Reprinted from THE REVIEW OF SCIENTIFIC INSTRUMENTS, Vol. 37, No. 1, 51–54, January, 1966
Printed in U. S. A.

Density Balance for Low Temperatures and Elevated Pressures*

ANTHONY F. GRIGOR AND WILLIAM A. STEELE
Whitmore Laboratory, Department of Chemistry, Pennsylvania State University, University Park, Pennsylvania 16802
(Received 1 September 1965)

An electromagnetic balance for the measurement of fluid density at low temperature and elevated pressure is described. The balance is operated by balancing the changes in gravitational forces due to the buoyancy of a sphere immersed in the fluid against the electromagnetic force between a current carrying coil and a permanent magnet. The apparatus, which requires a relatively small amount of fluid, is capable of high precision and can be operated at any temperature below ambient and at pressures up to 80 atm.

I. INTRODUCTION

ACCURATE *PVT* measurements on simple liquids over wide ranges of temperature and pressure are of considerable interest, theoretically as well as practically. The purpose of this work was to construct an apparatus capable of measuring the density of a liquid such as methane to an accuracy of 0.02% over a range of temperatures from the melting point to the critical region and at pressures up to 80 atm. In this way, isothermal compressibilities could be measured as well as molar volumes along the gas-liquid saturation line, and values of these properties in the neighborhood of the critical point could be obtained. It was also considered desirable to be able to work with relatively small quantities of liquid so that expensive (such as deuteromethanes) or hazardous (such as silanes) materials could be conveniently studied. Therefore, a density balance was constructed in which the apparent weight of a sphere immersed in the liquid could be measured by varying the current through a coil mounted on the balance beam and moving in the field of a permanent magnet. Although the use of electromagnetic balancing in the measurement of liquid density,[1,2] magnetic susceptibility,[3,4] and adsorption[5,6] is well-known, the requirements of high accuracy, elevated pressures, and low temperatures gave rise to a number of interesting features in the present apparatus. Furthermore the amount of liquid required to immerse the sphere could be made quite small by reducing the size of the chamber which enclosed it. In fact, by holding all volumes to a minimum, the apparatus required less than 3 cc of liquid for successful operation, compared to a value of 37 cc in a typical conventionally designed liquid density apparatus of comparable accuracy which was constructed by van Itterbeeck and co-workers.[7,8]

II. APPARATUS

A schematic diagram of the balance is shown in Fig. 1. A magnesium sphere (1) weighing 20.6563 g is suspended from the balance beam by a fine stainless steel wire. The substance to be measured is condensed into a spherical annulus with a volume of 2.6 cc, which is formed between the sphere and the cavity in a heavy copper block (2). The bottom half of the block contains a well (3) for a platinum resistance thermometer (calibrated by the National Bureau of Standards) and a heater is wound on the top half for changing the temperature. The sample cavity

* This research sponsored by the U. S. Air Force Office of Scientific Research, Office of Aerospace Research, Grant No. AF-AFOSR-118-63.

1 J. W. Beams and A. M. Clarke, Rev. Sci. Instr. **33**, 750 (1962).
2 C. W. Hargens, Rev. Sci. Instr. **28**, 921 (1957).
3 J. R. Singer, Rev. Sci. Instr. **30**, 1123 (1959).
4 T. R. McGuire and C. T. Lane, Rev. Sci. Instr. **20**, 489 (1949).
5 J. W. Beams, C. W. Hurlburt, W. E. Lotz, and R. M. Montague, Rev. Sci. Instr. **26**, 1181 (1955).
6 P. Cannon, Rev. Sci. Instr. **29**, 1115 (1958).
7 A. van Itterbeeck and O. Verbeke, Cryogenics **1**, 77 (1960).
8 A. van Itterbeeck, O. Verbeke, and K. Staes, Physica **29**, 742 (1963).

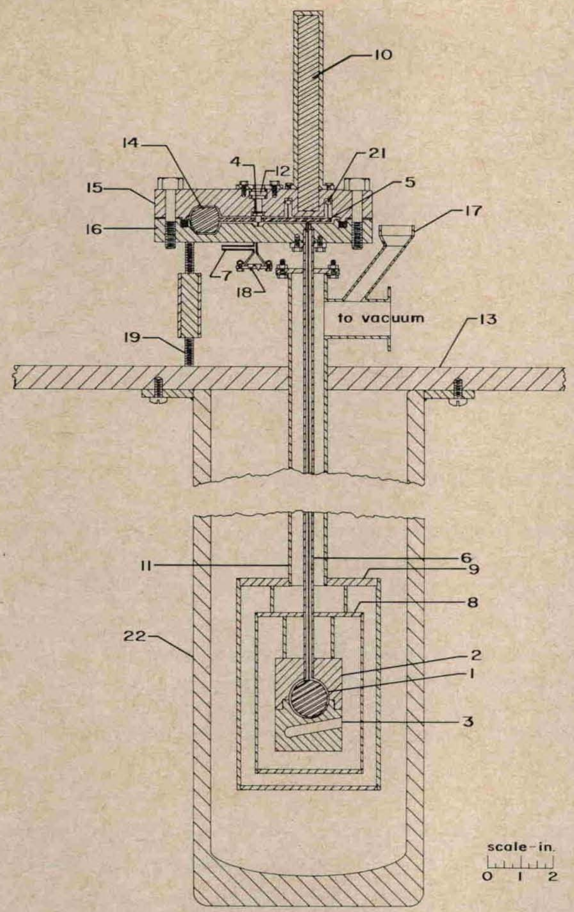

FIG. 1. A schematic diagram of the density balance is shown here. A portion of the tubing has been omitted, as indicated by the wavy lines; the apparatus above these lines is at room temperature, and that below the lines is submerged in the refrigerant. The parts shown here and in Fig. 2 include: 1—magnesium sphere suspended from the balance arm; 2—heavy copper block which holds the sample; 3—thermometer well; 4—mirror; 5—balance beam; 6—inlet tube from balance case to block; 7—tube connecting balance case to high pressure gas handling system; 8—thermal shield; 9—vacuum can; 10—bar magnet; 11—pumping tube for can; 12—glass plate for viewing mirror; 13—brass mounting plate; 14—silver counterweight; 15, 16—top and bottom sections of balance case; 17, 18—take-outs for leads from low temperature and from high pressure, respectively; 19—support screws for balance case; 20—mount for jewels; 21— coil; and 22—metal Dewar.

is connected to the room temperature portion of the apparatus by a 50 cm tube (6). The temperature control of the block is essentially that used in precision calorimetry; that is, it is enclosed by an electrically heated shield (8) located in the evacuated can (9) which provides thermal insulation between the block and shield, and the refrigerant held in the metal Dewar (22). The temperatures of the shield and the inlet tube (which could also be heated) were monitored by copper-constantan thermocouples which could be operated absolutely or differentially relative to a thermocouple on the copper block. The electrical leads went up the pumping tube (11) for the can and out a take-out cap (17) to the current sources and to a Leeds and Northrup type K-3 potentiometer, which was used to measure thermocouple emf and thermometer resistance. In order to ensure that the block was rigidly fixed relative to the room temperature portion of the apparatus, the inlet tube and pumping tube were made of thick wall stainless steel, and the shield-to-can and block-to-shield supports were stainless steel rods. The entire assembly was attached to a heavy brass plate (13) resting on levelling screws which allowed one to accurately center the magnesium sphere in the cavity in the block.

At its upper end, the suspension wire for the sphere was attached to one end of a brass bar 8 cm long and 3 mm square which served as the balance beam (5). The beam, which is shown in detail in Fig. 2, had a movable silver counterweight (14) on one end and a coil (21) of approximately 300 turns of No. 40 copper wire mounted concentric with the suspension wire. The beam was pivoted on two conical jewels mounted in the cross piece (20). The jewels rested on stainless steel pins. The position of the beam was observed optically by means of a mirror (4) mounted at the center of the beam and viewed through a thick glass plate (12) held by O-rings in the top section of the balance case (15). A permanent bar magnet (10), obtained from the Crucible Steel Company, was also fixed to this part of the balance case, outside of the high pressure volume of the system. This magnet was concentric to the coil mounted on the balance beam, and was located vertically so that the magnetic field was a maximum at the position of the coil when the beam was balanced. Under these conditions, the coil was in a field of $\sim$550 G which hardly varied over its vertical travel, which only amounted to 2 mm. Current leads to the coil led into mercury droplets in cavities in the bottom section of the balance case (16) so as to minimize damping of the beam motion. The leads continued from the droplets through a high pressure take-out cap (brass cones fixed with Araldite in a brass plate) to batteries and variable resistors which supplied the current to the coil. This current was accurately measured by determining the voltage drop across a 10 Ω standard resistor. The balance case was composed of two circular brass plates 17 cm in diameter in which cavities were milled to hold the balance beam, counterweight, and coil. The upper and lower plates were bolted together with an O-ring seal between them so that the top plate could be readily removed for adjustment of the beam. The bottom plate (16) was attached to the support plate for the apparatus (13) by means of three levelling screws [only one of which is shown (19)]; the inlet tube to the low temperature part of the apparatus was sealed to the bottom section of the balance case by O-rings, and the tube (7) connecting the

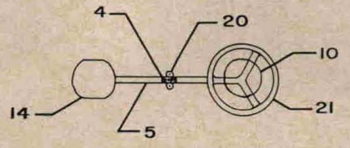

FIG. 2. A top view of the balance beam is shown here. The same numbering system is used as for Fig. 1.