# THERMAL ANALYSIS OF IMX-101 THROUGH LARGE SCALE SLOW COOK OFF TESTING

## NDIA Insensitive Munitions & Energetic Materials Technology Symposium 2009


**Alberto Carrillo***
**BAE SYSTEMS OSI, Holston Army Ammunition Plant**

# Why Investigate Thermal Hazards?

- Exposure to elevated temperatures over time can lead to
  - Decomposition
  - Self-heating
    - Point at which heat generated exceeds heat lost to surroundings
    - Can lead to both catastrophic and non-catastrophic events

- An assessment of an energetic material's thermal hazards are necessary to determine safety margins for processing and loading
  - Critical Temperature ($T_c$) is utilized most often to asses the thermal hazards associated with processing and loading of melt cast energetic materials
    - Defined as the lowest constant temperature at which a given material of a specific shape and size will catastrophically self heat
    - Affected by mass and shape
    - Several mathematical models for $T_c$ determination exist

# Required Testing for Qualification

- Mandatory Thermal Evaluations for Qualifications of Explosive
  - Critical Temperature Calculation
    - Test Method 1074 of MIL-STD-1751A
    - Mathematical determination utilizing Differential Scanning Calorimetry (DSC)
  - 1 Liter Cook Off Test
    - Test Method 1075 of MIL-STD1751A
    - Slow heating (3.3°C/hr) of material to determine self-heating temperature
    - Evaluation of decomposition reaction
  - Results from both tests and thermal models used to predict $T_c$ at large scale
    - Frank-Kamenetskii (F-K) model for conductive heat transfer with no agitation

$$T_c = \left( \frac{E_a}{R} \ln\left( \frac{d^2 \rho Q Z E_a}{T_c^2 \lambda \delta R} \right) \right)$$

    - Semenov model for convective heat transfer with agitation

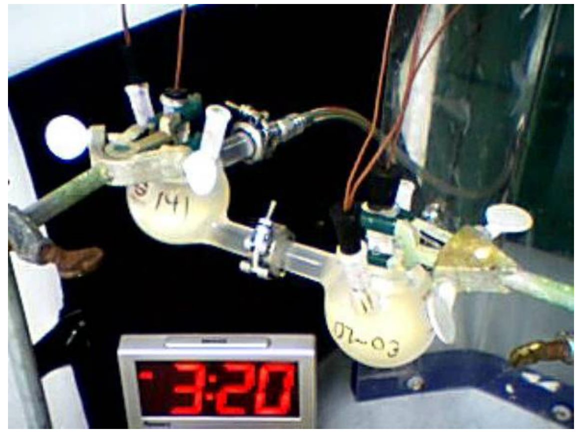
$$T_c = \left( \frac{E_a}{R} \ln\left( \frac{T_c^2 S \alpha R}{V \rho Q Z E_a} \right) \right)$$

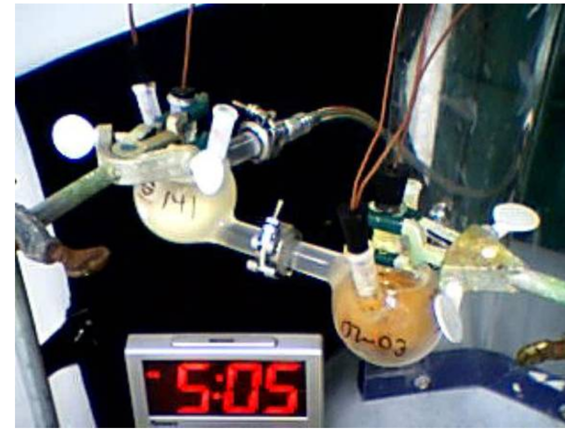
# Background

- IMX–101 is a non-traditional IM Melt Cast Formulation
  - Manufactured at HSAAP utilizing existing infrastructure
  - Melting point of 96°C to 98°C by DSC
  - Processed at greater than 100°C
- 1 Liter Cook Off
  - Experienced non-catastrophic event between 142°C and 149°C
  - Predictions for $T_c$ using F-K and Semenov models are extremely conservative
    - F-K extrapolates to value below melting point at large diameters
    - Semenov extrapolates to a $T_c$ of 130°C at large diameters
  - Result (with scaling effect considered), suggested IMX-101 was not safe to handle in large scale production as its $T_c$ was considered too low
- Holston Small Scale Cook Off Testing
  - Various amounts (small scale) tested at fast and slow heating rates also experienced mild thermal event between 145°C and 155°C
  - Similar finding to the 1 Liter Cook Off Test, suggests $T_c$ prediction is **not** affected by **reduction** in material mass

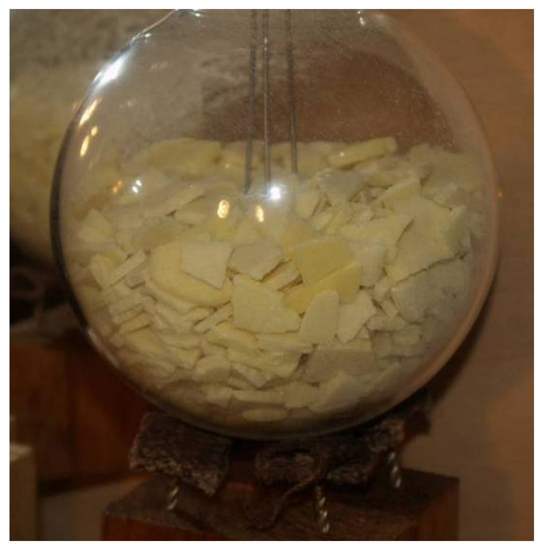
# Background (cont)

**Holston Small Scale Cook Off Test**




**1 Liter Cook Off Test**



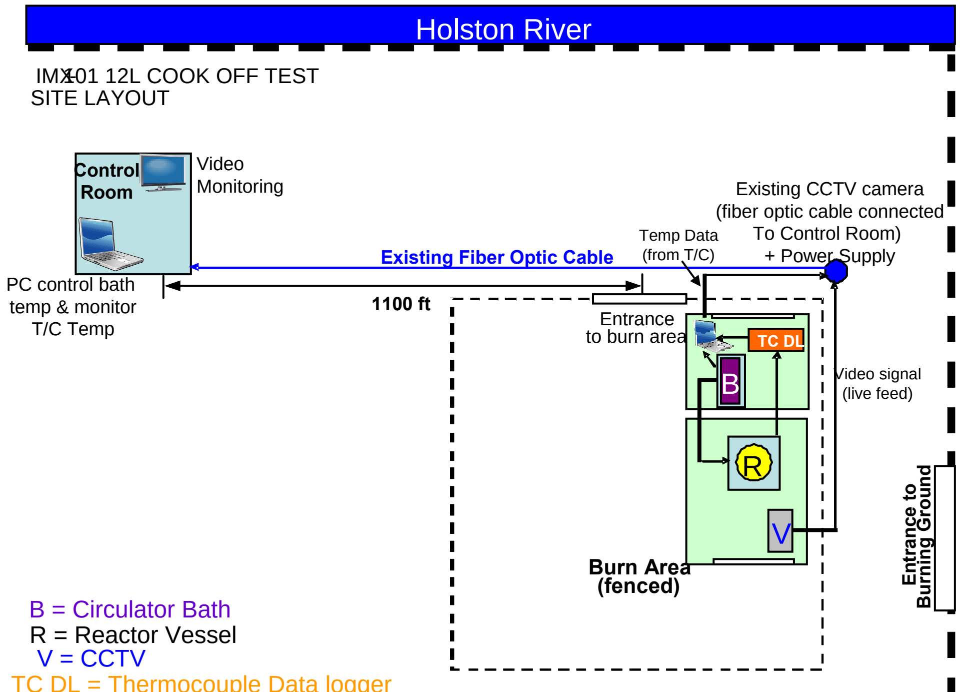
Photos of 1 Liter Cook Off Test courtesy of US Army Research Laboratory (ARL)

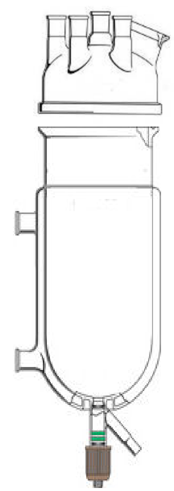
# Objective

- Determine whether the scaling effect for $T_c$, as predicted by both the F-K and Semenov Models exist for non-traditional melt pour formulation IMX–101 in a larger configuration
  - Compare $T_c$ between Small Scale, 1L, and 12L Cook Off Test
  - Demonstrate that thermal event is independent of kettle size

- 12 Liter Cook Off Test
  - Increase amount from 1 Liter Cook Off Test by factor of 12
  - Utilize 15 liter jacketed reactor with geometry closer to production melt kettles
  - Heat at rate of 3.3°C/hr

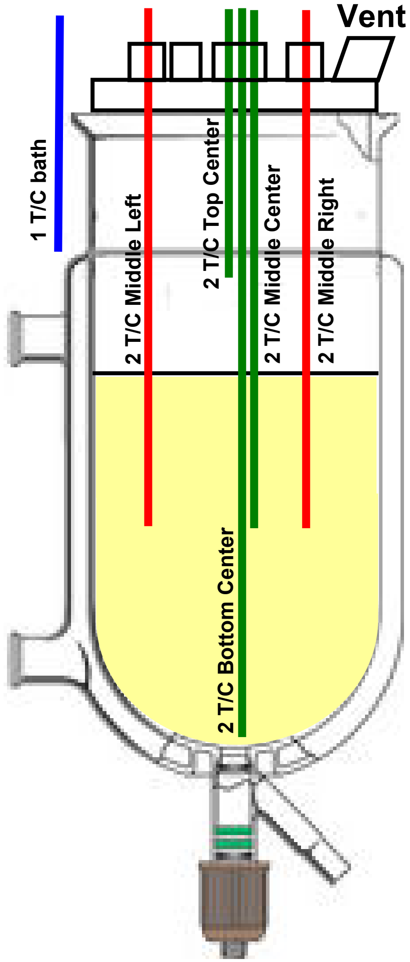
# 12 Liter Cook Off Test Site Layout

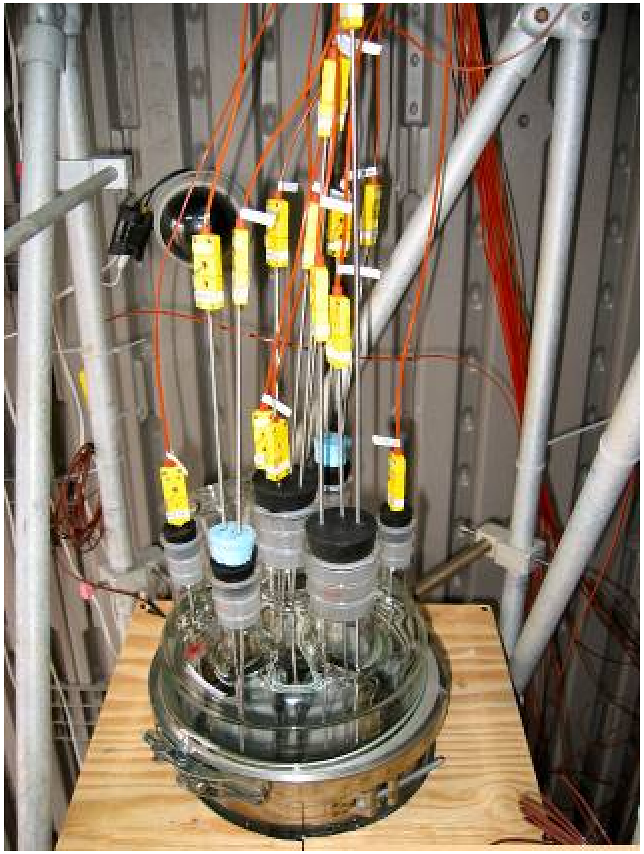


- Control Room located 1100 feet from actual test site
- All equipment must have remote capabilities
  - Data logger, CCTV, heating bath
- Tap into existing power and fiber optic network
- Containment of test vessel
  - 15 Liter reactor separate from other equipment
- Cannot enter test site upon start of test

# Thermocouple Placement



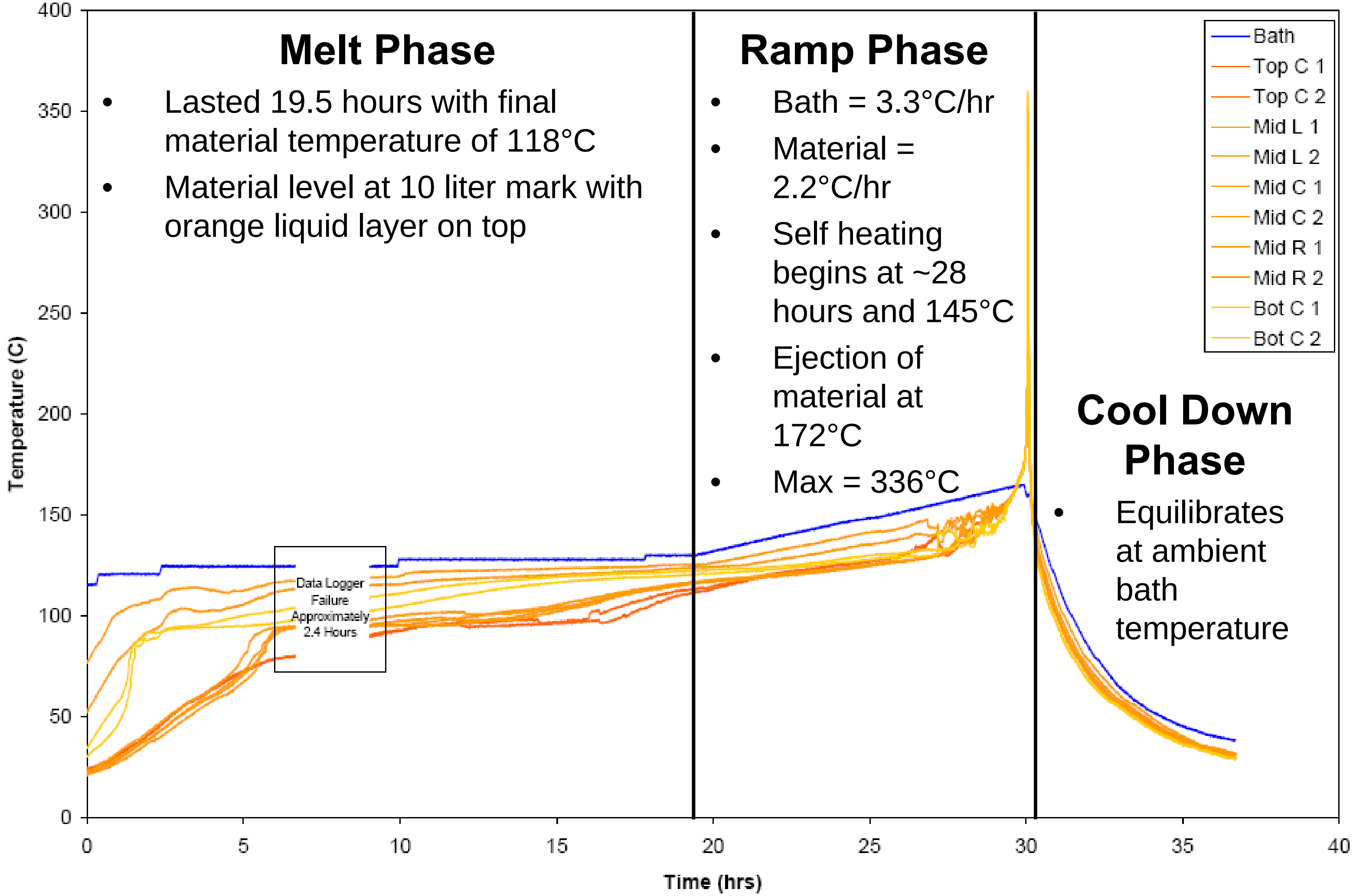



# Test Setup

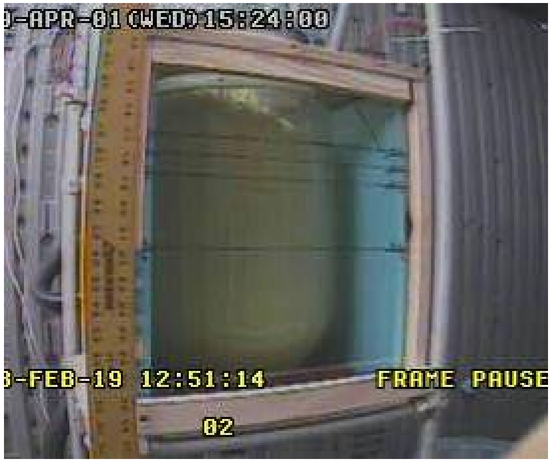

2009-APR-01(WED)15:22:25
01
02
2008-FEB-19 12:51:02
FRAME PAUSE
03
04

BAE SYSTEMS

# Thermocouple Data



# Melt Phase

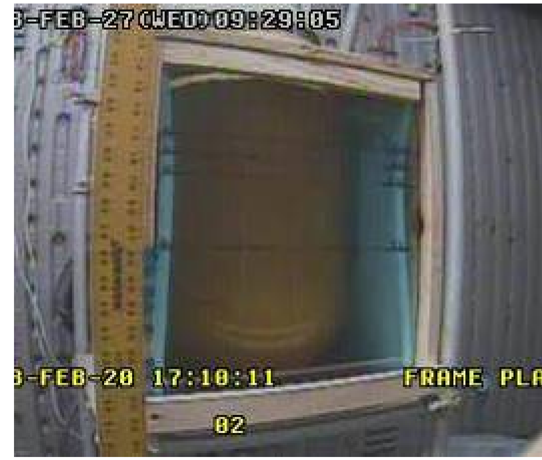





| Start of Melt Phase | Middle of Melt Phase | End of Melt Phase |
|---|---|---|
| 0 Hours into test | 8.5 Hours into test | 19.5 Hours into test |
| Bath Temp = 115°C | Bath Temp = ~124°C | Bath Temp = 130°C |
| Material Temp = 35°C | Material Temp = ~98°C | Material Temp = 118°C |

# Ramp Phase (Prior to 29 Hours)






| Early in Ramp Phase | Middle of Ramp Phase | Late in Ramp Phase |
|---|---|---|
| 20.5 Hours into test | 25.5 Hours into test | 28.5 Hours into test |
| Bath Temp = 134°C | Bath Temp = 150°C | Bath Temp = 160°C |
| Material Temp = 120°C | Material Temp = 130°C | Material Temp = 144°C |

# Ramp Phase (29 Hours)



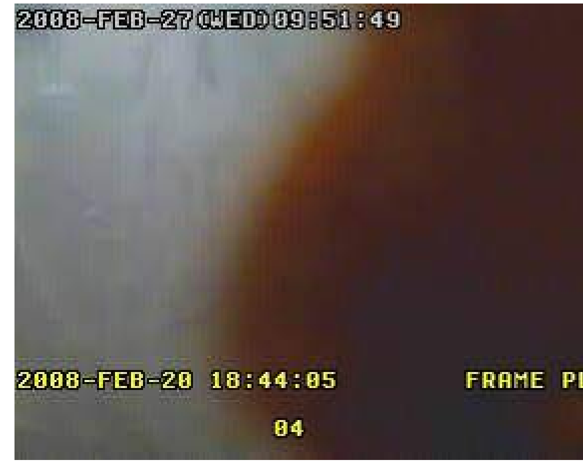

| Smoking Start | Self Heating at ~3x Rate | Beginning of Ejection |
|---|---|---|
| 29.1 Hours into test | 29.5 Hours into test | 29.9 Hours into test |
| Bath Temp = 162°C | Bath Temp = 163°C | Bath Temp = 164°C |
| Material Temp = 150°C | Material Temp = 158°C | Material Temp = 172°C |

# Ejection of Material


| | | | | | |
|---|---|---|---|---|---|
| **18:32:04** | **18:32:57** | **18:33:21** | **18:33:23** | **18:33:46** | **18:34:04** |
| **Bath 164.5°C** | **Bath 164.4°C** | **Bath 164.4°C** | **Bath 164.5°C** | **Bath 164.9°C** | **Bath 164.5°C** |
| **Material 170.7°C** | **Material 171.5°C** | **Material 171.6°C** | **Material 171.9°C** | **Material 172.2°C** | **Material 172.7°C** |

# Temperature Max/Camera Obscuration






| Maximum Material Temperature | Obscuration of Camera | Obscuration of Camera |
|---|---|---|
| 30.0 Hours into test | 30.1 Hours into test | 30.1 Hours into test |
| Bath Temp = 165°C | Bath Temp = 160°C | Bath Temp = 160°C |
| Material Temp = 336°C | Material Temp = 252°C | Material Temp = 252°C |

# Cool Down Phase




| Camera 1 | Camera 2 | Camera 4 |
|---|---|---|
| 30.8 Hours into test | 30.8 Hours into test | 30.8 Hours into test |
| Bath Temp = 123°C | Bath Temp = 123°C | Bath Temp = 123°C |
| Material Temp = 106°C | Material Temp = 106°C | Material Temp = 106°C |

# Post Test Observations

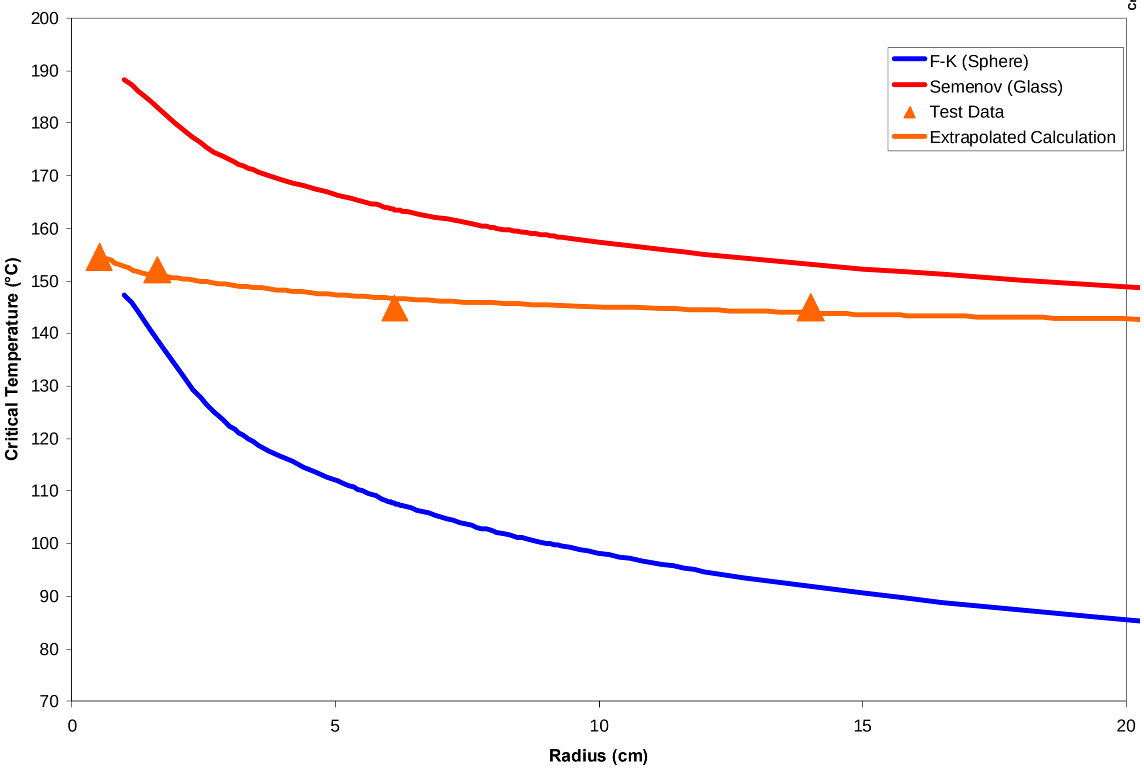
- Shed
  - Coated with yellow powder (inside and outside)
  - Considerable amount of ejected material with splatter on walls
  - No visual signs of burning or damage
- Reactor
  - Coated with material and undamaged
- Thermocouples
  - Still functioning
- Material
  - Post test analysis indicates ejected material is primarily IMX-101

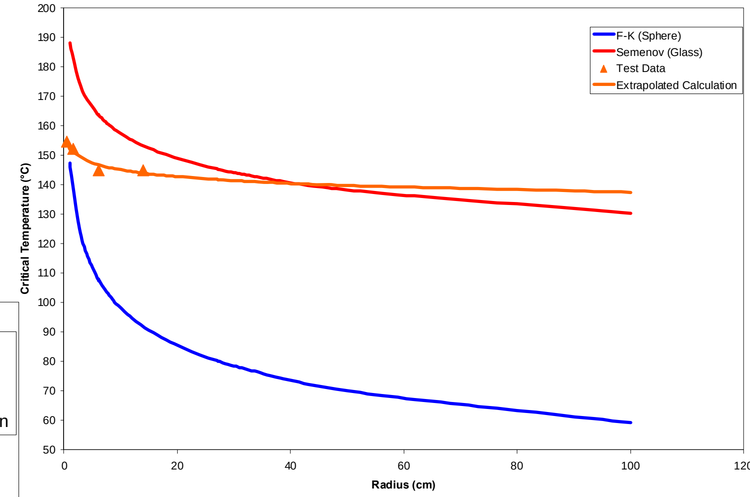





# Experimental Data vs. Predictions

- Test data holds to a shallow curve compared to F-K and Semenov models




- $T_c$ is similar at all sizes
  - Constant at 145°C ± 5°C
- Indicates predicting $T_c$ of IMX–101 may not follow traditional models

# Conclusions

- 12 Liter Cook Off Test
  - IMX–101 displayed start of self heating at 145°C and non-catastrophic thermal event at 172°C
  - Results similar to results from 1 Liter Cook Off Test and Small Scale Cook Off Testing

- IMX–101
  - Critical Temperature prediction based on the F-K and Semenov model did not apply to IMX-101
    - Scaling effect not applicable
  - Thermal event resulted in ejection of hot material from vessel, a non-catastrophic event

# Conclusions (cont)

- Thermal Hazards
  - Conservative estimates obtained from F-K and Semenov models
  - IMX-101 **proven to be safe** to process/load on large scale with appropriate control measures
  - Official statement from Energetic Materials Qualification Board:

> **"The EMQB considered the results of both tests and has affirmed an acceptable safety margin of ≥40°C over the typical IMX-101 processing temperature..."**
>
> **"The EMQB Explosives Subcommittee concurs that IMX-101 is safe to handle, process, and load in large production/loading environments"**

# Acknowledgements

- BAE Systems/HSAAP
  - Mr. Brian Alexander
  - Mr. Virgil Fung
  - Mr. Jim Owens
  - Mr. Curtis Teague
- RDECOM-ARDEC/PM-CAS
  - Mr. Charlie Patel
  - Mr. Crane Robinson
  - Dr. Kenneth Lee
- ARL
  - Dr. Brian Roos

# Questions?