Drop weight impact measurements of HE sensitivity: modified detection methods

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Abstract. High explosives small-scale sensitivity testing has been a hallmark of safety screening since WWII. Sensitivity testing was once crude and simple; broom sticks were used to scrape explosives on the floor while experimenters would look, listen, and smell for signs of a reaction. Since then, a wide variety of testing apparatus have been developed to explore the effects of different stimuli on explosives. In concert with the development of the machines themselves, the reaction detection methods have also evolved. This paper’s focus is on the Los Alamos National Laboratory’s (LANL) drop weight impact machine and reaction detection methods. A critical evaluation of results is presented with cautionary examples of false positives that can occur with non-explosive materials.

1. Introduction
Small-scale safety testing (SSST) is performed to determine the sensitivity of an energetic material to non-shock initiation scenarios. Results from the SSST enable a subject matter expert (SME) to make data-based decisions regarding the safe handling of a material for a given working condition. The SSST at Los Alamos includes vacuum thermal stability (VTS), differential scanning calorimetry (DSC), drop weight impact, friction, and electrostatic discharge (ESD) evaluation. It is important to note that though all of the SSST results are used to determine the sensitivity of an explosive, only three of the five tests are true sensitivity tests (impact, friction, and ESD), while VTS and DSC give information on compatibility and thermal behaviour of the sample. Drop weight impact sensitivity testing is the focus of this paper.

This general study explores the viability of new and modified reaction detection methods employed on the LANL Explosives Research Laboratory (ERL) Type 12 Drop Weight Impact Machine. From the drop weight impact machine’s inception to the present, some laboratories and testing facilities use operator judgment to determine explosive reactions or Go’s. Typically, the operator either listens for a reaction or looks for light during impact. Some reports indicate that at one time smell was commonly used for reaction detection. Smell should no longer be employed for reaction detection because a great deal of modern explosives work is devoted to the development of new molecules and formulations that may not thoroughly decompose, hence the inhalation of such products can have adverse effects on the human body. It is advantageous to have a reaction detection system that is operator independent. Since the early 1980’s, LANL has used two microphones in conjunction with sound level meters. Below we
provide a brief general description of detection methods, and summarize the results of our investigation into Go detection using a variety of diagnostics: a large bandwidth microphone, a series of strain gauges, and a digital single lens reflex camera (DSLR) [1].

2. Machine and Method

2.1. Tooling
Tooling for the Type 12 Drop Weight Impact Machine includes a 2.5 kg drop weight, a 0.8 kg striker, and an anvil. The striker and anvil are made from an A1-like tool steel and through-hardened to an HRC (Rockwell hardness) of 60. The testing faces of the striker and anvil are surface ground to 16 micro-inches. Energetic reactions, Go’s, are defined as the average of two sound meter levels greater than 120 dB. The 50% height (H50) is calculated using the Neyer D-Optimal method. Fifteen samples were tested for each H50 [2].

2.2. Sensitivity Testing
Sensitivity testing, a unique form of testing, is typically performed as a last resort due to the large consumption of material and the limited statistical value of the data generated. Sensitivity testing assumes the following: all samples are equal, there are no “duds”, a sample can only be tested once, and the applied stimulus to the sample must either result in a Go or a No-Go. Traditionally results are expressed as a 50% level of initiation or a threshold of initiation value (TIL) [3].

2.3. Go-criteria
The criteria for a Go must be defined at the start of the experiment. For explosives, light and acoustic output are among the most common Go criteria. Changing the Go criteria for a sensitivity test can dramatically affect the results. By defining and adhering to consistent Go criteria, multiple operators can generate statistically similar results.

2.4. Determination of 50% drop height (H50): Neyer’s D-optimal
The Neyer method was founded in 1994 and is based on an algorithm that uses maximum likelihood estimation (MLE) of $H_{50}$ and sigma (standard deviation) to adjust the stimulus levels during testing. This ensures that the estimates of both parameters are optimized simultaneously. The testing proceeds in a manner similar to the Bruceton approach – the outcome of a previous test determines the chosen stimulus level for the next test – but unlike the Bruceton method, the step sizes may change depending on the likelihood function. A commercial software package is used to compute the necessary stimulus level based on the MLE. Ultimately, the step size approaches values between 1 s and 2 s by construction. Two advantages of this method are: the $H_{50}$ and sigma are optimized together to better characterize the distribution; the adjustable step size allows the distribution to be probed using typically fewer tests than with the Bruceton method. On the other hand, this method requires a computer to calculate and adjust the step sizes between tests [2-4].

3. Experiment and Results and Discussion

3.1. Light detection
Almost all explosives and propellants emit visible light during rapid decomposition. Three explosives were used for this study: PBX 9502, HMX, and PETN. A DSLR was used to capture the emitted light. Specifically, the Nikon D7000 DSLR camera with a Nikkor 200mm focal length micro lens was used. The camera was set to 6400 ISO, f4.8, and a 1.6 second exposure. The light in the bay and outer room were turned off during testing. The shutter was opened before the weight was dropped and remained
open during the impact event and several rebounds. Images were processed by inverting their color maps in Photoshop and expanding the levels in each channel to fill the displayable color range. With this method, light emissions from reactions appear as intense blue-green areas on a washed-out blue-green background. A typical view captured by the camera is shown in figure 1(a). Examples of reactions are shown in figures 1(b) and (c). Figure 1(c) shows a deflagration in PBX 9502 due to grit-on-grit heat generation in drop weight impact. An example that does not exhibit a reaction is shown in figure 1(d). For comparison, full reactions of HMX and PETN are exhibited in figures 1(e) and (f).

**Figure 1.** (a) View shows the anvil (bottom) and striker (top). The samples are placed on 180 grit sand paper and put between the striker and anvil. (b) The dark blue spot at the striker-anvil junction is the light emission. Most events were of this color intensity and size. The vertical blue streak is stray light reflection from the striker. (c) This was the single strongest emission observed during all testing. (d) Impact data from drop test that showed no evidence of reaction: little light was detected, material was not consumed, and there was no excess noise detected. The anvil-striker junction is light green with no blue spots. (e) Light emission from 40 cm drop onto HMX. The intense emission has shifted the entire color scale for the image. (f) Light emission from 30 cm drop onto PETN.

This study did not lead to any conclusions as to the amount or intensity of light needed to define a Go; this is an unresolved topic in sensitivity testing. It would be problematical if any detection of light were used to define a Go, such as in the case of triboluminescent material. For this study, Wint-O-Green® Life Savers were chosen due to well-known triboluminescent behavior and its non-toxic nature (which was experimentally verified by the authors of this study). In this case, Wint-O-Green® Life Savers subjected to study on the drop weight impact would yield primary explosive-like results, if the triboluminescence that occurs during crushing of crystals was used as an indication of a Go [5]. This example is mentioned here to point out that light detection alone may be misleading as a Go criterion, and triboluminescence in explosive materials is also known [6].
3.2. Acoustic detection

The use of microphones began in the 1950’s with a “noisometer” at LANL. In previous years, typical Go / No-Go determination involved measuring the material response against a predetermined threshold sound intensity. With the acquisition of better microphones and calibrated read-outs, recording the actual decibel levels provides another level of insight into the impact event. Routine impact testing Go-detection at LANL uses two microphones (Bruel & Kjaer 4136) and two sound level meter (Bruel & Kjaer 2231) assemblies set to capture max impulse RMS pressure levels. The Go criteria are set to 120 dB. For solid explosives this method works with little ambiguity. For type 13 testing of liquid samples, this method may be misleading.

3.2.1. Drop weight impact of water. Dropping the weight onto a striker on a blank anvil (figure 3b and 3c) produces less than a 112 dB response at a 320 cm drop height. The threshold for Go / No-Go determination of 120 dB is well offset from this baseline and should allow good discrimination of reactions. Drop testing deionized water produces a response above the threshold and the assigned 50% level of 216 cm (figure 3a). This suggests that there is in fact some reaction or mechanical phenomenon occurring. A combination of variables could be responsible and would be understandable if the event occurred due to a phenomenon such as cavitation. This observation also suggests that liquid explosive materials should be examined with a more critical criteria before a 50% reaction level is assigned.
Figure 3 (a) $H_{50}$ of deionized water (216cm). (b) Blank greased anvil with 40mg water bead at the center. (c) Striker is suspended over the anvil with a magnet imposing a 2mm gap between the anvil and the striker.

An example of this is given with comparison of two aqueous-based liquid formulations. As shown in the figure 4, formulation 1 exhibits obvious Go events above 125 dB separated from No-Go events below 110 dB resulting in a 50% reaction height in the 75 cm range. Although formulation 2 is somewhat noisy, the sample is not obviously reacting – there is no clustering of Go and No-Go events. Applying a threshold level to formulation 2 would suggest a very narrow reaction distribution but varying the threshold slightly would move the 50% level around significantly. Formulation 2 may not be reacting at all, but exhibiting the same acoustic behaviour as pure water.

Figure 4. Acoustic drop-weight impact results.
3.3. **Dual strain gauge with advanced microphone**

We have encountered explosive formulations which are found to have significantly different sensitivities when tested by different labs. Our investigation has shown that the microphone or operator's frequency response differs from lab-to-lab. To reduce subjectivity and enhance acoustic sensitivity, we have explored implementing strain gauges on the anvil and striker and a broadband microphone to characterize the event, and a similar approach was used in ref [7]. Thus far, initial tests are promising. A Go produces a strong signal on the anvil and a very strong signal on the microphone that can be analysed for frequency. A No-Go from the same drop height shows very little response on the anvil and microphone. The delay between the strain signal and the microphone signal is due to the speed of sound in air (1100 ft/s): a three-foot microphone offset corresponds to around 2.7 ms.

![Figure 5. Dual strain gauge and microphone plots.](image)

4. **Summary**

As illustrated above, light, sound, and mechanical response are all viable methods in assisting in detection of reactions on the impact machine. Each method is economical, easy to install, but does come with some potential pitfalls, and illustrates that critical analysis of all results is required when determining the true sensitivity of an energetic material. These detection methods can be used to formulate Go criteria; using these modified methods improves the accuracy of sensitivity testing, avoiding false positives or negatives. This improves safety and saves time and money.

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