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Citation: Review of Scientific Instruments **25**, 295 (1954); doi: 10.1063/1.1771046
View online: https://doi.org/10.1063/1.1771046
View Table of Contents: http://aip.scitation.org/toc/rsi/25/3
Published by the American Institute of Physics

Laboratory and Shop Notes

Interferometer for the Measurement of Sedimentation in a Centrifuge*

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(Received August 31, 1953)

In both the rate of sedimentation and the sedimentation equilibrium methods of measuring molecular weights with the ultracentrifuge, the rotor speed, the rotor temperature, and the refractive index at various radial distances in the ultracentrifuge cell are usually measured while the centrifuge is spinning.[1] The development of the magnetically suspended vacuum-type ultracentrifuge[2] makes possible the determination and maintenance of the rotor speed and temperature with much greater reliability than the index of refraction in the cell so it has become important to adapt more precise methods to the latter measurement. The purpose of this note is to describe a modified Jamin interferometer method of determining this refractive index which not only gives improved precision at various radial distances but also at all points throughout the ultracentrifuge cell. This type of interferometer was adapted to this experiment after the development of the double ultracentrifuge cell[2,3] and after some other types[4] were found to be difficult to adjust.

Figure 1 illustrates the principle of the interferometer and Fig. 2 shows a schematic diagram of the apparatus used. In Fig. 1 light from a mercury or sodium arc A is made monochromatic by the filter F and parallel by the lens L_1. The diaphragm S limits the width of the parallel beam which falls upon the two accurately parallel interferometer plates I_1. The first plate is partially reflecting; i.e., it reflects less than half of the light and transmits the remainder. The second plate reflects practically all of the light striking it so that the two beams are formed as shown. The two beams first pass through a compensating cell K_2, outside the centrifuge, the purpose of which will be described later, and then into the double cell K_1 mounted in the ultracentrifuge rotor. The cell K_1 consists of two identical sector-shaped cells situated side by side in the same housing and having the same quartz windows.[3] One of the sectors contains the solvent and the other

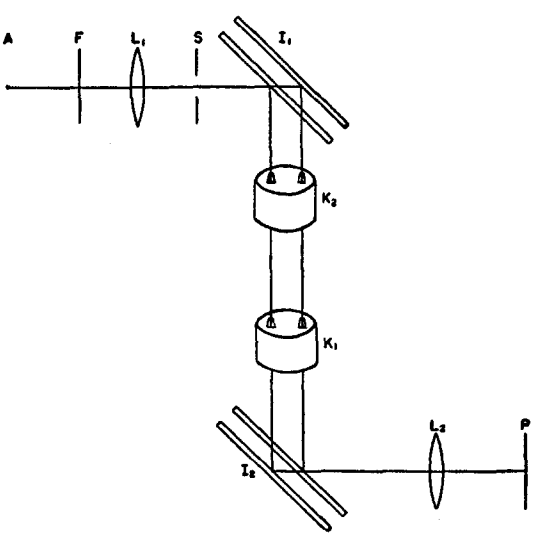

FIG. 1. Schematic diagram of simple Jamin-type interferometer.

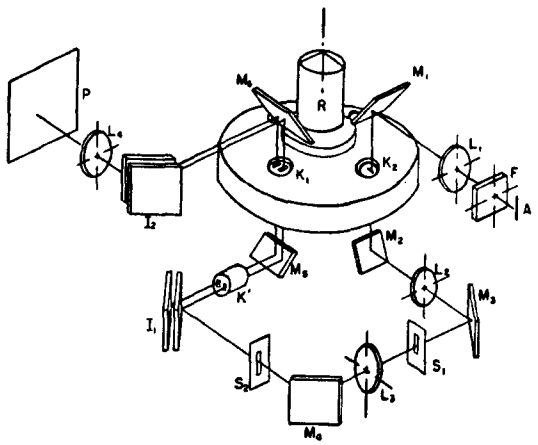

FIG. 2. Combination light chopper and interferometer.

the solutions in which sedimentation is measured. One of the interferometer light beams passes through the sector containing the solvent and the other through the solution. The two beams are then recombined by the second pair of interferometer plates, I_2, and the resulting fringes are focused on the photographic plate P by the lens L_2. If the two optically flat plates in I_1 and in I_2 are parallel to each other, respectively, the fringes will be at infinity. However, it is desirable in practice to form a sharp image of both the fringes and the ultracentrifuge cell K_1 on the plate P. This was accomplished by the well-known device of rotating one of the plates of I_2 at a small angle with respect to the other. The general theory of the Mach-Zehnder interferometer which is applicable to the interferometer here described has recently been given by Bennett and Kahl.[5] The interferometer described above may be considered to be either of the Jamin or Mach-Zehnder type.

Figure 3 shows the mounting of the adjustable interferometer plates I_2. The front surface "full silvered" optical flat is mounted in a heavy brass frame supported by leveling screws. The beam splitter also was an optical flat but "lightly silvered" to reflect about 30 percent and transmit 70 percent of the light. The reflecting surfaces of the two plates were separated as shown in Fig. 3 by two $\frac{1}{8}$-in. selected precision ball bearings and a screw adjustment. The screw adjustment was used to rotate the two plates. The interferometer element I_1 was identical to I_2 except that two additonal ball bearings replaced the adjustable screw. The use of ball bearings for spacers of Jamin interferometer plates has been described by Kuhn and Wheatley.[6]

Figure 2 shows the combination "light chopper" and interferometer used in the sedimentation experiments. Light from a capillary mercury arc A passes through an interference filter which removes all of the light except the green line and is focused by the lens L_1 on a narrow radial slit mounted in the centrifuge rotor[2] R at K_2 so that light passes only when K_2 is in the position shown. The light is then focused on a second narrow slit, S_1, and made parallel by the lens L_3. The width of the beam is limited by the slit diaphragm S_2, of about the same width as the sector-shaped cells in the centrifuge cell K_1. The beam is then split by the interferometer I_1 and passes through K^1 and K_1. The lens L_4 focuses the centrifuge cell K_1 on the photographic plate P. The wedge between the plates of I_2 is then adjusted to give clear interferometer fringes over the whole image of the cell K_1. The centrifuge rotor R, spins in a vacuum chamber so the mirrors M_1, M_2, M_5, and M_6 are used to send the light through the chamber. Since one of the light beams passes through the solution in K_1 and the other through the solvent, the two optical paths are not the same. Consequently, in order to get white light fringes for reference, a cell K^1, identical to K_1, is placed so that the

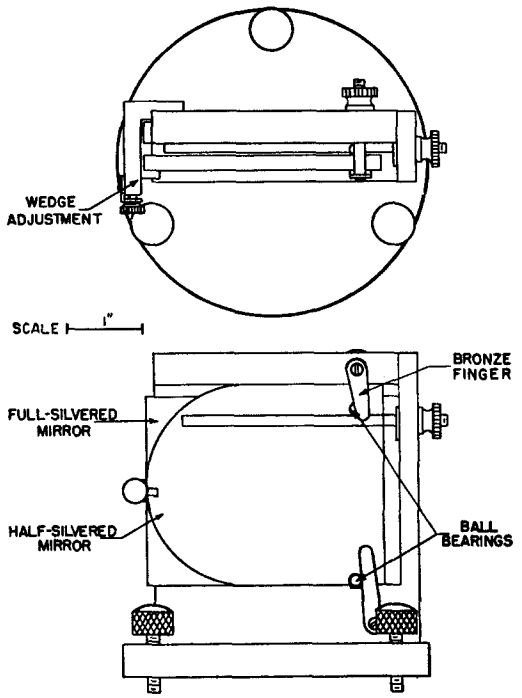

FIG. 3. Mounting of adjustable interferometer plates.

beam which passes through the solution in K_1 passes through the solvent in K^1, while in the other beam this order is reversed. In this way white light reference fringes are obtained before the centrifuge is started. During the sedimentation, white light reference fringes are obtained only for a narrow radial region in the cell where the concentration in the sector of K_1, which contains the solution, is approximately the same as at the beginning of the experiment.

At the beginning of the experiment the fringes are usually made parallel or perpendicular to the radial direction in K_1 and photographed both in monocromatic light and in white light. For white light an incandescent filament usually is placed in the position of A instead of the capillary arc, although generally it is only necessary to remove the filter F. When the centrifuge is spinning, light can pass S_1 once each revolution and only while the rotor turns through a very small angle, which is determined by the width of the slit in K_2, the optical properties and position of the lens L_2, and the width of the slit S_1. This angle is about 0.003 radian in these experiments. As a result, the fringes are sharp over the entire image of K_1 and the refractive index (and hence the concentration) can be determined perpendicular to the radius as well as parallel to the radius in K_1. It is for this latter reason that the Jamin type rather than the Rayleigh type of interferometer has been used in these experiments. Clearly the Rayleigh-type interferometer could be used with the double cell K_1 instead of the Jamin when desirable. The change in concentration ΔC due to a shift of one fringe is $\Delta C=\Delta n/k=\lambda/kt$ where n is the index of refraction, λ is the wavelength of the monochromatic light, k is the specific index of refraction increment, and t is the thickness of the cell K_1. It is clear that if one of the cells in the double cell distorts more or less than the other in the centrifugal field, the fringes also will move. However, the two cells have the same windows and housing and practically all of the distortions are compensated.[3] In practice a shift of 0.03 of a fringe can be measured. In Fig. 2, K_1 and K_2 are on rotor radii which make a right angle with each other. However, we have used the system with minor modifications when the angle is 180°.

The mirrors 1, 2, 3, 4, 5, and 6 were used as shown in Fig. 2 because of the geometry of the particular ultracentrifuge employed. Also, if a suitable capillary arc is not available, it may be replaced by any type of intense monochromatic source properly focused on a slit at A. Furthermore, if a nonsedimenting solute or substance were added to the solvent in the comparison cell in the proper amount to give white light fringes, the cell K^1 could be omitted. However, no sufficiently nonsedimenting solute has yet been found. The above interferometer may be used either in the rate of sedimentation or in the sedimentation equilibrium centrifuge methods of measuring molecular weights, but is especially adapted to the latter.

* Supported by U. S. Navy Bureau of Ordinance contract.
† U. S. Rubber Company fellow.
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A Simple Electrical Control For Automatic Toepler Pumps

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(Received October 16, 1953)

ALTHOUGH electrically operated automatic Toepler assemblies are commercially available, the construction and repair of such pumps generally necessitates the aid of a skilled glass blower. Of the electrical control units described in the literature, those that will operate over a wide range of pressures are somewhat complex,[1,2] and are not readily adaptable to existing Toepler pumps of the type represented by Fig. 1. Assembly of the Toepler pump and control system described below was accomplished cheaply and easily by research workers having only moderate glass-blowing skill. This control unit may easily be added to existing Toepler pumps.

The system herein described consists of a Toepler pump and a control mechanism, arranged as indicated in Fig. 1. The details of the control mechanism are shown in Fig. 2, and the electrical circuit is diagrammed in Fig. 3. The air leak is opened and closed by the action of a standard spring and solenoid combination. When the air leak is open the mercury falls in A and when it is closed the action of the mechanical pump (which is operated continuously) causes the mercury to rise in A.

To place the Toepler pump in operation, one turns on the mechanical vacuum pump and closes switches T_1 and T_2 (Fig. 3). Closing switch T_2 holds solenoid S_1 closed. When the mercury has drained out of the Toepler piston, the gas to be measured may be admitted into the Toepler pump. Switch T_2 is then opened. The spring tension of S_1 must be so adjusted that S_1 now opens the air leak, allowing the mercury to fall in reservoir A. When the mercury falls to a level where it no longer makes contact with electrode E_2, solenoid S_1 will close the air leak and keep it closed until the mercury has risen to a level where the mercury makes contact with electrode E_1. When the mercury makes contact with E_1, solenoid S_1 again opens the air leak. Thus the mercury in the reservoir is lowered and raised cyclically between levels L_1 and L_2. Levels L_1 and L_2 correspond to a point just below the tip of long electrode E_2 and a point just above the tip of short electrode E_1.

The rise and fall of mercury in the reservoir causes a corresponding fall and rise of mercury in the piston P of the Toepler