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Integrated Data Collection Analysis Program--Quarterly Review Meeting Summary

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Integrated Data Collection Analysis (IDCA) Program —Quarterly Review Meeting

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EXECUTIVE SUMMARY

On November 9 and 10, 2011 the IDCA had the annual quarterly meeting. The meeting started the afternoon of the first day with a tour of the NSWC IHD explosives safety testing and analysis facilities. The meeting on the second day addressed the formal sponsor review and further technical issues for the IDCA.

Examination of the IHD equipment during the tour, lead to a long discussion on liquid test methods. The discussion resulted in revision of liquid test methods in the impact test and selection of a new liquid test standard. In addition, modifications to friction, spark and thermal test methods were discussed.

The program review started with a summary of past performance to date. The proficiency test is almost complete. LANL and LLNL have completed examining all materials and released data reports. IHD is about 2/3 through the testing and expects to finish by the end of December. Tyndall is releasing reports on data already taken and is awaiting funding. The IDCA has issued 12 analysis reports, 70 data reports, and 8 outside presentations.

The focus of this year will be the revision and population of the DHS Small Scale Safety and Thermal (SSST) Test Guide, a reference guide of safety testing results on home made (improvised) explosives. LLNL and LANL will lead the effort. The first set of data to be included is from the results of the Proficiency Test. IHD and Tyndall are tasked with completing their measurements on the original set of materials for the Proficiency Test. All testing laboratories will do supplemental testing to resolve discrepancies that may have occurred in Proficiency Test data. SNL will be setting up a SSST testing facility and will join in the Proficiency Test if possible.

The DHS SSST Test Guide is being revised from the original format to include much more information about the materials, the testing methods and equipment design. The IDD server at LLNL is proposed to house the Guide temporarily. This server has a controlled access method in place. The ultimate place of the Guide and how it will be managed has yet to be decided.

The IDCA has committed to the International Round Robin (IRR) being sponsored by the Technical Support Working Group (TSWG). The IDCA will submit testing procedures and equipment identification to TSWG for each of the laboratories that will participate (so far all the IDCA participants). There are decision points about IDCA involvement in the IRR that depend upon the effectiveness of TSWG obtaining the test materials.

Several issues that have come about during the Proficiency Test were discussed.

- Thermal analysis of mixtures of KClO_3 /icing sugar reproducibility—There appears to be lack of homogeneity due to the small sample size required for DSC. The IDCA will continue to study this with the aim of publishing a definitive article on the subject.
- Statistical representation of Proficiency Test data—The Proficiency Test has yielded a very large amount of SSST testing data that allows for inter- and intra-laboratory performance comparisons. So far, on a limited set of comparisons for impact testing, there is a 5 to 20% variation within and among the participants. This is hardly statistical so statistical methods will be improved upon for this comparison data.
- Modifications of SSST testing—Discussion ensued on several subjects that either modify, improve or supplement SSST testing. Accelerated reaction calorimetry (ARC) confirmed measurements that substantiated DSC information that the IDCA has tested some very thermally sensitive materials, such as HP fuel mixtures and solid oxidizers with S added. Robocasting Enterprises is developing a new plate for the BAM friction testing system that is manufactured with more precision than the current stage. The IDCA will have a chance to test this new design when available.
- New research—Research topics under consideration for effort this FY are: adaptation of the DSC to better predict thermal instability in HMEs, effects of aging of HMEs on SSST testing results, understanding better the sandpaper grit size effect in impact testing, use of high speed cameras for detection of positive events, participation in the SMS User Group planned round robin, and potential participation of the Army in the IDCA Proficiency Test.

Because the FY 11 funding was received very late in the year work the flow was interrupted, so a new baseline on the unaccomplished deliverables needed to be defined. The new time line includes producing an analysis report every two weeks, milestones for the DHS HME SSST Test Guide, participation in the International Round Robin, and deadlines for upcoming quarterly meetings.

The meeting was completed with a presentation research on the drop hammer type 12-impact testing.

Keywords: Small-scale safety testing, proficiency test, round-robin test, safety testing protocols, HME, RDX.



ACTION ITEMS

The meeting resulted in many action items for the entire crew. These are listed below roughly by topic, and throughout the text.

1. *Revision of liquid testing methods—Mary and Geoff volunteered Daniel Preston to write-up his liquid test procedure; everyone will critique this method and compare how LANL does it.*
2. *Revision of liquid testing methods—IHD will perform liquid testing using cavity device and greased anvil*
3. *Revision of liquid testing methods—LANL and LLNL will chose some materials to redo with sandpaper.*
4. *Revision of liquid testing methods—LLNL will perform DH tests on HP/Flour, HP/Cumin with 180-grit sand paper.*
5. *New liquid testing standards—Mary will look into ordering n-propyl nitrate for everyone (drop ship from vendor on Geoff's pcard?); need to determine how many vials of n-propyl nitrate to buy for each lab.*
6. *Striker weight discussion—LLNL will see if there is a method to hold the 2.5-kg striker in position.*
7. *Striker weight discussion—LANL will share their magnet technique to hold the 2.5-kg striker with LLNL.*
8. *Striker weight discussion—LLNL will measure DH on RDX with a 1.0-kg striker.*
9. *Striker weight discussion—LANL and Tyndall say they have a 1.0-kg striker weight, and they will try a couple of materials and see what happens.*
10. *Friction testing—LLNL will look for a way to test where the BAM apparatus safety enclosure does not interfere with hearing positives (may be special cases approved by the safety people).*
11. *Friction testing—LANL can look for a temporary full enclosure to cover the BAM apparatus and try some retesting.*
12. *Friction testing—Everyone will send in pictures of how their BAM friction apparatus is enclosed or shielded.*
13. *ESD testing—Mary and Geoff have volunteered Daniel Preston to distribute the information on the Spark tester probe that he uses for calibration.*
14. *ESD testing—ESD tests on KP/Al with LLNL new spark tester.*
15. *Reports and Presentations—Any extra work that is done by anyone should be written into a report or memo so it can be assigned an IDCA report number for tracking (this includes work in addition to the data collection reports).*
16. *Compilation of the DHS SSST Test Guide—Peter is heading up the LLNL effort for the Test Guide.*
17. *Compilation of the DHS SSST Test Guide—Mary, Geoff, Daniel, etc., will lead the LANL effort for the Test Guide.*
18. *Compilation of the DHS SSST Test Guide—Tim will ramrod the effort of collecting high fidelity test data (should include HMX) as a link to historical databases.*
19. *Compilation of the DHS SSST Test Guide—Add n-propyl nitrate as liquid standard to IDCA testing.*
20. *Access to DHS SSST Test Guide—John will check on arranging access to the IDD server and putting the Test Guide on it.*
21. *Access to DHS SSST Test Guide—Laura and Greg will talk to Harry about putting the Guide on the IDD server.*

22. *Access to DHS SSST Test Guide—Laura will check with DHS to see if it is OK to put this on DTIC (Defense Technical Information Center).*
23. *International Round Robin—JGR will contact Beth Obregon to find out the link for the TSWG web site for the International Round Robin so the IDCA can have access, and the information can be easily shared.*
24. *International Round Robin—Everyone can release scale-up methods/data to the TSWG web-site (when we find the web site).*
25. *International Round Robin—Laura will tell TSWG we need the RDX soon (by Feb) or the IDCA will be unable to participate.*
26. *International Round Robin—Laura will ask if using European RDX would be easier to obtain for the whole group.*
27. *International Round Robin—All, IDCA is supposed to have document HP analysis procedures for checking concentration and purity. Make this happen.*
28. *KClO₃/icing sugar DSC issues—All, dig up DSCs of KC with different sugars.*
29. *KClO₃/icing sugar DSC issues—Mary will send out the post-test photographs that were taken during the sample size variation testing.*
30. *KClO₃/icing sugar DSC issues—Tim will come up with an experimental plan to prove heterogeneity of the KClO₃/sugar system for future discussions.*
31. *Statistical Analysis of IDCA Results—Geoff will test his proposed approach for statistical analysis of the Proficiency Test results on a material of his choice.*
32. *High-speed camera for positive event detection in SSST testing—Laura/John will draft paragraph to show support for this capability.*
33. *Participation in the SMS Round Robin—Laura/John will draft paragraph to show support for the SMS round robin.*
34. *Participation of the Army in the IDCA Proficiency Test—JGR will contact Roger Hale (Tooele Army depot) with the go ahead from DHS.*
35. *Participation of the Army in the IDCA Proficiency Test—John can give Laura a paragraph on how the Tooele interaction would affect the IDCA.*
36. *Other research topics, Methods report—Mary will get the Methods report finished. This will help us with the IRR methods analysis.*
37. *Other research topics, new BAM Friction plates— Mary will send out sample plates from Robocasting Enterprises to the team later if the prototypes work out*
38. *Other research topics, DSC use for thermal stability screening—Labs should provide some input to JGR on how to do this for now and maybe start small study later.*
39. *Other research topics, aging studies—Peter will measure the aging effect on KC/Dodecane on impact sensitivity (2 hrs., overnight, 4 days, and 1 week).*
40. *Other research topics, aging studies—Kirstin will measure the aging effects on HP/Flour (2hrs, overnight, 4days, and 1 week) using microcalorimetry.*
41. *Other research topics, DH sandpaper—We need someone to champion this—any interest? Peter and Dan can send sandpaper to Mary.*
42. *Rebaselining—Greg will get no cost extensions in early 2012 so that no one has to stop work.*

Keywords: Small-scale safety testing, proficiency test, round-robin test, safety testing protocols, HME, RDX.

1 INTRODUCTION

On November 9 and 10, 2011, the IDCA conducted the Annual Quarterly Review at Naval Surface Warfare Center, Indian Head Division, hosted by Kirstin F. Warner and crew. On November 9, 2011, the beginning of the meeting started with a tour of the IHD small-scale safety and thermal testing capabilities along with some other analytic testing and evaluation facilities. Following the tour, the team met to discuss some issues that have come about in the testing of liquid materials in the Proficiency Test. On the second day, the team met with the sponsor to review past performance, assess current status and develop future plans for FY 11 and possibly beyond. The sponsor also gave updates on current program issues and discussed deliverables for FY 11.

2 RESULTS

The Agenda for the meeting is listed in Appendix A. The results of the meeting will be listed in the same order as in the Agenda.

2.1 Tour of IHD Facilities

The core small-scale safety and thermal testing facilities at IHD were toured with discussions ensuing. In addition, micro calorimetry, ARC and isothermal reactivity testing were toured. At the drop hammer facility, the issue of how to test liquids became the center of the discussion. IHD presented their methodology that uses a modified liquid cell—the liquid cavity. This design is shown in Figure 1.

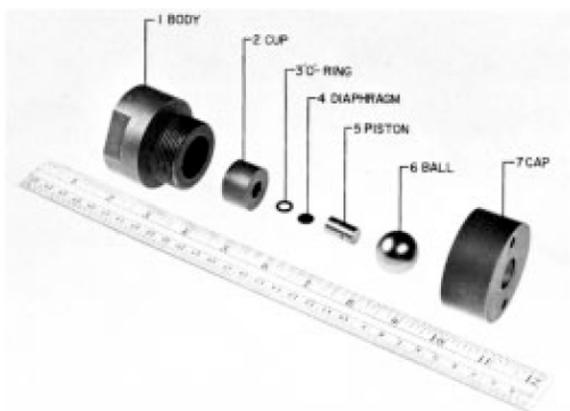


Figure 1. Liquid Cavity used for Drop Hammer Testing of Liquids.

This design comes from a 1960 JANNAF report. There was much interest in this design, so an action item was created to either find or make a drawing that could be shared among the team. Subsequently, Jason was able to find the original drawings and distributed them among the team on the following day.

Current liquid testing methods—Further discussion about how liquid testing is to proceed for the Proficiency Test proved very fruitful. In previous IDCA meetings and teleconference calls, the subject of drop hammer testing has been mostly focused on sand paper grit size for solid materials. The team has not discussed in detail how liquids should be tested for the Proficiency test. For liquid testing, LANL and LLNL use a greased anvil and no sandpaper, IHD uses the liquid cavity device except for the HP/flour mixture for which they used sandpaper, Tyndall uses greased anvil with an O-ring. These de-

scriptions represent three levels of confinement that are hardly uniform methods. As well, cursory examination of some results from LANL, LLNL, and IHD show the results are hardly uniform.

Revision of liquid testing methods—After a reasonable discussion, the team decided on some modifications, but, because much of the testing has already been done, retesting was decided on. For now, IHD will test using the cavity device as well as the greased anvil. Everyone else will run both greased anvil and sandpaper tests when appropriate. LANL and LLNL can go back and redo some of these tests. It was noted that some of the materials were not really liquids, but gooey and sloppy mixtures. The standard approach to these was to use sandpaper. *Action item: IHD will perform liquid testing using cavity device and greased anvil. Action item: LANL and LLNL will chose some materials to redo with sandpaper. Action item: Mary and Geoff volunteered Daniel Preston to write up his liquid test procedure; everyone will critique this method and comment how they do it. Action item: LLNL will perform DH tests on HP/Flour, HP/Cumin with 180-grit sand paper.*

New liquid testing standard—Examining the first draft of the Test Guide showed that liquid test standards have been used by LANL and LLNL—FEFO (1,1-[methylenebis(oxy)]-bis-[2- fluoro-2,2-dinitroethane) and TMETN (Trimethylolethane trinitrate, $\text{CH}_3\text{-C}(\text{CH}_2\text{-O-NO}_2)_3$). These standards are explosives and need special handling to distribute. Practically speaking, it is unlikely that these could be distributed in time for the end of the Proficiency test, so the team decided on a new standard—*n*-propyl nitrate. This material can be obtained as a regular chemical and could be distributed to the entire team fairly quickly. *Action item: Mary will look into ordering n-propyl nitrate for everyone (drop ship from vendor on Geoff's pcard?); need to determine how many vials of n-propyl nitrate to buy for each lab.*

Table 1. Drop Hammer (DH₅₀, cm) test results at different striker weights

TMETN (1.0-kg striker LLNL)	14
FEFO (1.0-kg striker LLNL)	32
PETN (1.0-kg striker LLNL)	10
HMX (1.0-kg striker LLNL)	23
PETN (2.5-kg striker LLNL)	15
HMX (2.5-kg striker LLNL)	32
TATP (2.5-kg striker LLNL)	11
HMTD (2.5-kg striker LLNL)	10
PETN (2.5-kg striker LANL)	13
HMX (2.5-kg striker LANL)	28
TMETN (2.5-kg striker LANL)	47
TATP (2.5-kg striker LANL)	6.5
HMTD (2.5-kg striker LANL)	4.2
HP (90)/NM 48.1/51.9 (1.0-kg striker LLNL)	33
HP (90)/NM 48.1/51.9 (1.0-kg striker LLNL)	31
HP (90)/NM 48.1/51.9 (1.0-kg striker LLNL)	27
HP (90)/NM 48.1/51.9 (2.5-kg striker LANL)	8.6
HP (90)/NM 48.1/51.9 (2.5-kg striker LANL)	8.8
HP (90)/NM 48.1/51.9 (2.5-kg striker LANL)	10.5

Striker weight discussions—Table 1 shows some DH50 test data from the first draft of the compendium and some recent Proficiency Test results. LLNL results on liquids were from tests that were done with

a 1.0-kg striker. LANL, Tyndall and IHD use 2.5-kg strikers. The data compares the performance of the same or similar materials when the test uses a 2.5-kg striker or a 1.0-kg striker. For standard military materials, such as PETN, the data from the use of the 1.0-kg striker shows a more sensitive material than the corresponding data from the use of the 2.5-kg striker. However, for the data for the HP 90/NM using the two different strikers, the 2.5-kg striker seems to indicate a much more sensitive material compared to the data on the same material obtained using the 1.0 kg-striker.

These results started a detailed discussion on the effects of different striker weights to answer the question can the data from a 1.0-kg striker be generalized with data from a 2.5-kg striker weight. A path to eliminate the issue of different striker weight is to make the striker weight uniform. However, LLNL is not sure that this can be done on the LLNL system. LLNL uses a 1-kg striker for liquids because of experimental configuration has not been modified to hold the striker offset with a 2.5-kg striker. With grease holding the liquid sample, the striker is not placed directly on the sample, but is a millimeter or so above (as not to squish the grease until the measurement). The other participants have equipment that has been modified to be able to hold this offset. The equipment at LLNL does not have this modification. *Action item: LLNL will see if there is a method to hold the 2.5-kg striker in position. Action item: LLNL will measure DH on RDX with a 1.0-kg striker. Action item: LANL will share their magnet technique with LLNL. Action item: LANL and Tyndall say they have a 1.0-kg striker weight, and they will try a couple of materials and see what happens.*

Friction Testing—LLNL data almost always indicates a material is more friction stable than data on the same material taken by the other laboratories. The reasons are still unknown. However, the team is considering that it might be due to the operators interpreting what a positive test is (not hearing the same). The LLNL friction testing equipment is in a closed box with a sucker hose (used to abate gases formed during the test). The equipment at the other labs have some shielding, but nothing as extreme as this. No resolution on the issue, but the team is going to try things related to the protective outer covering of the equipment. *Action item: LLNL will look for a way to test where the cover does not interfere (may be special cases approved by the safety people). Action item: LANL can look for a box to cover the BAM apparatus and try some retesting. Action item: Everyone can send in pictures of their set up with shielding in place.*

ESD Testing—Standardization here is also an issue, although the results for ESD testing (when LLNL custom built system is not included) results on the same material are fairly consistent among the participants even though there are some hardware differences. LANL uses brad nails from the hardware store. IHD uses Pfanstiehl phonograph needles. Also, after discussions at the SMS meeting, there is some concern about the calibration of the ESD equipment (capacitors, wiring, resistors, etc.). This will have impact if the IDCA plans to be a participant in the SMS round robin. *Action item: Mary and Geoff have volunteered Daniel Preston can distribute the info on the Spark tester probe that he uses for calibration.* LLNL now has a new ESD from SMS. Because KP/Al seems so sensitive, LLNL will retest KP/Al with the new spark tester. *Action item: ESD tests on KP/Al with LLNL new spark tester.*

2.2 Update of the IDCA

On November 10, 2011, the team met with the sponsor and the lead off presentation was an update for the program review on the IDCA. The presentation is attached as Appendix B.

Proficiency Test—Briefly, for collecting SSST test data, LANL and LLNL have completed all the testing and issued data reports. IHD is about 2/3 of the way through and is still measuring data on some mate-

rials. IHD expects to be finished by the end of December, 2011. AFRL is in the process of issuing some reports on data already taken and revising others, but is still waiting for funding to sort out.

Reports and Presentations—The IDCA has issued the following: 12 Analysis Reports that compares SSST data among the participants for each material, summaries results compared to military explosives standards, compares average SSST values to other sources, and discusses methods and IDCA issues; 70 Data Reports of data and supporting information on materials that have been tested; 8 Presentations (outside of the IDCA), 3 to TSWG International HME meeting, 1 to DOE, and 4 to outside open venues. **Action item: Any extra work that is done by anyone should be written into a report or memo with tracking number. This is work in addition to the data collection reports.**

2.3 DHS SSST Test Guide

Compilation of the DHS SSST Test Guide—LANL and LLNL will be the main drivers this year for the Testing Guide and will also do some retesting while IHD and Tyndall will finish testing the materials in the Proficiency Test. **Action item: Peter is heading up the LLNL effort for the test guide. Action item: Mary, Geoff, Daniel, etc., will lead the LANL effort.** The Proficiency Test data will be the first data that will be included in the Test Guide. After the format, storage and access are determined, the Guide will start including other DHS program data (NEXESS, IDD, Formulary), DOE and DoD historical data (IHD, LANL, and LLNL have large databases of historical data), and potentially data from international sources. The real challenge is how to assure any test results are worthy of inclusion. **Action item: Tim will ramrod the effort of High fidelity test data should include HMX and link to historical databases. Action item: Add n-propyl nitrate as liquid standard for our testing.**

Access to DHS SSST Test Guide—Access to the data will be one of the hardest issues to solve. The IDD server seems like a good place to start, but may not be the long-term solution. It has access control and has a staff that is used to working on large data sets. **Action item: John will check on arranging access to IDD server and putting the Guide on it. Action item: Laura and Greg can talk to Harry about putting the Guide on the IDD server.** DTIC is another place to put IDCA reports, at least, for other people to have access. **Action item: Laura will check with DHS to see if it is OK to put this on DTIC.**

2.4 International Round Robin

The IDCA will be participating in the International Round Robin (IRR) organized by the Technical Support Working Group (TSWG). Elaine Child temporarily replaces Shabana Haque. The IRR is broken into two parts—Phase 1, a paper comparison of testing methods, and Phase 2, an experimental testing of selected materials. IDCA will partake in Phase 1 and partially in Phase 2 (SSST testing only at this time).

TSWG has set up a website for the IRR. Laura tried to get on and found it was not trivial. JGR could not find it. **Action item: JGR contact Beth Obregon to find out the link and try to get everyone set up on it.** Phase 1 is not complete. Several members have not submitted their testing information, including most, if not all, the IDCA members. **Action item: Everyone can release scale up methods/data to the TSWG website (when we find it).** Phase 2 has yet to commence. The phase is testing at SSST testing levels followed by scale-up. Three materials have been tentatively identified, RDX, PLX, and HP/fuel. The fuel may turn out to be European methylated spirits, but that has not been decided. There also seems to be a hold up on the distribution of RDX, even domestically. **Action item: Laura will tell TSWG we need the RDX soon (by Feb?) or will be unable to participate. Action item: Laura will ask if using European RDX would be easier.** IDCA labs have the go-ahead to do IRR testing with current funds

if materials arrive. Also, because the HP will be purchased in the country that it is used in, analysis methods need to be shared. *Action item: All—IDCA is supposed to have document HP procedures for checking concentration and purity. Did that happen?* The IRR may not work out, so do not spend resources helping them get materials.

2.5 KClO₃/icing sugar DSC issues

When putting all the measurement data together on the KClO₃/icing sugar mixtures, the DSC data stood out—every laboratory reported different results. Figure 2 clearly shows these differences. Depending upon how the sample is prepared, the DSC will give you the low temperature exothermic event as well as higher temperature exothermic events.

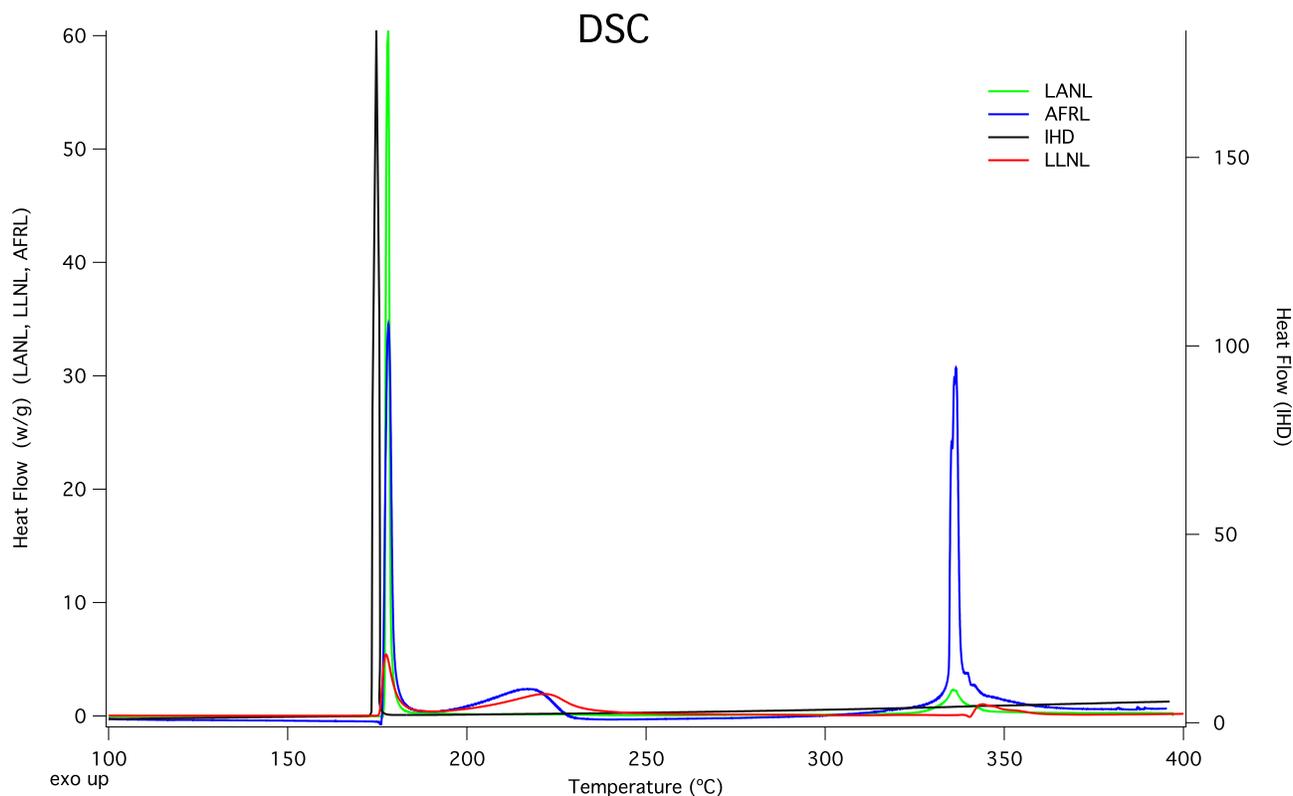


Figure 2. DSC of KClO₃/icing sugar

Reasons for inconsistent results—This prompted the team to undergo extensive discussions and extra efforts to understand the reason. Mary did some additional optical work, photographing the DSC pan before and after the DSC under magnification and at different sample sizes. She was asked to share her thoughts on the subject. That presentation is Appendix C. Mary presented photos of the materials to explain why the inconsistent results. It should be noted that all laboratories have seen all four of the profiles in Figure 2 at one time or another. Her premise is that the components can have trouble mixing when there are very small amounts on the sample pan. The low temperature peak is due to KC reacting with the sugar when the sugar melts, the middle peak is probably due to some carbonization of sugar, and the high temperature peak is due to the KC melting and contacting any residual fuel and then reacting. The team has plenty of DSC data, including with other sugars, which it would be good to com-

pare. *Action item: All—dig up DSCs of KC with different sugars. Action item: Mary will send out the post-test pictures.*

Proving heterogeneity—Tim would like to see about phase contrast images of materials in pans to check homogeneity. Also perhaps elemental analysis of different samplings could prove the heterogeneity. *Action item: Tim will come up with an experimental plan to prove this for a future teleconference call discussion.*

2.6 A Plan for Evaluating and Comparing Data Sets in the IDCA Collaboration

Geoff was asked by the team to come up with some nifty way to evaluate the inter- and intra-lab performance during the Proficiency test. Prior to this time JGR evaluated the performance by calculating the mean, deviation of the mean, and relative deviation of the mean for comparable data sets. However, knowing the brilliance of Geoff's understanding of statistical analysis (there are three kinds of lies: lies, damn lies, and statistics), Geoff was asked to address this issue in more detail. His presentation is Appendix D. His suggested approach is the following. Standard statistical approaches will let us evaluate results—report expected error in results at various confidence levels; report confidence intervals of mean values; compare multiple results simultaneously. New information obtained to help with Bruceton Testing Evaluation—additional functions—let us evaluate expected error in mean and std. dev.; goes into detail on multiple test comparison; procedures violate assumptions that can be evaluated for additional error with simulations. *Action item: Geoff will test this approach for a first material.*

2.7 SNL plan for IDCA for FY 11

Jason, being new to the IDCA, gave an informal presentation of a plan for the upcoming FY. He will be setting up a testing lab in a fully environmentally controlled transportainer. Jason has BAM friction and will get drop hammer when there is a place to put it, and will obtain Leroy's spark tester. At this time, the only thing missing is a DSC. He may work with EMCF for DSC, but this is difficult.

2.8 SMS Explosives Testing User Group meeting update

Many of the IDCA members went to this meeting in October in Park City, UT—Mary, Geoff, Jason, Jose, Peter, and JGR. Geoff presented some statistics and modeling, Jason presented his thesis work, and JGR presented IDCA Proficiency Test results to the group. There were many esoteric discussions about safety testing.

High Speed Camera—One item that got some of the team's attention was the use of a high-speed camera for positive/negative detection in SSST testing. This type of camera was shown to greatly assist in determining go/no go (it was not perfect and required some interpretation, but removed a lot of the uncertainty in current methods). Peter suggested having some support from DHS or others to help IDCA participants get funding for camera/software. *Action item: Laura/John can draft paragraph to show support for this capability.*

SMS also is planning a round robin. This RR would be different than the IDCA Proficiency Test because it would concentrate on calibration of equipment (by electronic means) and standardizing detection, such as using the high-speed camera. This RR will not be for some time, probably a year or so. At this time, Laura said it would be fine to participate if we could find funding. *Action item: Laura/John can draft paragraph to show support for the SMS round robin.*

Participation in the IDCA by Tooele Army Base—Roger Hale at Tooele interested in participating in the IDCA Proficiency test (relates to their program with Dugway). JGR said they would have to follow IDCA procedures if they are to participate—Tooele would have to use IDCA materials (if possible to get), and drying, screening methods, etc. Tooele does UN testing already. There is low liability associated with their participation (especially when no funding will be transferred). Getting the Army involved might allow IDCA an easier transition from DHS to DoD funding later or may also help with getting TSWG involved. We would have to ship materials to Tooele after John checks to see their requirements. **Action item: JGR will contact Roger with the go ahead from DHS. Action item: John can give Laura a paragraph on how the Tooele interaction would affect the IDCA.**

2.9 Other potential research topics

Appendix B shows a list of potential research topics taken from IDCA Analysis Report 008 (2010). This list has been kicked around for over a year and is sort of the wish list derived from experimental observations that pop up occasionally when the team has time to think about gaps in testing knowledge. This list combined with the issues that have come up during the testing phase—sand paper grit size, thermal behavior of KC/fuel materials, thermal issues with volatile samples, aging of materials, size effects—set the basis for future and side research efforts in the program. This discussion was open forum for anyone who wanted to share ideas and make comments.

IHD testing and support—Kirstin started with a few comments. This presentation is shown in Appendix E. One of the main points brought out were the issues about testing of HP/fuel mixtures, using the cavity cell (*vide supra*) and the need to possibly standardize liquid testing methods. This has been discussed above in detail and the team has reached at least a starting point. Kirstin also presented some Accelerated Reaction Calorimetry (ARC) testing her group had done on some of the Proficiency test materials, such as KC/sugar, gunpowder, UNi/Al/S. Table 2 shows that some materials had very low thermal onsets. This highlighted the thermal sensitivity of certain mixtures, such as the HP/Sugar as well as the effects of S in mixtures.

Table 2. Accelerated Reaction Calorimetry Results for Selected HMEs

Sample ID	Mass(g)	Exo. Onset, °C	Exo. Max, °C	Self-Heating Max, °C/Min
Bullseye Gunpowder	0.1015	145	195	216
AN/Gunpowder	0.1008	140	175	483
AN (60C-screen)	0.2522	260	275	0.05
KC/Sugar	0.1012	130	200	507
UN/Al	0.1001	116	128	0.38
UN/Al/S	0.1099	85	115	0.66
RDX	0.1004	196	271	387

International Round Robin—The IDCA can participate in IRR if RDX, at least, is distributed by February, 2012. Otherwise, Laura will pull the plug. **Action item: Mary will get the Methods report finished. This will help us with the IRR methods analysis.**

BAM Friction testing plate replacement—Robocasting Enterprises in New Mexico is developing newly designed ceramic BAM friction testing plates. These plates are made to higher precision than the plates provided by BAM. These may have better consistency than current plates and may eliminate some of the variation seen in the BAM testing results. Mary can measure their roughness beforehand with the LANL profilometer. Also, LANL may be able to put together larger effort in 2012 with small business program collaboration. **Action item: *Mary will send out samples to the team later if they work out.***

Fast screening method by DSC for thermal stability—This topic has been kicked around for a while. Some of the DSC programs use global kinetics to do assessment of thermal stability. Also, kinetics can be done by DSC (ASTM E698 for example). Fast is maybe a bad word choice. Want something that uses DSC to screen materials because DSC is such a common piece of equipment, can be fast, and uses a very small sample size. This would replace larger thermal stability tests that generally require longer times and much more material. Can look for correlations between DSC and other tests that directly tell larger scale stability (ARC, ODTX). Liquids are more reactive and may be more appropriate for this with mixtures being hardest materials to study. **Action item: *Labs should provide some input on how to do this for now and maybe start small study later (JGR to head collection of ideas?).***

Aging studies—Even at ambient temperature, aging can happen and play role in sensitivity. For example, LLNL has seen NG/NC mixtures became very sensitive after day 4. Gas bubbles formed which give sites for reactivity to impact. Others have seen HP/flour and HP/cumin to be more sensitive over time. NEXESS is doing some aging studies already. Should the IDCA do other mixtures? Maybe look at solid-solid mixtures that differ at each lab (KP/Al with sandpaper variation). KC/dodecane may be good since it includes solid-liquid mixture. KC/sugar held at 40 or 50°C in micro-calorimeter might be good as well. **Action item: *Peter will measure the aging effect on KC/Dodecane on impact sensitivity (2 hrs., overnight, 4 days, and 1 week).*** **Action item: *Kirstin will measure the aging effects on HP/Flour (2hrs, overnight, 4days, and 1 week) using microcalorimetry.***

Sandpaper studies—The IDCA has certainly seen differences in impact testing results with different sandpapers, depending upon the material studied. There has been some characterization by SEM but the results somewhat contradict the expected trend. LLNL has asked modelers to look at this. What else can be done to bring this to closure? A factorial design of experiment is proposed, probably 3 x 3 design, with the following: particle size control on powders by sieving; well characterized sandpaper of various grit sizes; selected combinations of mismatching sandpaper grit size with particle size; response factor would be drop weight height. Mixtures may be hard to control particle size (at least we could do particle size measurements). Coarse vs. fine PETN or HMX would show different particle sizes respond on single sand paper. Could make bimodal distributions as well to see if trend applies in mixtures. **Action item: *We need someone to champion this—any interest? Peter and Dan can send sandpaper to Mary.***

2.10 Rebaselining

In Appendix B Slide 9, the upcoming activities for the IDCA are listed with new deliverable dates. Greg has taken those and put them in a GANTT chart on Slide 34. The deliverables include due dates completing the analysis reports, due dates for parts of the Test guide, participation in the International Round Robin, and program review meetings. Not included are deliverables for any topics discussed in this meeting other than those just stated. IHD expects to be done with Proficiency testing in December. Because the status of the International Round Robin is not known for sure, Greg will readdress Gantt

charts in Feb if no IRR samples are available. Some slack is in the schedule but expect to be done by Sept 2012. The schedule shows 2 weeks per analysis report and JGR concurs. Because funding came so late, Greg will get NCEs for everyone who needs them. *Action item: **Greg will get no cost extensions in early 2012 so that no one has to stop work.*** Keep funding levels and remaining work in mind when considering travel.

2.11 Jason's Thesis Project

Jason was kind enough to brief us on his thesis work, "An Investigation of Modifications to the Type-12 Impact Sensitivity Test Apparatus for Explosives," that he did at EMRTC. This is shown in Appendix F. The work shows modifications in the Type 12 test based on strain gage measurements during impact. Striker had strong confinement and was sometimes difficult to remove. HDPE bushing was adopted by EMRTC after Jason's work. HDPE bushing cut the standard deviation in half. Heavens and Field 1974 Proc. Roy. Soc. Vol 338 pg 77-93 – release due to mechanical failure.

2.12 Future IDCA team contact

JGR will distribute meeting materials as soon as possible (if you are reading this, then he has). Laura would like a teleconference call the week after Thanksgiving to go over action items. The IDCA should try to have meetings more than once per year. Suggestions for next quarterly meetings: Tyndall in February some time, LLNL in summer, and LANL/SNL in early Oct (LLNL will fill in if Tyndall can not do it).

2.13 Detailed planning of the DHS HME SSST Test Guide structure

JGR lead a living discussion on what the Test Guide should look like—what should be included or requested from contributors.

General comments—May be useful to show graphs with error bars as well as tables. Organize guide into classes of materials, or by physical forms (good question).

Impact Testing—Information should include: Test date, formulation ratios, pressed vs. powder, physical appearance, mass of striker, type of sandpaper, mass of drop weight, test method, equipment description, detection method, number of drops in Bruceton, method of analysis, sample preparation and conditioning (dried, desiccated, shriveled), mixing conditions, age of sample, compositional analysis, purity, grade, particle size, batch number, linear vs. log steps in Bruceton. Separate section for historical data.

BAM Friction Testing—Information should include: equipment details (box, fan, lights), Go–No Go criteria, calibration (good question), equipment age, standards, verification frequency. Separate section for historical data.

ABL Friction Testing—Information should include: conditions of plate, wheel (roughness, hardness), plate velocity, retightening frequency. Separate section for historical data.

ESD Testing—Information should include: tape use, needles, voltage, capacitance, resistance, fixed needle vs. approaching, gap size. Separate section for historical data.

DSC Testing—Information should include: method, ramp rate, pan type, purge gas, calibration type, method of analysis, baseline interpolation type, modulation, onset determination. Separate section for historical data.

Other SSST Testing—Information can include, but not necessary to include: HME solubility, thermal tests (ARC, ODTX, etc), Henkin, isothermal DTA/TGA, Vacuum thermal stability, Chemical reactivity test, possibly test results from European tests if available.

Things not to mention—Do not try to assess hazard rating of materials. Want to avoid liability in the document.

3 ACKNOWLEDGMENT

This work was performed by the Integrated Data Collection Analysis (IDCA) Program, a five-lab effort supported by Los Alamos National Laboratory, Lawrence Livermore National Laboratory, Sandia National Laboratories, the Air Force Research Laboratory, and Indian Head Division, Naval Surface Warfare under sponsorship of the US Department of Homeland Security, Office of Science and Technology, Energetics Division. Los Alamos National Laboratory is operated by Los Alamos National Security, LLC, for the United States Department of Energy under Contract DE-AC52-06NA25396. Sandia is a multi-program laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy's National Nuclear Security Administration under Contract DE-AC04-94AL85000. This work was performed under the auspices of the U. S. Department of Energy by Lawrence Livermore National Laboratory under Contract DE-AC52-07NA27344. The Air Force Research Laboratory (AFRL/RXQF) and Indian Head Division, Naval Surface Warfare (NSWC IHD) also performed work in support of this effort. The work performed by AFRL/RXQF and NSWC IHD is under sponsorship of the US Department of Homeland Security, Office of Science and Technology, Energetics Division.

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Appendix A—Agenda for meeting

IDCA Quarterly Annual meeting, November 9-10, 2011 Naval Surface Warfare Center, Indian Head Division

November 9, 2011 Tour of NSWC-IHD small scale safety and thermal testing

1:00 pm to 1:15 pm	Pass office for badging	Everyone
1:15 pm to 4:00 pm	Tour of IHD SSST and Thermal testing and discussion of technical issues	Kirstin F. Warner Dan Remmers
5:00 pm to 7:30 pm	Dinner at Casey Jones	Optional

November 10, 2011 Review and Discussion Building 3133

7:45 am to 8:30 am	Pass office for badging and travel to Bldg. 3133.	Everyone
8:30 am to 8:35 am	Welcome, pastries, and evacuation plans	Kirstin F. Warner
8:35 am to 8:45 am	Words of wisdom and DHS update	Laura J. Parker
8:45 am to 9:30 am	IDCA Update and recent activities	John G. Reynolds
9:30 am to 10:30 am	Planning the DHS Test Guide	John G. Reynolds
10:30 am to 10:45 am	Break	Everyone
10:45 am to 11:00 am	International Round Robin	John G. Reynolds
11:00 am to 11:30 am	KC/Fuel thermal study	Mary M. Sandstrom
11:30 am to 12:00 noon	Statistics and modeling	Geoffrey W. Brown
12:00 pm to 12:15 pm	SNL plans for FY 11 (begin working lunch)	Jason J. Phillips
12:15 pm to 12:30 pm	ETUG meeting and how to integrate with SMS Round Robin (finish working lunch)	John G. Reynolds
12:30 pm to 1:15 pm	Other research topics	Everyone
1:15 pm to 2:30 pm	Re-baseline	Greg F. Struba
2:30 pm to 2:40 pm	Report release guidance	Laura J. Parker
2:20 pm to 3:00 pm	Jason's thesis work	Jason J. Phillips
3:00 pm to 4:00 pm	Discussion and Wrap-up	Everyone

Host:

Kirstin F. Warner, Ph.D.

Research, Development, Test and Evaluation Indian Head Division, NSWC

4104 Evans Way Suite 102

Indian Head, MD 20640-5102

Phone: 301-744-4525

Fax: 301-744-4445

Email: kirstin.warner@navy.mil

What I envision on how we will cover the topics

Welcome, pastries, and evacuation plans—John (and whoever else wants to) will bring coffee and donuts and junk); Kirstin will be official host and make sure we don't get hurt
Words of wisdom and DHS update –Laura will give us any update on the status of IDCA for the upcoming years, DHS for the upcoming years, and any official reported we need to do
IDCA Update and recent activities—JGR will review exactly where we are; recent activities (such as presentations and reports), and minimum IDCA efforts for this year
Planning the DHS Test Guide—JGR will lead a discussion on the structure; content, division of labor, where the initial guide will be housed.
Break—if we have time
International Round Robin—JGR will update current status and what is expected for participation
KC/Fuel thermal study—Mary will detail her before and after sample study for KC/sugar and all will discuss writing a report/paper on the over all effort
Statistics and modeling—Geoff will talk about his modeling and JGR will introduce the concept of how to statistically report intra- and inter-laboratory comparisons
SNL plans for FY 11 (begin working lunch)—Jason will go over new activity planned at SNL in the IDCA
ETUG meeting and how to integrate with SMS Round Robin (finish working lunch)—general comments from several of us on the SMS meeting, discuss how we might or want to be more involved, including with the SMS round robin that was proposed
Other research topics—Peter would like to discuss aging effects; Kirstin wants to discuss something, JGR will bring the list of topic from our summary report, and anything else can be proposed
Re-baseline—Greg will show the GANTT chart for this year from initial planning, and lead the discussion to revise the plan
Report release guidance—Laura will discipline us into turning in all reports and presentations in a timely manner, explain DHS guidelines, why we can not use DHS logos any more, DTIC and the LLNL HE Reference Guide, a distribution system upon request
Jason's thesis work—Jason did some very nice DH work for his thesis which he will share
Discussion and Wrap-up (for those who are left)—How about that logo (looks like JGR made it reminiscent of Cuba)? future meetings for the IDCA

Appendix B. Presentation—IDCA Update and Recent Activities



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Schedule for 11.10.11

7:45 am to 8:30 am	Pass office for badging and travel to Bldg. 3133.	Everyone
8:30 am to 8:35 am	Welcome, pastries, and evacuation plans	Kirstin F. Warner
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3:00 pm to 4:00 pm	Discussion and Wrap-up	Everyone



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IDCA Update and Recent Activities

John G. Reynolds, Lawrence Livermore National Laboratory, Livermore, CA USA
Mary M. Sandstrom, Los Alamos National Laboratory, Los Alamos, NM USA
Geoffrey W. Brown, Los Alamos National Laboratory, Los Alamos, NM USA
Kirstin F. Warner, Naval Surface Warfare Center, Indian Head, MD USA
LeRoy L. Whinnery, Sandia National Laboratories, Livermore, CA USA
Jason J. Phillips, Sandia National Laboratories, Albuquerque, NM USA
Timothy J. Shelley, Air Force Research Laboratory, Tyndall AFB, FL USA
Jose A. Reyes, Applied Research Associates, Tyndall AFB, FL USA
Peter C. Hsu, Lawrence Livermore National Laboratory, Livermore, CA USA

November 10, 2011, Naval Surface Warfare Center, Indian Head Division

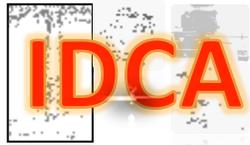


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Topics to cover

- **Overall goals**
- **Current status**
 - Proficiency Test Data report status
 - IDCA Report and Presentation List
- **Moving Forward**
 - FY11 planned activities
 - Planned presentations and publications
- **Research Topics**
- **DHS HME SSST Test Guide**
- **International Round Robin**



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IDCA Goals

- **Collect SSST test data of improvised explosives or HMEs**
 - Proficiency Test
 - International Round Robin
 - Exogenous Data
- **Determine if SSST test protocols need to be modified for handling HMEs vs. standard explosives**
 - Determine intra-laboratory variations in test results
 - Determine inter-laboratory variations in test results
- **Share and distribute SSST testing data that will help the community safely handle these materials**
 - Proficiency test
 - Methodology reviews
 - HME SSST testing guide



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PT—Data Report Status

Material	LLNL	LANL	IHD	Tyndall
RDX	Revised	Revised	Revised	Revised
KC/Sugar (100)	Revised	Revised	Revised	NA
KC/Sugar (received)	Revised	Revised	Revised	Received
KC/Dodecane	Revised	Revised	Revised	NA
KP/Al	Revised	Revised	Received	Received
KP/Charcoal	Revised	Received	Received	Need Report
KP/Dodecane	Revised	Received	Received	Need Report
SC/Sugar	Revised	Received	Received	Need Report
RDX 2nd Time	Received	Received	Need Report	Need Report
AN	Received	Received	Received	Need Report
AN/Gunpowder	Received	Received	Need Report	Need Report
Gunpowder	Received	Received	Received	Need Report
HP 70%/Cumin	Received	Received	Need Report	Need Report
HP 70%/Flour	Received	Received	Need Report	Need Report
HP 70%/Glycerine	Received	Received	Need Report	Need Report
RDX 3rd Time	Received	Received	Need Report	Need Report
PETN	Received	Received	Received	Need Report
HP 90%/NM	Received	Received	Need Report	Need Report
UNi/Al	Received	Received	Need Report	Need Report
UNi/Al/S	Received	Received	Need Report	Need Report
HMX	Received	Received	Received	Need Report
RDX 4th Time	Received	Received	Need Report	Need Report
I-RR Material #1				
I-RR Material #2				
I-RR Material #3				



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IDCA Report Status

- **12 Analysis Reports**
 - compares SSST data among the participants for each material
 - summaries results compared to military explosives standards
 - compares average SSST values to other sources
- **69 Data Reports—full SSST data reports**
 - from each participant for each material
 - primarily LANL, LLNL, and IHD
- **8 Presentations (outside of the IDCA)**
 - 3 to TSWG International HME meeting
 - 1 to DOE
 - 4 to outside interests



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Moving Forward

- **FY11 funding for IDCA**
 - LANL, SNL, IHD and LLNL have FY11 funding
 - AFRL will have FY12 funding
- **Start the DHS HME SSST Testing Data Guide (new name for Compendium)**
- **Completing FY10 testing report (write up is dependent on data reports)**
 - LANL and LLNL complete, all data reports handed in
 - IHD about ½ done
 - AFRL is now forging ahead
 - SNL has a new future
- **International Round Robin**
 - RDX should be distributed within a couple months (internationally)
 - First testing scheduled to be completed for 2012 International meeting?
- **Testing issues**
 - Sand paper grit size
 - Thermal behavior of KC/fuel materials
 - Thermal issues with volatile samples
 - Aging of materials
 - Size effects

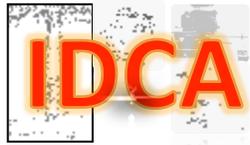


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Proposed Public Releases

- **Presentations**
 - SMS 2011 meeting in October 2011
 - IEEE HST meeting in November 2011
- **Manuscripts**
 - on results comparisons
 - resolution of testing issues
 - thermal issues with KC/fuel mixtures
 - thermal issue with volatile fuels
 - sandpaper grit size
 - sample preparation and handling (pretreatment and methods)



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Proposed deliverables

Deliverable	Due Date
Program Review meeting	11.10.11
KP/Al Analysis Report	11.15.11
SSST Testing Guide plan	11.17.11
KP/Charcoal Analysis Report	12.06.11
KP/Dodecane Analysis Report	12.20.11
SC/Sugar Analysis Report	01.10.12
AN Analysis Report	01.24.12
RDX Standard Run #2 Report	02.07.12
AN/Gunpowder Analysis Report	02.21.12
HP/Cumin Analysis Report	03.06.12
HP/Flour Analysis Report	03.20.12
Program Review meeting	03.14.12
HP/Glycerin Analysis Report	04.03.12
Gunpowder Analysis Report	04.17.12
RDX Standard Run #3 Report	05.01.12
International HME Meeting Program Update	05.01.12
International Round Robin plan	05.01.12
PETN Analysis Report	05.15.12
HP/Nitromethane Analysis Report	05.29.12
SSST Testing Guide first draft	06.04.11
UNi/Al Analysis Report	06.12.12
UNi/Al/S Analysis Report	06.26.12
HMX Analysis Report	07.10.12
RDX Standard Run #4 Report	07.24.12
Final Analysis of Proficiency Test Report	08.24.12
International Round Robin IDCA Comparison reports	09.03.12
SSST Testing Guide Interim Draft	09.12.12
FY 11 Program Review	09.19.12



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FY11 Topics

- **Continuation of the IDCA program for FY 2011**
 - Restructure the SSST Compendium
 - Population of the SSST Compendium
 - Reacquiring Proficiency Test Data with Modified Methods
- **Prioritization of future topics of interest to the IDCA**
 - Participation in the International Round Robin SSST testing
 - Developing a fast screening method by DSC for thermal analysis (relate to ARC, ODTX, APTAC, isothermal DSC)
 - Impact of aging of solid-solid, liquid-liquid and solid-liquid mixtures on testing sensitivity
 - Expansion of camera approach to SSST testing
 - Effects of impure source materials on testing
 - Effects of porosity of solid-solid and solid-liquid mixtures on sensitivity
 - Optimizing sandpaper for impact testing (design of experiments)
 - Developing methods so ABL vs. BAM data from different methods can be compared
 - Additional HME threats that challenge SSST Testing

From Integrated Data Collection Analysis (IDCA) Program—FY2011 Project Descriptions, IDCA Program Analysis Report 005, November 13, 2010.



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DHS HME SSST Test Guide Year 2011 Plan



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DHS HME SSST Test Guide

- What data should be included
 - Proficiency test data
 - Individual participant data
 - Historical data
 - Outside source data (FBI, URI, Army, other)
 - International sources (Canada, UK, France, Aussie)
 - Commercial sources (SMS, SEC, ARA)
- What format should be used
 - DH, Friction (ABL and BAM), spark, DSC
 - Vacuum stability, chemical reactivity, Henkel, ODTX. ARC,
- Who does what
- What delivery mechanism can be used
 - Initial phase
 - Final phase
- How do we do updates
- When do we do updates



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DHS HME SSST Test Guide

- Will be the definitive reference for small scale safety testing of home made explosives
- Data provided is from reputable sources that have been screened for
 - methodology
 - equipment
 - procedures
- Comparable data on standard reference explosives
- Detailed description of the testing equipment and procedures used for measuring the safety data
- Available for all who are legitimately working in HMEs (including International Partners)



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Program Plan for Guide

Three Parts

- Proficiency test—standardization of testing of materials and interpretation of results
- Collection of data—sources that are screened to assure high quality, properly obtained data
- Dissemination of information—web based, easily updated, access controlled



Part 1—Proficiency test

Did we cover all the bases in the Proficiency Test?

- **High fidelity testing data**
 - High quality data in a comparable format that the IDCA agrees on
 - Appropriate reference standards are measured—RDX and PETN
- **Assured testing methods**
 - Equipment is well documented and calibrated—is it?
 - Measurement Methods are well documented—not yet!
 - Analysis methods are well documented—how do we do this?
- **Understand SSST testing as applied to HMEs**
 - Broad range of HMEs has been selected—are there additional ones that need to be included?
 - Challenges in measurements are being compared—are we doing this to the fullest extent?
- **Understand the significance of variability in measured values from each participant**
 - Process of doing this, Geoff is helping



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Part 2—Collection of Data

- **Current data sources—laboratories**
 - LLNL, LANL, NSWC IHD, AFRL Tyndall
- **Current data sources—programs**
 - NEXESS testing, NEXESS Formulary, IDD, Proficiency test
- **Potential future data sources**
 - International (Round Robin, Canada, UK, Aussies, le Frenchies)
 - URI, FBI(?), ARA(?), SMS(?), SEC (?)
 - Historical data from DOE and DoD laboratories
 - Other?



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- **Web based access**
 - IDD server
 - HE Reference Guide
 - TSWG HME Web site
 - DHS
- **Access controlled**
 - Foreign national access
 - No export controlled information
 - No ITAR information
- **Uni- to multi-lateral agreements (?)**
- **Updates**
 - Staff to enter in new data
 - Committee to review new data

Part 3—Access

How do we do this?



Example of Impact Data

90% H ₂ O ₂ , 90% concentration (10% H ₂ O)	>177
90% H ₂ O ₂ /isopropanol, 80%/20% mix by weight (8% H ₂ O)	31
90% H ₂ O ₂ /black pepper, 70%/30% mix by weight (7% H ₂ O)	61
90% H ₂ O ₂ /cumin, 80%/20% mix by weight (8% H ₂ O)	41
90% H ₂ O ₂ /flour, 70%/30% mix by weight (7% H ₂ O)	70
90% H ₂ O ₂ /glycerol, 75%/25% mix by weight (7.5% H ₂ O)	82
90% H ₂ O ₂ /nitromethane, 48.1%/51.9% mix by weight (4.8% H ₂ O)	30
90% H ₂ O ₂ /sucrose, 65%/35% mix by weight (6.5% H ₂ O)	84
90% H ₂ O ₂ /fructose, 65%/35% mix by weight (6.5% H ₂ O)	63
90% H ₂ O ₂ /tang, 65%/35% mix by weight, (6.5% H ₂ O)	56
TMETN (note 1.0 Kg striker)	14
FEFO (note 1.0 Kg striker)	32
PETN (note 1.0 Kg striker)	10
HMX (note 1.0 Kg striker)	23
PETN (note 2.5 Kg striker)	15
HMX (note 2.5 Kg striker)	32
TATP (note 2.5 Kg striker)	11
HMTD (note 2.5 Kg striker)	10

To paraphrase, this ain't going to work



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Information

What tests to include?

- **Impact Sensitivity**
- **Friction Sensitivity**
- **Electrostatic Discharge**
- **Differential Scanning Calorimetry (DSC)**
- **Other**
 - Vacuum Thermal Stability (VTS)
 - Chemical Reactivity Test (CRT)
 - ARC and APTAC
 - One Dimensional Time to Explosion (ODTX)
 - Henkel test

What other information to include?

- **Aging Studies**
- **Additional Information on Hazards**
- **Nomenclature**
- **Radiography**
 - Z-Effective
 - μ s



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- Beta copy delivered to Sponsor in 2009 as hard copy and e-file
- Format is being revised
- Data from LLNL and LANL only
- Includes impact, friction, ESD, DSC and limited other thermal tests
- Methods and procedures will be added
- Data on selected HP/fuels and UN/fuels
- Additional reference materials will be completed
- Additional data from LLNL, LANL, IH and Tyndall ready to be added

Status of Compendium

Compendium on Small Scale Safety Data for HMEs
February 3, 2009
Revised February 26, 2009
LLNL IMS Tracking number 379548

Small Scale Safety Data for Home Made Explosives

John G. Reynolds¹, Becky Olinger², Peter Hsu¹, Doug Bauer³,
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⁴Transportation Security Administration
⁵Booze Allen Hamilton

This compendium addresses small scale safety testing of home made explosives (HMEs) for reference purposes. The data provided here is from reputable sources that have been screened for methodology, equipment and procedures. Not only the data is included, but comparable data on standard reference explosives is given, as well as a description of the testing equipment and procedures used for measuring the safety data.

Currently, data shown is only from Los Alamos National Laboratory (LANL) and Lawrence Livermore National Laboratory (LLNL). These are the only two laboratories that have contributed complete description of their methods, procedures and equipment. As this compendium is developed, this list of contributors will increase. Data from published sources will be included if the data is properly described, performance of reference compounds is available, as well as comprehensive description of the testing methods and procedures is available.

It is important to note that this data is for reference purposes only. It is not a substitute for deriving proper safe handling procedures of explosive materials. One needs to have a well-vetted internal procedure that meets this goal.

This compendium will continue to grow as more data is added and more sources are included. These initial chapters were chosen because LLNL and LANL already have a data on hydrogen peroxide/fuels and urea nitrate from previous testing. Data for other solid oxidizer/fuel mixtures and other non-tradition explosives will be added.



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Division of Labor

- **Proficiency Test is partially done**
 - IHD, Tyndall and SNL should continue producing data on the original list
 - All should retest when necessary
 - LLNL and LANL will begin accumulating data for Guide
 - Methods and procedures need to be written up
- **Find volunteers to help with the following elements of the Guide and overall small scale testing of HME**
 - Peer Reviewers of Documentation
 - Contributors to Guide
 - Method development to incorporate data that is incomplete or collected by other means
 - Different Procedures, Equipment, Standards, etc . . .
 - Data from International Sources



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International Round Robin Update



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International Round Robin

- **Formed through the Technical Support Working Group (TSWG) HME Working Group**
- **New contact for lead (acting)—Elaine Child at CPNI (UK)**
- **Last Update February 23, 2011**
- **Aims of International Round Robin**
 - To develop best practice guidelines and methods for safe handling, manipulation and scale up of improvised and novel materials.
 - To share lessons learnt from previous testing of improvised materials and to establish an effective mechanism e.g. web forum or network to allow the facile exchange of such information to international partners, and co-workers working in this field.
 - To ensure that the International community recognises the importance of conducting medium scale and thermal tests before scaling up to large kilogram quantities. At the moment, not all countries conduct these tests - this is not good practice, particularly when dealing with notoriously unpredictable materials.]
 - To recommend and implement a suite of small scale, medium scale and thermal tests which has been agreed by all parties, which will assist the International Community in the safety and hazard assessment and preliminary characterisation of novel and improvised explosives, prior to scale up to larger charge sizes.

Goal: To protect against HME accidents for workers internationally



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IRR Proposed Plan

IRR will be done in two phases

- **Phase 1 is capture testing methodologies by all participants**
 - What are the testing procedures and methods employed to assess the viability and thermal characteristics of an HME on the small, medium, and large scale
 - How to disseminate the information
 - Decide on what testing will be mandatory and what will be optional
- **Phase 2 is testing at small and medium scale**
 - 70 % HP semi conductor grade with 95% Ethanol (Sigma-Aldrich?) 3 to 1 ratio
 - PLX (95% nitromethane and 5% ethylene diamine) source not decided
 - RDX Type II Class 5 from Holston (TSWG to provide)



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Action Items!!!!

IRR—Current Status

- **ACTION 1**—Shabana to circulate the original guidelines and presentation (**completed**).
- **ACTION 2**—All Round Robin (RR) participants to send shipping address for samples to Beth Obregon (TSWG). Tim Shelley to assist John Reynolds as nominated deputy US National Labs POC (**outstanding**).
- **ACTION 3**—TSWG to set-up subgroup on HME Forum which is accessible to RR participants only (**completed**).
- **ACTION 4**—All to post current testing procedures (see guidelines for exam question) by end of March/beginning April. Please remember that data cannot be protectively marked. Data input at UNCLAS level was recommended (**outstanding**).
- **ACTION 5**—UK to share HP assaying methods with National Labs (**on-going**).
- **ACTION 6**—UK to share the specification of Industrial Methylated Spirits (IMS) so that this can be made in-house by all RR participants and will avoid issues with different sources. UK to specify the precursor specification for PLX - so that this can be made in-house (**on-going, details to come**).
- **ACTION 7**—National labs to explore funding options with TSWG and DHS (**completed?**).
- **Note 1**—We agreed that the first phase of work i.e data capture was still worth completing
- **Note 2**—Actual testing (phase 2) would be dependent on funding availability.
- **Note 3**—None of the parties present at the discussions are bound to completing phase 2 - in the absence of specific funding, this initiative was entirely voluntary.



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IRR—Example Canadian Testing Methods

TEST	CERL	DRDC Suffield	DRDC Valcartier
Small scale (< 10 g)			
DSC (30 mg)	X	X	X
DTA/TGA (30 mg)	X		
Accelerated Rate Calorimetry (1 g)	X		
Vacuum Thermal Stability (1 g)		X	
Thermal Stability Test: Steel cylinder (1-10 g)			X
BAM Impact (1-5 g)	X	X	X
Bureau of Explosives Impact (1-5 g)	X	X	
Type 12 Impact (1-5 g)	X		
BAM Friction (1-5 g)	X	X	X
Electro-Static Discharge (1-5 g)	X	X	X
Chemical and Physical Analysis (1 g)			
Melting Point	X	X	X
NMR			X
FTIR	X		X
Ion chromatography	X		
GC, GC/MS, HPLC			X
Raman			X
CHN			X
Bomb calorimeter			X
Medium Scale (5-1000 g)			
Small scale burn test (10-100 g)	X		
Princess incendiary spark test (5 g)	X		
Time/pressure test (5 g)	X		
Dewar test (500 g)	X		
Small-scale cook-off bomb (500 g)		X	
Cap sensitivity test (1 kg)	X	X	X
Minimum booster (500 g)	X	X	X
Critical diameter (confined/unconfined, 500 g)	X	X	X
Gap test (500 g)	X		
DDT test (500 g)	X		
Detonation velocity measurement (1000 g)	X	X	X
Large Scale (1-100 kg)			
Detonation velocity measurements (1-5 kg)	X	X	X
Detonation velocity measurements (10-100 kg)		X	
Blast overpressure measurements (1-100 kg)	X	X	X
Very large scale (>100 kg)			
Blast overpressure measurements (100-2000 kg)		X	



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How to evaluate performance in the Proficiency Test?



Impact Testing RDX (180-grit)

Rough estimation of performance for inter- and intra-laboratory comparisons

Lab	T, °C	RH, % ²	DH ₅₀ , cm ³	s, log unit
LLNL (180)	23.9	30	22.9	0.042
LLNL (180)	22.8	23	20.7	0.095
LLNL (180)	23.9	13	22.8	0.088
LLNL (180)	23.9	15	21.4	0.041
LANL (180)	22.3	< 16	22.0	0.030
LANL (180)	21.7	< 16	20.3	0.049
LANL (180)	21.7	< 16	20.0	0.049
LANL (180)	22.9	< 10	23.3	0.027
LANL (180)	21.8	< 10	23.1	0.048
LANL (180)	21.7	< 10	23.1	0.030
LANL (180)	23.1	< 10	19.6	0.062
LANL (180)	20.4	< 10	17.7	0.117
LANL (180)	21.2	< 10	19.2	0.143
IHD (180)	26	38	22	0.16
IHD (180)	26	38	19	0.18
IHD (180)	26	40	18	0.25
IHD (180)	26	40	18	0.11
AFRL (180)	22	43	15.1	0.10
AFRL (180)	23	45	13.1	0.17
AFRL (180)	27	57	17.6	0.09

- **LLNL 4 data points mean 22.0 ± 1.1, 4.7%**
- **LANL 9 data points mean 20.9 ± 2.0, 9.6%**
- **IHD 4 data points mean 19.2 ± 1.9, 9.9%**
- **AFRL 3 data points mean 15.3 ± 2.3, 15.0%**



Inter- and Intra-laboratory Comparisons

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Impact Data only

Simple evaluation → variation percent of mean

Laboratory based

- LLNL (RDX 120-grit) 1.7%
- LLNL (RDX 180-grit) 4.7%
- LLNL (KC/Sugar 120-grit) 7.2%
- LLNL (KC/Dodecane 120-grit) 5%
- LANL (RDX 150-grit) 4.8%
- LANL (RDX 180-grit) 9.6%
- LANL (KC/Sugar 150-grit) 11.7%
- LANL (KC/Sugar 180-grit) 9.2%
- LANL (KC/Dodecane 150-grit) 17.4%
- LANL (KC/Dodecane 180-grit) 18.7%

- IHD (RDX 180-grit) 9.9%
- IHD (KC/Sugar 180-grit) 18.7%
- IHD (KC/Dodecane 180-grit) 18.4%
- AFRL (RDX 180-grit) 15%
- AFRL (KC/Sugar 180-grit) 21%

Material based

- RDX (180-grit) 14.1%
- KC/Sugar (180-grit) 20.8%
- KC/Dodecane (180-grit) 18.4%



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KClO₃/Sugar Impact Data



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Lab ¹	Sample	Test Date	T, °C	RH, % ²	DH ₅₀ , cm ³	s, log unit ⁴
LLNL (120)	-100	1/22/10	23.3	21	14.5	0.065
LLNL (120)	-100	2/25/10	22.8	28	14.0	0.022
LLNL (120)	-100	2/16/10	22.8	28	16.1	0.020
LLNL (120)	AR	3/11/10	22.7	18	14.9	0.025
LLNL (120)	AR	3/12/10	23.3	24	15.0	0.029
LLNL (120)	AR	3/15/10	23.3	18	17.0	0.082
LANL (150)	-100	2/8/10	21.2	13.5	15.5	0.076
LANL (150)	-100	2/9/10	21.1	14.2	17.7	0.044
LANL (150)	-100	2/10/10	21.8	13.5	18.8	0.035
LANL (180)	-100	4/28/10	22.3	<10	10.7	0.076
LANL (180)	-100	4/29/10	22.1	<10	11.8	0.147
LANL (180)	-100	5/4/10	22.0	<10	9.2	0.062
LANL (150)	AR	3/1/10	22.1	15.1	15.3	0.131
LANL (150)	AR	3/2/10	22.3	16.0	13.4	0.049
LANL (150)	AR	3/3/10	21.8	12.2	16.3	0.049
LANL (180)	AR	4/28/10	22.5	< 10	11.0	0.139
LANL (180)	AR	4/29/10	21.5	< 10	10.7	0.105
LANL (180)	AR	5/4/10	21.0	< 10	9.5	0.043
IHD (180)	-100	1/21/10	26	40	14	0.07
IHD (180)	-100	2/3/10	27	40	15	0.18
IHD (180)	-100	2/3/10	27	40	14	0.14
IHD (180)	AR	8/4/10	20	45	9	0.120
IHD (180)	AR	8/12/10	20	50	11	0.090
IHD (180)	AR	8/19/10	20	50	11	0.070
AFRL (180)	AR	5/7/10	26.7	54	10.2	0.146
AFRL (180)	AR	6/23/10	26.1	57	6.9	0.279
AFRL (180)	AR	6/23/10	25.6	57	7.9	0.447



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Other Research Topics



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FY11 Topics

- **Continuation of the IDCA program for FY 2011**
 - Restructure the SSST Compendium
 - Population of the SSST Compendium
 - Reacquiring Proficiency Test Data with Modified Methods
- **Prioritization of future topics of interest to the IDCA**
 - Participation in the International Round Robin SSST testing
 - Developing a fast screening method by DSC for thermal analysis (relate to ARC, ODTX, APTAC, isothermal DSC)
 - Impact of aging of solid-solid, liquid-liquid and solid-liquid mixtures on testing sensitivity
 - Expansion of camera approach to SSST testing
 - Effects of impure source materials on testing
 - Effects of porosity of solid-solid and solid-liquid mixtures on sensitivity
 - Optimizing sandpaper for impact testing (design of experiments)
 - Developing methods so ABL vs. BAM data from different methods can be compared
 - Additional HME threats that challenge SSST Testing

From Integrated Data Collection Analysis (IDCA) Program—FY2011 Project Descriptions,
IDCA Program Analysis Report 005, November 13, 2010.



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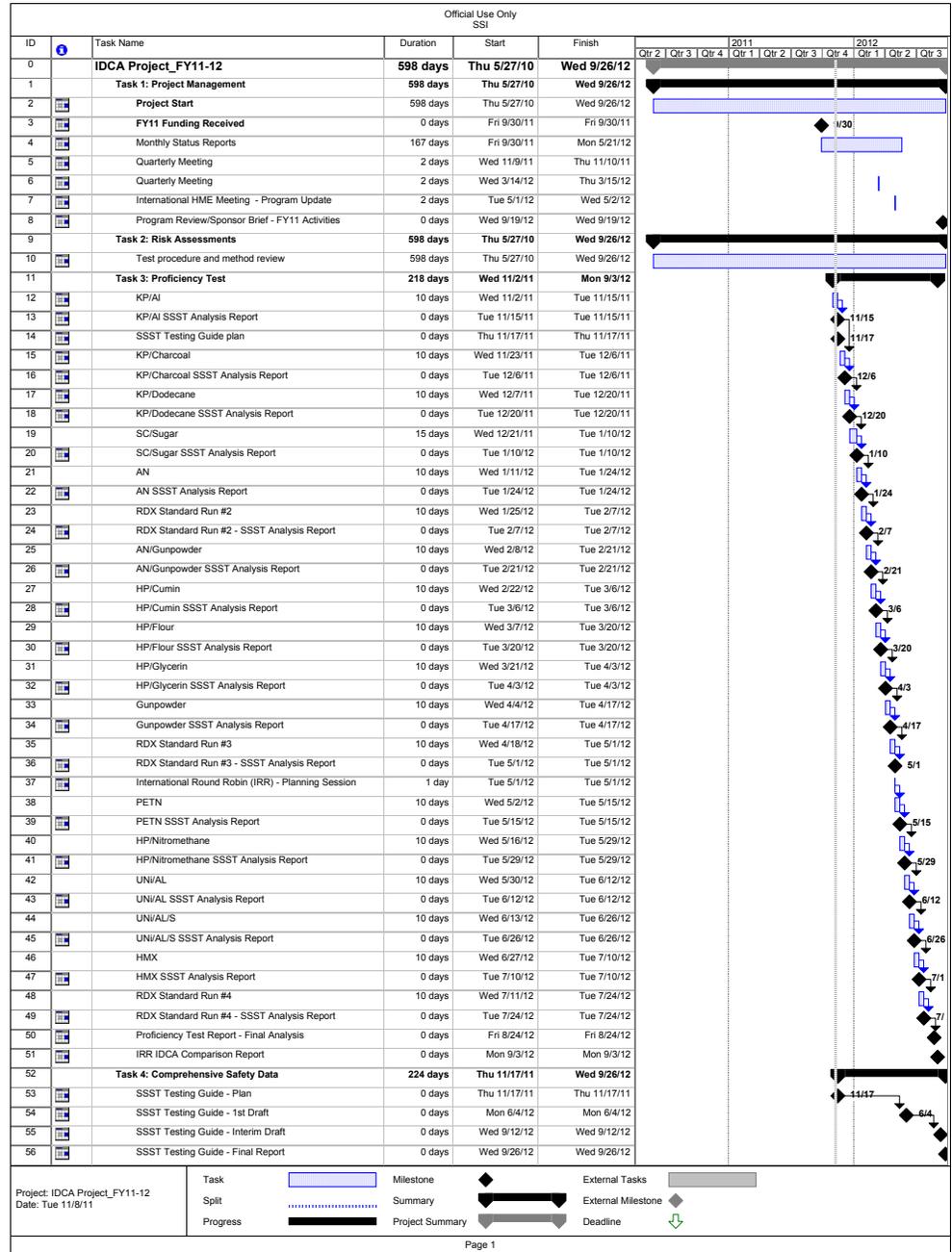
Re-baseline



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GANTT Chart





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The IDCA Logo



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Acknowledgments

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- The work was performed by the National Explosives Engineering Science Security (NEXESS) Center, a tri-lab effort supported by Sandia National Laboratories, Los Alamos National Laboratory, and Lawrence Livermore National Laboratory under sponsorship of the US Department of Homeland Security, Office of Science and Technology, Explosives Division, and the Transportation Security Administration. Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy's National Nuclear Security Administration under Contract DE-AC04-94AL85000. Los Alamos National Laboratory is operated by Los Alamos National Security, LLC, for the United States Department of Energy under contract DE-AC52-06NA25396. This work was performed under the auspices of the U. S. Department of Energy by Lawrence Livermore National Laboratory under Contract DE-AC52-07NA27344. The Air Force Research Laboratory (AFRL/RXQF) and The Indian Head Division, Naval Surface Warfare Center (NSWC, IHD) also performed work in support of this effort. The work performed by AFRL/RXQF and IHD, NSWC is under sponsorship of the US Department of Homeland Security, Office of Science and Technology, Explosives Division.



Homeland
Security



Appendix C. Presentation—Thermal Study of KClO_3 and Fuels

Thermal Analysis of KClO_3 /Sugar Mixtures

The Effect of Sample Size on DSC Analysis

Mary M. Sandstrom¹, Geoffrey W. Brown¹, Kirstin F. Warner², Daniel N. Sorensen², Timothy J. Shelley³, Jose Reyes³, Peter C. Hsu⁴, and John G. Reynolds⁴

¹Los Alamos National Laboratory, Los Alamos, NM USA

²Indian Head Division, Naval Surface Warfare Center (IHD-NSWC), Indian Head, MD USA

³Air Force Research Laboratory (AFRL/RXQF), Tyndall Air Force Base, FL USA

⁴Lawrence Livermore National Laboratory, Livermore, CA USA

Outline

- **Introduction**

- The results are in!
- How can they look so different if we are all doing the same thing?

Fear NOT!

We have the finest minds from the country's best institutions working on this problem.

- **Experimental**

- Vary the sample size from small to large
- Take pictures to see what they look like in the pan
- Run the DSC .

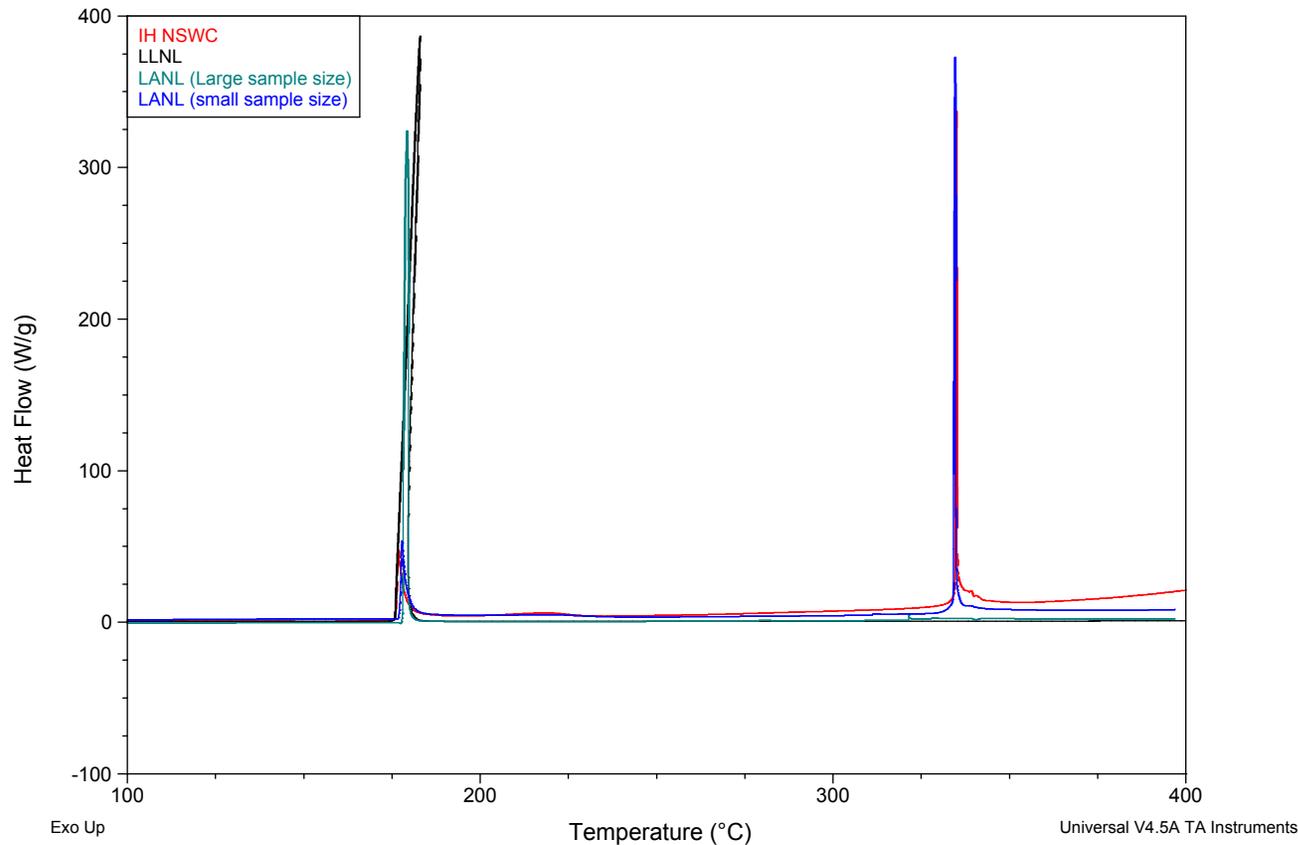
- **Results**

- **Conclusions**



Introduction

- During the testing of KC/Sugar (74%/26% wt/wt). Each of the participating labs observed very different results by DSC.



So why were we seeing single, double peaks, and sometimes even triple? How could LANL get all of them? (That's easy, because Mary will do **anything** to avoid sitting at her desk writing reports)

Introduction

GREAT SUGGESTIONS FROM THE TEAM:

- Aging effects
 - What is the reaction pathway?
 - What are the kinetics? How do we investigate that?
 - But we saw this in both fresh and aged sample
- Pan differences
 - Closed vs. pin-hole. (i.e. Totally confined vs. vented? Apples vs. oranges? Maybe, Maybe not?)
- Instrument differences
 - Q2000 vs. Q1000 vs. TA2920

Introduction

MORE GREAT SUGGESTIONS FROM THE TEAM:

- Samples size
 - Inhomogeneity of the sample
 - “Connectedness” in the pan
 - Sustainability of reaction

Investigation of sample size was the easiest and quickest option to investigate

Experimental

- A single batch of sieved KC/Sugar was prepared according to the IDCA mixing protocols. Materials used were the same as those used for the IDCA Round Robin study.
- Hermetically sealing Al pans with 70 μ m pin hole lids
- TA Instruments Q2000 DSC.
- Duplicate pans were loaded with 0.05mg, 0.10mg, 0.15mg, 0.20mg, 0.25mg and 0.30mg samples.
- Ramp rate was 10°C/min. One duplicate was run from 40°C to 250°C (sugar melt) and the other to 400°C (KC melt)

Experimental

Pictures were taken of each sample before it was sealed.

The samples were run up to either 250°C or 400°C.

The pans were reopened and pictures were taken of the residue left in the pan.

These results were then compared to the DSC traces

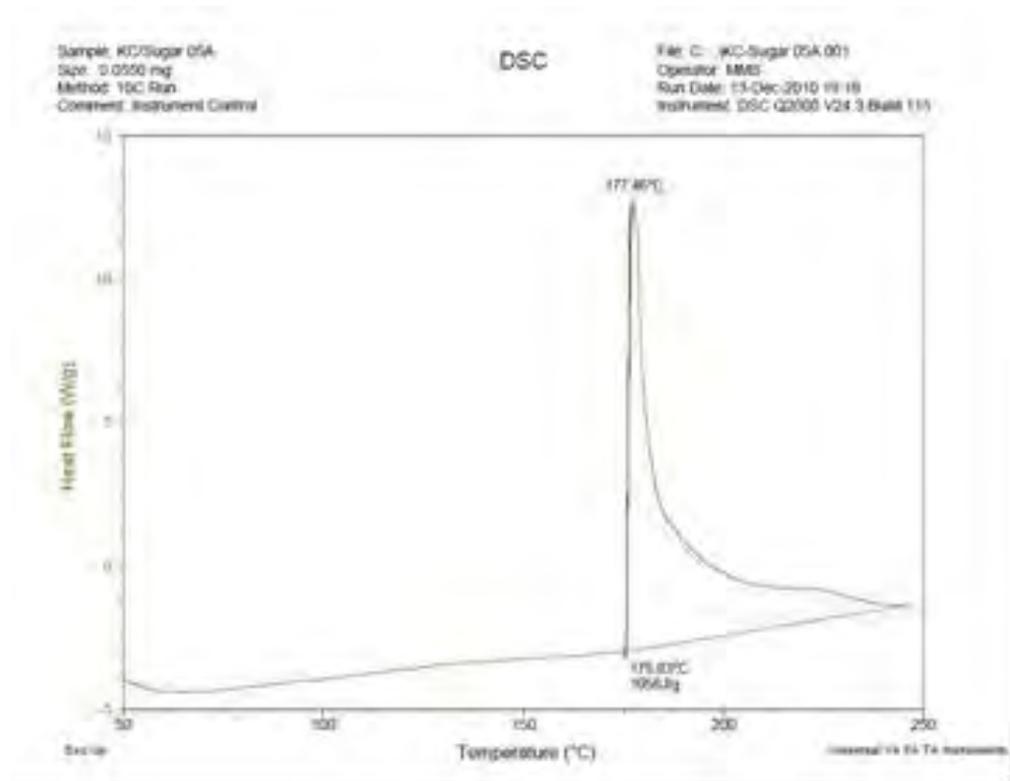
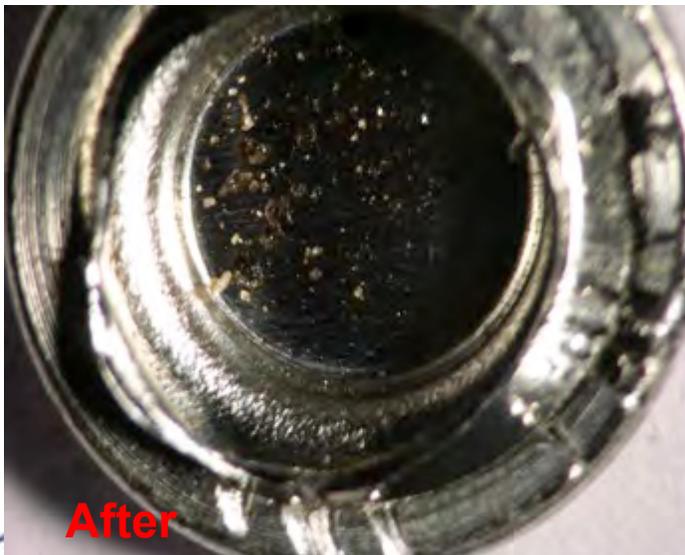
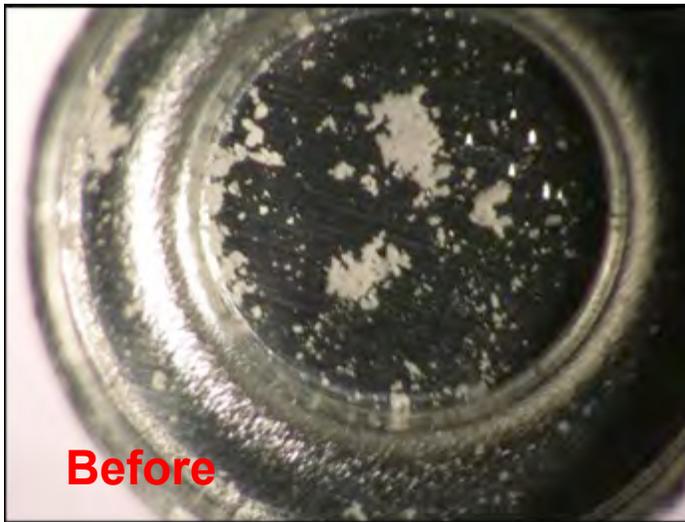


0.055mg sample

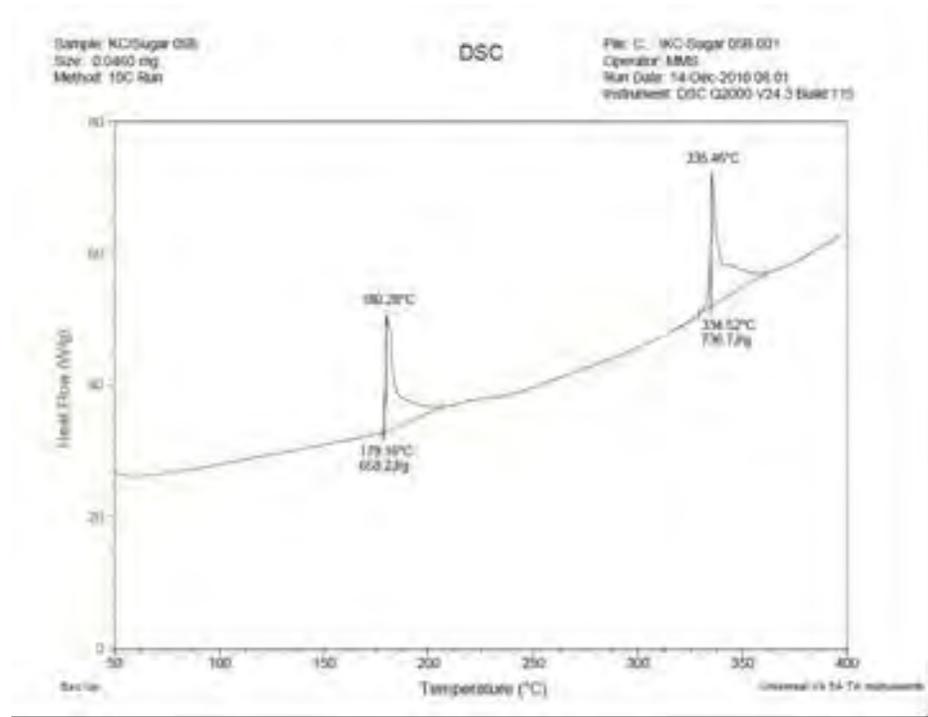
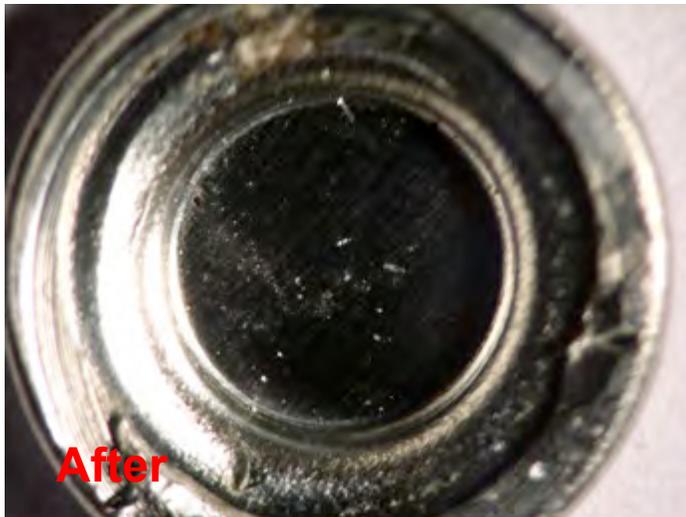


0.306mg sample

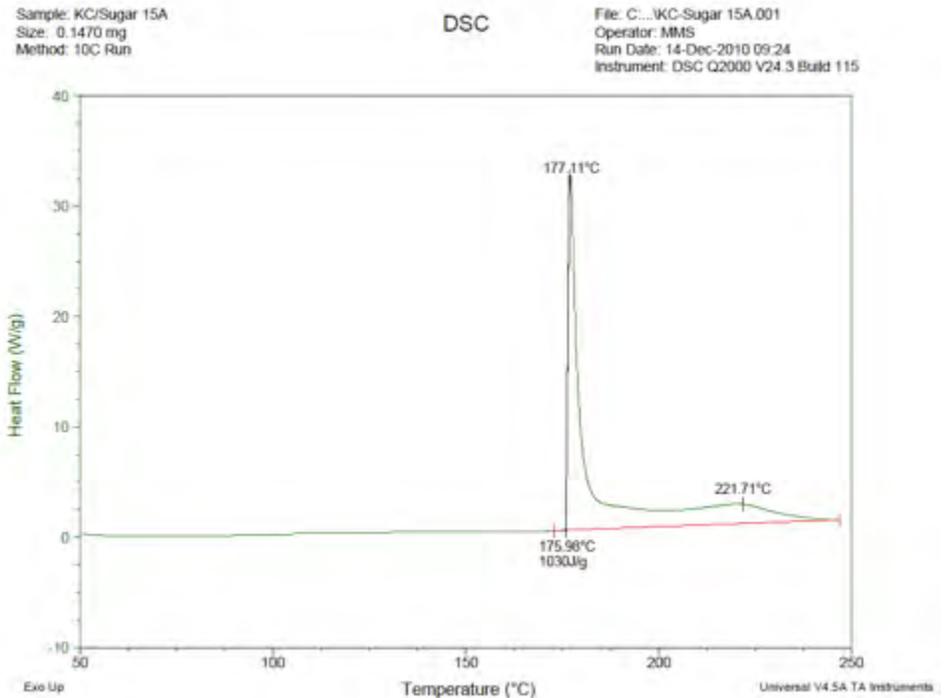
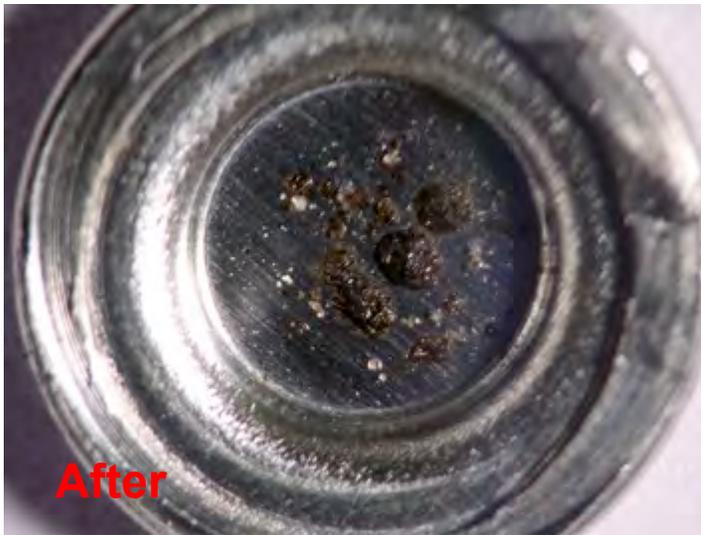
Results (0.05mg to 250°C)



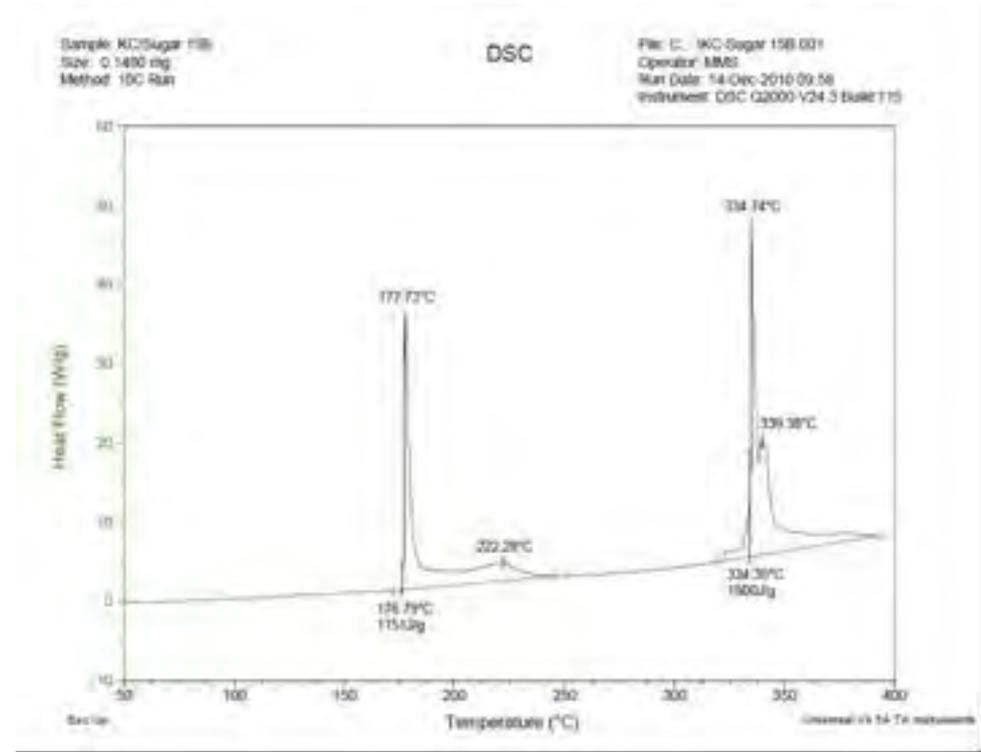
Results (0.05mg to 400°C)



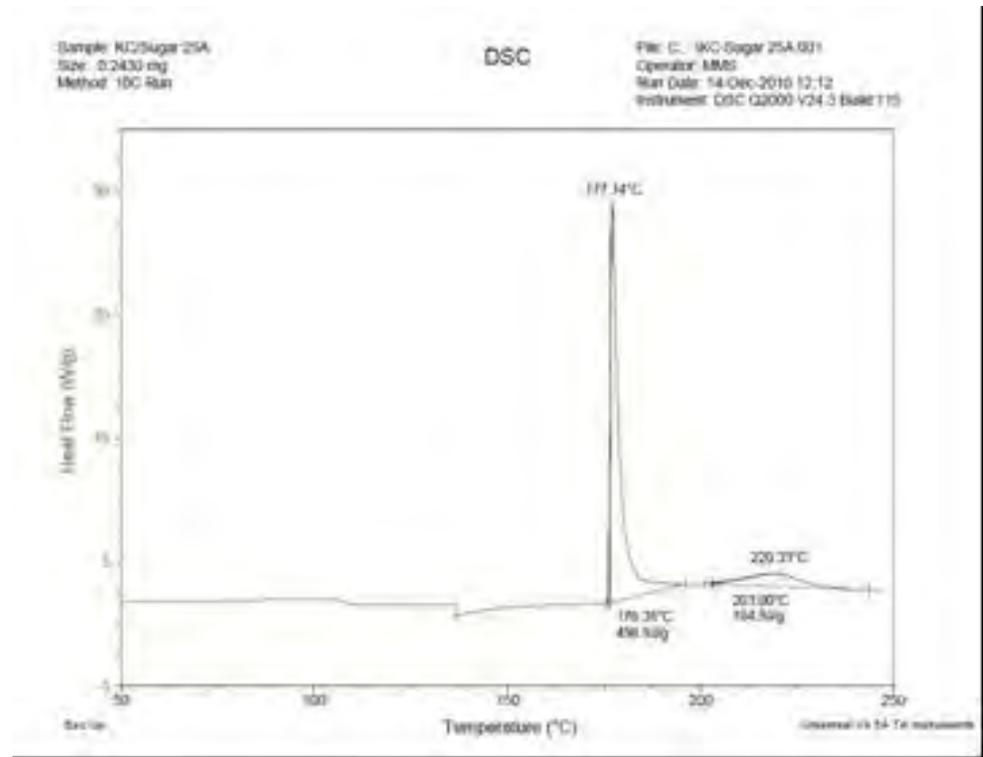
Results (0.15mg to 250°C)



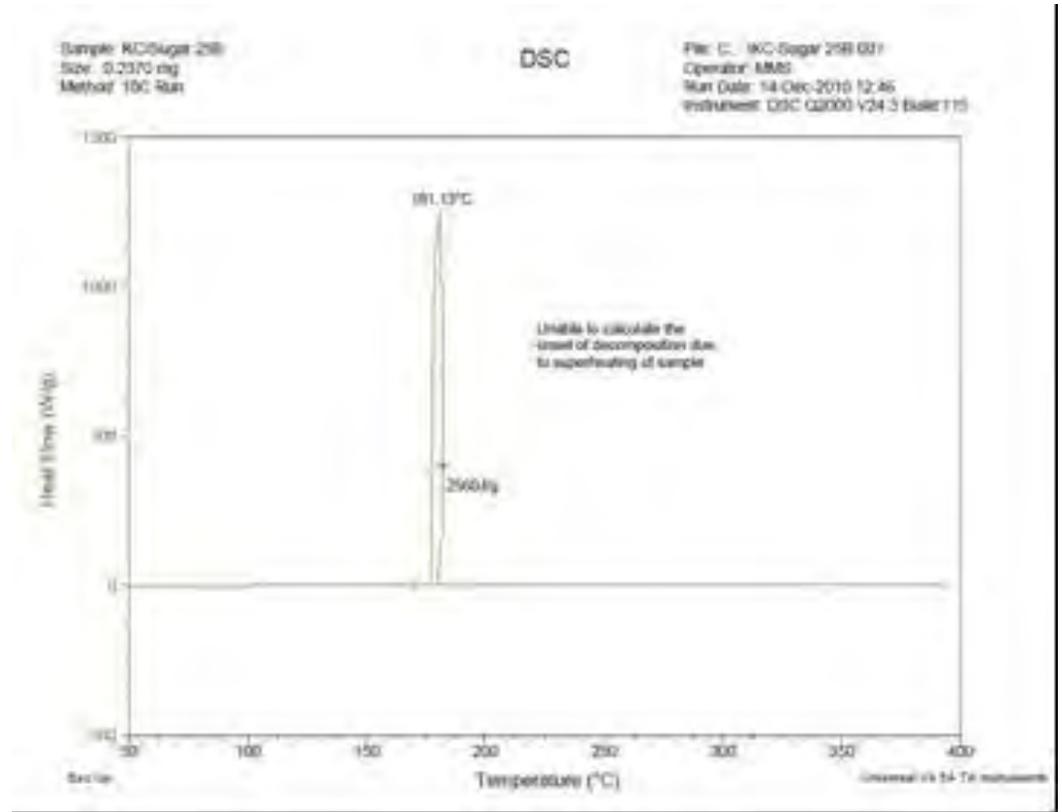
Results (0.15mg to 400°C)



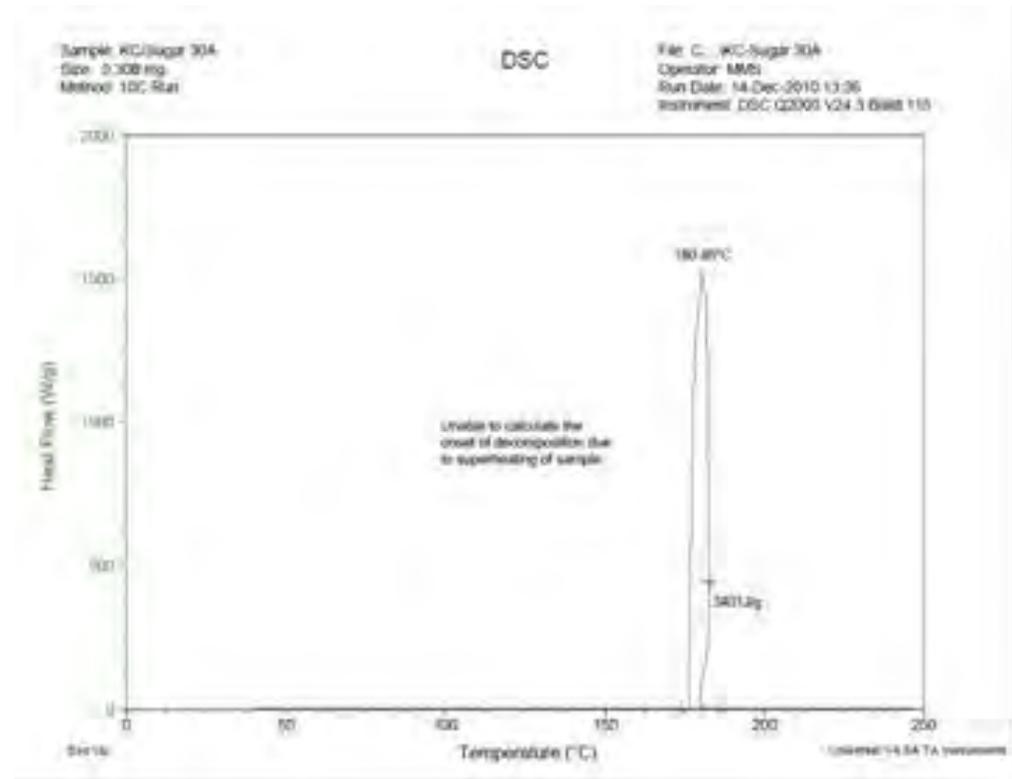
Results (0.25mg to 250°C)



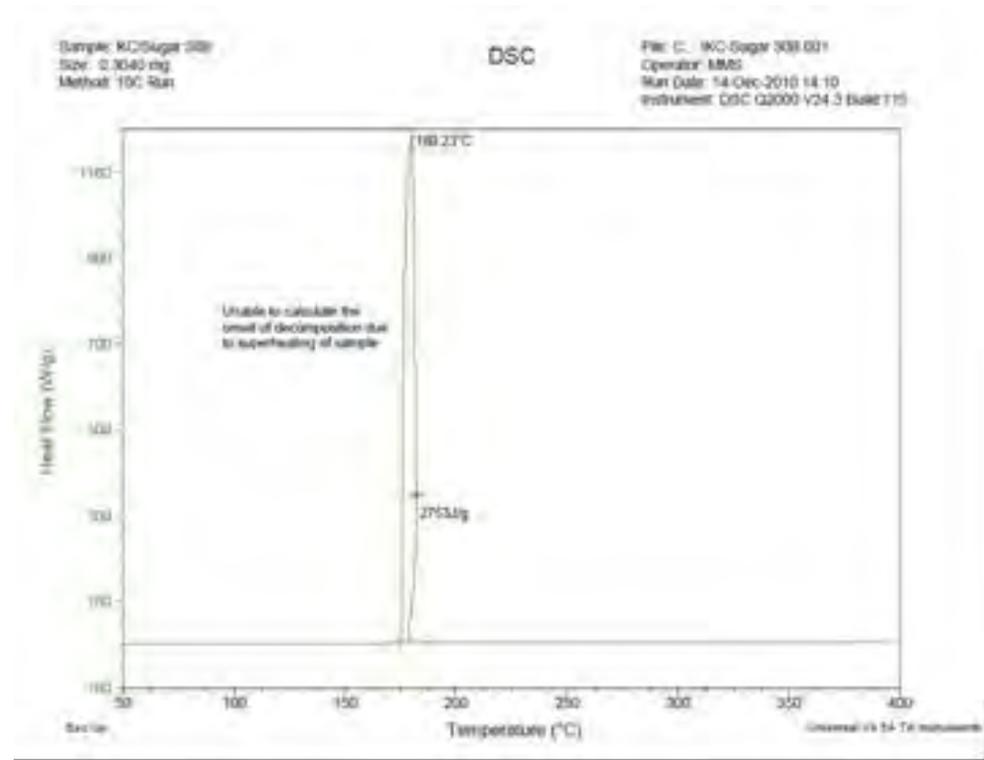
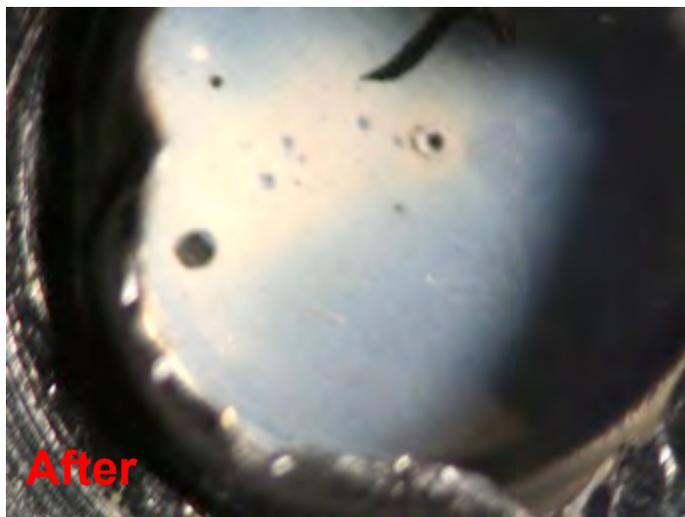
Results (0.25mg to 400°C)



Results (0.30mg to 250°C)



Results (0.30 to 400°C)

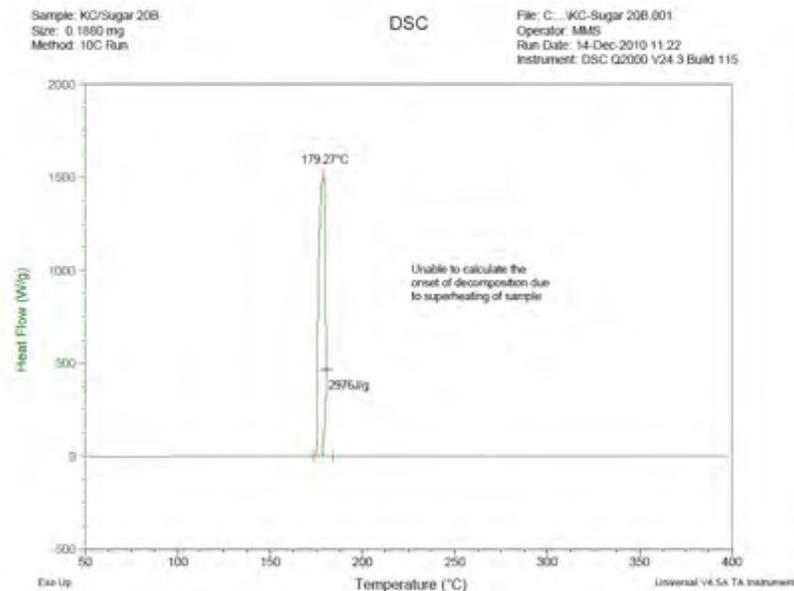


Discussion

- Inhomogenities
 - Difficult to tell because the materials have a similar appearance
 - Fuel rich vs oxidizer rich areas in pans with smaller sample sizes are more likely resulting in two (three) peaks
- Connectedness
 - Small samples have “discrete” piles that will be disturbed during sample prep and movement of the autosampler.
 - Can the melting sugar make contact with other materials in the pan before it is consumed.
- Sustainability of Reaction

Conclusion

- Let's get the truck out of the weeds...what does that mean in terms of safety testing?
- Do we care what 0.05mg of this sample is doing? This stuff is thermally unstable at the lower temperature...when the sugar starts to melt



And Now a Friendly Message from Your Friends in New Mexico

- Let's Do Another **QUARTERLY*** Meeting in the Beautiful Land of Enchantment!
- The First Week of October is Balloon Fiesta!



*Quarterly means every quarter

And Now a Friendly Message from
Your Friends in New Mexico



Hey Man!
You Can Tie Dye Your
Very Own
Lab Coat!

Just ask José how groovy
THAT is!

And Now a Friendly Message from Your Friends in New Mexico

- Come visit lovely Tucumcari, the former home of Becky Olinger



Maybe not.....

And Now a Friendly Message from Your Friends in New Mexico

- Soak your bones in the healing waters of Ojo Caliente
– (BYOS...Bring Your Own Spouse and don't be THAT guy.)



And Now a Friendly Message from Your Friends in New Mexico

Mary is running out of friends she can ask to feed her horse when she is on travel.



Appendix D. Presentation—Statistics for Participant Characterization

A Plan for Evaluating and Comparing Data Sets in the IDCA Collaboration

Can we say something meaningful about individual results and sets of results that appear different?

Geoff Brown
High Explosive Science & Technology (WX-7)
Los Alamos National Laboratory
Los Alamos, NM 87545



Slide 1



Two Questions and Some Answers

- 1 – What is our confidence that a single test is accurately probing the properties of the material?**
- 2 – What is our confidence that results from two or more labs are the same or are significantly different?**

Answers obtained with probability and statistics

Mark Twain :

“Figures often beguile me ... There are three kinds of lies: lies,
damned lies,
and statistics.”

Sampling Theory for Small Sample Distributions



Slide 2

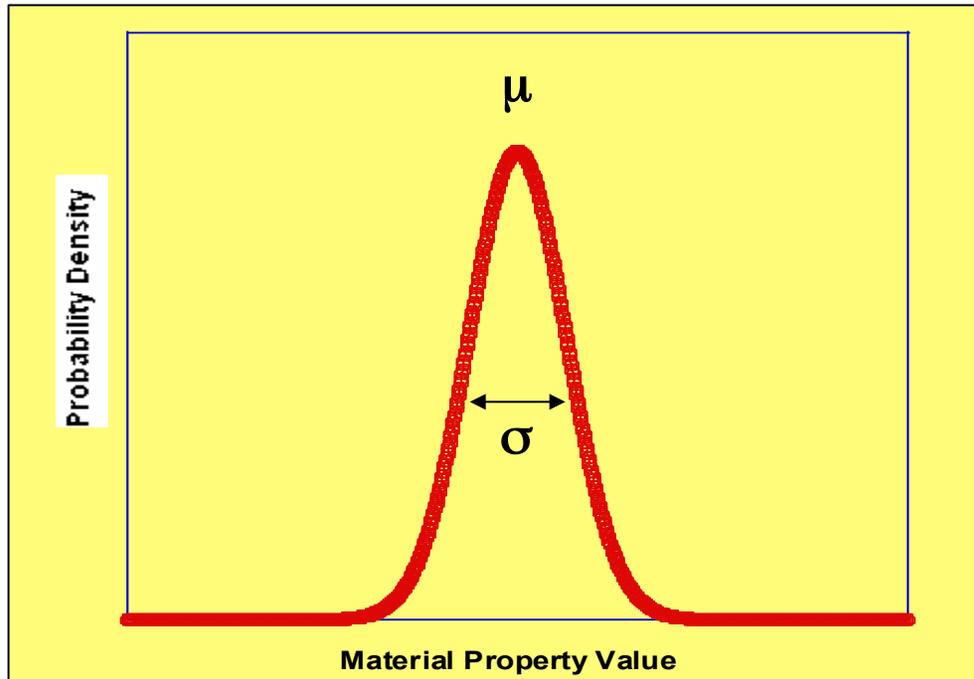
Operated by the Los Alamos National Security, LLC for the DOE/NNSA

Unclassified



What Are We Actually Measuring?

All IDCA Measurements are estimating mean and standard deviation



1 – “Identical” measurements repeated several times to observe “scatter” in the results. (Not error in method)

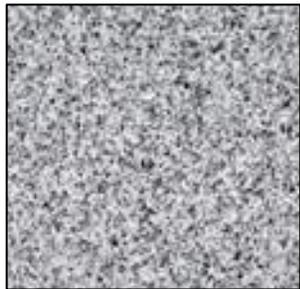
TIL values
DSC features

2 – Measurements repeated several times under different conditions to probe a response.

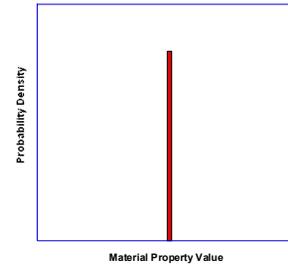
Bruceton and
Neyer tests

Material Property Measurements – TIL and DSC

Material Homogeneity Depends on Length Scale

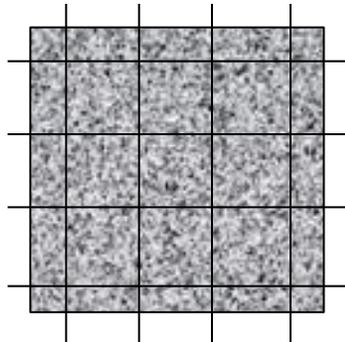


Measure full sample
Determine one
value

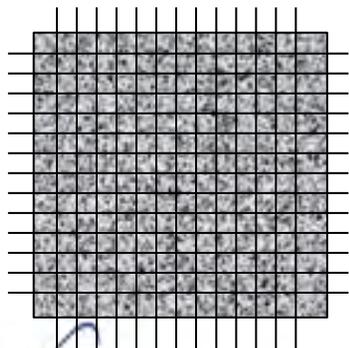
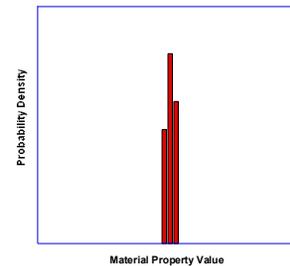


Last Case is IDCA

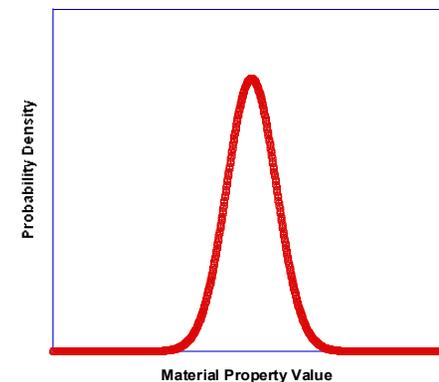
- Testing small fragments of larger sample
- Testing small amounts out of necessity



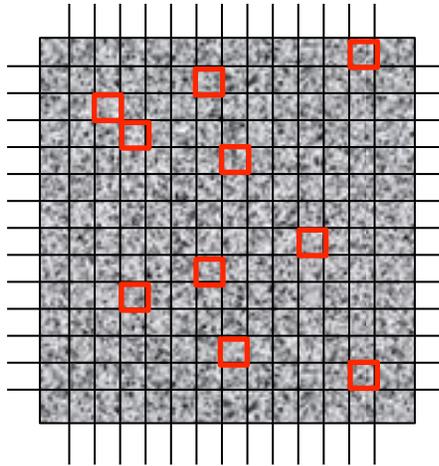
Sample coarsely
Determine several
values



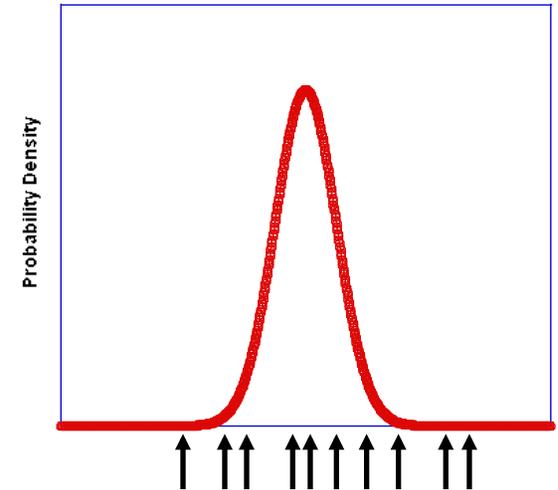
Sample finely – probing the
distribution of many values



Random Sampling Probes Material Property



Picking 10 random pieces from grid is picking 10 random levels along distribution's x-axis



Sample Mean

$$\bar{m} = \frac{m_1 + m_2 + m_3 + \dots}{n}$$

Sample Standard Deviation

$$s = \sqrt{\frac{(m_1 - \bar{m})^2 + (m_2 - \bar{m})^2 + (m_3 - \bar{m})^2 + \dots}{n - 1}}$$

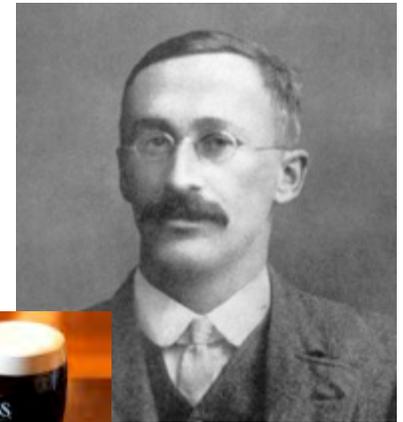
\bar{m} and s are estimates of μ and σ , the population distribution parameters

But what is our confidence or expected error in the reported estimates for the number of repeated measurements carried out?

“The Probable Error of a Mean” – Student’s t Distribution

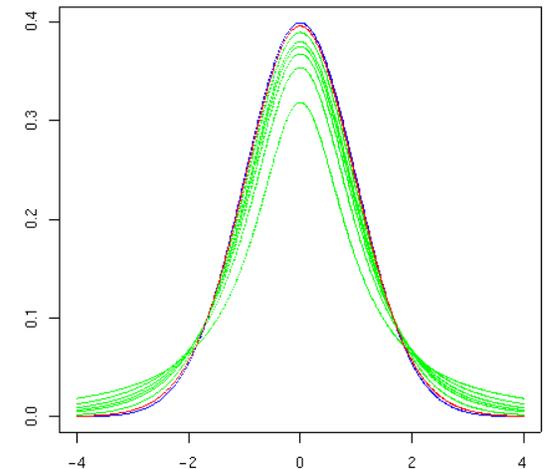
Problem solved in 1800’s in Germany and again by William Gosset in 1908 who put it in useful form in English.

- Gosset was chemist at Guinness brewery in Dublin
- Trained in chemistry and mathematics
- Examined materials (quality control) for the brewery
- Could not publish openly – rules or proprietary
- Published findings as “Student”



Same problem that we have – can only carry out small number of tests but need an estimate of mean with statement of confidence

- Probability and mathematical treatment of general case
- Small number of samples n from large population
- Calculated the distribution for various values of n
- Resulting distribution is not Gaussian for small n
- Illustrates “sampling error” and leads to sampling theory



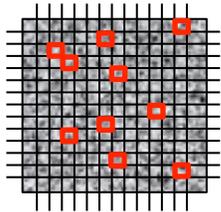
Student's t Distribution – How Do We Use It?

E is the error with probability $1-\alpha$ associated with using \bar{m} as an estimate of μ if n measurements produced a deviation of s

$$E < t_{\alpha/2} \frac{s}{\sqrt{n}}$$

With probability $1-\alpha$, μ lies within the interval bounded by the maximum error.

$$\bar{m} \pm t_{\alpha/2} \frac{s}{\sqrt{n}}$$



Example: Moisture Testing – Lab A measures the moisture 10 times and obtains $\bar{m} = 0.53\%$ and $s = 0.05\%$

10 measurements = 9 degrees of freedom

$t_{\alpha/2}$ for 9 df

95% ($\alpha=0.05$): 1.833

99% ($\alpha=0.01$): 2.821

95 % Level

$$E < 1.833 \frac{0.05\%}{\sqrt{10}} \quad E < 0.03\%$$

$$0.50\% < \bar{m} < 0.56\%$$

99 % Level

$$E < 2.821 \frac{0.05\%}{\sqrt{10}} \quad E < 0.04\%$$

$$0.49\% < \bar{m} < 0.57\%$$

How Do Results Vary with # of Tests and Probability level

Example: Moisture Testing – Lab A measures moisture
 $\bar{m} = 0.53\%$ and $s = 0.05\%$

Increasing Confidence: Higher Error and Larger Intervals

More Tests: Lower Error and Smaller Intervals

Degrees of Freedom	90% Expected Error , E	95% Expected Error , E	99% Expected Error , E	90% Confidence Interval	95% Confidence Interval	99% Confidence Interval
1	0.11	0.22	1.13	0.22	0.45	2.25
2	0.05	0.08	0.20	0.11	0.17	0.40
3	0.04	0.06	0.11	0.08	0.12	0.23
4	0.03	0.05	0.08	0.07	0.10	0.16
5	0.03	0.04	0.07	0.06	0.08	0.14
6	0.03	0.04	0.06	0.05	0.07	0.12
7	0.03	0.03	0.05	0.05	0.07	0.11
8	0.02	0.03	0.05	0.05	0.06	0.10
9	0.02	0.03	0.04	0.04	0.06	0.09
10	0.02	0.03	0.04	0.04	0.05	0.08
11	0.02	0.03	0.04	0.04	0.05	0.08
12	0.02	0.02	0.04	0.04	0.05	0.07
13	0.02	0.02	0.04	0.04	0.05	0.07
14	0.02	0.02	0.03	0.03	0.05	0.07
15	0.02	0.02	0.03	0.03	0.04	0.07
16	0.02	0.02	0.03	0.03	0.04	0.06
17	0.02	0.02	0.03	0.03	0.04	0.06
18	0.02	0.02	0.03	0.03	0.04	0.06
19	0.01	0.02	0.03	0.03	0.04	0.06
20	0.01	0.02	0.03	0.03	0.04	0.06

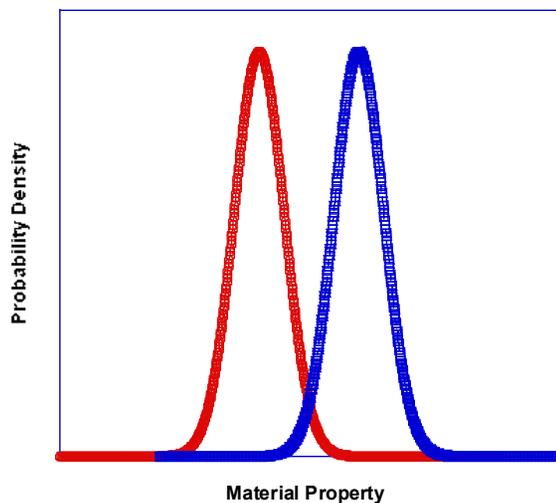
Comparison of Results from Multiple Laboratories

$$t = \frac{\bar{m}_1 - \bar{m}_2}{\sqrt{(n_1 - 1)s_1^2 + (n_2 - 1)s_2^2}} \sqrt{\frac{n_1 n_2 (n_1 + n_2 - 2)}{n_1 + n_2}}$$

Significantly different if $t >$ t-distribution value for chosen confidence level

“t-test” compares different means

ANOVA (Analysis of Variance) generalizes t-testing for comparison of multiple means.



Comparisons: Degree of Overlap of multiple t-distributions

Three Lab ANOVA test of DSC features from RDX. Used $\alpha = 0.05$, 90% Confidence Level

Feature	P-Value	Statistically Significant Difference	Largest Δ Between Labs	Reasonable single lab variation	Still Statistically Significant Difference
Endotherm 1 Onset	0.011	Yes	0.417 C	2 C	No
Endotherm 1 Minimum	0.720	No	0.246 C	2 C	No
Endotherm 1 Enthalpy	0.023	Yes	8.63 J/g	14 J/g (10%)	No
Endotherm 2 Minimum	0.032	Yes	0.973 C	2 C	No
Exotherm Onset	0.026	Yes	5.520 C	2 C	Maybe
Exotherm Maximum	0.507	No	0.950 C	2 C	No
Exotherm Enthalpy	0.018	Yes	82.4 J/g	200 J/g (10%)	No

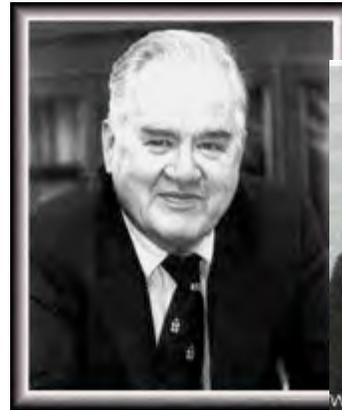
Bruceton and Neyer Testing Are Different

- Student's *t* and ANOVA are based on *random, independent* testing
- DSC and TIL – random samples from vial
- DSC and TIL – outcome of one measurement does not influence others
- Replicates of identical tests produce multiple values that lead to \bar{m} and s
- Bruceton and Neyer use random samples but test levels are not independent
- Outcome of one test influences the choice of next test level

Problem solved for Bruceton analysis when technique was developed

“Statistical Analysis for A New Procedure in Sensitivity Experiments” AMP 101.1R, SRG-P No. 40, J.W. Tukey, - (1944)

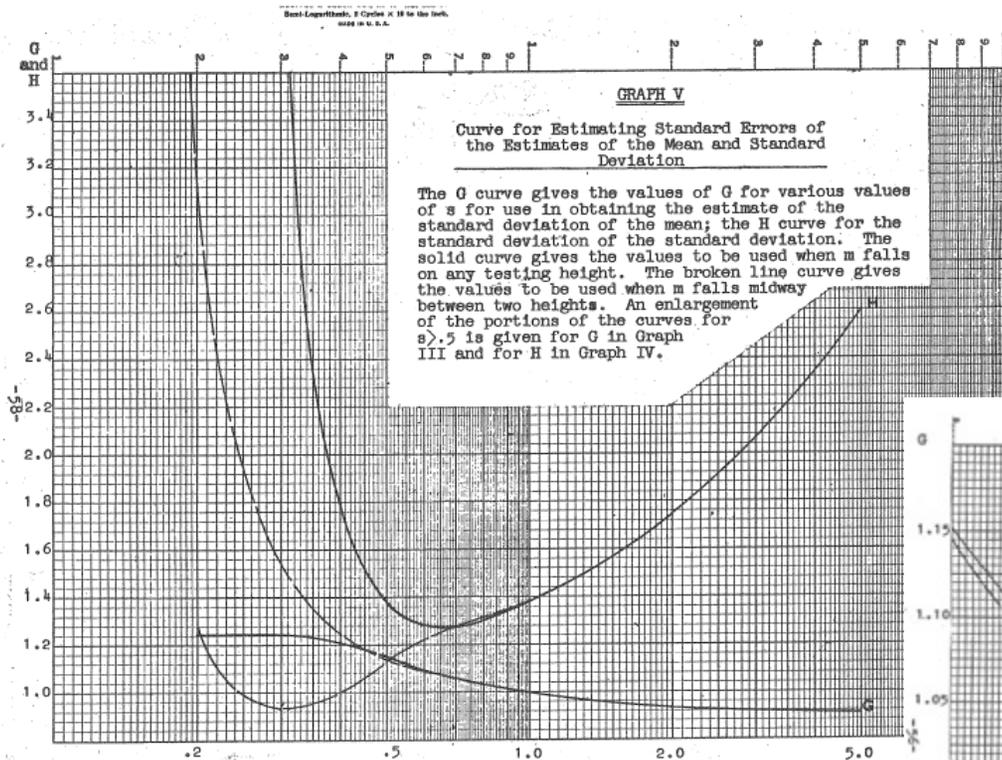
“A Method for Obtaining and Analyzing Sensitivity Data” – Dixon and Mood, J. Am. Stat Assoc – (1948)



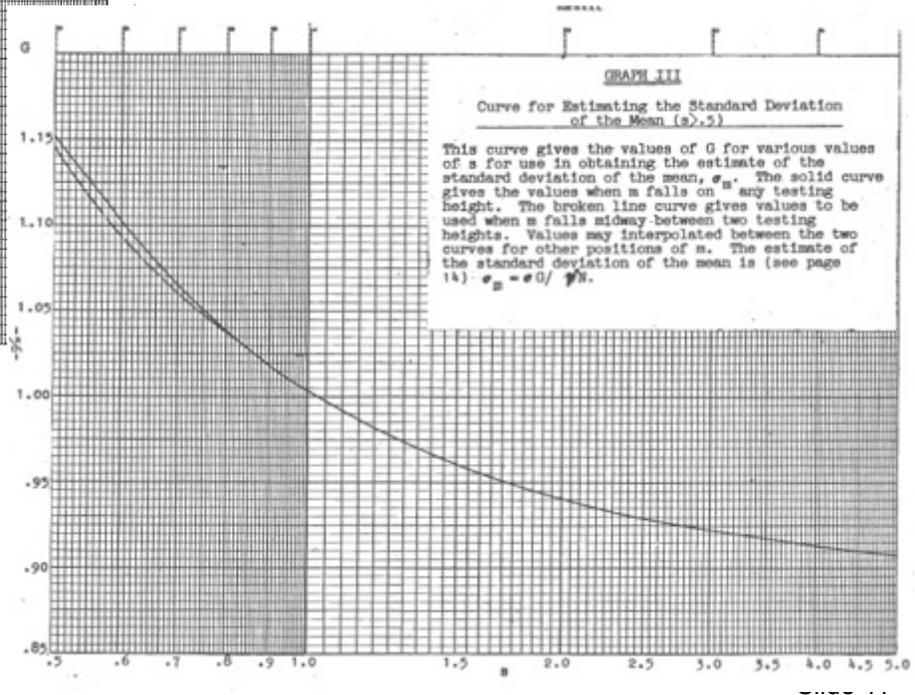
Standard Error s/\sqrt{n} has to be adjusted by a complicated function derived during maximum likelihood analysis treatment of procedure.

Standard Error of Mean from Bruceton Method

Depends on Complicated Functions and Spacing between Mean and Step Levels



$$\text{standard error} = \frac{Gs}{\sqrt{n}}$$



Can use the standard error to determine expected error and confidence intervals.

Comparing Results from Bruceton Method

t-testing is applicable if s is adjusted to G_s

Compares two different means

$$t = \frac{\bar{m}_1 - \bar{m}_2}{\sqrt{(n_1 - 1)(Gs)_1^2 + (n_2 - 1)(Gs)_2^2}} \sqrt{\frac{n_1 n_2 (n_1 + n_2 - 2)}{n_1 + n_2}}$$

Read G from plots or calculate

$$G^2 = \frac{\sum w_1}{\sum w_1} \left\{ \frac{z_{1-\alpha}^2}{q_{1-\alpha}} + \frac{z_1^2}{p_1} \right\}$$

χ^2 test all results to identify outliers within single lab and between labs

$$\bar{y} = \frac{\sum \frac{y_1}{\sigma_{y_1}^2}}{\sum \frac{1}{\sigma_{y_1}^2}}$$

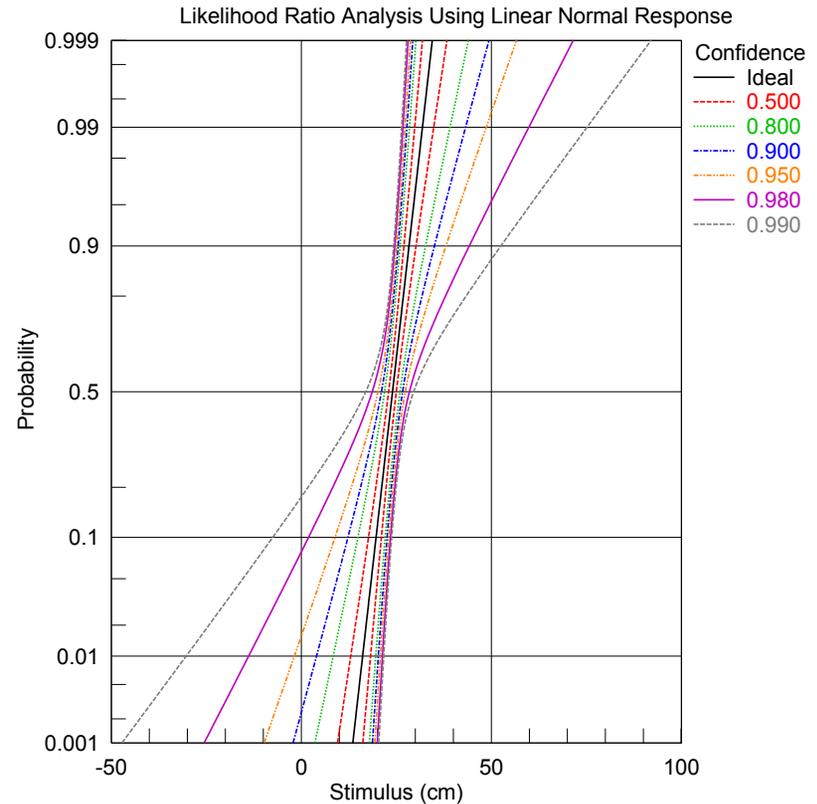
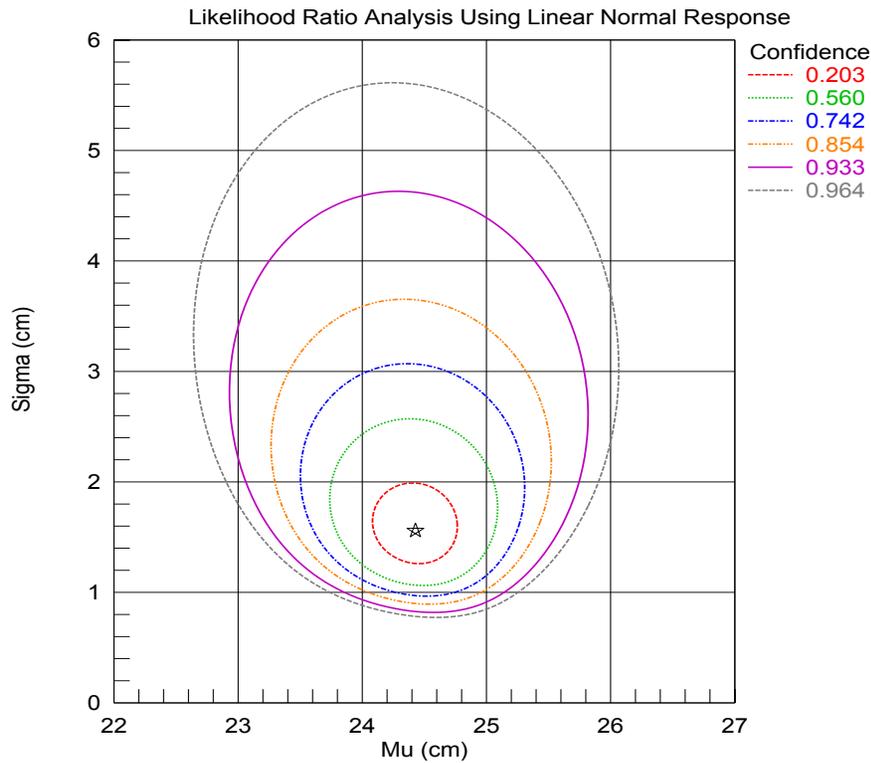
$$Q_r = \sum_1^r \frac{(y_1 - \bar{y})^2}{\sigma_{y_1}^2}$$

Compare Q_r to χ^2 table values for chosen confidence level. Remove results, re-evaluate.

H function allows standard deviations to be compared

Neyer d-optimal Method

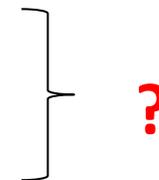
Built-in confidence level evaluation and result comparison



Mu	Sigma	Difference Confidence
24.4081,	3.53681,	
23.9931,	3.38301,	1.95902%,

How Will We Use These Errors and Confidence Intervals?

- Write comparison reports (4-5) for each family of materials?
- Add appendix to existing and future reports with statistics?
- At 95% and 99% levels, report
 - Confidence Interval for individual lab results
 - Expected Error for individual lab results
 - ANOVA or other comparison between all labs
 - Comparison of materials in the family
- Will provide an overall statistical evaluation of significance and similarity of results



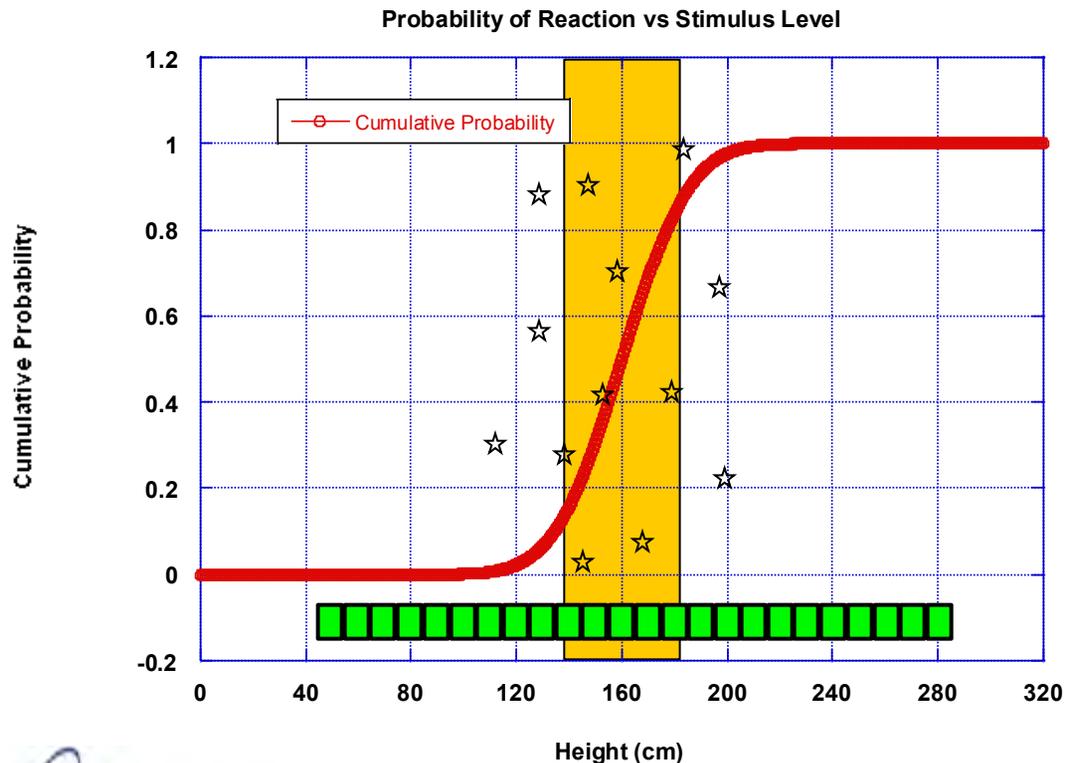
What's Missing?

Understanding the error induced by violating assumptions of the Bruceton Method.

Assumptions of the Bruce-ton Up-Down Test

- Stimulus levels are evenly spaced
- Reaction probability is Gaussian
- Step size is between $\frac{1}{2} \sigma$ and 2σ
- Sample size is large (> 50)

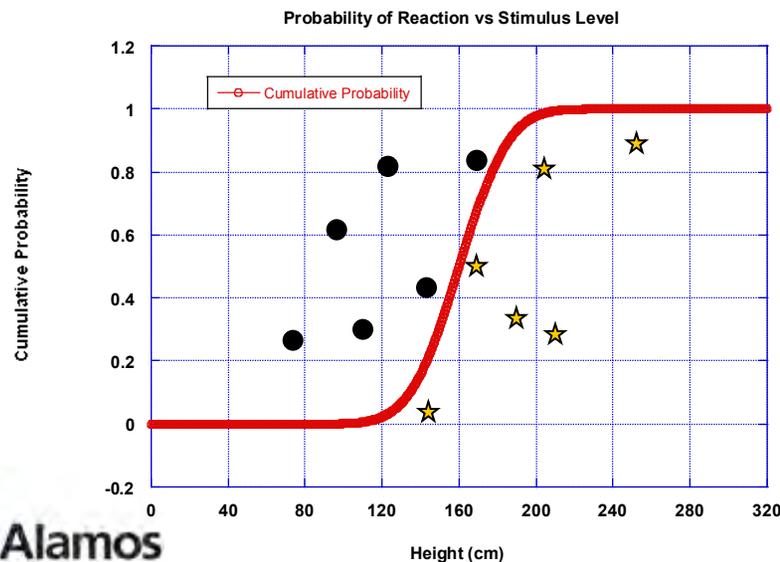
Simplifications for equation derivation



- Each lab's long standing procedures violate one or more of these assumptions
- Don't want to change procedures at this point
- Need to examine how much additional error to expect when each assumption is violated.
- Too many cases to treat theoretically.
- Use simulation to evaluate bounds of additional error.

Simulation Approach – Assume a Spherical Cow

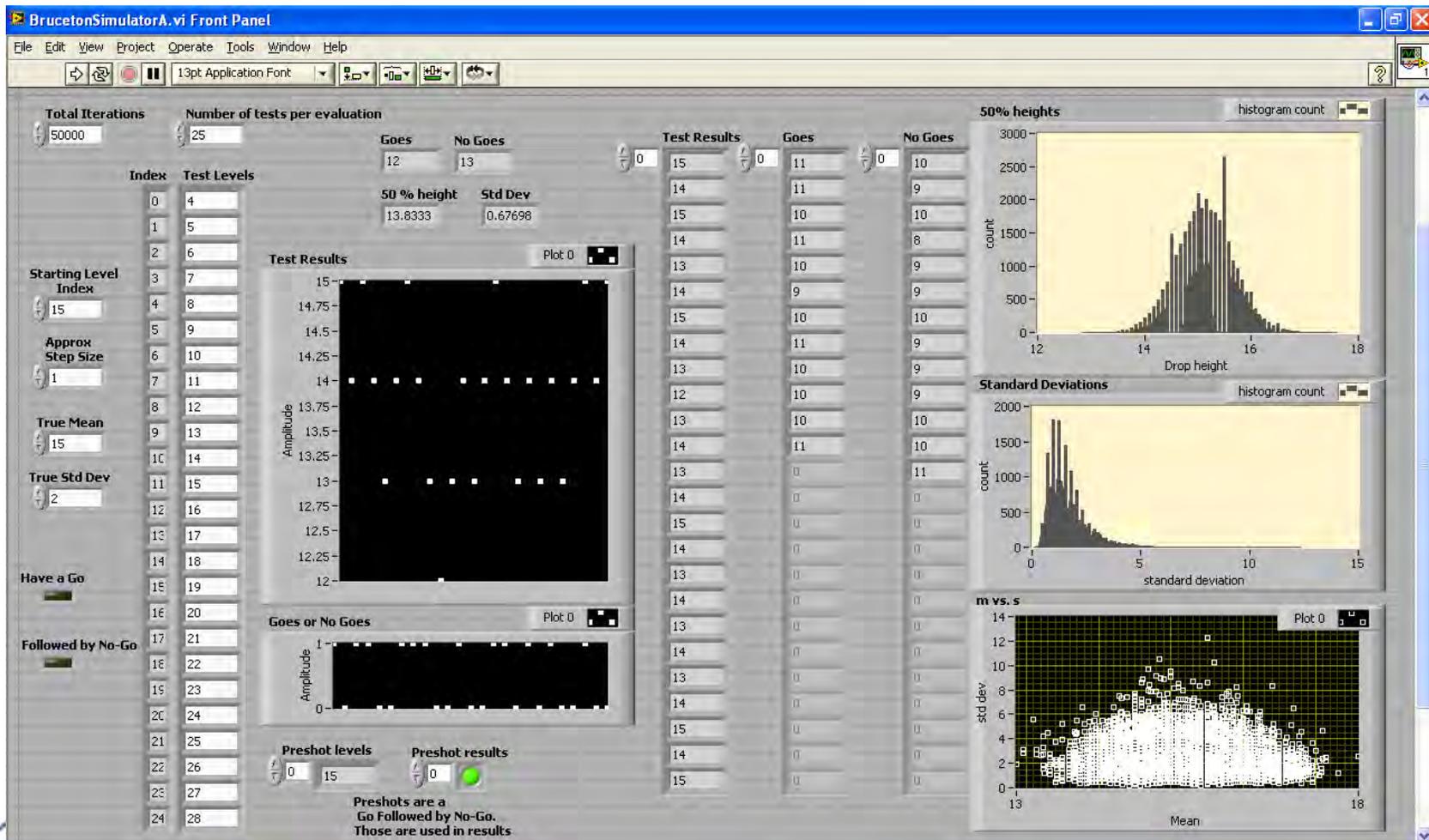
- Model a perfect material simply as a Normal distribution representing reaction probability
 - Eliminates sample / test inhomogeneity
- Model the Go / No-Go determination ideally with random number generator relative to perfect distribution
 - Eliminates operator inhomogeneity
- Run tens of thousands of Bruceton evaluations in the computer
- Evaluate probability of obtaining results outside of expected error calculated previously



- Program a distribution
- Choose stimulus level
- Generate random number
- Value > Cumulative is No-Go
- Value < Cumulative is Go
- Run Bruceton Up-Down Method
- Repeat thousands of times

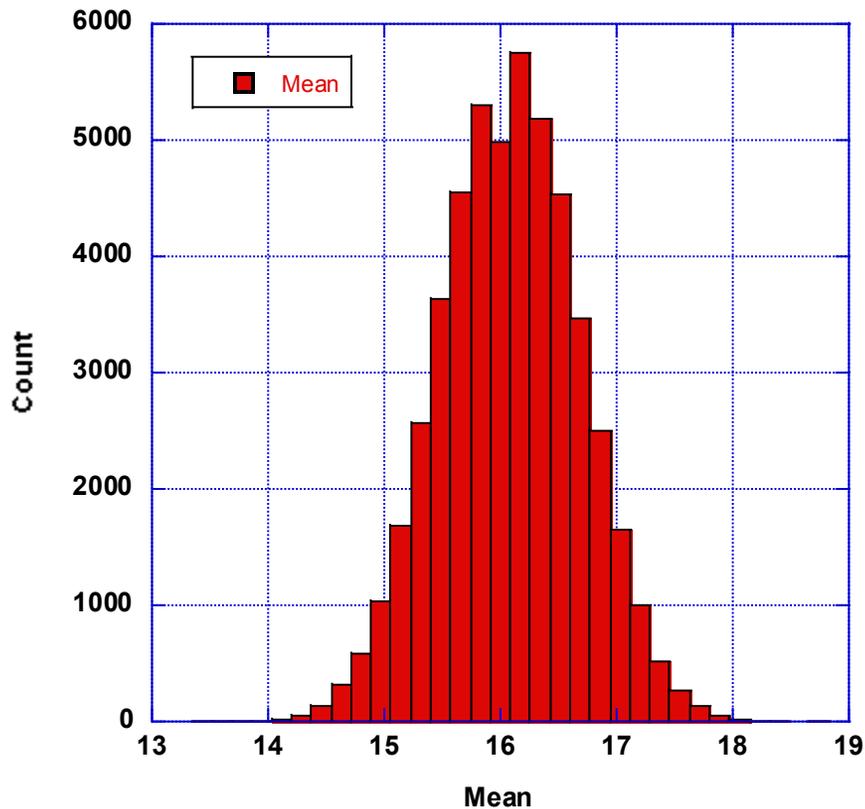
LabVIEW Bruceton Up-Down Simulator

- User selectable test levels, starting index, response function.
- Output file with all results for further investigation



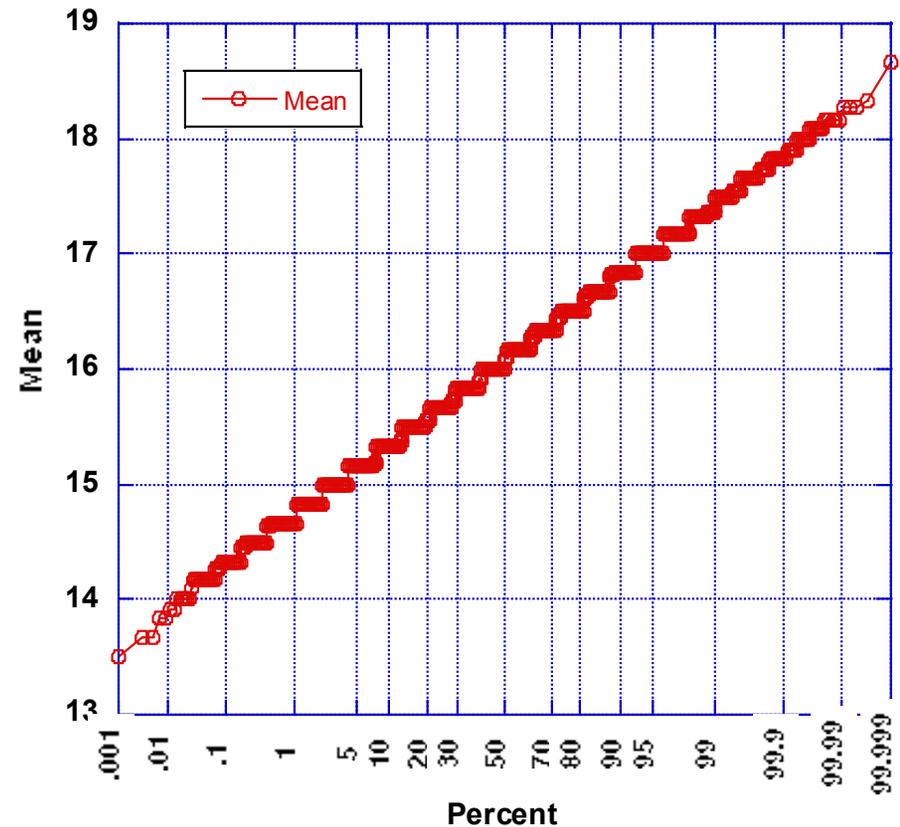
How to Look at Simulation Data

Histograms



Visually assess normality, spread, and quantization

Probability plots



- Assign percentiles to possible results
- Plot results vs probability.
- Normal is straight line.
- Read off Confidence intervals.

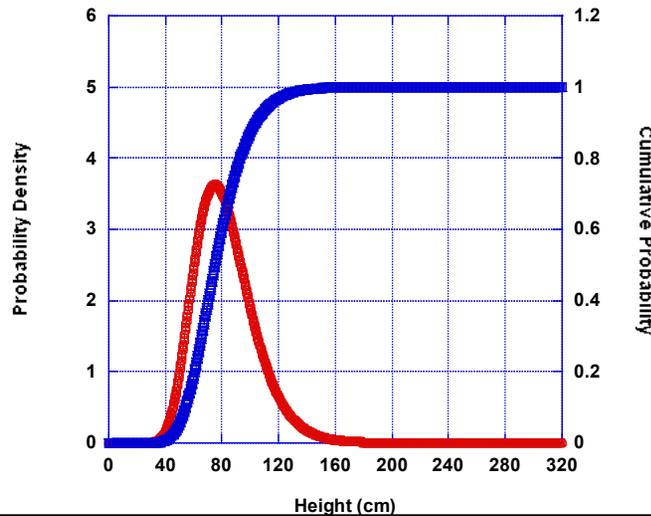
Assumptions We Violate in Established Procedures

Using Too Few Drops:
25 and 15 instead of
50.
(error will be
underestimated)

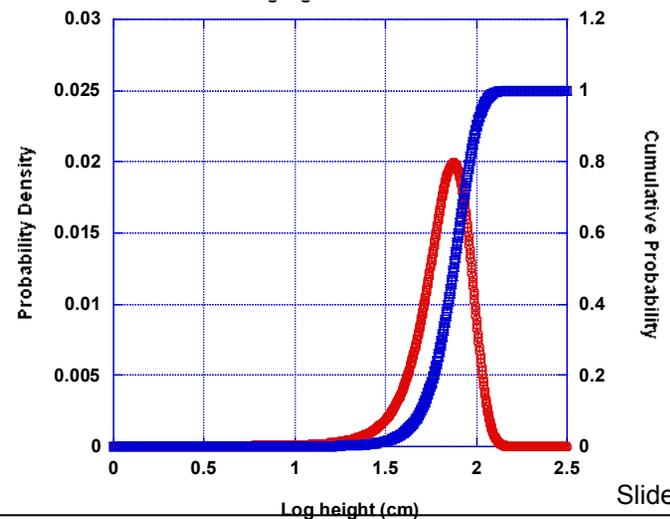
Ht (log)	Ht (cm)	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	Σ E	Σ N
1.5563	36.0																										0	0
1.5051	32.0										E																1	0
1.4548	28.5	E		E		E		E		N		E		E		E		E		E					E	10	1	
1.4065	25.5		N		N		N		N				N		N		N		N		E		E		N		2	9
1.3522	22.5																					N		N			0	2
1.3010	20.0																										0	0
#NUM!																											0	0
#NUM!																											0	0
#NUM!																											0	0
#NUM!																											0	0
#NUM!																											0	0
#NUM!																											0	0
Totals:																									13	12		

Incorrect step transform for sample distribution. (Don't know which one is correct.)

Non transformed steps with Log Normal Dist' n

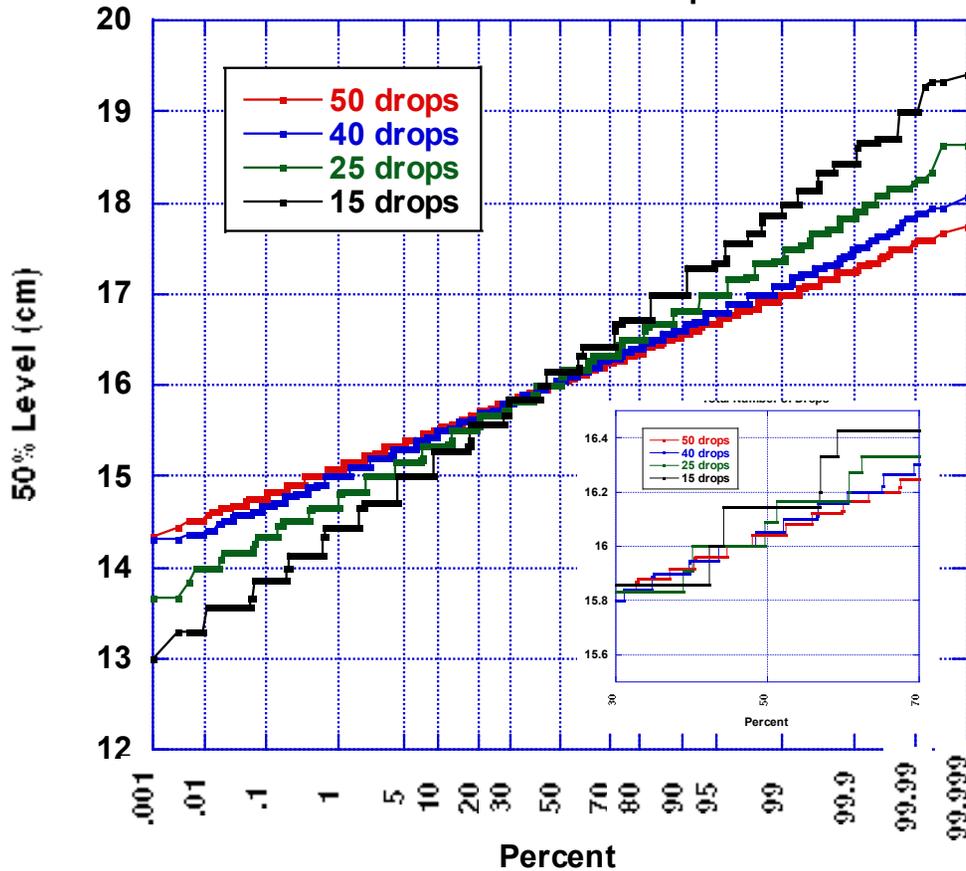


Log transformed steps with Normal Dist' n



50,40,25,15 Drops (Normal in Untransformed Steps)

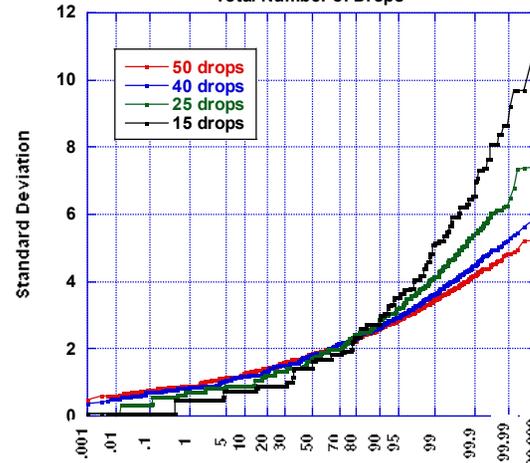
Dependence on 50% Level Probability on Total Number of Drops



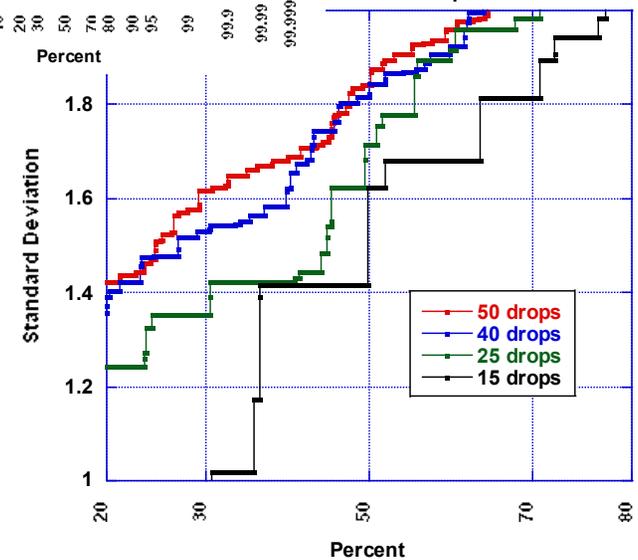
n=50: 1.9 cm
 n=40: 2.1 cm
 n=25: 2.7 cm
 n=15: 3.5 cm

98% Confidence Intervals

Dependence on 50% Level Probability on Total Number of Drops



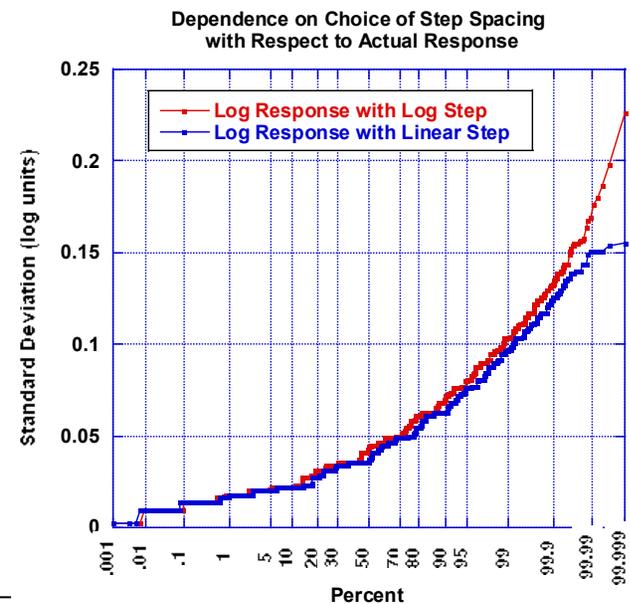
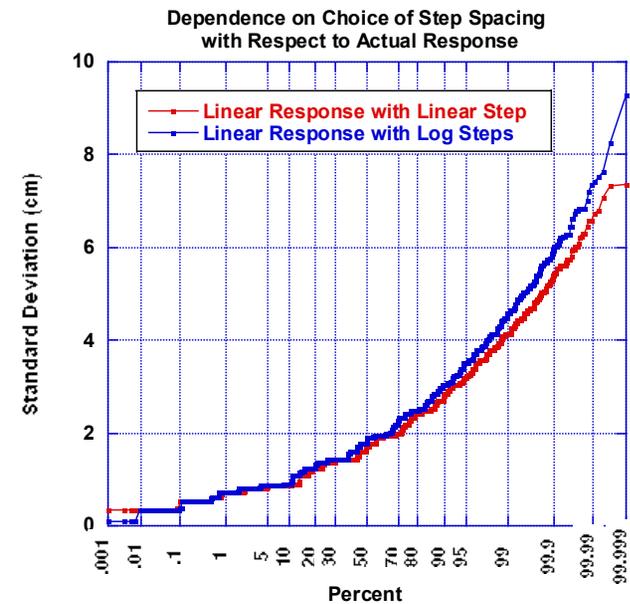
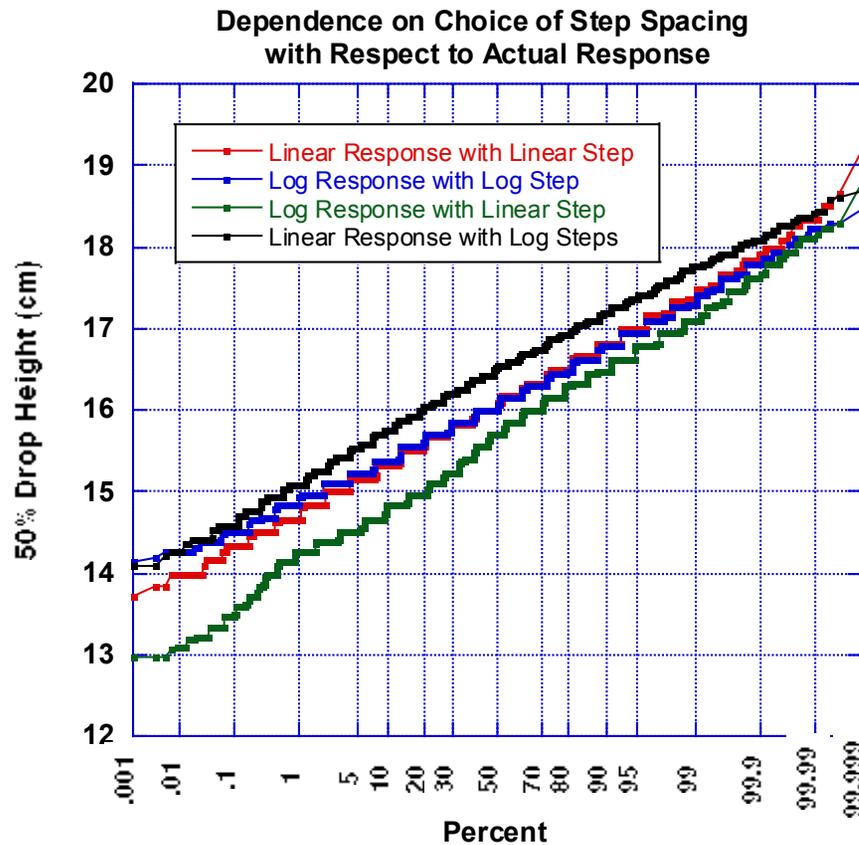
50% Level Probability on number of Drops



- Slightly higher error than \sqrt{n} .
- Twice the error in Std. Dev.



Effect of Step Transform vs. Response Type



- Wrong transform always misses the true mean
- Either one misses by similar amount.
- Similar errors in Standard Deviation.
- Have to evaluate magnitude vs. mean, step size, etc.

Summary and Conclusions

Standard statistical approaches will let us evaluate results

- Report expected error in results at various confidence levels
- Report confidence intervals of mean values
- Compare multiple results simultaneously

New information obtained to help with Bruceton Testing Evaluation

- Additional functions let us evaluate expected error in mean and std dev.
- Goes into detail on multiple test comparison

Procedures violate assumptions. Can evaluate additional error with simulations.

- Will let us determine magnitudes of error for various violations.
- Will have to be examined over range of parameters encountered.
- Examine cases for 15, 25, and 40 drops

Overall statistical approach assigns significance to differing results.

Tells us where to spend more effort examining procedures or detection methods and how often particular materials vary among participants.

Appendix E. Presentation—NSWC IHD IDCA Effort



NSWC IHD IDCA Effort

IHD IDCA Team: Daniel N. Sorenson, Daniel L. Remmers, and
Kirstin F. Warner

Kirstin F. Warner

Date: November 9-10 11





Financials

- **Title of Effort:** IDCA Support
 - **PR:** HSHQDC-10-X-00414
 - **Total Funding Received:** \$200,000 (FY 11)
-



Status/Outstanding Data

Sample ID	Status
HP/NM	SSST needed
HP/Glycerin	SSST needed
HP/Cumin	SSST ongoing
UN series	Awaiting SSST data

All Data will be submitted by Dec. 15



Testing Issues

- **Liquid/Paste Materials**

- Standardization/Differences between labs
- NSWIC IHD – Cavity Drop Impact Tester
 - HP/Glycerin
 - HP/NM

- **IHD will test HP mixtures using cavity drop impact and bare tools**

- **TEAM NEEDS TO AGREE**



HP/Flour BAM Data

BAM FRICTION TEST

sample name: hydrogen peroxide/flour
 sample ID: KFWII96-3
 sample prep: paste
 requester: Kirstin Warner, R11KW
 operator: D. Remmers

date: 9/21/11
 temperature: 24 °C
 relative humidity: 42 %

weight#	notch#	newtons	1	2	3	4	5	6	7	8	9	10	comments
9	6	360											
9	5	324											
9	4	288											
9	3	252											
8	5	216											
8	4	192											
9	1	180											
7	6	160											
7	5	144	1										black mark
6	6	120	0	0	1								black mark
6	5	108	0	0	0	0	0	0	0	0	0	0	no reactions
6	4	96											
6	3	84											
6	2	72											
6	1	60											
4	5	54											
4	4	48											
4	3	42											
3	5	36											
3	4	32											
3	3	28											
3	2	24											
2	6	20											
2	5	18											
2	4	16											
2	3	14											
2	2	12											
1	6	10											
1	4	8											
1	2	6											

1 = positive reaction (fire)
 0 = negative reaction (no-fire)

BAM threshold: **108** newtons

The BAM threshold is the level at which 10 negatives are observed with at least one positive at the next higher level.

Tested on BAM friction tester; with porcelain plates and porcelain pegs, in building 888 room 104

ARC Studies/Contribution to Compendium



- Thermal and pressure hazard parameters related to ARC data include:
 - onset temperature
 - adiabatic temperature rise
 - pressure developing rate (PDR)
 - self heating rate (SHR)
 - time to maximum rate (TMR)
 - evaluate worst-case energy release
 - probability of the occurrence of an incident, or the occurrence of a thermal runaway reaction



ARC Studies

Sample ID	Mass (g)	ARC Exo. Onset °C	ARC Exo. Max °C	ARC Self -Heating Max °C/Min
Bullseye Gunpowder	0.1015	145	195	216
AN/Gunpowder	0.1008	140	175	483
AN (60C-screen)	0.2522	260	275	0.05
KC/Sugar	0.1012	130	200	507
UN/AI	0.1001	116	128	0.38
UN/AI/S	0.1099	85	115	0.66
RDX	0.1004	196	271	387



Path Forward

- International Round Robin Participation
- Participation in Compendium
 - Aging Studies

Appendix F. Presentation—An Investigation of Modifications to the Type-12 Impact Sensitivity Test Apparatus for Explosives

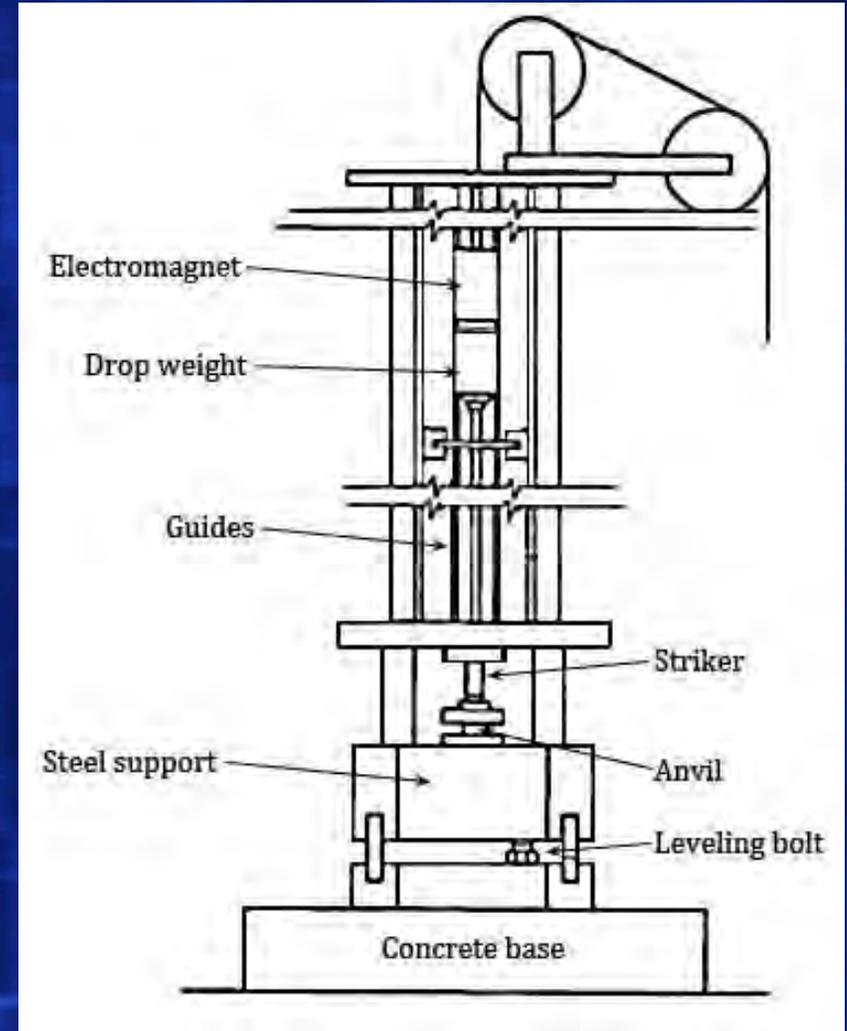
An Investigation of Modifications to the Type-12 Impact Sensitivity Test Apparatus for Explosives

Jason Phillips

NMT – Energetic Materials Research and Testing Center

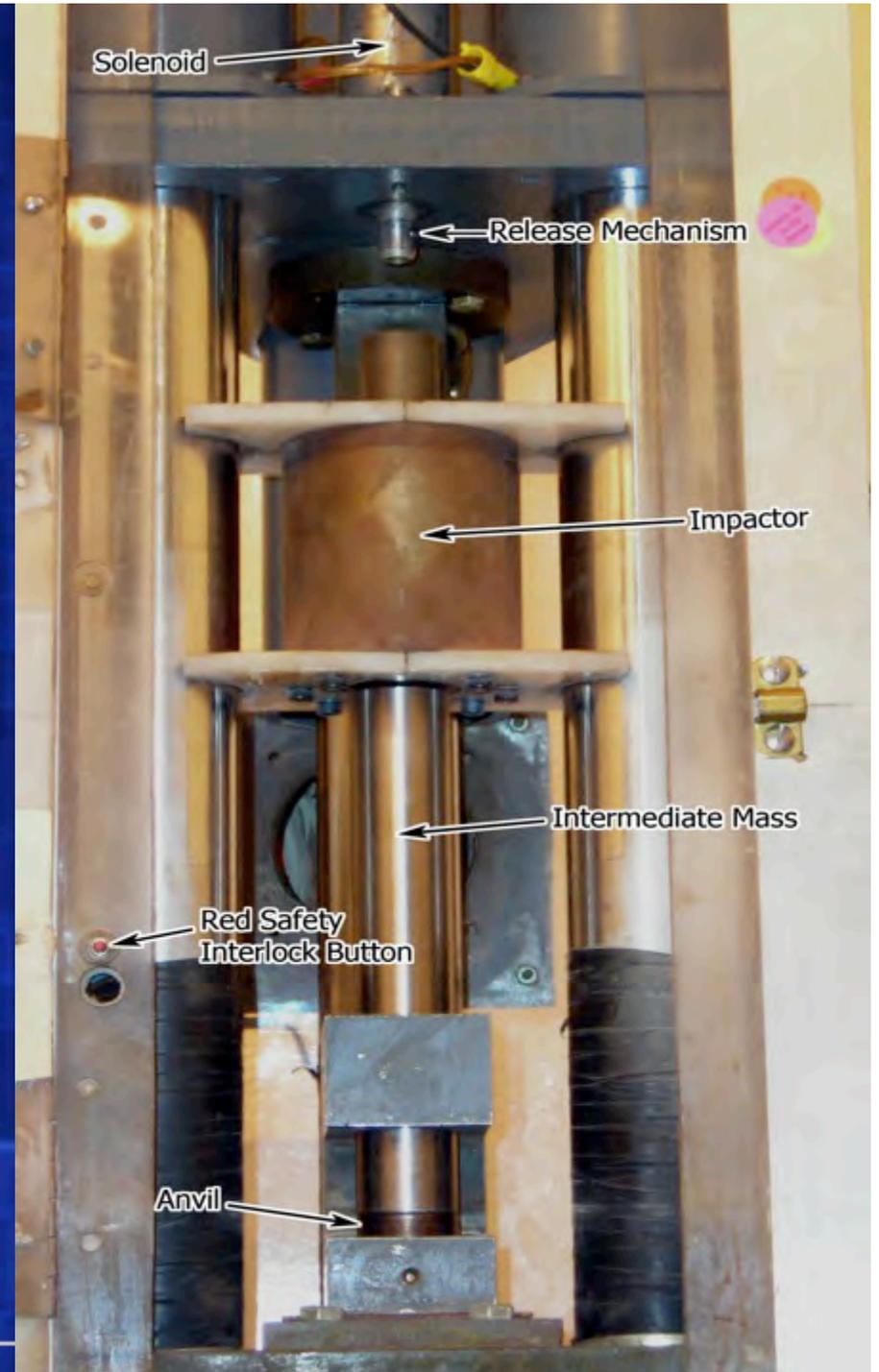
ERL Type-12 Impact Apparatus

- Variations of this design are in use at LLNL, LANL, China Lake, and EMRTC among others.
- Many subsequent designs are based on this apparatus.
- The Type 12/12A tooling utilizes a sandpaper sample surface. The type of sandpaper is not standardized among facilities. The mass of the drop weight varies between facilities and is not standardized.



EMRTC Type-12 Impact Apparatus

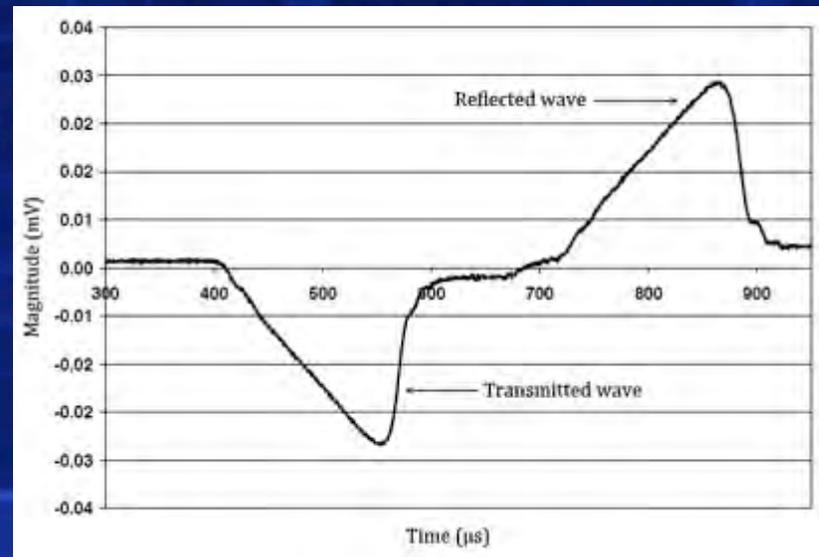
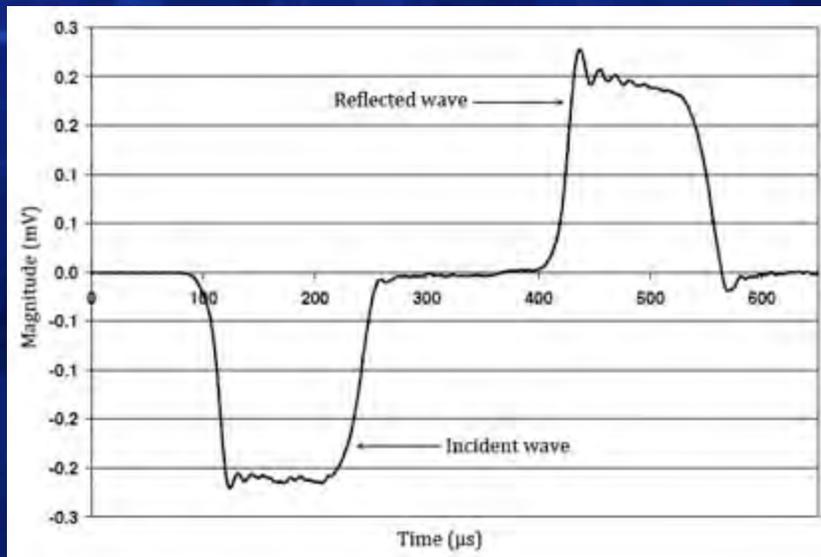
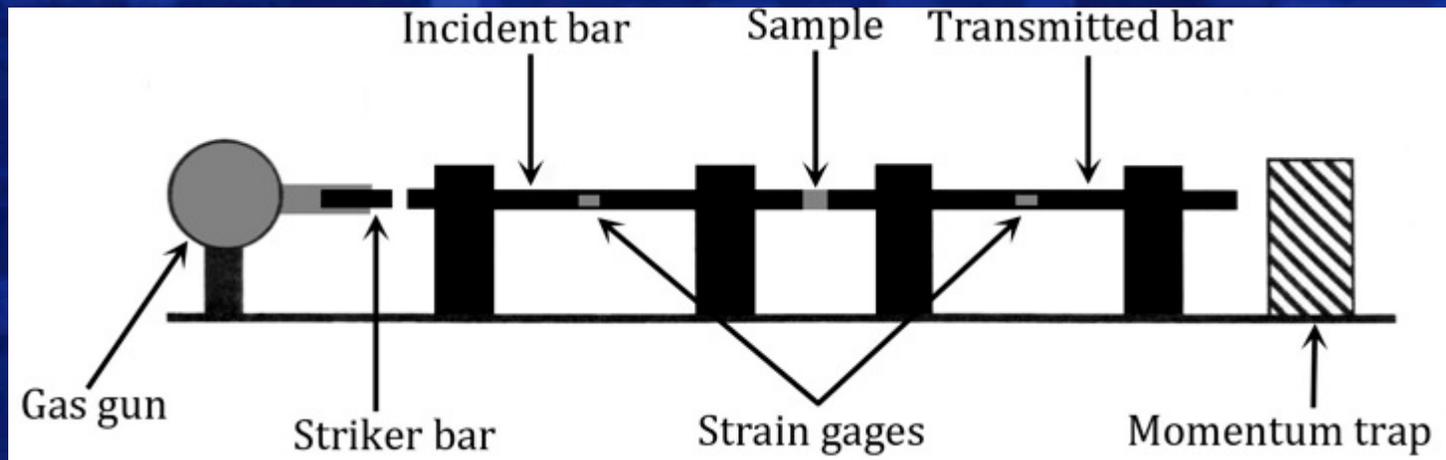
- Electronic height adjustment *via* a lift motor and threaded rod
- Electrically actuated solenoid used to release the weight
- No counterweight necessary
- Utilizes Type-12A tooling



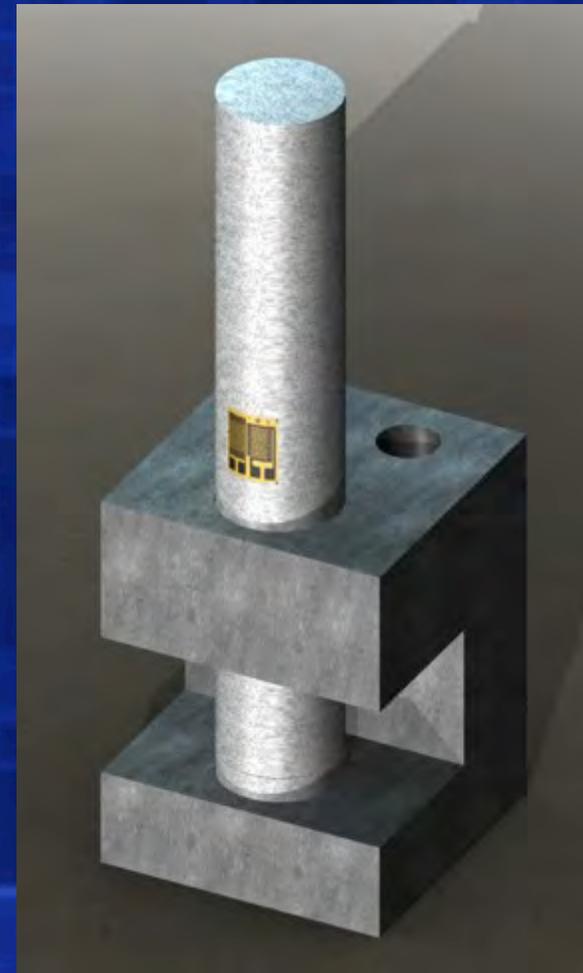
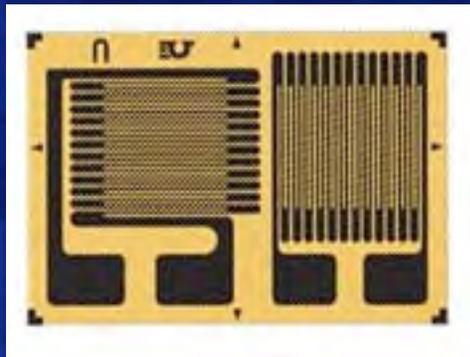
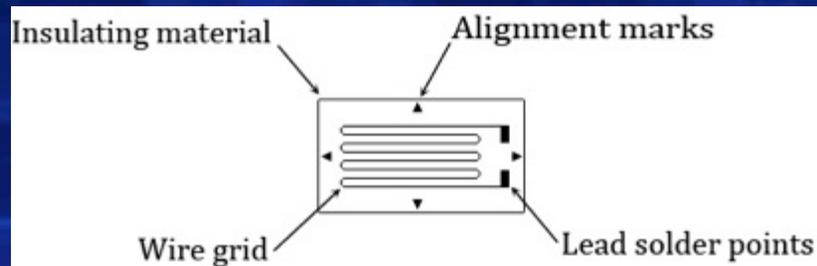
EMRTC Type-12 Tooling



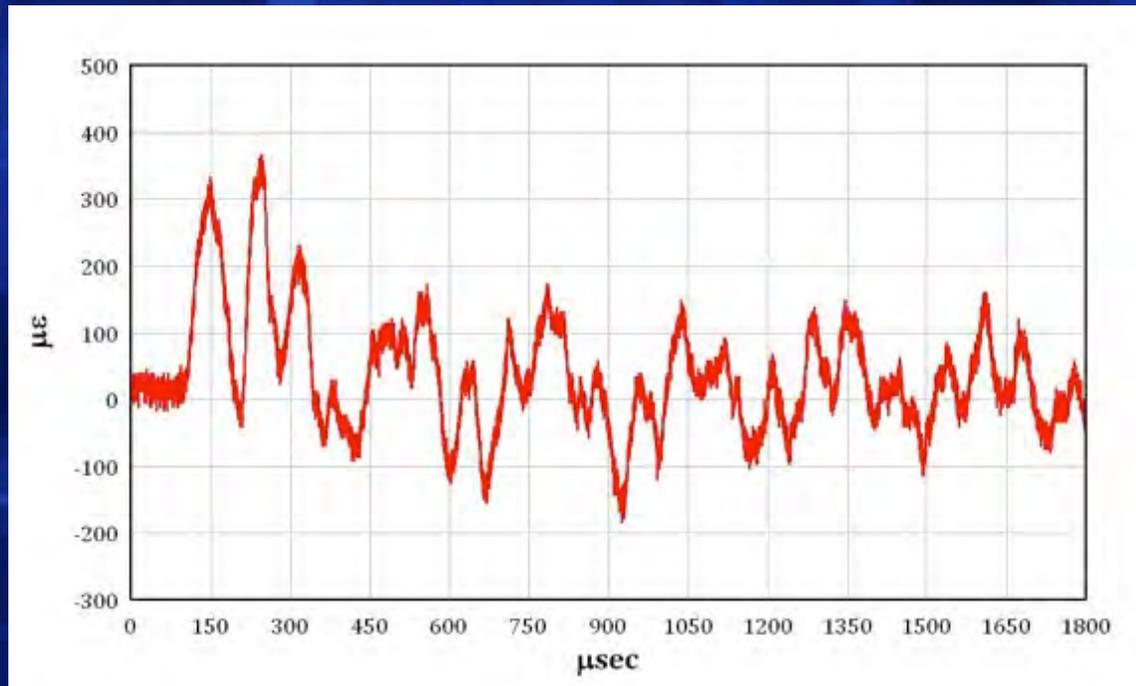
Split-Hopkinson Bar



The Resistance Strain Gage



Initial Results

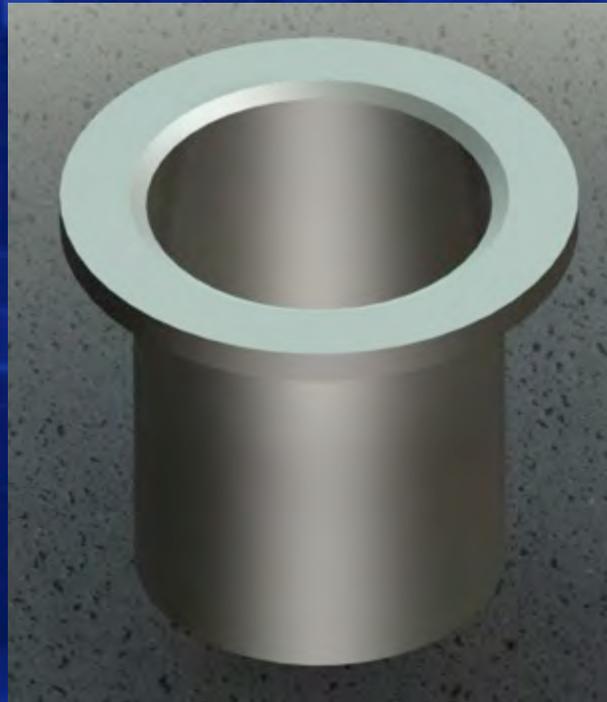


- Only recorded after multiple attempts
- Trigger manipulation did not solve this issue
- It was hypothesized that the steel-steel confinement of the striker bar in the tool holder was interfering with wave propagation

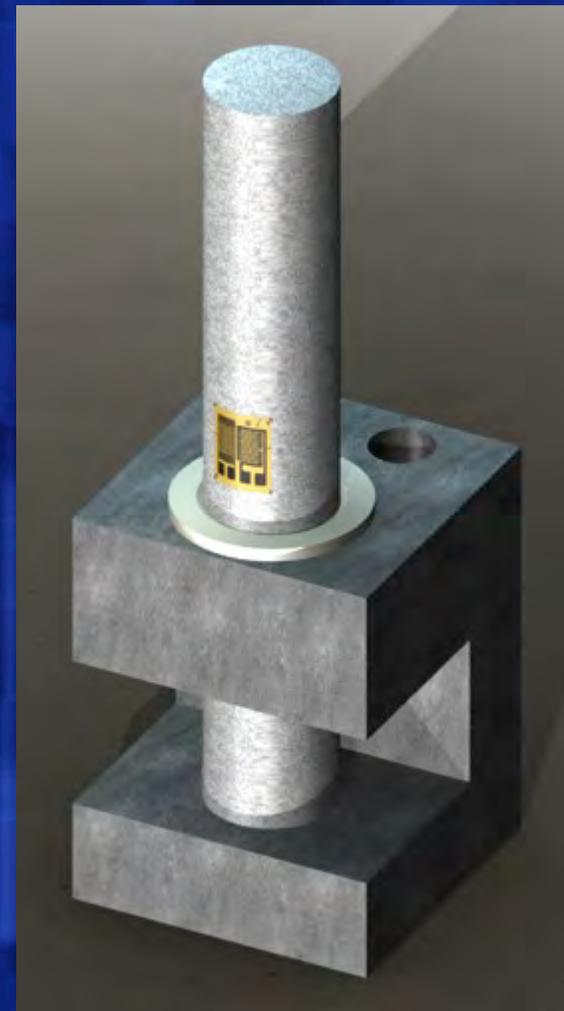
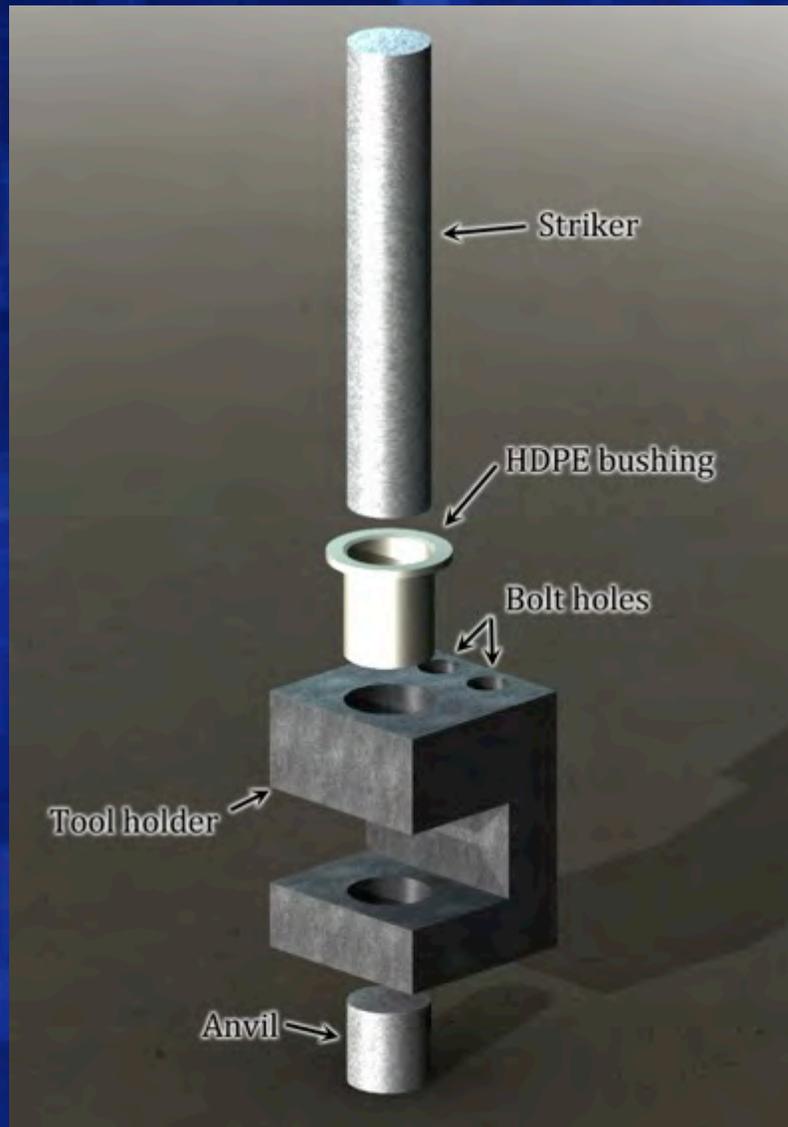
Proposed Solution

- Split-Hopkinson bars utilize linear bearings to limit all but axial movement while producing a minimum amount of radial confinement. In Type-12 tooling, there is very little clearance between the steel striker and steel tool holder.
- The proposed solution was to reduce rigid confinement to the striker bar by placing a softer material between the striker and tool holder.
- The tool holder was enlarged and an HDPE bushing was fabricated and installed to decrease rigid confinement of the striker in the radial direction

HDPE Bushing



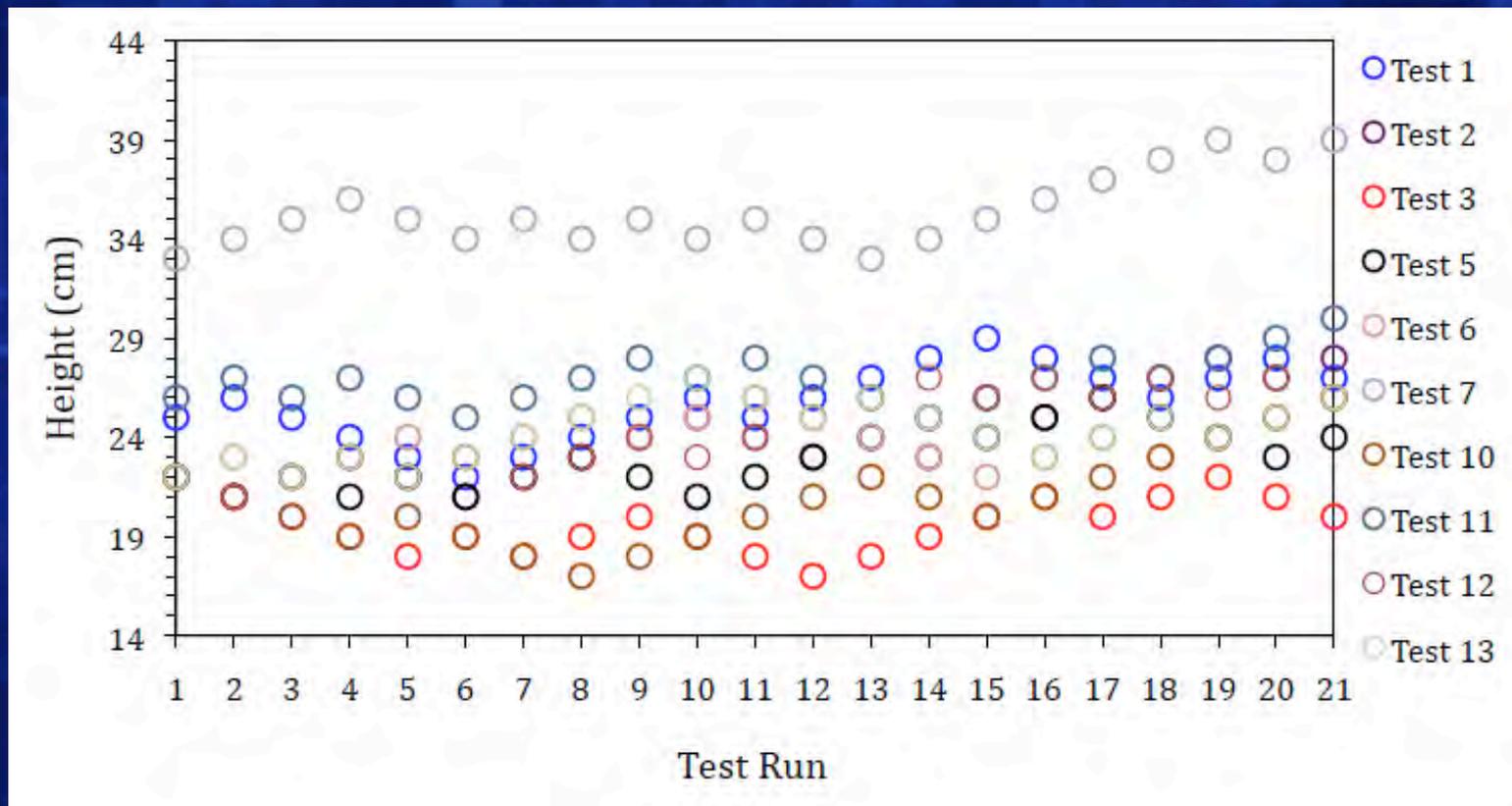
Modified Type-12 Tooling



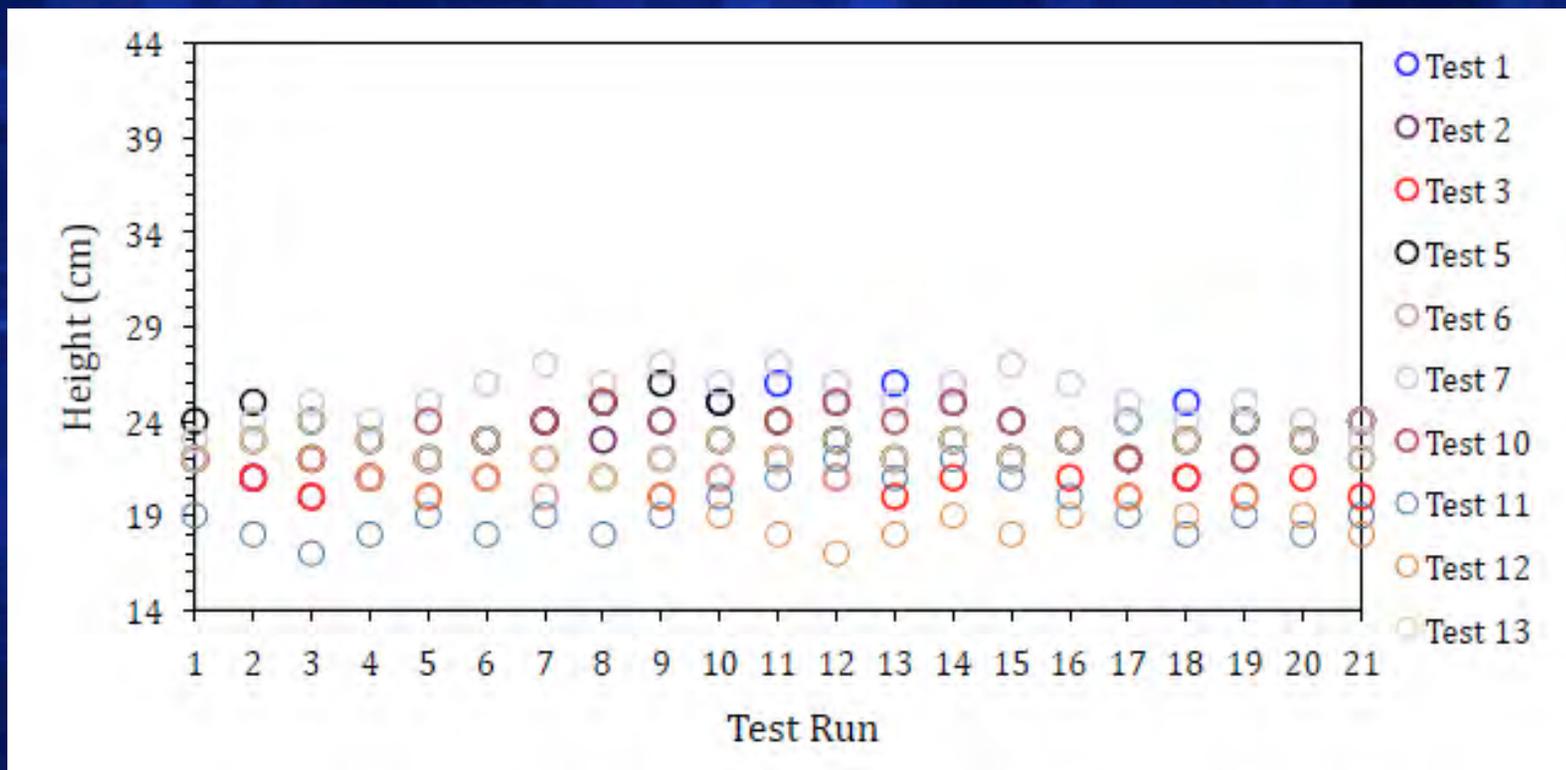
Tooling Comparison Testing

Type-12 vs. Modified Type-12

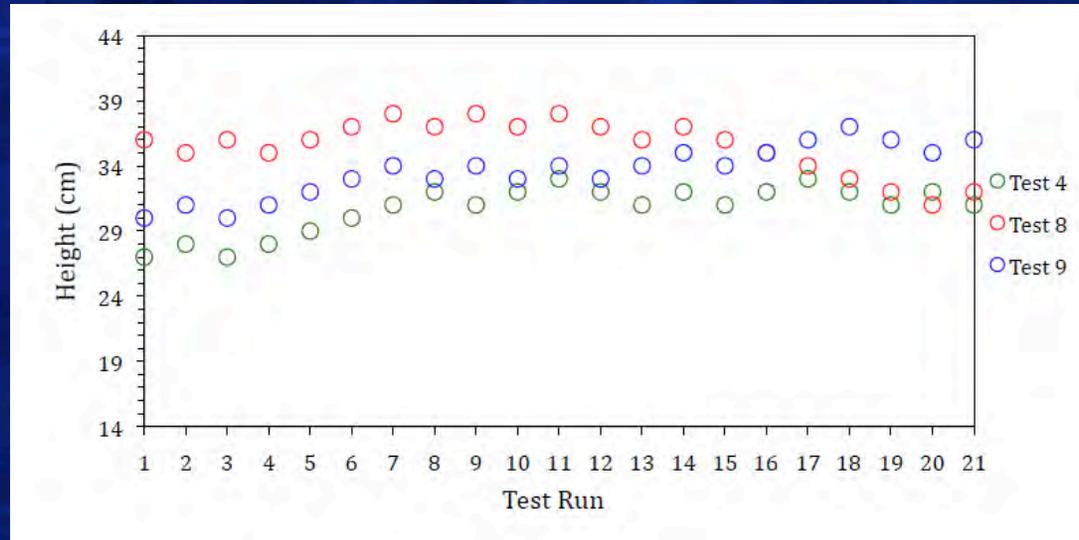
Type-12 Tooling



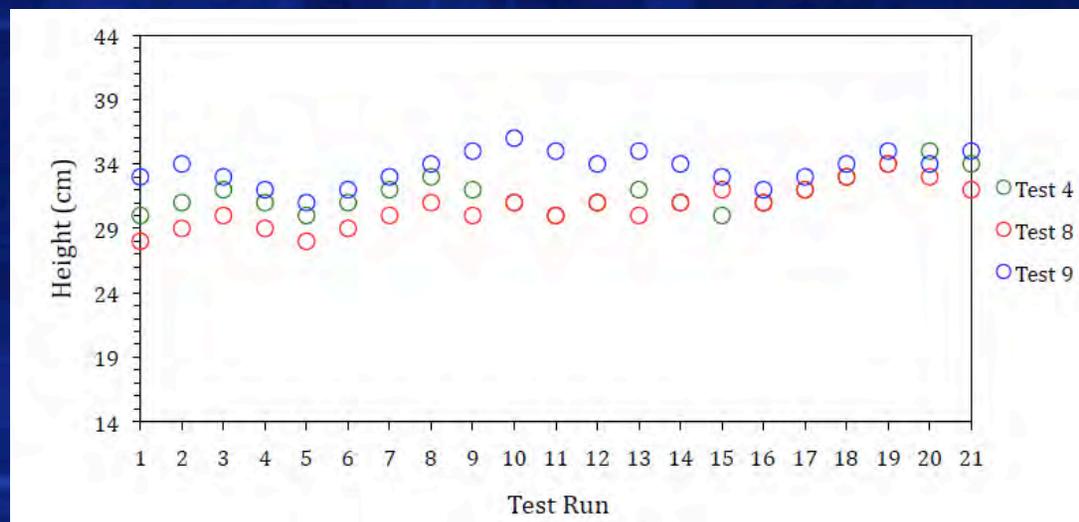
Modified Type-12 Tooling



Type-12 Tooling



Modified Type-12 Tooling



Tooling Comparison: RDX

Test	H ₅₀ Results with Std. Deviation (cm)	
	Type-12 Tooling	Modified Tooling
1	26.3 ± 4.8	23.8 ± 3.0
2	23.8 ± 6.0	22.2 ± 1.1
3	19.4 ± 2.4	20.8 ± 0.4
4	31.2 ± 3.3	31.6 ± 2.7
5	22.9 ± 3.0	23.5 ± 1.7
6	23.8 ± 1.6	21.8 ± 0.8
7	35.5 ± 5.3	25.4 ± 1.8
8	35.9 ± 5.1	30.9 ± 3.3
9	34.0 ± 5.3	33.6 ± 2.1
10	19.9 ± 2.9	23.4 ± 1.2
11	26.9 ± 2.2	19.3 ± 2.9
12	24.6 ± 6.3	19.8 ± 4.0
13	24.5 ± 2.9	22.7 ± 0.6
Mean H₅₀	26.8	24.5
Mean σ	3.9	2.0
% σ	14.7%	8.0%

Additional Material Testing

Material	H ₅₀ (cm)	σ (cm)	σ %
NH ₄ ClO ₄ + Al (70:30)	27.8	1.6	5.6
HMX	36.3	2.5	6.9
HNS II	36.3	2.3	6.2
LX-10	33.1	0.8	2.3
LX-14	31.7	2.9	9.1
PBXN-5	34.9	0.4	1.2
PETN	23.4	0.9	4.0

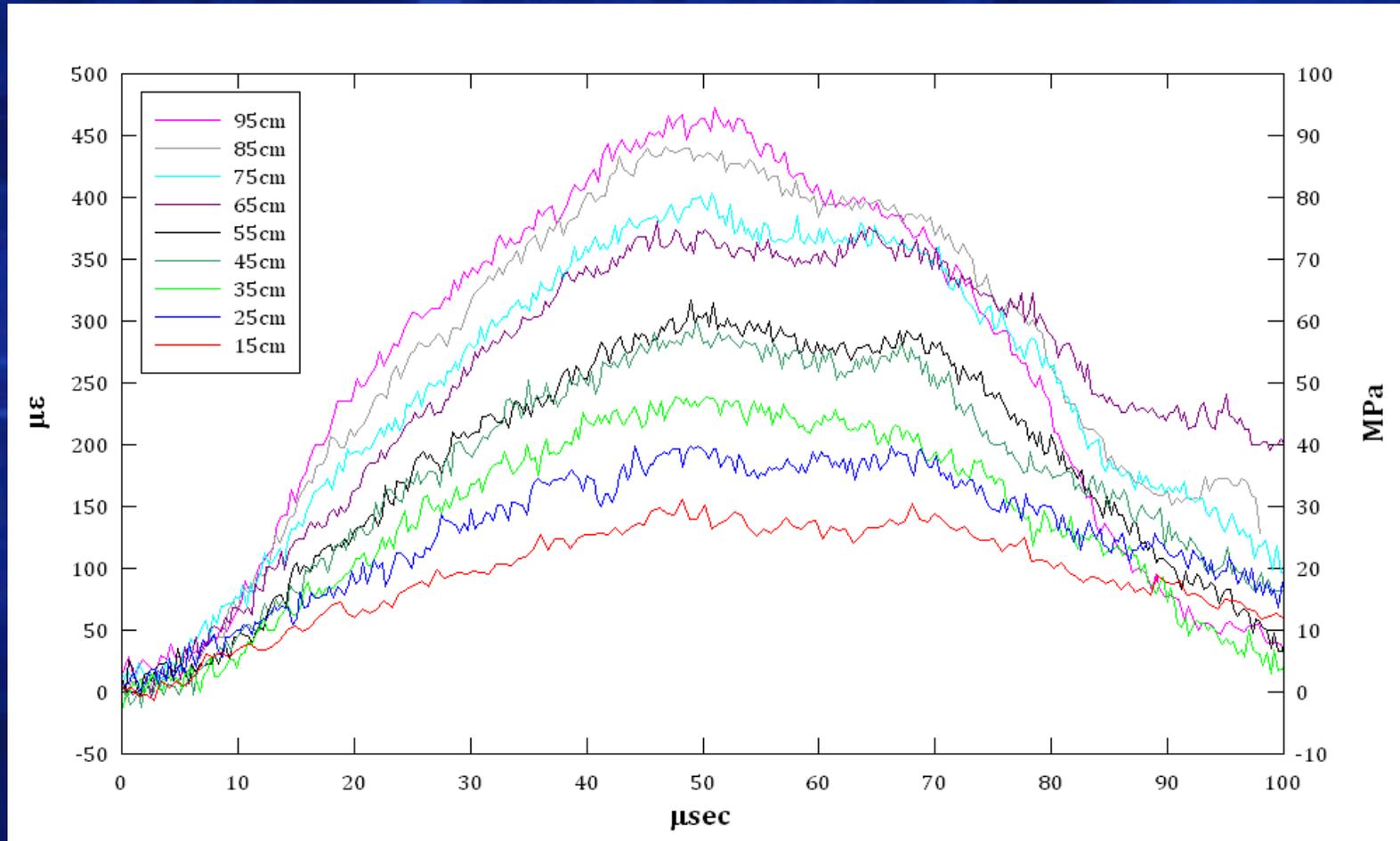
- It was determined that the modification to the Type-12 tooling created a significant improvement over the standard tooling
- The new tooling is now in use during sensitivity testing for EMRTC customers
- Since the modification created no adverse effects to impact testing, extended strain gage testing could continue

Strain Gage Testing

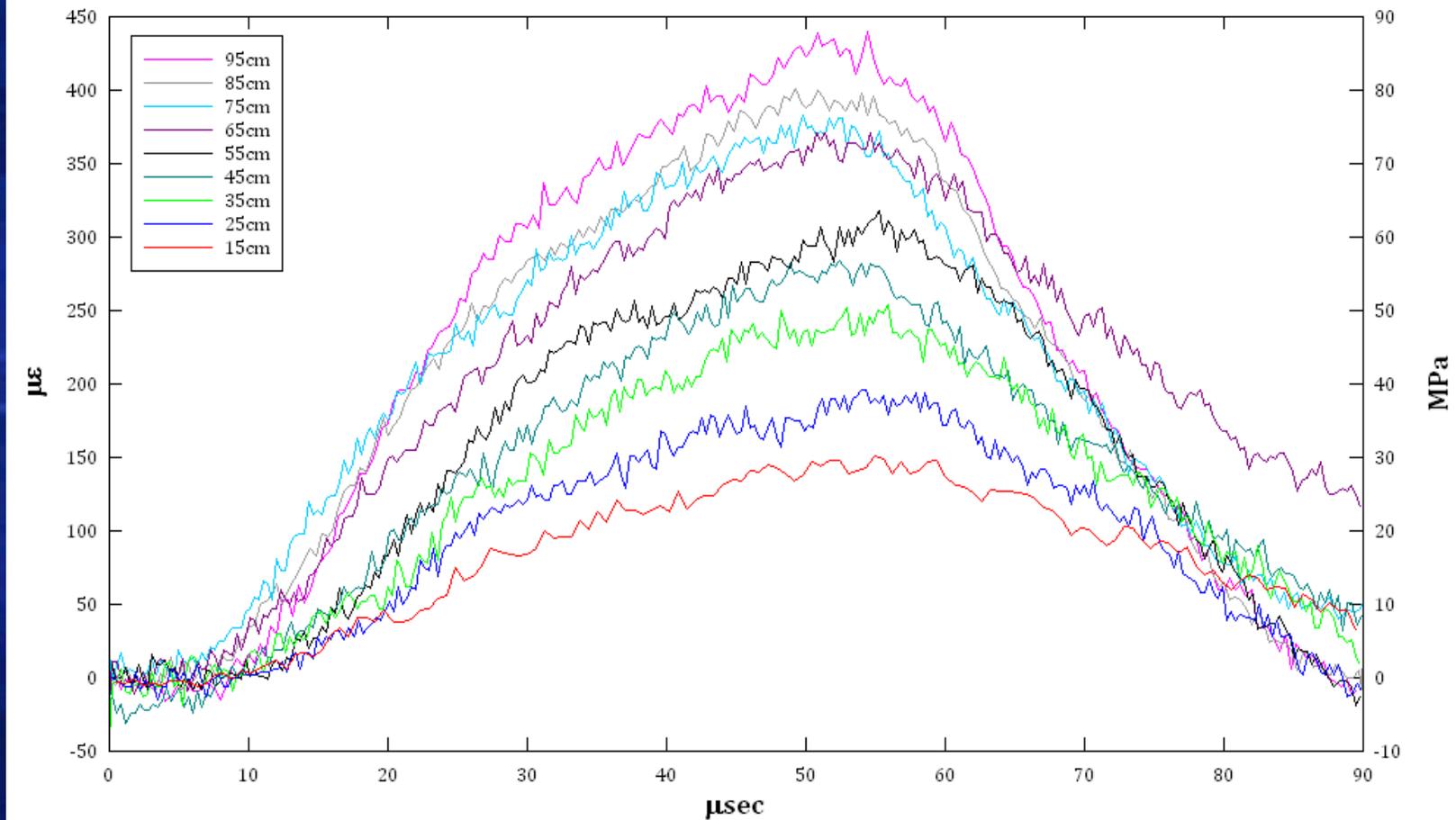
Impact Stimuli Measurements

- Solid lead core impactor (2.5kg)
 - Impacts with no material between the striker and anvil (steel-steel)
 - Impacts with only sandpaper between the striker and anvil (steel-paper)
- Lead shot core impactor (2.5kg)
 - This weight was examined using the steel-steel method
 - Due to the inconsistency of the waveform, this piece was not used for the duration of testing
- For conversion of strain to stress, the modulus of elasticity of the striker tool steel was approximated at 200GPa. (ASM Handbook, Vol. 8)

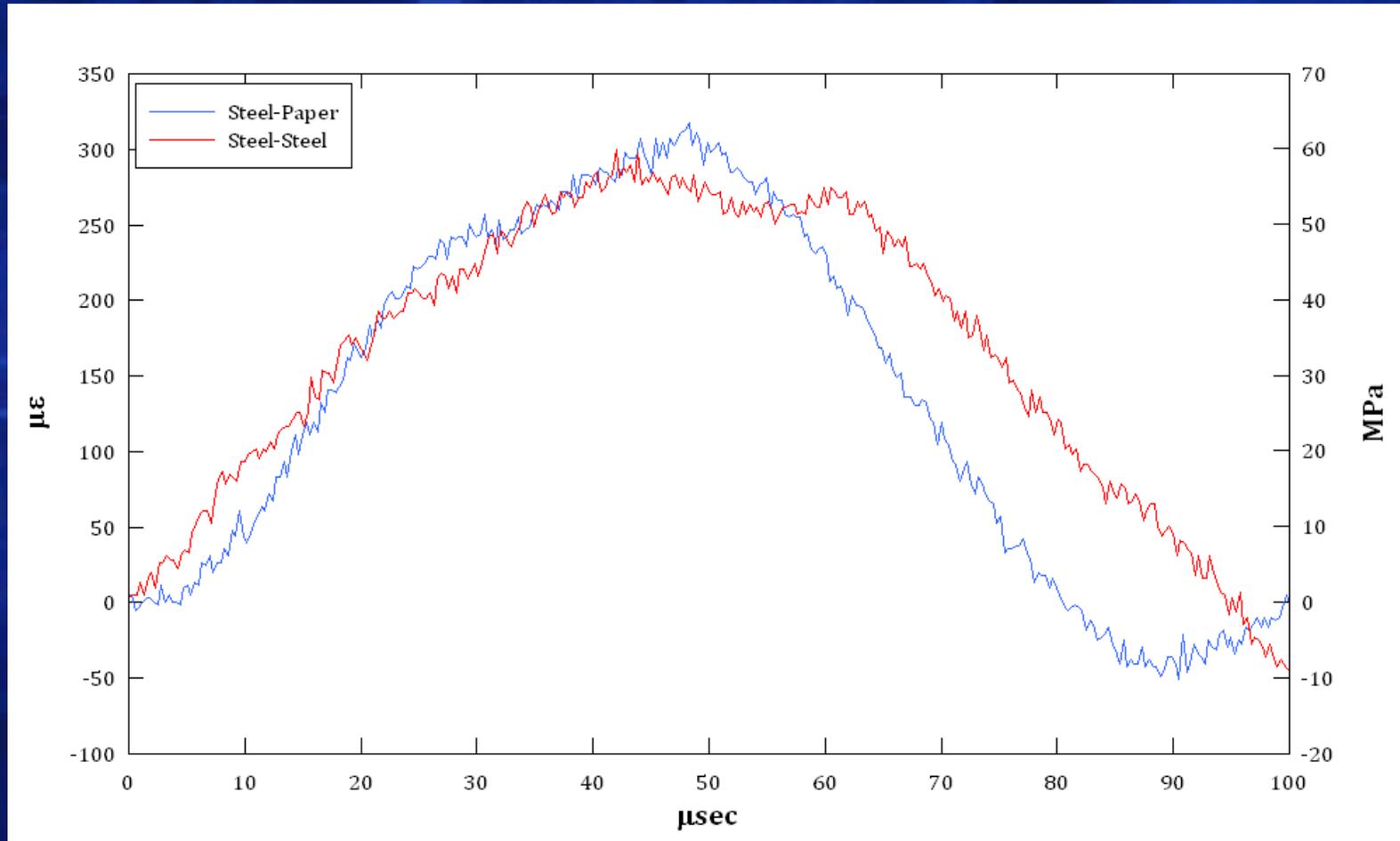
Solid Core Mass (steel-steel)



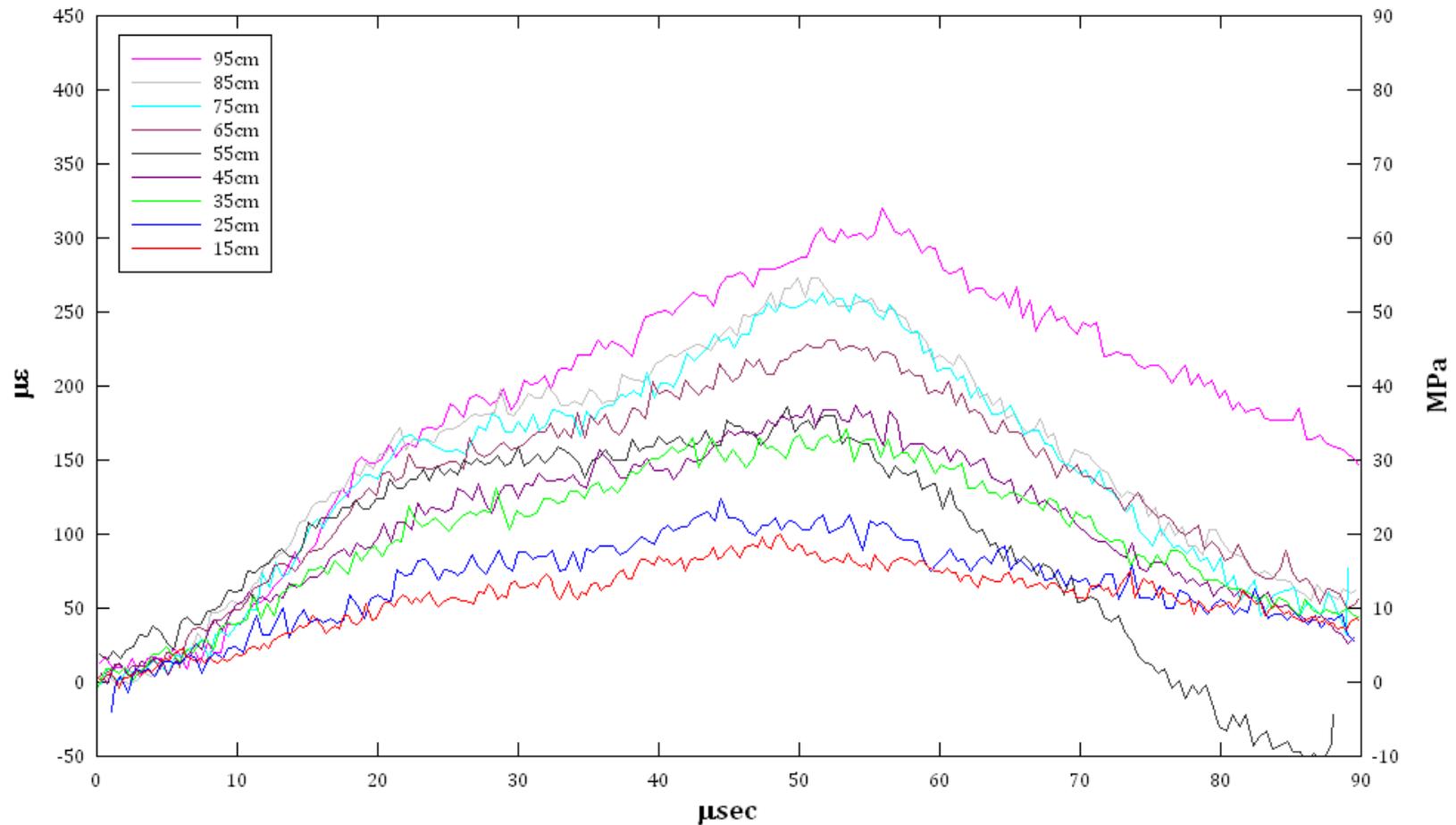
Solid Core Mass (steel-paper)



Type-12A vs. Type-12B Testing

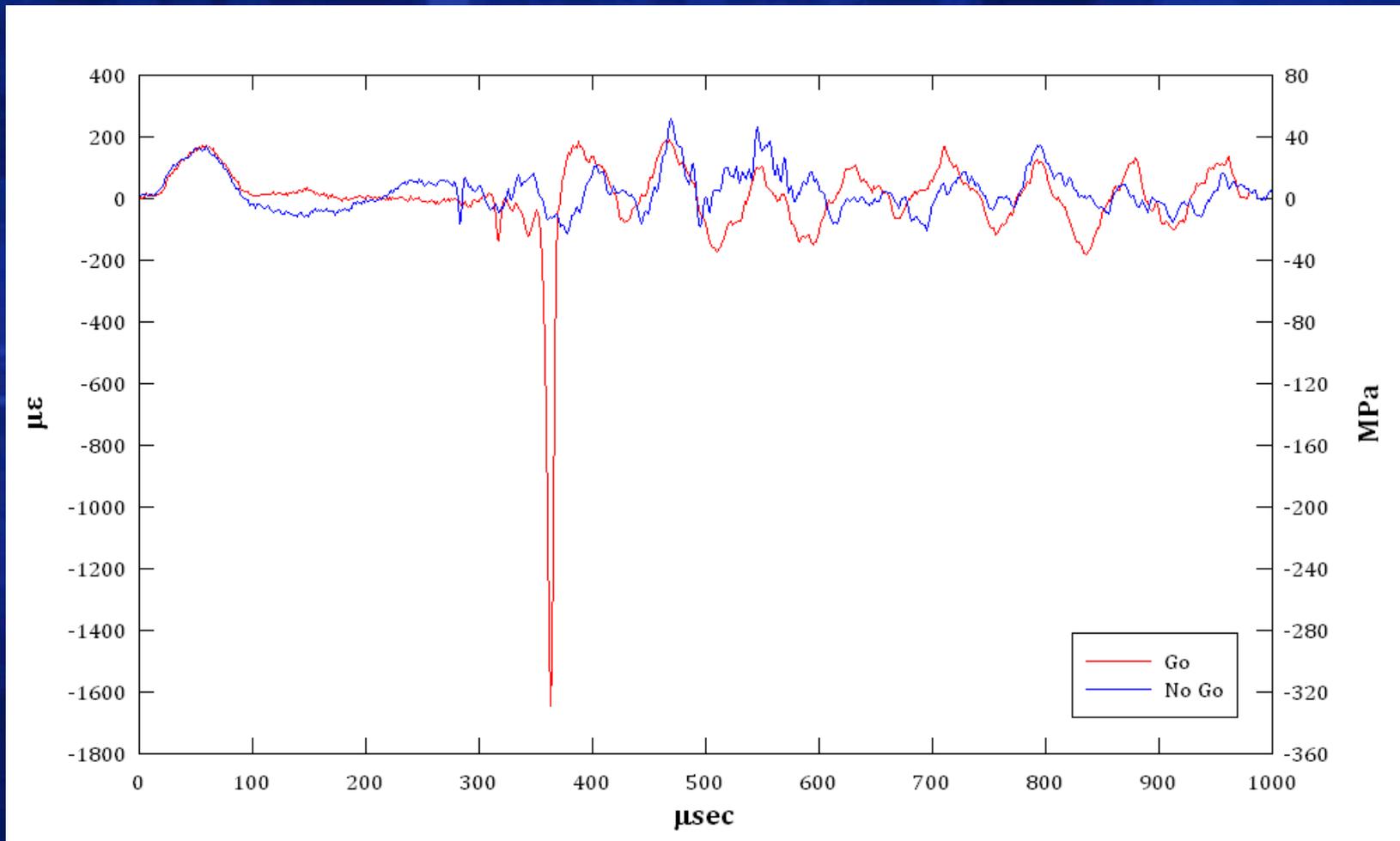


Lead Shot Mass (steel-steel)

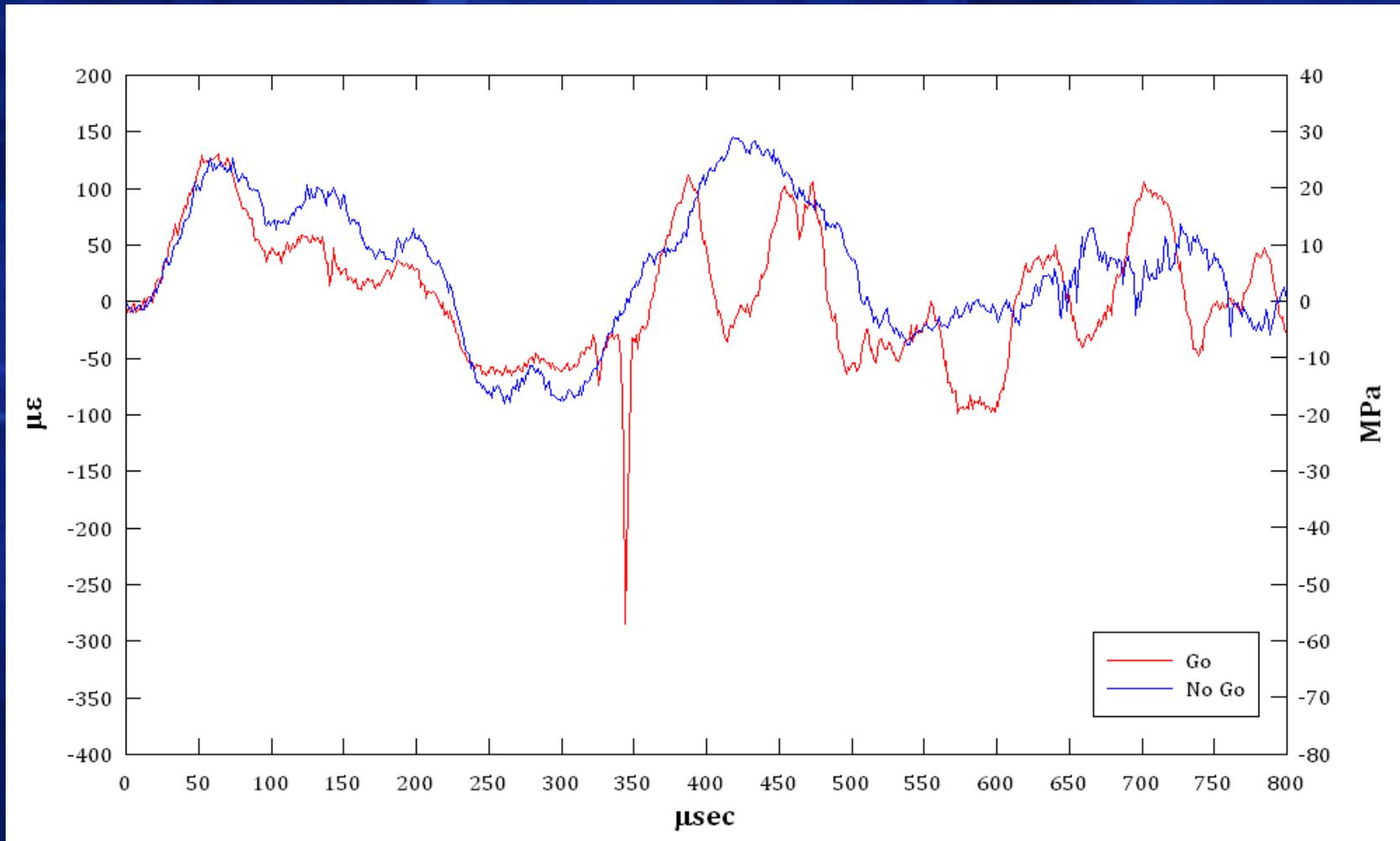


Explosive Materials Testing

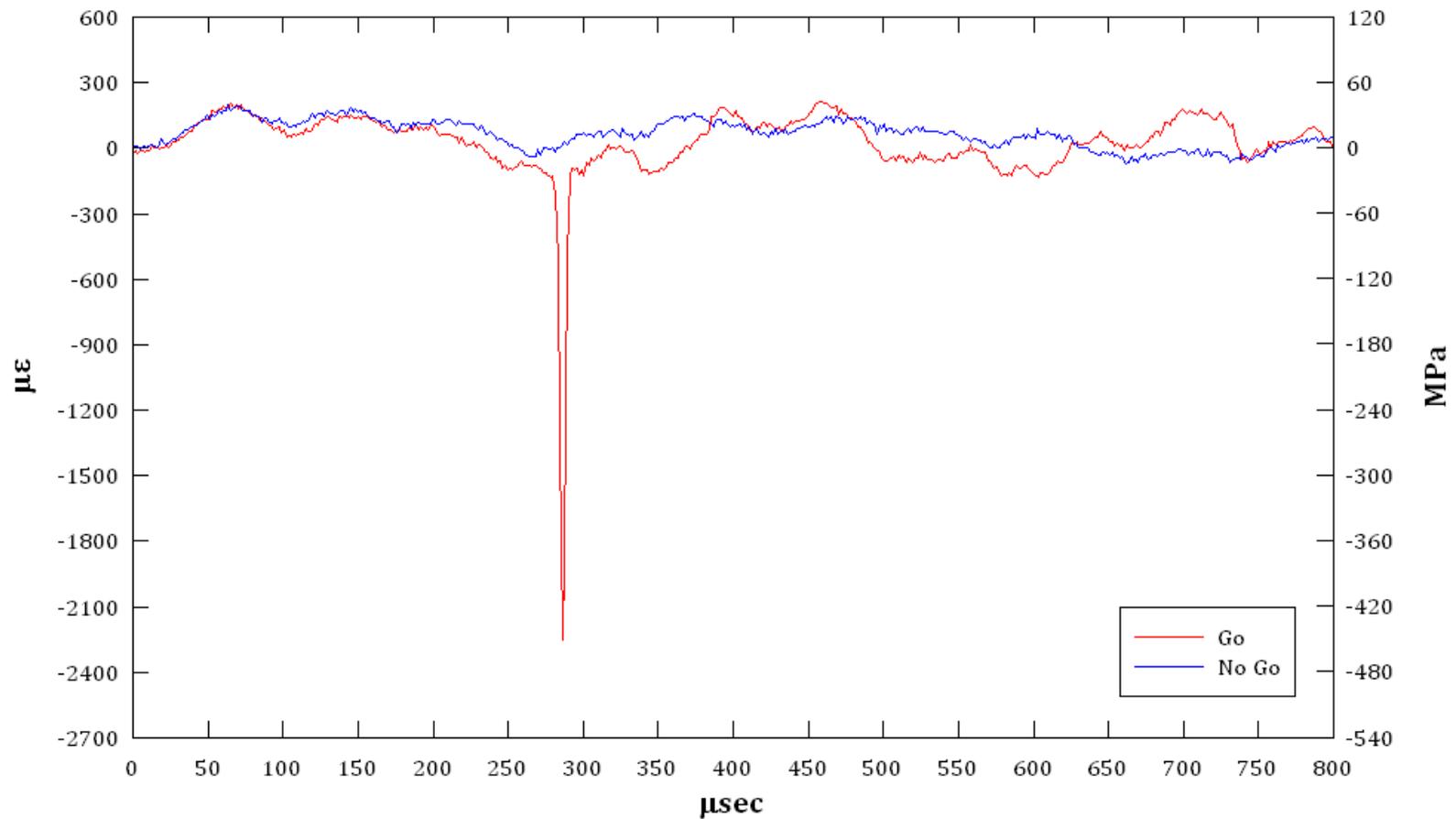
RDX: 20cm



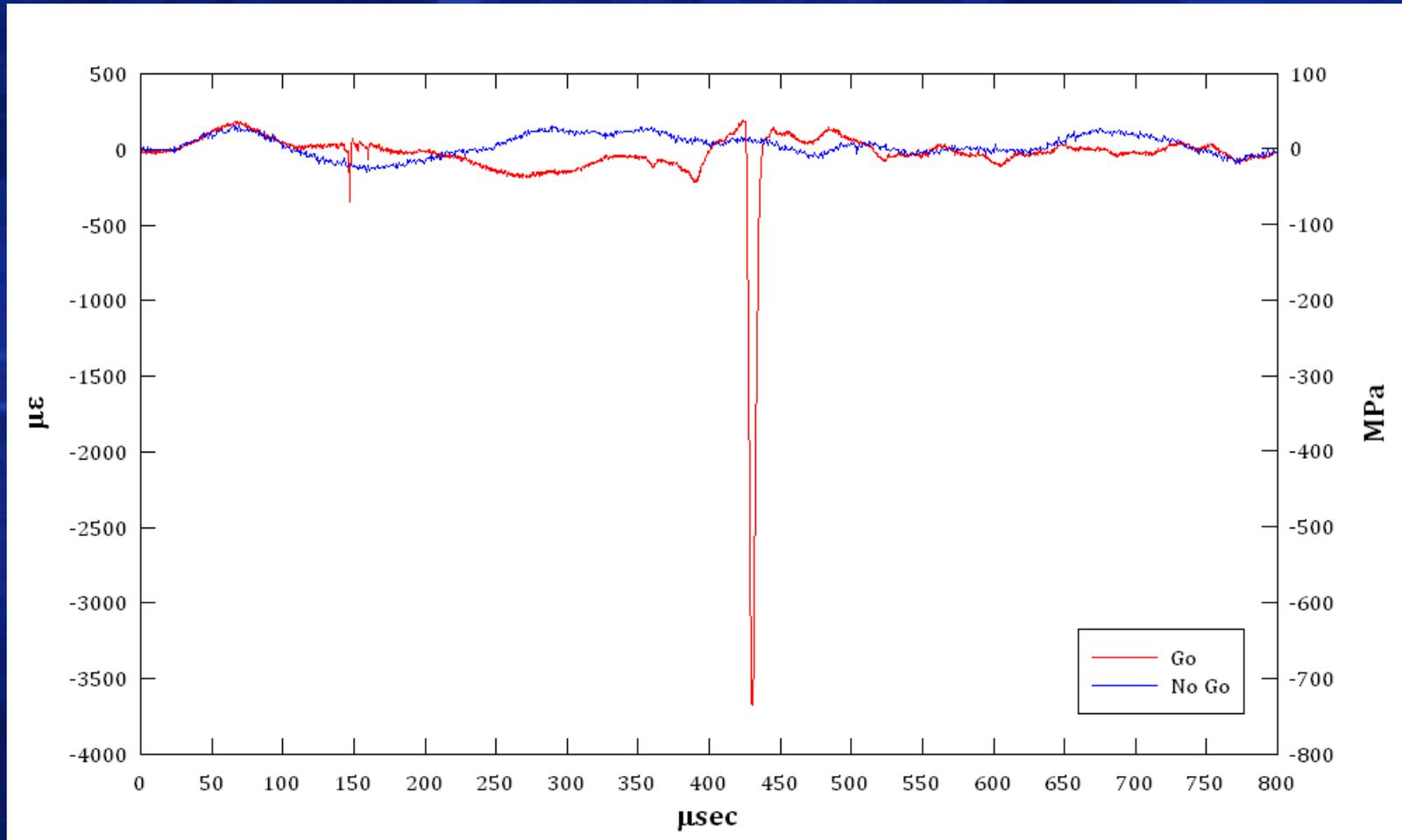
PETN: 15cm



HMX: 25cm



HNS II: 26cm

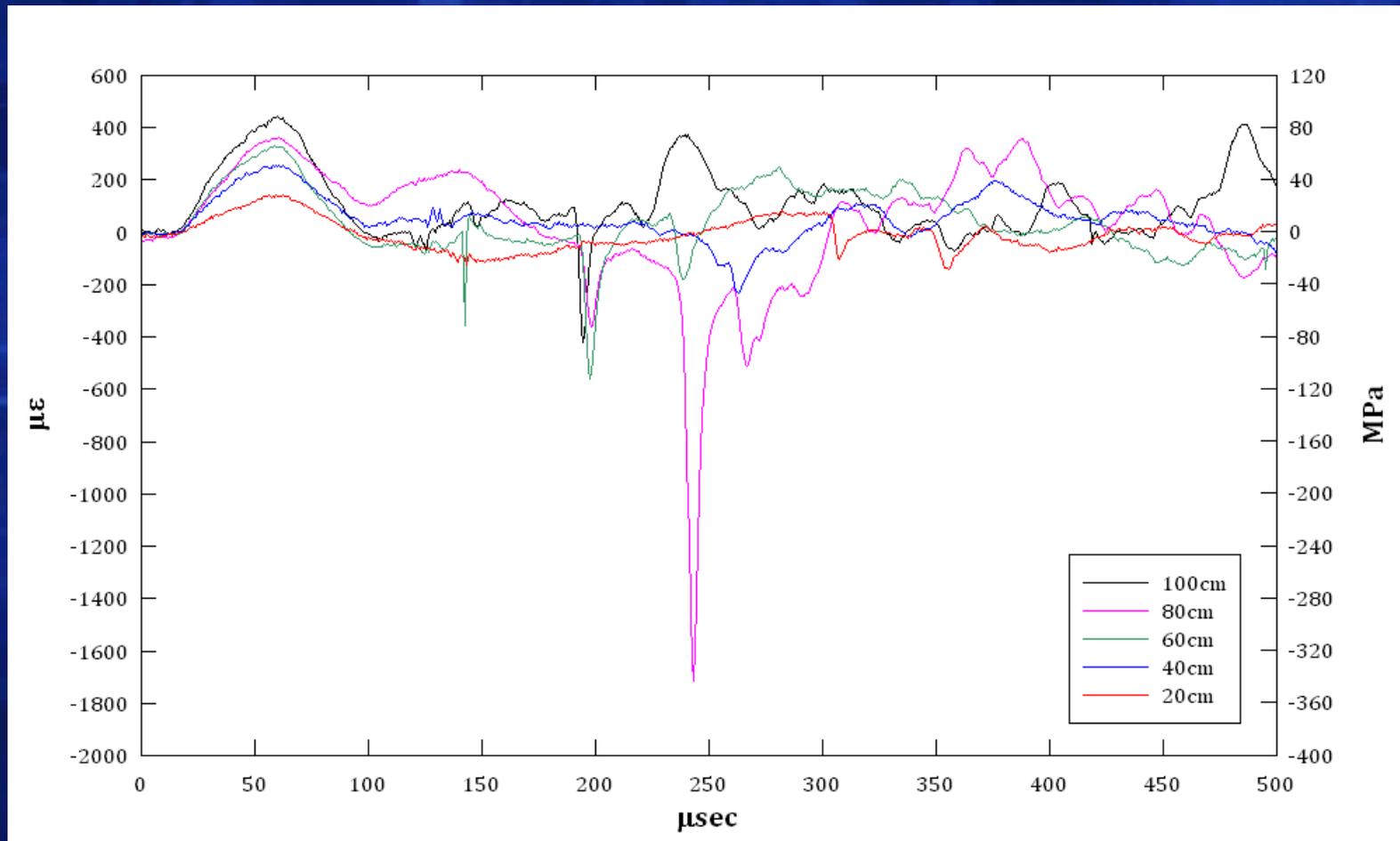


Testing of Inert Materials

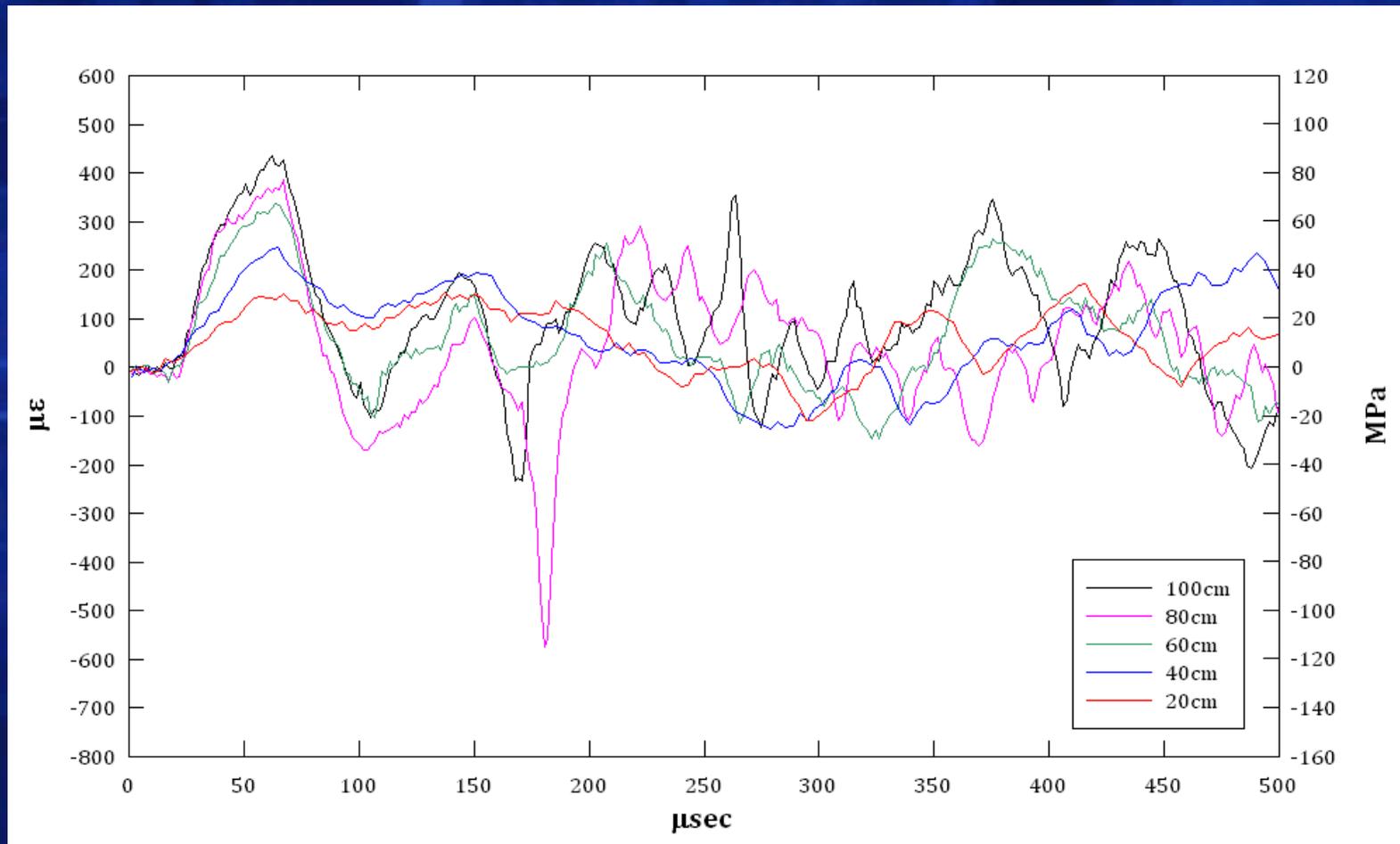
- Four inert materials were selected to study the occurrence of the rapid pressure releases seen during energetics testing
- Two factors were used in selecting these materials:
 - The material must be of similar consistency to the explosives being tested, usually a crystalline solid.
 - The materials must have varying melting points to distinguish any results due to melting.

Material	Melting Point °C
Salicylic Acid	159
Potassium Nitrate	334
Sodium Chloride	801
Sodium Carbonate	856

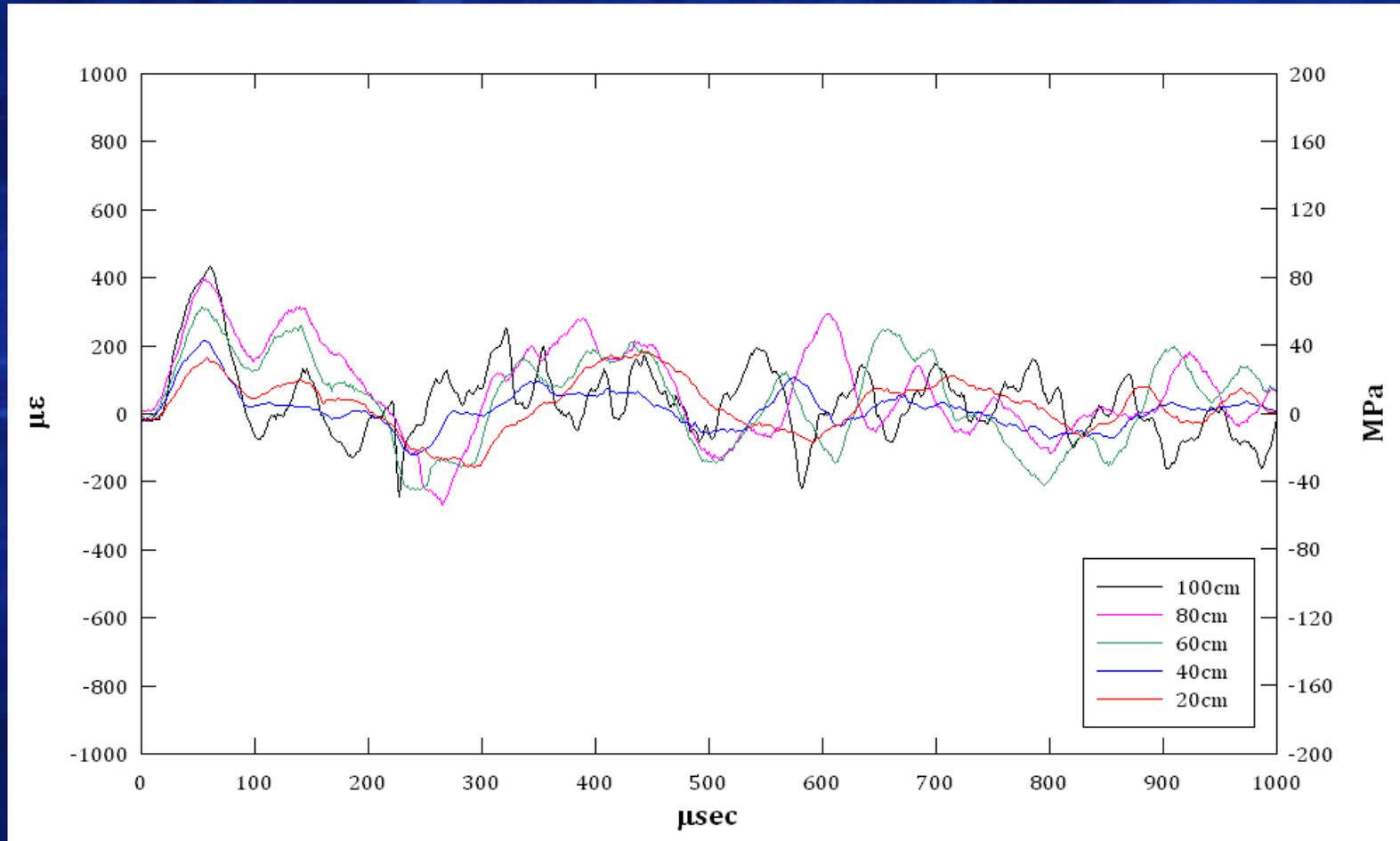
Potassium Nitrate



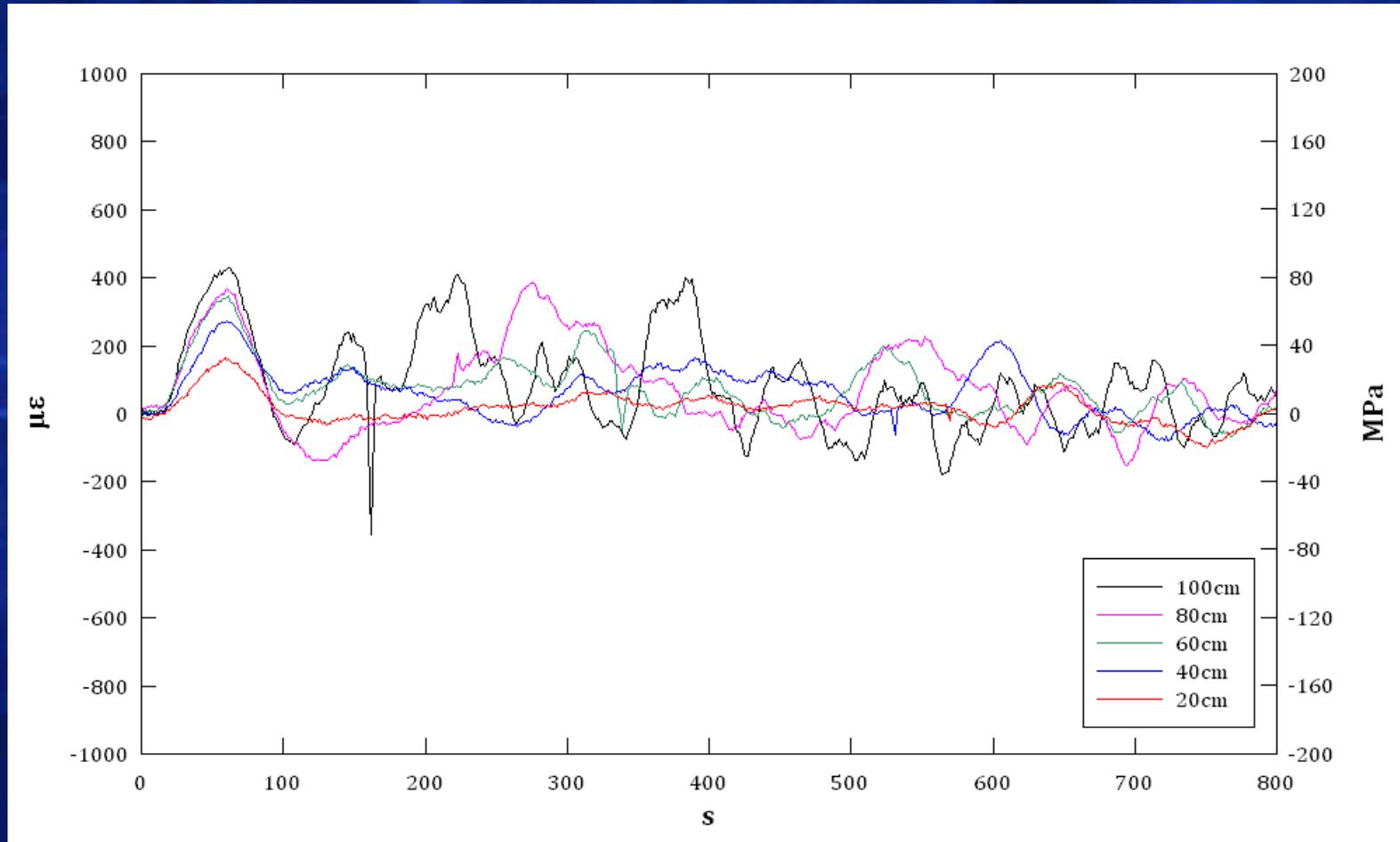
Salicylic Acid



Sodium Chloride

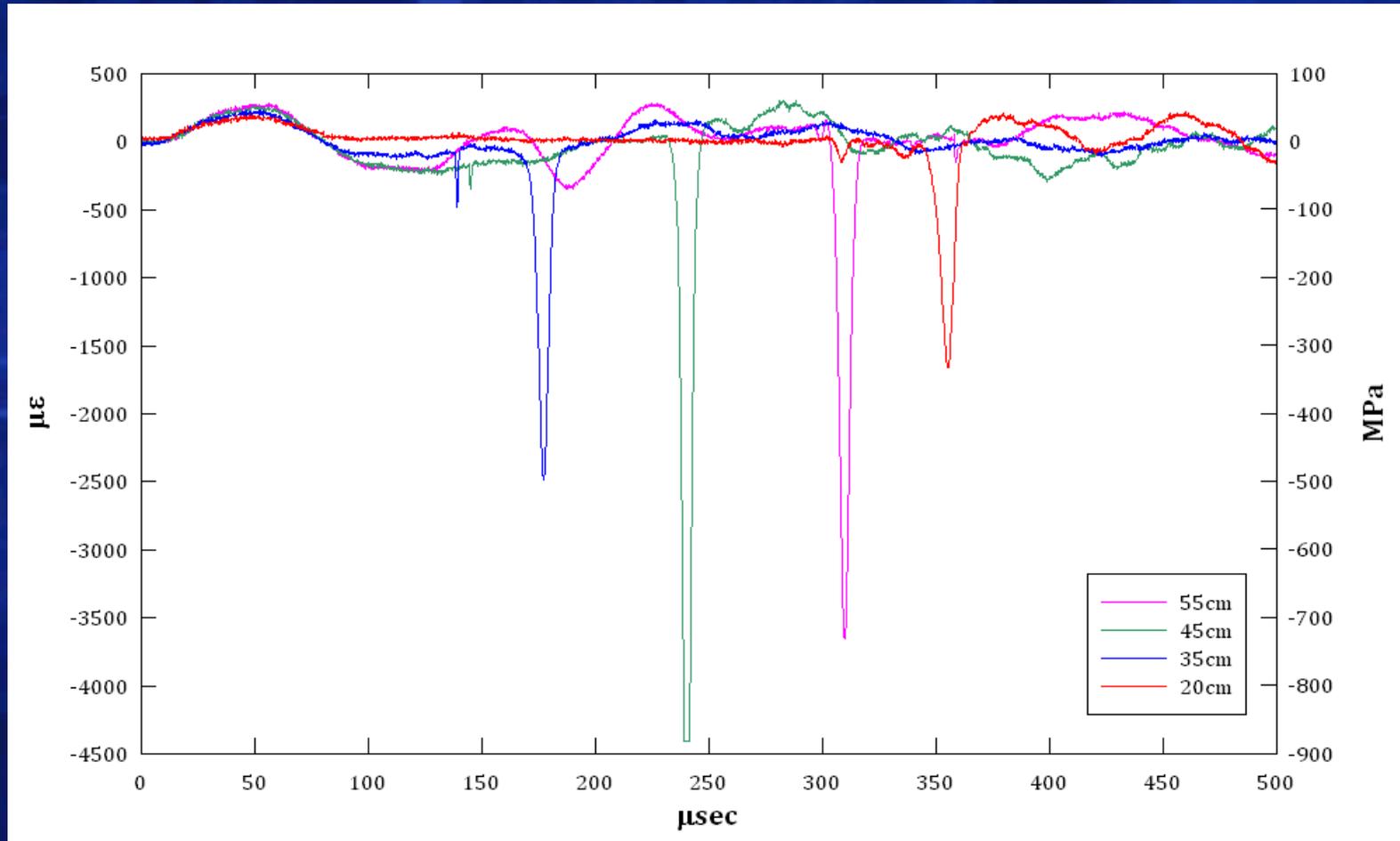


Sodium Carbonate

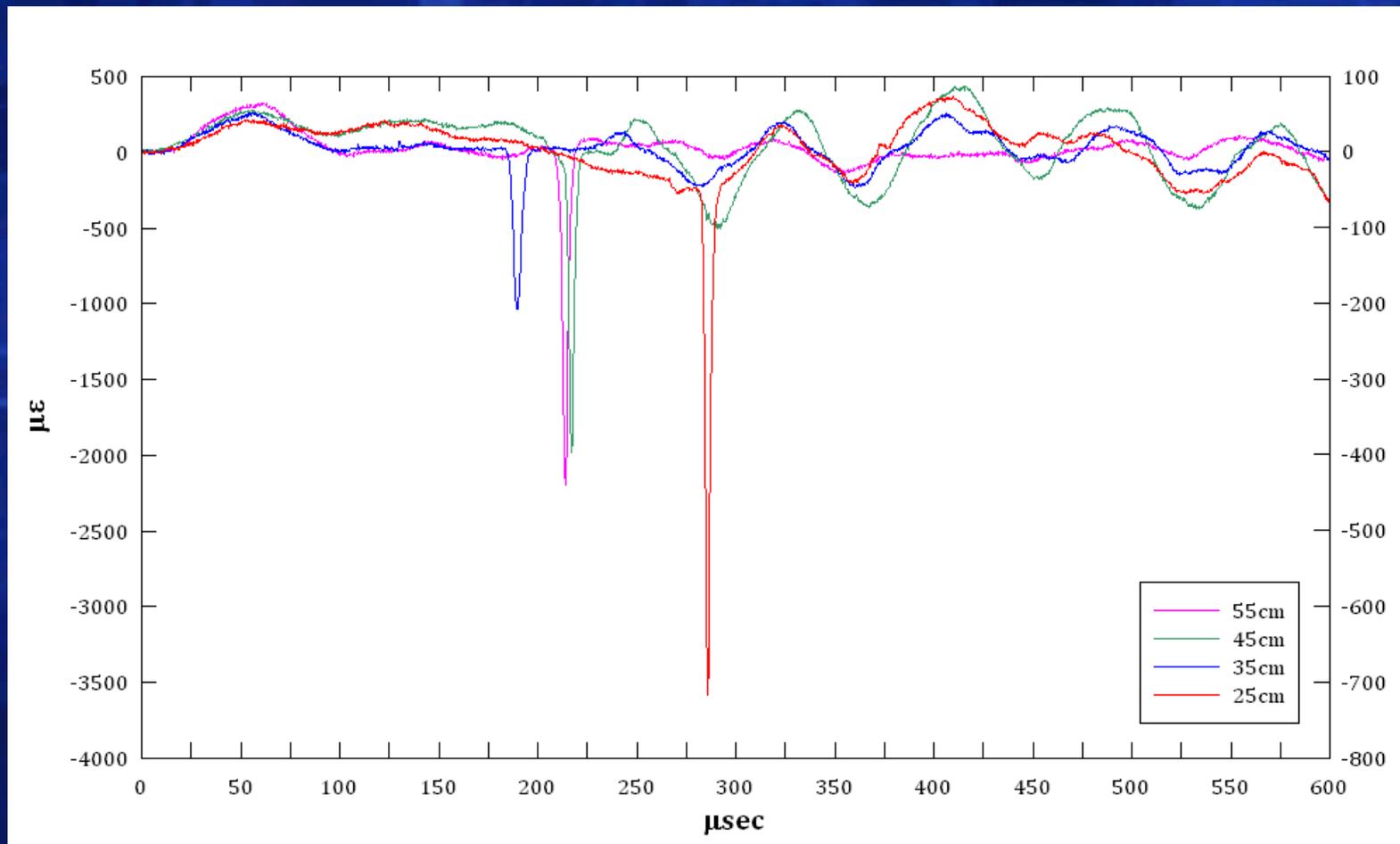


Positive Reaction Comparisons of Explosives

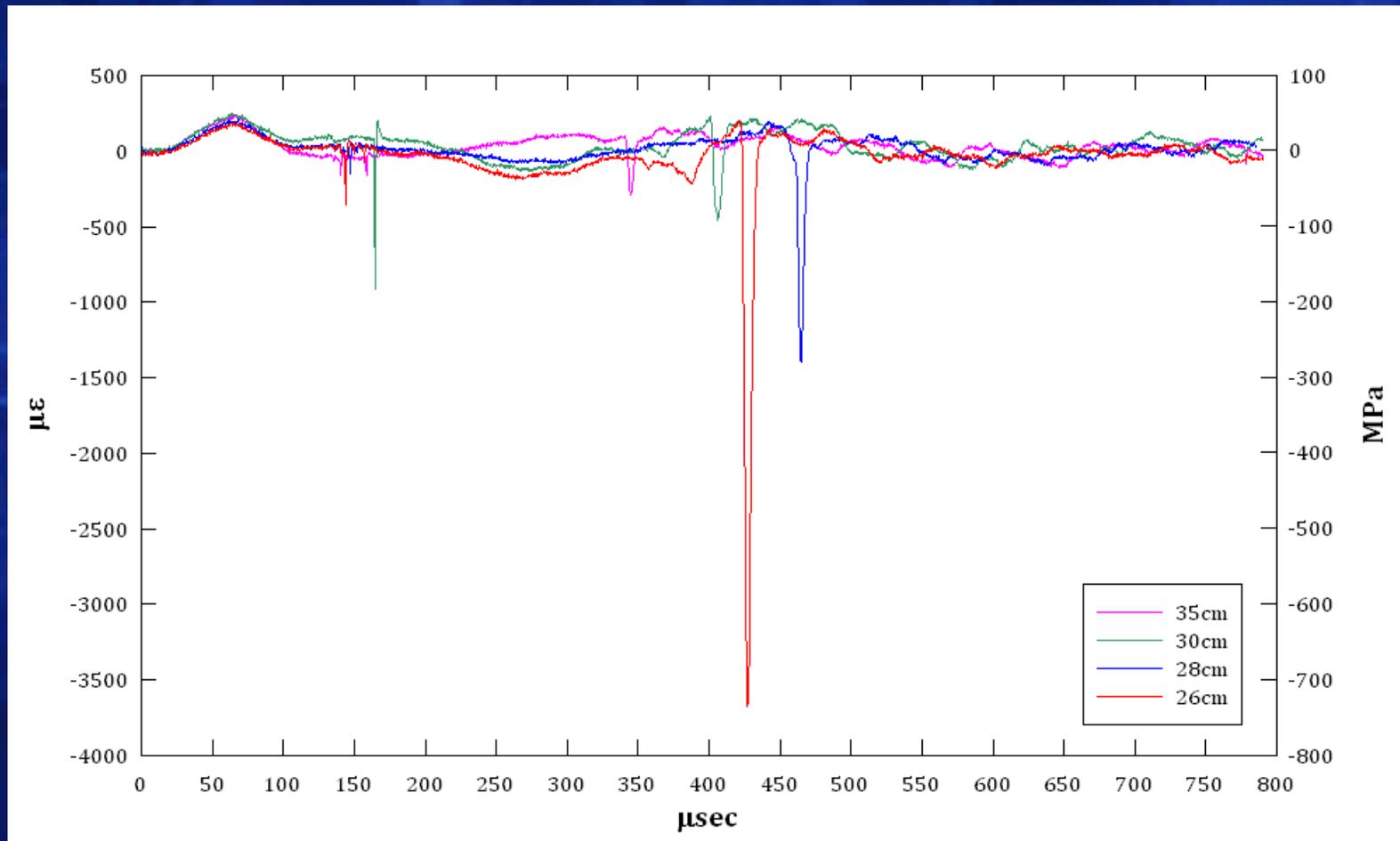
RDX: Go Comparison



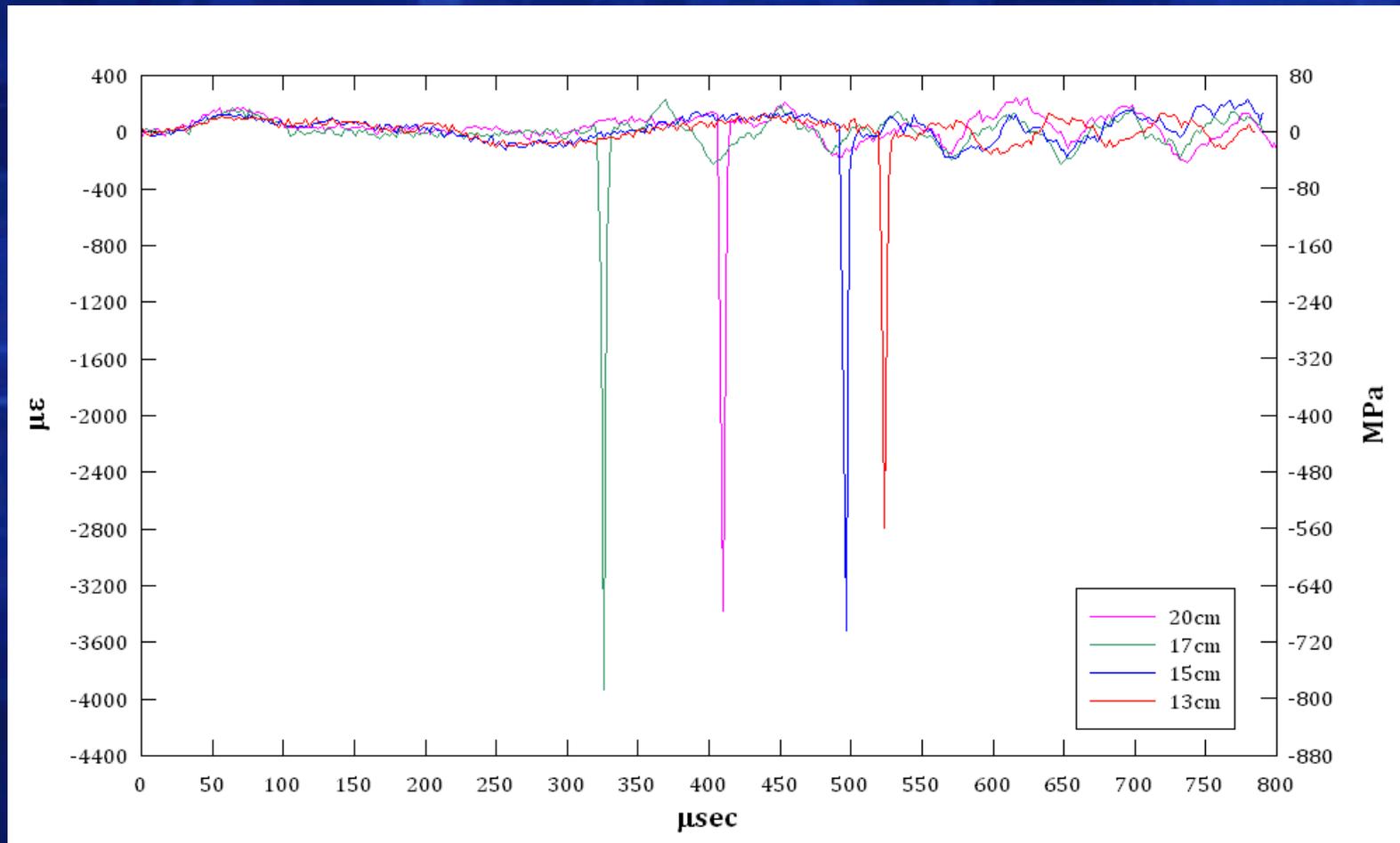
PETN: Go Comparison



HNS II: Go Comparison



CL-20: Go Comparison



Strain Gage Output Analysis

- Other materials tested including LX-14 and PBXN-5 show some form of rapid pressure drop. These spikes were only seen when the sample reacted.
- These drops in pressure may be indicative of mechanical failure of the sample, including melting, jetting, and plastic/viscous flow. This is supported by previous research by Heavens and Field in 1974.
- Various timing/amplitude of these spikes is likely affected by sample inhomogeneity.

Future Research: Sensitivity Tests

- HDPE bushings can be installed on similar drop weight instruments to decrease deviation of the results.
- Detailed studies of environmental factors will provide information necessary to reduce variation of sensitivity results.
- An investigation of the effect on sensitivity results by both the ambient temperature trends as well as the varying temperature of the tooling itself could also reduce deviation in H_{50} .
- Other factors such as humidity and ambient pressure should also be studied in detail.

Future Research: Strain Gage Testing

- Uniform sample geometry would allow for more detailed analysis including stress-strain levels in the sample, itself. Sample materials pressed into pellets or lightly confined into a fixed cross-sectional area would facilitate this.
- A longer striker bar would help isolate the incident wave and reduce interference due to reflected waves from the interfaces. This may also increase the clarity of waves created by sample response.
- A method of timing a Go reaction, such as a light detector used by Heavens and Field, would aid in the detailed interpretation of strain gage outputs.

Conclusions

- Current instruments designs are limited in that they only produce qualitative statistical data that must be compared to previous data to derive meaningful results.
- The newly modified tooling results in a 46% reduction in deviation among H_{50} values, displaying higher precision over previous designs.
- The addition of the strain gage can provide impact sensitivity data that presents a foundation to develop new standards to supplement the current drop height data collections.
- With further investigation, strain gage outputs may also be able to serve as an indicator of sample response, reducing dependence on operator judgment.

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