

Electrical Conductance of Directly Compressible Materials under Pressure

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Abstract □ An electrometer that allowed direct recording of resistance changes during compression was used to measure the conductances of sodium chloride, potassium chloride, ammonium chloride, and sodium citrate. The effects of initial particle size, lubricant level, moisture content, and compaction pressure were determined. Direct compression tableting diluents also studied were dextrose, sucrose, lactose, microcrystalline cellulose, and dibasic calcium phosphate. Distinctive conductance patterns were observed and are rationalized in terms of previously proposed bonding mechanisms.

Keyphrases □ Electrical conductances—various directly compressible inorganic chlorides and sodium citrate, effects of particle size, lubricant levels, moisture content, and compaction pressure □ Directly compressible materials—various inorganic chlorides and sodium citrate, electrical conductances, effects of particle size, lubricant level, moisture content, and compaction pressure □ Tablets—various directly compressible inorganic chlorides and sodium citrate, electrical conductances, effects of particle size, lubricant level, moisture content, and compaction pressure

This study was designed to evaluate measurement of the electrical conductance of directly compressible materials under pressure as a technique for investigating interparticulate bonding in compacts. The application of electrical resistivity measurements as a tool for studying applied and induced stresses in solid materials has been recognized (1–6) but is still incomplete. This technique has had only limited consideration as a means for investigating tableting.

BACKGROUND

Freshly prepared and aged silver bromide powders were compressed between two silver-plated plungers, which served as electrodes, and specific conductance was measured as a function of applied pressure (1). Specific conductance fell as silver bromide was aged (1). Electrical conductance also was used to describe sintering of powdered metals, and the effects of varying particle size and size distribution on copper powder sintering were noted. A relationship between tensile strength and electrical conductivity was developed and served to evaluate the quality of sintered copper (2).

The bonding in sodium chloride and copper sulfate compacts was studied by measuring electrical conductivity as a function of pressure, using 500–600 v ac (3). The specific conductivity increased with an increase in pressure up to a maximum value and then decreased with further increases in pressure (~1750 kg/cm²) (3).

In another study (4), a decade bridge was used to measure the electrical resistance of sodium chloride granules containing 8–10% graphite at low pressures (~70 kg/cm²). It was concluded that an initial packing stage was followed by a brief period during which the granular structure could support the imposed load without breakdown and then, finally, gradual failure of the granules to give a compact mass.

A modified Schering bridge circuit was used to measure the dissipation factor of sodium chloride undergoing compression in a polyester polymer die (5, 6). Hanus (5) correlated physical properties of sodium chloride crystals such as particle size, moisture content, porosity, and hardness of the compacts produced with calculated specific conductances at various pressures. Mohrle (6) studied the effect of addition of magnesium stearate and activated charcoal at a fixed pressure on the specific conductivity of sodium chloride.

In the present study, an electrometer that allowed direct recording of resistance changes during compression was used to measure the con-

ductances of sodium chloride, potassium chloride, ammonium chloride, and sodium citrate. The effects of initial particle size, lubricant level, and moisture content as well as compaction pressure were determined. To establish the value of applying the technique to other than ionic materials, several direct compression tableting diluents and binders also were included.

EXPERIMENTAL

Materials—USP grade sodium, potassium, and ammonium chlorides and sodium citrate (dihydrate) were fractionated using 20-, 30-, 40-, and 50-mesh U.S. standard screens. The sized chlorides were dried at 135° for 16 hr to remove all traces of moisture and other volatile impurities. The samples were stored in sealed containers in a desiccator over silica gel until used.

Moisture was added to samples of dried 30–40-mesh salt by directing moist air at the material tumbling in a coating pan for 15 min. Samples with different moisture levels were prepared by tumbling the damp material in a dry coating pan for varying times. The moisture content was determined gravimetrically.

In experiments where magnesium stearate was added, portions of the dried 30–40-mesh salts were blended with 0.1, 0.2, 0.5, and 0.75% magnesium stearate. The lubricant was dried for 7 days at 65° and passed through a 200-mesh screen before use. The lubricated salts were stored in sealed containers in a desiccator until needed.

The following materials were used as received: spray-dried lactose¹, microcrystalline cellulose², anhydrous dextrose³, polyethylene glycol 4000⁴, dibasic calcium phosphate dihydrate⁵, and sucrose⁵.

Instrumentation—A schematic diagram of the system used to measure the electrical resistance of materials under compression is shown in Fig. 1.

The base of a 2.5-cm diameter tablet punch was cemented to a square acrylic⁶ block with an epoxy adhesive. A steel collar placed over the base of the punch was fastened to the acrylic block for added support. A banana plug was welded perpendicularly to the middle of the tablet punch shaft. A cylindrical steel shield, 12.5 cm in diameter, was fastened to the acrylic block with metal brackets so that the banana plug was positioned opposite to a circular window in the shielding. This upper punch unit was fastened to the upper stage of a hydraulic press⁷ by large C clamps.

A 15 × 5 × 1-cm polished stainless steel plate resting on the lower stage of the hydraulic press served as the lower punch. The die was fabricated from a 5-cm diameter, 2-cm thick piece of polyethylene terephthalate⁸ ring, containing a 2.5-cm diameter cavity. It was positioned in the center of the steel plate to accommodate the sample. The acrylic block and polyethylene terephthalate die served to insulate the compacts from the grounded hydraulic press.

A motorization unit⁷ was used to control the application of pressure and the movement of the ram, whose speed could be varied from 0.1 to 0.9 cm/sec. The unit, when activated, drove the press to a preset pressure. If a fall in pressure occurred during the experimental run, the motorized unit was automatically activated to maintain the required pressure.

The shield and steel base plate were connected to the grounded hydraulic press. A shielded cable connected the punch to an electrometer⁹. The output of the electrometer was monitored continuously throughout the experimental run by a recorder¹⁰.

¹ Foremost-McKesson, Appleton, Wis.

² Avicel Ph-101, FMC Corp., Philadelphia, Pa.

³ J. T. Baker Chemical Co., Phillipsburg, N.J.

⁴ Ruger, New Brunswick, N.J.

⁵ Fisher Scientific Co., Springfield, N.J.

⁶ Lucite.

⁷ Model C, Fred S. Carver, Menomonee Falls, Wis.

⁸ Mylar.

⁹ Model 610 C, Keithley Instruments, Cleveland, Ohio.

¹⁰ Model 620, Leeds and Northrup, North Wales, Pa.

Table I—Reproducibility of Results Showing the Effect of Compression Time on the Conductivity of Dried 30–40-Mesh Sodium Chloride at 2700-kg Applied Load

Minutes	Apparent Specific Conductivity, $\text{ohms}^{-1} \text{cm}^{-1} \times 10^7$		
	Run 1	Run 2	Run 3
0	7.01	7.21	7.09
2	6.60	6.56	6.63
5	6.07	6.14	6.16
10	5.18	5.38	5.28
15	4.45	4.52	4.64
20	3.82	3.63	3.70
25	3.28	3.08	3.21
30	2.81	2.83	2.88

Procedures—A weighed quantity of material was transferred into the die resting on the steel plate. The lower stage of the hydraulic press was raised manually so that the tablet punch was positioned in the die cavity to rest on the surface of the granulation. The sample thickness was determined with a cathetometer¹¹, which was set to follow the movement of the base plate. The motorized unit was activated, and the electrometer and recorder were turned on.

The resistance at the moment the pressure reached its maximum value was designated as the zero-time reading. The thickness of the sample was again determined after 30 min, and corrections were made for elastic deformation of the insulating acrylic block. The specific conductance was calculated from resistances recorded at selected times during the run by:

$$\text{specific conductance} = \frac{\text{compact thickness}}{(\text{resistance})(\text{punch area})} \quad (\text{Eq. 1})$$

The accuracy of the measurements was checked by connecting standard resistors (1–100 megohm $\pm 1\%$) between the punch and plate. Resistances in excess of 10^{12} ohms were recorded in the empty die, depending on the distance set between the punch and plate. The range of measurement of the present equipment was limited to a maximum of 10^{10} ohms. All results reported are averages of replicated runs.

All reported results are for experiments run by operating the motorized unit at the lowest practical speed of 0.1 cm/sec with 6-g samples. Preliminary experiments indicated that initial observed conductance values were higher at lower operating speeds but approached the same values at 30 min. The observed specific conductances increased with an increase in weight up to 6 g; a further increase in weight resulted in a decline in specific conductance. Six grams was the maximum weight possible for most materials owing to the limitation imposed by the size of the die.

The reproducibility of experimental data is illustrated by the data

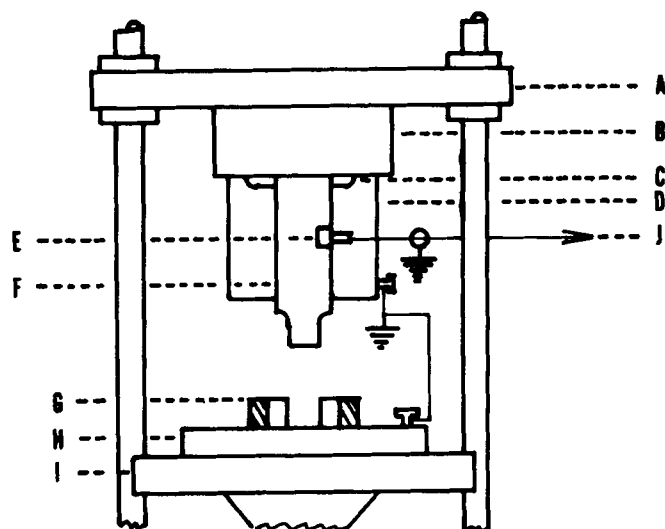


Figure 1—Schematic diagram of system used for measurement of conductance under load. Key: A, upper stage of hydraulic press; B, acrylic block; C, steel collar; D, steel shield; E, connection to electrometer; F, punch; G, polyethylene terephthalate die; H, steel base plate; I, lower stage of press; and J, shielded cable to electrometer.

Table II—Effect of Reduction in Applied Load (3600 kg) on Specific Conductances ($\text{ohms}^{-1} \text{cm}^{-1} \times 10^7$) and Thicknesses of Compacts Produced from 30–40-Mesh Salts

Salt	Initial Specific Conductance at 3600 kg	Specific Conductance at ~ 50 kg		Increase in Thickness, %
		0 min	30 min	
Sodium chloride	4.9	0.65	0.35	4.1
Potassium chloride	12.2	5.5	6.5	4.3
Ammonium chloride	1.9	0.9	1.2	2.7
Sodium citrate	0.1	0.2	0.3	0.67

summarized in Table I, which shows observed changes in specific conductance of sodium chloride compacts held at an intermediate pressure for three separate runs.

RESULTS AND DISCUSSION

The specific conductance of a pure sodium chloride crystal was reported to be $3.4 \times 10^{-17} \text{ohm}^{-1} \text{cm}^{-1}$ at room temperature (7). Most of this ionic conductance arises from valence vacancies. Although crystals possessing perfect lattices would conduct no current (8), all crystalline materials possess some electrical conductivity due to structural defects and impurities.

The conductance of a polycrystalline mass is the result of a complex interaction of factors, including the kind and concentration of impurities, lattice defects, structural defects such as cracks and fractures, and contact area. Most of the current flow takes place at the interface between the particles making up the mass. Thus, the greater the number of contact points, the greater the observed conductance.

Particles in a compact are forced into intimate contact under pressure, resulting in extensive areas of true contact. Particle rearrangements, fracturing, and plastic and elastic deformations take place. Consolidation processes predominate at low pressures, whereas deformations predominate at high pressures (9). The net effect of pressure during the compaction process should be an increase in conductivity up to the point where plastic deformation becomes significant. Interparticulate bonding reduces the interfacial contact area and, thus, the number of paths for current flow. The observed decrease in conductance is a consequence of healing and stress relaxations as well as bonding.

Interpretation of the conductances measured under the experimental conditions is further complicated by differences arising from ion character, variations in impurity content, and local temperature changes during compaction.

Conductivity at Constant Pressure—Studies of bonding in sodium

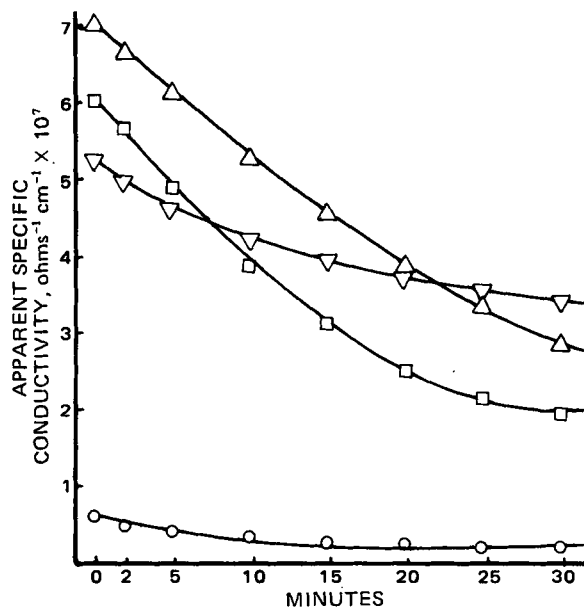


Figure 2—Specific conductivity of dried 30–40-mesh sodium chloride at 900 (O), 1800 (□), 2700 (Δ), and 3600 (▽) kg.

¹¹ Central Scientific Co., Chicago, Ill.

Table III—Effect of Initial Particle Size on Specific Conductances (ohms⁻¹ cm⁻¹ × 10⁷) and Densities of Salt Compacts

Salt	Mesh	Density	900 kg		Density	2700 kg	
			Specific Conductance			Specific Conductance	
			0	30 min		0	30 min
Sodium chloride	30-40	1.65	0.62	0.22	1.865	7.0	2.8
	40-50	1.596	3.0	0.05	1.835	4.7	0.93
Potassium chloride	20-30	1.60	5.7	1.5	1.865	17.2	9.8
	30-40	1.59	3.1	1.6	1.84	13.3	8.6
Ammonium chloride	40-50	1.58	1.6	0.08	1.85	7.9	6.5
	20-30	1.40	0.44	4.1	1.53	2.7	0.46
Sodium citrate	30-40	1.39	0.05	0.4	1.52	1.3	0.87
	40-50	1.38	0.14	0.29	1.50	0.28	1.16
Sodium citrate	20-30	1.41	0.008	0.038	1.51	0.009	0.033
	30-40	1.40	0.006	0.038	1.49	0.009	0.03

and potassium chloride in contrast to potassium citrate compacts suggest that consolidation occurs primarily by elastic and plastic deformation in the former case and by fragmentation and particle rearrangement in the latter case (10). Figures 2-5 show specific conductivity-time plots for 30-40-mesh sodium, potassium, and ammonium chlorides and sodium citrate at gauge pressures of 900-3600 kg. Zero time on these plots represents the point at which the applied pressure was maintained at a constant value.

Distinctive patterns were observed for each material. In both sodium and potassium chloride compacts, the conductance decreased with time, and the decrease was proportionally less for low, as compared to high, pressures. Cole *et al.* (10) reported that potassium chloride deformed at lower loads than did sodium chloride. This behavior was evident from the fact that the maximum initial conductance was approached at lower pressures with potassium chloride. The decrease in conductance at lower pressures suggests that more complete deformation, fracture, and bonding of crystals take place and that more pathways for current flow are destroyed in potassium chloride. The order of magnitude of observed conductances was potassium > sodium > ammonium chloride > sodium citrate.

Unlike sodium and potassium chlorides, the conductance of ammonium chloride (Fig. 4) increased with time under constant load except at the highest pressure. At the intermediate pressure, a distinct maximum was observed. It was suggested that ammonium chloride deforms by plastic flow (11), with little evidence of fracture and fragmentation. The increase in conductance is likely due to an increase in the number of interparticulate contact points as material flows into interparticulate space.

Sodium citrate (Fig. 5) showed a constant initial conductance, irrespective of the pressure employed to form the compact. Unlike ammo-

niun chloride, the rate of increase in conductance with time was greatest at lower pressures. Consolidation of sodium citrate is the result of fragmentation, which takes place on the initial application of the minimal load necessary to exceed the yield value of the salt. A constant initial conductance value is observed since a fixed number of contact points is made in the powder bed. The results suggest that the degree of fragmentation is constant, irrespective of the applied load. The observed trend to increased conductance with time probably reflects particle rearrangements resulting in increased numbers of contact points.

Table II summarizes data obtained in experiments in which the applied load was relaxed from its initial value to about 45 kg. The load was held at the initial value for 1 min. If compacts show significant elastic recovery, which results in a reduction in stress bonds and the effective area of contact, a reduction in conductance is observed. This reduction was evident with the chlorides; however, an order of magnitude increase in conductance was noted for sodium citrate. Sodium chloride showed a significant decrease in conductance, which further drifted downward with time. The proportionally smaller decreases observed with potassium and ammonium chlorides tended to increase with time. Increases in compact thickness were observed upon release of load. These increases were not significant for the citrate salt, while the sodium and potassium chlorides showed a significantly greater elastic recovery than ammonium chloride. The results reported for sodium chloride are in agreement with observations on sodium chloride tablets (12).

Potassium chloride showed intermediate characteristics, resembling sodium chloride at low loads and ammonium chloride at high loads. Unlubricated ammonium chloride produced translucent compacts even at the lowest applied loads, indicative of plastic deformation. At the

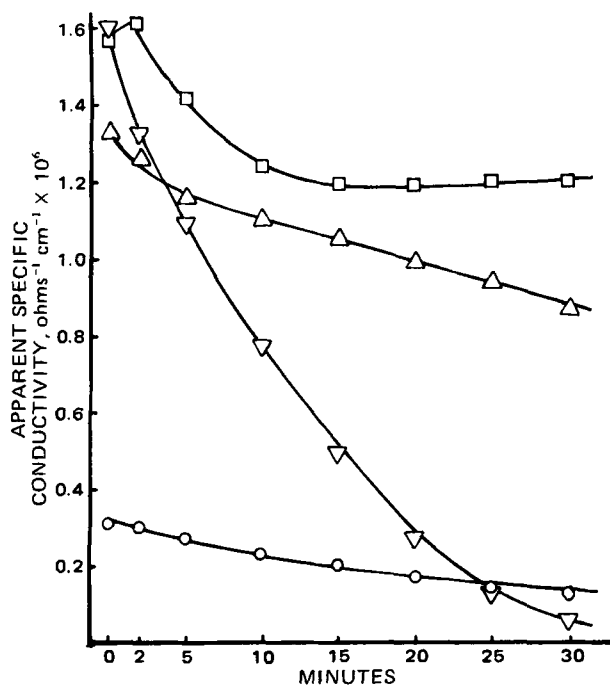


Figure 3—Specific conductivity of dried 30-40-mesh potassium chloride at 900 (O), 1800 (□), 2700 (Δ), and 3600 (▽) kg.

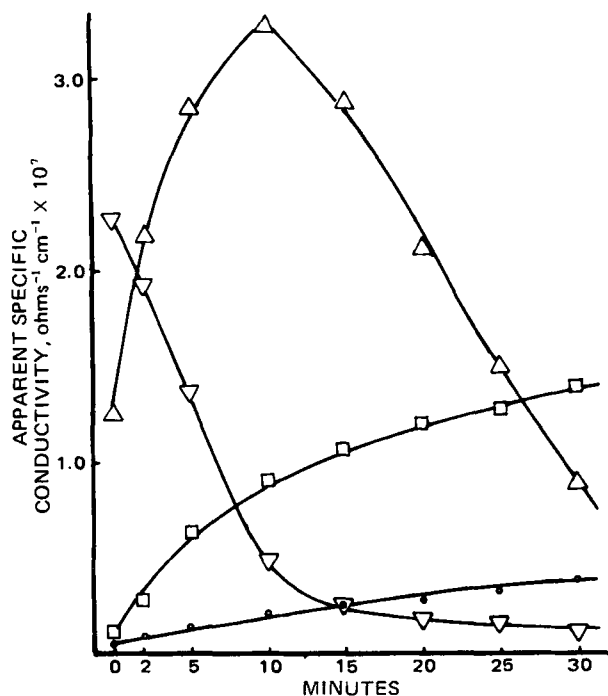


Figure 4—Specific conductivity of dried 30-40-mesh ammonium chloride at 900 (O), 1800 (□), 2700 (Δ), and 3600 (▽) kg.

Table IV—Effect of Magnesium Stearate on Specific Conductances ($\text{ohms}^{-1} \text{cm}^{-1} \times 10^7$) of Compacts Produced from 30-40-Mesh Salts

Salt	Magnesium Stearate, %	1800 kg		3600 kg	
		0	20 min	0	20 min
Sodium chloride	0	6.0	2.5	5.2	3.7
	0.1	1.35	0.05	1.9	0.96
	0.75	1.40	0.22	1.63	0.65
Potassium chloride	0	15.8	12.1	16.0	2.8
	0.1	0.77	2.03	28.6	16.9
	0.75	0.38	1.13	4.96	0.61
Ammonium chloride	0	0.11	1.22	2.3	0.17
	0.1	0.01	0.07	0.02	0.014
	0.75	0.007	0.05	0.01	0.015

highest loads, potassium chloride compacts tended to resemble more closely the ammonium chloride compact at low pressure than the more granular-like structure of the sodium chloride compact. Sodium citrate compacts produced at even the highest loads crumbled under mild stress, indicating a lack of significant bonding.

Effect of Initial Granule Size—Huffine (3) and Hanus (5) previously reported that the specific conductance of sodium chloride compacts under pressure tended to increase as the initial granule size was increased. These observations were generally confirmed by the data summarized in Table III, except for sodium chloride at low loads and sodium citrate, where conductance was independent of initial granule size. Differences in observed compact densities were not significant except for sodium chloride at the low load, where a less dense compact was attained with 30-40-mesh sodium chloride as compared to the 40-50-mesh granules. Larger granules are subject to greater deformation, resulting in more fractures and defects and, consequently, more paths for current flow.

Effect of Added Magnesium Stearate—Table IV summarizes the observed conductances of compacts containing added magnesium stearate. Figure 6 shows the anomalous results observed in potassium chloride compacts at 3600-kg applied load. In all other cases, addition of magnesium stearate reduced the specific conductance, with the greatest reduction seen at the lowest lubricant level studied. These results are in agreement with those reported by Mohrle (6) for sodium chloride compacts. The addition of even the lowest level of magnesium stearate significantly reduced bonding in salts at the lower applied loads. Compacts produced from lubricated ammonium chloride lacked the translucent structure characteristic of the unlubricated material.

Magnesium stearate potentially has two effects in compacts that can influence conductance. Principally, particles are coated with an insulating layer of the lubricant, which acts to decrease the effective conductance and to reduce bonding. In addition, the lubricant permits easier packing, producing compacts of maximum density with a minimum of particle deformation. The thickness of the lubricated compacts under pressure was somewhat smaller than the thickness of the unlubricated compacts but independent of the magnesium stearate level.

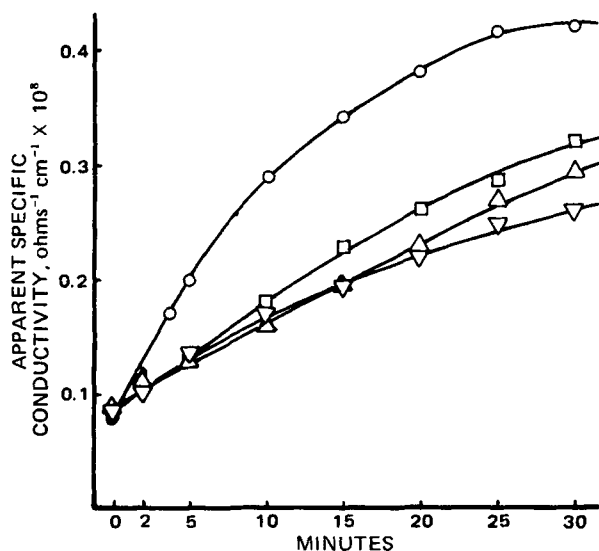


Figure 5—Specific conductivity of dried 30-40-mesh sodium citrate at 900 (○), 1800 (□), 2700 (Δ), and 3600 (▽) kg.

Table V—Effect of Added Moisture on Specific Conductances ($\text{ohms}^{-1} \text{cm}^{-1} \times 10^7$) of Compacts Produced from 30-40-Mesh Salts

Salt	Water, %	1800 kg		3600 kg	
		0	20 min	0	20 min
Sodium chloride	0	6.0	2.5	5.2	3.7
	0.023	4.74	0.73	5.0	2.48
	0.074	7.94	1.4	7.5	3.1
Potassium chloride	0	15.8	12.1	16.0	2.8
	0.035	3.7	5.5	5.1	4.3
	0.126	4.2	4.7	6.7	3.9
Ammonium chloride	0	0.11	1.22	2.3	0.17
	0.056	0.09	0.16	0.06	0.04
	0.158	0.06	0.09	0.09	0.06

The anomalous behavior evidenced by potassium chloride at the high loads, in which significant conductance increases were observed upon addition of 0.1% stearate, suggests that better packing, which resulted in more contact points, had the greater effect. Thus, a higher concentration of lubricant was necessary to sustain a conductance decrease. At the low pressures, while a decreased conductance was observed, the conductances increased rather than decreased with time.

Effect of Added Moisture—Table V summarizes observed conductances of compacts produced at 1800- and 3600-kg compressional forces from granulations containing low levels of added moisture. Figure 7 shows a representative run for ammonium chloride. The data indicate that as water is added, the conductance decreases and reaches a minimum value, after which an increase is observed. Similar conductance-time patterns were noted with sodium chloride. However, the large shifts in conductance observed with dry potassium and ammonium chlorides were absent after moisture was added.

Rees and Shotton (13), in their study of sodium chloride compaction, demonstrated that added moisture exerted a boundary lubricant effect, which facilitated consolidation. Dissolution of salts in moisture films resulting in "electrolyte solution" pathways should increase conductance. At the moisture levels studied, this increase was observed only in sodium chloride compacts. However, crystallization under pressure, which promotes the formation of salt bridges and the healing of defects, would lead to reduced conductance, an effect that should be most pronounced at the lowest moisture levels. The presence of salt bridges produced in this

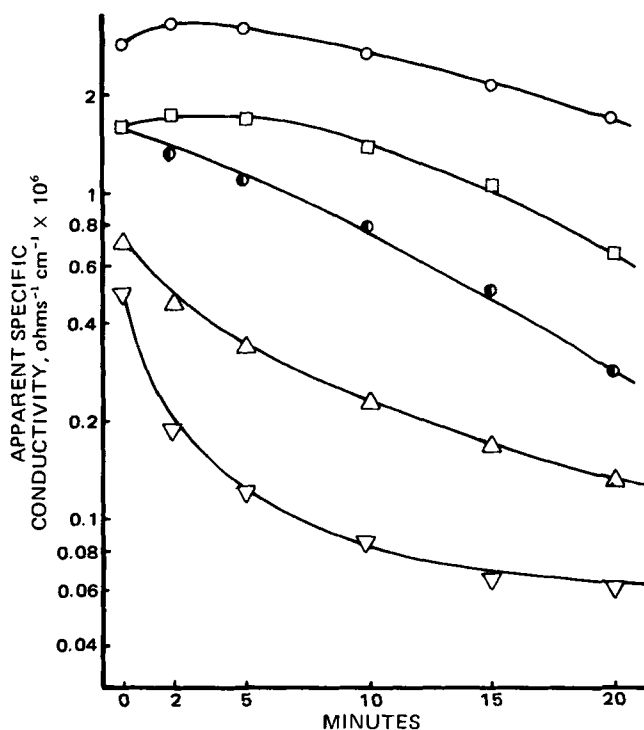


Figure 6—Effect of magnesium stearate on specific conductivity of dried 30-40-mesh potassium chloride under 3600-kg applied load. Key: ○, 0.0%; □, 0.1%; Δ, 0.2%; ▽, 0.5%; and ◇, 0.75% magnesium stearate.

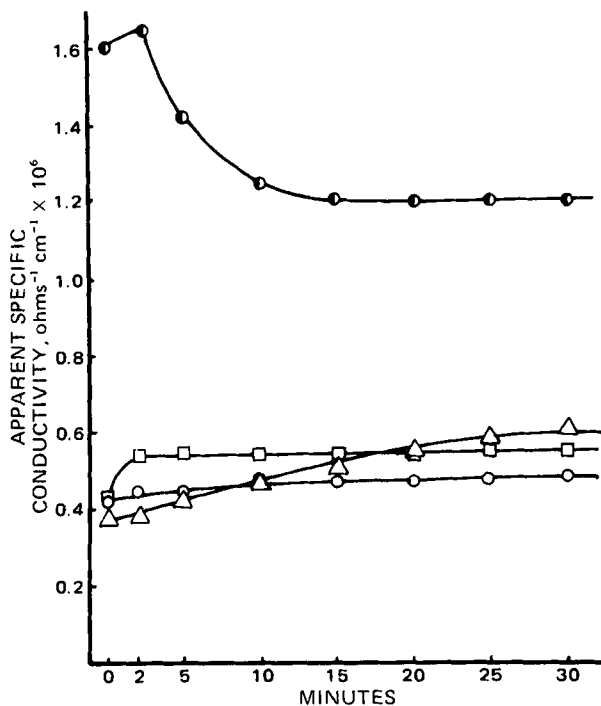


Figure 7—Effect of added moisture on specific conductivity of dried 30-40-mesh ammonium chloride under 1800-kg applied load. Key: ●, 0.0%; ○, 0.056%; □, 0.095%; and △, 0.158%.

fashion would result in a stable compact under pressure. This result seems to occur with the potassium and ammonium chloride compacts, since these salts are significantly more water soluble than sodium chloride.

Hanus (5) reported that the addition of moisture raised the conductance of sodium chloride at even the lowest levels added. However, sodium chloride compacts were viewed as leaky capacitors. The capacitance effects were more pronounced after the addition of moisture. In the present work, compacts are viewed as resistors—significant capacitance effects were absent under the conditions of measurement.

Direct Compression Tablet Diluents—Large changes in conductance characteristics of the chlorides were not observed except in the case of dibasic calcium phosphate dihydrate at 3600-kg pressure, which may have been the result of release of water of hydration (Table VI). Observed conductances were two to three orders of magnitude lower than the values obtained for the salts.

Compacts prepared from these diluents generally show stable physical characteristics on storage. For example, Horhota *et al.* (14) reported that dibasic calcium phosphate tablets stored under various conditions showed no changes in physical characteristics such as hardness. On the other hand, sodium chloride compacts showed considerable hardness increases on storage (5). A correlation between conductance and hardness was established (15). The data suggest that compacts that reach stable internal states rapidly are not affected by continuous application of pressure.

David and Augsburger (16) reported that in the times prevalent on a tablet press, plastic deformation was more predominant in microcrystalline cellulose as compared to dibasic calcium phosphate. Fracture, with the creation of new surfaces under compression, was more important with lactose and other sugars. No clear distinction could be made in interpreting the conductance patterns since polyethylene glycol, which bonds largely by plastic deformation, reached a stable conductance within 5 min whereas microcrystalline cellulose showed conductance increases at both high and low pressures and lactose showed increases in conductance at 1800-kg and decreases in conductance at 2700-kg loads. The lactose data

Table VI—Apparent Specific Conductances ($\text{ohms}^{-1} \text{cm}^{-1} \times 10^{10}$) of Direct Compression Diluents under Pressure

Diluent	Minutes	1800 kg	2700 kg	3600 kg
Dibasic calcium phosphate	0	7.0	9.1	1.2
	5	7.5	1.1	1.7
	30	6.7	1.1	2.3
Microcrystalline cellulose ^a	0	1.1	1.6	1.67
	5	1.16	1.6	1.7
	30	1.3	1.87	1.9
Spray-dried lactose	0	0.5	0.4	—
	5	0.38	0.5	—
	30	0.27	0.6	—
Sucrose	0	—	7.5	—
	5	—	7.53	—
	30	—	9.2	—
Dextrose	0	—	7.0	—
	5	—	7.3	—
	30	—	8.6	—
Polyethylene glycol 4000	0	—	67.6	—
	5	—	62.5	—
	30	—	62.0	—

^a Three-gram compacts.

are consistent with the view that fragmentation is the principal effect at low pressures while deformation resulting in increased bonding dominates at the higher loads. It should be emphasized that unlubricated materials of differing initial particle size are being compared.

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