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Development of a micro-dosing system for fine powder using a vibrating capillary. Part 2. The implementation of a process analytical technology tool in a closed-loop dosing system

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ABSTRACT

In an earlier study, a micro-dosing system for fine powder using a vibrating capillary which can precisely dose various lactose powders, was introduced. In scaling up to a multi-track dosing system, it was suggested that additional track cut-offs can improve the dosing performance by reducing the overruns. A non-contact in-line control unit within a closed-loop system is required to achieve this goal. Due to its very fast response time (a few milliseconds) a capacitive sensor was integrated together with multi track cut-offs into a closed-loop dosing system. With this improvement a standard dosing deviation as low as 0.1 mg was achieved. The results suggest its application in precise filling of fine pharmaceutical powders.

1. Introduction

Precise filling of fine powders can be found in many industrial operations. Such operations include the micro-dosing of powder in pharmaceutical research, development and production. For instance, the production of dry powder inhalers requires small quantity of fine powder being precisely metered into either capsules or blister pockets. A single dose ranges from only several milligrams up to 20 mg of powder. The validation of mass is done currently volumetrically. However, powders may have very different bulk densities depending on their manufacturing methods and handling processes during production. In some cases, the dosed value can be altered partly by changing the machinery parameter settings. For instance, the dosed weight of vacuum drum filler can be modified by altering the strength of suction pressure. However, in cases of formulation development where different powders are to be filled in small scale or in filling for pre-clinic research where the target dose might vary in a broad range, the only practical way is to change the dosing heads. As a result, it increases the process time and a number of dosing heads with different cavities are needed.

1.1. *A micro-dosing system for fine powders using a vibrating capillary*

Taking these demands into consideration, a simple dosing system with a straightforward construction was developed. The frequency and the amplitude of applied vibration (Matsusaka et al., 1995, 1996; Yang and Li, 2003; Yang and Evans, 2005; Jiang et al., 2006) were the contributing factors to the mean dosing value and the dosing accuracy. It was found that by careful selection of the capillary orifice, the start/stop of powder flow can be controlled by switching on/off the vibration (Yang and Evans, 2005). In a simple case, the desired dose can be controlled by adjusting the duration of vibration.

A 100% fill weight control can be achieved by implementing a closed-loop control using a weighing cell. However, it is difficult to implement it into an industrial scale production line due to several limitations (Chen et al., 2011). In this case, a closed-loop control using a non-contact in-line detection principle can provide more advantages. With this intention, a sensitive capacitive sensor was integrated into the dosing system presented.

1.2. *Measurement of the flow of powder bulk using a capacitive sensor*

A simple capacitive sensor has two parallel electrode plates. The permittivity change between electrodes causes a change of

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capacitance when powder passes through the sensor. The measurement is done by comparing the signal with a reference capacitor circuit. For a monolith, for example, a piece of tablet with a size smaller than the height of the electrodes obtains always a peak value. The peak value or the effective capacitance (C_{eff}) can be calculated from the capacitance values of two sensors which are connected in parallel, see Eq. (1):

$$C_{eff} = \frac{C_{solid} C_{gas}}{C_{solid} + C_{gas}} \quad (1)$$

In case of moving powder bulk which approximates to a continuum, its effective permittivity contributes by the fraction ratio of the solid material to the medium and their different electrical permittivity. Therefore, different functions are used to calculate the effective permittivity (Bruggeman, 1935). In recent years the industrial practice has focused mainly on the visualization of the powder flow regime (Rao et al., 2001; Chaniecki et al., 2006; Takei and Zhao, 2008; Sun et al., 2008; Niedostatkiewicz et al., 2010), for instance, to study the material distribution in a pneumatic conveyor for powder. In these studies, the signals collected by a few pairs of electrodes are used to visualize the material distribution in 2D or in 3D electrical capacitance tomography. Due to the complexity of such system and the lack of resolution, the quantitative measurement of a few milligrams of powder is less studied.

1.3. The phenomenon of self-valve and the need of an active track cut-off

Janssen proved that on large scale the weight of powder bed is mainly supported by the wall (Janssen, 1895). By analysing the micro-structures in photo-elastic granular material, it was found that the transmission of static forces in bulk powder is through a network built by anisotropic force chains (Howell et al., 1999a). They are formed by the spatial connections among particles (Howell et al., 1999b; Corwin et al., 2005).

In a capillary with a narrow orifice, the formation and the release of such micro-mechanical structures are influenced not only by the material properties but also by the local arrangement of particles. When the capillary has an orifice with the proper size, powder could be enclosed in it even without the need of a valve under the exit (Yang and Evans, 2005), which is called the *self-valve phenomenon*.

Once external vibration is applied to the powder bed and if the induced shear force exceeds the elastic threshold value, the network of force chains will be broken and powder starts to flow under gravity force. When the vibration is stopped, the particles rearrange themselves rapidly and establish a new network of force chains that stops the flow. The release and rebuild of such clogging structure take only several milliseconds in the setup presented here. Hence, discharge of powder is possible using a narrow capillary in a controlled manner by switching the vibration on/off.

On the other hand, the strength of these connections renders the cohesiveness among the particles. Feng and Hays (2003) compared the cohesive forces among particles and their forces of gravity. When the particle size decreases to 10 μm, the van der Waals forces between two particles are estimated to be at least a hundred-fold larger than their forces of gravity (10–500 nN to 0.005 nN). Consequently, the shear forces induced by the vibration alone might not be sufficient to overcome the cohesive forces among primary particles. However, most of large agglomerates break into small agglomerates due to the collision induced by the vibration (Matsusaka et al., 1995).

The effectiveness of such self-valve phenomena depends on a geometric factor: the ratio of the orifice diameter to the average particle size of powder. A small orifice has an increased risk to block the capillary even in presence of vibration, a too large orifice may lead to overrun even in absence of vibration. Furthermore, it could be problematic when powders have relative broad particle size distribution. In this case, the orifice of the capillary is often chosen for particles with large diameter in order to reduce the possibility of blockage. Consequently, the possibility of overruns is increased. An additional track cut-off is supposed to be helpful. Further, in scaling up to a multi-track dosing system, the fluctuation of powder flow and the minor difference of flow rate among diverse dosing heads could increase the flow rate variability. Again, a mechanism for individual adjustment of every single track is needed to meet the requirement of 100% control of fill weight.

In general, two conceivable strategies can be followed:

- the individual control of the vibration generators, and
- the individual cut-off of powder flow with one common vibration generator.

In the first concept, multiple actuators and signal controllers are needed which increases the complexity of the system and the cost. Accordingly, the system described here is based on the latter principle.

2. Materials and methods

2.1. Material

Several kinds of inhalation grade lactose powder were used in the experiments: Inhalac 70, Inhalac 120, Inhalac 230, Sorbolac 400 (all from Meggle GmbH, Germany, Wasserburg) and Respitose SV 003 (DFE Pharma, Germany, Goch). Their particle sizes and size distribution as well as other powder properties are shown in Table 1. A laser diffractometer (Sympatec HELOS GmbH, Germany, Clausthal Zellerfeld) was used to measure the volume particle size distribution. Powders were measured in dry form after dispersing in air using compressed air at a pressure of 3 bar (RODOS dispersion module). The bulk density, the tapped density and the Carr's index were taken from the specification provided by the manufacturers. The angle of repose were measured using a tester built according to DIN ISO 4324. The results were taken from the average value of five experiments with exception of Sorbolac 400, which did not pass the orifice despite stirring.

2.2. The machinery assembly and the procedure

A multi-track micro-dosing system for fine powder was assembled as shown in Fig. 1.

Powder was introduced through a small reservoir (1) into three capillaries (2) and from there to be dosed into cavities (5). The capillaries were bolted together to the actuator. An additional track cut-off (4) was installed under the capillaries, see Fig. 1. Under the cut-offs a capacitive sensor (3) was applied as the in-line non-contact controller. In Fig. 1 the track cut-offs (4) were removed to expose the capacitive sensor (3) (Advance Mass Verification sensor, Uhlmann VisioTec GmbH, Germany, Laupheim); in Fig. 1 the capacitive sensor (3) is removed to expose the additional track cut-offs (4).

The vibration parameters are set on frequency 338 Hz, amplitude corresponding to an input voltage 30 V. The strength of vacuum applied on the capillaries was measured with a pressure transmitter CPA2500 (WIKA Alexander Wiegand GmbH & Co. KG, Germany, Klingenberg). For ease of use, the vacuum was triggered on/off manually.

A few milligrams of powder pass through the sensor and cause the change of capacitance, which can be instantaneously calculated with a strength-time curve. The integral of curve is compared with a given target. Once the target value is achieved in one single track,

Table 1
Equivalent spherical volume diameter of powder used and their flow parameters.

Material	X (10) [μm]	X (50) [μm]	X (90) [μm]	Carr's index	Bulk density	Tapped density	Angle of repose
Inhalac 70	102.4	205.7	321.9	10.6	0.59	0.66	35
Inhalac 120	88	135.2	177.9	11.4	0.7	0.79	35.4
Inhalac 230	27.7	77.1	123.3	13.4	0.71	0.82	37.9
Respitose SV 003	34	66.2	104.4	19.2	0.63	0.78	38.7
Sorbolac 400	1.32	6.71	15.5	38.1	0.39	0.63	No flow

the cut-off mechanism (see Fig. 1) stops the flow immediately. All three dosing tracks are working in the same way and were synchronized.

However, to investigate the overruns, the track cut-offs in Fig. 1 were switched off. A sieved lactose monohydrate quality (Inhalac 120) was used. A factorial experiment plan was applied and the results were evaluated with the statistical software Minitab (Brandon Court, UK). The frequency, the amplitude and the clamp position of the capillary were studied systematically. The occurrence rates of overruns were taken as the output parameters. The frequency varied from 250 Hz to 450 Hz, the amplitude varied from 20 V to 45 V and the clamp position were fixed to two position 1 mm and 8 mm to the longitudinal centre of the capillary, respectively.

The functionality of two different kinds of track cut-off systems (Fig. 2) was examined in Section 3.3. The dosing results collected by the capacitive sensor were verified with a weighing cell of type AW-AD (Wipotec, Kaiserslautern, Germany).

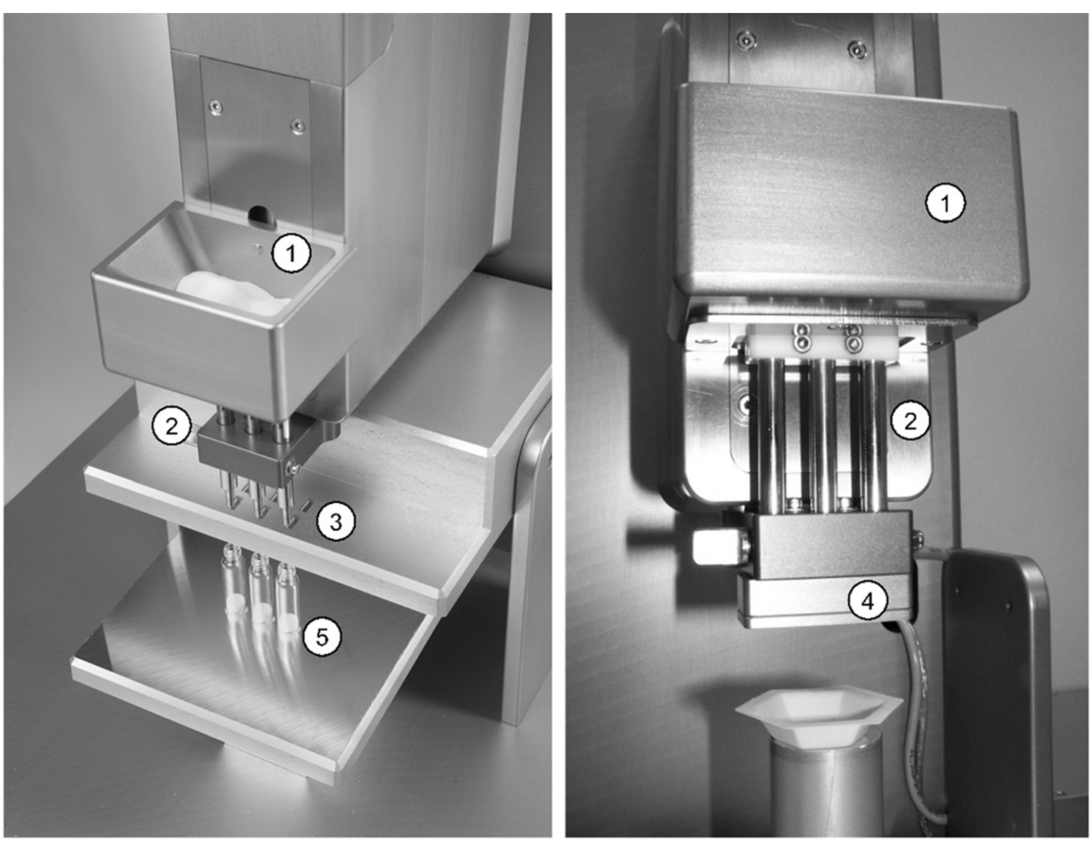
3. Results and discussion

As mentioned in Section 1, the precision of filling was determined by an accurate measurement of capacitance and an effective start/stop control. The contributing factors on the measurement, for instance, the physical properties of powders were discussed earlier (Chen et al., 2011). The focus of this study was particularly on the formation of powder clogging and its release in a narrow capillary which influenced the start/stop control.

3.1. The formation and the release of clogging in a narrow capillary

Four kinds of lactose powder were used to get understanding about the formation and the release of clogging. The capillary used was made of two identical halves. Its orifice diameter was about 0.8 mm. After 5 min long vibration, the capillary was carefully opened. Fig. 3 showed where the clogging builds up: it occurred in the most cases at the narrow end of the cone in the capillary regardless of the powder properties.

To study the patterns of particle movement in a narrow capillary, two kinds of lactose monohydrate, Inhalac 120 and Inhalac 230 were filled into a glass Pasteur capillary, respectively. A small portion of lactose monohydrate (Inhalac 120, coloured with E127) was used to visualize the movement of powder bed. A high speed



(a) three-track dosing system with an electrical capacitive sensor (b) dosing system with additional track cut-offs

Fig. 1. (a and b) A multi-track micro-dosing system integrated with closed-loop control.

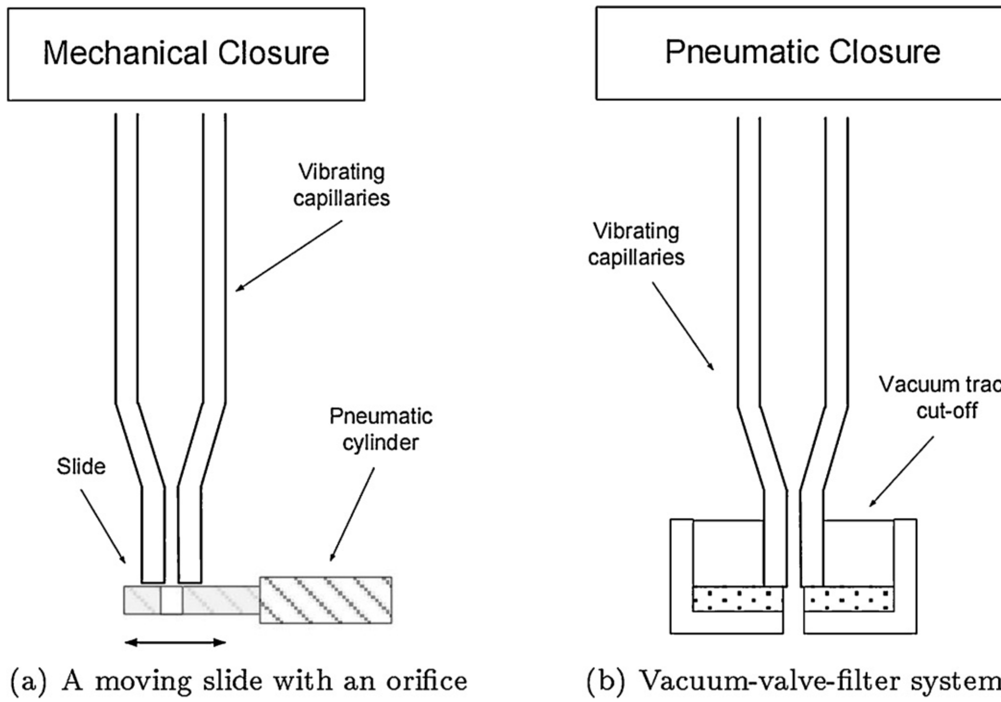


Fig. 2. (a and b) Two kinds of track cut-off with different designs.

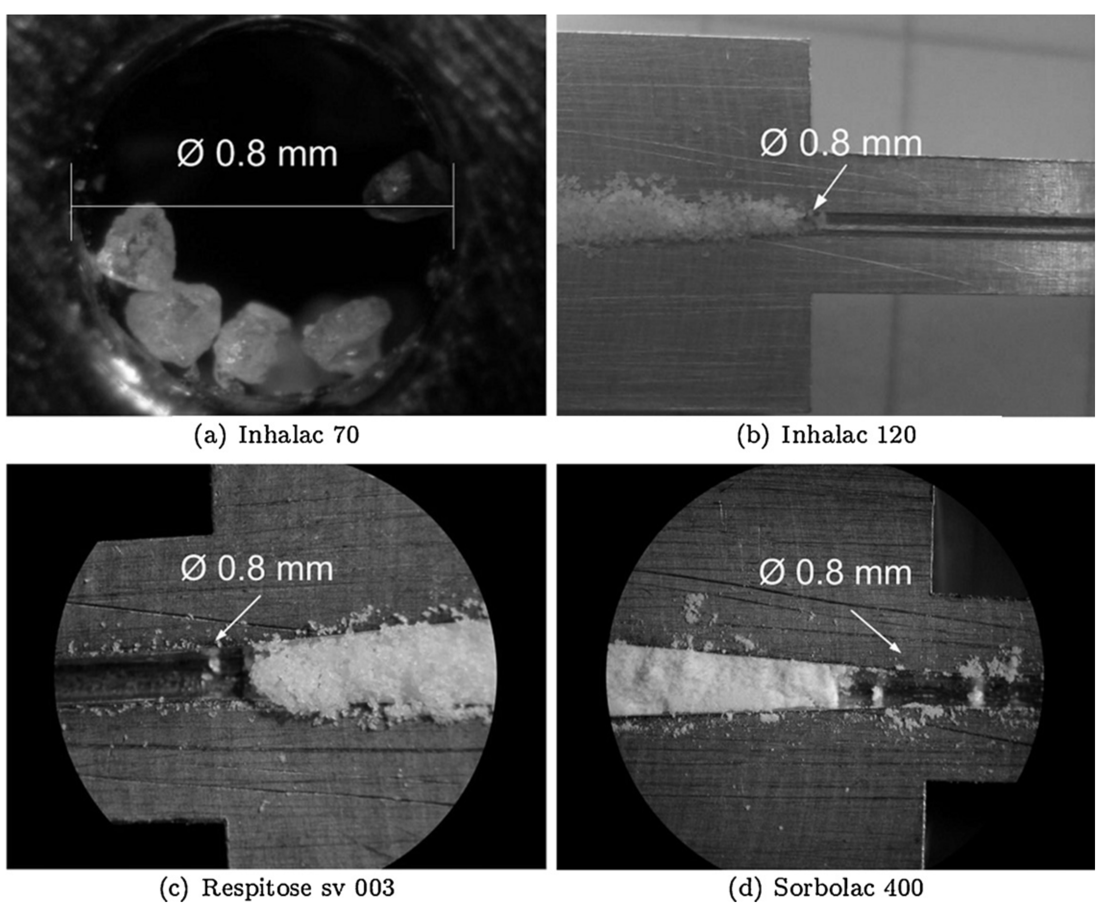
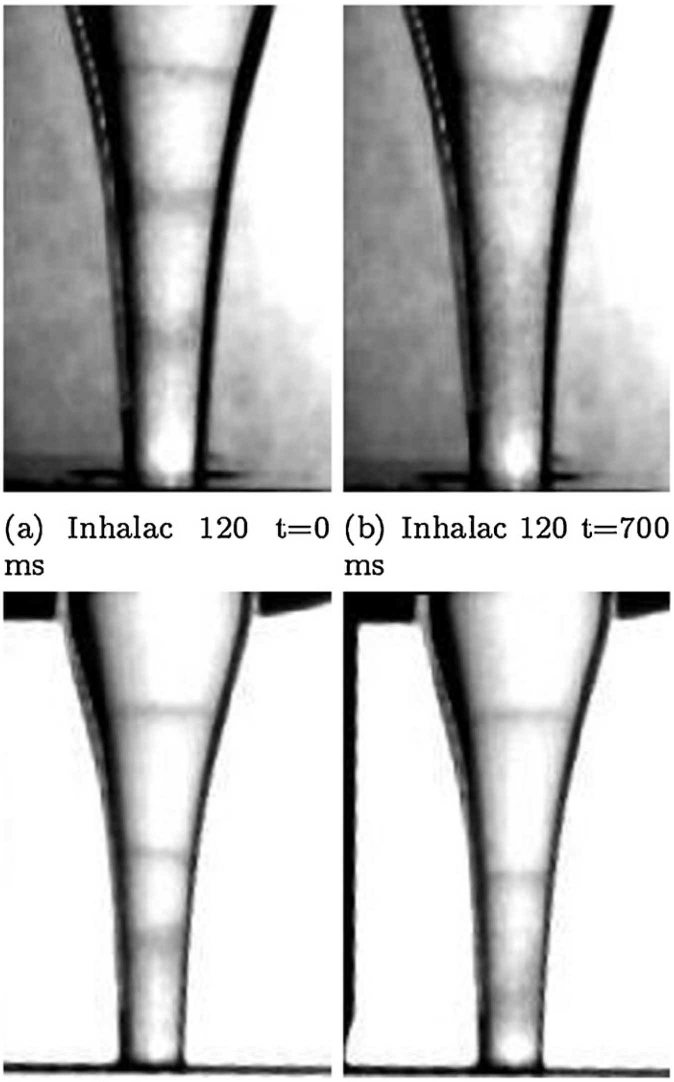


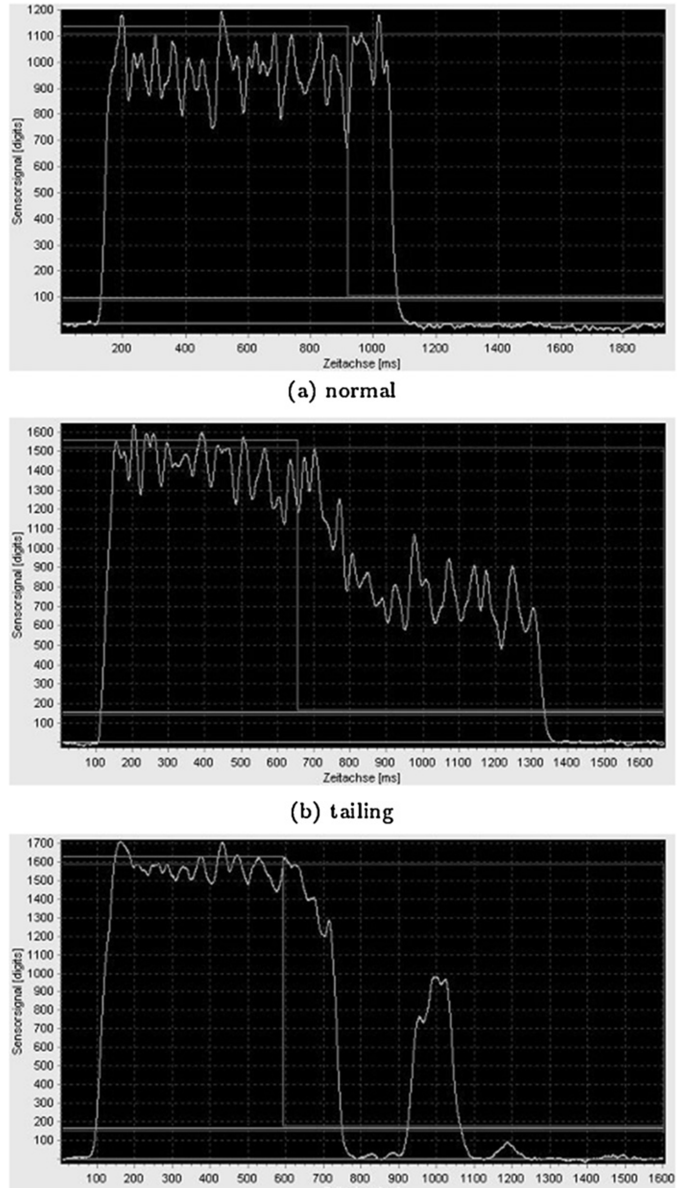
Fig. 3. (a–d) The clogging in a narrow two-piece capillary (Ø 0.8 mm).



(a) Inhalac 120 t=0 ms (b) Inhalac 120 t=700 ms

(c) Inhalac 230 t=0 ms (d) Inhalac 230 t=700 ms

Fig. 4. (a–d) The flow patterns of powder in a glass capillary.



(a) normal

(b) tailing

(c) pulsation

Fig. 5. (a–c) The signature of the flow behaviour with or without overruns.

camera (Festo AG & Co. KG, Germany, Esslingen) was focused on the cone of capillary and set on a frame rate of 200 pictures per second.

It was found that the layer of powder near the capillary wall turned rapidly cloudy under the vibration, see Fig. 4. As a contrast, those particles in deep layer in the centre moved down in rather a collective way. The reason is that the vibration is well damped in a large distance. Those particles in the middle of the powder bed cannot arrange themselves freely due to the damping effect. Therefore, the formation and the release of the clogging take place firstly at the narrow opening, where the vibration is transferred more effectively. Note that Inhalac 230 showed dilatant behaviour compared to Inhalac 120 due to its small particle size and the relative stronger cohesive forces among particles against the gravity forces.

3.2. Occurrence of overruns

Fig. 5 shows typical signatures of different dosing events in an electrical capacitance measurement: (a) a common dosing curve with clear start/stop (see Fig. 5(a)); (b) a tailing curve after stop of the vibration (see Fig. 5 (b)); (c) a dosing curve with pulsation (see Fig. 5(c)).

Here the frequency was set on 338 Hz; the amplitude was set on 30 V. The orifice diameter of the capillary used was about 0.8 mm. As a matter of course, a delayed stop of powder flow in Fig. 5(b) or an uncontrolled stop of powder flow in Fig. 5(c) leaded to a large variability of fill weight. A three-factor, two-level experiment was performed to investigate the influencing factors: the clamp position, the amplitude and the frequency.

In Fig. 6, a Pareto diagram on the left side indicates that all three factors have impact on the occurrence of the uncontrolled release. The figures on the right side show that the overruns occur more frequently under the vibration with lower amplitude and low frequency. Additionally, the clamp position increases the occurrence of uncontrolled release significantly. As discussed in Section 1, the transmission of vibration depends on the clamp position at the capillary. In fact, the clamp position also showed its contribution to the mean dosed value (Chen et al., 2011).

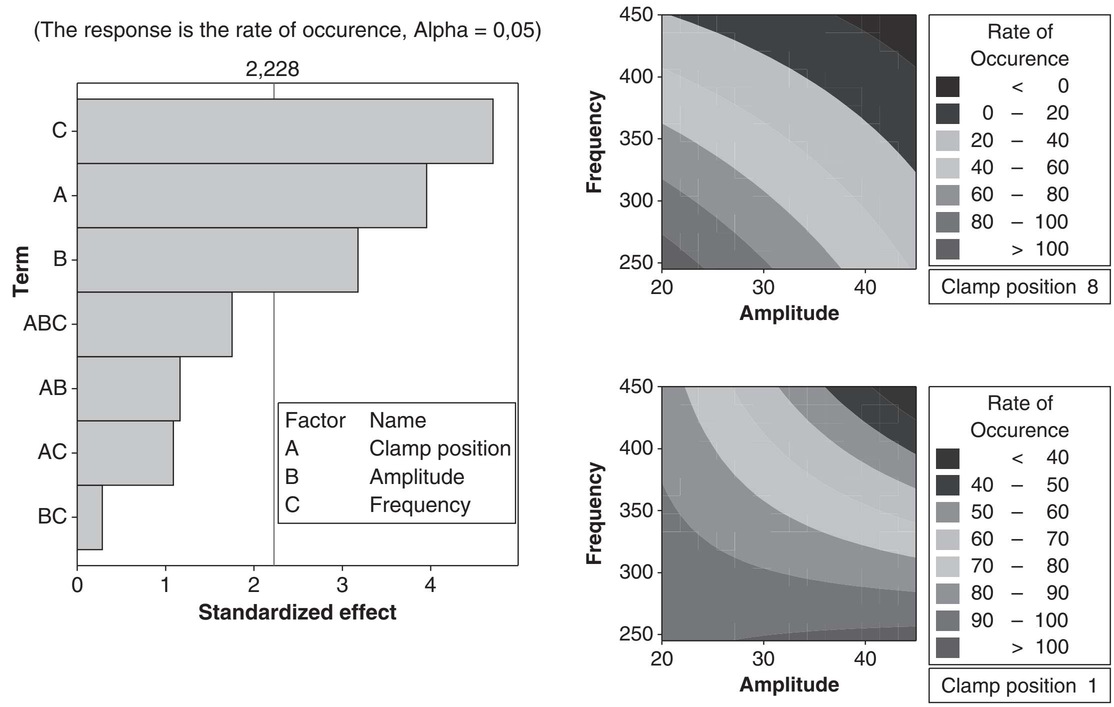


Fig. 6. The factors affecting the uncontrolled powder breakage.

3.3. Control strategy in a close-loop dosing system

In order to control the fill weight, an electrical capacitive sensor with two parallel electrodes was integrated into the dosing system, see Fig. 7. Depending on powder properties and the amount of powder passing through the sensor, the generated electrical signals differ from each other. These signals can be calculated with time-integral of the curve and described with a dimensionless "area under the curve". In an earlier study (Chen et al., 2011), these integral values were found to correspond very well to the quantities of powder with a broad range of pharmaceutical powders. Therefore, it is possible to use the capacitive sensor as a non-contact in-line

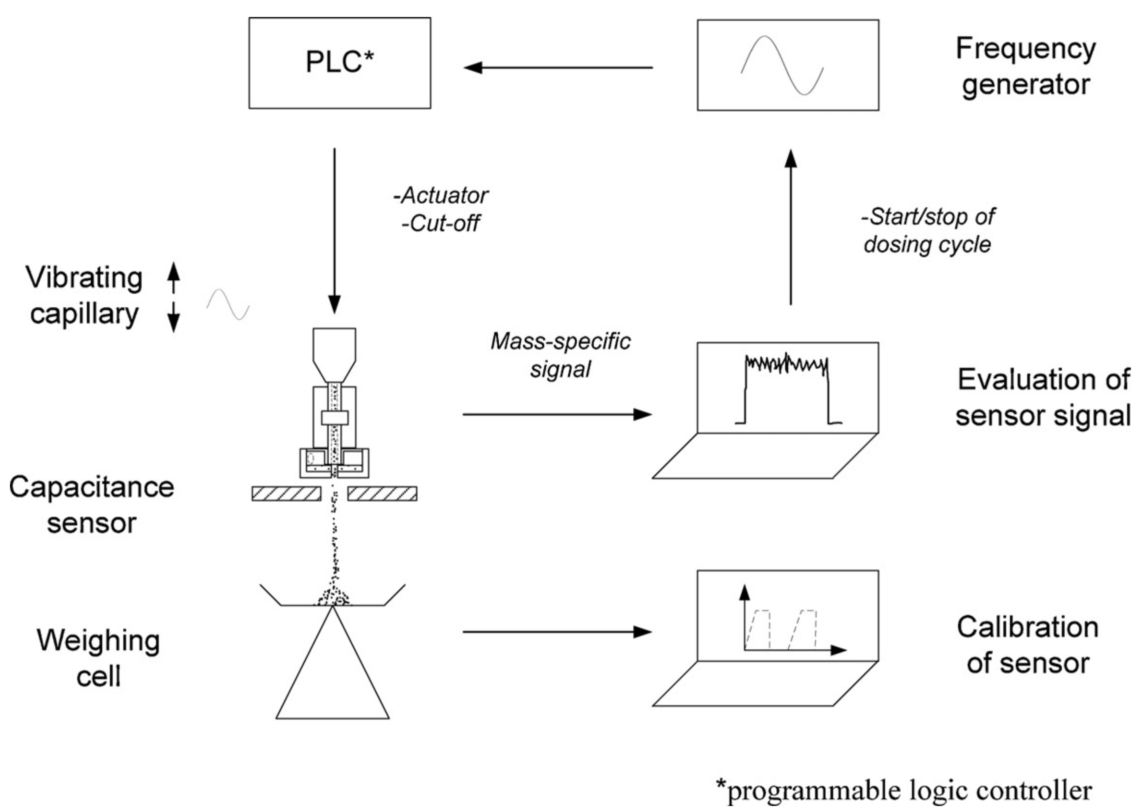


Fig. 7. A closed-loop micro-dosing system with a capacitive sensor as feedback control.

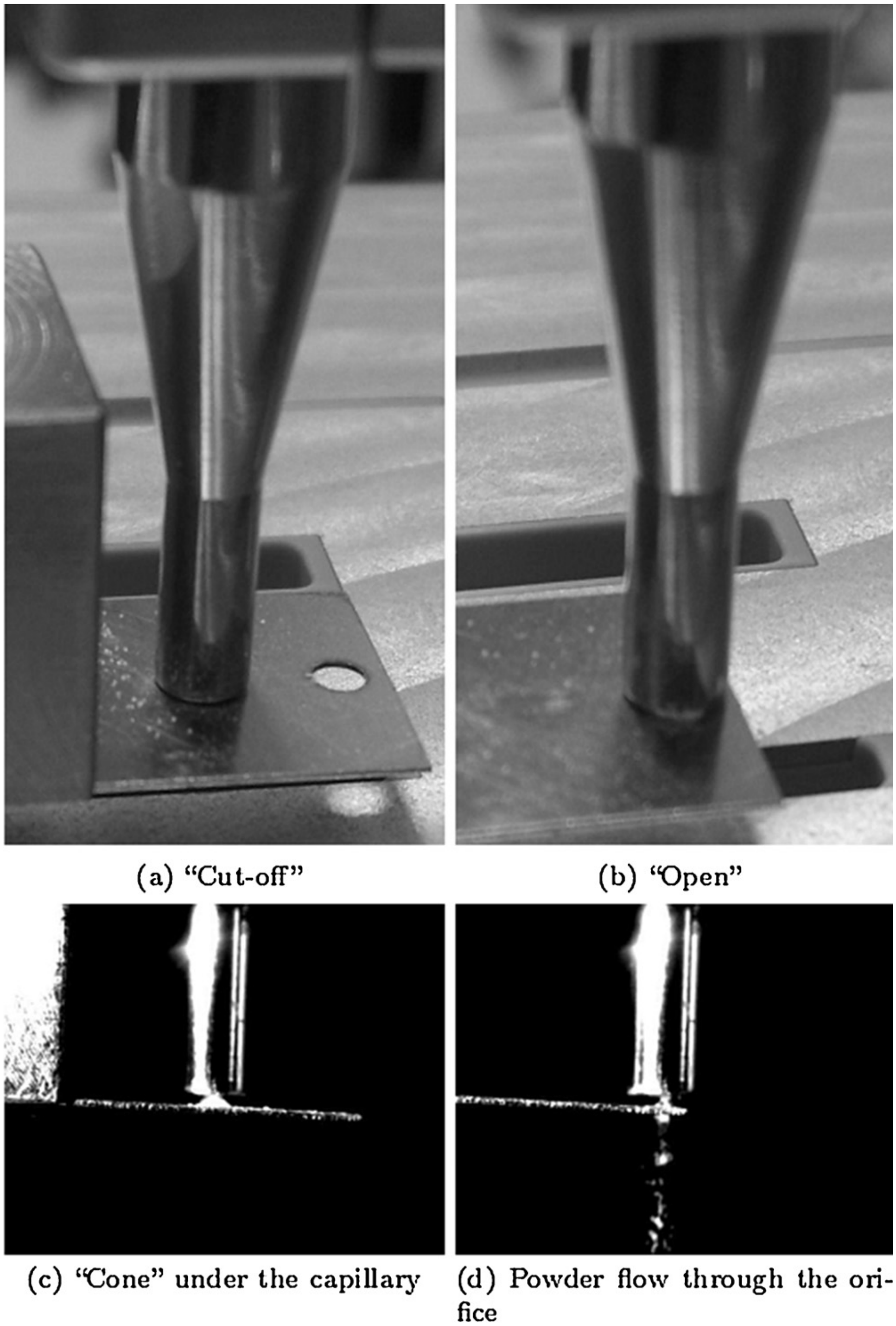


Fig. 8. (a–d) The working principle of a moving slide with an orifice.

control unit in a closed-loop dosing system. By comparing with a given target value, a feedback is sent to the system instantaneously. Once the target value is achieved, the dosing cycle must be stopped. In such case, an exact start/stop control is critical to dispense powder precisely. As shown in Fig. 7, a track cut-off is assembled under the capillary. The on/off of track cut-off is triggered by the control unit.

As mentioned in Section 1.3, there are two strategies to establish individual control of each dosing track. Instead of individually adjusting the vibration on each dosing unit, separate track cut-offs were implemented into the system. For comparison, two kinds of track cut-offs as described in Fig. 2(a) and (b) were assembled to the capillary individually.

3.3.1. *Cut-off with a straight-moving slide*

A moving slide with an orifice as shown in Fig. 2(a) was applied to the capillary. When the slide travels to the position "open", powder starts to flow through the orifice and is dispensed. Once the target value is achieved, the slide travels back to "stop" position and powder flow discontinues instantaneously. In this setup, powder forms a tiny cone directly under the orifice of the capillary, see

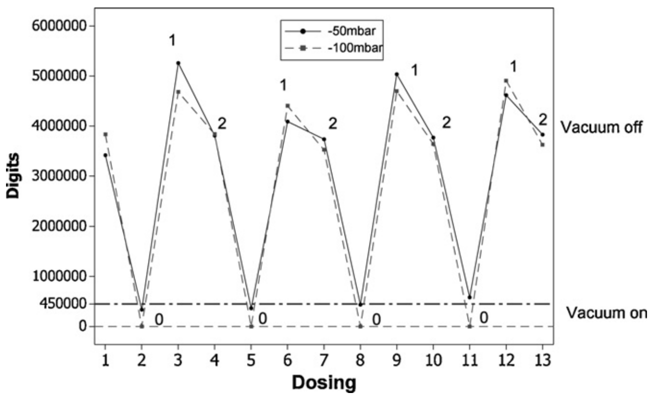


Fig. 9. The dosing chart under a local under pressure about 50 mbar and 100 mbar.

Fig. 8. However, it was observed that a certain distance between the slide and the capillary is needed to keep the capillary orifice free.

When the distance is too small, the capillary could be blocked. The observation with high speed photography discloses that the shear motion of the slide is the cause of blockage. Another argument against this approach is that an additional cleaning operation is needed to deal with the residual powder on the slide surface, introducing another source of error into the system.

3.3.2. *Cut-off with miniature vacuum–valve–filter system*

Another design is to apply a smooth vacuum to the orifice of the capillary by means of a set of filters and valves. The whole setup is shown in Fig. 1.

Powder flow is initiated immediately once the vibration starts. When the vacuum is switched on, the particles near the filter clog together due to the negative pressure and form a plug-like structure that stops the powder flow even in presence of the vibration. However, when vacuum is switched off, this clogging will be released instantaneously by the vibration, see Fig. 9. Therefore, the influence of local vacuum was examined with given parameters (Inhalac 120, frequency 338 Hz, amplitude 30 V). For ease of use, the valve was switched manually.

The lower part in Fig. 9 shows that a local under pressure 100 mbar less than the environment atmosphere is necessary to ensure an effective valve function. A local pressure of less than 50 mbar is not enough to cut off powder flow effectively, see also Fig. 10. However, once the vacuum is switched off, powder could be dosed again. An additional experiment indicated that applying a lower pressure with a great difference than the environment atmosphere did not show significant advantages. Moreover, the electric capacitive sensor gave more details about dosing events with or without vacuum. In Fig. 10, individual spectral curves show the effect of vacuum cut-off.

As mentioned in Section 1.3, the material properties influence the formation of plugs significantly. However, for a given powder, the balance of formation and release of clogging depends on the relative strength of applied vacuum and vibration. Additionally, a rapid formation of negative pressure is essential to ensure a precise cut-off of powder flow. Therefore, the construction of valve, pump and even the length of tube must be taken into account.

To validate the system, Inhalac 120 was introduced in the dosing system; the vibrating parameters were set on frequency 338 Hz, amplitude 33 V. In Table 2 the average dosed value from 100 cycles recorded by the weighing cell is compared with the results measured by the sensor; the target value was set on 1,000,000 digits.

Table 2
The effect of vacuum–valve–filter cut-off.

Variable	Weight [mg]		Sensor [digit]	
	With vacuum	Without vacuum	With vacuum	Without vacuum
Mean	3.62	5.05	1,530,336	278,6339
SD	0.09	0.36	14,246	199,674
Min	3.4	4.5	1,493,589	2,501,660
Max	3.8	6.7	1,567,219	3,768,812
RSD	2.49	7.08	0.93	7.17

Table 3
The improvement of dosing accuracy in gradual dosing.

Frequency 1/2 [Hz]	Amplitude 1/2 [V]	Mean [mg]/[digit]	SD [mg]/[digit]	RSD [%]
338/338	33/33	6.81/3003299.03	0.13/17754.37	1.91/0.59
338/345	33/33	6.65/2996667.02	0.15/22970.79	2.26/0.77
345/338	33/33	6.69/3006142.70	0.14/28549.73	2.14/0.95
338/338	33/40	6.30/2979425.37	0.11/25475.98	1.82/0.86
338/338	40/33	7.04/3020009.01	0.12/23350.95	1.72/0.77

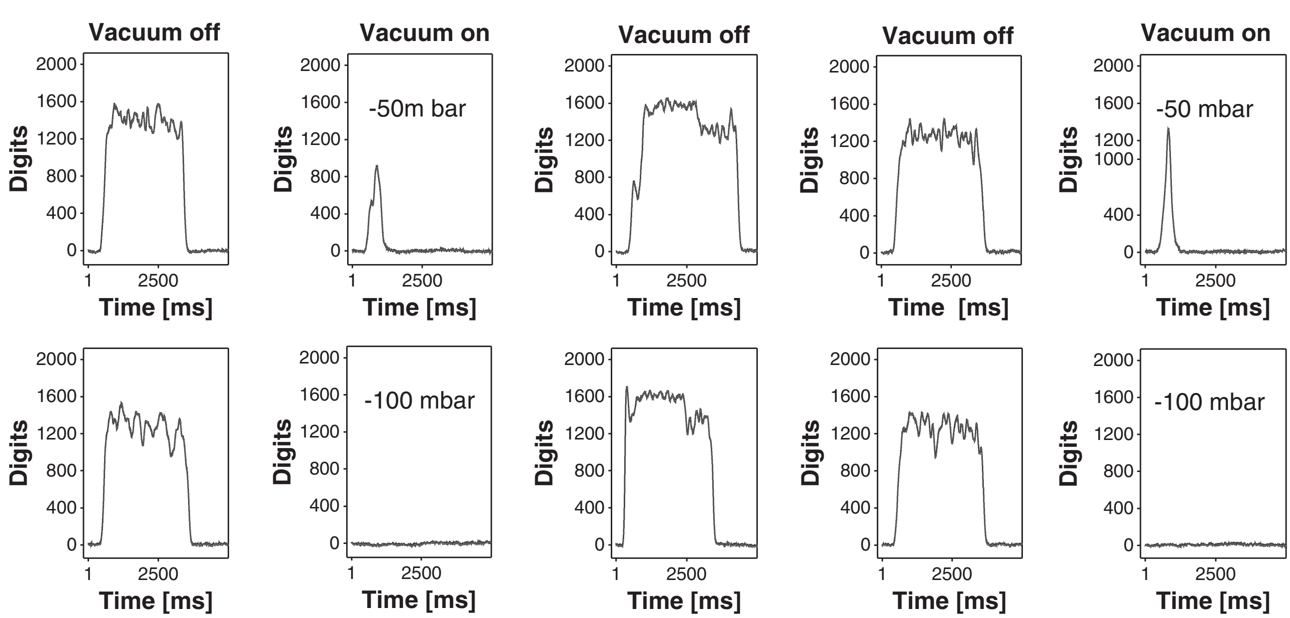


Fig. 10. The spectra curves of dosing with the vacuum-valve-filter cut-off (top line 50 mbar; bottom line 100 mbar).

As a result, the implementation of vacuum track cut-off increased the dosing accuracy in general by reducing overruns. As less of a surprise, the average dosed value with the track cut-offs is closer to target, although the dosed value is always more than the target value due to the response time needed in a common closed-loop dosing system.

3.3.3. Control of dosing process in a gradual manner

In a manual weighing operation, once the actual mass is close to the target value, only very little volume of material will be added stepwise. Despite the great flexibility it is a time consuming operation and could lead to unnecessary variability. An automated operation similar to manual weighing could be accomplished by the setups presented. The desired dosing amount is divided into two parts which could be dosed using different vibrating parameter settings. Once the dosed amount achieves the first level (for example 80% of target value), either or both of frequency and amplitude are changed (downgraded or upgraded) when necessary. Inhalac 120 was used again as reference material in this part of investigation. The target value was set at 2,500,000 digits according to the capacitive measurement. Due to the response time in the feedback system, the actual end point of dosing is about 3,000,000 digits. The average dosed value and their standard deviation were calculated from 100 dosing cycles; different parameter settings of vibration were compared in Table 3. Regardless of various settings of frequency and amplitude, the material delay is constant. There were no significant differences between weight control and measurement using the capacitive sensor. However, it is possible to approach the target value by simply varying the parameter settings.

It is worth mentioning that synchronization is needed in case of automated multi-track dosing system. Once the target value on one of three tracks is achieved, its own vacuum valving is immediately triggered and cuts off the powder flow. The dosing procedure will continue until the target values are achieved on each of the other tracks.

4. Conclusion

As part of the development of a micro-dosing system for fine powders, a capacitive sensor was integrated as feedback control to establish a 100% control of fill weight. Its implementation not only provided a deep insight in to the dosing process in terms of in-process control, but also improved the dosing performance significantly. Additionally, in order to reduce the overruns two different kinds of track cut-offs were compared. With a cut-off based on vacuum–valve–filter principle, a good uniformity of mass with a standard deviation of about 0.1 mg was obtained. The results suggest the potential application in many operations involving precise filling of small quantity of pharmaceutical powders.

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