1 millisecond, under the pressure difference of 1 kg/cm². Under the larger pressure difference, the more volume of gas will be sampled. The sampling may be more effective provided the cross-sectional area of the gas passage is widened.

The intended purposes were accomplished on the whole, although some problems remain to be discussed.

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The Free-Running-Type Equilibrium Ultracentrifuge*

By Yasuyuki GOTO**, Yuji MIYAZAWA***,
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The construction of a free-running-type equilibrium ultracentrifuge is reported in this paper. A centrifuge rotor, made of steel, weighing about 12 kg, is suspended magnetically in the vacuum chamber, the pressure of which is less than 10^{-5} mmHg, and its position is controlled automatically. Acceleration of the rotor to operating speed and braking of the running rotor are done by the driving motor whose shaft is connected to the rotor by a stainless steel rod. During the sedimentation experiment, the rod is disconnected from the rotor and then the rotor is kept running freely by its own moment of inertia. The sedimentation is observed with either a Rayleigh interferometer or a Schlieren optical system. Experiments on molecular weight determinations of digitonin gave good results.

1. Introduction

In the fields of physical chemistry and biology, the ultracentrifuge study is recognized as one of the most valuable methods for measuring the molecular constants of solute molecules. There are two ways of approach⁽¹⁾⁽²⁾, that is, 1) sedimentation velocity method, and 2) sedimentation equilibrium method. Sedimentation velocity method is carried out in a strong centrifugal field in order to make the solute molecules sediment rather fast. Molecular weight of solute molecules is calculated from the sedimentation constant using the theoretical formula based on hydrodynamics. Comparing the sedimentation

velocity method with the sedimentation equilibrium method, the latter has the advantage of being able to measure molecular weights of substances having relatively low molecular weights in a rather weak field of centrifugal force. Despite this advantage of the sedimentation equilibrium method, however, the sedimentation velocity method is used in many cases because of the speed of the measurement. Further reason is the many difficulties mainly involved in the stabilized rotation of ultracentrifuge rotor and the temperature control of the testing solution over a long period. To overcome these difficulties, Beams and his colleagues⁽³⁾ proposed a new type ultracentrifuge with a magnetically suspended rotor in a vacuum.

In this paper, the construction of a new well-improved sedimentation equilibrium ultracentrifuge of magnetic suspension type is reported as well as some experimental results obtained with this ultracentrifuge.

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2. Structure of free-running-type equilibrium ultracentrifuge

The main function of this ultracentrifuge is to measure the accurate molecular weights of materials, of which molecular weights are larger than several hundreds, and to obtain molecular weight distribution, according to the sedimentation equilibrium method. The ultracentrifuge mainly consists of a supporting frame for the main body, a supporting frame for the control circuit and measurement units, and other equipments, as seen in Fig. 1.

The supporting frame for the main body contains the following parts:

- (1) Main body
 - 1) Magnetic supporting device for rotor suspension
 - 2) Rotor
 - 3) Vacuum chamber
 - 4) Driving unit
- (2) Optical system
 - 5) Combined optical system by Schlieren cylindrical lens method and Rayleigh interference method
 - 6) Photographing apparatus
- 7), 8) Light source and its power source
- (3) Vacuum making system
 - 9) Oil rotary pump
 - 10) Oil diffusion pump
 - 11) Vacuum valve (air operated)
 - 12) Vacuum valve (magnetic)
 - 13) Liquid nitrogen trap
 - 14) Vacuum gauges (Philips and Geissler gauges)
- (4) Air compressor (for air valve, and also used to lift the vacuum case for changing the specimen)

The following circuits are accommodated in the control and the measurement operation frame:

- 15) Vacuum measurement circuit
- 16) Temperature control circuit
- 17) Rotating speed measuring circuit
- 18) Rotor position control circuit
- 19) Coil current supply circuit
- 20) Power source operation panel
- 21) Drift compensation circuit

Accessory units include an automatic voltage stabilizer, emergency batteries for power failure, and an "analytical cell".

The ultracentrifuge rotor, loaded with test specimens, is suspended in the vacuum chamber by controlled magnetic force. The driving rod passes through the center of the rotor cradle and is movable either upward or downward. The rotor is driven by the driving rod, which had been lifted upwards to be connected with the rotor. When the rotor is accelerated to the desired speed, the driving torque is decreased and the driving rod is disjointed from the rotor. Hereafter, the rotor rotates at a fairly constant high speed in the vacuum chamber by its own inertia, almost free from friction. Two cells, one cell contains the specimen solution and solvent and the other acts as a reference cell, are loaded into the holes in the rotor, which are parallel to the axis of rotation, 65 mm apart from the axis and located on the opposite sides around the axis to each other. The variation of the concentration of the solution in the cell under the centrifugal field is observed by means of the Schlieren or interference optical system to determine the molecular weight of the solute molecules.

3. Suspension of the rotor by magnetic attraction

3.1 Principle of magnetic support

Windage loss of the rotor and friction of the bearings are undesirable obstacles to keeping the stable rotation of the rotor at a high speed. In

Fig. 1 Skelton diagram of free-running-type equilibrium ultracentrifuge

other words, it would be ideal if the rotor could be freely suspended in vacuum. For this purpose magnetic attraction force was used as a counter-force against gravity. Thus a suspension coil was arranged as shown in Fig. 2, to support the rotor along the center line and just beneath the coil.

3.2 Magnetic support circuit

Fig. 3 is a block diagram of the circuit for supporting and stabilizing the rotor. A power amplifier excites the cylindrical supporting coil and creates a magnetic field capable of suspending a 12 kg rotor. The cylindrical coil consists of three bobbins, each 7 cm in inner diameter, 24 cm in outer diameter and 7.7 cm in height, and of 0.6 mm copper wire wound in 33 270 turns. Fig. 4 shows the attraction characteristics of the cylindrical coil used in the magnetic supporting device as shown in Fig. 2. The abscissa represents the positions of the rotor measured from the ceiling of the vacuum chamber, and the ordinate represents the coil current. Parameters a and b represent the distances from the origin to the central magnetic core and from the origin to the lower end of the cylindrical coil, respectively. It is shown in Fig. 4 that the cylindrical coil has a central magnetic core and the tip of the core protrudes from the bottom of the coil. The current compensating the gravity by magnetic attraction is approximately proportional to the distance between the central magnetic core and the rotor within the distance range of 4~18 mm. It is also evident that the coil current can be held at a minimum by lowering the position of the

central magnetic core.

The characteristics of the magnetic supporting device shown in Fig. 2 can be expressed by the following formulas

$$e(t) = L \frac{di(t)}{dt} + Ri(t) + A \frac{dx(t)}{dt} \quad \dots\dots\dots (1)$$

$$m \frac{d^2x(t)}{dt^2} = ki^2(t) - mg = k[i_0^2 + 2i_0 \cdot \Delta i(t) + \Delta^2 i(t)] - mg \quad \dots\dots\dots (2)$$

where L represents the coil inductance, R the direct current resistance of coil, A the coefficient of the voltage produced in the coil by rotor displacement, m the mass of the rotor, g the acceleration of gravity, k the coefficient of the coil magnetic attraction, $x(t)$ the position of the rotor, $e(t)$ the coil terminal voltage, $i(t)$ the coil current, and i_0 the coil current balancing rotor against the gravity. Applying Laplace transformation to the formulas (1) and (2) after omitting the square term in formula (2), and using the relation $ki_0^2 = mg$, the transfer function, $G_1(s)$, of the magnetic supporting device becomes

$$G_1(s) = \frac{X(s)}{E(s)} = \frac{1}{As} \cdot \frac{1}{T_1^2 s^2 + 2T_1 \zeta s + 1} \quad \dots\dots\dots (3)$$

where

$$T_1 = \sqrt{\frac{Li_0}{2Ag}}, \quad \zeta = \sqrt{\frac{R^2 i_0}{2^3 ALg}}$$

In order to stabilize the magnetic supporting control system, a gain adjustment and a phase compensation circuit are used in the feedback circuit as shown in Fig. 5.

Putting $T_2 = (\alpha + \beta/\alpha)CR'$, $T_3 = CR'$, the transfer function $H_1(s)$ of the circuit can be written as

Fig. 2 Skelton diagram of magnetic supporting system

Fig. 3 Block diagram of supporting circuit

Fig. 4 Magnetic attraction characteristics of supporting system

Fig. 5 Gain and phase compensation circuit $R=100 \text{ k}\Omega$, $C=0.03 \text{ }\mu\text{F}$, $R'=50 \text{ k}\Omega$

$$H_1(s) = \frac{\alpha(T_2s+1)}{T_3s+1} \quad \dots\dots\dots (4)$$

Then, the overall transfer function, $G(s)$, is given by

$$G(s) = K_f \cdot G_1(s) \cdot K_d \cdot K_m \cdot H_1(s) \\ = \frac{K_f \cdot K_d \cdot K_m}{As} \cdot \frac{1}{T_1^2s^2 + 2T_1\zeta s + 1} \cdot \frac{\alpha(T_2s+1)}{T_3s+1} \quad \dots\dots\dots (5)$$

where K_f represents the amplification factor of the power amplifier in the forward circuit, and K_d and K_m the amplification factors in the feedback circuit. The "Q" variation method is used for detecting the deviation of the rotor from the equilibrium position. The detection coil consisting of 0.6 mm copper wire of 10 turns, is wound around the rotor cradle of a steatite cylinder 5 cm in outer diameter, forming a tuning circuit for the frequency of 4 Mc at the grid of an oscillating circuit. The output of the position detector is indicated on the position meter. At the same time, the output is applied to the compensation circuit and adjustment is made automatically to meet the requirements for a stable control. The output of the compensation circuit is amplified by a mixing amplifier and fed back as the controlling signal.

When the measured constant, $K_f = 4.13$, $K_d = 169$, $K_m = 29$, $L = 40\text{H}$, $R = 826\Omega$, and $A = 12 \{V/(cm/sec)\}$, of the control circuit are applied to the loop transfer function $G(s)$ of formula (5), we obtain $T_1 = 0.024$, $\zeta = 0.25$. Adjusted the gain of the compensation circuit as $\alpha = 0.021$, $\beta = 0$, the gain and the phase characteristics are given by g_1 and θ_1 in Fig. 6. The chart shows that phase lag is too great to stabilize the rotor. When β is readjusted to 0.56, the time constants in formula (4) become $T_2 = 0.042$, $T_3 = 0.0015$. The gain and the phase in this compensation circuit become g_2 , θ_2 , g_3 , θ_3 , in Fig. 6. The gain and the phase characteristics of the loop transfer function $G(s)$

Fig. 6 Bode diagram of supporting system

in formula (5) are shown by $|G|$ and $\angle G$ in Fig. 6. The gain margin and the phase margin become 15 db and 22° , respectively, and the rotor can be suspended stably.

3.3 Characteristics of magnetic support circuit

The stability of the rotor suspended by means of the magnetic support circuit determines the value of this ultracentrifuge. The requirements of the stabilization of the control system are satisfied by adjusting the gain and the phase of the automatic control system and thereby the vertical oscillation of the suspended rotor is eliminated. The amplitude of the hunting is held below 1/100 mm. By applying the signal of a low-frequency oscillator to the control system, and by measuring the oscillation of the rotor, the frequency characteristic of the closed circuit is obtained as shown in Fig. 7. The resonance angular frequency of the closed loop is 15.5, and the peak, M_p , at that point is approximately 15 db, and the oscillation is damped when its angular frequency is more than 50.

3.4 Drift compensation of rotor position

In the sedimentation equilibrium experiment the change of position of the rotor owing to the drift becomes an important problem. Though this change is very slow, the rotor may deviate from the range of magnetic supporting and touch either the rotor cradle or the guard plate in the course of a long time operation. An effective countermeasure for the drift has been developed by using a minor loop to adjust the average excitation current of the coil to a fixed value.

Therefore, accurate control regarding the changes of the rotor position within a short period should be performed with the main feedback loop, whereas the rotor position should be controlled with a minor loop, when the change is comparatively slow, by checking the change of the coil current at the point of equilibrium. Fig. 8 shows the record of the suspended rotor position after the operation for 2.5 hours at 20 000 r.p.m. and approximately 6 hours.

Fig. 7 Closed loop frequency response of the whole except drift compensator

at 4 400 r.p.m., using a magnetic support circuit with a minor loop for drift compensation. Although there are vertical oscillations of approximately 0.5 mm when the driving rod is jointed and disjoined, the rotor continues to run smoothly keeping its vertical position within the limits of ± 0.15 mm after the driving rod is disjoined from the rotor.

3.5 Countermeasures against power failure

A grave danger may arise if the rotor deviates from the point of equilibrium as a result of some defects in the power source or control system, since the ultracentrifuge of this type has no part corresponding to the bearing in the conventional rotating machinery. In this system teflon blocks were used for the rotor cradle and the ceiling of the vacuum chamber. The magnetic force attracts the rotor towards the ceiling of the vacuum chamber. Whenever the current of the cylindrical coil drops to 290 mA or less, the power source of the coil is automatically switched from the control circuit to the battery and the rotor is attracted upwards. In this case, rotor continues to rotate, touching its coneshaped top with the teflon plate. If power failure continues over a long period, the contact friction between the surface of the rotor and the teflon plate serves to brake the rotor.

4. Driving part

For accelerating the rotor to a maximum of 30 000 r.p.m. or for stopping it, an AC commutator

Table 1 specifications of rotor

Maximum diameter	187 mm
Length of shaft	140 mm
Maximum applicable centrifugal force	6.5×10^4 g
Weight	12 kg
Moment of inertia	4.5×10^5 g-cm ²
Thermal capacity	1.4×10^6 cal

j; jointing of driving rod to the rotor
d; disjoining of driving rod from the rotor
a; acceleration region
r; free running region
b; deceleration region

Fig. 8 Records of rotor position

motor and step-up gears are used for the driving part, so that handling may be easy, and power supply may be directly obtained from an AC source. Non-magnetic stainless steel is used for the driving rod so that the magnetic effect may not reduce the stability of the suspended rotor when jointing and disjoining of the rotor with the rod take place. The driving rod is welded to the pinion shaft of the acceleration gear.

The pinion gear can be moved either up or down about 8 mm by changing the engaging position of the gears in the direction of the tooth width of the gears by the rack and pinion mechanism. Approximately 4 mm of the tip of the driving rod has a 1.8 mm square section which slides into the square groove on the bottom of the rotor.

Table 1 shows the specifications of the rotor used in this ultracentrifuge.

5. Revolution counter

The ultracentrifuge has three kinds of revolution counter. They are: (1) Driving part tachometer, (2) Precision tachometer for rotor, (3) Tachometer for synchronizing the rotation of the driving rod with that of rotor.

The second tachometer is to measure the free rotation of the rotor disjoined from the driving rod by the optical method. The pulses of the light which pass through the cell hole of the rotor are condensed on a photo-transistor and transformed into voltage pulses. The number of pulses can be counted up to the sixth digit by means of two decatrons and a magnetic counter. The third tachometer is used for synchronizing the rotation of the driving rod with that of the rotor, when jointing them for stopping the rotor, or changing the speed of the rotor. The Lissajous' figure is observed applying the output of the photo-transistor in tachometer circuit to the X axis of the cathode-ray oscilloscope and applying the output of the rotary generator of the driving rod to Y axis. After the rotation of the driving rod has been synchronized with that of the rotor, jointing of the driving rod to the rotor, can be easily done by looking through the window of the vacuum cylinder. Fig. 9 gives an example of measurement of the natural deceleration of the rotor while in free-running. The damping rate in this case is 3.6 r.p.m./hr.

6. Heating and temperature control of rotor

It is necessary to keep the temperature of test specimen at a constant condition in

order to bring the sedimentation equilibrium resulting from centrifugal force in balance with the diffusion of solute molecules. Furthermore, heating and temperature control are used for the ultracentrifuge research on solutions which are soluble only at high temperature, for example on the high polymers such as polyethylene. Although the quantity of test specimen for one measurement hardly exceeds 0.05 ~ 0.1 cc, the temperature of the entire rotor in which the test specimen is to be loaded must be controlled in order to keep the temperature of the test specimen constant. Since the rotor is suspended in a vacuum container, we used a thermister radiation thermometer. Moreover, the rotor surface is painted black, constant temperature water is circulated inside the vacuum cylinder wall, and a heat reflector is used between the rotor and the vacuum cylinder. A heater wiring with a diameter of 82 mm is set coaxially with the driving rod and is used to heat the rotor from the bottom of it. With the measurements of temperature by buried copper-constantan thermocouples in a stopping rotor, it was found that the temperature was controlled within the range of 0.2°C at 130°C.

7. Measurements of concentration distribution of test specimen⁽⁴⁾

The measurement of the distribution of solute molecules in a centrifugal field and the quantitative measurement of the change of concentration with respect to time are done through photographic recording of either the Schlieren or Rayleigh interference pattern by an optical system, which can be used for either of these two methods.

The structure of the cell for ultracentrifuge measurement is shown in Fig. 10. The solvent and solution are first poured through the two windows into the double-sector cell. When the light beam of wave length λ is divided into two and the one passes through the material, having the refractive index μ_0 and the other passes through the material, having the refractive index μ , and thickness of each material is d , the two beams interfere, because of

Fig. 9 Natural deceleration characteristic of rotor

the path difference of $(\mu_0 - \mu)d$. The interference stripes shift as many as $(\mu_0 - \mu)d/\lambda$ stripes, since the light wave number $(\mu_0 - \mu)d/\lambda$ is contained in this difference. In other words, if the wave-length and thickness of the test specimen d are known, the refractive index difference can be found by measuring the shift of the interference stripe. Moreover, the concentration variation of the test specimen can be known through the shift of the interference stripe by the measured refractive index, gradient of the concentration $\partial\mu/\partial c$.

Fig. 11 gives an example of measurement carried out with this ultracentrifuge. The distance between the tip of the cell and its bottom is the length of the cell in the direction of centrifugal force. The interference-stripe pattern can be divided into three parts from the side near the rotor axis toward the direction of centrifugal force. They are: (1) interference made by light beams, both passing through the air, (2) interference made by light beams, one passing through the air and the other through the solvent, (3) interference made by light beams, one passing through the solvent, the other through the solution. No stripe can be seen in part (2) since the interference order is too high.

Concentration gradient is formed in the direction of centrifugal force and the refractive index differs with the position in the cell, because of the occurrence of sedimentation, after centrifugal force has been applied to the test specimen.

Fig. 10 Construction of double sector cell

Fig. 11 Interference pattern of sedimentation

8. Measurement of molecular weight by sedimentation equilibrium⁽¹⁾⁽⁵⁾⁽⁶⁾⁽⁷⁾

When centrifugal sedimentation is carried out over a long period under the designated conditions, the sedimentation and the diffusion of the solutes are eventually balanced, that is a state of sedimentation equilibrium. In this state, the relation between the molecular weight M of the solute and the concentration gradient, formed within the cell in the direction of centrifugal force, is:

$$RTd\ln C = M(1 - \bar{v}\rho)\omega^2 x dx \quad (6)$$

where R represents the gas constant, T the absolute temperature, C the concentration, $\bar{v}$ the partial specific volume of solute, ρ the specific gravity of solvent, ω the angular velocity of rotation, and x the distance from the center of rotation in the direction of centrifugal force.

If the compression ratio can be disregarded in the two-component system, formula (6) can be integrated and becomes

$$M = \frac{2RT \ln C_b/C_a}{(1 - \bar{v}\rho)\omega^2(x_b^2 - x_a^2)} \quad (7)$$

Suffixes a and b represent the surface and the bottom of the solution column, respectively. Generally speaking, high polymer consists of components having various molecular weights, and, therefore, the measurement of molecular weight gives different values depending on the method of obtaining the average. It follows, therefore, that molecular weight cannot be determined by formula (7) because it is based on the homogeneous system with respect to the molecular weight of solute. The following formula is used in the multicomponent system which has a molecular weight distribution.

$$M_w = \frac{2RT(C_b - C_a)}{(1 - \bar{v}\rho)\omega^2 C_0(x_b^2 - x_a^2)} \quad (8)$$

where $M_w = \sum C_i M_i / \sum C_i$ represents the average molecular weight, i the component, and C_0 the initial concentration. Formulas (7) and (8) are valid only for ideal solutions. Therefore, the discrepancy between the ideal solution and the test solution becomes large as the concentration of the solution increases. This effect is best illustrated by

Virial expansion. If we denote the calculated molecular weight, by formulas (7) or (8), at a given concentration C as M^{app} , the actual molecular weight, M_0 , is given by

$$1/M^{app} = 1/M_0 + 2A_2C \quad (9)$$

where A_2 is referred to the second Virial's coefficient.

We measured the molecular weight of digitonin ($C_{56}H_{92}O_{29}$) in order to determine the accuracy of the ultracentrifuge measurement. Digitonin is a crystalline substance produced of Merk. Its melting point T_m is 235.5°C. We used this without any further purification in subsequent measurements since T_m coincides with the value 235~6°C, given in the literature⁽⁸⁾. And a 50 volume percent solution of water and ethyl alcohol was used as the solvent in order to carry out the measurement in a state as close to ideal solution as possible. The ethyl alcohol was distilled twice after dehydration with quicklime. The fraction at a distilling temperature of 78.3°C was collected. The specific gravity of the solvent was 0.9324 at 25°C. The concentration dependence of refractive index, $\partial\mu/\partial c$ of the digitonin solution was measured as 0.138 ml/g, using a Zeiss interferometer, calibrated by the aqueous solution of the sucrose supplied by the American National Bureau of Standard. It was after 50 hours that the shift of the interference stripes was found to cease and that an equilibrium condition was attained.

The molecular weight was calculated according to formula (7) by measuring the change of interference stripes on the photograph taken after reaching the sedimentation equilibrium, by means of a comparator with an accuracy of 0.001 mm. The results and the conditions of the measurements are given in Table 2. " M_{calc} " is 1229.35 obtained from the molecular formula of digitonin. The molecular weight thus obtained has an error margin of only 1 % at most, using formula (7) and taking into account of the relative error of measurement.

9. Conclusion

Research work on the construction of the free-running-type ultracentrifuge and the sedimentation

Table 2 Results of the molecular weight measurement

No.	Concentration g/100 ml	Final speed of rotation r.p.m.	Duration of rotation hr	Measured molecular weight M_{obs}	Relative error
					$\left \frac{M_{obs} - M_{calc}}{M_{calc}} \right \%$
1	0.2306	14170	33	1231	0.12
2	0.4646	15870	55.5	1236	0.54
3	0.6006	20306	67	1230	0.02

Rotor temperature 22°C

equilibrium were reported. This research was carried out as part of the research program for the development of a free-running-type equilibrium ultracentrifuge launched by the Tokyo Kyoiku University subsidized by the Ministry of Education. We wish to express our gratitude to Prof. A. Kotera, Prof. K. Suzuki and their colleagues of Tokyo Kyoiku University, who gave us both the opportunity for the research and invaluable advices. We also note with gratitude that we could accomplish this work in a relatively short period thanks to the cooperation and guidance of Dr. S. Sasaki, Dr. T. Nakada, and Dr. O. Taniguchi who have played prominent roles in the field of ultracentrifuge research for the past 10 years.

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On the Roller Straightener*

(2nd Report, Straightening of Round Bars, Pipe and Tubings)

By Haruo TOKUNAGA**

The rotary straighteners for straightening round bars and tubes are discussed.

The author found that the residual stress distribution in the bar straightened by the rotary straightener represents an alternate vortex of tensile and compressive stress zones around the center of the bar, which is not affected by the repeated rotary bending. To obtain better straightness, therefore, the bar has to be bent more severely at the point under the rolls, and receive more multiple rotary bending near the outlet in the straightener. For the rotary plastic bending of the bar, the direction of the bending moment does not coincide with that of the bending of the bar, and there is some angle between both directions, and therefore power is consumed for the rotary plastic bending of the bar. From this point of view, the author derived a formula to calculate the power required for straightening of the bar.

1. Introduction

For the straightening of the round bars and tubings, the rotary straighteners having oblique rollers are mainly used, because the bars and tubings can not obtain better straightness by the parallel roller straightener used for the straightening of the shapes and profiles, since on the parallel roller straightener, the round bars and tubings rotate on the rollers, when the bending moment is applied. There are many different types of the rotary straighteners according to

the arrangements of the rollers, number of rollers, the types of roller drivings and so on, and in all of the rotary straighteners, the rollers of concave hyperbolic contour are used, but in very special cases, the cylindrical or the drum type ones are used.

It is the principle of the rotary straightener that the bars and tubings advancing in the rotary straightener are applied with a rotary plastic bending moment, and very few scholars have hitherto, studied the fundamental of the process^{(1)~(4)}.

The 5-roller straightener is the typical one of the rotary straighteners; it is shown in Fig. 1 (A).

In the present paper, the author studies the

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