

TGA Q Series 操作訓練

2013-8-2

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TGA Q Series 操作訓練說明

- TGA操作軟體
- TGA機台操作與機台校正
- 如何做一個簡單樣品實驗
- 分析軟體(簡易示範)
- 平時保養注意事項

TGA 機型介紹

TGA Q50



TGAQ500



TGAQ5000



Q500/Q5000 控制方式



Standby	Run 1	37.95°C
Control status		
Complete		
Control command		
Furnace	↓	↑
Open	↓	↑
Apply		
Start	Stop	Control
Display	Calibrate	



Q50 控制方式



開機前注意事項

□ 開氮氣、空氣, 出口壓力15~20 psi



□ 注意 heat exchanger 水位與管線流暢度

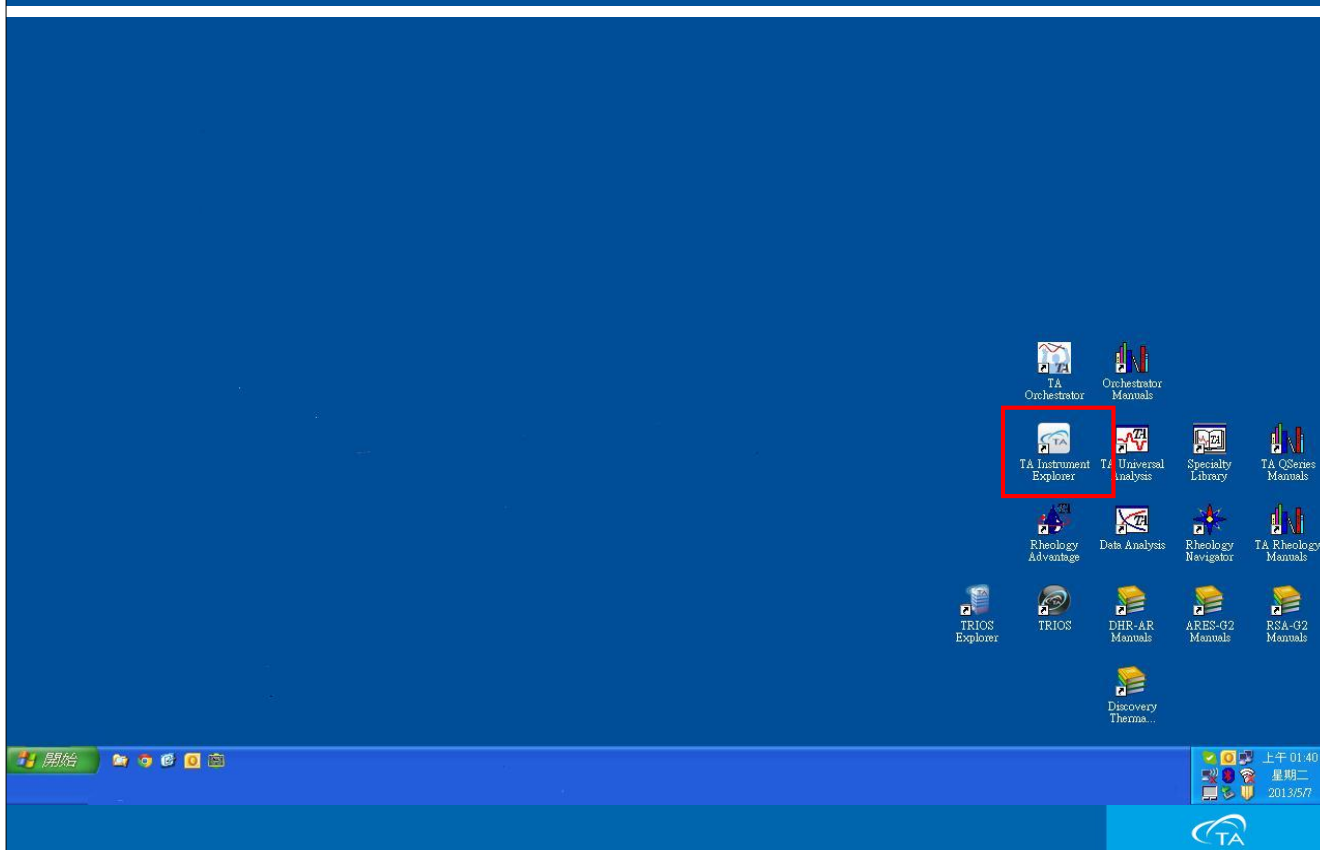


TGA 開機順序

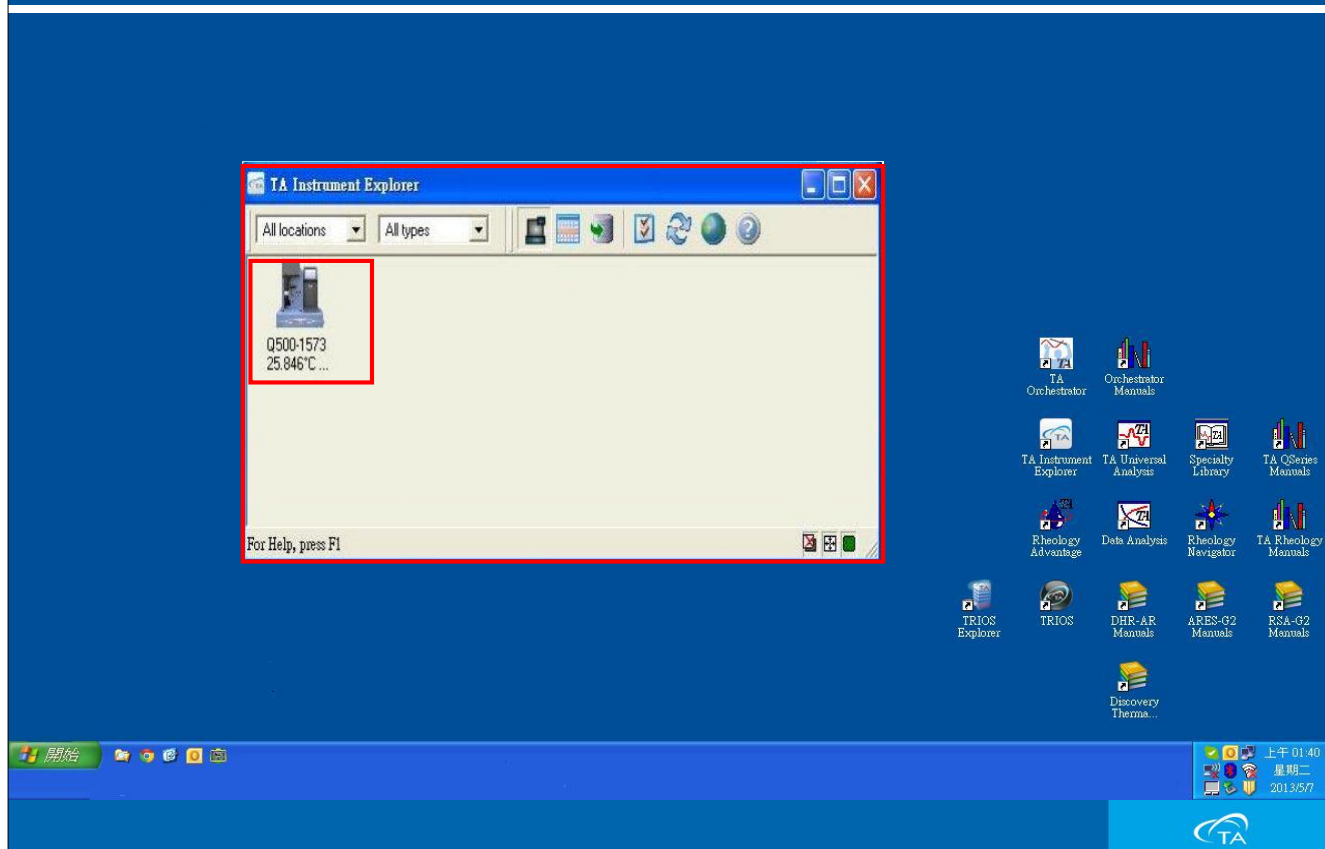
- 開電腦
- 開TGA電源,約兩分鐘後,Ready燈亮或LCD螢幕出現TA圖樣
- 執行 Instrument Explorer
- 點選TGA圖示,開啟連線控制視窗



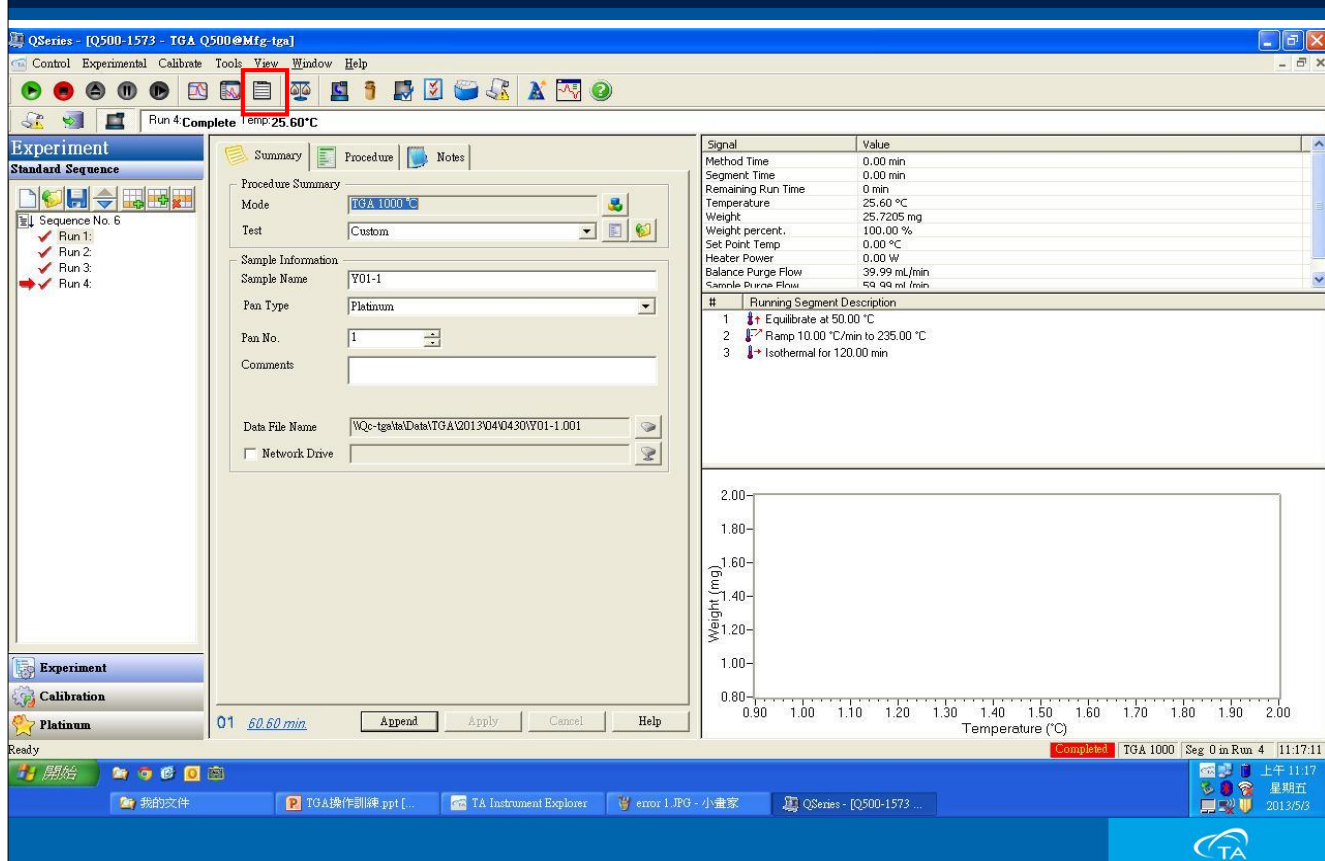
TGA操作軟體介面說明



TGA操作軟體介面說明



TGA操作軟體介面說明



TGA操作軟體介面說明

QSeries - [Q500-1573 - TGA Q500@Mfg.tga]

Control Experimental Calibrate Tools View Window Help

Run 4 Complete Temp: 25.60°C

Experiment

Standard Sequence

Sequence No. 6

Run 1: ✓
Run 2: ✓
Run 3: ✓
Run 4: ✓

Procedure Summary

Mode: TGA 1000 °C

Test: Custom

Sample Information

Sample Name: Y01-1

Pen Type: Platinum

Pen No.: 1

Comments:

Data File Name: WQc-tga\data\TGA\2013\04\0430\Y01-1.001

☐ Network Drive

01 60.60 min

Append Apply Cancel Help

Signal	Value
Method Time	0.00 min
Segment Time	0.00 min
Remaining Run Time	0 min
Temperature	25.60 °C
Weight	25.7205 mg
Weight percent	100.00 %
Set Point Temp	0.00 °C
Heater Power	0.00 W
Balance Purge Flow	39.99 mL/min
Sample Purge Flow	50.00 mL/min

#	Running Segment Description
1	Equilibrate at 50.00 °C
2	Ramp 10.00 °C/min to 235.00 °C
3	Isothermal for 120.00 min

Weight (mg)

Temperature (°C)

Completed TGA 1000 Seg 0 in Run 4 11:17:11

Ready

開始 我的文件 TGA操作訓練 ppt TA Instrument Explorer error 1.JPG 小畫家 QSeries - [Q500-1573 ...]

上午 11:17 星期五 2013/5/3

TA

即時訊號

TGA操作軟體介面說明

QSeries - [Q500-1573 - TGA Q500@Mfg.tga]

Control Experimental Calibrate Tools View Window Help

Run 4 Complete Temp: 25.60°C

Experiment

Standard Sequence

Sequence No. 6

Run 1: ✓
Run 2: ✓
Run 3: ✓
Run 4: ✓

Procedure Summary

Mode: TGA 1000 °C

Test: Custom

Sample Information

Sample Name: Y01-1

Pen Type: Platinum

Pen No.: 1

Comments:

Data File Name: WQc-tga\data\TGA\2013\04\0430\Y01-1.001

☐ Network Drive

01 60.60 min

Append Apply Cancel Help

Signal	Value
Method Time	0.00 min
Segment Time	0.00 min
Remaining Run Time	0 min
Temperature	25.60 °C
Weight	25.7205 mg
Weight percent	100.00 %
Set Point Temp	0.00 °C
Heater Power	0.00 W
Balance Purge Flow	39.99 mL/min
Sample Purge Flow	50.00 mL/min

#	Running Segment Description
1	Equilibrate at 50.00 °C
2	Ramp 10.00 °C/min to 235.00 °C
3	Isothermal for 120.00 min

Weight (mg)

Temperature (°C)

Completed TGA 1000 Seg 0 in Run 4 11:17:11

Ready

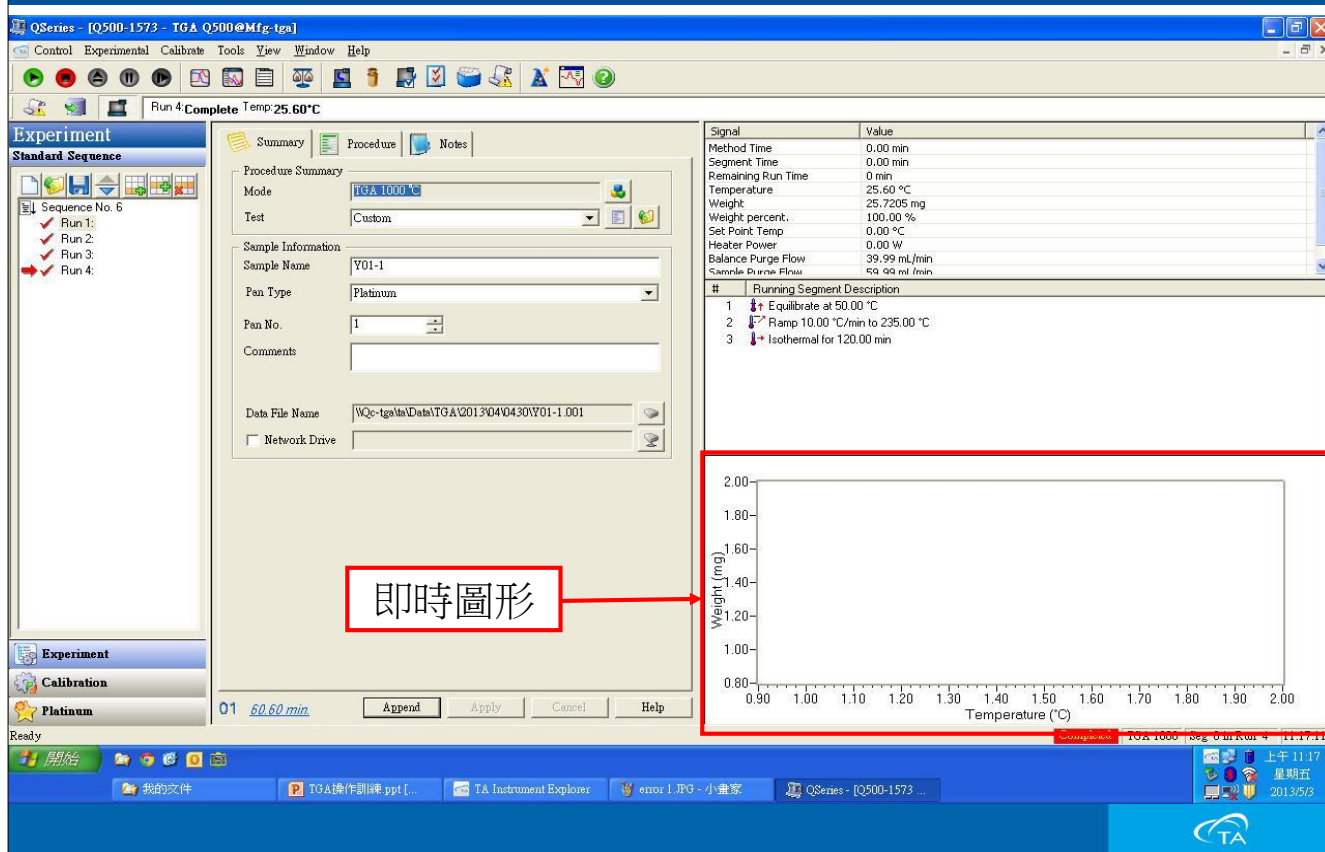
開始 我的文件 TGA操作訓練 ppt TA Instrument Explorer error 1.JPG 小畫家 QSeries - [Q500-1573 ...]

上午 11:17 星期五 2013/5/3

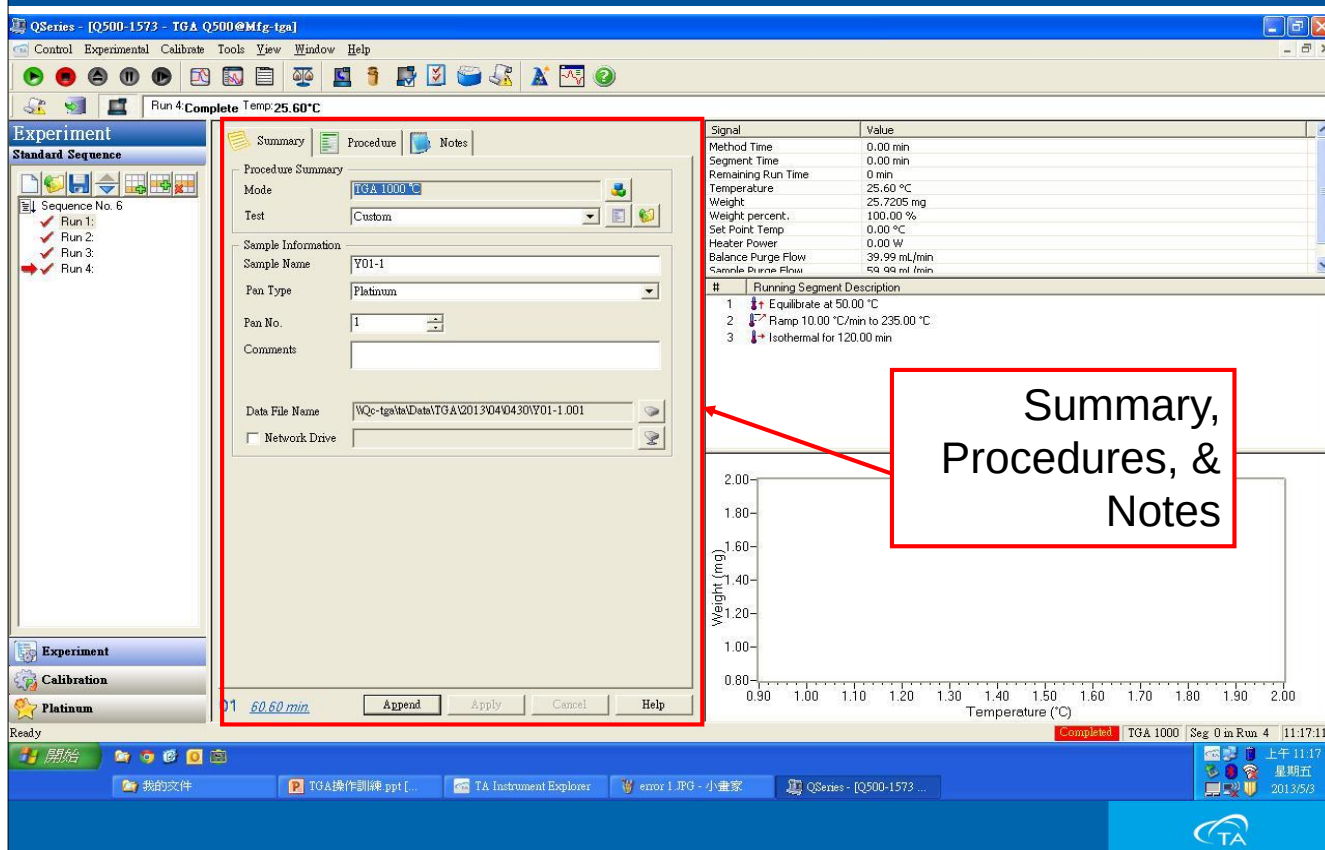
TA

實驗條件

TGA操作軟體介面說明



TGA操作軟體介面說明



TGA操作軟體介面說明

QSeries - [Q500-1573 - TGA Q500@Mfg-tga]

Control Experimental Calibrate Tools View Window Help

Run 4: Complete Temp: 25.60°C

Experiment

Standard Sequence

- Sequence No. 6
- Run 1: ✓
- Run 2: ✓
- Run 3: ✓
- Run 4: ✓

Experiment

Calibration

Platinum

Procedure Summary

Mode: TGA 1000 °C

Test: Custom

Sample Information

Sample Name: Y01-1

Pan Type: Platinum

Pan No.: 1

Comments:

Data File Name: WQc-tga\data\TGA\2013\04\0430\Y01-1.001

☐ Network Drive

Append Apply Cancel Help

Signal Value

Signal	Value
Method Time	0.00 min
Segment Time	0.00 min
Remaining Run Time	0 min
Temperature	25.60 °C
Weight	25.7205 mg
Weight percent.	100.00 %
Set Point Temp	0.00 °C
Heater Power	0.00 W
Balance Purge Flow	39.99 mL/min
Sample Purge Flow	50.00 mL/min

Running Segment Description

#	Running Segment Description
1	↑ Equilibrate at 50.00 °C
2	↑ Ramp 10.00 °C/min to 235.00 °C
3	↑ Isothermal for 120.00 min

Weight (mg)

Temperature (°C)

Completed TGA 1000 Seg 0 in Run 4 11:17:11

Ready

我的文件 TGA操作訓練 ppt TA Instrument Explorer error 1.JPG - 小畫家 QSeries - [Q500-1573 ...]

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TGA操作軟體介面說明

QSeries - [Q500-1573 - TGA Q500@Mfg-tga]

Control Experimental Calibrate Tools View Window Help

Run 4: Complete Temp: 25.52°C

Experiment

Standard Sequence

- Sequence No. 6
- Run 1: ✓
- Run 2: ✓
- Run 3: ✓
- Run 4: ✓

Experiment

Calibration

Platinum

Weight (mg)

Temperature (°C)

Signal Value

Signal	Value
Method Time	0.00 min
Segment Time	0.00 min
Remaining Run Time	0 min
Temperature	25.52 °C
Weight	25.7204 mg
Weight percent.	100.00 %
Set Point Temp	0.00 °C
Heater Power	0.00 W
Balance Purge Flow	40.03 mL/min
Sample Purge Flow	60.00 mL/min

Running Segment Description

#	Running Segment Description
1	↑ Equilibrate at 50.00 °C
2	↑ Ramp 10.00 °C/min to 235.00 °C
3	↑ Isothermal for 120.00 min

Completed TGA 1000 Seg 0 in Run 4 11:20:00

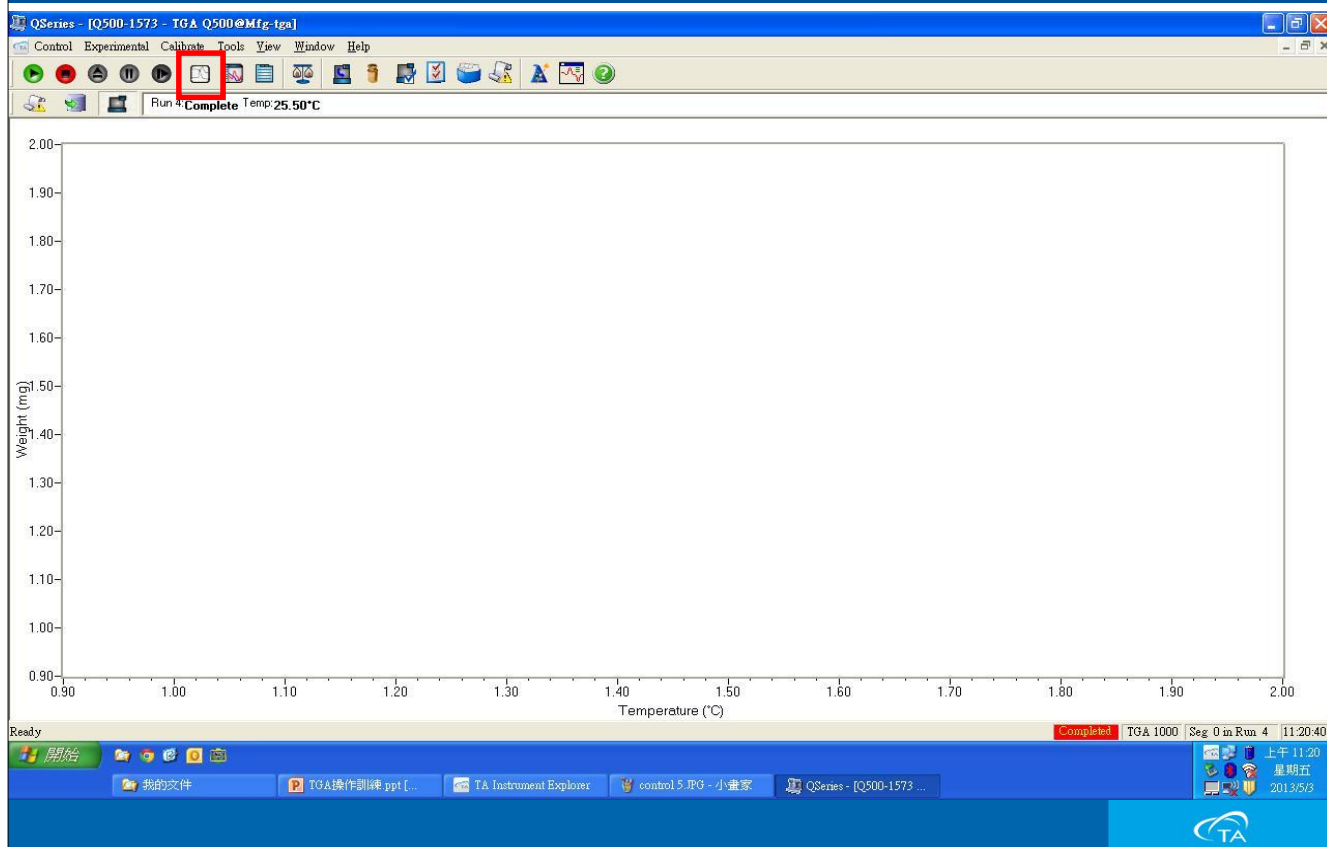
Ready

開始 我的文件 TGA操作訓練 ppt TA Instrument Explorer control 4.JPG - 小畫家 QSeries - [Q500-1573 ...]

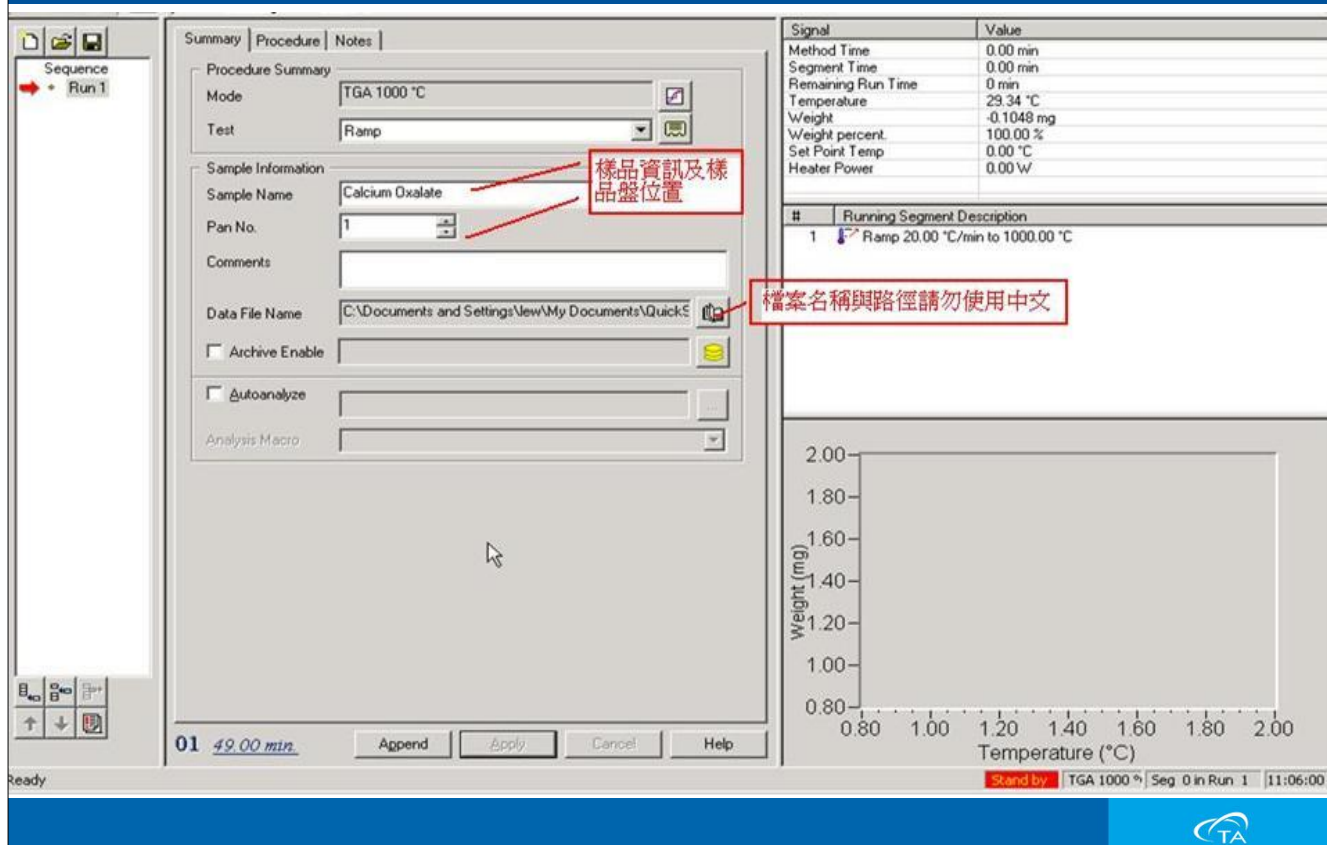
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TGA操作軟體介面說明



TGA操作軟體介面說明



TGA操作軟體介面說明

The screenshot shows the QSeries - [Q500-1573 - TGA Q500@Mfg.tga] window. The 'Experiment' tab is active, displaying a 'Standard Sequence' with four runs. The 'Method' editor is open, showing a 'Custom' test with two segments: '1 Equilibrate at 50.00 °C' and '2 Ramp 10.00 °C/min to 600.00 °C'. The 'Signal' table on the right lists various parameters like Method Time, Segment Time, Remaining Run Time, Temperature, Weight, Weight percent, Set Point Temp, Heater Power, Balance Purge Flow, and Sample Purge Flow. A graph at the bottom right shows Weight (mg) vs Temperature (°C) with a red line indicating the current run.

Signal	Value
Method Time	0.00 min
Segment Time	0.00 min
Remaining Run Time	0 min
Temperature	25.58 °C
Weight	25.7204 mg
Weight percent	100.00 %
Set Point Temp	0.00 °C
Heater Power	0.00 W
Balance Purge Flow	39.97 mL/min
Sample Purge Flow	50.00 mL/min

TGA操作軟體介面說明

The screenshot shows the 'Method' editor with a 'Method Contents' dialog box open. The dialog box displays a 'Segment list' with various options: Jump, Equilibrate, Initial temperature, Ramp, Isothermal, Step, Increment temperature, Repeat, Repeat until, Abort next segment on limit, and Sampling interval. The 'Ramp' option is selected. The 'Method Contents' dialog box also shows a 'Name' field with 'Ramp' and a 'Segment Description' table with two segments: '1 Ramp 20.00 °C/min to 500.00 °C' and '2 Select gas 2'. The 'OK' button is highlighted.

Signal	Value
Method Time	0.00 min
Segment Time	0.00 min
Remaining Run Time	0 min
Temperature	27.23 °C
Weight	-71.7221 mg
Weight percent	100.00 %

實驗條件編輯

TGA操作軟體介面說明

The screenshot shows the TGA software interface with the 'Notes' tab selected. A red box highlights the 'Mass Flow Control Settings' section, which includes fields for 'Balance', 'Sample', 'Flow Rate', and 'Auto Analyze'. A red arrow points to the 'Operator' field with the text '操作者氣體流速設定' (Operator Gas Flow Rate Setting). The 'Running Segment Description' table is also visible.

Signal	Value
Method Time	0.00 min
Segment Time	0.00 min
Remaining Run Time	0 min
Temperature	25.57 °C
Weight	25.7205 mg
Weight percent	100.00 %
Set Point Temp	0.00 °C
Heater Power	0.00 W
Balance Purge Flow	39.99 mL/min
Sample Purge Flow	60.03 mL/min

#	Running Segment Description
1	Equilibrate at 50.00 °C
2	Ramp 10.00 °C/min to 235.00 °C
3	Isothermal for 120.00 min

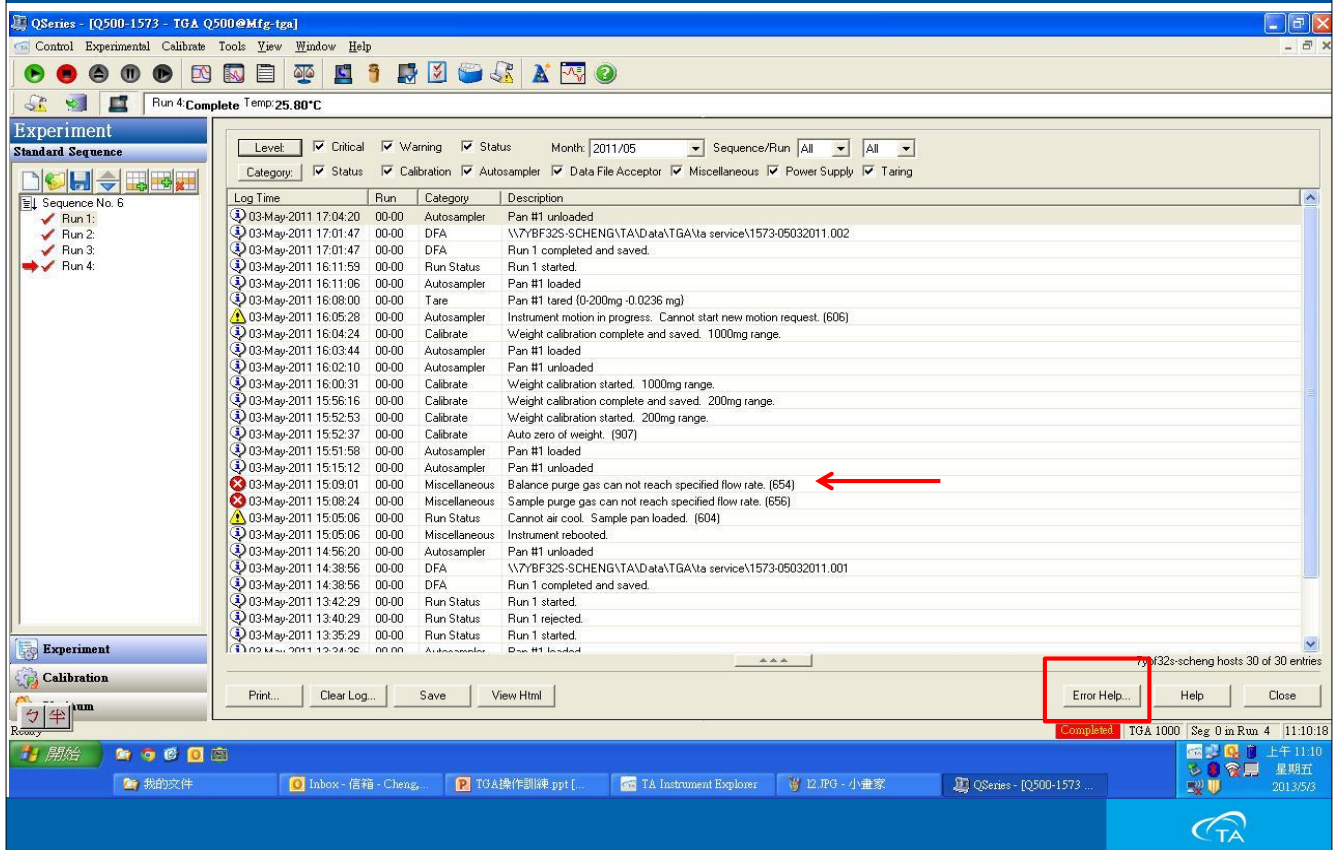
Weight (mg) vs Temperature (°C) graph showing a baseline at 2.00 mg.

TGA操作軟體介面說明

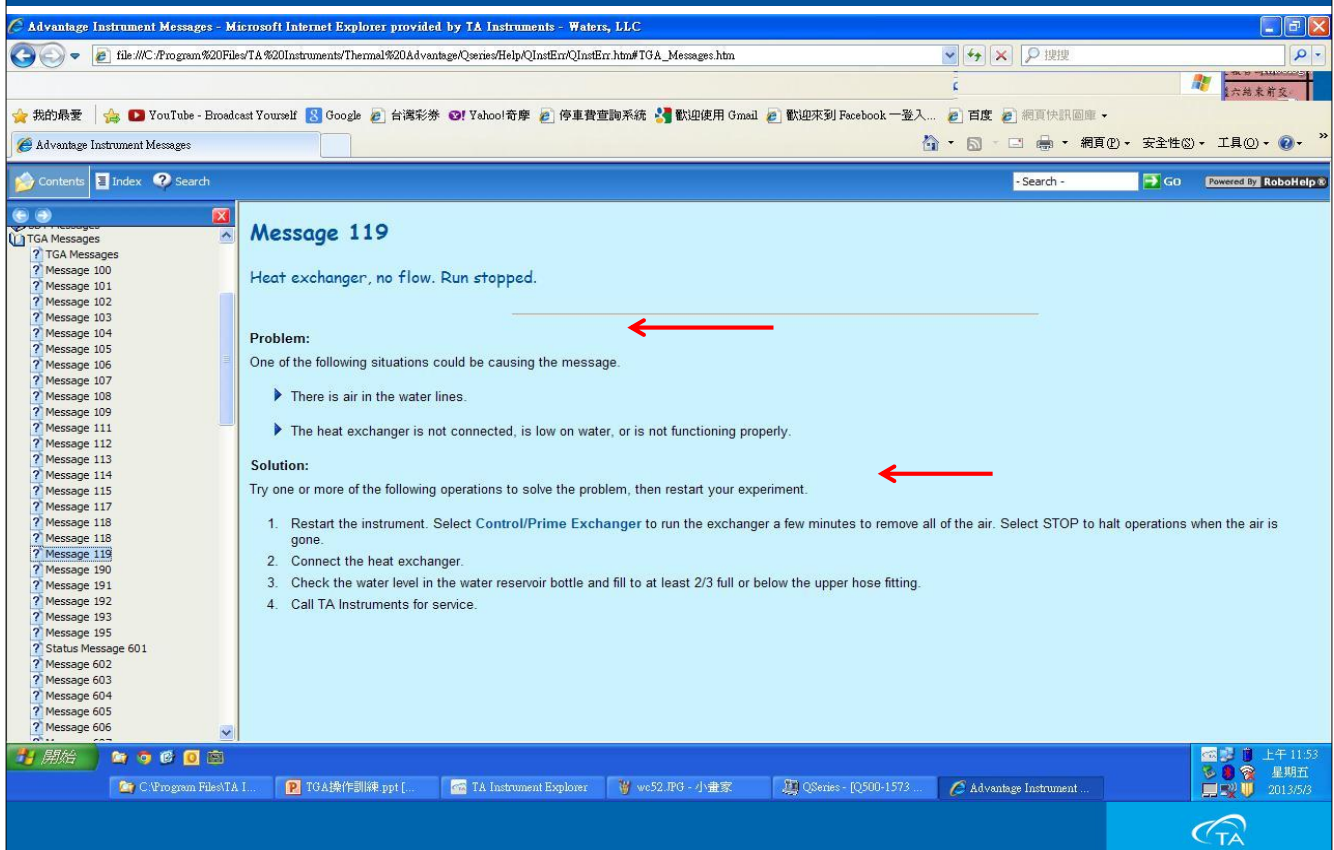
The screenshot shows the TGA software interface with the 'Instrument Preferences' dialog box open. The 'MFC Purge' tab is selected, showing settings for 'Gas #1' and 'Gas #2', both set to 'Nitrogen'. A red box highlights the 'Gas #1' and 'Gas #2' dropdown menus, with a red arrow pointing to them and the text 'GAS1/GAS2的氣體種類設定' (Gas1/Gas2 Gas Type Setting). The 'Stop experiment when flow rate deviates from the set value' checkbox is checked.

GAS1/GAS2的氣體種類設定

TGA操作軟體介面說明



TGA操作軟體介面說明



實驗氣體流速設定

□ For EGA furnace

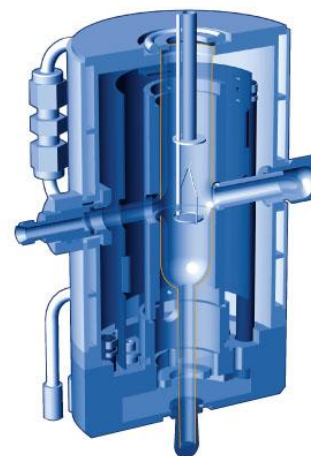
□ 90 ml/min 爐子

□ 10 ml/min 天平

□ For Q5000 furnace

□ 25 ml/min 爐子

□ 10 ml/min 天平



TGA校正操作說明

□ 重量校正

ASTM International Standard E2040 Standard Test Method for Mass Scale Calibration of Thermogravimetric Analyzers

ASTM International Standard E617 Class 1 Tolerance (100 mg Mass)

OIML R111 Class E2 Tolerance (1 g Mass)

標準砝碼 100mg / 1000mg

□ 溫度校正

Curie Point Transition Method

ASTM 1582 - Standard Practice for Calibration of Temperature Scale for Thermogravimetry

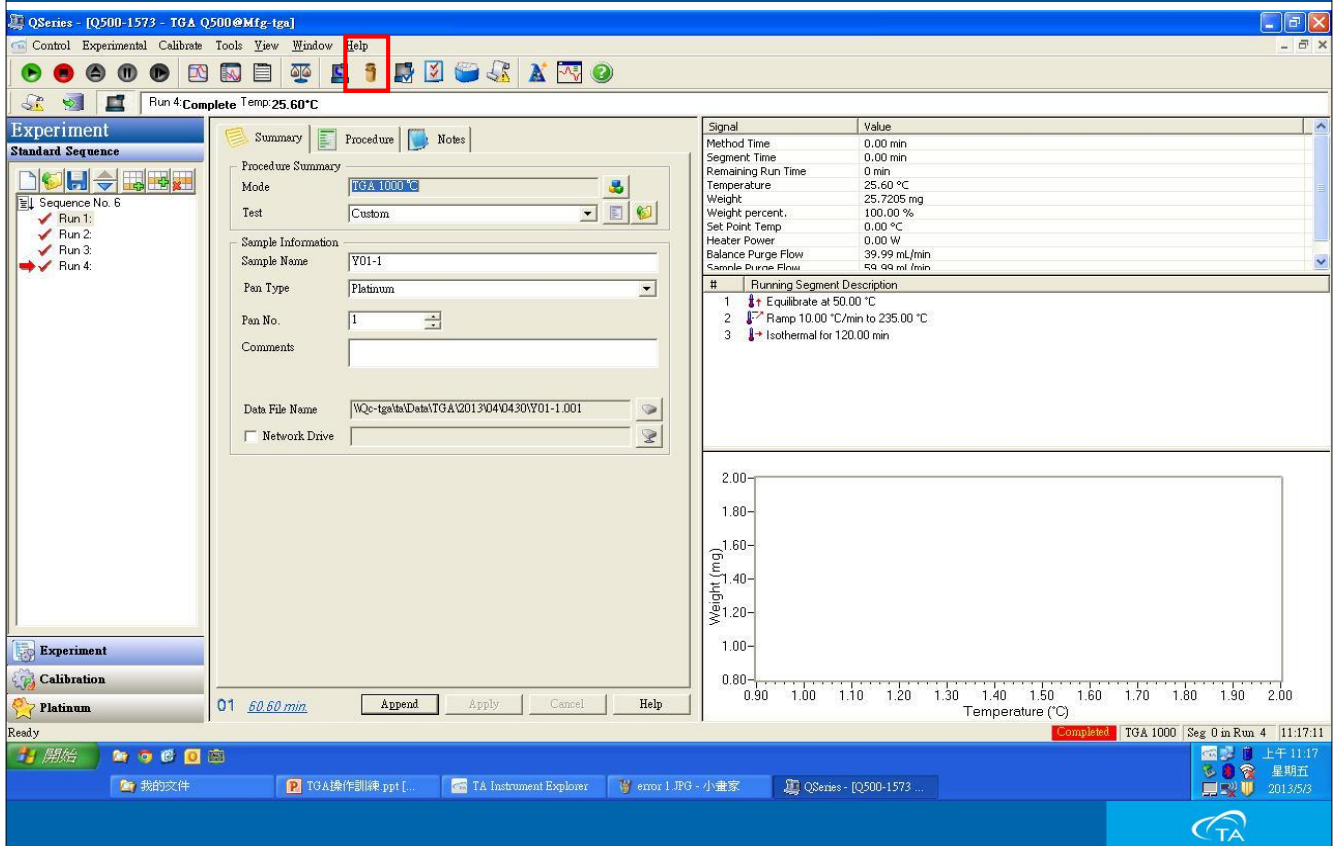


重量校正

- 步驟一：Electronic Balance Tare
 - 步驟二：200mg範圍歸零
 - 步驟三：200mg範圍校正(使用真實100mg砝碼)
 - 步驟四：1000mg範圍歸零
 - 步驟五1000mg範圍校正(使用真實1000mg砝碼)
- 請使用砝碼夾及乾淨的白金盤



重量校正



重量校正

QSeries - [Q500-1573 - TGA Q500@Mfg-tga]

Control Experimental Calibrate Tools View Window Help

Run 4: Closing Temp: 24.02°C

Experiment
Standard Sequence

Sequence No. 6
Run 1: ✓
Run 2: ✓
Run 3: ✓
Run 4: *

Procedure Summary
Mode: TGA 1000 °C
Test: Custom

Sample Information
Sample Name: Y01-2
Pan Type: Platinum
Pan No.: 4
Comments:
Data File Name: \VQc-tga\data\...
☐ Network Drive

Signal Value
Method Time: 0.00 min
Segment Time: 0.00 min
Remaining Run Time: 0 min
Temperature: 24.02 °C
Weight: 5.6874 mg
Weight percent: 100.00 %

Q500-1573 - TGA Q500@Mfg-tga - Weight Calibration - step 1 of 5

Electronic Balance Tare

1) Place an emptied pan, of the same type and size as the sample, on the tare side of the balance.
2) Press Load Pan to load an empty sample pan on the sample side of the balance and close the furnace.
3) Press Next to begin electronic tare

Load Pan
Unload Pan

Next > Cancel Help

Electronic Balance Tare

Weight (mg)
Temperature (°C)

04 144.10 min Append Apply Cancel Help

Ready Stand by TGA 1000 Seg 0 in Run 4 11:35:36

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上午 11:35 星期五 2013/5/3

TA

重量校正

Q500-0022 - TGA Q500@Tga Mfg - Weight Calibration - step 2 of 5

Standby

Zero 200 mg Range

1) Electronic tare complete. Press Next to begin zeroing 200mg range.

200mg範圍歸零

Next > Cancel Help

TA

重量校正

Q500-0022 - TGA Q500@Tga Mfg - Weight Calibration - step 2 of 5



Standby

Zero 200 mg Range

- ✓ 1) Electronic tare complete. Press Next to begin zeroing 200mg range.
- 2) Wait until the balance is stable.

Uncalibrated Weight 0.0058

Stable

0 1 2 3 4 5 6 7 8 9 10

Stability (ug/sec)

200mg範圍歸零

< Back

Accept >

Cancel

Help



重量校正

Q500-0022 - TGA Q500@Tga Mfg - Weight Calibration - step 3 of 5



Standby

Calibrate the 200 mg Range

- 1) Press Unload Pan to unload the sample pan.
- 2) Place a 100 mg class M weight in the sample pan.
- 3) Press Load Pan to load the sample pan.
- 4) Enter the exact mass of the standard mg.

Load Pan

Unload Pan

請放100mg砝碼

Next >

Cancel

Help



重量校正

Q500-0022 - TGA Q500@Tga Mfg - Weight Calibration - step 3 of 5



Standby

Calibrate the 200 mg Range

- ✓ 1) Press Unload Pan to unload the sample pan.
- ✓ 2) Place a 100 mg class M weight in the sample pan.
- ✓ 3) Press Load Pan to load the sample pan.
- ✓ 4) Enter the exact mass of the standard mg.
- 5) Wait until the balance is stable.

Uncalibrated Weight 99.9882 Stable

0 1 2 3 4 5 6 7 8 9 10

Stability (ug/sec)

200mg範圍校正

< Back Accept > Cancel Help

TA

重量校正

Q500-0022 - TGA Q500@Tga Mfg - Weight Calibration - step 4 of 5



Standby

Zero 1000 mg Range

- 1) Press Unload Pan to unload the sample pan.
- 2) Remove the weight from the sample pan.
- 3) Press Load Pan to load the sample pan.

1000mg範圍歸零

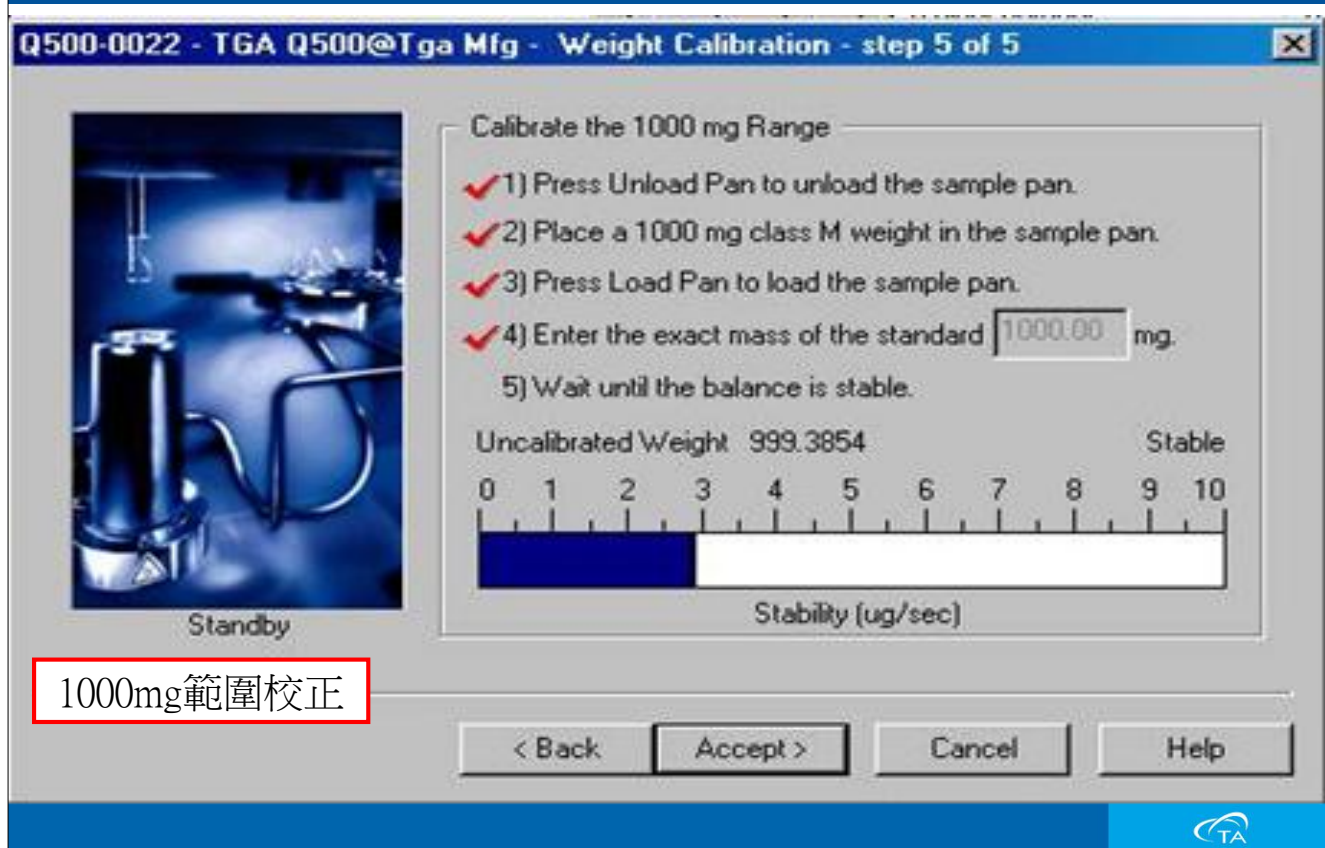
Load Pan

Unload Pan

Next > Cancel Help

TA

重量校正



重量校正



溫度校正

- 實驗條件
 - 平衡的起始溫度設定在標準物質的起始居禮點之下100°C。
 - 居禮點-100°C = 起始溫度
 - 加熱速率與往後的實驗相同，加熱到居禮點以上約50°C。
 - 居禮點+50°C = 終點溫度
 - 舉例 NICKEL
 - Equilibrate 250
 - Ramp 20°C/min to 400
- 校正實驗前，請先清除 "Temperature Table"。
- 請設定實驗結束後的選項為 "Furnace Closed"。這是預防實驗結束後、爐子自動開啟壓在磁鐵尚且傷害TGA本身。
- 使用支撐物 將磁鐵墊爐子正下方在。

溫度校正

The screenshot shows the TGA Q500 software interface. A red box highlights the 'Temperature Table...' option in the 'Calibrate' menu. Another red box highlights the 'Temperature Table - Q500-0014 - TGA Q500@Demo Lab' dialog box. The dialog box contains a 'Calibration Table' with the following data:

	Observed Temperature °C	Correct Temperature °C
Point 1:	150.22	154.16
Point 2:	355.15	358.28
Point 3:	0.00	0.00
Point 4:	0.00	0.00
Point 5:	0.00	0.00

The 'Reset' button is highlighted with a red box. The background shows a graph of Weight (mg) vs Temperature (°C) and a table of values.

溫度校正

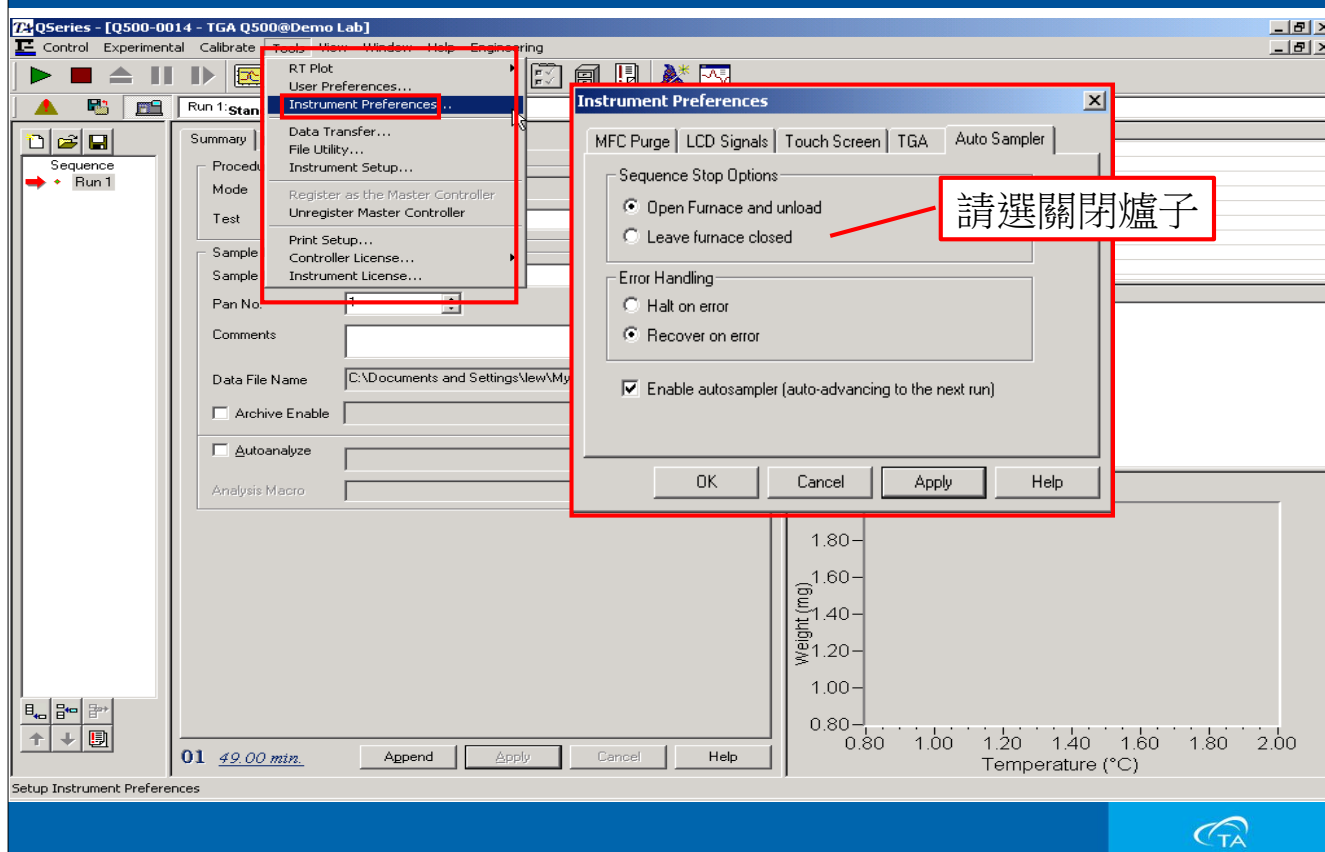
The screenshot shows the TGA Q500 software interface. A red box highlights the 'Post Test Parameters' dialog box. The dialog box contains the following information:

Method End Conditions:

- Furnace: ☐ Open and unload ☒ Closed
- ☒ Air Cool for 20 min

The 'OK' button is highlighted with a red box. The background shows a graph of Weight (mg) vs Temperature (°C) and a table of values.

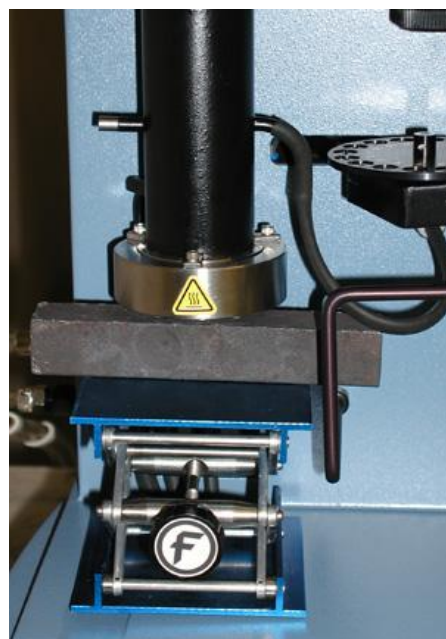
溫度校正



溫度校正

□ 使用支撐物並將磁鐵墊爐子正下方。

□ 溫度校正品 Nickel



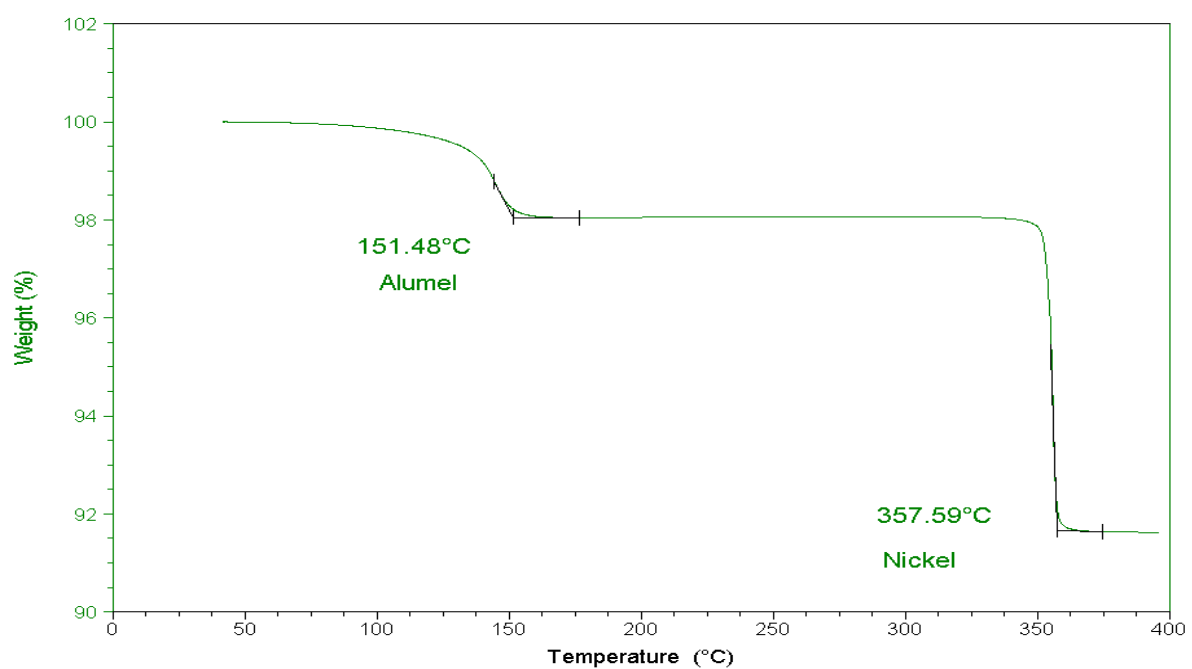
溫度校正

- 使用 Universal Analysis 分析檔案
- 找出居禮溫度誤差值
- 回填 'Temperature Table'

4

3A

溫度校正



TA

溫度校正

Temperature Table - Q500-0014 - TGA Q500@Demo Lab

	Observed Temperature °C	Correct Temperature °C
Point 1:	151.48	154.16
Point 2:	357.59	358.28
Point 3:	0.00	0.00
Point 4:	0.00	0.00
Point 5:	0.00	0.00

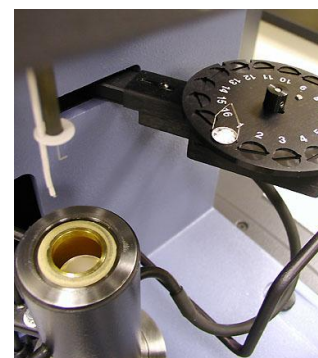
填回量測溫度

如何做一個簡單實驗

□ 實驗前準備事項



□ 樣品準備



樣品準備

- ❑ 樣品的表面積最大化能提昇重量損失解析度與溫度再現性。
(Powders best)
- ❑ 樣品重量
10-20mg 適用大部分實驗
50-100mg 適用易揮發性樣品
- ❑ 大部分TGA儀器上會有重量基線飄移($\pm 0.025\text{mg}$)、如10mg的樣品約0.25%
- ❑ 請使黃銅製鑷子來排除靜電影響
- ❑ 每一次實驗前要對乾淨的盤子做歸零動作(Tare)
- ❑ 樣品要平鋪於樣品盤內
- ❑ 液態樣品 - 要使用液態盤並實驗前上蓋打孔



如何做一個簡單實驗

QSeries - [Q500-1573 - TGA Q500@Mfg.tga]

Control Experimental Calibrate Tools View Window Help

Run 4 Complete Temp: 25.60°C

Experiment

Standard Sequence

Sequence No. 6

- Run 1: ✓
- Run 2: ✓
- Run 3: ✓
- Run 4: ✓

Experiment

Calibration

Platinum

Ready

01 60.60 min

Append Apply Cancel Help

Procedure Summary

Mode: TGA 1000°C

Test: Custom

Sample Information

Sample Name: Y01-1

Pen Type: Platinum

Pen No.: 1

Comments:

Data File Name: WQC-tga\data\TGA\Q2013040430Y01-1.001

Network Drive:

Signal

Signal	Value
Method Time	0.00 min
Segment Time	0.00 min
Remaining Run Time	0 min
Temperature	25.60 °C
Weight	25.7205 mg
Weight percent	100.00 %
Set Point Temp	0.00 °C
Heater Power	0.00 W
Balance Purge Flow	39.99 mL/min
Sample Purge Flow	59.99 mL/min

Running Segment Description

#	Running Segment Description
1	Equilibrate at 50.00 °C
2	Ramp 10.00 °C/min to 235.00 °C
3	Isothermal for 120.00 min

Weight (mg)

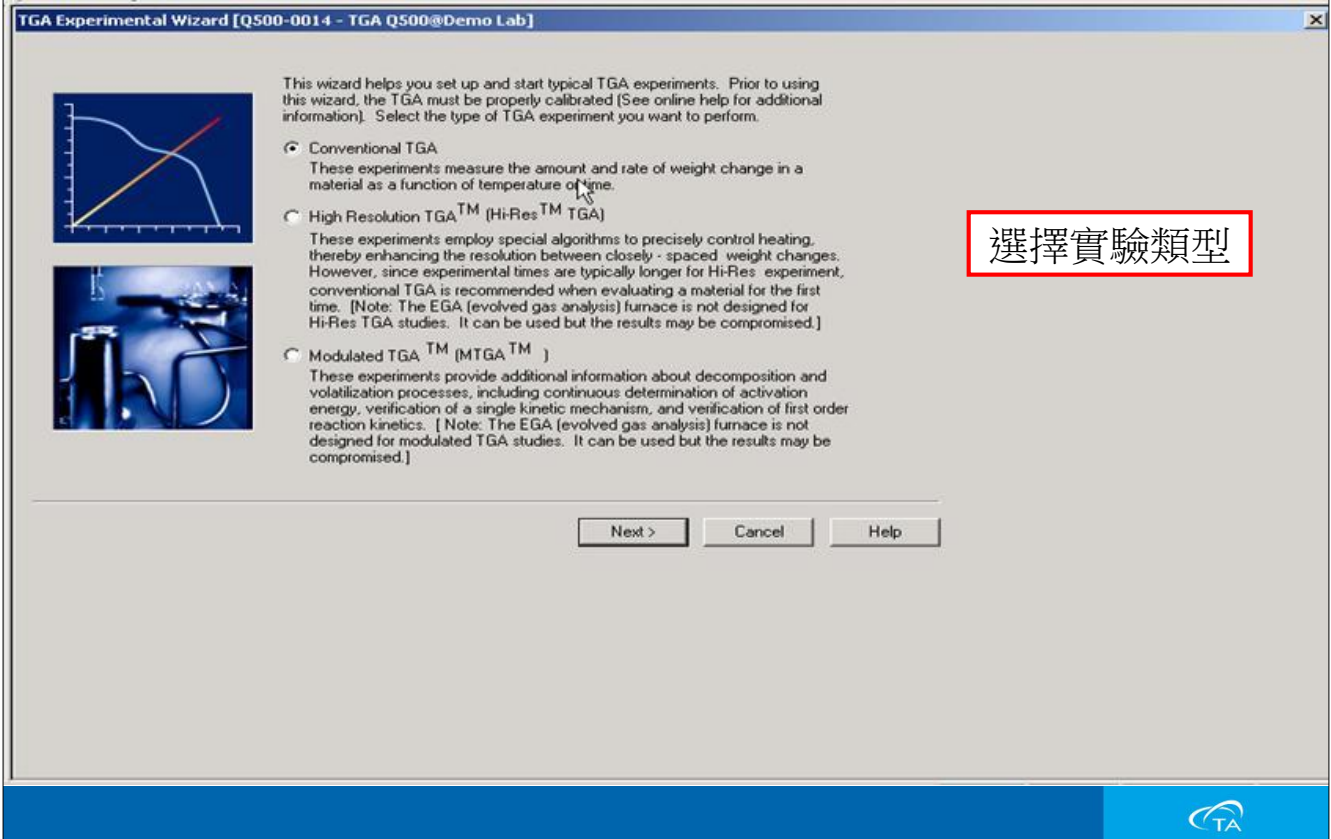
Temperature (°C)

Completed TGA 1000 Seg 0 in Run 4 [11:17:11]

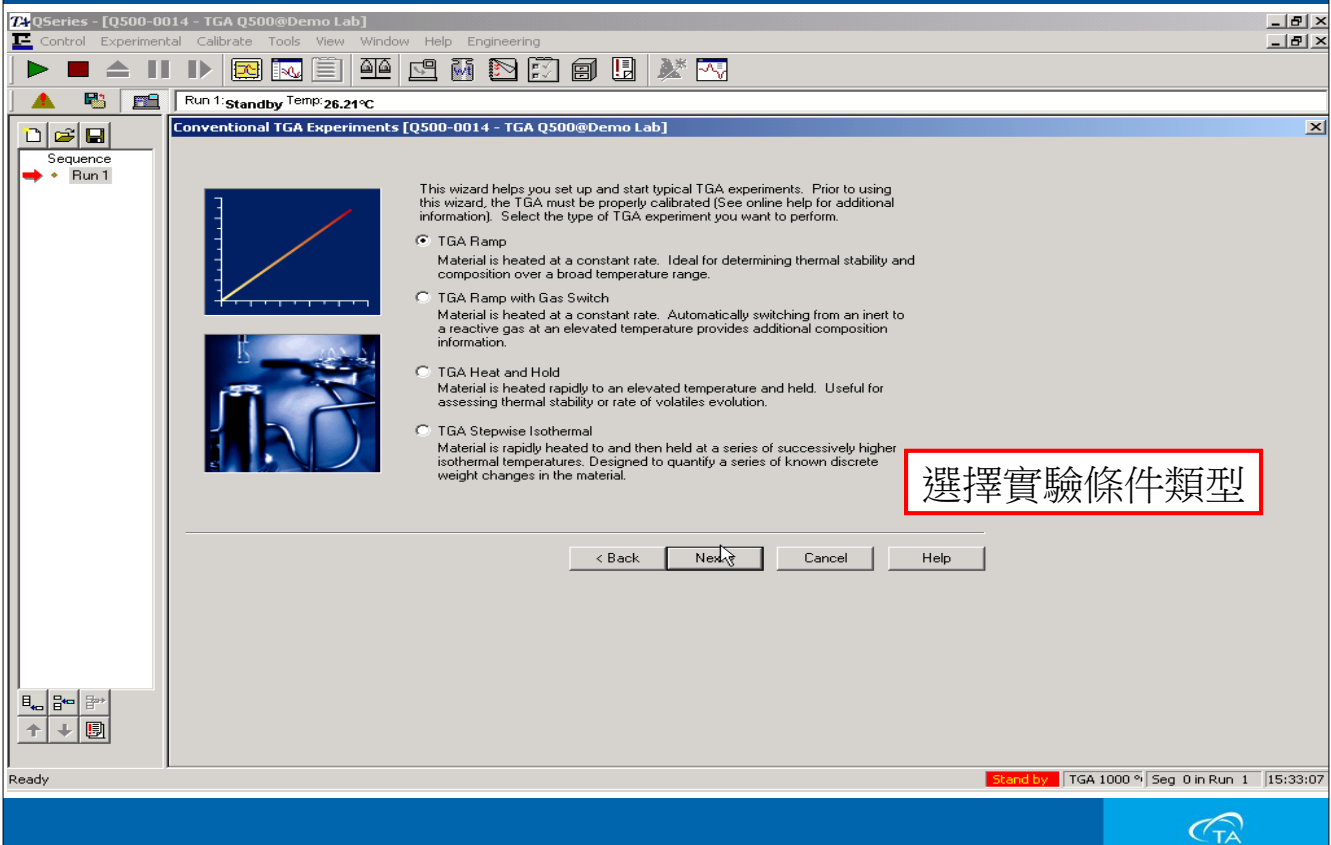
我的文件 TGA操作訓練 ppt TA Instrument Explorer error 1.JPG - 小畫家 QSeries - [Q500-1573 ...]

上午 11:17 星期五 2013/5/3

如何做一個簡單實驗

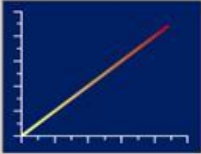


如何做一個簡單實驗



如何做一個簡單實驗

Experimental Parameters: TGA Ramp [Q500-0014 - TGA Q500@Demo Lab]



This experiment is designed to heat the sample at a constant rate. Ideal for determining thermal stability and composition over a broad temperature range. The final temperature should not exceed 600°C for aluminum pans and 1000°C for platinum or alumina (ceramic) pans.

Enter the desired parameters:

Heating rate: °C/min

Final temperature: °C

輸入實驗參數

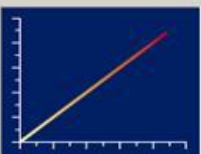
Advanced Parameters... Post-Test Conditions...

< Back Next > Cancel Help



如何做一個簡單實驗

Experimental Parameters: TGA Ramp [Q500-0014 - TGA Q500@Demo Lab]



This experiment is designed to heat the sample at a constant rate. Ideal for determining thermal stability and composition over a broad temperature range. The final temperature should not exceed 600°C for aluminum pans and 1000°C for platinum or alumina (ceramic) pans.

Enter the desired parameters:

Heating rate: °C/min

Final temperature: °C

Advanced Parameters... Post-Test Conditions...

Advanced Parameters

Data Sampling Interval: sec/pt

OK Cancel Help

< Back Next > Cancel Help



如何做一個簡單實驗

Experimental Parameters: TGA Ramp [Q500-0014 - TGA Q500@Demo Lab]

This experiment is designed to heat the sample at a constant rate. Ideal for determining thermal stability and composition over a broad temperature range. The final temperature should not exceed 600°C for aluminum pans and 1000°C for platinum or alumina (ceramic) pans.

Enter the desired parameters:

Heating rate: °C/min

Final temperature: °C

Advanced Parameters... **Post-Test Conditions...**

Post Test Parameters

Method End Conditions

Furnace: ☒ Open and unload ☐ Closed

☒ Air Cool for min

OK Cancel Help

< Back Next > Cancel Help



如何做一個簡單實驗

Experimental Summary [Q500-0014 - TGA Q500@Demo Lab]

General

Instrument: Q500-0014 - TGA Q500
Location: Demo Lab
Mode: TGA 1000 °C
Test: Ramp
Sample Name: Nickel & Alumel
Signal List:
1. Temperature (°C)
2. Time (min)
3. Weight (mg)
4. Temperature Amplitude (°C)
5. Sample Purge Flow (mL/min)
6. Balance Purge Flow (mL/min)

Method

Name: Ramp
Segment List:

< Back **Next >** Cancel Help

檢視實驗設定



如何做一個簡單實驗

Sample Information [Q500-0014 - TGA Q500@Demo Lab]



Sample Information

Sample Name:

Comments:

Data File: 

Pan No.:

Auto Analysis

☐ Autoanalyze

Analysis Macro:

< Back Next > Cancel Help

輸入樣品名稱



如何做一個簡單實驗

Sample Information Notes [Q500-0014 - TGA Q500@Demo Lab]



Notes

Operator:

Pan Type:

Extended Text:

Mass Flow Control Settings

Balance: Flow Rate: mL/min

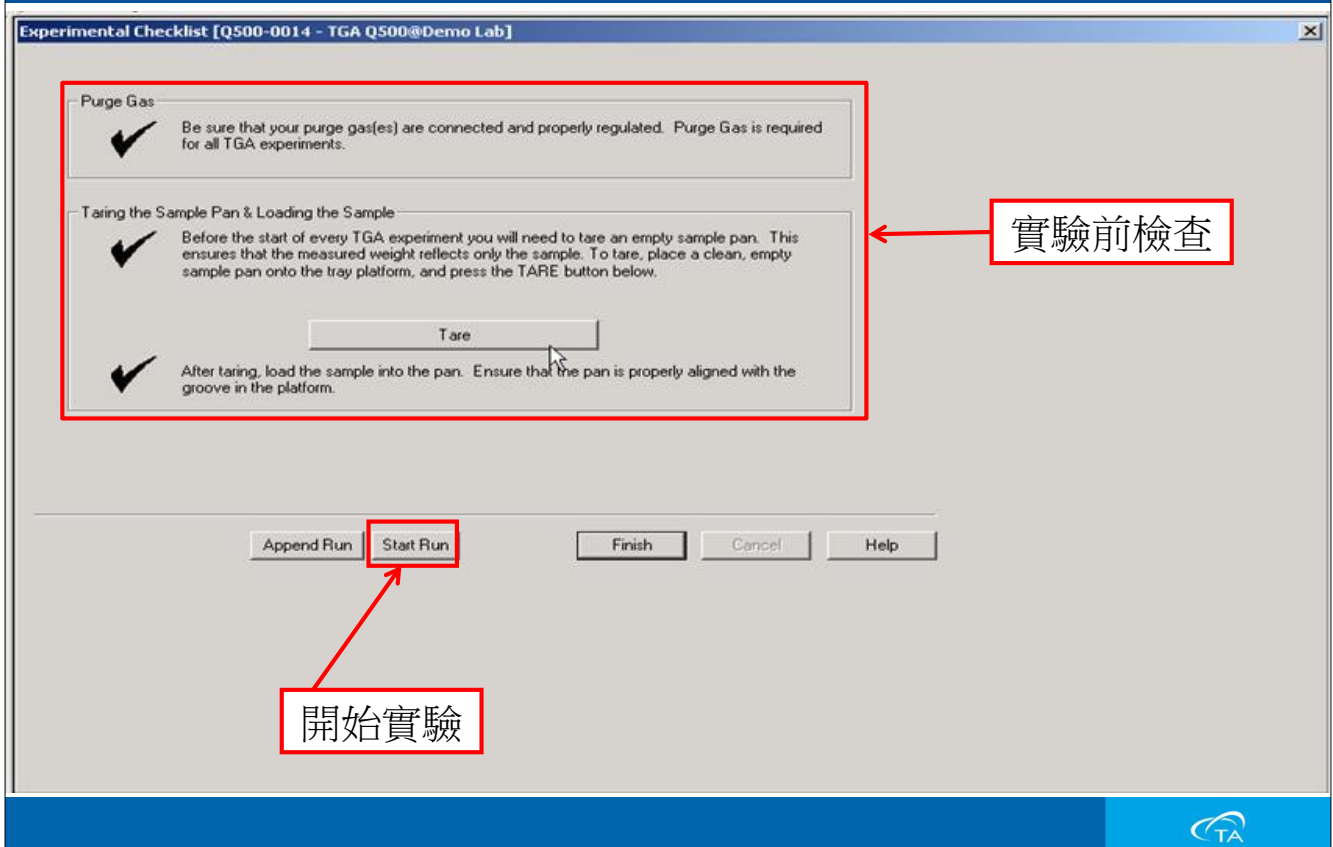
Sample: Flow Rate: mL/min

< Back Next > Cancel Help

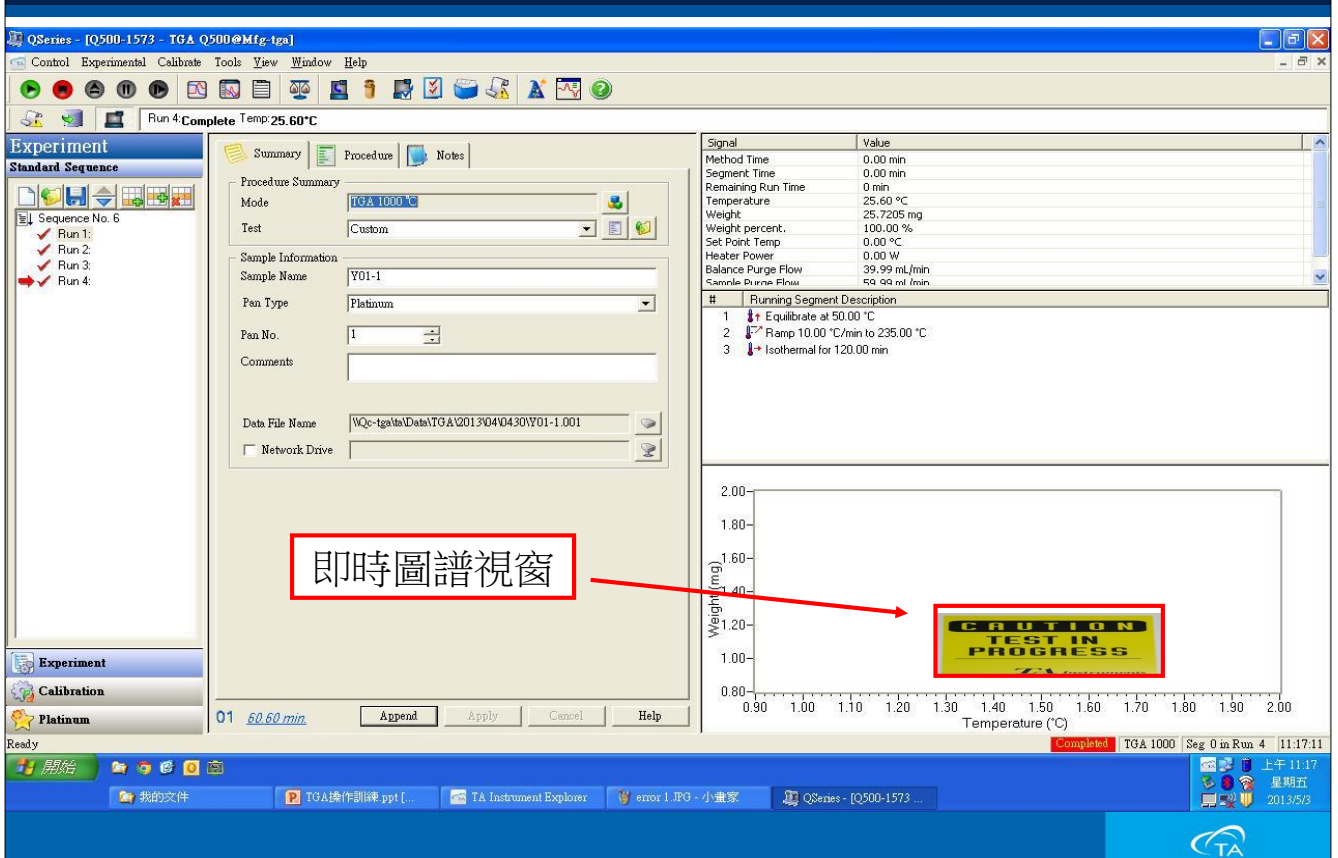
輸入使用者名稱
/樣品盤種類/
實驗註解



如何做一個簡單實驗



如何做一個簡單實驗



分析軟體(簡易示範)

QSeries - [Q500-1573 - TGA Q500@Mfg.tga]

Control Experimental Calibrate Tools View Window Help

Run 4: Complete Temp: 25.60 °C

啟動分析軟體

Experiment
Standard Sequence

Sequence No. 6
Run 1: ✓
Run 2: ✓
Run 3: ✓
Run 4: ✓

Procedure Summary

Mode: TGA 1000 °C
Test: Custom

Sample Information

Sample Name: Y01-1
Pan Type: Platinum
Pan No.: 1
Comments:
Data File Name: YQc-tga\data\TGA\Q013040430\Y01-1.001
☐ Network Drive

Signal Value

Signal	Value
Method Time	0.00 min
Segment Time	0.00 min
Remaining Run Time	0 min
Temperature	25.60 °C
Weight	25.7205 mg
Weight percent	100.00 %
Set Point Temp	0.00 °C
Heater Power	0.00 W
Balance Purge Flow	39.99 mL/min
Sample Purge Flow	50.00 mL/min

Running Segment Description

#	Running Segment Description
1	Equilibrate at 50.00 °C
2	Ramp 10.00 °C/min to 235.00 °C
3	Isothermal for 120.00 min

Weight (mg)

Temperature (°C)

0.90 1.00 1.10 1.20 1.30 1.40 1.50 1.60 1.70 1.80 1.90 2.00

0.80 1.00 1.20 1.40 1.60 1.80 2.00

01 60.60 min Append Apply Cancel Help

Ready

Completed TGA 1000 Seg 0 in Run 4 11:17:11

我的文件 TGA操作訓練 ppt TA Instrument Explorer error 1.JPG - 小畫家 QSeries - [Q500-1573 ...]

上午 11:17 星期五 2013/5/3

TA

分析軟體(簡易示範)

Universal Analysis 2000

File View Macros Window Help

開啟檔案

Open Data File

Look in: TGA

CaOx 052802.001 TGAKIN.004

Mtga-eva.001

TGA-Caox.001

TGAKIN.001

TGAKIN.002

TGAKIN.003

File name: CaOx 052802.001

Files of type: All Files (*.*)

File path: C:\TA\Data\TGA

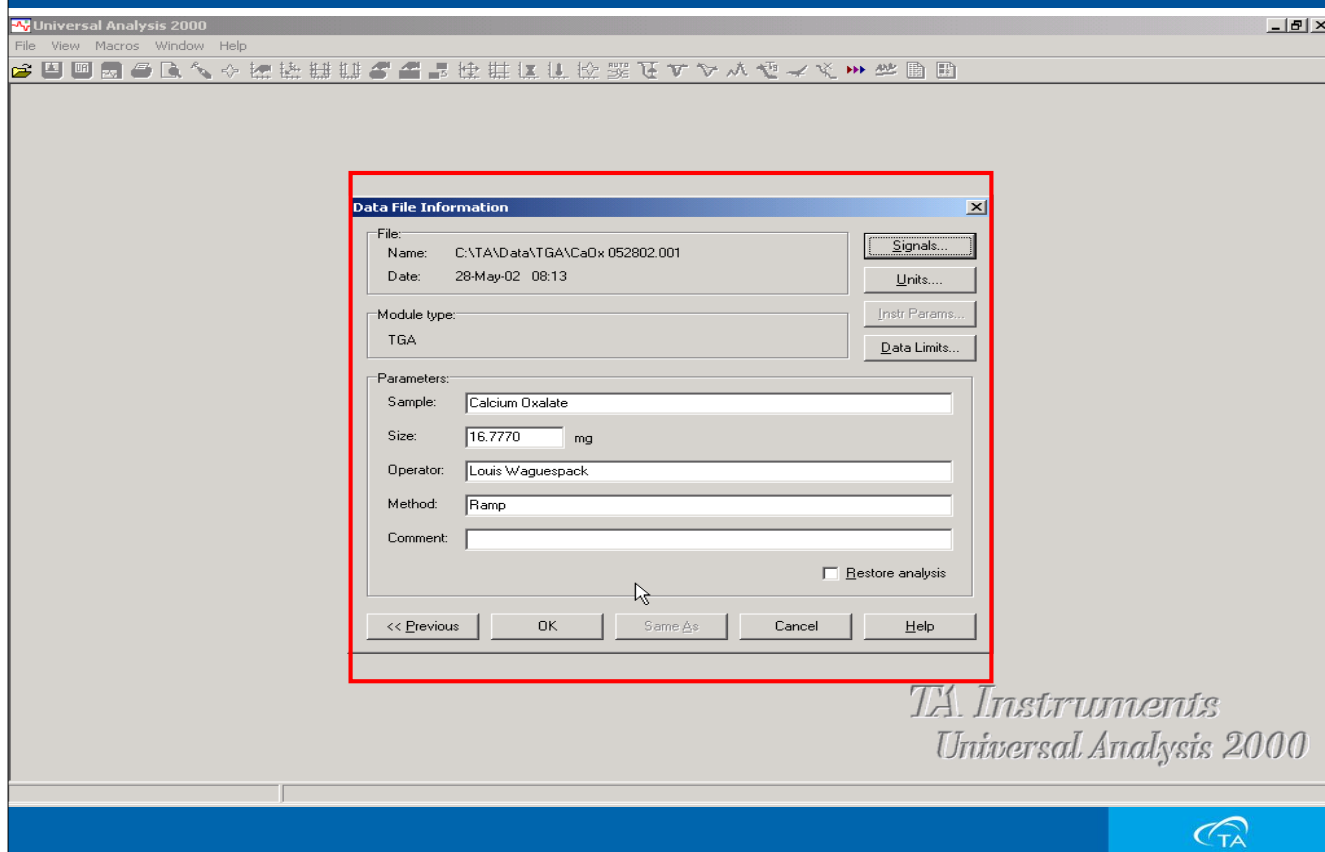
Open Cancel Help

Run 1 (0)
Instrument TGA Q50 V4.1 Build 141
Module TGA
Sample Calcium Oxalate
Size 16.777 mg
Method Ramp
Operator Louis Waguespack
Comment
Date 28-May-2002
Time 08:13

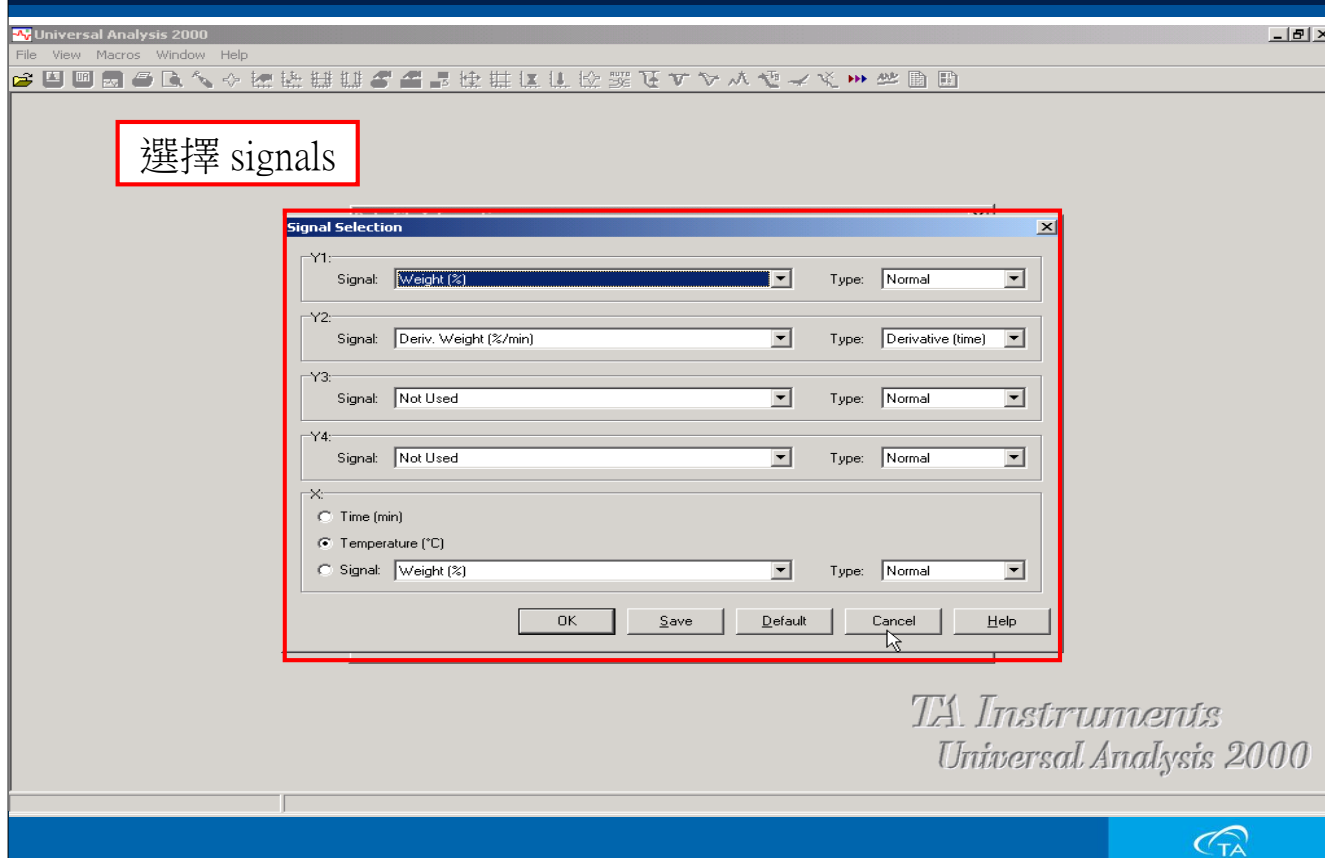
TA Instruments
Universal Analysis 2000

TA

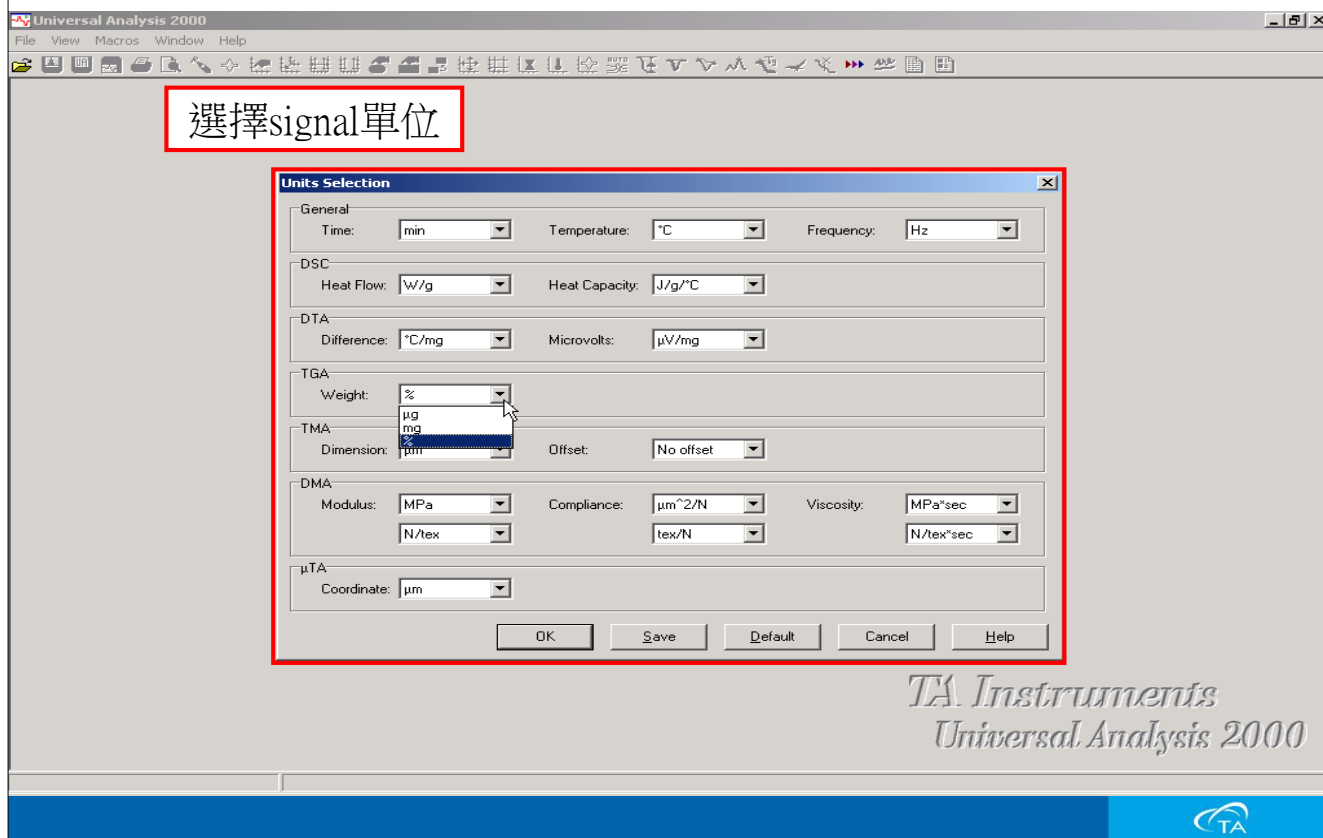
分析軟體(簡易示範)



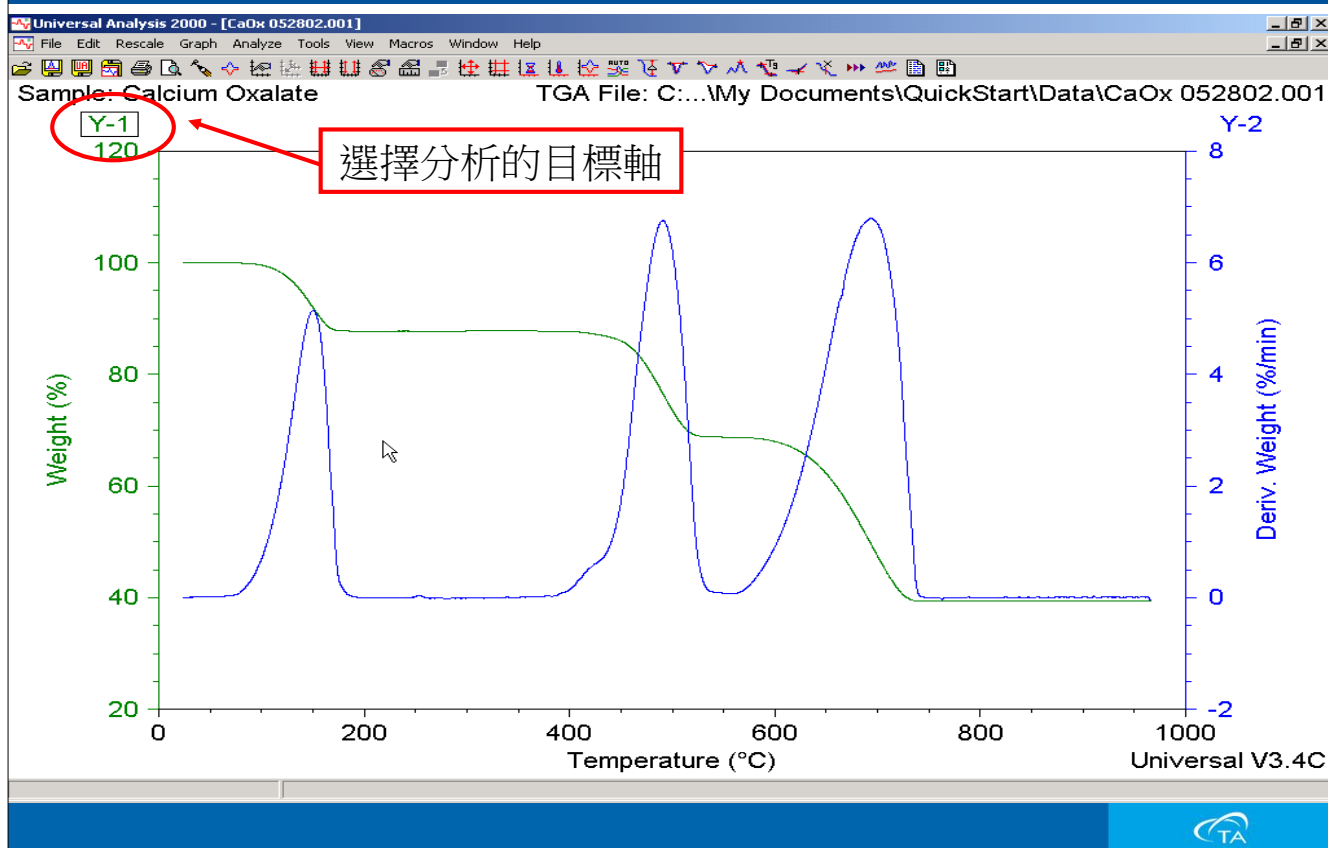
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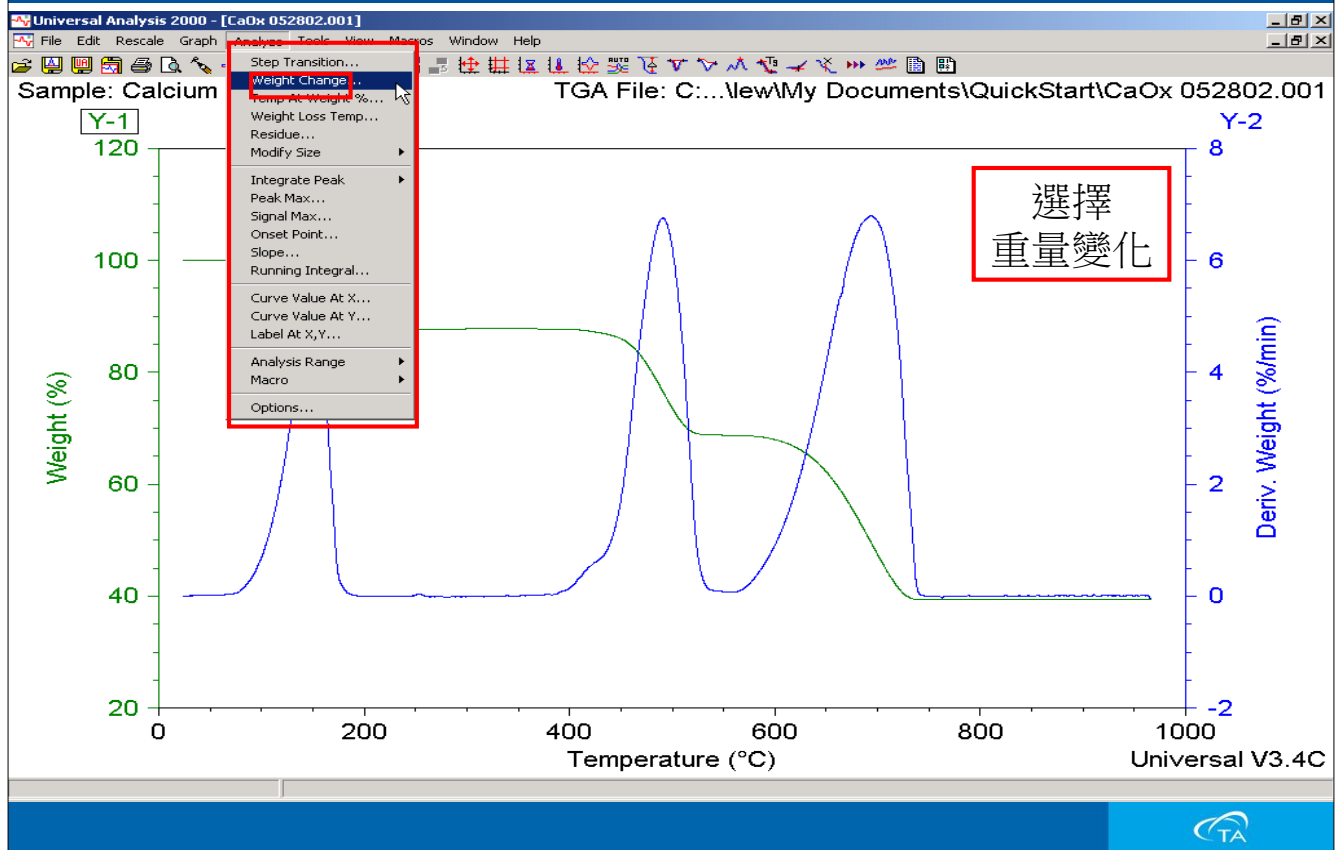
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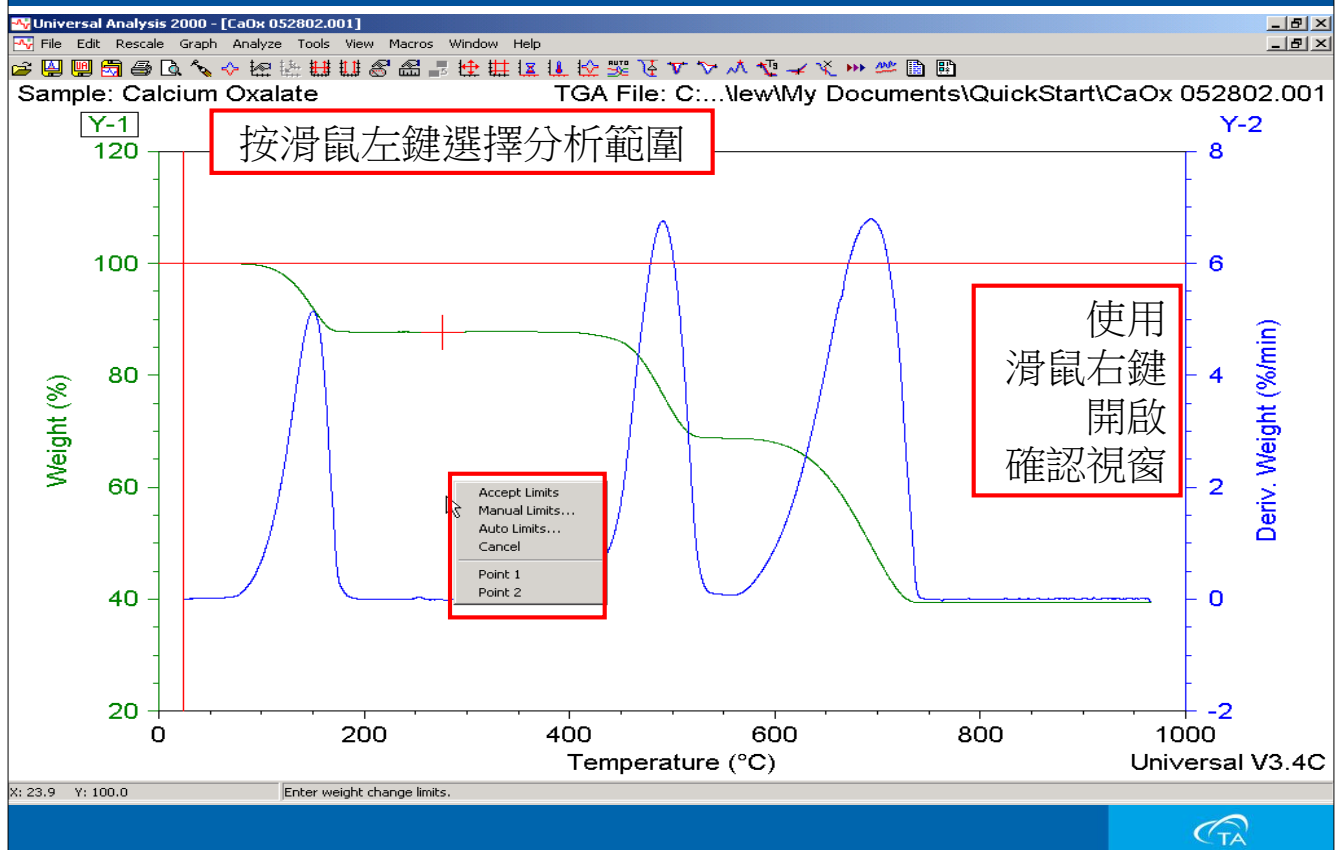
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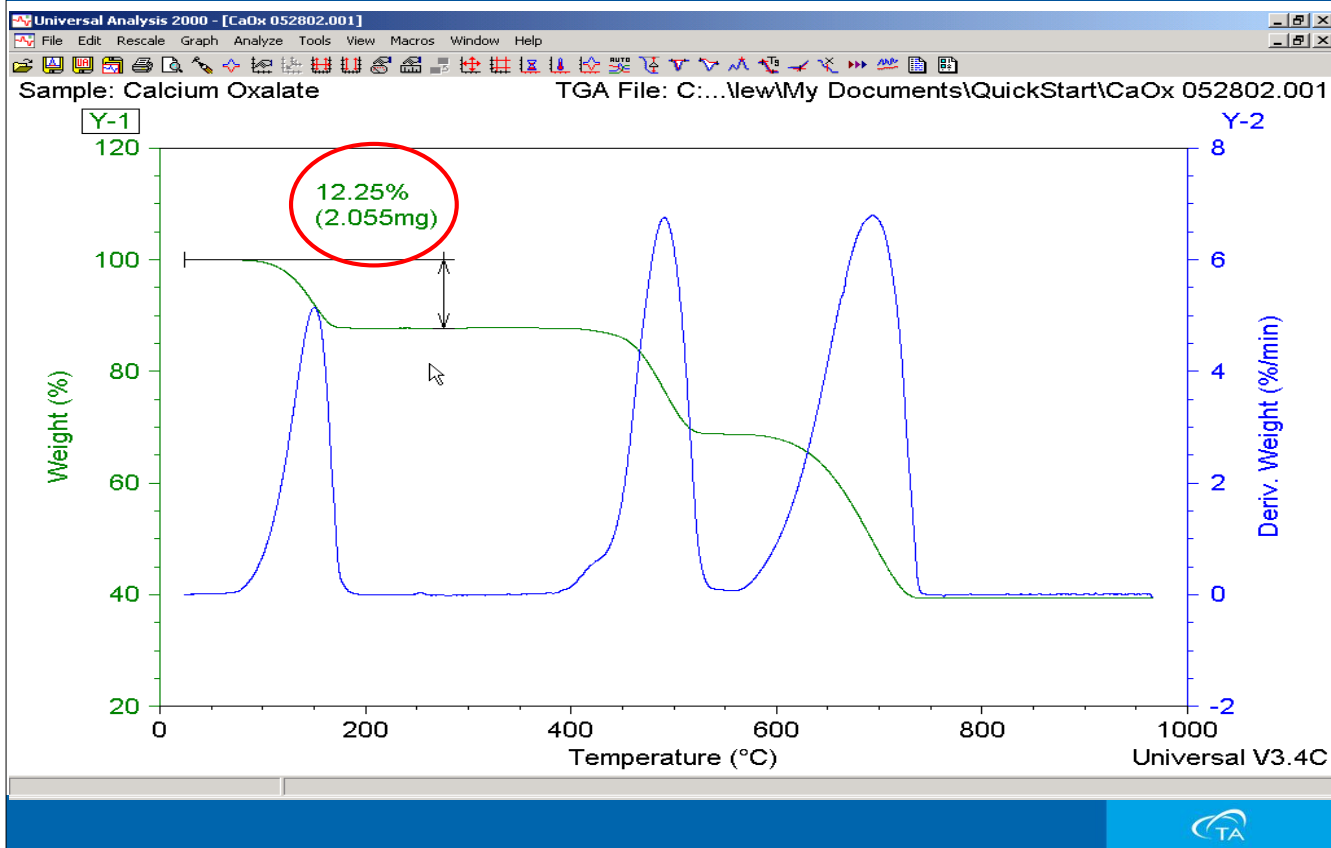
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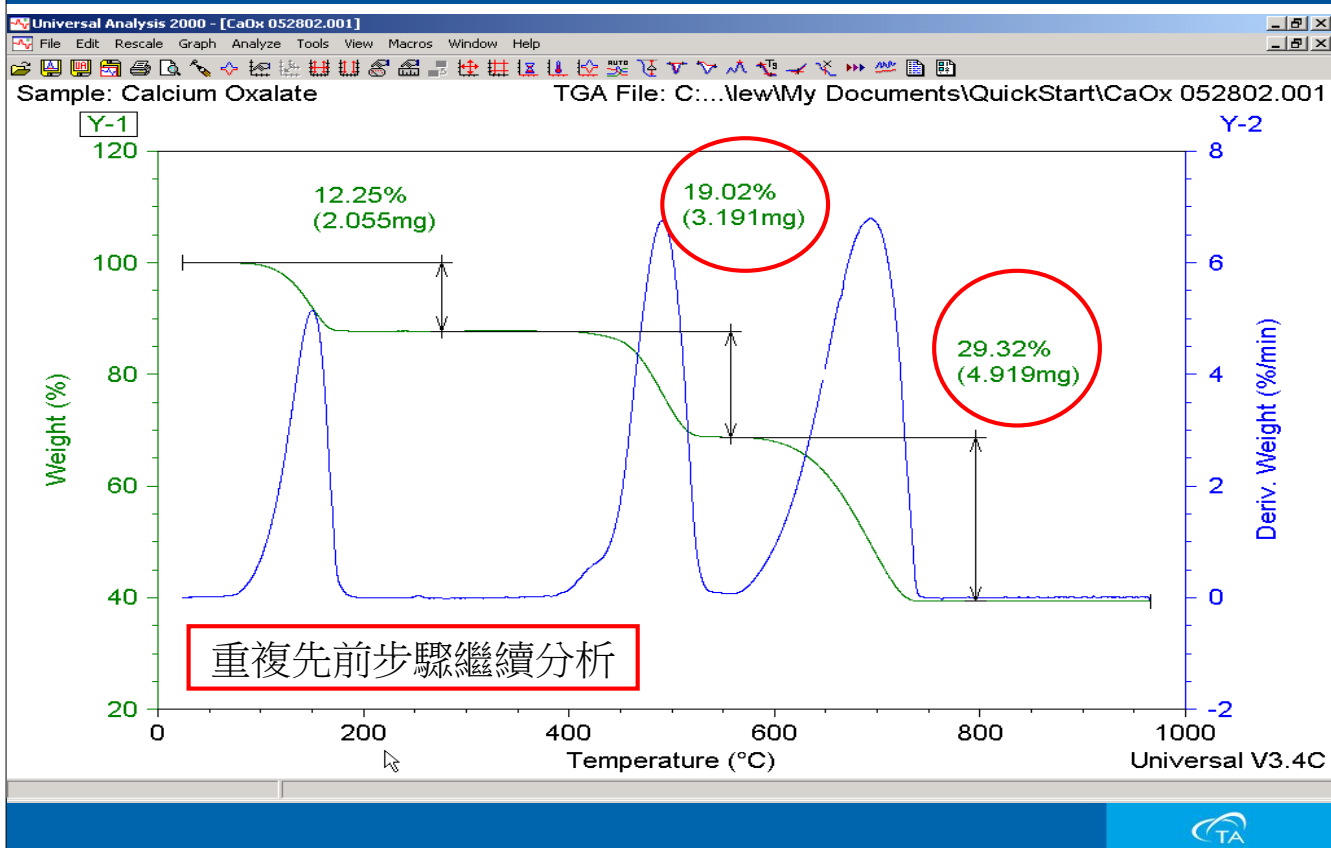
分析軟體(簡易示範)



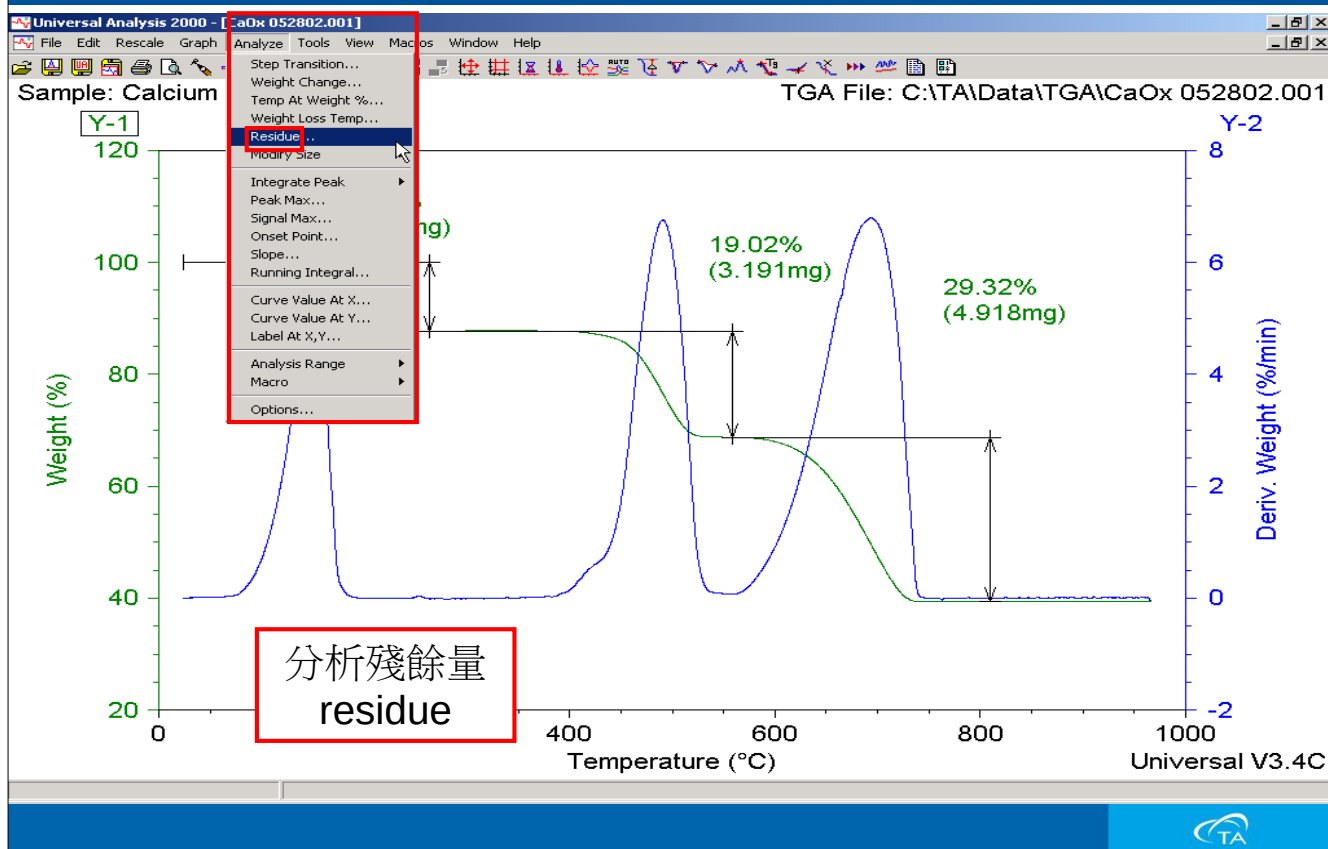
分析軟體(簡易示範)



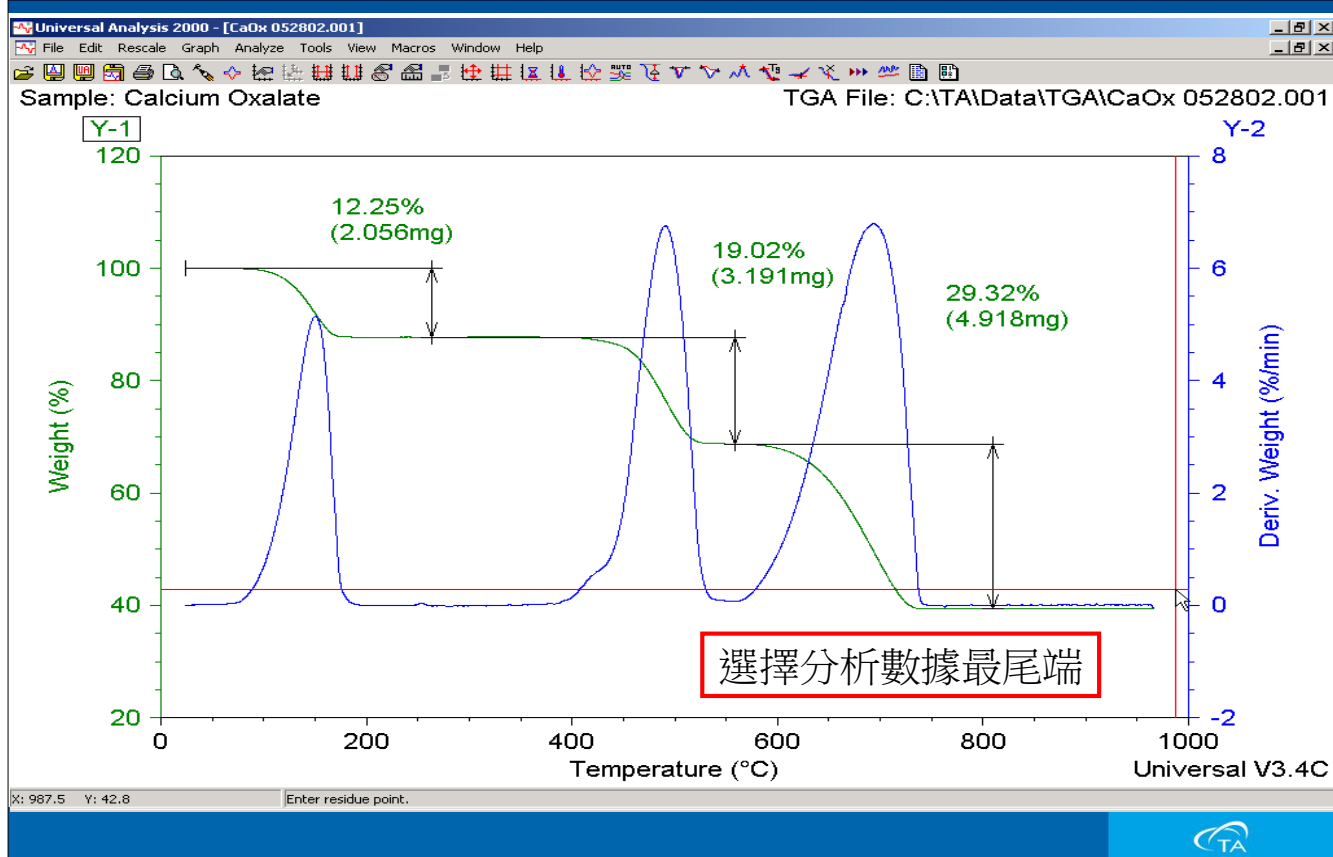
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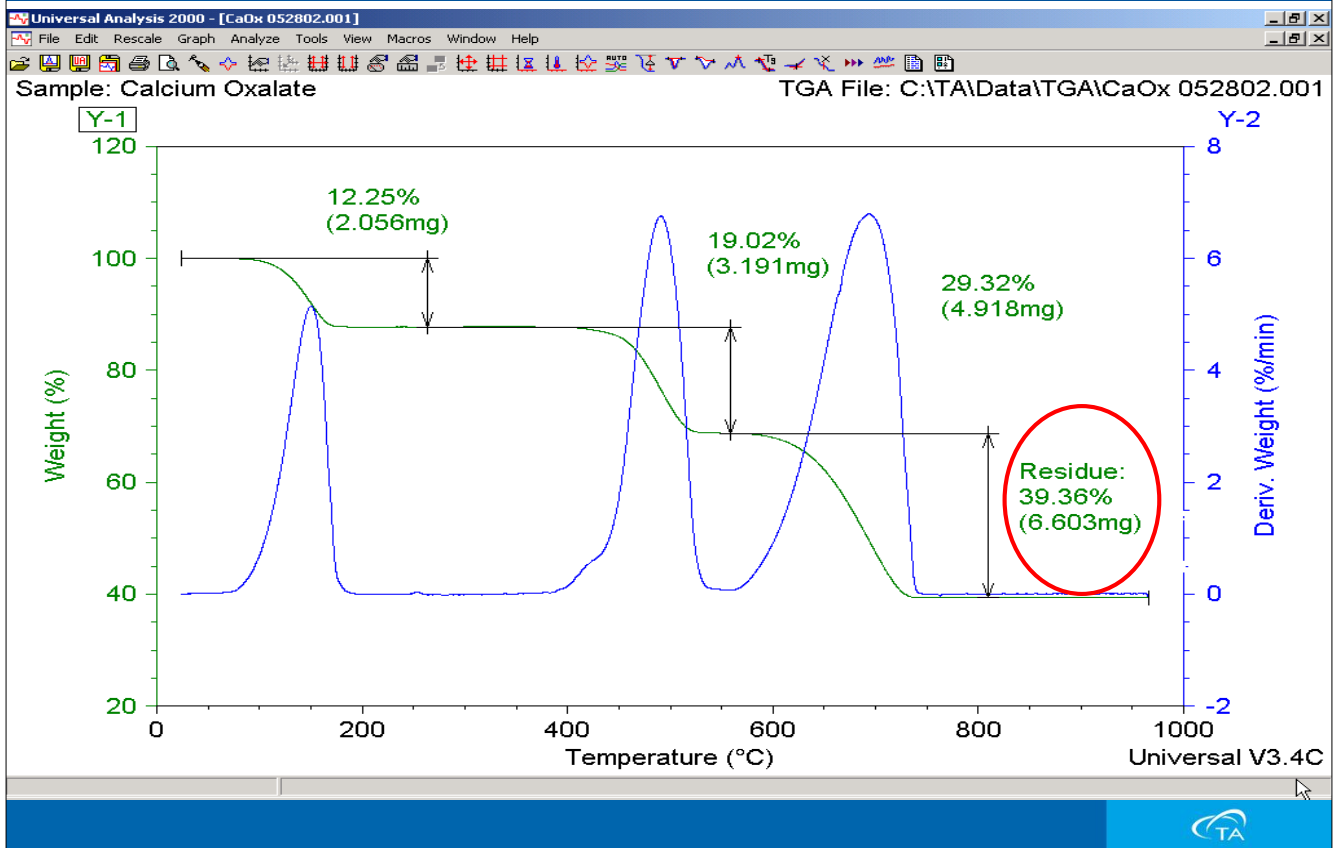
分析軟體(簡易示範)



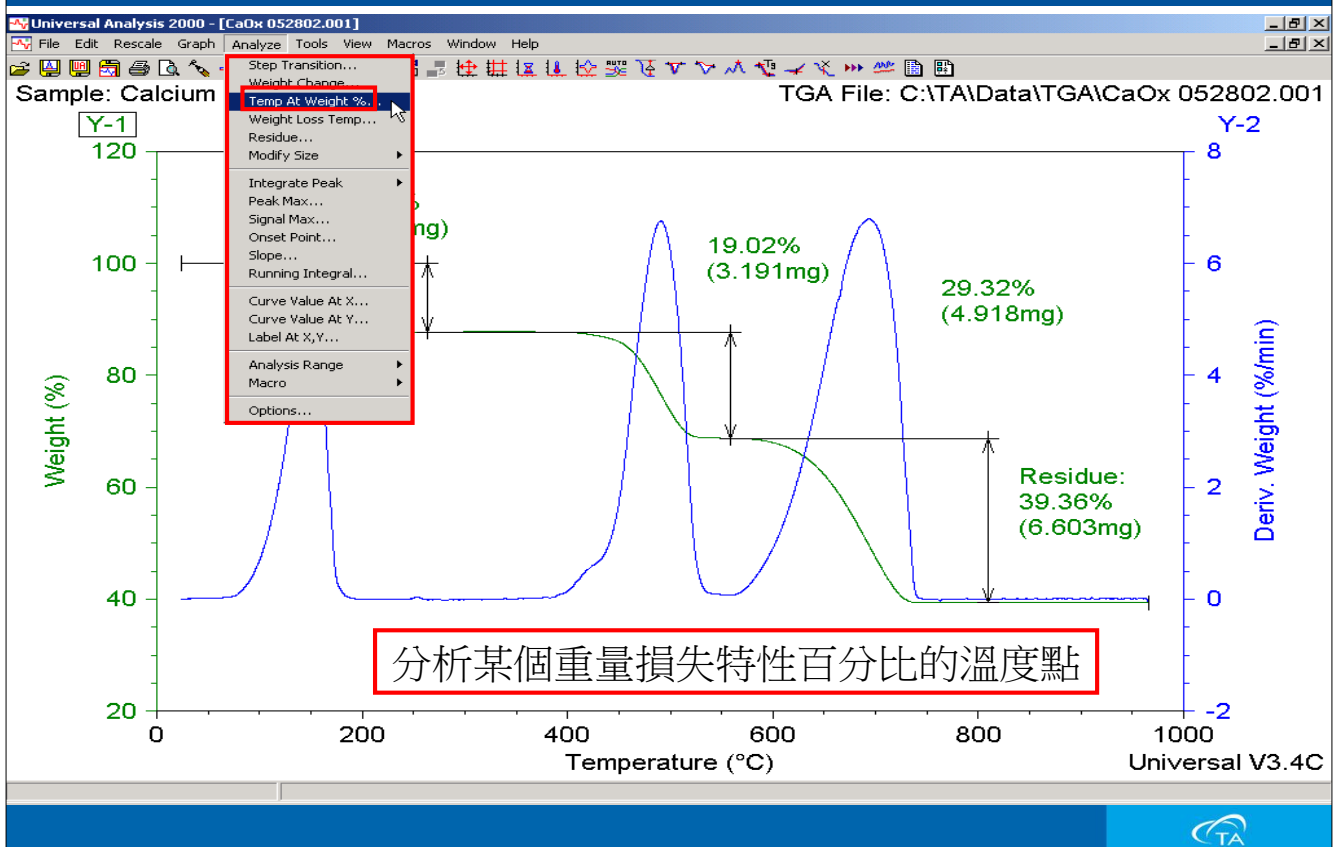
分析軟體(簡易示範)



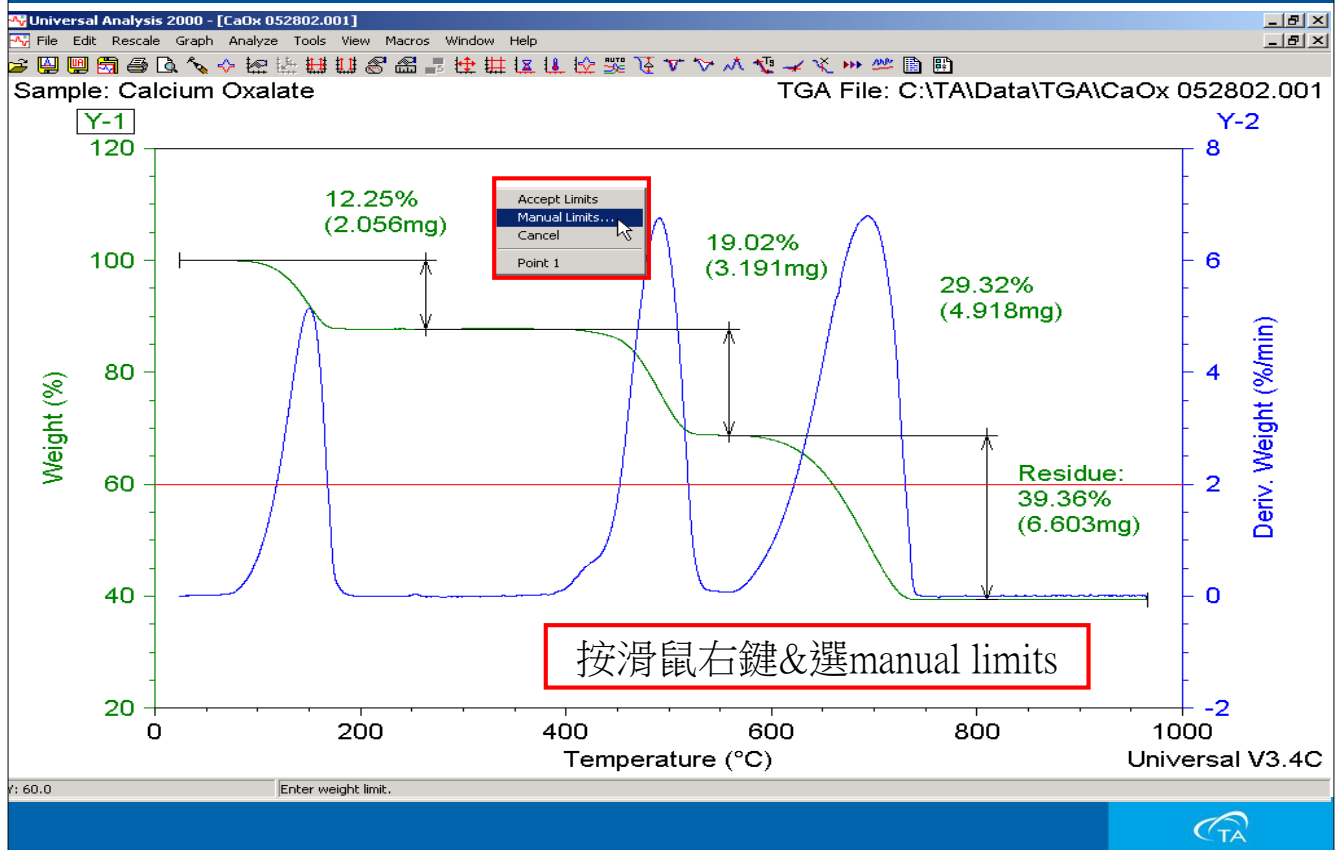
分析軟體(簡易示範)



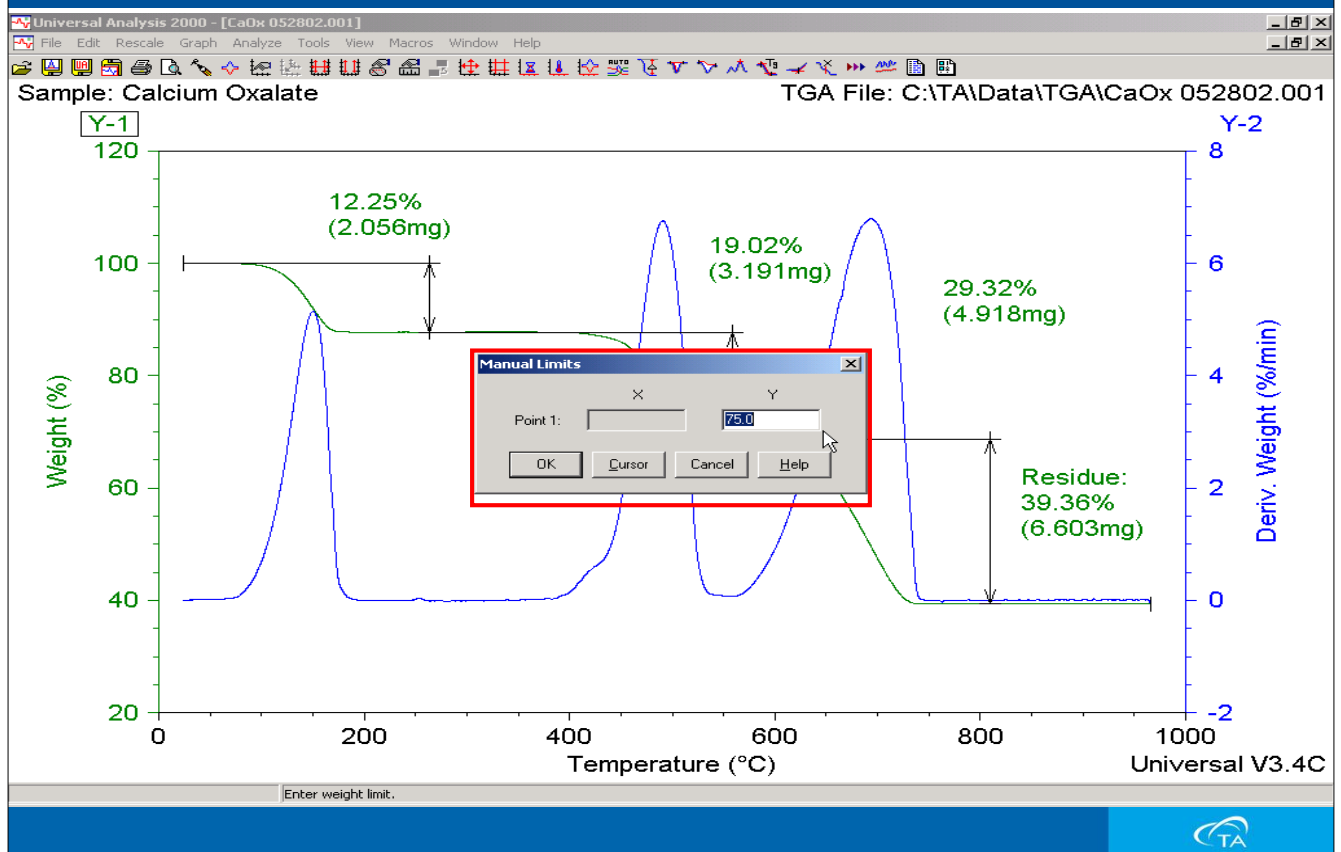
分析軟體(簡易示範)



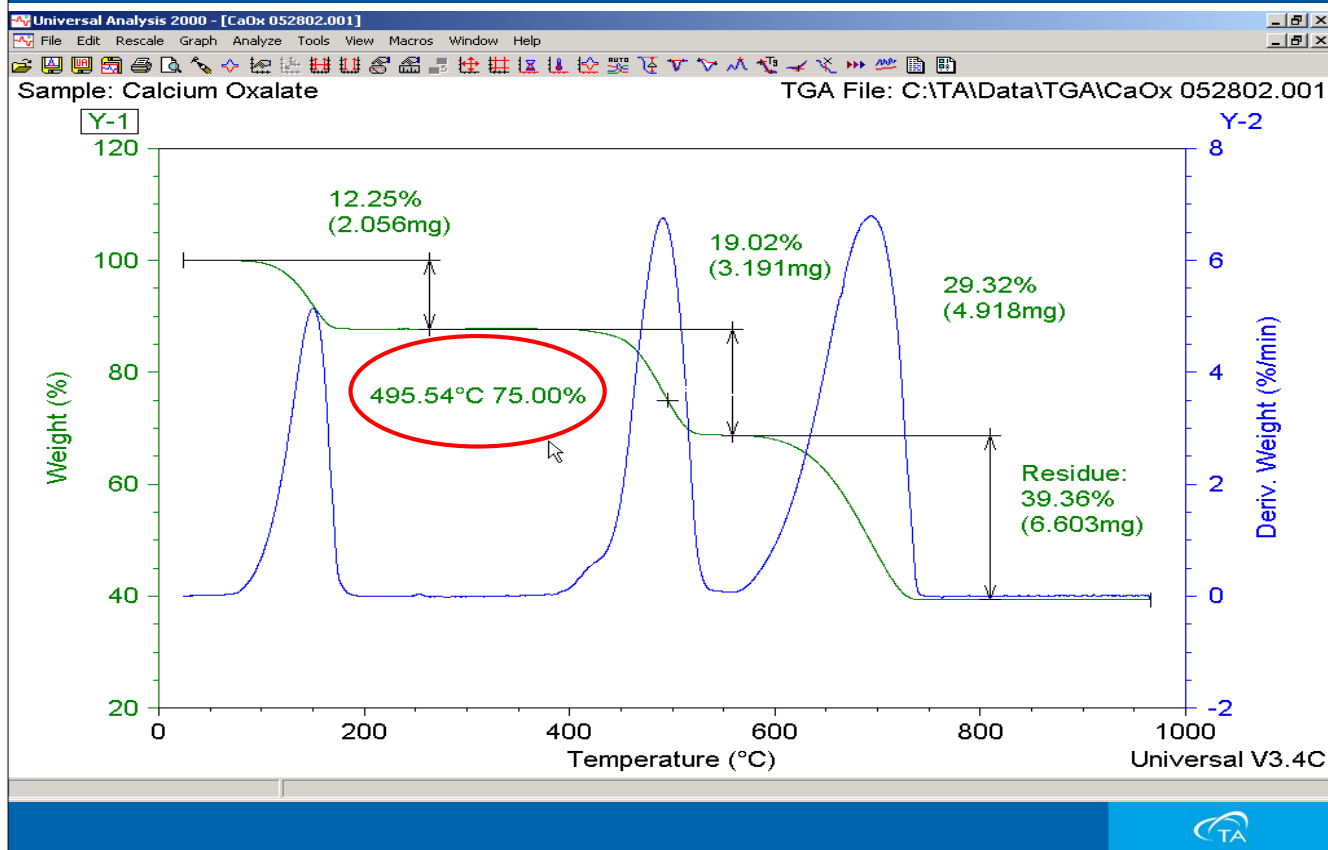
分析軟體(簡易示範)



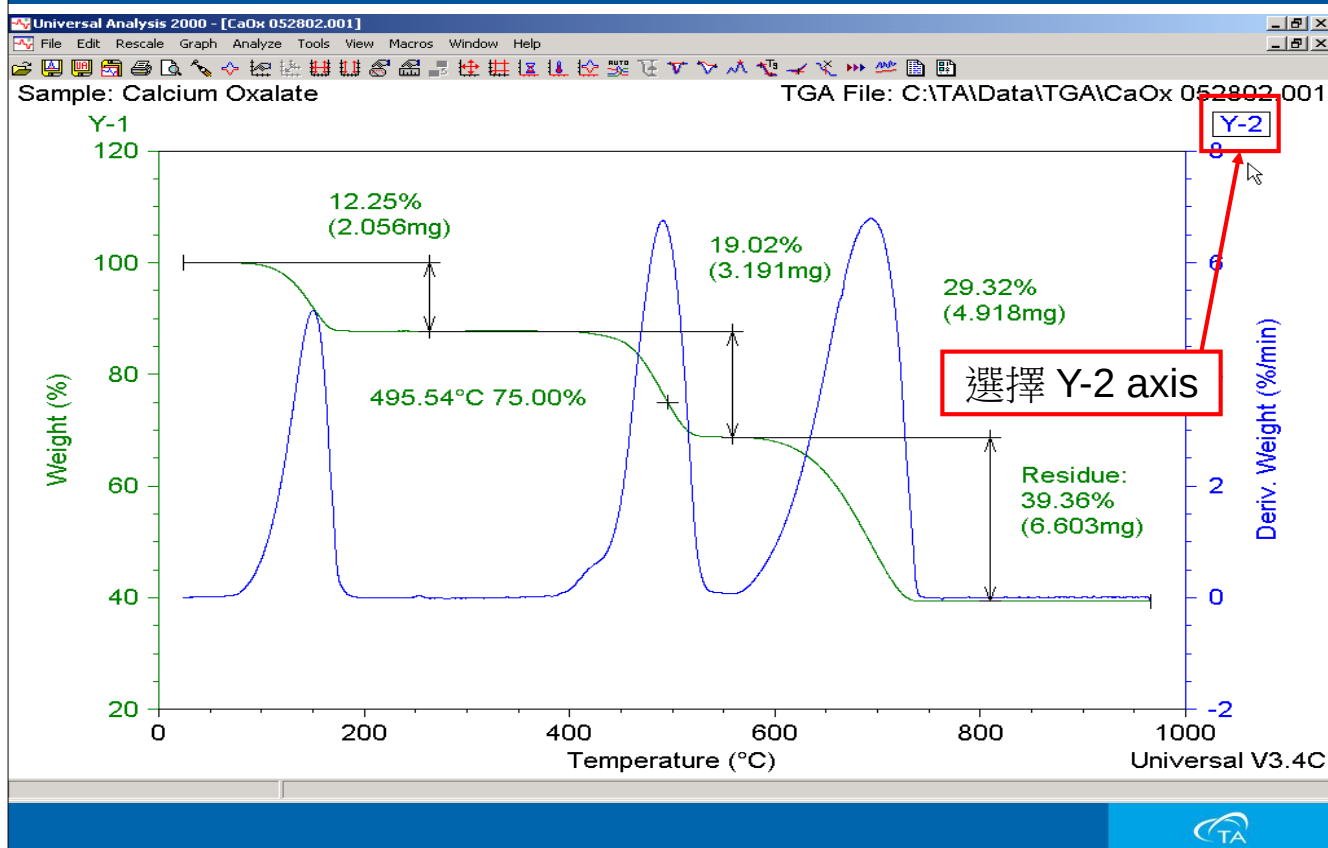
分析軟體(簡易示範)



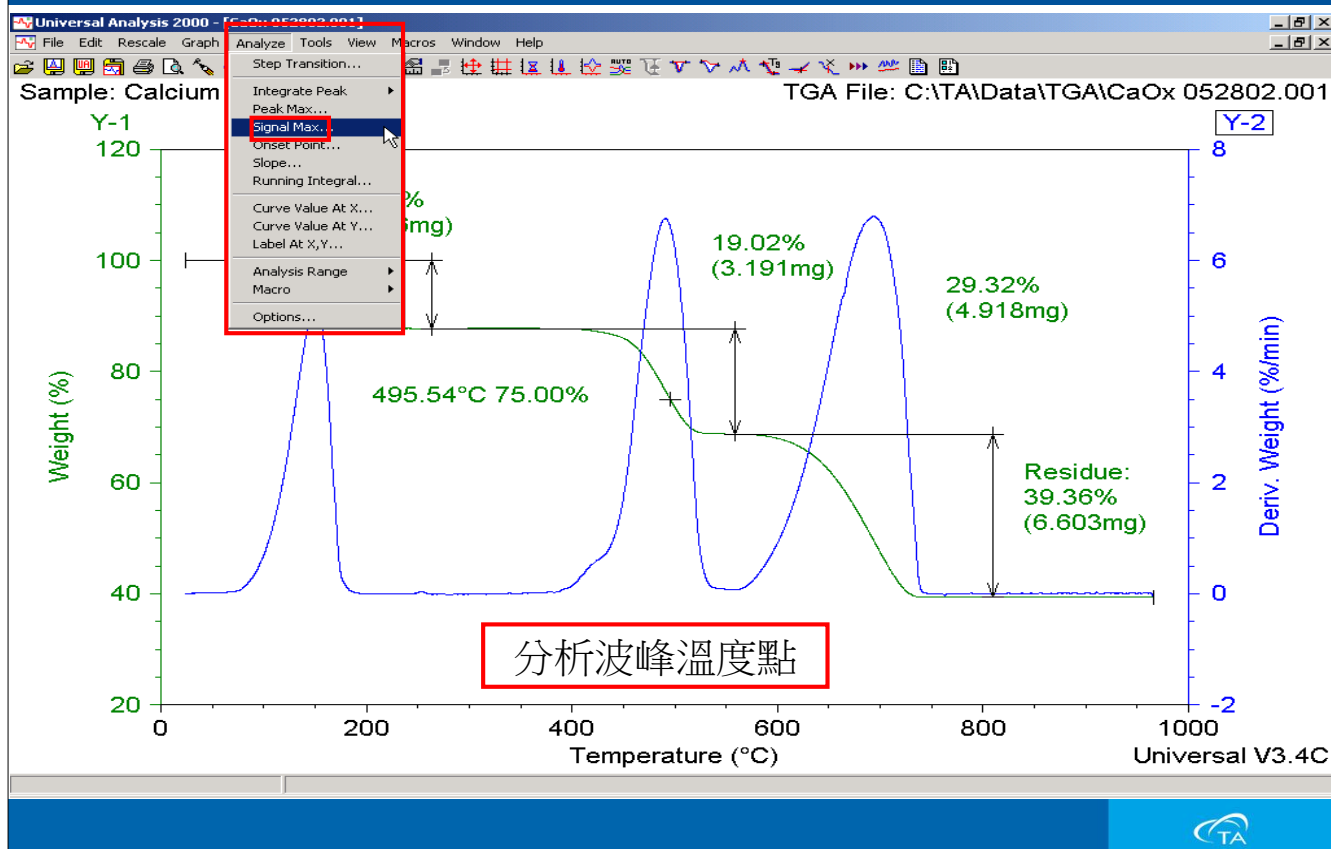
分析軟體(簡易示範)



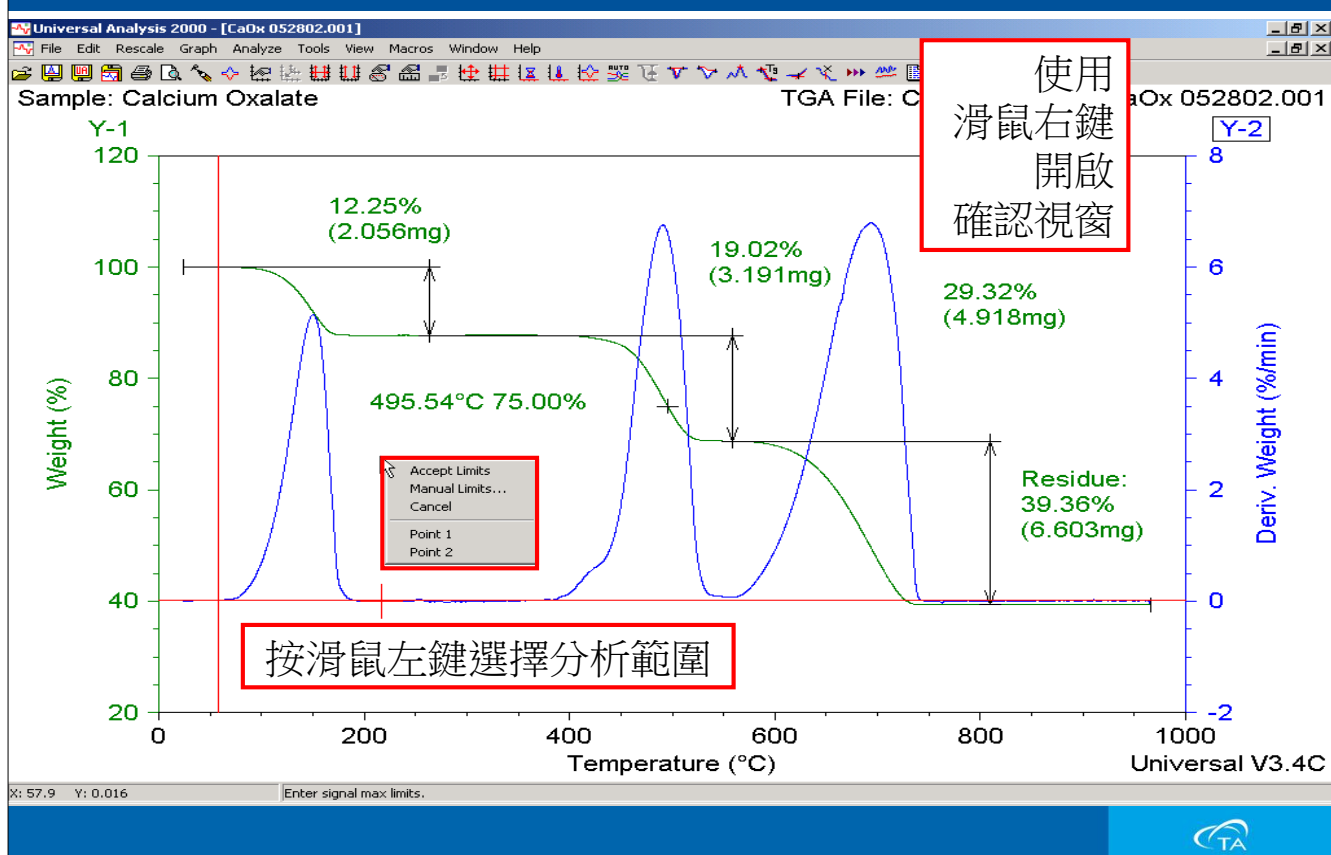
分析軟體(簡易示範)



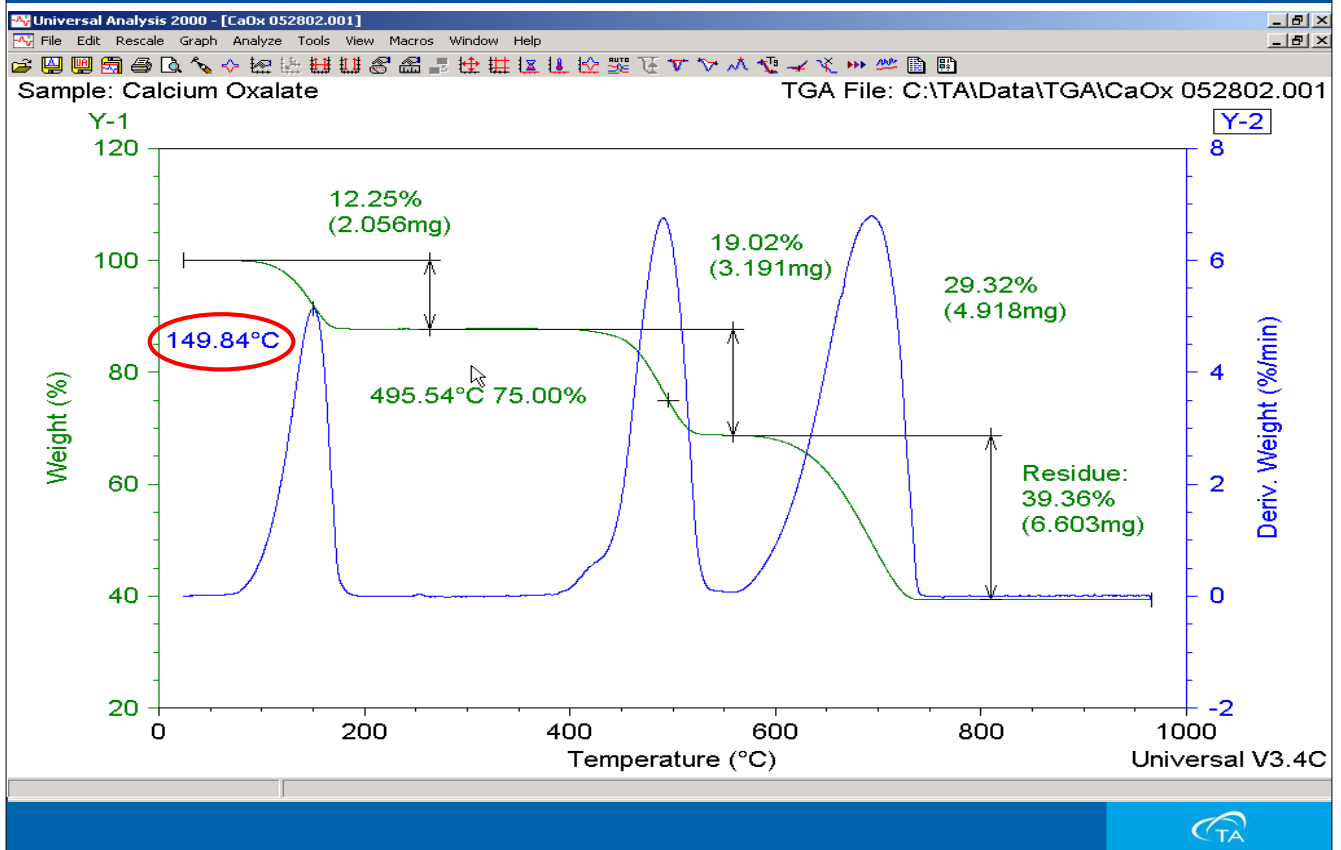
分析軟體(簡易示範)



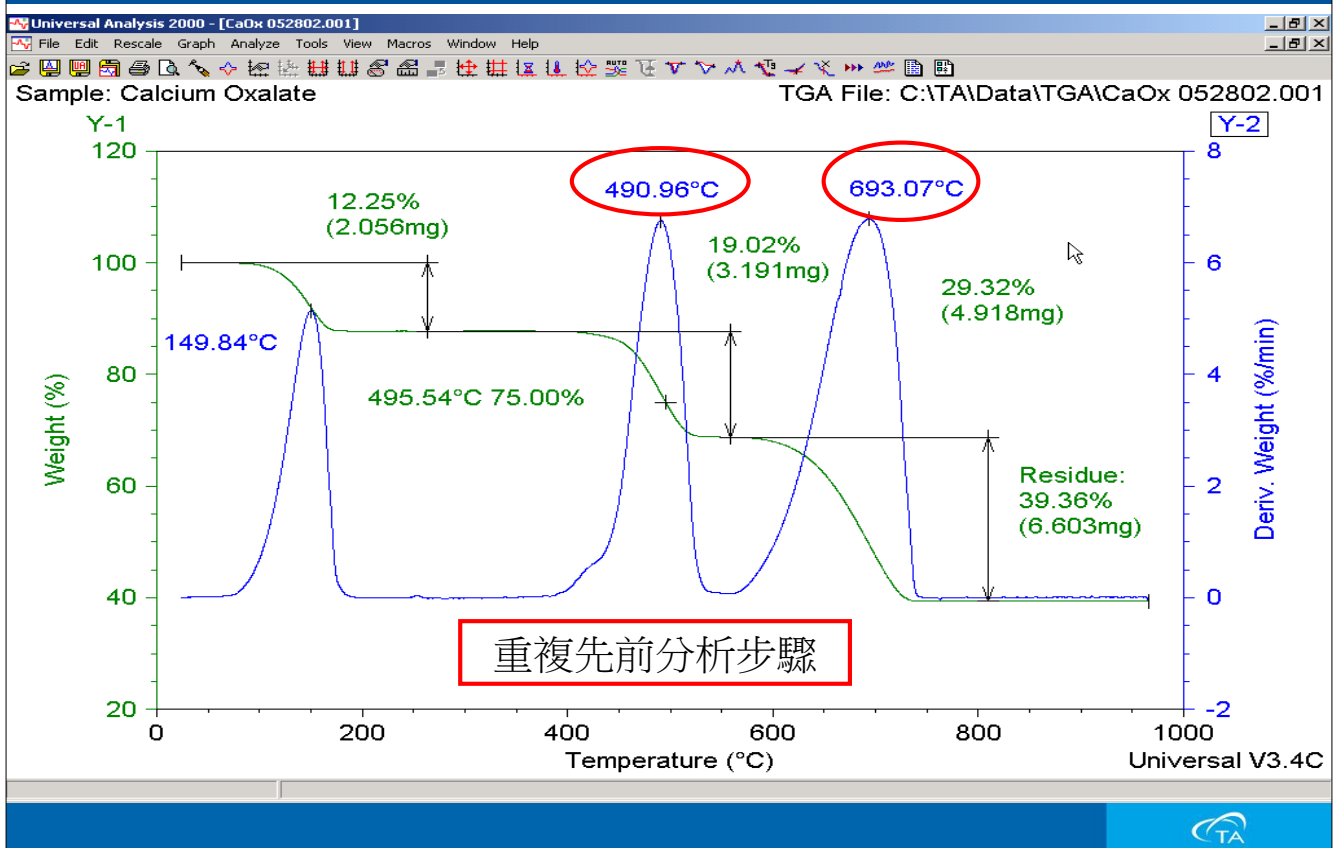
分析軟體(簡易示範)



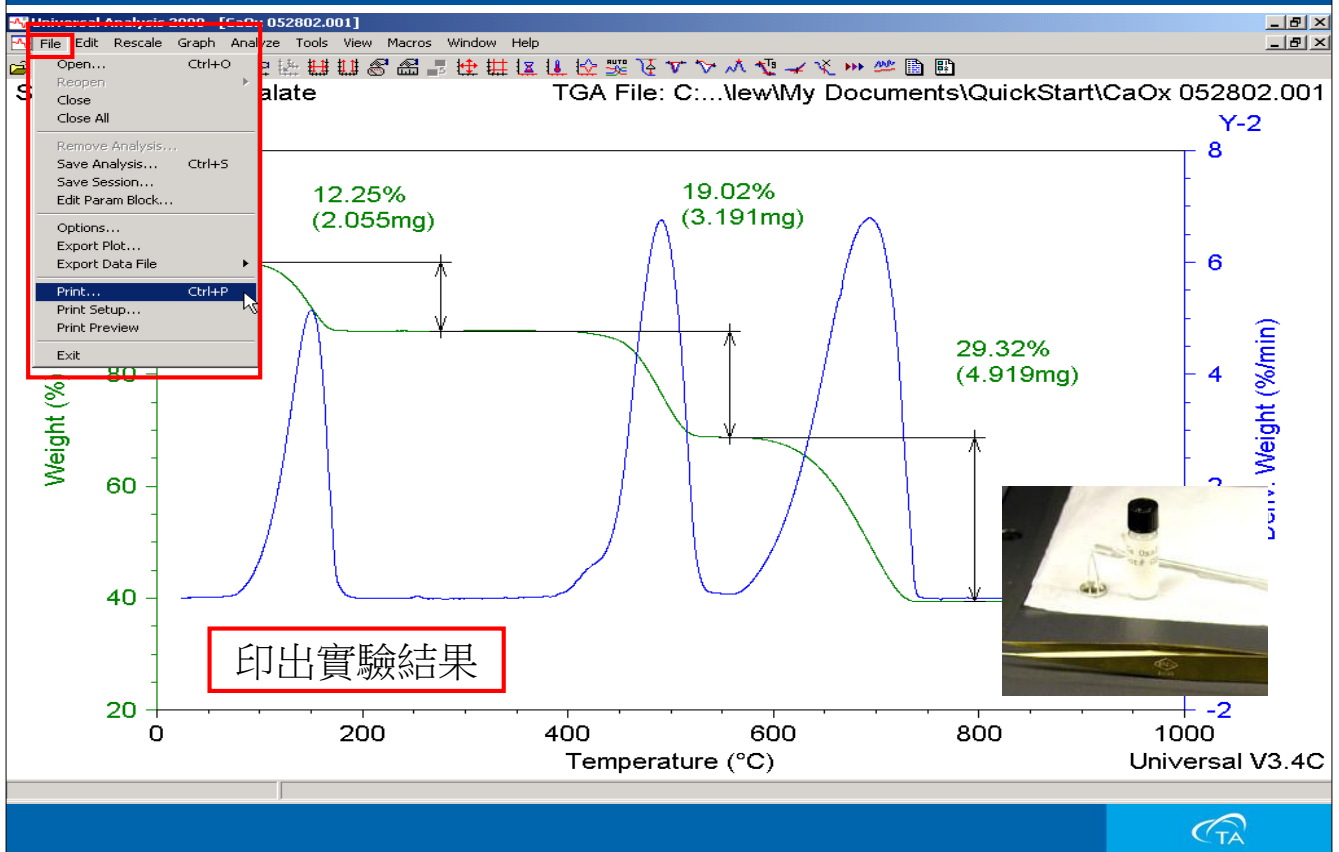
分析軟體(簡易示範)



分析軟體(簡易示範)

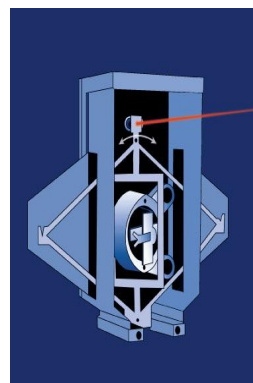
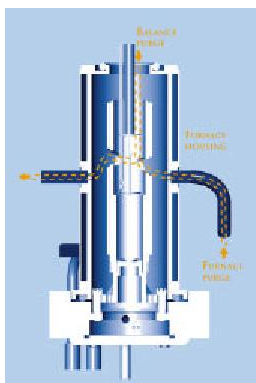


分析軟體(簡易示範)



平時保養注意事項

- TGA常有問題的部分
不外乎Furnace, Thermocouple, Balance, 白金吊絲
- 白金掛勾彎曲可以拆下, 使用兩片平板物品或是載玻片輕壓白金掛勾滾直
- 樣品平台校正



平時保養注意事項

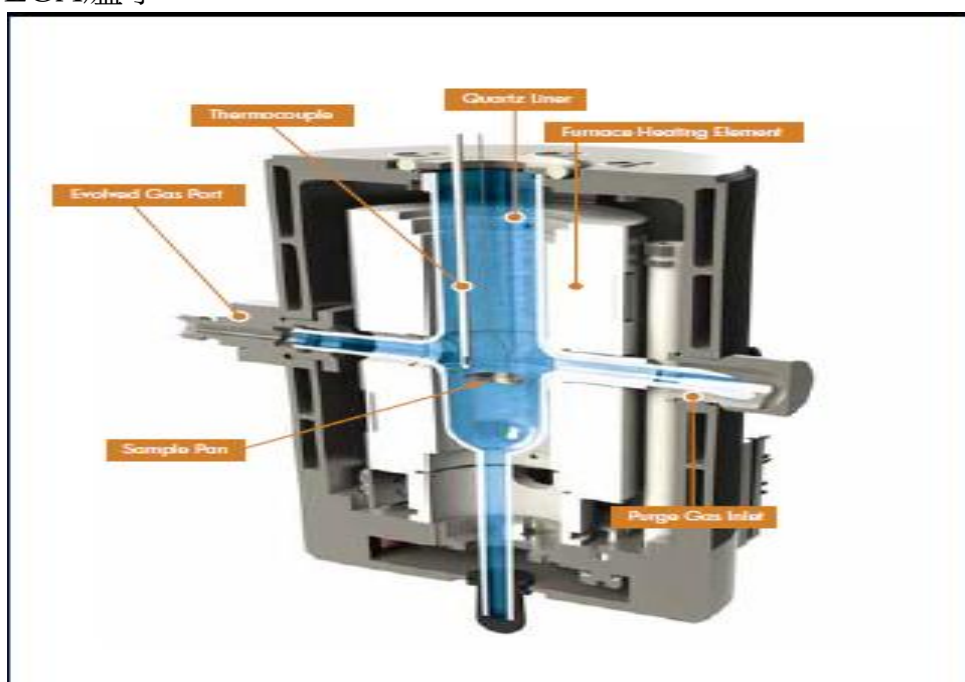
- TGA爐壁需定期清潔 並保持黃銅色般乾淨
- 爐子排氣出口的清潔, 並維持暢通
- 白金掛鉤的垂直性與調整TGA機台水平, 樣品盤需在爐口正中心



TA

平時保養注意事項

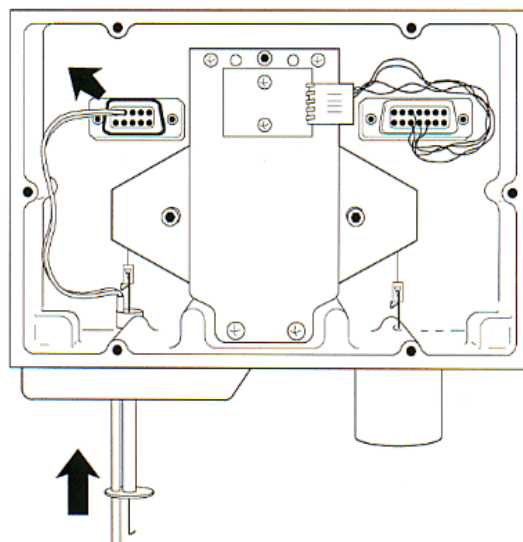
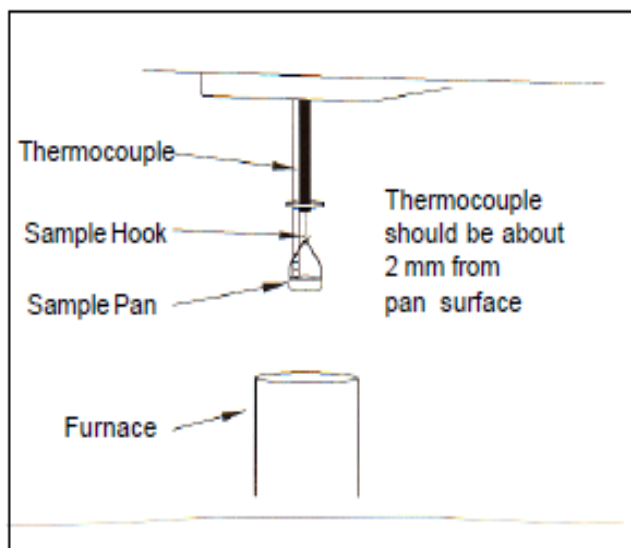
- EGA爐子



TA

平時保養注意事項

- 簡易更換熱電偶
- 抽出舊熱電偶, 注意熱電偶與樣品盤高度間距約2mm 高度



樣品平台校正

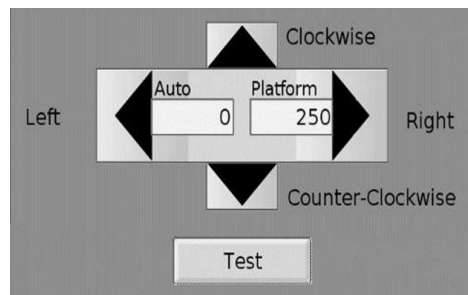
□ 快速平台校正

1. 檢查白金掛勾 是否彎曲
2. 檢查樣品盤是否變形
3. 檢查TGA機台水平(樣品盤需在爐口正中心)
TGA掛空盤並調整平台與樣品盤的間距1mm
4. 選擇觸控螢幕Calibrate/Platform(Autosampler), 擺放空盤於平台上
5. 調整平台手臂與白金掛鉤的最佳交接位置
6. 進樣測試、如有不佳 重複調整直到最佳位置



樣品平台校正

□ QNX



Auto Function	Description
Counter-Clockwise Arrow	<i>AutoTGA Only:</i> Moves the sample tray counter-clockwise one position.
Clockwise Arrow	<i>AutoTGA Only:</i> Moves the sample tray clockwise one position.
[Auto Number]	Displays the relative position of the tray. Used with the Counter-Clockwise and Clockwise buttons to adjust the tray position.
Test	Moves the sample tray the number of positions you selected and saves the information.



樣品平台校正

□ 舊版



Key Name	Description
CCW	<i>AutoTGA Only:</i> Moves the sample tray counter-clockwise one position.
CW	<i>AutoTGA Only:</i> Moves the sample tray clockwise one position.
[Number]	Displays the relative position of the tray. Used with the CCW, CW, LEFT, and RIGHT keys to adjust the tray position.
Left	Moves the sample arm to the left one position.
Right	Moves the sample arm to the right one position.
Test	Verifies the calibration adjustment you have made.
Save	Stores the calibration values.
Exit	Leaves the tray calibration touch screen menu.



TGA故障排除

- Error 66 / Error 81
- Error 119
- TGA不掛白金盤 卻告知已有懸掛樣品盤
- 白金掛勾與白金盤沾黏無法退盤



TGA故障排除

Advantage Instrument Messages - Microsoft Internet Explorer provided by TA Instruments - Waters, LLC

file:///C:/Program%20Files/TA%20Instruments/Thermal%20Advantage/Qseries/Help/QInstErr/InstErr.htm#TGA_Messages.htm

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Advantage Instrument Messages

Contents Index Search

Search - GO Powered By RoboHelp

Message 66

Instrument temperature limits exceeded. Run terminated.

Problem:

One of the thermocouples in the instrument is indicating a temperature that is outside the instrument's operating limit.

The following signals are checked for each instrument:

- ▶ DSC: Tzero temperature °C, heater temperature °C, flange temperature °C, heater temperature mV, Tzero mV
- ▶ DMA: Sample temperature °C
- ▶ SDT: Sample and heater temperature °C
- ▶ TGA: Sample and heater temperature °C
- ▶ TMA: Sample and heater temperature °C

Solution:

Try one or more of the following:

1. Try running the sample in an inert atmosphere and/or to a lower temperature. If the sample starts to burn during an experiment, it can raise the temperature beyond the limit.
2. DSC: Turn the RCS or LNCS on at least 20 minutes prior to starting the run.
3. Check the thermocouples to make sure they are clean and that they are showing reasonable values on the RealTime Signal Display screen.
4. Reset the saved instrument parameters.

Call TA Instruments for service, if the problem persists.

Windows taskbar: C:\Program Files\TA... TGA操作訓練 ppt [TA Instrument Explorer error 119.JPG - 小畫家 QSeries - [Q500-1573... Advantage Instrument...

上午 11:57 星期五 2013/5/3



TGA故障排除

Advantage Instrument Messages - Microsoft Internet Explorer provided by TA Instruments - Waters, LLC

file:///C:/Program%20Files/TA%20Instruments/Thermal%20Analysis/Advantage/QSeries/Help/QuickStart/InstErr.htm#TGA_Messages.htm

我的最愛 Advantage Instrument Messages

Contents Index Search

Message 81

Bad temperature reading. Hardware error. Run terminated.

Problem:
The difference between the heater temperature thermocouple and the sample temperature thermocouple is too large.

Solution:
Try one of the following procedures to solve the problem, depending on the instrument currently displaying the error. Then restart the experiment.

1. Check the sample thermocouple and replace if necessary. (DSC, TGA, TMA, SDT)
2. Check the reference thermocouple continuity, it should be about 2 ohms. (SDT)
3. Check the heater thermocouple and replace if necessary (DSC, TGA)
4. Clean the furnace housing. (TGA standard furnace, TMA, SDT)
5. Call TA Instruments for service.

下午 12:00 星期五 2013/5/3

TGA故障排除

Advantage Instrument Messages - Microsoft Internet Explorer provided by TA Instruments - Waters, LLC

file:///C:/Program%20Files/TA%20Instruments/Thermal%20Analysis/Advantage/QSeries/Help/QuickStart/InstErr.htm#TGA_Messages.htm

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Advantage Instrument Messages

Contents Index Search

Message 119

Heat exchanger, no flow. Run stopped.

Problem:
One of the following situations could be causing the message.

- There is air in the water lines.
- The heat exchanger is not connected, is low on water, or is not functioning properly.

Solution:
Try one or more of the following operations to solve the problem, then restart your experiment.

1. Restart the instrument. Select Control/Prime Exchanger to run the exchanger a few minutes to remove all of the air. Select STOP to halt operations when the air is gone.
2. Connect the heat exchanger.
3. Check the water level in the water reservoir bottle and fill to at least 2/3 full or below the upper hose fitting.
4. Call TA Instruments for service.

上午 11:53 星期五 2013/5/3

TGA 關機順序

□ 關機步驟：

- 等待TGA後的空氣冷卻自動停止或是爐溫回到室溫
- 完成後,執行Control \ Shutdown Instrument
- 出現Shutdown視窗,按Start
- 此時視窗自動關閉,TGA進行關機前參數回存動作
- 等待TGA前方Ready燈熄滅或LCD螢幕提示可以關機後,便可關閉TGA電源
- 關電腦
- 關氮氣/空氣



TA Universal Analysis 分析軟體操作解說

UA分析軟體實際演練說明



現場TGA操作問題 Q & A



TA Taiwan 維修人員

□ 北區維修:

張永威

□ 手機 0972-633337

□ E-mail AChang@tainstruments.com

□ 中區維修:

張瑞銓

□ 手機 0972-633336

□ E-mail RChang@tainstruments.com

□ 南區維修:

□ 程鈴雄

□ 手機 0972-633338

□ E-mail SCheng@tainstruments.com



2013年TA技術講座

TGA機台的原理與應用

日期	地點
2013年5月16日(四)	國立成功大學國際會議廳第二演講室 台南市大學路1號 (光復校區)
2013年8月2日(五)	國立台灣大學化學系潘貫講堂 台北市羅斯福路四段1號
2013年10月4日(五)	逢甲大學學思樓國際會議廳九樓 台中市西屯區文華路100號

許炎山

TA Instruments, Waters LLC

美商沃特斯國際股份有限公司台灣分公司

TA Taipei office: 104臺北市長安東路1段23號4F之5

Tel: 02-25638880 Fax: 02-25638870

C/P: 0928-168676 E/M : jhsu@tainstruments.com



開宗明義

量測物質重量隨環境與溫度變化之技術，統稱為TGA

- 溫度
 - 室溫以下
 - 室溫以上 到 1000°C
 - 室溫以上 到 1500°C
 - 室溫以上 到 >1500°C
- 壓力
 - 常壓
 - 高壓
 - 負壓
- 環境
 - 鈍氣(如氮氣、氬氣、氫氣)
 - 含氧(如空氣、氧氣)
 - 含濕(RH控制)
 - 特殊(反應型成分、腐蝕型成分、爆炸型成分...等)



TGA 951 : 水平爐體, 水平氣滌



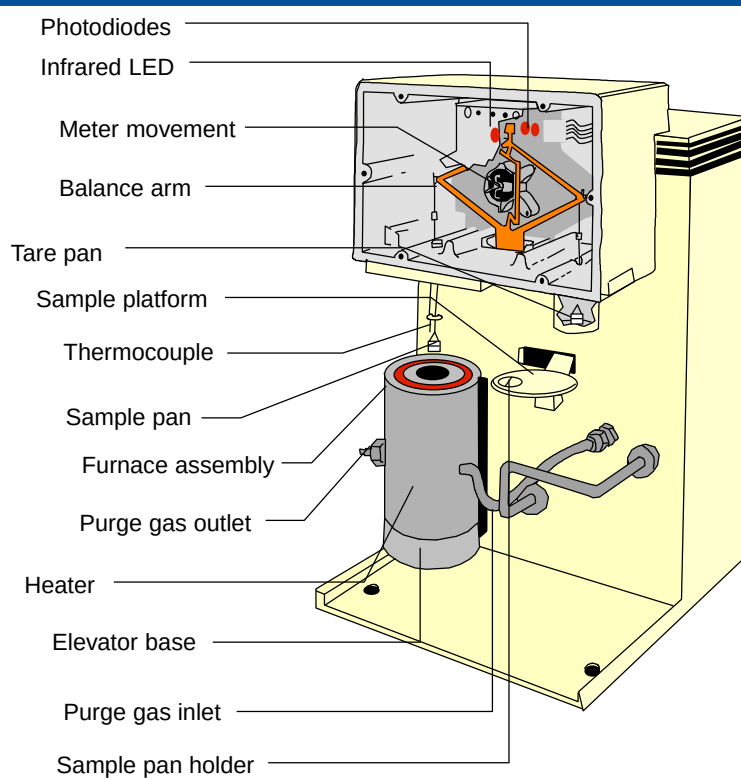
TGA 2950 : 垂直爐體, 水平氣滌



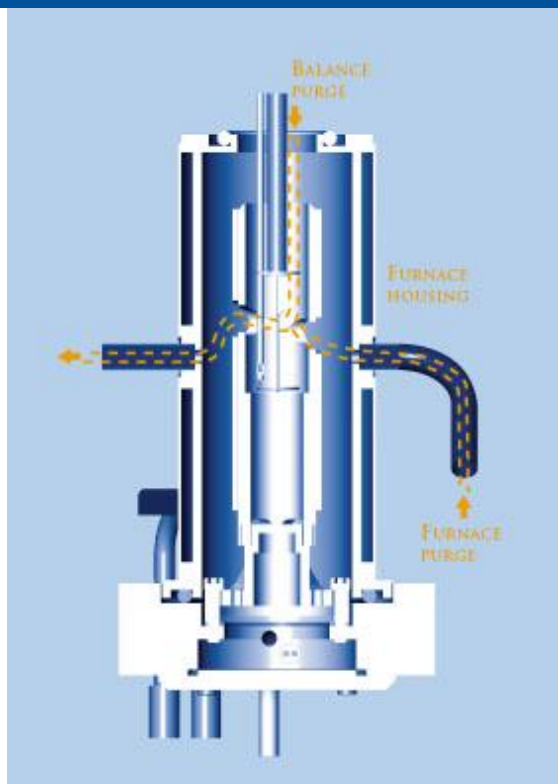
TGA Q500 : 垂直爐體, 水平氣滌



TGA: Schematic Diagram



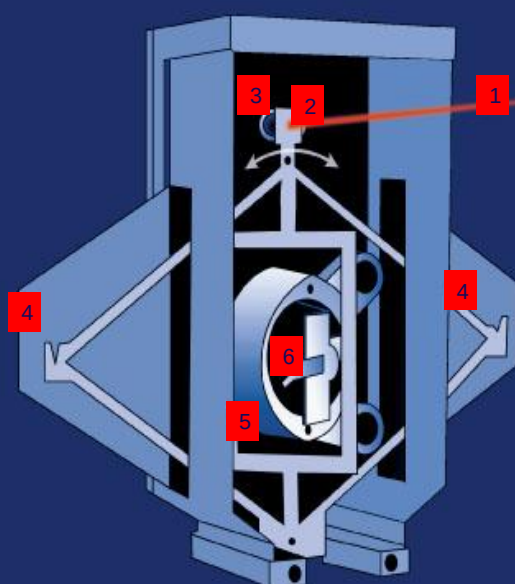
Unique Gas Flow Design Benefits



There is a fresh, continuous, horizontal sweep of purge gas across the sample. This provides better resolution of transitions because of faster burn-offs. Using designs where the sample is in a sample cup or in a furnace cup prevent a fresh sweep of gas across the sample. A stale pocket of gas can form over the top of the sample causing non-repeatable decomposition profiles.



Dual Range Microbalance



1. Light Source (not shown but the light beam is hitting the flag)
2. Flag (attached to the balance arm frames)
3. Photodiodes (one of two is barely visible behind the flag)
4. Balance Arms
5. Meter Movement
6. Taunt Band location (not visible)



Dupont DSC 910 TGA 951 and 990 DSC / TGA System

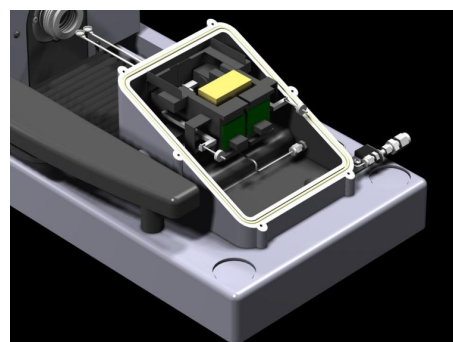
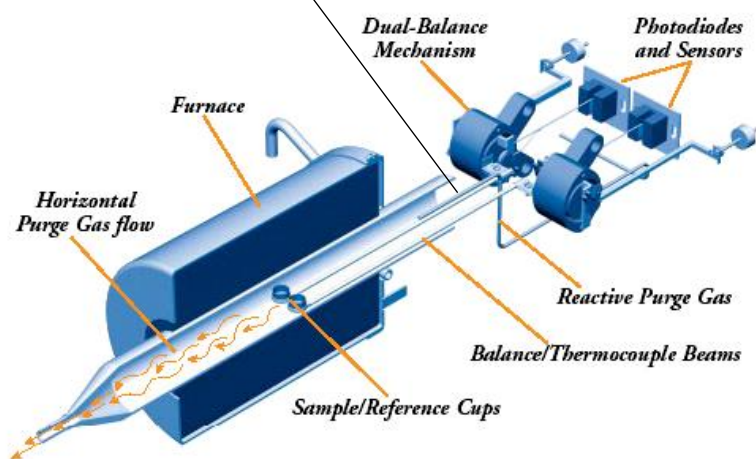


Q600 Simultaneous DSC-TGA



Reactive Gas Tubing and Inlet

內建獨立之反應性氣體管路, 直接
送入到樣品盤之上方避免擴散!



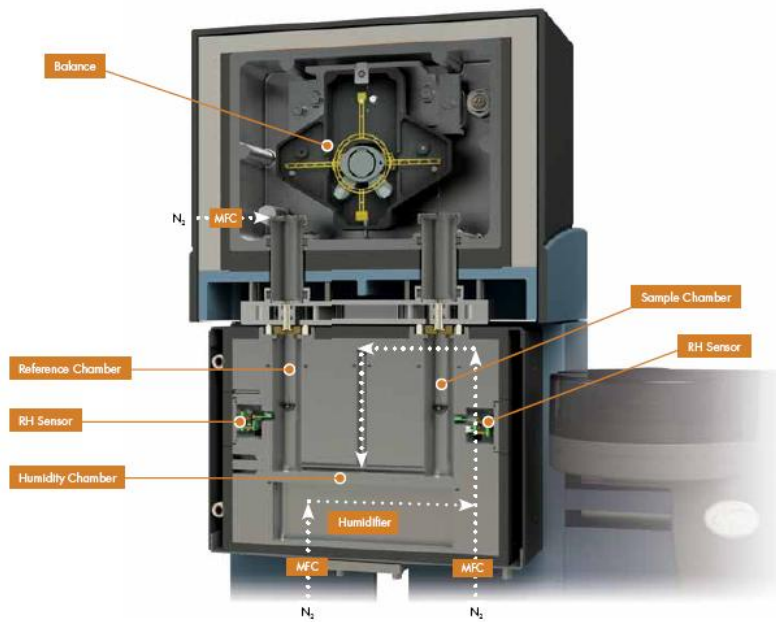
TGA-HP50



Pressure Limit :
50 bar
High Vacuum :
 1×10^{-6} torr
Temperature:
to 800 ° C



Dynamic Vapor Sorption Q5000 SA



Dynamic Vapor Sorption VTI-SA⁺



TGA General Considerations

(Experimental Effects)



TGA: What TGA Can Tell You

- Thermal Stability of Materials
- Oxidative Stability of Materials
- Composition of Multi-component Systems
- Estimated Lifetime of a Product
- Decomposition Kinetics of Materials
- The Effect of Reactive or Corrosive Atmospheres on Materials
- Moisture and Volatiles Content of Materials



TGA: Mechanisms of Weight Change in TGA

- Weight Loss:
 - Decomposition: The breaking apart of chemical bonds.
 - Evaporation: The loss of volatiles with elevated temperature.
 - Reduction: Interaction of sample to a reducing atmosphere (hydrogen, ammonia, etc).
 - Desorption.
- Weight Gain:
 - Oxidation: Interaction of the sample with an oxidizing atmosphere.
 - Absorption.

All of these are kinetic processes (i.e. there is a rate at which they occur).



TGA Curves are not 'Fingerprint' Curves

Because most events that occur in a TGA are kinetic in nature (**meaning they are dependent on absolute temperature and time spent at that temperature**), any experimental parameter that can effect the reaction rate will change the shape / transition temperatures of the curve. These things include:

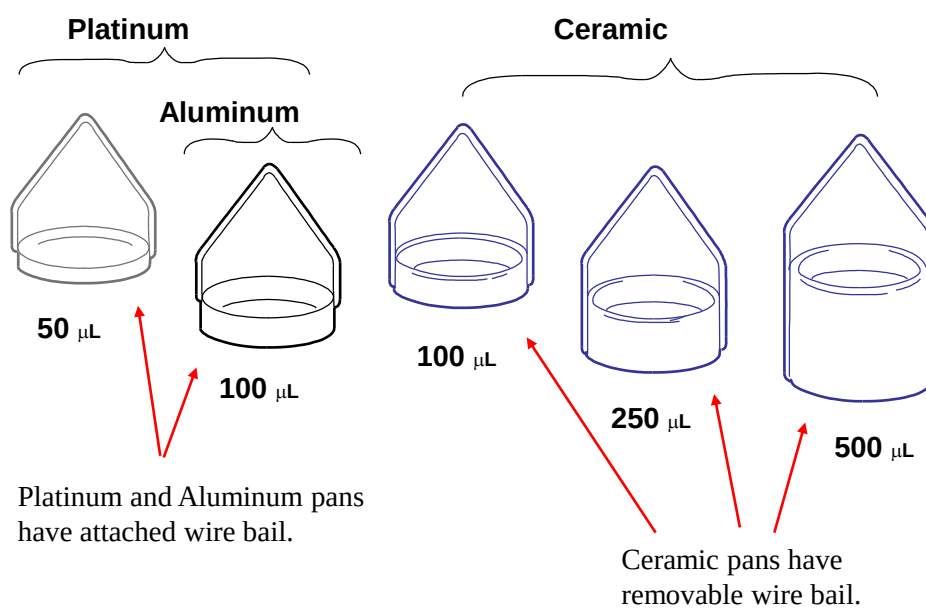
- Pan material type, shape and size.
- Ramp rate.
- Purge gas.
- Sample mass, volume/form and morphology.



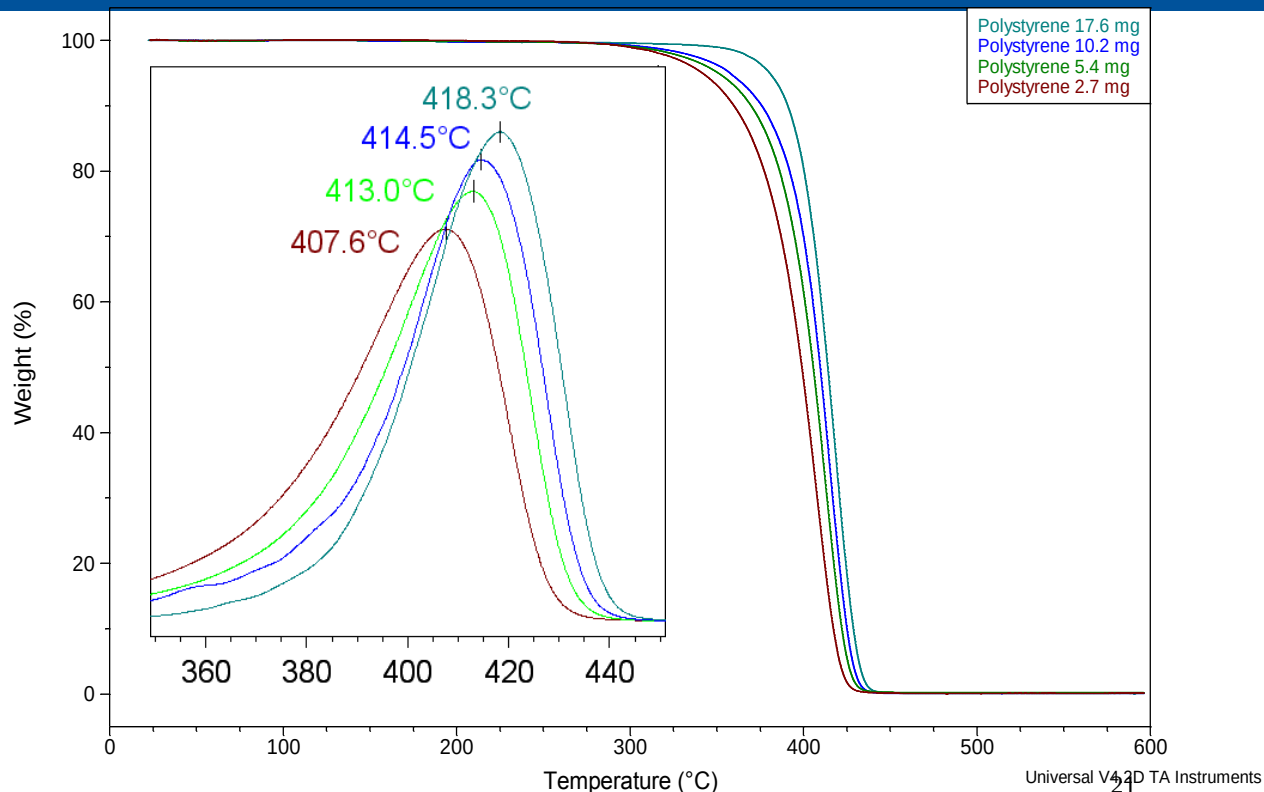
TGA: Q Series MFC and GSA



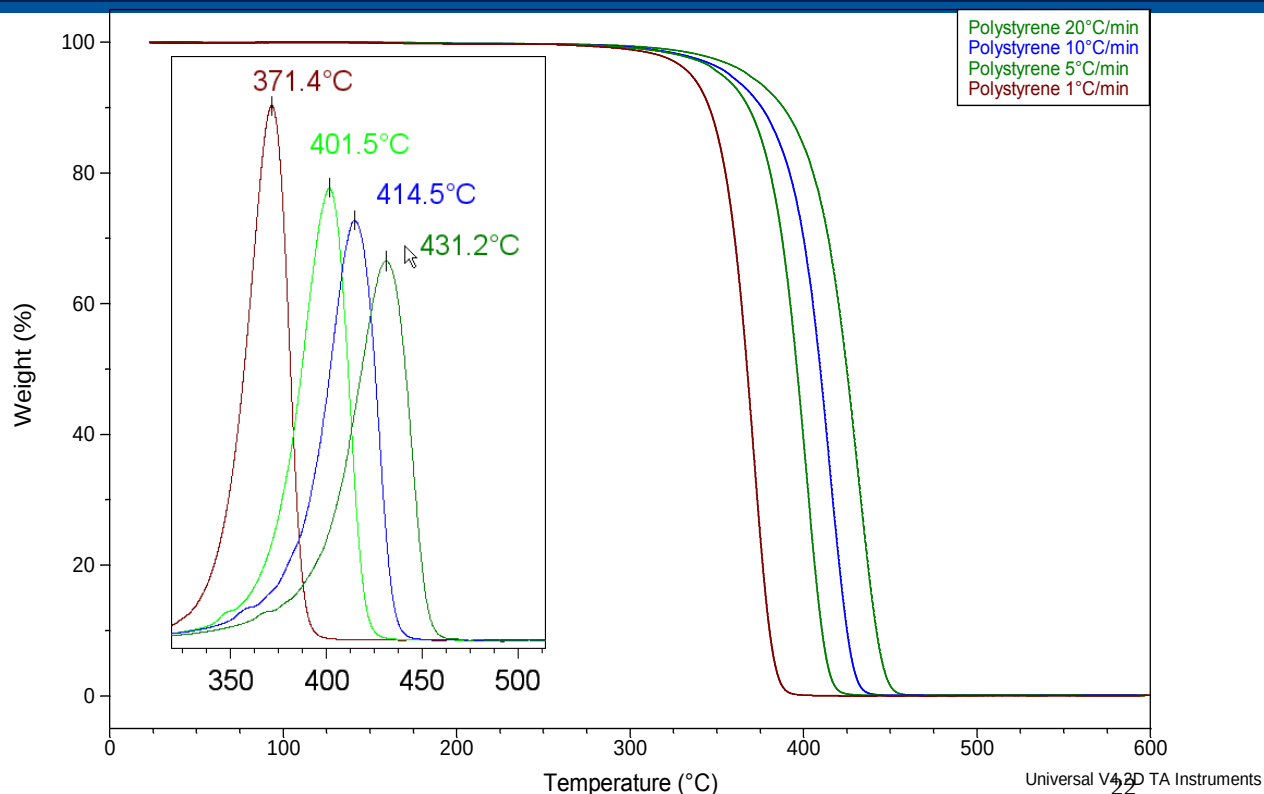
TGA: Sample Pans - Types/Sizes



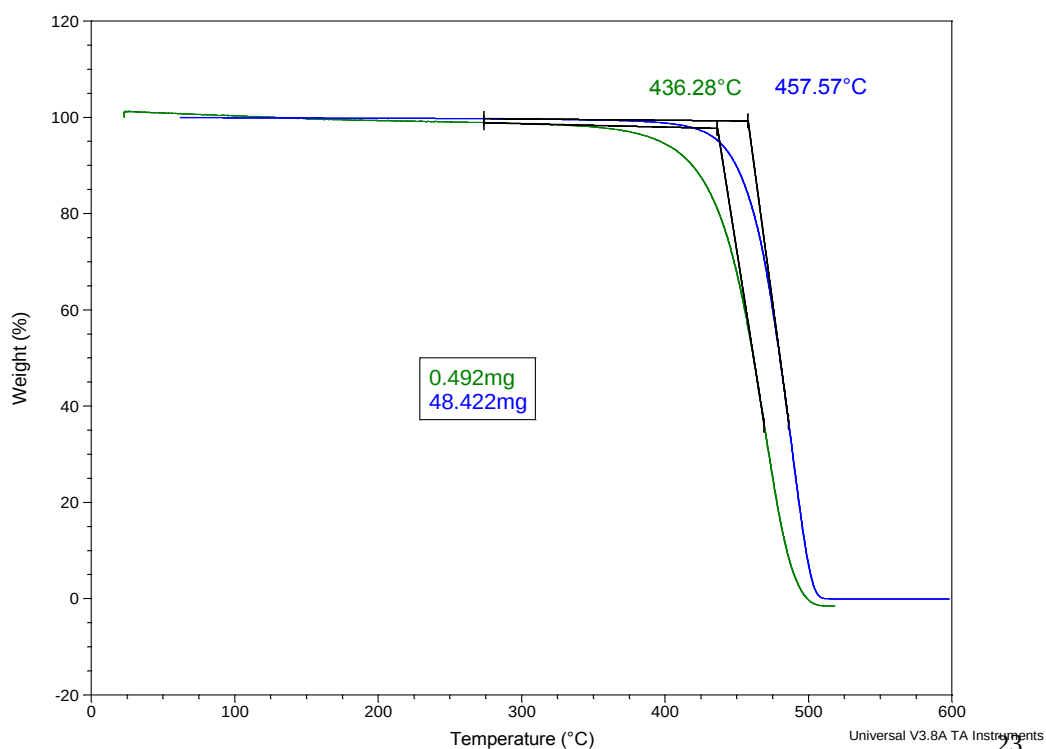
Effect of Sample Size on Decomposition Temperature of Polystyrene



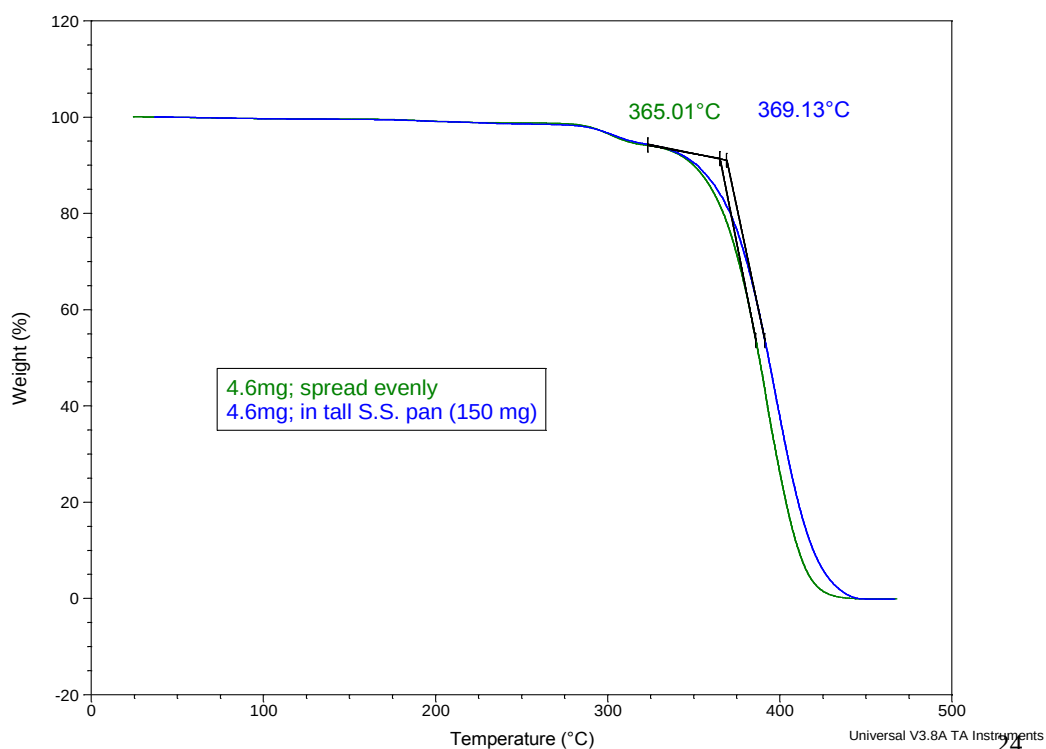
Effect of Heating Rate on Decomposition Temperature of Polystyrene



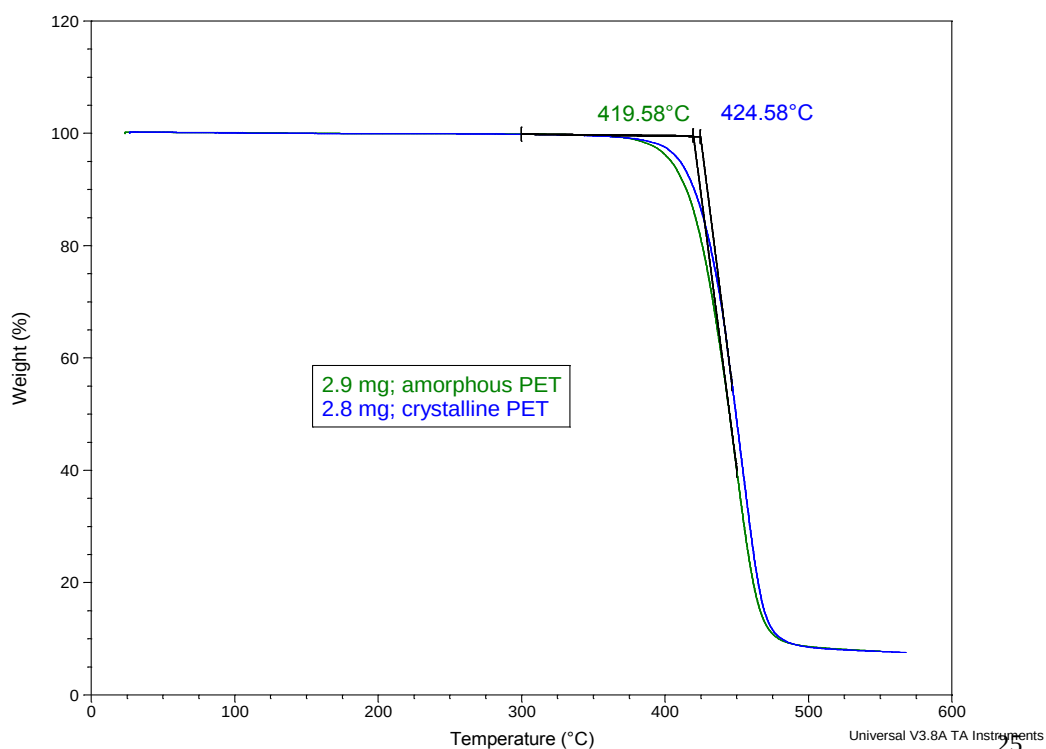
Mass Effect – Semi-crystalline PE



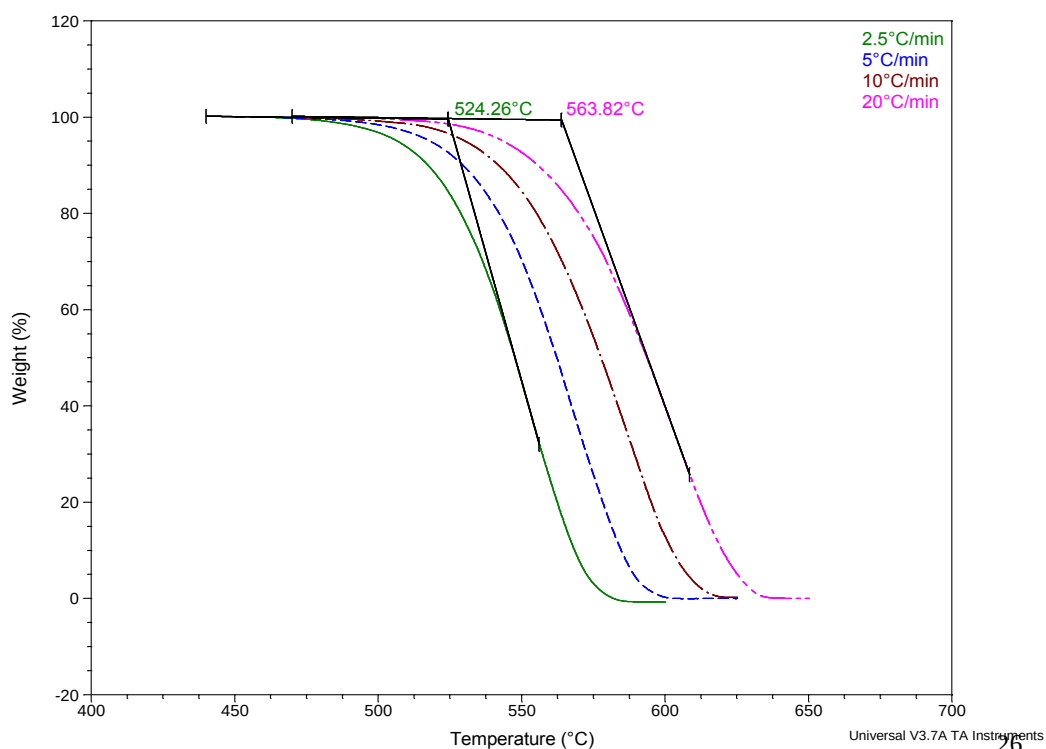
Pan Shape Effect – Amorphous PMMA



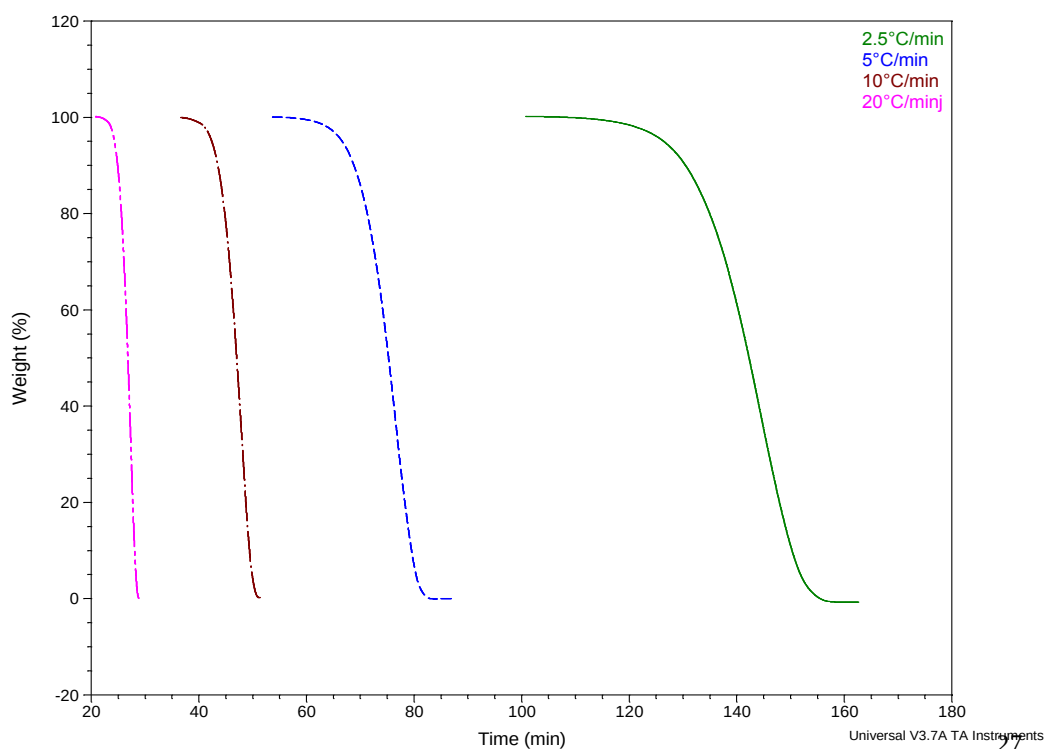
Sample Morphology Effects – PET



Shift in Onset with Ramp Rate



Time to Complete Degradation at Higher Temperatures



TGA Basic Applications

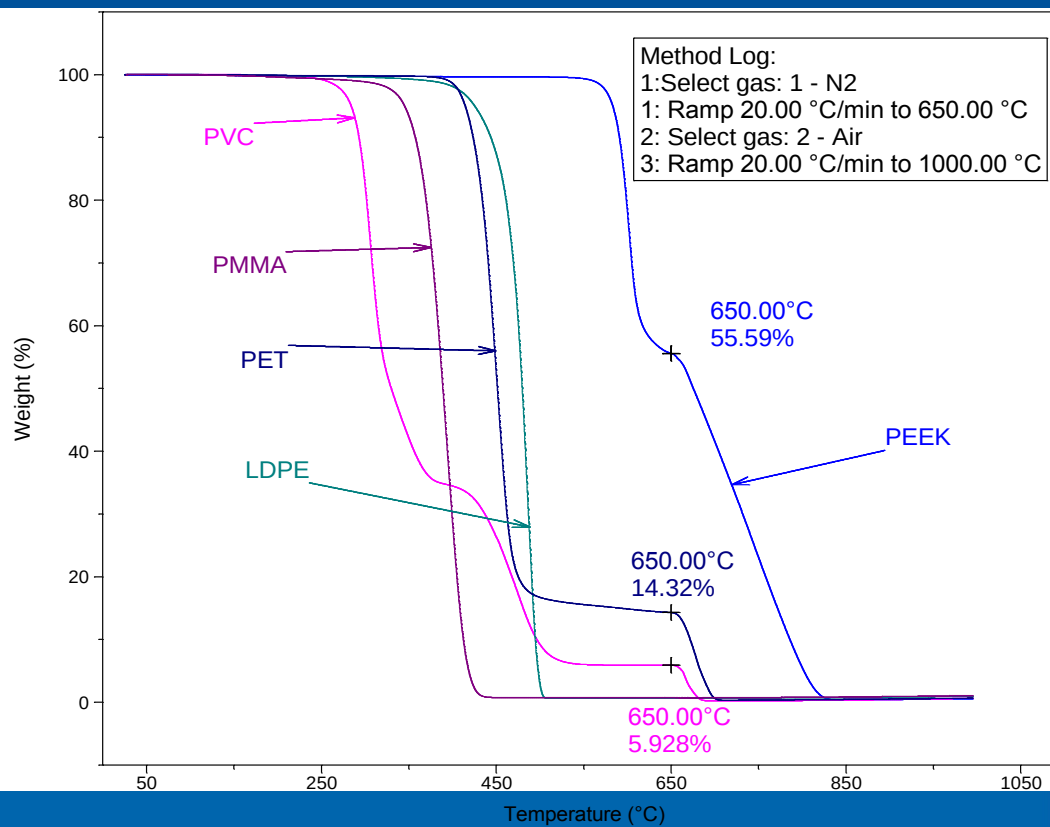
- Thermal Stability
- Compositional Analysis
- Oxidative Stability

- Thermal Stability
- Compositional Analysis
- Oxidative Stability

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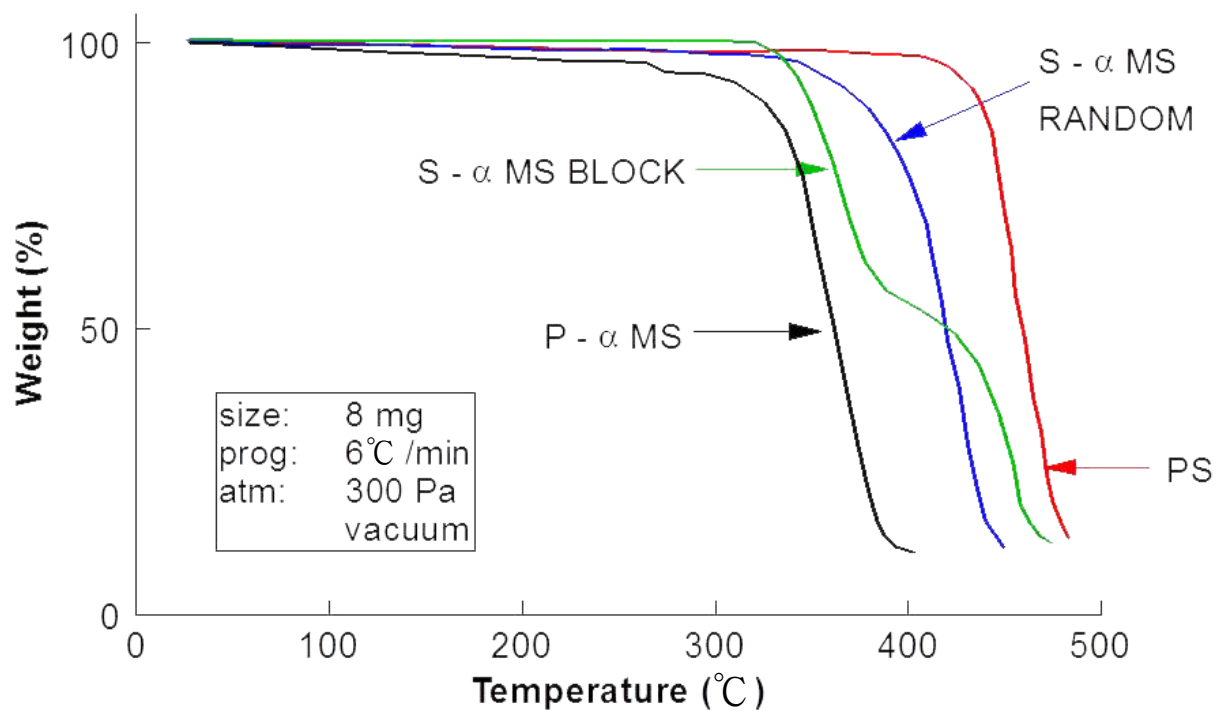
Thermal Stability of Polymers



30



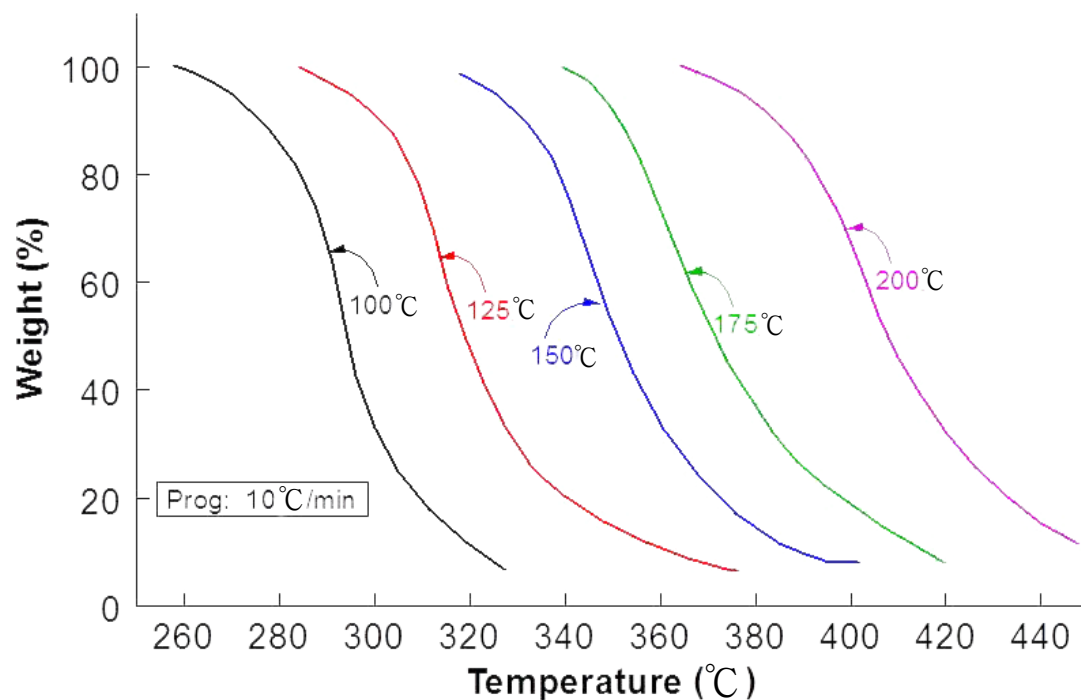
Block versus Random Copolymers



31



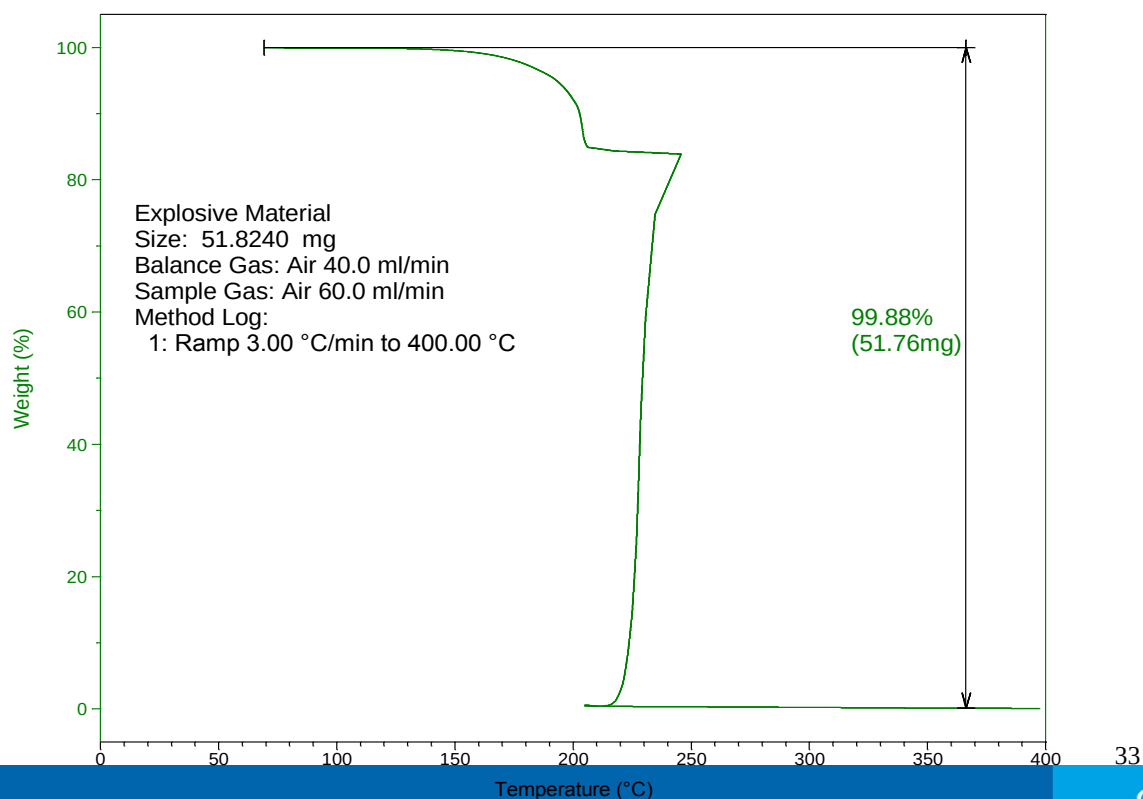
Decomposition of Epoxies Cured at Different Temperatures



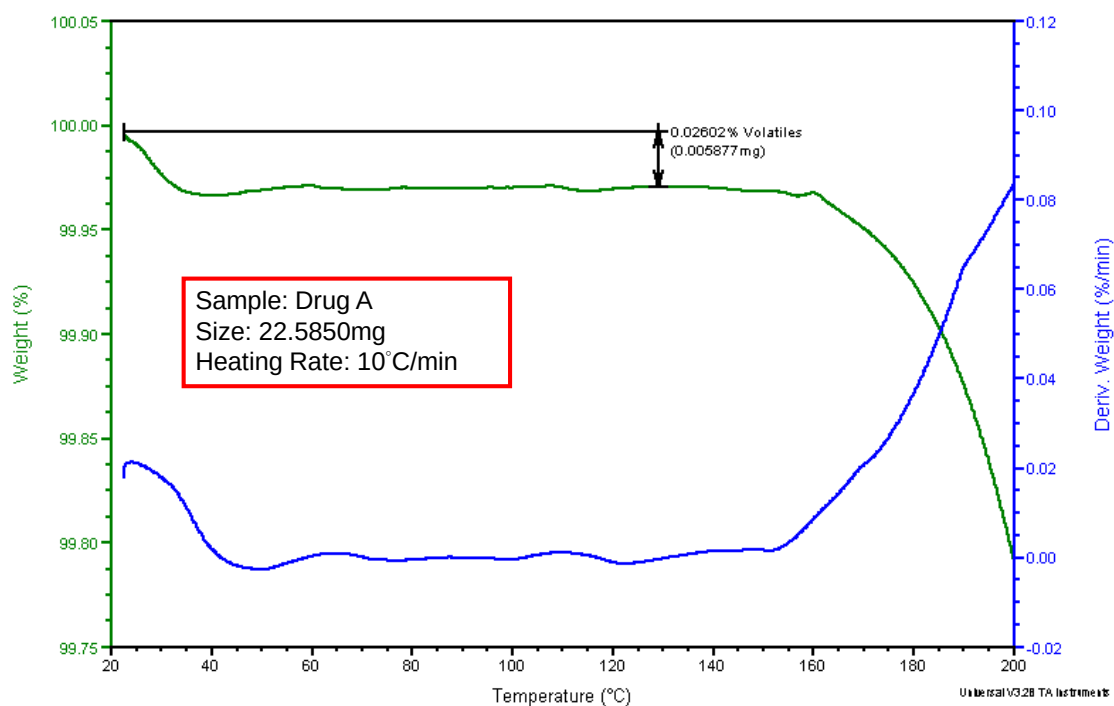
32



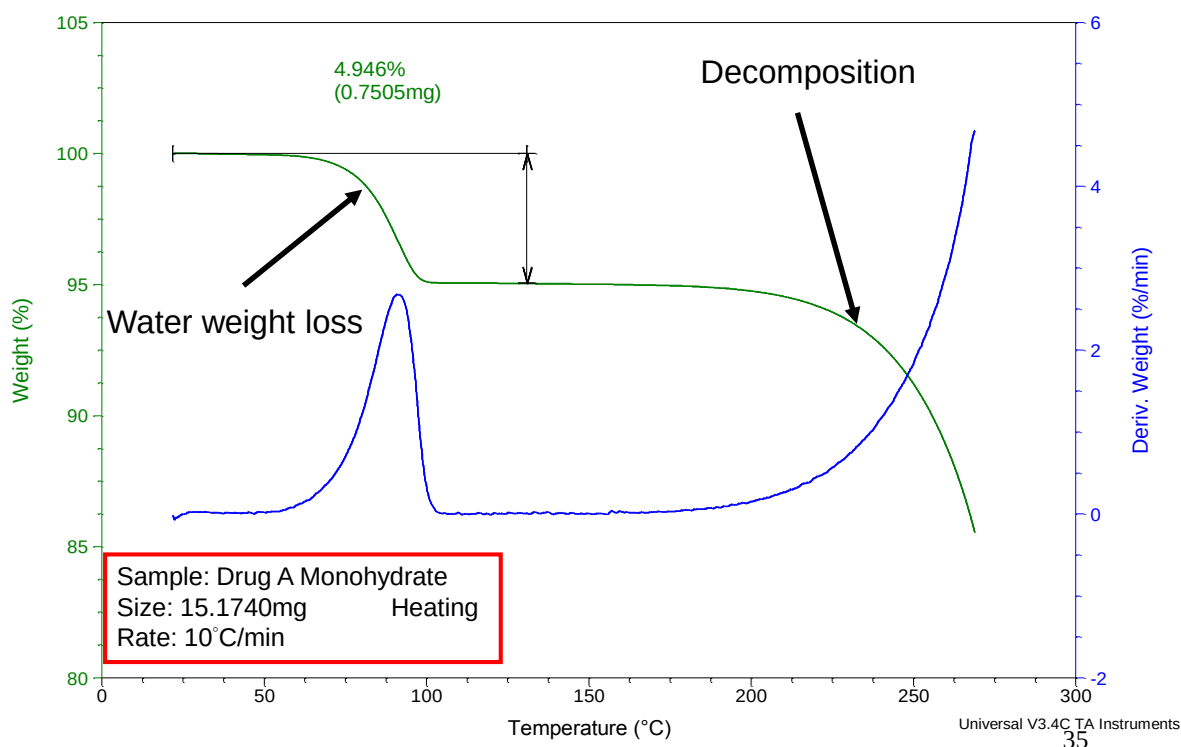
Thermal Stability of an Explosive Material



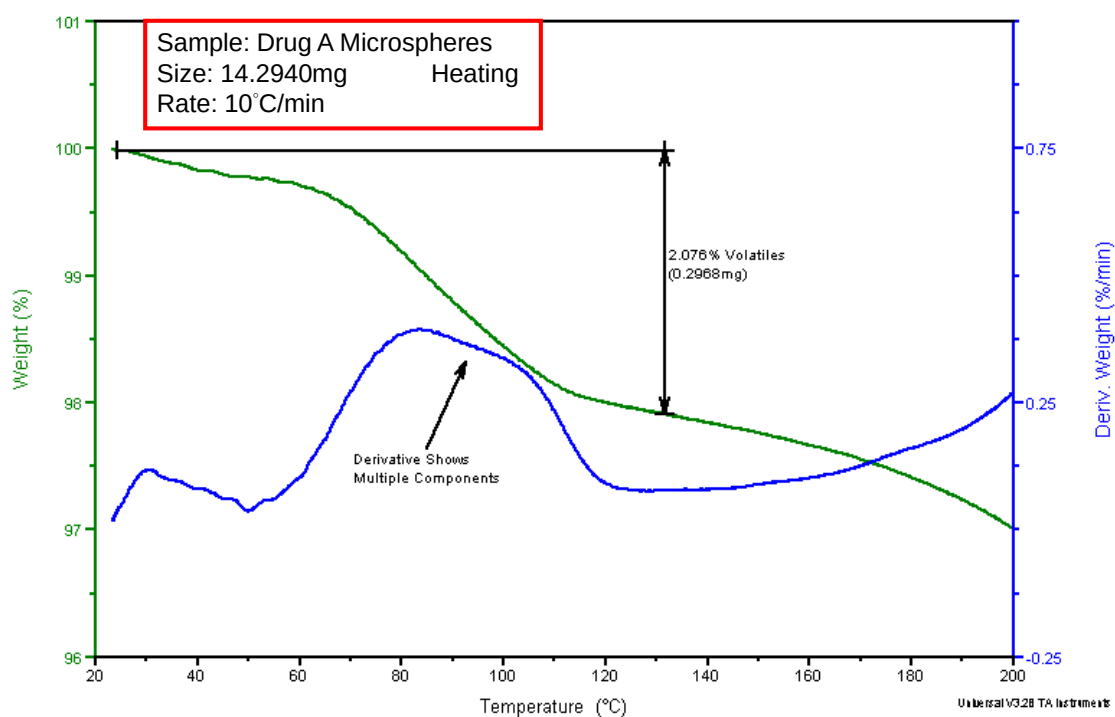
TGA of Drug A



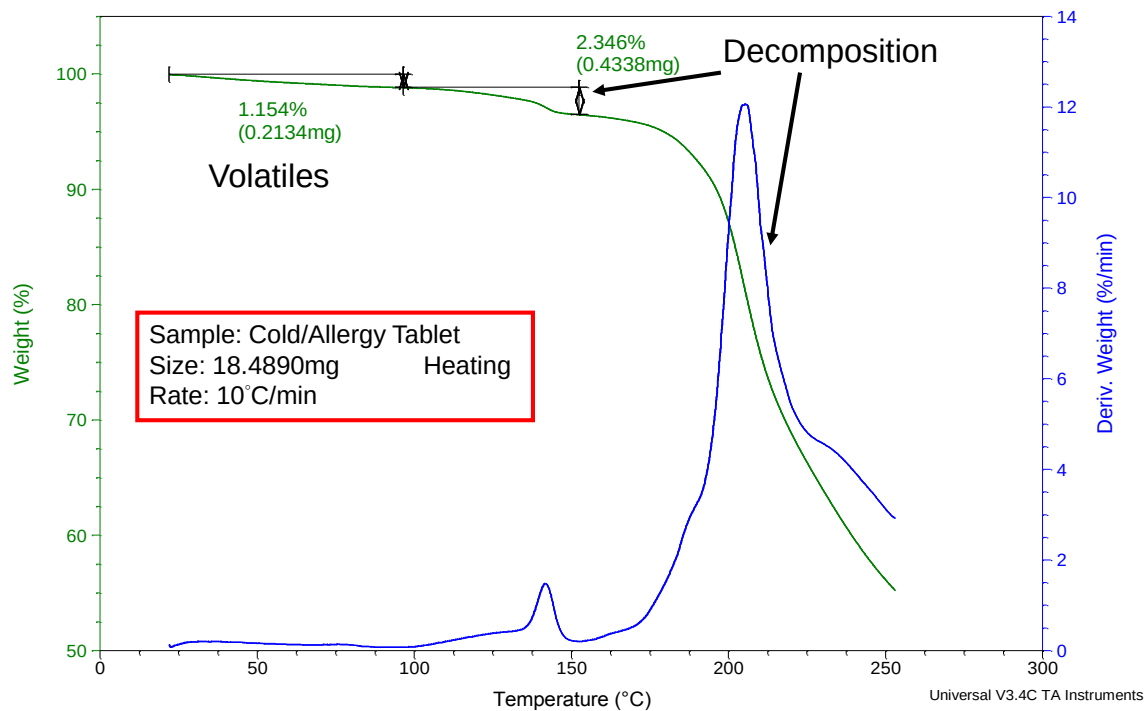
TGA of Drug A Monohydrate



TGA of Drug A Microspheres



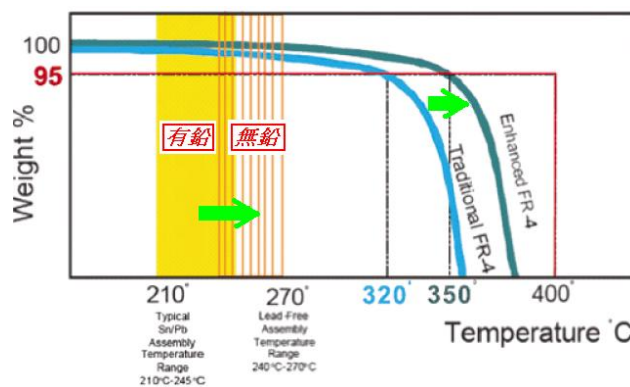
TGA Analysis of Cold/Allergy Tablet (冷過敏片)



37



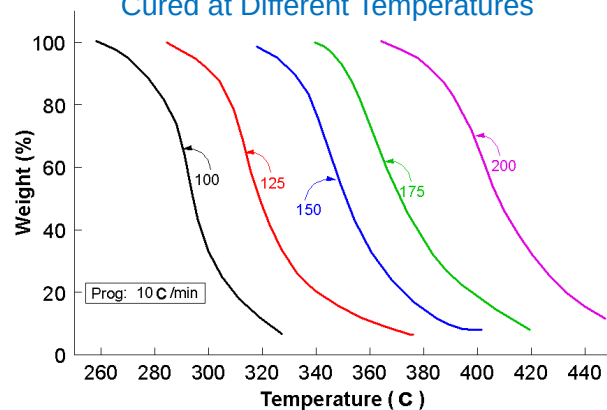
Decomposition of Epoxies



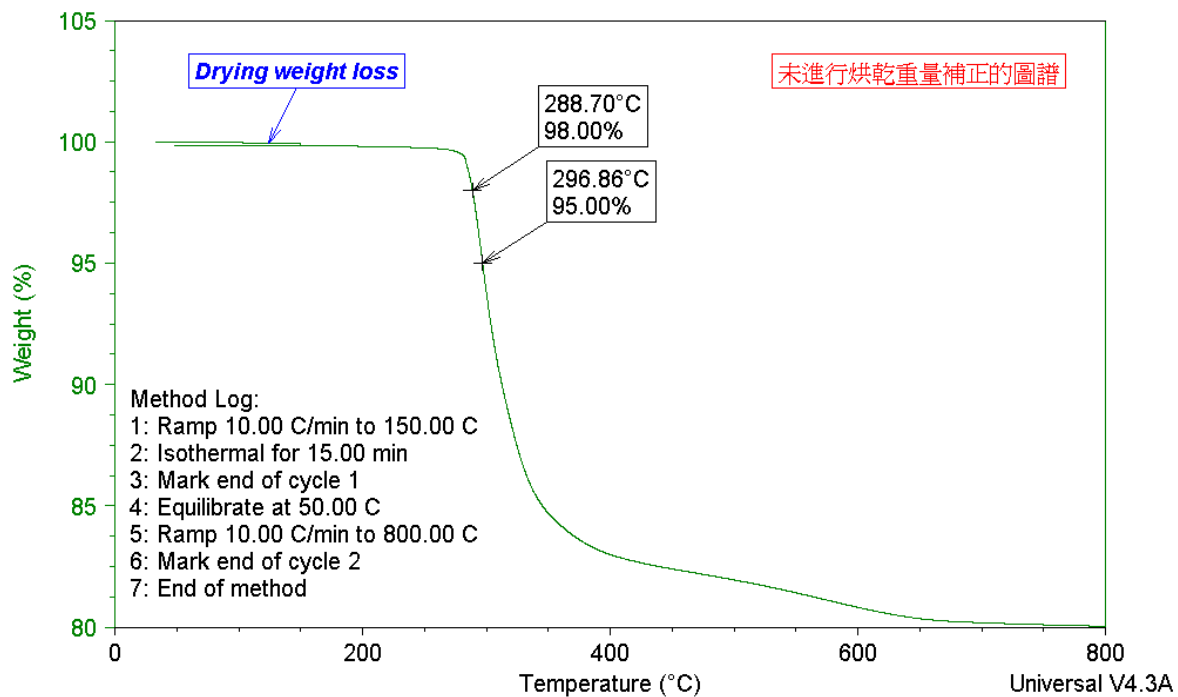
Td (裂解溫度)

一般 : > 310°C
Mid : > 325°C
High : > 340°C

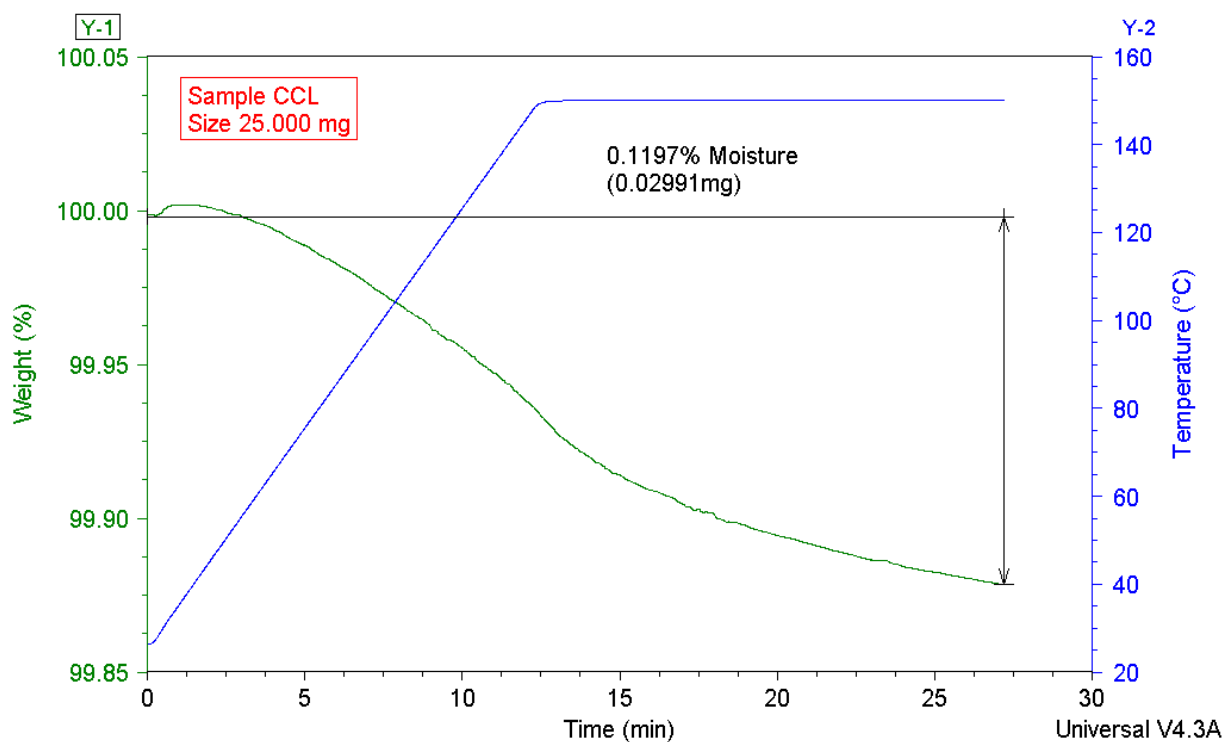
Cured at Different Temperatures



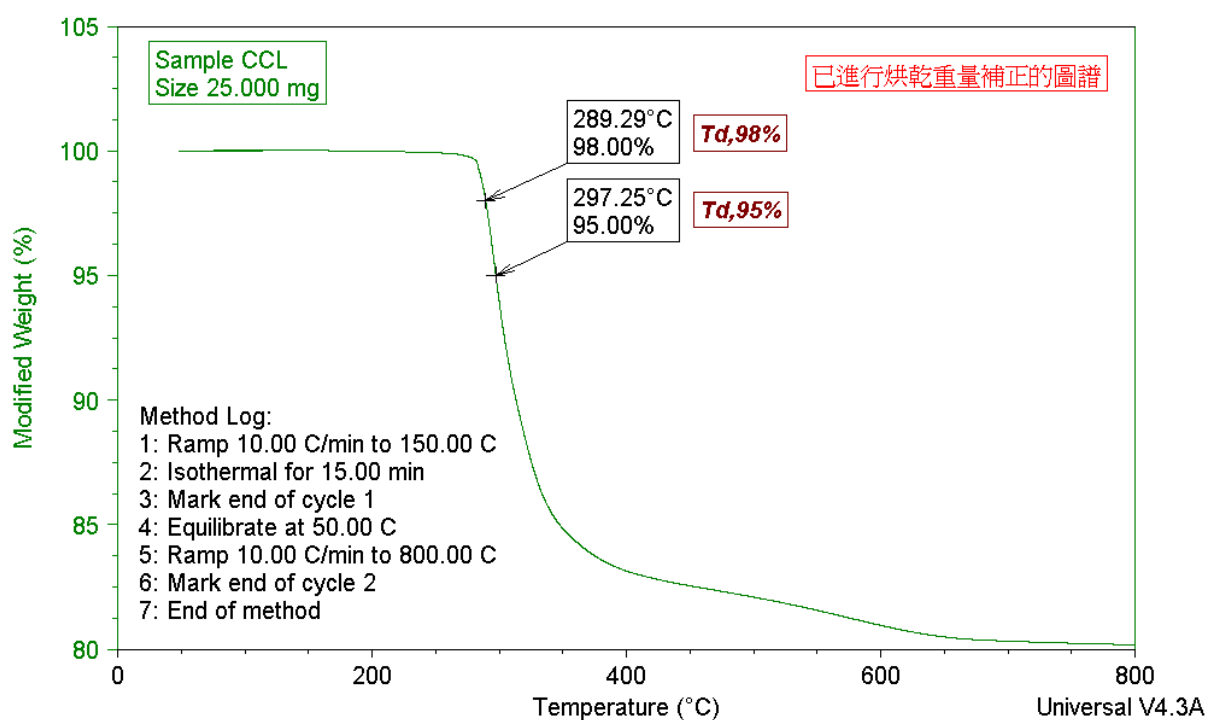
Td of TGA - Modified IPC-TM-650 2.4.24.6



Modified IPC-TM-650 2.4.24.6—Moisture Content

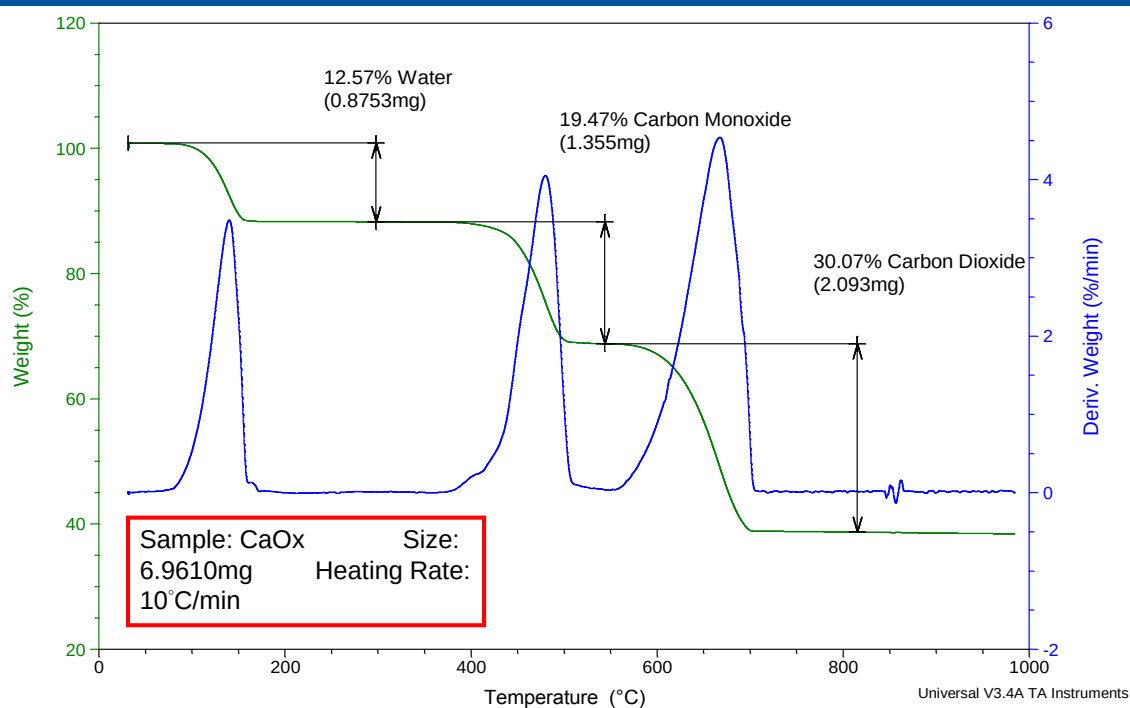


Td of TGA - Modified IPC-TM-650 2.4.24.6—Td



- Thermal Stability
- Compositional Analysis
- Oxidative Stability

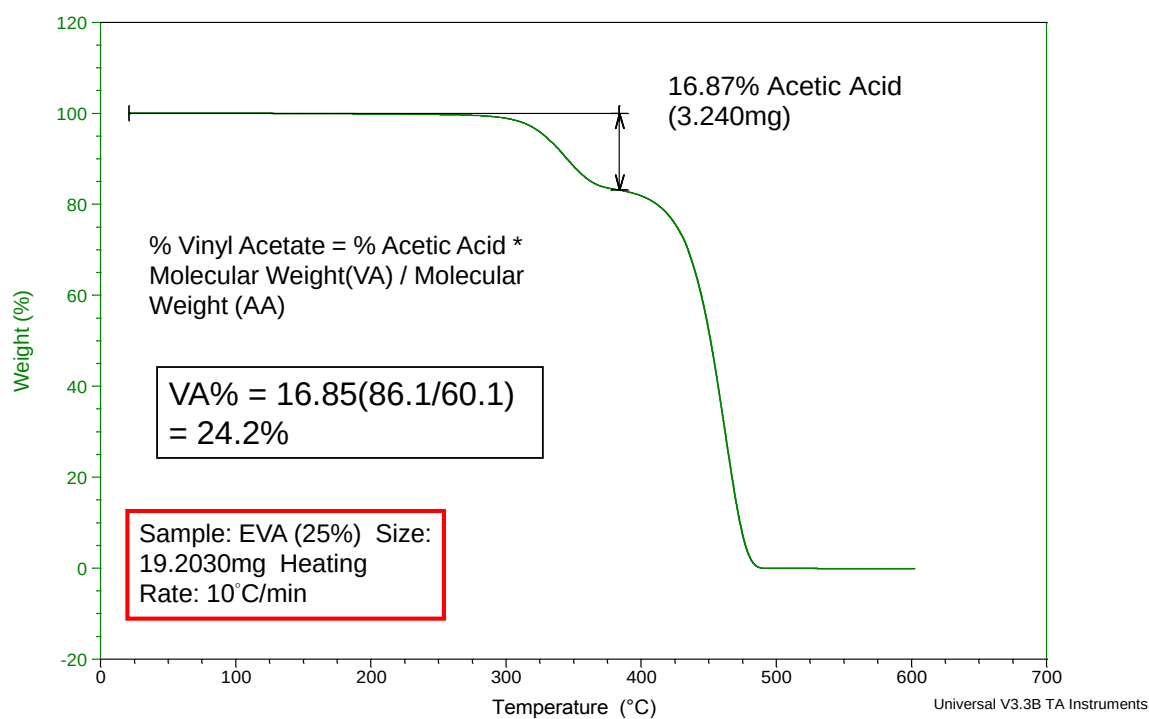
Calcium Oxalate



43



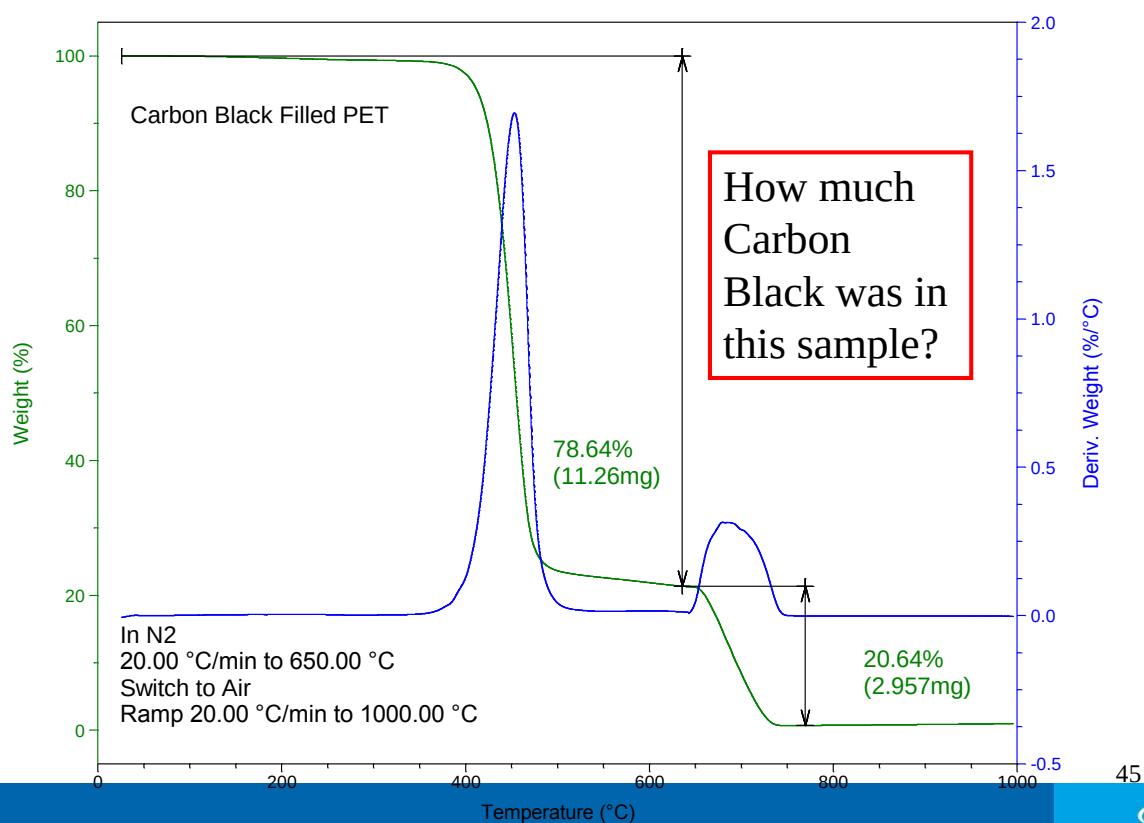
EVA Copolymer



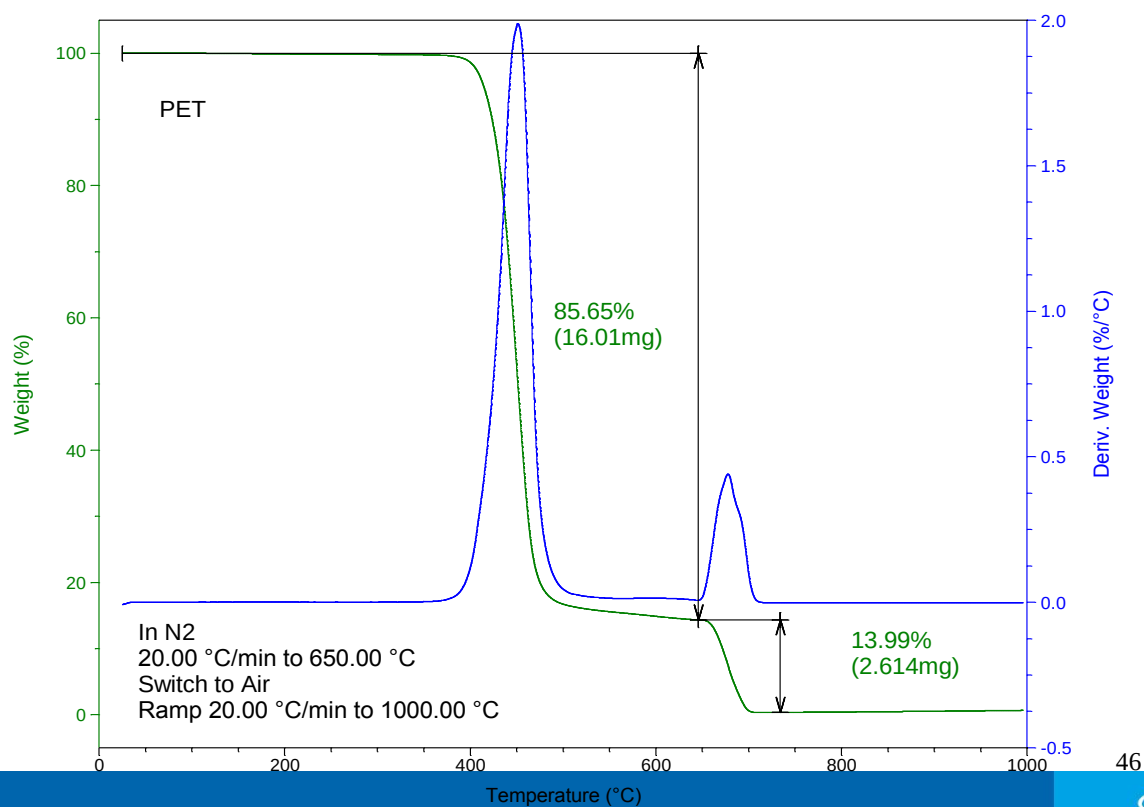
44



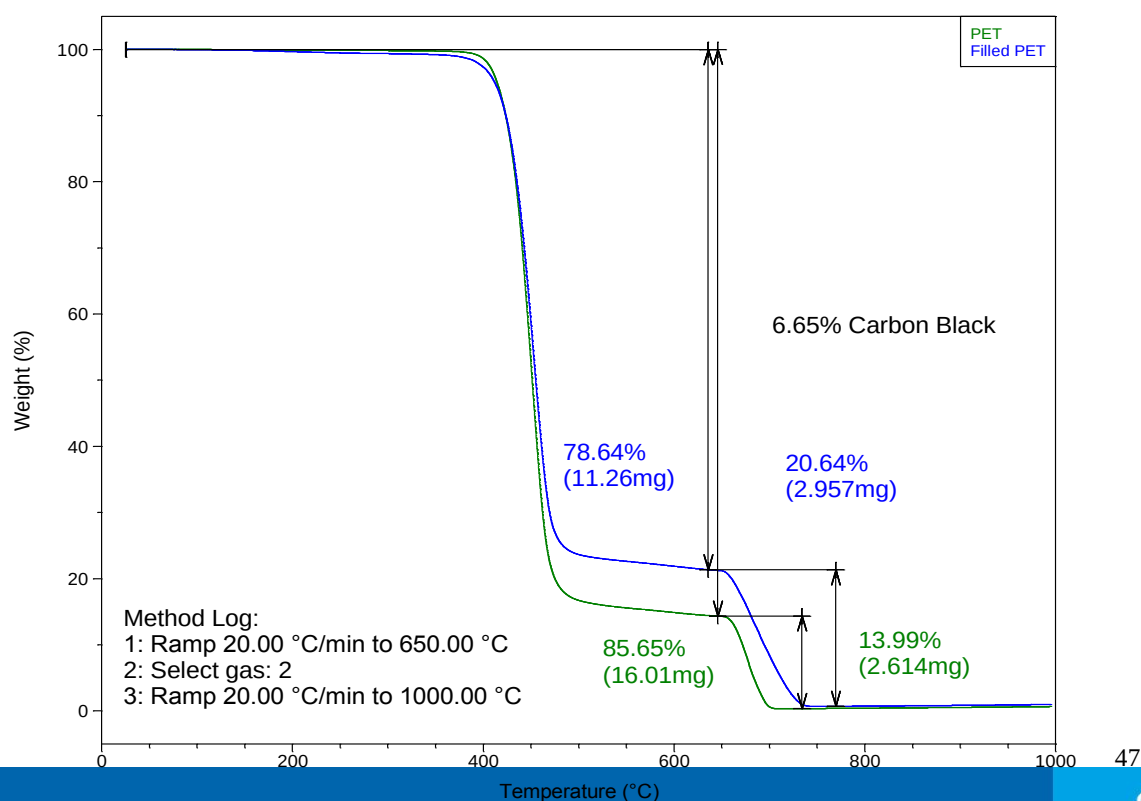
PET w/ Carbon Black Filler



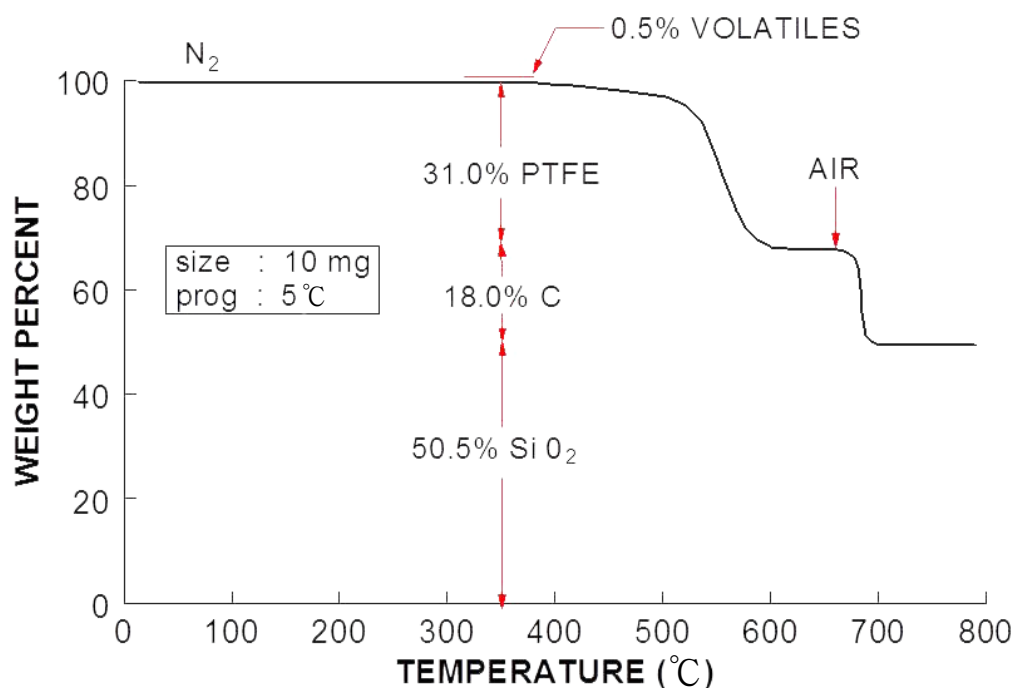
PET



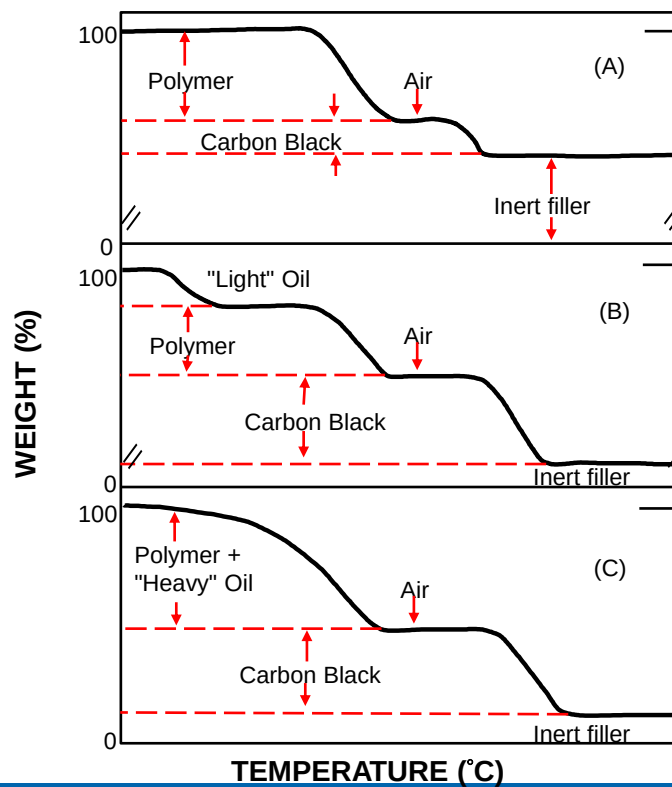
Comparison of Filled & Un-Filled PET



Composite Analysis



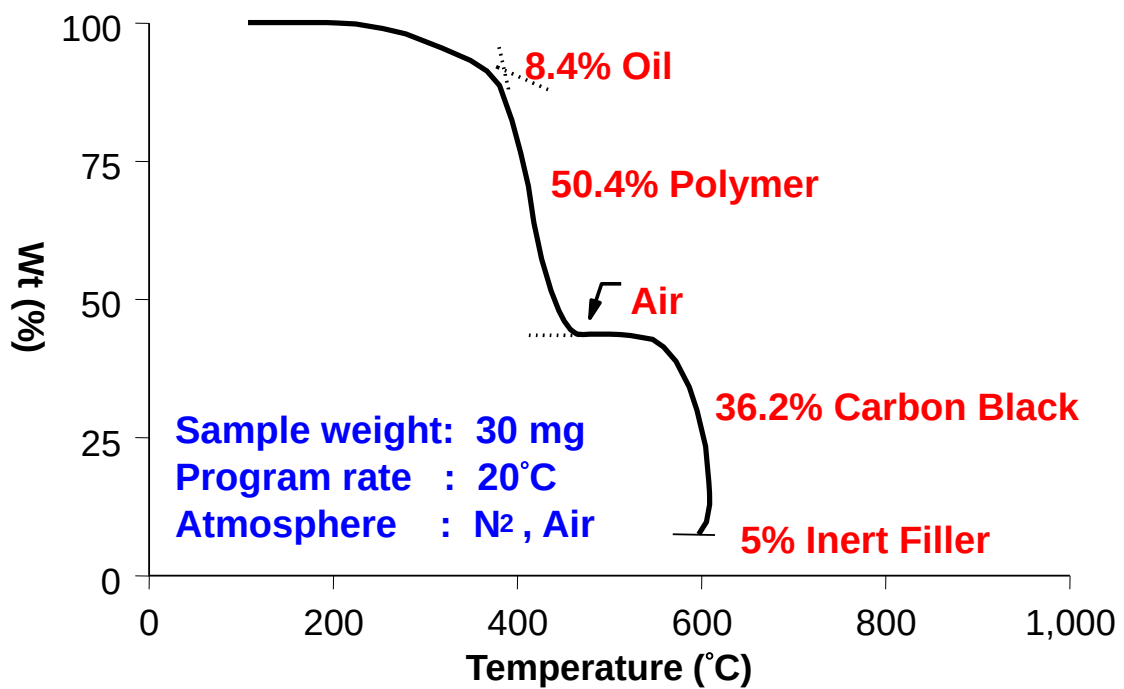
Filled Polymer Analysis



49



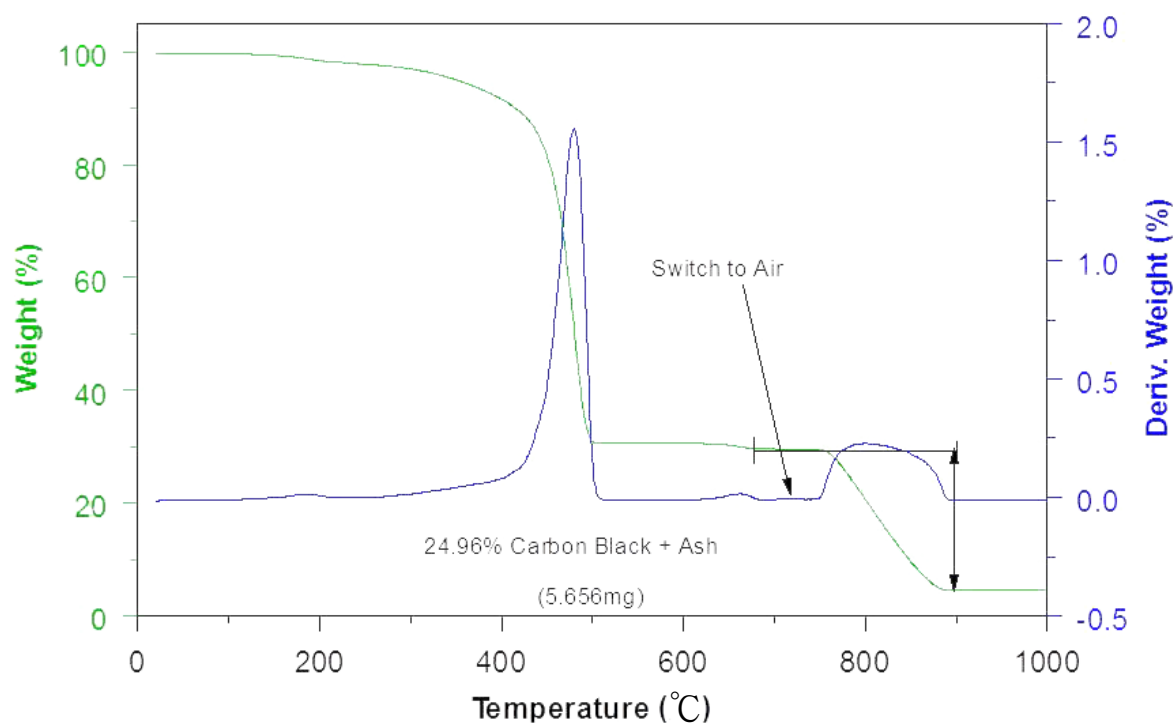
Styrene-Butadiene Rubber Analysis



50



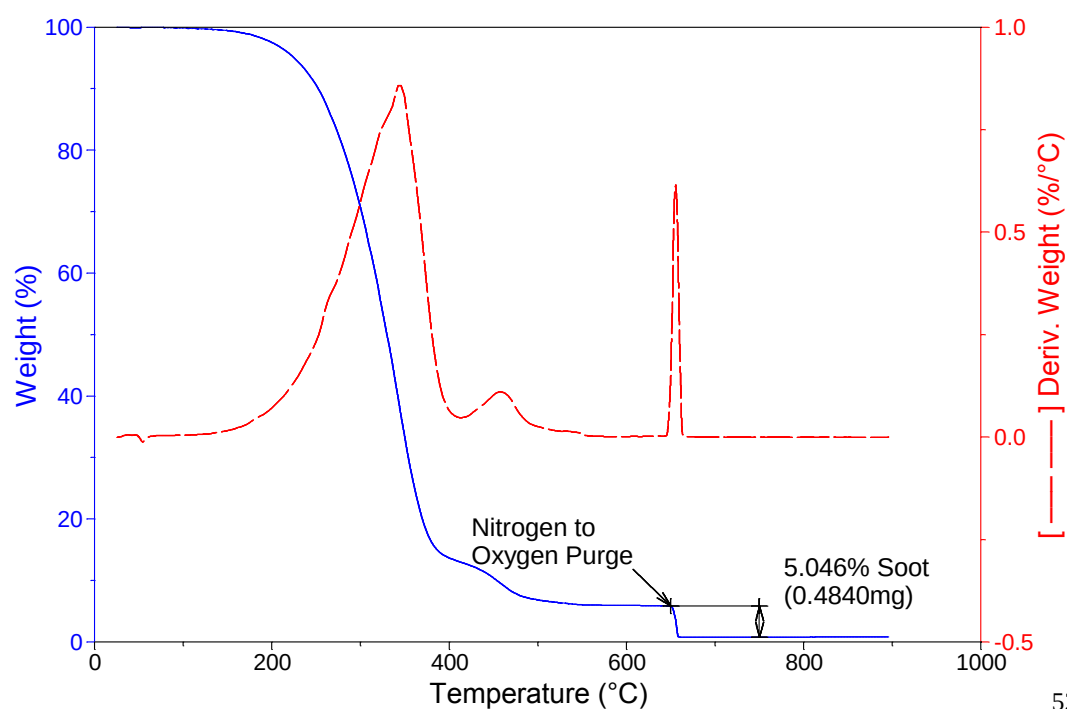
EPDM Rubber Analysis



51



Engine Oil Soot Analysis



52

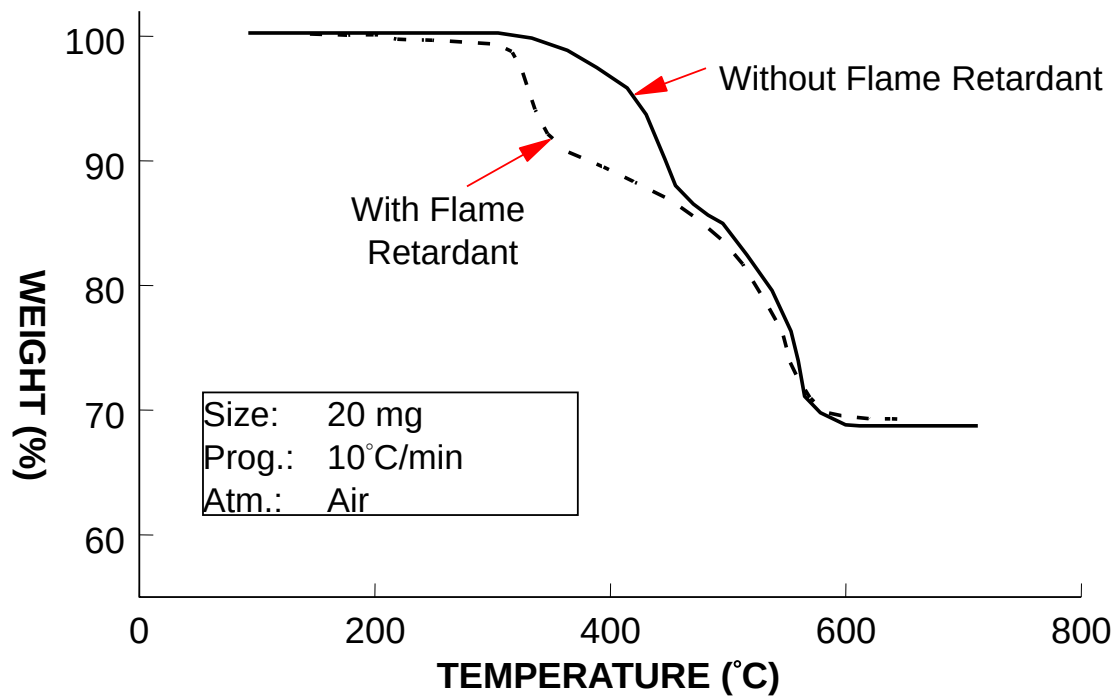


- Thermal Stability
- Compositional Analysis
- Oxidative Stability

53



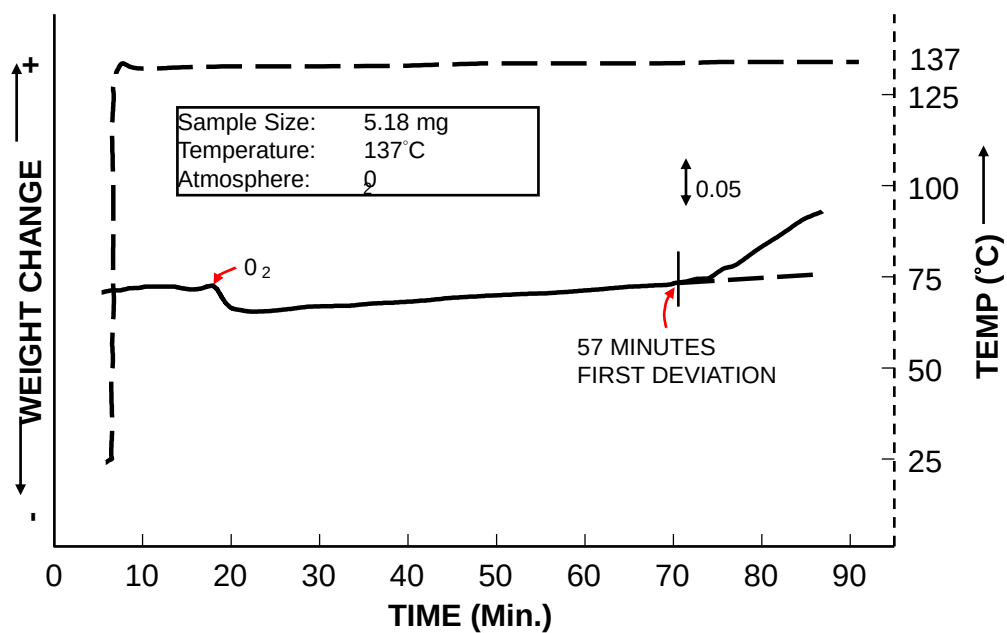
Oxidative Stability of Epoxy



54



Vegetable Oil Oxidative Stability



55



Advanced Applications



Standard TGA

Means of Enhancing Resolution

- Slower Heating Rate
 - longer runs.
- Reduced Sample Size
 - detection of small weight losses compromised.
- Change Purge Gas
 - not applicable in all cases.
- Pin-hole Hermetic Pans
 - not applicable in all cases.

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Hi-Res™ TGA - Alternate Methods

- Dynamic (Hi-Res) TGA
- Step - Wise Isothermal TGA

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Automated Stepwise Isothermal TGA (SWI)

- Heating stops (goes isothermal) when a certain rate of weight loss is reached, then resumes after this rate falls below a second defined value
- Operator defines the values for the rate of weight loss
- Incorrect values can cause artifacts
- Correctly set up, can give excellent resolution, but takes quite a bit longer

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Automated Stepwise Isothermal

- Advantages
 - Sample held isothermal until transition completed - thus excellent resolution of overlapping transitions
 - Permits careful control of reaction environment
 - Available on all TA Instruments TGA's
- Disadvantages
 - Difficult method development. May require several scans to optimize run conditions
 - Inappropriate parameter choices may produce artifacts
 - Long run time
- Utility
 - Routine Analysis of similar samples

60



Typical SWI Thermal Method

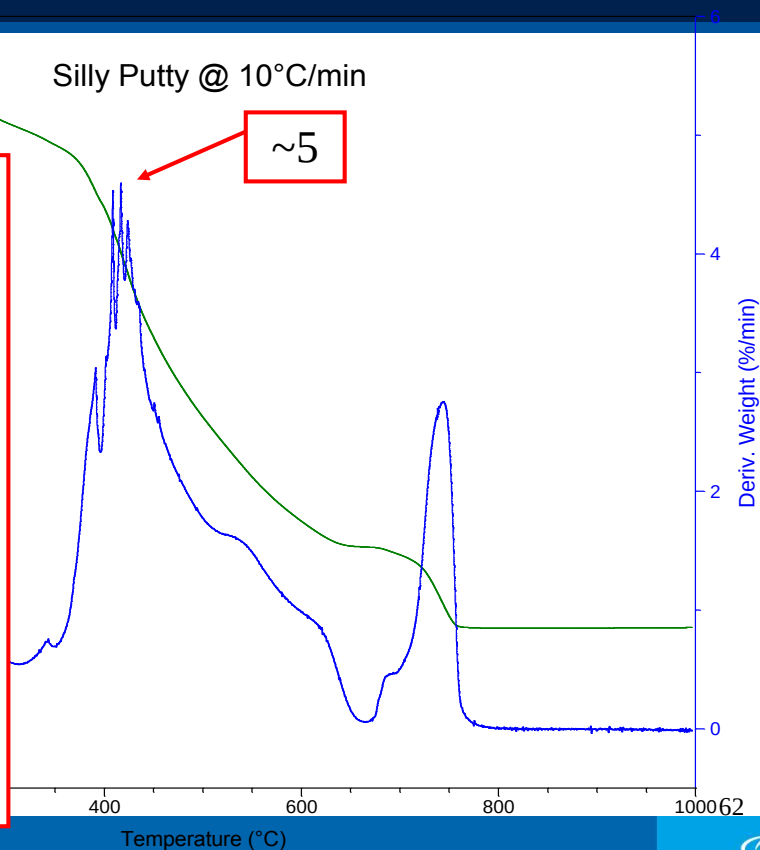
1. Abort next segment if $\%/min > 5.0$
2. Ramp $10^{\circ}C/min$ to $1000^{\circ}C$
3. Abort next segment if $\%/min < 0.5$
4. Isothermal 1000 min
5. Repeat 1 until $1000^{\circ}C$

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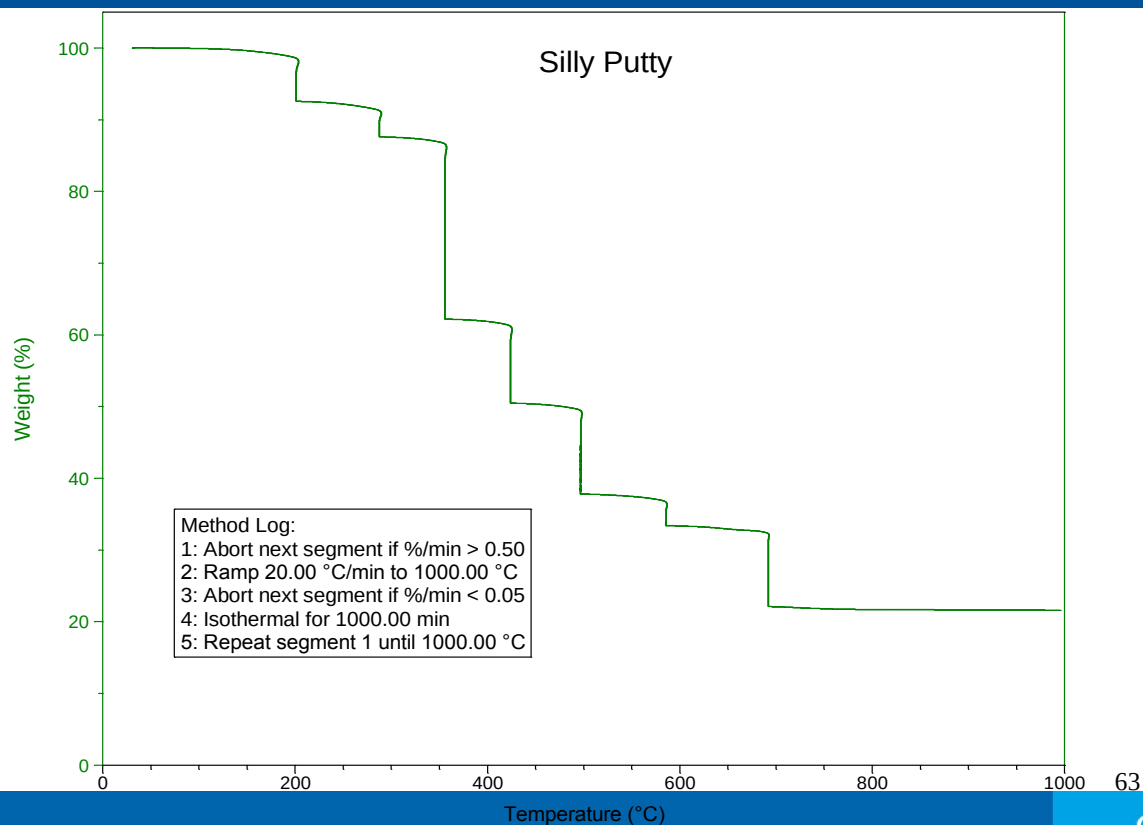


How to set Parameters for SWI

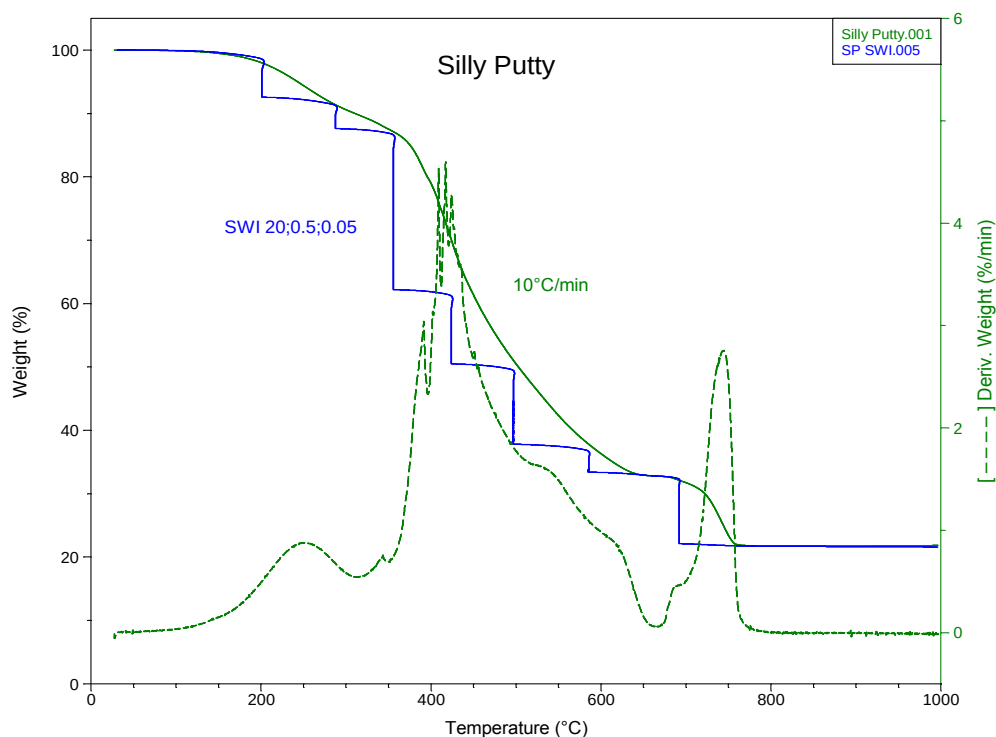
1. Run sample @ $10^{\circ}C/min$
2. Get peak value of time based deriv
3. Use $1/10^{th}$ of this value for entrance threshold
4. Use $1/10^{th}$ of entrance threshold for exit threshold



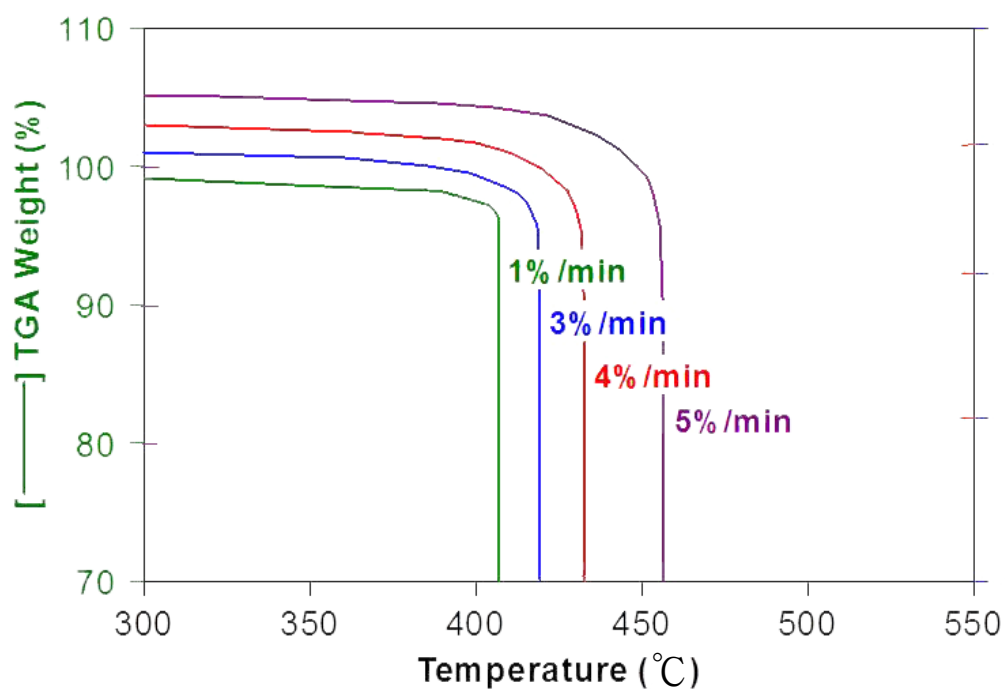
SWI of Silly Putty



Comparison of SWI & Std TGA



SWI - Effect of Entrance Threshold on Transition Onset

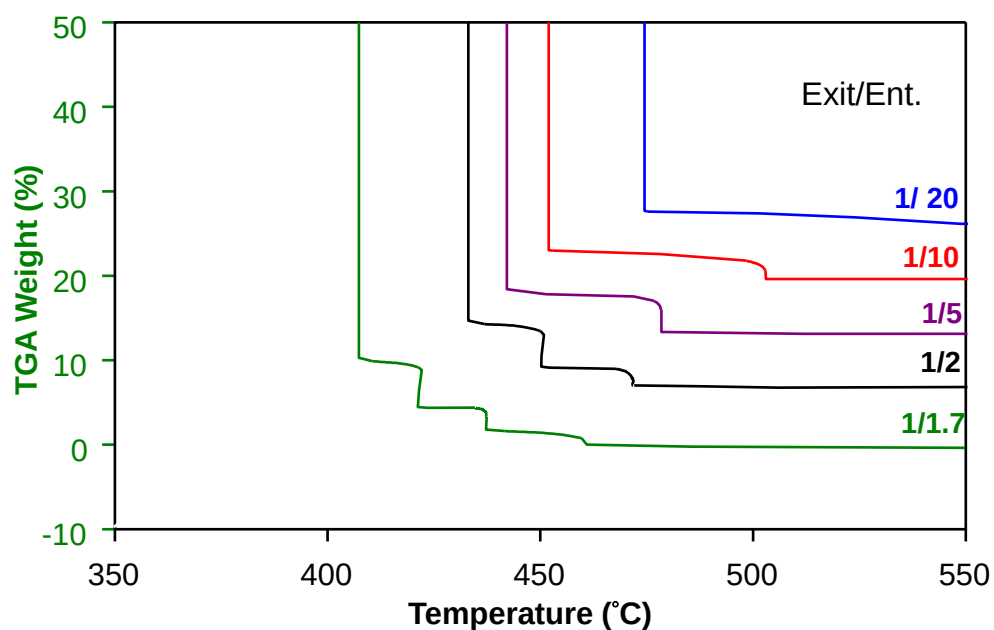


65



SWI - Effect of Threshold Ratio on Transition End

1%/min. Exit Threshold, x%/min. Entrance Threshold

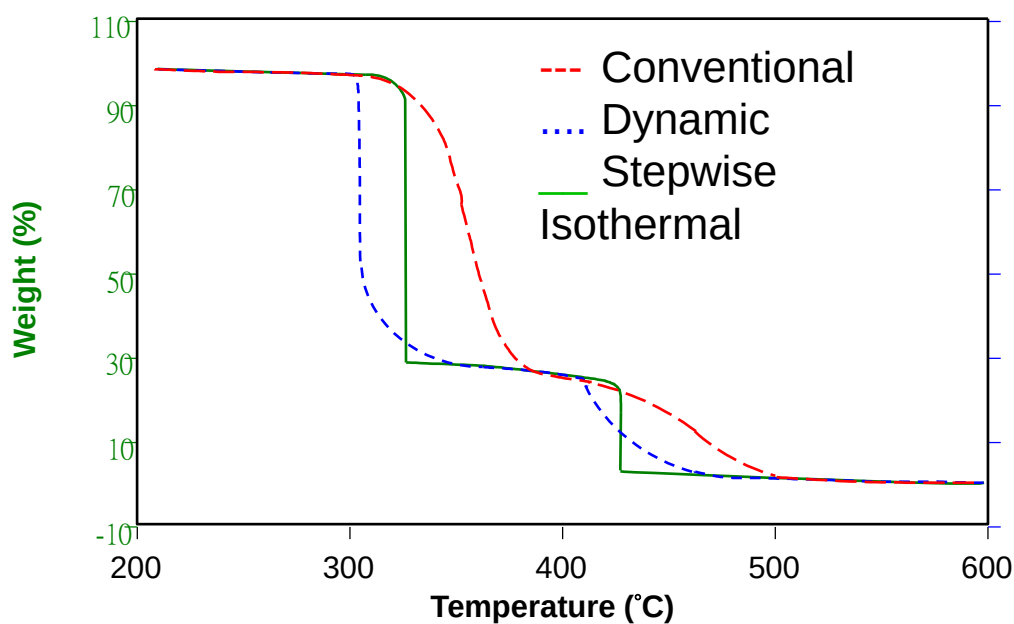


* Note: Curves have been shifted relative to the y-axis to facilitate comparison

66



Poly(vinyl acetate) Comparison of Modes



67



Hi-Res™ TGA - What is Dynamic Hi-Res TGA?

Heating rate is continuously varied in response to actual rate of weight loss

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Hi-Res™ TGA - Dynamic Rate Advantages

- Relatively simple to develop method
- Rapid survey over wide temperature range with excellent resolution
- Need to know little/nothing about sample
- High resolution with equal/better productivity, even on unknowns

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Hi-Res™ TGA - Example of Dynamic Rate Method

1. Sensitivity 2.0
2. Ramp **50°C/min**, **Res. 5.0** to **1000°C**

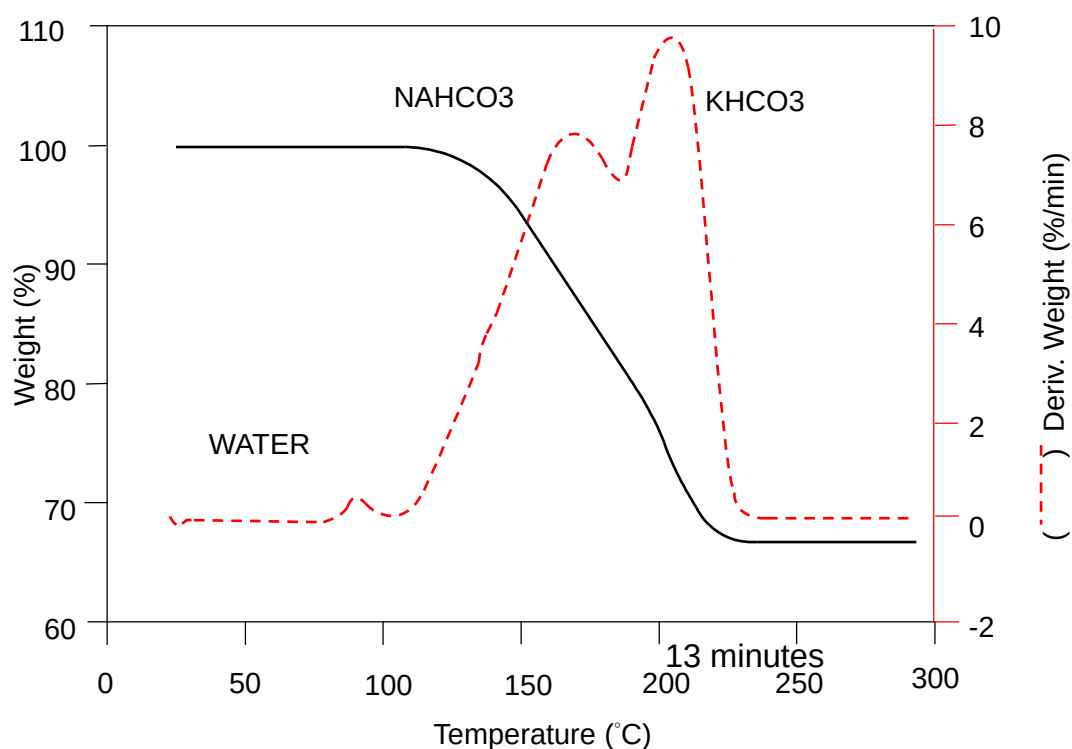
50°C/min : Ramp rate
Res. 5.0 : Resolution setting (0 to + 8.0)
2.0 : Sensitivity setting (1.0 - 8.0)
1000°C : Final temperature

Note: In Hi-Res TGA, the ramp rate is continuously changing – therefore, comparing temperatures to standard runs is not possible.

70



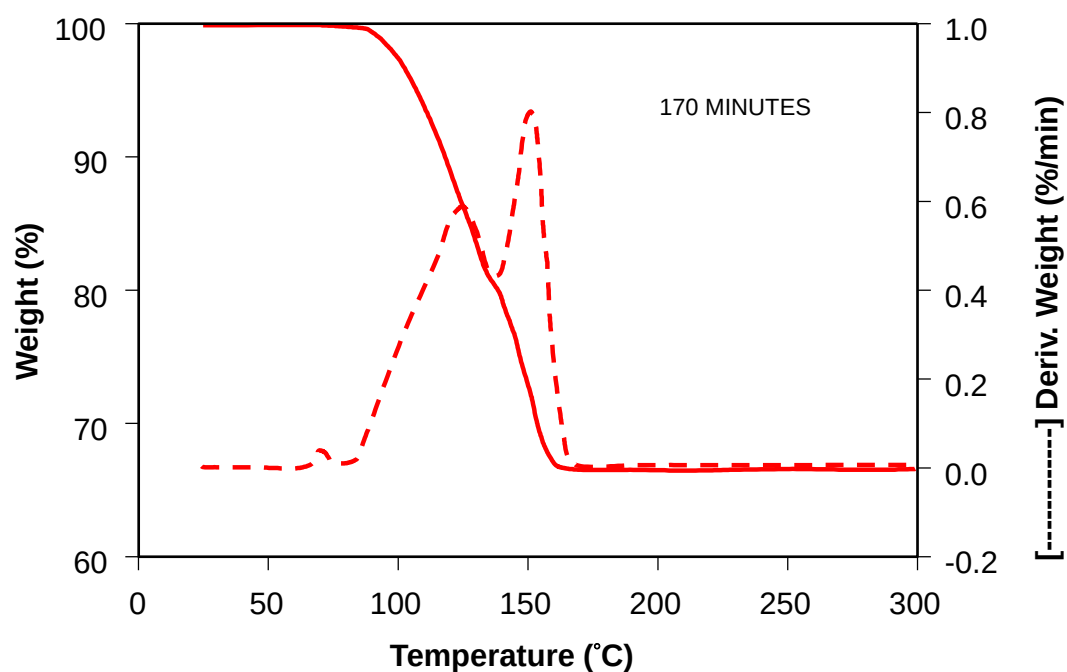
Bicarbonate Mixture - Conventional TGA at 20°C/min



71



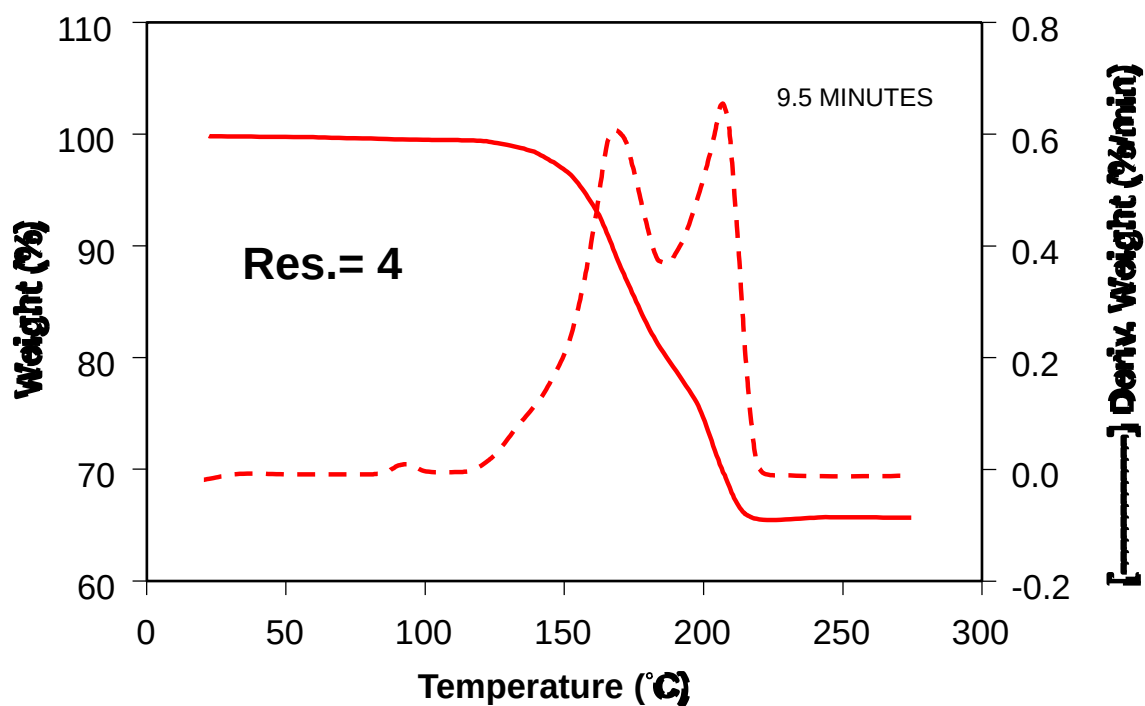
Bicarbonate Mixture - Conventional TGA at 1°C/min



72



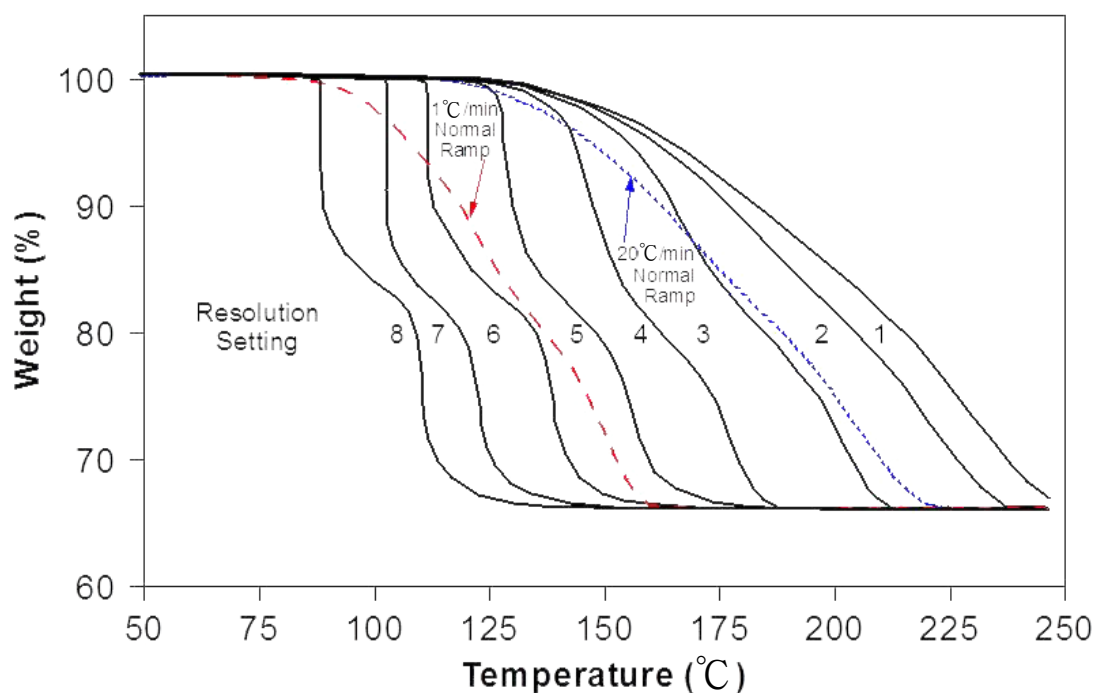
Bicarbonate Mixture Hi-Res™ TGA (Dynamic Rate)



73



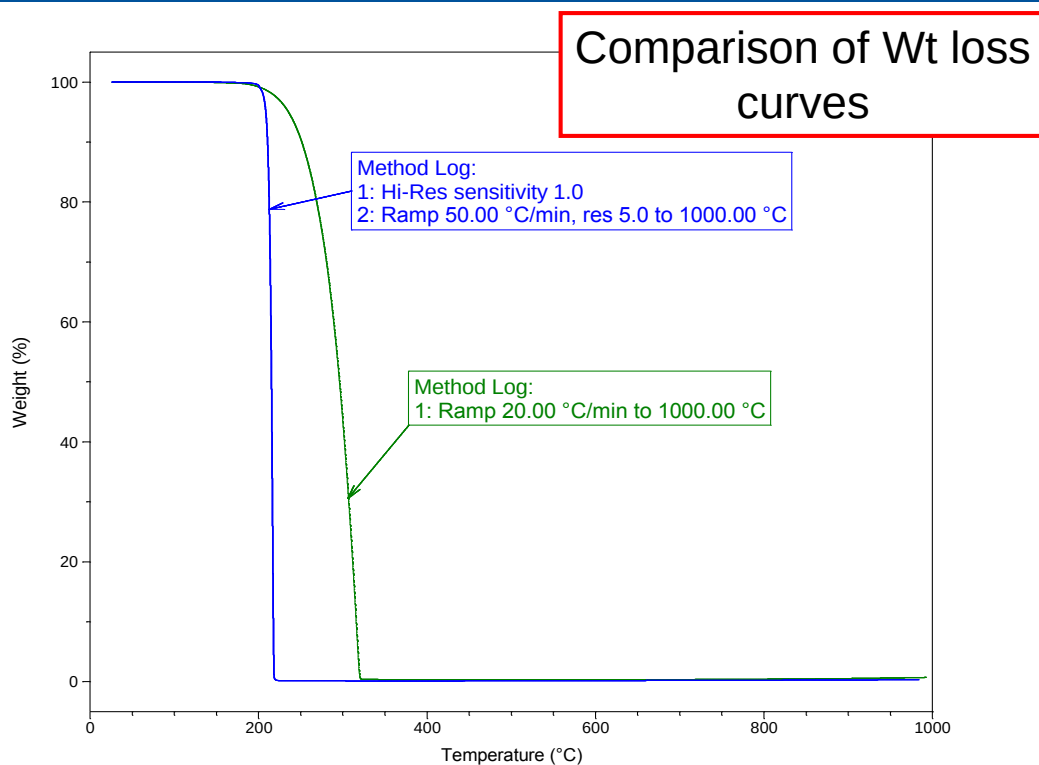
Varying Resolution Setting Mixture 50/50 $\text{KHCO}_3/\text{NaHCO}_3$: Air Purge



74



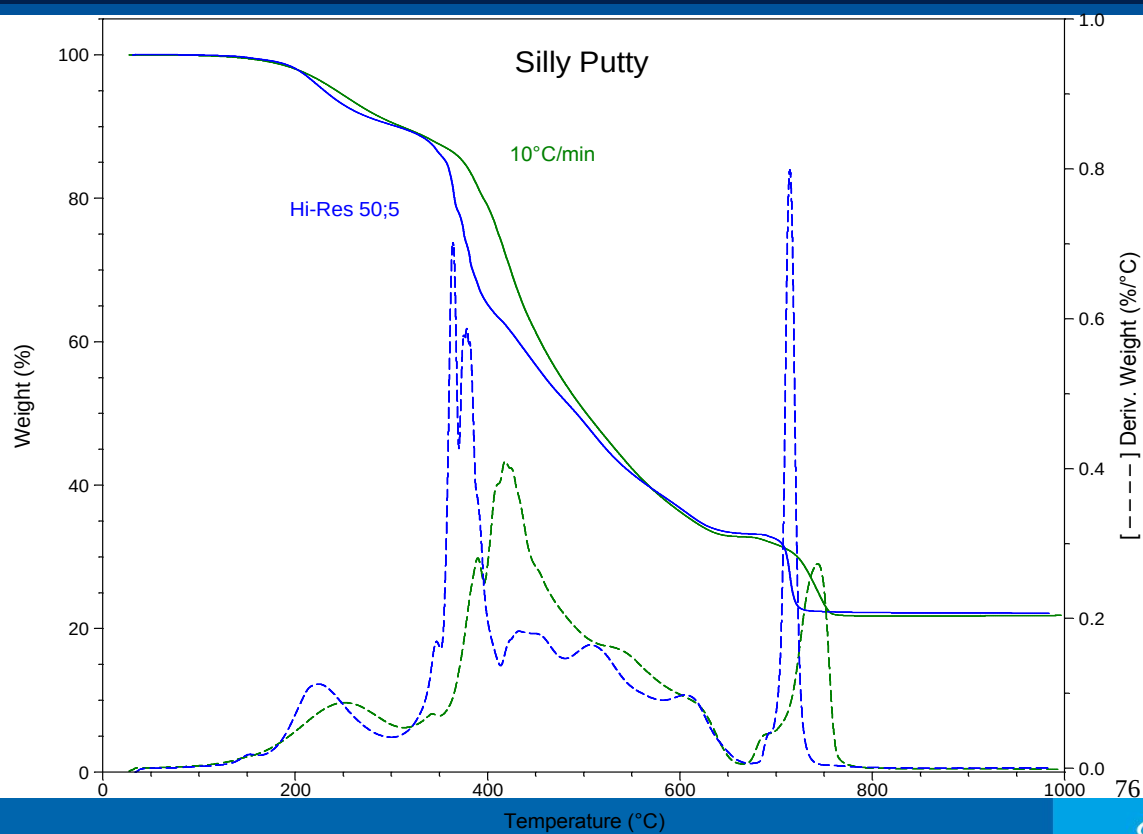
Acetaminophen Std TGA Vs. Hi-Res™



75



Comparison of Hi-Res™ & Std TGA



76



TGA

Decomposition Kinetics



Decomposition Kinetics Background

- Includes isothermal and constant heating rate methods.
- Constant heating rate method is the fastest and will be discussed here.
- Based on method of Flynn and Wall – Polymer Letters, **19**, 323, (1966). Requires collection of multiple curves at multiple heating rates.
- Ultimate benefit obtained in 'Life-Time' plots.



TA Specialty Library -- TGA Kinetics

- Requires at least 3 TGA runs at different heating rates or 1 Modulated TGA® run
- Calculates Activation energy & conversion curves
- Ultimate benefit is predictive curves “Lifetime Plots”

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Kinetic Analysis

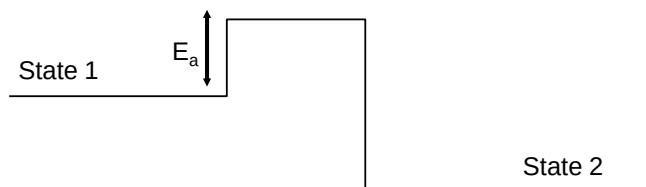
- The rate at which a kinetic process proceeds depends not only on the temperature the specimen is at, but also the time it has spent at that temperature.
- Typically kinetic analysis is concerned with obtaining parameters such as activation energy (E_a), reaction order (k), etc. and/or with generating predictive curves.

80



Kinetic Analysis, con't.

Activation energy (E_a) can be defined as the minimum amount of energy needed to initiate a chemical process.

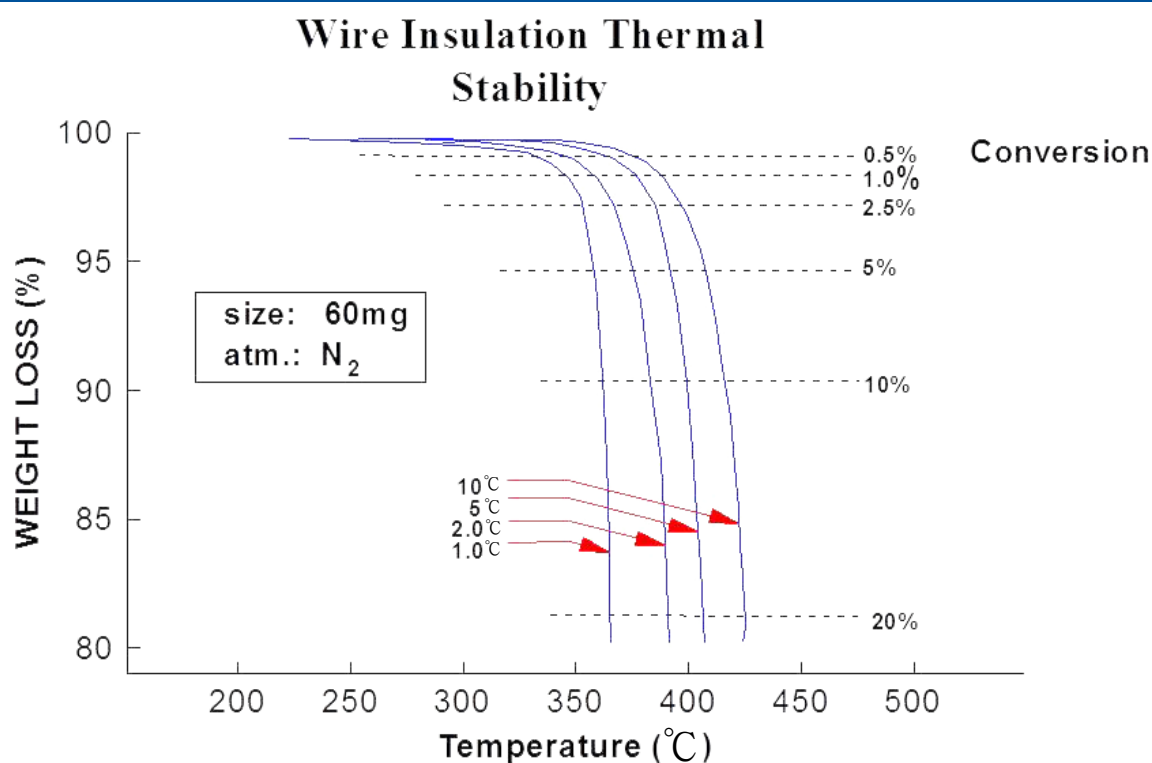


With Modulated TGA, E_a can be measured directly.

81



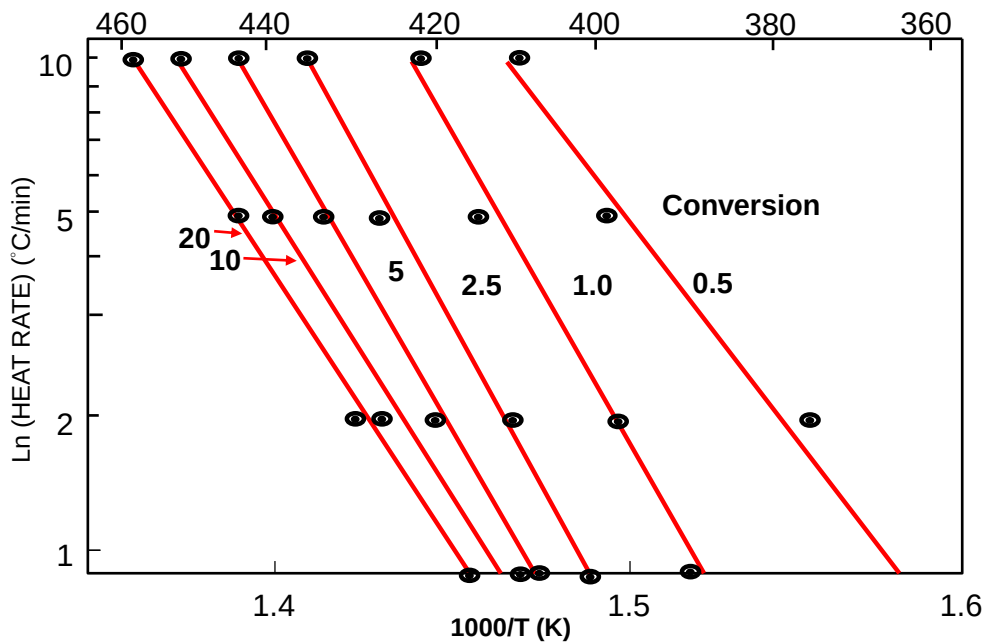
TGA Kinetics - Wire Insulation Thermal Stability



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TGA Kinetics - Heating Rate vs. Temperature

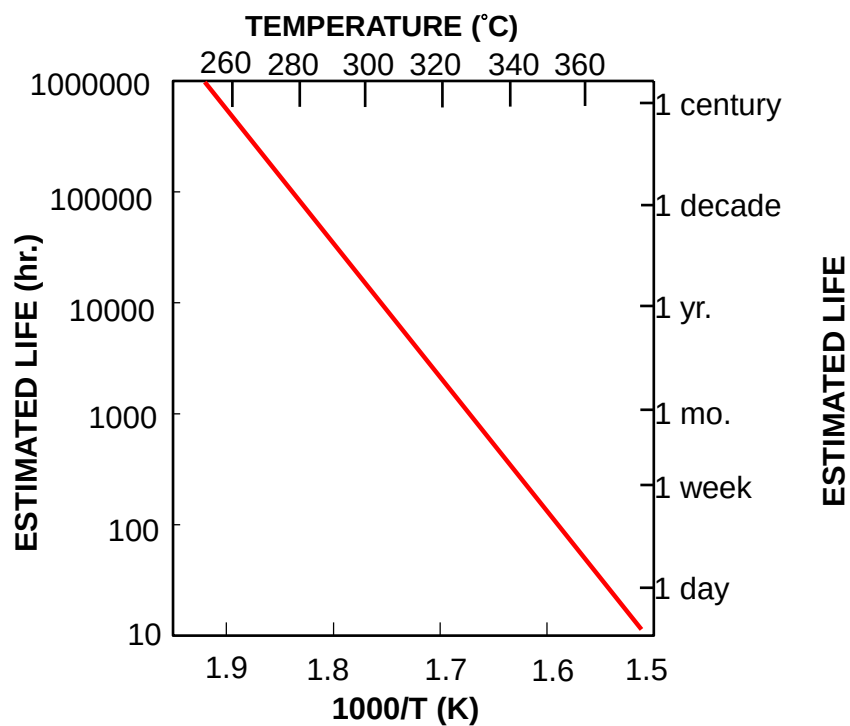


Activation Energy (E_a) $\propto$ Slope

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TGA Kinetics - Estimated Lifetime



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Thermogravimetry Under Extreme High Heating Rate Conditions



What Constitutes Extreme Conditions?

- High Heating Rates
 - Kinetic Studies
 - Sample Throughput



Rapid Heating Thermal Analysis

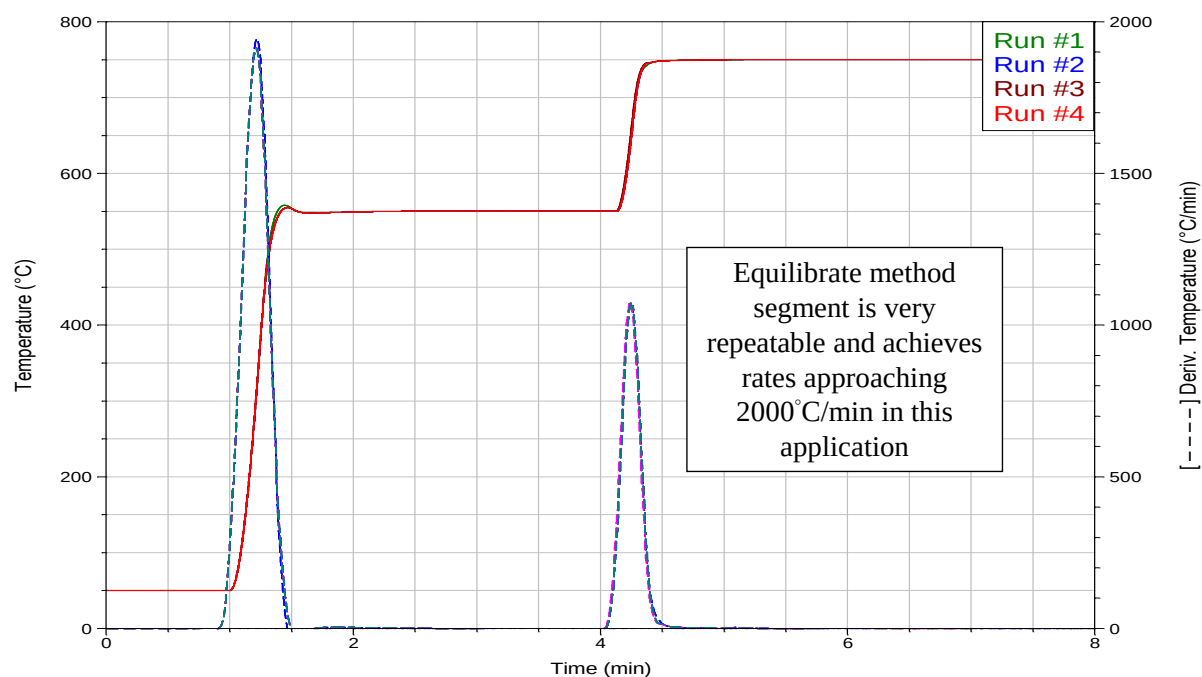
- Gasification, combustion, and volatilization are complex processes.
- Thermal treatments by different heating rates and time/temperature relationships can result in different chemical decomposition products.
- As an example, heating at a high rate can result in the thermal degradation of a component that would otherwise volatilize at a slow heating rate.
- Rapid heating rates, as on the TA Instruments Discovery TGA and pyrolysis GC/MS, provide powerful techniques to investigate the time/temperature relationships



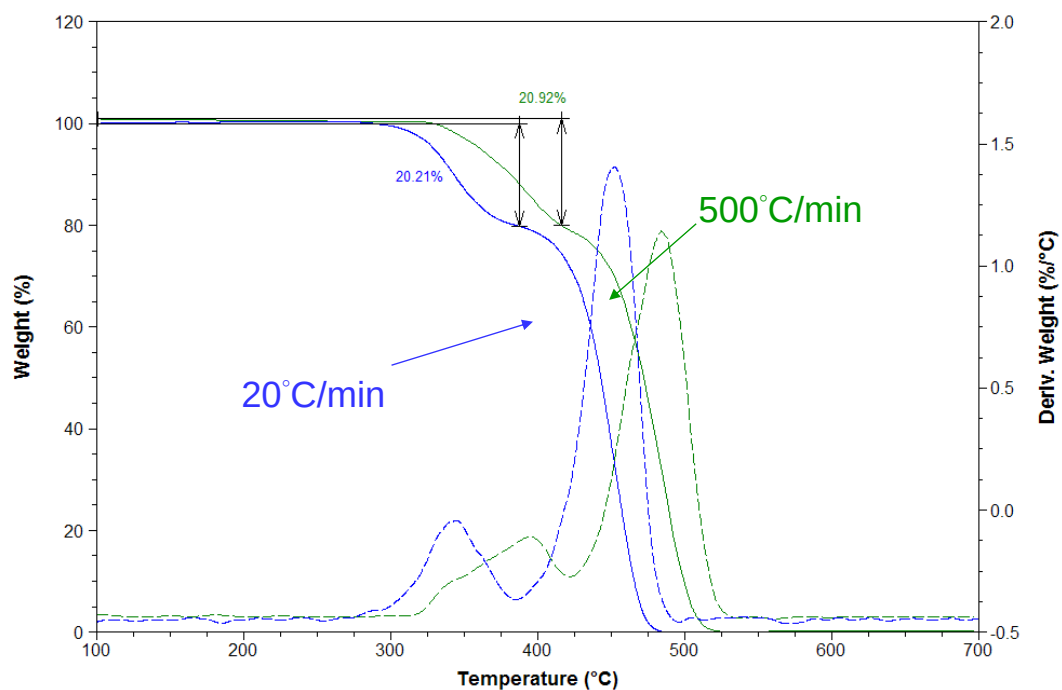
Discovery TGA & Q5000 IR Innovative IR-Heating Furnace



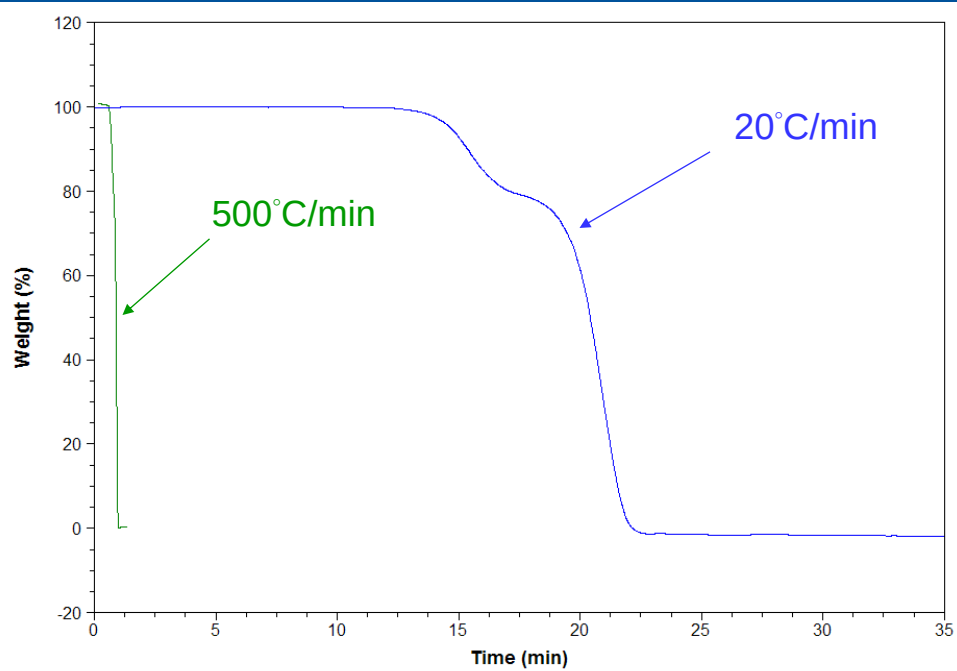
DTGA Ballistic Heating Performance



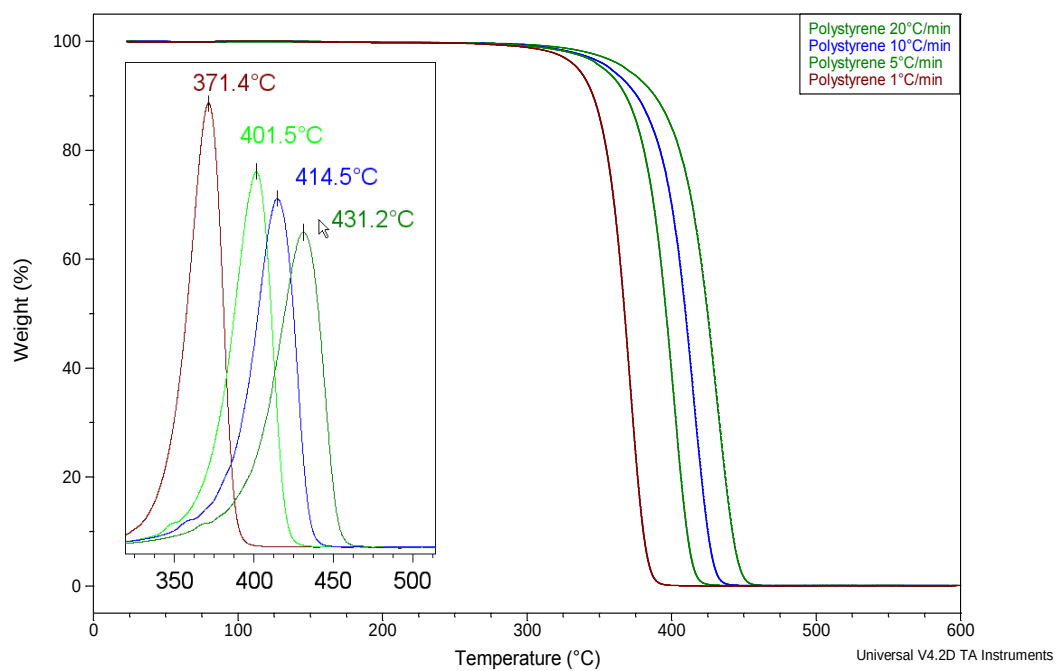
DTGA Heating Rate Comparison- Temperature



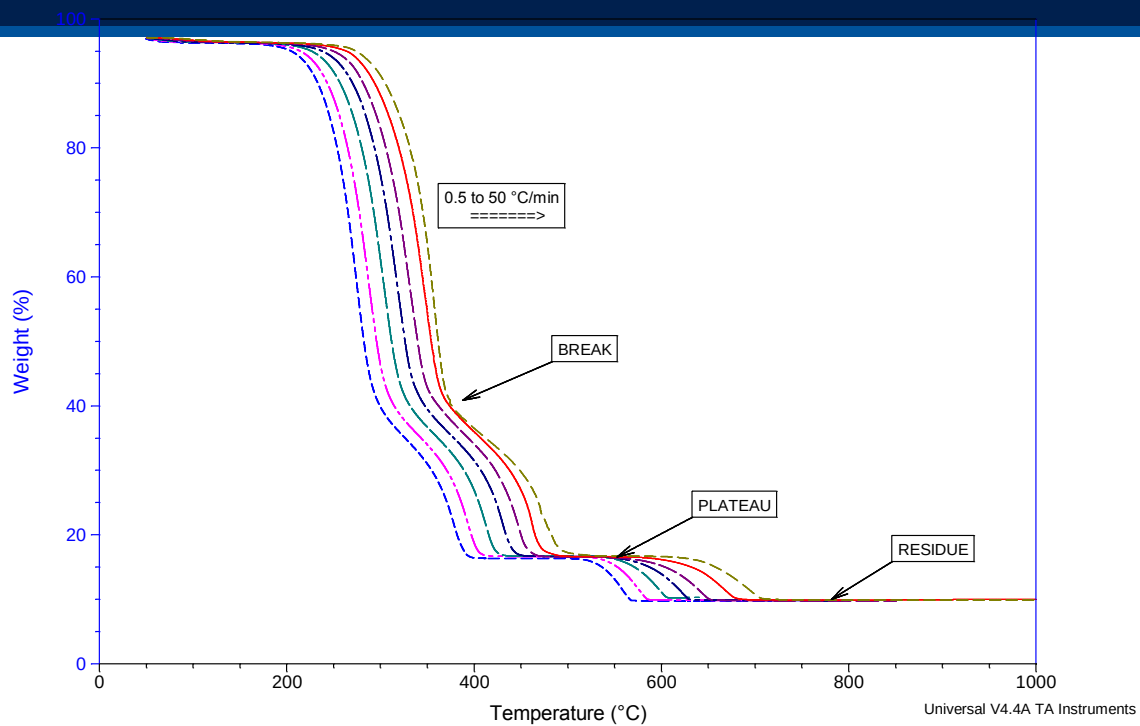
DTGA Heating Rate Comparison-Time



Effect of Heating Rate on Decomposition Temperature of Polystyrene



TGA: Copier Paper in Air

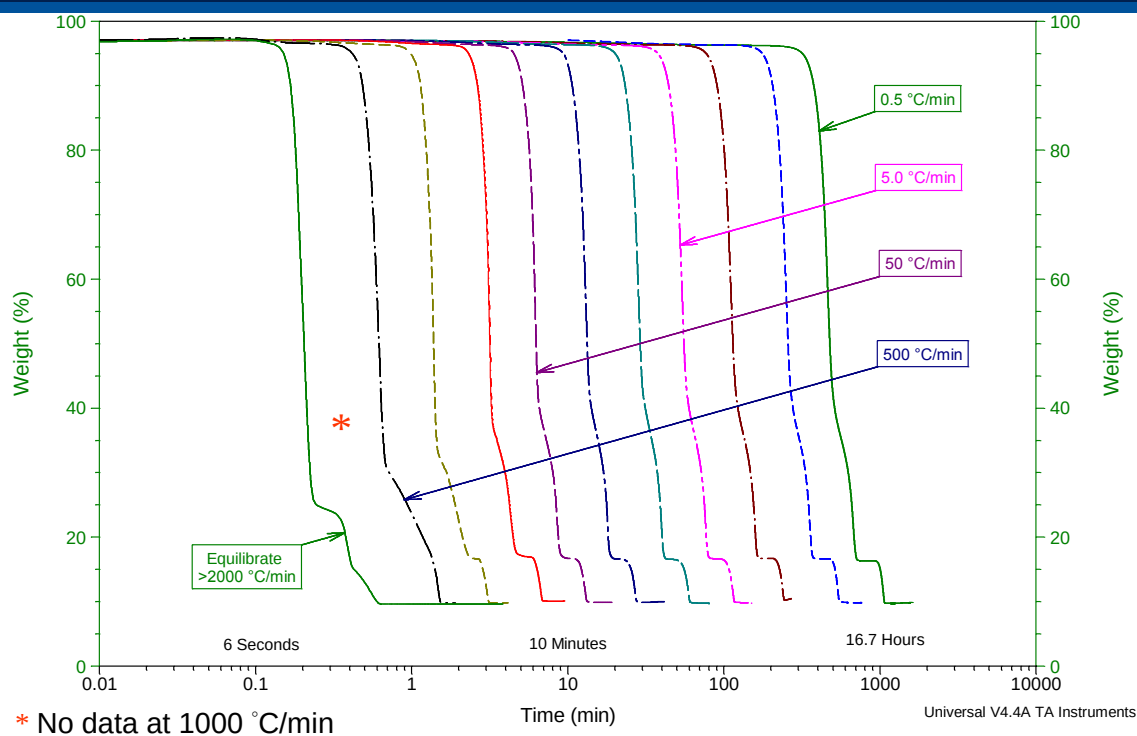


TGA: Quantitation of Copier Paper

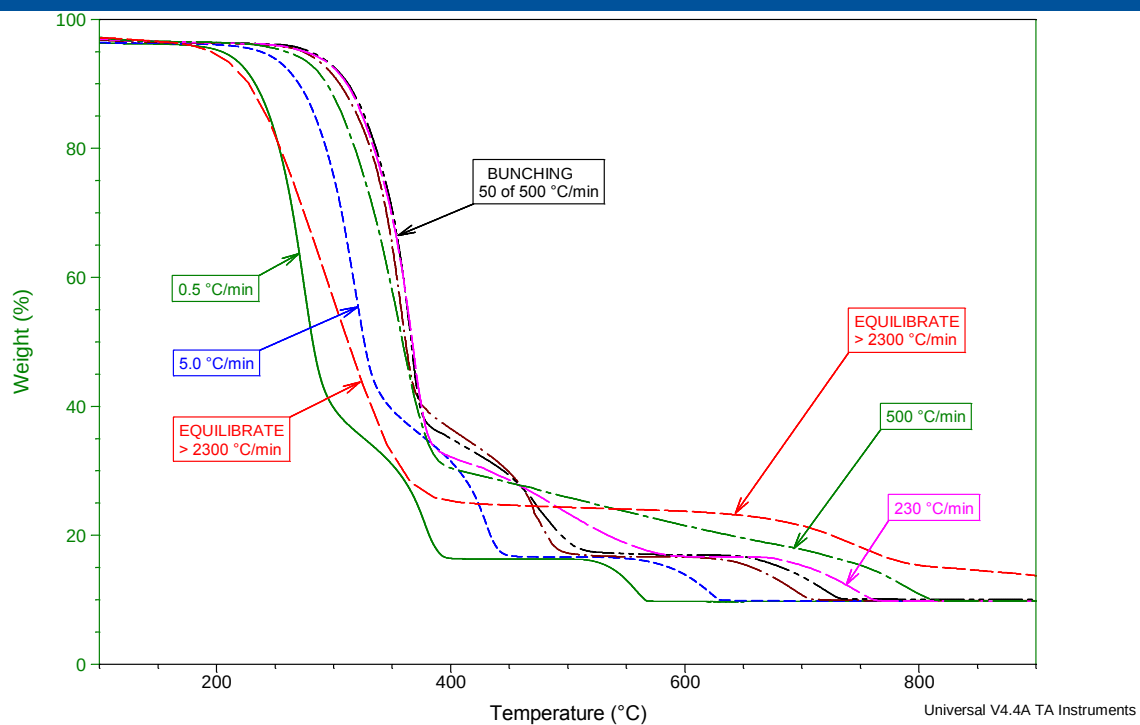
HEATING RATE °C/min	BREAK weight %	PLATEAU weight %	RESIDUE weight %
0.5	40.63	16.32	9.87
1	41.09	16.64	9.85
2.3	41.27	16.70	-
5	41.55	16.66	9.79
10	41.82	16.57	9.74
23	41.40	16.63	9.90
50	40.35	16.70	9.88
100	37.67	17.06	10.08
230	33.53	16.68	9.83
500	31.46	-	9.77
-	-	-	-
Equilibrate > 2300 °C/min	25.06	-	9.66
Average	41.29	16.66	9.84
STD DEV	0.41	0.19	0.11



TGA: Semi-Log Plot of Copier Paper



TGA: High Heating Rates of Copier Paper

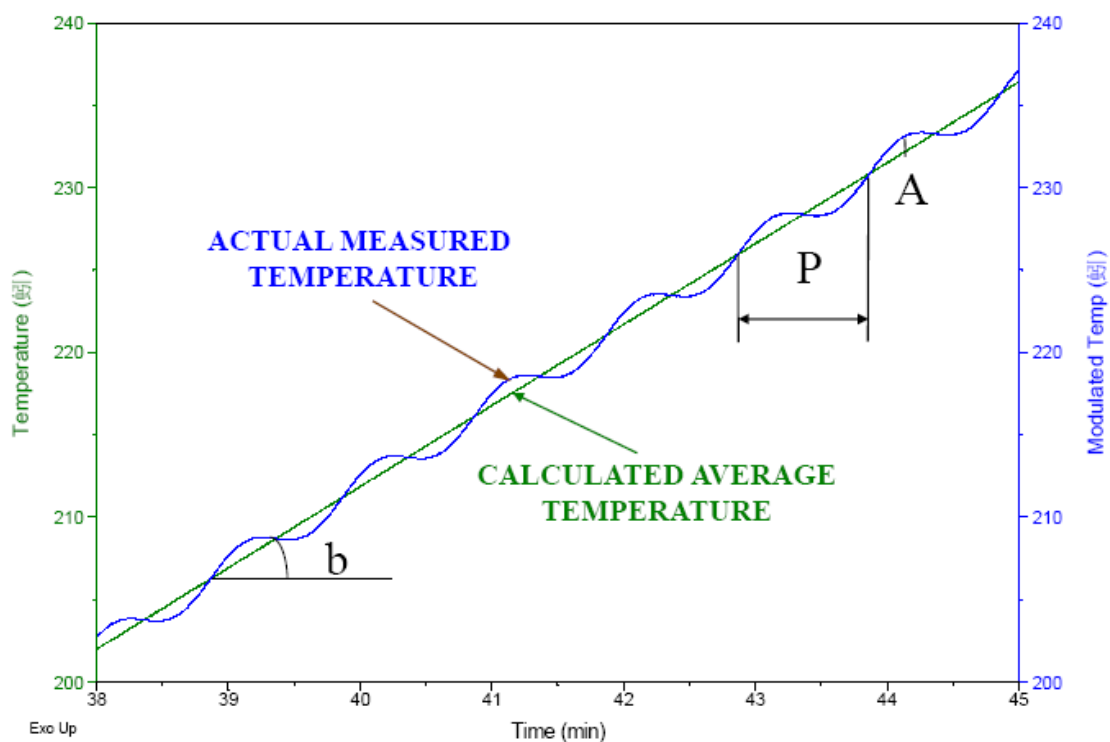


Modulated TGA Technology

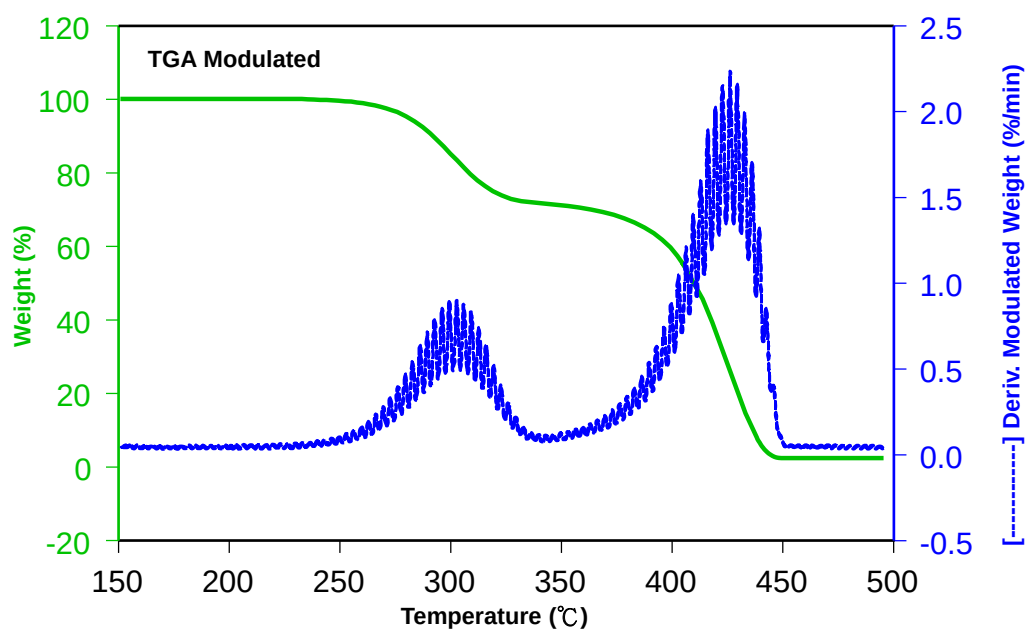
**A New Approach for
Obtaining Kinetic Parameters**



Temperature Change in MDSC and MTGA



MTGA OF 60% EVA WITH LINEAR RAMP



TA



GENERAL THEORY OF MTGA

$$\frac{d\alpha}{dt} = C \frac{dT}{dt} + f(t, T)$$

Rate of Weight Loss Zero Kinetic Component



ARRHENIUS AND GENERAL RATE EQUATIONS

$$\frac{d\alpha}{dt} = k(T) [f(\alpha)] = Z [f(\alpha)] e^{(-E/RT)}$$

Where:

α	= reaction fraction
$d\alpha / dt$	= rate of reaction
$k(T)$	= rate constant at temperature T
T	= absolute temperature
$f(\alpha)$	= kinetic expression
Z	= pre-exponential factor
e	= natural logarithm base
E	= activation energy
R	= gas constant



KINETIC EXPRESSION RATIO

$$\frac{d\alpha_1}{dt} = Z [f(\alpha)_1] e^{(-E/RT_1)}$$

$$\frac{d\alpha_2}{dt} = Z [f(\alpha)_2] e^{(-E/RT_2)}$$

$$\frac{d\alpha_1}{d\alpha_2} = \frac{[f(\alpha)_1] e^{(-E/RT_1)}}{[f(\alpha)_2] e^{(-E/RT_2)}}$$

Where: $d\alpha_1$ = rate of weight loss at temperature T_1
 $d\alpha_2$ = rate of weight loss at temperature T_2
 $f(\alpha_1)$ = kinetic expression at the value of $d\alpha_1$
 $f(\alpha_2)$ = kinetic expression at the value of $d\alpha_2$



FACTOR JUMP EQUATION AT CONSTANT CONVERSION

$$E = \frac{R T_1 T_2 \ln(d\alpha_1 / d\alpha_2)}{T_1 - T_2}$$

Where: $d\alpha_1$ = rate of weight loss at temperature T_1
 $d\alpha_2$ = rate of weight loss at temperature T_2
 R = gas constant



FOURIER TRANSFORMATION YIELDS

$$T_1 = T + A,$$

$$T_2 = T - A,$$

$$L = \ln(d\alpha_1 / d\alpha_2), \text{ and}$$

$$f(\alpha_1) = f(\alpha_2) \Rightarrow \ln[f(\alpha_2) / f(\alpha_1)] = 0$$

Where: T = average temperature
 A = temperature (half) amplitude
 L = (full) amplitude of
 $\ln$ (rate of weight change)



MODULATED TGA EQUATION

$$E = \frac{R (T^2 - A^2) L}{2A}$$

Where: E = activation energy
 R = gas constant
 T = average temperature
 A = temperature (half) amplitude
 L = $\ln (d\alpha_1 / d\alpha_2)$ = amplitude of
 $\ln$ (rate of weight change)



FIRST ORDER PRE-EXPONENTIAL FACTOR

$$\ln Z = \ln [d\alpha/(1-\alpha)] + E/(R T)$$

Where: Z = pre-exponential factor
 E = activation energy
 R = gas constant
 T = temperature
 $d\alpha$ = rate of weight
 loss
 $(1-\alpha)$ = weight fraction

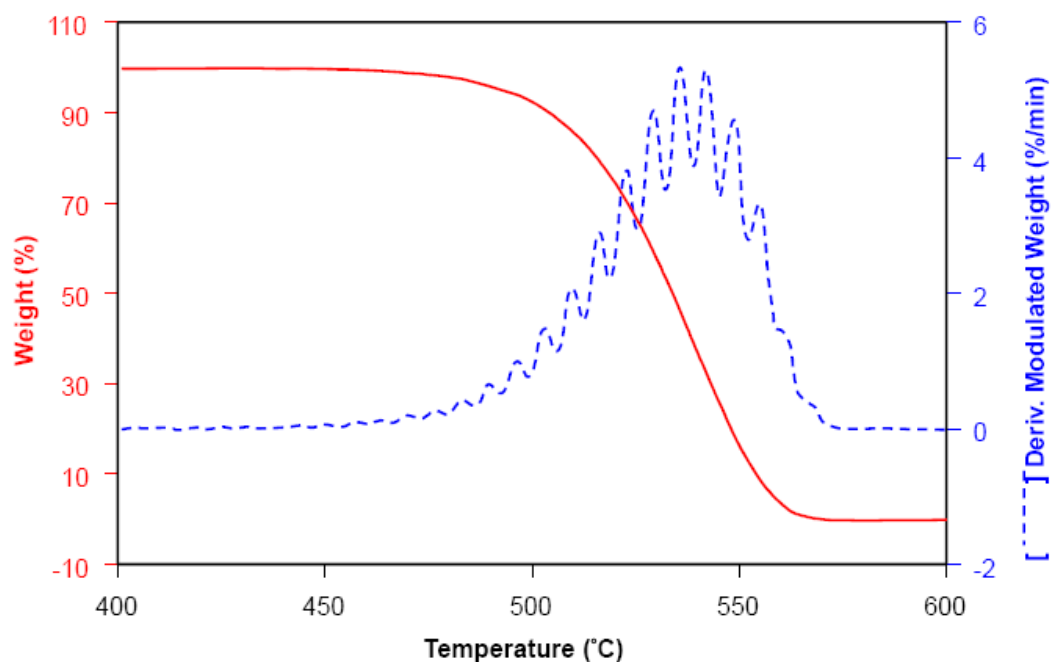


FAST FOURIER TRANSFORMATION

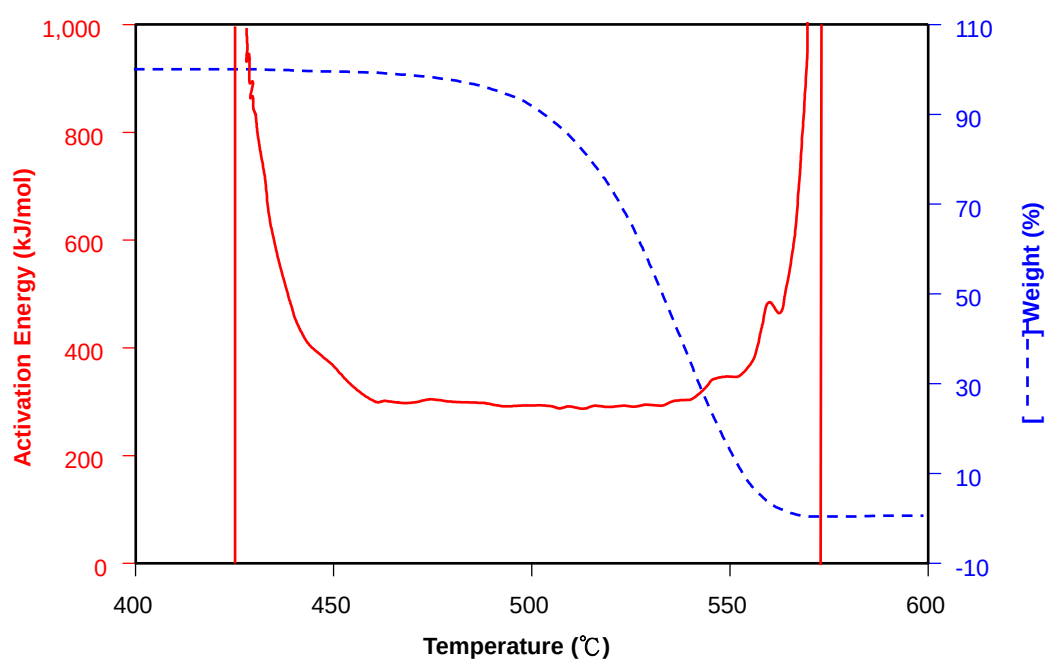
- Fast Fourier Transformation yields continuous kinetic parameters



LINEAR HEATING PROFILE FOR POLYTETRAFLUOROETHYLENE



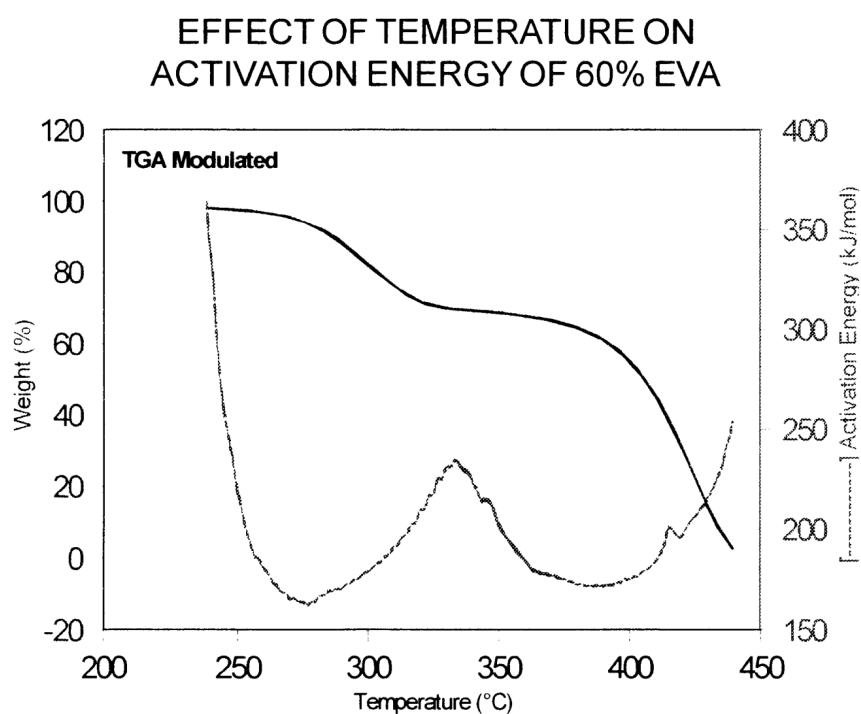
TGA: MTGA - Activation Energy for Polytetrafluoroethylene



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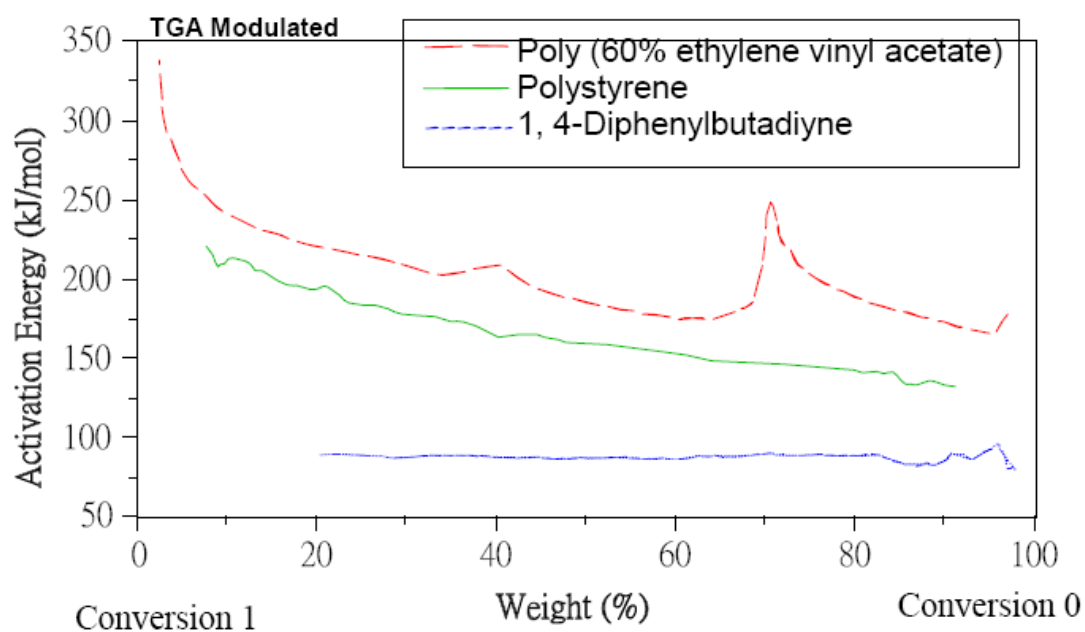


EVA(60% V_{ac})的分解活化能溫度歷程圖



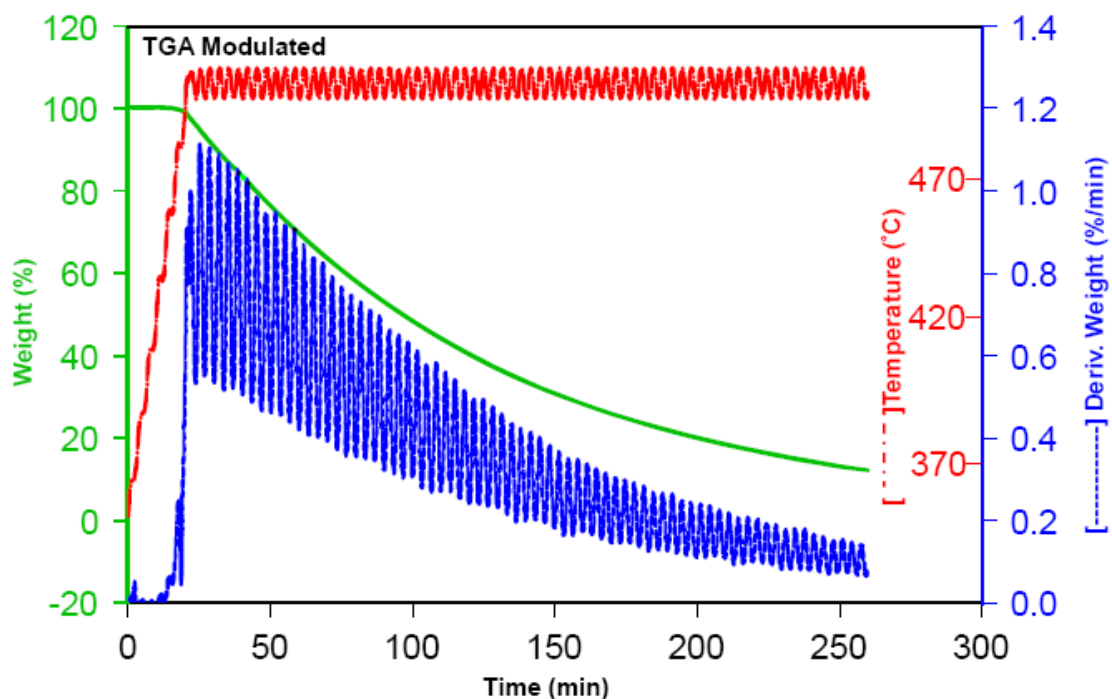
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EFFECT OF CONVERSION ON ACTIVATION ENERGY

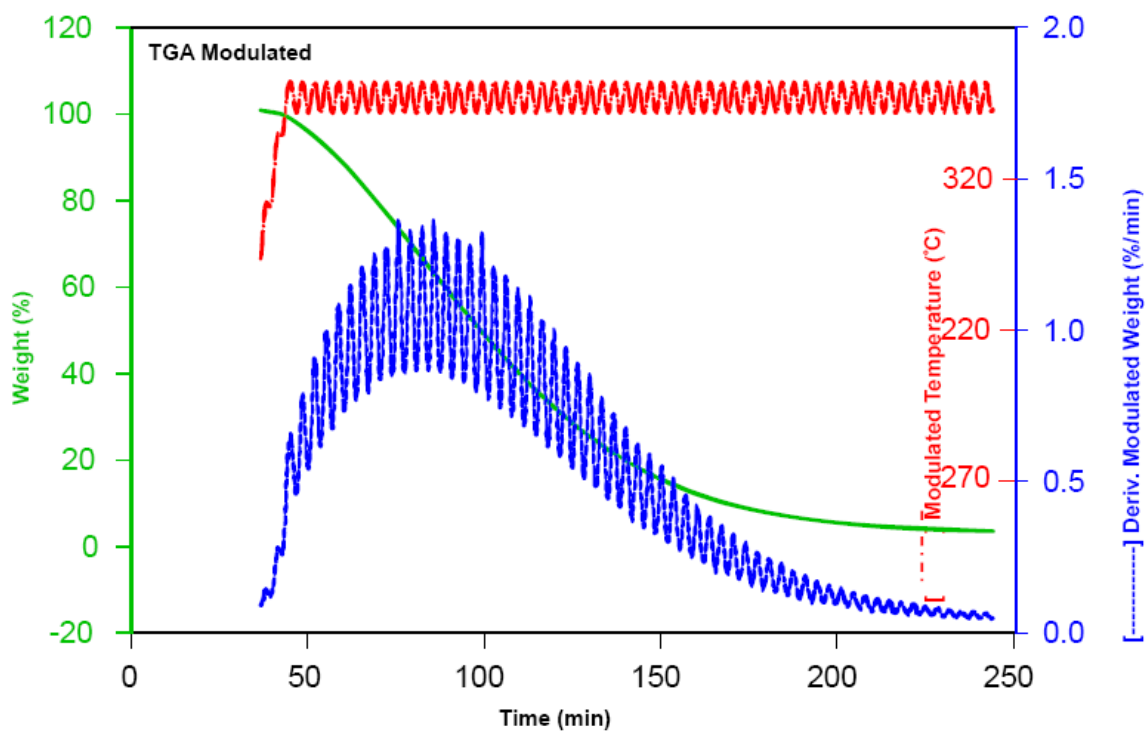


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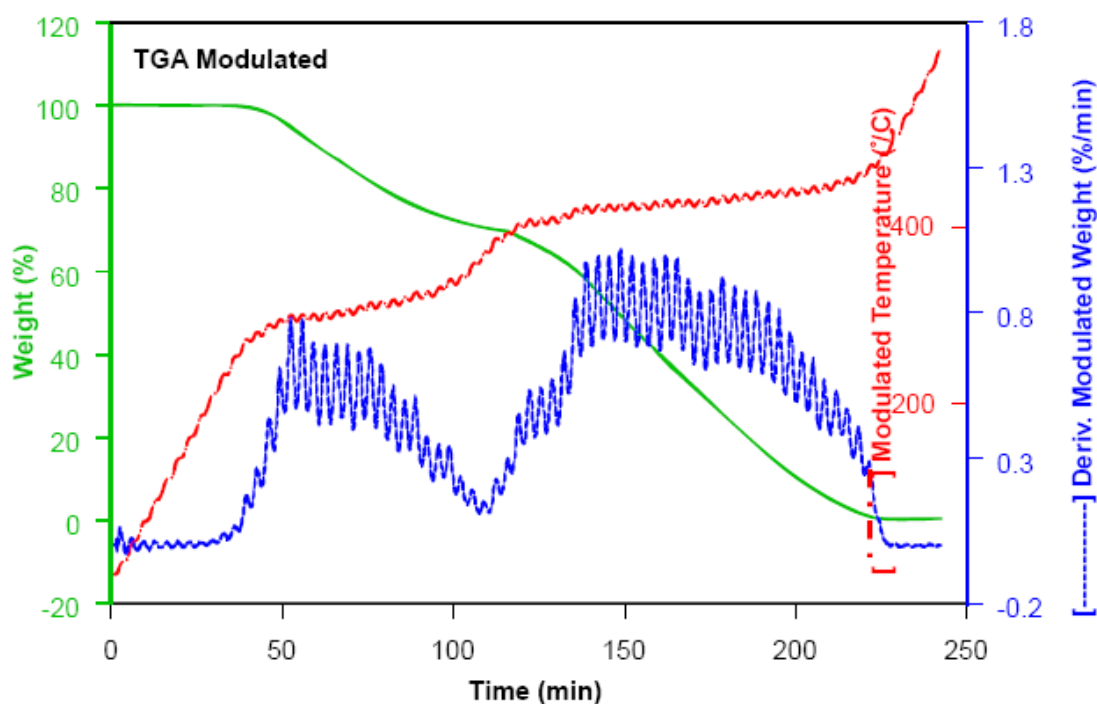
QUASI-ISOTHERMAL MTGA OF PTFE



QUASI-ISOTHERMAL MTGA OF PS



MTGA OF 60% EVA WITH DYNAMIC HEATING RATE



MTGA REPEATABILITY POLY (60% ETHYLENE VINYL ACETATE)

Side Group Loss		Backbone Decomposition	
Activation Energy (kJ/mol)	Log Pre-Exponential Factor (1/min)	Activation Energy (kJ/mol)	Log Pre-Exponential Factor (1/min)
166.2	13.141	176.0	11.601
165.6	13.191	173.6	11.491
163.4	13.041	174.6	11.601
171.7	13.381	173.8	11.421
168.3	13.351	175.1	11.541
163.9	12.951	170.2	11.141
mean	166.5	173.9	11.47
std. dev.	3.1	2.0	0.17
RSD	1.9%	1.2%	1.2%



MTGA KINETICS COMPARISON

	Activation Energy (kJ/mol)		Logarithm of Pre-Exponential Factor (1/min)	
	ASTM ¹ E1641	MTGA ²	ASTM ¹ E1641	MTGA ²
Poly(ethylene)	190	190	12.9	12.8
Poly(tetrafluoroethylene)	316	341	19.1	21.1
Poly(styrene)	173	182	13.0	14.0
Poly(ethylene vinyl acetate)	183	167	14.5	13.2
	289	174	20.4	11.5
Dicumyl Peroxide	104	101	12.0	11.8
1,3 Diphenylbutadiyne	81	99	8.1	10.4
Calcium Oxalate · H ₂ O	117	121	13.8	13.5
Calcium Oxalate	207	194	14.0	12.2
Calcium Carbonate	210	188	10.5	8.7

¹Repeatability RSD = 2.8%

²Repeatability RSD = 1.7%



MTGA EXPERIMENTAL CONDITIONS

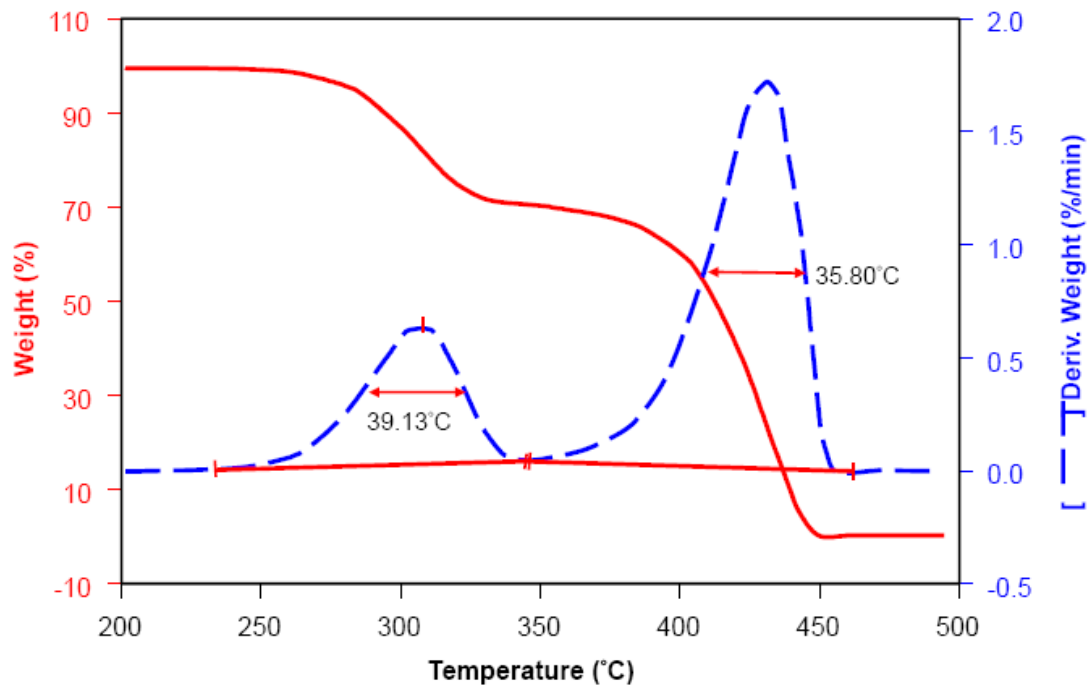
- Period : > 200 seconds
(temperature range dependent)
- Amplitude : 4 - 5 ° C
- Cycles Across Transition : > 5

Temperature Program

- Isothermal or
- Heating Rate : < 2 ° C/min



DETERMINATION OF FULL WIDTH TEMPERATURE AT HALF HEIGHT



BENEFITS OF MTGA METHOD

- SINGLE EXPERIMENT
- INCREASED PRODUCTIVITY
- MODELFREE
- EASY OF USE
- OBTAIN Z WITH ASSUMPTION OF 1st ORDER MODEL
- COMPLETE KINETIC INFORMATION
- E & Z AS A FUNCTION OF CONVERSION
- FOLLOW PROCESS CHANGES



Evolved Gas Analysis



Why Use Evolved Gas Analysis?

- TGA measures weight changes (quantitative)
- Difficult to separate, identify, and quantify individual degradation products (off-gases)
- Direct coupling to identification techniques (Mass Spec, FTIR) reduces this problem



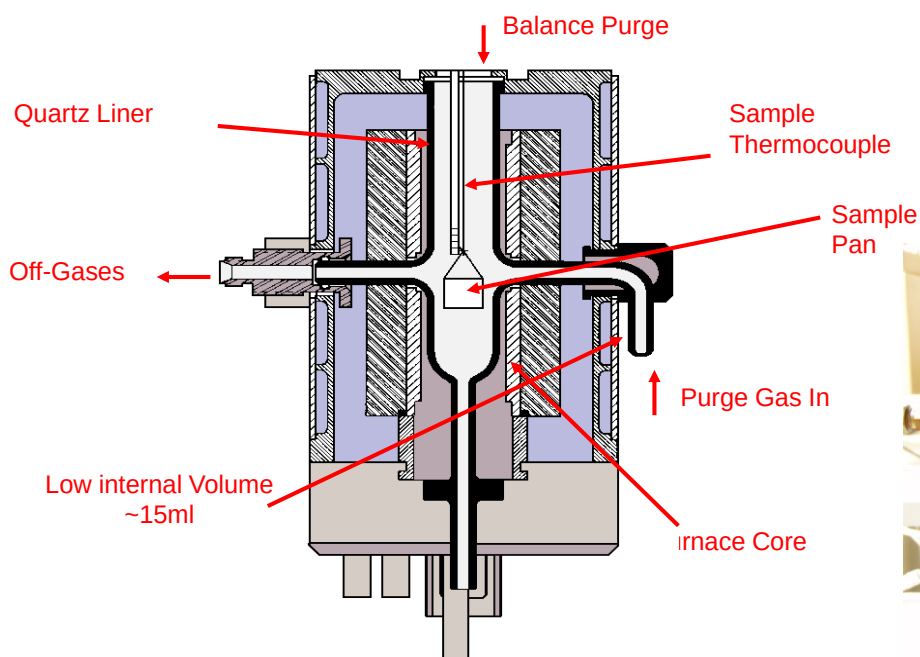
TGA-EGA: Typical Applications

- Polymers (composition, hazard evaluation, identification)
- Natural Products (contamination in soil, raw material selection {coal, clays})
- Catalysts (product/by-product analysis, conversion efficiency)
- Inorganics (reaction elucidation, stoichiometry, pyrotechnics)
- Pharmaceuticals (stability, residual solvent, formulation)

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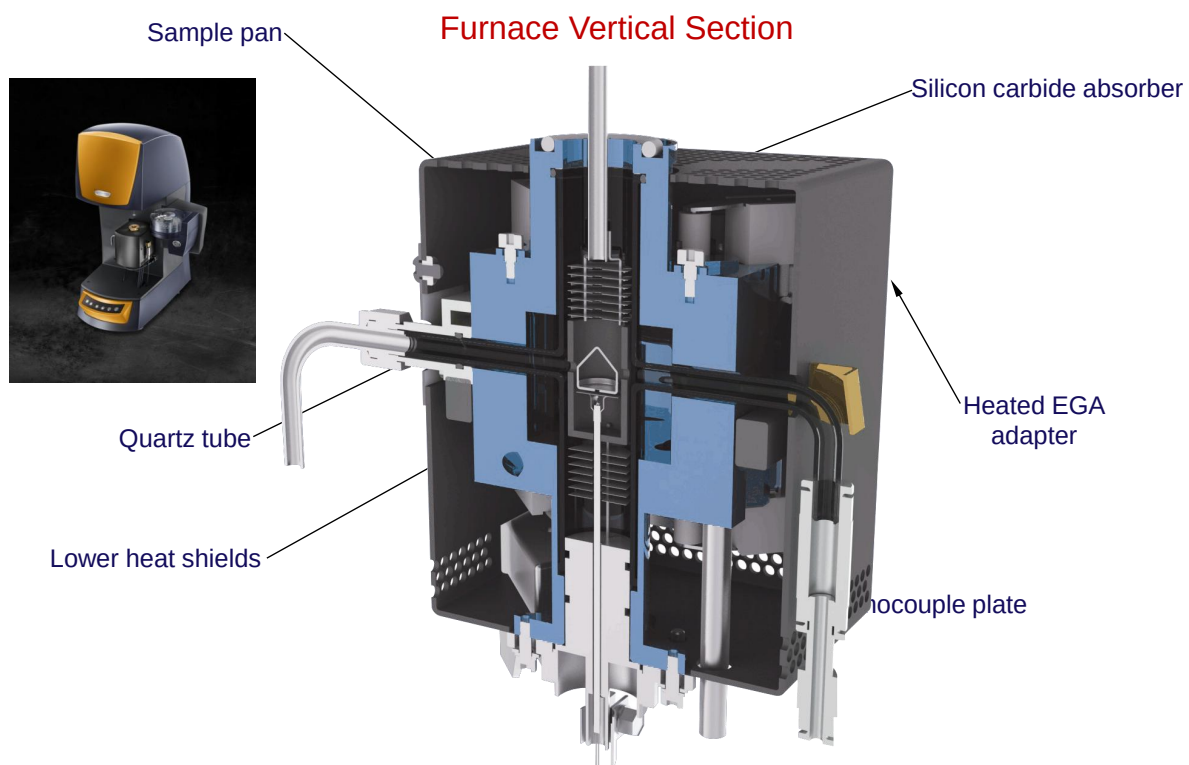
Q50/Q500 EGA Furnace Schematic



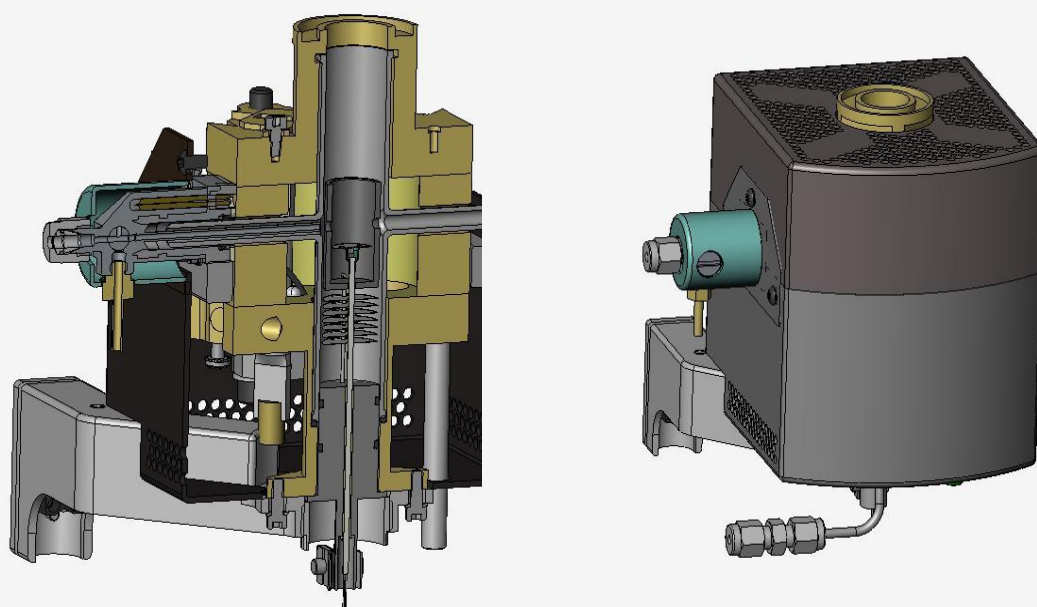
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Discovery TGA & TGA Q5000 IR



Discovery TGA Heated FTIR Adapter



Mass-Spectrometry Benefits

- Additional information for the interpretation of the reactions in the TGA results
- Sensitive method for the analysis of gaseous reaction products
- Exact control of the furnace atmosphere before starting and during the experiment
- Location of air leaks around the furnace

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TGA-Mass Spectroscopy

Advantages:

- Higher sensitivity and wider dynamic range than FTIR (1ppm vs. 10ppm).
- Measures non-IR absorbing gases.
- More rapid response.

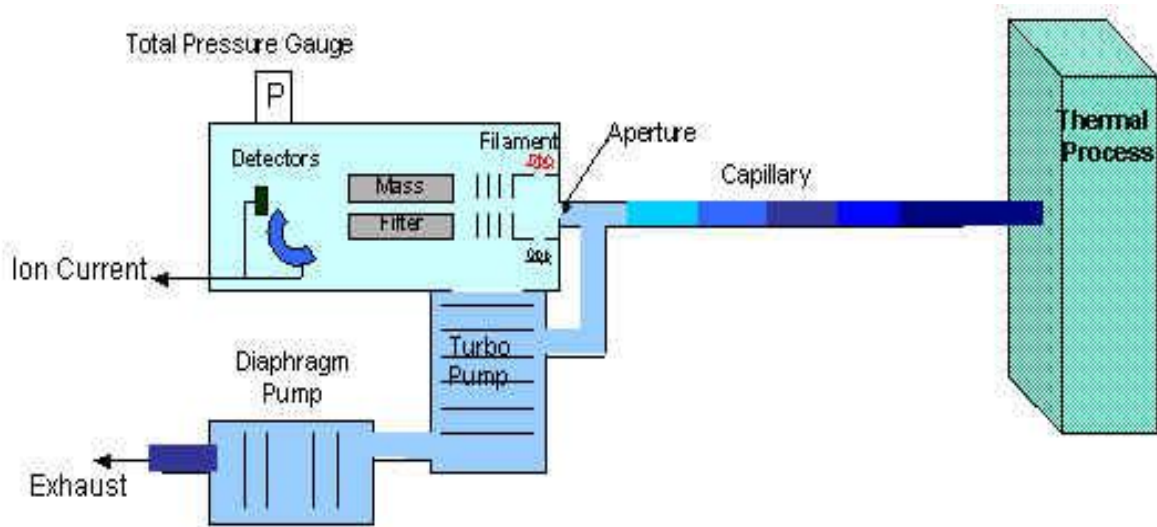
Disadvantage:

- Cannot distinguish between isomers. (e.g. N₂ and CO)

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TGA-MS: System Schematic



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TGA - FTIR

FTIR (Fourier transformation infrared spectroscopy):

Advantages:

- On-line measurement
- Hydrocarbons are easy to identify

Disadvantages:

- No detection of inert gases (no dipole moment)
- Detection of inorganic gases limited

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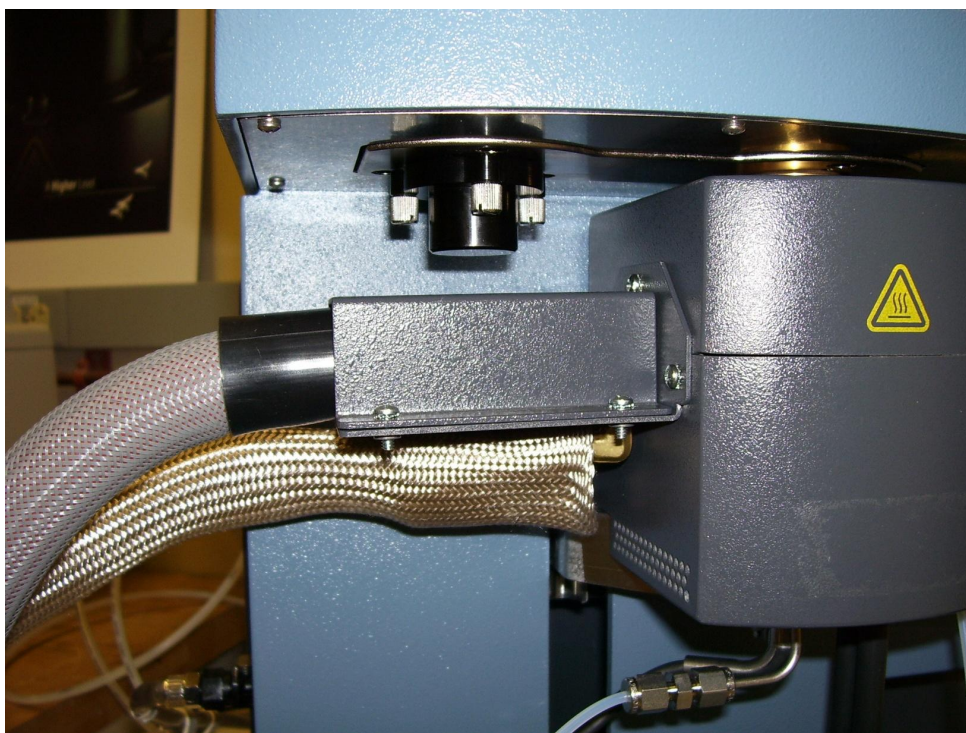
TGA-FTIR: System Schematic



TA Instruments Universal Analysis software supports the importation of MS (trend analysis) and FTIR data (Gram-Schmidt and Chemigram reconstructions), allowing TGA and EGA data to be displayed on a common axis of temperature and/or time.



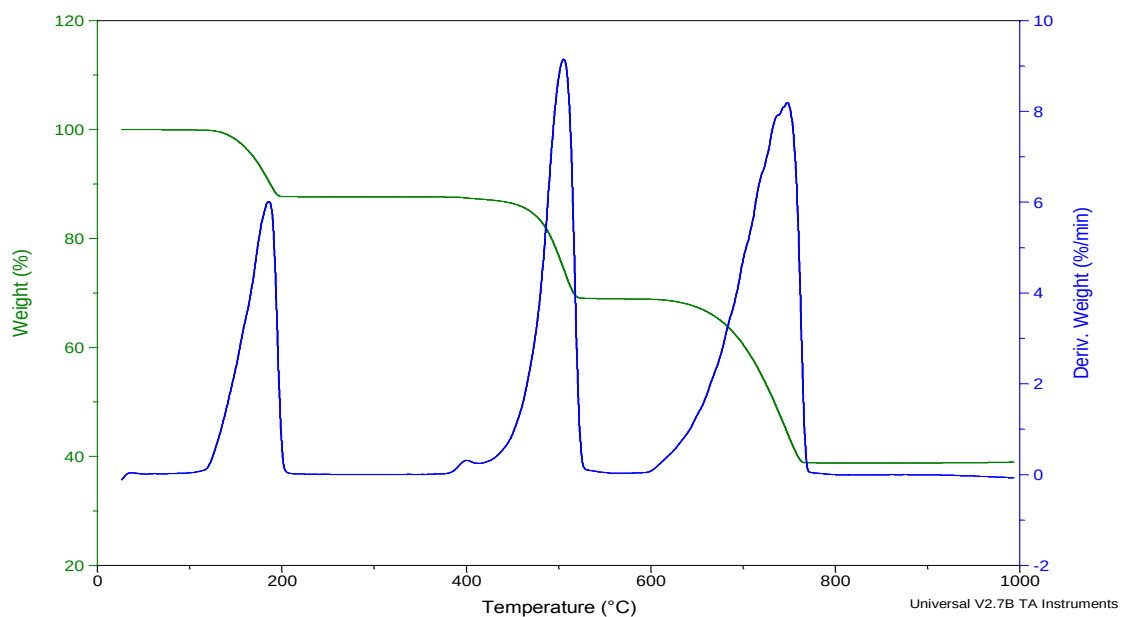
MS & FTIR Attached to Interface



TGA of Calcium Oxalate

Sample: Calcium Oxalate Monohydrate
Size: 17.6070 mg
Method: RT→1000°C @ 20°C/min

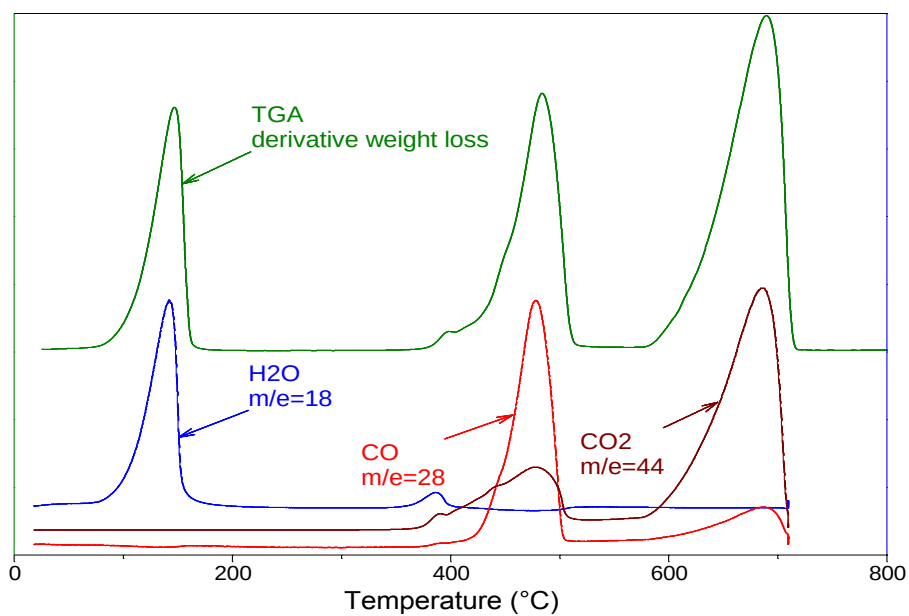
TGA



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TGA-MS Calcium Oxalate



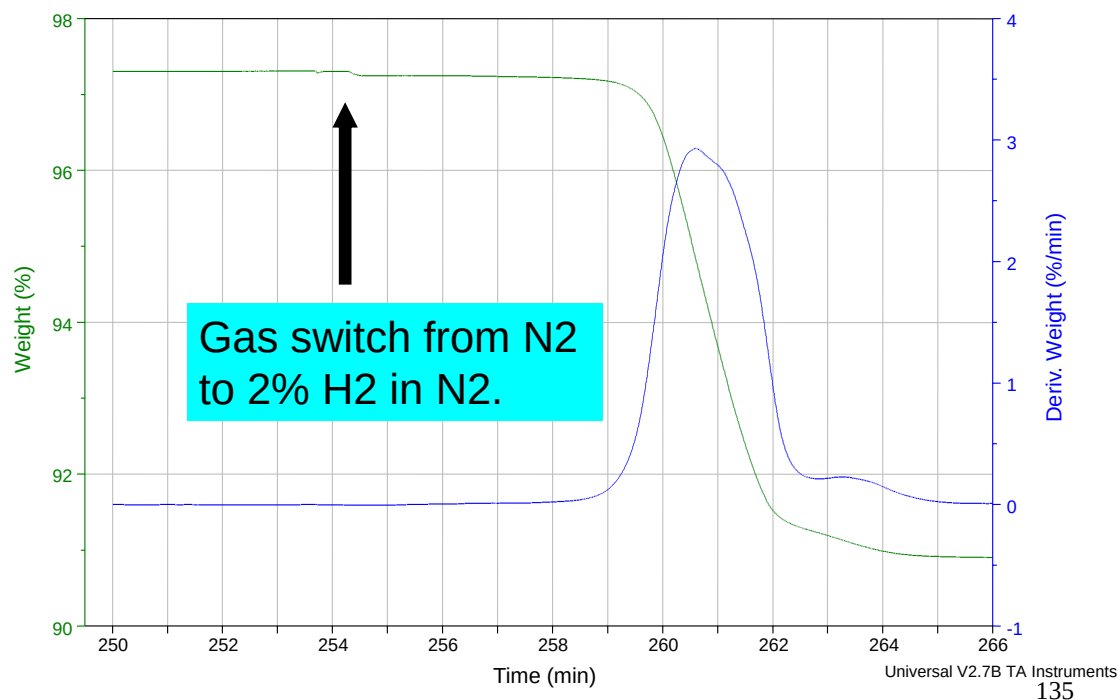
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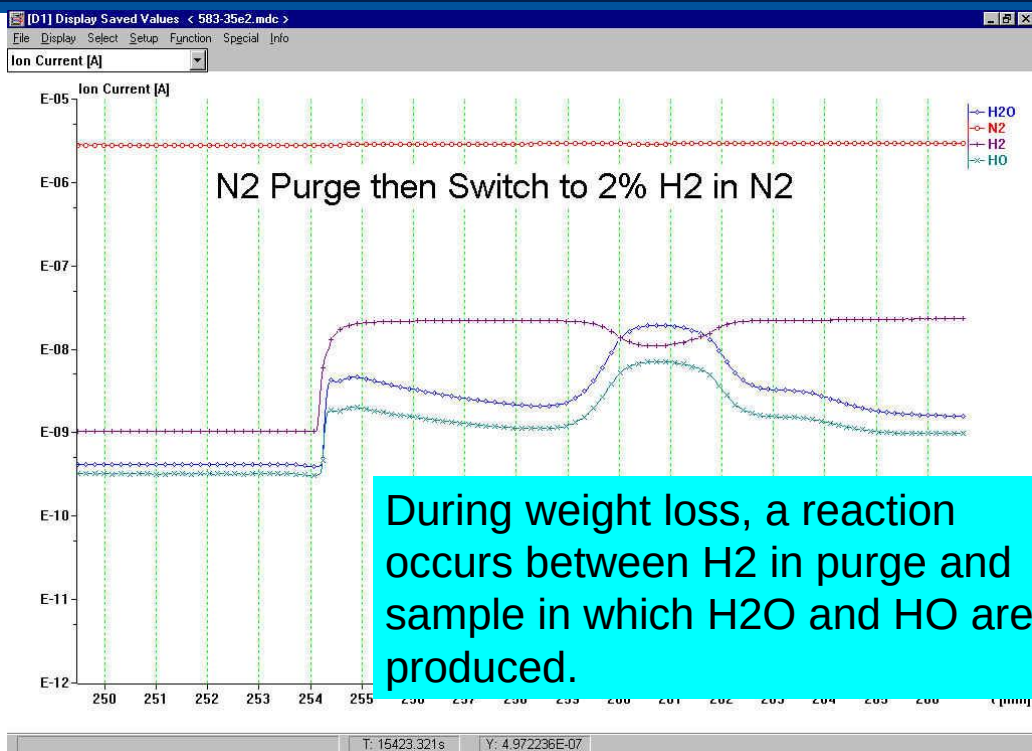
TGA-MS

Sample: 583-35-E
Size: 19.6330 mg

TGA

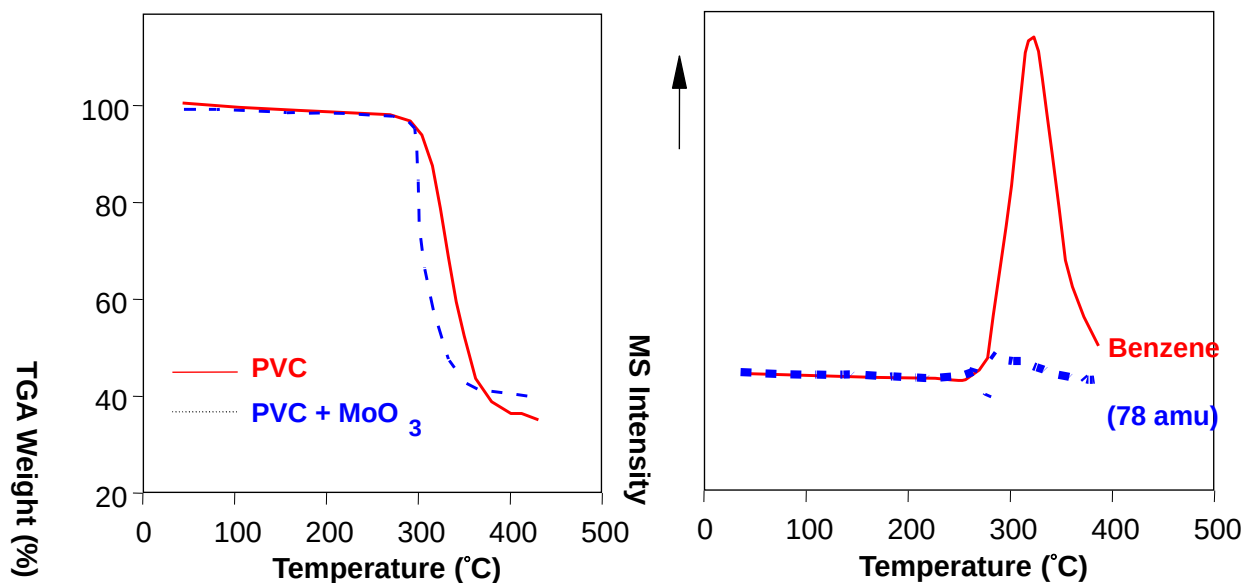


TGA-MS



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Smoke Generation in Flame Retarded Polymers (PVC)

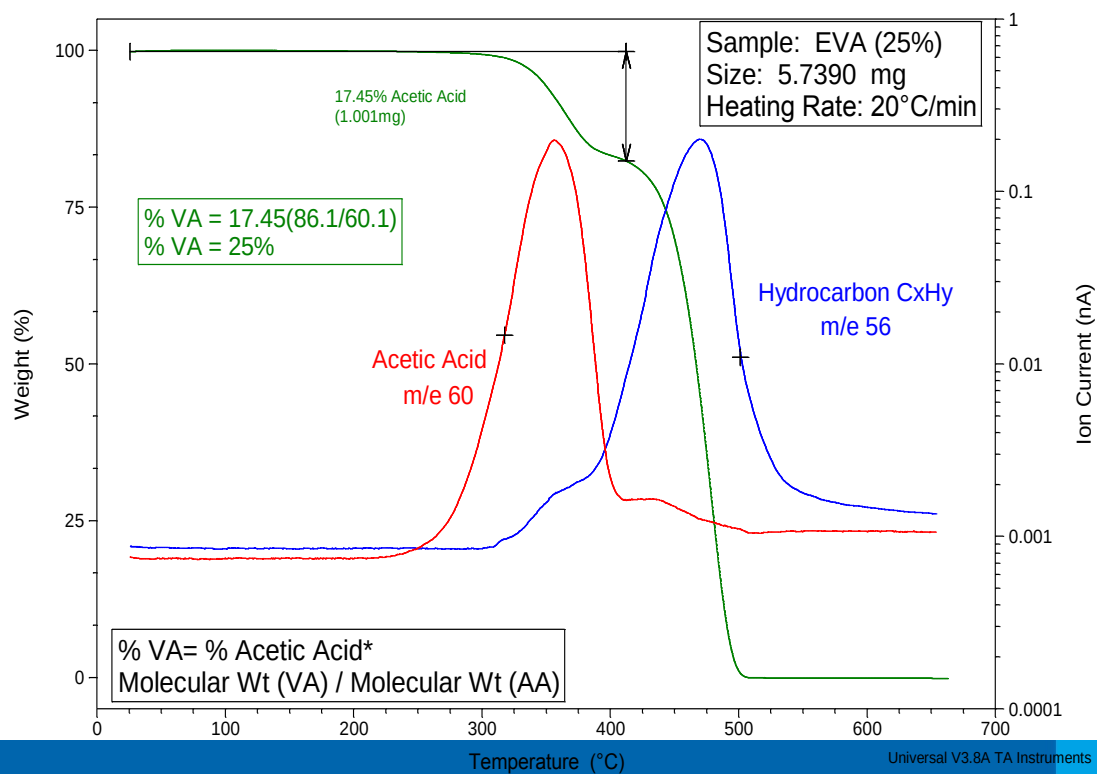


Benzene is a component of smoke. Much reduced in the flame retardant sample.

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Compositional Analysis by TGA-MS



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Universal V3.8A TA Instruments



Thank You

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Thermal Analysis, Rheology,
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Thank you for your attention!

