

the task of developing the air-driven spinning top of Henriot and Huguenard [*Compt. rend.*, **180**, 1389 (1925); *J. Phys. Radium*, **8**, 433 (1927)] as a Svedberg ultracentrifuge, of equal power and possibilities but at an expense so low as to make it fairly generally available. Out of many designs, two may be mentioned here. With the latter we are able to obtain sedimentation velocity measurements similar to that illustrated in the accompanying photograph, which shows all four characteristic features of such measurements.

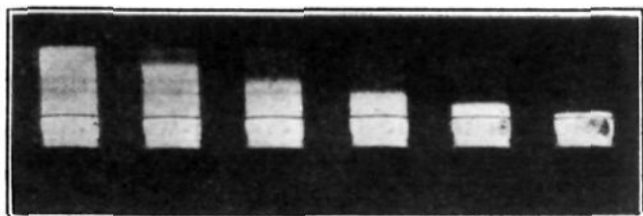


Fig. 1.

An essential part of both designs is that a stationary periscope reaches through a hole in the hollow conical base of the steel rotor and serves to pass light of any desired wave length in the visible or ultraviolet through the cell to a camera. The rotor revolves around the periscope, driven by air supplied at constant pressure and adjusted temperature. The speed of rotation is very accurately measured by beating the note produced by the top either directly or with the assistance of a photoelectric cell against a standard variable oscillator. Speeds are limited solely by the strength of the strongest materials available.

The former cell consists of two disks of crystal-line quartz 30 mm. in diameter made water-tight by an expanding rubber seal between them stretched over a baffle plate of hard rubber in which sectorial openings contain the liquid. A fortunate accident enabled us to observe the sedimentation velocity of a boundary in aqueous solution of mercuric chloride: $S = 0.891 \times 10^{-13}$ cm./sec./dyne; theory = 0.890×10^{-13} .

The latter cell is of a Svedberg type, about one quarter of the size of his, but with a more homogeneous centrifugal field (14% compared with 20%). It fits into a hole in the steel rotor. The photograph refers to the respiratory protein of the blood of the earthworm at 2020 r. p. s. at 22.5°: $S = 72 \times 10^{-13}$.

In the photograph, the uppermost light portion is oil resting on the solution (5% blood of *lumbricus terrestris* in 1% potassium chloride). There is but one boundary and the solution be-

neath is homogeneous, indicating only one size of protein particle. The boundary becomes blurred with time through diffusion and the solution below becomes more dilute through radial expansion and increasing velocity. The immobile streaks were due to a smear of cement.

An elegant alternative solution is that of Beams and Pickles recently communicated to *Science*. They have sedimented diluted human blood (not hemoglobin itself).

We envisage four different fields for the air-driven ultracentrifuge. First, that of Svedberg above, applicable to all molecules. Second, immobilized systems in the hollow spinning top perfected by Henriot and Huguenard (see letter recently submitted to *Nature* on the many kinds of measurement so afforded). Third, sedimentation of the Bechhold type in the hollow top. This is presumably the kind of sedimentation we have observed with hemoglobin, methylene blue, etc., during the past few years. Fourth, convectionless sedimentation in a mechanically immobilized liquid of any kind (including for example virus, phage or sucrose).

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RECEIVED MARCH 23, 1935

RADIO-CESIUM ACTIVATED BY NEUTRONS

Sir:

All cases of radioactivity induced by neutron bombardment which have been investigated have been shown to be β -activity. However, it seemed possible that radio-caesium might be an exception to the rule and emit a positron since the emission of an electron would result in the formation of Ba^{134} which apparently does not exist in nature, while the emission of a positron would form a known isotope of xenon, Xe^{134} . The caesium case is also simplified by the fact that it has only one isotope, 133, and that the observed activity, of 100 minute half-life, is hydrogen sensitive, *i. e.*, is due to the capture of a slow neutron. We have therefore determined the sign of the particle and have found that in this case too it is an electron. The measurements were made with a screen-wall Geiger-Müller counter by bending the particles around a lead shield 5 mm. thick with a field of 5000 gauss. The observed effect decayed with a half-life of 80-100 minutes, and we also find the

activation greatly increased by a hydrogenic environment.

Since the Ba^{134} resulting from the decomposition is not found by mass spectrum analysis, it may be presumed to be unstable. However, we have added a small amount of barium to cesium nitrate activated by neutrons and upon precipitating the barium chemically, have found that the barium shows no activity of half-lives between ten minutes and three days. Chemical separations also show the absence of radio-iodine or radio-xenon.

We are indebted to Dr. L. R. Taussig and the University of California Hospital for 150 milligrams of radon used in the experiments.

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RECEIVED MARCH 25, 1935

THE REACTION OF FLUORINE WITH NITRIC ACID AND WITH SOLID POTASSIUM NITRATE TO FORM NO_3F

Sir:

Cady [THIS JOURNAL, 56, 2635 (1934)] has recently discovered that when fluorine is bubbled through 3 *N* nitric acid, the remarkable compound NO_3F is formed. Through Dr. Cady's kindness his apparatus was made available to us and we have repeated the preparation. The resulting material has all the properties described by him. It boils at about -42° , it liberates iodine from iodides and, in the gaseous form, it explodes when heated to some 200 or 300° .

We have found that NO_3F reacts very slowly with VO^{++} to give vanadic acid, and that no appreciable reaction takes place with Mn^{++} in 6 *N* H_2SO_4 , as judged by color changes. When, however, the gas is brought in contact with solutions of silver nitrate, a black precipitate (Ag_2O_3) forms at once. Mixtures of Mn^{++} and Ag^+ in 6 *N* H_2SO_4 reacted with the NO_3F to give permanganate. The reaction with silver ion is convenient for testing purposes if ozone and OF_2 are known to be absent.

We have also found that fluorine reacts readily and efficiently with solid potassium nitrate to form a substance that appears to be NO_3F . The gas explodes on heating, it reacts with iodide ion and silver ion in the manner described above, and the boiling point agrees, qualitatively, with that given by Cady for NO_3F . Twenty cc. of the substance in the liquid state (at -79°) was prepared.

By allowing it to boil at atmospheric pressure for a time it was freed from any of the more volatile impurities. The vapor, at atmospheric pressure, was then passed through dilute nitric acid to remove any hydrolyzable impurities. It was then distilled from a trap cooled to -78° into a trap immersed in liquid air. The resulting material had, when molten, and unlike the material prepared from nitric acid, a very slightly yellowish color. Due to some other substances, or to a slow decomposition, an apparently constant pressure of from 5 to 10 mm. developed above the material cooled with liquid air, even after repeated pumping. The solid material, while under vacuum in the trap cooled with liquid air, eventually exploded very violently (danger!), and for no obvious reason.

CONTRIBUTION FROM
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1,2-BENZOPYRENE

Sir:

In preparing a quantity of 1,2-benzopyrene required in a study which is being carried out in collaboration with the Office of Cancer Investigations, U. S. Public Health Service, the synthesis of Cook and Hewett [*J. Chem. Soc.*, 398 (1933)] has been modified to advantage in certain details, the over-all yield from pyrene being 36%.

The condensation of pyrene (0.6 mole) with succinic anhydride (0.72 mole) in nitrobenzene solution (600 cc.) with aluminum chloride (190 g.) was carried out as described [THIS JOURNAL, 54, 4351 (1932)] for the similar condensation of acenaphthene. Sodium β -1-pyrenoylpropionate was collected after one crystallization and washed free of tars with alcohol and ether. The material was directly pure and suitable for reduction; yield 90–94%. The method of reduction of Cook and Hewett was not improved in yield (71–75%, pure), but the process was simplified. After acidifying the solution of γ -1-pyrenylbutyric acid, the suspension was heated at the boiling point for one hour to dehydrate the hydroxy acid and give a granular product. This was extracted with hot sodium bicarbonate solution, then with cold soda solution, and the combined filtrates were acidified and digested. The crude acid (m. p. 181–183°) was crystallized once from xylene (m. p. 187–188°).