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THE SURFACE HARDNESS DISTRIBUTION OVER 'TILTAB' TABLETS

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ABSTRACT

A modified microindentation apparatus is described and shown to be a satisfactory technique to monitor the surface hardness and resilience of tablets with unusual surface curvatures, in this case those associated with the SK&F novel tilting tablet concept. The indentation test has been able to pin-point differences in properties over the tablets and has demonstrated various trends in surface hardness for these new shapes. 'Ridaura' tablets, which are square, bevel edged with normal convex surfaces bearing a raised dome on both faces, show stronger surface characteristics around the periphery and lower surface hardness over the dome. The elastic quotient is also reduced at the top of the dome and this may well be due to the reduced consolidation in this region during compression. Two other tablet shapes were also examined: 200 mg and 800 mg 'Tagamet' tablets. The 200 mg tablet is a round shape similar in profile to the square 'Ridaura' product. There is a quantitative similarity between the data of both surfaces for

this tablet with harder areas at the periphery, but unlike the 'Ridaura' it also demonstrates a measurable reduction in surface hardness and resilience at the peak of the tilt feature. The more complex oval 800 mg 'Tagamet' 'Tiltab' tablets have generally higher surface hardness and resilience. It is believed that a slightly modified formulation may also have contributed to these significantly higher volumes.

It is concluded that the microindentation apparatus may well be a useful tool to assist in the optimisation of formulations of 'Tiltab' tablets, as well as in determining the most appropriate processing conditions.

INTRODUCTION

'Tiltab' tablets have been developed recently by Smith Kline & French (Tovey, 1987). They are compressed tablets which have a raised central portion on both faces which impart an angle of tilt to the tablets when they are lying on a flat surface. The original objective of this novel design was to create a tablet which would show an increase in height when lying on a flat surface, but without an increase in tablet weight or volume. This would enable the tablets to be picked up more easily by, for example, arthritic patients. Thus for 'Ridaura' tablets (Auranofin, SK&F) a square shape with central dome-shaped projections was devised. This tablet shape has the added advantage that it will not roll as it tilts up from the horizontal. A further general advantage of 'Tiltab' Tablets is that they are clearly different from conventionally shaped tablets. This makes them easily recognisable and may help to avoid confusion between products and thereby assist compliance.

'Tiltab', 'Ridaura' and 'Tagamet' are registered Trade Marks of Smith Kline and French Labs Ltd.

The variation in tablet thickness as a result of this shape changes the degree of consolidation of the tablets, i.e. the powder in the region of the raised central region is less consolidated than that of the outer flatter portion. This can create processing difficulties resulting from variable strength and abrasion resistance over the tablet. This is particularly important during film coating when the tablets are subjected to high abrasion forces during the first few minutes of the coating process, before a protective film has been applied. Indeed, most of the development problems experienced with 'Ridaura' 'Tiltab' tablets lay in the achievement of a tablet core suitable for film coating. The general handling of 'Tiltab' tablets in bulk could also be a problem for tablets with potentially weak spots on the most exposed areas of their surface.

The measurement of point surface indentation is one test which can be used to quantify mechanical properties at localised points over the tablet surface rather than the properties of the tablet as a whole. Surface penetrometry has already been used to examine the effect of punch curvature and compaction pressure (Aulton, 1981) over the surface of tablets. In this present work a modified microindentation apparatus has been used to study the distribution of hardness and elastic quotient over the surfaces of commercially available 'Tiltab' tablets.

APPARATUS

The apparatus used is based on that described by White and Aulton (1980). Briefly, this apparatus consists of a vertically-supported shaft, on the lower end of which is an indenting sphere and at the upper end is a loading platform onto which weights can be applied to provide a constant indentation force. The depth of penetration of the indenter The present apparatus, whilst retaining the same principal of action to that

described above, has been extensively modified to suit the unique characteristics of 'Tiltab' tablets. The modifications include a) a universally-jointed vice to grip a range of tablet shapes and to orientate the tablets so that the indentation is always normal to the curvature of the surface at the point of test, b) a precision lowering device for the indenter shaft housing to compensate for the wide variation of tablet thicknesses over the tablet and c) a pneumatic device to lower the indentation load without initial impact overload.

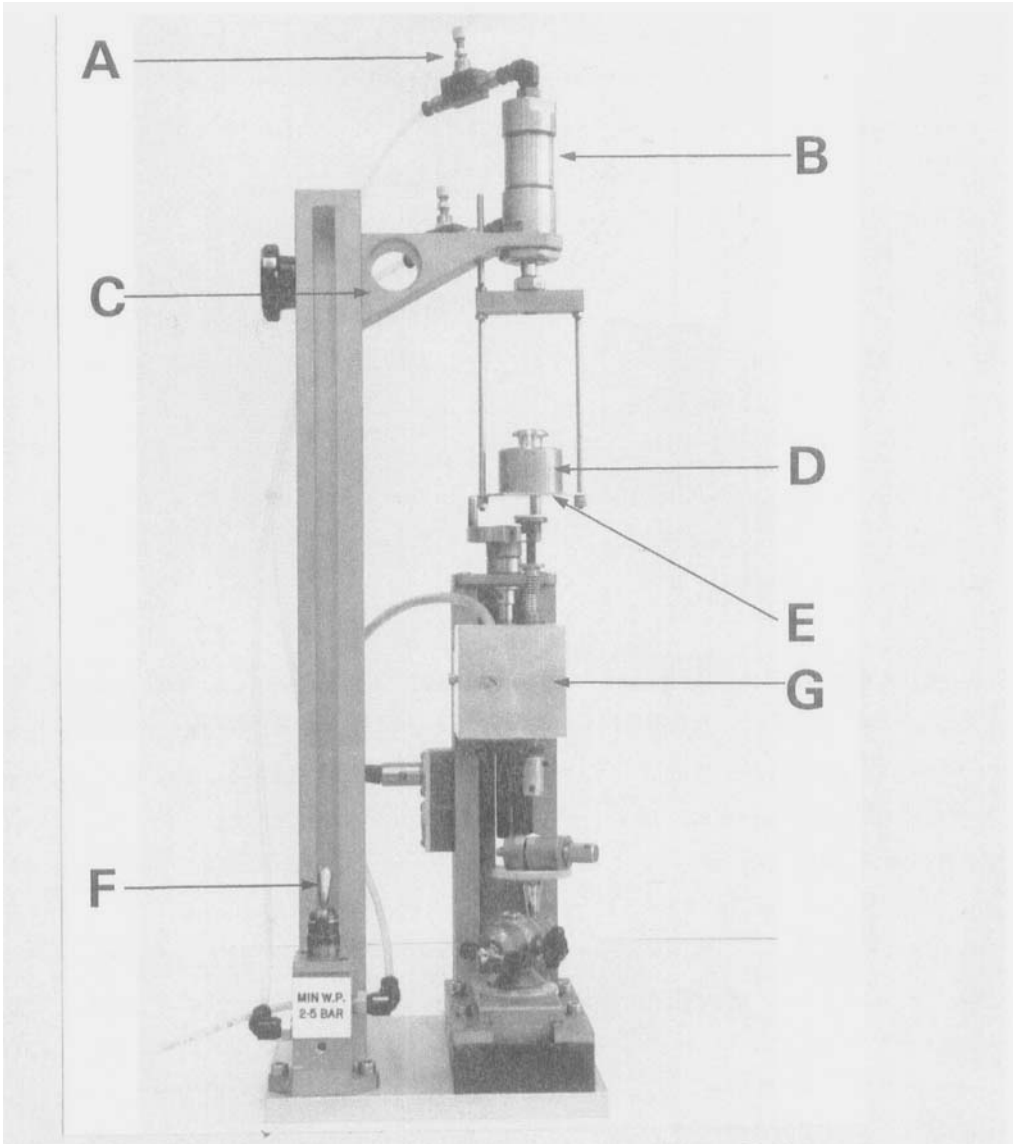
Figure 1 is a general view. This shows the pneumatic piston and its control switch for raising and lowering the indentation load, which is mounted on a height adjustable arm supporting a platen for carrying the test load. Figure 2 is a close up view of the indenter set-up showing the very low pitch screw threaded rod on which is mounted the indenter and LVDT assembly, the position of which can be adjusted by means of the control wheel. It is locked in place from the back of the apparatus. Figure 2 clearly shows how the mounting stage for the test specimens can be moved in all directions using a combination of the allen bolts holding the stage itself, and the two adjustments on the support for the mini vice holding the tablet under test.

METHODS

The modified apparatus has been tested and validated and indentation tests performed over the surfaces of uncoated cores of three commercially available tablets - 'Ridaura', 'Tagamet' 200 mg and 'Tagamet' 800 mg.

Calibration of Indenter

The LVDT (Penny and Giles, P1S 1304) was calibrated *in situ* by the use of slip gauges at an electrical load of 10 kohms. The



A - Control valves for pneumatic loading device
B - Pneumatic piston
C - Adjustable support arm
D - Test weight
E - Loading platen (free to move on supports)
F - Control switch for loading platen
G - Indenter assembly

FIGURE 1
GENERAL VIEW OF MICROINDENTER APPARATUS

entire movement of the transducer was first calibrated using 1 mm slip gauges to determine the central zero output point, and the central region (± 1 mm) was further calibrated at 0.1 mm intervals. The voltage output varied from +12 to -12 volts and further calibration was performed over the region of +1 to -1 volt using 0.01 mm increments. Linear regression of that data gave a slope of -8.538×10^{-2} mm/volt (correlation coefficient R = 0.9992) and thus, in use, the LVDT gave an output to the chart recorder as follows:

1 volt = 85.38 μm and 100mvolt = 8.54 μm

Indentation Testing

The technique for each individual indentation was as follows. With the tablet held firmly in the jaws of the clamp its position was adjusted, by a suitable combination of the three adjustment points on the universally-jointed stage, so that the point on the surface to be measured was normal to the line of the indentation. The indenter was gently lowered onto the tablet surface, at which point the voltage output began to change. After checking that the indenter was still normal to the test surface the indenter was locked in place. The indenter was now in position ready for the test and in contact with the tablet surface under a small pre-load of about 0.05N. The test was commenced by placing the appropriate weight (Table 1) on the loading platform (see Figures 1 and 2) and starting the chart recorder. The indenting load was lowered onto the platen of the indenter by operating the control switch on the hydraulic system. The same switch was used to raise the weight from the indenter after a fixed indentation time of 30 seconds. The test was continued for a further 15 seconds to monitor surface recovery after load removal. Preliminary experiments had shown these times to be satisfactory since the materials displayed

TABLE 1

Summary of Indentation Test Conditions

Tablet Type	Indenter Sphere Diameter (mm)	Indentation		Recovery Time (seconds)
		Time (seconds)	Load (N)	
'Ridaura'	1.66	30	2N	15
'Tagamet' 200 mg	1.66	30	2N	15
'Tagamet' 800 mg	1.66	30	3N	15

little time-dependent deformation under the conditions of the test. The tablet under test was then moved to the next indentation position and the process repeated. Details of the test parameters used for each tablet type are given in Table 1

The above procedure was repeated at a number of points over the surface of each tablet. These test positions are shown in Figure 3 for 'Ridaura', Figure 4 for 'Tagamet' 200 and Figure 5 for 'Tagamet' 800. Ten tablets were tested at each of these positions.

RESULTS AND DISCUSSION

The indentation data is presented as two values. Firstly, Brinell hardness (Pa), as defined by $F/(\pi\ D\ h_1)$, where F is the applied load (N), D is the diameter of the indenting sphere (m) and h_1 (m) is the depth of penetration of the indenter at the end of the loading period. Secondly, as Elastic Quotient

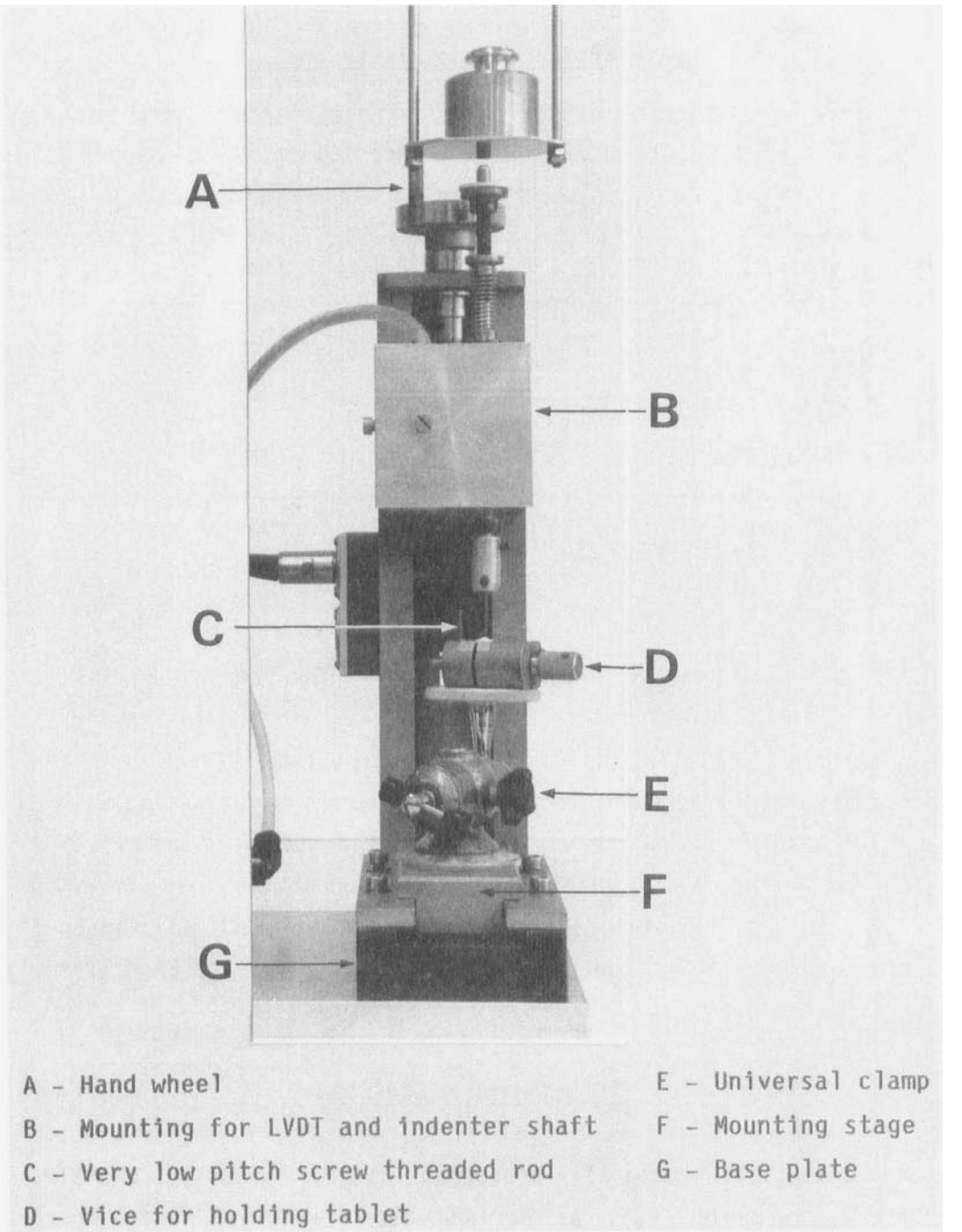


FIGURE 2
CLOSE UP OF INDENTER ASSEMBLY AND UNIVERSAL MOUNTING

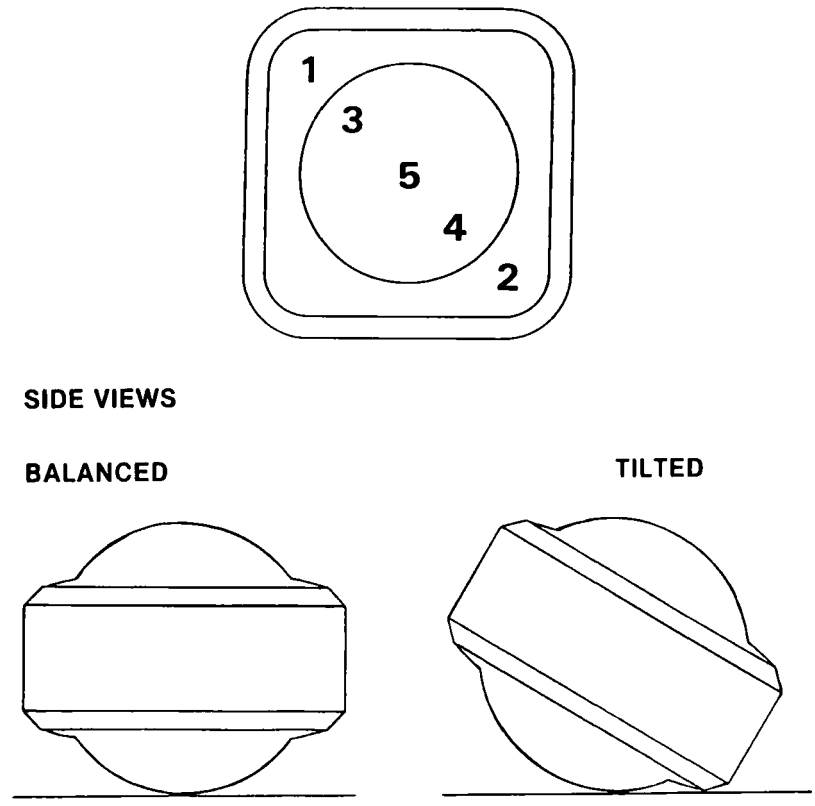


FIGURE 3
INDENTATION POSITIONS ON 'RIDAURA' TABLET

(EQ) which is ratio of the depth of recovery of the indentation on removal of the load (Δh) to the depth of indentation under load (h_1). It is a measure of the resilience of the material. The following are the mean of ten readings from ten separate tablets. The mean coefficient of variation of the readings on any particular tablet ranged from 0.16 to 0.19. This is typical of microindentation testing of tablets and is a function of the inherent variation in the tablet surface, rather than any

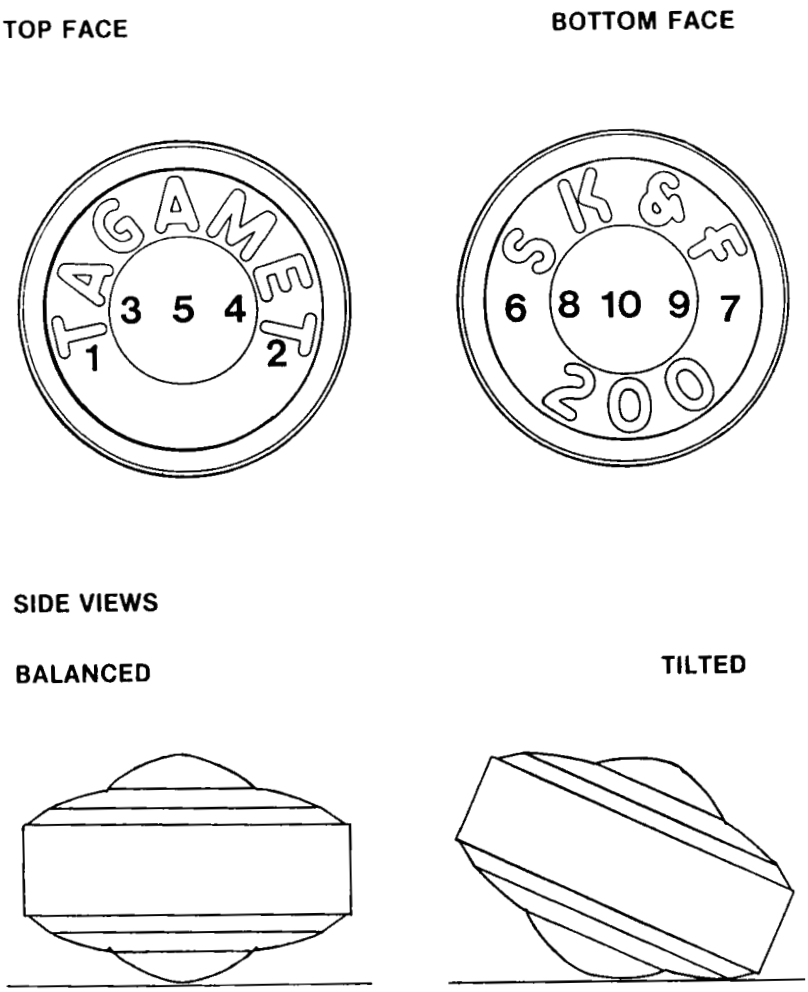


FIGURE 4
INDENTATION POSITIONS ON 'TAGAMET' 200MG TABLET

scatter due to the mechanism of the test. The test positions on the tablet are indicated in Figure 3 for 'Ridaura', Figure 4 for 'Tagamet' 200 mg and Figure 5 for 'Tagamet' 800 mg. In some cases, where appropriate, data from radially symmetrical points have been combined.

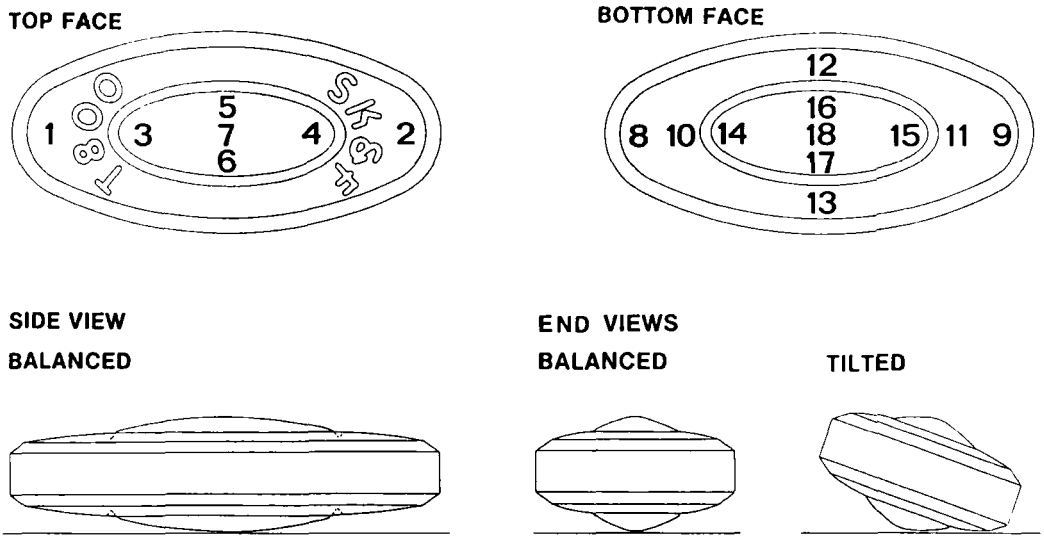


FIGURE 5
INDENTATION POSITIONS ON 'TAGAMET' 800MG TABLET

Table 2 shows the indentation data for Ridaura tablets. The top and bottom surfaces of 'Ridaura' tablets are indistinguishable and thus the data refers to a combination of both.

Positions 1 and 2 are the flatter areas of the tablet and, as can be seen, these are the hardest parts of the tablet surface. This is not unexpected since these are the zones which have been more highly consolidated during compression. The points over the dome of the tablet (positions 3 to 5) have lower hardness values. It is interesting to note the reduction in elastic quotient, and thus resilience, at the top of the dome. This may well reflect the nature of the compact itself in the region of the dome, rather than being a purely surface related phenomenon. The tablet will show reduced consolidation in the region of the domes thus allowing more particle orientation during the indentation process, and hence reducing the recovery of the surface when the indenting load is removed.

TABLE 2

Brinell Hardness and Elastic Quotient data for 'Ridaura' Tablets

Position on tablet (refer to Figure 3)	Brinell hardness (MPa)	Elastic Quotient ($\Delta h/h_1$)
1 and 2	27.0	0.65
3 and 4	20.7	0.65
5	21.4	0.56

Nevertheless, the differences are not great and indicate that with this particular geometry and formulation little trouble should be expected due to undue friability at the dome. Indeed, the initial efforts during tabletting tended to over emphasize the potential friability of the dome, with the result that capping was a far greater problem. Once it was established that the dome could be made at a surface hardness sufficient to withstand abrasion during the coating process without using excessive compaction force, then most of the practical difficulties had been overcome. Unfortunately, from time to time some batches of tablets do exhibit a tendency to erosion on the domes during coating. The use of this technique should allow the investigation and possible identification of the causes of this problem, and hence lead to a solution.

Indentation data for both surfaces of 'Tagamet' 200 mg tablets are shown in Table 3.

TABLE 3

Brinell Hardness and Elastic Quotient data for 'Tagamet' 200 mg Tablets

Position on tablet (refer to Figure 4)	Brinell hardness (MPa)	Elastic Quotient ($\Delta h/h_1$)
Top Face		
1	20.5	0.64
2	20.2	0.77
3	17.0	0.61
4	17.2	0.59
5	16.1	0.50
Bottom Face		
6	22.6	0.56
7	23.0	0.56
8	18.0	0.68
9	18.0	0.70
10	16.1	0.38

The results for 'Tagamet' 200 mg show again that the flattest areas of the tablet (positions 1, 2, 6 and 7) are the hardest and have the highest resilience due to their greater consolidation. The sides of the central dome have intermediate mechanical properties whilst the tablet is softest at the peak of the dome and again shows low resilience at this point. There is quantitative similarity between the data of both upper and lower surfaces.

As with the 'Ridaura' 'Tiltab', the shape for 'Tagamet' 200 mg is an empirically derived best option for the required design, working with a fixed formulation. The initial shape, which was

rejected, gave a product with a peak to the tilt feature rather than the more rounded shape shown in Figure 4.

Further modifications were made, but only to the embossing of the punch with the various monogramming options, and the line surrounding and highlighting the tilt feature.

With experience gained from many years of manufacturing 'Tagamet' 200 mg and from other 'Tiltab' products in development, it was not felt that any difficulties would be encountered in producing this new 'Tiltab' tablet. However, as the relatively low hardness for the tip of the tilt feature indicates, together with the low resilience at this point (suggesting a less consolidated structure compared to the edges of the tablet) some problems of erosion on coating might have been expected and were in fact encountered. It should be remembered that these data are from typical tablets currently being produced for commercial purposes. Like the 'Ridaura' tablet the problems were overcome by balancing the need for a hard tilt feature against the potential for capping of the tablet as a whole, not just the dome. Some factors which have been investigated empirically have been moisture content and granule size distribution, and it is hoped that this will form part of a subsequent study.

Table 4 shows the indentation data for 'Tagamet' 800 mg tablets.

The shape of the 'Tagamet' 800 mg tablet is more complex (see Figure 5) but the results follow similar trends to those of 'Ridaura' and 'Tagamet' 200 mg. The flatter areas of the tablet (positions 1, 2, 8, 9, 10 and 11) are the hardest, with the tablet being softer over the dome. Again the characteristically low resilience at the peak of the dome is repeated. Positions 12 and 13 showed a surprisingly low hardness and resilience compared with other data from the flatter regions of the

TABLE 4

Brinell Hardness and Elastic Quotient Data for 'Tagamet' 800 mg Tablets

Position on tablet (refer to Figure 5)	Brinell hardness (MPa)	Elastic Quotient ($\Delta h/h_1$)
Top Face		
1	36.4	0.72
2	35.8	0.63
3	34.2	0.57
4	30.0	0.51
5	25.9	0.72
6	28.0	0.70
7	31.0	0.51
Bottom Face		
8	39.8	0.77
9	38.4	0.77
10	39.6	0.69
11	37.3	0.68
12	28.6	0.49
13	29.0	0.59
14	30.3	0.55
15	32.3	0.57
16	34.9	0.70
17	34.1	0.71
18	36.4	0.49

tablet. This presumably reflects the nature of the distribution of forces through the elongated shape of this tablet as it is compressed, with propagation towards the ends of the compact being greater than that normal to the direction of compaction.

In the light of experience gained with other 'Tiltab' products the design of the 'Tagamet' 800 mg tablet was selected

to give the minimum changes in surface curvature as would be consistent with a 'Tiltab' as defined in the relevant patents (Tovey, 1983, 1986). Thus fewer problems were expected in commercial production with this design of 'Tiltab', and this has indeed been the case. The generally higher surface hardness and resilience across this tablet, as indicated by these data are probably the result of this design, coupled with the slightly different formulation used in this higher potency 'Tagamet' tablet. Very few problems associated with the tilt feature of 'Tagamet' 800 mg tablets have been encountered in practice.

CONCLUSIONS

The modified microindentation apparatus described here has been shown to be a satisfactory technique to monitor the surface hardness and resilience of tablets with unusual surface curvatures, in particular the range of SK&F 'Tiltab' tablets. The indentation test itself has been shown to pin-point differences in properties over the tablets and has shown differences between various areas of the 'Tiltab' tablet shape, some expected and some not. The designs of the three 'Tiltab' tablets investigated were derived empirically, and the degree of processing problems, in particular those related to surface erosion of the tilt feature or to capping of the tablet, has been shown to be related to the surface hardness and resilience of the different 'Tiltab' tablet shapes. It is hoped that the use of this apparatus and microindentation testing will enable the shape of future 'Tiltab' tablets to be optimised to reduce problems in practice. It is also recognised that formulation factors play a key role in the ability of 'Tiltab' tablets to withstand the stresses of film coating and general handling. This technique should therefore be extremely useful in determining both the optimum excipients to use in new

formulations, and the nature of the more important processing parameters for existing products being converted to the 'Tiltab' tablet format.

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