Chem. Pharm. Bull. 23(2) 326—332 (1975)

UDC 615.011.4.014

Compaction of Powders. III.[1] Comparison of Compaction Behaviours among Various Powders

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(Received July 15, 1974)

Various powders are compressed at the constant rate of displacement of upper punch equal to 0.332 mm/sec for the comparison of the results to those in the cases of potassium chloride and lactose powder reported in the previous paper. Consequently, from the patterns of changes of pressure-transmission-ratio with the increase of upper punch pressure, the powders are divided into three classes, as followings; (1) KCl type, for which the pressure-transmission-ratio takes the maximum value at the almost constant porosity (about 32%) without reference to material and net volume, (2) lactose type, for which the values of the ratio decrease monotonously, and (3) the powders not belonging to KCl and lactose type, for which the values of the ratio do not decrease. Besides, the comfirmity of the results to the Janssen's equation is checked up.

Furthermore, the powders are compressed continuously under the constant pressure of upper punch for ten hours for the purpose of examining the creep properties.

Introduction

In the previous paper,[3] the authors reported that the potassium chloride and lactose powder showed the very different behaviours, when they were compressed from one side by upper punch at the constant rate of displacement of upper punch equal to 0.332 mm/sec and under the constant pressure of upper punch for ten hours. That is, in the compression at the constant rate of displacement, the values of pressure-transmission-ratio to lower punch took the maximum at half-way in the case of potassium chloride powder and decreased monotonously with the increase of upper punch pressure in the case of lactose powder. Further, when the potassium chloride powder was compressed continuously for ten hours under the constant pressure of upper punch higher than 625 kg/cm^2, the values of the ratio and the porosity of powder bed tended to approach to 100% and zero, respectively, while in the case of lactose powder they took the constant values after about five hours in spite of considerable amounts of void left still.

In the present work, identical measurements are made with various powders for the comparison to the potassium chloride and the lactose powder.

Experimental

The preparations of powders, the measurements of specific gravities of materials and the compressions of powders are made with the same methods as in the previous paper.[3]

In Table I, the mesh size and the specific gravity of each compressed powder are shown. The mesh sizes of most powders are uniformed from 42 to 65 mesh. But, in the cases of potato starch, acacia and aluminium hydroxide powder, the fractions of 200 to 250 mesh are compressed, for the fractions of 42 to 65 mesh cannot be obtained owing to the small particle sizes of these powders. Further, as the particle sizes of calcium stearate powder do not exist within the sieve range and the particles of sodium stearate powder are flakelike, these two powders are not sieved. Three aniline salts are not also sieved. For the sodium bromide powder, only a part of measurements is made, since this powder is so hygroscopic that the specific gravity decreases gradually.

1) Part II: J. Okada and Y. Fukumori, *Yakugaku Zasshi*, 94, 285 (1974).
2) Location: *Yoshida-Shimoadachi-cho, Sakyo-ku, Kyoto.*
3) J. Okada and Y. Fukumori, *Chem. Pharm. Bull.* (Tokyo), 22, 493 (1974).

Table I. Compressed Powders

Material	Mesh	Density (g/cm^3)	Material	Mesh	Density (g/cm^3)
KCl	42—65	1.988	$Al(OH)_3$	200—250	2.437
KBr	42—65	2.748	$NaHCO_3$	42—65	2.220
KI	42—65	3.125	CMC-Na	42—65	1.551
NaCl	42—65	2.165	Aspirin	42—65	1.394
NaBr	42—65	2.609	Phenacetin	42—65	1.237
NH_4Cl	42—65	1.538	Na-stearate	unsieved	1.112
NH_4Br	42—95	2.436	Ca-stearate	unsieved	1.100
Potato starch	200—250	1.464	Aniline-HCl	unsieved	1.209
Lactose	42—65	1.537	Aniline-HBr	unsieved	1.648
Sucrose	42—65	1.595	Aniline-HI	unsieved	1.882
Acacia	200—250	1.489			

Results and Discussion

When the powders shown in Table I are compressed at the constant rate of displacement of upper punch, they are divided into the following three classes according to the patterns of changes of the pressure-transmission-ratio with the increase of upper punch pressure.

1. Powders Behaving Similarly to the Potassium Chloride Powder (KCl Type)

When KCl, KBr, KI, NaCl, NaBr, NH_4Cl, NH_4Br and potato starch powder are compressed at the constant rate of displacement of upper punch equal to 0.332 mm/sec, the relationships of the pressure of upper punch and the pressure-transmission-ratio to the thickness of powder bed are shown in Fig. 1, where the values of pressure-transmission-ratio are plotted for the range of upper punch pressure higher than 30 kg/cm^2. It is clear that the values of pressure-transmission-ratio take the maximum at halfway for these powders. Besides, the values of the thickness of powder bed at these maximum points are nearly equal, although

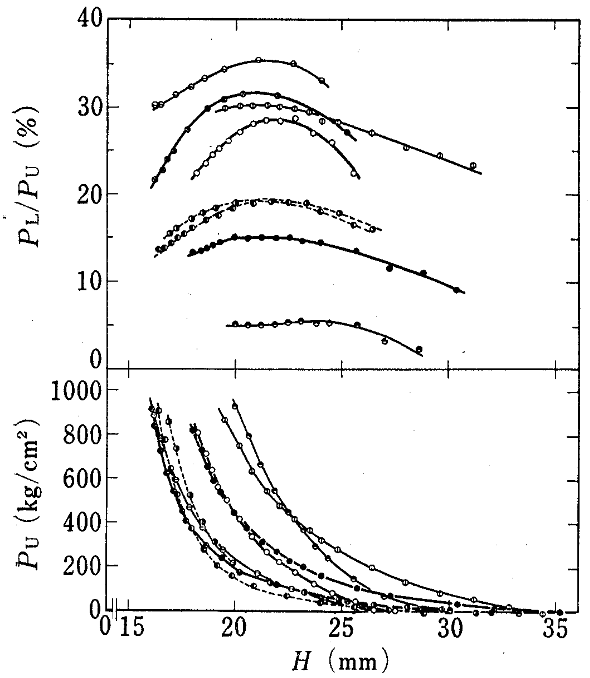

Fig. 1. Relationship between the Thickness of Powder Bed (H) and Upper Punch Pressure (P_U) or Pressure-Transmission-Ratio (P_L/P_U, P_L: Lower Punch Pressure) when Compressed at the Constant Rate of Displacement of Upper Punch Equal to 0.332 mm/sec

net volume (cm^3): 0.7545, H (mm) at the porosity equal to zero: 14.87

—○—: KCl, —●—: KBr, —⊗—: KI, —◑—: NaCl
—⊖—: NaBr, ---◑---: NH_4Cl, ---◐---: NH_4Br,
—◓—: potato starch

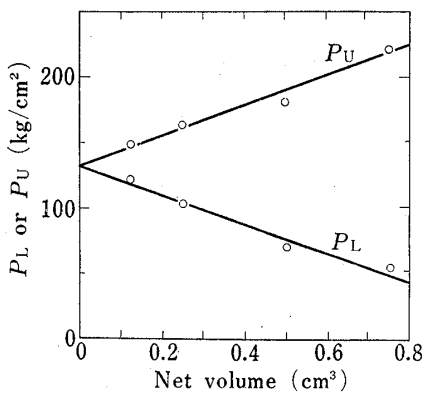

Fig. 2. Upper or Lower Punch Pressure at the Maximum of Pressure-Transmission-Ratio in the Case of Potassium Chloride Powder

Table II. Porosity (%) at the Maximum of Pressure-Transmission-Ratio

Material	Net volume (cm^3) 0.1258	0.2515	0.3773	0.5030	0.7545	Mean
KCl	34.2	32.0	—	32.2	32.8	32.8
KBr	35.2	29.6	—	31.2	29.5	31.4
KI	31.0	30.9	—	30.0	29.3	30.3
NaCl	33.5	29.8	29.0	28.5	29.7	30.1
NaBr	—	—	—	—	30.4	—
NH_4Cl	35.0	33.6	—	34.3	31.8	33.7
NH_4Br	33.1	30.5	—	32.0	32.2	32.0
Potato starch	33.8	32.2	—	33.4	35.7	33.8

total mean; 31.9%

Table III. Upper and Lower Punch Pressure (kg/cm^2) at the Maximum of Pressure-Transmission-Ratio

Material		Net volume (cm^3) 0.1258	0.2515	0.3773	0.5030	0.7545	A[a)]	Mean[b)]
KCl	P_U	149	164	—	181	220	134	132
	P_L	122	103	—	69	63	129	
KBr	P_U	117	178	—	201	323	83	95
	P_L	90	98	—	58	49	106	
KI	P_U	103	103	—	121	141	91	90
	P_L	83	71	—	56	44	88	
NaCl	P_U	359	418	507	569	589	335	330
	P_L	293	285	273	242	191	324	
NH_4Cl	P_U	63	64	—	74	120	44	49
	P_L	49	38	—	24	23	53	
NH_4Br	P_U	51	59	—	64	74	48	47
	P_L	40	35	—	20	14	45	
Potato starch	P_U	206	281	—	339	361	202	183
	P_L	140	116	—	51	20	163	

a) values extrapolated to zero net volume
b) mean of the two values of A

the values of the upper and the lower punch pressure are different with each sample powder.

In Table II, the values of porosity at the maximum of pressure-transmission-ratio are shown for each net volume of compressed powder. These values are almost constant without reference to material and net volume, taking the total mean value equal to 31.9%. This fact suggests that there is one of the causes in the state of particle packing for the values of pressure-transmission-ratio to take the maximum.

The upper and the lower punch pressure at the maximum of pressure-transmission-ratio values are plotted to the net volume for the potassium chloride powder in Fig. 2. The upper punch pressure decreases lineally and the lower increases lineally with the decrease of net volume, and the upper and the lower punch pressure take the nearly equal value, when they are extrapolated to the zero net volume.

For the powders belonging to KCl type, the upper and the lower punch pressure at the maximum of pressure-transmission-ratio and the values extrapolated to the zero net volume are shown in Table III. As the upper and the lower punch pressure at the maximum points take the different values according to the net volume, these values are not adequate as the characteristic pressure at the maximum points. Furthermore, the arithmetic mean of the upper and the lower punch pressure at the maximum points cannot represent the average state of powder bed at the maximum of pressure-transmission-ratio, because the distribution of axial pressure is not linear generally. But, the smaller is the net volume, the more exactly the arithmetic mean can represent the average states. Accordingly, the means of extra-polated values are most representative of the average states of powder bed at the maximum points.

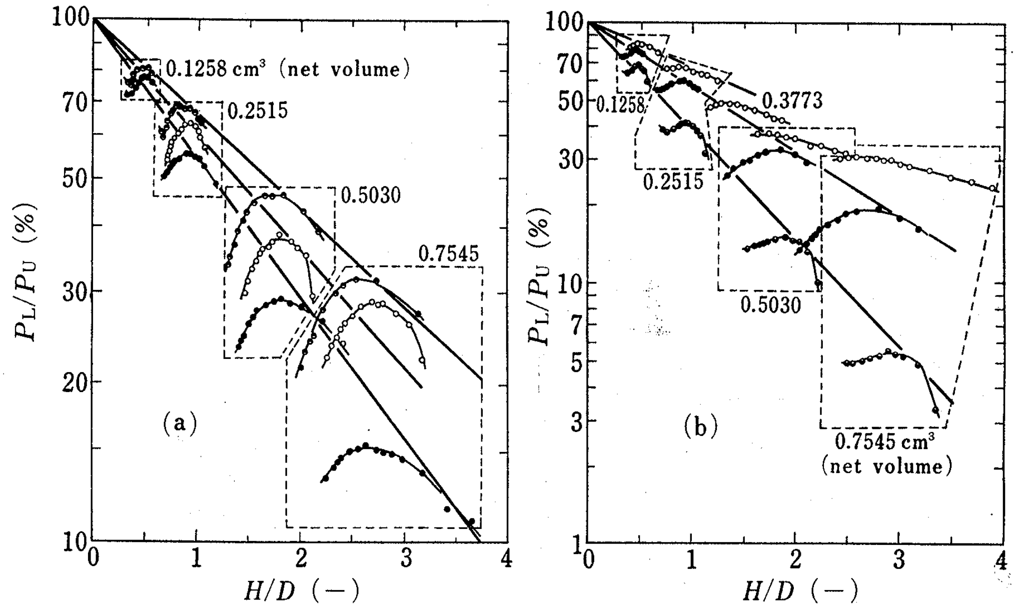

Fig. 3. Relationship between Common Logaritbm of Pressure-Transmission-Ratio and the Thickness Divided by the Diameter of Die (D) in the Case of KCl Type

(a) ○: KCl, ●: KBr, ◓: KI
(b) ○: NaCl, ●: NH_4Br, ◒: potato starch

In Fig. 3, the common logarithm of pressure-transmission-ratio (P_L/P_U) is plotted to the thickness (H) divided by the diameter of die (D), following the Janssen's equation;[4,5]

$$\ln (P_L/P_U) = -4\mu KH/D,$$

where μ is the coefficient of friction between the die wall and the powder, K is the ratio of the horizontal to the vertical pressure, P_L is the upper punch pressure and P_U is the lower punch pressure. As the data for the ammonium chloride powder fit closely to those for the ammonium bromide powder, they are not shown in Fig. 3. The data for each powder do not fit to one straight line, which passes 100% of pressure-transmission-ratio at the zero thickness, over all the range of pressure, since the ratio decreases with the decrease of thickness at the high pressure. Besides, in the cases of the sodium chloride and the potassium chloride powder, the values of pressure-transmission-ratio at the large net volume are influenced by the net volume. Although the values of pressure-transmission-ratio increase with the decrease of the thickness at the low pressure, the data do not necessarilly seem to fit to the Janssen's equation even within this range of pressure.

For the purpose of magnifing the accuracy of data in the range of pressure below the maximum point, the powder, the net volume of which is 4.0243 cm³ corresponding to 0.6303 of H/D at the zero porosity, is compressed in the cylindrical die of 20.11 mm inside diameter. In this case, the compressions are made at the rate of displacement of upper punch equal to 0.820 mm/sec so as to coincide the rate of increase of strain with that in the case of 8.02 mm inside diameter. The results showed that in the cases of potassium bromide, potassium

4) H.A. Janssen, *Z.-V.D.I.*, **39**, 1045 (1895).
5) K. Schneider, *Chemie Ing. Techn.*, **41**, 51 (1969).

iodide, ammonium chloride and ammonium bromide powder the data in the range of pressure below the maximum points fit to the Janssen's equation. These powders have such the tendency that the rate of increase in the upper punch pressure below the maximum points is much smaller than the others as shown in Fig. 1.

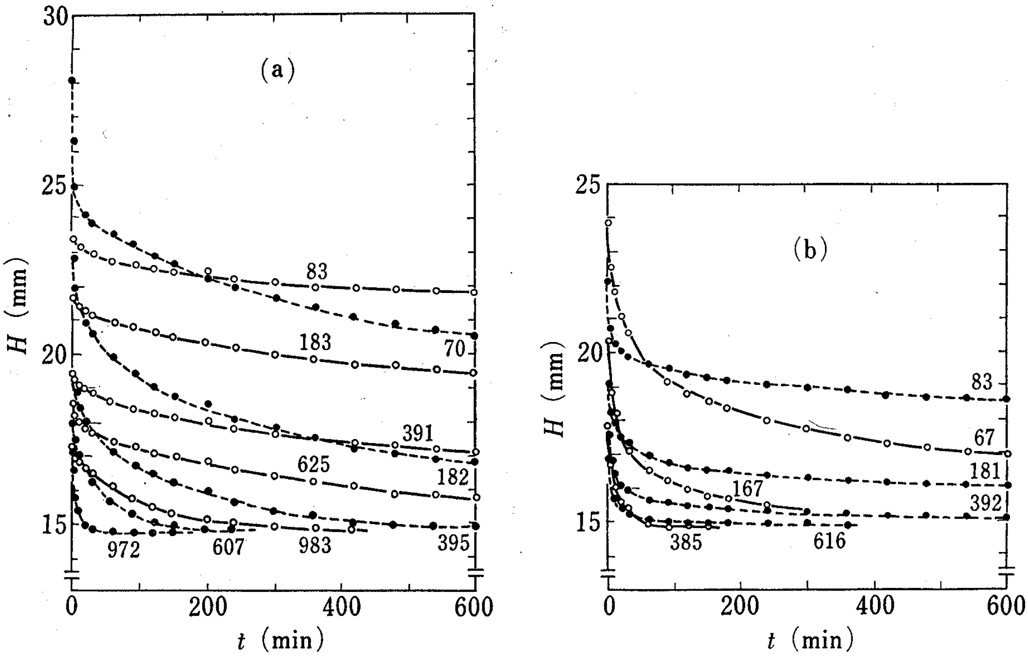

Fig. 4. Relationship between the Thickness of Powder Bed and Compressing Time when 0.7545 cm³ of Net Volume is compressed under the Constant Upper Punch Pressure

(a) —○—: KCl, ---●---: KBr (b) —○—: KI, ---●---: NH_4Br
The numbers in Fig. show the upper punch pressure (kg/cm²).

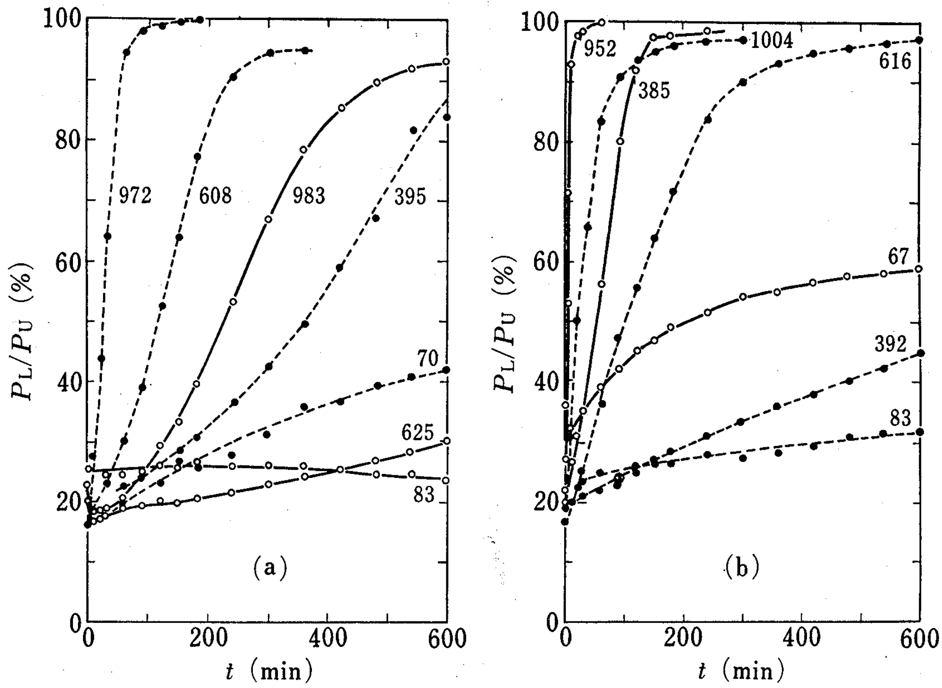

Fig. 5. Relationship between Pressure-Transmission-Ratio and Compressing Time when 0.7545 cm³ of Net Volume is compressed under the Constant Upper Punch Pressure

(a) —○—: KCl, ---●---: KBr (b) —○—: KI, ---●---: NH_4Br
The numbers in Fig. show the upper punch pressure (kg/cm²).

When the powders shown in Fig. 1 (except for NaBr powder) are compressed continuously under the constant pressure of upper punch reached finally by the compression at the constant rate of displacement of upper punch, only in the cases of potassium bromide, potassium iodide and ammonium bromide, the values of porosity and pressure-transmission-ratio tend to approach to zero and 100%, respectively, as well as in the case of potassium chloride powder.[3] The results are shown in Fig. 4 and 5. It is clear from Fig. 4 and 5 that the creep flow is easy in the order of potassium iodide, ammonium bromide, potassium bromide and potassium chloride. Although the ammonium chloride powder behaves similarly to ammonium bromide powder in the case of the compression at the constant rate of displacement, it does not tend for the porosity and the pressure-transmission-ratio to approach to zero and 100%, respectively. As the time scale of these phenomina is far larger than that in the case of the compression at the constant rate of displacement of upper punch equal to 0.332 mm/sec, it is difficult to discuss directly the relations between Fig. 1 or 3 (compression at the constant rate of displacement of upper punch) and Fig. 4 or 5 (compression for ten hours under the constant upper punch pressure).

2. Powders Behaving Similarly to the Lactose Powder (Lactose Type)

When the lactose, sucrose, aluminium hydroxide and acacia powder are compressed at the constant rate of displacement of upper punch, the results are plotted in Fig. 6, following the Janssen's equation. For these four powders, the values of pressure-transmission-ratio decrease at the large net volume with the increase of upper punch pressure contrary

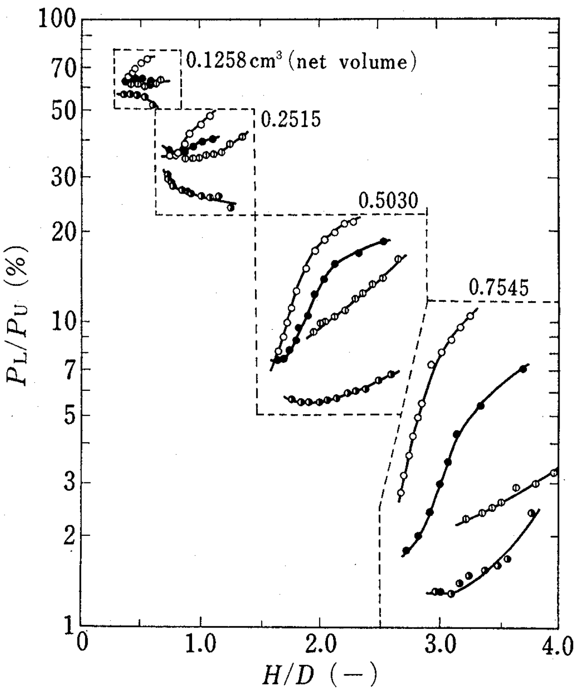

Fig. 6. Relationship between Common Logarithm of Pressure-Transmission-Ratio and the Thickness Divided by the Diameter of Die (D) in the Case of Lactose Type

●: lactose, ○: sucrose, Φ: $Al(OH)_3$, ◑: acacia

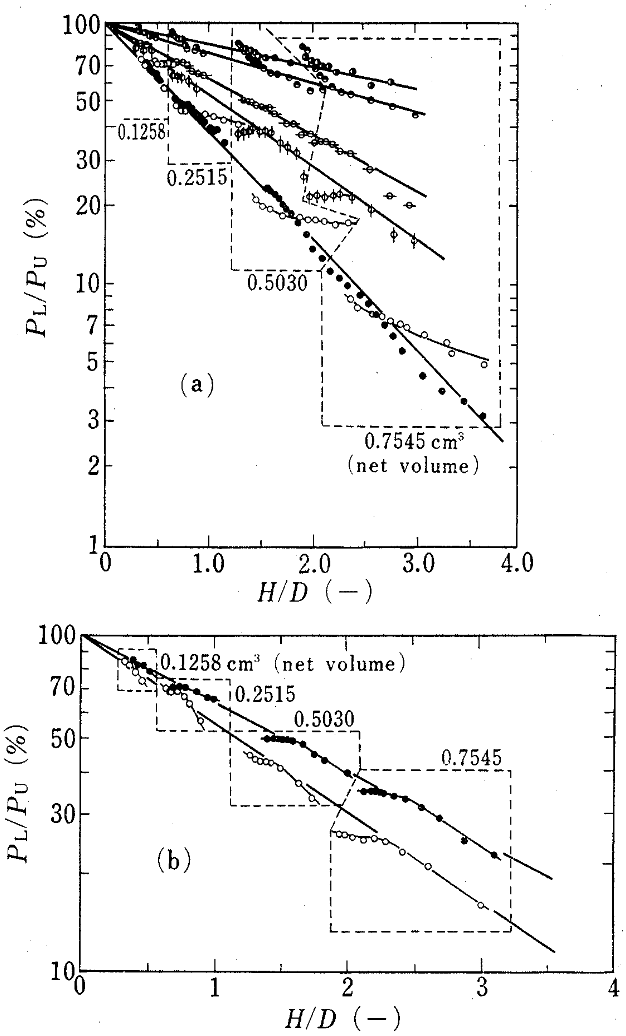

Fig. 7. Relationship between Common Logarithm of Pressure-Transmission-Ratio and the Thickness Divided by the Diameter of Die (D) in the Cases of Powders not Belonging to KCl and Lactose Type

(a) ○: CMC-Na, ●: $NaHCO_3$, Φ: aspirin, ⊖: phenacetin, ◑: Na-stearate, ◓: Ca-stearate
(b) ○: aniline-HCl, ●: aniline-HBr

to the Janssen's equation. At the small net volume, the tendency of decrease becomes to be not remarkable except for the sucrose powder.

When these powders are compressed continuously under the constant pressure of upper punch, they show the same apparent creep properties as in the case of lactose powder.[3)]

3. The Other Powders

Each powder, the results for which are shown in Fig. 7, has the different tendency from that of the potassium chloride and lactose powder. (As the data for aniline hydroiodide powder fit closely to those for aniline hydrobromide powder, they are not shown in Fig. 7.) That is, in the cases of these powders, the values of pressure-transmission-ratio do not decrease with the increase of upper punch pressure. For the aspirin, phenacetin and three aniline salts powders, the values of pressure-transmission-ratio tend to become almost constant at the high pressure of upper punch, that is different from the value of the potassium chloride powder. The results for sodium bicarbonate powder fit to the Janssen's equation over all the range of these measurements. In the cases of sodium and calcium stearate powder, which are used as the lubricants, the values of pressure-transmission-ratio are very large and besides increase remarkablly at the high pressure of upper punch with the increase of upper punch pressure. It can be said that the sodium and calcium stearate powder have very adequate properties as the lubricants.

When the powders shown in Fig. 7 are compressed under the constant pressure of upper punch, only three aniline salts behave similarly to the potassium chloride powder, and others behave similarly to lactose powder.

Conclusion

From the patterns of changes of pressure-transmission-ratio with the increase of upper punch pressure, when the powders are compressed at the constant rate of displacement of upper punch equal to 0.332 mm/sec, the powders are divided into three classes, as followings;

(1) KCl type, for which the values of pressure-transmission-ratio take the maximum at half way.

(2) Lactose type, for which the values of pressure-transmission-ratio decrease monotonously.

(3) The other powders, for which the values of pressure-transmission-ratio do not decrease.

In the cases of the powders belonging to KCl type, the values of porosities at the maximum points of pressure-transmission-ratio are almost constant (about 32%) without reference to material and net volume, although the characteristic pressures at the maximum points are different among these powders.

For the sodium bicarbonate powder belonging to the third class, the results agree fairly well with the Janssen's equation over all the range of measurements, although for the other powders in this experiment the results agree with the Janssen's equation within only a part of range of upper punch pressure at the best.

When the powders are compressed continuously under the constant pressure of upper punch, only the potassium bromide, potassium iodide, ammonium bromide and aniline salts (hydrochloride, hydrobromide and hydroiodide) powders have the same apparent creep properties as in the case of the potassium chloride powder, that is, the values of porosity and pressure-transmission-ratio tend to approach to zero and 100%, respectively.