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## Magnetic Suspension Balance Method for Determining Densities and Partial Specific Volumes\*

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(Received April 13, 1962)

Two modifications of the magnetic suspension balance are described for determining densities and partial specific volumes. Both modifications are capable of high precision. Also, they require comparatively small amounts of the materials and short times for the determinations.

IN order to determine molecular weights by the ultracentrifuge method, it is necessary to know the partial specific volume  $\bar{V}$  of the substance and the density  $\rho$  of the solution.<sup>1,2</sup> With modern equilibrium ultracentrifuging techniques,<sup>3</sup> usually the least precisely known factor which enters into the determination of the molecular weight is the partial specific volume. Aside from this, the partial specific volume itself is of considerable importance for properly characterizing a substance.<sup>1,4</sup> In the past, a number of methods have been used successfully for the measurement of  $\bar{V}$ . These methods essentially consist of measuring the density of the solution at a series of concentrations. The standard pycnometer technique is perhaps the most widely used but the magnetic float method originally described by Lamb and Lee,<sup>5</sup> and further developed by MacInnes and his collaborators,<sup>6</sup> is the most accurate. Unfortunately, both of the above methods require a comparatively large amount of the substance in solution when precision is required. Often the substances are difficult to obtain in large enough quantities and there is a need for methods which require smaller amounts of material for the measurements. In this paper, two variations of a magnetic balance<sup>7</sup> method are described in which the magnetically floated body is stationary and which use relatively small quantities of the substance for the measurements.

Figure 1 shows a schematic diagram of the first method. A ferromagnetic body  $W$  is surrounded by a transparent glass or plastic container  $C$ , which is filled to the level  $L$  with the solution whose density is to be determined. This container is surrounded by a circulating water or liquid bath which maintains the temperature constant to the order of 0.001°C. The float  $W$  is magnetically suspended freely in the solution by the air core solenoid  $S$ , situated

above the chamber  $C$ . The signal from the sensing coil  $P$  located outside and on the bottom of  $C$ , when applied to the electronic circuit, regulates the current through  $S$  in such a way as automatically to maintain  $W$  at a given vertical position. The axially symmetrical magnetic field of  $S$  holds  $W$  in the desired horizontal position. When properly adjusted, no motion of  $W$  can be observed in the microscope  $T$ . The force on  $W$  in the upward direction  $z$ , due to  $S$  is  $F = M dH/dz$ , where  $M$  is the magnetic moment of  $W$ . If  $W$  is made of a "soft" magnetic material,  $F = KH \partial H / \partial z = K_1 \cdot I^2$ , where  $K$  and  $K_1$  are constants of the apparatus,  $H$  is the magnetic field of  $S$ , and  $I$  is the current in  $S$ . A pair of Helmholtz coils  $H-H$  are so positioned that they produce a magnetic field at  $W$  in the  $z$  or vertical direction. Consequently, in addition to the force on  $W$  due to the magnetic support current, the Helmholtz coils superpose a steady field which from the relation  $F = KH \partial H / \partial z$  produces a force on  $W$  in the  $z$  direction. Small corrections for the magnetic susceptibility of the solution and cooling water, of course, must be made when the highest accuracy is required.

Before using this method, the apparatus must be calibrated by measuring the current through the solenoid when  $W$  is supported at a given height in different liquids with known densities. This height is determined by focusing the microscope  $T$  on a fine mark on  $W$  and is so chosen that  $W$  is well above the bottom of the container but still completely covered at the top. Pure water, sucrose solutions,

\* Supported by Navy Bureau of Weapons and Army Research Office (Durham).

<sup>1</sup> T. Svedberg and K. O. Pedersen, *The Ultracentrifuge* (Oxford University Press, New York, 1940).

<sup>2</sup> L. G. Longworth, *Proc. Natl. Acad. Sci.* **36**, 502 (1950).

<sup>3</sup> J. W. Beams, R. D. Boyle, and P. E. Hexner, *Rev. Sci. Instr.* **32**, 645 (1961).

<sup>4</sup> G. N. Lewis, and M. Randall, *Thermodynamics* (McGraw-Hill Book Company, Inc., New York, 1923).

<sup>5</sup> A. B. Lamb and R. E. Lee, *J. Am. Chem. Soc.* **35**, 1666 (1913).

<sup>6</sup> D. A. MacInnes and M. O. Dayhoff, *Rev. Sci. Instr.* **22**, 642 (1951); and *J. Am. Chem. Soc.* **74**, 1017 (1952).

<sup>7</sup> J. W. Beams, C. W. Hulburt, W. E. Lotz, Jr., and R. Montague, Jr., *Rev. Sci. Instr.* **26**, 1181 (1955).

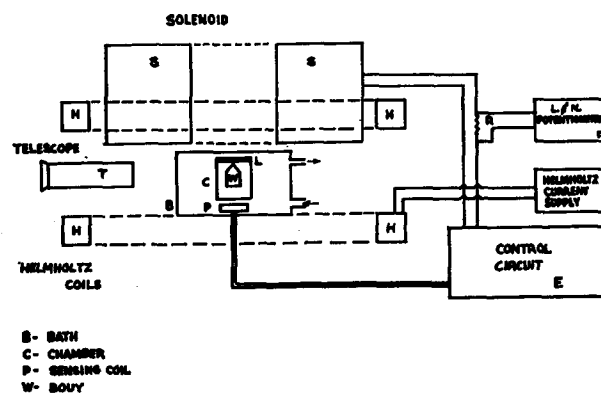


FIG. 1. Diagram of first magnetic balance method of determining densities.

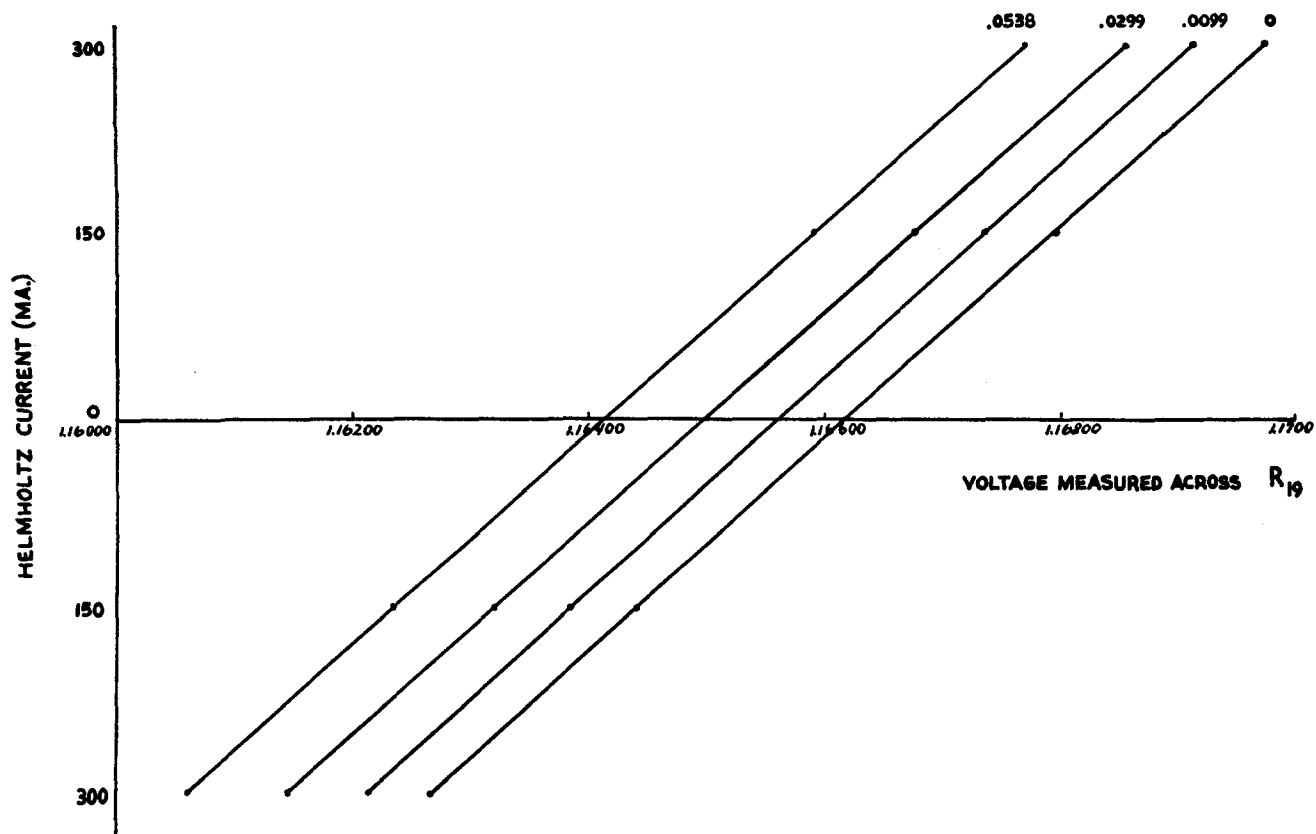


FIG. 2. Helmholtz current as a function of solenoid current (concentration by weight of aqueous solution of sucrose).

and NaCl solutions of known concentrations were used for the calibrations. In addition to the current through S required to support W at the given height in each of the solutions, curves of solenoid current versus Helmholtz current were taken. Typical curves are shown in Fig. 2. This procedure makes it possible to obtain better precision in determining the support currents and hence in the density measurements. After calibration the above procedure is carried out to determine the unknown densities of solutions at various known concentrations of the substance under investigation.

The solenoid is composed of three coils containing 40 000 turns of No. 25 AWG enameled copper wire with a total resistance of 2200  $\Omega$ . The coils are stacked one on top of the other to form a solenoid with inner diameter of 1.5 in., outer diameter of 9 in., and 4 in. high. The coils are separated by thin brass plates which are cooled by the circulating cooling liquid. The two Helmholtz coils are carefully wound on a plywood ring of 12 in. i.d. and 14 in. o.d. Each coil is made of 100 turns of No. 25 AWG enameled copper wire. Current through the coils is measured by calibrated ammeters. The current in the solenoid S is measured by a special potentiometer arrangement. The float W which weighed 0.0421 g, is a carefully machined cylinder of Permalloy covered with a thin coating of a

substance which protects it and prevents wetting of its surface by the solution. If necessary, it can be enclosed in a glass tube. Permalloy was used because it is magnetically soft. The container C surrounding W was made of glass and could be tightly sealed. In most of the experiments, it contained 0.2 cm<sup>3</sup> of liquid. This, in turn, was surrounded by a circulating water bath made of Lucite. Figure 3 shows the electronic support circuit. It is very similar to those previously described in detail.<sup>8,9</sup> The solenoid currents required in the experiments varied from 150 to 250 mA.

In most of the experiments, the densities of the solutions could be determined to 0.01% which gave a precision of about three places in the partial specific volumes. This accuracy could be increased if the need arises.

Figure 4 shows a schematic diagram of the second method used. W<sub>c</sub>, which contains the solvent filled to the level L and the ferromagnetic body W, is supported by a fine nonmagnetic wire from one arm of a sensitive balance B. W may be magnetically supported freely by the air core solenoid similar to that used in Fig. 1. The sensing coil P is separately mounted well below the bottom of W<sub>c</sub> and

<sup>8</sup> J. W. Beams, *Science* **120**, 619 (1954).

<sup>9</sup> J. W. Beams, J. B. Breazeale, and W. L. Bart, *Phys. Rev.* **100**, 1657 (1955).

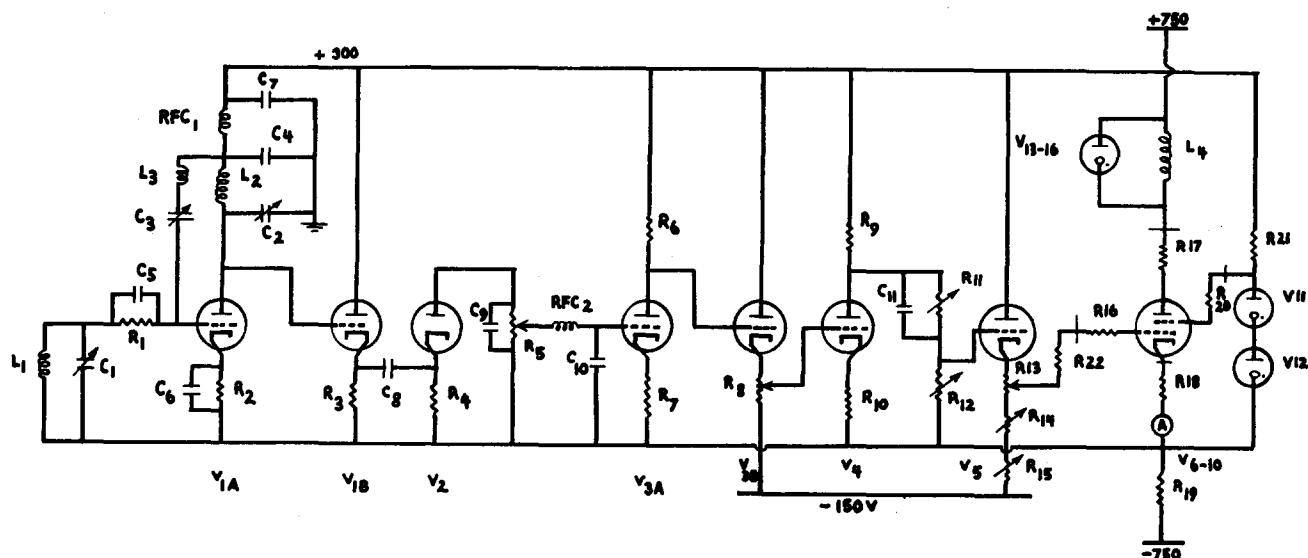


FIG. 3. Magnetic support control circuit.

$L_1$	25 T no. 32	(Q coil)	$C_{1,2}$	140 $\mu\mu\text{F}$	$R_1$	8.5 k $\Omega$	$R_{12}$	100 k $\Omega$	$V_1$	12AU7
$L_2$	30 T no. 16		$C_3$	25 $\mu\mu\text{F}$	$R_2$	2.7 k $\Omega$	$R_{13}$	75 k $\Omega$	$V_2$	6AL5
$L_3$	6 T no. 20		$C_{4,6,7,8,10}$	0.001 $\mu\mu\text{F}$	$R_3$	100 k $\Omega$	$R_{14}$	5 k $\Omega$	$V_3$	6SN7
	cold end of $L_2$		$C_5$	400 $\mu\mu\text{F}$	$R_4$	500 k $\Omega$	$R_{15}$	750 $\Omega$	$V_{4,5}$	6J5
RFC <sub>1,2</sub>	2.5mH		$C_8$	500 $\mu\mu\text{F}$	$R_5$	2 M $\Omega$	$R_{16,22}$	10 k $\Omega$	$V_{6-10}$	6L6 in parallel
$L_4$	40 000 T no. 25	(Solenoid)			$R_6$	150 k $\Omega$	$R_{17}$	33 $\Omega$	$V_{11,12}$	OC3
					$R_{7,10}$	1.5 k $\Omega$	$R_{18}$	500 $\Omega$	$V_{13-16}$	OD3 in series
					$R_8$	50 k $\Omega$	$R_{19}$	6.2 $\Omega$		
					$R_9$	47 k $\Omega$	$R_{20}$	33 $\Omega$		
					$R_{11}$	1.2M $\Omega$	$R_{21}$	3 k $\Omega$ (10 W)		

does not influence the weight of  $W_c$ . The procedure consists first in weighing the air tight plastic or glass container with the balance B.  $W$  is then placed in the container and another weighing made on the balance. The container is next filled to some level  $L$  with the pure solvent and again weighed on B. Next,  $W$  is freely suspended in the solvent and another weighing made with B. From these measurements, the density  $\rho$  and volume  $v$  and weight of the solvent may be determined if the volume  $E$  of  $W$  has been previously determined with a liquid of known density. A known amount of solute  $Q$  is next added to the container and

the weight recorded by the balance first with no current in the solenoid and  $W$  resting on the bottom of  $W_c$  and second with  $W$  suspended by the solenoid. From these two weighings the density of the solution  $\rho_1$  and the volume of the solution  $v + \Delta v$  may be obtained. If the weights recorded on B are  $X_1$ ,  $X_2$ ,  $X_3$ , and  $X_4$ , respectively, then

$$W_c + W + \rho v = X_1,$$

$$W_c + \rho E + \rho v = X_2,$$

$$W_c + W + \rho_1(v + \Delta v) = X_3,$$

$$W_c + \rho_1 E + \rho_1(v + \Delta v) = X_4.$$

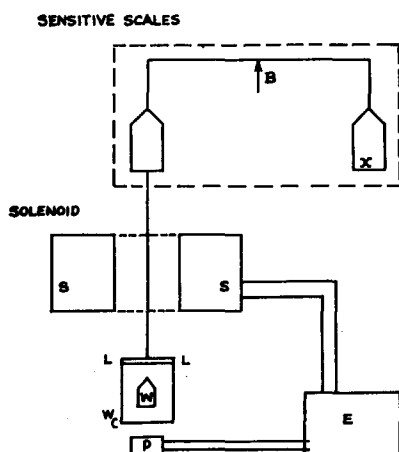


FIG. 4. Schematic diagram of second magnetic balance method.

A second known quantity of the solute  $Q_2$  is next added and the weighings repeated, etc. These data give the densities and  $\Delta v$ 's at various concentrations. From these values, the partial special volume  $\bar{V}$  and the apparent specific volumes  $\varphi$  can be determined.<sup>1,4,6</sup>

It will be observed that the precision of the method is determined by the precision with which the weighings on the balance B can be carried out and upon the volumes of  $W_c$  and  $W$ . Also, the magnetic suspension circuit may be much simpler than that shown in Fig. 1 since no current measurements need be made. In addition, a much smaller solenoid S with a core<sup>8,9</sup> may be used provided the core loses its magnetism when the current is turned off. We have

used Permalloy powder cores successfully. The float W need not be made of magnetically soft material. We have used magnetic stainless steel covered with different coatings such as Teflon for most of the measurements. Furthermore, it is not necessary to make up separate solutions of known concentrations as in the first method. The second method has been used to determine the partial specific volumes of sucrose and a number of salts and the results obtained are in good agreement with those found by others.<sup>1,6,10</sup> Most of the values obtained were reliable to between 1.0 and 0.1%. In the present apparatus, slowly circulating air is used to hold the temperature constant. This is possible because the measurements can be made in a few minutes. However, liquid cooling could, of course, be used if necessary. The principal source of error in both of the above methods is the possible adsorption of solute on the magnetically suspended body W. This phenomenon

<sup>10</sup> H. S. Harned and B. B. Owen, *Physical Chemistry of Electrolytic Solutions* (Reinhold Publishing Corporation, New York, 1950).

has been investigated by Hulburt,<sup>11</sup> who has shown that it is small if the proper coatings are placed on W. One important advantage of the above methods is that W does not move with respect to the walls of the container. This eliminates the effect of viscosity as well as any wall effect and makes possible the use of very small volumes of the material. The extent of the vertical movement of W can be roughly estimated from the fact that with a well-regulated servo circuit of Fig. 3 the gravitational field is balanced by the magnetic field to about one part in  $10^6$ . Since the reaction time of the circuit is the order of  $10^{-3}$  sec, W will fall a distance  $d = \frac{1}{2} at^2 = \frac{1}{2} 10^{-12} g \sim 10^{-9}$  cm which is the order of an atomic diameter. At the same time, when it is desired to stir the solution, or remove the air bubbles from the surface of the float, W can be "bounced" up and down in the solution to accomplish this.

We are much indebted to Dr. C. W. Hulburt for his help during the initial stages of this work.

<sup>11</sup> C. W. Hulburt, Dissertation, University of Virginia (1956).

## Reflecting Variable Bent Crystal Spectrometer\*

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(Received February 16, 1962)

A reflecting variable bent crystal spectrometer is described which in some forms varies between Johann and Johansson arrangements. The variable bent crystal and its spectrometer arrangement differ from the Harker concept. The variable control features of this spectrometer permit compensation for tolerances, application, and aging. As a result this combination of events has already resulted in achieving experimental signal-to-noise ratios as high as 3100 to 1 using a mica crystal on pure silicon. Other features permit direct comparison of relative x-ray intensities without diffraction volume correction.

### 1. INTRODUCTION

IN order to satisfy conditions of high sensitivity and variable discrimination in x-ray microanalysis the bombarding electron beam must, first, be of adequate intensity for rapid and accurate measurement of characteristic emissions, and secondly, be capable of high and low discrimination. The need of high discrimination arises from the fact that the minimum detectable concentration is lowered as the discrimination of the spectrometer increases due to improved signal-to-noise ratio. Line separation in the L series, for example, is enhanced. On the other hand, for raster scanning x-ray microanalysis one wishes a low

Q spectrometer for large area scan as one starts to go off the Rowland Circle.

### 2. BACKGROUND

Liebhafsky<sup>1</sup> shows the two typical reflecting arrangements, namely, (Johann—Fig. 1) where the crystal grating has a radius of  $R$  and the inner surface of the grating must lie on a circle  $R/2$ , the focal circle; and Johansson's arrangement (Fig. 2) where the crystal is bent to  $R$  and ground to  $R/2$  so that the x rays make the same angle with the crystal planes as in the Johann arrangement, but the grinding has brought these planes to lie on the focal

\* Presented November 3, 1961 at the Nineteenth Annual Pittsburgh Diffraction Conference.

<sup>1</sup> H. A. Liebhafsky, H. G. Pfeiffer, E. H. Winslow, and P. D. Zeman, *X-Ray Absorption and Emission in Analytical Chemistry* (John Wiley & Sons, Inc., New York, 1960).