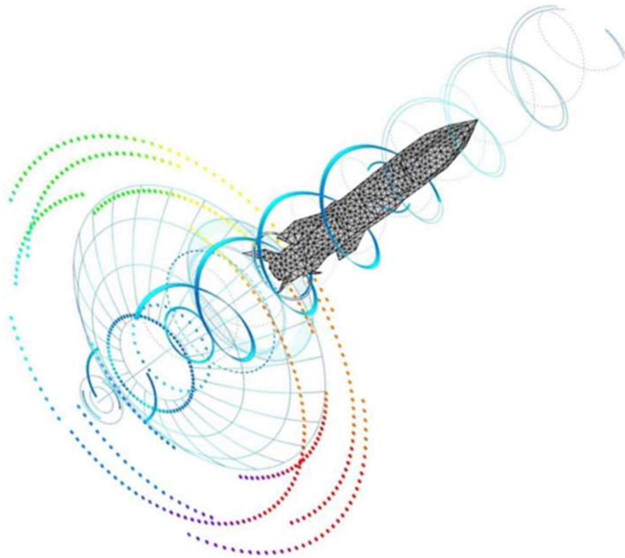
The importance of good analysis; ensuring the UK Energetics industry takes full advantage of technological advancements



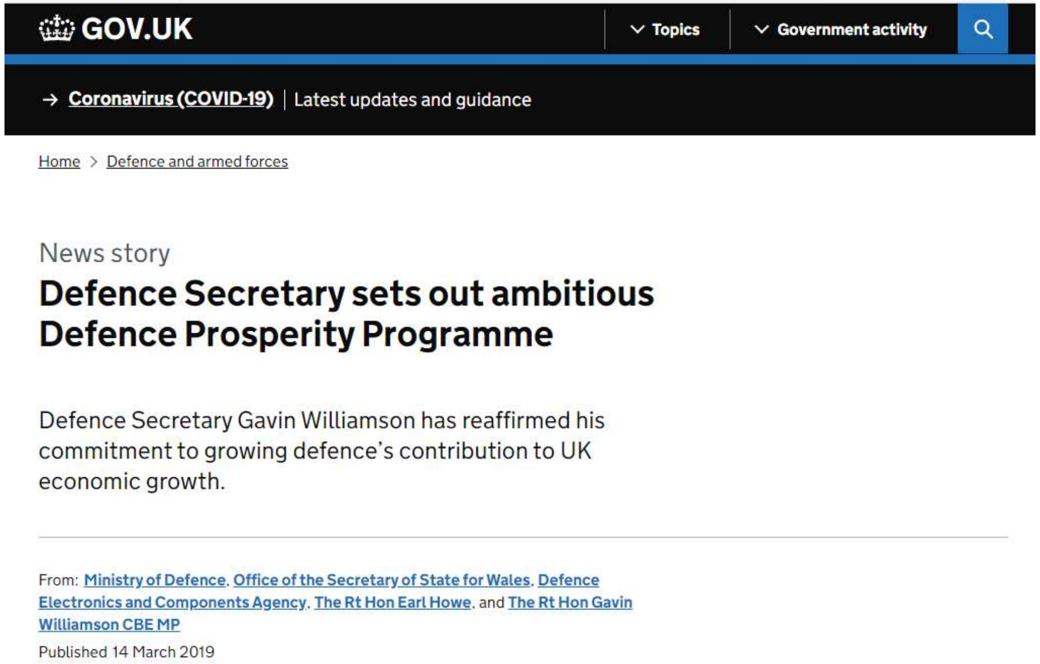
Ruth Pettifer

QINETIQ/22/01066

April 2022

Introduction

- We are in an era of unprecedented change with regards to technological advances
- We will need to take advantage of these to meet the aims of important issues for the UK such as:
  - Sovereign capability
  - Security of supply
  - SQEP sustainment
- However, we need to learn lessons from our past and ensure that the field of energetics analysis keeps pace



https://www.gov.uk/government/news/defence-secretary-sets-out-ambitious-defence-prosperity-programme--2

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Some lessons - #1 scientists need to learn how to communicate

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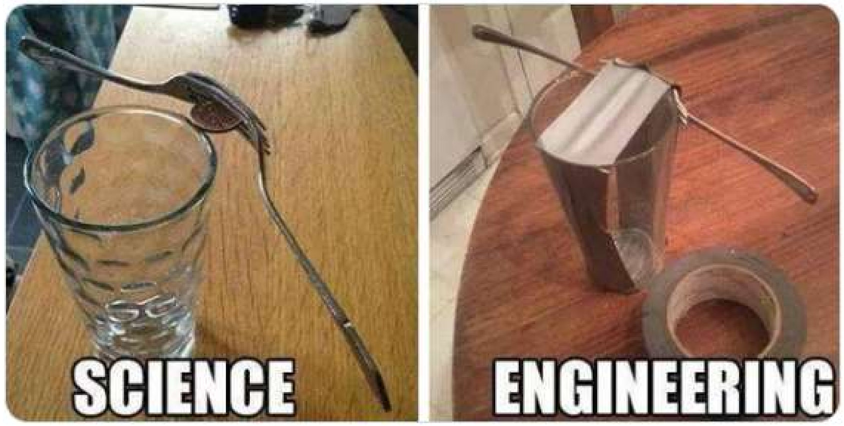
Weird Science
@weird_sci

The difference between science and engineering.



9:35 pm · 30 Dec 2015 · SocialOomph

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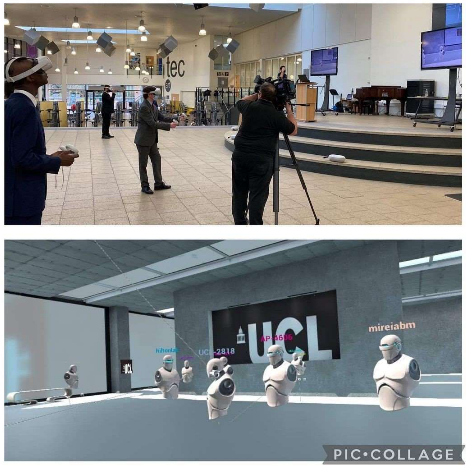
Some lessons - #2 we need to take ideas from other industries and rapidly employ them in the UK energetics industry

Instagram

hiltonlab
UCL School of Pharmacy

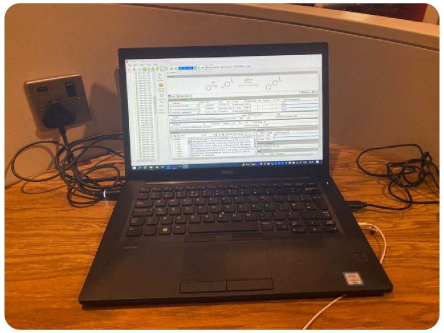


27 likes
hiltonlab #virtualreality #outreach - an amazing way to reach schools and bring them into #digitaltwin laboratories of #ucl
27 January

Tweet

stephen hilton
@hiltonlab

How can you not have an #ELN in your lab???
Out the lab, yet still able to access old group data on experiments from four years ago. Electronic lab notebooks 😀 #RealTimeChem



UCL School of Pharmacy and 3 others
18:52 · 08 Mar 22 · Twitter for iPhone

13 Likes

Tweet

stephen hilton
@hiltonlab

Looking back a few years ago to when we had 8 @Biotage Horizons in the lab. Most from auctions - old tech, but amazing to be able to rapidly and automatically purify compounds. This started us on the path to design & #3dprint our own kit. You can see examples in the pic

22:11 · 17 Feb 22 · Twitter for iPhone

1 Retweet **13** Likes

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Some lessons - #3 we have to find a way of working smartly and collaboratively

SMART ASSESSMENT & TESTING

Dr Peter Barnes - MOD DOSG ST6*
Dr Mike Sharp – MOD DOSG ST6a

NDIA IM & EM Technology Symposium

Miami - 16 October 2007

de&s

BACKGROUND

- Smart assessment & testing of munition safety requirements has been a major objective of DOSG for several years
- Smart assessment & testing is defined as the assessment of intelligent data by competent staff
- Intelligent data are defined as results which help to achieve a desired aim or objective
- Don't do the same old tests just because we have always done them
- Do not 'gold plate' assessments; set pragmatic levels of confidence for particular objectives
- This presentation is a first review of issues that have arisen in trying to achieve smart assessment and testing

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Some lessons - #4 some tests performed at manufacture are inadequate for life prediction purposes

Test	Comment
Abel heat test	Use should be limited to nitrate ester manufacture only
Density	Unlikely to change with ageing
Dutch Test (110°C)	Stability check test
Bergmann-Junk Test (132°C)	Stability check test
Vacuum stability	Important to determine likelihood of material to gas but small scale STANAG test may not be representative of gassing in a large scale solid rocket motor. Need to be able to determine the concentration of gases
Quantification of compounds by gravimetric methods	Some compounds are unlikely to change with ageing. Others may change but should look to quantify by more modern methods or understand what is actually changing e.g. molecular mass of nitrocellulose

Some lessons - #5 we should be performing targeted testing – failure modes and mechanisms

- Monday 15:20 – 16:00. Improved methodology for life assurance of guided missiles from a prime perspective, Gaynor Olliver, MBDA
- Tuesday 11:35 – 12:05. Agile qualification of future minimum-smoke rocket propellants, Phil Gill, Roxel

There are many reasons why new tests need to be developed

- Understanding behaviour of materials under actual conditions
  - E.g. ignition, small scale tests not representative, understand actual hazard and risk, operation under extreme environmental conditions, to quantify failure modes and mechanisms
- Provide more accurate predictions of life of asset which can then be validated through in-service data
- Take advantage of technological advancements such as increase in instrumentation ability
  - Ensure that the right tests are instrumented correctly to give the right information at both the system and sub system level
- To enable the increased use of predictive and descriptive modelling
  - To reduce test and evaluation costs and risk both safety and project related
- Investment in better raw material characterisation may reduce issues in the expensive products they go into
  - We do not want to find out there is something wrong with our RDX when we trial our missile

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Modelling – another challenge for the UK energetics industry

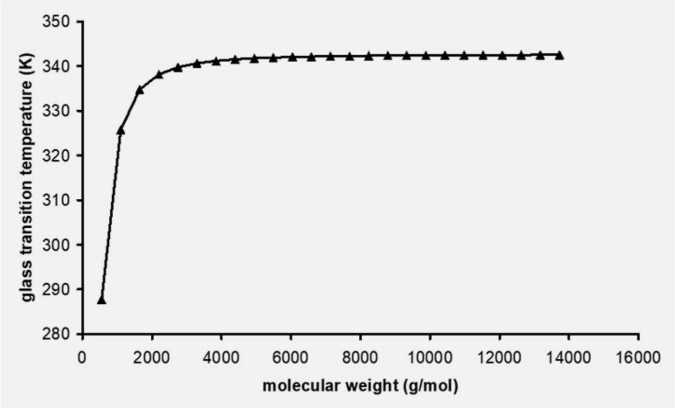
- To predict the life time of an energetic material, there are four factors which need to be known[1]:

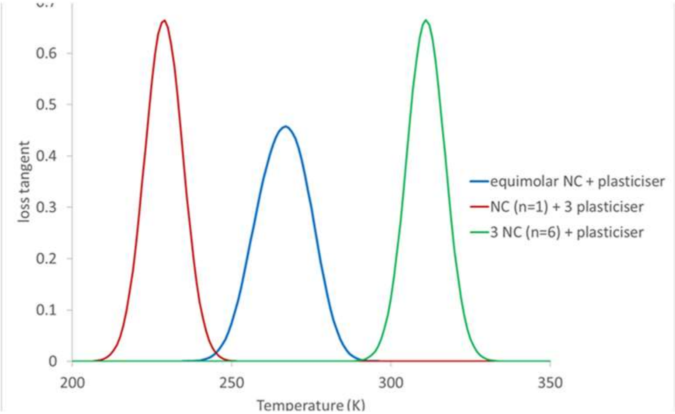
1. The key ageing mechanisms.

2. The rate expression and so the temperature dependence for these ageing mechanisms from kinetic measurements.

3. The service environment of the asset across storage, deployment and operational use.

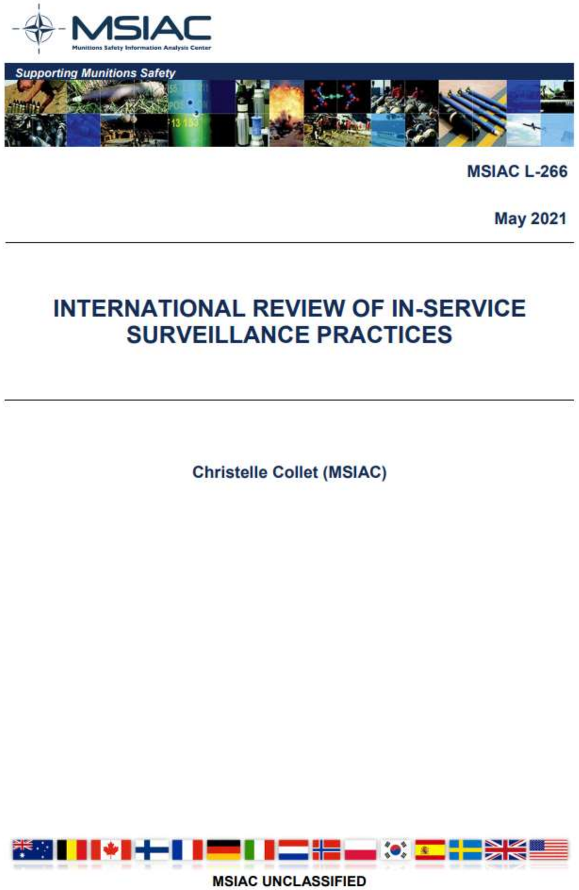
4. The design limits of the energetic component.





[1] A. Rix, "WSTC0503 Health Usage Monitoring Systems - Strategy for Lifing Algorithms for Complex Munitions," QinetiQ/19/03516/4.0, UK, 2021.

How do we get new or improved tests into service and is this agile enough for the modern era?


MSIAC
Munitions Safety Information Analysis Center
Supporting Munitions Safety

MSIAC L-266

May 2021

INTERNATIONAL REVIEW OF IN-SERVICE SURVEILLANCE PRACTICES

Christelle Collet (MSIAC)

MSIAC UNCLASSIFIED

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Round robins and collaborative working

Munitions Safety Information Analysis Center
Centre d'information et d'analyse sur la sécurité des munitions

MSIAC
Supporting Munitions Safety

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L-127

April 2006

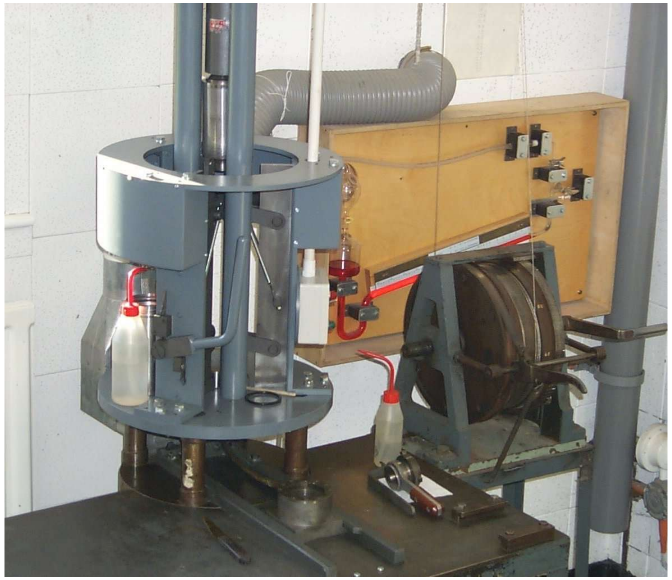
RS-RDX ROUND ROBIN (R4)
PRELIMINARY RESULTS ANALYSIS

By

Duncan Watt (MSIAC)

Ruth Doherty and Lori Nock
(NSWC-IHD)

The importance of good analysis – capture all of the information, in the right way



MAKING SENSE OF SENSITIVITY DATA

Ruth M. Doherty
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Abstract:
Sensitivity to a range of stimuli, as measured in small-scale tests, is routinely reported for new energetic materials. Often insufficient information is given for the results to be interpreted in the context of the behaviour of other energetics. This paper focuses on small-scale impact sensitivity testing and the information about the testing that is needed for a meaningful evaluation of those results to be made. The discussion addresses impact sensitivity tests, but the lessons are applicable to other types of small-scale sensitivity testing as well.

Keywords: *small-scale, sensitivity, explosives, impact, Bruceton, D-Optimal, Probit*

1. INTRODUCTION

Among the most important assessments performed on new energetic materials is the evaluation of the response of the material to a variety of mechanical and thermal stimuli, including impact and friction. The ability to respond to such stimuli is referred to as sensitivity or sensitiveness. These terms are often used interchangeably, but a distinction between response to unplanned stimuli (*sensitiveness*) and response to a planned level of stimulus (*sensitivity*) is sometimes made. The former is important in the context of safety and hazards, whereas the latter is an important consideration for reliability of function. In this paper the term *sensitivity* will be applied to both categories. While papers in the literature reporting new energetic compounds routinely include results of small-scale sensitivity tests, those reports often omit information on the testing procedure and data analysis methodology, thus making it difficult for the reader to judge the reliability of the results.

One of the current topics of interest in the energetic materials community is the development of insensitive energetic compounds (i.e., compounds that require a relatively strong stimulus to initiate a reaction), so the comparison of test results for a new compound will often emphasize the results of small-scale impact and friction sensitivity testing, comparing the results to a compound that is a candidate for replacement. An example of this is found in reference [1]. The authors state "Compound 5 has the properties of a high explosive with a performance close to that of the common high explosive RDX (l,3,5-trinitro-l,3,5-triazacyclohexane) but with lower sensitivity to impact and friction." The results are presented as "Dropweight test (cm)" and "Friction Test (N)." The equipment used for the friction testing is identified in the experimental section, but no other information is provided about the impact testing. Values are given for both RDX and Compound 5, but there is no indication of the source of the data for RDX.

It is known that many factors influence the measured values of impact sensitivity of energetics, including some obvious ones, such as the mass of the dropweight employed and the criteria for determining whether an individual trial is positive, indicating presence of reaction (GO),

1

R. Doherty, "Making sense of sensitivity data," in 21st NTREM conference, Pardubice, Czech Republic, 2018.

The importance of good analysis – report your variables and know that they will affect the results

WILEY-VCH

On the use of differential scanning calorimetry for thermal hazard assessment of new chemistry: Avoiding explosive mistakes

Sebastian P. Green,[a,b] Katherine M. Wheelhouse,[c] Andrew D. Payne,[d] Jason P. Hallett,*[b] Philip W. Miller*[a] and James A. Bull*[a]

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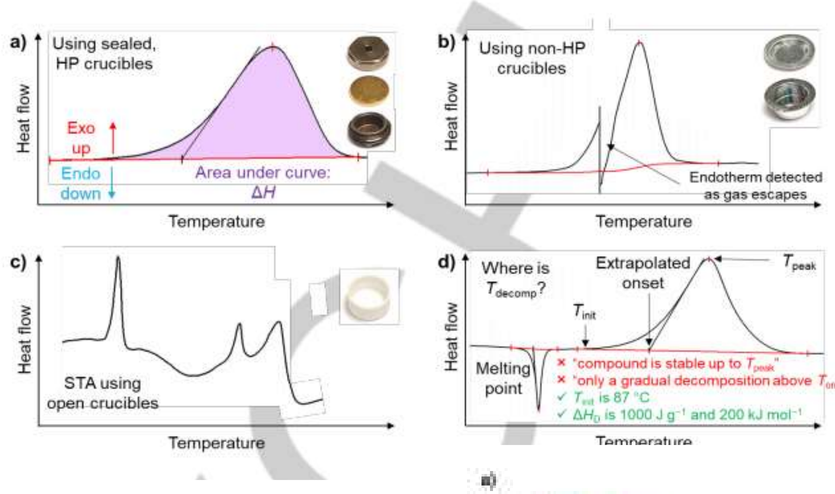
Supporting information for this article is given via a link at the end of the document.

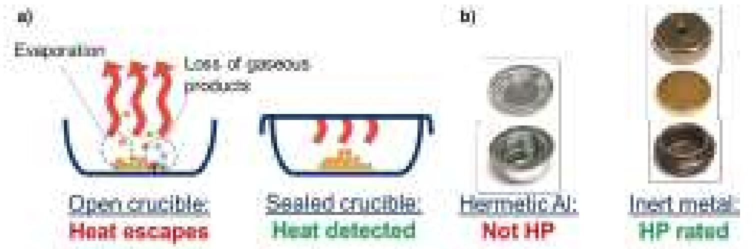
Abstract: Differential scanning calorimetry (DSC) is increasingly used as evidence to support a favourable safety profile of novel chemistry, or to highlight the need for caution. DSC enables preliminary assessment of the thermal hazards of a potentially energetic compound. However, unlike other standard characterisation methods, which have well defined formats for reporting data, the current reporting of DSC results for thermal hazard assessment has shown concerning trends. Around half of all results in 2019 did not include experimental details required to replicate the procedure. Furthermore, analysis for thermal hazard assessment is often only conducted in unsealed crucibles, which could lead to misleading results and dangerously incorrect conclusions. We highlight the specific issues with DSC analysis of hazardous compounds currently in the organic chemistry literature and provide simple 'best practice' guidelines which will give chemists confidence in reported DSC results and the conclusions drawn from them.

The development of novel chemical reagents is an essential step in the advancement of synthetic methodology, access to new chemical space, and ultimately the discovery of new medicines and materials. Chemists have become acutely aware of the potential safety hazards of newly discovered reagents, and the impact it can have on their wider use and applications. In particular, a misunderstanding or misjudgement in the safety profile of a new compound or process can have serious unforeseen consequences. The use of differential scanning calorimetry (DSC) is becoming a common means for organic chemists to either support the favourable safety profile of their new reagent or process, or to evidence the need for caution. Inclusion and discussion of potential hazards is laudable and is one way of helping improve awareness and attitudes towards safety within the chemistry community, which can be deficient – a claim particularly levelled towards academia.[1,2]

DSC is an invaluable tool for chemists, enabling a 'first look' at a potentially energetic material. Using small quantities (approximately 5 mg), a rapid assessment of the thermal stability and the energetic yield of a compound or reaction mixture can be obtained.[3,4] DSC is considered a screening technique, with limited sensitivity, but the results are accurate and robust enough to determine process temperatures to avoid thermal decomposition (often using T_{D24}: the temperature at which the time to maximum decomposition rate under adiabatic conditions is 24 h),[5] estimate the severity of an exotherm (often using the adiabatic temperature rise, ΔT_{ad}),[5] and even make predictions whether impact sensitivity and explosivity are a concern by using the Yoshida correlation.[6] Such data, particularly on novel or under-explored compounds and reagents, is informative to chemists wishing to employ new synthetic methods. There are other thermal hazard tests available, such as accelerating rate calorimetry (ARC), reaction calorimetry, impact sensitivity or explosivity tests, but none are as ubiquitous or simple to use as the humble DSC. Recently, there have been studies seeking to characterise and compare the thermal and process hazards of compound classes, such as peptide-coupling reagents,[7] oxidisers,[8] *N*-heterocyclic iodanes,[9] and diazo compounds;[10] or processes such as Pd-catalysed cross-coupling,[11,12] TIPS-EBX[13] and NaH with DMF/DMSO/DMAc.[14]

In contrast to these in-depth studies, DSC data reported in the mainstream organic chemistry literature generally forms a small part of a much broader demonstration of the synthetic utility of a reagent or process, perhaps used to make cautious statements about the stability or hazardous nature of a reagent. Compared to the rigorous reporting standards in place for characterisation data, it seems that all too often the vital experimental details required to reproduce a DSC experiment are lacking. This may be a consequence of the lack of specific rules in place for publishing this type of data and a lack of specialist knowledge at the author, reviewer and publisher levels. Additionally, DSC experiments are frequently performed, or results interpreted, without adherence to best practices (Figure 1), possibly indicating a lack of familiarity with the technique as applied to thermal hazard assessment. As a consequence, common errors are made which could lead to the dangerously misleading conclusion that a new reagent is safer or does not exhibit a significant exotherm when this may not be correct. We previously reported on how the use of unsealed crucibles led to the incorrect conclusion that 2-azido-4,6-dimethoxy-1,3,5-triazine (ADT) had no exothermic decomposition and was "intrinsically safe"; when in fact it is highly energetic.[15]





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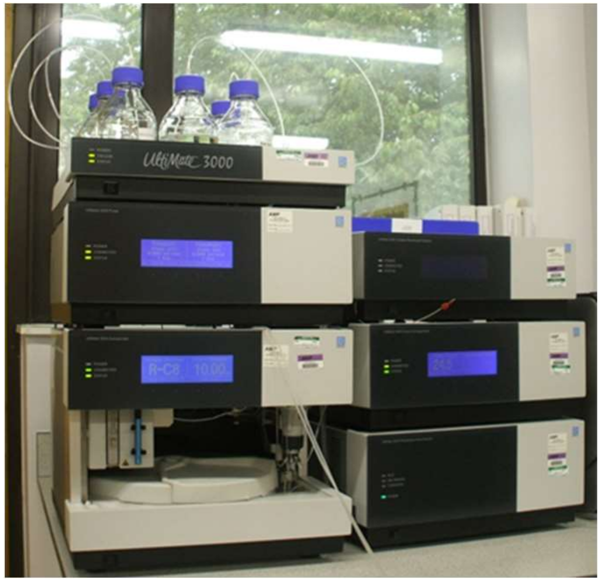
The importance of good analysis – know when the result is ‘wrong’ / ‘different’ and either resolve or report limitations

- AOP-48 stabiliser analysis usually performed using high performance liquid chromatography (HPLC)
  - Eluent, column, column temperature, flow rate and wavelength can all be varied which will affect results
- Are standards being used for quantification?
- Limits of detection should be determined
- Standard deviation of results should be reported
- Accuracy of reported results



Component expected %	Result test house A	Result test house B
90% ± 0.5%	50% ± 2%	105 % ± 0.001%

The importance of good analysis – know when the standard is appropriate for your material and what you can do to achieve meaningful data

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SUBJECT: EXPLOSIVES, NITROCELLULOSE BASED PROPELLANTS, STABILITY TEST PROCEDURE AND REQUIREMENTS USING HEAT FLOW CALORIMETRY

Promulgated on 09 March 2007

J. MAJ
Major General, POL(A)
Director, NSA

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MSIAC Munitions Safety Information Analysis Center
Supporting Member Nations in the Enhancement of their Munitions Life Cycle Safety

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June 2018

LIFE ASSESSMENT OF NITROCELLULOSE-BASED ENERGETIC MATERIALS: LOVA, FLARE AND CCC

by
Dr Matthew Andrews (EM TSO), Christelle Collet (PT TSO), Rebecca Millar (Stokes Fellow 2018), Emmanuel Schultz (Eurenco, formerly PT TSO)

This paper was presented at 8th International Nitrocellulose Symposium, Bergerac, France, 5th-7th June 2018

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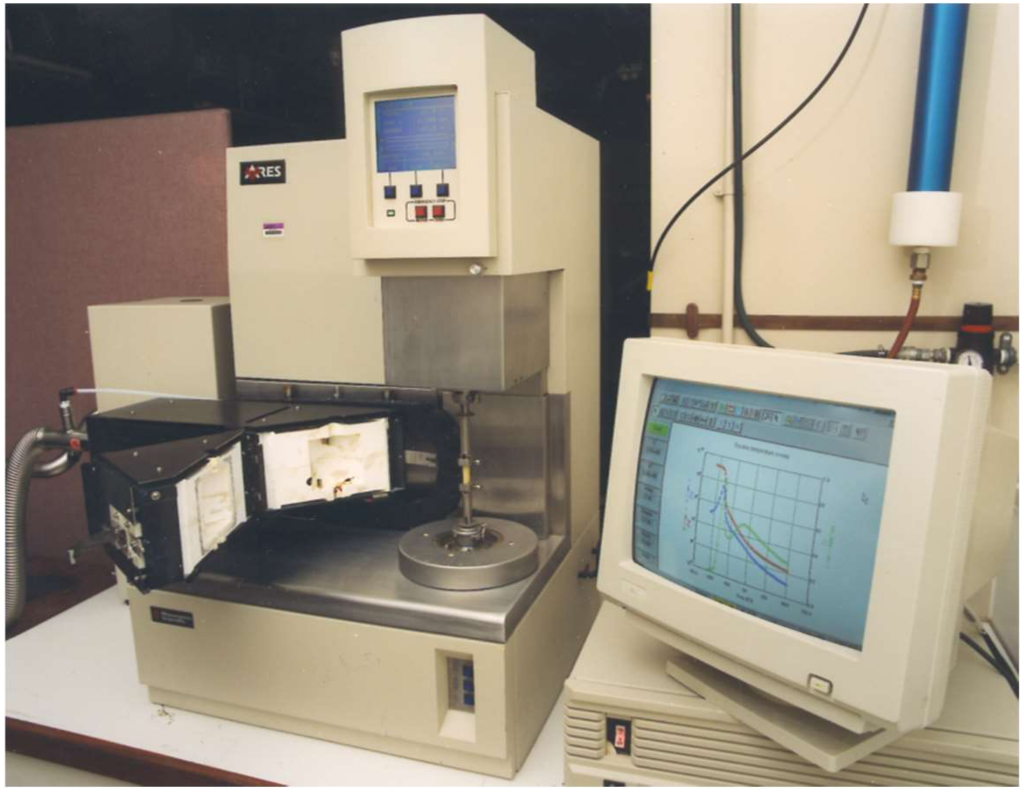
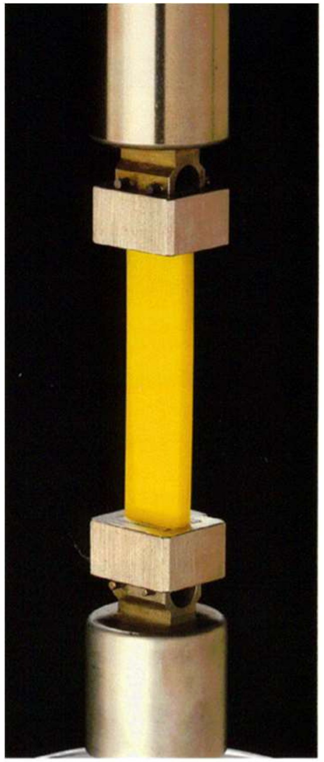
The importance of good analysis – know what your customer needs and provide it





Final remarks

- Should we be standardising analytical methods across all test houses?
- How do we ensure only the best test methods are applied and new techniques get accepted and routinely into a more agile manner?
- How do we ensure those with less experience build expertise quickly and don't make the same mistakes as others?
  - caveat: mistakes are a very good way to learn
- How do we attract and retain SQEP?
- What can we learn from our overseas partners?
- How can we work together more collaboratively for the benefit of the UK energetics industry?

https://www.etcmd.com/

Early careers day 2016

Acknowledgements

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