

Flow properties of powdery or granular filling substances of dangerous goods packagings—Comparison of the measurement of the angle of repose and the determination of the Hausner ratio

Eva Schlick-Hasper  | John Bethke | Nico Vogler | Thomas Goedecke

Federal Institute for Materials Research and Testing (BAM), Berlin, Germany

Correspondence

Eva Schlick-Hasper, Division 3.1 Dangerous Goods Packagings, Federal Institute for Materials Research and Testing (BAM), Berlin, Germany.

Email: eva.schlick-hasper@bam.de

To ensure safety when transporting dangerous goods, it is important to specify the flow properties of the respective solid filling substance of the packagings. For this purpose, the angle of repose is currently used for the UN approvals in Germany. Measurements were carried out on 12 powdery or granular substances applying the angle of repose measuring methods customary in the test centres. The results of the methods differ significantly from each other. In addition, some of the techniques cannot be applied for very cohesive or coarse-grained materials. The results for the angle of repose show a strong scatter for some constellations (coefficient of variation more than 20%). Safety during transport of dangerous goods cannot be guaranteed with this currently practised system of measuring the angle of repose. As a consequence, an alternative parameter to characterize the flow properties of bulk materials should be used in the recognized test centers for dangerous goods packagings, such as the Hausner ratio. This approach leads to more precise test results for the substances examined (maximum coefficient of variation 2.8%). It also has advantages in terms of applicability and occupational safety. Since the flow properties are safety-relevant, both in terms of mechanical safety and safety against the release of dangerous substances, the testing practice in the recognized test labs should be improved and standardized.

KEYWORDS

angle of repose, flow properties, flowability, Hausner ratio, powders, sift-proofness

1 | INTRODUCTION

The UN Model Regulations (UN 6.1.5.2.1¹) state that substances to be transported in dangerous goods packagings may be replaced by other substances during the design type tests, unless this would invalidate the test results. For solids, this substance must have the same physical properties (mass, grain size) as the substance to be carried.

The physical properties of the replacement filling substance are to be documented in the test report.

In the case of German approvals for packagings for solid dangerous goods, the test report and the certificate of approval must also indicate the minimum angle of repose of the filling substances for which the packaging may be used.² In this context, the angle of repose serves as a parameter to describe the flow behaviour

This is an open access article under the terms of the [Creative Commons Attribution](https://creativecommons.org/licenses/by/4.0/) License, which permits use, distribution and reproduction in any medium, provided the original work is properly cited.

© 2022 The Authors. *Packaging Technology and Science* published by John Wiley & Sons Ltd.

of a bulk material. The user must ensure that only those filling substances are transported in the approved packaging whose angle of repose is higher than the angle of repose specified in the approval according to the design type test report. The higher the angle of repose of a bulk material, the worse its flow properties.

The flow properties of bulk solids affect both mechanical safety of the packaging and safety against the release of substances under normal conditions of carriage.

With regard to the mechanical safety in the event of an impact, Luxenhofer³ examined the behaviour of enclosed bulk materials in the event of an impact, using bags as an example. Flow phenomena are responsible for the tearing of the bag upon the impact. The poorer the flowability of the bulk solid, the lower its damaging effect on the packaging wall.³ Depending on the bulk material, part of the vertical load is deflected horizontally in the event of an impact. The resulting horizontal flow movement stresses the packaging wall when decelerating the particle mass, which can lead to failure.⁴ Good flowability, expressed by a low angle of repose or a low Hausner ratio, leads to a greater damage effect and thus to a reduction in safety.⁵

But the flow behaviour of a powdery or granular substance is also of fundamental importance for the release of particles in form of bulk solids. For sift-proofness under normal transport conditions, for example, under the influence of vibrations, there are various influencing factors. On the one hand, the weak points of the packaging play a role, but on the other hand, also properties of the powdery or granular substance, such as the flow properties. In the case of a powder with good flowability, powder can emerge from insufficiently pasted joints already during manual filling.^{2,6}

Reports from practice of dangerous goods controls show that dangerous goods are repeatedly released during transport from packagings that are actually intact.^{2,7,8} Figure 1 shows the release of a flammable solid of UN No. 1328 from the closure of a 5M2 paper bag, the substance labelled as 'free-flowing' on the packaging. In Figure 2, a corrosive solid of UN No. 3262 escapes from the stitched seam of a 5M2 paper bag.

The areas of the packaging affected here, the internal sleeve valve and the stitched joint, are typical weak points for particle release.² However, good flowability also favours the release of a bulk material. As with the first aspect, mechanical safety, it also applies here that good flowability, that is, a low angle of repose or a low Hausner ratio, reduces the level of safety.

The specification of the flow properties is therefore important in practice for the safety of the transport of dangerous goods, both with regard to mechanical safety and sift-proofness.

The problem is that different methods for the measurement of the angle of repose are currently used in the recognized test centres for dangerous goods packagings in Germany, as no specific method is prescribed for measuring this parameter.² It is unclear how reproducible the results of the various methods are and for which types of powdery or granular substances the individual techniques are in principle suitable. The certificate of approval also does not state which



FIGURE 1 Release of a flammable solid (UN No. 1328) from the closure of a 5M2 paper bag (Police North Rhine-Westphalia Münster)



FIGURE 2 Release of a corrosive solid (UN No. 3262) from the seam of a 5M2 paper bag (Police North Rhine-Westphalia Münster)

measuring method was used for the determination of the angle of repose.

Therefore, the aim of the present work is to systematically examine the methods as they are currently applied in the test centres for measuring the angle of repose. This topic has not yet been investigated against the background of the test practice in the test labs for dangerous goods packagings. The materials chosen are 12 non-hazardous powdery or granular substances with different particle sizes and particle shapes. They were conditioned in a controlled atmosphere of $23^{\circ}\text{C} \pm 2^{\circ}\text{C}$ and $50\% \pm 2\%$ relative humidity (UN 6.1.5.2.3¹) before starting the tests.

In addition, an alternative approach for characterizing the flow properties of bulk solids is investigated, the determination of the Hausner ratio. This dimensionless parameter represents the quotient of the tapped density to aerated bulk density. The individual methods are assessed with regard to their repeatability, their applicability to certain types of solid filling substances and their suitability for handling hazardous substances.

It is essential to know the limitations of existing methods.⁹ This is relevant for the test labs for dangerous goods packagings and for manufacturers of bulk solids and the users of the packagings. The

results are used to identify opportunities to improve testing practice for the determination of flow properties of filling substances of dangerous goods packagings. This serves to ensure safety when transporting dangerous goods.

2 | THEORETICAL BACKGROUND—CHARACTERIZATION OF FLOW PROPERTIES OF BULK SOLIDS

The flow properties of a powdery or granular product are of decisive importance for the filling process of a packaging, the behaviour during transport and handling and for emptying the packaging. In the case of dangerous goods packagings, the flow properties of the filling goods are also important for safety reasons, as they have an influence on the sift-proofness of the packaging during transport and on the mechanical behaviour in the event of an impact.

Flowability is the property of bulk materials characterized by the freedom of the constituent particles, or groups of particles, individually to move when the bulk material is put in motion by some force, gravity or otherwise.¹⁰ The flow of a powder is a complex phenomenon that is sensitive to properties of the powder itself and external factors such as humidity. Within the powder, many parameters affect flow properties.¹¹ The main influencing factors are particle size and particle shape and shape and width of particle size distribution.^{12–15} Further named influencing factors are surface roughness, material density, electrostatic characteristics,¹¹ chemical composition and moisture content.¹⁴

Generally, the flowability of a powder increases with increasing particle size.^{15,16} For large particles, the forces of gravity and inertia are large compared to the interparticle forces. Consequently, larger particles have an individual mobility that tends to result in ‘free flow’ characteristics. For very small particles, the interparticle force particles are large compared to the inertial and gravitational forces, resulting in poor particle mobility and a cohesive flow behaviour.¹⁷ Flowability of bulk solids having the same median x_{50} increases with decreasing width of the particle size distribution.¹⁵

There are various parameters for describing the flow properties of a bulk material and a number of measurement techniques. Marchetti and Hulme-Smith¹¹ give a classification of flowability testing methods. There is a need in industry for a quick and reproducible method of measuring the flowability of bulk solids.^{18,19}

To characterize the flow behaviour of solid filling substances, the recognized test centres for dangerous goods packagings in Germany are currently measuring the angle of repose. This parameter is stated in the test report and in the approval certificate. However, there are no agreements about which measuring method must be selected. The angle of repose is considered to be a simple approach to approximate the flowability of a powdery or granular material.²⁰

Another commonly used simple method for characterizing the flow behaviour of solid substances is the determination of the Hausner ratio.^{11,18,20,21} Therefore, this parameter is also considered in this work as a possible alternative to the angle of repose.

In the pharmaceutical industry, too, the determination of the angle of repose and the Hausner ratio are among the most frequently cited methods for characterizing the flow properties of powders and bulk solids.¹⁹

In addition to determining the flow properties, which serve to characterize the bulk material, there is another area of application:

During the industrial packaging process, bulk material devices, silos and feed hoppers are required for filling a package with a powder or bulk material. In this case, the flow properties are important for the design of such apparatus in order to avoid flow problems as flow disturbances or demixing. Schulze²² names suitable processes for such large-scale industrial applications. If the focus is on such technical apparatus designs, parameters other than the angle of repose and the Hausner ratio are also used.

In the context mentioned in this work, these two parameters are used to characterize the flow properties of the bulk solid, as they are also applied in test laboratories for comparative measurements of the flowability of different products.

2.1 | Angle of repose

2.1.1 | Definition

The angle of repose is defined as the angle of inclination of the free surface to the horizontal of a bulk solid heap.²⁰ Assuming a conical heap, the angle of repose α is the angle between the conical slope of a pile of solids solid materials and the horizontal base.^{19,23–25} It is related to the interparticle friction and the resistance during the movement of the particles against each other.¹⁹ The angle of repose is a criterion for the flowability of a powder and a fundamental parameter in bulk material storage. Generally, the angle of repose is an easy parameter to measure and gives a rough estimation of the cohesive behaviour of powders.²⁰

The steeper the angle of repose, the higher the cohesiveness of a bulk solid; the lower the angle of repose, the more fluid-like and free-flowing the bulk solid.^{23,25} When characterizing the flowability based on the angle of repose, there are different classifications in the form of reference tables.

TABLE 1 Classification of flow behaviour of solids based on the angle of repose according to Carr²⁵

Angle of repose (°)	Denomination
25–30	Excellent
31–35	Good
36–40	Fair
41–45	Passable
46–55	Poor
56–65	Very poor
>66	Very, very poor

According to Carr,²⁵ the angle of repose is a direct indication of the potential flowability of dry solids, as shown in Table 1. This classification is also used in the pharmaceutical literature.¹⁹ Although a simple and common technique, the angle of repose displays a reasonable correlation with flowability rating.²³

There are also other classifications of flow behaviour based on the angle of repose, which differ in some details from Carr's scheme.^{18,24,26} There is also the general criterion that cohesive powders generally have angles of repose above 40°, free flowing powders below that.^{16,18,27,28} Due to the fine differentiation in the evaluation of the angles and their widespread use, the original classification of Carr (Table 1) is used as reference in this work.

2.1.2 | Influencing factors

There are different statements regarding the influence of the **particle size** on the angle of repose. Fine powders, which are generally cohesive, have angles of repose greater than 40°²⁸; for granular materials (larger than 100 µm), the angle of repose does not appear to change in any systematic way with particle size. The angle of repose gradually decreases as the mean size increases, indicating that the powders change from cohesive to more free flowing with increasing mean size.^{18,29} The angle of repose decreases with increasing grain size for diameters lower than 50 µm³⁰; afterward, they are constant. As a contrast, however, there are also reports that there is a critical particle size at which either an increase or decrease in particle size results in an increase in the angle of repose.^{20,31}

Kalman²⁰ gives an overview of the literature on the dependence of the angle of repose on the **moisture content**. For pulverized coal, the increase in moisture content will decrease the flowability.³¹ Brown and Richard²⁷ conclude that generally cohesive powders, such as those containing particles smaller than 100 µm in size, or those with high moisture content, generally have high angles of repose.

In this work, the influence of a different moisture content on the flow behaviour is not examined since the preconditioning is carried out in a constant standard climate.

2.1.3 | Systematics of measuring methods

The angle of repose is not an intrinsic property of the powder but depends strongly on the method and equipment used for forming the heap and measuring.^{11,19,20,27,30} Therefore, the published values of the angle of repose are seldom comparable.^{18,20}

There are two experimental parameters that distinguish the methods: the distance between the funnel and the base (fixed height of funnel or raising the funnel as the heap grows, which leads to a variable funnel height) and the geometry of the base (fixed base size or geometrically large base area, enabling the formation of a variable cone diameter of material).¹⁹ In the case of a fixed base area without lateral boundary, for example in form of a circular platform, excess material is allowed to fall from the base plate.^{20,29}

Kalman²⁰ points out that different names are often used in the literature for the same method or that the same name is given to different methods. This leads to inconsistency and ambiguity.

The four most common methods used are as follows^{27,28}:

- The fixed height cone method ('free base piling'^{20,31}): Fixed height of funnel, variable base size
- the fixed base cone method: fixed base size, variable funnel height
- the tilting table method (sliding angle of repose^{20,31}) and
- the rotating cylinder method²⁰

In contrast to the second measuring principle listed, the test method 'fixed base piling'^{20,31} consists of a fixed base size and a fixed height of funnel.

The angle measured using variants 1–3 is also referred to as the static angle of repose, the angle measured using variant 4 as the dynamic angle of repose.^{18,20,27} The measurements of variants 1 and 2 are carried out on conical heaps, and the measurements of variants 3 and 4 relate to a plane surface.²⁷ The angle of repose can be directly measured, or it can be calculated by measuring the heap height and heap diameter, which assumes a perfectly conical heap and which results in an average angle.^{20,32} In addition to the perfect conical shape of the powder pile with straight slopes, piles with concave or convex shapes can also arise.³²

Although measuring the angle of repose is a simple and quick method, there is no general agreement about the best design and size of the experimental set-up, nor about the optimum amount of sample that should be used^{18,28}; the measurement method and tools must be standardized.²⁰

Within the scope of this work, only those procedures are considered in more detail, which are currently used in the recognized test centres for dangerous goods packagings in Germany.

2.2 | Hausner ratio

2.2.1 | Definition

For the determination of the Hausner ratio, the tapped bulk density ρ_t and aerated bulk density ρ_a of a powdery or granular substance are measured. The Hausner ratio is defined as follows^{16,18,19,21}:

$$H = \frac{\rho_t}{\rho_a} \quad (1)$$

Different terms are used in the literature for these two densities. Synonyms for the tapped bulk density are also tap density^{23,29} or apparent density after tamping³³; expressions for the aerated bulk density are also apparent density,^{11,34} poured density,¹¹ free bulk density²⁰ or loose bulk density.³⁵

In this work, the terms tapped density and aerated bulk density are used. Measuring these two densities is common practice to characterize the behaviour of powdered and granular materials during

storage, processing and transport.⁹ The aerated bulk density is useful for predicting the maximum volume an aerated material will occupy in a container; the tapped bulk density gives a measure of the volume occupied by a bulk material that has been vibrated.⁹

The Hausner ratio is an indirect measure of the property of a bulk material to reduce its volume under mechanical influence. It is also a measure of the ability to compress and of the interaction between the particles. In a free-flowing powder, these interactions are less important, and the values for bulk density and tapped density are closer together. A low Hausner ratio means that the material flows more easily. In the case of less flowable substances, there are significantly higher interactions between the particles, and the Hausner ratio is higher. In particular, the cohesive forces are higher.^{19,20} The classification of flow behaviour of dry solids according to Carr²⁵ that is generally accepted is shown in Table 2.^{6,19–21,29,36}

The use of such reference tables, even though the ranges defined are to a certain degree arbitrary and the descriptions are necessarily qualitative, can certainly be useful for a first rough classification.²⁹ The Hausner ratio is an easy, quick and largely accurate technique widely used in industry for predicting flow behaviour in different technical areas.^{12,16,19,23}

2.2.2 | Influencing factors

With almost all powders, the Hausner ratio decreases as **particle size** increases because this is accompanied by a decrease in cohesiveness.^{16,18} Below a Hausner ratio of 1.25, interparticle forces become less significant with the increase of particle size; above the range of 1.25 ... 1.4, cohesivity increases rapidly with the decrease of particle size.¹⁶ A critical mean size above which the increase in the mean size would see no significant effect on cohesiveness cannot be categorically be determined.¹⁶ In contrast, Lumay et al.³⁰ found that the Hausner ratio is almost constant for particles larger than about 50 μm .

Hausner ratio is virtually independent of the **relative humidity** of the air surrounding the particles even though both the aerated and the tapped bulk density vary with the relative humidity.^{9,16,37}

In terms of **particle shape**, non-spherical particles tend to result in poorer flowability (higher Hausner ratio values) than spherical particles.^{16,20}

TABLE 2 Classification of flow behaviour of solids based on the Hausner ratio according to Carr²⁵

H [–]	Flow behaviour
1.00–1.11	Excellent
1.12–1.18	Good
1.19–1.25	Fair
1.26–1.34	Passable
1.35–1.45	Poor
1.46–1.59	Very poor
>1.60	Very, very poor

2.2.3 | Systematics of measuring methods

The Hausner ratio is not an intrinsic material property but depends on the measurement method used.^{12,19} Important parameters that influence the determination of the aerated bulk density and the tapped density are, for example, the weight of the sample, the diameter of the tamping cylinder, the number of impacts and any rotation of the sample during the tamping.¹⁹

There are basically three options for determining the aerated bulk density: measuring the volume of a known mass of the powder sample in a measuring cylinder (method 1) or determining the mass of a known volume of powder that has passed a volumeter (method 2) or that has been filled into a weighing vessel with a known volume (method 3). The tapped density is obtained by mechanically compressing a sample in a graduated measuring cylinder or a weighing vessel with a defined volume.¹⁹ An overview of apparatus and methods of measuring these densities can be found in literature.^{9,16,19,25}

The methods used in this research are detailed in Section 4.

3 | MATERIALS

Twelve bulk solids were examined for their angle of repose and their Hausner ratio. Chrome ore sand (>46% Cr_2O_3 , >25% FeO , >15% Al_2O_3) was obtained from Mineralwerk Hamm GmbH, Hamm, Germany. All other substances were obtained from KSL staubtechnik GmbH, Lauingen, Germany. Esplas H130 is a polymethylmethacrylate (PMMA). The Eskal powders (Eskal 10, Eskal 15, Eskal 30, Eskal 80, Eskal 150) and Eskal grained types (Eskal 0.5–1.0, Eskal 2.8–4.0) consist of natural calcium carbonate CaCO_3 (>99.10%). Escor 1.8–3.4 is a corundum (>95% Al_2O_3). The composition of the two Arizona dusts ISO 12103-1 (A2 fine and A4 coarse) is mineral silica/quartz (>97% silicon dioxide) with other oxides.

Table 3 lists parameters for the particle size characterization of the 12 substances obtained from measuring the **particle size distribution**. The index '3' in these characteristics indicates a volume-related quantity type. From the measured particle size distribution (cumulative distribution and density distribution), different parameters can be determined for the characterization of particle collectives. The median $x_{50,3}$ means that 50% of the total amount of the particle collective is below this value, determined from the cumulative distribution curve. The mode $x_{\text{mod},3}$ is the particle size at which the density distribution has a maximum.^{6,38} The measurements were also used to calculate the relative span as defined in Baesso et al.²⁹:

$$\text{span} = \frac{x_{90,3} - x_{10,3}}{x_{50,3}} \quad (2)$$

Two techniques were used for measuring the particle size and particle size distribution within this work: For Eskal 10, Eskal 15, Eskal 30, Eskal 80, Eskal 150, Esplas H130 and the two Arizona dusts, laser diffraction analysis was applied. This method is based on the

TABLE 3 Parameters for the characterization of the particle size of the 12 bulk solids

Substance name	CAS No.	$x_{mod,3}$ (μm) (mode)	$x_{min,3}$ (μm)	$x_{10,3}$ (μm)	$x_{50,3}$ (μm) (median)	$x_{90,3}$ (μm)	$x_{max,3}$ (μm)	Span (–)
Chrome ore sand	1308-31-2	330.0	74.0	158.7	274.0	442.0	1030.0	1.03
Esplas H130	9011-87-4	196.7	0.9	35.9	137.1	290.3	541.5	1.86
Eskal 10 ^a	1317-65-3	11.2	0.2	4.6	11.5	20.2	75.3	1.35
Eskal 15	1317-65-3	18.9	0.2	10.4	17.7	25.9	83.0	0.87
Eskal 30	1317-65-3	32.1	0.3	20.7	30.6	43.8	74.7	0.76
Eskal 80	1317-65-3	97.2	0.1	53.9	89.7	125.4	205.3	0.79
Eskal 150	1317-65-3	136.9	0.5	86.8	128.8	168.2	212.3	0.63
Eskal 0.5–1.0	1317-65-3	942.3	123.3	580.7	785.0	1015.7	1122.3	0.55
Eskal 2.8–4.0	1317-65-3	3626.0	110.0	2741.5	3393.5	3998.0	5704.0	0.37
Escor 1.8–3.4	1344-28-1	3031.0	1096.5	2259.5	2794.0	3247.0	3697.5	0.35
Arizona dust quartz A2 fine	14808-60-7	3.3	0.3	1.6	6.6	43.6	135.0	6.34
Arizona dust quartz A4 coarse	14808-60-7	64.7	0.4	2.8	26.2	97.7	240.7	3.62

^aEskal 10 contains a flow agent, 0.25% calcium stearate (by weight).

utilization of diffraction effects, which are caused by the interaction between the laser light and the particles to be examined. The HELOS/BR system was used in combination with the wet dispersing unit SUCELL (Sympatec GmbH, Clausthal-Zellerfeld, Germany). For the optical setting, the components R1, R3 and R5 (0.1–875 μm) were combined, and the dispersion was done with 2-propanol.

For chrome ore sand, Eskal 0.5–1.0, Eskal 2.8–4.0 and Escor 1.8–3.4, a dynamic image analysis was carried out using the CAMSIZER P4 system (Microtrac Retsch GmbH, Haan, Germany). The measuring range was 30 μm –30 mm, and a dry dispersion was performed.

Table 3 shows that for the various Eskal fractions, the values for the median increase continuously from 11.5 μm (Eskal 10) to 3393.5 μm (Eskal 2.8–4.0). There is also the tendency that from Eskal 10 to Eskal 2.8–4.0, the values for the relative span decrease from 1.35 to 0.37. The examined substance with the smallest median is Arizona dust A2 fine (6.6 μm), and the substance with the largest median is Eskal 2.8–4.0.

A **particle shape analysis** was carried out by dynamic image analysis. For Eskal 10, Eskal 15, Eskal 30, Eskal 80, Eskal 150, Esplas H130 and the two Arizona dusts, the QICPIC system combined with the disperser OASIS/L was applied (Sympatec GmbH, Clausthal-Zellerfeld, Germany). The measuring range M6 (5–1705 μm) was used, and the dispersion was done with 2-propanol. For chrome ore sand, Eskal 0.5–1.0, Eskal 2.8–4.0 and Escor 1.8–3.4, the dynamic image analysis by CAMSIZER P4 (measuring range 30 μm –30 mm) was used for the particle shape analysis. Both systems (QICPIC and CAMSIZER P4) are working according to ISO 13322-2.³⁹

From the recordings, the particle circularity ψ_c ^{13,40,41} (sphericity⁴²) was established for each substance. It represents the degree to which the particle is similar to a circle, taking into consideration the smoothness of the perimeter. This means circularity is a measurement of both the particle form and roughness. Thus, the further away from a perfectly round, smooth circle a particle becomes, the lower the

TABLE 4 Particle shape factors circularity ψ_c and aspect ratio ψ_a , derived from particle shape analysis

Substance name	ψ_c (–)	ψ_a (–)
Chrome ore sand	0.89	0.91
Esplas H130	0.66	0.57
Eskal 10	0.88	0.61
Eskal 15	0.85	0.70
Eskal 30	0.85	0.79
Eskal 80	0.87	0.87
Eskal 150	0.66	0.59
Eskal 0.5–1.0	0.84	0.88
Eskal 2.8–4.0	0.87	0.9
Escor 1.8–3.4	0.87	0.89
Arizona dust quartz A2 fine	0.89	0.66
Arizona dust quartz A4 coarse	0.78	0.66

circularity value.¹³ This parameter is calculated by the ratio of the perimeter of the equivalent circle to the real perimeter. Its value is between 0 and 1 ($\psi_c = 1$ for a circle). The smaller the value, the more irregular the shape of the particle, since an irregular shape causes an increase of the perimeter.⁴² The perimeter of all non-circular areas is larger than that of a circle of the same area.⁴⁰

In addition, the aspect ratio ψ_a was determined by particle shape analysis.⁴¹ It gives an indication for the elongation of the particle⁴² (rather round or rod-shaped). Table 4 displays the results for the circularity ψ_c and the aspect ratio ψ_a , obtained for the mode $x_{mod,3}$, since this then corresponds to the largest proportion of the particles recorded.

Deviations for one and the same material in relation to these two values can be caused by differences in the processing of the material. The precondition for comparable values is the identical material

processing (duration and energy and method for grinding the starting material).

It is not easy to make a comparison between samples of different materials. However, it is noticeable that the values for circularity ψ_c and aspect ratio ψ_a for Eskal 2.8–4.0 and Escor 1.8–3.4 are very close together.

4 | METHODS

4.1 | Preconditioning of the powdery and granular substances

Before the tests are carried out, the bulk solids are conditioned for at least 120 h in an atmosphere having a controlled temperature of $23^\circ\text{C} \pm 2^\circ\text{C}$ and a relative humidity of $50\% \pm 2\%$ according to UN 6.1.5.2.3.¹ This standard climate is chosen because most test centres for dangerous goods packagings, especially those for packagings made of paper and fibreboard, can implement this standard climate. According to UN 6.1.5.2.3, at least 48 h are prescribed for preconditioning.

The moisture content after preconditioning is determined for each material according to DIN EN ISO 17892-1⁴³ on different portions of the laboratory sample. The resulting mean moisture content for all substances was less than 0.15% by weight (dry basis).

After preconditioning, the sample quantity required to carry out a single test is taken and subjected to the measurements (4.2, 4.3.1) within 1 min under a fume cupboard. For measuring the tapped bulk density (4.3.2), the required sample quantity is dried in an oven at $105^\circ\text{C} \pm 2^\circ\text{C}$ as prescribed in DIN EN ISO 787-11³³ for at least 2 h prior to testing.

4.2 | Measurement methods for the angle of repose

4.2.1 | Method according to ISO 4324

The experimental set-up for this method is shown in Figures 3 and 4.^{44,45}

The funnel outlet is initially closed; 150 mL of the powdery or granular sample are loaded in the glass funnel, and the outlet is opened. The bulk material is released through the funnel outlet (outlet diameter: 10 mm) to flow and form a conical pile of material on the circular base plate made of transparent plastic, which has a fixed diameter of 100 mm. The use of the agitator is optional. The height distance between the base plate and the funnel outlet is 75 mm. Excess powder will fall laterally from the base plate. The height of the powder cone is measured using a slewable measuring bar.^{44,45} The angle of repose is calculated from this measured height h of the pile and the fixed diameter of the circular base plate^{32,44,45}:

$$\tan \alpha = \frac{2h}{100} = \frac{h}{50} \quad (3)$$



FIGURE 3 Method according to ISO 4324: Test set-up—Arizona dust A4 coarse

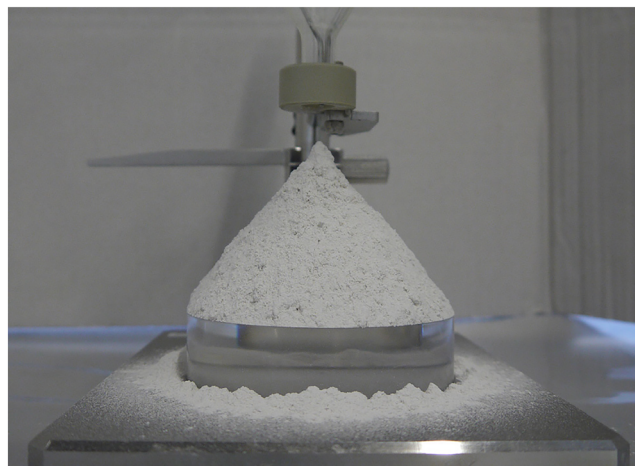


FIGURE 4 Method according to ISO 4324: Test set-up (detail)—Arizona dust A4 coarse

A minimum of five tests are carried out for each material. Stirring with the agitator was used for the following substances to facilitate their flow out of the funnel: Eskal 10, Eskal 15, Eskal 30, Escor 1.8–3.4, Arizona dust quartz A2 fine, Arizona dust quartz A4 coarse. With regard to the systematics of the test procedures given in Section 2.1.3, the

method according to ISO 4324 can be assigned to the ‘fixed base piling’ measuring principle.

4.2.2 | ‘Funnel test’—Test method used in the test labs for dangerous goods packagings

The experimental set-up is shown in Figure 5. This equipment and procedure correspond to the method currently applied in the test centres for dangerous goods packagings.

The measuring equipment consists of a stainless steel stamp whose circular platform has a diameter of 50 mm. The outlet diameter of the plastic funnel is 30 mm. The funnel is placed with its outlet opening on the test stamp and is filled with the bulk solids sample; 100 mL of the substance to be tested are placed in the funnel. The funnel is fixed with both hands in the middle of the test stamp and slowly pulled upwards until the bulk material runs out and forms a conical pile on the platform. Excess particles are allowed to drain away past the circumference of the platform. The angle of repose of the heap is measured by applying an inclinometer (Angle Star DP-45 Digital Protractor, MEAS Deutschland GmbH, Dortmund, Germany). The inclinometer is held parallel to the slope of the heap, and the measured value for the angle of repose is read. Two values are determined for each pile (right and left slope). At least five experiments are conducted on each substance.

This procedure can be assigned to the fixed base cone method from Section 2.1.3.

4.2.3 | ‘Plate method’—Test method used in the test labs for dangerous goods packagings

This method is used in two variants. The geometry of the test set-up is the same, but the material of the plates is different. The experimental set-up with plexiglass plates is the original technique used in the test centres for dangerous goods packagings in Germany. In order to investigate the influence of the plate material, plates were also manufactured of mineral glass. Figure 6 displays the experimental set-up with plexiglass plates, Figure 7 with mineral glass plates.



FIGURE 5 ‘Funnel test’: Test set-up—chrome ore sand

The inside dimensions of the container are 940 mm length, 220 mm height and 30 mm distance between the plates. The plastic funnel has an outlet diameter of 20 mm, and the height of the funnel outlet above the rectangular base area of the test set up is 220 mm. The funnel outlet is initially closed; 1.5 L of the bulk material sample are loaded in the plastic funnel, and the outlet is opened. The bulk material is released through the funnel outlet to flow and form a pile between the plates. The angle of repose of the heap is measured by applying an inclinometer (Leica Disto D3a, Leica Geosystems AG, Heerbrugg, Switzerland). The inclinometer is held parallel to the slope of the heap, and the measured value for the angle of repose is read. Two values are determined for each pile (right and left slope). At least five heaps are formed for each substance.

The plate method cannot be clearly assigned to one of the basic measuring methods mentioned under Section 2.1.3. It is a variant similar to the ‘fixed base piling’ (fixed base size and fixed height of funnel), but the base area here is rectangular and not circular. In addition, the long side surfaces of the container limit the expansion of the bulk material in this direction, so that the resulting bulk material heap is not shaped like a cone but that of a wedge.



FIGURE 6 ‘Plate method’ (plexiglass): Test set-up—Eskal 0.5–1.0

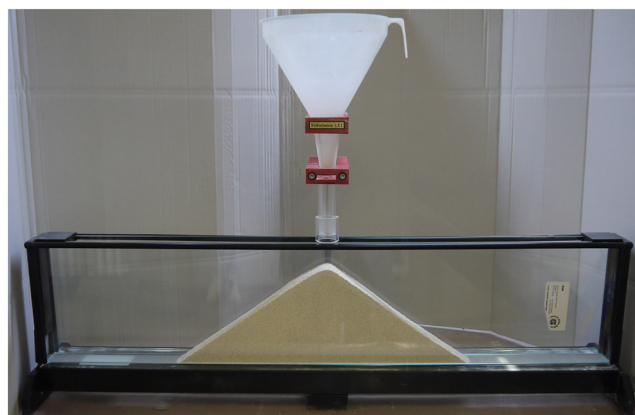


FIGURE 7 ‘Plate method’ (mineral glass): Test set-up—Eskal 0.5–1.0

ratio (Table 4), that is the most uniform grain shape, resulting in a good flowability. In this case, the influence of the friction of a different plate material is likely to have a greater effect on the angle of repose than with materials having high interparticle forces.

The results of the ANOVA just indicate that there is a significant difference between the measuring methods but does not explain the reasons for this. Therefore, in the following, the results and observations for the measurements are discussed in more detail for each substance.

The angles of repose of **chrome ore sand** (Figure 8) are slightly higher than 31° for all four methods and can therefore be assigned to the area of good flow behaviour according to Carr's classification (Table 1). When flowing, chrome ore sand forms heaps with a uniform, symmetrical, conical heap shape (ISO method and funnel test, see

Figure 5) and straight slopes in the plate apparatuses. The coefficients of variation c_v are a maximum of 3% for all methods.

For **Esplas H130** (Figure 9), the flowability is slightly worse than for chrome ore sand according to all methods. For Esplas H130, the classification according to Carr (Table 1) leads to different results for the four measurement methods: The mean values for the two plate methods with values $\bar{\alpha} \approx 34^\circ$ can also be assigned to the area of good flow behaviour according to Carr, while the mean values of the ISO method and funnel test (both slightly higher than 36°) are assigned to the 'fair' area. With this substance, too, the heap shape is basically conical for the ISO 4324 method and the funnel test and characterized by straight slopes for all methods, but the slope shows more irregularities. It can therefore not be ruled out that the formation of the cone is slightly different each time, which has a greater effect on

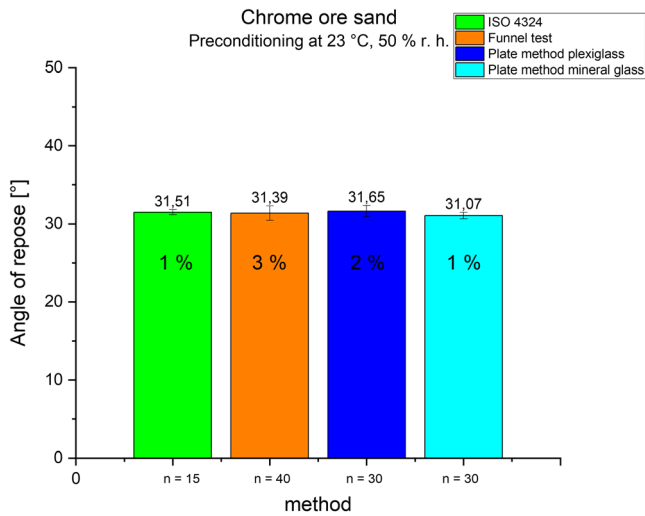


FIGURE 8 Angle of repose for chrome ore sand

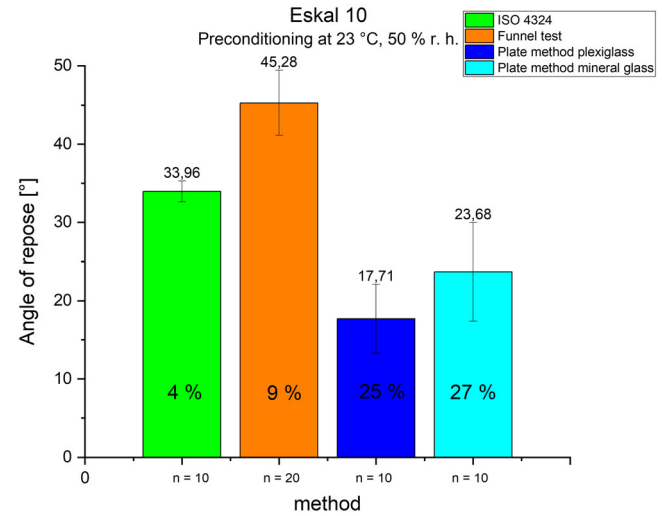


FIGURE 10 Angle of repose for Eskal 10

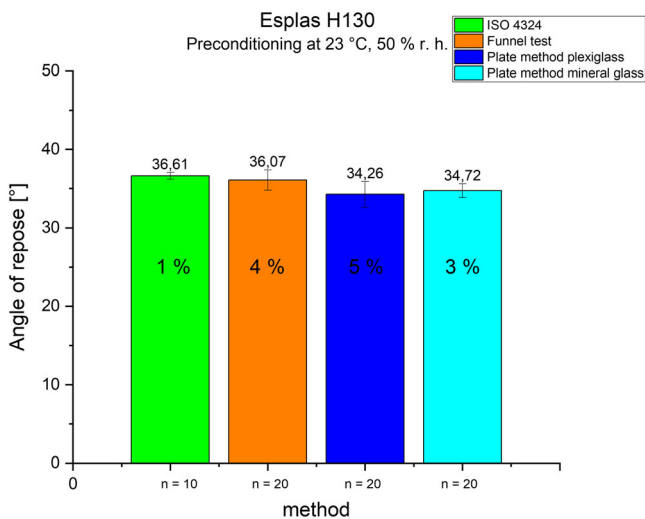


FIGURE 9 Angle of repose for Esplas H130

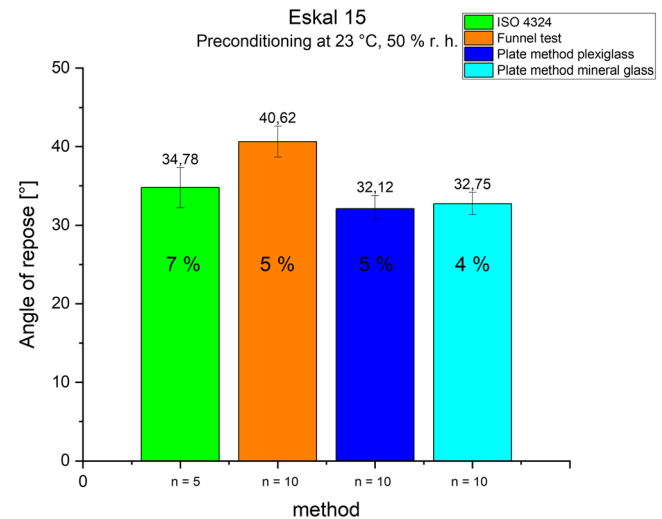


FIGURE 11 Angle of repose for Eskal 15

the scattering of the measured values, especially with the long slopes of the plate methods.

With the ISO 4324 method, even with Esplas H130, the scatter of the measured values is comparatively small ($c_v = 1\%$). In this method, the total height of the cone is measured, and the angle of repose is calculated according to Equation 1. Therefore, averaging is carried out,^{20,32} and minor local irregularities of the slope have no effect. Even if this approach is viewed critically by Lumay et al.,³⁰ because the actual heap shape is not considered, it has an advantage here. With the funnel test and the plate methods, on the other hand, it cannot be ruled out that the influence of the operator plays a role when measuring the angle of repose with the inclinometer, which would also result in poorer reproducibility.¹⁵ In the case of the funnel test, a possible operator influence is given not least by the fact that the funnel does not have a fixed height but is instead pulled upwards during outflow.

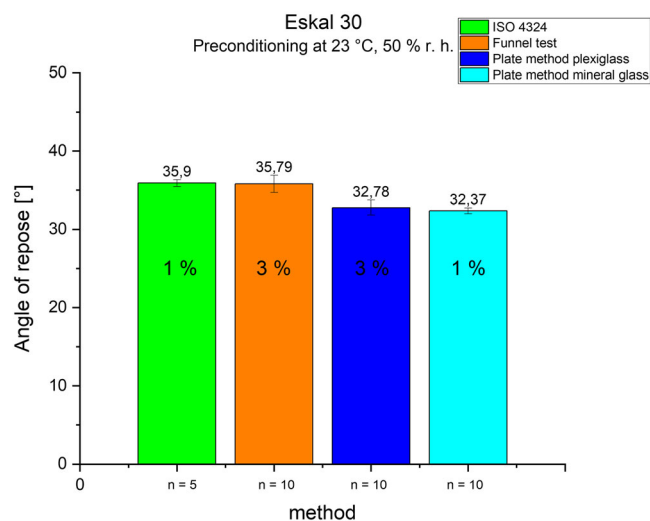


FIGURE 12 Angle of repose for Eskal 30

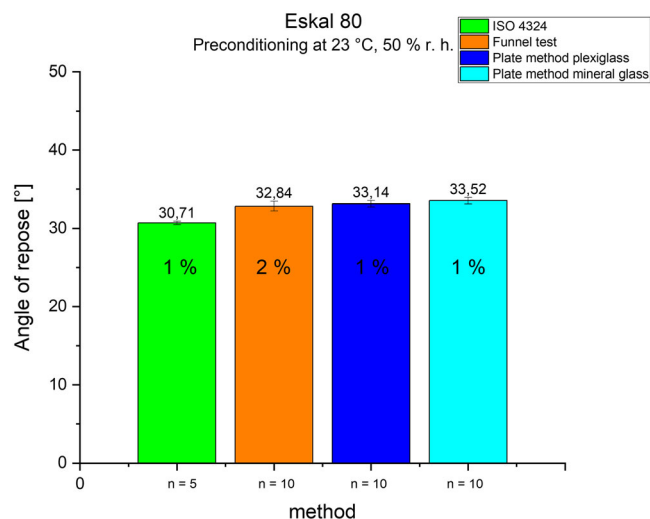


FIGURE 13 Angle of repose for Eskal 80

The results for Eskal 10, Eskal 15, Eskal 30 and Eskal 80 (Figures 10–13) are considered together. The values of mode and median (Table 3) show that these bulk solids tend to become coarser in this order.

As indicated in Table 3, Eskal 10 is a special case because the manufacturer has added a flow agent to this substance. However, the angles of repose resulting from the application of the four methods are contradictory. Figure 10 shows that for Eskal 10, the mean values determined using the four methods differ greatly from one another. The classification of flowability according to Carr's scheme (Table 1) therefore results in the following flow behaviour: ISO method ($\bar{\alpha} \approx 34^\circ$): good; funnel test ($\bar{\alpha} \approx 45^\circ$): transition between passable and poor; plate method (plexiglass) ($\bar{\alpha} \approx 18^\circ$) and plate method (mineral glass) ($\bar{\alpha} \approx 24^\circ$): formally better than excellent. Only for the ISO method, the value for c_v is comparatively low with a value of 4%; for the two plate methods, this value is even 25% or more. The heaps formed for Eskal 10 using the ISO method and the funnel test show a highly irregular, non-conical, asymmetric shape (Figure 14). Despite the addition of the flow agent, larger agglomerates of particles are formed with this substance, which contribute to the irregular shape of the heap. This heap shape is typical for a highly cohesive powder.³⁰

In both plate methods, the slope visible in the side view is also irregular, non-symmetrical and characterized by a rather concave shape (Figure 15). During the testing of Eskal 10 with all four



FIGURE 14 Funnel test—Eskal 10

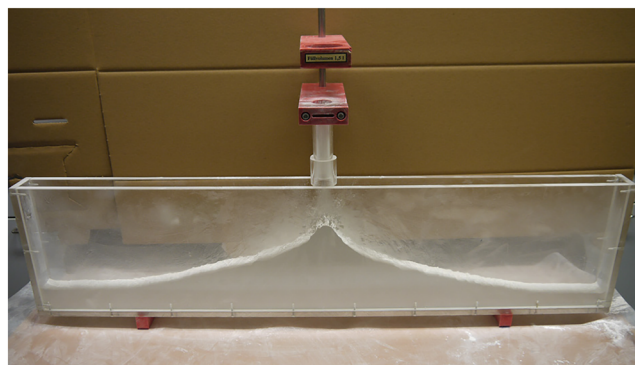


FIGURE 15 Plate method (plexiglass)—Eskal 10

methods, there was a high level of dust formation. The dust deposited visible in Figure 15 comes from only one measurement. Especially with the plate method, with its large geometric dimensions and the large amount of sample that needs to be handled, emptying the apparatus after each single measurement and cleaning the apparatus is an important practical aspect. The question arises as to how occupational health and safety can be guaranteed if dangerous original bulk solids are tested in actual laboratory operation instead of non-hazardous filling goods.

Because of the presence of agglomerates in Eskal 10, the formation of the shape of each heap seems to be to some extent arbitrary. Due to the irregular shape of the slope, it is difficult to apply the respective inclinometer to the slope in a reproducible manner when using the funnel test and the plate methods. This also contributes to poor repeatability. For this substance, too, the ISO method has the

best repeatability (smallest value for c_v), since this type of angle of repose determination results in an averaging along the slope.

With increasing particle size for the same substance (in the order **Eskal 10, Eskal 15, Eskal 30, Eskal 80**), the heap shapes show the tendency to approach a uniform, conical, symmetrical shape (ISO method and the funnel test) or a uniform, symmetrical shape with straight slopes (plate methods), respectively. In the case of the funnel test and the plate methods, this, in turn, makes it easier to place the inclinometer on the slope and read off the measured value. The mean values of the individual methods continue to converge. For Eskal 80, the values for c_v for the four techniques are a maximum of 2%.

The results for **Eskal 150, Eskal 0.5–1.0, Escor 1.8–3.4** and **Eskal 2.8–4.0** are also considered together (Figure 16–19). As visible from the values for mode and median (Table 3), these substances have an increasing grain size in this order. The mean values for the ISO

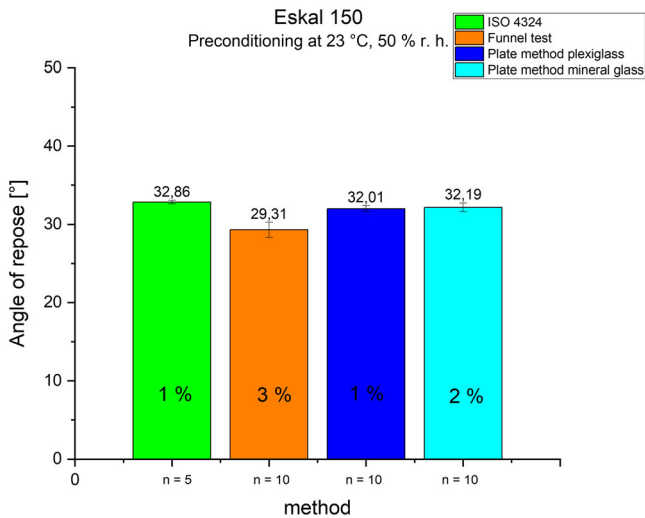


FIGURE 16 Angle of repose for Eskal 150

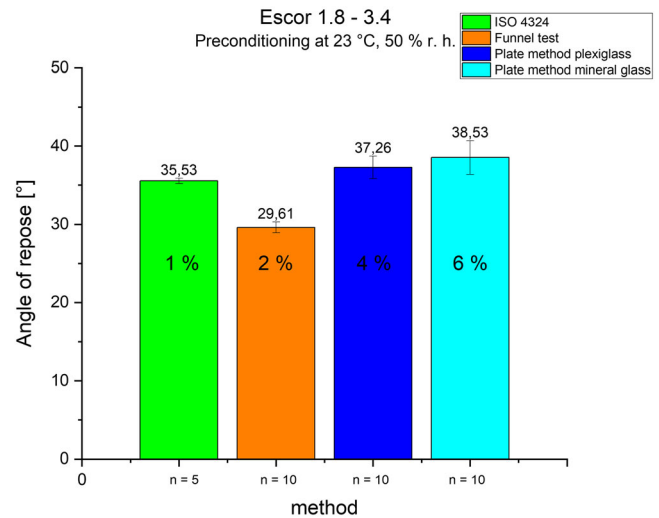


FIGURE 18 Angle of repose for Escor 1.8–3.4

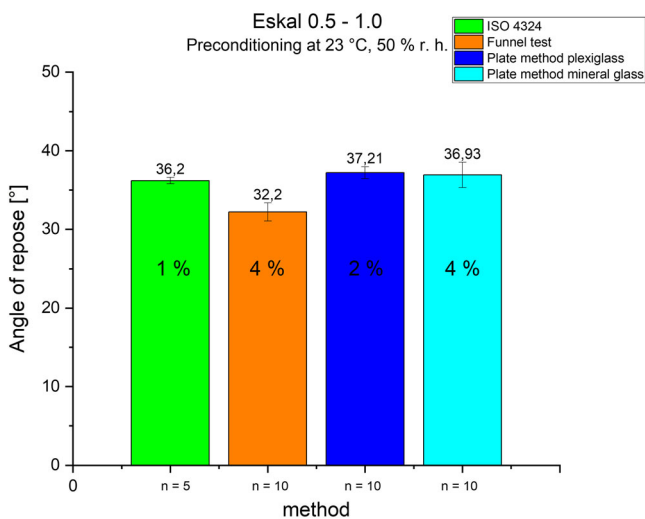


FIGURE 17 Angle of repose for Eskal 0.5–1.0

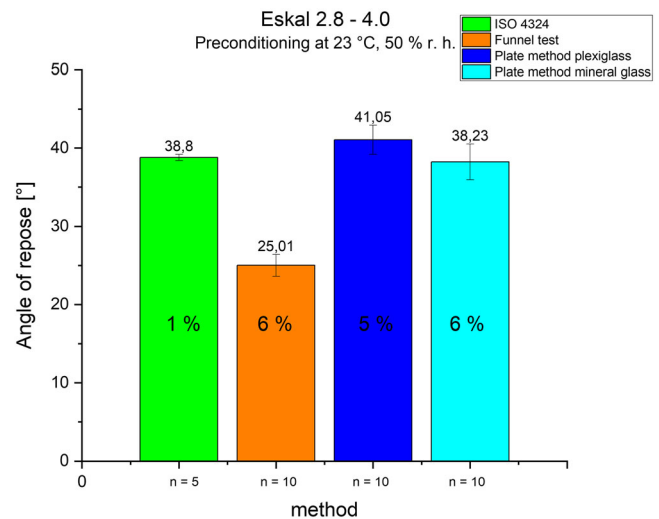


FIGURE 19 Angle of repose for Eskal 2.8–4.0

method and for the plate methods are on a similar level for all four substances. However, it is noticeable that the mean value of the funnel test, compared to the other methods, continues to fall for coarser materials. The explanation for this can be observed in Figure 20: The coarser the grains, the higher their tendency to slide off the relatively small stamp in the funnel test. The cone of bulk solids can therefore not build up properly, and the angle of repose is too flat.

In practice, this could mean that the minimum angle of repose is given as too low in the certificate of approval because it was measured with the funnel test, although the minimum angle of repose should actually be higher for the respective design type. This could lead to insufficient sift-proofness and a reduction in mechanical safety during transport if the packaging is filled with bulk materials with too high flowability.

In all methods, the heaps have a conical overall shape for all methods. However, as visible in the example of Eskal 2.8–4.0 in the plate method (mineral glass) (Figure 21), the edge lines of the slope are slightly wavy due to the coarseness of the material. This can make it more difficult to apply the inclinometer in a reproducible manner. Again, the ISO method has the best repeatability (smallest value for c_v).

Figures 22 and 23 show the results for Arizona dust quartz A2 fine and Arizona dust quartz A4 coarse. For these two substances,



FIGURE 20 Funnel test—Eskal 1.8–3.4

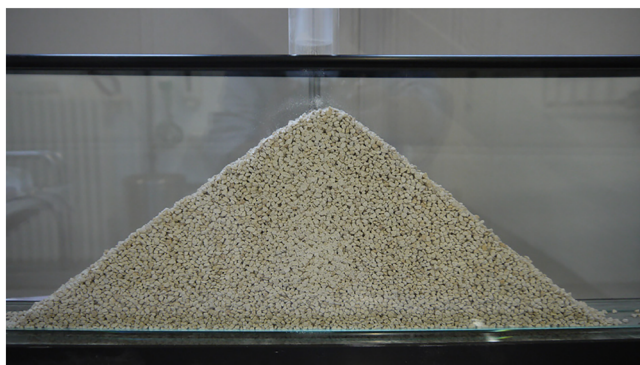


FIGURE 21 Plate method (mineral glass)—Eskal 2.8–4.0

only the ISO 4324 method was applicable. Its test equipment has an agitator, which can be used if necessary for poorly flowable materials. The shape of the resulting heap is conical with a pronounced apex (see Figures 3 and 4), which is a common phenomenon for cohesive powders.²⁷ The funnel test and the plate methods could not be carried out with these two substances because their funnels became blocked, which is an indication of the cohesiveness of these two substances.²⁸ The mean angles of repose for the two Arizona dusts, with values of about 53°, are the highest values measured of all 12 substances. According to Carr's scheme, they have poor flow properties.

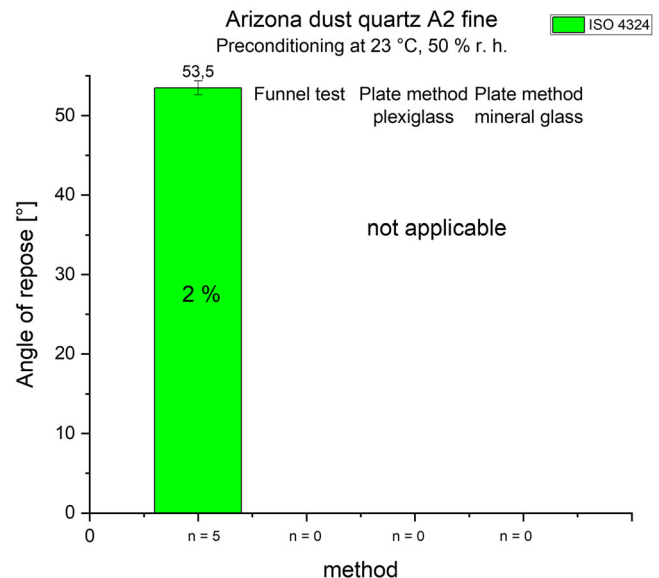


FIGURE 22 Angle of repose for Arizona dust quartz A2 fine

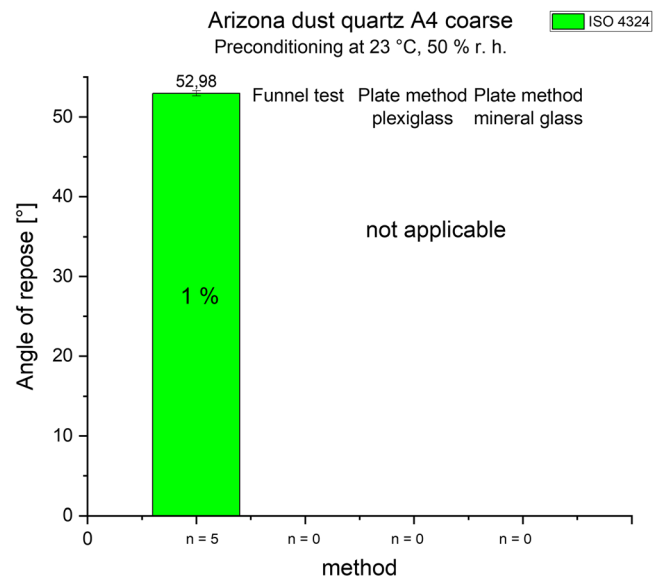


FIGURE 23 Angle of repose for Arizona dust quartz A4 coarse

Nevertheless, the variation coefficient is comparatively small with values of a maximum of 2%.

5.2 | Summary

As explained in Section 4.2, the methods for determining the angle of repose examined here and currently in use in the test centres for dangerous goods packagings are based on different principles of heap formation and measurement as well as a different geometry of the experimental set-up and a different sample size. As just shown, the measurement values of the individual methods for certain substances often differ greatly from one another or have a strong scatter. These findings confirm the generally known fact that the results of different methods are rarely comparable (see Section 2.1.3).

Overall, the results can be summarized as follows:

- **Practical applicability:** The ISO method is the only method that can be used for the two Arizona dusts that flow poorly according to Carr's scheme. The funnel test is not suitable for comparatively coarse-grained substances since the coarse granules slide off to the side from the stamp.
- **Repeatability:** Overall, the ISO method has the lowest scatter of the measured values for all substances, that is, the best repeatability. The other methods are more sensitive to operator influence.
- **Occupational health and safety:** All methods of measuring the angle of repose shown here are based on the principle that a defined quantity of powdery or granular substance is poured out so that a heap forms on an open surface or in an apparatus. In terms of occupational safety, this procedure is fundamentally to be seen as critical, since in the case of dusty and dangerous substances, appropriate precautions must be taken. It should therefore be considered to use a completely different quantity than the angle of repose to determine the flow properties of bulk solids.

6 | HAUSNER RATIO

Table 6 shows the results for the determination of the Hausner ratio (mean value \bar{H} , standard deviation σ and coefficient of variation c_v) as well as the classification of the flow behaviour of the respective substance according to Carr²⁵ (Table 2). Figure 24 presents the results.

The results of the Hausner ratio show a comparatively low scatter for the 12 substances examined (c_v max. 2.8%). The smallest determined Hausner ratios have values of 1.06 (for Eskal 2.8–4.0 and Escor 1.8–3.4), which means excellent flow behaviour according to Carr's classification (Table 2), and the highest value is 1.50 (Arizona dust quartz 4), which corresponds to very poor flow behaviour.

For the seven Eskal fractions (from Eskal 10 [median: 11.5 μm] to Eskal 2.8–4.0 [median: 3393 μm]), there is a tendency towards a general decrease in the Hausner ratio with increasing particle diameter, that is, an improvement in the flow properties. This corresponds to the knowledge in Section 2.2.2: With smaller particles, the interparticle forces are more dominant than with large particles, which impairs

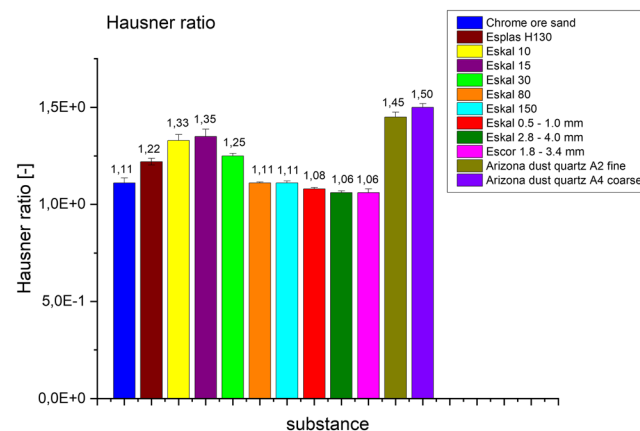


FIGURE 24 Hausner ratio for the 12 substances

TABLE 6 Results for the Hausner ratio

Substance name	\bar{H} (°)	σ (°)	c_v (%)	Classification (Table 2 ²⁵)
Chrome ore sand	1.11	0.026	2.4	excellent
Esplas H130	1.22	0.017	1.4	fair
Eskal 10	1.33	0.030	2.2	poor
Eskal 15	1.35	0.038	2.8	poor
Eskal 30	1.25	0.013	1.0	fair
Eskal 80	1.11	0.011	1.0	excellent
Eskal 150	1.11	0.006	0.5	excellent
Eskal 0.5–1.0	1.08	0.007	0.6	excellent
Eskal 2.8–4.0	1.06	0.010	1.0	excellent
Escor 1.8–3.4	1.06	0.020	1.8	excellent
Arizona dust quartz A2 fine	1.45	0.026	1.8	poor
Arizona dust quartz A4 coarse	1.50	0.020	1.3	very poor

the flowability. An exception to this is that the Hausner ratio of Eskal 10 is slightly lower than that for Eskal 15. According to theory, better flowability (i.e., a lower Hausner ratio) would have been expected with Eskal 15. The addition of the flow agent to Eskal 10 (see Table 3) can be an explanation for this.

However, the coarser the Eskal fractions, the lower the further decrease in the Hausner ratio. The resulting classification according to Carr is 'excellent' for the substances from Eskal 80 to Eskal 2.8–4.0, and there is no further improvement in the classification. This corresponds to the observations of Lumay³⁰ that the Hausner ratio remains almost constant for particle sizes larger than 50 μm .

It is also noticeable that the flowability of the finer Arizona dust quartz A2 ($\bar{H} = 1.45$) is better than that of Arizona dust quartz A4 ($\bar{H} = 1.50$). The other parameters of the particle size distribution in Table 3 also suggest that Arizona dust quartz A2 should actually be less flowable than Arizona dust quartz A4. A possible explanation for the better flow behaviour can be found in the measured values for the particle shape in Table 4. With the same value for the aspect ratio ($\psi_a = 0.66$), the value for the circularity ($\psi_c = 0.89$) is larger for Arizona A2 than for Arizona A4 ($\psi_c = 0.78$). Higher circularity tends to result in better flowability (see Section 2.2.2). The actual flow behaviour, expressed as Hausner ratio, is probably influenced by superimposing effects of particle size, particle size distribution and particle shape.

A direct comparison between the Hausner ratio of the different materials is only possible to a limited extent, since morphology of the grains (particle size, particle shape) is different in addition to the chemical composition. However, it is striking that the two coarsest substances, Eskal 2.8–4.0 and Escor 1.8–3.4, have the same measured value for the Hausner ratio. The values for their median and span (see Table 3) and their shape factors (circularity and aspect ratio, see Table 4) are comparable. This may be a reason for the coincident value of their Hausner ratios.

7 | SUMMARY

The results of determining the Hausner ratio can be summarized as follows:

- **Practical applicability:** The underlying measurements for tapped density and aerated bulk density were applicable for all 12 substances examined.
- **Repeatability:** The maximum coefficient of variation resulting from the application of this procedure to the 12 substances is 2.8%.
- **Occupational health and safety:** For the measurements of aerated bulk density and tapped density, it is also necessary to decant the respective substance. However, the respective sample quantity is taken up in a measuring cylinder with a defined volume, and a significantly smaller sample amount is spilled freely onto a surface. This leads to easier handling and cleaning of the equipment. In particular with regard to occupational health and safety, this approach can be rated as more favourable.

7.1 | Comparison of the measurement of the angle of repose and the determination of the Hausner ratio

Both the measurement of the angle of repose and the determination of the Hausner ratio are common methods for assessing the flow behaviour of powders and bulk materials. However, comparing these two techniques shows the following:

- **Practical applicability:** For the angle of repose methods, only the ISO method was applicable to all 12 substances. For the determination of the Hausner ratio, however, an application was possible for all substances examined. This also includes substances with very poor flow behaviour or with a coarse grain size.
- **Repeatability:** Especially with the poorly flowable materials Eskal 10 and Eskal 15, none of the four methods of measuring the angle of repose has an acceptable value for the coefficient of variation for both materials. It is commonly accepted that the measurements of the angle of repose are poorly reproducible with cohesive substances,²⁹ which was also observed in the measurements here. The situation is different when determining the Hausner ratio, and the maximum coefficient of variation is 2.8%. With the exception of these two substances, the coefficient of variation for the ISO method is also comparatively small at a maximum of 2%.
- **Occupational health and safety:** In the context of the test centres for dangerous goods packagings, the determination of the Hausner ratio brings advantages in terms of occupational safety compared to the angle of repose measurement methods.

8 | CONCLUSION

To ensure safety when transporting dangerous goods, it is necessary to establish a uniform procedure for determining the flow behaviour of powdery or granular filling substances. This is not possible with the common methods currently used in the test centres for dangerous goods packagings in Germany for measuring the angle of repose, as shown in exemplary measurements on 12 non-hazardous substances.

The measured values of the four methods applied differ significantly or in certain cases show poor repeatability. These findings confirm the disadvantages of angle of repose measurement methods known from the literature. For special cases of bulk solids (e.g., strongly cohesive or coarse-grained substances), some of these methods cannot be used. In the case of very dusty or dangerous filling goods, it is difficult to ensure occupational safety. It follows that safety during transport of dangerous goods cannot be guaranteed with the currently practised system of measurement of the angle of repose.

The following remedial actions are conceivable:

1. As a minimum, the test report and the approval certificate should at least state the method used to determine the respective angle

of repose. This would make it possible to reproduce the measurement experimentally.

- In order to standardize the test practice, one could agree to a prescribed method for measuring the angle of repose, which would have to be used in all test centres. Of the methods examined here, the ISO method shows the best potential for this.
- An alternative parameter for characterizing the flow properties should be considered. When defining a possible alternative to the measurement of the angle of repose, it should be taken into account that it must also be suitable for the application in the recognized test labs and in the industry, in particular an easy-to-use method.
- The determination of the Hausner ratio proposed in this work is basically suitable for this. In addition to better repeatability of the results and reliable applicability, this technique also brings advantages when handling hazardous filling goods. The classification of the Hausner ratio according to the scheme of Carr enables a direct statement to be made about the flowability of bulk solids.

The implementation of the fourth measure would have significant effects in practice. On the one hand, the testing laboratories would have to change their test procedures from measuring the angle of repose to determining the Hausner ratio, and on the other hand, all future certificates of approval of the competent authority would have to be changed to this new specification system. Instead of a minimum angle of repose, a minimum Hausner ratio would be listed on the approval certificate. This would affect all future approvals for dangerous goods packagings in Germany.

Further investigations will deal with the influence of different preconditioning (e.g., in a very dry or very humid climate) on the flow behaviour of filling goods. In addition, the measurements will also be extended to dangerous original filling goods in order to further shed light on the aspect of the necessary occupational safety.

The results are relevant for the test centres for dangerous goods packagings, the manufacturers of bulk solids and the subsequent users of the packaging. Since the flow properties of powdery or granular substances have an impact on both mechanical safety and sift-proofness, improvement and standardization of testing practice for the determination of the flow properties are recommended for the area of dangerous goods packagings.

ACKNOWLEDGEMENT

Our special thanks go to Ms. Deborah Ngoue for carrying out the measurements and to Mr. Ackermann and Mr. Senkel, Police North Rhine-Westphalia Münster, department Schwerlastgruppe of the Autobahnpolizei Münster, for the friendly provision of the photos (Figures 1 and 2). Open Access funding enabled and organized by Projekt DEAL.

TABLE OF SYMBOLS

Symbol	Description	Unit
c_v	Coefficient of variation	-
H	Hausner ratio	-
h	Height of the pile	m
x	Particle size	μm
$x_{max,3}$	Largest particle size (based on quantity type volume)	μm
$x_{min,3}$	Smallest particle size (based on quantity type volume)	μm
$x_{mod,3}$	Mode (based on quantity type volume)	μm
$x_{10,3}$	Particle size below which 10% of the particle quantity is located (based on quantity type volume)	μm
$x_{50,3}$	Median (particle size below which 50% of the particle quantity is located; based on quantity type volume)	μm
$x_{90,3}$	Particle size below which 90% of the particle quantity is located (based on quantity type volume)	μm

TABLE OF GREEK SYMBOLS

Symbol	Description	Unit
α	Angle of repose	$^\circ$
ρ_a, ρ_t	Aerated bulk density, tapped bulk density	kg/m^3
σ	Standard deviation of α	$^\circ$
Ψ_a	Aspect ratio	-
Ψ_c	Circularity	-

DATA AVAILABILITY STATEMENT

The data that support the findings of this study are available from the corresponding author upon reasonable request.

ORCID

Eva Schlick-Hasper  <https://orcid.org/0000-0003-4486-2465>

REFERENCES

- UN Model Regulations. *Recommendations on the Transport of Dangerous Goods*. Vol. I and II. Twenty-second revised. United Nations: New York and Geneva; 2021.
- Schlick-Hasper E, Bruchno M, Goedecke T. Release of powdery or granular substances from intact dangerous goods bags during road transport—Analysis of the causes and development of a test concept. *Packag Technol Sci*. 2021;34(7):435-446. doi:10.1002/pts.2575

3. Luxenhofer K. Über das Verhalten eingeschlossener, fließfähiger Füllgüter bei Stossbeanspruchung im Hinblick auf die Bemessung von Säcken. PhD-Thesis. München: Technische Hochschule München, 1970.
4. Kaltenborn S. Festigkeitsuntersuchungen an flexiblen Schüttgutbehältern bei auftretenden Stoßbelastungen. PhD-Thesis. Clausthal-Zellerfeld: Technische Universität Clausthal, 2006.
5. Lengas N, Müller K, Schlick-Hasper E, Neitsch M, Johann S, Zehn M. Development of an analysis and testing concept for the evaluation of impact targets in the mechanical safety testing of dangerous goods packagings. *Packag Technol Sci*. 2022; doi:[10.1002/pts.2656](https://doi.org/10.1002/pts.2656)
6. Schlick-Hasper E, Eiben M, Drousch B, Heinrich A, Goedecke T, Kraume M. Influence of the length of the internal sleeve valve on the release of powdery substances from the closures of valved bags for dangerous goods. *Packag Technol Sci*. 2018;31(8):532-544. doi:[10.1002/pts.2377](https://doi.org/10.1002/pts.2377)
7. Ackermann M. Verpackungsversagen in der Praxis - Polizeikontrollen mit nachhaltiger Wirkung. In: 16. Gefahrgut-Technik-Tage Verpackungen + IBC. Berlin, November 2017. Hamburg: Ecomed-Storck Verlag, 2017.
8. Schlick-Hasper E, Goedecke T, Ackermann M, Senkel U. Staubdichtheit von Gefahrgutsäcken und -FIBC - Überblick über Probleme in der derzeitigen Transportpraxis. *Tech Sicherheit*. 2020;10(3):10-15. doi:[10.37544/2191-0073-2020-03-10](https://doi.org/10.37544/2191-0073-2020-03-10)
9. Cain JH, Quesnel DJ. A comparison of density measurements for a cohesive powder. In: *Powder & Bulk Solids*. Rosemont (Chicago), IL, 1996.
10. ANSI/CEMA Standard No. 550-2020 Classification and definition of bulk materials. Naples, FL: Conveyor Equipment Manufacturers Association, 2020.
11. Marchetti L, Hulme-Smith C. Flowability of steel and tool steel powders: a comparison between testing methods. *Powder Technol*. 2021; 384:402-413. doi:[10.1016/j.powtech.2021.01.074](https://doi.org/10.1016/j.powtech.2021.01.074)
12. Kaleem MA, Alam MZ, Khan M, Jaffery SHI, Rashid B. An experimental investigation on accuracy of Hausner ratio and Carr index of powders in additive manufacturing. *Met Powder Rep*. 2000; doi:[10.1016/j.mprp.2020.06.061](https://doi.org/10.1016/j.mprp.2020.06.061)
13. Olson E. Particle shape factors and their use in image analysis - part 1: theory. *J GXP Compl*. 2011;15(3):85-96.
14. Li Q, Rudolph V, Weigl B, Earl A. Interparticle van der Waals force in powder flowability and compactibility. *Int J Pharm*. 2004;280(1-2):77-93. doi:[10.1016/j.ijpharm.2004.05.001](https://doi.org/10.1016/j.ijpharm.2004.05.001)
15. Schulze D. *Pulver und Schüttgüter - Fließeigenschaften und Handhabung. Powders and Bulk Solids - Behavior, Characterisation, Storage and Flow*. 2. Auflage. Berlin, Heidelberg: Springer; 2009 ISBN: 978-3-540-88449-1.
16. Abdullah EC, Geldart D. The use of bulk density measurements as flowability indicators. *Powder Technol*. 1999;102(2):151-165. doi:[10.1016/S0032-5910\(98\)00208-3](https://doi.org/10.1016/S0032-5910(98)00208-3)
17. Forsyth AJ, Hutton S, Rhodes MJ. Effect of cohesive interparticle force on the flow characteristics of granular material. *Powder Technol*. 2002;126(2):150-154. doi:[10.1016/S0032-5910\(02\)00046-3](https://doi.org/10.1016/S0032-5910(02)00046-3)
18. Geldart D, Abdullah EC, Hassanpour A, Nwoke LC, Wouters I. Characterisation of powder flowability using measurement of angle of repose. *China Particulol*. 2006;4(3-4):104-107. doi:[10.1016/S1672-2515\(07\)60247-4](https://doi.org/10.1016/S1672-2515(07)60247-4)
19. *Europäisches Arzneibuch Ph. Eur., Grundwerk 2017*. 9. Ausgabe. Deutscher Apotheker Verlag; 2017 ISBN: 978-3-7692-6641-2.
20. Kalman H. Quantification of mechanisms governing the angle of repose, angle of tilting, and Hausner ratio to estimate the flowability of particulate materials. *Powder Technol*. 2021;382:573-593. doi:[10.1016/j.powtec.2021.01.012](https://doi.org/10.1016/j.powtec.2021.01.012)
21. Chao PC. Easy flow—Kontinuierliche Feuchtgranulation am Beispiel von Calciumcarbonat. PhD-thesis. Bonn: Rheinische Friedrich-Wilhelms-Universität Bonn, 2013.
22. Schulze D. Zur Fließfähigkeit von Schüttgütern - Definition und Messverfahren. *Chem Ing Tech*. 1995;67(1):60-68. doi:[10.1002/cite.330670105](https://doi.org/10.1002/cite.330670105)
23. Cain J. An alternative technique für determining ANSI/CEMA standard flowability ratings for granular materials. *Powder Handl Process*. 2002;14(3):218-221.
24. ISO 3435: 1977 Continuous mechanical handling equipment—classification and symbolization of bulk materials.
25. Carr RL. Evaluating flow properties of solids. *Chem Eng*. 1965;72(3): 163-168. doi:[10.1001/archopht.1965.00970040165007](https://doi.org/10.1001/archopht.1965.00970040165007)
26. Carr RL. Particle behaviour storage and flow. *British Chem Eng*. 1970; 15(12):1541-1549.
27. Brown RL, Richards JC. *Principles of Powder Mechanics*. First ed. Oxford: Pergamon Press; 1970.
28. Geldart D, Mallet MF, Rolfe N. Assessing the flowability of powders using the angle of repose. *Powder Handl Process*. 1990;2(4):341-346.
29. Baesso I, Karl D, Spitzer A. Characterization of powder flow behavior for additive manufacturing. *Addit Manuf*. 2021;47:1-14. doi:[10.1016/j.addma.2021.102250](https://doi.org/10.1016/j.addma.2021.102250)
30. Lumay G, Boschini F, Traina K, et al. Measuring the flowing properties of powders and grains. *Powder Technol*. 2012;224:19-27. doi:[10.1016/j.powtec.2012.02.015](https://doi.org/10.1016/j.powtec.2012.02.015)
31. Wang W, Zhang J, Yang S, Zhang H, Yang H, Yue G. Experimental study on the angle of repose of pulverized coal. *Particuology*. 2010; 8(5):482-485. doi:[10.1016/j.partic.2010.07.008](https://doi.org/10.1016/j.partic.2010.07.008)
32. ASTM D6393-21 standard test method for bulk solids characterisation by Carr indices. West Conshohocken, PA: American Society for Testing and Materials, 2021.
33. DIN EN ISO 787-11: 1995-10 Allgemeine Prüfverfahren für Pigmente und Füllstoffe Teil 11: Bestimmung des Stampfvolumens und der Stampfdichte. General methods of test for pigments and extenders determination of tamped volume and apparent density after tamping. Beuth-Verlag: Berlin, 1995.
34. DIN EN ISO 60: 2000-01 Kunststoffe - Bestimmung der scheinbaren Dichte von Formmassen, die durch einen genormten Trichter abfließen können (Schüttdichte). Plastics - determination of apparent density of material that can be poured from a specified funnel. Beuth-Verlag: Berlin, 2000.
35. ASTM D7481-18 standard test methods für determining loose and tamped bulk densities of powders using a graduated cylinder. West Conshohocken, PA: American Society for Testing and Materials, 2018.
36. Akseli I, Hilden J, Katz JM, et al. Reproducibility of the measurement of bulk/tapped density of pharmaceutical powders between pharmaceutical laboratories. *J Pharm Sci*. 2019;108(3):1081-1084. doi:[10.1016/j.xphs.2018.10.009](https://doi.org/10.1016/j.xphs.2018.10.009)
37. Geldart D, Harnby N, Wong AC. Fluidization of cohesive powders. *Powder Technol*. 1984;37(1):25-37. doi:[10.1016/0032-5910\(84\)80003-0](https://doi.org/10.1016/0032-5910(84)80003-0)
38. Teipel U, Winter H, Cäsar J, Gromov A. Characterization of test dust for product qualification. *Bulk Solids Powder*. 2008;3(4):155-161.
39. ISO 13322-2: 2006-11 Particle size analysis - Image analysis methods - Part 2: Dynamic image analysis methods.
40. Stieß M. *Mechanische Verfahrenstechnik-Partikeltechnologie 1*. 3. Auflage. Berlin, Heidelberg: Springer; 2009 ISBN: 978-3-540-32551-2.
41. DIN ISO 9276-6: 2012-01 Darstellung der Ergebnisse der Partikelgrößenanalysen - Teil 6: Deskriptive und quantitative Darstellung der Form und Morphologie von Partikeln. Representation of results of particle size analysis - Part 6: Descriptive and quantitative representation of particle shape and morphology Beuth-Verlag: Berlin, 2012.
42. Sympatec GmbH. Particle shape analysis—particle shape and shape descriptors. <https://www.sympatec.com/en/particle-measurement/glossary/particle-shape/>. Last access: 2021-12-17.

43. DIN EN ISO 17982-1: 2015-03 Geotechnische Erkundung und Untersuchung - Laborversuche an Bodenproben - Teil 1: Bestimmung des Wassergehalts. Geotechnical investigation and testing - laboratory testing of soil - part 1: Determination of water content. Beuth-Verlag: Berlin, 2015.
44. ISO 4324: 1977-09 Surface active agents—powders and granules—measurement of the angle of repose.
45. DIN ISO 4324: 1983-12 Tenside - Pulver und granulate - Bestimmung des Schüttwinkels surface active agents - powders and granulates -measurement of the angle of repose. Beuth-Verlag: Berlin, 1983.
46. Hedderich J, Sachs L. *Angewandte Statistik*. 14. Auflageed. Berlin, Heidelberg: Springer; 2012. ISBN: 978-3-642244001. doi:[10.1007/978-3-642-24401-8](https://doi.org/10.1007/978-3-642-24401-8).
47. Fahrmeir L, Künstler R, Pigeot I, Tutz G. *Statistik Der Weg zur Datenanalyse*. 7. Auflageed. Heidelberg: Springer; 2010 ISBN: 978-3-642-01938-8.

How to cite this article: Schlick-Hasper E, Bethke J, Vogler N, Goedecke T. Flow properties of powdery or granular filling substances of dangerous goods packagings—Comparison of the measurement of the angle of repose and the determination of the Hausner ratio. *Packag Technol Sci*. 2022; 1-18. doi:[10.1002/pts.2678](https://doi.org/10.1002/pts.2678)