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# Acoustic emission of pharmaceutical materials: the effect of compression speed, ejection, lubrication and tablet weight

M.J. Waring [1], M.H. Rubinstein [2] and J.R. Howard [3]

[1] *Conratec B.R.L., Clwyd (U.K.),* [2] *School of Pharmacy, Liverpool Polytechnic, Liverpool (U.K.) and* [3] *Fisons Pharmaceuticals, Loughborough (U.K.)*

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## Summary

The acoustic emission from sodium chloride, paracetamol and lactose were measured during compaction and ejection using a portable activity meter connected to a transducer held against the die. In order to distinguish between particle-to-particle and particle-to-die wall emissions, tablets were compressed both in a lubricated and an unlubricated die. It was found that compacts of sodium chloride were little affected by lubrication, the acoustic counts not being much greater in an unlubricated die compared with similar tablets made in a lubricated die. This suggested that the emissions during compression are mainly due to particle-to particle and not particle-to-die wall interactions. However, with the other materials acoustic emission was approximately proportional to the peak ejection force with about twice the total acoustic counts being recorded from unlubricated samples compared with lubricated compacts. It would seem therefore that lubrication modifies the acoustic emission during compression and ejection and that the magnitude of the effect is material-dependent. It was observed that with sodium chloride and lubricated paracetamol samples, as the tablet emerged from the die and relaxation of the compact commenced, there was further acoustic emission demonstrating the tremendous strains to which tablets are subjected to as they leave the constraints of the die. As the tablet weight of sodium chloride was increased a correspondingly higher number of acoustic counts were recorded. This was directly related to the increased number of particles present in heavier tablets causing more particle-to-particle interactions during compression. It was found that a linear relationship existed between compression energy and total acoustic counts. The total acoustic counts from sodium chloride was unaffected by changes in compression speed from 2 to 50 mm/min. However, the amplitude and peak counts of the reorganisation acoustic peak (monitored during the initial stages of consolidation) increased with higher compression rates. This showed that early in the compression cycle, particle interactions were higher as dwell time was reduced, but that overall, the acoustic counts were constant for the duration of the whole compression cycle.

## Introduction

A previous study demonstrated that acoustic emission (A.E.) or stress wave analysis was a useful technique for material evaluation during tablet compression (Waring et al., 1987). The study showed that a number of pharmaceutical materials could be characterised acoustically. The acoustic profile of the materials during compression was shown to have 3 distinct stages: a reorganisation stage characterised by high emissions, a relatively quiet period during consolidation and a postcompression acoustic peak. Particle size effects were also observed with sodium chloride and a free-flowing form of lactose. Although numerically

*Correspondence*: M.H. Rubinstein, School of Pharmacy, Liverpool Polytechnic, Byrom Street, Liverpool L3 3AF, U.K.

greater acoustic counts were detected during compression compared with ejection the work did not establish that the emissions observed were the result of either particle-to-particle or particle-to-die wall interactions. In order to elucidate what proportion of the acoustic emission signal is generated from particle-to-particle interaction, this study was initiated to examine the effect of die wall lubrication on the acoustic profile of tablets of sodium chloride, paracetamol, lactose B.P. and a free-flowing form of lactose. In this study the emissions from sodium chloride were shown to be least affected by particle-to-die wall interactions and so this material was further used to evaluate the effect of compression speed and tablet weight.

## Materials and Methods

The following materials were used in this study: sodium chloride, paracetamol (B.D.H. Chemicals Ltd., Poole, U.K., Analar Grade), lactose BP and Zeparox, a free-flowing form of lactose (Dairy Crest Whey Division Ltd., Thames-Ditton, U.K.). Paracetamol and lactose BP were used as received, in the case of sodium chloride and Zeparox size fractions of 200-250 μm and 32-63 μm respectively were obtained by sieving (Fritsch Model 3.502, Idar-Oberstein, F.R.G.) for 20 min at amplitude 4. Compression was performed on an Instron Universal Tester (Instron Ltd., High Wycombe, U.K.,) and the acoustic emissions (A.E.) were analysed using a portable activity meter (P.A.M.) (Dunegan P.A.C., Cambridge, U.K.) as described previously (Waring et al., (1987). Throughout the study both acoustic counts and average signal level (A.S.L.) were measured as indicated in Fig. 1 for each compression cycle. Additionally the total counts for compression were computed from the acoustic count profile and compression energy obtained from the integrator on the Instron. To avoid interference emanating from the crosshead drive system on the Instron when using the wide bandwidth (20–600 kHz) it was found necessary to operate the P.A.M. at the narrow bandwidth (95–600 kHz).

*Calibration of the acoustic signal*

Validation of acoustic emission experiments is particularly difficult, since for true calibration a reliable source which will generate repeatable amplitudes is required. Parker in 1979 described 4 methods and concluded that a "point contact" device which resulted in a thermal stress pulse on the specimen provided a reproducible approximation to acoustic emission. A simpler method, however, is obtained by fracture of a pencil lead onto the surface of the specimen, the Hsu-Neilson test. This method was used in this study and is judged to be a reliable technique for producing repeatable amplitude levels. However, the acoustic analyser in this study uses an averaging technique for amplitude analysis. This resulted in wide variations in the captured amplitude signal depending on the time in the averaging cycle at which the pencil lead fracture was detected. For this reason it should be noted that the results obtained in the study are comparative since the system geometry,

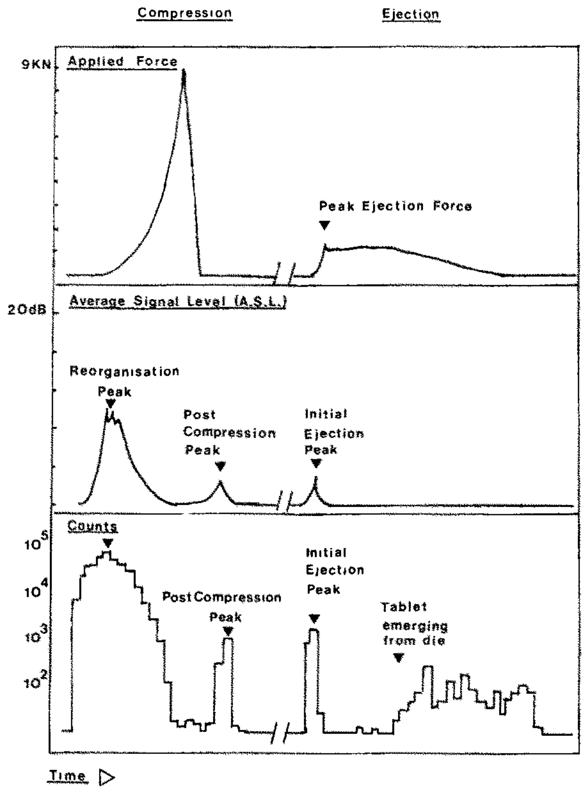


Fig. 1. Acoustic and force profiles of sodium chloride, 200–250 μm. All profiles to the same time base.

acoustic couplant thickness and applied load of the wave guide against the die wall influence the amplitude of the emissions detected by the transducer.

*Effect of lubrication*

In order to evaluate the effect of lubrication on the A.E. during compression 5 tablets were compressed in an unlubricated die and the acoustic profile compared with 5 tablets from a die lubricated with a solution of 10% stearic acid in chloroform. In the case of paracetamol, contamination of the die from the unlubricated samples resulted in only 3 lubricated samples being available for comparison.

*Effect of compression speed*

The effect of compression speed on the emissions from sodium chloride was evaluated at a range of compression speeds from 2 mm/min to 50 mm/min using a constant tablet weight of 225 mg. Replicate samples were taken at each speed.

*Effect of tablet weight*

Sodium chloride was compressed at a series of tablet weights from 100 mg to 500 mg at 100-mg increments. Replicate samples were taken at each weight and the compression speed was maintained at 10 mm/min.

## Results and Discussion

*Effect of lubrication*

Sodium chloride is least affected both acoustically and mechanically as shown in Fig. 2, indicating that the material is showing little resistance to particle-to-die wall reaction. Moreover, this also suggests that the emissions during compression are the result of particle-to-particle and not particle-to-die wall interactions. The other materials, paracetamol, Zeparox and lactose, are all brittle in character and were all affected to approximately the same extent by lubrication. As can be seen from Figs. 3–5 regardless of magnitude approximately twice the total acoustic counts were recorded from unlubricated samples compared with lubricated compacts. The twofold increase in acoustic counts with unlubricated lactose and Zeparox is mirrored by a twofold increase in peak ejection force. However, there is a significant difference in the total counts measured from unlubricated and lubricated paracetamol (Fig. 5), which is not reflected in the values of ejection force. Sodium chloride and lubricated paracetamol samples demonstrated interesting acoustic ejection profiles in that once the tablet was moving and the initial acoustic peak complete, the ejection became acoustically quiet until the tablet started to emerge from the die and relaxation of

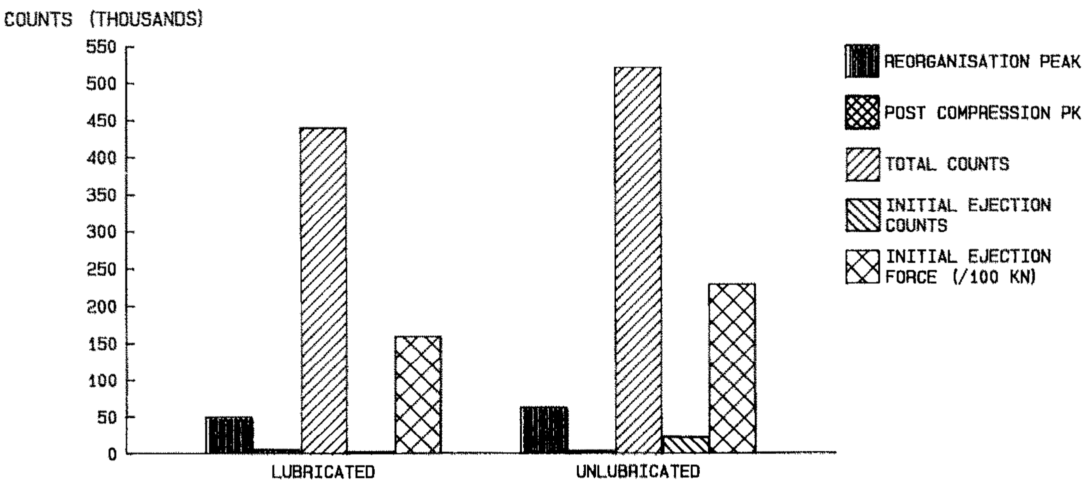


Fig. 2. The effect of lubrication on the A.E. from sodium chloride, 200–250 μm.

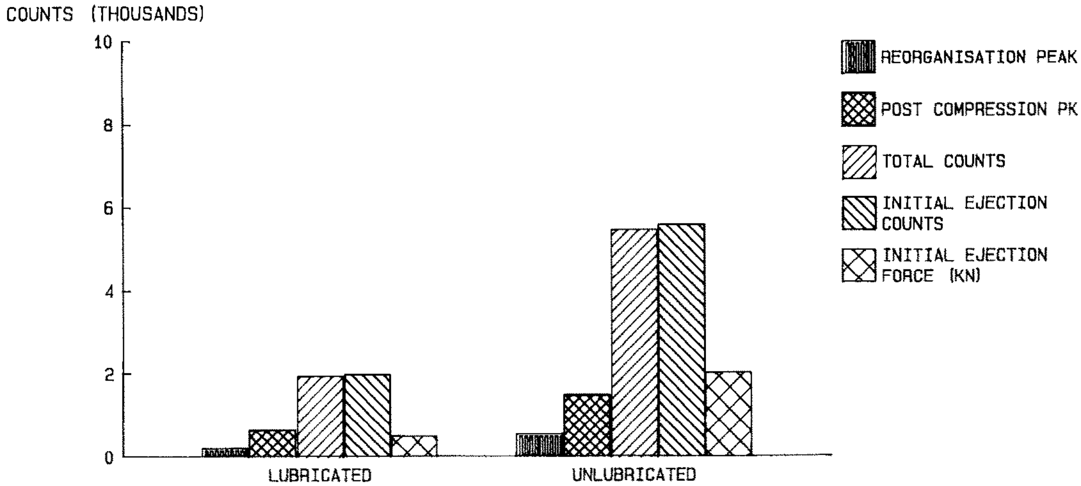


Fig. 3. The effect of lubrication on the A.E. from lactose monohydrate.

the compact commenced (Fig. 1). A similar phenomenon was observed by Rue and Barkworth (1980) who compared the cumulative acoustic emissions from sodium chloride tablets with a concomitant increase in tablet crushing force. In Rue's work however the transducer was mounted directly onto the tablet and monitored the stress relaxation of the tablet after ejection. The relative remoteness of the transducer in this study serves to demonstrate the tremendous strains to which tablets are subjected to as they leave the constraints of the die. It would seem from Figs. 2–5 that lubrication modifies the A.E. during compression and ejection, the magnitude of the effect, however, appears material-dependent. Initially it would also appear likely that materials such as

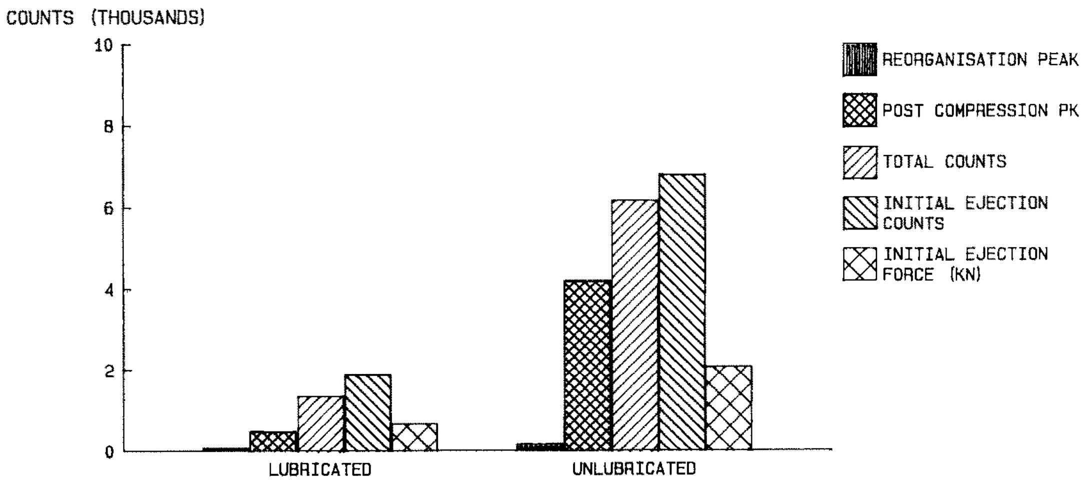


Fig. 4. The effect of lubrication on the A.E. from: Zeparox, 32–63 $\mu$m.

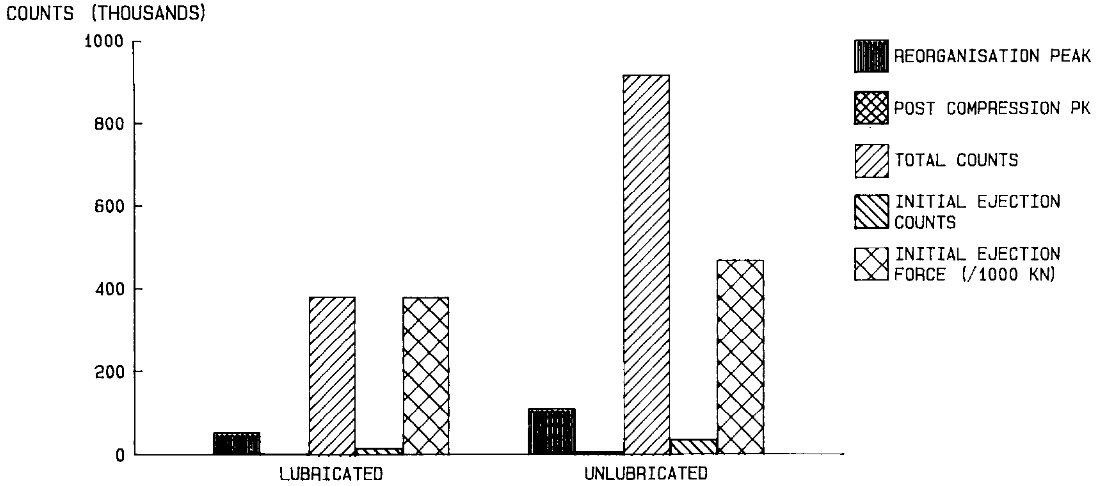


Fig. 5. The effect of lubrication on the A.E. from paracetamol.

sodium chloride, which are essentially self lubricating, are least affected acoustically by lubrication.

*Effect of tablet weight*

Increasing the tablet weight of sodium chloride tablets does not appear to significantly affect the postcompression or initial ejection peaks. However, there is an increase in both the reorganisation peak (Fig. 6) and the total counts measured during compression (Fig. 7). The higher number of counts observed is probably directly related to the increased number of particles present causing more particle-to-particle interactions during compression. As can be seen from Fig. 7, a linear relationship between total counts and compression weight

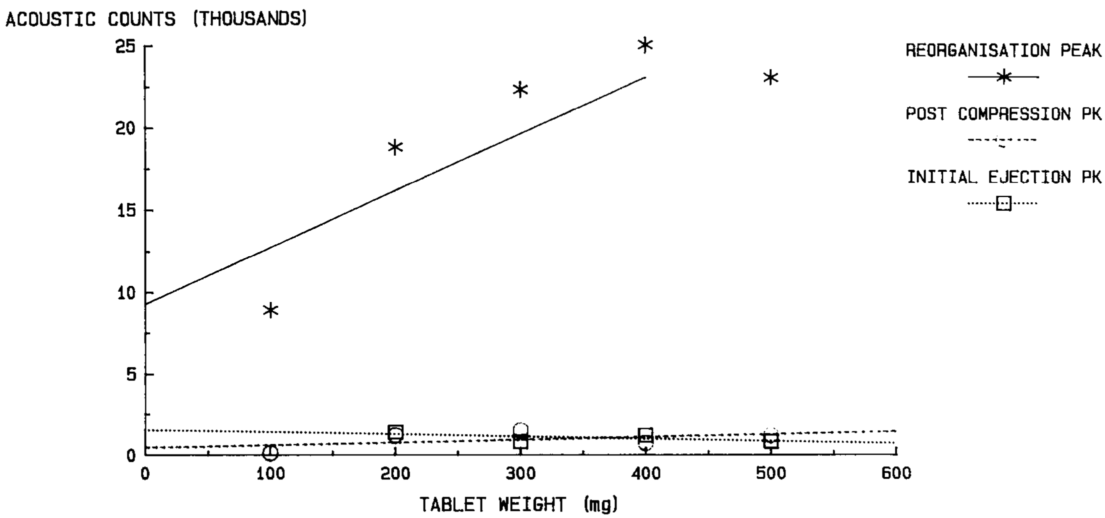


Fig. 6. The effect of tablet weight on the A.E. from sodium chloride, 200–250 μm, during compression and ejection.

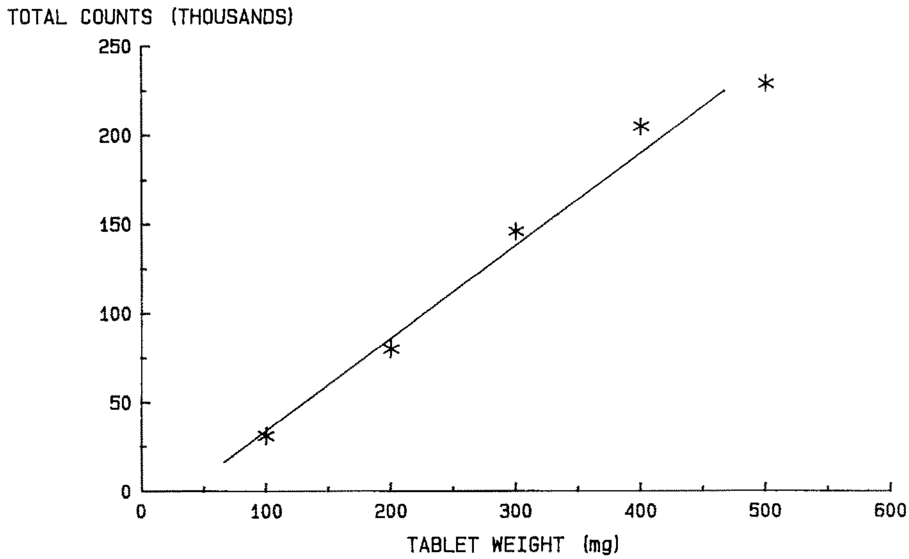


Fig. 7. The effect of tablet weight on the total acoustic counts measured during compression of sodium chloride, 200–250 μm.

is observed. The linear relationship between total counts and tablet weight may also be extended to counts and energy of compression. Energy of compression increases with the larger number of particle-to-particle interactions which in turn produces higher acoustic counts demonstrating the relationship between compression energy and acoustic energy. Improved accuracy is obtained in the measurement of total counts and this is reflected in the good linearity observed in the total counts plot (Fig. 7) compared with the plot of the rapidly changing reorganisation peak (Fig. 6). The slight increase in the counts measured in the post-compression peak (Fig. 6) is likely to be the result

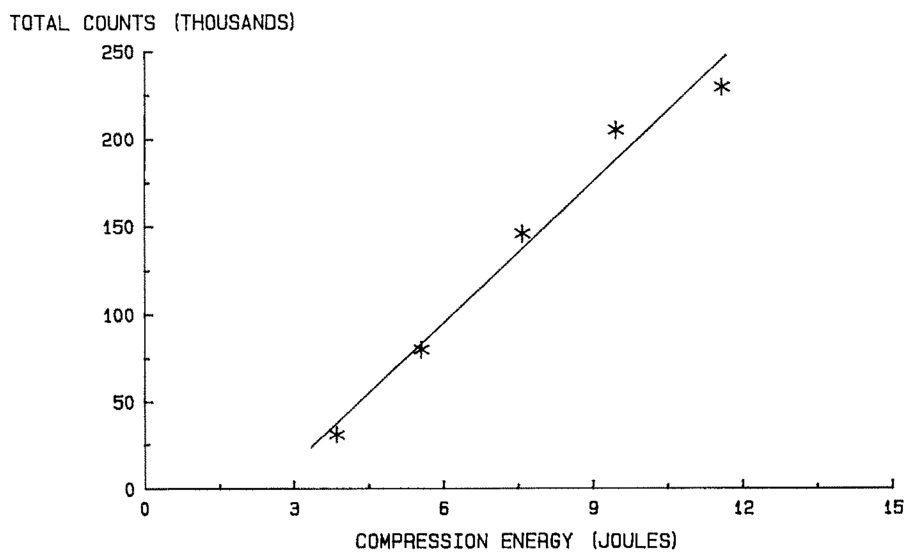


Fig. 8. The effect of compression energy on the total acoustic counts measured during compression of sodium chloride, 200–250 μm.

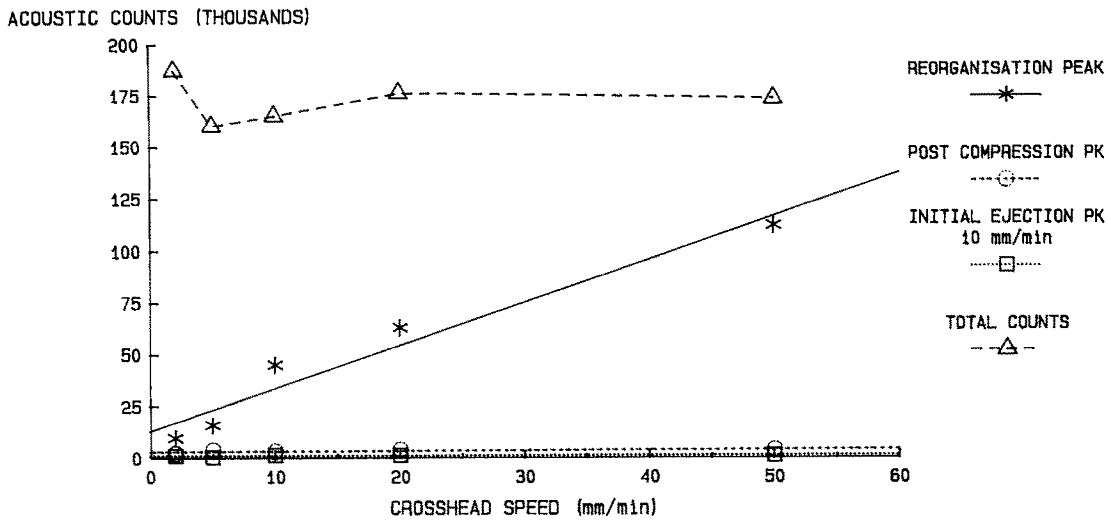


Fig. 9. The effect of compression speed on the A.E. from sodium chloride, 200–250 μm. Ejection speed maintained at 10 mm/min.

of greater contact with the die at higher tablet weights causing more emissions as the tablet relaxes. The greater die wall contact does not appear to affect the initial ejection peak which further indicates the good lubrication properties of sodium chloride.

*Effect of compression speed on sodium chloride*

Both the acoustic counts and average signal level remain constant over the range of speeds examined for the postcompression and initial ejection peaks (Figs. 9 and 10) indicating that recovery within the die is not affected by the speed of compaction. However, the amplitude and peak counts of the reorganisation peak are increased with higher compression rates (Figs. 9 and 10) indicating an acoustic emission rate response to compression speed. The total counts for the dura-

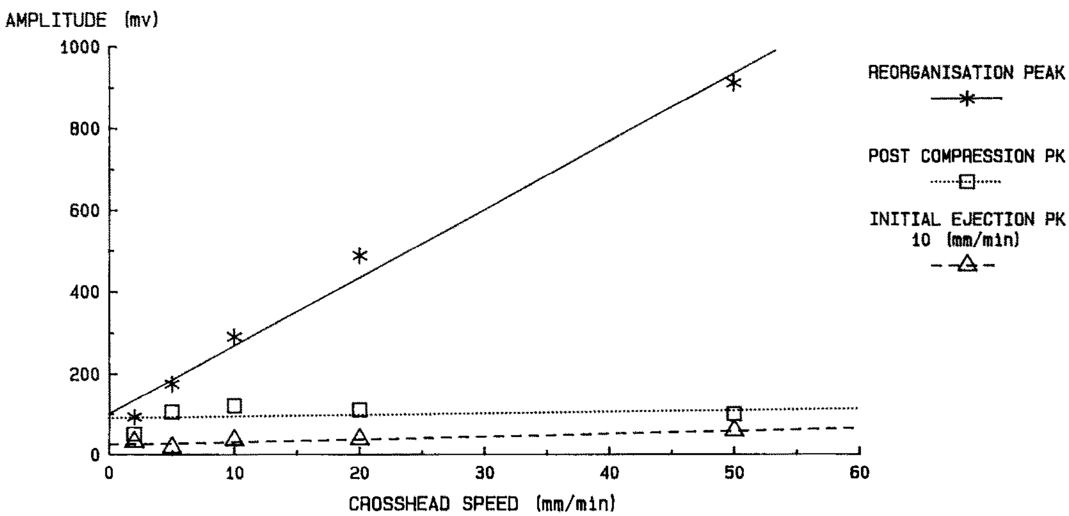


Fig. 10. The effect of compression speed on the amplitude of the acoustic emissions from sodium chloride, 200–250 μm.

tion of compression is relatively unaffected by compression speed suggesting that the acoustic energy remains constant for the range of tableting speeds selected but that the emission rate indicated by the reorganisation peak is directly related to the rate of change of compression speed. Compression energy was not affected over the range of speeds selected. Ejection speed was kept constant at 10 mm/min and ejection energy was also unaffected by change in compression speed. Although it would have been useful to examine higher compression speeds the peak counts measured for the reorganisation peak was greater than the P.A.M. could accommodate.

Further work is required to characterise the acoustic emissions and to quantify the relationships observed; however, the technique of A.E. appears to be a unique method for real-time analysis of the compression cycle.

## References

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