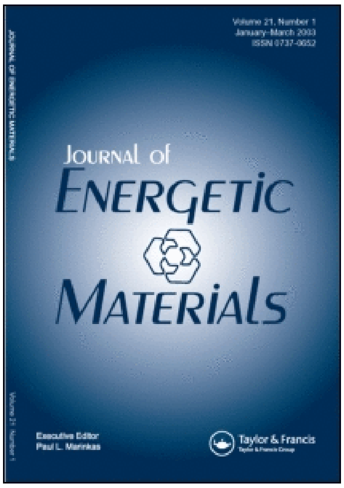

Journal of Energetic Materials
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713770432

Impact sensitivity of nitroglycerin

Marvin L. Jones[a]; Edmund Lee[a]
[a] United Technologies, San Jose, CA

To cite this Article Jones, Marvin L. and Lee, Edmund(1997) 'Impact sensitivity of nitroglycerin', Journal of Energetic Materials, 15: 2, 193 — 204
To link to this Article: DOI: 10.1080/07370659708216083
URL: http://dx.doi.org/10.1080/07370659708216083

IMPACT SENSITIVITY OF NITROGLYCERIN

Marvin L. Jones and Edmund Lee
United Technologies
PO Box 49028
San Jose, CA 95161

ABSTRACT

The impact sensitivity of liquid nitroglycerin, 50% initiation level, was observed to be near 1.8 kg*cm or 3.3×10^3 j/m^2. The impact sensitivity of solid nitroglycerin, 50% initiation level, was observed by extrapolation to be near 200 kg*cm or 4.4×10^5 j/m^2. The impact sensitivity of a mixture of solid and liquid NG was observed to be comparable to solid NG at the two stimulus levels tested. Thus, the presence of solid nitroglycerin reduces the impact sensitivity of liquid nitroglycerin.

The 50% initiation level for Class A cyclo-tetramethylenetetranitramine was observed to be near 150 kg*cm or 3.3×10^5 j/m^2 using the same instrument.

INTRODUCTION

A number of weather related events during the last several years prompted a review of the sensitivity of

Journal of Energetic Materials Vol. 15, 193-204 (1997)
Published in 1996 by Dowden, Brodman & Devine, Inc.

nitroglycerin (NG) at low temperatures. Urbanski[1] reported sensitivity values determined by Hackel[2] for liquid NG and solid NG. The results indicate a 6 to 8 times greater shock initiation stimulus is required to generate an explosion with frozen NG than with liquid NG. Neither the type of stimulus nor explosion were defined. Also, the labile form of NG crystals requires a greater shock stimulus than the stable form of NG crystals. Cronquist[3] reported the impact sensitivity of liquid NG to be 0.6 to 0.7 kg*cm, of solid NG to be 1.0 to 1.5 kg*cm, and of a mixture of liquid and solid NG to be 0.3 to 0.4 kg*cm. Cronquist reported the method of obtaining the liquid and solid mixture was to impact the solid, melting a portion, and then re-impact the sample.

The impact sensitivity of NG mixtures has been observed to be more sensitive than the values reported by Cronquist. Cronquist did not report on his test apparatus. Also, the method of preparing the liquid and solid NG mixtures may have compromised the sample. For these reasons, it was decided to investigate the impact sensitivity of solid and liquid NG.

The impact sensitivity of cyclo-tetramethylenetetranitramine (HMX) was determined for comparison.

MATERIALS

A 10% mixture of NG in lactose was obtained from ICI Americas Inc., Specialty Chemicals Division, Wilmington, DE. The lactose was removed by repeated washing with water and chloroform. The NG was dried in a desiccator over phosphorus pentoxide.

Small quantities of NG were placed in aluminum dishes and cycled between cold-boxes at 0°, -15°, and -40° C numerous times without formation of crystals. Crystals of sodium chloride and potassium nitrate were added to separate dishes and the temperature recycled. When nucleation occurred, a small quantity of crystals was transferred to other dishes of NG near 0° C and crystallization proceeded. Additional solid NG was prepared as required.

The solid NG was somewhat opaque and not transparent crystals as shown by Brun.[4] The presence of a trace of water was suspected. Naoum[5] reported the solubility of water in NG as 0.1 to 0.2%. A differential scanning calorimetry (DSC) trace of the solid NG did confirm the presence of water with its endotherm at 0° C. The DSC trace also revealed the NG to be the stable form with a melting point of 13.0° C. The material was used without further purification.

Small NG crystals were prepared by placing several drops of NG in an aluminum dish. After cooling to near 0° C, an NG crystal was introduced and the mixture gently stirred. The NG would freeze rapidly in thin sheets which could be broken by flexing the aluminum dish. The NG would crumble into small crystals.

Larger, 2 to 4 mm diameter, crystals of NG were formed by placing several drops of NG in a pre-cooled aluminum dish that had previously contained NG crystals. The separated NG drops would freeze into hemispherical crystals overnight.

HMX was obtained from Holston Army Ammunition Plant. It was dried and used as is.

PROCEDURE

The Olin Impact Test Apparatus was used. Liquid NG was tested using ASTM Designation: D 2540 - 66 T method. The procedure for preparing the sample in the sample holder is described in the ASTM method and will not be repeated here. The impact hammer had a diameter of 8.30 mm. A 'First-Bounce" catching device was not used. A trial that failed to initiate on the initial impact but initiated on the rebound was considered a failure. The model impact test apparatus used had a 1 kg hammer.

Weights could be added in increments of 0.25 kg up to a total of 6 kg, including the hammer.

The same test apparatus was used for testing HMX and solid NG. The sample was confined in a brass cup with an internal diameter of 8.03 mm. The steel hammer had a diameter of 7.62 mm. There was a sample holder where the brass cup would set on an anvil about 2 cm thick. The hammer was held in the vertical position in contact with the solid in the cup. The sample holder was then placed in the impact test apparatus for testing.

The drop heights were selected to increase the stimulus about 50% above the previous level. The drop heights used with liquid NG with the 1 kg weight were 1, 2, and 3 cm plus 1.5 kg and 1 cm. The drop heights used with solid NG and liquid-solid mixtures with either the 2 or 3 kg weights were 32 and 48 cm. Numerous drop heights were used with the 2, 3, 4, and 5 kg weights when testing HMX.

The impact tests on HMX and liquid NG were performed at room temperature, 20 to 25° C. The impact tests with solid NG, with and without liquid NG, were performed between 5 and 10° C. This temperature was obtained by constructing a dam around the base of the impact machine and adding ice. Water was allowed to accumulate to a depth of about 1.5 cm. The sample holder and hammer were placed in a pan of ice water to keep the temperature as consistent as possible.

The first 30 or so trials with solid NG were performed with 0.05 to 0.1 gram of NG crumbs. The final 30 or so trials were performed with two hemispheres of solid NG in each cup.

The trials with liquid and solid NG were prepared by placing 2 hemispheres of solid NG in a cup and adding 2 drops of liquid NG. The trial was performed without delay.

It was common for solid NG to extrude out of the cup during an impact event. The hammer and cup had to be allowed to warm, thawing the NG, in order to remove the cup. Liquid NG was observed to extrude (squirt) from the cup during the impact events with solid and liquid NG. The NG would be frozen by the time the sample holder was removed from the apparatus. Again, the hammer and cup had to be allowed to warm in order to remove the cup.

The impact energies of the stimuli were estimated from the potential energy of the weight falling a given height. There was no correction for energy loss due to friction, misaligned apparatus, poor energy transfer, or for rebounds. The impact areas were estimated from the diameters of the hammers, 8.30 mm for liquids and 7.62 mm for solids.

Initiation was defined by a loud report.

RESULTS

The probit or probability method[6] was used to determine the threshold initiation level (TIL). The TIL was defined as the stimulus level where no initiation events were observed in 20 trials (10 trials for tests with solid NG present) with one or more initiation events being observed at the next higher level, a nominal 50% increase in stimulus. The stimulus was varied by changing the drop height and/or the weight on the hammer.

The results for liquid NG are shown in Table 1. The drop heights and weights are included.

Table 1

Impact Test Results for Liquid Nitroglycerin

Weight	Drop Height	Stimulus		Number of	
kg	cm	kg*cm	j/m^2	Trials	Initiation
1.0	1.0	1.0	1.8×10^3	20	0
1.5	1.0	1.5	2.7×10^3	10	5
1.0	2.0	2.0	3.6×10^3	10	4
1.0	3.0	3.0	5.4×10^3	10	9

Few initiation events were observed with solid NG. A complete probit or probability series was not completed. Normally, it is desirable to continue to increase the stimulus until 80% or more initiation events are observed at a stimulus level. Half or more of the impact trials were initiating on the first or second rebound. For this reason, it was decided to limit the test trials with solid NG.

The results for solid NG are shown in Table 2. The drop heights and weights are also included.

Table 2
Impact Test Results for Solid Nitroglycerin

Weight	Drop Height	Stimulus		Number of	
kg	cm	kg*cm	j/m^2	Trials	Initiation
2.0	32	64	$1.4x10^5$	20	0
3.0	32	96	$2.1x10^5$	18	1
3.0	48	144	$3.1x10^5$	10	2

A limited number of impact trials were performed with the liquid and solid NG mixtures at the two lowest levels tested with solid NG. The results with liquid and solid were comparable to the results with solid NG. The results are listed in Table 3.

Table 3
Impact Test Results for Mixtures of
Solid and Liquid Nitroglycerin

Weight	Drop Height	Stimulus		Number of	
kg	cm	kg*cm	j/m^2	Trials	Initiation
2.0	32	64	$1.4x10^5$	10	0
2.0	48	96	$2.1x10^5$	10	1

The impact sensitivity of Class A HMX, nominal 180 micron particle size diameter, was determined for comparison. Numerous impact test levels were evaluated.

The results for HMX are shown in Table 4. The drop heights and weights are also included.

Table 4
Impact Test Results for HMX

Weight	Drop Height	Stimulus		Number of	
kg	cm	kg*cm	j/m^2	Trials	Initiation
5.0	9	45	$1.0x10^5$	20	0
4.0	14	56	$1.2x10^5$	10	1
3.0	21	63	$1.4x10^5$	32	1
5.0	14	70	$1.5x10^5$	13	1
4.0	21	84	$1.8x10^5$	10	2
3.0	32	96	$2.1x10^5$	30	3
5.0	21	105	$2.3x10^5$	15	2
2.0	56	112	$2.4x10^5$	10	1
4.0	32	128	$2.8x10^5$	30	8
3.0	48	144	$3.1x10^5$	35	5
5.0	32	160	$3.5x10^5$	20	12
3.0	56	168	$3.7x10^5$	30	17
4.0	48	192	$4.2x10^5$	10	9
4.0	56	224	$4.9x10^5$	10	7
5.0	48	240	$4.2x10^5$	10	10

DISCUSSION

The behavior of liquid NG to impact stimulus was what is expected for a sensitive material. A TIL was defined and initiation events were observed at the next higher level. There were 90% initiation events at the third level above the TIL. The data are shown in probability format in Figure 1. The TIL was observed to be 1 kg*cm. This value is equivalent to 1.8×10^3 j/m^2, neglecting impact losses. The 50% initiation value was interpolated to be 1.8 kg*cm. This value is equivalent to 3.2×10^3 j/m^2. Solid NG is about two orders of magnitude less sensitive that liquid NG. The data are shown in probit format in Figure 2. The TIL was observed to be 64 kg*cm. This value is equivalent to 1.4×10^5 j/m^2. The 50% initiation value was obtained by extrapolation of the two impact values where initiation events were observed. The value is near 200 kg*cm or 4.4×10^5 j/m^2.

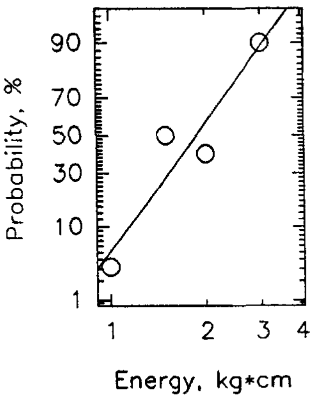

FIGURE 1
Impact Sensitivity
Liquid Nitroglycerin

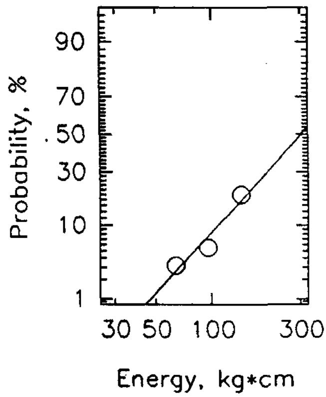

FIGURE 2
Impact Sensitivity
Solid Nitroglycerin

In order for NG to initiate, the material, or at least a kernel of sufficient size to support reaction, must be heated to a critical temperature. When this occurs, the reaction reaches run away conditions. This results in an initiation. An impact event can generate hot spots that do not reach the critical temperature. When a second impact such as a rebound occurs, the hot spot can be further heated. If the critical temperature is reached, the reaction can reach run away conditions resulting in an initiation event. This would explain initiation on the first or even second rebound where the stimulus is much less than the initial impact.

The observation that solid NG is much less sensitive to impact than the liquid can be attributed to the heat sink of melting at 12° C. In order for material to reach the critical temperature, the material must melt, at least the material in the kernel, and the kernel heated further. The heat of fusion is 33.2 cal/g. When considering the heat capacity of the solid, 0.315 cal/g/deg C, or the liquid, 0.356 cal/g/deg C, the heat of fusion would be equivalent to lowering the initial temperature of the liquid almost 100° C.

The impact trials performed with a mixture of liquid and solid NG indicate the mixture is comparable in sensitivity to the solid alone. The mixture is certainly not more sensitive as suggested by Cronquist. The heat sink of the solid material made the mixture less sensitive.

The impact sensitivity of solid NG, with or without liquid present, is comparable to the impact sensitivity of HMX. This can be seen by comparing the HMX results in

Table 4 with the NG results in Tables 2 and 3.

ACKNOWLEDGEMENT

This work was supported by the US Navy on Contract N00030-93-C-0068.

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