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TA Instruments

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

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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

RI Technologies

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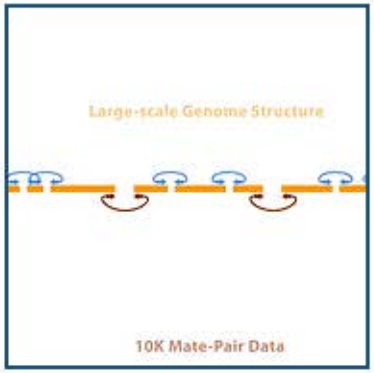

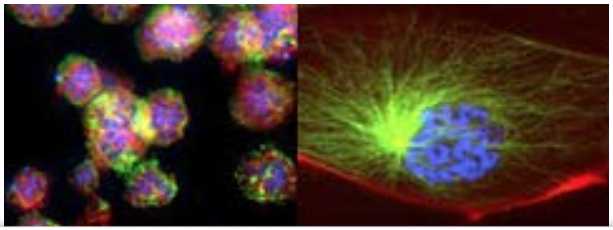
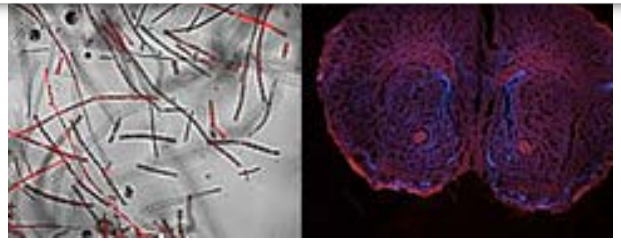

Life Science Research

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

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Thermogravimetric Analysis (TGA)

Thermogravimetric Analysis

Heat ⬇ **Gravity or weight** ⬇ **To measure** ⬇

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)

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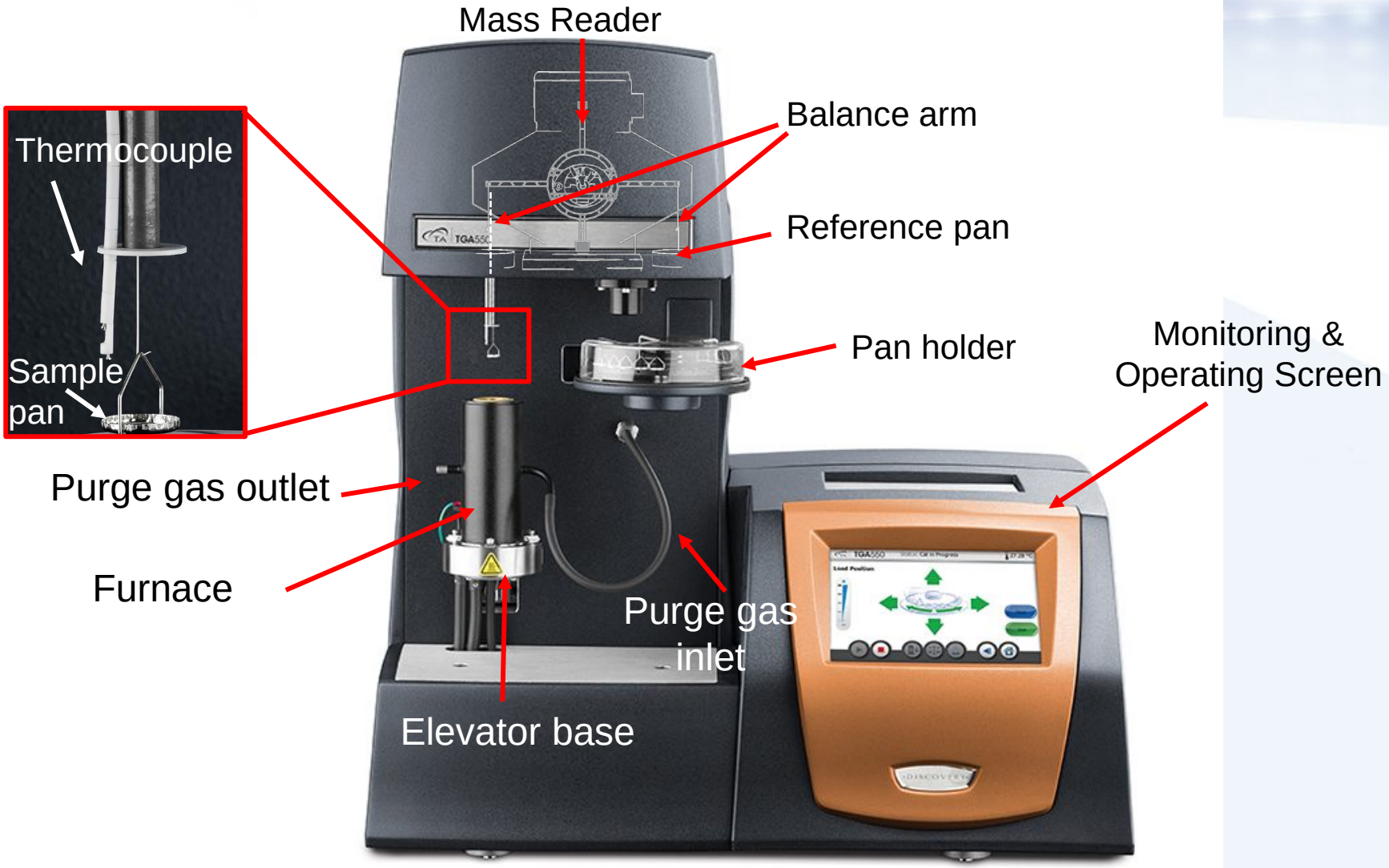
TGA: What does it look like?



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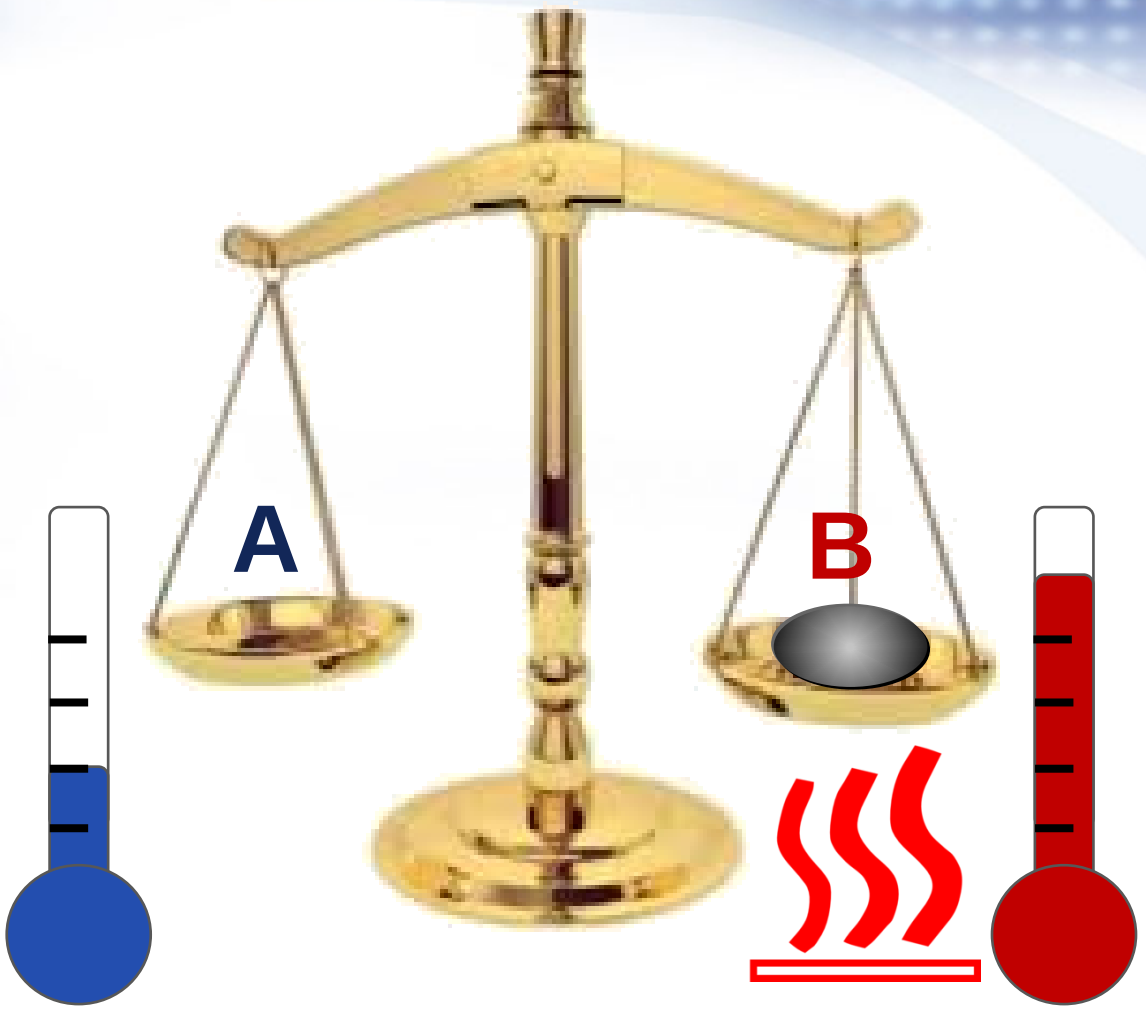
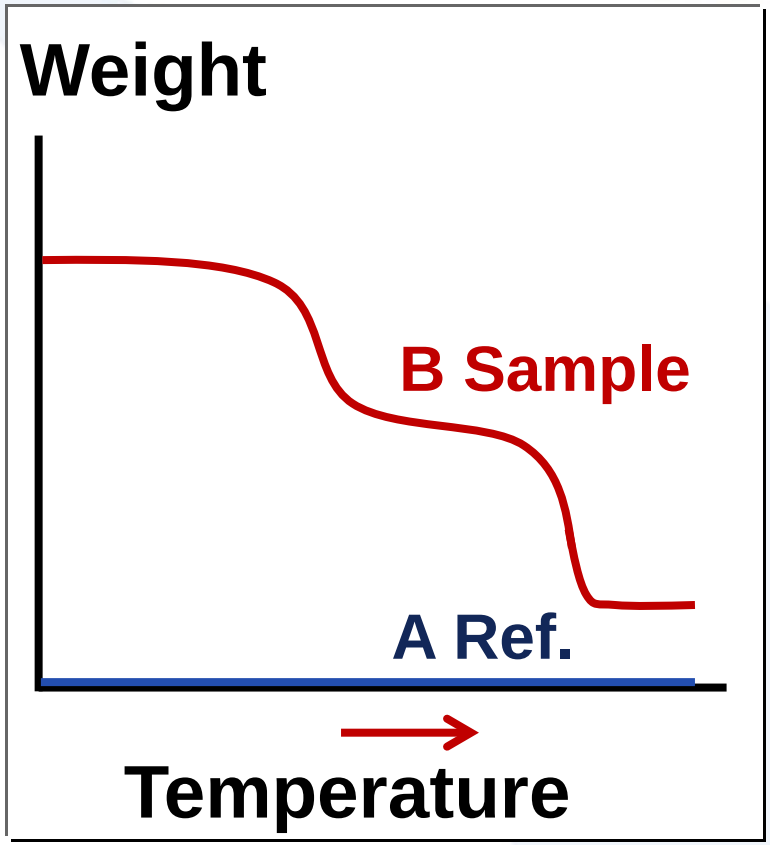
How does it work?



Reference PAN
(Room temp)

Sample PAN
(Heat)



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Summary

- 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.

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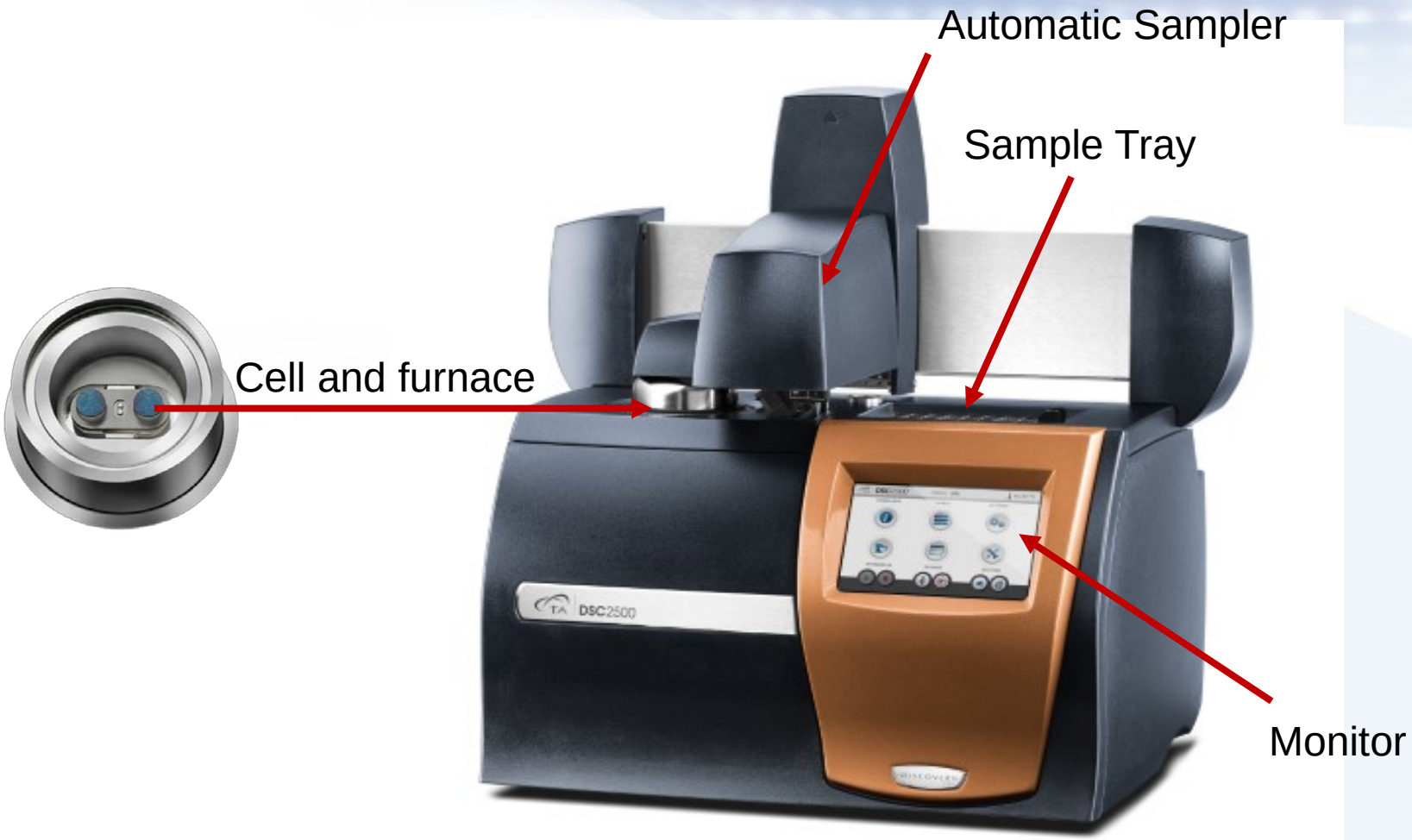
DSC: What does it look like?



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Heat flow equation

Watts = J/s

Total Heat Flow

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

J/°C

°C/min

Heating Rate

J/s

Heat flow that is function of time at an absolute temperature

Heat Capacity

- Glass Transition

Kinetic

- Crystallization
- Cure reactions
- Volatilization
- Decomposition
- Denaturation

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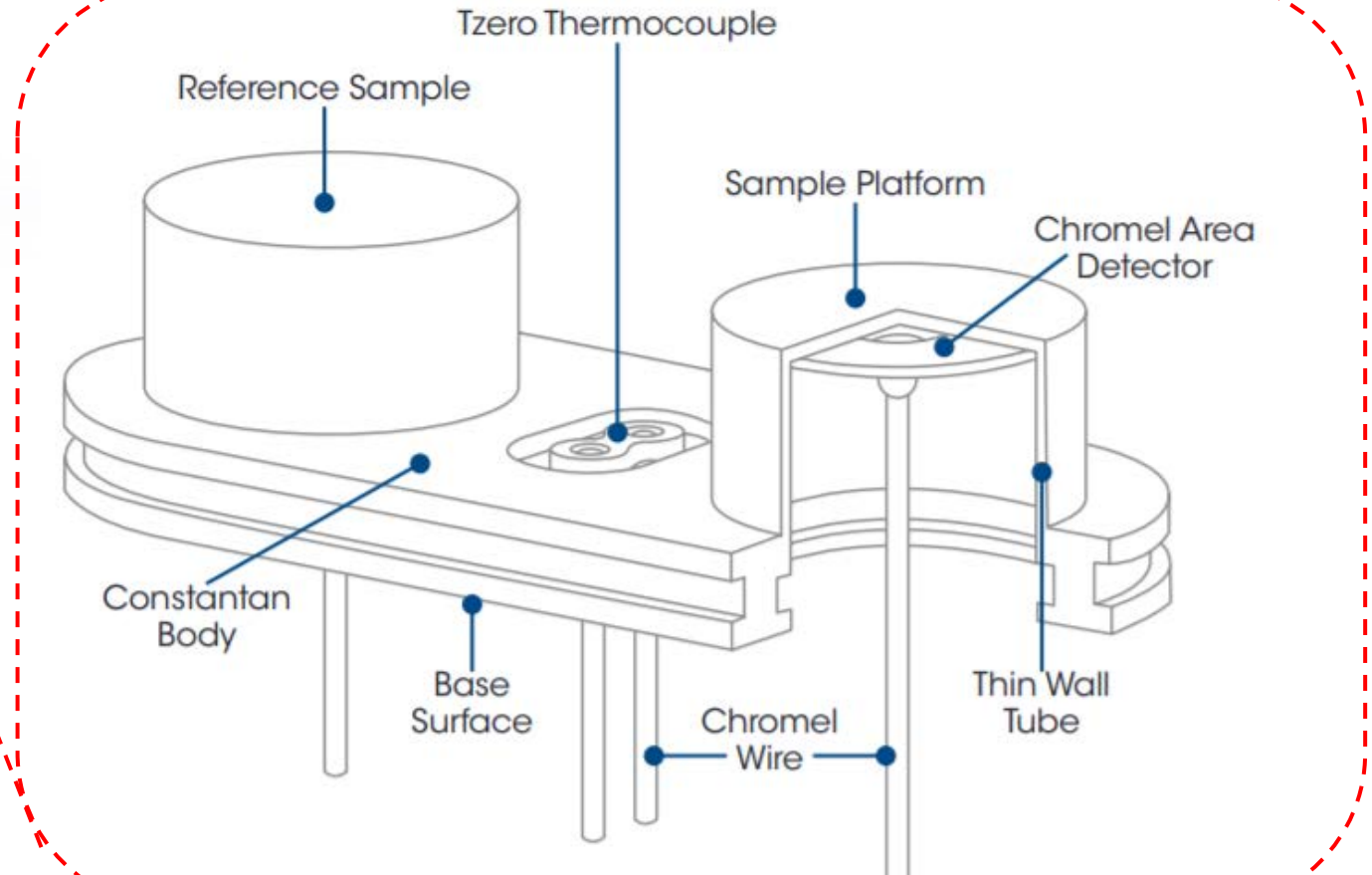
Inside the cell



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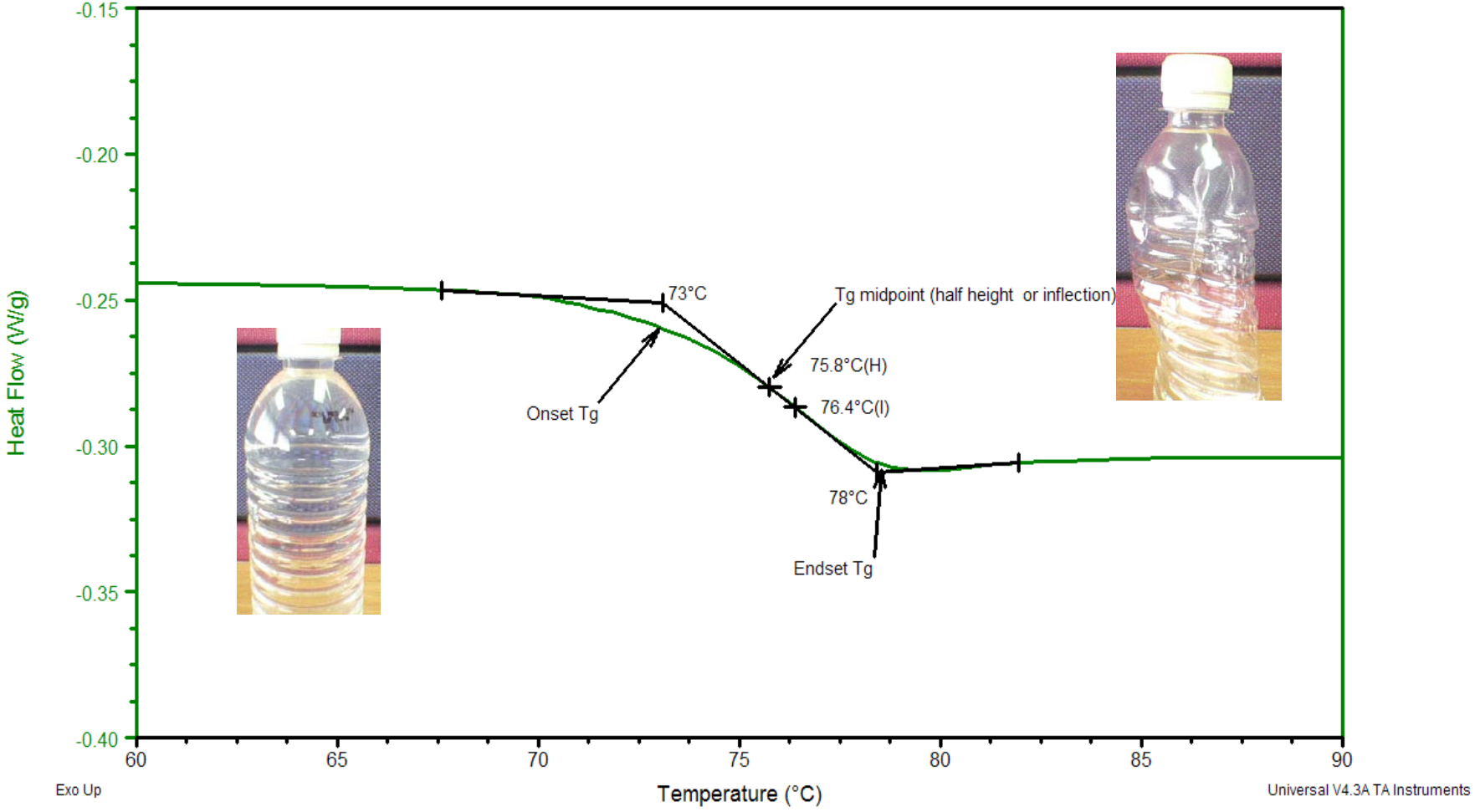
Glass Transition of PET

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

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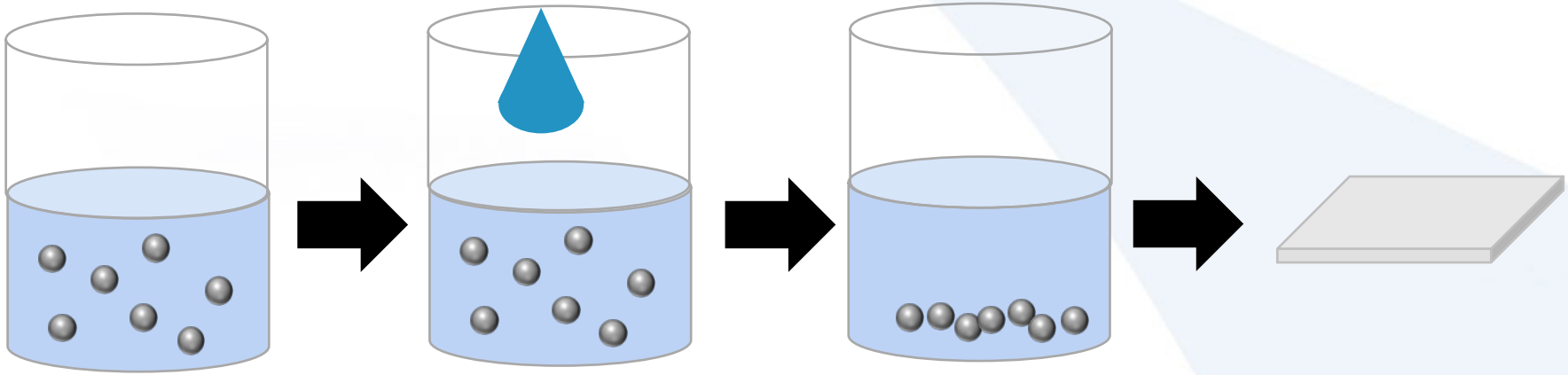
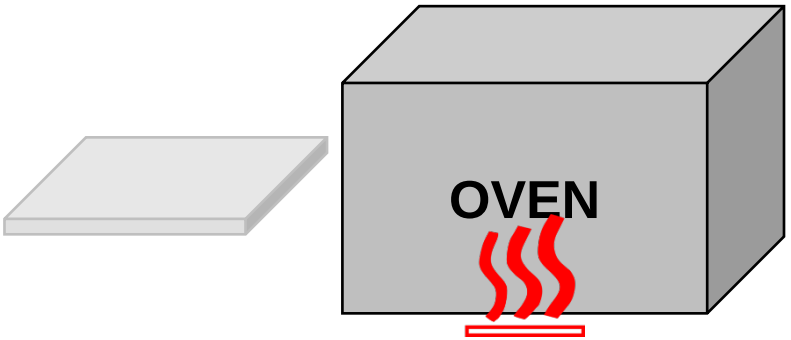
การหาปริมาณเนื้อยางในน้ำยางและในยางแห้ง

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

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

$$\%DRC = \frac{\text{น้ำหนักยางที่แห้งแล้ว x 100\%}}{\text{น้ำหนักก้อนยางสด}}$$





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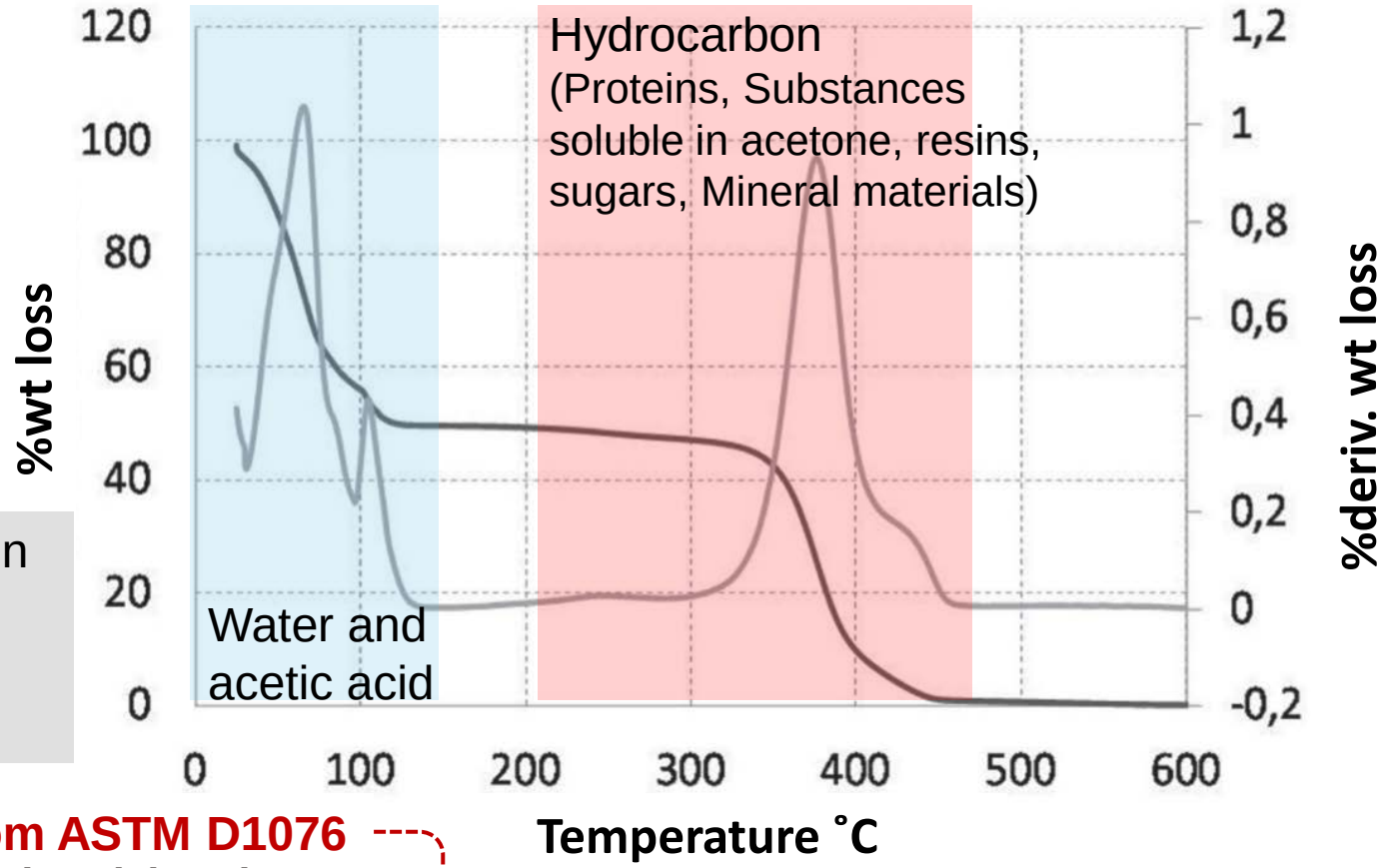
Conventional method vs TGA

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%

rev.ion, 2012;25(2):57-65. Bucaramanga (Colombia)

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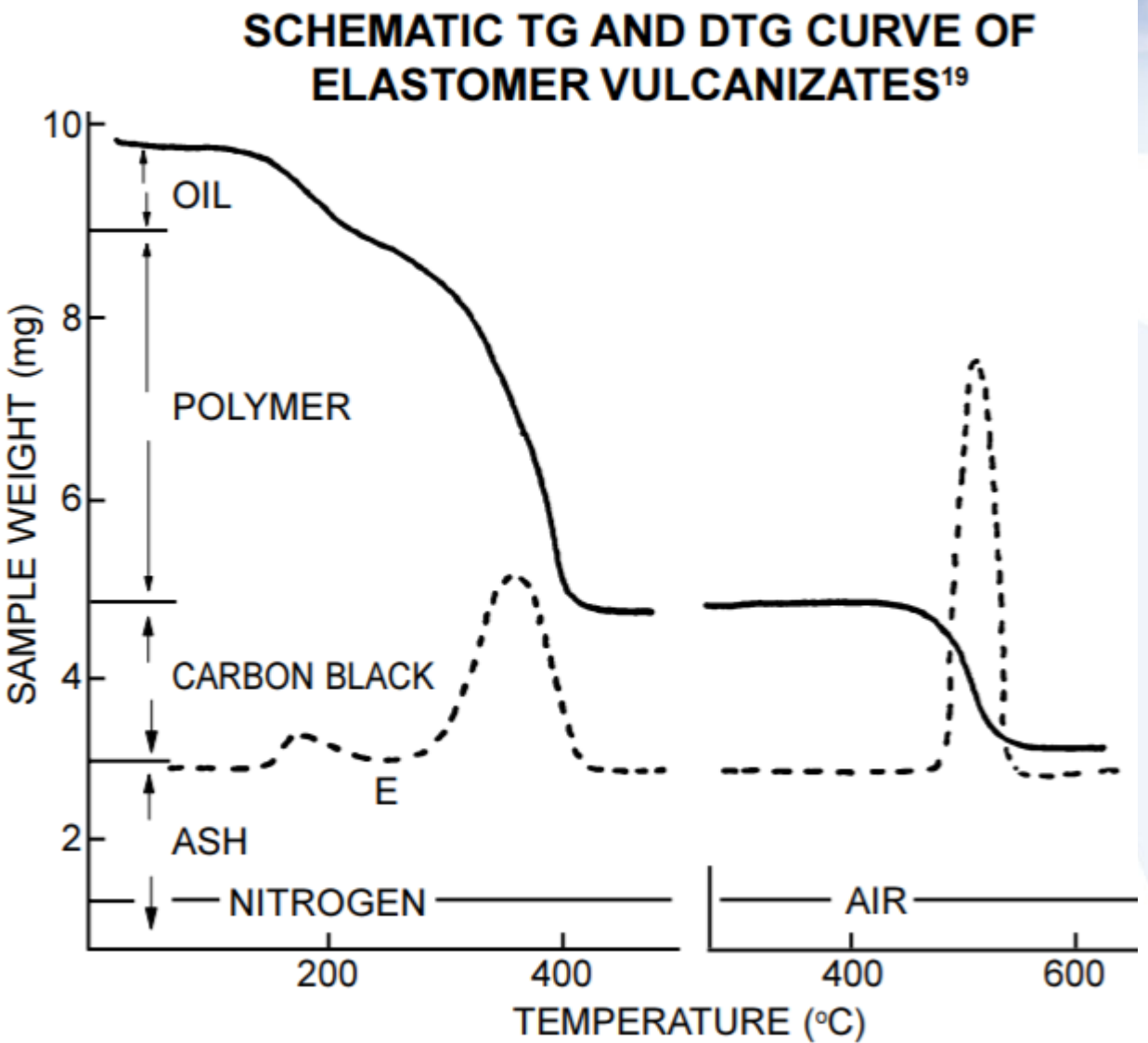
Decomposition of Vulcanizates



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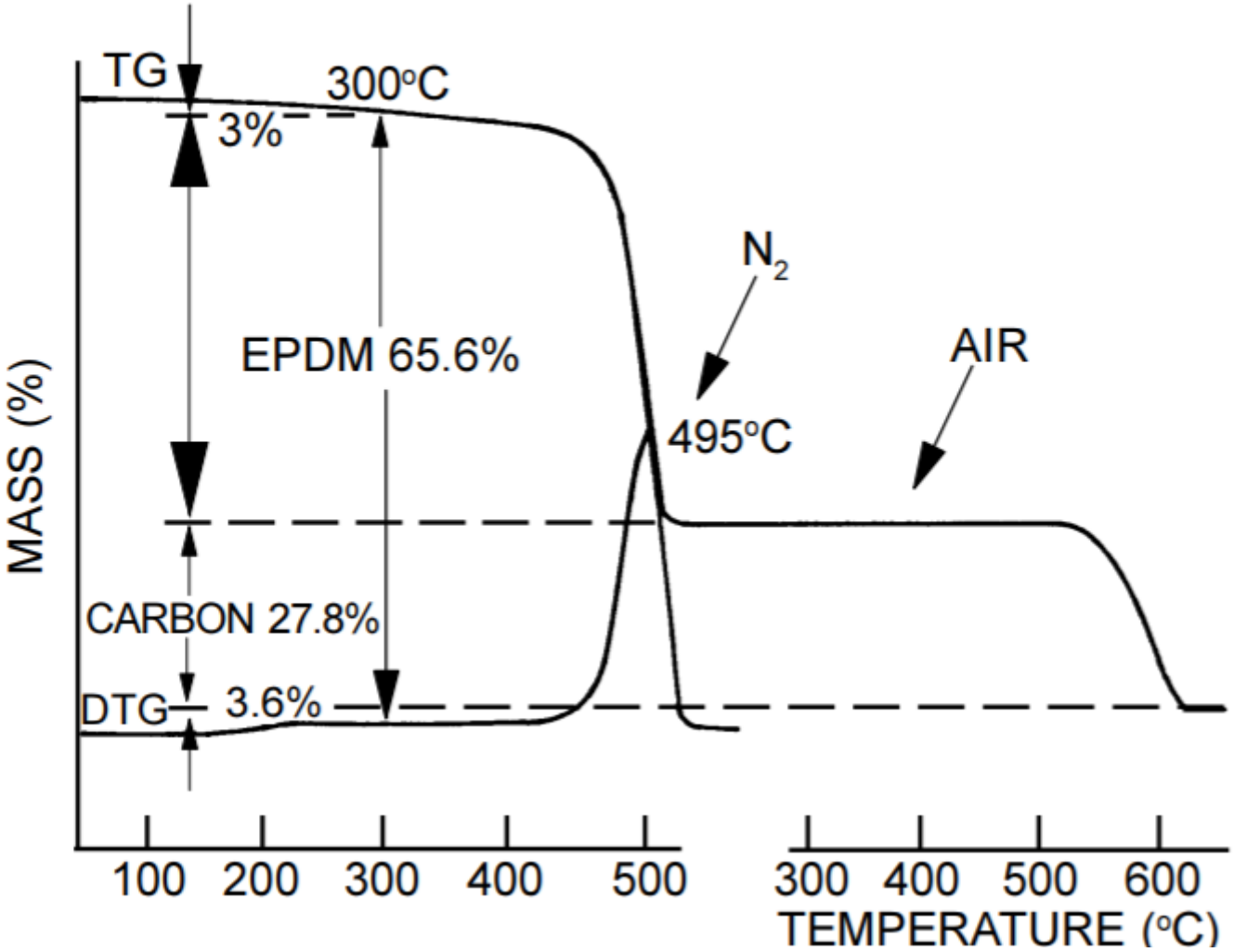
Decomposition of EPR Vulcanizates

TG-DTG CURVES OF EPR VULCANIZATES



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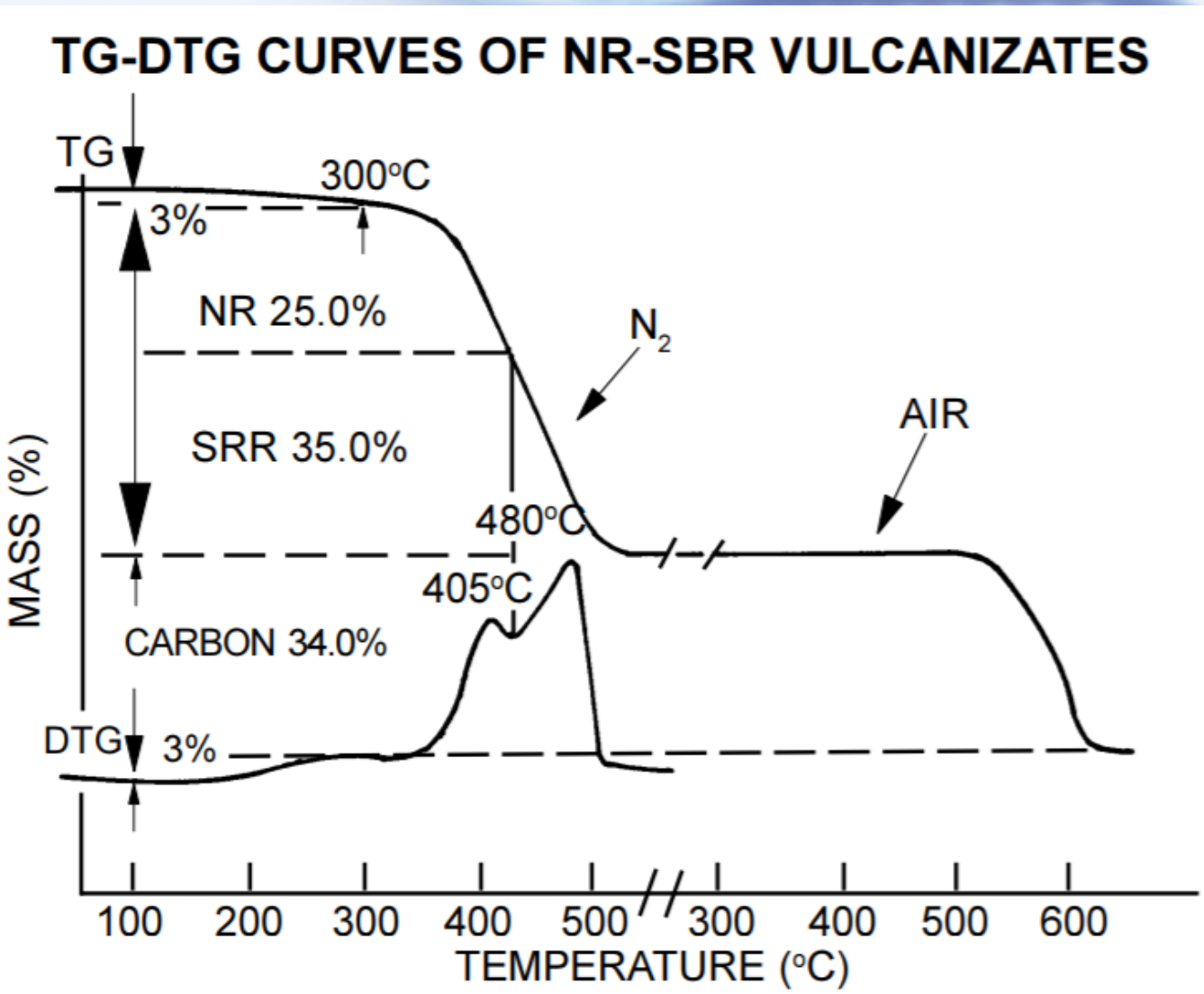
Decomposition of NR-SBR Vulcanizates



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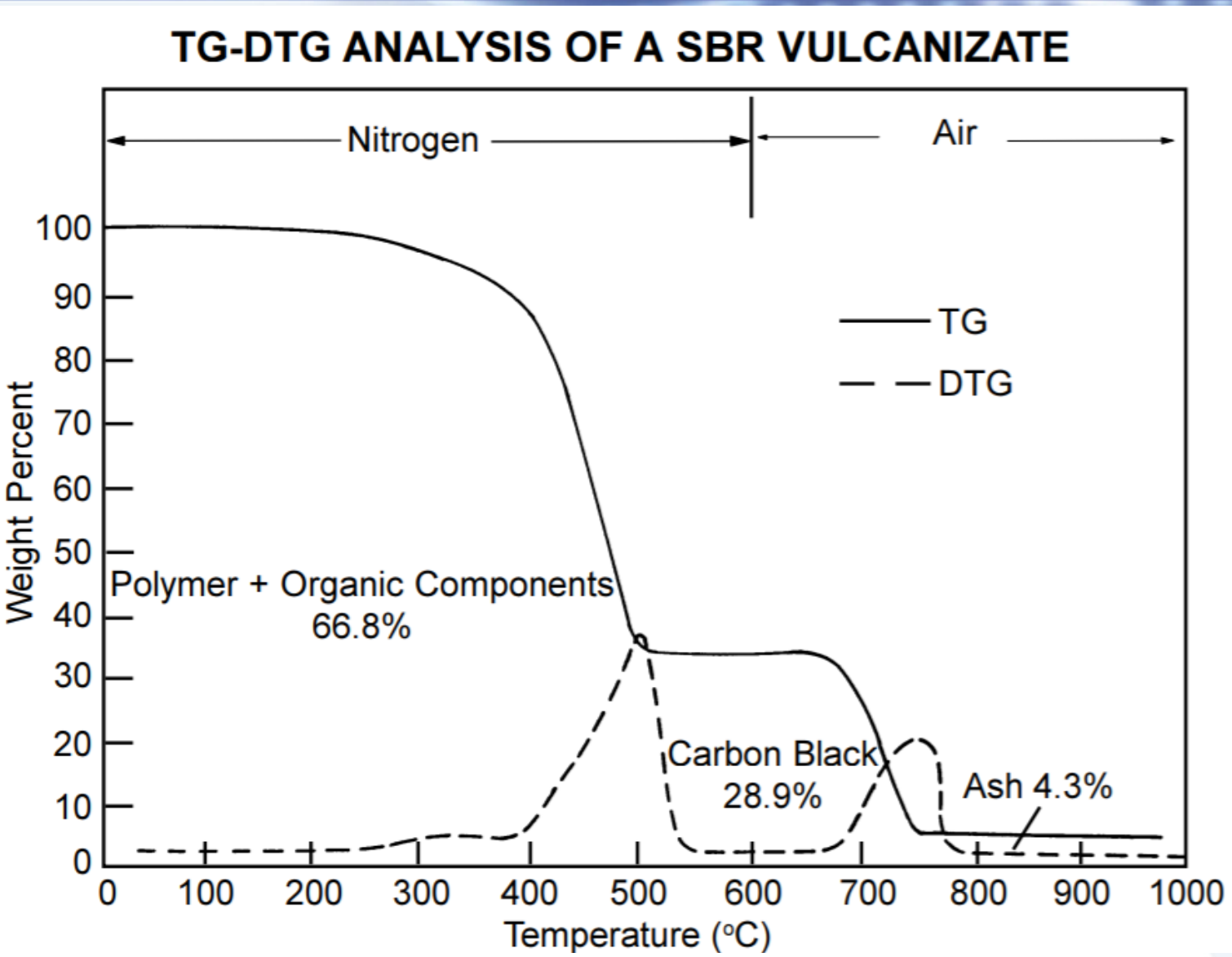
Decomposition of SBR vulcanizate



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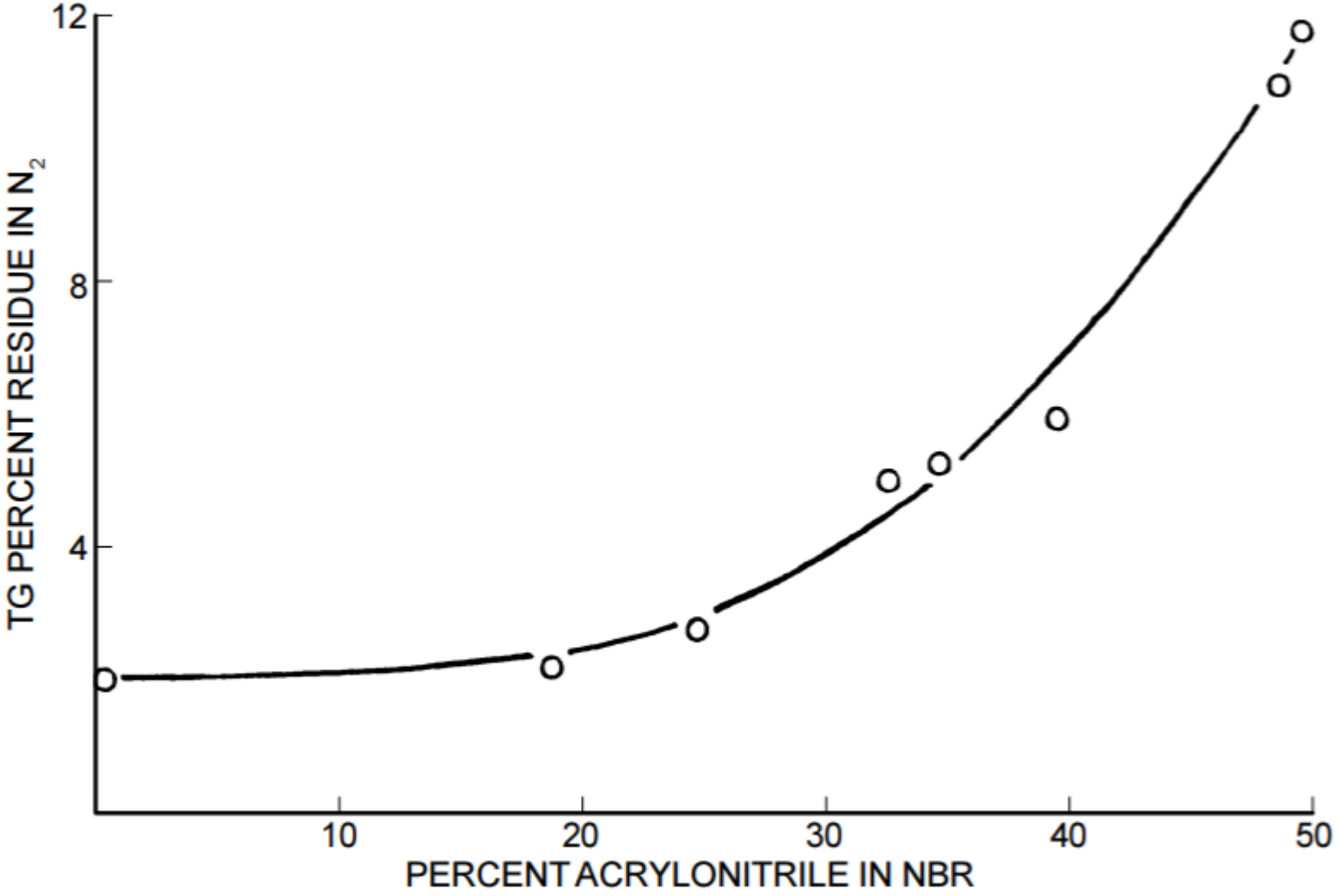
Carbon Residue in N2 with Nitrile Content of NBR Elastomer



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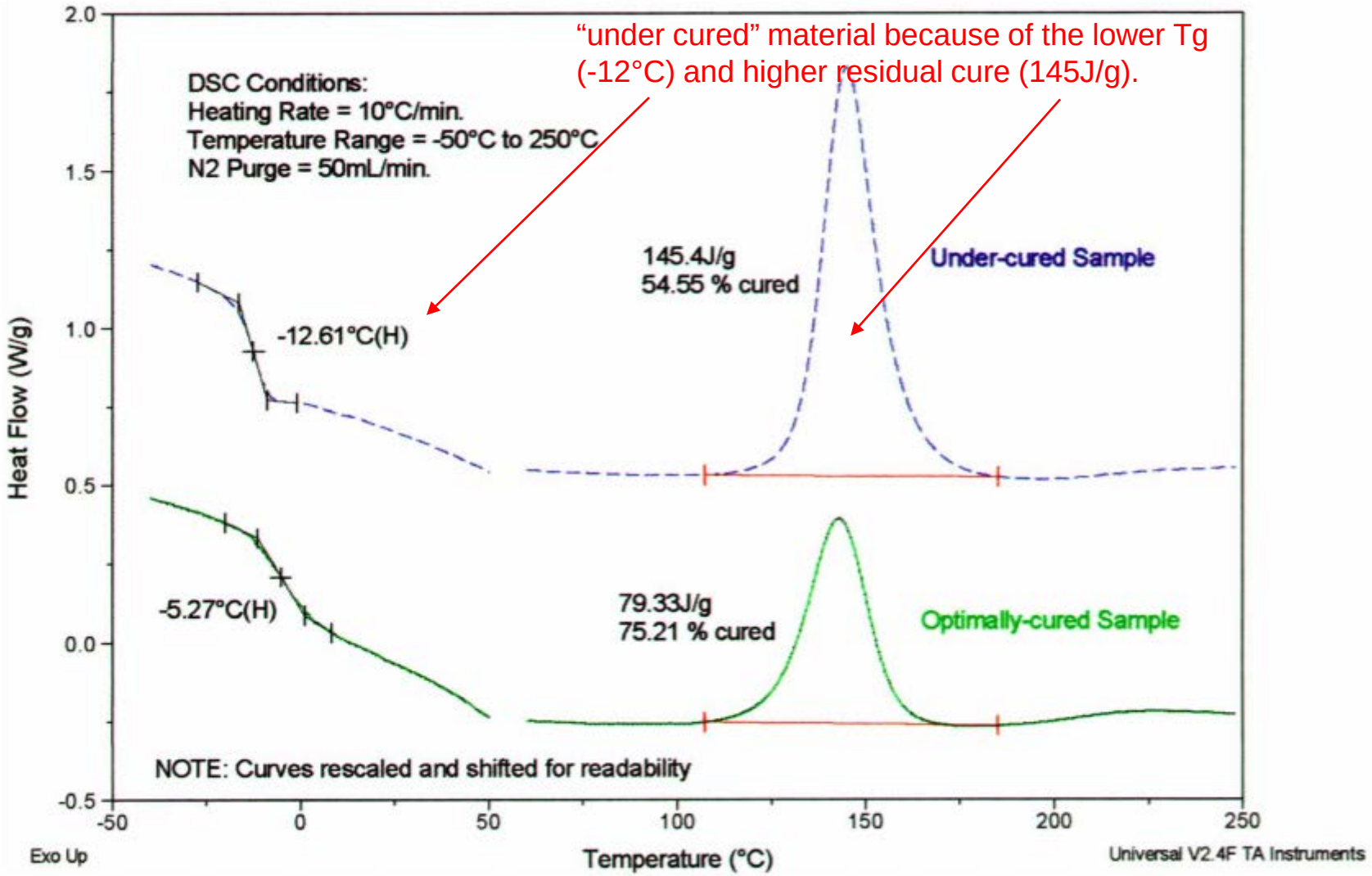
Characterization of the %Cure of Thermosetting Resins by DSC



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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

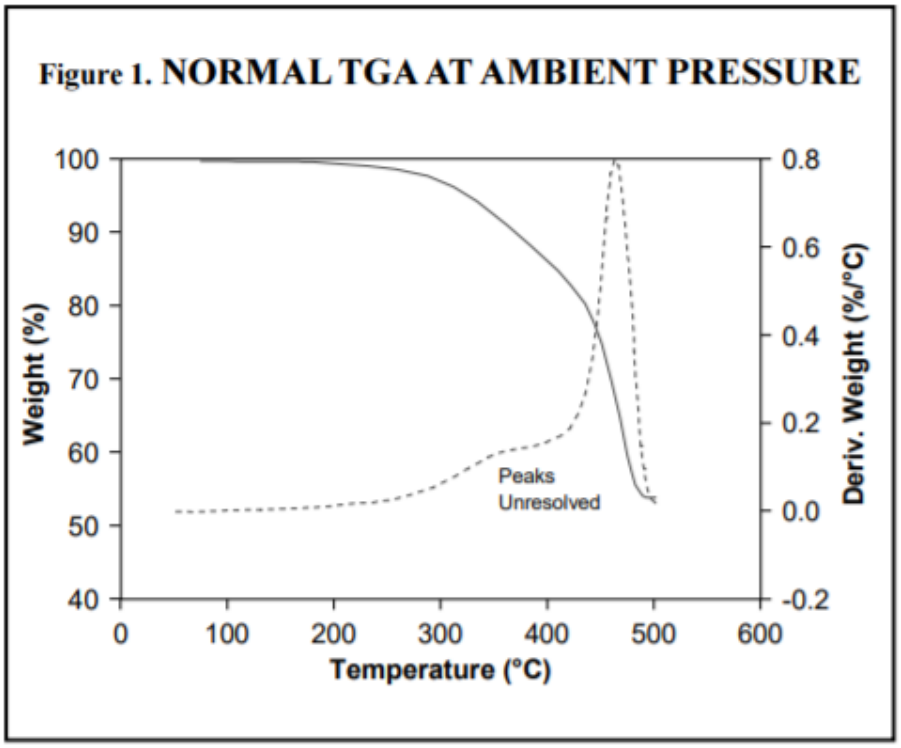
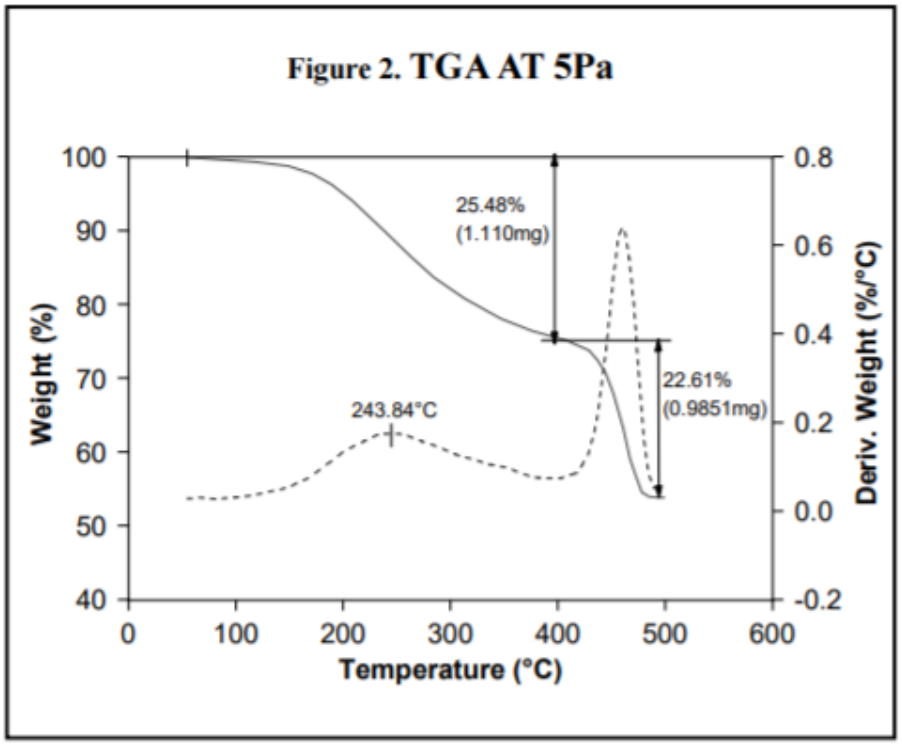


Figure 2. TGA AT 5Pa



Rubber begins to pyrolyze

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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
Using Hi-resolution mode	good result with short test time

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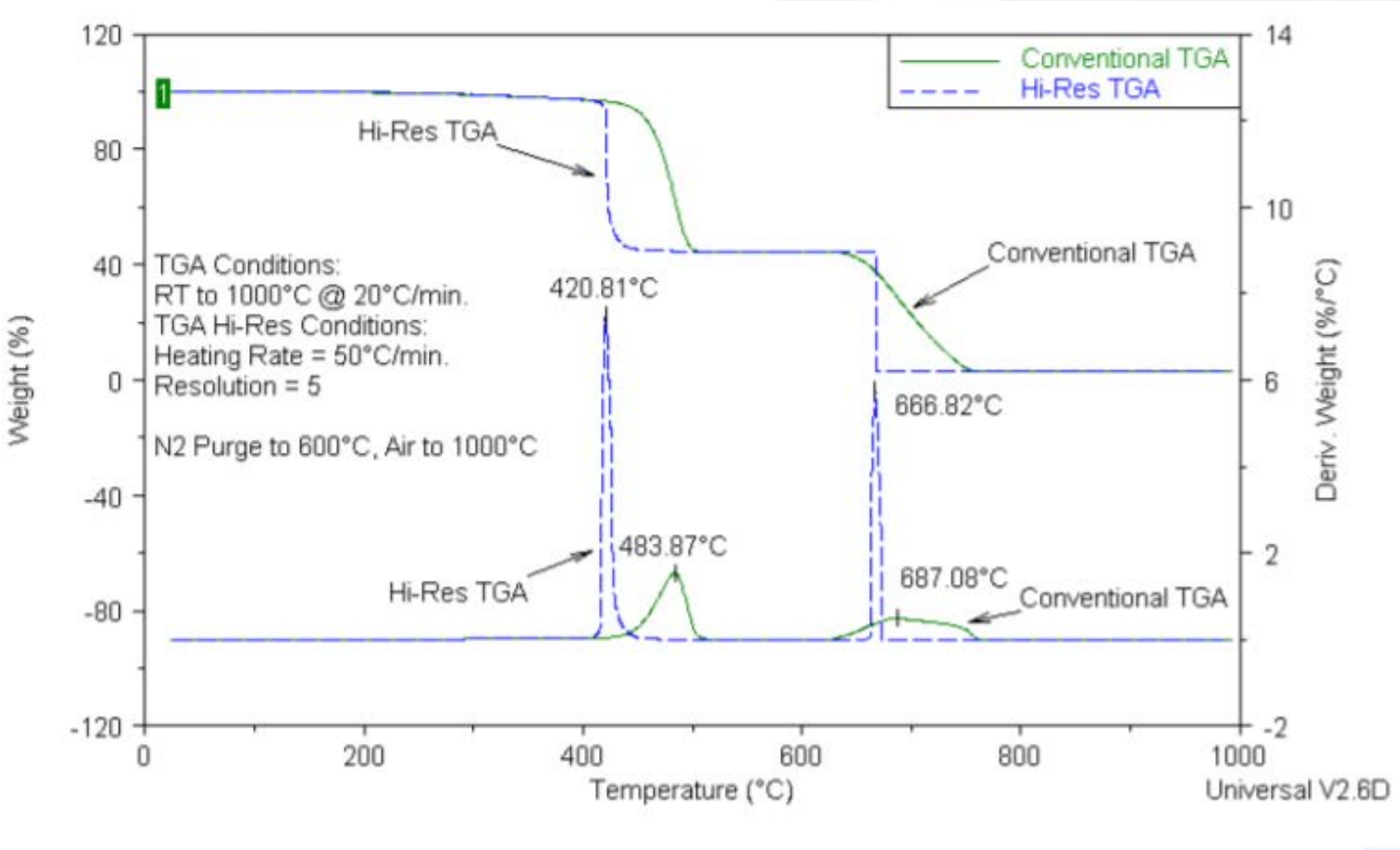
Characterization of EPDM Rubber by TGA and Hi-Res TGA



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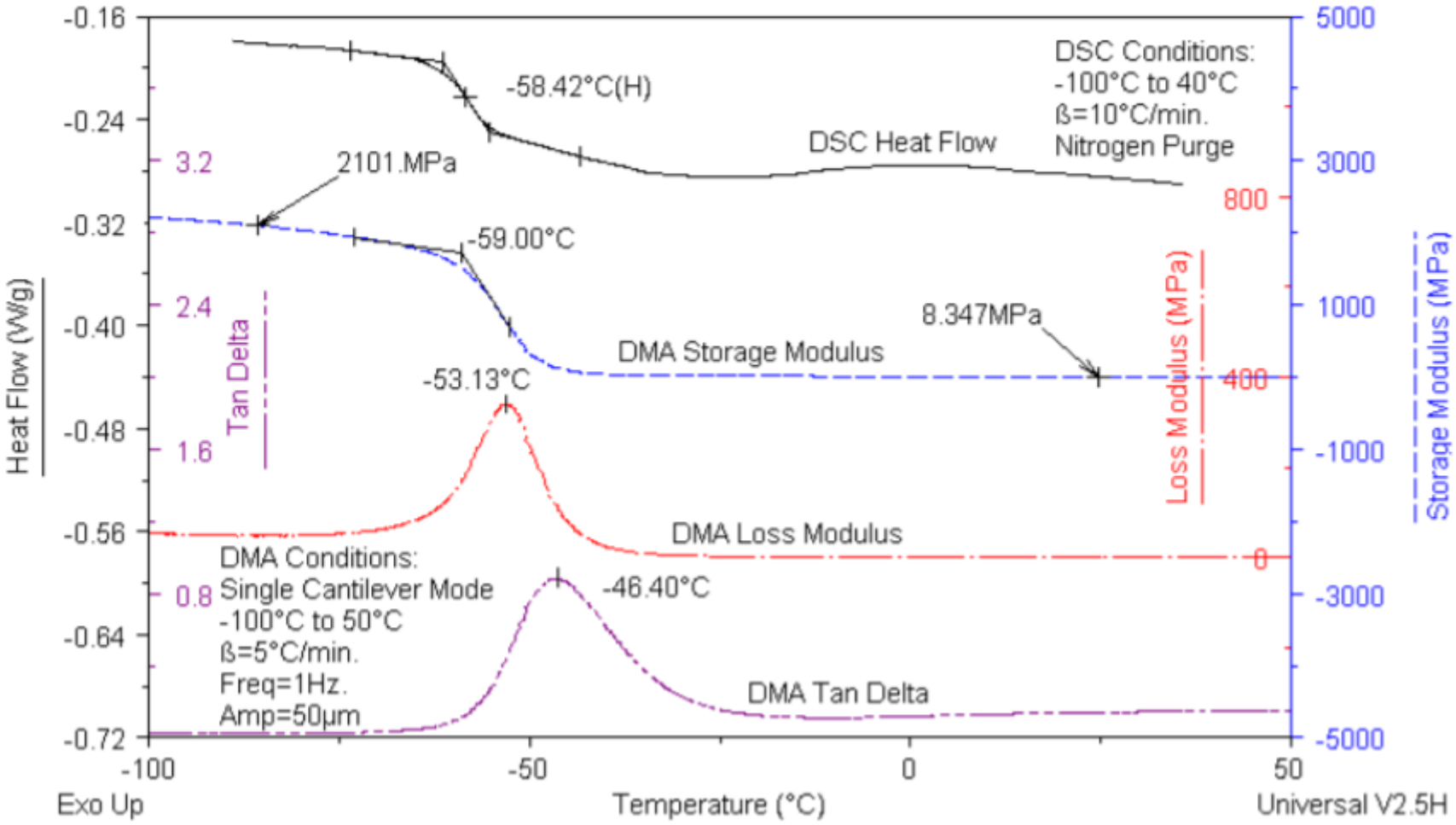
Characterization of EPDM Rubber by DSC and DMA



Easy Analysis by

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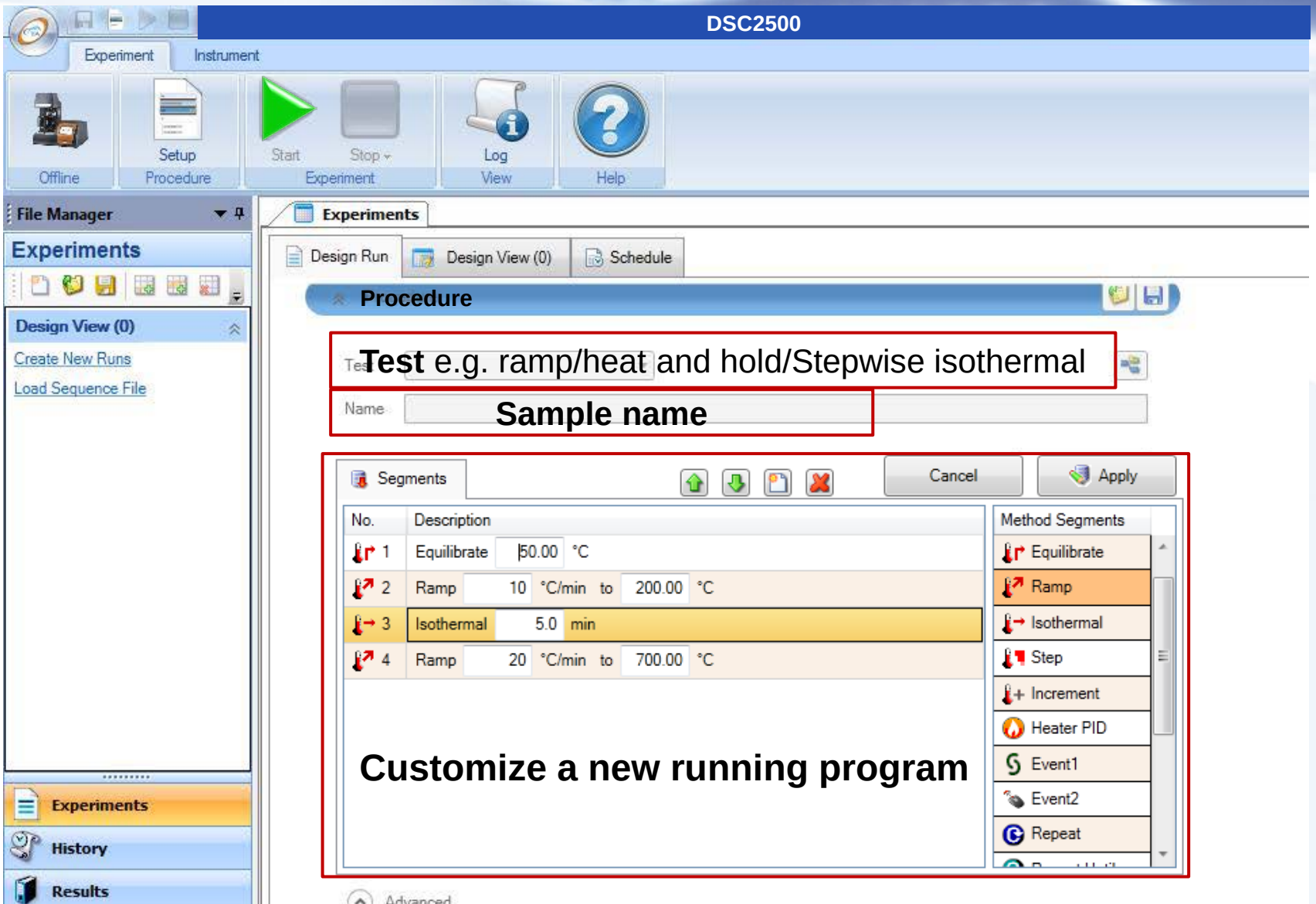
Programing the Run



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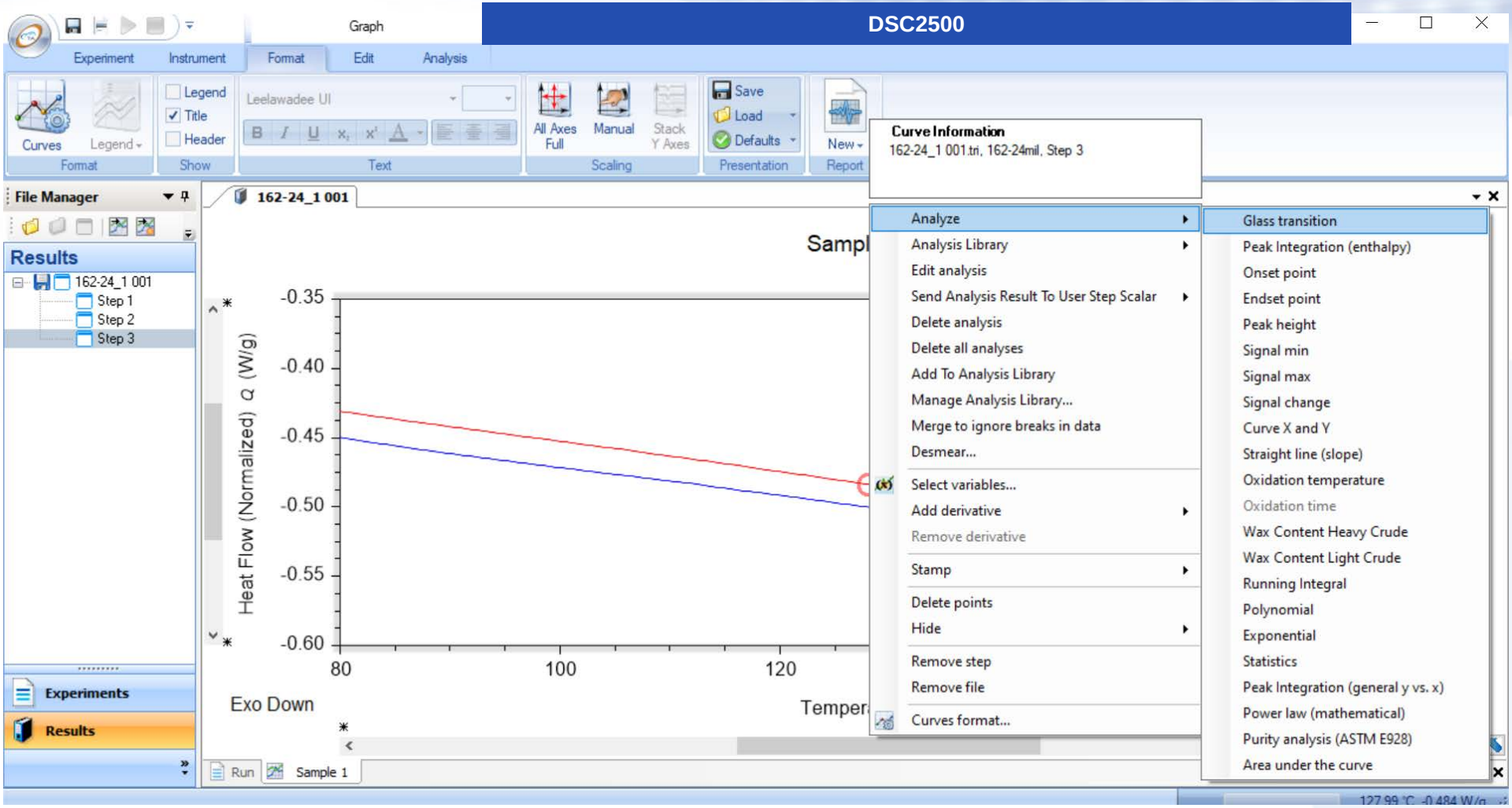
Result Analysis

DSC2500



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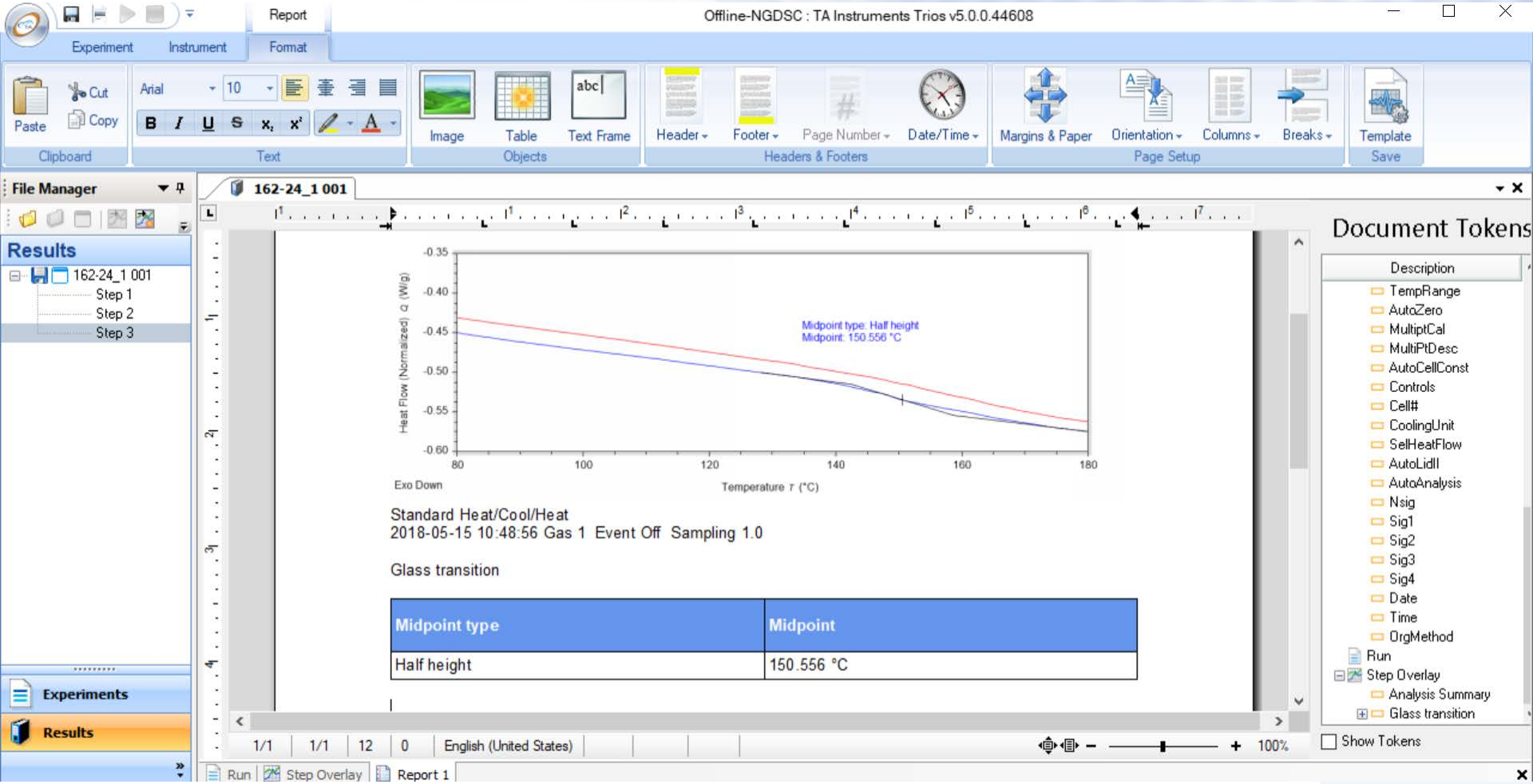
Report Generate



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Q&A

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