A Safe and Effective Method To Remotely Mix Small Quantities of Energetic Compositions

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Acknowledgements

- Project team consisted of the following:
  - Investigators: Brad Cragun, Paul Braithwaite
  - Technicians: Dean Child, Colton Potter, Marc Hall
- Formulating, mixing, and testing all performed at ATK facilities in Promontory, Utah

Background & Introduction

- Hand mixes of small quantities of new energetic formulations have traditionally been made for safety screening as part of the scale-up process for new formulations.
  - Operators directly exposed to live materials
  - Mixing may be inadequate or inhomogeneous
  - Reproducibility varies from operator to operator – or within the same mix series.
- Remote mixing of small, safety screening sized mixes provide the following:
  - Improved safety
  - Thorough mixing
  - Reproducibility
  - Improved efficiency

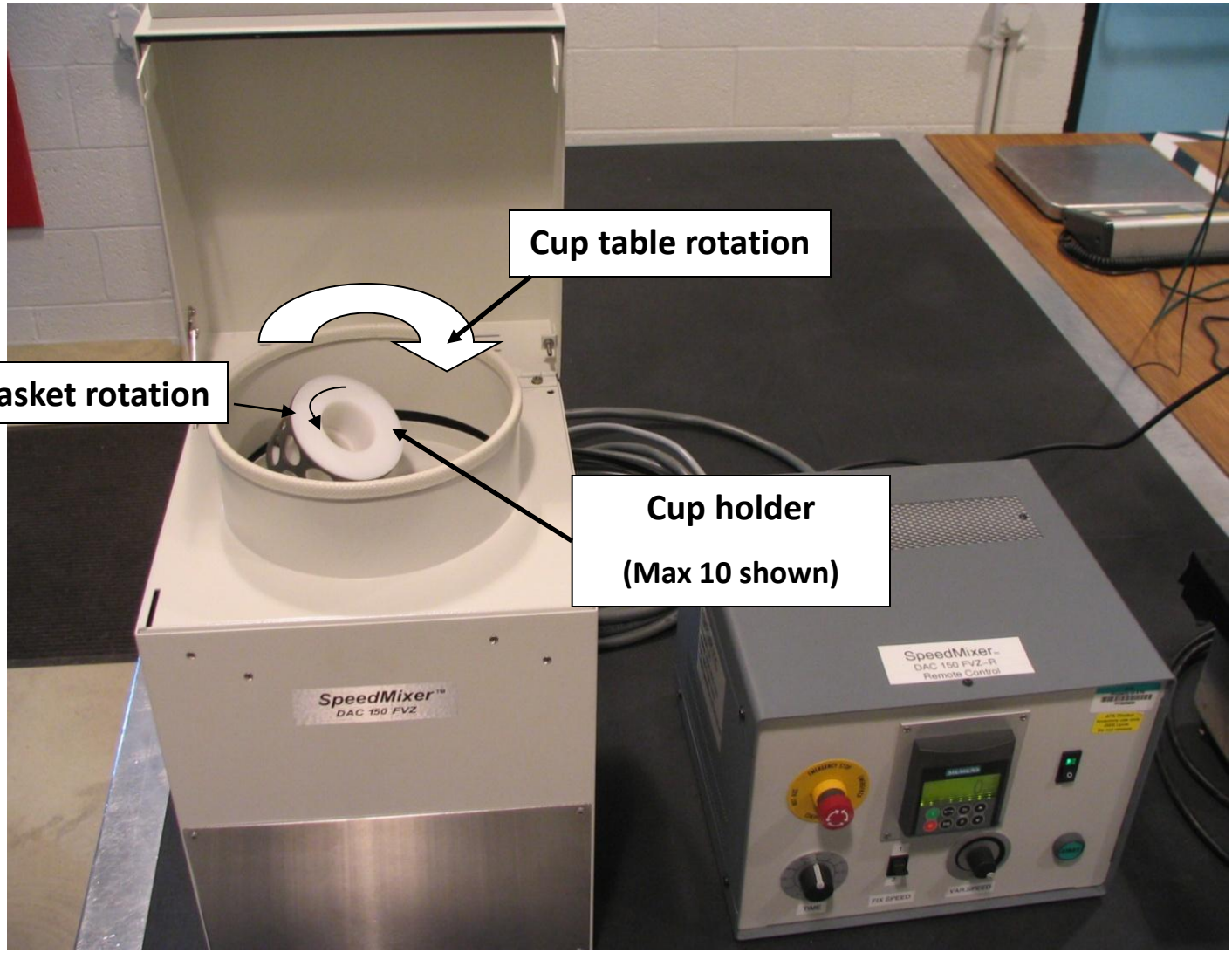
New Energetic Formulation Development

- Begins with individual and binary DSC compatibility testing.
- Transitions to small scale (~10 gram) mixes
  - Requires the evaluation of:
    - New ingredients
    - New particle sizes
    - Different combinations of ingredients
    - New methods of combining materials
    - Analogous mixing/processing methods intended for larger mixersBegins
- Early information gathered includes:
  - ➢ Processibility:
    - ✓ First look at binder-filler interactions
    - ✓ Ball park viscosity
  - ➢ Laboratory handling safety data:
    - ✓ Friction
    - ✓ Impact
    - ✓ ESD
    - ✓ Thermal Stability

Mixer Acquisition Background

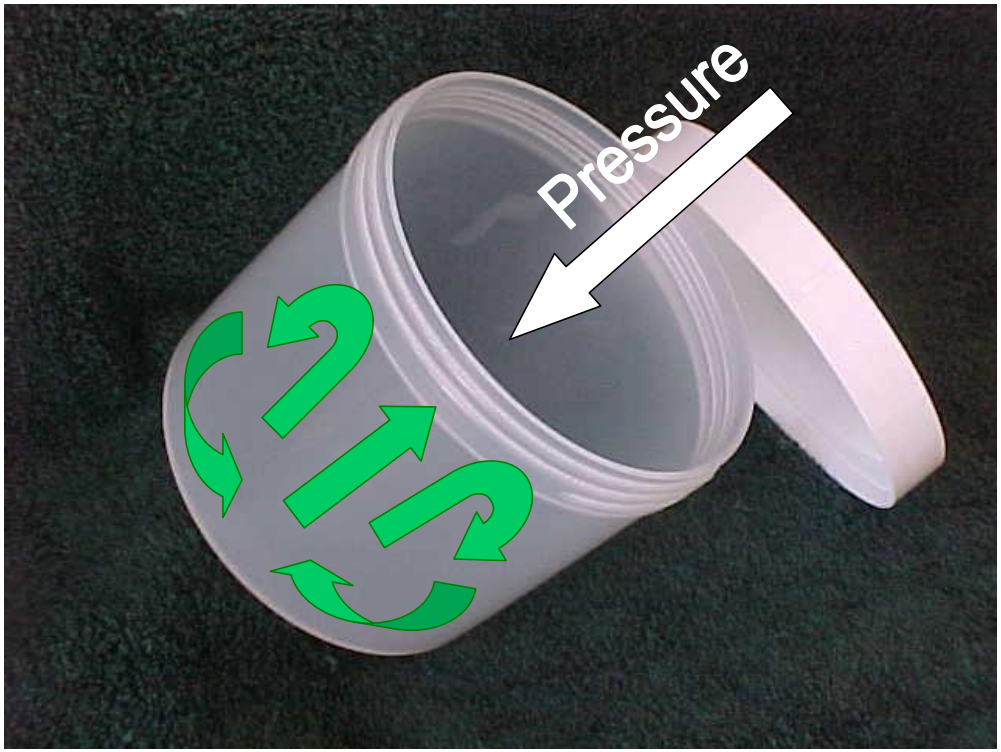
- Corporate safety audit suggested to look into alternatives to hand mixing.
- Previous small scale mixers did not provide consistent quality.
- Centrifugal mixer identified.
  - Advantages of:
    - No blades
    - Mixes made and delivered in the same cup
    - No (or very minimal) clean up
    - Ease of remote operation
    - Mixes follow same order of addition as likely scale up mixes
    - Minimal facility requirements
    - Minimal air entrainment in sample
    - Relatively easy to move (portable)
    - Low preventive and ongoing maintenance costs
    - 5 – 100 gram sample weight capability
    - Mixing does not generate an explosive atmosphere

Centrifugal Mixer Setup

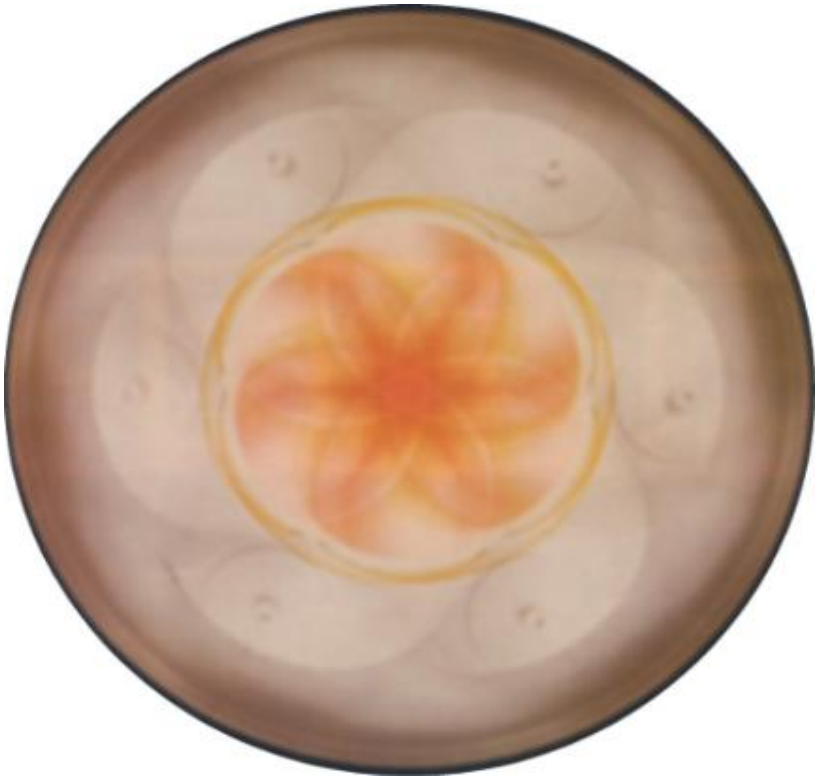


Mixing Motion Within the Cup

Vertical Motion

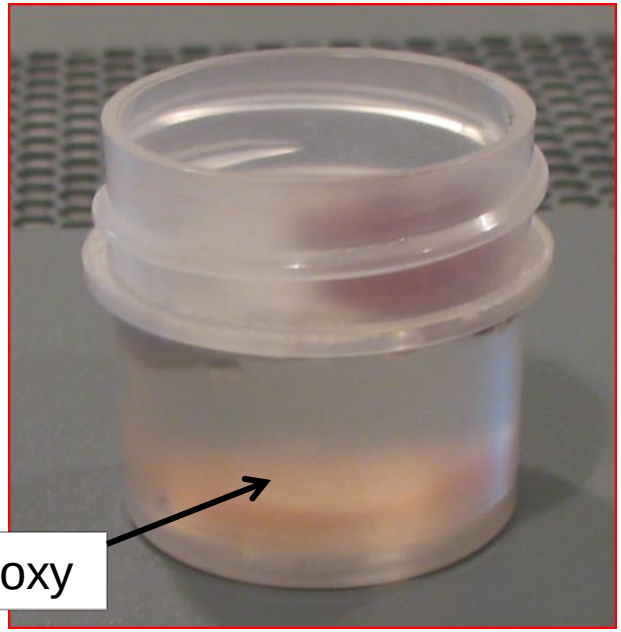


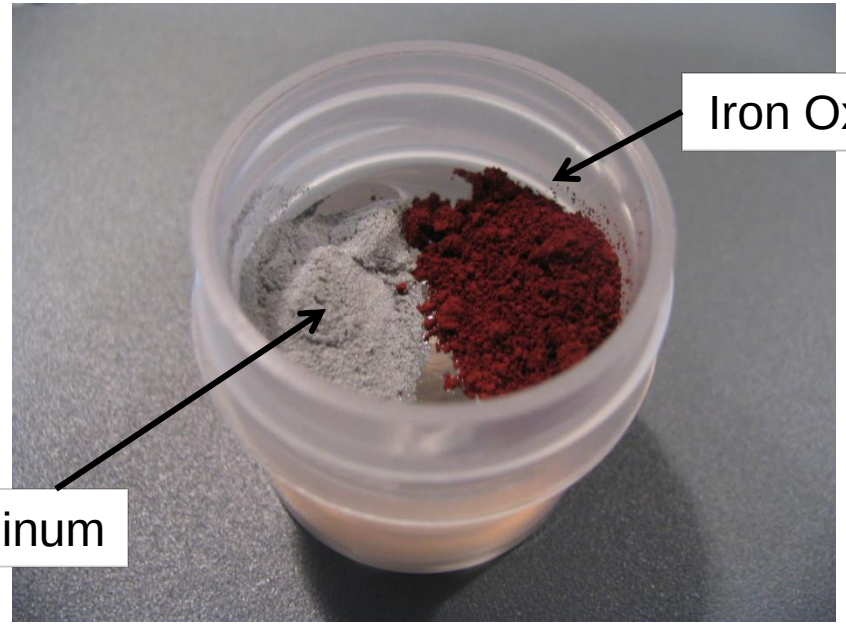
Horizontal Motion

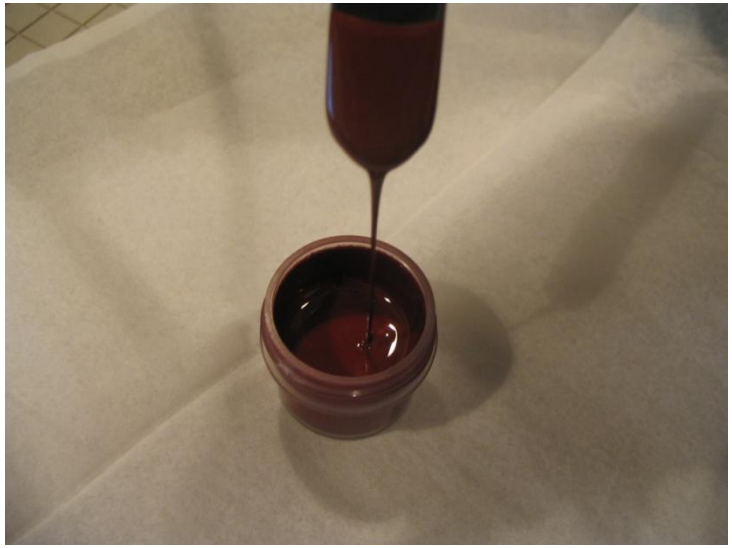


Flow motion is down the walls of the cup and up the middle

Mixing at the 10-Gram Cup Scale



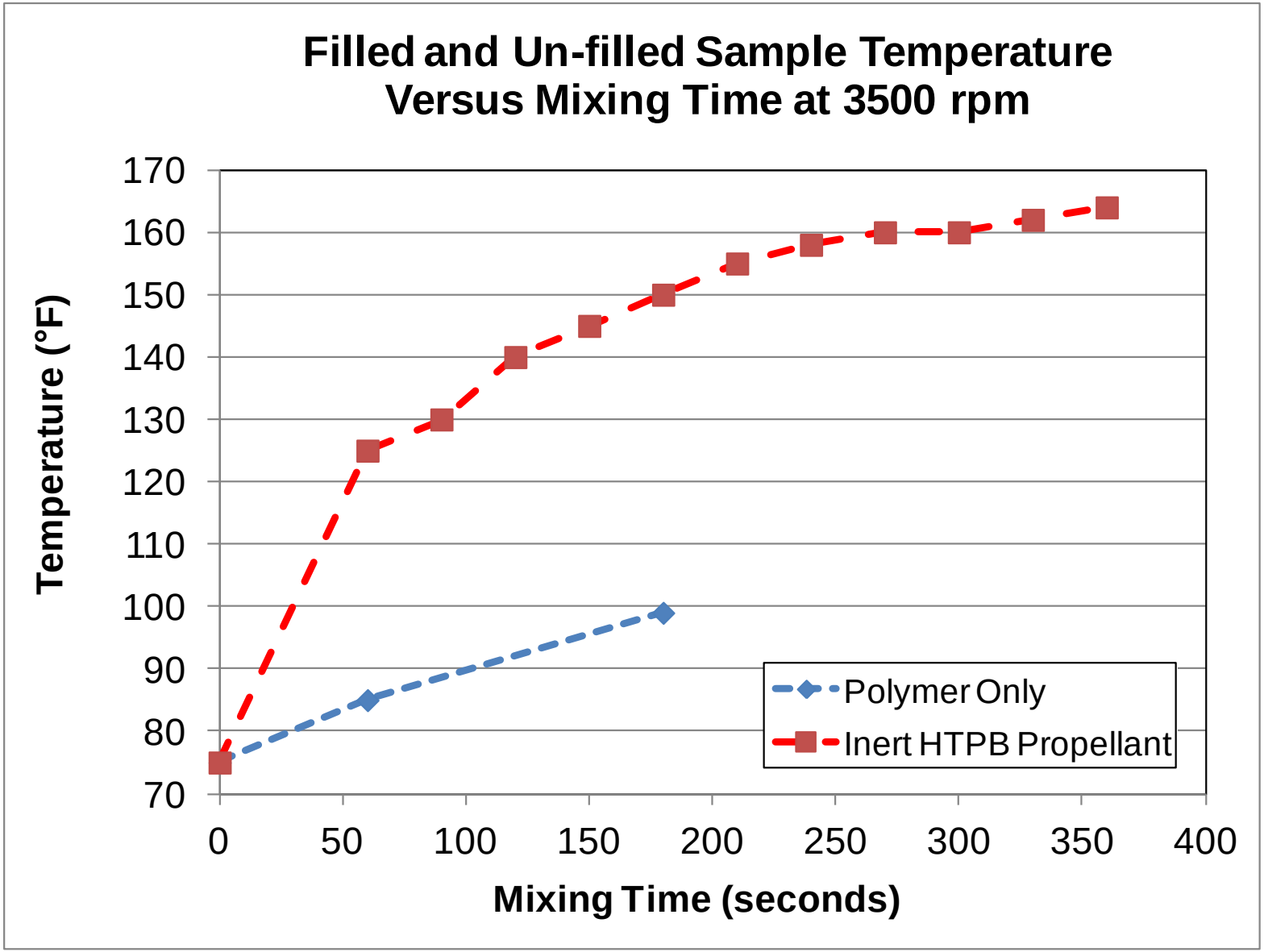




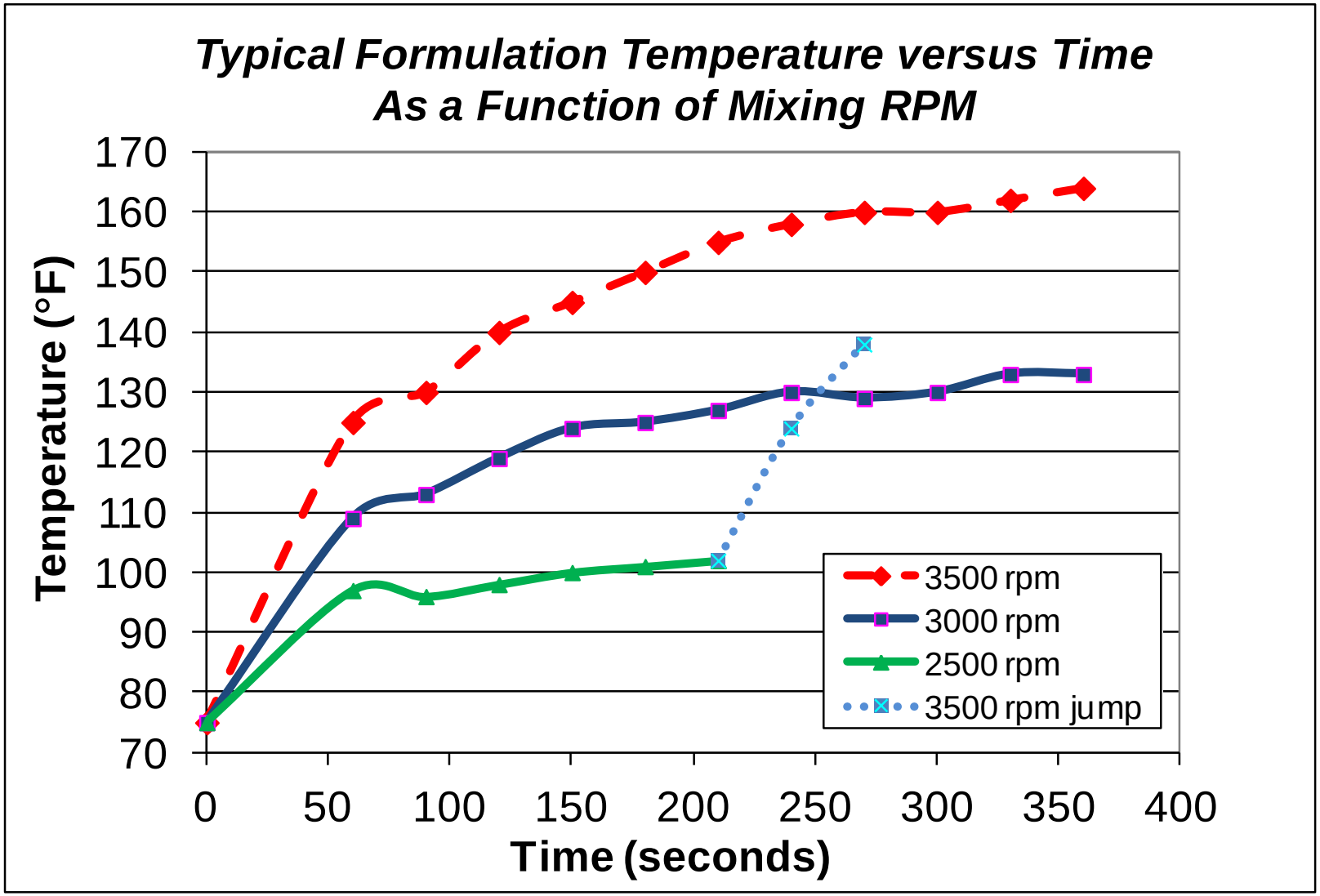
Considerations

- Ingredient densities
  - ✓Volumetric loading considerations
- Potential heat generation
  - ✓Bulk density
  - ✓Formulation detail
    - •Percent dry ingredients
    - •Percent solid ingredients
- Precision requirements
  - ✓Need adequate balances
    - •Associated with small batch size

Temperature Response in Filled and Un-filled Systems



Profiling a Formulation



Hand Mixing vs. Remote Mixer Processing

Metric	*Mixing Process*	
	Traditional Hand Mixing	**Remote Speed Mixer**
Capital equipment	**Advantage** - no capital required.	**Minor disadvantage** - small capital investment needed.
Process equipment	**Neutral** - low cost vials and spatulas. Laboratory hood (formulation dependent.)	**Neutral** - low cost vials and spatulas. Laboratory hood (formulation dependent.)
Personel protective equipment	**Neutral** - standard laboratory coats, protective eyeware and portable shields.	**Neutral** - standard laboratory coats and protective eyeware.
Operator exposure	**Slight disadvantage** - mixing process is attended and has higher exposure.	**Advantage** - mixing process is remote which minimizes operator exposure.
Mix quality and Reproducibility	**Disadvantage**: • No deaeration • Quality depends on skill of technician • Poorly coated solids are likely	**Advantage**: • Mixing naturally deaerates • Reproducible/not operator dependent • Vigorous mixing leads to well coated solids
Reliability of mix	**Disadvantage** - Non-homogeneous samples can produce erratic safety test results and misleading processing information.	**Advantage** - more homogeneous samples produce representative safety data and processing results.

Lessons Learned

Multiple Formulation Types Screened:

- Cast-cure
- Dry blend
- Pressed
- Melt-pour

Process works best with cast cure & dry blend

Frictional heating can be substantial for some compositions:

- Dry blends
- High density

❖ Heating effects can be minimized by mixing for short time periods with short delays between mixing periods.

Summary & Conclusions

- Remote processing of small safety screening sized samples of energetic materials have been demonstrated to be safe and efficient.
  - Homogeneity, reproducibility, and ergonomics are improved over hand mixes.
  - Applicable to a range of formulation types
  - Minimal clean-up
  - Relatively easy installation
- Evaluation of new mixing technology is an ongoing initiative which has produced substantial improvements