Research Article

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Impact and Friction Sensitivities of PETN: I. Sensitivities of the Pure and Wetted Material

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Dedicated in Memoriam to Prof. Dr. Peter Elsner

Abstract: Pentaerythritol tetranitrate (PETN) is a sensitive and brisant explosive. PETN is transported wetted (25 %) with water to limit its impact and friction sensitivities. Literature on its sensitivities in function of its water content is controversial as the measurements were performed by several operators and laboratories rendering difficult to compare the values. Literature studies of mechanical sensitivity methods show the weaknesses and problems of mechanical measurements. Indeed, it is important to analyze a sample with standardized machines and by a single operator. During this work, pure PETN samples with water contents of 0 to 35 % were prepared and the water content was measured by Karl-Fischer titration. The sensitivities were analyzed by the "BAM Fallhammer" and the "BAM Friction Apparatus". The resulting trends were analyzed and discussed with regard to their meaning for handling safety. The study should help to better assess dangers when working with wet PETN (10–25 %) in order to avoid accidents.

Keywords: PETN · Explosives · Impact Sensitivity · Friction Sensitivity · Testing and Assessment

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1 Introduction

1.1 About Pentaerythritol Tetranitrate

The Tetranitrate of pentaerythritol, PETN, is a versatile explosive used in boosters, detonating cords, detonators etc. [1–3] Industrial PETN large-scale manufacturing has a long tradition reaching back to the 1930's, but accidents still occur on plants. After nitration, the nitrate is filtered, washed, neutralized, and mostly recrystallized. [4] In a dry state, an impact sensitivity of 3–4 J and a friction sensitivity of about 60 N renders the material sensitive to very sensitive. [5-6] To ensure safe packing and transportation, traditionally water is added. The sensitivity of dry pentaerythritol tetranitrate is extensively studied, but surprisingly very few data is known on the impact and friction sensitivities of the wetted material. In the ICI research brochure no. 46 by Gow, it is stated that the mechanical sensitivity is gradually decreasing, from 2–3 J to 5–6 J with rising water content, from 0 % to 35 %. [7] Coffey et al. measured impact sensitivities of PETN with a particle size of 5 μm. At a vacuum dry state ($<$ 0.1 % water), PETN shows an increased sensitivity of 2 J compared to air dry with 3 $\pm$ 0.5 J. [8] More recent data is measured by the UN in the report UN/SCETDG/47/INF.8. The spanish experts show that a water content of 9 % decreases the sensitivities to 25 J and 80 N. [9] In the following report UN/SCETDG/49/INF.9 the german experts discovered an inhomogeneous distribution of water in the delivered samples. Homogenized samples were measured dry, 4 J and 54 N, with 9 % water, 25 J and 60 N, and 15.2 % water, 25 J and 72 N. [10] A large decrease in impact sensitivity besides a rather small decrease in friction sensitivity is observed. From a purely economic point of view, the lowest possible water content is beneficial, whereas insensitive material is preferable for safe handling and transportation. A complete set of data is necessary so that the appropriate water content can be selected for each application and the danger can be better assessed (Figure 1).

In this study, the influence of water on the mechanical sensitivity of pure PETN is investigated. The difference between pure and industrial PETN is the presence of small

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Figure 1. The importance of mechanical sensitivity.

ammounts of Dipentaerythritol hexanitrateDIPEHN in the industrial PETN. A study on the properties of industrial PETN is ongoing.

1.2 Assessment of Impact Sensitivities

The traditional method to detect a reaction towards a mechanical stimulus is the determination of the impact sensitivity (Figure 2). When enough impact energy or more accurate plastic deformation is applied on a sample, ignition can occur [11]. The ignition sites are called hotspots and are caused through adiabatic shear bands and gas compression [12–14]. For PETN the initiation through hotspots is believed to happen at 400 to 500 °C [15].

The "ERL Type 12 Drop Weight Impact Sensitivity Apparatus" [16] and the "BAM Fallhammer" [17] are two widely used and established instruments. The ERL machine has a plane anvil on which the sample is hit by a striker sitting above it. The energy on the striker is generated by a 2.5 kg weight. In contrast, the BAM apparatus uses a sample holder that confines the sample and is exchanged every measurement. A 1 kg, 5 kg, or 10 kg weight from a certain height (1–40 cm) delivers an energy of 1–40 J on the sample.

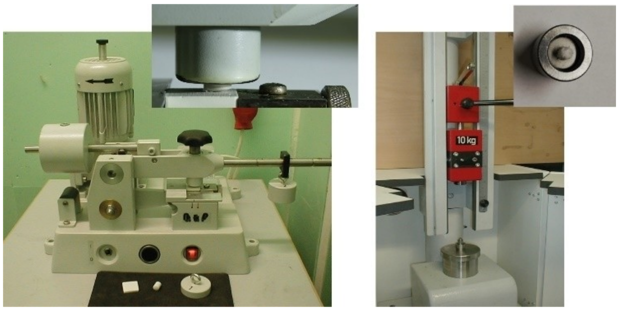

Figure 2. Friction and impact test apparatus used in this work.

Single test results are "no reaction", "decomposition" or "explosion" and those are assigned to the result "positive" or "negative" depending on the procedure.

At the end of a test series an energy is determined at which a certain probability of a positive result is given. Typically, an E50, 50 % probability, value is determined on the ERL device, usually with the Bructon or Neyer D method [18–19]. The detection is often performed with the help of measuring instruments (microphone etc.). In every case strict criteria should be defined before the experiment and one should be aware of possible pitfalls when using technical help [20]. The BAM device can be used for E50 values but more common are "one out of six" measurements. The one out of six method means that at least one positive result occurs in six trials at certain energy, but six negative results are obtained with lower energy. Therefore, the lower detonation limit is detected, which is different and not directly comparable to the 50 % probability value. The detonation is usually detected by the operator which is possible because the sample is confined and the positive result is usually audible [17].

The Fallhammer method comes with some general drawbacks. Depending on the apparatus and ist wear, different energies than the theoretically calculated are delivered into the sample. Energy is lost due to elastic deformation or friction of the apparatus [8]. Several studies were performed to compare sensitivity results of different institutions. With uniform test regulations and identical samples, large deviations between different laboratories are observed. This is the case for both BAM and ERL instruments [21–23]. An intense study by Marrs et al. shows that Fallhammer experiments with PETN are reproducible over decades at the same institute, with some deviation. Furthermore, it becomes clear that PETN is excellently suited to evaluate experiments acoustically [24]. There are some materials (e.g. thallium azide) where it is difficult to detect the actual sensitive character via this method [25]. All studies attribute deviations to the operator, protocol, or environment. It can be concluded that the comparison of data between laboratories should be done with care.

1.3 Assessment of Friction Sensitivities

The friction sensitivity testing is the second most used mechanical sensitivity test (Figure 2). Over time the "BAM friction apparatus" [17] has become established, but friction tests with an impact component such as the Ball Drop Impact Test [26] are also known. The friction measurement in combination with the impact test gives a good overview of the mechanical sensitivity of a material. The BAM apparatus consists of a fixed porcelain peg and a moving porcelain plate. The plate is moved one centimeter forth and then returns to the starting position. The peg is part of a one-sided lever and the force is generated by nine different weights that can be fixed in six notches on the lever. Thus 0.5 to

360 N force can act on the specimen. Evaluation is similar to the impact test, E50 is determined with STANAG [27], one out of ten with LLNL [28–29] and one out of six with the UN procedure [17]. Drawbacks are comparable to those of the impact method. The porcelain surfaces and the operation of the apparatus are major uncertainty factors.

2 Experimental Section

2.1 Sample Specification

A sample of pure PETN delivered from the "Fraunhofer ICT" was used for this study. The purity was determined by elemental analysis, IR spectroscopy and ^{1}H, ^{13}C and ^{14}N NMR. Thermal properties were determined by differential thermal analysis (DTA) and differential scanning calorimetry (DSC) (Supporting Information). The DSC measurements show that no change in the melting or decomposition point is observed due to wetting.

The morphology of the dry sample was analyzed by scanning electron microscopy (SEM) and optical microscopy. The particle size distribution was analyzed using sieves with a mesh size of 100, 300, 500, 600, and 1000 micrometers.

Analysis of pentaerythritol tetranitrate: DTA (5 °C min^{-1}, onset): 143 °C (melt.); Sensitivities: BAM Fallhammer: 3.5 J, friction tester: 54 N; IR (ATR) $\tilde{v}$ (cm^{-1}) = 2986 (w), 2904 (w), 1638 (s), 1630 (s), 1473 (m), 1396 (w), 1305 (m), 1283 (s), 1266 (s), 1036 (m), 998 (s), 939 (m), 833 (s), 752 (s), 701 (s), 619 (s), 458 (m); Elem. Anal. ($C_5H_8N_4O_{12}$, 316.15 g mol^{-1}) calcd.: C 19.00, H 2.55, N 17.72 %. Found: C 19.01, H 2.28, N 17.47 %; ^{1}H NMR (DMSO-D$_6$, 400 MHz, ppm) $\delta = 4.70$ (s, 8H); ^{13}C NMR(DMSO-D$_6$, 101 MHz, ppm) $\delta = 70.3$, 40.8; ^{14}N NMR (DMSO-D$_6$, 29 MHz, ppm) $\delta = -45$.

The morphology and particle size distribution are shown in Figure 3. The nitrate ester crystallizes in elongated blocks that are partly grown together. The crystal surface is smooth and the edges are sharp and not crushed. The mean particle size is at 200 to 400 µm. Particles below 100 and above 600 µm are rare.

2.2 Sample Preparation

About one and a half gram of dry PETN was weight in and wetted to the desired water content. The sample was shaken for two hours with an overhead shaker and additionally mixed with a spatula directly before each measurement. As water is only attached loosely to the crystalline material it evaporates, settles, or attaches to the walls of the vessel. Therefore, the water content was determined by coulometric Karl-Fischer titration with a 899 coulometer and 860 KF Thermoprep oven from Metrohm. [30] Before and after each friction sensitivity measurement a representative sample (ca. 20 mg) was taken. This sample was analysed by the Karl-Fischer device using the oven method. Therefore, water was extracted from the sample oven (80 °C) into the cell (nitrogen flow). A sample was considered dry at water content below 0.1 %. Table 1 shows the measured water contents after preparation and after a friction sensitivity measurement was performed. The average of three meas-

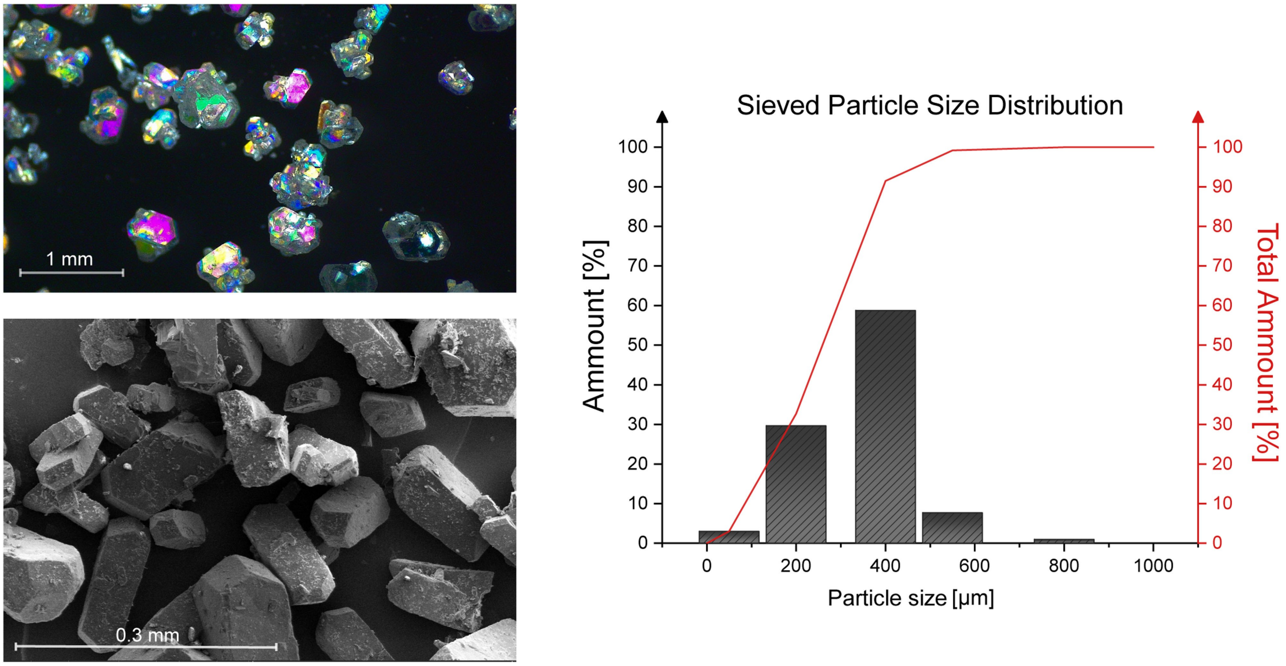

Figure 3. Optical microscop image (top left), SEM image at 350x magnification (bottom left) and particle size distribution (right).

Table 1. Water content as determined by coulometric Karl-Fischer titration. A triple determination with standard deviation is shown.

Water content	Initial [%]	After friction sensitivity determination [%][31]
dry	<0.1	<0.1
5 %	4.6 ± 0.1	4.3 ± 0.3
10 %	9.5 ± 0.2	9.4 ± 0.1
15 %	14.7 ± 0.7	13.5 ± 0.7
20 %	19.0 ± 0.7	17.9 ± 0.7
25 %	25.3 ± 0.9	23.1 ± 1.6
30 %	30.3 ± 2.3	26.6 ± 1.9
35 %	36.4 ± 2.3	34.5 ± 0.25

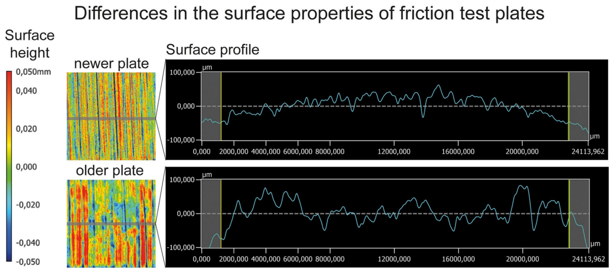

Figure 4. Surface height and profile of BAM friction test plates.

urements was calculated and given with its standard deviation. At 5–20 % water the samples taken shows to be drier than initially prepared. This is believed to happen due to evaporation and the separation of water. In contrast at 25 % and higher the samples show higher water contents.

At this point, water begins to separate from the PETN and when a sample is taken water attaches to the spatula due to surface tension. The water content generally drops a few percent during sensitivity measurement with a larger deviation at higher contents. (The raw data can be found in the Supporting Information).

2.3 Sensitivity Measurement

The impact sensitivities were determined on a "BAM Fallhammer" by Reichel & Partner. Steel rings and collars are purchased from OZM Research (2020). For 1–5 J the 1 kg weight and for energies above that the 5 kg weight were used. The contact surfaces of the guiding rail and the weight was greased with powdered graphite [32]. The result "explosion" was assigned to a positive test and "decomposition" or "no reaction" was assigned to a negative test. The samples were taken with a cylindrical 40 mm^3 measure and tapped directly into the sample holder. The friction sensitivities were measured on a "BAM Friction Apparatus" by Reichel & Partner. Porcelain plates and pegs are purchased from OZM Research (2020). The surface of new (2020) and old plates, which appear much rougher, was compared with the Keyence 3D profilometer VR-5200 [33]. The two plates were scanned and analyzed for their surface heights and profiles (Figure 4). The older plate shows significantly fewer and wider grooves with greater differences in height. Whereas the newer plates consist of many small furrows with less depth. It is believed that this contributes to the sensitivity especially for different particle sizes. Therefore, only the newer 2020 plates were used.

A small amount of sample about 20 to 30 mm^3 was taken and put on the porcelain plate underneath the porcelain peg. Due to the adherent nature of the sample, accurate dosing and placement was difficult. This was also due to the fact that fast handling was necessary to prevent the sample from drying out. Three different phenomena were observed which are shown in Figure 5. The sample can be offset from the center of the plate. When the peg is moved down on the sample it distributes differently. (1.) If the sample is in front of the peg, the PETN-water mass forms a slippery film where the peg might slide on. (2.) The sample is placed in the center and a mix of sliding and scratching of the porcelain surfaces is observed. (3.) The sample is placed behind the moving direction of the peg and mostly scratching is observed. It is believed that case (3.) shows the highest and case (1.) the lowest sensitivity. In this work mostly, methods (1.) and (2.) were used. For the determination specifically, the one out of six methods (1 of 6) was selected, as the lower detonation limit is determined. When it comes to safety assessment this value is more reasonable than an E50.

As the accuracy of those measurements is limited, each measurement was done triple and the standard deviations were calculated. The machines (Figure 2) and procedures are described in ST/SG/AC.10/11/Rev.6 (s. 13.4.2.3.3).[17]

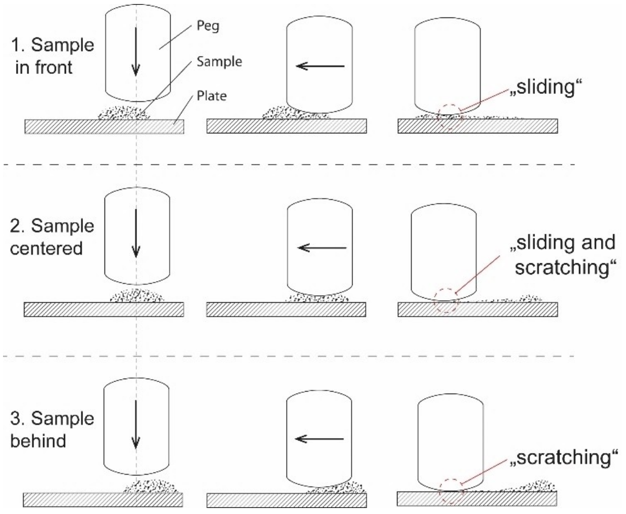

Figure 5. Differences in the way the sample is placed between the porcelan parts within the BAM friction tester.

3 Results and Discussion

The mechanical sensitivities of pure PETN were determined ranging from a dry sample to water contents up to 35 %. The measurements were repeated three times and the mean value with standard deviation is given. The red colored triangles indicate literature known measurements on BAM devices. The red line indicates the limiting load for safe transportation according to the United Nations handbook "Transport of Dangerous Goods".

3.1 Impact Sensitivity

The impact sensitivity of the dry sample is at 3.5 ± 0.5 J and therefore similar to the UN measurements (red). The explosion of the dry sample is sharp and clearly audible. Adding water of an amount of 2.5 % is not decreasing the sensitivity remarkably. An energy necessary for explosion of 4.3 ± 0.5 J is observed. At 5 % water, the sensitivity decreases slightly while the deviation increases to 7.5 ± 2.0 J. Wetting to 10 % causes a strong decrease in sensitivity with 16.7 ± 2.4 J. For 20 % and 25 % water content, no explosion is observed below 20 J with a mean sensitivity of 21.7 ± 2.4 J. With a water content of 35 %, a sensitivity of 25 J was measured in a single determination. In the regions of lower water contents, an exponential decrease of the sensitivity is observed. From 10 % and higher the sensitivity decreases only slightly with the addition of more water. The measurement results of the UN experts show slightly lower sensitivities.

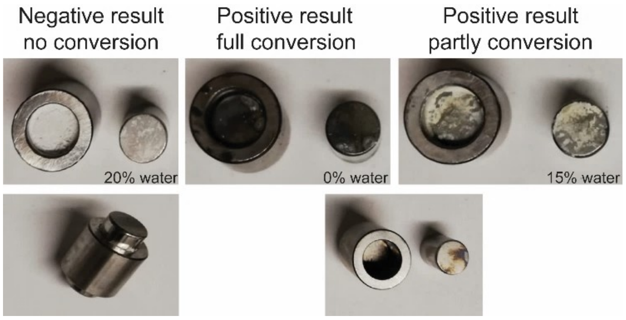

Figure 6. Fallhammer specimen holder after various tests. Negative tests remain closed (bottom left) positive tests are usually opened through the detonation (bottom right).

Dry samples or samples wetted up to 5 % mainly show complete conversion of the sample when a positive result is detected (Figure 6). The higher the water content, the lower the conversion. With the lower conversion, the noise of the positive result is generally less loud. It is believed, that the desensitizer, in this case water, inhibits propagation. Whereas it is not given that initiation is diminished by the addition of water. It cannot be excluded, that small initiation sites that are not propagating are formed. Furthermore, great care should be taken against drying out or settling of the water when working with wet PETN. In contrast to wax or plastic, water is only loosely attached to PETN and separates over time.

3.2 Friction Sensitivity

The dry sample shows a friction sensitivity of 50 ± 4 N with loud and partly propagating explosions [34]. This is comparable to the results by the UN experts. The water contents in Figure 7 are corrected to the actual water contents after measurement from Table 1. Wetting causes the sensi-

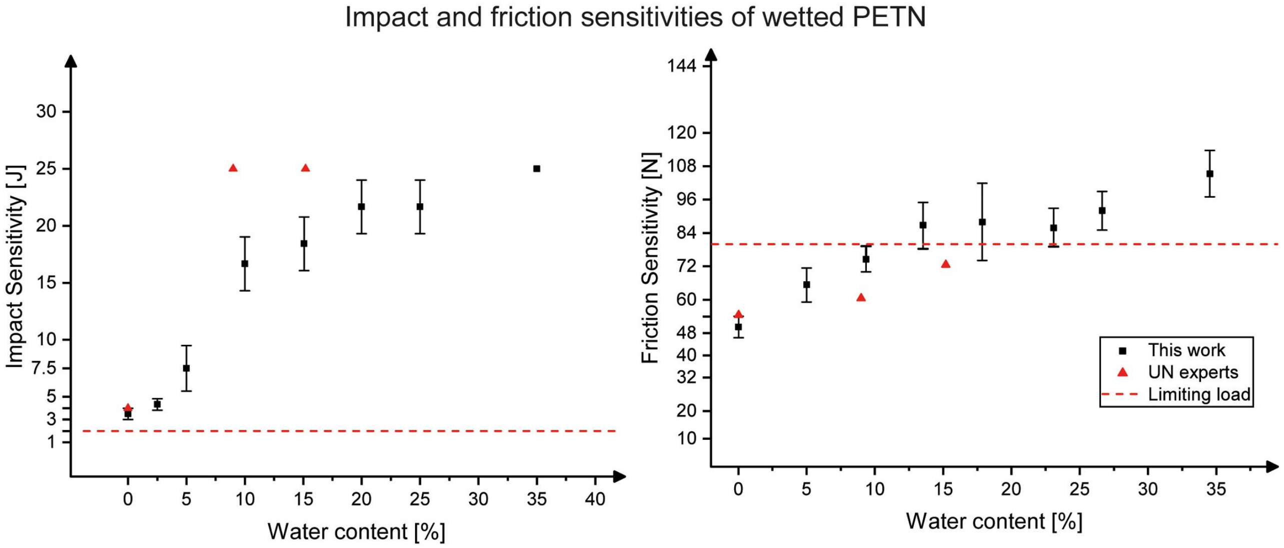

Figure 7. Mechanical sensitivities of wetted PETN ranging from 0 to 35 % water. Left: Impact sensitivity, Right: Friction sensitivity.

tivity to drop linear to 87 ± 8 N at 15 % water. The sensitivity keeps in this range until 25 % water. Up to 35 %, a decrease is observed with a sensitivity of 105 ± 8 N. The measurements by the UN experts are slightly more sensitive which can be attributed to different samples (Figure 7 red triangles).

The noise and propagation of the explosion decreases when water is added. This reduction in the intensity of a positive result is not captured by the measurement or evaluation. Often, very quiet crackling can be heard that is barely audible with hearing protection, which makes evaluation difficult. We also believe that mainly propagation is inhibited and initiation is less detected due to the decreasing noise of very small explosions.

4 Conclusion

Impact and friction sensitivity measurements are two well established methods for the determination of mechanical sensitivity. The literature was reviewed carefully showing that good accuracy can only be achieved through high standardization.

PETN with a mean particle size of 200 to 400 μm, crystallized in partly intergrown blocks, was used. The water loss during sensitivity determination was detected by Karl-Fischer titration and found to be in the lower percent area.

The dry sample shows a sensitivity of 3.5 ± 0.5 J and 50 ± 4 N. When wetted the mechanical sensitivity is generally decreased. The impact sensitivity shows an exponential decrease up to 10 % water to 7.5 ± 2.0 J. The friction sensitivity is decreasing linear up to 15 % and 87 ± 8 N. Between 15 and 25 % water content, the mechanical sensitivities stagnate. Up to 35 % water, a decrease is observed to 25 J and 105 ± 8 N. The authors believe, like stated before, that desensitization is caused by inhibition of propagation. The initiation can still occur in wet samples.

For the handling, it can be concluded that PETN below 10 % water is still sensitive and should be handled as the dry material. Above 15 % water, impact forces are of less concern but sensitivity towards friction and shear is still given. Smooth or soft surfaces (Teflon, plastic) and low shear stress are beneficial. Rough surfaces (metal, porcelain) and mixtures with hard particles (sand) should be avoided. Higher water contents (35 %) are reducing the sensitivity further but in every case, the material stays sensitive to moderately sensitive.

Wet samples always tend to separate and release water and get therefore dry and sensitive again. Wet PETN delivered in larger containers or bags shows a separation of water due to condensation or settling. The water content represents an average value and lower water contents can be expected at the top layer.

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[30] One measurement takes aprox. 20 min at room temperature.

[31] https://www.metrohm.com/en_us/products/2/8600010.html (accesed 04/**2022**).

[32] The decomposition products cause coating and corrosion on the device. Regular cleaning with a brush and cloth, as well as lubrication with graphite are necessary. Our experience shows that a dirty instrument determines a lower sensitivity (e.g. PETN 5 J).

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