Nitrocellulose: DOSG Propellant Qualification Requirements

MP Sloan, DOSG ST1, 11/05/2010

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CONTENTS

- Cellulose and Nitrocellulose
  - UK Nitrocellulose Requirement
  - Recent Nitrate Ester Source Changes
  - Naval Rocket Motor Investigations
  - Proposed Future Policy
- Nitrate Ester Propellant Stability Test Policy
  - Current Requirements
  - Proposal

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UK MoD Requirement

- Continuity of Supply of Nitrocellulose Propellants
- Consistency of Propellant Quality
  - Performance
  - Safety Characteristics
  - Service Life

UK MoD Requirement: Background

- Propellant Supply Chain Unstable
- Gun Propellants have proven tolerant to source changes
- Rocket Propellant position more complex:
  - Lower Performance, cartridge load propellants re-qualifications have also been shown tolerance to source changes
  - High Performance rocket motors more ‘eventful’

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  - High Performance rocket motors more ‘eventful’
- Problems in requalification
- Problems in Service
  - Multiple simultaneous obsolescence issues

Cellulose/Nitrocellulose Supply 1990-2005

- Cotton Linter background since 1990:
  - Holden Vale Linters, Dumfries NC
  - Holden Vale Linters, Bishopton NC
  - Temmings Linters, Bishopton NC
  - Temmings Linters, Bishopton NC, Muiden Plant
  - Temmings Linters, Bergerac NC
  - Temmings Linters, Wimmis

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  - Milouban Linters, Wimmis
  - *Bergerac future material/SNPE-SAFRAN uncertainty*
  - *Replacement Source Linters, Wimmis*

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UK Naval Missile NC Associated Issues

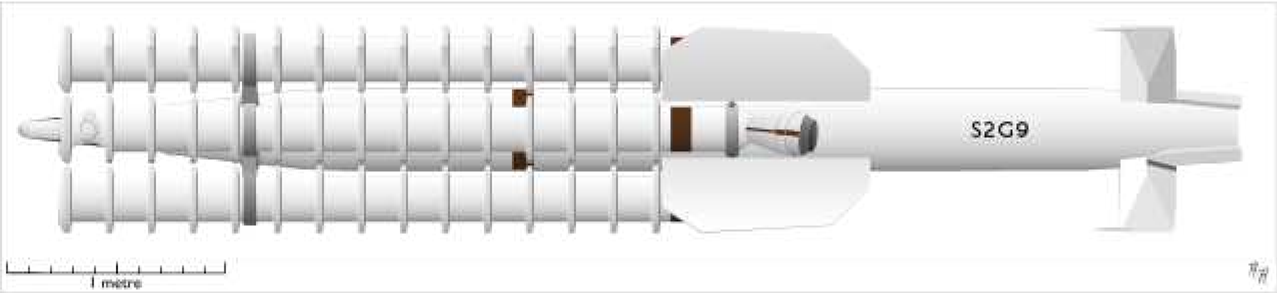
- Sea Slug ‘Retriever’ Boost manufacture
- Sea Dart ‘Chow’ Boost manufacture
- Sea Wolf Charge
- Sea Dart ‘Chow’ Service Life and other propellant bondline issues

UK Rocket Motor NC Associated Issues

- Sea Slug ‘Retriever’ Boost manufacture
- Sea Dart ‘Chow’ Boost manufacture
- Sea Wolf Charge Propellant Setback
- Sea Dart ‘Chow’ Service Life and other propellant bondline issues

- In all these cases problems identified at Rocket Motor Manufacture, Proof or in-service NOT during propellant level proof which was successfully passed

Sea Slug: 1961-1995



Sea Slug Manufacturing Issues

- Sea Slug – mid 1970's
  - Boost grain propellant granules would not coalesce fully towards end of cast grain
    - Sample (short) charges cast satisfactorily with acceptable, unexceptional proof results
    - Over 120 full size motors cast to recover production capability

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    - Problem vanished when a linter preparation process change reversed

Sea Dart: 1973 - 2013

Sea Dart: CMCDB Boost

Sea Dart Manufacturing Issues

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Propellant Porosity

1 mm

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Sea Wolf Propellant Grain

- Closure of RO Bishopton required move from Wood to Cotton origin nitrocellulose
- Initial Propellant manufacture and motor development successful

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- UNTIL…

Sea Wolf Rocket Motor Failure

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Sea Wolf Propellant Grain

- ‘New’ charge design required different charge specifications to survive setback/pressurisation
- Quantitative Linkage between propellant mechanical properties and charge safety factors never established

Sea Dart Service Life and other propellant bondline issues

- UK Cast Double Base propellant have historically used Duplex Propellant Adhesives:
  - Exterior layer bonds to case insulation
  - Interior layer bonds to propellant
    - Propellant bonding achieved by NC diffusion during propellant cure

Firing of 12 Year Old Rocket Motor

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Sea Dart Boost Failure

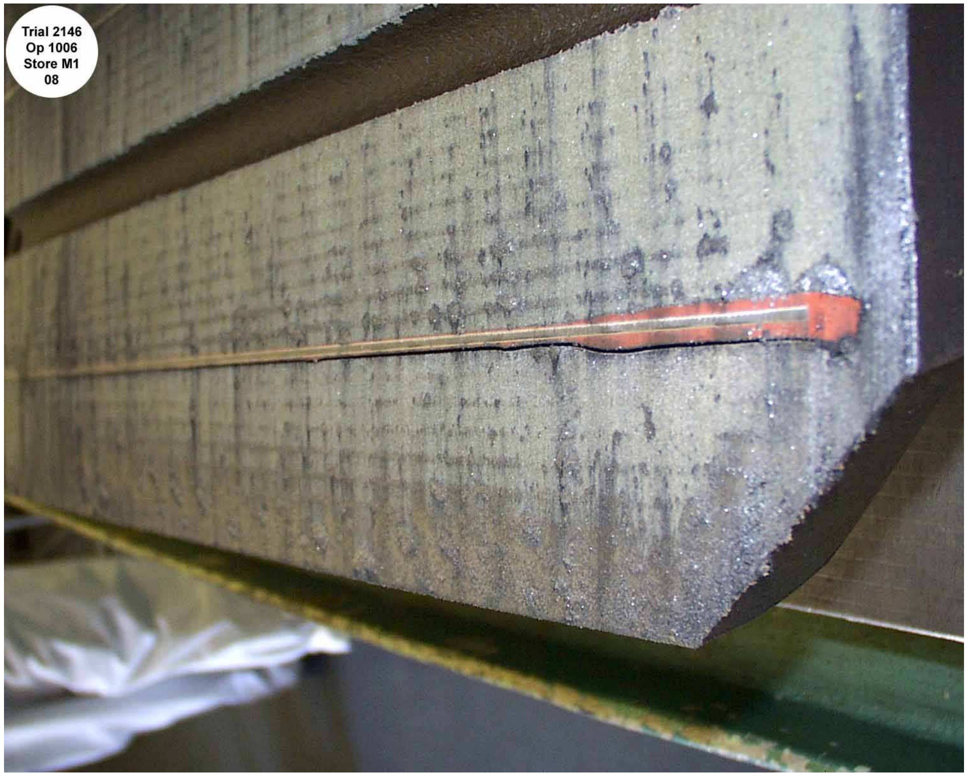
- Cause identified as propellant to insulation bondline failure
- Rocket Motor was beyond service life
- But older motors had been fired without fault

Sea Dart Boost Failure: Role of NC?

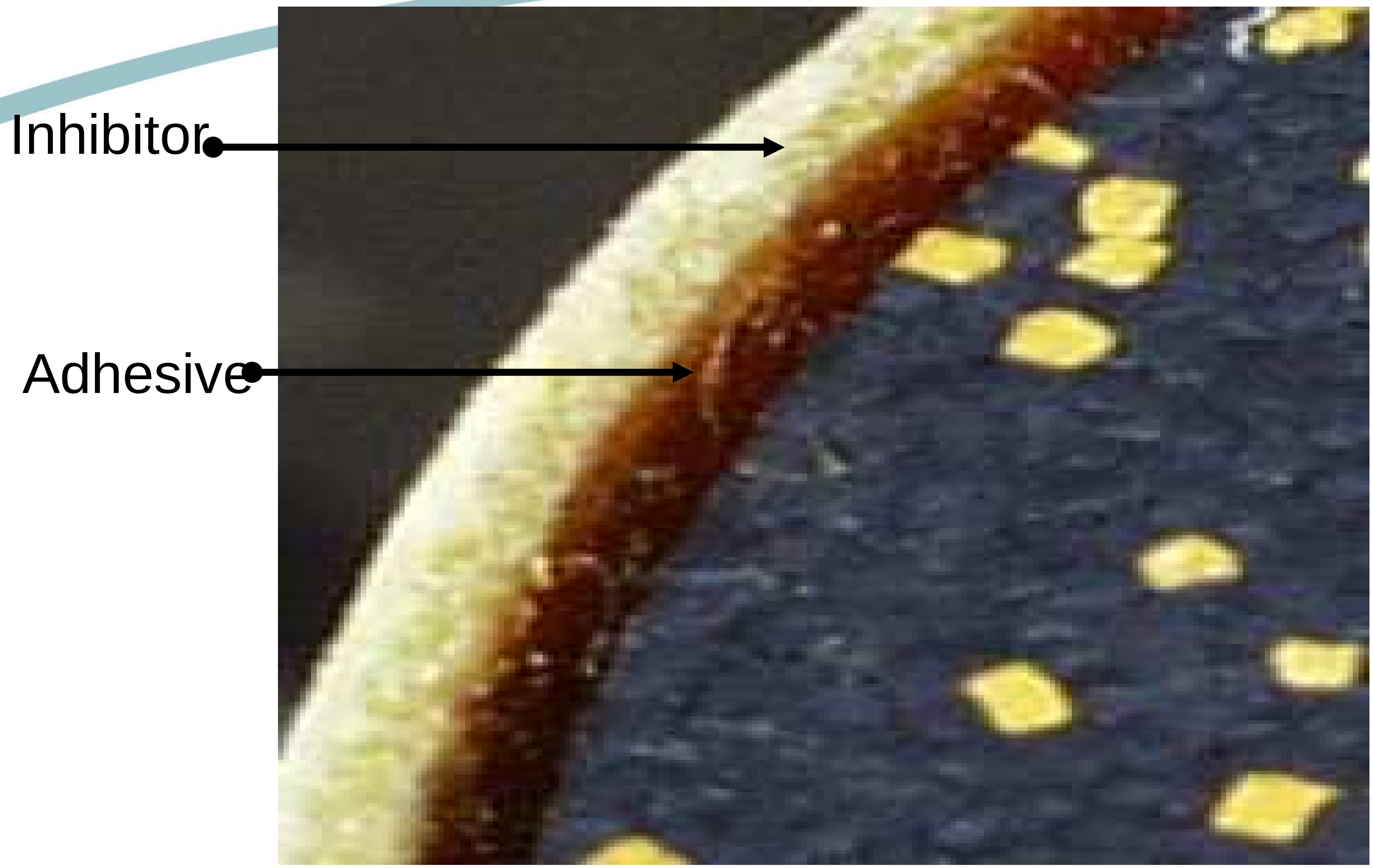
- Cause identified as propellant to insulation bondline failure
- Rocket Motor was beyond service life
- But older motors had been fired without fault

- Nitrocellulose source had changed
  - Bondline variability dates from this change

Propellant Separation – 12 Year Service Motor



Propellant to Inhibitor Separation



Bishopton Closure: CDB Rocket Propellants

- Loss of UK Military Grade Cellulose Nitration Capability
  - Switch to non UK sources initially unsuccessful
    - NC/NG Paste (‘Galette’) would not process
    - Major changes to process required
    - Initial Ballistics unplateaunised (‘Tram Lines)
    - Multiple iterations and delays
  - Eventual success

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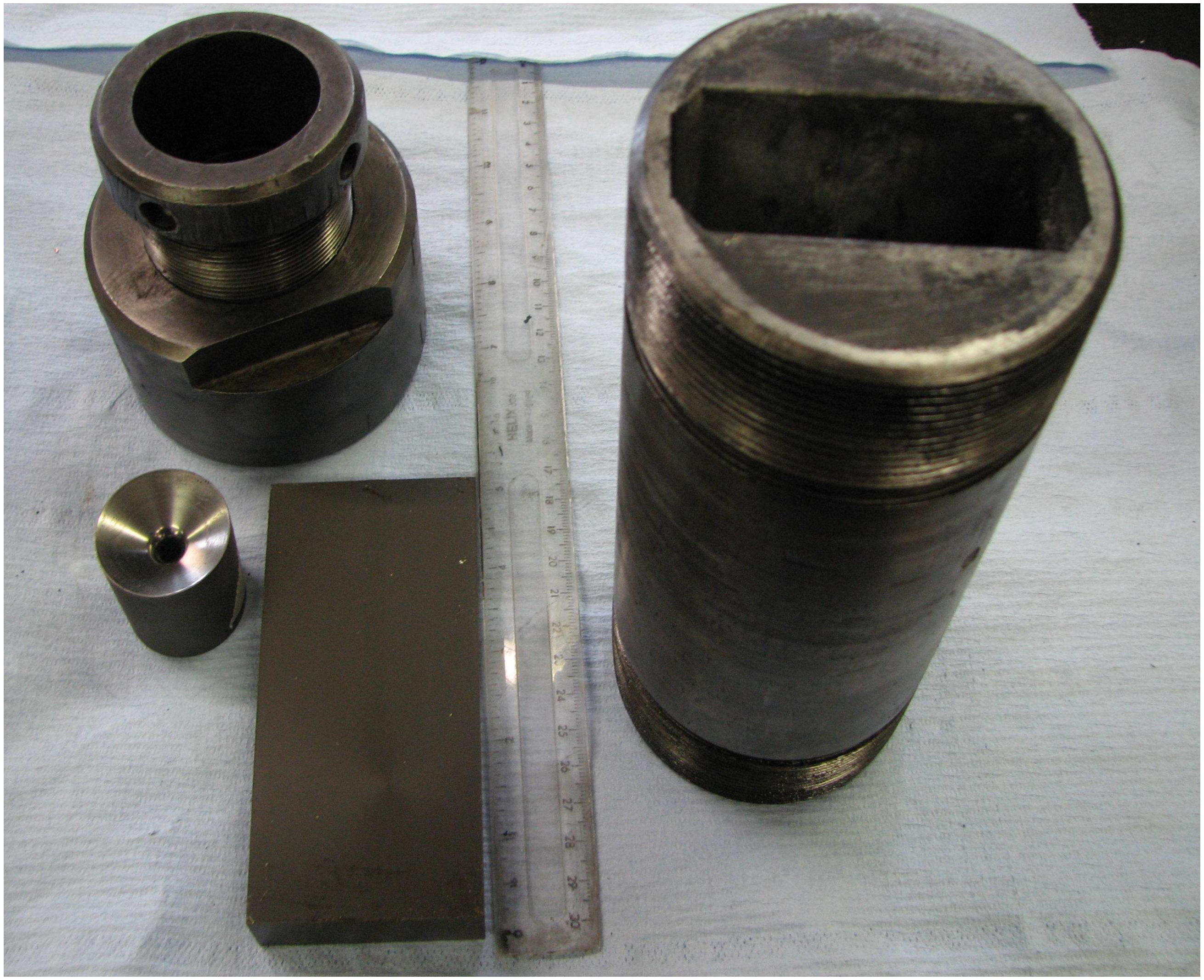
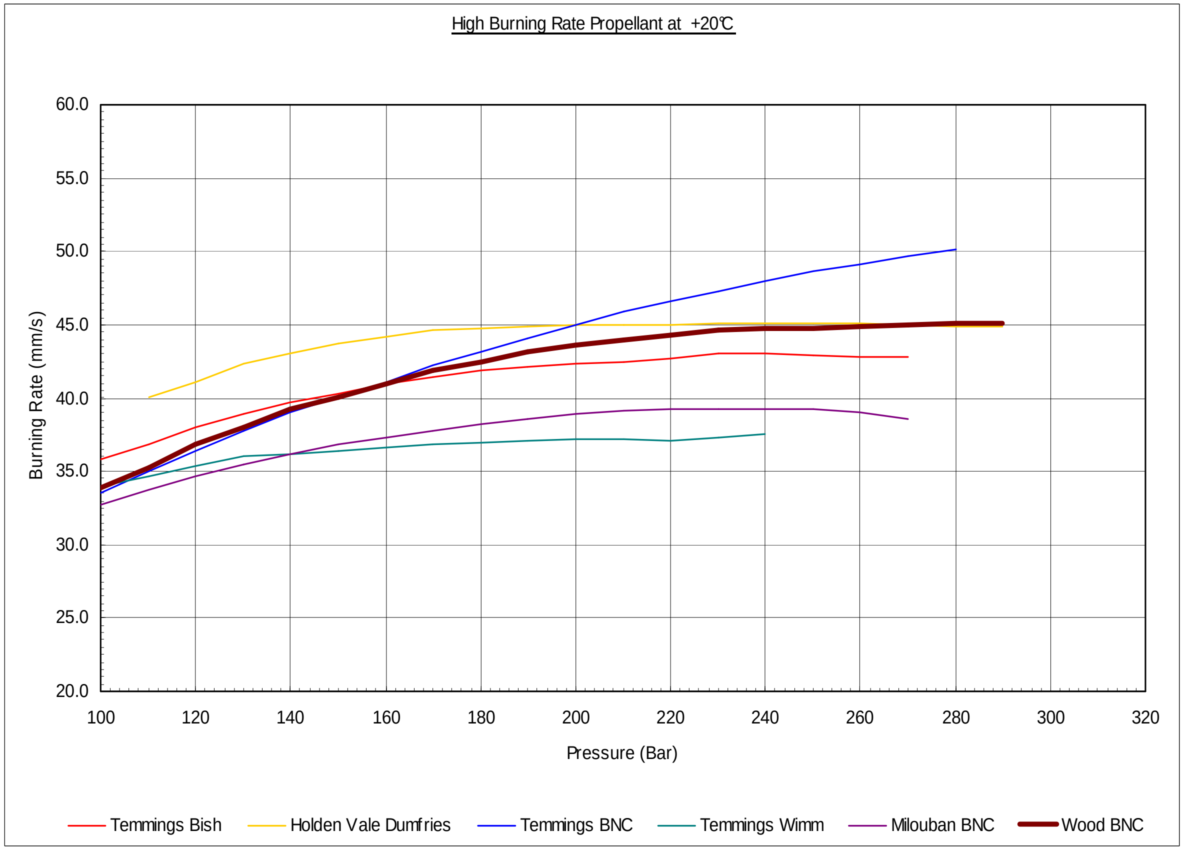
Ballistic Spectrum (Slab) Motor



Measures Burn-Rate Across a Wide Pressure Range.

Ballistic Performance – NC/Cellulose Types



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    - Initial Ballistics unplateaunised ('Tram Lines)
    - Multiple iterations and delays
  - Eventual success
  - Root causes of difficulties not fully understood
  - Propellant tolerance to a 'wood' NC apparently greater than a 'flock' source switch

Large Rocket Motor Problems: Common Causes

- Reliance on small scale proof testing gave false confidence of tolerance to NC changes
- Lack of understanding of key nitrocellulose parameters

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    - NC solubility
    - NC gelatinisation
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- Lack of understanding of key nitrocellulose parameters
  - For propellant mechanical properties, ballistics and bonding:
    - NC molecular weight distribution
    - NC solubility
    - NC gelatinisation
    - NC diffusion
  - None of these parameters was adequately understood

DOSG/DA/AWE/Roxel Research Programme

- Aims:
  - Develop a reliable NC molecular weight distribution test method
  - Monitor NC molecular weight during service life
    - Develop extraction techniques
  - Establish linkage between NC parameters and propellant service life limiting failure modes

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  - Programme Continuing…

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Conclusion

- High Performance Rocket Motors can be sensitive to nitrocellulose parameters
- The resultant failure modes have not always been apparent at propellant proof level
- Lower Performance Rocket Motors and gun propellants have demonstrated insensitivity to cellulose/nitrocellulose source change
  - Recent changes in cellulose flock source have not produced any clearly discernable difference in propellant or weapon system performance

Proposed Way Forward – Gun/LP RM

- DOSG ST1 as UK National Authority for Explosive Qualification accept that changes between alternative cellulose flock sources do not require individual STANAG 4170 qualification
  - Propellant physical, chemical, hazard and performance proof parameters must be consistent with production experience of qualified source
  - DOSG ST1 may specify propellant/type tests where individual safety related or life limiting failure modes have been identified

Proposed Way Forward – High Performance RM

- UK currently has relatively few high performance rocket motors in production or development
- The numbers required per Tranche is relatively low

- DOSG ST1 proposes that a stock of cotton linters sufficient to complete development and First Tranche Production be procured as early as possible in the project
  - Concerns have been raised about possible cotton linter ageing mechanisms
  - DOSG ST1 has no experience of these and considers the risk insignificant with respect to the proven risk from cellulose source changes

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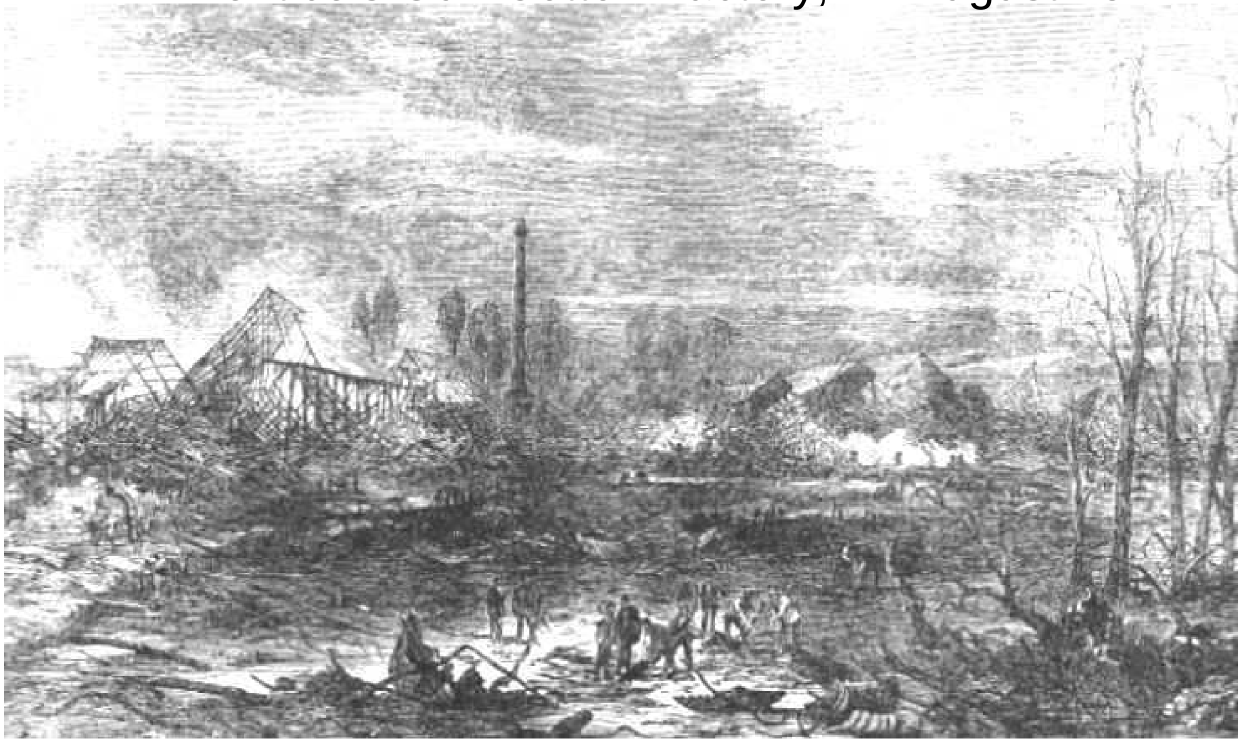
Chemical Stability of Nitrocellulose Propellants

- Historic UK Civil and Military Tests
  - Abel Heat Test
  - 80^{O}C Self Heating Test (originally ‘Silvered Vessel Test’)
  - 65.5^{O}C Chemical Stabiliser Consumption Test

- Test Requirements included in:
  - HSE Conditions for Authorisation (Now Superseded)
  - JSP482 MoD Explosive Regulations
    - JSP 762 Weapons… Through Life Capability Management

Abel Heat Test: Response to Various Accidents

Prentice's Gun Cotton Factory; 11 August 1871



1914 - Requirement that "all nitro –compound explosives… satisfy the conditions of the Abel Heat Test."

Abel Heat Test for NC: STANAG 4178 Edition 2

- 65.5 and 76.6^{O}C Abel Heat Test retained as a 'Purity Test'
- Historically Bergmann-Junk and 135^{O}C Methyl Violet Test NC Stability Tests generally used outside UK

- 'Purity Test' view has (broadly) prevailed in the UK for decades:
  - HS(G).114 (1994): "The Abel heat test is not an absolute measure of stability."
- Since NC manufacture in the UK has ceased, Abel Heat Test as applied to NC seen as an academic issue
  - UK MoD makes neither NC nor NC/NG Paste
  - DOSG's interest is in Propellant Stability

Abel Heat Test for NC Propellant

- MoD DOSG regard the Abel Heat Test as a useful comparative test – when intelligently applied
- Large Calibre Gun Propellant Surveillance/Environmental Test Trial
  - Two Triple Base Propellant Type
  - 42 Samples of Propellant 1
  - 28 Samples of Propellant 2

Large Calibre Propellant Trial

Abel Heat Test Time at 65.5ºC

Heat Test Time/mins	2 1/2	3	3 1/2	4	4 1/2	5	5 1/2	6	6 1/2	7	7 1/2	8	8 1/2
Occurrence Propellant 1	1	1	14	38	12	9	5	2	0	0	0	1	1
Occurrence Propellant 2	0	3	5	23	15	8	2	0	0	0	0	0	0

UK Propellant: ≥ 10 minutes Abel Heat Test at Manufacture

Rounds had experienced Prolonged UK storage then Overseas Deployment and Finally Environmental Testing at 63ºC

All samples passed AOP-48 (Ed. 1) Chemical Stabiliser Consumption (Carbamite) and Sentenced as having a further 10 Years Service Life

Large Calibre Propellant Trial: Questions

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  - What was the safety significance?
    - (BR1203 ≤ 4 minutes Heat Test required immediate disposal)

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- Once established that results were far lower than at manufacture:
  - What was the cause of the Low Heat Tests?
  - What was the safety significance?
    - (BR1203 ≤ 4 minutes Heat Test required immediate disposal)
  - Was AOP-48 Stabiliser Consumption Sufficient to confirm Safety and Service life?
    - Propellant > 7.0% Carbamite – unlikely to fail AOP-48
      - Tested and Passed 70 times!

Proposed Use of Abel Heat Test for NC Propellants

- Abel Heat Test should not be used as the primary measure of NC based propellant stability – prefer AOP-48 Ed 2
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  - What is the Safety Significance?
    - Carry out additional test to confirm safety:
      - 80^{O}C Self Heating Test
      - 90^{O}C Mass Loss Tests
      - Heat Flow Calorimetry/Accelerated Rate Calorimetry

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Stability Tests for Nitrate Ester Propellants

- DOSG are concerned that nitrate ester propellant stability experience is being lost:
  - Inadequate understanding of nitrate ester failure modes
  - Instances of failure to understand ‘catastrophic’ decomposition is a possibility – e.g. Gas-Crack

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- DOSG are concerned that nitrate ester propellant stability experience is being lost:
  - Inadequate understanding of nitrate ester failure modes
  - Instances of failure to understand 'catastrophic' decomposition is a possibility – e.g. Gas-Crack
  - Blind reliance on AOP-48 testing to 'Prove' stability
    - Has led to dangerous trial design

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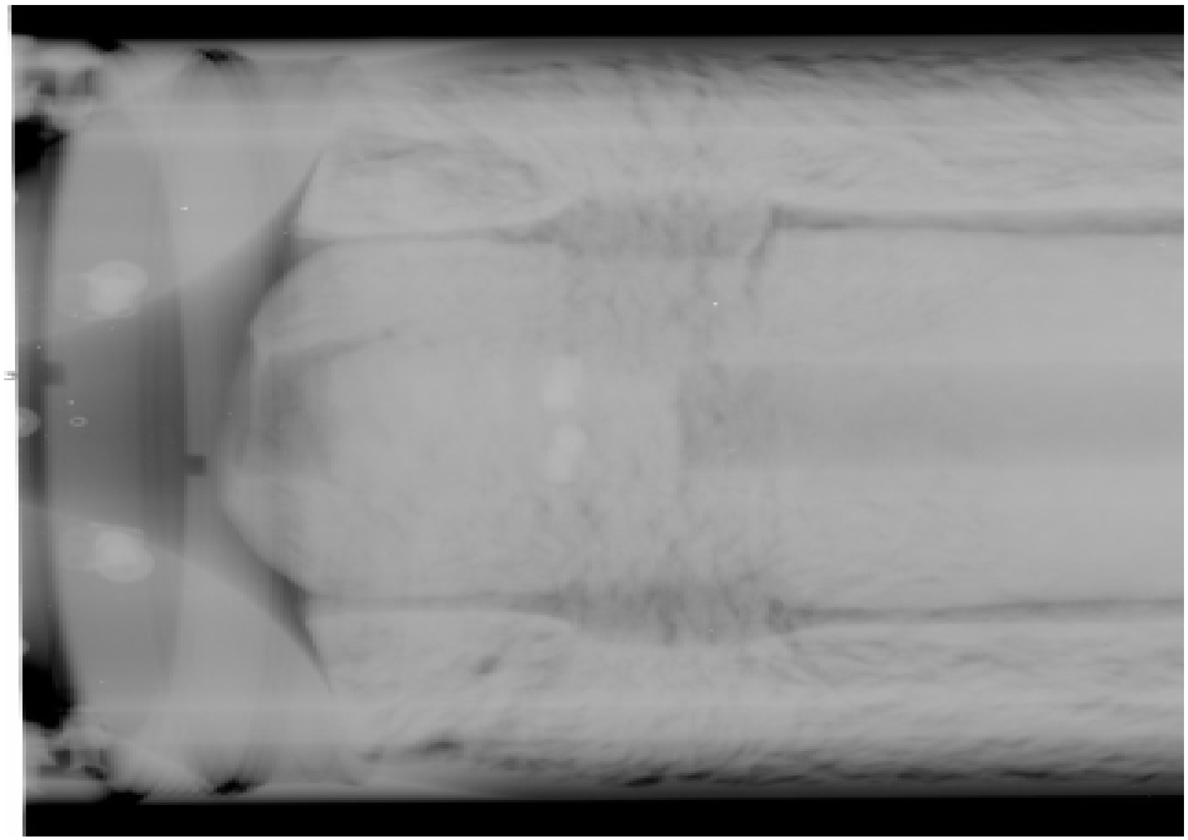
Nitrate Ester Propellant: Dangerous Trial Design



"But it Passed AOP-48…"

Conclusions: Nitrate Ester Testing

- A wide range of tests exist
- It is rarely a case of ‘good’ and ‘bad’ tests
- Rather ‘appropriate’ and ‘inappropriate’ tests

Conclusions:

- A wide range of tests exist
- It is rarely a case of ‘good’ and ‘bad’ tests
- Rather ‘appropriate’ and ‘inappropriate’ tests

- NC propellants are not entirely understood
- NC propellants will be required for decades to come
- Further research is essential for both safety and reliability

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