A Tubular Vacuum-Type Centrifuge

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longer spectral region may be covered by taking a continuous record with two complete revolutions of the drum. Thus in C the record starts at 100μ at 1,1 on the left and reaches 150μ at 2,2, starting the second rotation of the drum at 3,3 and attaining 200μ at 4,4. This record was made to locate the blaze of a grating having 87 lines per inch. The spectrum is not pure but satisfactorily locates the blaze at about 150μ.

These records have been chosen to show something of the flexibility of the instrument and the character and quality of its recordings. It has become customary to give the apparatus no attention after being started on a record; the experimenter return at the close of one records and starts another. The three records of B would require about 5 hours for the recordings alone. The time for changes in adjustments, the introduction of gases, etc., varies widely with circumstances. The 25 or more lines of the record E can be completely measured and computed in about $2\frac{1}{2}$ hours or during the time another similar record is being recorded.

The authors are indebted to Mr. H. M. Foley for running record D, which was included among the records recently published, and to Mr. Nelson Fuson for all of the others. They wish to express their appreciation also for the exceptional quality of the work done on this spectrograph by the staff of the instrument shop.

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A method of spinning tubular rotors to high speeds in a vacuum is described. The rotors are spun by air-supported, air-driven turbines situated below the vacuum chamber. The maximum rotational speed attainable is set only by the bursting strength of the tubular rotor. The material to be centrifuged enters the spinning tube at the top at a continuous rate and is collected in light and heavy fractions at the bottom. The apparatus has been used for centrifuging materials in gaseous, vapor and liquid states.

THE vacuum-type air-driven ultracentrifuge[1, 2, 3, 4] has been found to be very efficient because of the absence of stirring or remixing of the materials during the centrifuging. Also the maximum centrifugal force attainable is set only by the bursting strength of the rotor. However, the quantity of material which can be centrifuged per unit of time is somewhat limited because of the size and shape of the rotors. This limitation results from two causes; first, for dynamical reasons the rotors are never made much longer than their diameters, or rather the moment of inertia about the axis of rotation is always greater than that about a perpendicular axis which limits the usable capacity of the centrifuge, and second, the material does not pass through the centrifuge continuously but the rotor must be stopped and removed from the vacuum chamber for emptying and refilling. In order to overcome these handicaps the vacuum-type tubular air-driven centrifuge[5] has been developed. In this apparatus the material to be centrifuged enters at the top and is collected in two fractions at the bottom of the spinning tube. Also improvements in the air cushion support make it possible to support very heavy rotors so that the length of the spinning tube and hence the rate of centrifuging may be made comparatively large. These tubular centrifuges have been run successfully with the air support and drive either above or below the spinning tube but in this paper details will be given only for the case where they are below.

[1] Beams and Pickels, R. S. I. **6**, 299 (1935).
[2] Bauer and Pickels, J. Exp. Med. **64**, 503 (1936); **65**, 565 (1937).
[3] Wyckoff and Lagsdin, R. S. I. **8**, 74 (1937).
[4] Beams, J. App. Phys. **8**, 795 (1937). Beams, Linke and Sommer, R. S. I. **9**, 248 (1938).
[5] Beams, Linke and Skarstrom, Science **86**, 293 (1937).

Separations of material in the liquid, gaseous and vapor states have been carried out with essentially the same design of machine. The only differences were the details of the internal construction of the spinning tube.

Figure 1 shows a scale drawing and Figs. 2 and 3 photographs of the apparatus used for the separation of gases and vapors. The rotating parts consist of the air-driven, air-supported turbine T, the tubular rotor R and the flexible tubular shafts S_1, S_2 and S_3. S_1 passes through the vacuum-tight oil gland G_1, S_2 and S_3, pass through the vacuum-tight oil glands G_2 and G_3 while S_2 passes through the vacuum-tight oil gland G_4. G_1 and G_2 seal the vacuum chamber V while G_3 and G_4 make it possible to separate the fraction of the material flowing out through S_2 from that flowing out between the coaxial tubes S_2 and S_3. S_1, S_2 and S_3 are stainless steel hypodermic needle tubing 0.109″, 0.075″ and 0.125″ outside diameters, respectively. An enlarged cross-sectional drawing giving the design of the vacuum-tight oil glands which are all alike except for dimensions is shown in Fig. 1(b). A rod of brass or Duralumin is machined to the shape of Q. A channel is bored along its axis about $\frac{1}{8}''$ larger than the shaft S. Plugs U from $\frac{1}{4}''$ to $\frac{3}{8}''$ long, made of bearing metals, such as hard Babbitt, bronze or Oilite, are screwed and soldered into the ends of this channel as shown. These plugs are first bored and afterward carefully reamed until the polished shaft S will just slip through without forcing. Q is mounted in round rings W made of flexible oil-resistant material such as Duprene. Vacuum pump oil (Cenco 93050B) is forced into each gland through O. If made properly, only a cubic centimeter or so will leak out of the plugs U per hour. This oil is always collected, filtered and used again. It will be noted that heat generated in the bearings is conducted by the oil to the large metal plates in which each gland is mounted. As a further precaution the outlets O_1', O_2', O_3' and O_4' are added so that the oil may be circulated in order to maintain the shafts at uniform temperature. Water cooling of U has also been used for this purpose but circulation of the oil is preferable. In practice it was found unnecessary to circulate the oil except when large shafts were used at very high rotational speeds and liquids were

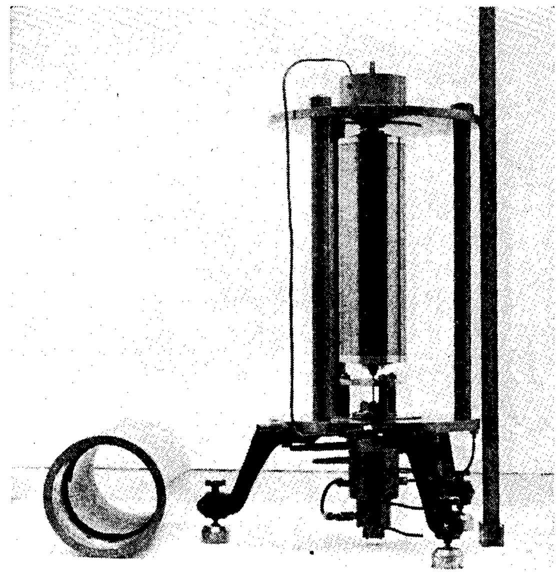

FIG. 2. Photograph of a tubular vacuum-type centrifuge with vacuum chamber removed. At the left is the vacuum chamber, at the right a scale in inches.

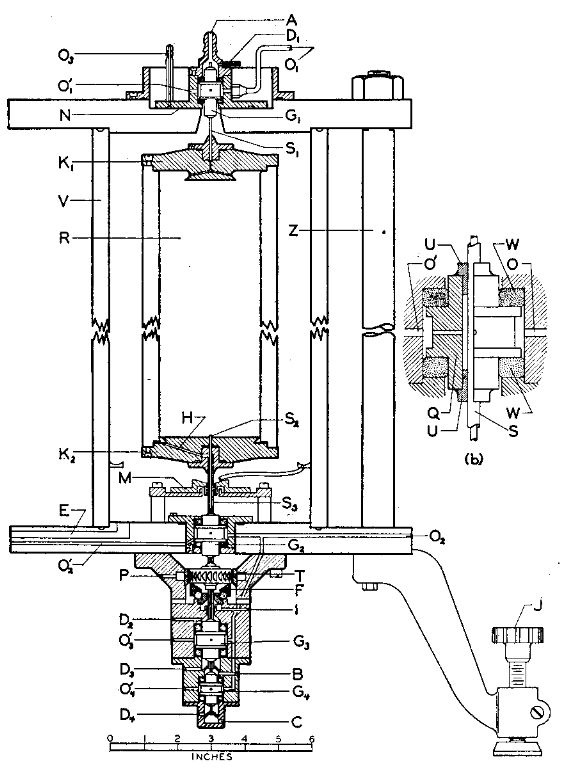

FIG. 1 (a). Drawing of tubular vacuum-type centrifuge used to centrifuge gases and vapors. (b) Enlarged drawing of vacuum-tight oil gland.

being centrifuged slowly. In most cases O_1', O_2', O_3' and O_4' were plugged. The "pill box" shaped turbine T is made of Duralumin. Its diameter is approximately determined by the size of R. For the 4″ tube R shown in Figs. 1, 2 and 3 it was $1\frac{1}{8}$″. T is supported on the air cushion formed between its flat under surface and the Bakelite collar F. F in turn is flexibly supported on a round Duprene or rubber ring. The free air space between F and T is made small to insure stability for heavy rotors. T is driven by directed air jets which issue from channels in the inner cylindrical wall of the surrounding air box and impinge upon its flutings. The channels have their axes in a horizontal plane. If they are extended they should form an envelope to a circle with a diameter about $\frac{3}{16}$″ less than that of the turbine T. The size and number of the channels are not critical and should depend upon the size of T and the air supply available. In Fig. 1 the air box has 8 channels bored with twist drill size 53, while T has 23 flutings. The rotor R is a hollow steel tube. In Figs. 1, 2 and 3 its external diameter is 4″ with approximately $\frac{1}{2}$″ wall thickness. It was heat treated (Chrome-Moly. SAE 4130X) steel. The end caps K_1 and K_2 were also of alloy steel or Duralumin ST 14. They were put on with steel screws and made vacuum tight by rubber, Duprene or soft metal washers. The shafts S_1, S_2 and S_3 were soldered into K_1 and K_2. The Duralumin plate M, which carries a bearing made of leather, is free to slide over the oiled flat upper surface of its support. The bearing is lubricated by the oil leaking through G_1 and N which is caught by the trap on the inner wall of V. This freely moving bearing mounted on M is of value during the acceleration of R to the first few hundred revolutions per second. At higher speeds the leather bearing is usually loose enough not to touch the shaft. M is not absolutely essential for satisfactory operation of the machine and has been omitted many times without serious consequences. The three upright steel rods Z are used to support the upper plate while running R without the vacuum chamber V for purposes of adjustment. They also make the apparatus more rigid. The vacuum chamber V has its ends flat and parallel and ground to fit the upper and lower plates so that a good vacuum can be maintained with ordinary vacuum wax. The upper gland G_1 is mounted on a movable metal plate N, the under surface of which is ground to fit the upper surface of the top plate of the vacuum chamber. Vacuum pump oil under a few pounds pressure is forced in at O_3 so that N is supported upon a thin film of oil. It was found that only a few cc of oil leaked into the vacuum chamber through this floating bearing per hour.

To operate the centrifuge the axis of rotation is first made vertical by the three leveling screws J and a spirit level. The supporting air is then forced into I at a pressure which will support freely the rotating members. With the design shown, this pressure is not critical and rotors from one to 50 lbs. have been used on the same support by simply changing the air pressure on I. In the apparatus of Fig. 2 the supporting pressure was from 30 to 35 lbs./in.^{2} and required about 4.3 cu. ft. of air per minute reduced to standard conditions. D_2 was adjusted so that the air escaping through it was sufficient to blow out oil leaking through G_3. The supporting pressure was also applied to all of the oil glands. For very heavy rotors, G_3 was the only gland to which the necessarily high supporting pressure was applied. Also for this case heavier oil was used. The vacuum chamber is then evacuated through E to a pressure below a micron and the driving pressure applied through P. As the rotors speed up R passes through certain rather sharp frequencies where the floating plate N is observed to move around or "wobble" freely upon its oil cushion. Above or below these frequencies, N remains stationary and seeks a position which makes the axis of rotation vertical. It has been the writer's experience that it is very difficult to get R through these critical frequencies when N is fastened solidly to the upper plate. Invariably

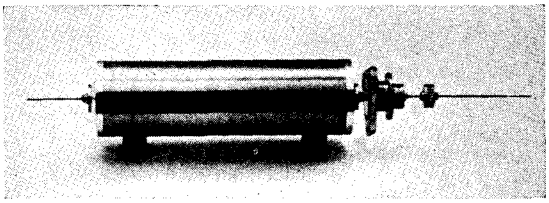

FIG. 3. Photograph of tubular rotor with tubular shafts, air turbine, sliding bearing and demountable lower oil gland which seals the vacuum chamber, all attached ready for mounting.

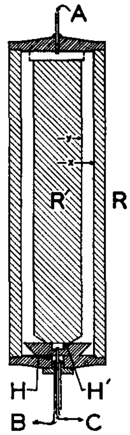

FIG. 4. Drawing of tubular rotor used for centrifuging liquids.

the shafts are sheared off. However this never happens with N floating on oil. Also the acceleration of R remains practically uniform in passing through these frequencies. The machine is, of course, always operated at rotational speeds well removed from these critical frequencies. With a pressure of 60 lb./in.^{2} applied to P, the rotor R in the machine shown in Figs. 1, 2 and 3 attained almost 1200 r.p.s. in 30 min. This required about 28 cu. ft. of air reduced to standard conditions per minute. The rotational speed then remained constant with an applied pressure to P of from 30 lb./in.^{2} to 40 lb./in.^{2}. Because of the large moment of inertia of the long cylinder the rotational speed can be made very constant if the driving pressure is carefully regulated.

When gases or vapors are centrifuged, the material enters at A through S_1 and flows into R at the top. As it passes down R separation takes place. The lighter fraction flows down through S_2 and is collected at C while the heavier fraction flows between the circular flange and the inner wall of R (0.002″ clearance), then through the channels H (as great a number of channels H are provided as possible) into the space between the coaxial tubes S_2 and S_3 and is collected at B. The ratio of the quantity of light material collected at C to the quantity of heavy material collected at B can be regulated by adjustable valves at B and C. Oil traps at D_1, D_3 and D_4 are provided for collecting the oil leaking from the glands G_1, G_3 and G_4. It should be especially noted that the oil pressure in G_1, G_3, and G_4 should always exceed the pressure of the gas or vapor at A, B, and C, respectively.

When the centrifuge is used to separate materials in the liquid state the internal construction of the rotor R is shown in Fig. 4. It is essentially the same as in Fig. 1 except that a solid rod R' is mounted inside in order to direct the liquid flow through the strongest part of the centrifugal field. Where back diffusion is slow, as in the case of liquids, this arrangement greatly increases the rate of separation. R' also makes it possible to anchor the end plates of R more securely and thus relieve some strain upon the screws in K. Since the bursting speed of an empty spinning tube is roughly proportional to x^2w^2, where x is the radius and w the angular speed, while the centrifugal force is proportional to xw^2, smaller diameter tubes R are usually used for centrifuging liquids. In most of the experiments with liquids "rustless" steel cylinders from 2″ to 3″ in diameter with $\frac{1}{2}$″ wall thickness have been used. As yet, an upper limit to the length of the spinning tube R has not been found. Hence, the upper limit to the rate of centrifuging, which is directly proportional to the length of R, has not been reached. Also, the turbine T can always be made to spin much faster than the bursting speed of the tube so that the separation could be much increased if stronger materials could be found for R. The above machine was originally designed for the separation of isotopes, but it should be applicable to many other centrifuging problems. It should be especially useful for the purification of large quantities of biological materials which are not injured by contact with oil.

The writer is greatly indebted to Mr. Fritz Linke and Mr. Phillip Sommer, instrument makers, for skillfully constructing all of the above machines as well as for much valuable assistance with their design. It is also a pleasure to acknowledge a grant from the Division of Natural Sciences of the Rockefeller Foundation, which has made possible the development of the above apparatus.