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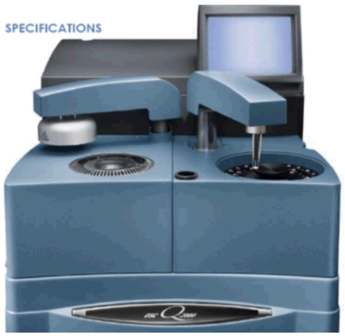

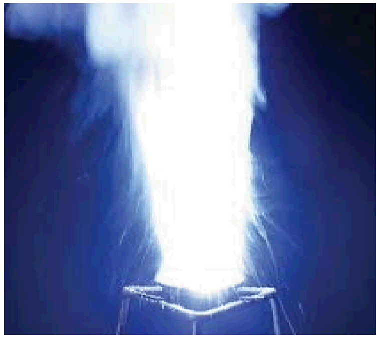

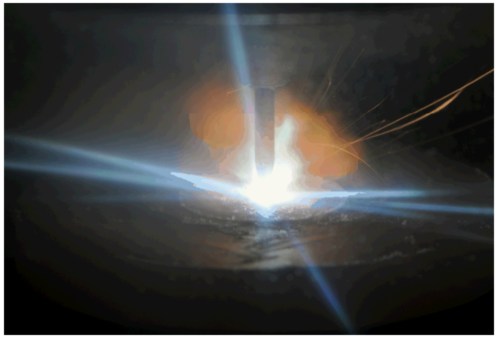

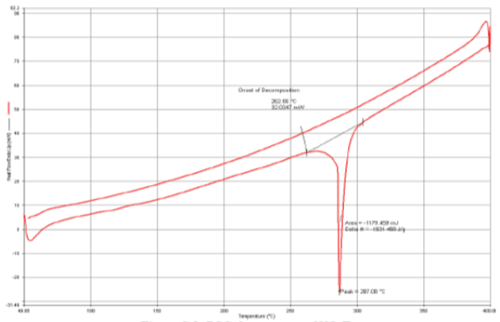

Figure C-1: DSC thermogram of NQ_2Tz

SNL Energetics Compatibility Capabilities

JOWOG presentation: 2/16/18

Colin Pollard

U.S. DEPARTMENT OF ENERGY

NNSA National Nuclear Security Administration

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Material Compatibility Testing

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- Methods derived from requirements in STANAG 4147, “Chemical compatibility of ammunition components with explosives”
  - DSC
  - Dynamic TGA
  - Isothermal TGA
  - Pinch-off Tubes (POT) tests
- Other Methods
  - Access to ARC systems (not typically used)
  - Investigating obtaining a Simulated Bulk Auto-Ignition Test (SBAT) instrument
- The following information is taken from the Design Guide

General Approach

Sandia National Laboratories

Processing compatibility and long term compatibility

DSC	1-2 mg	Relatively inexpensive

Long term powder storage stability

TGA	1-2 mg	Relatively inexpensive

Techniques used for material down-selection

Long term compatibility

POT & other follow-on analyses	25 mg energetic 25 mg inert	moderate expensive

materials with high probability of going into a component

Component accelerated aging

numerous analyses	Full components	Expensive, time intensive

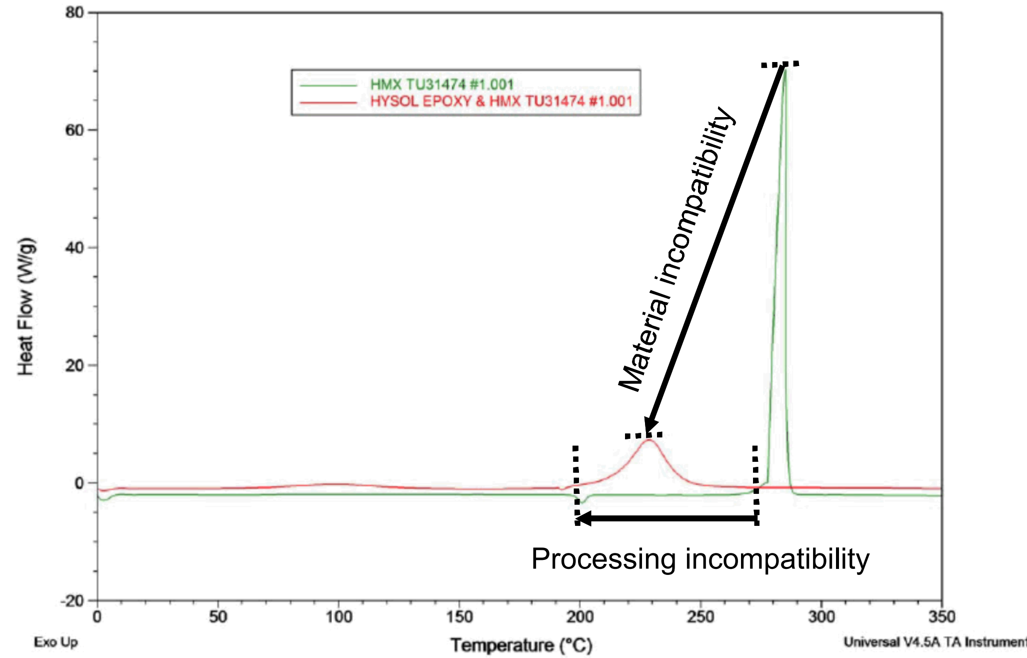
materials as near to final configuration as possible

DSC Testing

Processing compatibility and long term compatibility		
DSC	1-2 mg	Relatively inexpensive

- TA Q2000 systems (two) and one Q200
- Configuration
  - Pinhole lid or solid; solid hermetic seal is preferred
  - 10°C/min from ambient T to 300°C

HMX and Amine: Massive change in output and onset



<u>Shifts in Onset</u>

<Δ4°C = compatible

>Δ20°C = incompatible

Δ4°C to 20°C = requires follow-on POTs

Changes in peak shapes can also indicate incompatibilities

TGA: Dynamic and Isothermal

Long term powder storage stability		
TGA	1-2 mg	Relatively inexpensive

- TA Q5000 series TGA system
- Configuration
  - Test should be (not always) conducted under flow of nitrogen at 50cc/min unless material can form nitride(s)

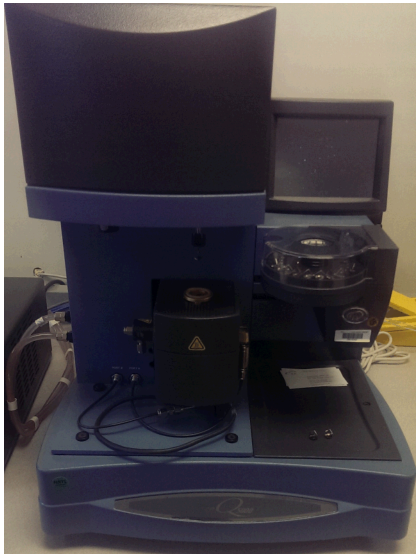
Dynamic (standard)

- 10°C/min

Isothermal

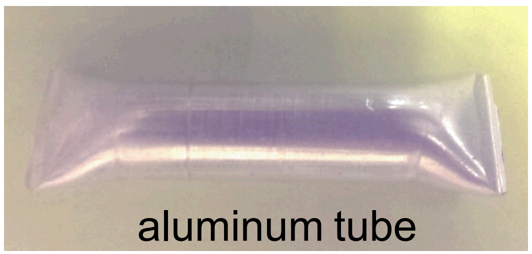
- Test time should be 1000 min
- Test temperature is that of the first exothermic peak in DSC data or 20°C below the weight loss derivative peak found through dynamic TGA analysis

<u>Increase in Weight Loss</u>
<Δ4% = compatible
>Δ20% = incompatible
Δ4°C to 20% = requires follow-on POTs



Pinch-Off Tube Tests:

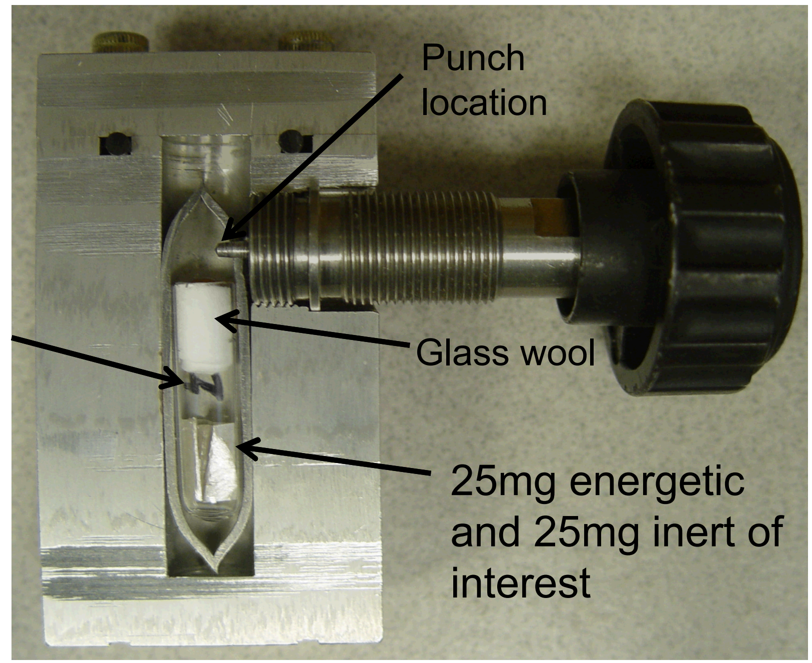
Purpose: to test each and every material in direct contact with the energetic material



hermetically sealed using a special tool

Cutaway of punch rig with POT

borosilicate thimble



Age 30 days at 70°C → Punch tubes to collect gas → Gas analysis via GC/MS → Quantify N_2, N_2O, NO, NO_2, CO, and CO_2

5x outgassing volume warrants concern

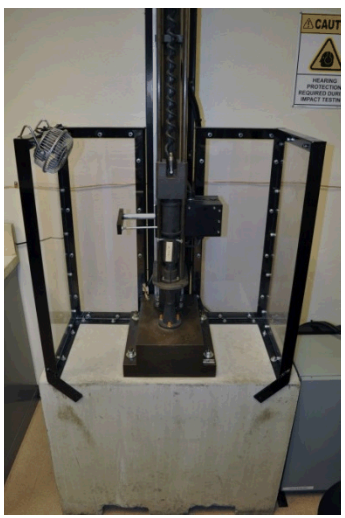
SNL method was adapted and scaled down from STANAG 4147 CRT

SNL POT Method	STANAG 4147 CRT
• 25 mg energetic material 25 mg inert	• 5g of sample material
• 70°C for 30 days	• 100°C for 40 hours or • 70°C for 24 days
• 50μL of decomposition gas warrants concern (scaled from STANAG) • 5x outgassing volume decomposition gas indicates incompatibility	• 5mL of decomposition gas indicates incompatibility

In general, the POT test is more conservative than STANAG

Small Scale Sensitivity Testing (SSST)

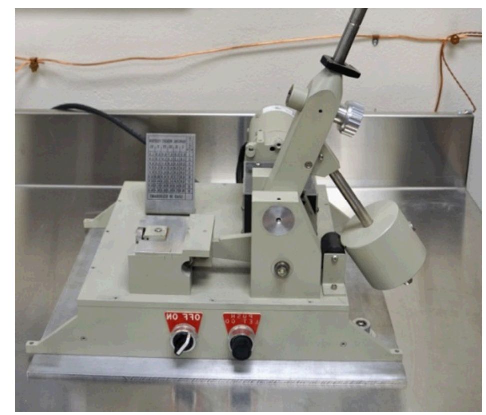
- Assesses an energetic materials response to common stimuli:
  - Impact
  - Electrostatic discharge (ESD)
  - Thermal (DSC)
  - Friction
- Provides safety testing of new or modified energetic materials.
- Used to understand changes in safety characteristics of aged energetic materials.



Drop-hammer Impact test (MBOM)

ESD

Thermal



Friction

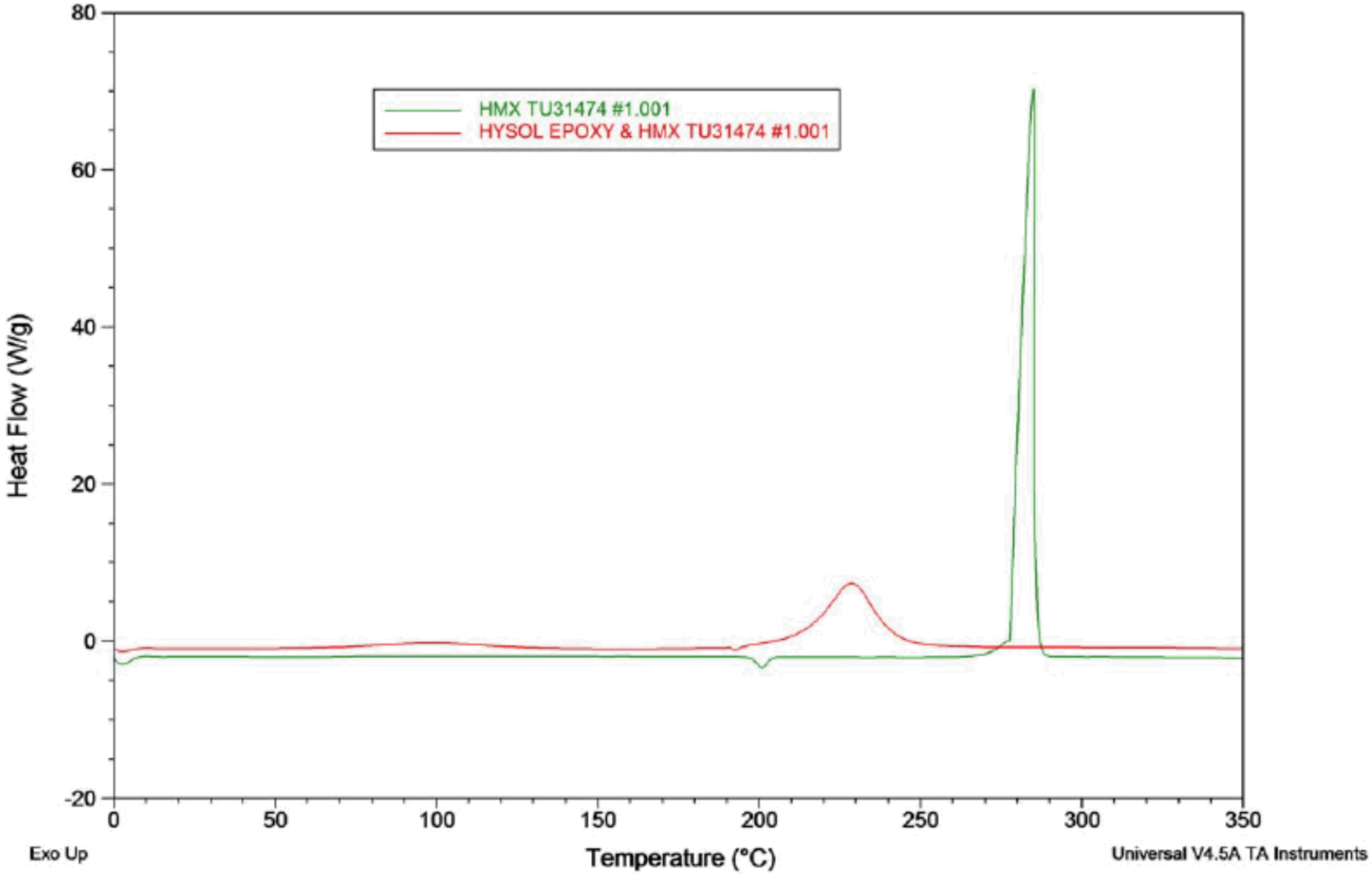
Future Direction: Simulated Bulk Auto-Ignition Test (SBAT)

- In future, we are looking to stand-up an SBAT as well
  - Can be equipped with pressure transducers to measure gas output
  - Bridges the gap between 1mg quantities and bulk loads

Questions?

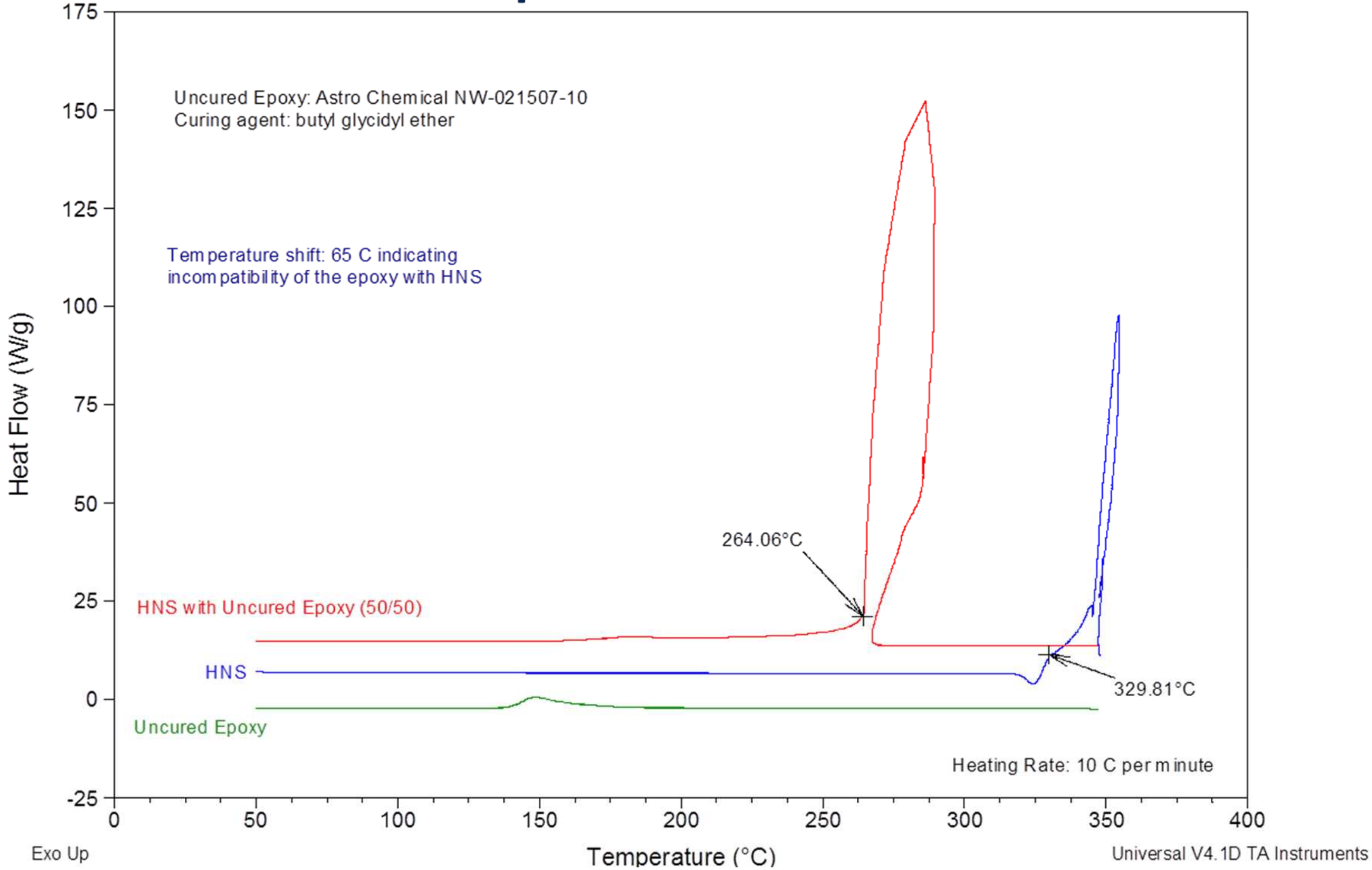
Data Samples: DSC, TGA, and POT

DSC Data



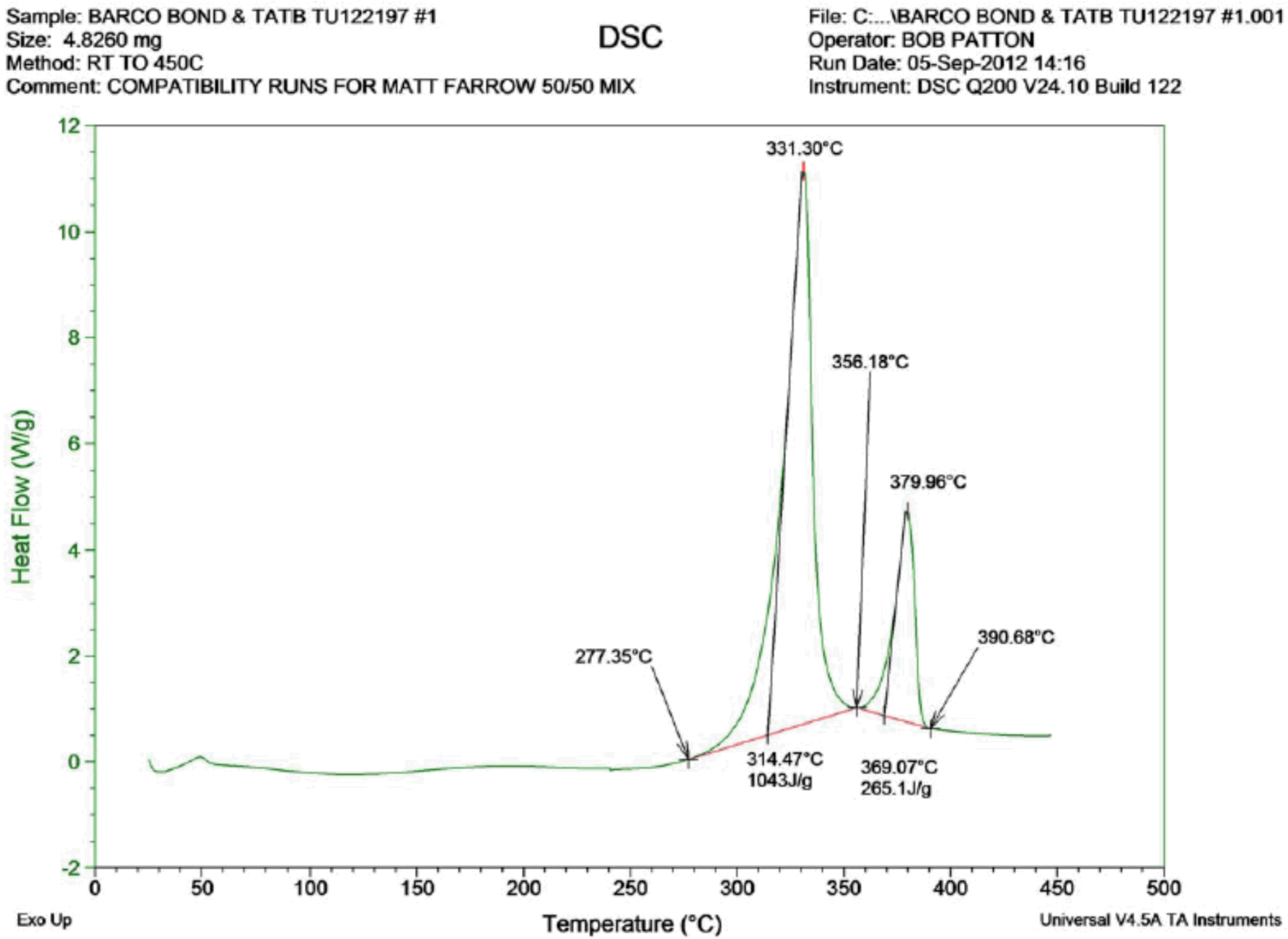
- HMX and Amine example: Massive change in output and onset

Another example



- HNS and uncured epoxy: Sample data

TATB vs Epoxy:



Analysis Technique Issues/Concerns

DSC/TGA Issues?

- Small sample sizes: how well does it correlate with larger masses?
  - What about pellets? Could an epoxy permeate a consolidated sample enough to cause significant output or safety issues before it cures or reacts completely?
- Peak T vs. Onset T?
  - Peak T is typically fairly consistent while onset seems to be more operator dependent… but what about instrument drift?

POT Test: Pitfalls?

- Gas analysis: how reactive are the liberated species?
  - Samples are physically sealed in an inert tube but the gases do get out. What about side reactions with the metal?
  - Over a month or a year, what's really happening at the metal surface?
    - Should we try SEM/EDS and look?
  - How complete is the gas sampling?
    - A vacuum manifold and cold trap has been the standard for a long time, but there has not been a flow-through of gas to ensure all the product gases reach the analyzer
    - There is a new system undergoing validation for that reason now.
- How hermetic are the seals?
  - First question that comes up commonly when the test is described.
  - Multiple tubes are prepped; losses are very rare and easily seen in the data