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AN EXPERIMENTAL COMPARISON OF THREE DOCUMENTED TEST METHODS
FOR THE EVALUATION OF FRICTION SENSITIVENESS

by

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ABSTRACT

Several different test methods can be used to determine the sensitiveness of an explosive or energetic material to initiation by friction. In this paper we examine the results for seven explosives obtained using three commonly used friction test methods. The suitability of each method as a means of quantifying friction sensitiveness is discussed, and the advantages and disadvantages of the individual techniques are highlighted.

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INTRODUCTION

Reviews of accidents in the USA involving high explosives, propellants^{1,2}, and pyrotechnics³ have indicated that friction is the major cause of ignitions. Explosives accident records held by the UK Health and Safety Executive (HSE) were recently analysed⁴ and similar conclusions drawn: 59% of accidents could be ascribed to friction effects, while events initiated by impact accounted for only 9%.

Measurement of the friction sensitiveness of an explosive or energetic material (ie. the ease with which it may be ignited or initiated by a friction stimulus) therefore forms an important part of any hazard evaluation process. In the UN scheme for the Transport of Dangerous Goods⁵, for example, an assessment of mechanical sensitiveness is required in Test Series 3.

As part of its remit to provide a technical base for the development of HSE policy and guidance on explosives, HSE's Commercial Explosives Section recently undertook a programme of work to assess three of the commonly used friction test methods. Two of the selected tests are leading UN Methods (with the recent addition of a new Russian method, UN Test Series 3 currently has four tests for friction). The third test is widely used in the UK and included in the Ministry of Defence Sensitiveness Collaboration Committee Manual⁶.

Consideration is currently being given to revision of the UN Tests and Criteria Manual⁵ and proposals have been made to select

single test methods for particular properties. The data gathered in our studies serve to highlight the advantages and disadvantages of two of the current UN methods for evaluating friction sensitiveness and should provide useful input to forthcoming discussions on changes to the UN scheme.

EXPERIMENTAL

Friction Test Methods

Rotary Friction Test

This test method is described in detail in both the SCC and UN Manuals^{6,5}. Briefly, the test comprises a steel wheel in contact (under pressure at 275 KPa) with a steel anvil. A 15 μ l portion of the sample is placed between the two surfaces and the wheel is then rotated $1/6$ of a turn (60°) at a given speed. The speeds at which the wheel is spun are defined on a logarithmic scale through 100 rpm with a step increment of 0.1. If an ignition (defined as an audible event, generation of sparks, or evidence of combustion) is observed the next experiment is carried out at one speed level down from the previous one. Similarly, if a non-ignition is observed the next experiment is carried out at one speed level up.

Fifty of these experiments are performed to complete a test, stepping up and down using the Bruceton⁷ procedure. The results are used to determine the speed at which there is a 50% chance of a positive event: this speed is called the 50% or Median Speed and is quoted as the test result.

A Figure of Friction for a sample can be determined by comparing the median speed for the test material to the median speed obtained when testing RDX, which is used as a standard for this test and assigned a Figure of Friction of 3.0. The Figure of Friction of the sample is then given by

$$3.0 \times \text{median speed sample} / \text{median speed RDX standard}$$

BAM Friction Test

This test is also known as the Koenen Friction Test and a full description is given in the UN Manual⁵. The test consists of a porcelain peg in contact (under load) with a porcelain plate. A 10 μ l portion of the sample is placed between the peg and the plate and the apparatus is configured so that the plate is moved back and forth once a distance of 10mm.

Different loads in the range 5-360N can be used on the peg. The first experiment is carried out at 360N, and if a non-ignition is observed a further five experiments are done using the same load. If, after six experiments, an ignition has not been observed, the sample is said to have a limiting load of >360N. However, if an ignition is observed during the six experiments testing is stopped at this load and continued at the next load down. This process is repeated until six non-ignitions are observed using a particular load.

The test result is the lowest load at which a positive event is observed, and is quoted as the Limiting Load.

Mallet Friction Test

A full description of this test, which uses a range of mallet and anvil combinations, can be found in the SCC manual⁶.

The anvils used are made from steel, softwood, hardwood and yorkstone, and the range of mallets comprises a steel tipped mallet, a nylon tipped mallet and a wooden mallet.

Tests are carried out using the following combinations: steel mallet on steel anvil, nylon mallet on steel anvil, wooden mallet on hardwood anvil, wooden mallet on softwood anvil and wooden mallet on yorkstone anvil. For each experiment, approximately 100 μ l of the sample is spread out on the anvil and then struck a glancing blow with the mallet. The sample is struck five times for each experiment unless an ignition is observed. If no ignitions occur for all five blows then the result is noted as negative. If an ignition is observed then no further blows are performed for that particular experiment and the result is noted as positive.

Ten of these experiments are carried out for each mallet/anvil combination except for steel on steel where the test method stipulates that twenty experiments are carried out.

The overall test results are quoted in the following ways:

1. If none out of ten (or none out of twenty for steel on steel) ignitions are observed then the percentage ignitions for that particular mallet/anvil combination is quoted as 0%.
2. If six or less ignitions out of ten are observed (or twelve out of twenty) then the percentage ignitions is quoted as 50%.

3. If more than six out of ten (or twelve out of twenty) ignitions are observed then the percentage ignitions is quoted as 100%.

Test Materials

Seven explosives were selected, covering a range of sensitivenesses to friction, in order to evaluate the discriminatory aspects of the three test methods.

The materials used in the comparison studies are listed in Table 1. Note that UK RDX made by the Woolwich Process, rather than RDX made by the Bachmann Process, was used (see footnote to Table 1).

Test Procedure

Each material was tested three times, apart from RDX/TNT which was only tested once by the Mallet Friction Test because of a limited sample availability.

The prescribed test procedures were adhered to for all the tests.

RESULTS AND DISCUSSION

Table 2 summarises the results obtained using the Rotary Friction, Mallet Friction and BAM Friction Tests.

Using this information it is possible to rank the seven materials tested in the following orders of sensitiveness to friction stimuli:

Rotary Friction Test:

LDNR > PETN > HMX > RDX > Tetryl > RDX/TNT = TNT⁸

Mallet Friction Test:

LDNR > PETN = HMX = RDX > Tetryl = RDX/TNT = TNT

TABLE 1

Explosives used in Comparison Studies

Explosive	Chemical Name
LDNR	Lead Dinitro Resorcinate
RDX*	Cyclo-1,3,5-trimethylene-2,4,6-trinitramine
RDX/TNT†	Type A: 60/40 mix of cyclo-1,3,5-trimethylene-2,4,6-trinitramine and trinitrotoluene with 1% beeswax as additive
PETN	Pentaerythritol tetranitrate
HMX	Cyclo-1,3,5,7-tetramethylene-2,4,6,8-tetranitramine
Tetryl	Trinitro-2,4,6-phenylmethylnitramine
TNT	Trinitrotoluene

* UK RDX is made by the Woolwich Process and is different from US RDX made by the Bachmann Process. Whereas the former has minimal HMX (<0.2%), US RDX can contain up to 12% HMX which will result in the material being more friction sensitive.

† Because of the differences in US and UK RDX outlined at *, samples of RDX/TNT formulated in these countries will differ.

TABLE 2
Friction Sensitiveness Test Results

SAMPLE	ROTARY FRICTION TEST		BAM FRICTION TEST		MEAN LIMITING LOAD (N)		MALLET FRICTION TEST					
	FIGURE OF FRICTION (FOF)	MEAN FOF	LIMITING LOAD (N)	MEAN LIMITING LOAD (N)	STEEL ON STEEL	NYLON ON STEEL	WOOD ON SOFTWOOD	WOOD ON HARDWOOD	WOOD ON YORKSTONE			
LDNR	<0.7	<0.7	<5	<5	100%	50%	100%	100%	100%			
	<0.7		100%		50%	100%	100%	100%				
	<0.7		100%		50%	100%	50%	100%				
PETN	2.3	2.4	120	73	50%	0%	0%	0%	0%			
	2.7		50%		0%	0%	0%	0%				
	2.3		50%		0%	0%	0%	0%				
HMX	2.8	2.5	160	147	50%	0%	0%	0%	0%			
	2.5		50%		0%	0%	0%	0%				
	2.1		50%		0%	0%	0%	0%				
RDX	3.0	3.0	240	173	50%	0%	0%	0%	0%			
	3.0		50%		0%	0%	0%	0%				
	3.0		50%		0%	0%	0%	0%				
Tetryl	6.3	6.3	>360	>360	0%	0%	0%	0%	0%			
	6.1		0%		0%	0%	0%	0%				
	6.4		0%		0%	0%	0%	0%				
RDX/TNT	>8.2	>8.2	>360	>360	0%	0%	0%	0%	0%			
	>8.2		0%		0%	0%	0%	0%				
	>8.2		0%		0%	0%	0%	0%				
TNT	>8.2	>8.2	>360	>360	0%	0%	0%	0%	0%			
	>8.2		0%		0%	0%	0%	0%				
	>8.2		0%		0%	0%	0%	0%				

BAM Friction Test:

LDNR > PETN > HMX > RDX > Tetryl = RDX/TNT = TNT

Although the three tests rank the sensitiveness to friction in essentially the same order, each displays different degrees of sensitivity and reproducibility.

From Table 2 it can be seen that the Rotary Friction Test gave reproducible results for all the seven materials tested. The method was able to discriminate between all the explosives apart from RDX/TNT and TNT which were both less sensitive than the lower limit of accurate measurement. LDNR was found to be so friction sensitive as to be beyond the upper limit of sensitiveness measurement of the test⁹.

We ascribe the differences between our results for the explosives TNT and tetryl, and those quoted in the UN Manual ⁵, to the use of different methods of assessing whether an ignition has occurred. Experience in this laboratory has shown that the visual detection of sparks is the most accurate means of determining whether an explosion has taken place. The use of other techniques, such as audible detection or the examination of the contact surfaces for evidence of combustion (blackening), is much more subjective. There is an additional concern in examining whether sample blackening has occurred, since this could entail the exposure of the operator to toxic decomposition products.

The results obtained with the Mallet Friction Test were found to be reproducible but not particularly selective. The test gave

many results rated at 0% ignitions and was unable to distinguish between TNT, RDX/TNT and Tetryl which were so insensitive to friction as to be beyond the measurable limit. RDX, HMX and PETN were all ranked the same, with positive results being obtained only when using the steel on steel combination.

The BAM Test results, Table 2, indicated that this method was unable to distinguish between Tetryl, RDX/TNT and TNT, judging them to be of equal sensitiveness and beyond the limit of accurate evaluation. The limit of sensitiveness measurement is lower than that for the Rotary Friction Test since Tetryl as well as RDX/TNT and TNT lie outside the range. As with the Rotary Friction results, LDNR was so friction sensitive as to be beyond the upper limit of measurement. Those materials with friction sensitivenesses that could be numerically quantified using the apparatus (PETN, HMX and RDX) displayed a range of values in the three individual determinations. Although the method was less reproducible than the Rotary Friction Test it could clearly distinguish between the friction sensitivenesses of these three explosives.

Since the individual BAM results in Table 2 cover a range of numerical values, our data are in satisfactory agreement (within one load increment) with those published in the UN Manual⁵.

We note that, whereas the UN Manual gives finite results for the friction sensitiveness of the explosive TNT when using either the Rotary Friction Test or the BAM Friction Test, in our studies

the material was beyond the limits of measurement of the two test methods. This may indicate that the explosive stocks were of a different nature, or that the blackening of the sample was taken as the basis of a positive test result.

It is felt that the relatively poor reproducibility of the BAM test could be improved by performing a second series of six trials at the load two levels below the limiting load. A similar procedure is employed with the BAM Impact Test and this method is capable of generating reproducible results.

From our present studies, and practical experience gained with the three test methods over a number of years, it is clear that each apparatus has certain advantages and disadvantages.

The Rotary Friction method has the advantage over the other tests of employing an internal calibration standard: in accordance with documented procedures^{5,6} the sensitiveness of the explosive under test is compared with the results obtained for RDX. Another advantage lies in the mechanical nature of the test which ensures that the energy transmitted to the sample is constant and reproducible at each speed: this probably accounts for the good repeatability. In our work, however, we have noted that when the apparatus is subjected to frequent use at the maximum speed of 398 rpm it can be prone to mechanical failure. We also feel that, since the detection of a positive event is reliant on the operator's senses, it is subjective and could be improved by using gas sensors in the enclosed area surrounding the sample under

test. Although the detection of events in both the Mallet Friction and BAM Friction Tests is also dependent on the operator's senses, neither of these methods is capable of being readily modified in this manner.

We also note that whereas positive events were easily discerned with certain explosives when using the Rotary Friction Test (eg PETN, RDX, HMX) since they reacted violently, this was not found to be the case with the other methods investigated, when only mild cracking and spark production was detected.

The Mallet Friction Test offers a clear advantage over the Rotary Friction and the BAM methods in using a range of test surfaces. The Rotary Friction utilises only steel on steel whereas the BAM examines a porcelain on porcelain combination. The latter system was presumably selected because of the increased likelihood of generating local hot spots when using friction materials of low thermal conductivity¹⁰; these rubbing surfaces are, however, less relevant to the types of situations likely to be encountered, for example, during manufacturing:

As a method of evaluating pure friction effects, the Mallet Friction Test is poor since the glancing blows to the test surface will combine a degree of impact with the main shearing force. The test is also crude in nature, with the energy imparted into the sample being dependent on the technique of the operator. There is the likelihood of inconsistency between blows and between the modes of operation of individual test operators.

The Mallet Friction Test can, however, provide a useful technique for the investigation of incidents, since these often occur in situations where both impact and friction forces can be important.

Although the BAM Test does not use a reference standard, it should have the advantage, because of its mechanical nature, of ensuring that the energy transmitted to the sample is constant and reproducible for each load. However, with loads of 80N or greater we have noted that, even in the absence of test material, substantial heating occurs at the interface between the porcelain surfaces. This induces both a red glow and the production of glowing sparks: both these heat sources could affect the results with substances having low decomposition temperatures.

With the BAM apparatus, it was also apparent that movement of the porcelain peg could cause the test sample to be pushed aside, thus leaving little material to experience friction forces between the contact surfaces.

CONCLUSION

The studies to evaluate three commonly used friction test methods have indicated that, although all the methods examined were able to rank seven explosives in approximately the same order of friction sensitiveness, the Rotary Friction was the most reproducible test and that with the widest range of applicability. The Rotary Friction Test was the only apparatus for which all the explosives PETN, HMX, RDX and Tetryl gave a measurable result.

It is apparent that the BAM Test, in its current form, is not suitable for accurately quantifying friction sensitiveness. Several areas for improvement (eg. non-reproducibility of results, excessive generation of heat, and the pushing aside of sample) have been identified.

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8. The symbol > is used to indicate "more sensitive than", and the symbol = is used to designate materials of equivalent friction sensitiveness..
9. For extremely friction sensitive materials, the SCC method enables non-standard testing to be done using a reduced load specification.
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