Effects of Selected Variables on Salt Compression

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ABSTRACT

The use of pressure to consolidate loose salt into products of a more useful or salable configuration is common in the salt industry and is accomplished by batch or continuous compression.

These products must resist handling and shipping damages, withstand disintegration when stored under brine, present a salable appearance and obtain a high yield of usable product. Methods of measuring some of these parameters in terms of strength and density by use of laboratory testing are discussed.

Several variables, among which are applied pressure, moisture, additives, temperature, crystal structure, lubricants, dies, and time will affect the ability of a compressed salt to meet the required production and sales parameters. The effects of these variables on several operating problems are covered as they have been noted in laboratory testing and production operations in an effort to establish the degree of effectiveness of certain practices that have been accepted over a period of time.

INTRODUCTION

The purpose of compressing salt is to obtain a more salable product. Compression results in the cementation and/or fusion of individual salt particles into a mass consolidated to varying degrees. It is the purpose of this presentation to discuss methods of measuring these degrees and the variables that affect the process of consolidation.

Salt pressing is generally done in two ways. Continuously, as exemplified by forcing salt between two confined rolls where cementation is caused by "pinching" the salt into a continually smaller space. Rolls can be either smooth or pocketed and produce what is either compacted sheets or briquettes. Secondly, batch, as exemplified by placing salt into a die and applying pressure through means of a punch or punches to the confined salt. This unit process is typical of the blocks, but is also seen in canning tablets. The key difference between the two methods is the division of the pressing cycles. Batch consisting of filling, application of pressure, withdrawal of punch, ejection and preparation for refill, while continuous blends all steps.

As the salability of the product is the purpose of compaction, it is important that the factors influencing salability be specified. Other than price, which is in the hands of marketing, production must be concerned with four major factors:

1. *Resistance to Handling:* Products must be strong enough to prevent excessive breakage in packaging, screening, shipping and utilization. This capability will be referred to as "Dry Strength."
2. *Resistance to Wet Disintegration:* Products must resist the tendency, when stored under brine, to revert to unconsolidated crystals or "mushing." This capability will be referred to as "Wet Strength."
3. *Appearance:* Esthetic appearance should equal or better competition and ideally should resemble a crystal.
4. *Yield:* All pressed products ideally should yield 100% pressed product of the salt input, so that waste or recycle is held to a minimum. "Recycle" being defined as that salt which has passed through a pressing cycle and is not part of the yield. Yield is a factor of the dry strength at moment of pressing.

Measurement of these criteria has been rather subjective and efforts have been made to determine methods of objective measurement and to establish the effects of variables, especially in the laboratory, as a means of estimating effect on production without the cost of full scale testing.

PROCEDURES

Measurement of the two resistances has been accomplished by establishing the pressure required to fracture a pressed product. To insure reproducible results, a standard means of applying pressure was required and a unit was constructed (Fig. 1).

The testing unit consists of three pneumatic cylinders which permit a relatively wide range of pressures as areas of cylinders are on the ratios of 1:3:7. All points of pressure applications should be of same radius. In use, the pressed product is placed on the anvil and the pressure required to crush the product is measured. In the case of many small items, such as water softening buttons, all buttons in a sample are placed in turn on anvil and tested at a set pressure, recording the number of buttons crushed at that pressure. Pressure is then raised by a fixed increment and remaining buttons tested, the procedure being continued until all buttons are crushed. A weighted average is then obtained which is used as a measure of strength.

In the case of relatively large products, such as the laboratory test cylinders to be later described, a slightly different procedure is used. The product is placed on anvil and reducing valve is gradually opened, continually increasing pressure until product is crushed. The reading on valve gauge is then used to calculate the pressure required to crush product. The method selected is based only on ease of operation, but same procedure obviously should be used on all comparative products.

The testing unit is utilized to measure both resistance to handling and to wet disintegration and are expressed as "dry strength" and "wet strength" respectively; values are expressed in kilograms.

"Dry strength" is measured by testing the product under ambient conditions, while "wet strength" is measured after the product has been stored under saturated brine for a predetermined period of time. In these tests this has been standardized at three days, but any time can be used. Inasmuch as "wet strength" has been found to be dependent on time of exposure, time must, of course, be kept constant.

Since both these "strengths" can be shown to be directly correlated with mass, no attempt should be made to compare directly two products of widely different mass. In addition, wet strength is a function of dry strength. Therefore, a ratio is used in expressing many values which indicates the proportion of strength lost under brine storage.

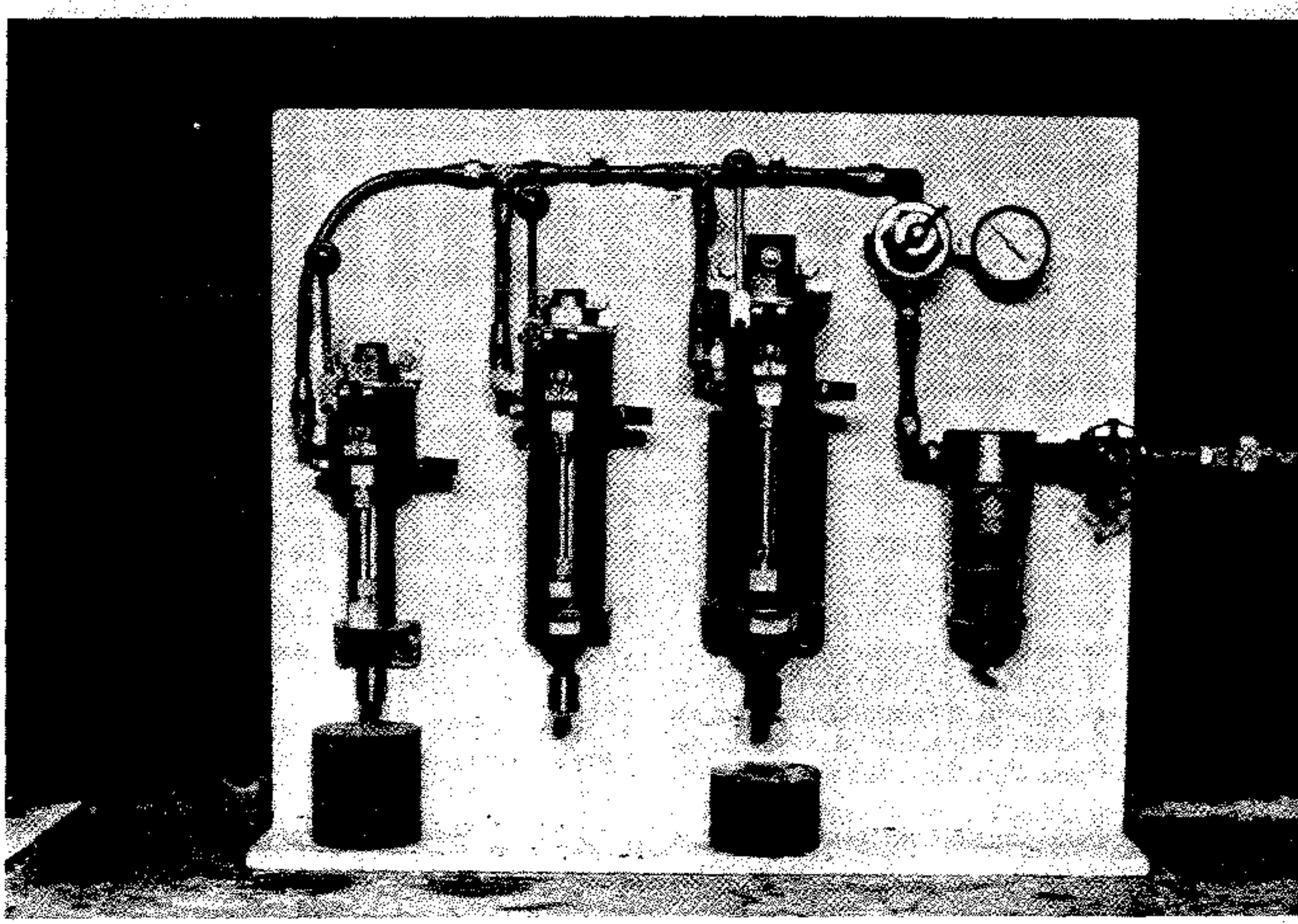

Figure 1. Photo of Pressure Tester.

$$\frac{\text{Dry Strength} - \text{Wet Strength}}{\text{Dry Strength}} = \text{Loss Ratio}$$

It is felt that such a value can offer a means of comparing two different products.

In order to evaluate the variables, it is also necessary to produce products under known conditions. This was done by developing a small die that could be used on a standard Carver laboratory press capable of developing 24,000 psi (10,909 Kg/cm^2). Die dimensions were selected to reproduce on a small scale the 50 lb. block proportions and to permit scaleup of pressures available on our block presses. On this basis, the taper of dies were scaled from actual block press dies as was diameter-length ratio; dimensions are given in Figure 2. Sufficient leeway was also allowed to permit mass variations.

Use of these test cylinders enables density of the product to be calculated from the dimensions. This has proven to be a valuable unit for comparison. In the case of an irregular product, namely water softener briquettes, etc., a method was developed using a scaled up pycnometer, which was made up on special order by the Arthur H. Thomas Company of Philadelphia, Pennsylvania, and consists of a 250 ml Erlenmeyer flask with a 34/45 ground glass joint, an extended pycnometer arm, a 34/45-10/30 reducer and a usual pycnometer thermometer (Fig. 3). The method follows the typical method of weighing under liquid to establish specific gravity. In this case, the liquid consists of saturated brine of measured gravity. The neck of the flask is such that relatively large buttons and pieces of compacted salt can be placed in bottle. The product is added to the flask (at least 100 grams) and followed by brine in usual manner, weighing and technique being similar to a standard pycnometer test. Brine should be added in such a manner to eliminate air bubbles. This method has proven valuable, as it permits a relatively large sample to be taken, overcoming, through averaging, some of the shortcomings of the method such as variations in liquid displacement of air voids. At this point, it should be emphasized that these methods are dependent in all cases on statistics. Obviously, variations can be expected to be considerable and the importance of sufficient samples cannot be stressed too strongly. No less than 10 buttons should be tested for strength and, for dependable results, a sample of 50 or so is preferred.

By use of these procedures, it has been possible to compare the effects of some variables on pressed products. Among the variables that were investigated are:

1. Pressure

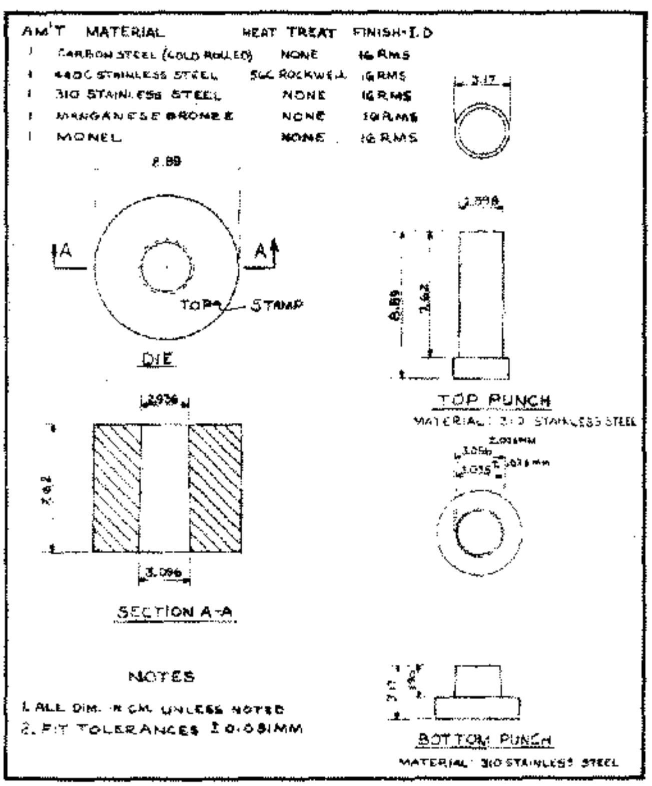

Figure 2. Lab Die Dimensions.

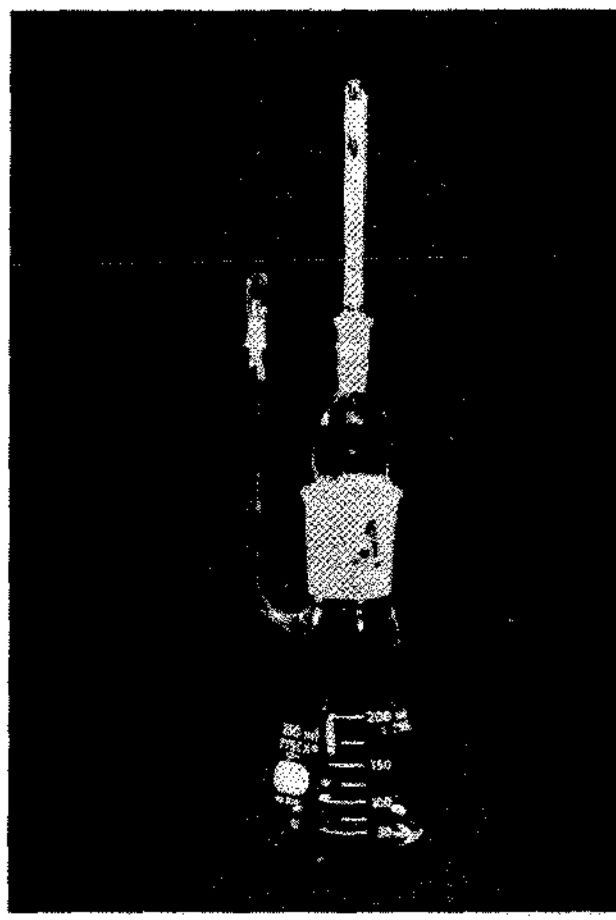

Figure 3. Pycnometer Photo.

2. Temperature
3. Moisture
4. Time
5. Additives
6. Lubricants
7. Crystal Variations
8. Die Materials

PRESSURE

This variable produces the greatest overall effects. It correlates directly with density (Fig. 4) and also with dry and wet strength (Fig. 5). In general, appearance and yield also increase with pressure.

Pressure can be expressed in several ways, dependent on definition. As used here, "applied pressure" will be taken to mean the total available pressure in kilograms. "Forming pressure" will be taken to mean the applied pressure proportioned over the entire surface area of the product, (top, bottom and sides) in kilograms/square centimeter and "release pressure" will mean the pressure in kilograms/square centimeter, applied over the entire surface area required to eject the product from the die.

Slope of curve (applied pressure vs. density) is gradual, almost linear, and is typically shown in Figure 4. Note that density, even at relatively high pressures, does not approach theoretical density of salt, assumed here to be 2.16. The correlation between applied pressure and dry and wet strength is also nearly linear and it can thus be assumed that density and crush strength are also positively correlated (Fig. 5).

The increment between wet and dry strength is shown to be relatively constant. This loss, divided by the dry strength, results in a loss ratio which will be expressed as such in following tables. The loss in strength increases, although at a decreasing rate, over time and thus the length of brine exposure must be specified.

The relationship between the forming pressure and the ejection or release pressure is essentially linear. The slope is dependent on several factors, covered later, but an average can be assumed at 0.33. Release pressure is an important variable, as considerable breakage can occur at ratio increases. Relatively small increases from 0.30 to 0.40 can have disastrous results on block quality and die life. Release pressure is dependent to a great part on sidewall area,

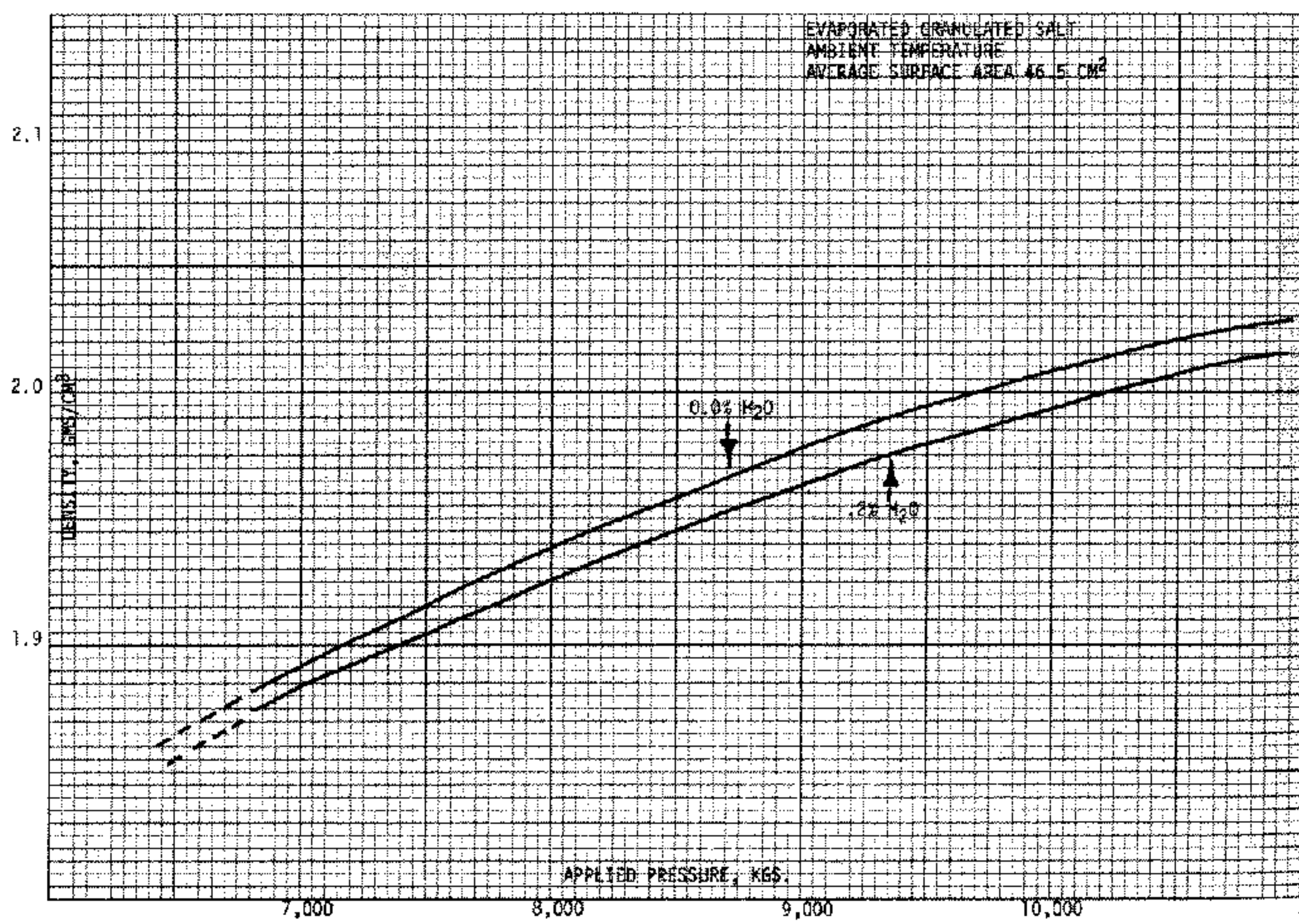

Figure 4. Applied Pressure vs. Density.

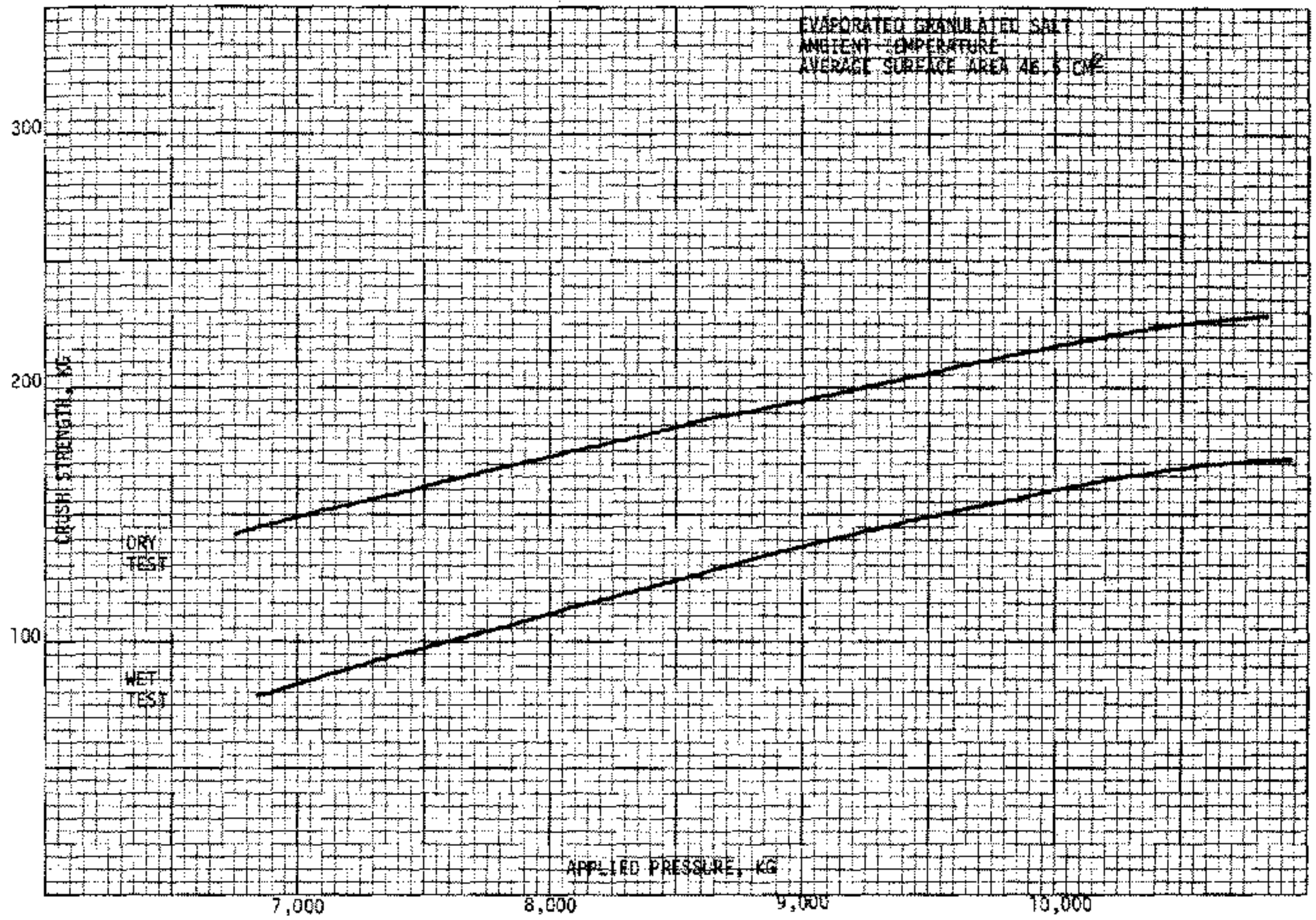

Figure 5. Applied Pressure vs. Fracture Strength.

which means that geometry is important. All other factors being equal, sidewall height should be considered approximately equal to the diameter.

The method of application of pressure is also of importance. A press can be operated so that pressure is applied with one dynamic punch and one static punch, one dynamic and one floating punch, or two dynamic punches. It is possible in the laboratory, using the aforementioned dies, to simulate a static bottom punch or a floating bottom punch by the use of #5 rubber stoppers (Fig. 6).

There will always be areas of varying density within a pressed product due to the effect of sidewall and intercrystalline friction. In a process with a floating punch, the minimum density will be located in the center of the product, while a fixed punch will result in the minimum density being located near the bottom. Unfortunately, this is not an optimum location, as the edges of a pressed product are very prone to damage. In a fixed bottom punch system then, this point of lowest density and lowest strength will coincide with the point of maximum exposure to damage. For this reason, the use of floating bottom punch is recommended or, ideally, a double acting upper and lower punch.

Figure 7 indicates the actual positions of the upper and lower punches at various applied pressures. Note that area marked "S" will be of minimum density. On Figure 8 the resultant density of the product with and without a floating punch is shown over various forming pressures. Note that density of a floating punch product is higher at any given forming pressure. Although a product of equivalent density can be made with a fixed punch, it will require the expenditure of considerably more work.

The increment can be estimated under these conditions at about 20–40 Kg/cm^2, dependent on density selected. The larger increments being required as desired density increases. Selecting an example of 165 Kg/cm^2 forming pressure (which approximates an average block press) from Figure 8, the floating die produces 1.926 density as against the 1.886 density of the fixed punch. It would require 192 Kg/cm^2 on the fixed punch to reach the 1.926 density. This is an approximate 16% increase in applied pressure. This operating increase should be considered

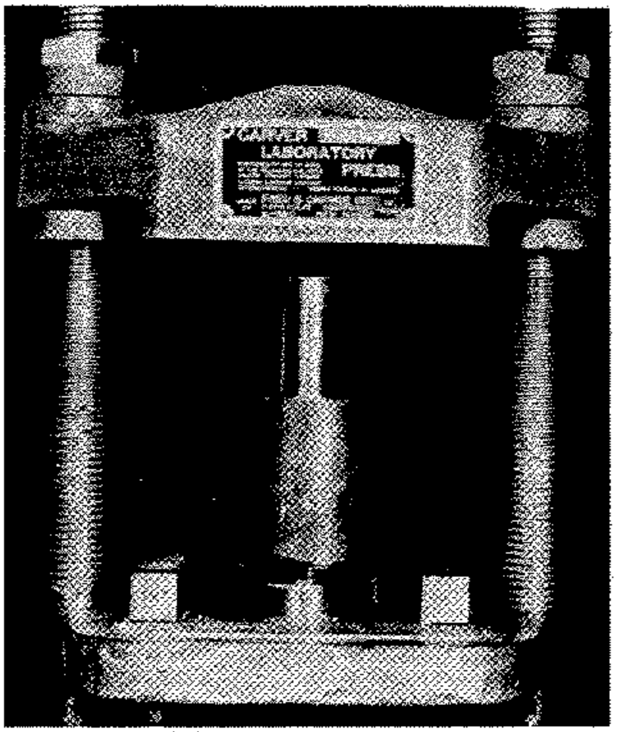

Figure 6. Photo of Carver Press with Die Installation.

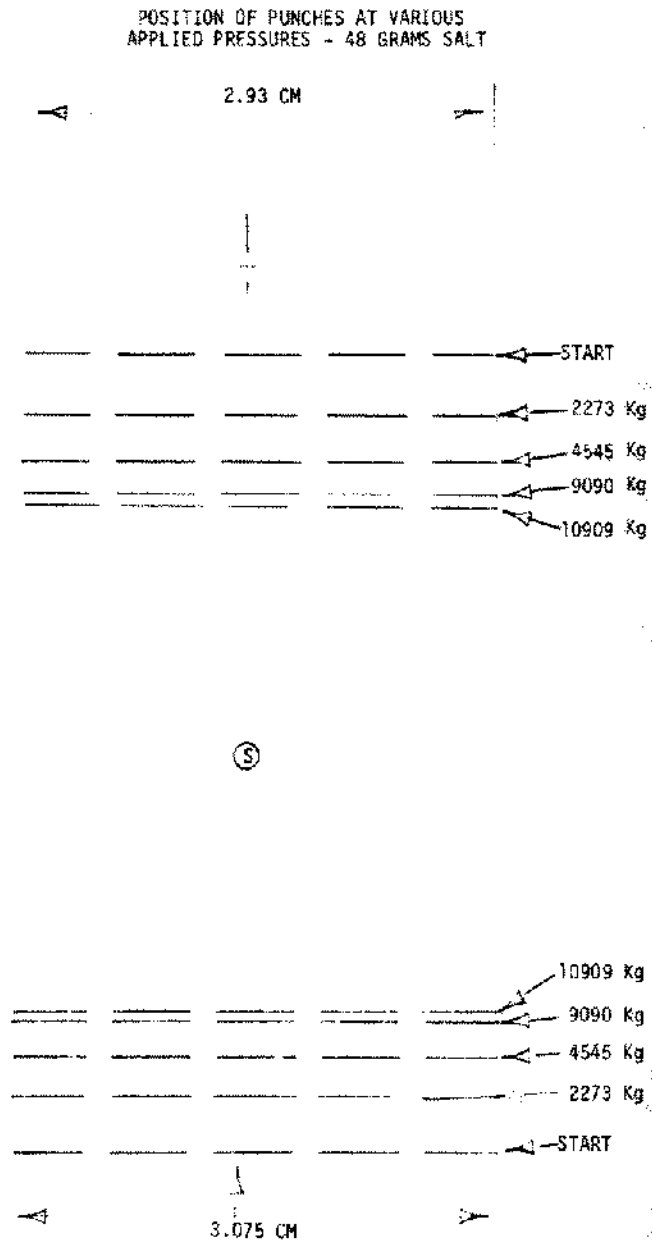

Figure 7. Sketch of Punch Positions During Pressing, 48 grams salt.

when the lower first cost of a fixed punch seems desirable. Pressure can thus be said to have the following effects within the ranges tested. As applied pressure increases, density, fracture strength (dry and wet) and release pressure will all increase. The use of a floating or double action punch is to be preferred.

TEMPERATURE

As salt approaches a temperature of 200°C., it assumes a plastic flow (Halbouty, 1967). Attempts were made in laboratory to simulate the advantages of increased plasticity. If the salt is heated to about 150°C., and then pressed, a much denser product will result, given the same applied pressure. In the typical mid-range values, raising temperature will permit the same density to be obtained by using approximately 13% less work. Conversely, by the use of equal work and higher temperatures, a denser product with greatly improved appearance results. In addition, the dry and wet strength is usually increased considerably. Unfortunately, the use of heat does have a disadvantage. The release pressure is increased considerably as compared to ambient conditions, the effect being such that removal is virtually impossible as salt approaches 200°C. The change in density due to heat is shown in Figures 8 and 9. The slope remains approximately the same, but density has increased markedly.

MOISTURE

The amount of water present has an important effect on the parameters, especially dry strength, which also directly effects yield. It should be realized that moisture can exist within salt crystals as well as on surface. When pressure is applied, this moisture can be released as crystals are molded. This effect is the probable cause of many observations that "such and such a salt" presses better than others, rather than differences in crystal structure. If salt is to be heated, the addition of moisture is, of course, academic, so moisture comparisons are made only between various salts at ambient conditions.

The effects are varied as regards density. Moisture decreases the density in evaporated granulated salt, but increases the density in others. The effect on release is also varied, dependent on other factors. In all untreated salt, the addition of moisture increased the dry strength considerably and, for this reason, its use seems indicated.

TIME

Over a period of time, a pressed product assumes a higher crush strength. This process commonly being known as "aging." A typical curve is given in Figure 9. Note that product containing moisture always tests higher than product without.

ADDITIVES

There has been considerable discussion on the effects of Yellow Prussiate of Soda (YPS) when salt is pressed into blocks, buttons, etc. YPS was found to have a definite effect on crush strength, both dry and wet. Results comparing salt with and without YPS are shown in Figures 10 and 11. Note that the presence of YPS produces a salt having less dry strength when moisture is added. This is in marked contrast to all untreated salt. In addition, the slope of the curves with YPS salt are considerably flatter than untreated salt, especially in salt with moisture. The effects of YPS on salt without added moisture are not marked, other than flattened slope. YPS has apparently removed some of the positive correlation of density and strength. YPS use in pressed products should thus be based on individual studies of conditions.

LUBRICANTS

A good many lubricants have been tested over the years. The best lubricant located at this time is calcium stearate, which is dusted on die before pressing. (If calcium stearate is mixed with salt before pressing, an adverse effect will result as regards dry strength.) Calcium stearate is relatively inexpensive and is approved by the

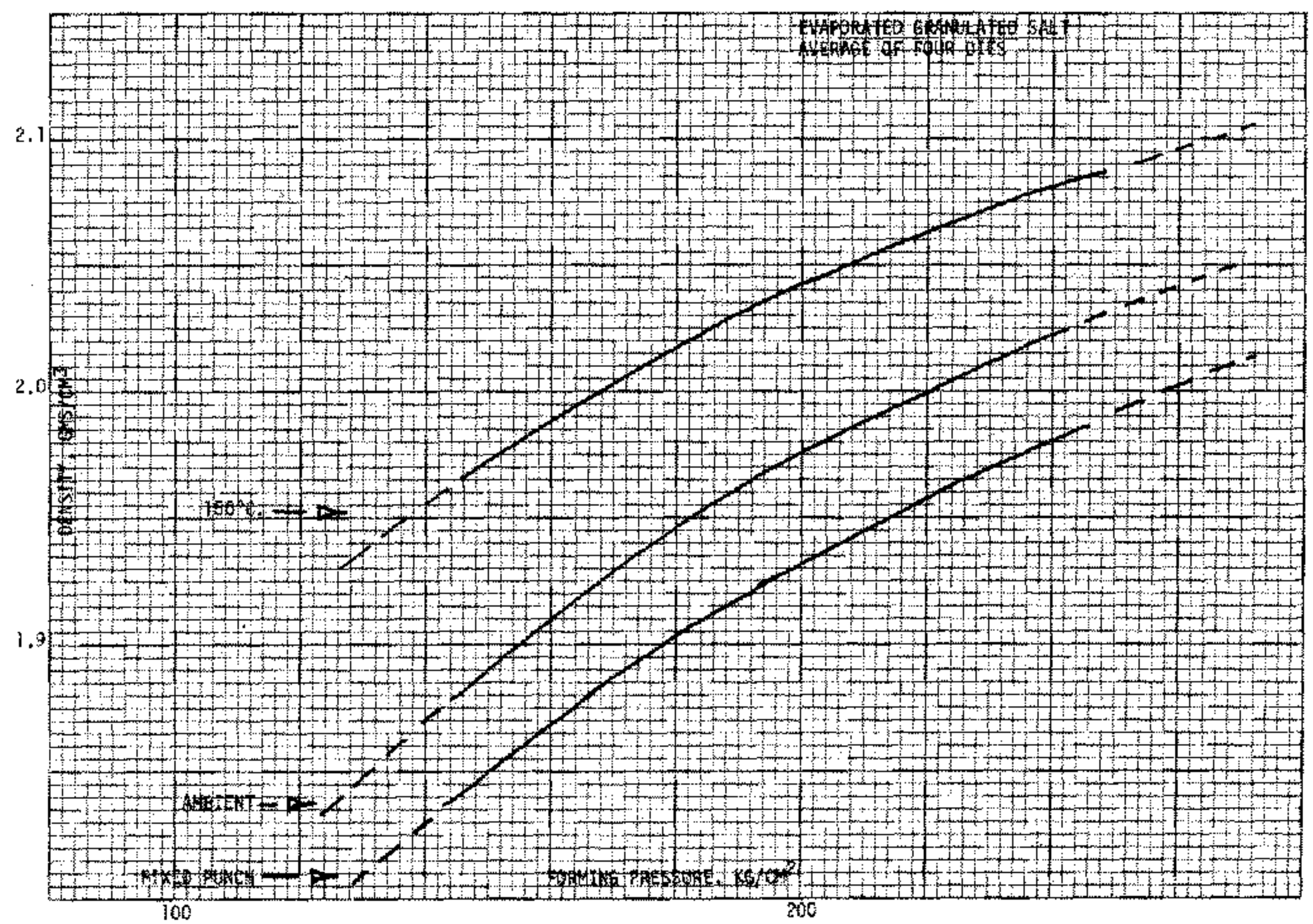

Figure 8. Forming Pressure vs. Density. (a) Fixed, (b) Floating

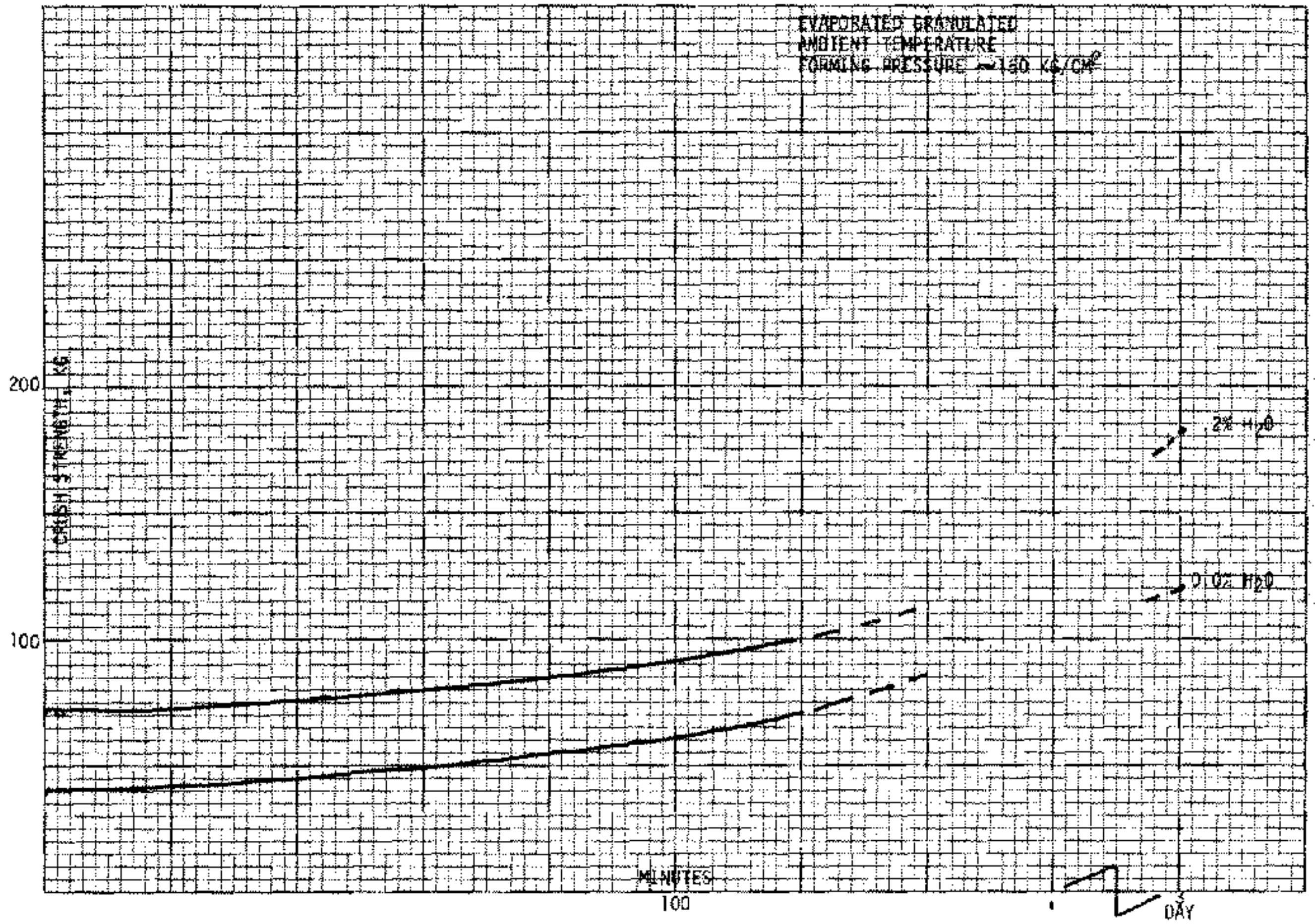

Figure 9. Crush Strength vs. Time.

Federal Drug Administration in food grades. In use, powder can be blown into die before each pressing operation. Results with the lubricant are quite dramatic—product is considerably denser, appearance is greatly enhanced (glossy surface), dry strength is increased, and release pressure is much reduced. Again, an equal product can be made with lower energy or a better product with the same energy. Material is especially useful if salt is heated, since release pressure can be reduced to satisfactory limits, as is illustrated in Table I.

TABLE I

Effects of calcium stearate used as a dusted lubricant at a forming pressure of about 245 Kg/cm^2.

Die	Temperature	Release Ratio	Density	Additives
Bronze	Ambient	.30	2.0288	—
Bronze	Ambient	.21	2.0466	Calcium Stearate
Bronze	150° C.	.57	2.0842	—
Bronze	150° C.	.15	2.1421	Calcium Stearate
310 S.S.	Ambient	.45	2.0173	—
310 S.S.	Ambient	.22	2.0515	Calcium Stearate
310 S.S.	150° C.	.55	2.0603	—
310 S.S.	150° C.	.21	2.1151	Calcium Stearate

CRYSTAL VARIATIONS

There are obviously infinite variations possible here and only a few will be mentioned as each would have to be tested separately. The salt used in the foregoing pressure tests was screened to remove all +45 mesh material and tests were then duplicated. Comparison is given in Table II. As can be seen, the use of the finer salt produced a product that was less dense per given applied pressure. It also produced a product that was stronger in resistance to handling, but note that wet strength has decreased while the loss ratio between the wet and dry increased very considerably the finer the salt. In other words, this salt is proportionately considerably lower in "mush" resistance. In appearance, unfortunately, a subjective rating, the use of the –45 mesh salt was an improvement.

Another crystal variation of importance results when salt passes through a continuous press and is recycled. This process tends to crush individual particles and a very

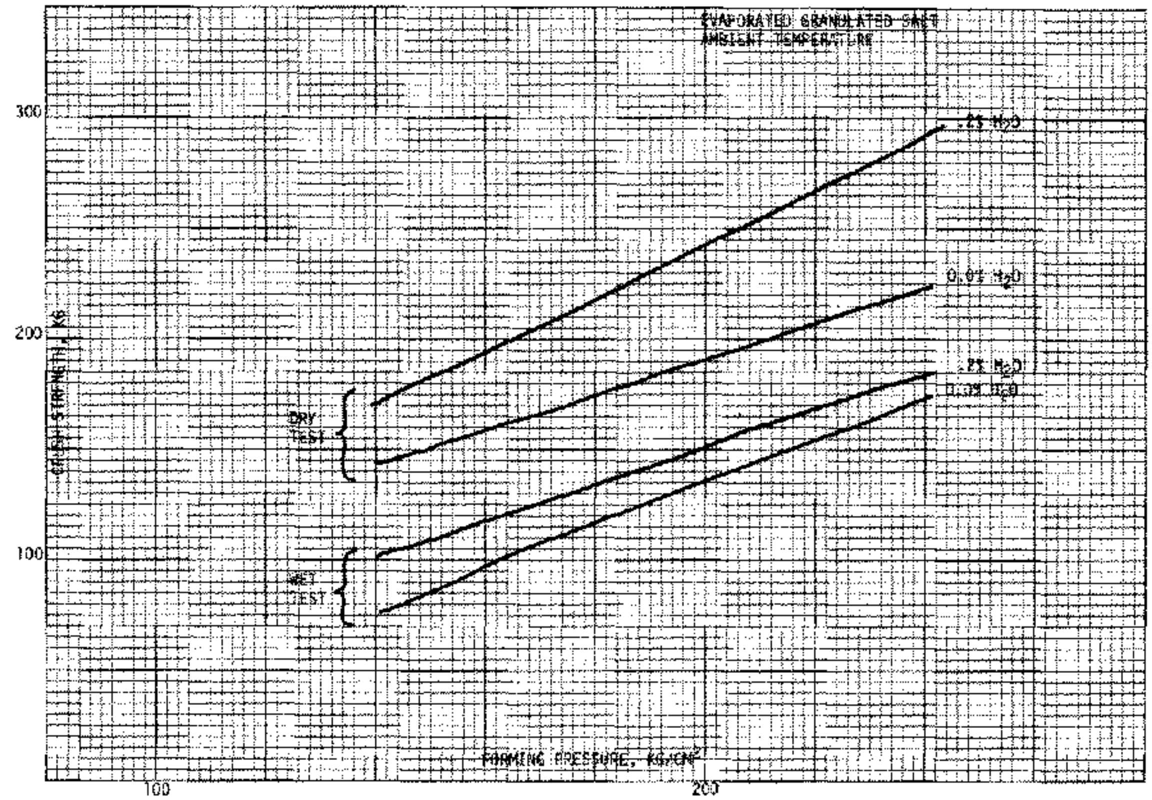

Figure 10. Crush Strength with H_2O.

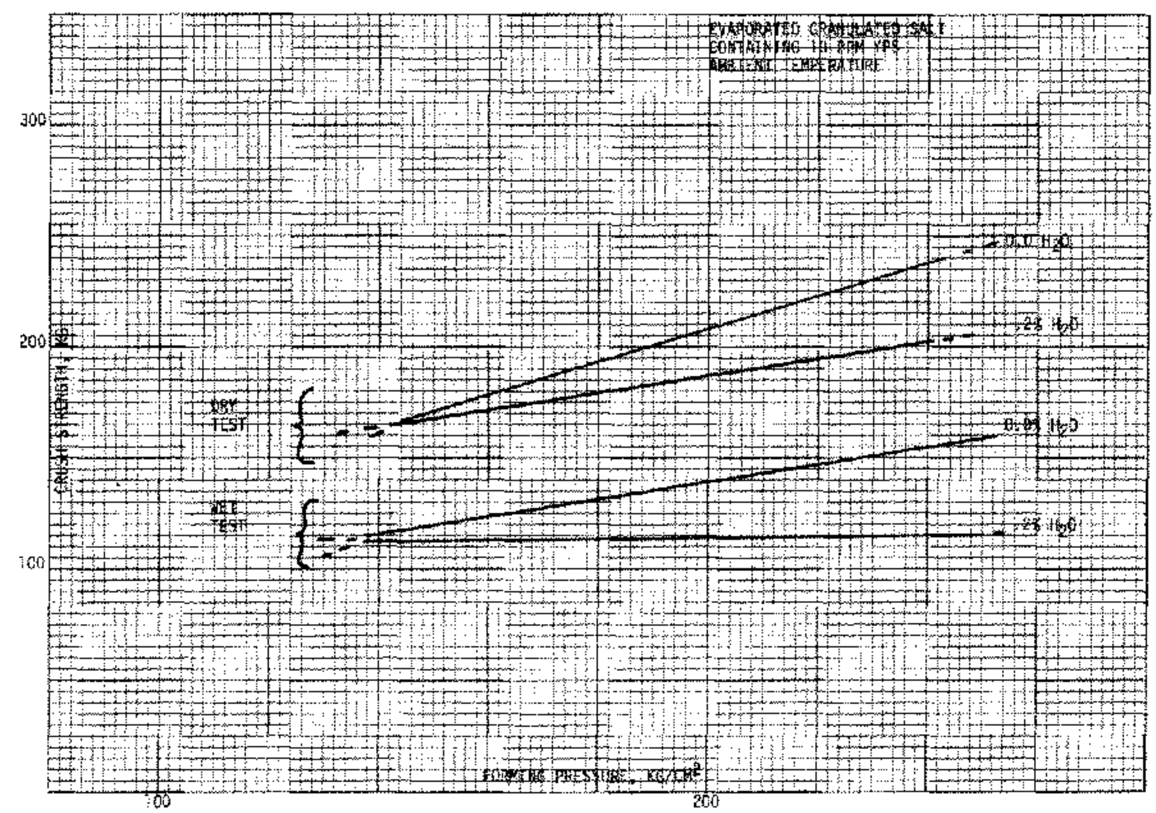

Figure 11. Crush Strength with H_2O and YPS.

TABLE II

Values averaged over all applied pressures

Average with Bronze Die

Conditions: Salt, Temperature, Additives	Density GM/CM^3	Pressures Kg/CM^2: Forming	Release	Ratio	Crush Strength Kg: Dry	Wet	Loss Ratio
Evaporated—Gran Ambient	1.9731	192	58	.30	198	148	.25
Evaporated—Gran Ambient, .2% H_2O	1.9680	192	58	.30	248	166	.33
Evaporated—Gran 150°C.	2.0449	196	111	.57	279	199	.29
Evaporated—Gran Ambient, —45 Mesh	1.9647	191	58	.30	287	146	.49
Evaporated—Gran Ambient, —45 Mesh, .2% H_2O	1.9685	191	53	.28	297	160	.46
Evaporated—Gran Ambient, 10 PPM YPS	1.9631	191	65	.34	221	161	.27
Evaporated—Gran Ambient, 10 PPM YPS, .2% H_2O	1.9756	192	63	.33	201	143	.29
Evaporated—Gran Ambient, Fixed Punch	1.9421	190	50	.26	224	—	—
Evaporated—Purified Ambient	1.9567	190	95	.50	235	147	.38
Evaporated—Purified Ambient, .2% H_2O	1.9639	190	86	.45	315	131	.47
Evaporated—Gran Ambient, Recycle	1.9490	190	66	.35	250	0	1.00
Evaporated—Gran Ambient, Recycle, .2% H_2O	1.9895	193	43	.22	363	0	1.00
Evaporated—Gran 150°., Recycle	1.9987	193	111	.57	388	0	1.00
Northern Rock Ambient, —30 Mesh	1.9690	191	78	.41	277	—	—
Northern Rock Ambient, —30 Mesh, .2% H_2O	1.9883	193	51	.26	421	—	—
Northern Rock 150° C., —30 Mesh	2.0372	196	118	.60	344	—	—
Ohio Rock Ambient, —30 Mesh	1.9806	192	59	.31	268	—	—
Ohio Rock Ambient, —30 Mesh, .2% H_2O	1.9868	192	51	.27	324	—	—
Ohio Rock 150° C., —30 Mesh	2.0284	195	90	.46	175	—	—

TABLE III

Values averaged over all applied pressures

Average of all Dies

Conditions: Salt, Temperature, Additives	Density GM/CM^3	Pressures Kg/CM^2: Forming	Release	Ratio	Crush Strength Kg: Dry	Wet	Loss Ratio
Evaporated—Gran Ambient.	1.9602	191	65	.34	186	129	.31
Evaporated—Gran 150° C.	2.0340	196	106	.54	253	197	.22
Evaporated—Gran .2% H_2O, Ambient	1.9456	190	68	.36	236	147	.38
Evaporated—Gran —45M, Ambient	1.9473	190	74	.39	244	112	.54
Evaporated—Gran —45M, .2% H_2O Ambient	1.9456	190	68	.36	273	135	.51
Evaporated—Gran 10 PPM YPS, Ambient	1.9477	190	75	.39	203	136	.33
Evaporated—Gran 10 PPM YPS, .2% H_2O, Ambient	1.9590	191	71	.37	187	118	.37
Evaporated—Gran Fixed Punch	1.9152	188	65	.30	210	—	—

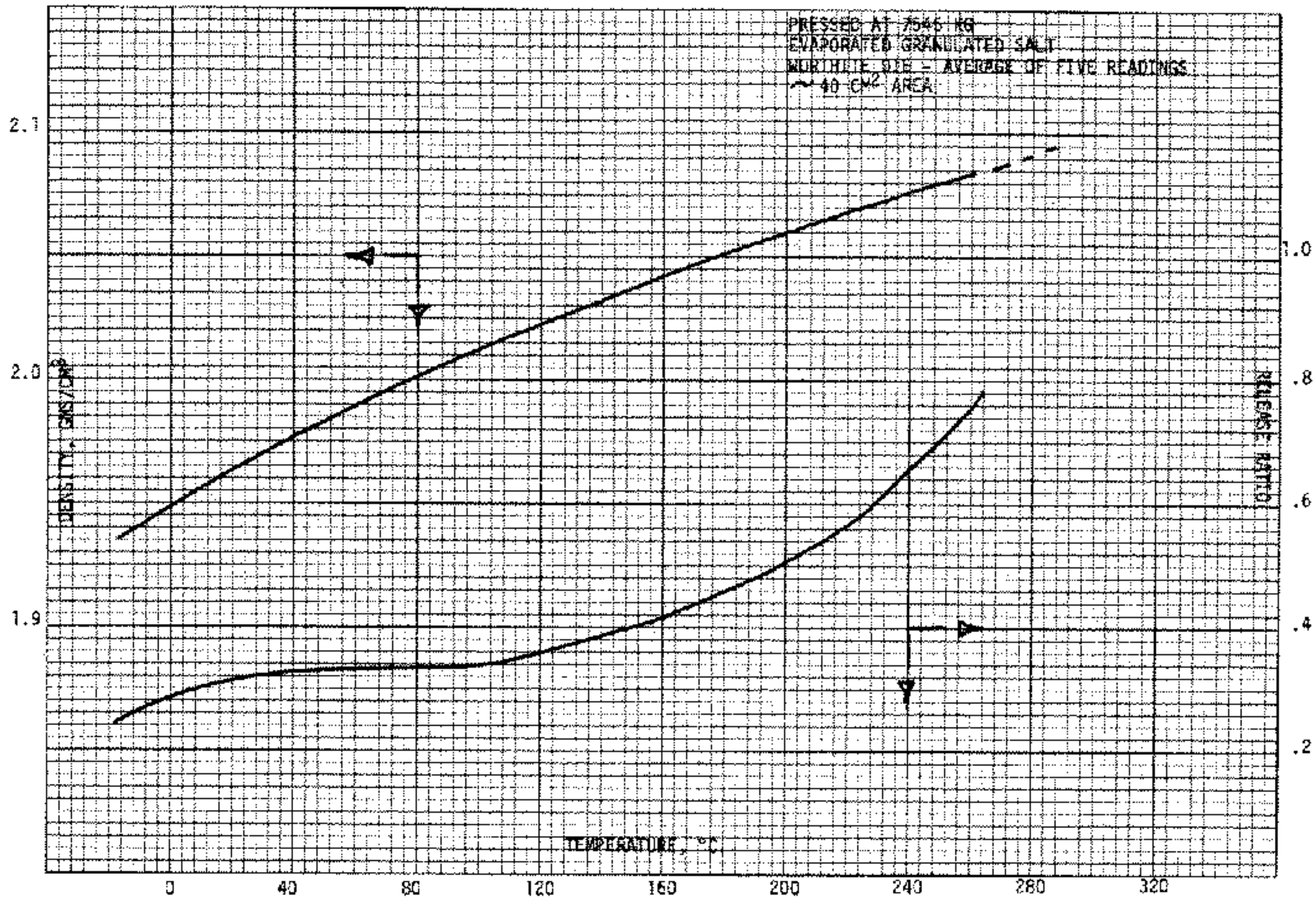

Figure 12. Density vs. Temperature.

TABLE IV

Screen Analysis

USS Mesh	Granulated	−45M	Purified	Recycle	Northern Rock	Ohio Rock
25	.1	–	–	–	–	–
35	16.5	–	4.0	16.1	14.1	9.8
45	54.2	–	59.7	43.2	23.5	27.1
60	25.5	86.8	32.1	28.8	17.5	22.7
80	3.2	11.3	3.2	6.1	11.8	16.7
Pan	.5	1.9	1.0	5.7	33.1	23.7

fine salt can result. A series of tests was made using only the bronze die, as time was short. In addition, a third purified grade salt was tested as another variation. Results are given in Table III, comparing the results between several salt variations on the bronze die only. The finer salts again give a product superior in dry strength, but inferior in wet strength. Note that the use of recycle salt has resulted in a product that has no wet strength at all! All samples disintegrated completely under brine. The lower density products disintegrate in one day under brine, while even the highest density fell apart in three days. At this time, there is no reliable data on the percentage of recycle salt that can be accepted in a pressed product. It is obvious that the use of 100% recycle is catastrophic and it would follow from these results that the use of recycle salt should be kept to a minimum, with none preferred. Probably no more than 10–15% should ever be present if product is to be used in brine applications. In dry applications, recycle salt is fully usable, producing products of equal or better dry strength than a mill run salt. The addition of heat to recycle salt again improves density and dry strength, but wet strength is still zero. The release ratio is almost doubled (Table III).

Some results are indicated using rock salt screened to remove the +30 mesh fractions. In all cases, rock salt was found to be fully usable as a pressed product. No wet strength tests were run, as rock salt products are not felt to be generally suited for brine applications.

DIE MATERIAL

The dies as used in these experiments consisted of four materials; manganese bronze, monel, 310 S.S., and 440 C (age hardened), all of which have been or are in use as production liners. A considerable difference was found in the product produced in each material. Averaged overall:

	Average Density Ratio	*Release*
Bronze	1.969	.31
Monel	1.954	.35
310 S.S.	1.946	.37
440 C	1.936	.45

These factors are, of course, not the only input considerations. 440 C, although poor in release characteristics and resultant density, is hard and does not suffer abrasion while the use of stearate can eliminate the release problems. Bronze, though good in laboratory factors, is soft and erodes rapidly, but can be easily machined. Choice must be made by an individual economic study.

CONCLUSIONS

It has been the purpose of this study to show that evaluations can be made on pressed salt products by the use of laboratory studies and that such data can then be used for engineering and economic studies. By use of this equipment, many of the old beliefs on pressing salt can be properly assigned causes. In many cases, the opinions were usually right, but the causes may be erroneous. Unless the true cause is found, resultant errors may be expensive. It is much cheaper to experiment with a Carver Press than a 750 ton press.

REFERENCES

Halbouty, M. T., 1967. *Salt Domes.* Houston, Texas, Gulf Publishing Company. p. 35, Chapter 3.