

Analysis Techniques

Thermal, IR, Surface Area, and Particle Size

February 19, 2007

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Principal Technologist**



Thermal Analysis Technique

Differential Scanning Calorimetry (DSC)

**Uses: Melting & Boiling Temperatures
Specific Heat & Heat Capacity
Crystallization Time & Temperature
As well as %Crystallinity
Heat of Reaction & Fusion**

**Four DSCs Q1000, 2910 LNCA,
2910 RCS, and 2910**

Q1000 DSC

- **Temperature Range: -90C to 550C, typical to 400C**
- **Uses Inert Purge Gas**
- **Uses Refrigerated Cooling**
- **Auto Sampler for pans**
- **Sensitivity 0.2 micro watts**



2910 DSC LNCA

- **Temperature Range: -150C to 500C**
- **Uses Liquid Nitrogen Cooling**
- **Uses Inert Purge Gas**
- **Auto Sampler for pans**
- **Sensitivity 2.0 micro watts**



2910 DSC RCS

- Temperature Range: -90C to 400C
- Uses Refrigerated Cooling
- Uses Inert Purge Gas
- Auto Sampler for pans
- Sensitivity 2.0 micro watts



2910 DSC

- **Temperature Range: Room Temperature to 600C**
- **Uses No Cooling**
- **Uses Inert Purge Gas**
- **Manual Sample Loading**
- **Sensitivity 2.0 micro watts**

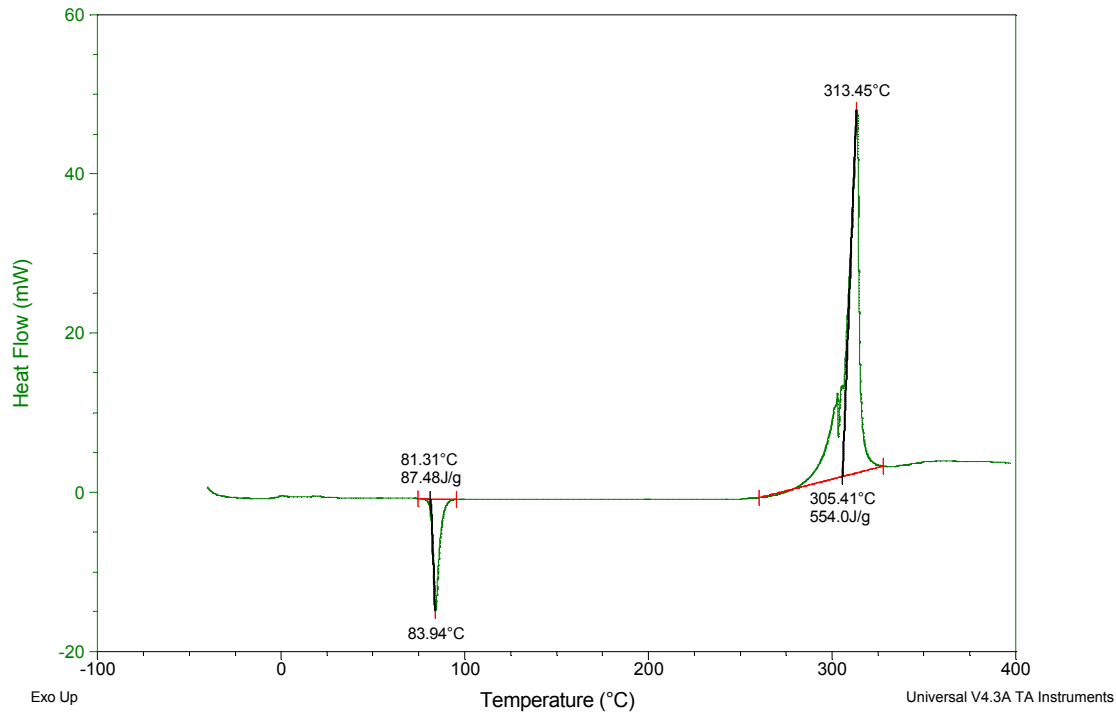


DSC Plot of TNT

Sample: TNT
Size: 4.0020 mg
Method: 0 TO 400 10C/MIN
Comment: Max volume

DSC

File: C:\DSC\2910\Rachel\De-Mil\TNT.001
Operator: Rachel Carlson
Run Date: 15-Apr-2005 08:40
Instrument: 2910 DSC V4.4E





Q1000 and 2910 Cells





Thermal Analysis Technique

Thermo Mechanical Analysis (TMA)

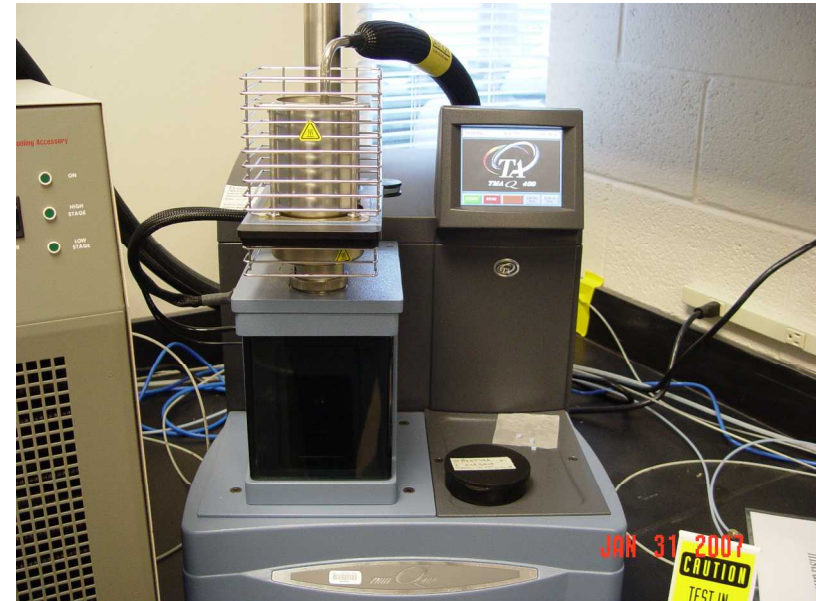
Uses:

- Measure Softening Point**
- Measure Glass Transition**
- Measure Expansion Coefficient**



Q400 TMA

- **Temperature Range: -70C to 400C**
- **No purge Gas Used**
- **Uses Refrigerated Cooling**
- **Uses Interchangeable Probes**
- **Sensitivity:
15 Nanometers for Length
and 0.0001 Newton for
Force**





Q400 TMA

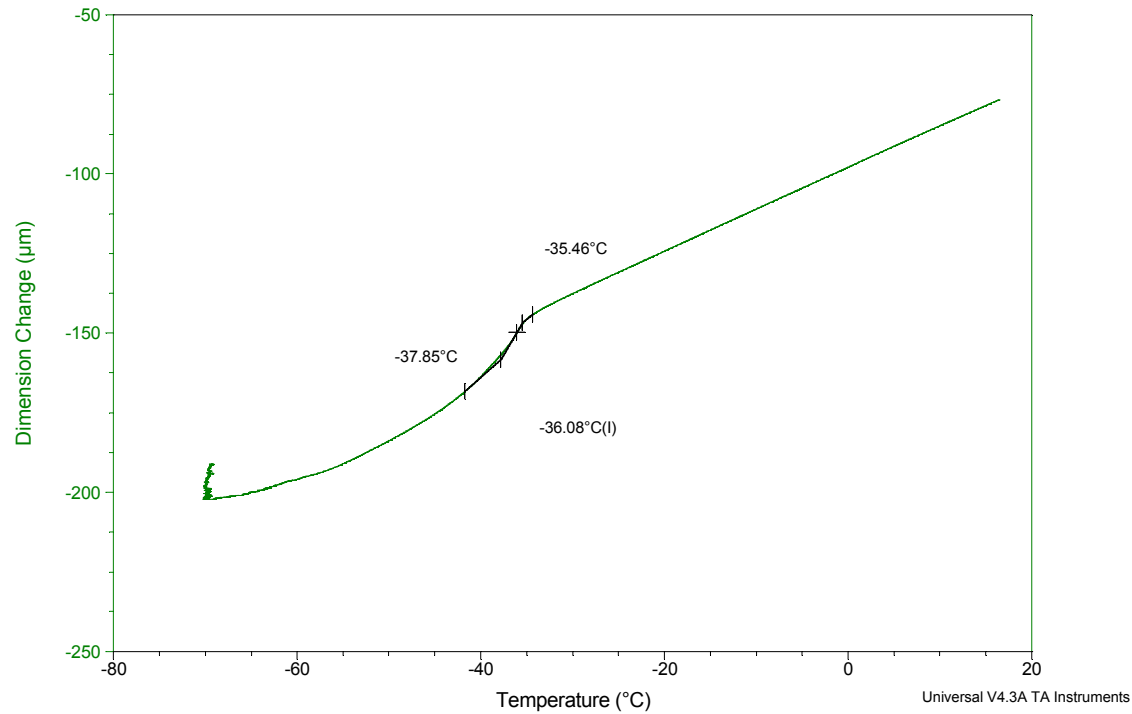


TMA Plot of RTV

Sample: RTV 630 Bulk run 2
Size: 4.4599 mm
Method: -70 to 20 at 2C/min

TMA

File: C:\...Erin\RTV630\RTV630Bulk9-6-06.001
Operator: Erin Shrouf
Run Date: 06-Sep-2006 14:22
Instrument: TMA Q400 V7.0 Build 80





Thermal Analysis Technique

Simultaneous Differential Thermal Analysis and Thermogravimetric Analysis with DSC Capability (SDT)

Uses: Same as DSC

Weight Losses or Gains

**Temperature Difference Between
Sample and Reference**

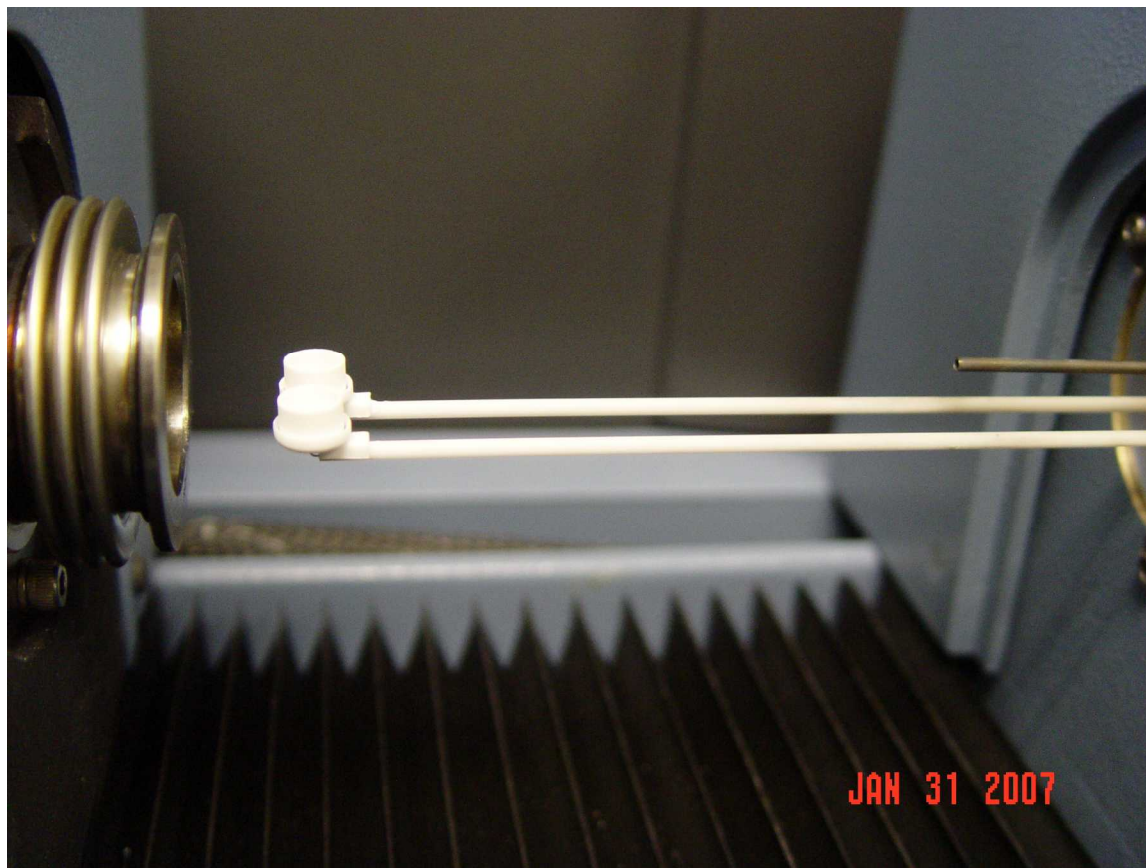
Q600 SDT

- **Temperature Range: Room Temperature to 1500C**
- **Inert or Oxidizing Purge Gas**
- **Uses No Cooling**
- **Uses Platinum or Alumina Cups 40 or 90 Micro Liter**
- **Sensitivity: 0.1 Micro Grams for Weight, 1 Micro Watt for DSC, and 0.001C Temperature Difference for DTA**





Q600 SDT

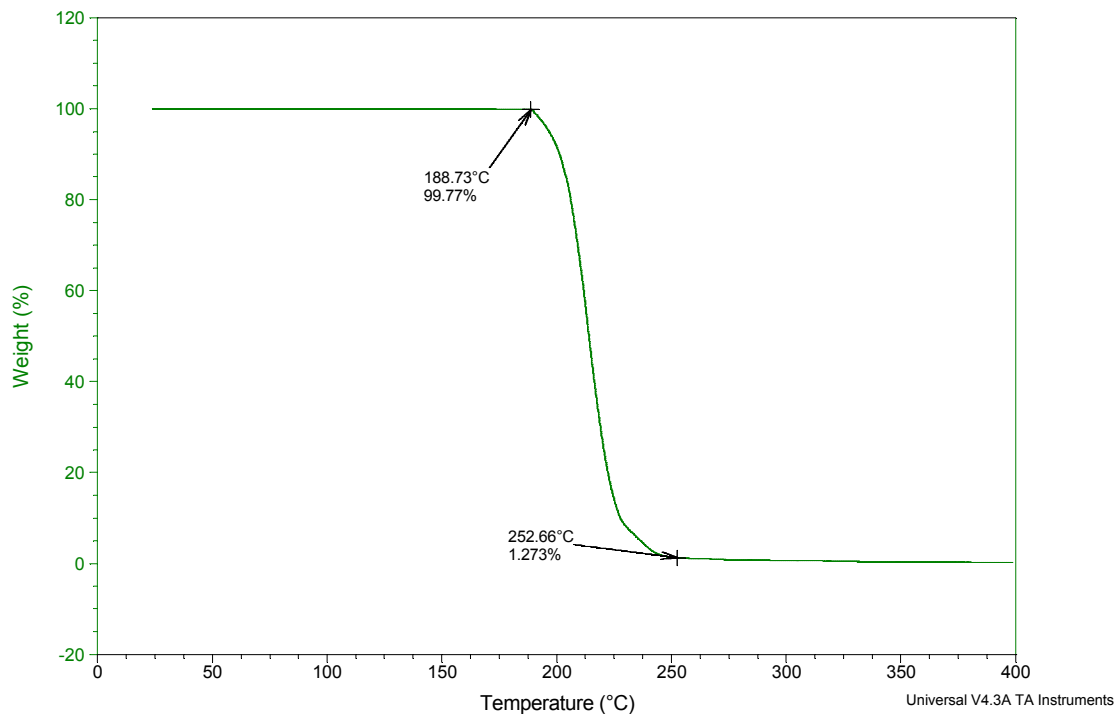


SDT Plot of RDX (TGA) 1C/min

Sample: RDX run 2 at 1C/min
Size: 5.4910 mg
Method: RT to 400C at 1C min
Comment: RDX TU 65138

DSC-TGA

File: C:\...\RDX_1C_min_SDT.002
Operator: Rachel Carlson
Run Date: 20-Feb-2006 09:11
Instrument: SDT Q600 V8.0 Build 95

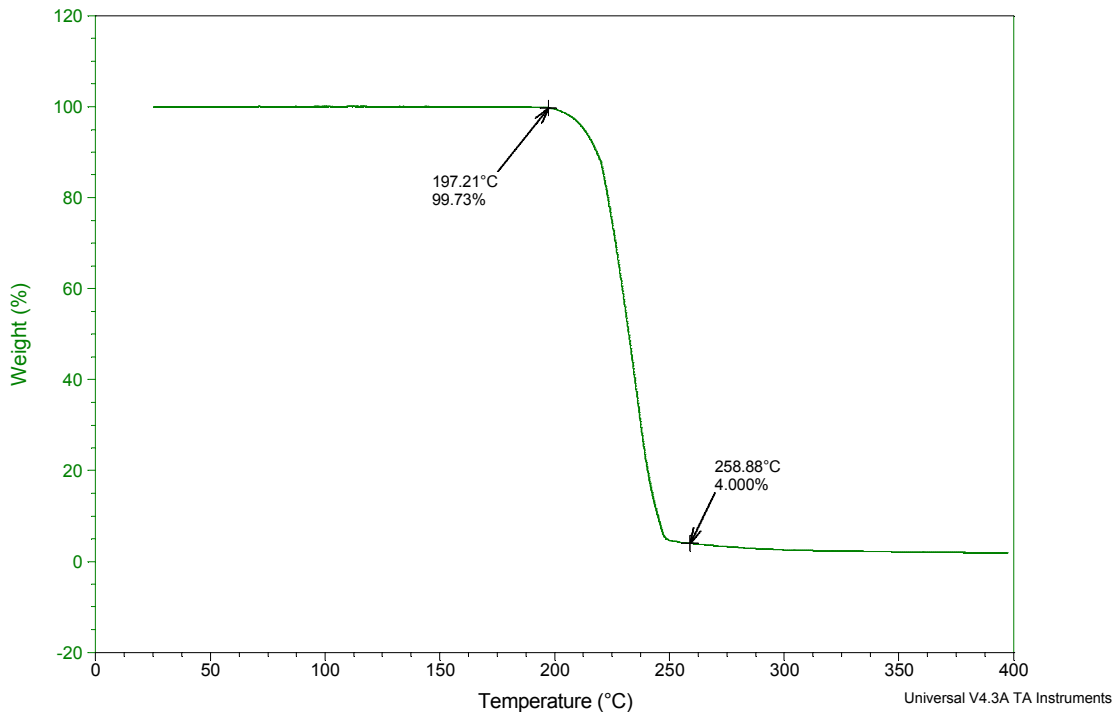


SDT Plot of RDX (TGA) 5C/min

Sample: RDX run 2
Size: 5.0290 mg
Method: RT to 400C at 5C min
Comment: RDX TU 65138

DSC-TGA

File: C:\...\RDX_5C_min_SDT.002
Operator: Rachel Carlson
Run Date: 16-Feb-2006 11:23
Instrument: SDT Q600 V8.0 Build 95

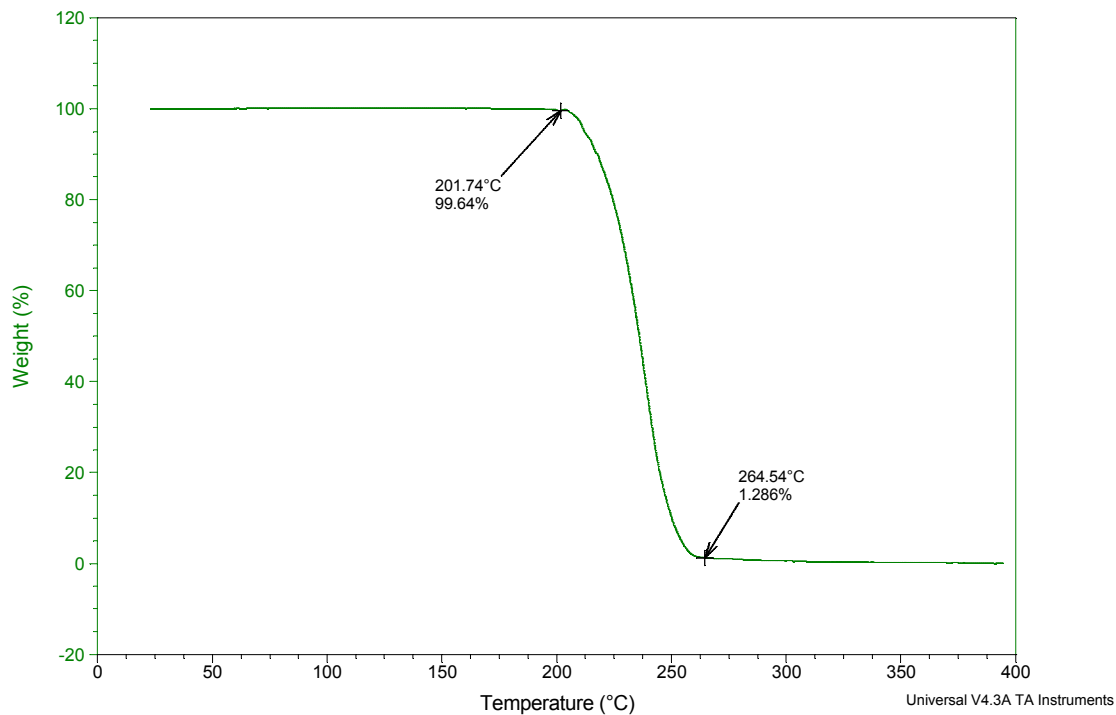


SDT Plot of RDX (TGA) 10C/min

Sample: RDX run 2 at 10 C/min
Size: 5.1040 mg
Method: RT TO 400C 10C MIN
Comment: RDX TU 65138

DSC-TGA

File: C:\...\RDX_10C_min_SDT.002
Operator: Rachel Carlson
Run Date: 01-Mar-2006 16:07
Instrument: SDT Q600 V8.0 Build 95

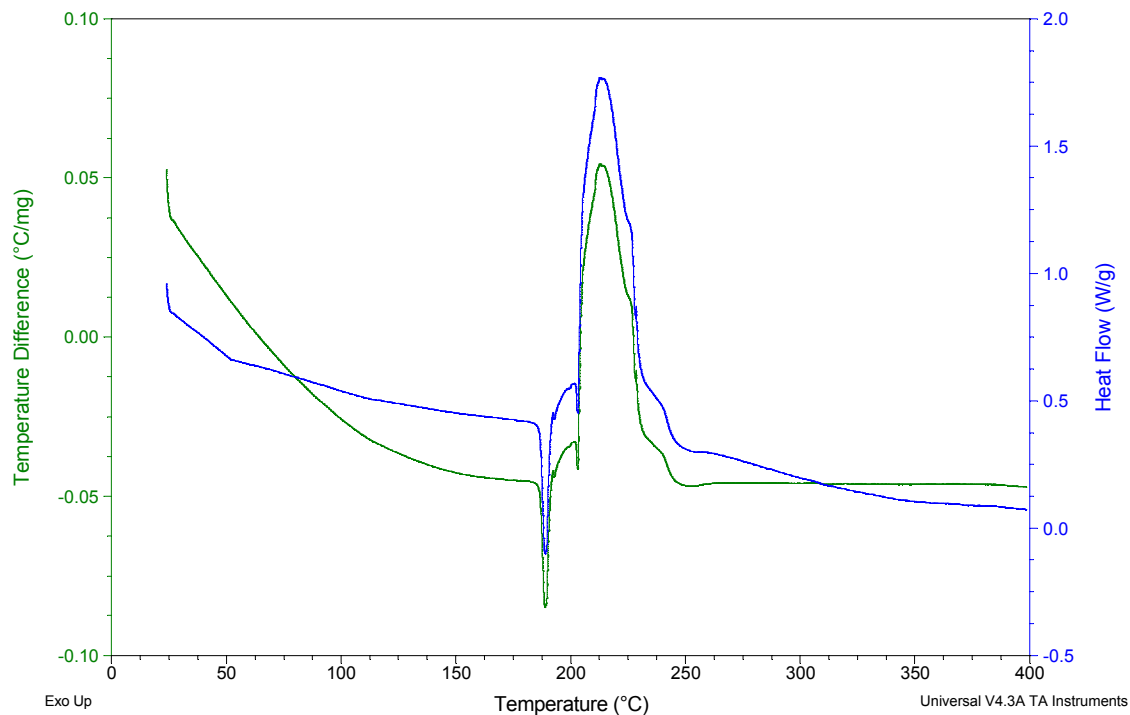


SDT Plot of RDX (DTA & DSC) 1C/min

Sample: RDX run 2 at 1C/min
Size: 5.4910 mg
Method: RT to 400C at 1C min
Comment: RDX TU 65138

DSC-TGA

File: C:\...\RDX_1C_min_SDT.002
Operator: Rachel Carlson
Run Date: 20-Feb-2006 09:11
Instrument: SDT Q600 V8.0 Build 95





Thermal Analysis Technique

Thermo Gravimetric Analysis (TGA)

**Uses: Weight Losses or Gains Due to
Decomposition or Oxidation
Can Also be linked to FTIR with
Evolved Gas Furnace and Flow Tube**

2950 TGA

- **Temperature Range: Room Temperature to 1000C**
- **Auto Sampler for Cups**
- **Inert or Oxidizing Purge Gas**
- **Uses No Cooling**
- **Uses Platinum 50 or 100 Micro Liter Cups, Alumina 100, 250, or 500 Micro Liter Cups**
- **Sensitivity: 0.1 Microgram in 1 to 100 Milligram Range and 1 Microgram in 100 to 1000 Microgram Range**

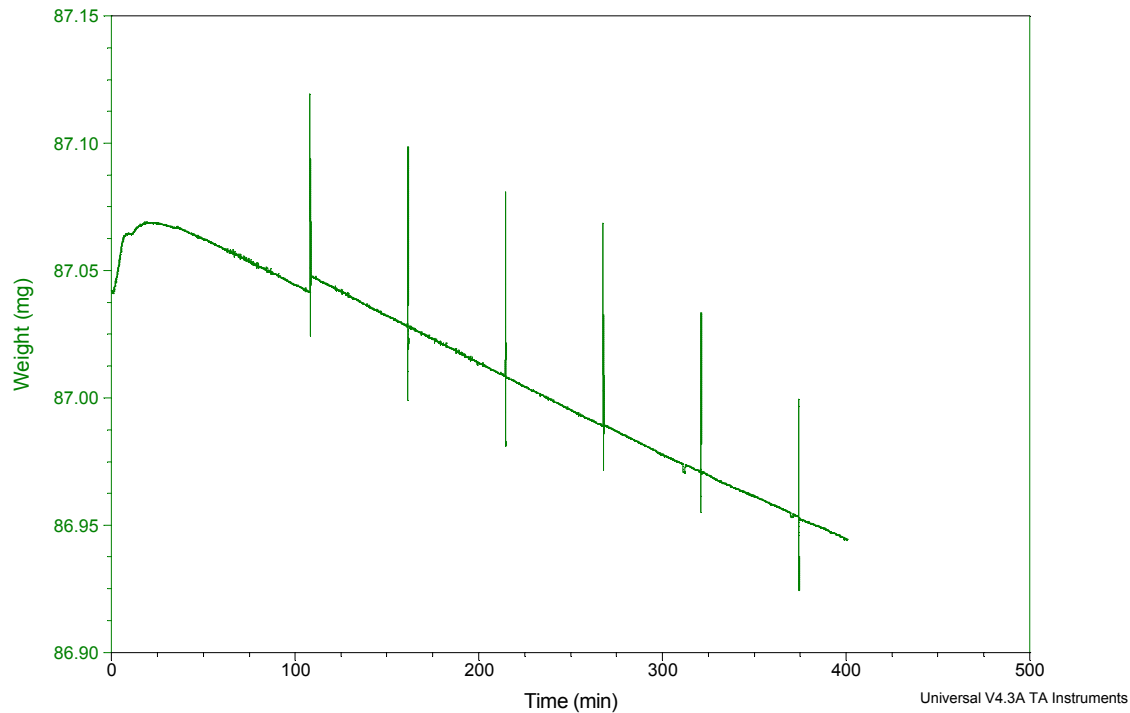


TGA Plot of C4 at 85C

Sample: C4 @ 85C PT PAN #1
Size: 87.0420 mg
Method: C4 EVAP AT 85C
Comment: RUNS FOR JIM PHELAN

TGA

File: C4 85C EVOLVED GAS HEATED OUTLET PT
Operator: BOB PATTON
Run Date: 23-Jun-2005 10:19
Instrument: AutoTGA 2950HR V6.1A





Thermal Analysis Technique

Dynamic Mechanical Analysis (DMA)

Uses: Gives Storage & Loss Modulus

Gives Storage & Loss Compliance

**Gives the Stress and Strain on
Sample Plus Force Applied to Sample**

**Gives the Dimension Change of Sample
with the Stress, Strain, and Force
Applied to Sample**

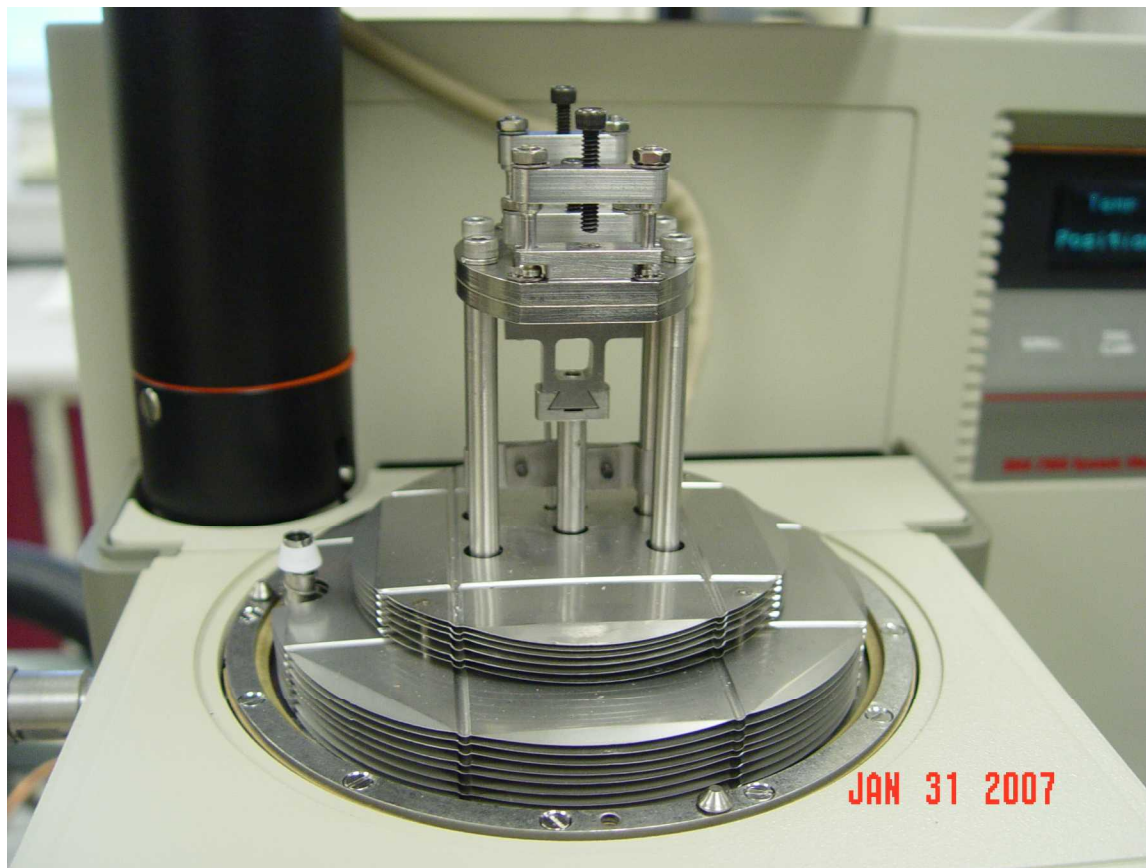
2980 DMA

- **Temperature Range: -145C to 600C**
- **Uses Refrigerated Cooling**
- **Uses Air or Nitrogen Purge**
- **Uses Tensioning Clamps:**
3 Point Bend 5, 10, 15, 20,
& 50 Millimeter Lengths,
Shear Sandwich, Film &
Fiber, and Compression 15
& 40 Millimeter
- **Uses Non Tensioning**
Clamps: Single and Dual
Cantilever 8 & 16, 15 & 30,
and 20 & 40 Millimeter





2980 DMA

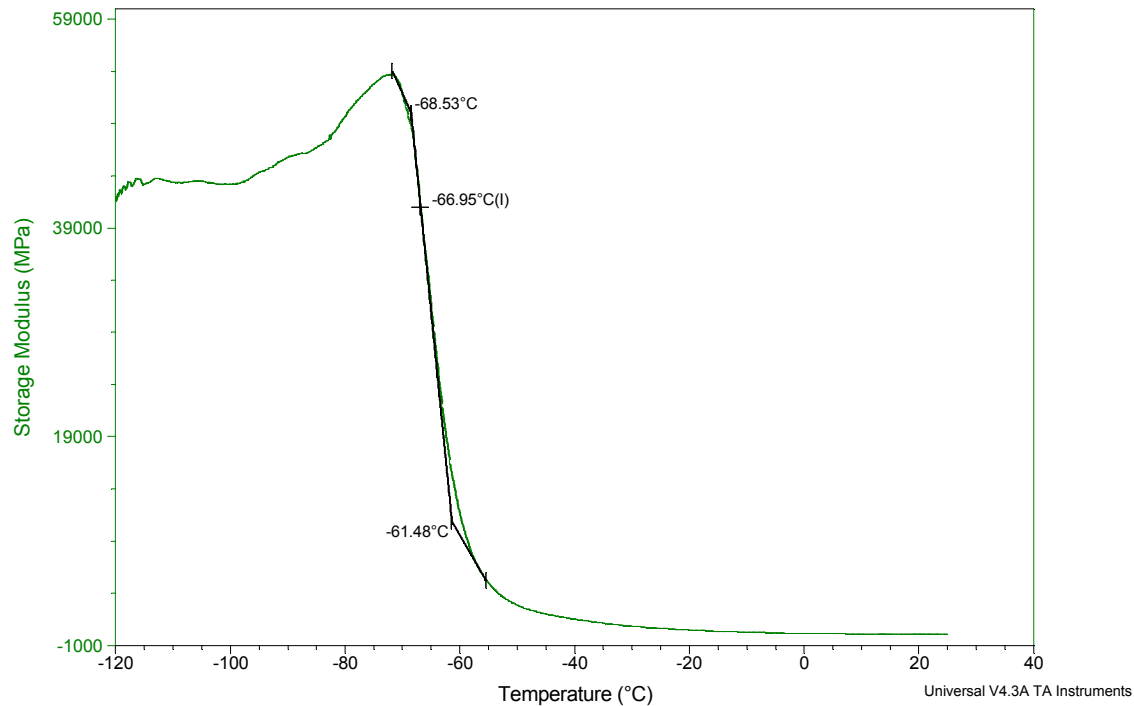


DMA Plot of Hipel 430

Sample: HIPEL 430 TU48150 RUN #2
Size: 15.1550 x 3.5550 mm
Method: HIPEL 430
Comment: 3uM AMP, 8N SF, 1HZ FREQ 150% FORCE TRACK

DMA

File: C:\...DMA\HIPEL 430\HIPEL430 TU48150.02
Operator: Bob Patton
Run Date: 30-Oct-2000 11:41
Instrument: 2980 DMA V1.5B





Thermal Analysis Technique

Bomb Calorimetry

**Uses: Gives Calorific Output
(Calories per Gram) of Materials
That Burn i.e. Pyrotechnics and
Heat Powders for Thermal
Batteries**

Parr 1720 Adiabatic Calorimeter





Thermal Analysis Technique

Accelerating Rate Calorimeter (ARC)

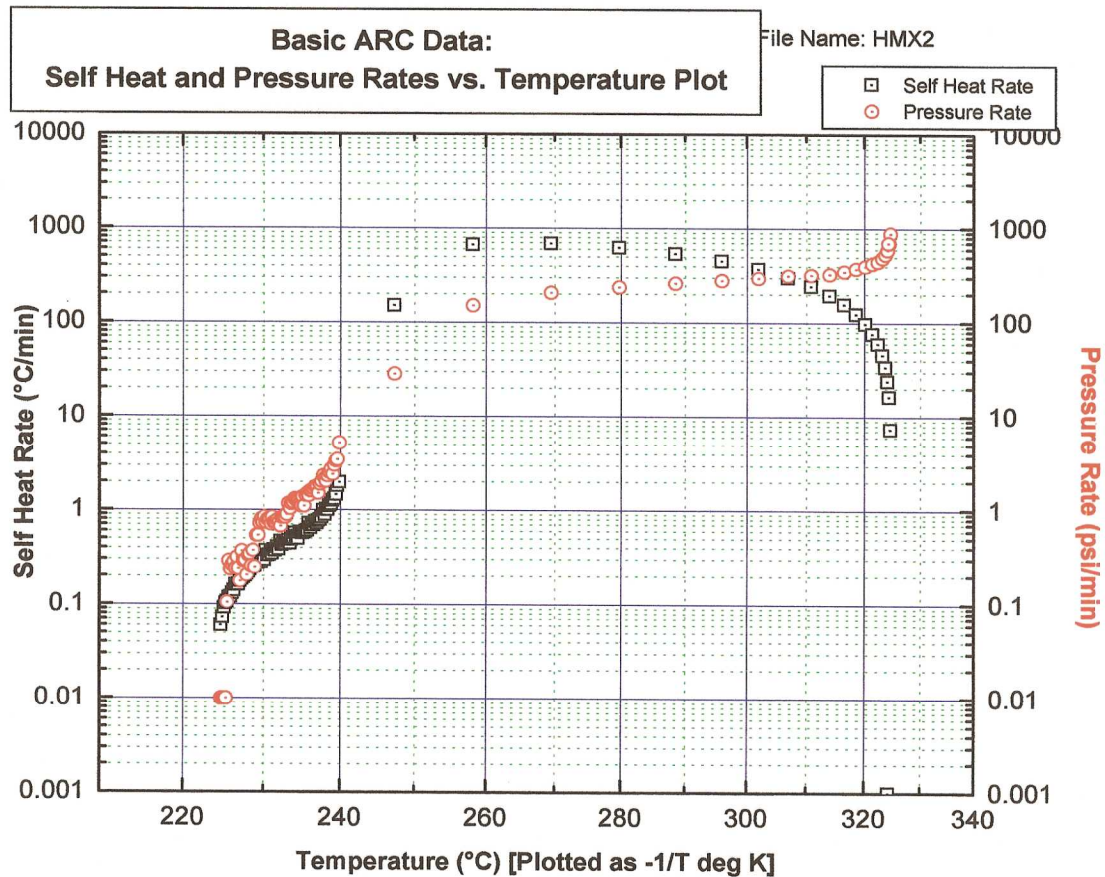
Uses: Provides Heat Rate and Pressure Rate Information for the Sample

Accelerating Rate Calorimeter

- Temperature Range: Room Temperature to 500C
- Pressure Range: Atmospheric to 2000 PSI
- Uses Spherical Bombs with .250" Stem Sealed with Swagelok Fitting
- Heat Generation Detection Limit 0.02C per Minute



ARC Plot of HMX





Fourier Transform Infrared Analysis (FTIR)

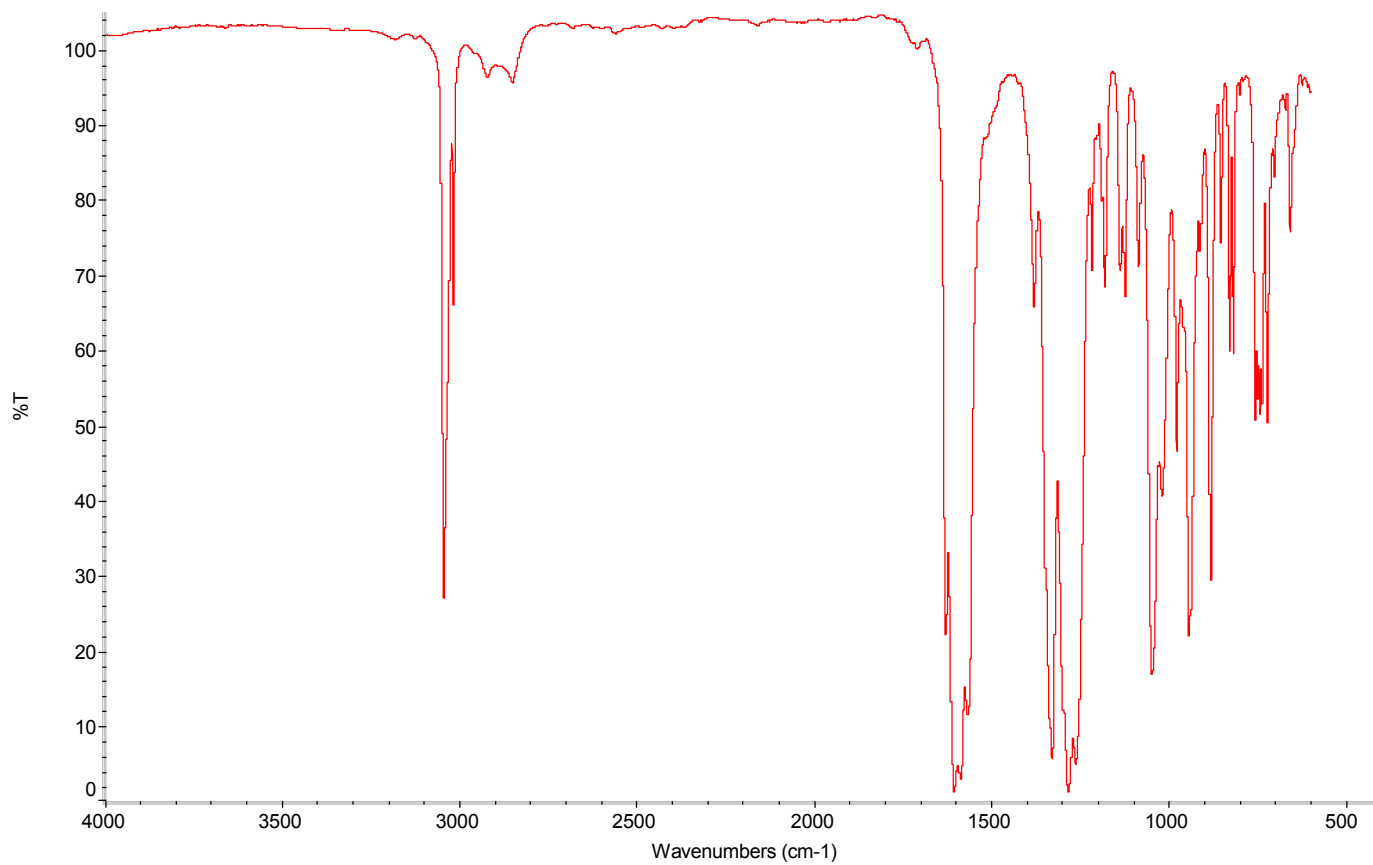
Uses: ID Materials using very small amounts of sample and gases using the AEM. With the microscope a single crystal is enough. With the main bench more sample is required, from 3 to 50 milligrams. Samples used in the TGA for evolved gas ID need to have about a 50% weight loss or a 10 milligram loss at a minimum with a purge flow of 40 to 60 cc/min. to get a good signal.

Nicolet 760 Magna Bench with Microscope





IR Plot of CL20





Particle Size Analysis

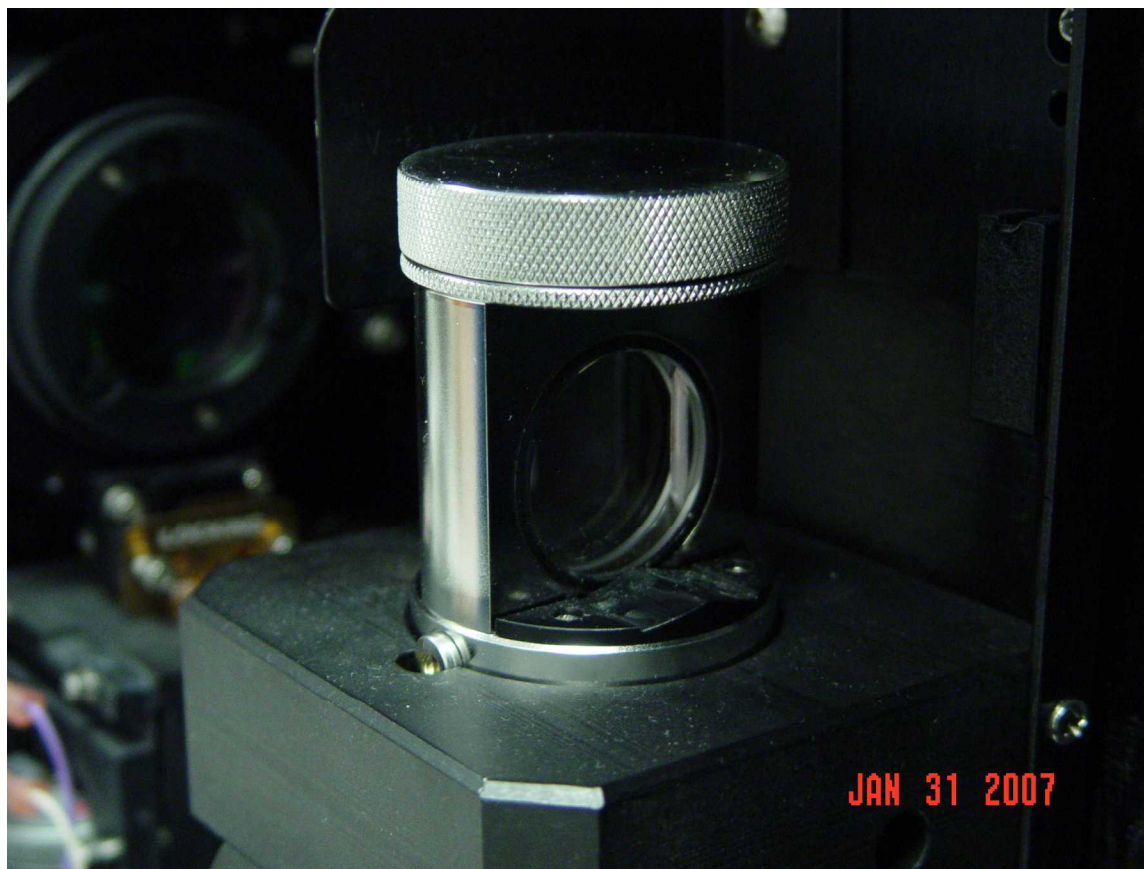
Uses: Give particle size data for a material. Lower limit is 0.4 microns and upper limit is 900.0 microns. Materials are usually put in a 50/50 saturated solution of Isopropyl Alcohol and DI water, sonicated for 5 to 60 seconds and then analyzed. The instrument uses Laser Light Scattering and a circulating system to collect the data.

Coulter LS100





Micro Volume Module



Plot of HNAB Powder

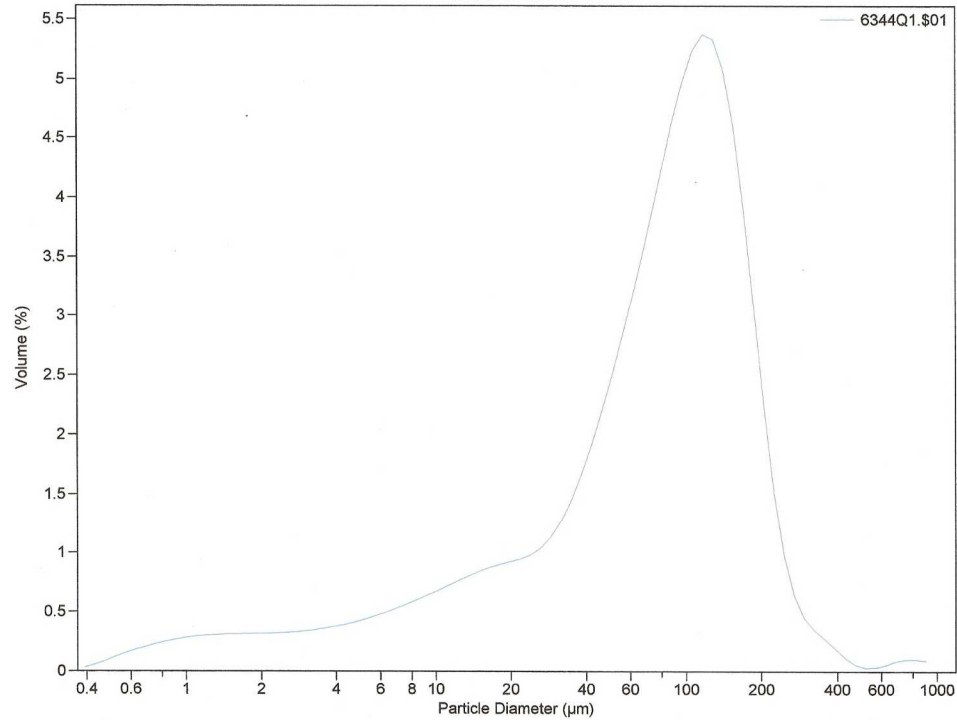


LS Particle Size Analyzer

Sandia National Labs Department 2555

16 Feb 2007

Lot 6344-11H -01





Surface Area Analysis

Uses: Determine the Surface Area of a material using gas adsorption. Nitrogen is used for materials with a surface area higher than 0.5 meters squared per gram and Krypton is used for materials with a surface area lower than 0.5 meters squared per gram. Heating mantels and vacuum is used to prepare samples for analysis.

Micromeritics ASAP2405

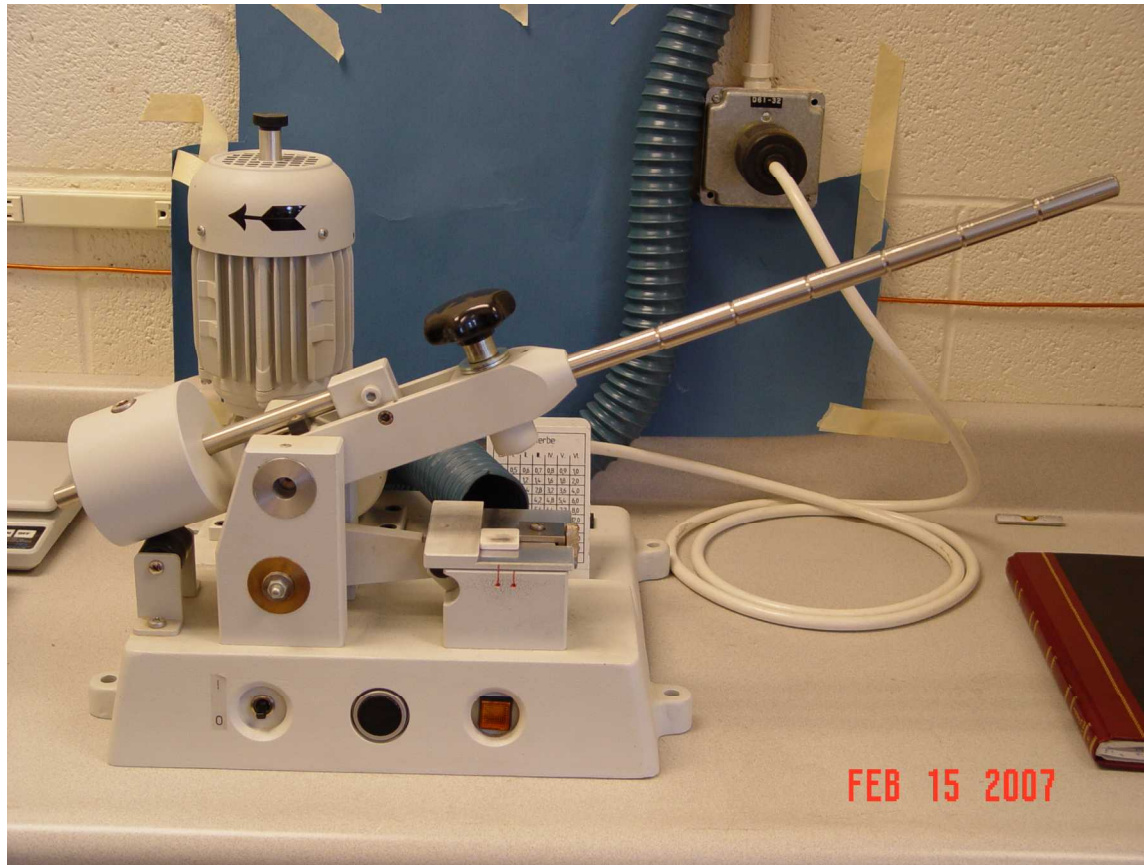




Friction Testing

Uses: Determine the friction sensitivity of a material using the BAM method. The BAM method is where a reaction occurs once in six tries at a weight. The weight range is 0.5 kilograms to 24 kilograms.

Haake Friction Tester



Friction Tester Setup

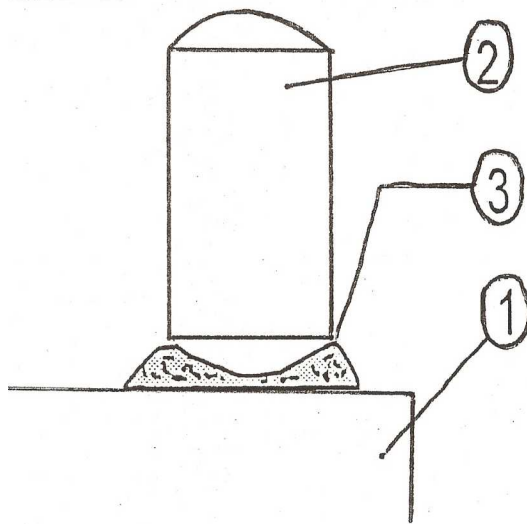


Fig. 2
Setting the pin on the specimen
(starting position)

- 1 Porcelain plate
- 2 Porcelain pin
- 3 Material



Summary

- **For Thermal, IR, Particle Size, and Surface Area Analysis, as well as Friction Testing Contact:**

Tom Massis, Dept. 2555, 844-1540

Bob Patton, Dept. 2555, 844-4375

Rachel Carlson, Dept. 2555, 845-0501