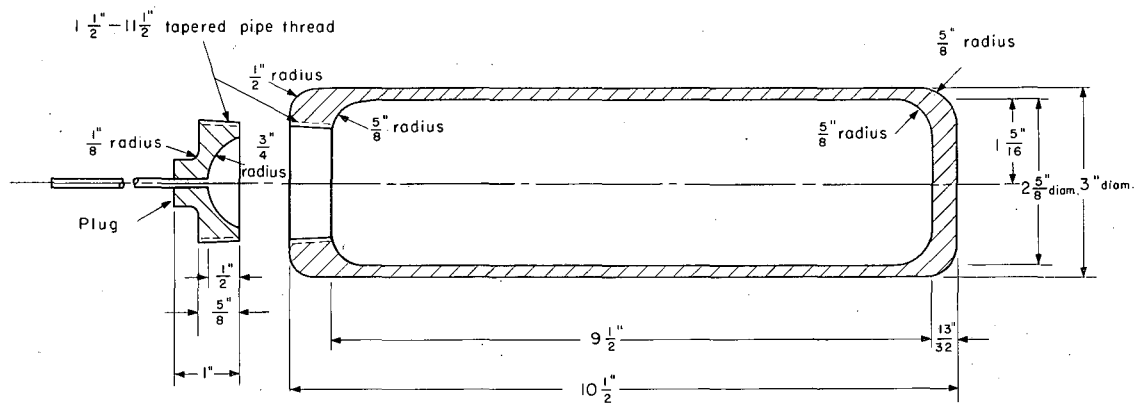
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FIGURE CAPTIONS

Fig. 1 Pressure vessel - longitudinal section, exploded view.

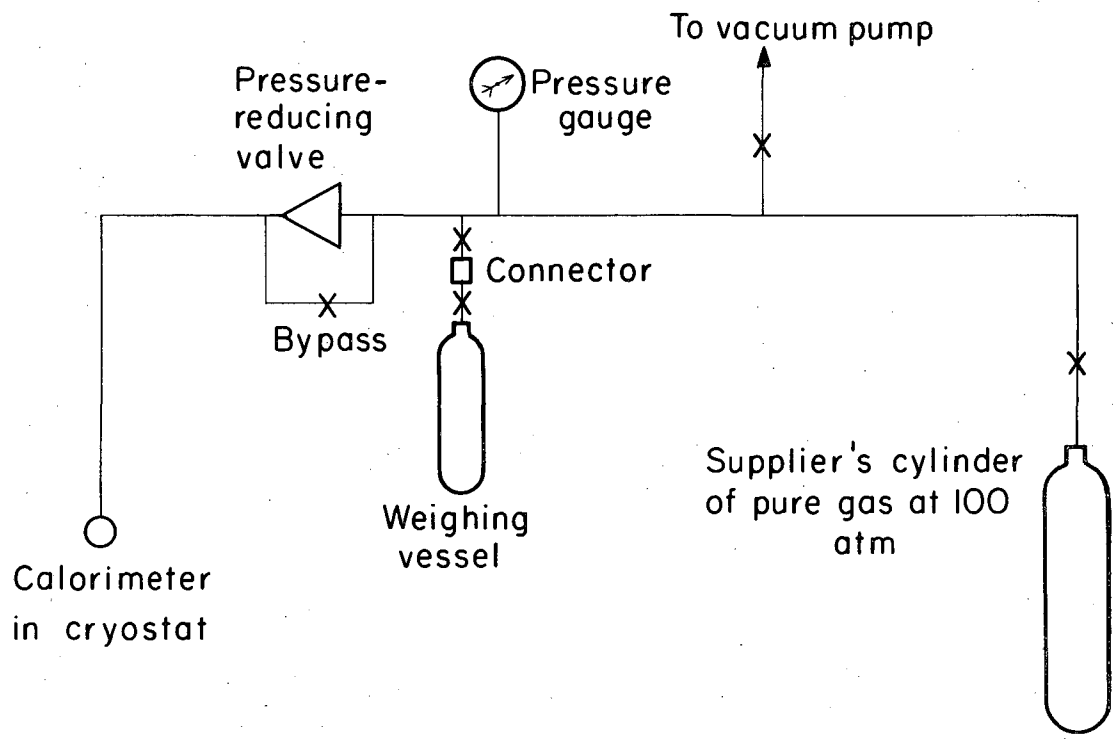
Dimensions are in inches.

Fig. 2 Gas handling system.



MUB 12055

Fig. 1



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Fig. 2

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