

# TGA & DSC for rubber application

โดยผู้เชี่ยวชาญด้าน Thermal Analysis  
ดร.ชลันดา จุฬาคำ

วันที่ 22 พฤษภาคม 2563  
เวลา 14:00 – 14:30 น.

ท่านที่ไม่ได้ลงทะเบียนล่วงหน้า  
กรุณาแจ้งชื่อและemail ทางแชท  
เพื่อให้ RI ส่งสไลด์สัมมนาไปให้ค่ะ



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โทร 02-3180948 / 083-4816285



# RI Technologies

Enabling Next-Generation Science

**Gregor Kent:**

**Founder and Chief Executive Officer**

- Since 1989, Gregor has been leading and building the RI Group of Companies including
  - Research Instruments Pte Ltd (Singapore),
  - Research Instruments Sdn Bhd (Malaysia),
  - ***RI Technologies Ltd (Thailand)***
  - Origen Laboratories Pte Ltd (Singapore).
  - Research Instruments Co Ltd (Vietnam)

# Brands we represent

 sage science

  
FLUXION

 RESEARCH  
INSTRUMENTS

 BioTek®  
A part of **Agilent**

  
proteintech®  
Antibodies | ELISA kits | Proteins

  
Quanterix  
The Science of Precision Health

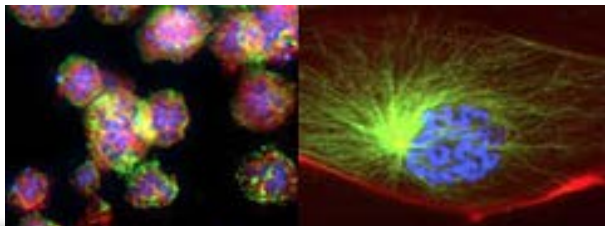
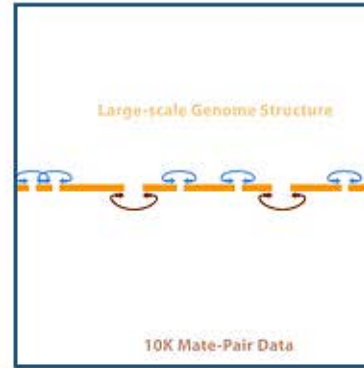
 Seahorse *Bioscience*  
A part of **Agilent Technologies**

  
nanocelllect:  
Biomedical, Inc.

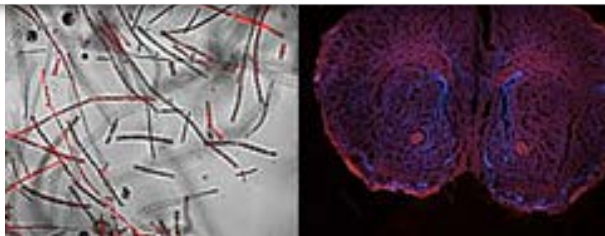
  
NanoEnTek  
Revolutionary Wave for Human Welfare

  
TA  
Instruments

  
GenapSys



Life Science Research



Analytical Resolution

# TA Instruments

- ▶ Headquarters in New Castle Delaware.
- ▶ Manufacturing sites in Delaware, Utah, Wisconsin and Germany.
- ▶ Direct sales & service in 20 different countries
  - ▶ North America
  - ▶ Western Europe
  - ▶ Asia





# Theory of DSC and TGA

The background of the slide is white with abstract, overlapping geometric shapes in various shades of blue (light blue, medium blue, and dark blue) on the right side, creating a modern, technical aesthetic.

## **Thermo**gravimetric Analysis



**Heat**



**Gravity**

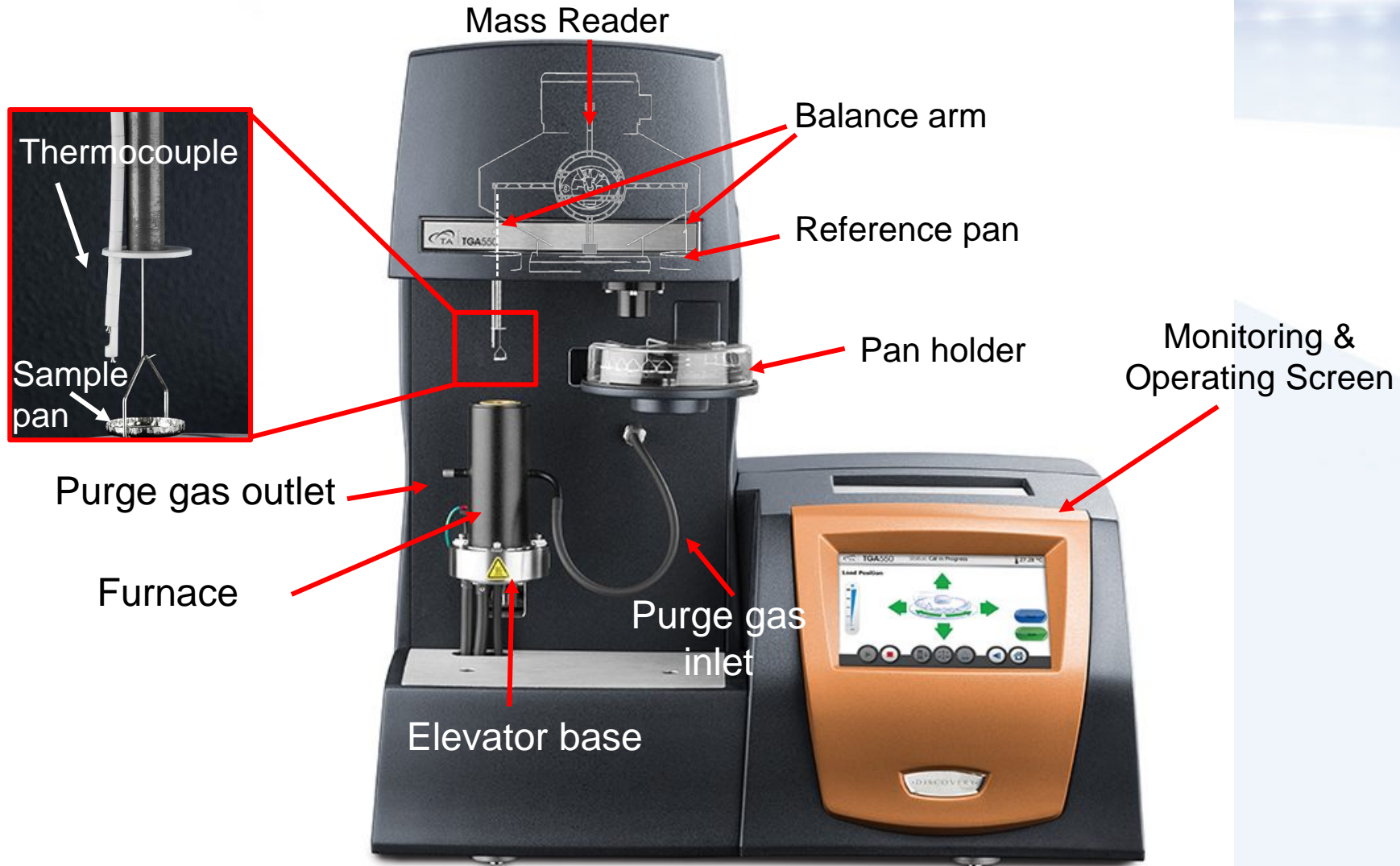


**To measure**

**or weight**

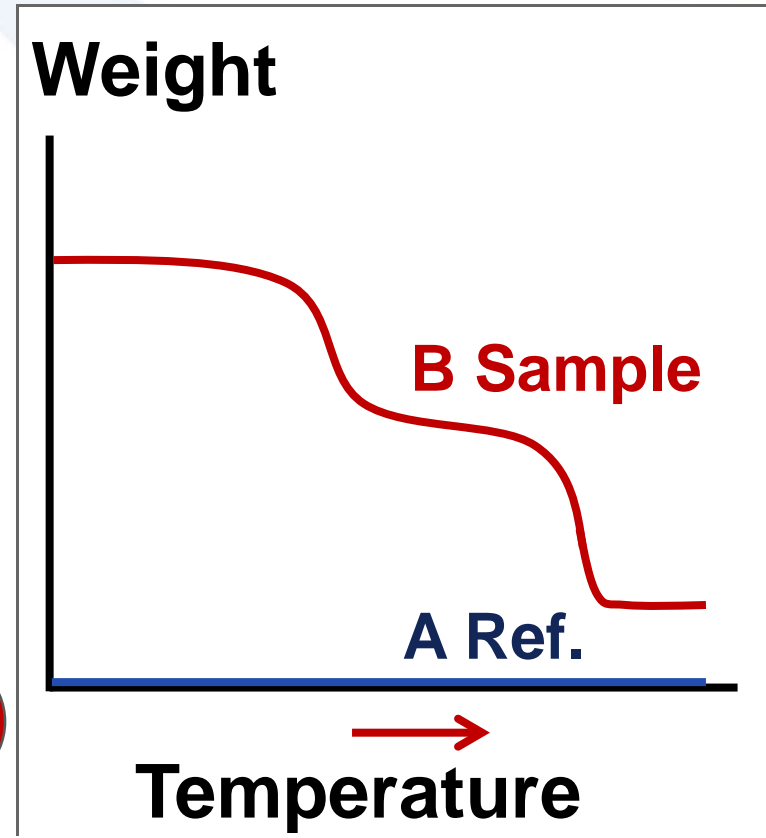
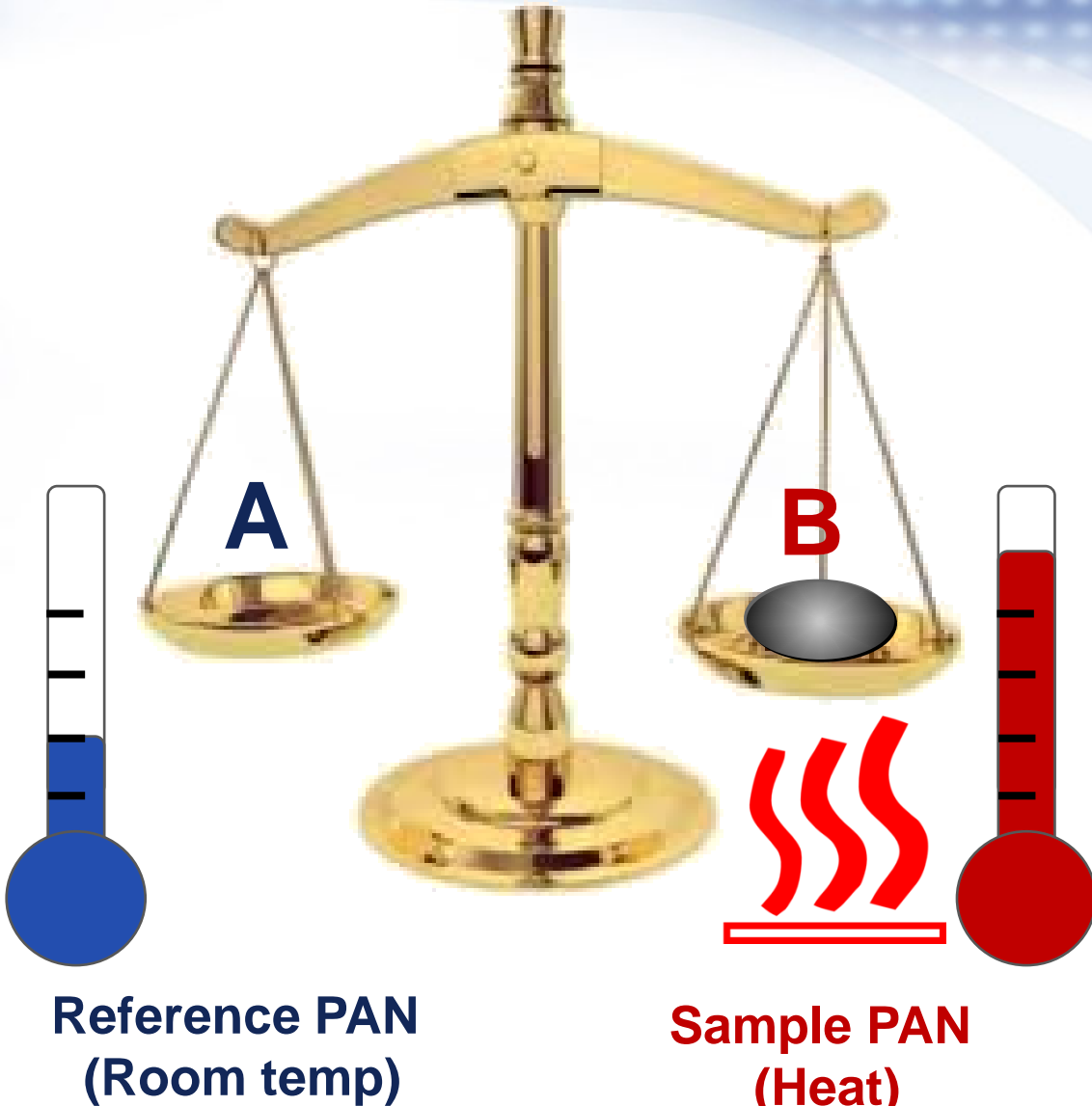
Measurement of amount and rate change in the weight of a material as a function of **temperature** or **time** in a controlled atmosphere (**gas** used)

# TGA: What does it look like?





# How does it work?





- Measuring weight change and kinetic study related to temperature change
- Samples ; solid or liquid (polymers, rubbers, composites, drugs, ceramics, etc.)
- Helpful tool to understand material behavior in production process
- Qualitative and quantitative analyzing composition and stability of material



# **Differential Scanning Calorimetry**

**A DSC measures the difference in **Heat Flow Rate** between a sample and inert reference as a function of time and temperature.**

# DSC: What does it look like?



# Heat flow equation

**Watts = J/s**  
Total Heat Flow

**J/°C**  
Heat Capacity

**°C/min**  
Heating Rate

**J/s**  
Heat flow that is function of time at an absolute temperature

$$\frac{dH}{dt} = C_p \frac{dT}{dt} + f(T, t)$$

**Heat Capacity**

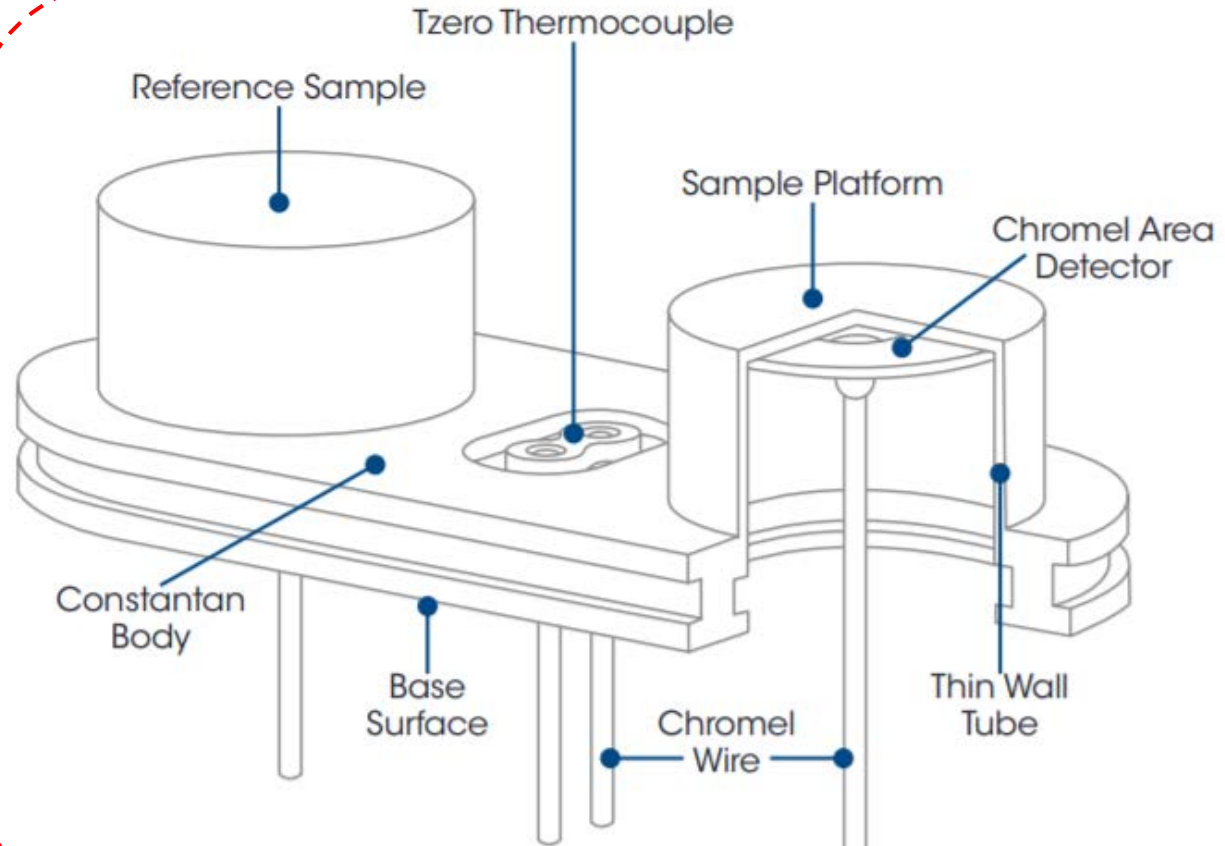
Glass Transition

**Kinetic**

Crystallization  
Cure reactions  
Volatilization  
Decomposition  
Denaturation

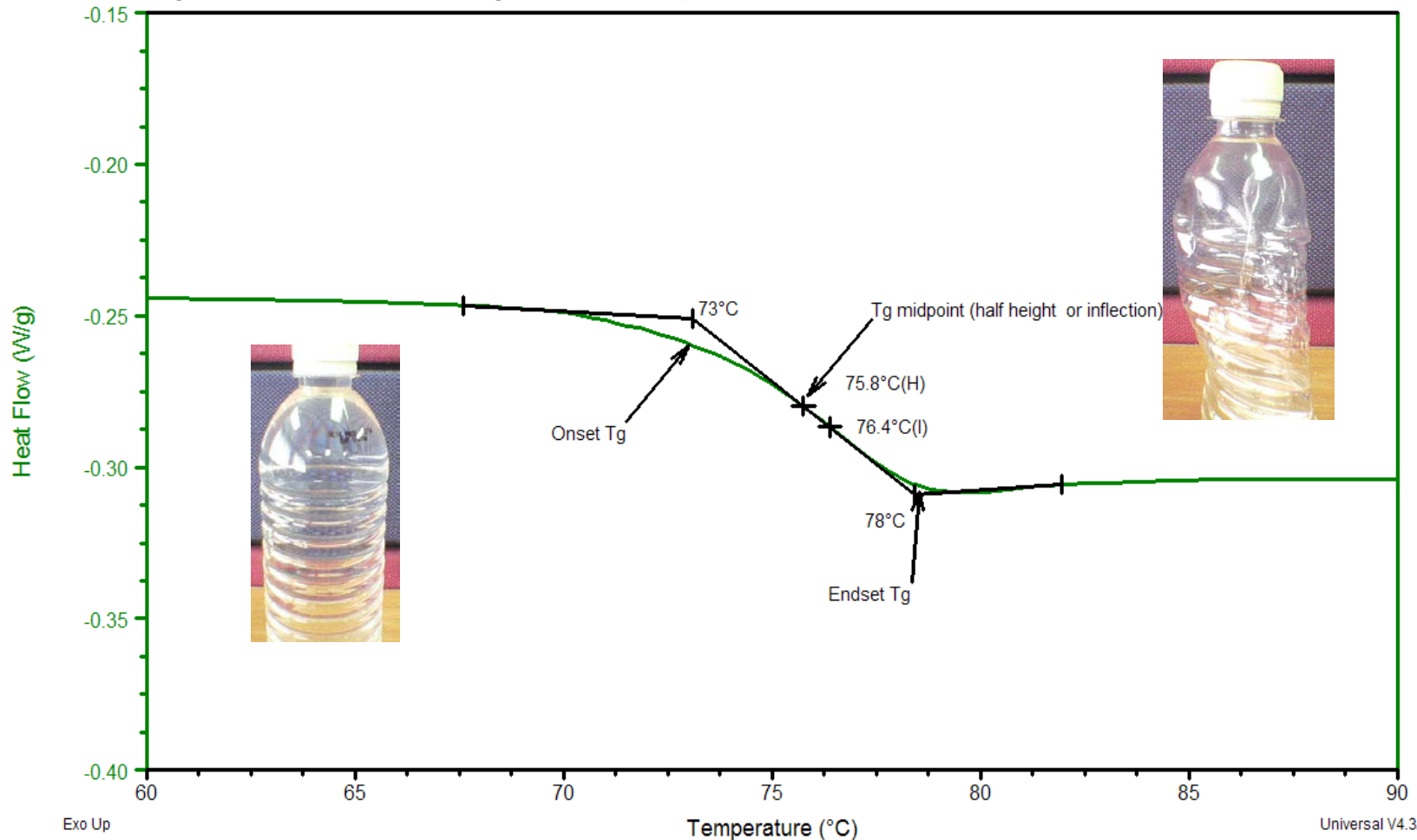


# Inside the cell



# Glass Transition of PET

The Glass Transition is always a temperature range of several to more than 10 degrees depending on many factors

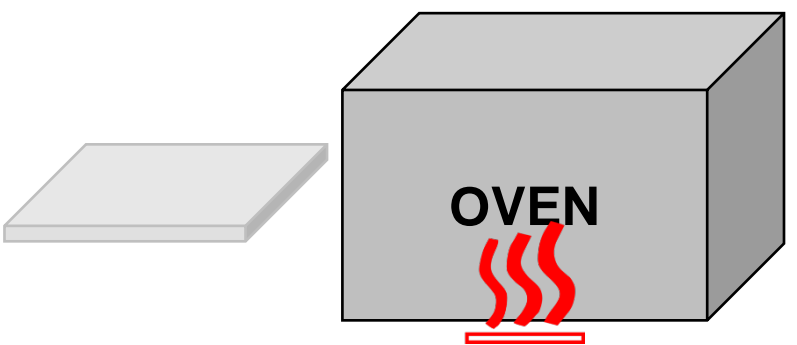
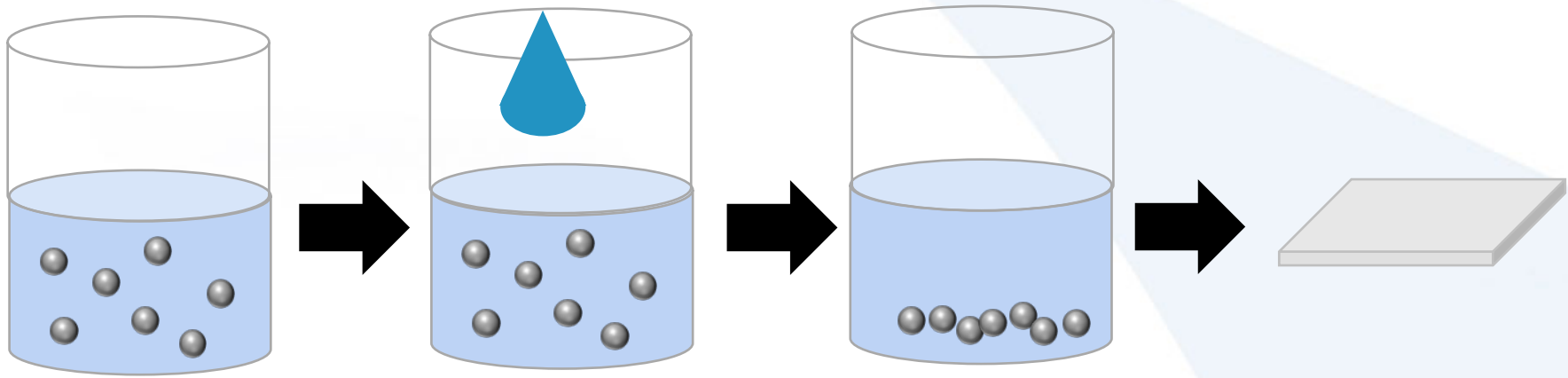


# Rubber Application on DSC and TGA

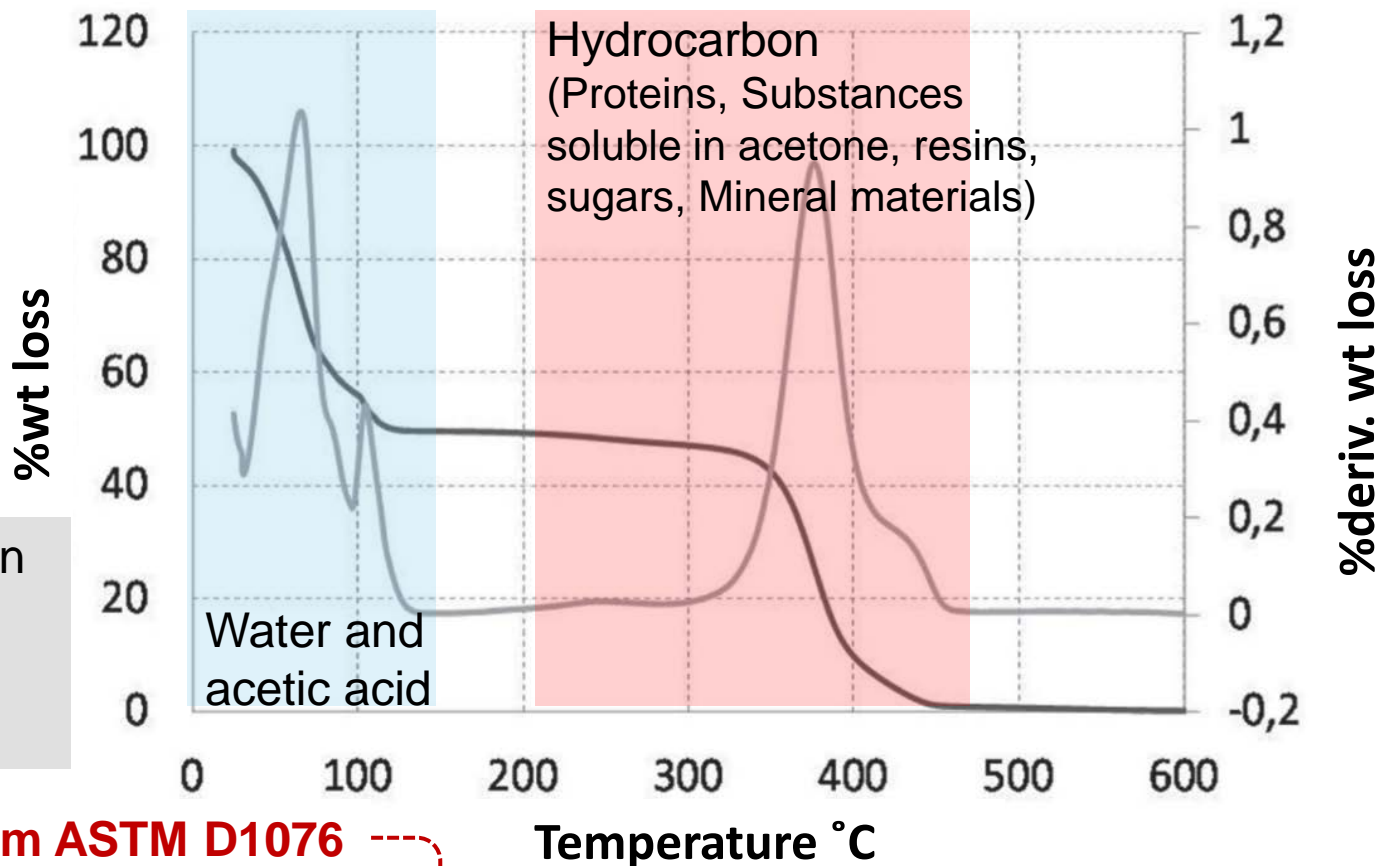
## Total solid content (TSC) and dry rubber content (DRC)

$$\%TSC = \frac{\text{น้ำหนักยางแห้ง} \times 100 \%}{\text{น้ำหนักน้ำยางสด}}$$

$$\%DRC = \frac{\text{น้ำหนักยางที่แห้งแล้ว} \times 100\%}{\text{น้ำหนักก่อนยางสด}}$$



## TG and DTG curves of the characterization of the preserved natural latex

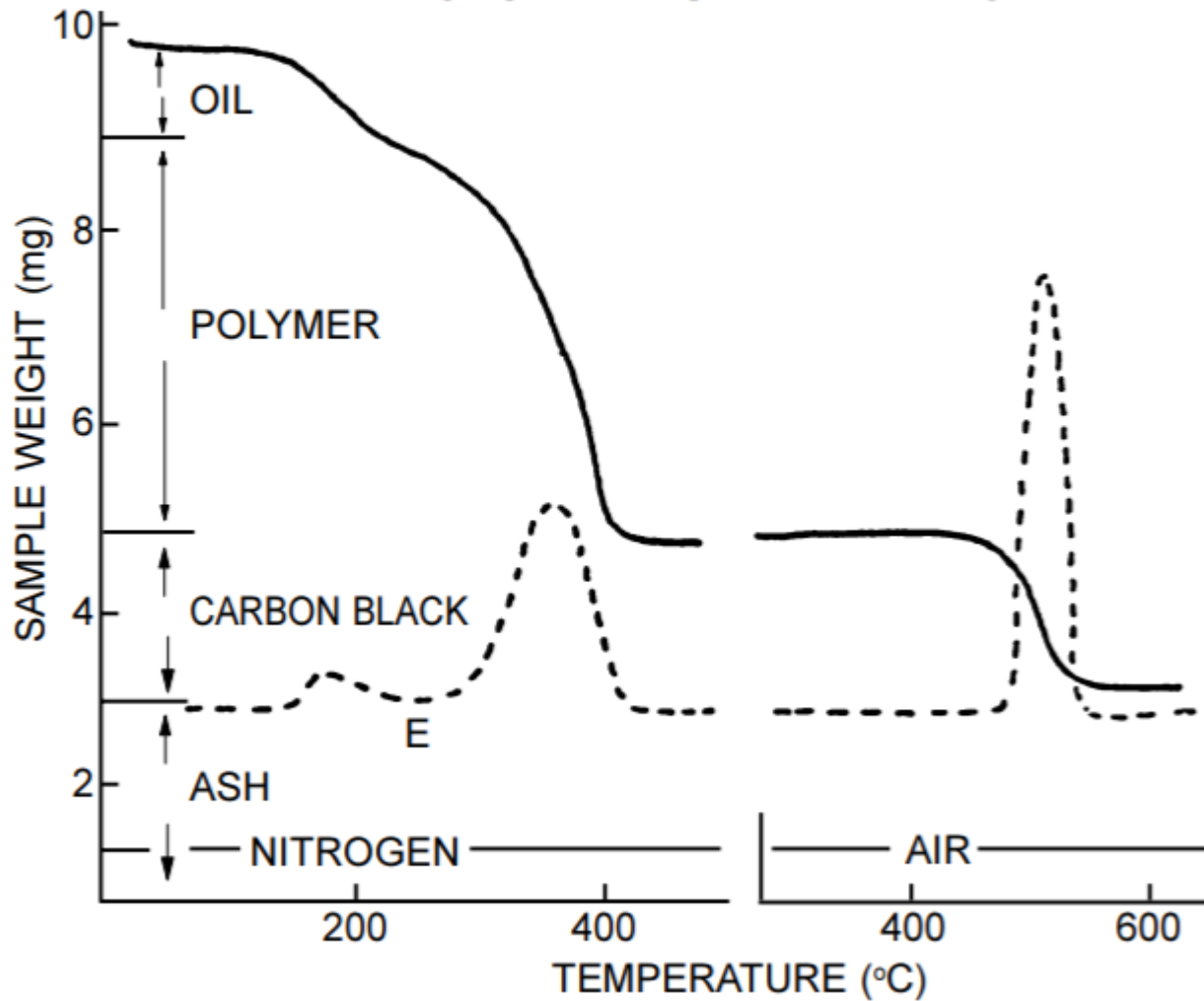


See results within  
**2 hrs**  
Save at least  
**14 hrs!**

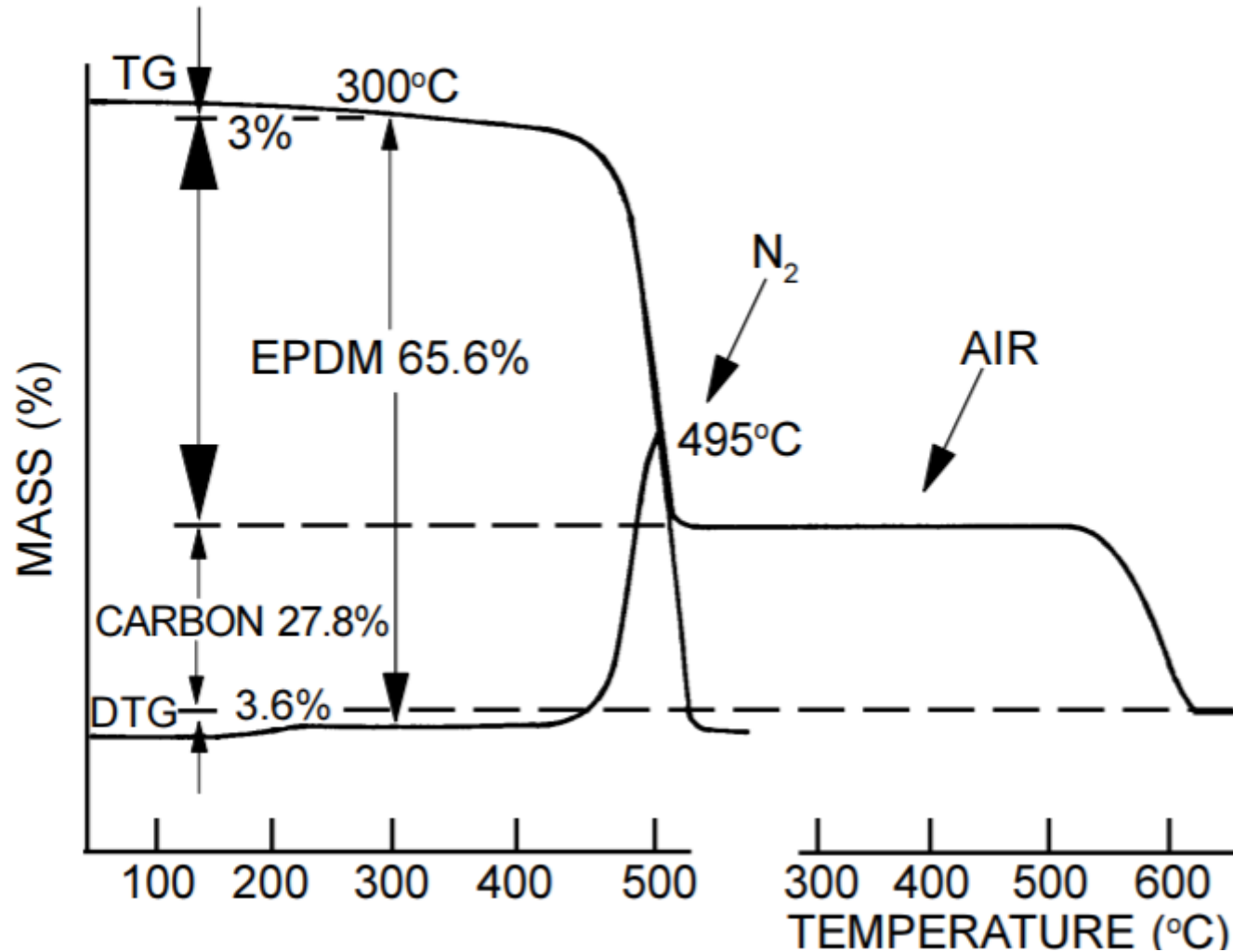
**Difference from ASTM D1076**  
Total solid content (TSC) (%wt) : 0.45%  
Dry rubber content (DRC) (%wt) : 0.13%



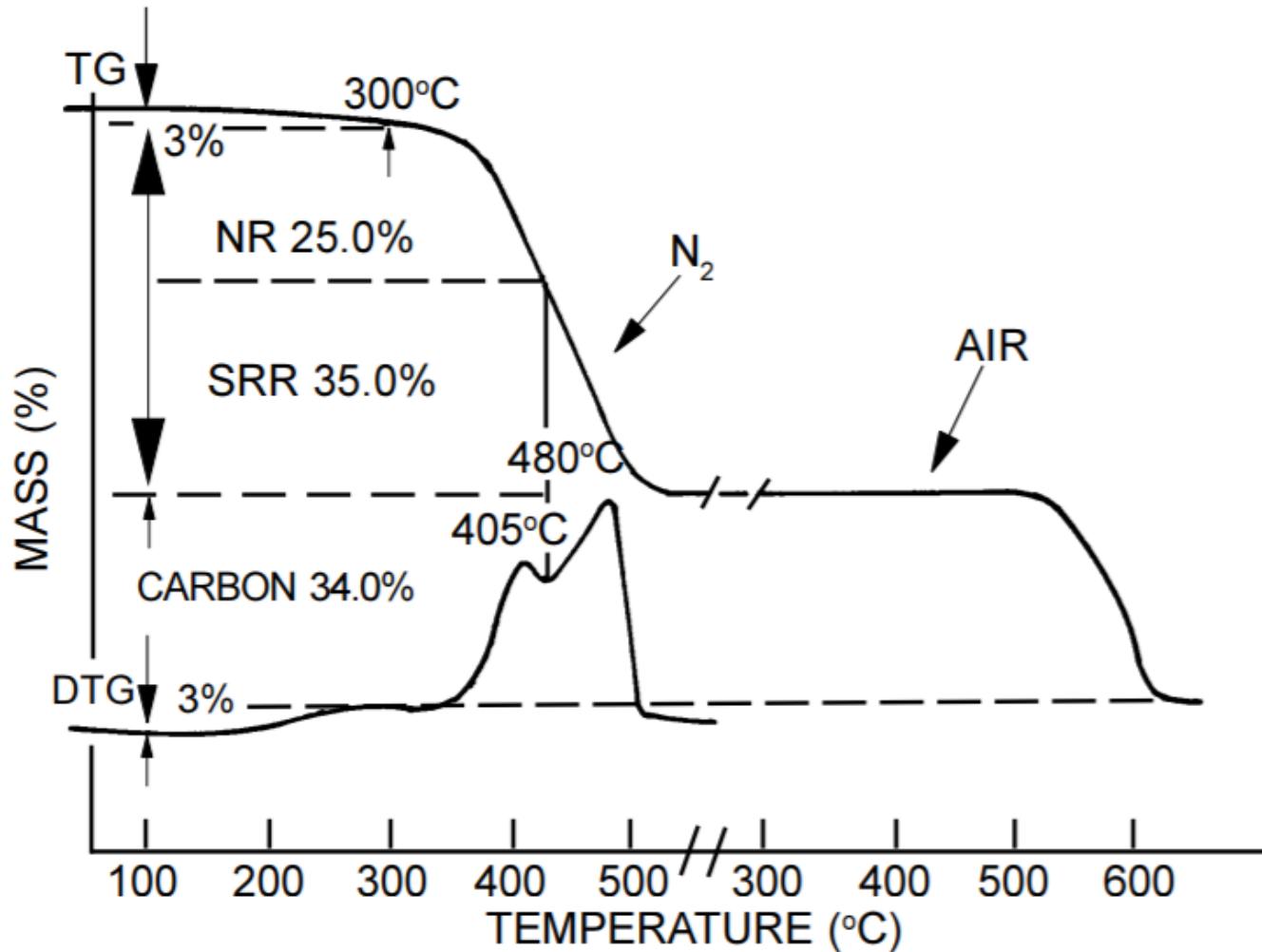
## SCHEMATIC TG AND DTG CURVE OF ELASTOMER VULCANIZATES<sup>19</sup>



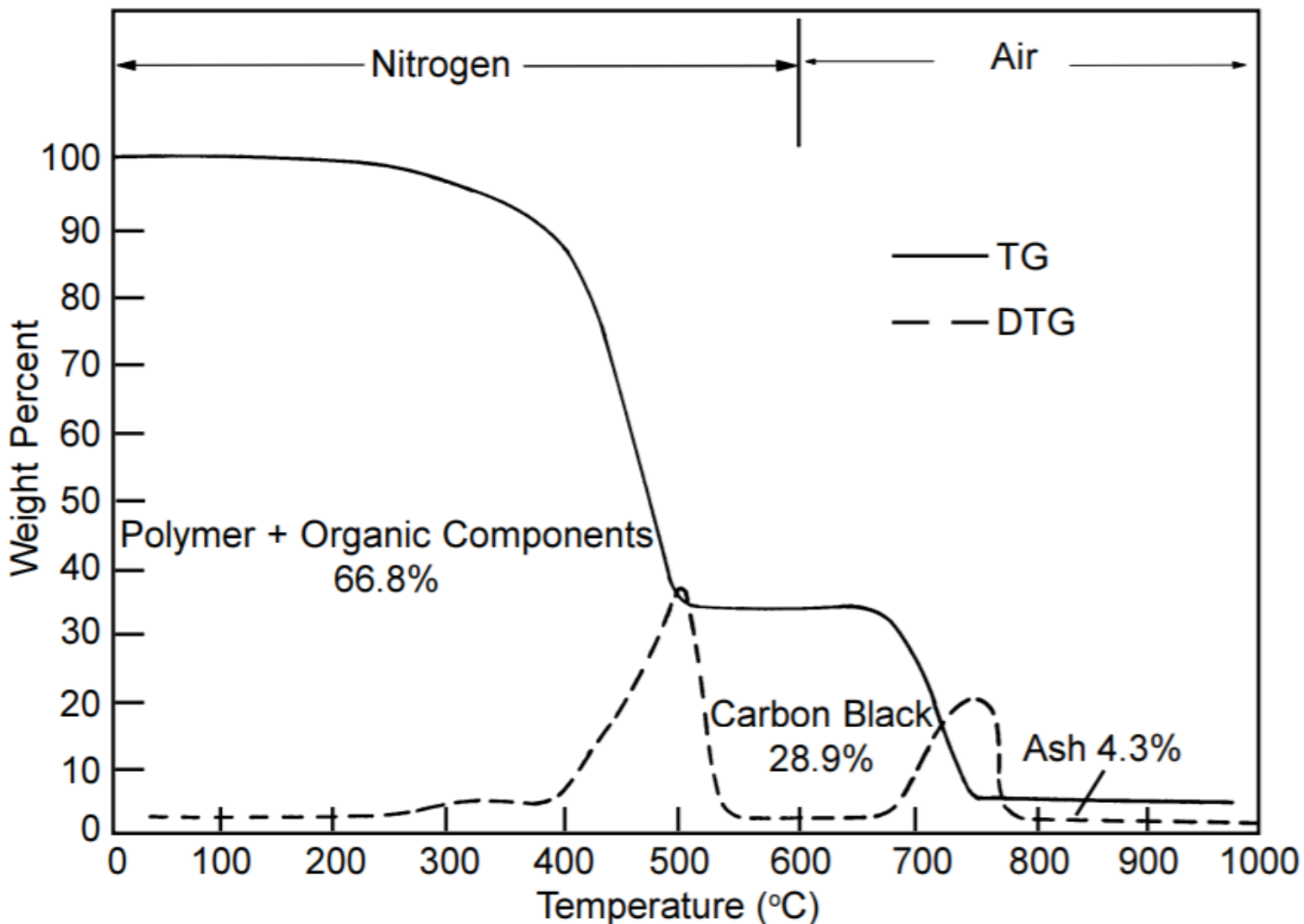
## TG-DTG CURVES OF EPR VULCANIZATES



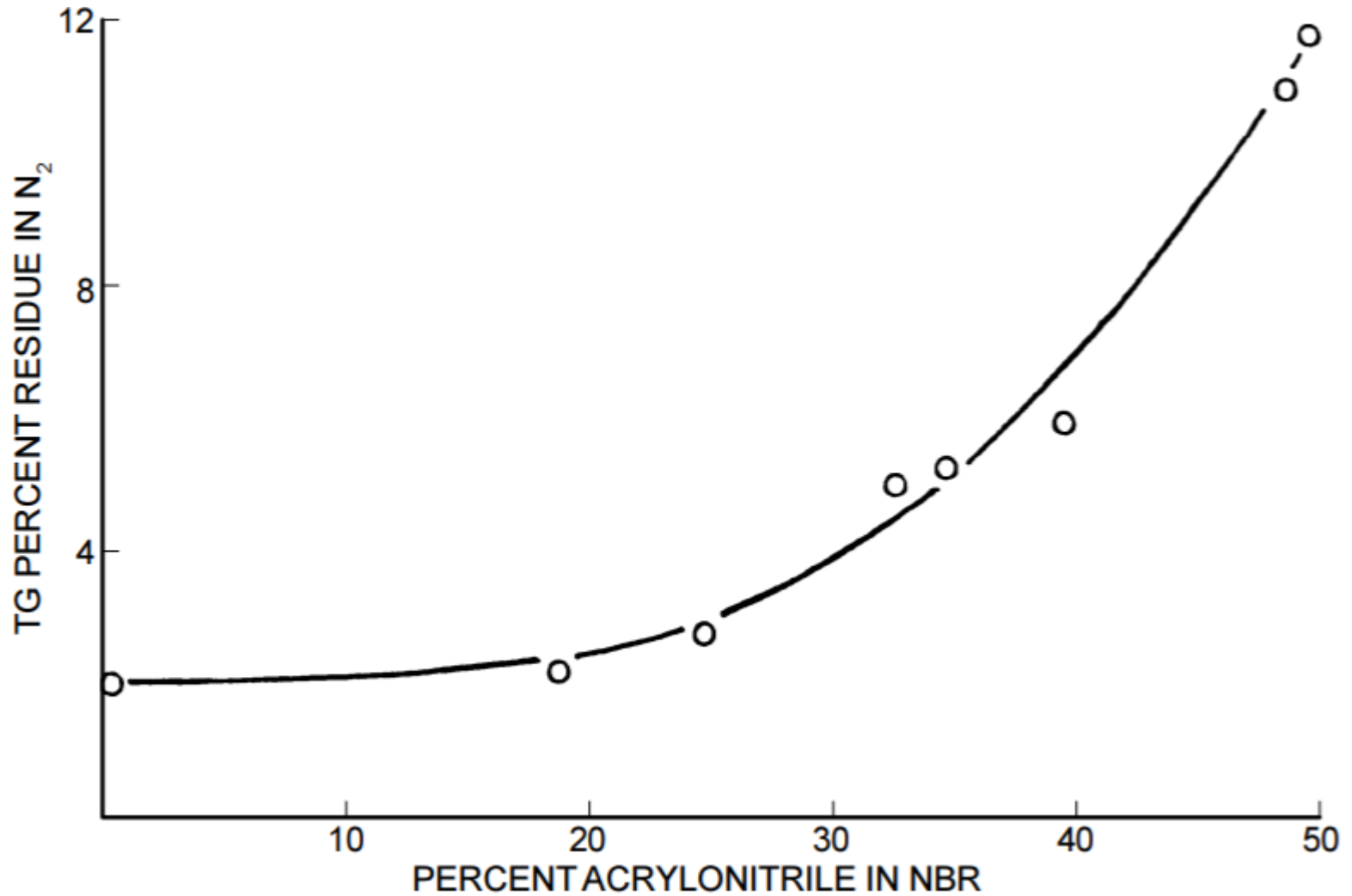
## TG-DTG CURVES OF NR-SBR VULCANIZATES



## TG-DTG ANALYSIS OF A SBR VULCANIZATE

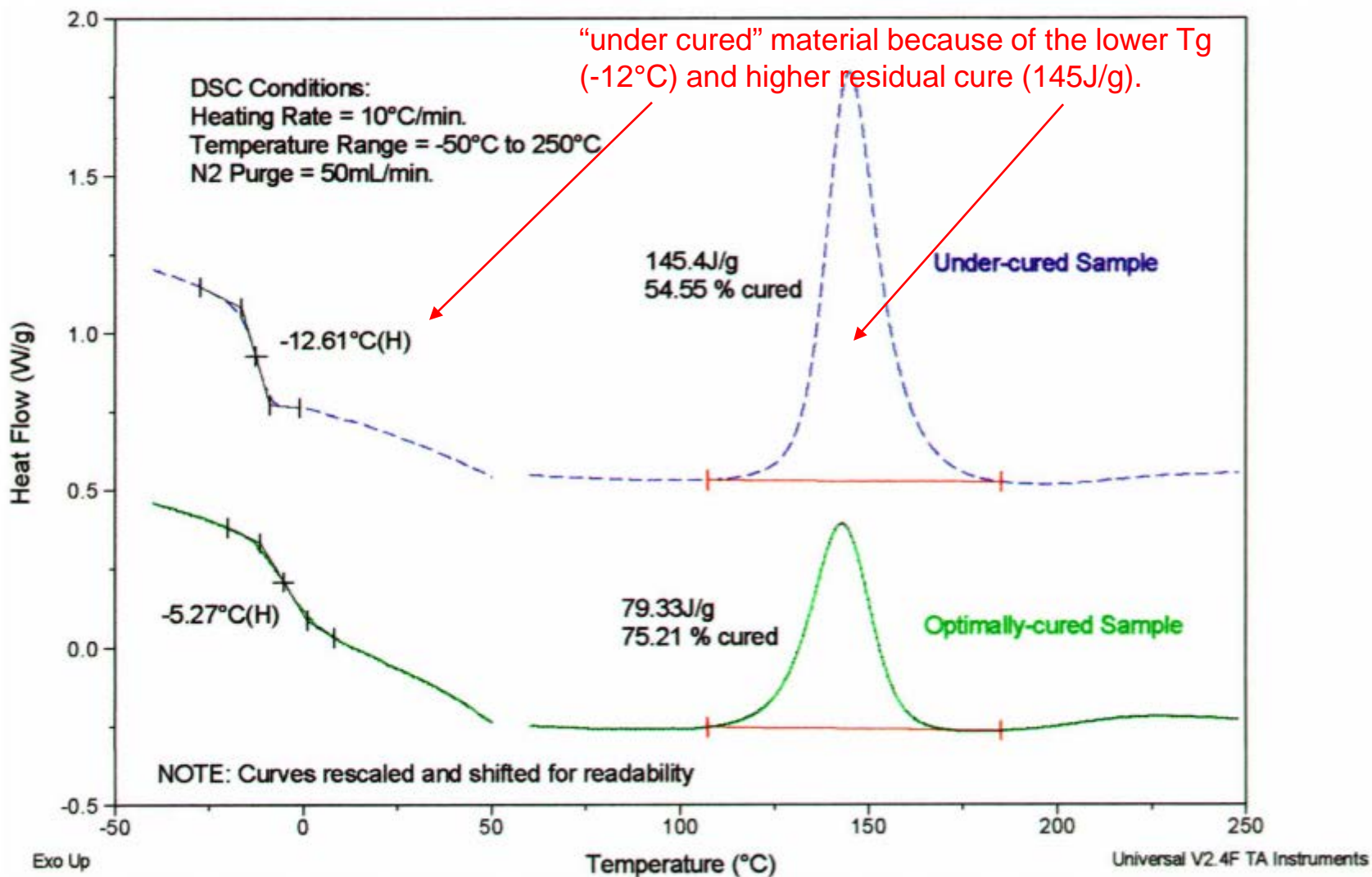


# Carbon Residue in N<sub>2</sub> with Nitrile Content of NBR Elastomer





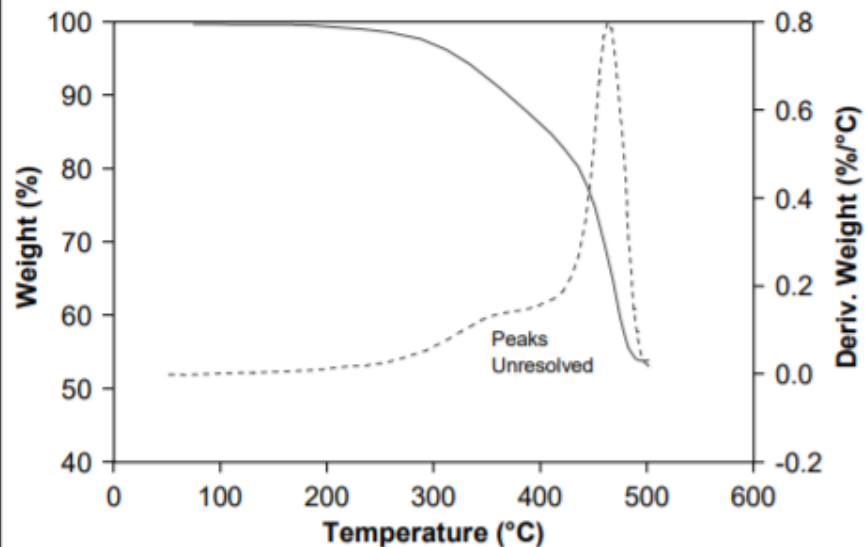
# Characterization of the %Cure of Thermosetting Resins by DSC



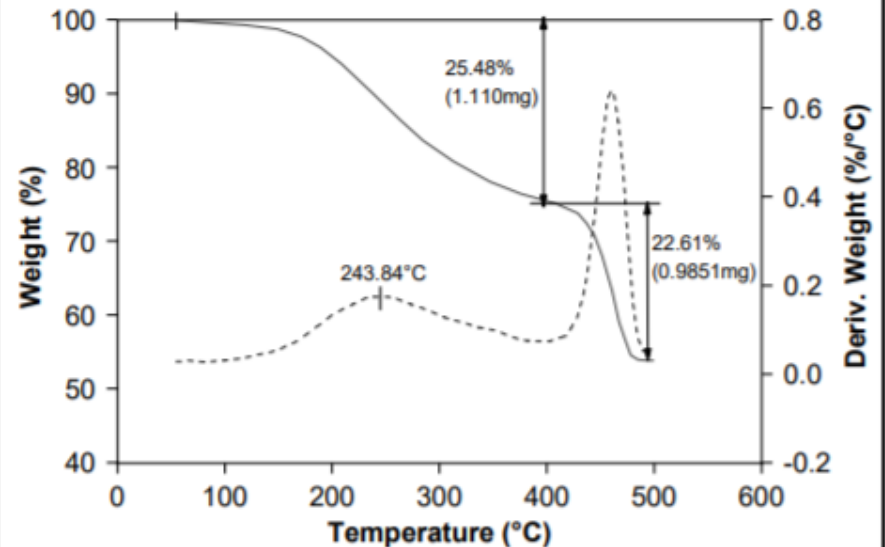
# Determination Of Oil in Rubber by Vacuum TGA

Many rubber products are made of simple mixtures of oil, raw rubber, and carbon black. End-use performance of these materials is dependent on batch composition, with variations of even 1 or 2% causing significant differences. For this reason, manufacturers of tires, seals, gaskets, and vibration-damping mounts require a way to quickly assess batch composition.

**Figure 1. NORMAL TGA AT AMBIENT PRESSURE**



**Figure 2. TGA AT 5Pa**

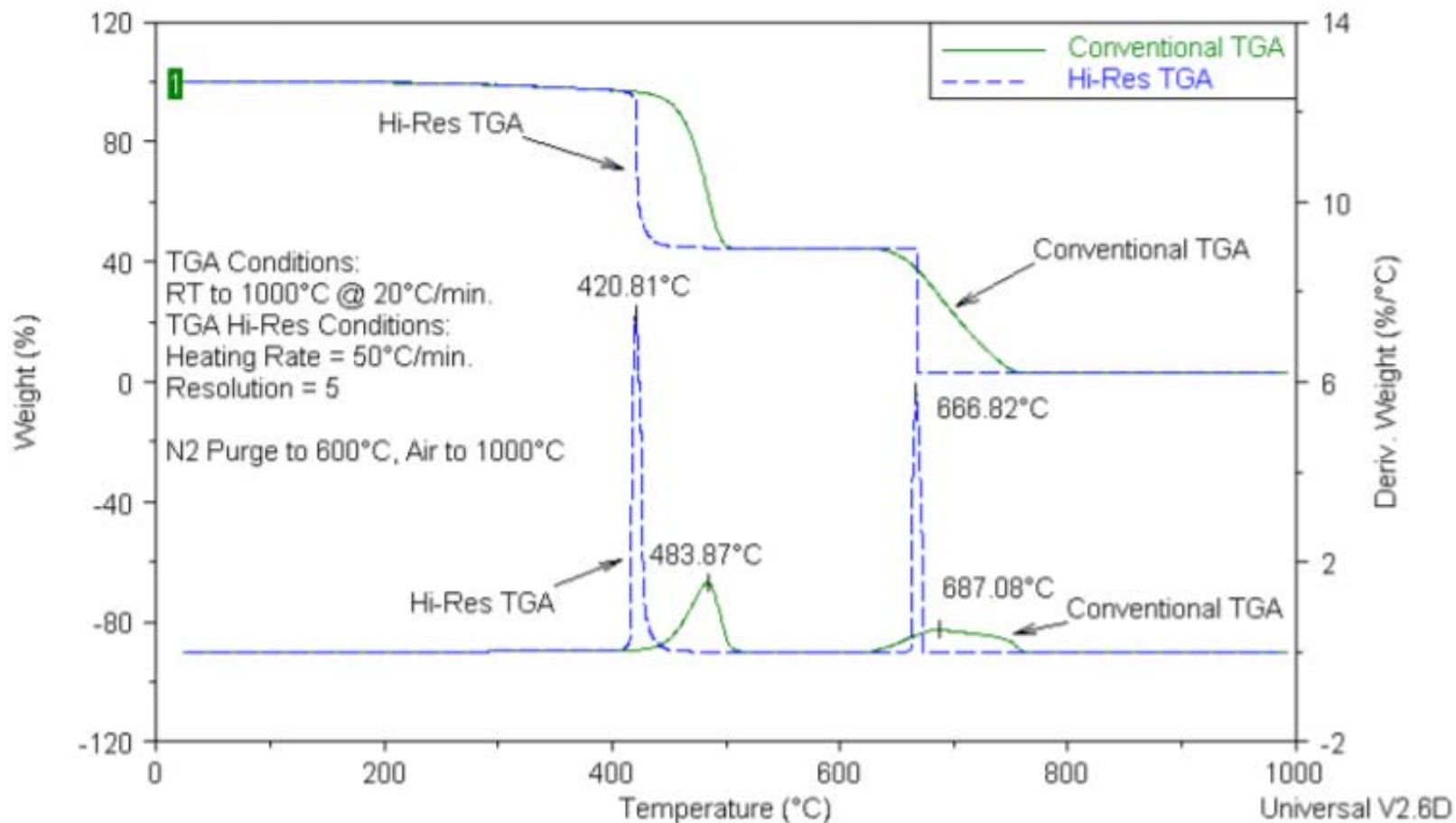


Rubber begins to pyrolyze before all of the oil evaporates

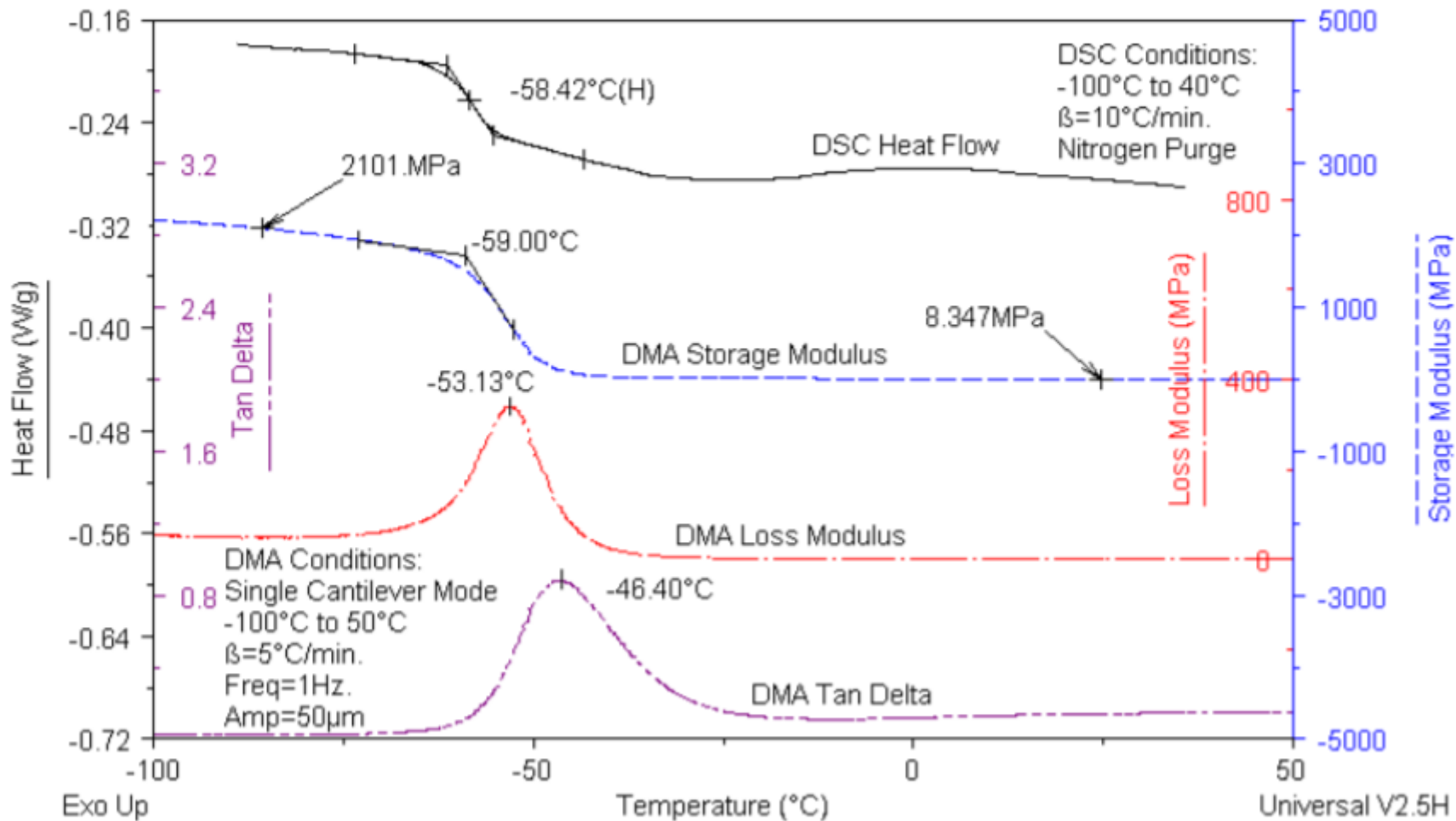
# How to increase Resolution?

Solutions	Consequences
Slowing down the ramp rate	Increase in test time
Reducing sample mass	May give issues with sensitivity (Sample depending)
Reducing sample size	Sample behavior may not changed in some cases
Change Purge Gas	not applicable in all cases
Pin-hole Hermetic Pans	not applicable in all cases
<b>Using Hi-resolution mode</b>	<b>good result with short test time</b>

# Characterization of EPDM Rubber by TGA and Hi-Res TGA



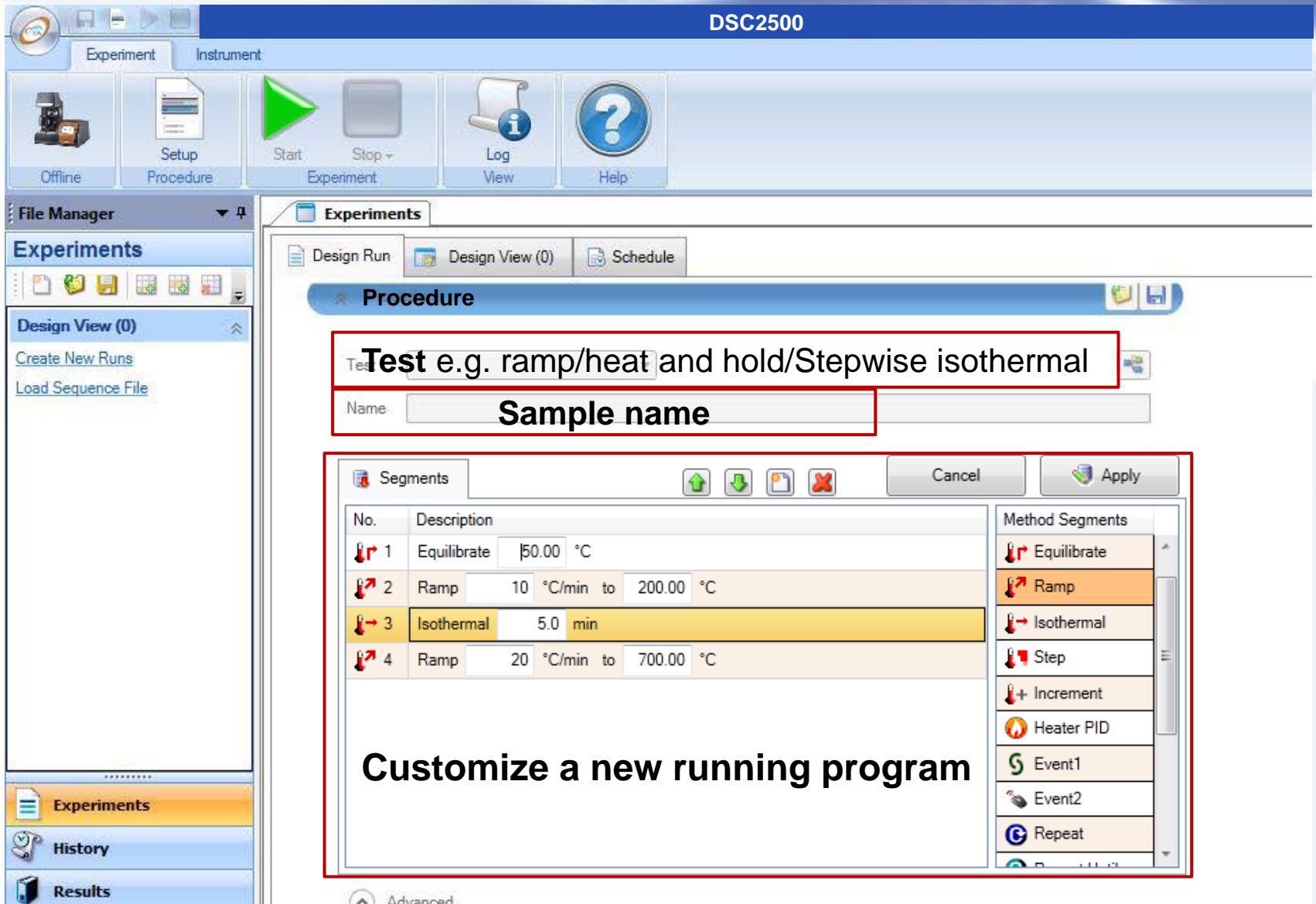
# Characterization of EPDM Rubber by DSC and DMA





Easy Analysis by





The screenshot shows the DSC2500 software interface. The top bar displays 'DSC2500'. Below it is a toolbar with icons for Offline, Setup Procedure, Start, Stop, Log View, and Help. The left sidebar contains a File Manager with sections for Experiments, Design View (0), and Results. The main window is titled 'Experiments' and shows a 'Procedure' dialog box. The dialog box has a 'Test' field containing 'Test e.g. ramp/heat and hold/Stepwise isothermal', a 'Name' field containing 'Sample name', and a 'Segments' table. The 'Segments' table has columns for 'No.', 'Description', and 'Method Segments'. The 'Method Segments' list includes Equilibrate, Ramp, Isothermal, Step, Increment, Heater PID, Event1, Event2, and Repeat. The 'Isothermal' segment is highlighted in yellow. A red box highlights the 'Test' field, the 'Name' field, and the 'Segments' table. The text 'Customize a new running program' is overlaid on the bottom of the dialog box.

**DSC2500**

Experiment Instrument

Offline Setup Procedure Start Stop Log View Help

File Manager Experiments

Design View (0)

Create New Runs Load Sequence File

Experiments

History Results

Design Run Design View (0) Schedule

Procedure

Test **Test e.g. ramp/heat and hold/Stepwise isothermal**

Name **Sample name**

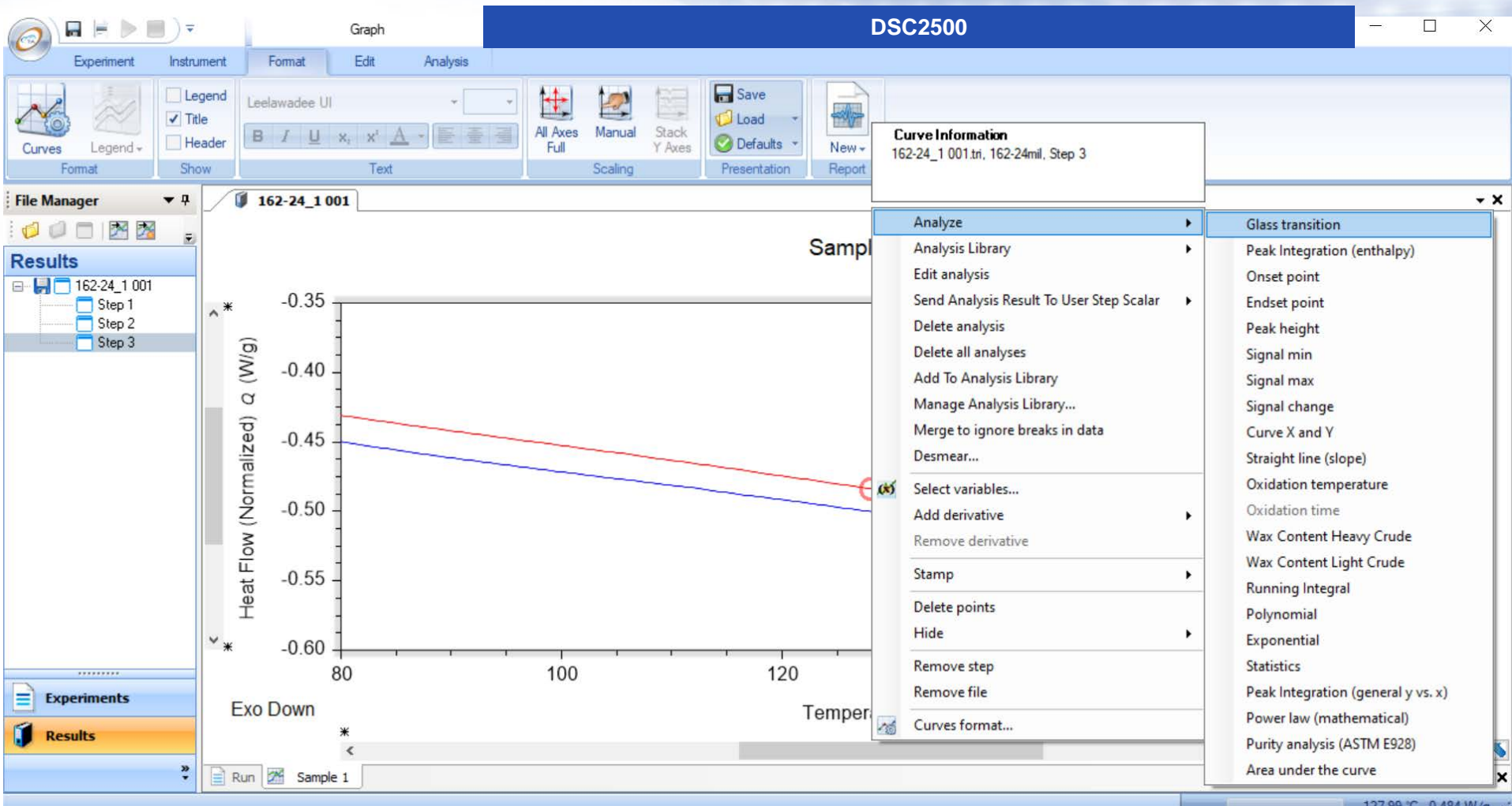
Segments

No.	Description	Method Segments
1	Equilibrate 50.00 °C	Equilibrate
2	Ramp 10 °C/min to 200.00 °C	Ramp
3	<b>Isothermal 5.0 min</b>	Isothermal
4	Ramp 20 °C/min to 700.00 °C	Step

Cancel Apply

**Customize a new running program**

Advanced



Offline-NGDSC : TA Instruments Trios v5.0.0.44608

Report

Experiment Instrument Format

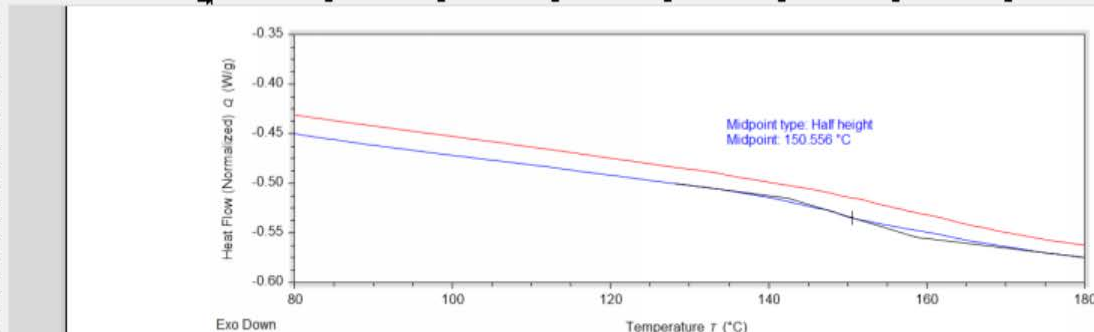
Paste Cut Copy B I U S x, x' Image Table Text Frame Header Footer Page Number Date/Time Margins & Paper Orientation Columns Breaks Template Save

File Manager 162-24\_1\_001

Results

- 162-24\_1\_001
  - Step 1
  - Step 2
  - Step 3

Experiments Results



Heat Flow (Normalized)  $q$  (W/g)

Temperature  $T$  (°C)

Midpoint type: Half height  
Midpoint: 150.556 °C

Exo Down

Standard Heat/Cool/Heat  
2018-05-15 10:48:56 Gas 1 Event Off Sampling 1.0

Glass transition

Midpoint type	Midpoint
Half height	150.556 °C

Document Tokens

- TempRange
- AutoZero
- MultiPtCal
- MultiPtDesc
- AutoCellConst
- Controls
- Cell#
- CoolingUnit
- SelfHeatFlow
- AutoLid1
- AutoAnalysis
- Nsig
- Sig1
- Sig2
- Sig3
- Sig4
- Date
- Time
- OrgMethod
- Run
- Step Overlay
- Analysis Summary
- Glass transition

1/1 1/1 12 0 English (United States) 100%

Run Step Overlay Report 1





**info@ri.co.th**