



Solid Oxidizer Development

Naval Surface Warfare Center
Dahlgren Division
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Solution Decontamination Development

- Desirable Attributes:
 - Efficacy (broad spectrum or niche)
 - Compatibility with military materials
 - Non-hazardous to user or environment
 - User-friendly
 - Low logistical burden



Peracetic Acid (PAA)

Effective Active Ingredient

- Found to be an excellent decontaminant
 - 100% kill of *Bacillus* endospores in lab tests
 - Selective oxidation of HD to less toxic sulfoxide
 - Selective P-S bond cleavage in VX to avoid formation of toxic EA 2192
- Advantages over halogenated oxidizers:
 - Less corrosive
 - Lower toxicity
 - Less harmful to the environment



Peracetic Acid (PAA) Limitations

- Traditionally 15-30% in solution
 - Wasted weight and footprint on water
 - $\geq 43\%$ PAA forbidden for transport
- DOT Hazard Class 5.2 (Organic Peroxide)
 - Shipping and transportation limitations
 - Storage and handling requirements

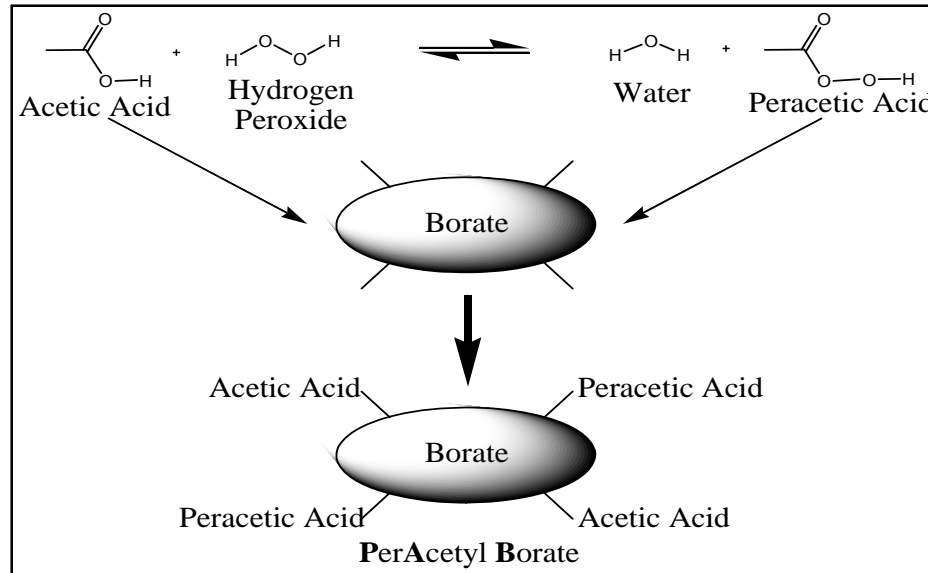
Logistical Challenge

Hauling Water

HAZMAT



Solid Oxidizer: PES-Solid



- Solid
- Stable on extended storage
- Good water solubility
- **Peracetic acid** released immediately upon addition to water

PES-Solid Challenges

- Officially classified as Class 5, Division 5.2 Organic Peroxide, Type F, UN3110
- Operationally relevant limitations not fully understood
 - Storage condition requirements
 - Shelf life
 - Solubility parameters
- Not available above bench scale production capacities



The Path Forward: PES-Solid Development

- Optimize PES-Solid for safety & affordability in storage, transportation, and use.
- Characterize PES-Solid with respect to operational feasibility and use in hazard mitigation and decontamination applications.



Reclassification Effort

- Solvay Chemicals has world-class facilities and substantial experience in required UN Orange Book test methods for organic peroxides (5.2)
- Data currently being gathered to apply for official reclassification to Type G
 - Type G requires a Self Accelerating Decomposition Temperature (SADT) of equal to or higher than 60°C
 - DSC shows 1 endotherm (150°C) and 1 exotherm (170°C)
 - Preliminary results on new batch shows SADT of 60°C

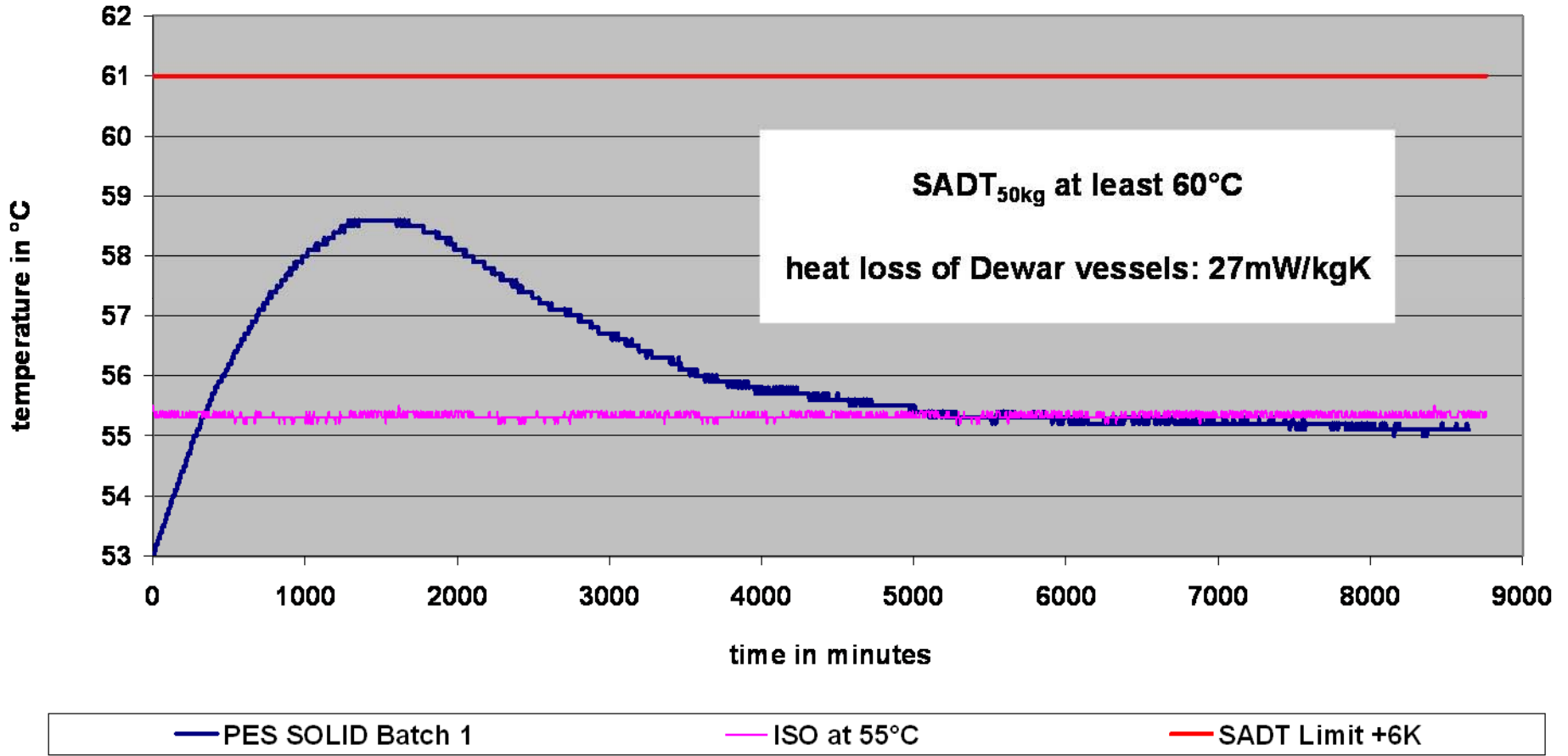
Type G organic peroxides exhibiting thermal stability are not subject to the requirements of Division 5.2.





Solvay Chemicals GmbH - Transport & Product Safety

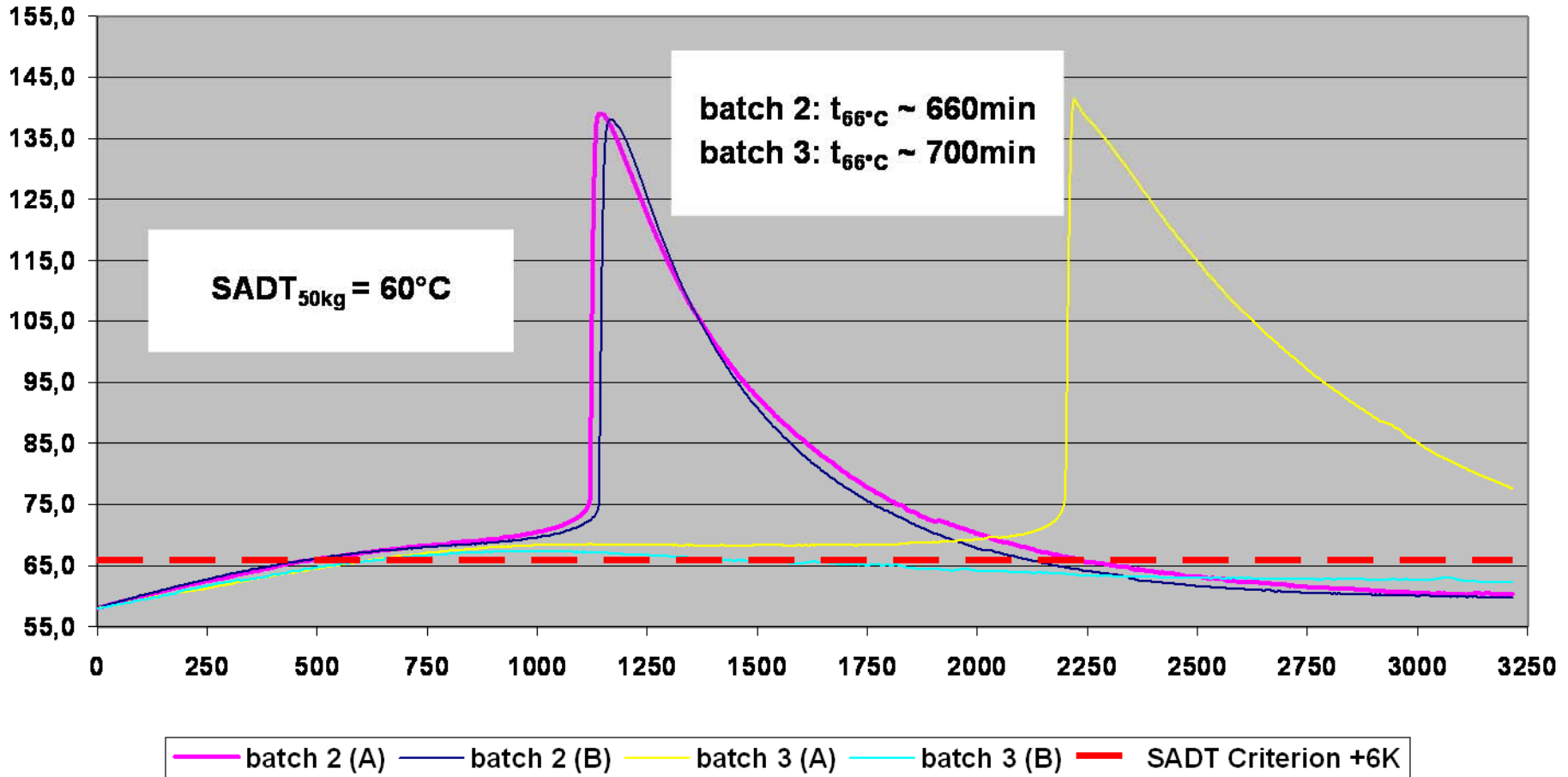
Heat Accumulation Storage Test UN H.4 - PES Solid Batch 1 - 2009/08/05





Solvay Chemicals GmbH - Transport & Product Safety

UN H.4 at 60°C - batch 2 and 3 from August / September 2009



Shelf Stability of Pure PES-Solid: Initial Solvay Results

- 4 weeks storage at 55°C (131°F)
 - 0.3g samples of 2009 material
 - Retained most of original PAA
 - Batch 1: Average 89% at 4 weeks
 - Mixture of Batch 2 & 3: Average 94% at 4 weeks
 - Variation in data
 - Likely due to small sample size
 - Currently testing with 50g samples

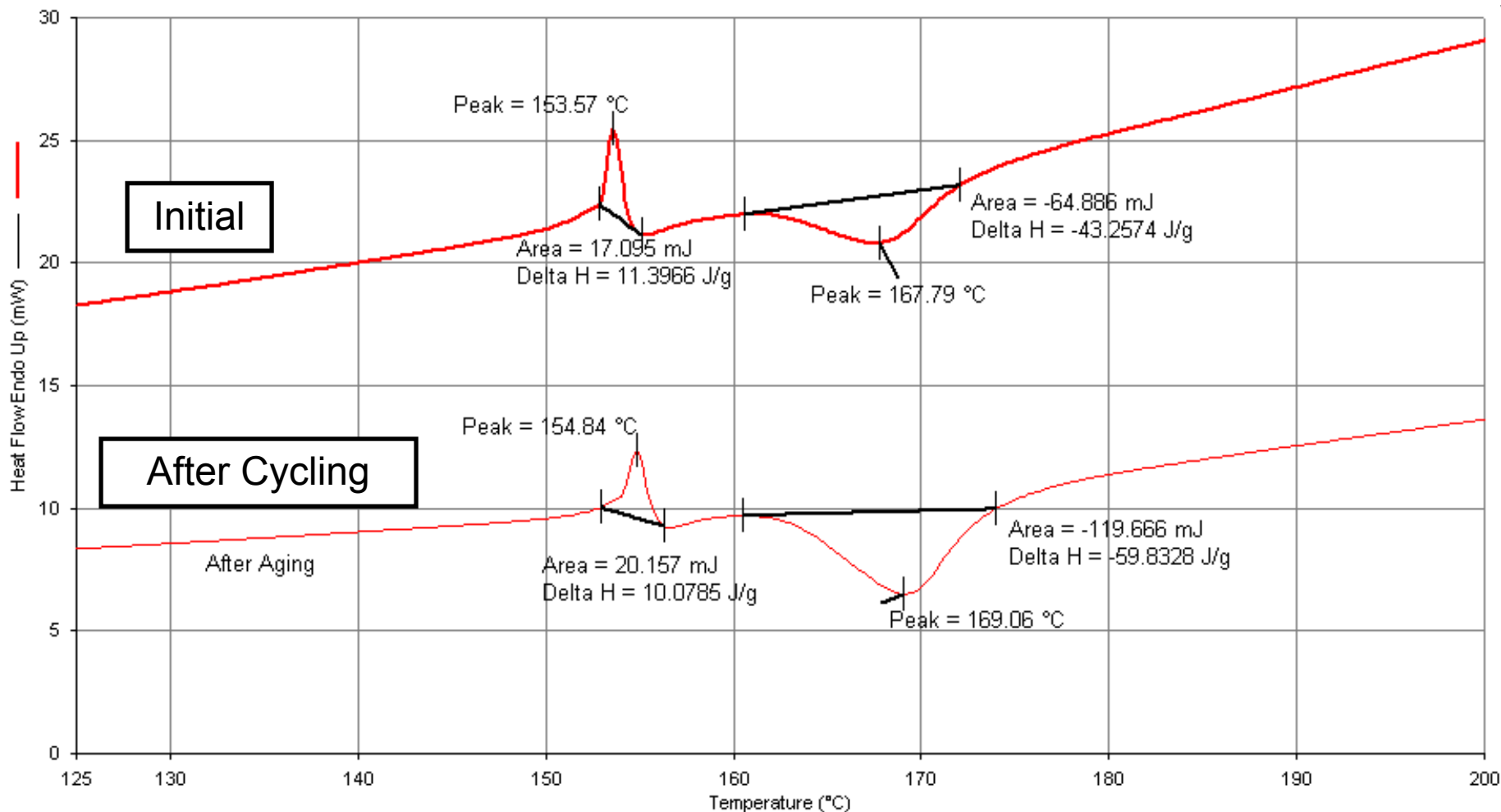


Shelf Stability of Pure PES-Solid: Initial NSWC Results

- 1½ weeks of temperature cycling
 - 10g samples of lab-scale material
 - 20% RH held constant
 - 4 hr at 50°C, 2 hr at 20°C, 15 min ramps
- Results
 - No loss of original PAA content outside of calculated error
 - Average sample mass loss of 2.6%
 - No significant shift in DSC peaks



DSC Data: Pure PES-Solid



Solubility Landscape Mapping

- Up to 200 g/L dissolved in < 5 minutes:
 - Deionized/Distilled laboratory water
 - Potomac River in Dahlgren, VA
 - Decon station of USS Winston Churchill in Norfolk, VA
- Final pH adjusted to 7 & 9 with 3M NaOH
 - Data obtained at 20°C
 - pH 7 from 20 – 200 g/L
 - pH 9 from 20 – 75 g/L



Solvay's Capacity Upgrade

- Solvay Chemicals holds the patent for the production of PES-Solid but initial lab-scale capacity was too low to support large scale tests
- New single pot batch reactor installed
 - 15 kg/batch current capacity
 - 200kg from new process will be provided in FY10
 - Multiple batches successfully completed & qualified

Current Pilot Facility Capacity: 2 ton/year using current single pot production process.



Decontaminant Efficacy Testing

- Simulant Testing in progress
 - Chemical Testing with simulant in vials
 - BSL2 Biological Testing with *B. anthracis* Δ Sterne on coupons
- CWA and BWA testing planned
 - HD, VX, and G-series
 - *B. anthracis* Ames on military surfaces
- Simulant/Agent correlation determination

Did PES-Solid process changes affect efficacy?



Future Efforts

- Scale-Up to determine relevant dissolution times
- Expand solubility data set
- Expand material compatibility data set
- Additional stability testing at Solvay & NSWCCD
- Official re-classification testing & application by Solvay Chemicals, Inc.



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- Decon Sciences group from ECBC

