The new promising test procedure suitable for the energetic materials sensitivity testing

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Abstract

Paper describes comparison of the two well-known and well established statistical testing procedures with the newly developed testing procedure. The standardized BAM friction sensitivity test is used for the comparison of the sensitivity data. The first used method is Bruceton staircase, also known as "UP and DOWN". The result of the Bruceton staircase method is value of the F_{50} point. The second used method is Probit analysis. The result of the Probit analysis is the complete sensitivity curve. The newly developed method is a modified Neyer's sensitivity D-optimal test method. The parameters of the sensitivity curve are continuously estimated using maximum likelihood algorithm. The newly tested algorithm differs from the Neyer´s method by altering the way next level suggestion based on the all previous stimulus levels. The friction sensitivity test was carried out with the selected primary explosive and one pyrotechnic mixture. Comparison of the materials sensitivity obtained by the mentioned testing procedures is provided. It was found the main advantage of the new method is time and cost effectivity. Moreover, it requires less sample counts while providing a maximum statistical data in the form of complete sensitivity curve. Another advantage of the new method is its independence on the beforehand knowledge of the real value of stimulus level, thus significantly reduce incorrect estimate of the initial test conditions.

Keyword: DDNP; Bruceton staircase; Probit analysis; statistical methods; sensitivity to friction

1 Introduction

The sensitivity of explosives is one of their crucial parameters limiting their usage. Various tests were developed to test the sensitivity to a variety of initiating events (impact, friction, electric spark, bullet impact, etc.). Many of these tests have one thing in common and that is the characteristic sigmoidal curve of sensitivity of explosive vs. energy of external stimulus (figure 1). This curve represents a well-known fact that a single sharp boundary between energy levels causing initiation ('go' level) and not causing initiation ('no go' level) does not exist. Rather there are three intervals: first includes low values of stimulus energy (the sample never reacts), opposite is the region of very high values of stimulus energy (the sample always reacts). The third, most important, interval lies between these two extremes and the energy of the impulse causes sample reaction with certain probability. Generally, the low values interval is connected with safety and the high values interval is associated with the reliability.

The sensitivity curve is mathematically represented as the cumulative distribution function, normal and lognormal probabilities are the most used ones.

Historically various methods were developed to determine the whole sensitivity curve or some important points (e.g. 50% or 10% probability level) [1-10]. Regardless on the technical issues of the particular test, these procedures have to handle the random nature of the tested energetic materials response and the fact once the sample is tested, it is affected by testing or destroyed thus can't be tested again. This behavior requires utilizing so called "single shoot" test procedures and the associated necessary statistical analysis of experimental data in order to get information about the sensitivity to the tested stimuli.

One of the most widely spread methods is the up-and-down method (also known as staircase, Bruceton staircase) used for determination of the 50% probability level (the level of stimulus where the sample will react with the 50% probability). The single value is usually reported (50% probability level) as a result, but together with the standard deviation, it can be used to calculate the whole sensitivity curve [8].

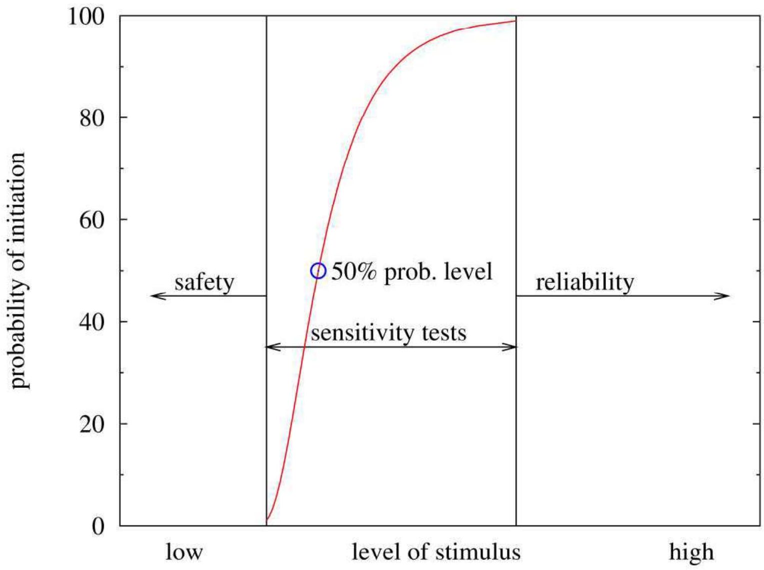

Figure 1. The sensitivity curve.

The Probit method can be used to reconstruct the sensitivity curve from test data. The curve is of lognormal shape and it is estimated from the linear dependence of a probability of ignition and the logarithm of a corresponding stimulus level [7]. Alternatively, the parameters of a lognormal probability distribution can be obtained by the Maximum Likelihood Estimation [9]. The Probit method is reliable, but quite laborious and time and cost inefficient.

The high efficiency is promised by Neyer D-Optimal experiment design [10], but this method is not widespread. The parameters of a probability distribution are estimated by Maximum Likelihood and the level of next shot is proposed to gain maximum information about the distribution.

In this paper, we would like to introduce the new algorithm, or a new experimental design, called FEST (Fast and Efficient Sensitivity Testing). The parameters of lognormal probability distribution are also estimated by Maximum Likelihood, but simpler algorithm for proposing of a next shot level is used in comparison with Neyer.

We chose tho samples and the sensitivity to friction to demonstrate using of FEST and to compare the results to Bruceton staircase and a Probit method.

2 Experimental section

The sensitivity to friction was determined for two samples (one pyrotechnic mixture and one primary explosive) using BAM friction apparatus.

2.1 Materials

The pyrotechnic mixture KFCN III [11] contained 50% w. of potassium ferricyanide and 50% w. of potassium perchlorate. Both components of size d_{90}= 25 µm were mixed in 3D dry mixing machine Turbula (50 rpm, 10 min.).

The second measured sample was DDNP (6-Diazo-2,4-dinitrocyclohexa-2,4-dien-1-one) prepared according to [12].

2.2 Apparatus

The friction sensitivity was measured using BAM friction apparatus FSKM-10 utilizing porcelain pegs (Ø10x15 mm) and porcelain plates (25x25x5 mm), manufactured by OZM Research, Czech Republic. The measuring equipment and test setup was in compliance with the standard [2].

2.3 Friction sensitivity measurement

The friction sensitivity was determined with the three methods. Bruceton staircase was used as a well-known method, also prescribed in the Czech Defense Standard [5]. Thirty shots were performed with the standard step size. The sensitivity curve was then calculated as a normal probability cumulative distribution function with the parameters F_{50} and the standard deviation.

Probit analysis was used to calculate the sensitivity curve as precisely as possible. Therefore 30 shots were used at each of the five levels of friction force, 150 shots for the sample in total. The parameters of a lognormal probability distribution were than estimated by maximum likelihood from the whole dataset. The 95% confidence bands for the curve were calculated according to [13].

Third and last method was FEST. Using this algorithm, the sensitivity curve (lognormal) is estimated after every shot and it is possible to see, how it is upgraded and becoming more precise. The determination was ended after 50 shots.

3 Results and discussion

The data from Bruceton staircase are in tables 1 (KFCN III) and 2 (DDNP). The calculated values of F_{50} and S are also stated. The ratio S/D is inside the interval (0.5; 2) and the determination is therefore valid.

Table 1. Record of measurement by Bruceton staircase for KFCN III.

F[N]	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20
4									-		-									
5						-		+		+		-								-
6	-		-		+		+						-						+	
7		+		+										-		-		+		
8															+		+			

F[N]	21	22	23	24	25	26	27	28	29	30
4					-				-	
5		-		+		-		+		+
6	+		+				+			
7										
8										

F_{50} = 5.71 N
S = 1.71 N
S/D = 1.71

The measured probabilities of initiation used for the Probit analysis are summarized in tables 3 and 4. The parameters of sensitivity curves (lognormal probability distribution) were calculated using maximum likelihood estimate. The resulting sensitivity curves are, together with their 95% confidence limits, plotted in the final graphs 3 and 4.

The progresses of friction sensitivity measurements using FEST algorithm are plotted in figure 2 (KFNC III on the left and DDNP on the right). Black circles correspond to failures and red crosses correspond to initiations. The parameters of sensitivity curves (lognormal probability distribution) were calculated using maximum likelihood after each shot. The resulting sensitivity curves after 20, 30, 40 and 50 shots are plotted in the final graphs 3 and 4.

Table 2. Record of measurement by Bruceton staircase for DDNP.

F[N]	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20
7					-														-	
14				+		-				-				-				+		-
21	-		+				-		+		-		+		-		+			
28		+						+				+				+				
35																				

F[N]	21	22	23	24	25	26	27	28	29	30
7										
14										-
21	-								+	
28		-		-		-		+		
35			+		+		+			

F_{50} = 21.7 N
S = 10.6 N
S/D = 1.51

Table 3. Probabilities of initiation for KFCN III (Probit analysis).

F [N]	P [%]
3	26.67
4	30.00
6	50.00
8	56.67
9	83.33

Table 4. Probabilities of initiation for DDNP (Probit analysis).

F [N]	P [%]
7	26.67
11	26.67
14	46.67
18	60.00
25	86.67

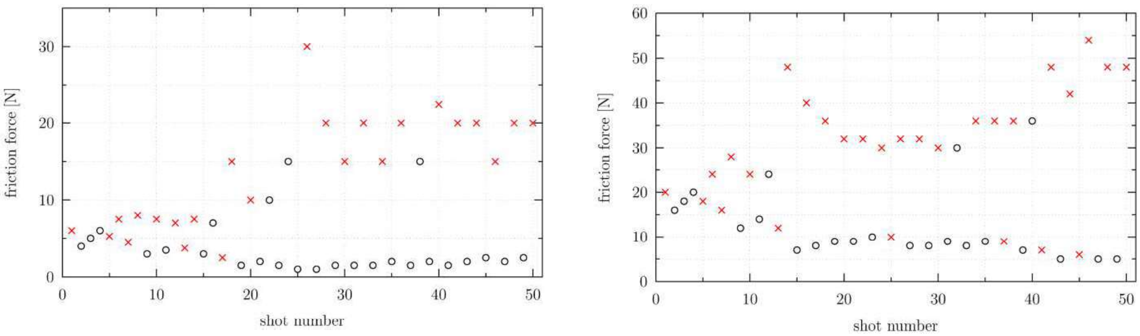

Figure 2. Sensitivity measurement progress of KFCN III (left) and DDNP (right) using FEST. Black circles correspond to failures and red crosses correspond to initiations.

The comparison of all used methods for the sensitivity determination can be seen in figures 3 and 4, for KFCN III and DDNP, respectively. The curves from Bruceton staircase method have a different shape in both cases. Bruceton staircase method was performed with standard steps; therefore the resulting curves correspond to normal probability distribution. Probit analysis and FEST result in lognormal probability distribution. According to our opinion, lognormal distribution corresponds more to the reality. Bruceton staircase is a quick method, how to find the F_{50} value, but the tails of the distribution are not very precise. Another cost, for the speed, is occasional inaccuracy of determined F_{50} value (as can be seen in the DDNP case).

The sensitivity curve from the Probit analysis can be considered as close to the true curve, as it results from 150 shots per sample. Thereafter we can conclude that in the 95% confidence bands (light grey areas) we have the data representing true material sensitivity.

The new methodology presented here, FEST, looks promising. With about 30 shots it is possible to estimate the F_{50} value which is within the confidence region. Moreover, the determination is always valid. In case of Bruceton staircase there is a possibility of the initial incorrect selection of the step size – when the *S/D* lies outside the interval (0.5; 2) than the whole trial have to be repeated with the corrected step, which is another drawback of the staircase method.

The tails of the FEST sensitivity curve falls into the 95% confidence limits of Probit analysis curve within the 40 or 50 shots. That is fairly less than number of shots for precise Probit analysis (150 shots was used in this work). Of course, more shots means more precision, this is without any doubt. However lowering the laboriousness (thus overall test costs) is also attractive. And we believe, that FEST algorithm is a step in the right way.

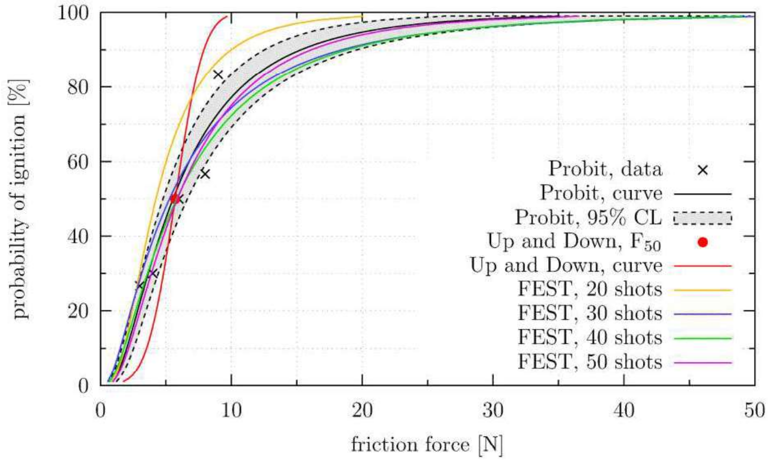

Figure 3. Comparison of friction sensitivity curves for KFCN III, obtained by Bruceton staircase, Probit analysis and FEST.

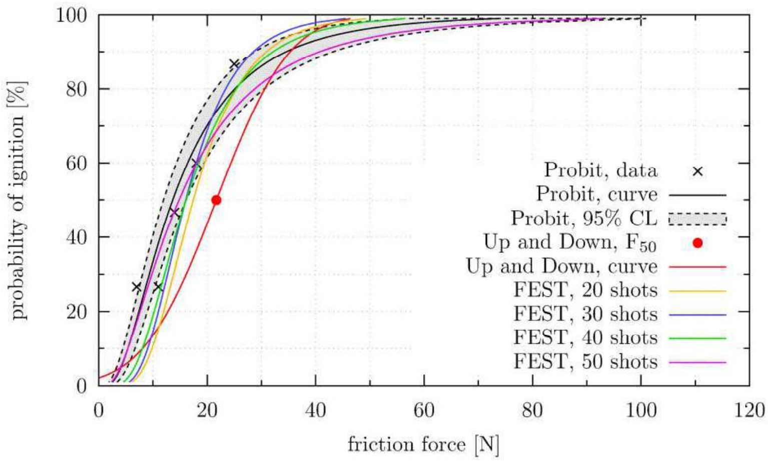

Figure 4. Comparison of friction sensitivity curves for DDNP, obtained by Bruceton staircase, Probit analysis and FEST.

4 Conclusion

A new algorithm for the sensitivity testing, FEST, was introduced. Using the determination of sensitivity to friction, the FEST results were compared to standard methods – Bruceton staircase and the precise Probit analysis. Our preliminary results show better FEST algoritm performance than both these methods. If a goal is determination of 50% level, the similar performance of FEST and Bruceton staircase was observed with the similar number of shots (30). In case of FEST, there is no option to choose the bad step size – the determination is always valid. If a goal is to calculate the whole sensitivity curve, within 50 shots can be reached when using FEST method. The using of Probit analysis requires large series of shots in order to get relevant data. At the end, the FEST algorithm is fast and effective method of statistical evaluation of the "single shoots" samples, which bring a considerable time and cost savings while keeping the without compromising result credibility.

Acknowledgments

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References

[1] Recommendations on the transportation of dangerous goods, manual of tests and criteria, 3rd edition, United Nations, New York and Geneva, 1999.
[2] STANAG 4487 Explosives, Friction sensitivity tests, 2002.
[3] STANAG 4489 Explosives, Impact sensitivity tests, 1999.
[4] STANAG 4490 Explosives, Electrostatic discharge sensitivity tests, 2001.
[5] COS 137601 Organizace a metody schvalování způsobilosti výbušnin pro vojenské účely, 4th ed., Praha, 2018 (in Czech).
[6] MIL STD 650 Explosive: Sampling, inspection and testing, 1962.
[7] Selesovsky J., Pachman J., "Probit analysis – a promising tool for evaluation of explosive´s sensitivity", *Central European Journal of Energetic Materials*; 7(3), 2010.
[8] Dixon W. J., Mood A. M., "A method for obtaining and analyzing sensitivity data", *Journal of the American Statistical Association*, 43(241), 1971.
[9] Finney D. J., Probit analysis, Cambridge University Press, 3rd edition, Cambridge, UK, 1971.
[10] Neyer B.T., "A D-optimality-based sensitivity test", *Technometrics*, 36(1), 1994.
[11] Schuster J., Study of the pyrotechnic compositions containing unconventional fuels, MSc. thesis, University of Pardubice, 2018.
[12] F.M. Garfield, H.W.U. Dreher, Manufacture of diazodinitrophenol, US Patent 2,408,059 (1946)
[13] Cheng R.C.H., Iles T., "Confidence bands for cumulative distribution functions of continuous random variables", *Technometrics*, 25(1), 1983.