Release of powdery or granular substances from intact dangerous goods bags during road transport—Analysis of the causes and development of a test concept

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Abstract

The leaks of dangerous goods from actually intact bags detected in the years 2018 to 2020 tend to be at an almost constant high level. These releases of powdery or granular dangerous goods represent violations of the sift-proofness required in the dangerous goods regulations. This article first analyses the causes. The components of the bags that are affected by leaks are micro-perforations, joins and closures, in particular internal sleeve valves. A distinction must be made between bags closed in conformity with or contrary to the manufacturer's instructions. The particle release is determined by a number of influencing factors of the filling substance, the packaging and other boundary conditions. Therefore, a comprehensive test concept is developed in this work, which takes all these factors into account. The application of this test concept facilitates the planning of the test setup and the experiments. On this basis, the complex mechanisms involved in the release of solid substances can be systematically investigated in the test laboratory. To prevent releases of powdery or granular substances from intact bags, it is necessary that the user has access to the closing instructions and the relevant properties of the test substance used for the design-type approval. Further experimental investigations are needed to assess whether filling substances change their properties during transport and whether this enables them to escape.

KEYWORDS

bags, dangerous goods packagings, leak, particles, sift-proofness

1 | INTRODUCTION

During the transport of solid dangerous goods by road, dangerous goods are repeatedly released from bags that are actually intact. $^{1-3}$ This can involve both powdery and granular substances. Depending

on the type of substance, this can pose health hazards for people or hazards for the environment or material goods.

The international UN Model Regulations prescribe that all packagings for solid dangerous goods should be sift-proof (UN 4.1.1.14⁴). This term is synonymous with the packaging being absolutely

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impermeable to dry contents (UN $1.2.1^{4.5}$). This requirement also applies to the joins and closures of individual design types (e.g., paper bags, UN $6.1.4.18.1^4$). But this requirement is often not complied with in the practice of transport of dangerous goods bags by road. There is a need for an analysis of the causes of these substance releases and for a systematic test concept. $^{1.5}$

This work therefore initially provides an overview of the leakage of solid substances from intact dangerous goods bags during road transport detected by the German Police North Rhine-Westphalia Münster in the years 2018 to 2020. Exemplary substance releases from 2020 are shown. A systematic analysis of the causes is carried out. On this basis, an experimental investigation concept for the various release mechanisms is developed, which takes into account factors of the packaging as well as its contents. The application of this test concept is explained using two different examples. In the conclusions, possible remedial measures to prevent the leakages are suggested. An outlook on further necessary investigations is given.

There are also cases in which solid substances leak out of damaged bags. Mechanical damage during transport can occur, for example, during loading or unloading or through incorrect use of lashing devices or through the application of unsuitable means of load securing. In particular, bags are often damaged by using edge gliders or by tightening them with lashing straps.³

The focus of this article is on the leakage from actually intact, not damaged bags, under normal transport conditions.

2 | RELEASES OF POWDERY OR GRANULAR FILLING SUBSTANCES FROM DANGEROUS GOODS BAGS DURING ROAD TRANSPORT FROM 2018 TO 2020

In the course of random checks of the transport of dangerous goods by the German Police North Rhine-Westphalia Münster, Department Schwerlastgruppe of the Autobahnpolizei Münster (highway patrol Münster), an almost consistently high level of dangerous goods leaks from intact bags was found over the past 3 years. Table 1 compares the total number of detected releases of dangerous goods from intact bags from 2018 to 2020.

The UN numbers mentioned in the table represent the following substances (dangerous goods list of the UN Model Regulations⁴):

- UN 1328: HEXAMETHYLENETETRAMINE
- UN 1486: POTASSIUM NITRATE
- UN 1498: SODIUM NITRATE
- UN 1727: AMMONIUM HYDROGENDIFLUORIDE, SOLID
- UN 1823: SODIUM HYDROXIDE, SOLID
- UN 2811: TOXIC SOLID, ORGANIC
- UN 2813: WATER-REACTIVE SOLID
- UN 3077: ENVIRONMENTALLY HAZARDOUS SUBSTANCE, SOLID
- UN 3181: METAL SALTS OF ORGANIC COMPOUNDS, FLAMMABLE

 TABLE 1
 Detected releases of solid dangerous goods from intact bags, total numbers per year (Police North Rhine-Westphalia Münster)

	2018	20192	2020		
Total number of releases	19	13	22		
Areas affected by the leaks	Paper bags 5M2: Valves: 6 Sewn joins/closures: 7 Pasted joins/closures: 1 Micro-perforations: 1	Paper bags 5M2: Valves: 2 Sewn joins/ closures: 5 Pasted joins/closures: 1 Micro-perforations: 0	Paper bags 5M2: Valves: 2 Sewn joins/closures: 10 Pasted joins/closures: 4 Micro-perforations: 0		
	Plastics bags 5H: Valves: 1 Sewn joins/ closures: 1 Micro-perforations: 2	Plastics bags 5H: Micro-perforations: 2	Plastics bags 5H: Sewn joins/closures: 3 Microperforations: 3		
		Paper bags 5M1: Sewn joins/closures: 1 Leaking part not clearly identifiable: 2			
Released dangerous goods	UN 1486: 3 UN 2813: 1 UN 3077: 13 UN 3263: 1 UN 3378: 1	UN 1328: 1 UN 1486: 1 UN 1823: 1 UN 3077: 8 UN 3181: 1 UN 3262: 2 UN 3288: 1 (these figures also include leaks from damaged bags)	UN 1486: 2 UN 1498: 1 UN 1727: 1 UN 1823: 1 UN 2811: 1 UN 3077: 13 UN 3253: 1 UN 3261: 1 UN 3378: 1		

- UN 3253: DISODIUM TRIOXOSILICATE
- UN 3261: CORROSIVE SOLID, ACIDIC, ORGANIC
- UN 3262: CORROSIVE SOLID, BASIC, INORGANIC
- UN 3263: CORROSIVE SOLID, BASIC, ORGANIC
- UN 3288: TOXIC SOLID, INORGANIC
- UN 3378: SODIUM CARBONATE PEROXYHYDRATE

After a slight decrease in 2019, an increase to a similar level as in 2018 was recorded in 2020. In all 3 years, the proportion of substances of UN number 3077 is the highest. Annual differences in the detected number of releases may be due to the fact that the number of inspections fluctuates per year and that controls are only carried out on a random basis. Therefore, the estimated number of unreported cases is likely to be higher.¹

The data recorded in Table 1 are leaks that can be assessed objectively and factually on site by the police during their controls. For the scientific analysis of the causes of exit, the following information would also be relevant in a first step:

- Was the bag as used in conformity with the packaging design type (material, composition, construction)?
- Had the substance as transported similar physical properties as the substance which was applied at the design type test?
- Were the other conditions in conformity with the design type approval (e.g., gross mass)?

For the objective factual assessment of violations during the daily work of the control officers, such further technical information relating to the approval is not legally required. Scientific or advanced technical procedures or verification procedures are neither possible nor intended by the police. Such further investigations can only be attempted by fine authorities, public prosecutors or courts with the inclusion of official reports. The necessity of this is then decided on a case-by-case basis. With the normal release of dangerous goods from bags, the technical causes are almost never investigated further. Exact

driving routes are also not required for the police findings and are therefore not determined.

For this reason, the data listed in Table 1 cannot be used to assess whether or not all the requirements according to the approval and the underlying test report were met in the specific cases.

3 | SUBSTANCE RELEASES FROM INTACT BAGS IN 2020—ANALYSIS OF THE CAUSES

Figures 1 to 13 display exemplary releases of hazardous powdery or granular substances from dangerous goods bags in 2020, detected by the German Police North Rhine-Westphalia Münster. Affected packaging design types are bags of codes 5H3 (woven plastics bags—water resistant), 5H4 (plastics film bags), and 5M2 (paper bags, multi-wall, and water resistant).

As just mentioned, in the case of these identified releases, it is ultimately unknown whether the packaging concerned was used in accordance with its design type approval or not.

Figures 1 and 2 show the release of potassium nitrate (UN No. 1486) from the micro-perforations of a 5H4 bag. Micro-perforations are intentionally created holes within the layers of a bag, which are intended to allow air to escape from the inside during industrial filling process.¹

There are several explanations for the leakage visible here. On the one hand, particles can escape if the perforations in the various layers of the bag are not offset. Particles can then migrate directly to the outside. On the other hand, it is also possible that particles initially migrate through the micro-perforations of one of the layers and accumulate between the layers. The particles can then migrate from this interspace through the perforations of the next layer and thus escape to the outside. The precondition in both cases is that the cross section of the particles is small enough to fit through the cross section of the perforation openings. In the present case, it is unclear whether the filling material was unsuitable for the bag, that is,



FIGURE 1 Release of a UN No. 1486 substance from the microperforations of a 5H4 bag (Police North Rhine-Westphalia Münster)



FIGURE 2 Release of a UN No. 1486 substance from the microperforations of a 5H4 bag (detail) (Police North Rhine-Westphalia Münster)



FIGURE 3 Release of a UN No. 3077 substance—leaking internal sleeve valve of a 5M2 bag (Police North Rhine-Westphalia Münster)



FIGURE 6 Release of a UN No. 3077 substance—leaking stitched closure of a 5H3 bag (Police North Rhine-Westphalia Münster)



FIGURE 4 Release of a UN No. 3261 substance—leaking pasted join of a 5M2 bag (Police North Rhine-Westphalia Münster)



FIGURE 7 Release of a UN No. 3077 substance—leaking stitched closure of a 5M2 bag (Police North Rhine-Westphalia Münster)



FIGURE 5 Release of a UN No. 3077 substance—leaking pasted closure of a 5M2 bag (Police North Rhine-Westphalia Münster)

whether it already had this small particle size when it was filled or whether the small, emerging particles were created by abrasion during transport. The release of a powdery substance of UN No. 3077 through the internal sleeve valve of a 5M2 bag is visible in Figure 3. There are valves that are not pasted, heat-sealed, or sewn, but just turned inwards; in the case of powdery substances, the escape of solid filling substances often occurs.^{1,3} The shorter the length of the internal sleeve valve and the better the flowability of the powder, the higher is the amount of powder that is released from a bag as bulk solids in a drop test.¹

Figure 4 displays the release of a powdery UN No. 3261 substance through the pasted join of a 5M2 bag. In the case of powder with good flowability, powder can emerge from insufficiently pasted joins already during manual filling.¹ In the case of the leakage shown in Figure 4, it is unclear whether the joins were not adequately pasted during production or whether the joins were loosened during road transport.

The pasted closure of 5M2 bags can also be the leaking part; see Figure 5. This weak point can as well be caused either by an inadequate gluing during production or by loosening during transport.



FIGURE 8 Release of a UN No. 3077 substance—leaking stitched closure of a 5M2 bag (Police North Rhine-Westphalia Münster)



FIGURE 11 Release of a UN No. 3077 substance—leaking stitched join of a 5M2 bag (Police North Rhine-Westphalia Münster)



FIGURE 9 Release of a UN No. 3077 substance—leaking stitched closure of a 5M2 bag (Police North Rhine-Westphalia Münster)



FIGURE 12 Release of a UN No. 3077 substance—leaking stitched join of a 5M2 bag (Police North Rhine-Westphalia Münster)



FIGURE 10 Release of a UN No. 3077 substance—leaking stitched closure of a 5M2 bag (Police North Rhine-Westphalia Münster)

Figures 6-10 show the release of UN No. 3077 substances from the stitched closures of 5M2 bags. The particles can exit both from the seam holes themselves (Figures 6 and 9) or from the interspace between the individual stiches (Figures 7, 8, and 10). In the first case, the cross-section of the seam holes is relevant for the passage of the particles and, in the second case, the length of the open space.

Figures 11 and 12 display the release of powdery substances through the stitched joins of 5M2 bags. The particles can emerge through the seam holes themselves or through the space between the different layers of the double-stitched seam.

In the cases shown in Figures 6-12, it is unclear whether a filling substance with an unsuitable particle size was chosen. A large number of test reports do not contain any information on the particle size of the test substance used for the design type approval. This makes it difficult for the user to choose the substance that can be transported in the respective design type.

There are also cases in which the bags are closed contrary to the manufacturer's instructions. In most of these cases, the bags are only provisionally closed with adhesive tape; a release of particles from these closures is likely to occur.³ Figure 13 shows a damaged area that is only temporarily sealed with adhesive tape. Figure 14 shows a closure that is only provisionally sealed with adhesive tape, from which the product is leaking.³



FIGURE 13 Release of a UN No. 3077 substance—provisionary closed damaged area (adhesive tape) in a 5H4 bag (Police North Rhine-Westphalia Münster)



FIGURE 14 Release from the closure of a paper bag that is only provisionally sealed with adhesive tape (Police North Rhine-Westphalia Münster)

The substance releases from intact bags visible here contradict the requirement of sift-proofness of dangerous goods bags in the UN Model Regulations. All these mechanical transport processes of individual particles or agglomerates through the various components of the bags just shown are caused by vibrations or shocks occurring during road transport.

Theoretically, also a pressure difference can be the driving force for the release of powdery substances from intact bags. ⁵ During road transport, an external lower pressure could occur during the passage of mountain passes.

Figure 15 summarizes the affected components of the leaking bags.

This figure displays only the leaking components of the bags that are factually recognizable for the police on site. A statement about the use of the packaging according to its design type approval cannot be made from this. The numbers in brackets indicate the number of cases detected in 2020 for the affected component (see Table 1).

When substances are released from intact bags, a distinction can be made between bags that are closed according to their approval and those that are closed contrary to the manufacturer's instructions. In the latter case, the contents are likely to escape from the provisionally sealed closure. If the bags are properly closed, the following critical components for the release can be distinguished: surfaces, joins, and closures. A release through intact surfaces can only take place through micro-perforations. Leaking joins can be pasted, heat-sealed, or sewn. If the weak point for the leakage is the closure, this can also be pasted, heat-sealed, or sewn, or it can be an inner sleeve valve, which is merely folded inwards.

Figure 16 shows the relationship between the influencing factors relevant to the release of solid substances from intact dangerous goods bags during road transport.

The weak points of the components affected by leakage are already shown in Figure 15. The duration of release can either take place over a longer time period during transport or the release can be caused by a sudden singular event. Such impacts may be caused by a drop of a single packaging, shocks during transport, or hard placement of a pallet. As mentioned above, both vibrations and shocks during transport, as well as a pressure differential between the inside of the bags and the environment, can be the driving force for the transport of material. The combined effect of vibrations and low pressure on the release from dangerous goods packagings for liquids has already been investigated in Singh et al.

The *powdery or granular substance* can be characterized by different parameters.

The form of release of particles can take place both as coherent collective of particles (bulk solids) and in the form of an airborne transport of particles (solid aerosol).⁵

The particles can either be in their original *form* or there can be changes in the properties of the particle collective during transport, for example, by abrasion. The stacking pressure exerted on the individual particle layers as well as friction can lead to a comminution of the original particle collective. As consequence, one and the same bag could be leakproof to the original filling material with its original particle size distribution, but not to the more fine-grained substance formed during transport.

The name and description of the dangerous substance (according to the UN Dangerous Goods List, UN 3.2.1⁴) determines its UN number and the packing group and thus also the type of packaging in which the substance may be transported.

Various geometric and mechanical quantities are summarized as *physical properties* in this overview. Particle shape and particle size or particle size distribution are basic geometric parameters for describing a powdery or granular substance. The mechanical parameters include, for example, the hardness of the particles, the solid density, the bulk density and the tamped density of the bulk material. For German UN approvals of bags, the bulk density of the contents must always be specified.

The flow properties are of fundamental importance for the release of the particles in the form of bulk solids. The flowability of a powdery or granular substance can be described by various

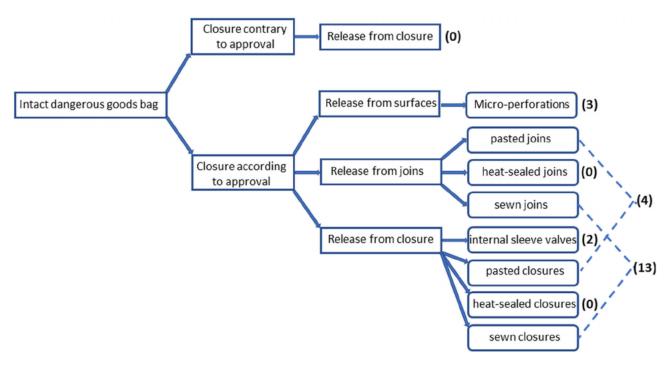


FIGURE 15 Components of the bags affected by leakage

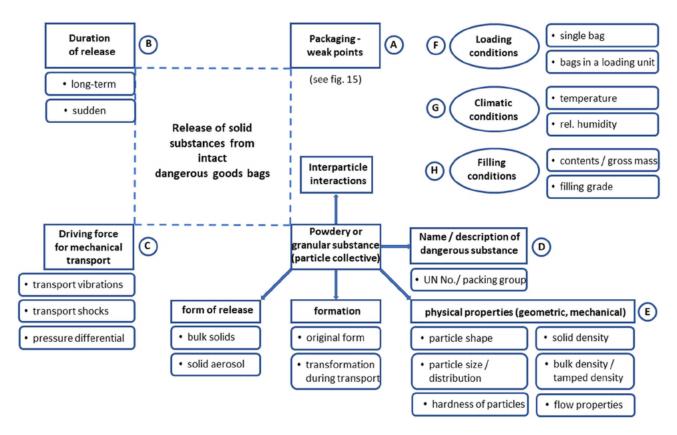


FIGURE 16 Influencing factors relevant for the release of solid substances from intact dangerous goods bags during road transport

parameters. For German UN approvals, the angle of repose of the filling substance has to be specified. However, no specific method has yet been defined for measuring this quantity. The Hausner factor, the quotient of tamped and bulk density, is also an established parameter for describing the flow behaviour of bulk solids. 1,7

The *interparticle interactions* are influenced by both the climatic conditions, the loading conditions and the filling conditions. The interactions between the particles include, for example, physical adsorption, elastic and plastic deformation, ductile and brittle fracture, solid electrification and particle attrition.⁸ The adhesion between particles and the tendency to form agglomerates are also interparticle properties.

Both of these properties are influenced by the *climatic environmental conditions*, especially the relative humidity. In general, a high moisture content enhances the stickiness of powders and prevents flow.⁹

The *loading conditions*, that is, the position of a certain bag within the loading unit, also have an influence on the condition of the powdery or granular substance and thus on its release. The possibility of the formation of finer fractions within a bag through the pressure of layers above it has already been mentioned. The load on the filling contents of single bag is therefore different from that in a bag in the lowest layer of a pallet. The hardness of the particles is an important physical variable that influences the interparticle interactions and thus the formation of finer particles through abrasion or crushing. It is currently not determined within the scope of the type approval of dangerous goods bags.

The interparticle properties just listed are not directly adjustable influencing parameters. Rather, they result from the effect of the transport conditions, that is, the climatic conditions and the loading conditions, on the powdery or granular filling substance.

The filling conditions of the respective bag, for instance, the mass of the filling substance, the gross mass of the bag or the resulting degree of filling, are also important factors influencing the behaviour of bag and its contents during transport and therefore the tendency for leakage.

For the development of a test concept to investigate the release mechanisms of solid substances from intact bags, only those influencing factors can be selected as parameters that can be directly set and varied for a test program. These influencing factors, which can be directly specified for a practical test, are marked in Figure 16 with the capital letters A to H. It is apparent that the investigation of particle release is a complex issue, since a large number of combinations of the individual parameters is possible.

4 | DEVELOPMENT OF AN EXPERIMENTAL TEST CONCEPT

For a comprehensive experimental investigation of the leakage of powdery or granular substances from intact dangerous goods bags, the parameters of following categories have to be set for the laboratory tests. The designation of the categories of test parameters is chosen analogously to the influencing factors relevant for the release in Figure 16.

(A) Type of packaging and weak point to be investigated

Both the specific design type of the dangerous goods bag (5H woven plastics or plastics film bag; 5L textile bag; 5M paper bag)

and the weak point to be examined must be selected. Components of the bags that are frequently affected by leaks have already been shown in Figure 15. In addition, it must be determined whether the bag is to be tested as a whole or whether only the critical weak point of the bag is to be investigated. This has a direct impact on the test design. Regarding the release from micro-perforations, for example, a test procedure could be developed in which the side surfaces of a bag are cut out and clamped in a special device and only these are subjected to a certain filling substance. The position of the microperforations in the individual layers could be varied in a test program.

- design type of dangerous goods bag
- · weak point of packaging
- extent of examination (entire packaging or just the affected area [cut-out])

(B) Test duration

During the transport of solid dangerous goods, it cannot be excluded that the properties of the originally filled powdery or granular substance will change during the transport (see Figure 16, formation). In order to consider such effects of long-term release, the test duration should be selected accordingly or abstracted, for example, when performing vibration tests. Analogous to the release of substances, there are the following two variants:

- long-term test (for the investigation of a longer process during transport)
- short-term test (for sudden release)

(C) Test method

Depending on the driving force for the substance release, a corresponding test method or a combination of these must be selected. In general, vibration tests or shock tests, which simulate the vibration spectrum of road transport to be considered, are appropriate methods. If the cause for the mechanical release is a shock due to the drop of an individual packaging, a drop test can also be used as a method to investigate basic effects. If a pressure difference is additionally responsible for the leakage, this can be taken into account in the test program, for example, by lowering the pressure within a test chamber.

- · vibration test
- · shock test
- · drop test
- · differential pressure test
- combination of test methods

The parameters used for the test method must also be specified, for example, drop height and drop orientation in a drop test or applied vibration spectrum in a vibration test.

As explained above at the end of Section 2, in most cases of releases of dangerous goods from bags detected by the police, no information is available about the exact route taken. Therefore, a

specific test program for the vibration test during road transport cannot be recommended across the board. An overview over the broad variety of road vehicle vibration simulation for packaging testing purposes is given in Lepine et al. 10 Even if there are limitations on these, 10 typical or assumed standard vibration spectra are often used in practice. For example, a first step could be to use the vibration spectrum in ASTM D4169-16.^{5,11}

(D) Powdery or granular test substance—name and description

The product name and description of the solid test substance and its UN number and packing group, if it is a dangerous good, must be given.

(E) Powdery or granular test substance—physical properties

The following physical properties are essential to characterize the product used in relation to the release of particles:

- Particle shape
- particle size/particle size distribution
- bulk density and/or tamped density
- flow properties

(F) Arrangement of the test sample(s) during the test

During transport, the position of a bag within a loading unit can be different. Both individual packaging and a complete loading unit can be subjected to a specific test procedure. In the latter case, the type of load securing with which the bags are secured on the loading unit is also relevant. For example, a vibration test can be carried out on a single bag as well as on a pallet loaded with bags.

- single bag
- defined arrangement of several bags

(G) Climatic conditions (for pre-conditioning and testing)

The climatic conditions for the pre-storage of filling substance and bags as well as for carrying out the actual test must be specified. The temperature and the relative humidity are particularly important here.

(H) Filling conditions of the test sample(s)

With regard to the filling conditions of the test sample(s), the filling mass of the test substance, the gross mass of the bag or its filling grade must be specified for the test.

(X) Form of release to be investigated

During the transport of dangerous goods, the release of powdery or granular substances from intact bags constitutes a violation of the requirement for sift-proofness and is therefore an undesirable process. Therefore, in Figure 16, the form of release is not marked as a directly adjustable parameter for a test program. The release can either take place as a leakage of bulk solids or as escape of a particleair flow (solid aerosol). However, the form of release to be examined determines the sensors required for the test setup. Therefore, this category is defined as part X of the test concept.

Table 2 provides an overview of the points of the test concept just listed. In this table, points B, C and X have been combined into the category "Parameters related to the test method." Points D and E

TABLE 2 Test concept for the investigation of the release of powdery or granular substances from intact dangerous goods bags

Designation	Category	Parameters to be specified
A	Type of packaging and weak point to be tested	 design type of dangerous goods bag weak point of packaging extent of examination (entire bag or cut-out)
B, C, X	Parameters related to the test method	Type of test: - vibration test - shock test - drop test - differential pressure test - combination of test methods and applicable test parameters Test duration: - long-term test - short-term test Form of release: - bulk solids - solid aerosol
D, E	Powdery or granular test substance	Name and description: - chemical designation/ product name - UN number (if applicable) - packing group (if applicable) Physical properties: - particle shape - particle size/particle size distribution - bulk density and/or tamped density - flow properties
F	Arrangement of the test sample(s) during the test	single bagdefined arrangement of several bags
G	Climatic conditions (for pre-conditioning and testing)	- temperature - relative humidity
Н	Filling conditions of the test sample(s)	filling mass of test substancegross mass of the bagfilling grade

have been grouped together to form the category "Powdery or granular test substance."

This scheme can also be used to classify experiments in which no packaging or packaging component is involved, but in which the change of the properties of the filling substance under certain conditions is examined separately in suitable test devices. Category A of the test concept would then be omitted. In the context of preliminary studies, for example, it could be investigated on a small scale to what extent interparticle abrasion occurs under a specific vibration exposure and under the application of a specific load.

By defining the parameters of the individual categories of this test concept, the test setup and the required sensors can be planned on this basis.

In practice, an important question is whether there is a demand to individually test all relevant solid dangerous goods or whether these could possibly be summarized in sensible categories. A possible creation of categories would result from the respective particle sizes. Examples of the particle size ranges of the solid phase of two-phase disperse systems are given in Stieß. 12 According to this, the particle sizes of bulk solids range are between 10^{-6} m and 10^{-2} m.

In the pharmaceutical sector, powders are two-phase disperse systems with a grain size of up to 1 mm (10⁻³ m).¹³ According to Herzfeldt, ¹⁴ particle sizes up to 2 mm are still referred to as coarsegrained powders. However, the flow properties are also relevant with regard to particle exit. A final categorization of solid substances with regard to their physical properties for the release can only be made through a systematic implementation of tests.

The test concept can also be used to experimentally investigate the influence on particle release when packaging is not used in accordance with the design type approval.

5 | EXAMPLES FOR THE APPLICATION OF THE TEST CONCEPT

Two examples for the application of the test concept are given below. Details of the examinations can be found in previous studies. ^{1,15} The parameters of the individual categories of the test concept are shown in Table 3 as an overview. In both cases, basic issues should be investigated that can be caused by a single sudden impact.

5.1 | Example 1: Drop tests to determine the sudden release due to impacts of two powdery substances in the form of bulk solids from the internal sleeve valves of a 5M2 paper bag

The aim of these measurements¹ was to investigate the sudden release of two powdery substances (Esplas H130 and zinc oxide

"Rotsiegel" [UN No. 3077]) from the internal sleeve valves of a 5M2 paper bag (max. gross mass: 25 kg) with pasted joins when subjected to an impact load. The focus was to investigate the influence of the length of the internal sleeve valve on the amount of particles released upon sudden impact for different filling products.

A drop test from different heights served as the test method (flat drop on a wide face). In addition, the length of the internal sleeve valve was varied. Since gross leaks often occur in practice with this type of quasi-open closure (see Figure 3), the focus of these measurements was the determination of the mass of particles escaping in the form of bulk solids. Two different powdery filling substances with different flow properties were used. A single bag should be tested at a time.

5.1.1 | Result of the application of the test concept

The evaluation of the categories of the test concept showed that a drop test apparatus according to ISO $7965-1^{16}$ represents a suitable test setup for this type of question. Since the measurement of the amount of particles that had escaped as bulk solids was to be carried out, a laboratory balance was chosen as the sensor.

Already during the manual filling process of the test samples the pasted joins of almost all test samples were not sift-proof to the Esplas H130 powder, due to its high flowability. The shorter the length of the inner valve, the higher the mass of powder that escapes upon impact.¹

5.2 | Example 2: Drop tests to determine the sudden release due to impacts of a powdery substance in the form of a solid aerosol from the pasted joins and closure of a 5M2 paper bag

The aim of these measurements¹⁵ was to investigate the sudden release of a powdery substance (zinc oxide "Pharma 8" [UN No. 3077]) as airborne particle transport from an entire 5M2 paper bag with pasted joins and closure (max. gross mass: 26 kg) when subjected to an impact load.

As in Example 1, drop tests were carried out as flat drops on a wide face. The test samples were originally closed and filled by the

TABLE 3 Examples for the application of the test concept

No.	Α	В	С	х	D	E	F	G	Н
1 ¹	5M2, internal sleeve valve, entire bag	short-term (sudden impact)	drop test	bulk solids	Esplas H130; zinc oxide "Rotsiegel"	see Schlick- Hasper ¹	single bag	preconditioning at 23°C, 50 % rel. humidity	see Schlick- Hasper ¹
2 ¹⁵	5M2, pasted joins and closure, entire bag	short-term (sudden impact)	drop test	solid aerosol	Zinc oxide "Pharma 8"	see Bruchno ¹⁵	single bag	Preconditioning at 23°C, 50 % rel. humidity	see Bruchno ¹⁵

manufacturer (pasted closure and pasted connections). Since a gross leak of particles escaping in the form of bulk solids was not expected for this question, the focus was on measuring the amount of airborne particles released after the impact.

5.2.1 | Result of the application of the test concept

The drop test apparatus already used in Example 1 was used as the core piece of the test setup. In order to record the airborne particles suddenly escaping after the impact of the bag, the experimental setup was expanded to include an automatically closing test chamber. After the impact of the bag, the homogenized particle-air mixture was fed to an aerosol particle size spectrometer. This sensor enables both the determination of the mass of the particles released into the air and the determination of their particle size distribution.

Although the bags were originally filled and closed, the results indicated that they were not sift-proof. In all drop tests, an airborne particle release was measured. The amount of particles escaping from the bags was compared with regard to the exceedance of occupational exposure limits. Airborne particles can cause health problems if inhaled. If a single bag drops under the idealized conditions of this test setup, no hazard is expected.¹⁵

Figure 17 shows how the test setup used for the measurements in Example 1 was expanded for the investigations in Example 2.

These two examples show that the application of a systematic test concept has advantages when planning the test setup. If the experimental setup is carried out in a modular way, its basic elements can also be used to investigate other questions. In a next step, a model shaker could be inserted into the test chamber shown in

Figure 17. This modified test setup could then be used to determine the long-term release from a single bag as an airborne particle flow due to vibration. The sensor system of the experimental setup would then already have been tested. This simplifies the implementation.

6 | CONCLUSIONS

There are various causes for the release of powdery or granular substances from intact dangerous goods bags. These are systematically analysed in this work.

Bags that are not closed according to the manufacturer's specifications are likely to leak. The leakages detected in practice are influenced by factors that affect both the respective packaging with its individual weak points and the respective filling material with its substance-specific chemical and physical properties. In addition, there are other influencing factors, such as the duration of release, the underlying driving force for the transport of substances, the form of release and a potential change in the properties of the product. The prevailing climatic conditions and the conditions within a loading unit also play a role.

For a comprehensive experimental investigation of the release mechanisms from intact dangerous goods bags, these parameters must be specified. Therefore, a test concept was developed in this work, which considers the relevant categories for the release of powdery and granular substances from bags. The application of this test concept in test laboratories enables the planning of the test setup and the selection of the required sensors. This procedure leads to synergy effects if individual components of an experiment set-up can also be used for other issues.

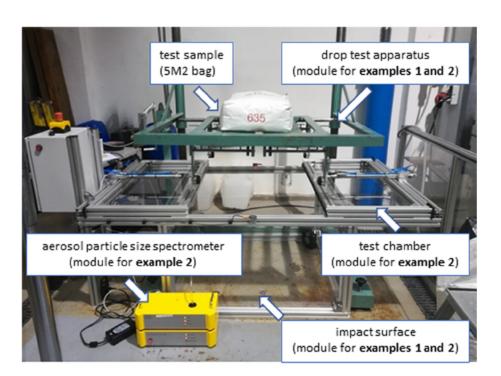


FIGURE 17 Modules of the experimental set-up used for the investigations in Examples 1 and 2

In order to prevent leakage from intact bags, some remedial measures can be derived from the cases shown. On the one hand, it must be ensured that the manufacturer's closing instructions are accessible to the subsequent user. On the other hand, the test report should include all physical properties of the test substance that are relevant for the release of solid substances. This information must also be made available to the user. This is to prevent unsuitable contents from being filled into the bags.

The flow properties are of particular importance for the release of the particles in the form of bulk solids. For this reason, measurements are presently being carried out at BAM in order to compare various methods currently used in testing practice for determining the flow properties. Both the reproducibility of the individual methods and their advantages and disadvantages of the application to certain extreme cases of solid substances are to be worked out.

Experimental studies are necessary to assess whether original filling substances change their properties during transport and whether these processes enable or facilitate leakages. Therefore, measurements are planned to investigate the possible formation of finer fractions from coarse filling substances on a small scale. There is a need to further experimentally investigate the complex mechanisms of product release from intact bags. The systematic test concept makes an important contribution to this.

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