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OZM Ball Drop Impact Tester (BIT-132) vs. BAM Standard Method – a Comparative Investigation

Michael S. Gruhne,[a] **Marcus Lommel,**[a] **Maximilian H. H. Wurzenberger,**[a] **Norbert Szimhardt,**[a]
Thomas M. Klapötke,*[a] **and Jörg Stierstorfer***[a]

Abstract: Safety, performance, cost efficient synthesis and toxicity are the most important aspects of modern explosives. Sensitivity measurements are performed in accordance with different protocols all around the world. Sometimes the BAM drop hammer does not accurately reflect the sensitivity of an energetic material, in particular the sensitivity of primary explosives. Therefore, we present here preliminary results obtained using the novel ball drop tester (BIT-132), manufactured by OZM research, following MIL-STD-1751 A (method 1016). The ball drop impact sensitivity tester is a device in which a free-falling steel ball is dropped onto an unconfined sample, and is expected to produce more realistic results than the currently commonly used BAM method. The results obtained using the probit analysis were compared to those from the BAM drop hammer and friction tester. The following sensitive explosives were investigated: HMTD, TATP, TAT, Tetrazene, MTX-1, KDNBF, KDNP, K_2DNABT, Lead Styphnate Monohydrate, DBX-1, Nickel(II) Hydrazine Nitrate, Silver Acetylide, AgN_3, $Pb(N_3)_2$ RD-1333, AgCNO, and $Hg(CNO)_2$.

Keywords: Impact Sensitivity · Ball Drop · Primary Explosives · Safety · Probit Method

1 Introduction

In the field of applicable energetic materials chemistry, the safe handling of explosives is as important as a tailored performance or an economic synthesis. Nowadays, theoretical calculations based on quantum chemical models or structural relationships are possible and allow an estimation of the sensitivity of an energetic material toward mechanical or electrostatic stimuli [1].

Nevertheless, the experimental determination of sensitivities cannot be replaced by calculations, since many reliable calculations focus on a limited set of CHNO-based compounds and are based on empirical data. Unfortunately, sensitivity measurements are performed and evaluated using different methods all over the world, making comparisons of the sensitivity values difficult. The method for impact sensitivity testing which is recommended by the UN is the BAM (Bundesanstalt für Materialforschung und -prüfung) drop hammer, and has therefore become the most frequently used standard measuring method [2]. Besides its major advantages, some particular aspects must be taken into account when using the BAM drop hammer. The substance is placed between two steel cylinders enclosed by a steel guide, and a weight is dropped onto the steel cylinders from variable heights which may lead to an ignition. This ignition scenario facilitates the interpretation of the testing outcome, but it doesn't correspond to realistic conditions as the setup could cause hot spots during impact, leading to ignition [3]. An ignition could also be induced by adiabatic compression of the air trapped between the two cylinders [4].

An alternative method which can be used is the ball drop impact tester (BIT) in accordance with MIL-STD-1751 A, method 1016 [5]. Similar ball drop impact testing devices are mostly used in the US. In this method, a free-falling steel ball is dropped onto an unconfined layer of substance. This testing scenario takes more realistic circumstances into account, e.g. the slight spin of the ball when hitting the sample [6]. This enables an alternative, more realistic result, to be obtained which allows a simpler and more reliable ignition mechanism and therefore safer handling of energetic materials. The lack of uniformity of results, especially regarding ball drop impact sensitivities, (e.g. energy, force, height, etc.) and different evaluation procedures (E_{50}, no-fire-level, 1-out-of-10, etc.) are problems that urgently needed to be addressed. Consequently, an intensive study ap-

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[a] *M. S. Gruhne, M. Lommel, M. H. H. Wurzenberger, N. Szimhardt, T. M. Klapötke, J. Stierstorfer*
Department of Chemistry
Ludwig-Maximilian University of Munich
Butenandtstr. 5–13, 8137 Munich (Germany)
**e-mail: tmk@cup.uni-muenchen.de*
jstch@cup.uni-muenchen.de
Homepage: http://www.hedm.cup.uni-muenchen.de

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plying the BIT method to the most common sensitive energetic materials under uniform testing conditions was long overdue [7,8,9]. In order to obtain the largest possible data set, the probit method is used as the evaluation method of choice [10].

In this work, a variety of different sensitive energetic materials were chosen in order to compare the ball drop impact sensitivities. The selected primary explosives included a range of typical, commercially used ones such as tetrazene (**4a**, **4b**), KDNBF (**6**), lead styphnate monohydrate (LS, **9a**, **9b**), nickel(II) hydrazine nitrate (NHN, **11**), AgN_3 (SA, **13a**, **13b**), $Pb(N_3)_2$ RD-1333 (LA, **14**), AgCNO (SF, **15**)), and mercury fulminate (MF, **16**). In addition, several homemade explosives such as HMTD (**1**), TATP (**2**) and silver acetylide (**12**) were chosen, as well as a series of potential green primary explosives (TAT (**3**), MTX-1 (**5**), KDNP (**7**), K_2DNABT (**8**), and DBX-1 (**10**)).

2 Experimental Section

CAUTION! *All of the compounds which were investigated are potentially explosive energetic materials, which show increased sensitivities toward various stimuli (e.g. elevated temperatures, impact, friction or electrostatic discharge). Therefore, proper safety precautions (safety glasses, face shield, earthed equipment and shoes, leather jacket, Kevlar gloves, Kevlar sleeves and ear plugs) must be worn while synthesizing and handling these compounds.*

Each compound that was tested was synthesized on a 1.5–3 g batch size, depending on the sensitivity of the compound and its bulk density. This should guarantee that all measurements could be carried out from the same batch. The crystal shapes and sizes were established using light microscopy, and the results - together with the particle size distributions - are shown in Figures S16–17 and Table S17.

2.1 Synthesis of the Explosives

Common primary explosives were synthesized according to standard literature procedures. The organic peroxides **1** and **2** were prepared by the reaction of hexamine (**1**) or acetone (**2**) with hydrogen peroxide under acid catalysis [11,12]. Compound **3** was precipitated from a mixture of acetone and water by the reaction of cyanuric chloride and sodium azide [13]. Tetrazene (**4b**) was synthesized by dissolving aminoguanidine bicarbonate in acetic acid and further reaction with sodium nitrite [14]. Compound **5** was obtained by treating tetrazene with sodium nitrite [15]. Compound **6** was obtained using a two-step synthesis, in which picryl chloride was reacted with sodium azide and potassium carbonate [16]. The reaction of 3-bromo-2,4,6-trinitroanisole with potassium azide and diethyl carbonate resulted in compound **7** [17].

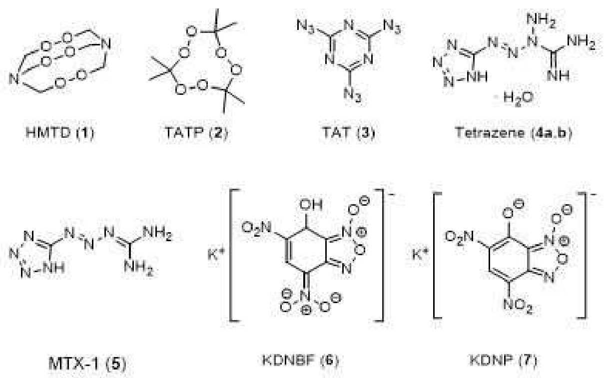

Chart 1. Prepared primary explosives **1–7**.

The 1,1′-Dinitramino-5,5′-bistetrazole salt **8** was prepared from hydrazine and diethyl carbonate according to WO 2018209366 A2 [18]. Dissolving an aqueous solution of styphnic acid in magnesium oxide and subsequent reaction with lead(II) nitrate yielded compound **9b** [19]. Compound **10** was obtained by conversion of sodium 5-nitrotetrazolate dihydrate into the copper(I) salt [20]. The nickel complex **11** was prepared by adding hydrazine hydrate to nickel nitrate [21]. Silver acetylide (**12**) was synthesized by passing acetylene through silver nitrate solution in aqueous ammonia [22]. Silver azide (**13b**) was synthesized by a simple metathesis reaction starting from sodium azide and silver nitrate [23]. The fulminate salts **15** and **16** were obtained by dissolving the respective metal in nitric acid and then pouring the reaction mixture onto ethanol [21,24].

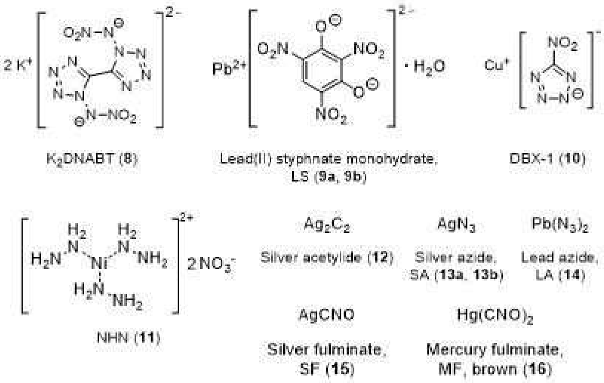

Chart 2. Synthesized primary explosives **8–16**.

Compounds **4a**, **9a**, **13a**, and **14** were provided by DynITEC GmbH Germany, whereas **4b**, **9b**, and **13b** were synthesized in the lab according to literature procedures.

2.2 Sensitivity Measurements

The ball drop experiments were carried out using the BIT-132 ball drop impact tester (OZM Research, Czech Republic) [25] following MIL-STD-1751 A (method 1016) [5]. A steel ball (0.50–2.00 inch, 8.35–534.70 g) is rolled of a steel guidance and dropped onto the explosive compound with a certain spin.

The sample layer was prepared as follows: The explosive compound was placed onto a steel target platform using a 30 mm^3 volumetric spoon. The sample was then spread out on the platform, resulting in a homogenous layer of 0.33 mm height (Figure 2). Steel balls with diameters of 0.50 inch (1.27 cm, 8.35 g) and 0.75 inch (1.91 cm, 28.20 g) were used. For every steel ball, a different steel ball guide was used to ensure a proper drop [26].

Figure 1. OZM BIT-132 (left side) and its release mechanism (right side).

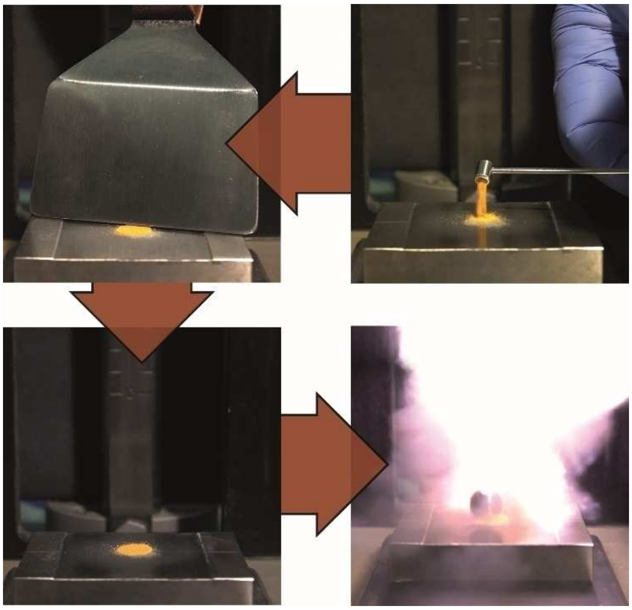

Figure 2. Stepwise sample preparation.

Any visual observation of decomposition was considered a positive result. After each trial, the remaining material was disposed of and the target block loaded with a freshly prepared layer. The steel balls were replaced for each substance. The impact energy was calculated from the product of the drop height, the mass of the steel ball used during the experiment and the gravitational constant. The initial drop height was chosen after several preliminary attempts, in which the height was determined at which the majority of the tests were positive. The probit analysis was used to evaluate the results [9]. The probability of ignition of each compound was determined using 6 test heights, with 10–15 trials at each height. The probabilities obtained were expressed as probits and the linear regression between probits and natural logarithms of the impact energy was performed. The sensitivity curve was obtained by backward transformation of the regression line into the probability-impact energy coordinates. Details of the 1 of 6 method that was used when a probit analysis wasn't possible, can be found in the general methods in the Supporting Information.

Additionally, the impact and friction sensitivities were determined according to the BAM (Bundesanstalt für Materialforschung) standard methods. The impact sensitivity tests were performed according to STANAG 4489 [27] with a modified instruction [28] using a BAM drop hammer [2]. Steel guide rings and steel cylinders for BAM drop hammers were obtained from OZM Research, Czech Republic [24]. The impact energy was calculated as explained above for the ball drop device. Friction sensitivity tests were performed according to STANAG 4487 [29] with a modified instruction [30] using a BAM friction tester [2]. Porcelain plates and pins were obtained from OZM Research, Czech Republic [24]. The friction force was calculated using the lever rule. The limiting values of the impact energy and friction force were determined in accordance with the method recommended by the UN for testing impact and friction sensitivities (1 of 6 method), according to ST/SG/AC.10/11/Rev.6 (s. 13.4.2.3.3) [31].

3 Results and Discussion

A key factor influencing the results was the type of steel ball used for each measurement. For compounds **4**, **5**, **11** and **16**, a smaller ball size at the same energy did not result in complete detonation or deflagration of the sample, and only a slight crackling was observed. This complicated the interpretation of a positive result by acoustic signals. It is assumed that the deflagration to detonation transition (DDT), shockwave sensitivity or critical diameter of each substance is an essential factor. This circumstance also influences any visual evaluation, since remaining substance does not necessarily indicate a negative test in every case.

The use of larger steel balls solves the problem, since the larger surface area of the bigger steel ball enables a larger amount of material to react, making evaluation clearer. However, larger steel balls tend to mask the sound of the detonating sample due to the louder impact noise. In addition, the use of the larger steel ball dusts the solid that would remain after a negative test, making visual evaluation nearly impossible.

Sensitivity measurements were carried out as described for each primary explosive. Standard deviations of the associated E_{50}, and $E_{16.6}$ values can be found in the supporting information (Table S1), together with plots of the results of each individual compound (Figure S1–15). In addition, detailed data on the results of each substance can be found in the Tables S2–S16.

In addition to primary explosives, some high explosives were also investigated. An evaluation with the probit method was not possible for any of these cases. In addition to the problems mentioned above with respect to the ignition of substances, a strong grain size dependence was observed. PETN, RDX and TKX-50 could be ignited only using the smallest grain sizes which were obtained after flash crystallization or sieving. Larger grain sizes showed lower impact sensitivities. In the case of FOX-7, no ignition was observed at all. Due to this behavior, we decided to investigate these substances in more detail in future work.

During the characterization of each metal azide (**13**, **14**), variations in the sensitivity were observed. A proper probit analysis leading to reliable sensitivity data was not possible. Extending the test setup, for example the number of test heights and the number of tests per selected height, is assumed to correct statistical variations in compounds **13** and **14**.

In the case of silver fulminate (**15**), no probit evaluation was possible because a no-fire-level could not be determined. The Ball Drop Impact Sensitivity (BDIS) of these compounds was determined using the 1 of 6 method.

The results of the sensitivity measurements are shown in Table 1 together with the sensitivity data determined using BAM standard methods. In order to compare the substances examined with probit analysis to those compounds for which it was not possible, the 16.6 % ignition probability values of these substances were calculated. This corresponds approximately to the probability level represented by the 1 of 6 method.

The probit method was successfully used for the evaluation of the BDIS of compounds **1**–**12** and **16**. The respective E_{50}, and $E_{16.6}$ values together with the sensitivity values according to BAM, as well as the particle size distributions are compiled in Table 1.

The diagrams shown in Figures 3 and 5 and 6 are combined according to their respective slopes. Figures 3 and 4 show the curves with the highest gradients. Compound **4 b** was placed in Figure 4 to give a better comparison of the influence of the manufacturing process and the particle size of tetrazene. The improvised explosive HMTD (**1**) is the most sensitive compound investigated by the probit method ($E_{16.6} = 4$ mJ). It is assumed that silver fulminate is even more sensitive, since a probit-based analysis was not possible. The $E_{16.6}$ level is even closer to the lowest measurement limit (1 of 6 = ≤ 4 mJ).

Table 1. Sensitivity data of the compounds **1**–**16**.

Compound		Ball drop impact sensitivity (mJ)		BAM friction sensitivity 1 of 6 (N)	BAM impact sensitivity 1 of 6 (J)	Particle size dist. (μm)
		E_{50}	$E_{16.6}$			
HMTD	(**1**)	6	4	0.3	1.5	50–200
TATP	(**2**)	18	13	0.4	≤ 1	< 30
TAT	(**3**)	27	21	0.3	≤ 1	50–300
Tetrazene STANAG 4170	(**4 a**)	10	5	2.5 ($> 7^{c}$)	1.5 ($> 1^{c}$)	< 30
Tetrazene	(**4 b**)	33	21	2.5	1.5	400–1000
MTX-1	(**5**)	14	10	2	2	< 30
KDNBF	(**6**)	19	13	2	1.5	< 30
KDNP	(**7**)	56	40	12	3	< 30
K_2DNABT	(**8**)	31	25	≤ 0.1	≤ 1	500–1500
LS STANAG 4170	(**9 a**)	28	19	1 ($> 0.5^{c}$)	7 (1.75^{c})	40–140
LS	(**9 b**)	22	15	0.45	8	< 30
DBX-1	(**10**)	39	21	≤ 0.1	≤ 1	< 30
NHN	(**11**)	175	134	15	20	< 30
Silver acetylide	(**12**)	29	14	≤ 0.1	≤ 1	50–150
AgN_3 STANAG 4170	(**13 a**)	n.d.^{a}	29^{b}	≤ 0.1 ($\leq 0.1^{c}$)	≤ 1 ($> 2.25^{c}$)	< 10
AgN_3	(**13 b**)	n.d.^{a}	29^{b}	≤ 0.1	3	< 30
$Pb(N_3)_2$ RD-1333	(**14**)	n.d.^{a}	37^{b}	≤ 0.1 ($\leq 0.1^{c}$)	4 ($> 1.75^{c}$)	< 30–50
AgCNO	(**15**)	n.d.^{a}	≤ 4	$\leq 0.1^{c}$	5	100–200
$Hg(CNO)_2$, brown	(**16**)	21	16	2.5	2.5	50–300

[a] No probit analysis possible. [b] Determined by 1 of 6 method. [c] Sensitivity data according to supplier.

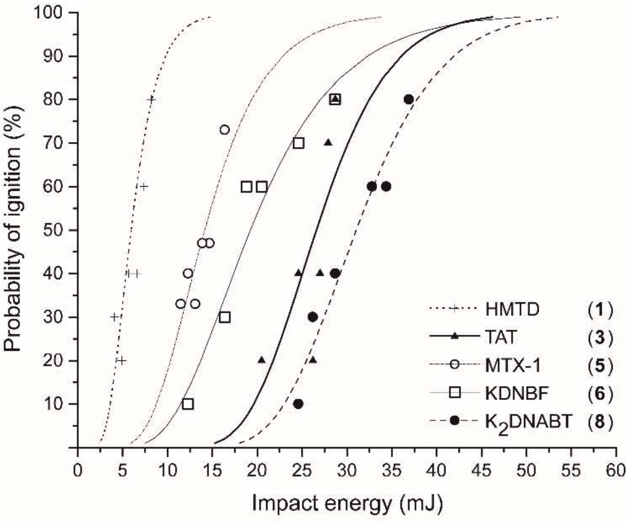

Figure 3. Probit curves of compounds **1**, **3**, **5**, **6**, and **8**.

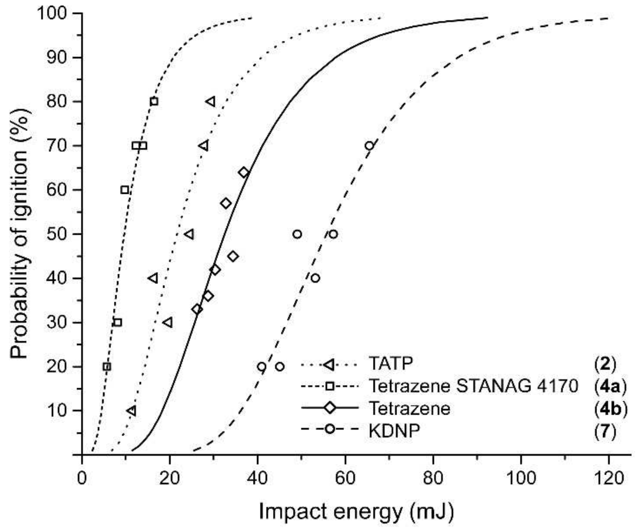

Figure 4. Probit curves of compounds **2**, **4 a**, **4 b**, and **7**.

However, this observation is only partly consistent with the sensitivity data according to BAM, as silver fulminate (**15**) has an unexpectedly high tolerance to impact (FS: 0.3 N (**1**), $\leq$0.1 N (**15**); IS: 1.5 J (**1**), 5 J (**15**)). Among the curves shown in Figure 3, K_2DNABT (**8**) has the highest $E_{16.6}$ value (25 mJ). This is in strong contrast to the extreme sensitivity data determined with the BAM standard methods ($<$ 0.1 N, $<$ 1 J). The same applies to TAT (**3**), which has a similar $E_{16.6}$ value (21 mJ) at very low friction and impact sensitivities according to BAM (FS: 0.3 N, IS: $\leq$ 1 J).

In case of tetrazene (**4**), larger crystals show the same sensitivity toward friction (**4 a**: FS: 2.5 N vs. **4 b**: FS: 2.5 N), while in the case of lead styphnate monohydrate (**9**) the trend indicated by the ball drop impact tester was confirmed for the friction sensitivity (**9 a**: FS: 1 N, $E_{16.6}$: 19 mJ vs. **9 b**: FS: 0.45 N, $E_{16.6}$: 15 mJ). Identical sensitivity data were observed for both types of silver azide (**13 a**, **13 b**). This observation fits with expectations since both compounds have approximately the same particle size, whereby the commercial product **13 a** consists of agglomerates of smaller particles (Figure 7).

With regard to the measured data (Table 1), however, these grain sizes do not seem to have any influence on the sensitivity toward mechanical manipulation. Nickel(II) hydrazine nitrate (**11**) turned out to be the most insensitive compound according to BAM (FS: 15 N, IS: 20 J). The data determined using the ball drop impact tester in this case agrees with the $E_{16.6}$ of 134 mJ.

Regarding lead styphnate monohydrate (**9**) and the already mentioned silver fulminate (**15**), a significantly higher

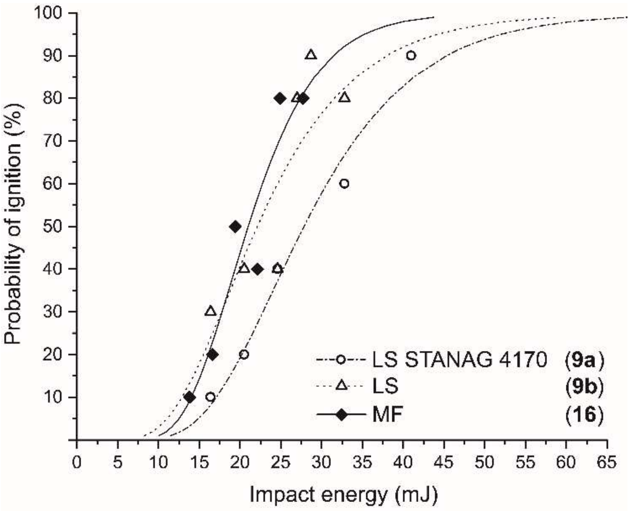

Figure 5. Probit curves of compounds **9 a**, **9 b**, and **16**.

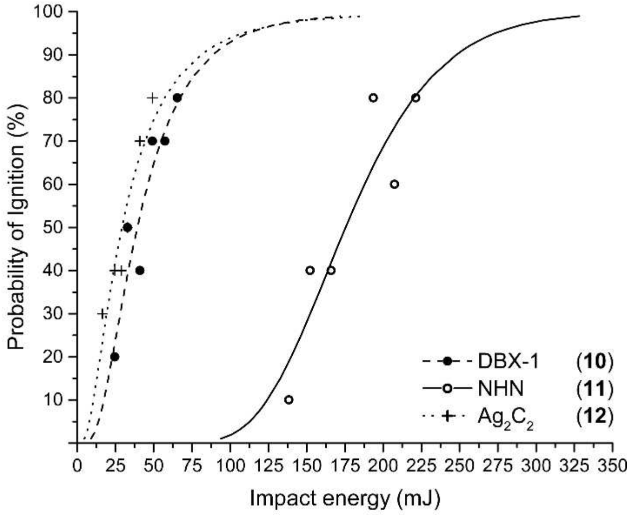

Figure 6. Probit curve determined for compounds **10**–**12**.

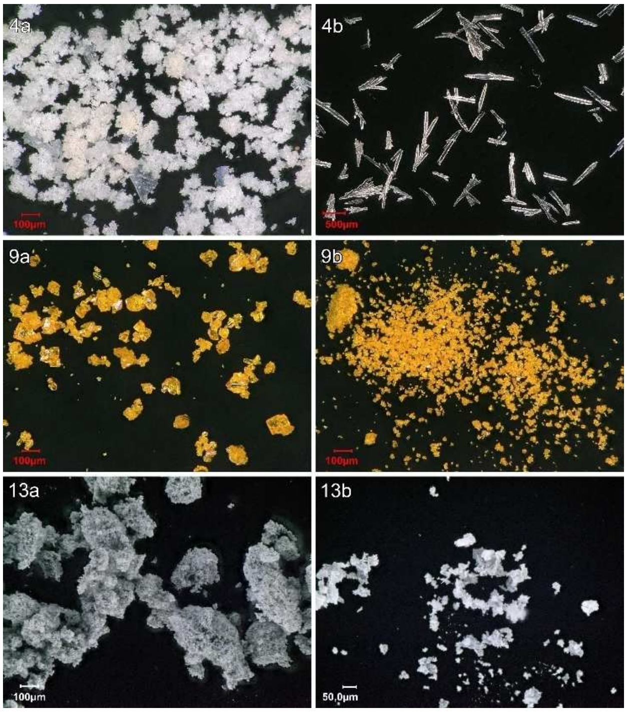

Figure 7. Different crystal shapes and particle size distributions of the compounds Tetrazene (**4**), Lead-styphnate Monohydrate (**9**), and Silver Azide (**13**).

discrepancy between the ball drop impact sensitivities and impact sensitivities according to BAM was observed (**9a**: $E_{16.6}$: 19 mJ, IS: 7 J; **9b**: $E_{16.6}$: 15 mJ, IS: 8 J). It is assumed that grain size effects play a major role here. In addition, a comparison with literature values again reveals the problem of comparing sensitivity values. According to *Köhler*, lead styphnate monohydrate possesses an impact sensitivity of 2.5–5 J [32,33].

The United States Army Material Command reported an impact sensitivity of 3.4 J for the Picatinny Arsenal apparatus, as well as 0.17 J determined using the Bureau of Mines apparatus. [34] In both cases, no conclusions can be drawn about the measured particle size distribution, which is known to have a large influence on the results. Regarding the data by *Köhler*, no reference to the measuring instrument is given. This lack of a uniform specification of the measuring methodology also leaves open whether the results presented are E_{50}, no-fire or BAM 1 of 6 values.

A general relationship between both of the impact sensitivity testing methods was not found. It was generally observed that low BDIS values are accompanied by low friction sensitivity data. (Figure 8) The only significant exception was KDNP (**7**). At low BDIS values, KDNP (**7**) shows a lower sensitivity to friction, which is comparable to that of NHN (**11**) (**7**: $E_{16.6}$: 40 mJ, FS: 12 N; **11**: $E_{16.6}$: 134 mJ, FS: 15 N).

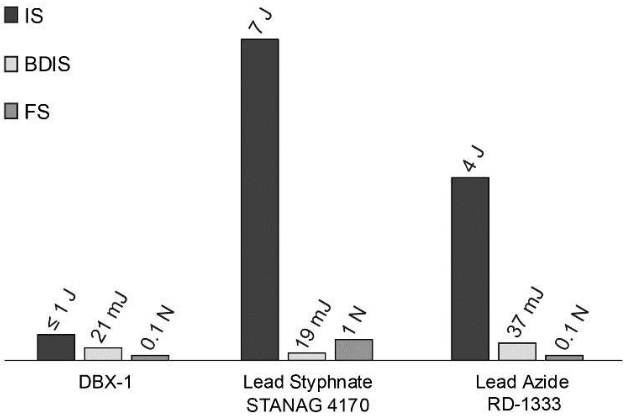

Figure 8. Comparison of the sensitivity data of compounds **9a** and **14**.

4 Conclusion

The BIT-132 Ball drop impact tester from OZM Research was used to evaluate the sensitivity of well-known, sensitive energetic materials. In addition, the BAM standard methods were used to determine sensitivities for all compounds. The sensitivity data obtained using the BIT-132 apparatus was evaluated using a probit analysis. In cases where this was not possible, the BAM 1 of 6 method was applied.

As shown by the examples of lead styphnate and lead azide, it is impossible to correlate the most recent BIT results with the data obtained using the BAM drop hammer (1 of 6). This is a result of the different test set-ups and therefore different ignition types. A closer correlation between the sensitivity to friction and ball drop impact sensitivity was observed. It is assumed that a combination of friction and impact is exerted on the substance by the spin of a falling steel ball.

Furthermore, it was found that there is a strong coherence between the particle size and the sensitivity towards BDIS of the compounds. Smaller particle sizes clearly showed more sensitivity, which is in strong contradiction to earlier assumptions that larger crystal sizes lead to drastically higher sensitivities. For the well-known explosives PETN, RDX, FOX-7 and TKX-50 o ignitions could be observed at certain grain sizes. So the current BIT has limited suitability for characterization of secondary explosives.

The authors would suggest the use of balls with a constant diameter and different densities. The 0.75 inch or the 1.00 inch ball, which were used in the set-up described in this work, are preferred for evaluating primary explosives.

Since most of the above problems do not apply to the testing of primary explosives, the device is perfectly suitable for testing primary explosives.

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