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OHMS RESEARCH AND DEVELOPMENT FORUM:  
ENERGETIC MATERIALS PANEL

# UN Manual of Tests & Criteria

# Areas Identified for Improvement: Ideas for Potential R&D Projects

Presented by

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16 April 2015

- Areas identified for improvement/change:
  - Exclusion of insensitive substances from Class 1
  - UN Series 4 (b) (ii) 12-meter drop test criteria
  - UN Series 6 (c) External fire (bonfire) test specifications
    - Burning Time and Heat Flux Qualifiers
    - Measurement of Flame Distances
    - Minimum Test Quantities
  - Risk and approval for small quantities of explosives
  - Definition of desensitized explosives

Referencing United Nations (UN) Recommendations on the Transport of Dangerous Goods, Manual of Tests and Criteria, fifth revised edition (2009) as the Explosives Test Manual (ETM).

# Exclusion of Insensitive Substances from Class 1



Exclusion from Class 1 for substances

UN Series 4 (b) (ii)  
12-m (40-ft) drop

UN Series 6 (c)  
External fire (bonfire)

Burning time and  
heat flux qualifiers

Measurement of  
flame distances

Minimum test  
quantities

Risk and approvals  
for small quantities

Desensitized  
explosives

- **IMPORTANT:** Route for insensitive substances to be excluded from Class 1 (currently no protocol).
- **ISSUE**
  - Potential for future changes to the ETM's Figure 10.2 Class 1 Acceptance Procedure: UN Series 1 / 2
    - In the past, test series could only be performed on substances NOT manufactured with the view to producing a practical explosive or pyrotechnic effect
    - IGUS initially appears to be supportive of removing this restriction (Box 2)
    - If change is approved, it will permit UN Series 1 / 2 to be performed on explosive substances for Exclusion from Class 1
  - SMS was asked to write a white paper to this regard (due June 2015)

# Exclusion of Insensitive Substances from Class 1



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- SMS recently performed testing on two propellants from different manufacturers that would be excellent candidates to test the proposed change
  - One propellant manufacturer in Alabama, one in Nevada
  - Both propellants are very insensitive at ambient pressures
    - Not unusual to pass the UN Series 1 (a) UN Zero Gap test (larger critical diameter)
    - Not unusual to pass the UN Series 1 / 2 (c) (ii) Time/pressure test since the propellant is extremely difficult to ignite
    - The insensitive propellants may fail the Koenen test since they are designed to require confinement and pressure in order to sustain a reaction
  - One (Alabama) has already passed UN Series 4 & 6 and is currently recommended for Division 1.4S
  - One (Nevada) has already passed the UN Zero Gap test with no damage to the witness plate

# Exclusion of Insensitive Substances from Class 1



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- **OBJECTIVE**

- Receive sponsorship from U.S. DOT PHMSA R&D group to procure the insensitive propellants and subject the propellants to the full UN Series 1 / 2 tests

- **EXPECTED OUTCOME/PRODUCT**

- Testing would form a technical basis for the white paper and proposed change to the ETM
- Proposed change would give insensitive substances a route to be Excluded from Class 1, similar to articles

- **FUNDING/SCHEDULE**

- \$10K for procurement and transport of the insensitive propellants
- \$25K for performance of UN Series 1 / 2 on the two propellants
- ASAP

# Exclusion of Insensitive Substances from Class 1



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- **ISSUE 2**

- UN Series 1 / 2 (c) (ii) Time/pressure test specifies the usage of **primed cambric** for ignition source
  - Not readily available in the U.S.
    - Stock in Canada, Great Britain, and Sweden

- **OBJECTIVE**

- Develop an alternative equivalent to primed cambric
  - Potential candidates: pyrogen, black powder, smokeless powders, etc.

- **EXPECTED OUTCOME/PRODUCT**

- Proposal to IGUS for a recommended equivalent alternate for incorporation in the ETM

- **FUNDING/SCHEDULE**

- TBD

# UN Series 4 (b) (ii)

## 12-meter (40-foot) Drop Test



Exclusion from Class 1 for substances

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12-m (40-ft) drop

UN Series 6 (c)  
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Burning time and heat flux qualifiers

Measurement of flame distances

Minimum test quantities

Risk and approvals for small quantities

Desensitized explosives

- **IMPORTANCE:** Threat to safety of test personnel
- **ISSUE**
  - Conflicting test specifications
    - 14.5.2.1 Introduction: This test determines whether a test unit can withstand a free-fall impact without producing any significant fire or explosion hazard.
    - 14.5.2.4 Test criteria: The test result is considered "+" and the packaged substance or article(s) too dangerous to transport if a fire or explosion resulted from impact.
    - 14.5.2.3 Procedure: The test unit is dropped...The test unit should then be further examined to determine if any ignition or initiation has occurred.
      - Examination for “any ignition or initiation” requires unnecessary hazardous investigation

# UN Series 4 (b) (ii) 12-meter (40-foot) Drop Test



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- Examination for “any ignition or initiation” requires unnecessary hazardous investigation:
  - Handling and opening compromised outer packagings (metal drums, wood crates, fiberboard boxes)
  - Opening and handling compromised intermediate/ inner packagings to access and inspect the substance or article
  - Disassembly of articles that may fully contain/ conceal “any ignition or initiation”.
    - Potentially encounter out-of-place explosives



# UN Series 4 (b) (ii) 12-meter (40-foot) Drop Test



Exclusion from Class 1 for substances
UN Series 4 (b) (ii) 12-m (40-ft) drop
UN Series 6 (c) External fire (bonfire)  Burning time and heat flux qualifiers
Measurement of flame distances
Minimum test quantities
Risk and approvals for small quantities
Desensitized explosives

- **OBJECTIVE**

- Propose change to the ETM, replacing “The test unit should then be further examined to determine if any ignition or initiation has occurred.” with “Examine the test area for evidence of significant fire or explosion.”

- **EXPECTED OUTCOME/PRODUCT**

- U.S. DOT PHMSA sponsored white paper to IGUS that is vetted, approved, and incorporated into the ETM.

- **FUNDING/SCHEDULE**

- TBD

# UN Series 6 (c)

## External Fire (Bonfire) Test



Exclusion from Class 1 for substances
UN Series 4 (b) (ii) 12-m (40-ft) drop
UN Series 6 (c) External fire (bonfire)

Burning time and heat flux qualifiers

Measurement of flame distances

Minimum test quantities

Risk and approvals for small quantities

Desensitized explosives

- **ISSUES**

- Burning Time and Heat Flux Qualifiers
- Measurement of Flame Distances
- Minimum Test Quantities

# UN Series 6 (c): Burning Time and Heat Flux Qualifiers



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UN Series 4 (b) (ii) 12-m (40-ft) drop
UN Series 6 (c) External fire (bonfire)
Burning time and heat flux qualifiers
Measurement of flame distances
Minimum test quantities
Risk and approvals for small quantities
Desensitized explosives

- **IMPORTANT:** Division 1.3/1.4/1.4S Burning Time and Heat Flux Qualifiers are a **significant parameter/variable directly affecting the final hazards classification** of materials
- **ISSUE 1**
  - Dr. Spencer Watson's 2013 Research
    - Model & empirical qualifiers based off of a series of tests
    - Test data for propellants (critical reference/baseline material) did not fit the model and were apparently disregarded
- **ISSUE 2**
  - Challenges in measuring burning time
    - Test criteria assessed on basis of burning time per mass
    - Rarely linear burn rate on bonfire
      - Burning rate for one packaging often like a bell curve
      - Very difficult to discern burning times of adjacent materials



Basis for UN 6c burning time criteria.pdf

# UN Series 6 (c): Burning Time and Heat Flux Qualifiers



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Burning time and heat flux qualifiers
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Desensitized explosives

- **ISSUE 3**
  - Heat flux measurement and qualifiers used for “articles and low energy substances”; not aware of anyone employing these instruments for commercial
    - Utilized routinely for U.S. DoD classification testing.
- **ISSUE 4**
  - Heat of combustion changes burning time and heat flux qualifiers; no accepted or recommended methodologies for determining heat of combustion
- **ISSUE 5**
  - Simple scaling of current burning time qualifiers to mass can be improved with implementation of a chart, like that used to assess projection energy

# UN Series 6 (c): Burning Time and Heat Flux Qualifiers



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Exclusion from Class 1 for substances

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Burning time and  
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Measurement of  
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Minimum test  
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Desensitized  
explosives

- **OBJECTIVE**

- Collect test videos and heat flux test data from the explosive test labs and evaluate typical product burning times and heat flux measurements
- Determine whether an improved set of test criteria can be recommended to IGUS for adoption in the ETM.
  - For example, perhaps the burning time should be measured using a controlled ignition test (confined/unconfined) employing the smallest packaging unit and a standardized ignition source/ location (eliminating many of the variables)
    - Criteria established for burning rate of flash powder

# UN Series 6 (c): Burning Time and Heat Flux Qualifiers



Exclusion from Class 1 for substances
UN Series 4 (b) (ii) 12-m (40-ft) drop
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<b>Burning time and heat flux qualifiers</b>
Measurement of flame distances
Minimum test quantities
Risk and approvals for small quantities
Desensitized explosives

- **EXPECTED OUTCOME/PRODUCT**
  - Updated test criteria incorporated into the ETM for identifying and classifying Division 1.3 materials presenting a mass fire hazard through improved burning time and/or qualifiers
- **FUNDING/SCHEDULE**
  - TBD

# UN Series 6 (c): Measurement of Flame Distances



Exclusion from Class 1 for substances
UN Series 4 (b) (ii) 12-m (40-ft) drop
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Burning time and heat flux qualifiers
Measurement of flame distances
Minimum test quantities
Risk and approvals for small quantities
Desensitized explosives

- **IMPORTANCE:** Prescribed Division 1.3/1.4/1.4S flame distances are a **significant parameter/variable directly affecting the final hazards classification** of materials
- **ISSUE**
  - Fireballs or jets of flame are classified as :
    - Division 1.4S if they extend 1 meter or less from the flames of the fire
    - Division 1.4 if they extend more than 1 meter from the flames of the fire
    - Division 1.3 if they extend beyond any of the witness screens (positioned 4 meters from the edge of packages)
      - Using a different frame of reference that is problematic problematic if the wind is significantly blowing near 6 m/s
        - » Less ambiguous if replaced with 3 - 4 meters from the flames of the fire

# UN Series 6 (c): Measurement of Flame Distances



Exclusion from Class 1 for substances

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Burning time and heat flux qualifiers

Measurement of flame distances

Minimum test quantities

Risk and approvals for small quantities

Desensitized explosives

- Very rare that radius of fireballs from Division 1.3 propellants meet or exceed the specified distance
  - Heated fireballs will rapidly rise (rather than expand)
  - Will typically first fail the Division 1.3 burning-time qualifier
- **OBJECTIVE**
  - Collect test videos from the explosive test labs and evaluate typical product fireball sizes for Division 1.3
  - Determine whether an improved set of test criteria can be recommended to IGUS for adoption in the ETM.
- **EXPECTED OUTCOME/PRODUCT**
  - Updated test criteria incorporated into the ETM for identifying and classifying Division 1.3 materials presenting a mass fire hazard through improved fireball size assessment.
- **FUNDING/SCHEDULE**
  - TBD

# UN Series 6 (c): Minimum Test Quantities



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Desensitized explosives

- **IMPORTANCE:** Prescribed minimum quantities may not provide any additional information for unambiguous classification than equivalent smaller quantities
- **ISSUE**
  - Prescribed minimum quantities (at least 0.15 m<sup>3</sup> with a minimum of three packages)
    - Small articles in small packages require 6 - 18 packages to meet this volume requirement and often tens of thousands of articles
      - High asset cost when small, hand made, and/or not intended for transport in large quantities
      - Cleanup of thousands and tens of thousands of reacted/unreacted articles unnecessary when hazard could have been adequately classified using significantly lesser quantities

# UN Series 6 (c): Minimum Test Quantities



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Desensitized explosives

- New Klieboldt test to be added to next revision of ETM to alleviate this problem for ammunition.
- Companies currently making decision to go at risk and provide lesser quantities for testing.
- **OBJECTIVE**
  - Determine whether lower test quantities are appropriate in certain circumstances to enable unambiguous characterization of the hazard classification.
- **EXPECTED OUTCOME/PRODUCT**
  - Provisions to be added to the ETM permitting lower test quantities in the UN Series 6 (c) External fire (bonfire) test if such quantities permit unambiguous characterization of the hazard.
- **FUNDING/SCHEDULE**
  - TBD

# Risk and Approvals for Small Quantities



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Risk and approvals for small quantities
Desensitized explosives

- **IMPORTANCE:** No classification route for approval of R&D and lab-scale quantities outside of DOT-SP 8451
- **ISSUE 1**
  - DOT-SP 8451 allows for transport of new, unapproved explosive substances in a special shipping container (up to 25 grams N.E.W. PETN equivalency)
    - New explosives
    - Heavy and time consuming
  - DOT-SP 8228 allows for transport of 35-gram explosives samples for laboratory examination to U.S. Gov't laboratories
    - New explosives
    - Light and easy but limited usage

# Risk and Approvals for Small Quantities



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Risk and approvals for small quantities

Desensitized explosives

- New explosive substances
  - UN Series 3 (small quantity) > **Not forbidden**, Division 1.1
    - Impact and friction sensitivity tests: <10 grams N.E.W.
    - Thermal stability test: 6 - 150 grams N.E.W.
    - Small-scale burning test: 20 - 220 grams N.E.W.
    - Total: 50 - 375 grams N.E.W. typical
  - UN Series 5/6 (large quantity) > **Division 1.1 - 1.5**
    - Single package and bonfire tests: 100 - 400 lbs N.E.W.
- **OBJECTIVE**
  - Determine an approval route for R&D materials in 10 and 100 g quantities (mini Single package and bonfire tests)
- **EXPECTED OUTCOME/PRODUCT**
  - Proposal to IGUS for incorporation in the ETM
- **FUNDING/SCHEDULE**
  - TBD

# Risk and Approvals for Small Quantities



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Desensitized explosives

- **ISSUE 2**
  - Materials contaminated with limited quantities of explosives
    - Industrial hygiene samples
    - Equipment used for processing explosives to be returned to the manufacturer to be refurbished
      - Disassembled, cleaned and free from hidden spaces
- **OBJECTIVE**
  - Develop threshold quantities of concern and an approval route for materials below that threshold
- **EXPECTED OUTCOME/PRODUCT**
  - Approval route for materials below a threshold of concern
- **FUNDING/SCHEDULE**
  - TBD

# Definition of Solid & Liquid Desensitized Explosives



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flame distances

Minimum test  
quantities

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for small quantities

Desensitized  
explosives

- **ISSUE**
  - Current definitions based upon solubility of the explosive in the wetting agent (Liquid: dissolved or suspended in water or other liquid; Solid: homogeneous solid mixture wetted with water or alcohols)
    - Apparent discrepancy -- potential considerations for 1) flammability of wetting agent, 2) liquid explosives, 3) desensitizing agents besides wetting agents (e.g. soil)
  - PG I vs PG II for desensitized (1H2 for liquids not available)
- **OBJECTIVE**
  - Evaluate the adequacy of the current definition of desensitized explosives
- **EXPECTED OUTCOME/PRODUCT**
  - Proposal to IGUS to revise the definition of desensitized explosives based on findings of evaluation
- **FUNDING/SCHEDULE**
  - TBD

# Conclusions



- Identified areas of improvement based on SMS's testing experience and interfacing with clients
  - Some may have potential for further development or investigation by PHMSA R&D
  - Explosives represent only one area of PHMSA stewardship
- Technical expertise of PHMSA transportation specialists and culture of improvement for hazards classification system
- Discussion / questions?