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MOM-3170 TYPE SMALL-SIZE HIGH PERFORMANCE ULTRACENTRIFUGE

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Development in engineering always proceeds in steps: first the existing machines and equipment are being developed and improved, or new, though essentially similar facilities produced. At this stage development is rather slow, but after a while, it becomes necessary to start from entirely new bases. A good example is the development of the radio valve followed by that of the transistor. The situation is similar in the field of analytical ultracentrifuges. Up to now development has been restricted to the improvement of the complex, large-size and heavy weight machines. We have attempted to embark on a new path and to produce a small-size, light-weight, simple ultracentrifuge with an accuracy and automation exceeding those of the known types.

Thus our intention was to bring the analytical ultracentrifuge "down to earth" and at the same time to raise its accuracy in order to ensure the conditions for its wide range acceptance as demanded by rapidly progressing biological research, as well as by the interests of other sciences. The main advantages of the Type 3170 analytical ultracentrifuge as compared to the earlier models are

1) Reduced weight and dimensions (about 50 per cent of the earlier types),

2) Simplicity,

3) Low energy requirement (appr. 3 kW),

4) Extensive automation and simple operation,

5) High accuracy,

6) Adaptability to preparative operations.

The analytical fields of application of the Type 3170 ultracentrifuge are: all fields where ultracentrifuge measurements are required, such as molecular biology, biology, virus research, diagnostics, plastics, oil and rubber industries.

The apparatus is suitable for velocity, equilibrium and density gradient measurements.

The types of measurements which may be performed by means of an analytical ultracentrifuge are determined by the optics of the apparatus, and the assortment of the analytical cells. The type 3170 apparatus incorporates Philpot-Svensson and Interference optics, and ultraviolet absorption optics to be built in subsequently will also be shortly available.

The most important analytical cell types supplied with the apparatus or available separately are as follows: single and double sector cells, density gradient cells, synthetic boundary cells, mechanical separation cells, and multibore cells for equilibrium measurements. The cell centerpieces are made of aluminium and plastics in various dimensions. In addition to these types there are also separation cells for electron microscopes, and cells to permit the measurement of the equilibrium of labelled radioactive solutions. Naturally, several types of analytical rotors are constructed for the many different cells.

Hungarian Optical Works (hereafter: MOM) continues to increase its type 3170 rotor and cell assortment.

Actually, density gradient measurements performed by using a swinging bucket rotor are also considered as analytical measurements

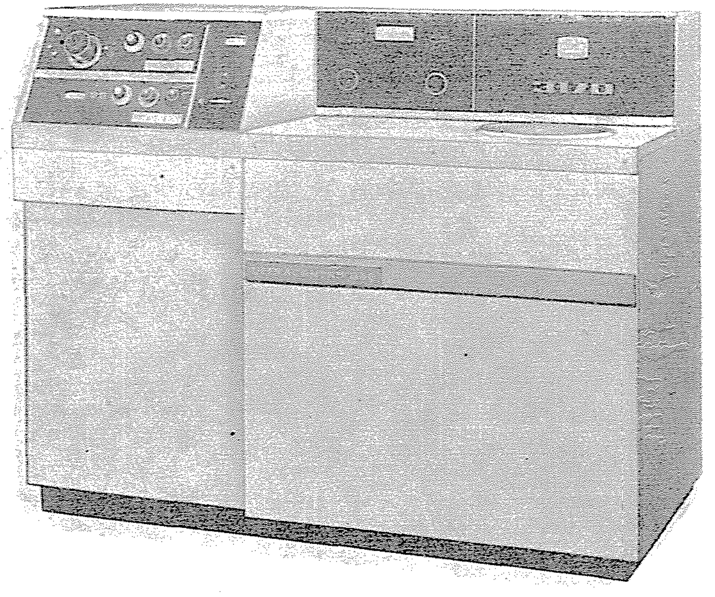

Fig. 1

for which the Type 3170 apparatus is particularly suited.

The type 3170 ultracentrifuge can be supplied with several different preparative rotors, up to 0.4 liter volume, and a maximum centrifugal force of 100 g. The other preparative rotors are smaller, but capable to produce greater centrifugal forces.

By means of the insert producing high temperatures which is now under development the apparatus will be suitable for high temperature measurements (up to 150 °C) as well.

In designing the apparatus, all factors determining the accuracy of an analytical ultracentrifuge were taken into consideration. It was attempted to find a compromise between the different requirements and to put the emphasis on the most important criteria. The accuracy requirements of analytical measurements are in the order of importance:

1) Satisfactory optical image,
2) Accurate temperature measurement,
3) Satisfactory speed control.

Maximum errors are generally committed in the evaluation of the photos, particularly if their quality is poor. The quality of the Philpot-Svensson or Interference photos depends, in addition to the quality of the optics, also on the deformation of the analytical cell and rotor during revolution. For this reason, in Type 3170 a completely new cell and rotor construction has been employed which will reduce these unfavourable deformations. The vibration-free operation of the rotor and the oil-free condition of the vacuum space are, of course, similarly important and it was endeavoured to realize these objectives as closely as possible.

With Type 3170, photography is on film. Its great advantage is the possibility of more than 6 exposures (16) and, according to practical experience, the greater number of exposures improves accuracy indirectly. In this case film shrinkage does not lead to any measurement error, since it is several orders lower than the error in evaluation and thus may be entirely neglected. Another advantage of the film is its higher sensitivity.

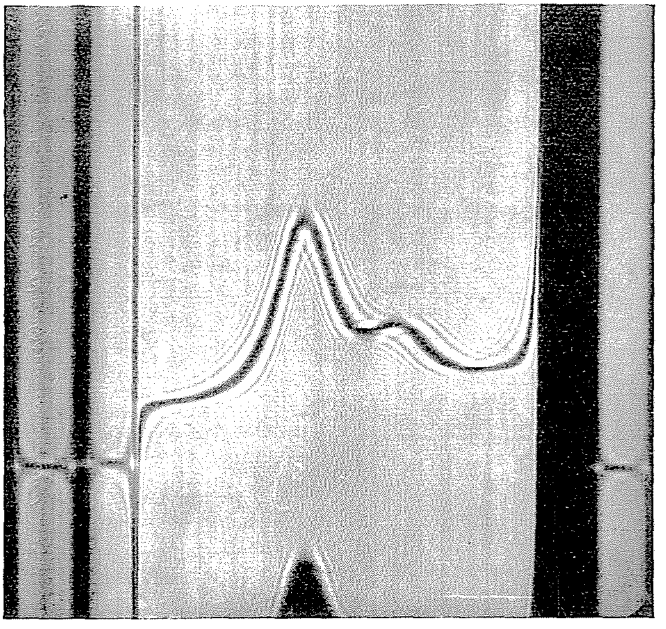

Fig. 2

The second most important accuracy requirement is the measurement and control of the temperature. With respect to temperature measurements, however much necessary they are, no excessive precision requirements can be specified, partly because of the temperature differences within the rotor, and partly owing to the difficulties of temperature calibration. In our opinion the 0.2 °C temperature accuracy specified is quite realistic, and a higher accuracy could only be achieved if it were possible to measure the temperature directly in the cell instead of along the rotor shaft. However, for the time being this is not feasible in any of the available apparatus.

Since only relative values are involved, accurate temperature control is comparatively far more easy to accomplish and is necessary, at that, in order to prevent convection currents within the solution in the cell. The accuracy of temperature control in Type 3170 is within 0.1 °C.

Control of the speed will be discussed later. It will suffice here that increasing the accuracy of speed control beyond a certain limit will not lead to higher accuracy of the measurement.

Comparison of measurements with ultracentrifuge 3170 with data rendered by other apparatus seemed to be in favour of the former.

Operation of the ultracentrifuge including rotor replacement is very simple. Rotors, including the analytical types, must be placed on the swivel pin from above, as usual with preparative types, whereby the awkward assembly commonly required by analytical devices is eliminated. Replacement of the analytical cells is similarly much simpler. Installation and starting of the apparatus are so simple that it is impossible to make a mistake. The individual operation components are separated in space according to their functions. In spite of its simplicity, the ultracentrifuge is automatic and equipped with safety devices. In automation an optimum solution was attempted, that is without impairing reliability for the sake of spectacular features. Speed and temperature control as well as photography are automatic.

The diffusion pump will switch off automatically when the brake is in operation, the centrifuge stops automatically in case of oil shortage, etc. The built-in safety equipment does not permit the operation of the rotors at speeds beyond the permissible maximum.

The apparatus has further built-in safety devices. The automatic assemblies consist of simple and mostly reliable mechanical components.

Each part of the apparatus is within an enclosed unit of 1.70×0.75 m base dimensions and 600 kg weight. The equipment can travel on four wheels. The cover can be removed and replaced in a few seconds for maintenance or repair. Each component is readily accessible. Optical observation is possible by the operator either standing or sitting. The photo unit is similarly easy to reach and to operate.

The instrument is painted grey, with black, red, and chromium trimmings. This equipment was awarded at the 1967 Budapest International Fair the "Most attractive product" distinction, and a Golden Prize at the 1968 Leipziger Messe.

The internal arrangement is articulated according to functions and not crowded.

The rotor of the ultracentrifuge Type 3170 has, unlike that of earlier solutions with their bearing fitted above, similarly to the preparative centrifuges its bearing below. Thus the driving mechanism is underneath the vacuum chamber. One advantage of this solution, as mentioned earlier, is easy rotor assembly and the other is the prevention of the lubricating oil of the bearing to enter the vacuum chamber, that is, oil mist is never encountered.

Driving of the Type 3170 ultracentrifuge is by a 1 HP asynchronous motor joined to a fluid coupling which drives by means of a tining belt in turn a plastic gear accelerator on the shaft of which is the high rpm rotor.

Apart from a few exceptions, all analytical and preparative centrifuges of the world employ plastic gear accelerators, and this is not without reason. Modern engineering often returns by discovering new methods or materials to old, proven machine elements. The same applies to plastic gears, since with driving mechanisms of this type a maximum speed of 100 000 rpm may be achieved. Plastic gear life is practically infinite, only the small steel wheel meshing with it may become somewhat burnished.

The gear type accelerators generally used are driven by controlled speed, brush-type, series wound motors. Usually these motors present the main problem, particularly with respect to the brush. Our solution is entirely different: Type 3170 is driven by a simple asynchronous motor, while speed control and starting are performed by the fluid coupling generally used in the car industry. When switched on the asynchronous motor immediately assumes its full speed, while the coupling is still "slipping", but then the latter takes over acceleration. The efficiency of the system is characterized by the possibility of accelerating the analytical rotor by means of a 1 HP asynchronous motor to a speed of 60 000 rpm within less than 9 min. With the desired speed achieved, rotor revolution can be regulated with the utmost accuracy by modifying the oil charge of the coupling (thus the coupling always slips the more, the lower the speed).

Driving between the hydraulic clutch and the gear type driving mechanism is transmitted by a special timing plastic belt with a stranded steel cable insert. This timing belt represents a completely new machine element combining the advantages of gear and belt drives and has already begun to gain wide acceptance. The same belt is employed in the Fiat-125 cars for driving the valve gear. It does not require any lubrication and has a practically infinite life.

The smooth operation of the driving mechanism system is characterized by the possibility of producing perfect ultracentrifuge photographs.

The life of the driving mechanism is determined by the high-speed shaft and its two high-speed bearings only. Although this life is about 1000 hours in practice, there are two driving mechanisms supplied with the apparatus. Replacement of the driving mechanism takes about 15 min. The used mechanism requires only bearing and shaft replacement. Since the driving motor includes no brushes, it neither requires any maintenance.

According to the experiences collected so far, the driving system is reliable, and capable to withstand even severe operation conditions.

The low energy requirement of the apparatus may be attributed to the fact that the driving motor is only of 1 HP, that is, about 750 watt. This, in turn, may be mainly attributed to the special accelerator properties of the hydraulic clutch which enable the driving electromotor to operate at full speed as soon as it is switched on when the rotor yet hardly rotates. It is not necessary, therefore, to use an electromotor producing an excessive momentum at low speed, and requiring for this a high starting output.

As mentioned above, speed is controlled by changing the oil quantity in the hydraulic clutch. Oil feed and removal is by a small-size gear pump.

Control is by a differential gear which compares the speed of a synchronous motor to that of the shaft extending from the driving mechanism. However, to make control feasible at several speeds, the system includes a small quick-change gear box whereby 32 discrete speeds can be set. Thus the reference frequency is rendered by the mains which is generally accurate enough in every country, since extensive networks are interconnected. The accuracy of speed control is 0.2 per cent at the speed set which is quite sufficient for ultracentrifuge measurements. If average rpm during precipitation is desired with a higher accuracy, it may be measured by means of a simple stop-watch. The apparatus has a drum counter similar to a motor car distance counter from which the number of revolutions can be read off at any moment. Revolution per time gives the speed. It must not be forgotten that in case of sedimentation measurements the average rate and not the momentary value should be used in the calculations assuming, naturally, that speed does not fluctuate beyond a certain limit which is ensured by the apparatus. For information on this point the apparatus has a pointer type speedometer.

Measurement of the number of revolutions by the stop-watch as referred to above is of an absolute character, as the drum counter receives the revolution from the driving mechanism through a 1 : 100 mechanical transmission. This measurement is the most accurate of all methods used so far, although, in our opinion, such accuracy is but seldom required.

The temperature of the rotor is measured by a thermistor sensing thermal radiation. This thermistor is in the geometrical axis of the rotor, and extends into the black tube projecting from the top of the rotor. Its holder is insulated from the environment and, as its temperature is more or less identical with that of the rotor, the error caused by ambient heat conduction is eliminated.

Rotor temperature can be measured with an accuracy of 0.2 °C. The advantages of the system as compared to the hitherto employed rotor incorporated thermistor solutions are, first, that the thermistor does not rotate and, second, that mercury contact and the uncertainty caused thereby are omitted. Temperature measurements should take into account that not all the points of the rotor body, and particularly of the analytical cell and of the liquid in it have absolutely identical temperatures and, consequently, no precision exceeding 0.2 °C has any sense, not even when temperature is measured with a greater accuracy, since it is measured in the rotor shaft and not in the cell. Temperature measurements are performed by an electronic device which regulates rotor temperature with an accuracy of 0.1 °C by means of a small heater located next to the rotor. If the rotor is to be removed, both the thermometer and the small heater may be pushed back and will not be in the way.

Cooling of the vacuum chamber, that is of the rotor environment is by a small, air-cooled, completely enclosed compressor type cooler of 0.3 kW output with Freon-12 cooling medium. Evaporation is in the tubes soldered on the copper jacket enclosing the rotor.

Jacket temperature is controlled by a separate automatic device. The cooling system is reliable and so efficient that rotor temperature can be adjusted down to -5 °C at any ambient temperature. At low speeds under 30 000 rpm even a temperature of -10 °C may be maintained.

A separate heating insert easy to build into the vacuum chamber later has been developed for high temperature measurements. With a special rotor temperatures may be measured up to $+150$ °C by this method. The heater jacket is within the cooling mantle, but cooling also operates during heating to prevent the temperature rise of the vacuum chamber and the lining.

As mentioned before, Type 3170 has Philpot-Svensson and Interference optics which proved to be satisfactory in all earlier MOM ultracentrifuges, Some modifications have been made in the frame, arrangement, easier access and cleaning of the optics. Photography is automatic. Exposure and interval times may be preset but, of course, manual exposure is also feasible. No. 120 film rolls are used on which 16 Philpot or 64 Interference photos may be taken. This film is everywhere available and also used by photoamateurs. As a novelty texts on small boards such as identifying captions, dates, etc. may also be photographed on the film. The automatic photo-device consists of a small casket equipped with automatic transfer mechanism, and of a timer actuated by a synchronous motor. The visual observation mirror is automatically released during exposure. The light source of the optical unit is a 200-watt mercury vapour tube and the unit is completed with a phase plate.

The vacuum in the apparatus produced by a prevacuum and diffusion pump may reach 10^{-3} Hg mm.

The thickness of the armour protecting against eventual rotor explosions is greater than that of any other similar equipment: 48 mm for the inner and 12 mm for the outer ring. This thick armour as well as the special fastening of the cover provide for absolute safety in case of explosion.

Ultracentrifuge Type 3170 is suitable for both routine operations and the most delicate research work. Thus the apparatus can be used for prolonged equilibrium measurements as well as for any preparative purpose. It is, therefore, an apparatus which may — we hope — satisfy the most diverse requirements of the customers.

The apparatus is being continuously developed. This applies, above all, to its accessories such as the rotors, cells, zone rotors, etc., but also to other components available for subsequent installation.

Type 3170 Ultracentrifuge is expected to represent a useful tool in scientific research.