J. Pharm. Pharmacol., 1964, **16**, *Suppl.*, 111*T*–120*T* Received March 9, 1964

Some observations on the effect of lubrication on the crushing strength of tablets

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The effect of mean compaction pressure, base particle size, lubricant particle size, and lubricant concentration, on crushing strength of tablets has been determined for crystalline aspirin, hexamine, sucrose and sodium chloride, and simple granulations of sucrose and of hexamine. Magnesium stearate powder was used as a lubricant. The lubricant decreased the strength of all tablets, the reduction being greatest for the crystalline materials. Variation of lubricant particle size had no effect on strength, and the behaviour of the granulations was not predictable from the characteristics of the crystalline materials. The results are compared with those of other workers.

MUCH of the previous research on the problems of pharmaceutical tabletting has involved the use of standard granulations containing adhesive and lubricating agents. Strickland (1956), and Munzel & Kagi (1954) have investigated the distribution of lubricant in a granulation and its role in reducing friction, but paid slight attention to the effect of lubricant on tablet strength. Shotton & Ganderton (1961) found that coating of simple crystalline materials with stearic acid interfered with the mechanism of failure under a crushing load.

We record some observations on the effect of magnesium stearate on the crushing strength of tablets of crystalline materials and of two simple granulations prepared without binders.

Experimental

Crystalline sucrose, hexamine, aspirin and sodium chloride (B.P. or B.P.C. quality) were agitated in small quantities for 15 min on B.S. sieves using an "Inclyno" machine, and 30–40, 40–60, 60–80, 80–100 and −100 mesh fractions were collected.

Granulations of sucrose and of hexamine were prepared by hand from material which had been ball-milled until all passed a 100 mesh sieve. The powder was damped with distilled water and pressed through a 30 mesh sieve. Both granulations were dried at room temperature (2 hr); the hexamine granulation was further dried at 50° and 29 inches mercury vacuum (5 hr); the sucrose granulation at 60° (2 hr). The dried granulations were sieved to give a range of sizes.

A range of lubricant particle sizes was obtained by making a granulation of −100 mesh magnesium stearate (technical grade) using chloroform to produce a coherent mass. The size fractions were obtained by sieving the dried granulation for 15 min. The lubricant was mixed

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with the base material by tumbling in a glass jar (5 min), and, unless otherwise stated, was added as a —100 mesh powder.

All materials were compacted in a hand-operated Lehmann single-punch tablet machine, using a $\frac{1}{2}$ inch plane faced, cylindrical punch and die set instrumented in the manner of Shotton & Ganderton (1960). The upper punch was longer than is normally used (7 cm) to allow a greater degree of adjustment when compressing the granulations. The material to be tabletted was hand filled into the die, the weight of sample being sufficient to produce a tablet 0·4 cm thick at zero porosity. During the compaction cycle the force on the upper punch, the force on the lower punch, and the force necessary to eject the tablet were recorded. The weight and thickness of the tablets were measured before estimation of crushing strength on the apparatus described by Shotton & Ganderton (1960).

The following variables were investigated:

1. The effect of mean compaction pressure on lubricated and unlubricated materials using 40–60 mesh sodium chloride and 30–40 mesh fractions of the other materials. 2% magnesium stearate of —100 mesh was used as lubricant.

2. The effect of base particle size, in the presence and absence of 2% lubricant, the mean compaction pressure being constant.

3. The effect of lubricant concentration, mean compaction pressure remaining constant.

4. The effect of lubricant particle size, mean compaction pressure being constant, and lubricant concentration 2%.

The mean compaction pressure used in (2), (3) and (4) varied with the strength characteristics of the base material. Sucrose and sucrose

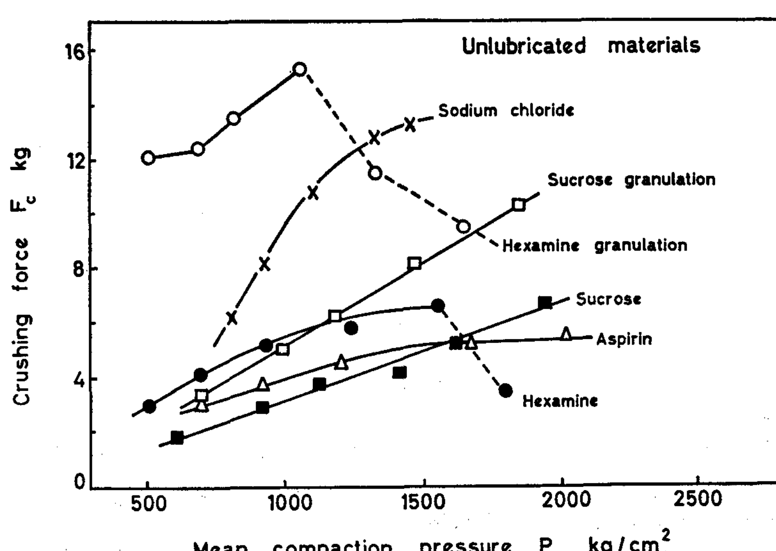

FIG. 1. Effect of mean compaction pressure on crushing strength.

granulation could not be tabletted without some form of die-wall lubrication. The die was lubricated by pressing a tablet containing 30% magnesium stearate in the die before the compaction of each tablet of sucrose or sucrose granulation. At no time was there any lubricant material which could affect particle bonding of unlubricated base.

To obtain reproducible results with hexamine and hexamine granulation the material was heated at 50–55° under 29 inches Hg vacuum for 2½ hr immediately before use.

Results

The results are presented graphically in Figs 1–7. Each point represents the mean of 5 tablets. Particle sizes where given refer to the mean of the sieve apertures by which the size range was classified:

Sieve classification, mesh	20–30	30–40	40–60	60–80	80–100
Mean particle size, μ ..	625	435	315	220	170

1. THE EFFECT OF MEAN COMPACTION PRESSURE ON CRUSHING STRENGTH

The relation between crushing strength and mean compaction pressure for unlubricated materials varies with the material being tabletted (Fig. 1). For sucrose and sucrose granulation there appears to be a direct relationship between crushing strength and the applied pressure, whereas for aspirin and hexamine, and sodium chloride up to 1,450 kg/cm^2, the relation is logarithmic. Hexamine and hexamine granulation show peak values for crushing strength after which the strength falls off with increasing pressure. This reduction in strength is associated with a pressure at which lamination of the tablet is evident. For hexamine granulation an increase in compaction pressure from approximately 500 kg/cm^2 to

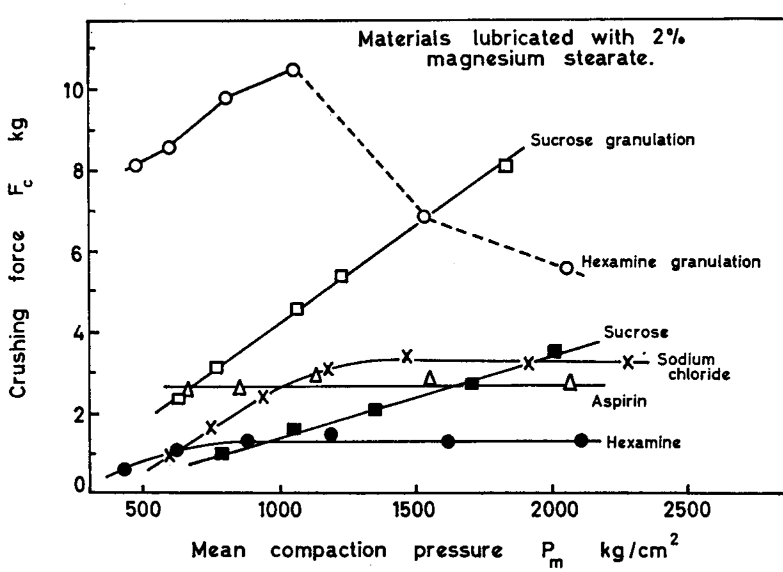

FIG. 2. Effect of mean compaction pressure on crushing strength.

700 kg/cm^2 has little effect on the crushing strength. Of particular note is the much greater strength of tablets made from granulations compared to those made from the crystalline base materials.

The addition of 2% magnesium stearate powder reduces the strength of all tabletted materials (Fig. 2), the reduction being less for the granulations. Increased compaction pressure has no effect on the crushing strength of lubricated aspirin, and only a slight increase in strength with increased pressure is observed with lubricated hexamine.

For hexamine and hexamine granulation the broken lines link results obtained from laminated compacts.

2. THE EFFECT OF BASE PARTICLE SIZE IN THE PRESENCE OF LUBRICANT

Results for lubricated and unlubricated materials are given in Figs 3, 4 and 5. The crushing strength of sodium chloride tablets, lubricated and unlubricated, compacted at a constant pressure, shows a minimum value over the approximate size range 220–315 μ (Fig. 3). In contrast, tablets of aspirin exhibit maximum strength over this same size range.

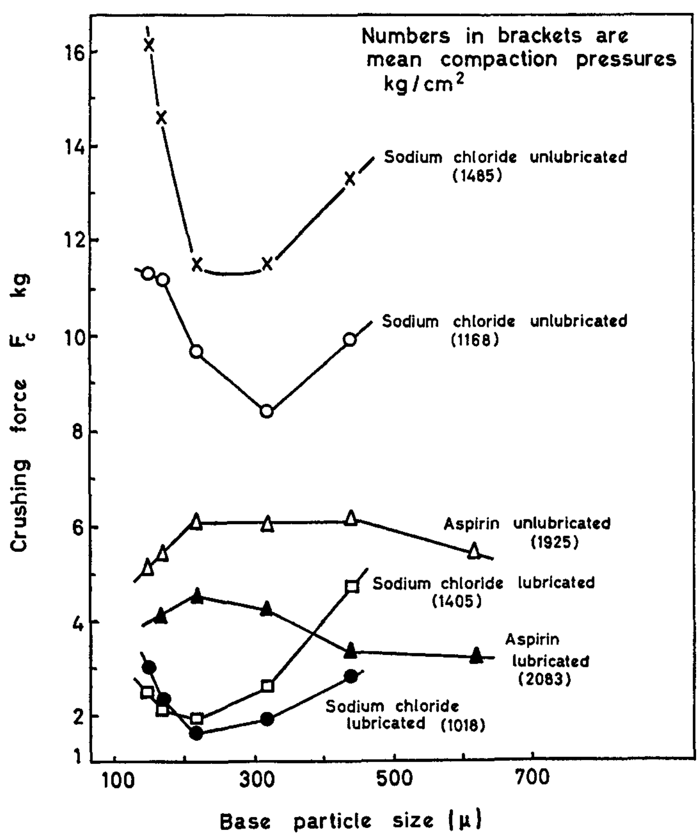

FIG. 3. Effect of base particle size on crushing strength.

For both these materials the presence of lubricant causes a reduction in crushing strength without altering the overall effect of base particle size.

A reduction in the particle size of sucrose, when lubricated, had no effect on crushing strength when the mean compaction pressure was 889 kg/cm^2 or 1,841 kg/cm^2 (Fig. 4). Sucrose granulation was also unaffected by changes in particle size when unlubricated, but tablet strength decreased slightly with decreasing particle size in the presence of lubricant.

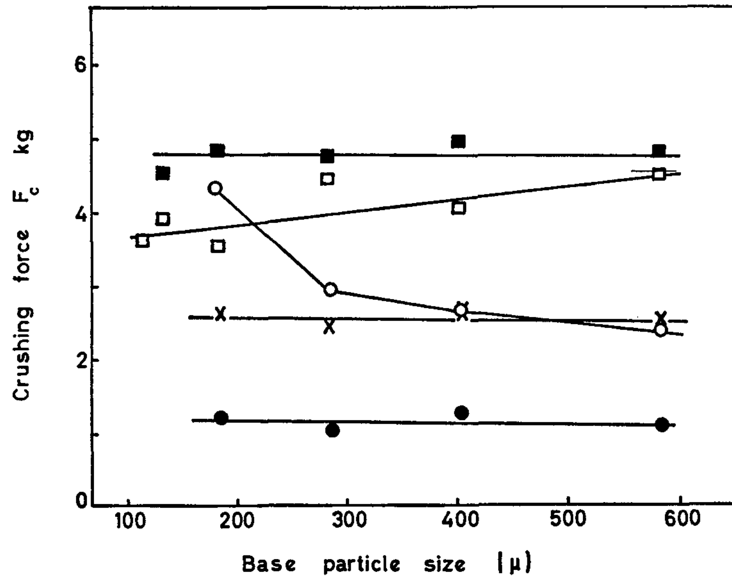

FIG. 4. Effect of base particle size on crushing strength of sucrose compacts.

		P_m (kg/cm^2)
■	Sucrose granulation unlubricated ..	981
□	Sucrose granulation lubricated ..	885
○	Sucrose unlubricated	992
●	Sucrose lubricated	889
X	Sucrose lubricated	1841

The effects of particle size on the strength of hexamine tablets are complicated (Fig. 5). With unlubricated hexamine crystals, a reduction in particle size produces an increase in strength to a maximum at 40–60 mesh, any further reduction in size producing weaker tablets. When lubricant is present, the strength decreases with decreasing particle size. The strongest tablets of hexamine granulation were those made with large sizes (20–30 mesh). The presence of lubricant markedly reduces crushing strength as granulation size decreases, the effect being much more pronounced than with the sucrose granulation.

3. THE EFFECT OF LUBRICANT CONCENTRATION

Not unexpectedly (Fig. 6), an increased concentration of lubricant produced weaker tablets, although for all materials used, other than hexamine granulation, an increase in concentration above 0·5% produced only a slight effect on crushing strength. Results for hexamine granulation are different in form.

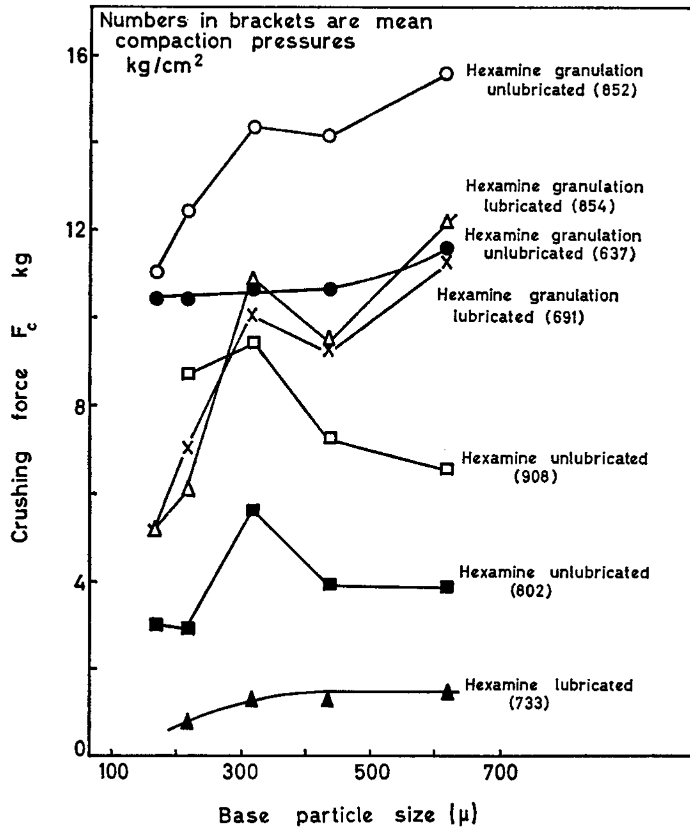

FIG. 5. Effect of base particle size on crushing strength of hexamine compacts.

4. THE EFFECT OF LUBRICANT PARTICLE SIZE (FIG. 7)

A reduction in lubricant particle size below 30–40 mesh (435 μ) has little or no effect on crushing strength.

Discussion

The strength of a tablet depends on the area of intimate contact between particles and the adhesive strength over the whole area. The work of Bowden & Tabor (1954) in the field of friction and lubrication has indicated that the strongest bonds are formed between clean surfaces, so that the addition of a lubricant to a material to be compacted might be expected to interfere with the adhesive bond between particles. The present work indicates that such interference occurs, for apart from the reduction in values of crushing strength when lubricant is present, visual observations of the fractured surfaces of tablets made from crystalline materials showed little particle-particle bonding.

Apart from interference by the lubricant forming a physical barrier between particles, the reduction of interparticular friction might well reduce the amount of clean reactive surface produced by shear at the sliding contact areas between particles. The fact that lubricated granu-

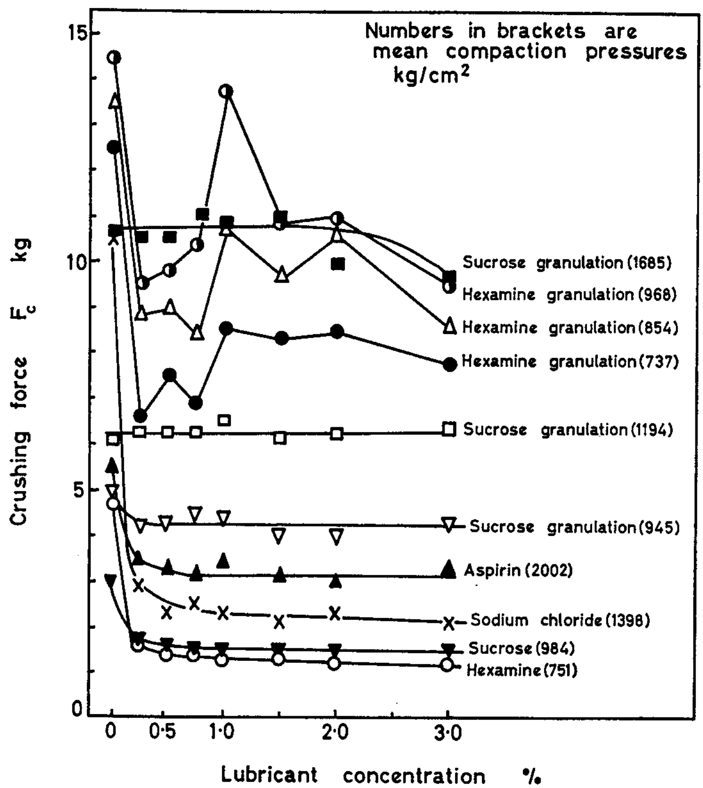

FIG. 6. Effect of lubricant concentration on crushing strength.

lations still produce strong tablets can be accounted for by the observations of Higuchi, Rao, Busse & Swintosky (1953), Higuchi, Elowe & Busse (1954) and Elowe (1954), who found that the surface area of standard granulations increased to a maximum and then decreased as compacting pressure was increased. With a granulation lubricated externally with magnesium stearate powder, most of this new and reactive surface will remain uncontaminated by the lubricant and produce strong bonds. Crystalline substances do not undergo the same degree of fragmentation and will be affected by the lubricant to a greater extent. Table 1 compares the crushing strength of tablets made from lubricated and unlubricated materials at a mean compaction pressure of 1,000 kg/cm^2.

TABLE 1. THE EFFECT OF LUBRICANT ON CRUSHING STRENGTH
Mean compaction pressure = 1,000 kg/cm²
Lubricant: 2% magnesium stearate — 100 mesh

Material 30–40 mesh except*	Crushing strength kg		Reduction in strength %
	Unlubricated	Lubricated	
Sucrose	2·8	1·4	50·0
Sucrose granulation	4·9	4·0	18·4
Hexamine	5·4	1·3	76·0
Hexamine granulation	14·6	10·5	28·1
Aspirin	3·6	2·7	25·0
Sodium chloride* 40–60 mesh	9·1	2·6	71·4

A linear relationship between strength values and the logarithm of the compaction pressure is quoted for a number of materials (Ganderton, 1962; Higuchi & others, 1953, 1954; Nelson, 1956). We have found unlubricated sodium chloride to deviate from a logarithmic relationship at 1,450 kg/cm², the maximum pressure attainable with the apparatus used. However the presence of a lubricant facilitates compaction to a greater pressure level and Fig. 2 confirms that for sodium chloride the logarithmic

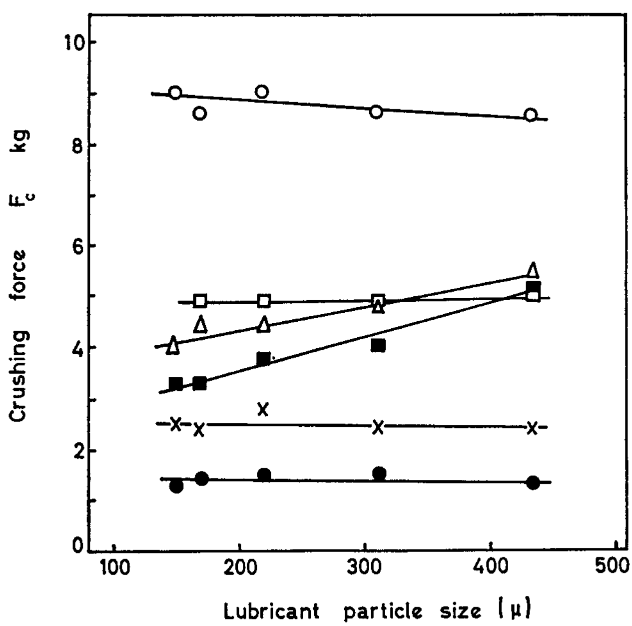

FIG. 7. Effect of lubricant particle size on crushing strength.

		P_m (kg/cm²)
○	Hexamine granulation	728
□	Sucrose granulation	961
△	Aspirin	2025
■	Sucrose	1911
X	Sodium chloride	1418
●	Hexamine	757

relationship breaks down at approximately 1,500 kg/cm². Such a deviation was also noted by Higuchi & others (1954) for sulphadiazine granulation.

On the other hand Huffine (1953) states that the breaking strength of tablets of sodium chloride and of sucrose is directly proportional to the applied pressure, a view supported by the work of Henderson (1962) on sodium chloride and sodium bromide, although his results for tablets of urea, potassium chloride and potassium bromide might be represented equally well by a logarithmic relationship.

That 2% 30–40 mesh lubricant should interfere with bonding to the same extent as −100 mesh powder, was considered surprising (Fig. 7). However, since the lubricant particles were preformed granules of fine powder it is possible that when mixing the lubricant with the base material, sufficient fine powder was transferred to the surface of the base particles to reduce the strength of the bond. A breaking down or spreading of the larger lubricant particles was not evident.

Little work has been reported on the comparative effect of lubricant concentration on the crushing strength of tablets made from crystals and granulations prepared without adhesives. Strickland (1956) states that increasing concentration produces softer tablets but said the effect was negligible at concentrations below 1%. The present work indicates that for crystalline materials 0·25% lubricant produces almost the maximum reduction in strength, and the effect of lubricant concentration on simple granulations varies with the nature of the base material.

The crushing strength of tablets made from the sucrose granulation is only slightly affected by increased lubricant concentration. This is consistent with the hypothesis of appreciable fragmentation producing a clean reactive surface for re-bonding. Using hexamine granulation, 0·25% lubricant produces a marked reduction in strength but as the concentration increases to 1% the strength increases before again decreasing. No explanation is offered for this behaviour which was absent in the crystalline hexamine, but similar behaviour was reported by Ganderton (1962) who lubricated crystalline hexamine with hard paraffin. Work in progress is designed to clarify this point.

The effect of particle size on the crushing strength of tablets made from unlubricated crystalline materials is related to the mechanism of failure (Shotton & Ganderton, 1961). Aspirin has a weak interparticulate bond and when unlubricated shows little particle size effect except for a weakening with 80–100 mesh fractions. In the presence of lubricant, a reduction in size from 20–30 to 60–80 mesh increases the crushing strength of the aspirin compact, with no appreciable effect on particle bonding. This is possibly due to a more uniform distribution of the applied pressure. The lubricant abolishes particle size effects with crystalline hexamine, but has no effect on sodium chloride, which is a harder, cubic, crystal capable of penetrating the lubricant film.

The effect of base particle size on tablets of unlubricated hexamine granulation (Fig. 5) is difficult to interpret; it is unlike that for hexamine crystals or for the granulation of sucrose. The observed behaviour of hexamine granulation is not due to mechanical defects, e.g., no laminations were found in any tablets of this material at the mean compaction pressures used.

The results from lubricated granules suggest that hexamine granules tend to deform whilst sucrose granules fracture. If, as suggested earlier, fracture of granules is necessary to obtain satisfactory tablet strength in the presence of a lubricant, the bonding of those granules which only deform will be weakened by lubricant films. However, even those granules which fill the void space in the compacting mass largely by deformation, are likely to be subjected to some degree of fragmentation when the granules are large; the importance of fracture and fragmentation becoming less as the granule size decreases. The lubricant powder will have relatively less adverse influence on the bonding of larger granules than on that of smaller ones, because of the improved bonding which arises after fragmentation. Thus where fracture of the hexamine granules is most probable (with 20–30 mesh granules, Fig. 5) the tablet strength is greatest, but the loss of strength is more rapid with the hexamine granulation when granule size is reduced than with sucrose granulation.

Visual observation of the fractured surfaces of all tablets of crystalline materials showed that magnesium stearate powder affected the mode of bonding in the same manner as that recorded by Shotton & Ganderton (1961) for coatings of stearic acid. No visual difference was observed in the fractured surfaces of tablets made from granulations with and without lubricant. Of particular note is that lubrication eliminated the capping effect in crystalline hexamine but not in the hexamine granulation (Cf. Figs 1 and 2). If the elimination of capping is due to stress relief at the crystal boundary, as suggested by Shotton & Ganderton (1961), it would appear that 2% magnesium stearate applied externally to hexamine granules is insufficient to allow stress relief between granules.

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The paper was presented by DR. LEWIS.