Thermogravimetric Analyzer
熱重量分析儀

TGA：重量變化的量測儀器

TGA: 定義

熱重量分析儀(Thermogravimetric Analysis,TGA)係指將樣品置於一受控制的環境下給予一可控制的溫度程式, 然後量測其重量隨時間或溫度而變化的回應. 通常透過此種分析可獲得樣品耐熱性及組成份的資訊. 在其間樣品可因為種種物理或化學變化而引起重量的減少或增加, 使用者必須對樣品具備足夠的認識才能加以判斷究屬何者?

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TGA: 告訴您哪些事?

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

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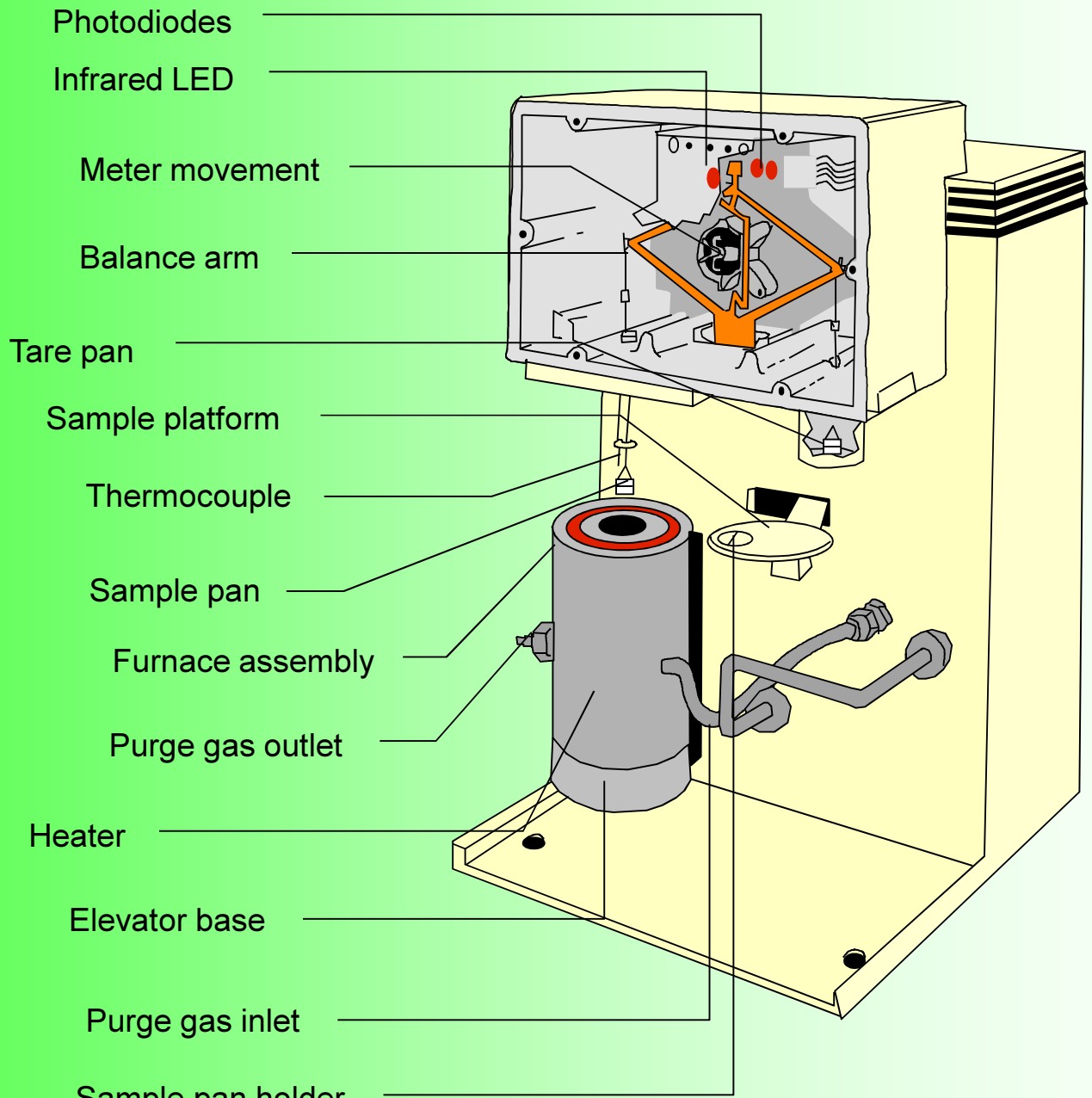
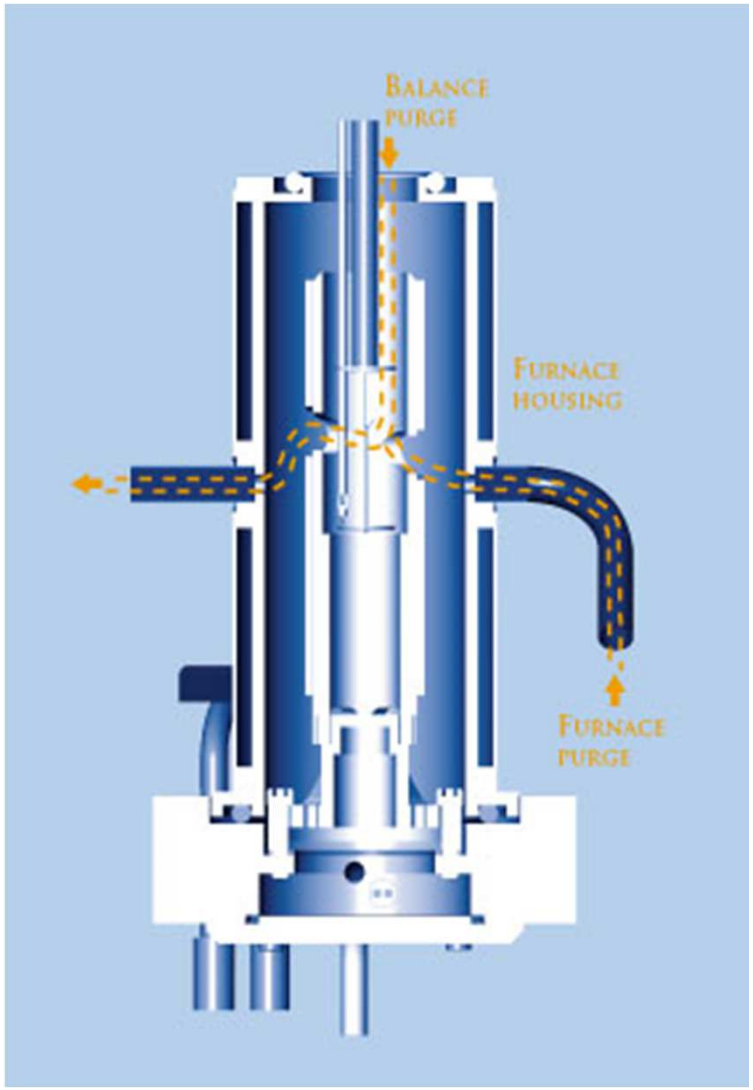
TGA 2950/2050/Q50/Q500: 外觀結構圖



獨特的加熱爐設計
Responsive Heater System

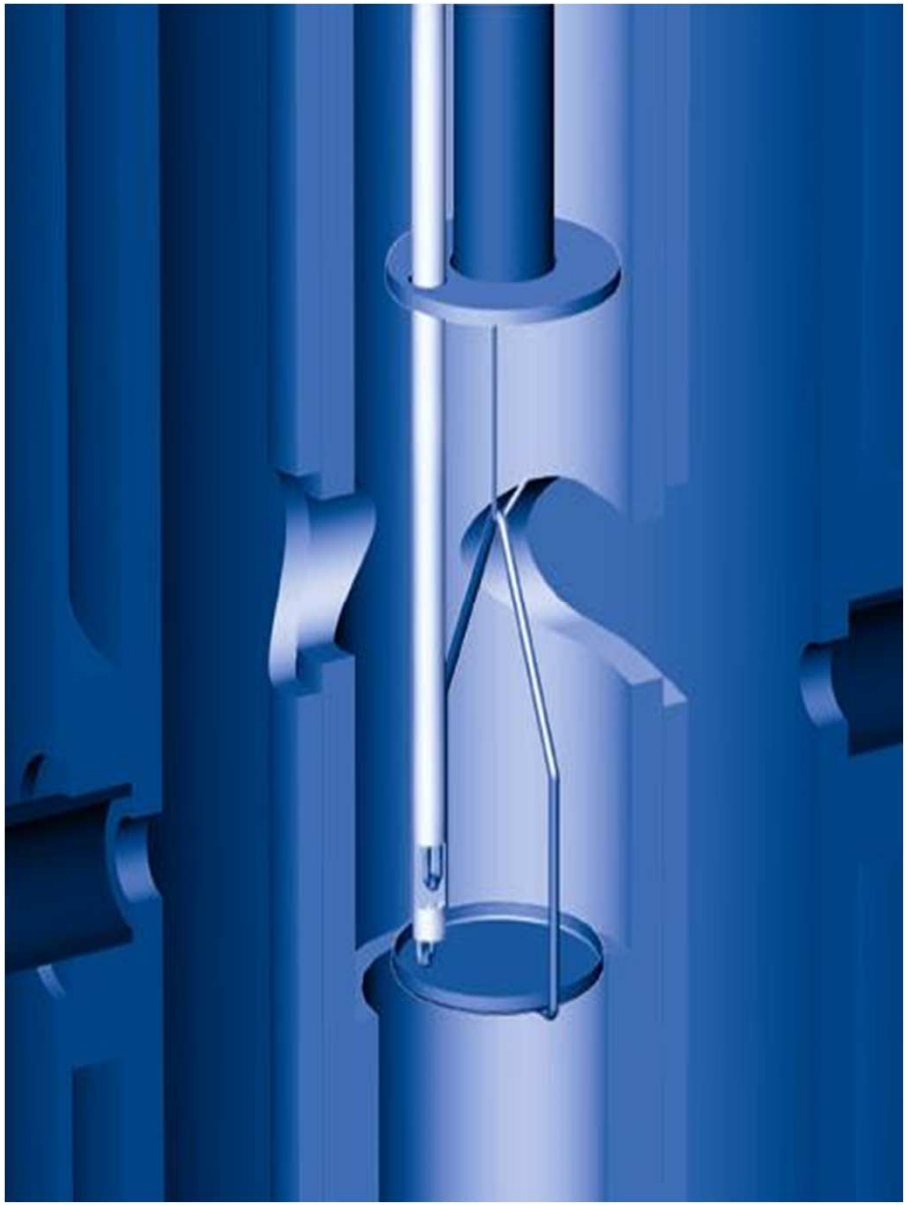
1) Low mass furnace core can achieve heating rates up to 200 deg/min easily.
2) Water cooled jacket provides ability to quickly reach isothermal equilibrium.
3) Automated furnace movement
4) Easy connection for off gases
5) Easy furnace removal for cleaning
6) Forced Air Cooling

獨特的加熱爐設計
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.

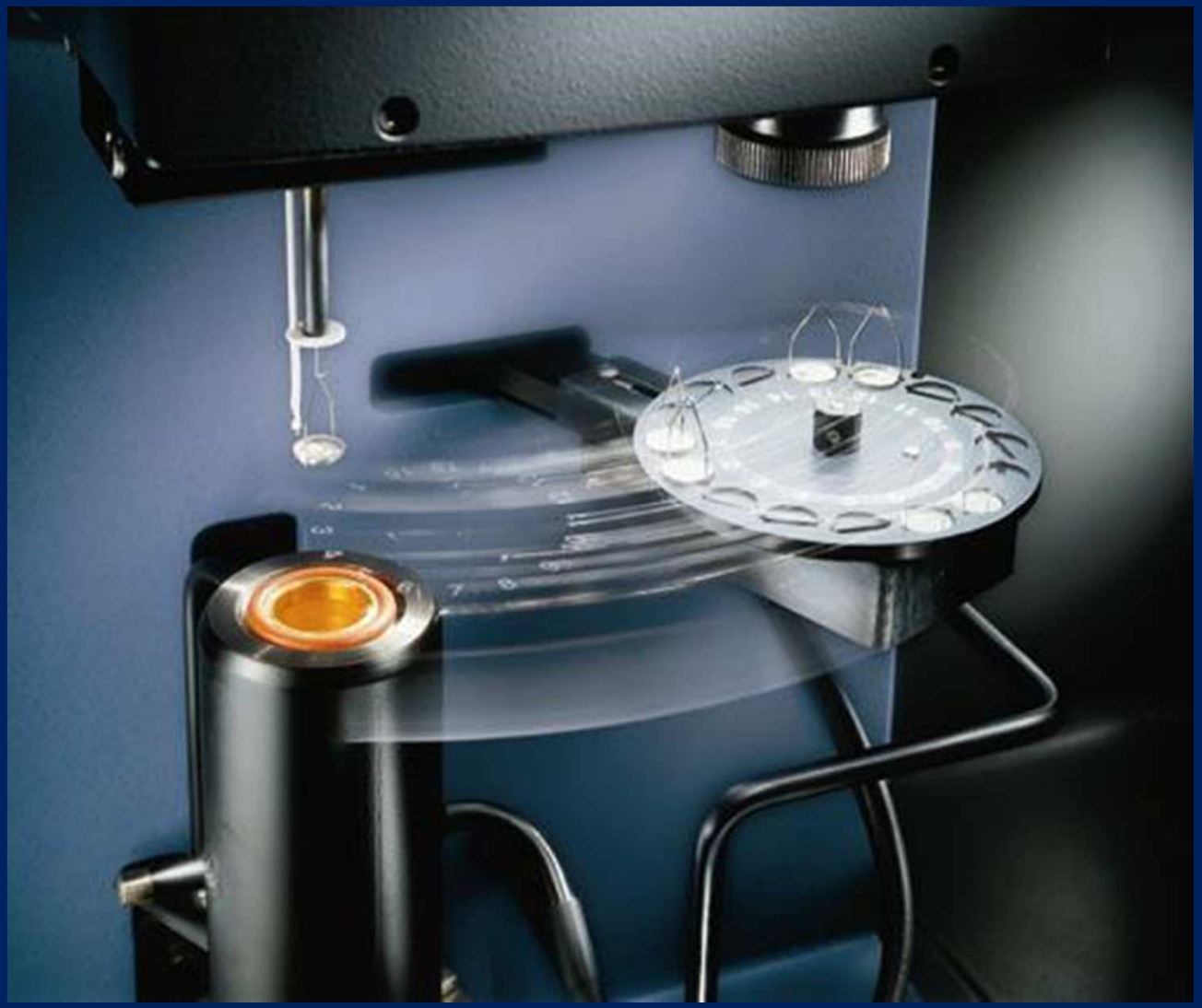
獨特的加熱爐設計
Same Sample/Control Thermocouple



The same thermocouple used for accurate measurement of the sample temperature is also used for controlling the furnace. The benefit of this unique capability is a highly responsive furnace. In particular, this design provides superior isothermal temperature control. A second thermocouple in the same ceramic sleeve is a backup thermocouple for safety purposes.

Q500 Upgrade Option

Reliable Autosampler

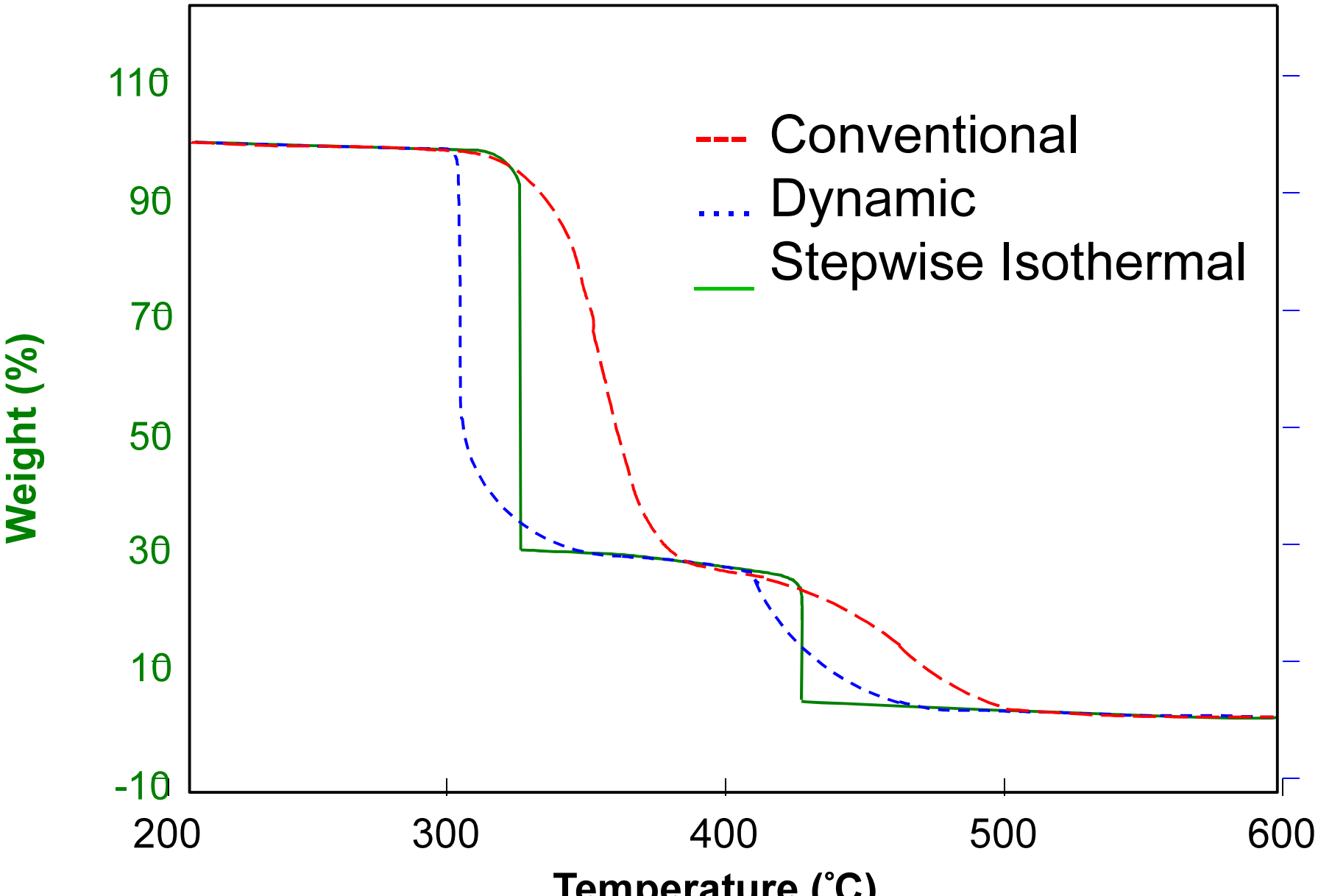


Q500 Upgrade Options

- **Hi-Res™ TGA**
- **Modulated ™ TGA**

Hi-Res™ TGA

PVC TGA Thermoplot Compared



Q 50/500 TGA

Choice of sample pans

TGA:

- **Experimental Note**

TGA: 影響基線穩定的因素

- 實驗桌面的穩定性
- 白金懸吊絲的狀況
- 金屬懸吊管的狀況
- TGA機台的水平
- 加熱爐的清潔程度
- 洗滌氣體的流量與穩定性

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TGA: 洗滌氣體(Purge Gas)

- 最常用的是氮氣
- 氦氣的基線最佳
- 若成分間彼此對氧化的速率有差異時, 則空氣可以增進解析度
- 可用草酸銅(Copper oxalate)來偵測是否有殘氧的污染

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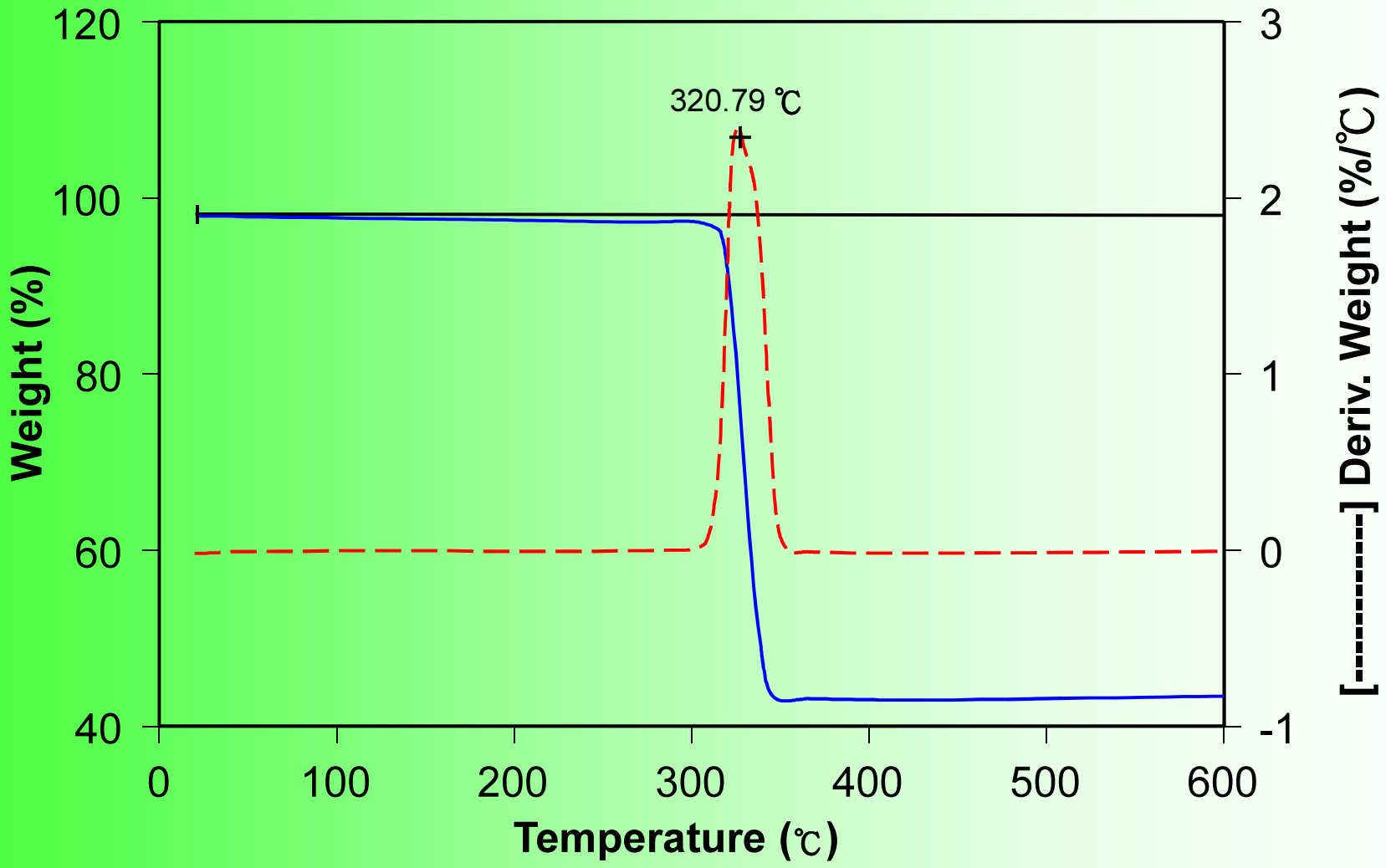
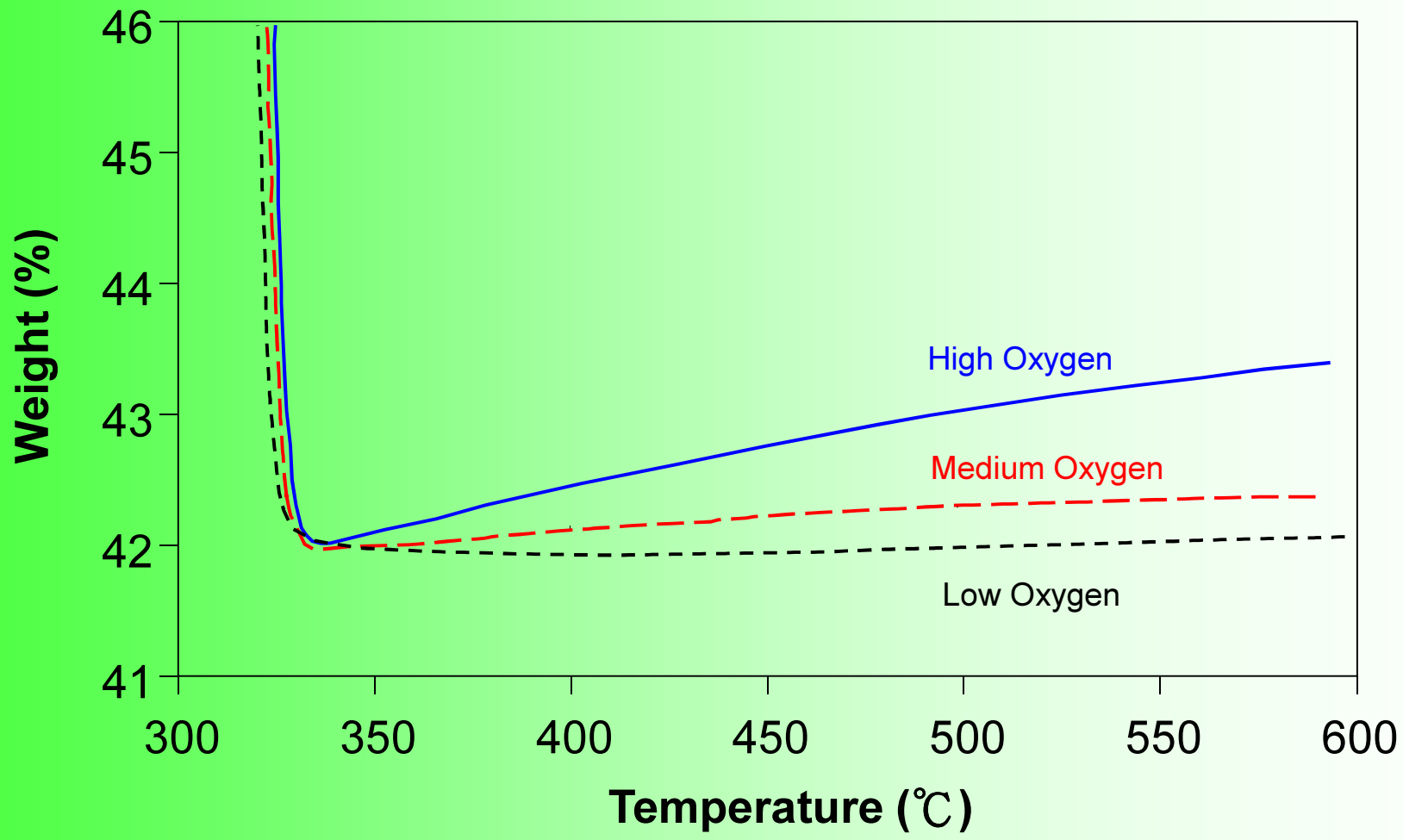
TGA: 使用草酸銅(Copper Oxalate)檢查氮氣中是否殘存氧氣?(全圖)



TGA: 使用草酸銅(Copper Oxalate)檢查氮氣中是否殘存氧氣?(部分放大圖)



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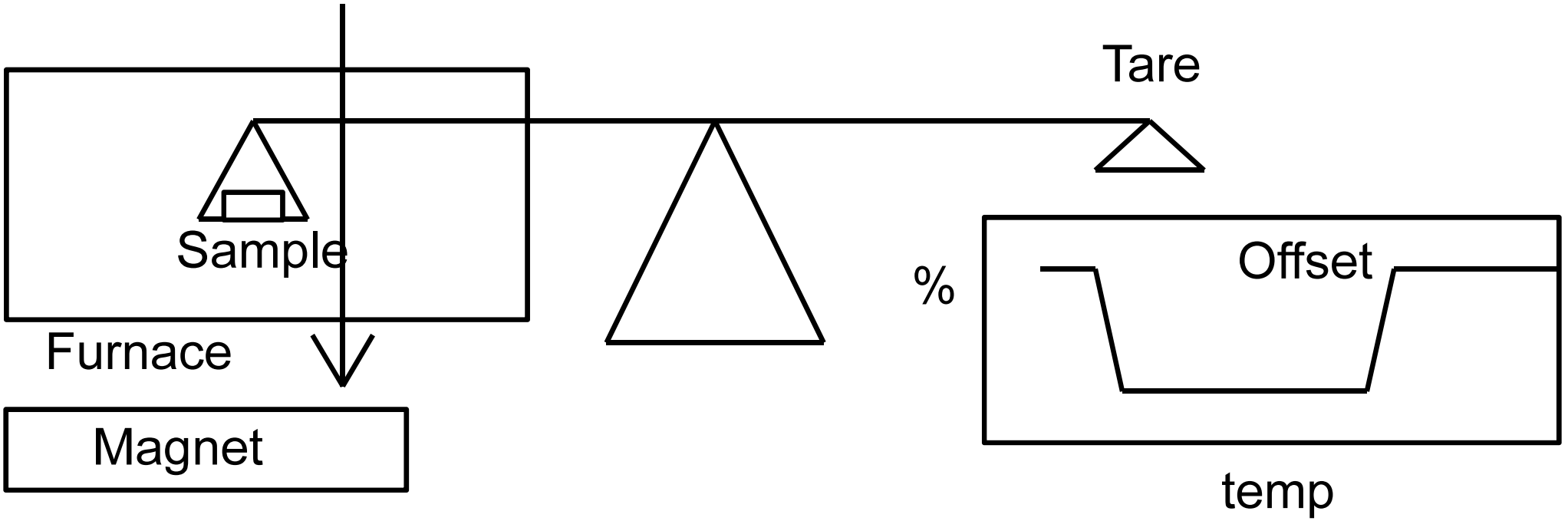
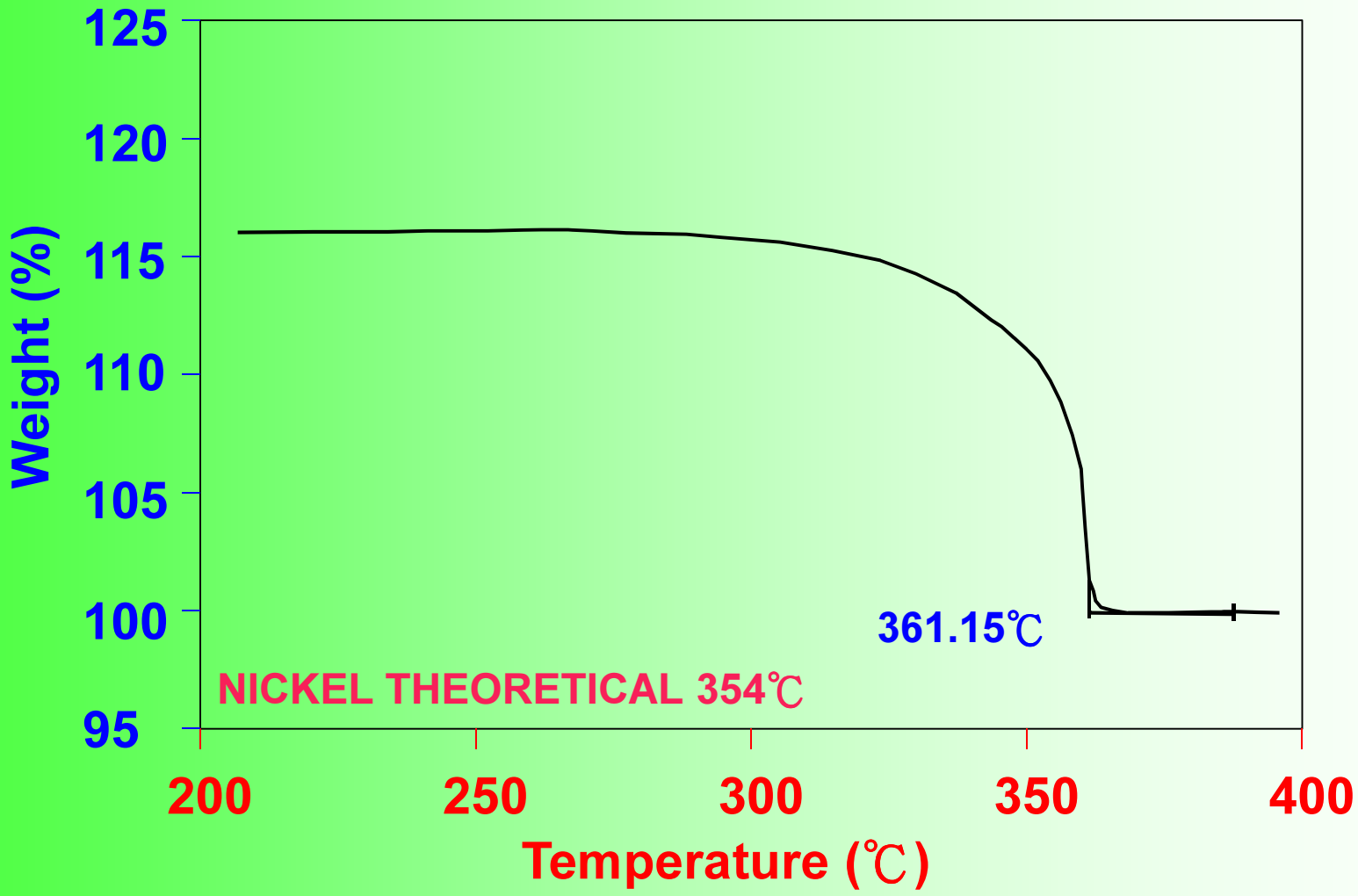
TGA: 溫度校正 - 居禮點(Curie Point)轉移法

Vertical Balance Configuration - TGA 2050/2950

Attraction of Sample to Magnet
Results in Initial Weight Gain



TGA/SDT:溫度校正 -
居禮點(Curie Point)轉移實例



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TGA: 如何製備樣品?

- 越大的總表面積有越佳的解析度及溫度再現性
- 樣品重量

  一般樣品用 **10-20mg**

  揮發性樣品用 **50-100mg**
- 大多數情形**TGA**本身的基線漂移量大約有**+0.025mg**，意即對一 **10mg** 樣品量而言有 **+0.25%** 的誤差
- 使用銅製鑷子以減少靜電干擾
- 每次實驗都要用乾淨的樣品盤歸零
- 樣品要平均分布在底面
- 液態樣品則事先封妥在有針孔的密封樣品盤內

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TGA: 選用適當的樣品盤(1)

- Platinum (useful for most materials)
  - Easy to Clean
  - Nonporous
  - Alloys with most metals
- Alumina (Ceramic)
  - Corrosives/Inorganics
  - Large samples
- Aluminum (TGA) (designed for one-time use)
  - Lower cost
  - Lower temperature limit (<=600)

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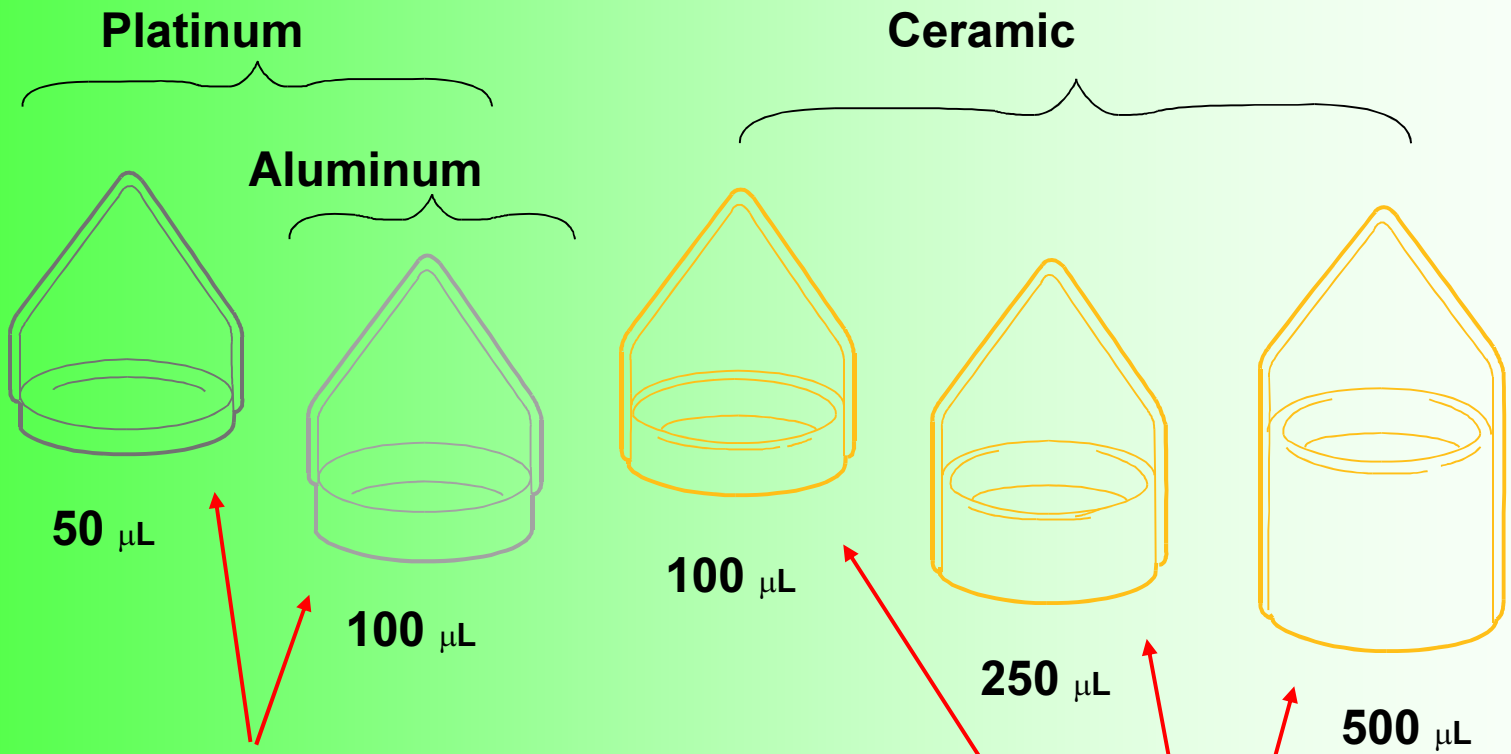
TGA:選用適當的樣品盤(2)



Platinum and Aluminum pans have attached wire bail.

Ceramic pans have removeable wire bail.

TGA/SDT: 如何清潔樣品盤?

- All sample pans are reusable (except Aluminum)
- Flame remaining residue from pan with torch (do not flame Aluminum pans)
- Scrape off remaining ash (DSC fiberglass brush)
- Tare clean sample pan before each run

TGA: 代表性的應用例子

- Thermal Stability
- Compositional Analysis
- Oxidative Stability

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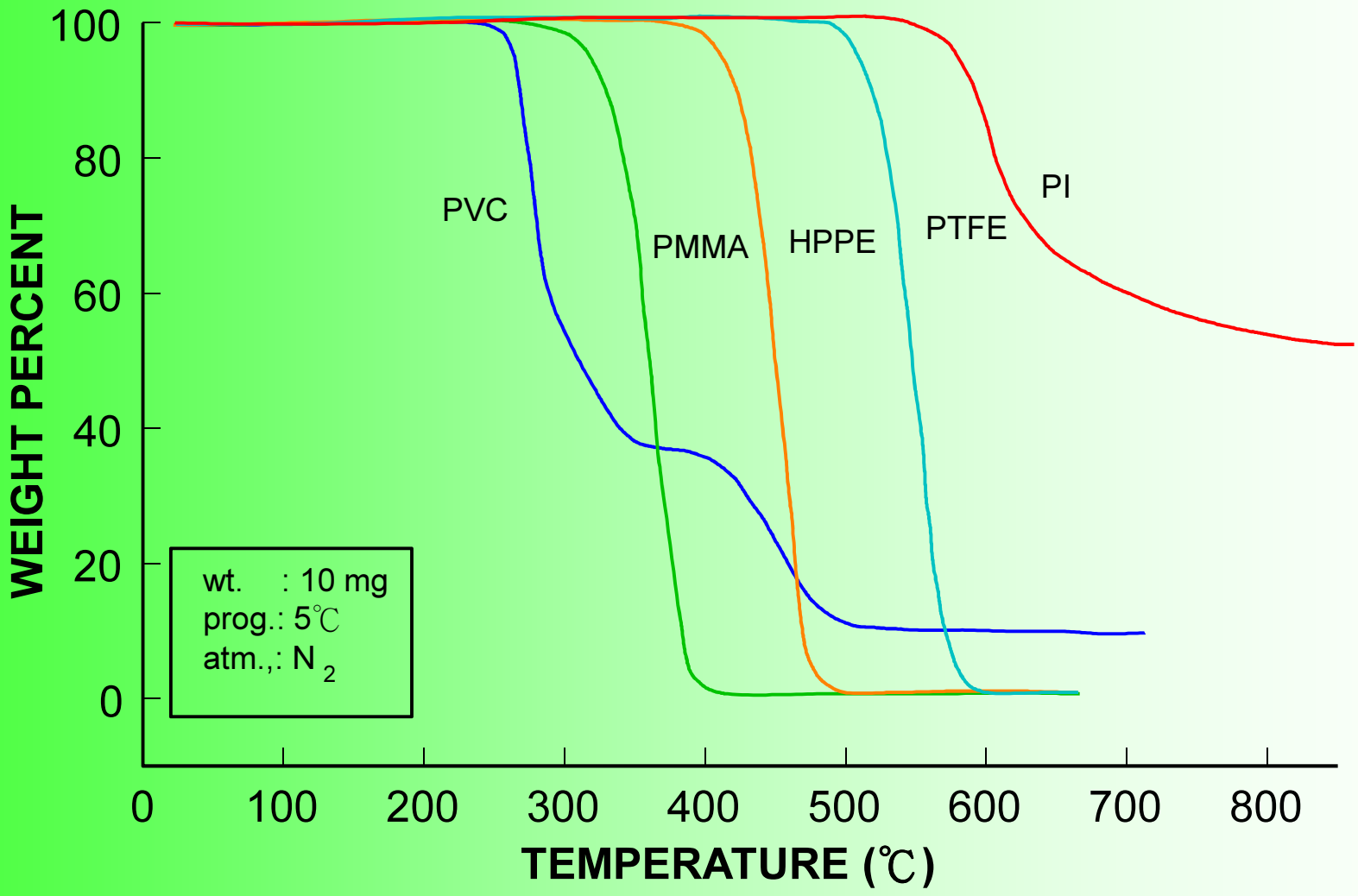
TGA: 評估高分子材料耐溫性與特性分解圖



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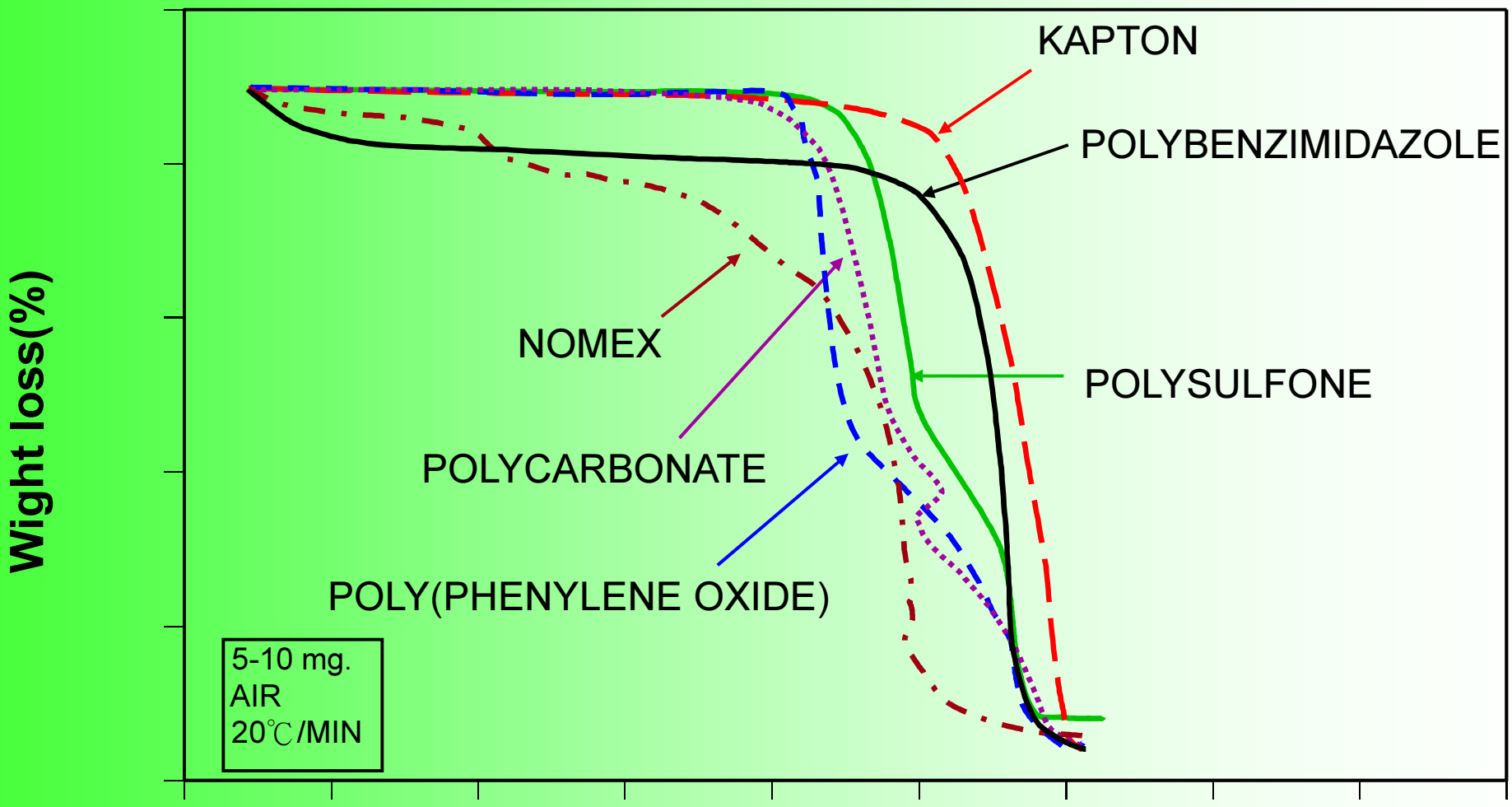
TGA:評估高分子材料耐溫性



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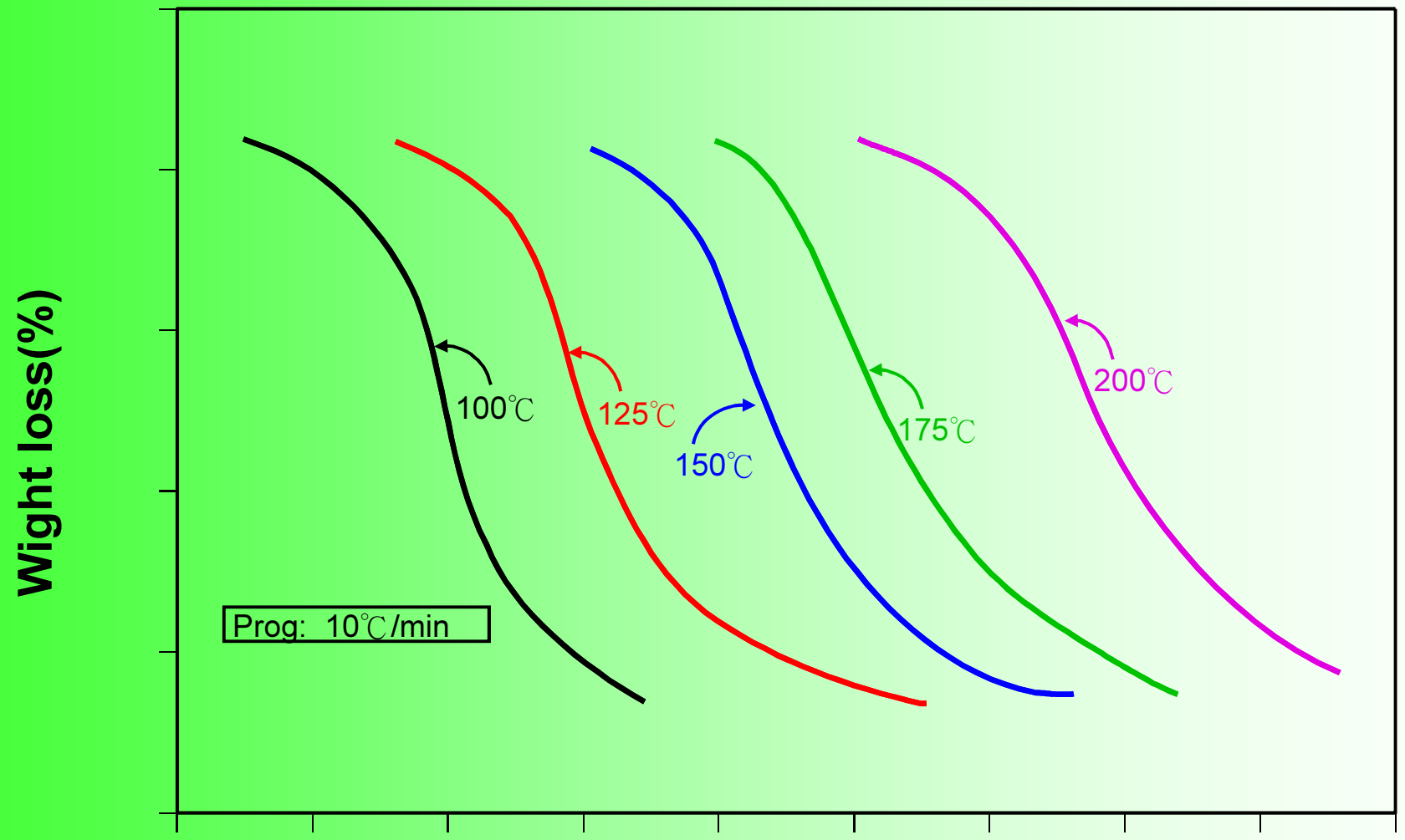
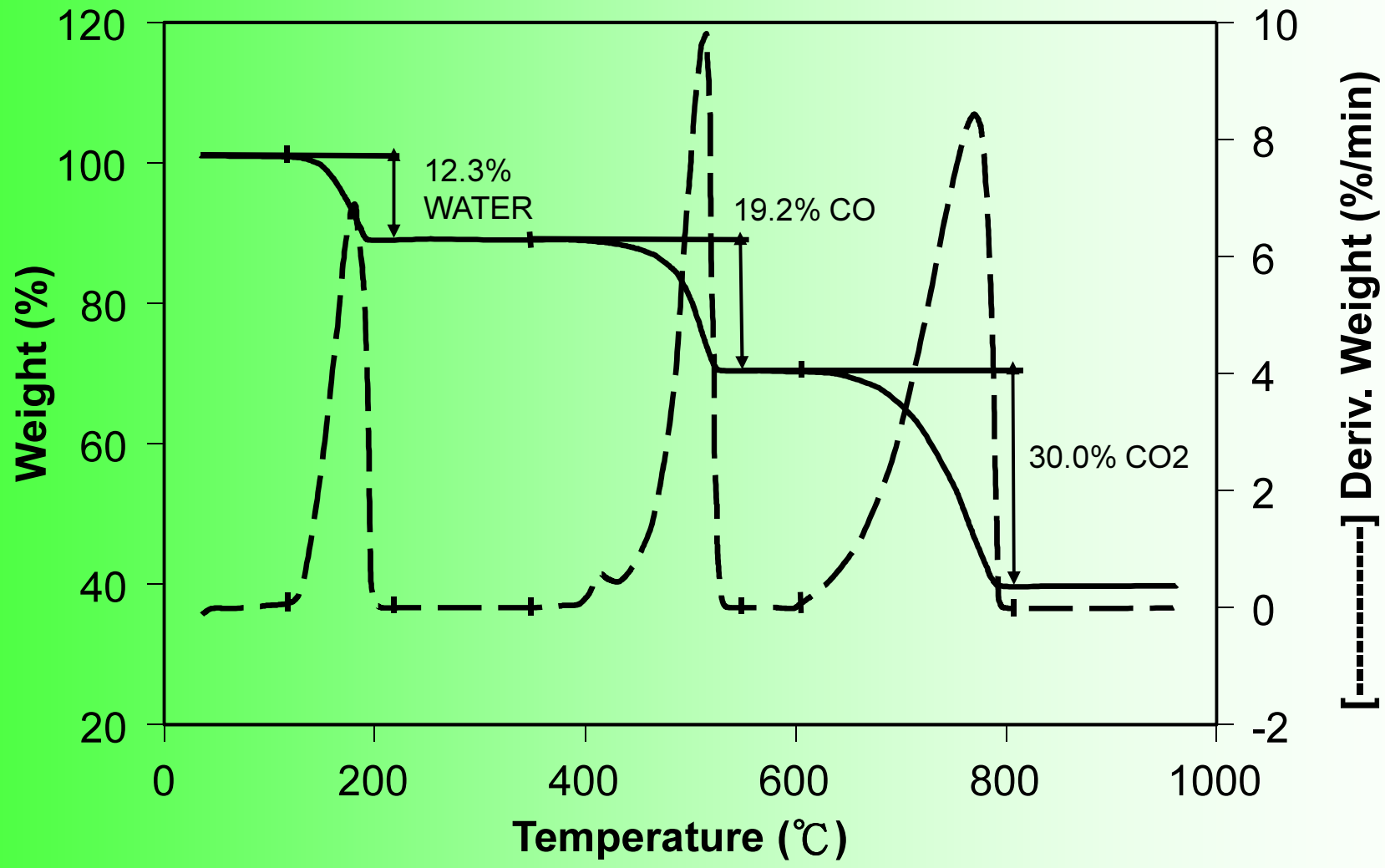
TGA: 觀察在不同反應溫度下之環氧樹脂的熱分解特性



TGA:成分分析的應用-
草酸鈣(Calcium Oxalate)



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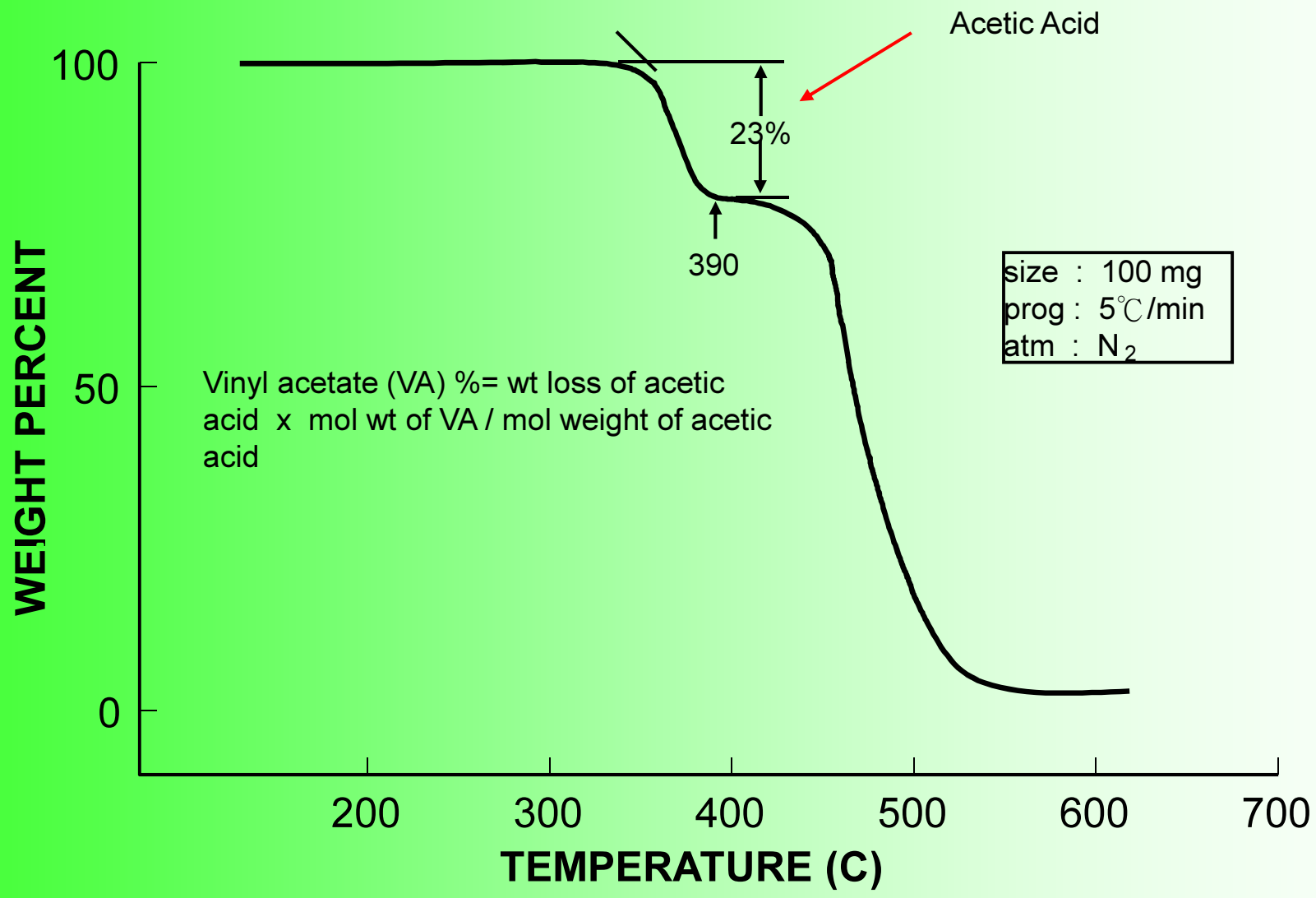
TGA:成分分析的應用-
EVA 共聚合物 [J. Chiu, Appl. Polym. Sym., 2, 25 (1966)]



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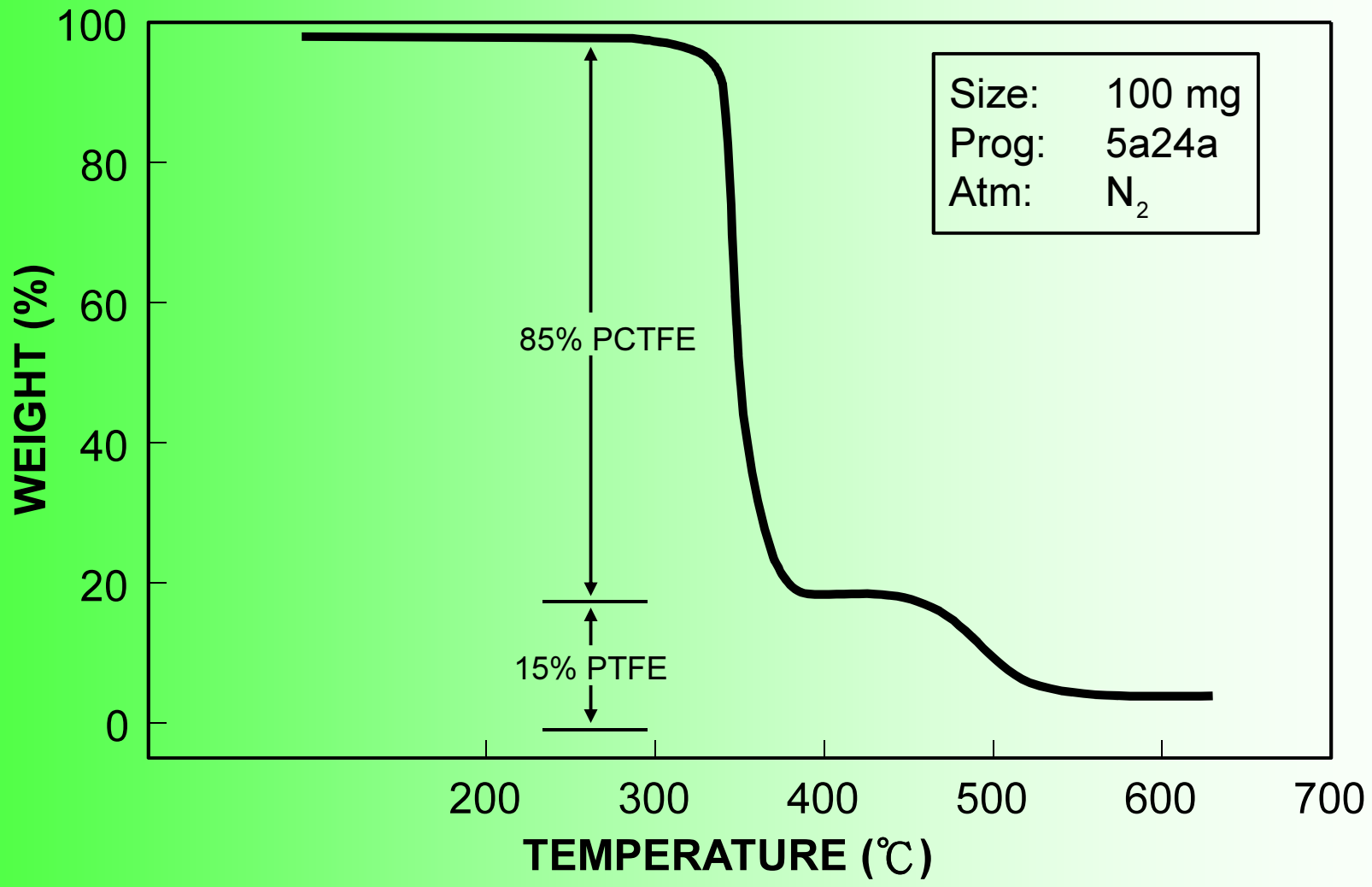
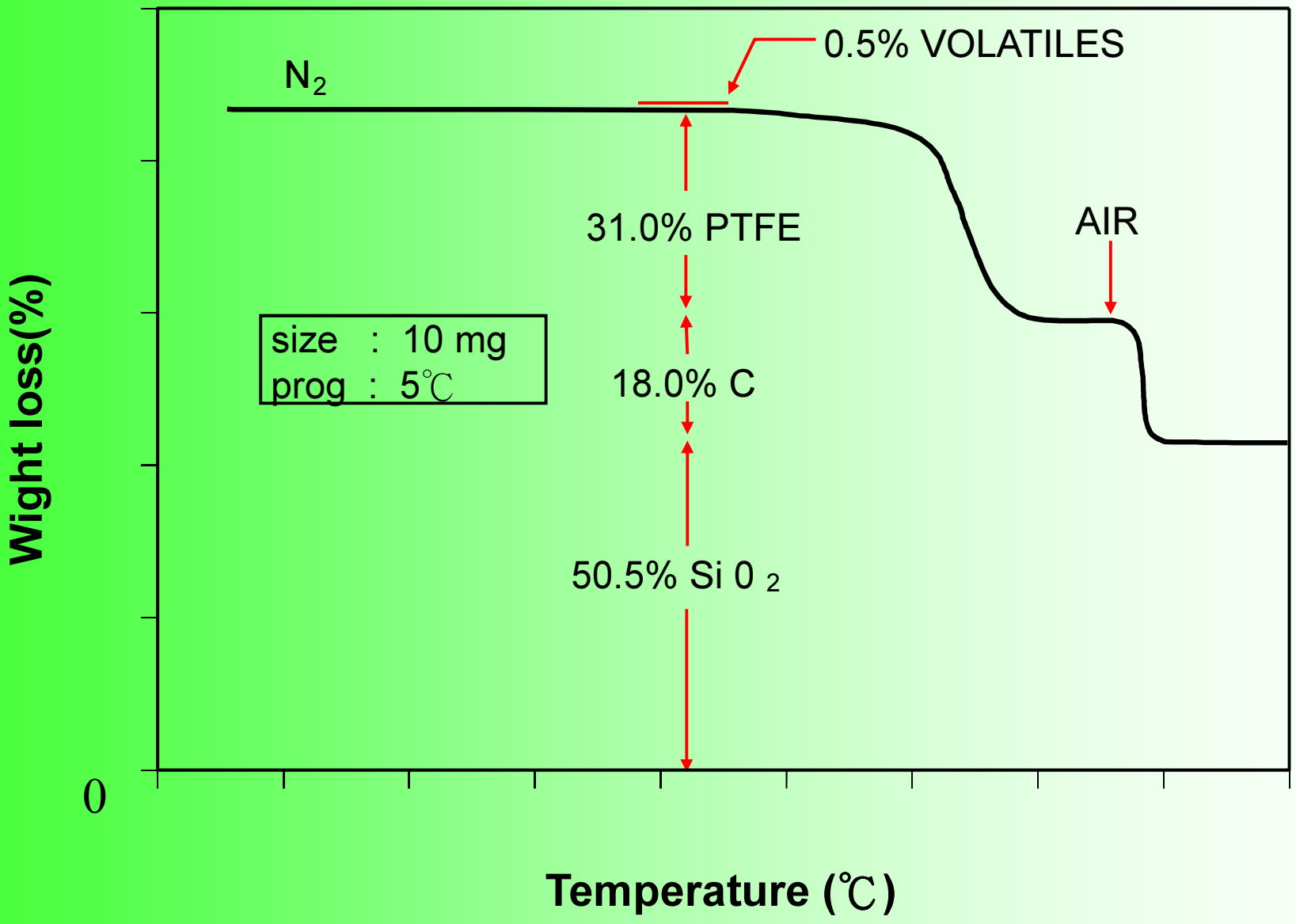
TGA:成分分析的應用-
氟碳共聚物 Fluorocarbon Mixture

Polychlorotrifluoroethylene/Polytetrafluroethylene

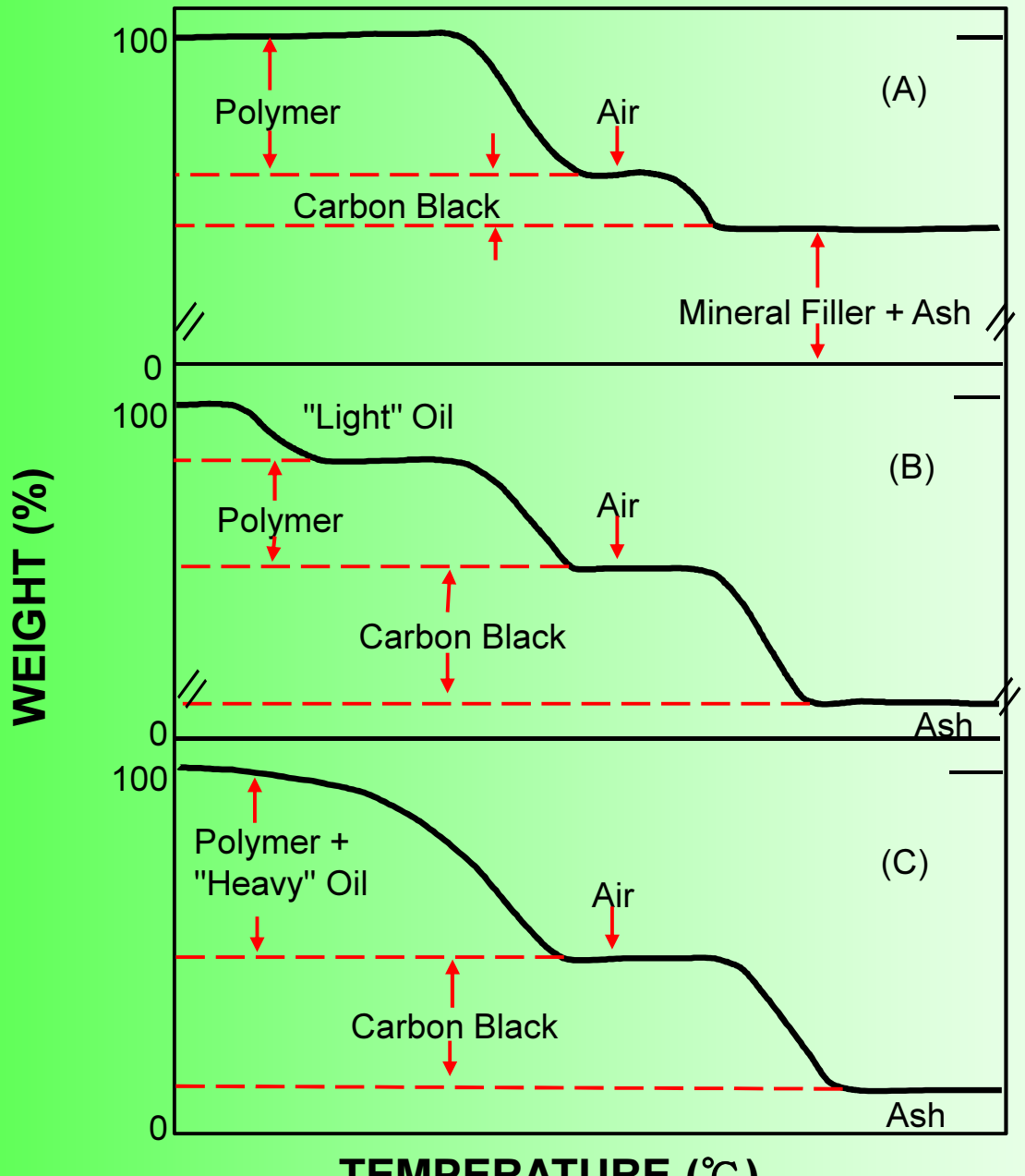


J. Chiu, Appl. Palm. Sym., 2, 25 (1966)

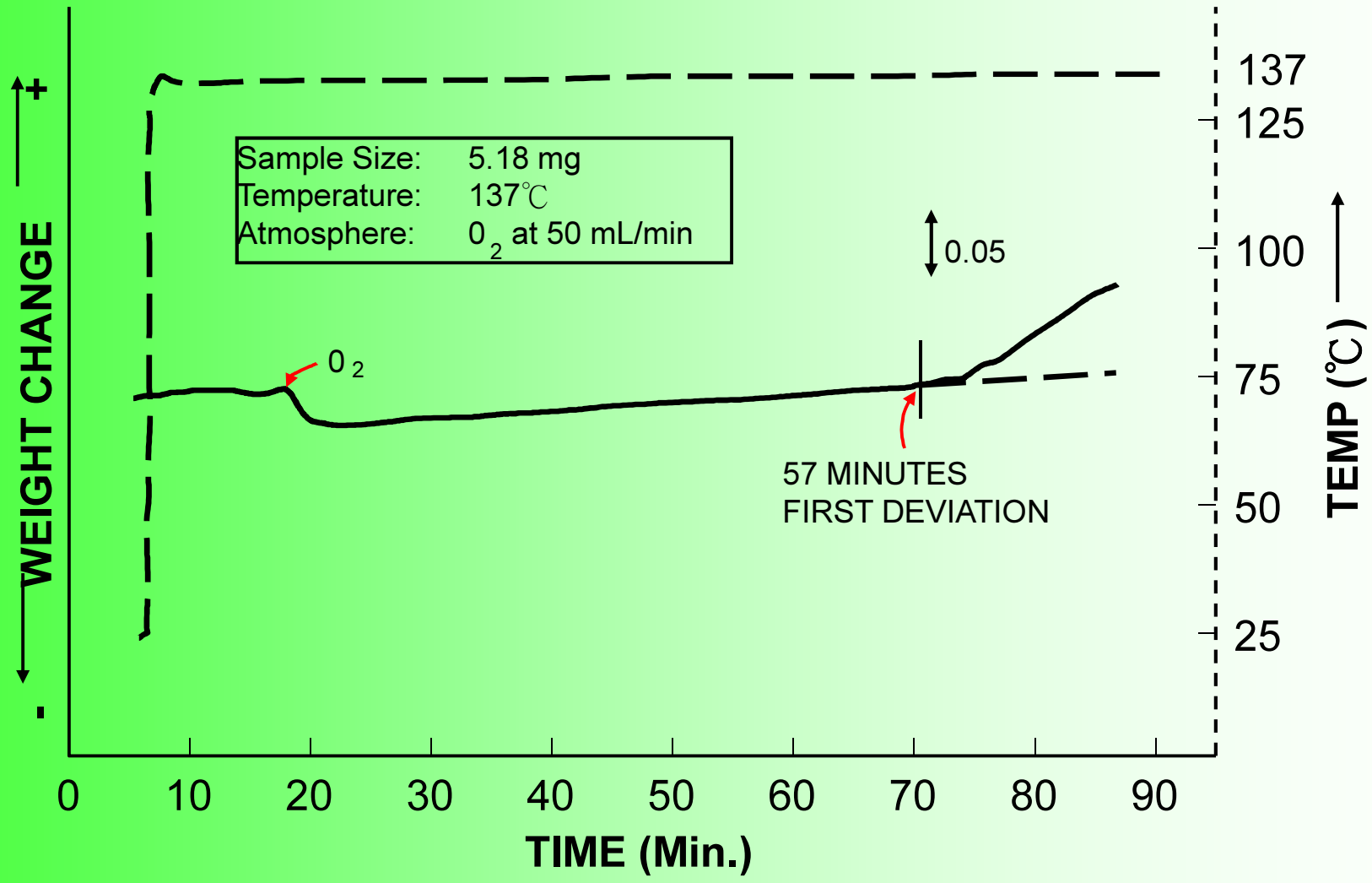
TGA:成分分析的應用-
複合材料的分離技巧



TGA:成分分析的應用-輪胎的分離技巧



TGA:氧化安定性的應用-
植物油(Vegetable Oil)



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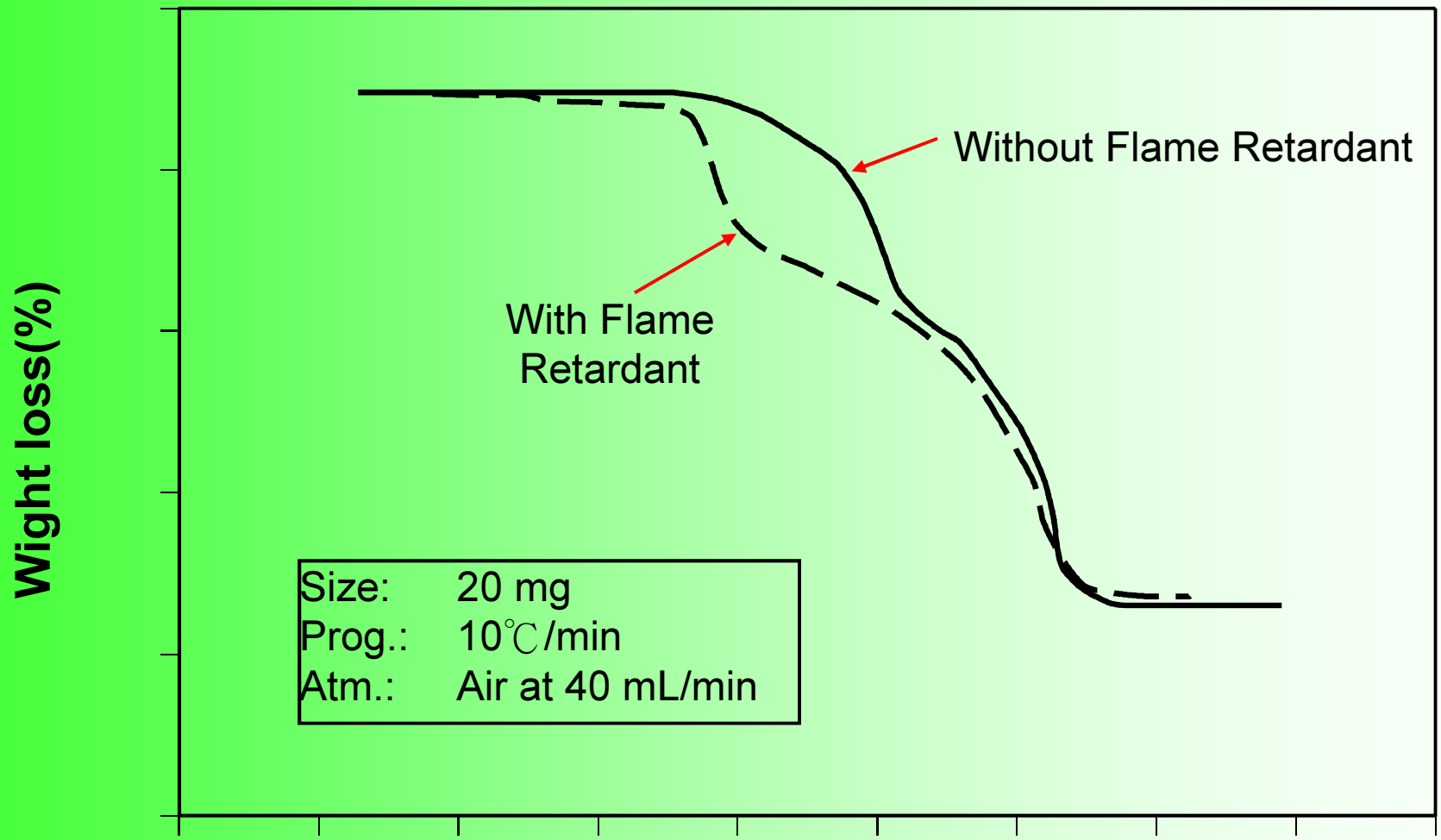
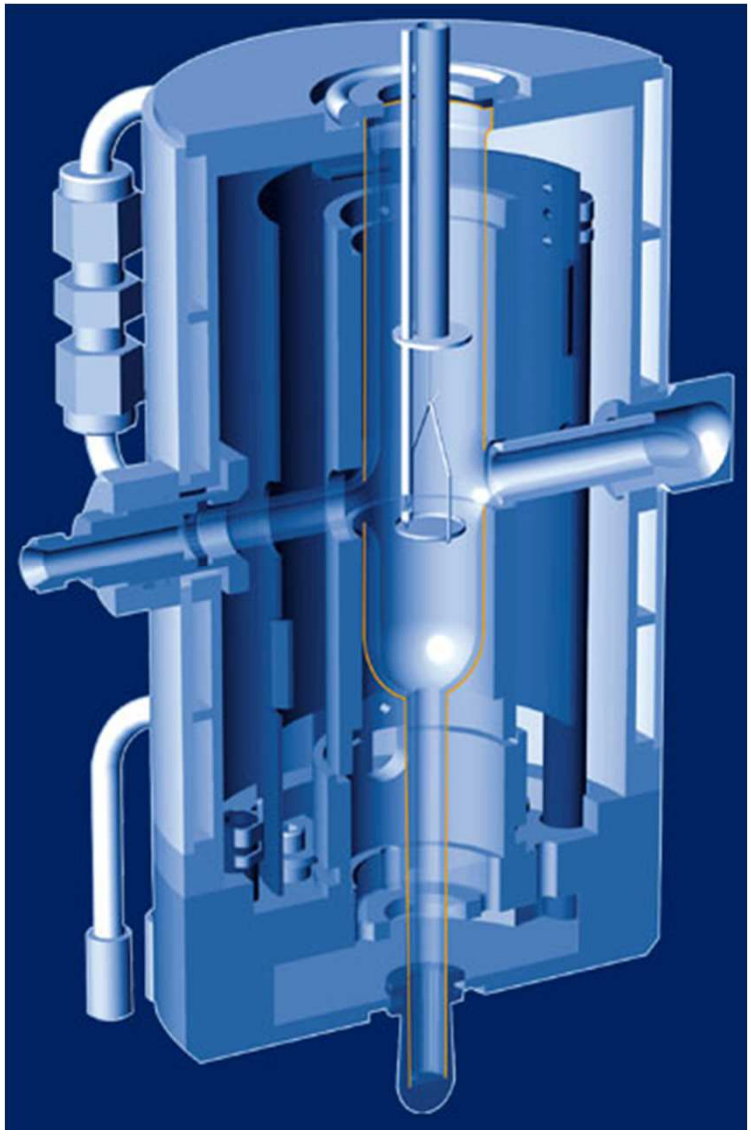
TGA:

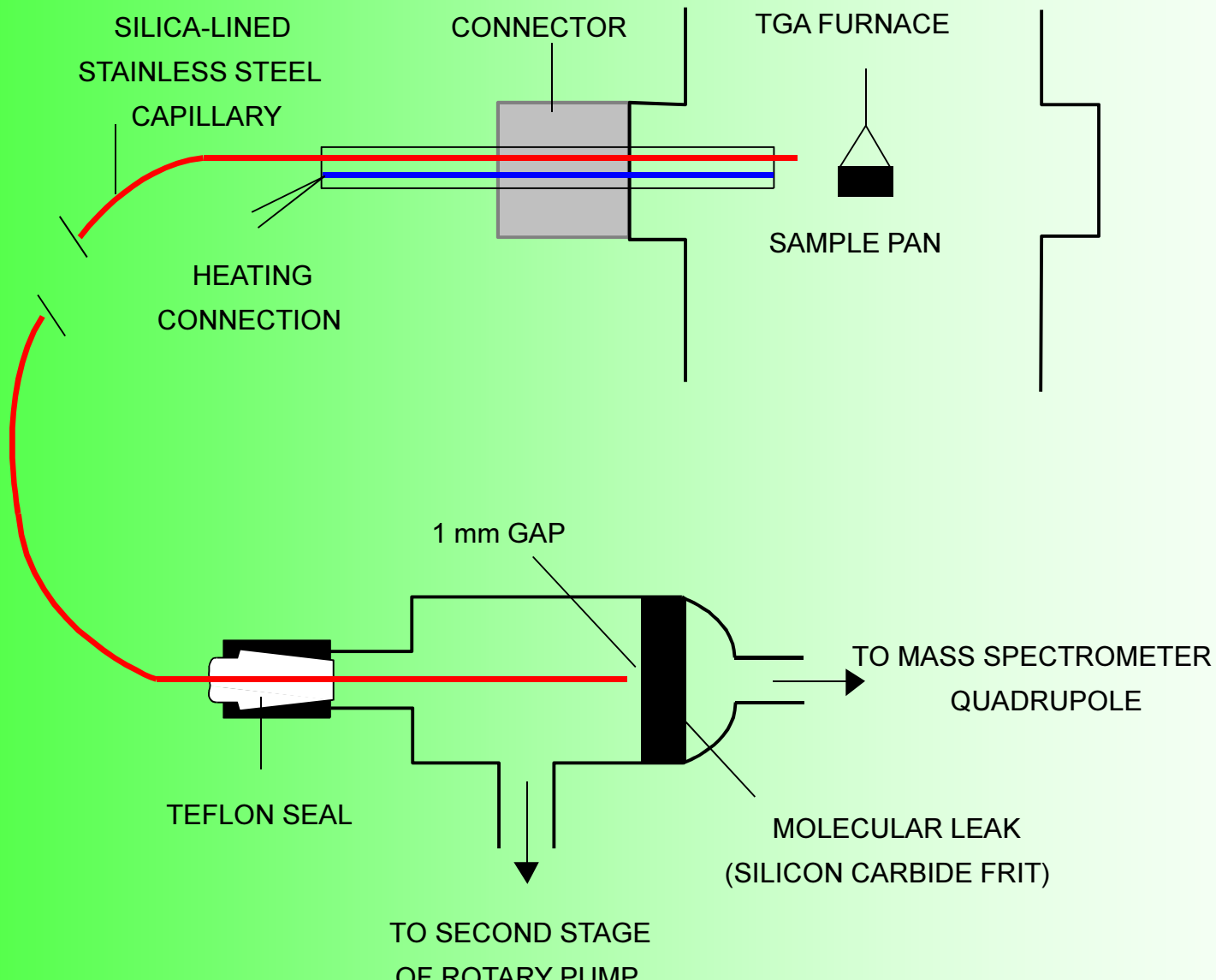
- **TGA-MS**

TGA:氧化安定性的應用-環氧樹脂(Epoxy)

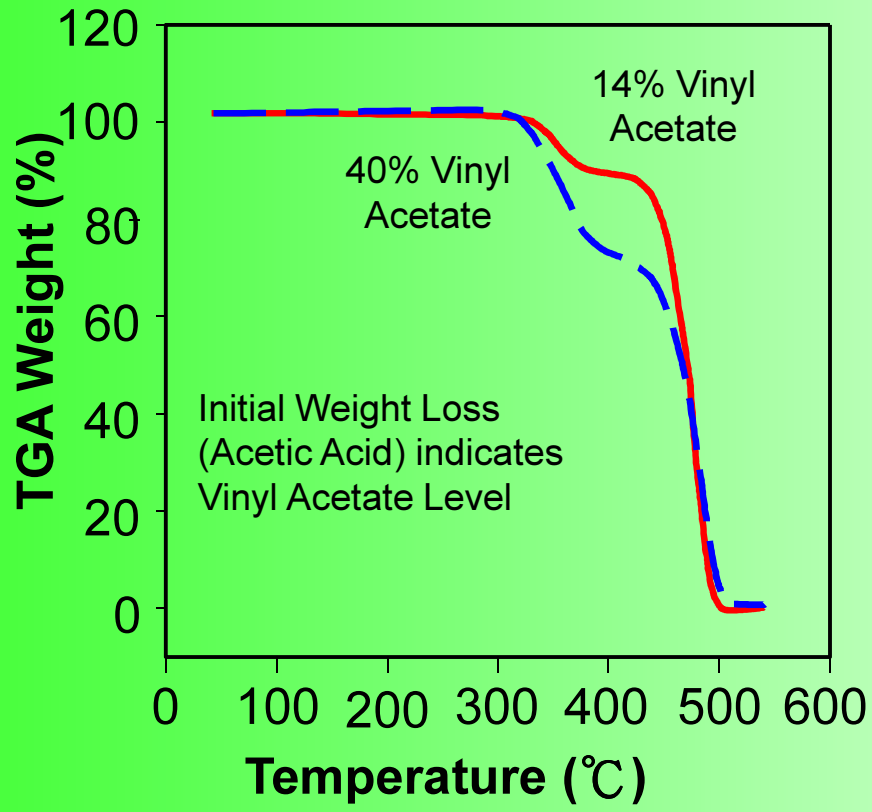


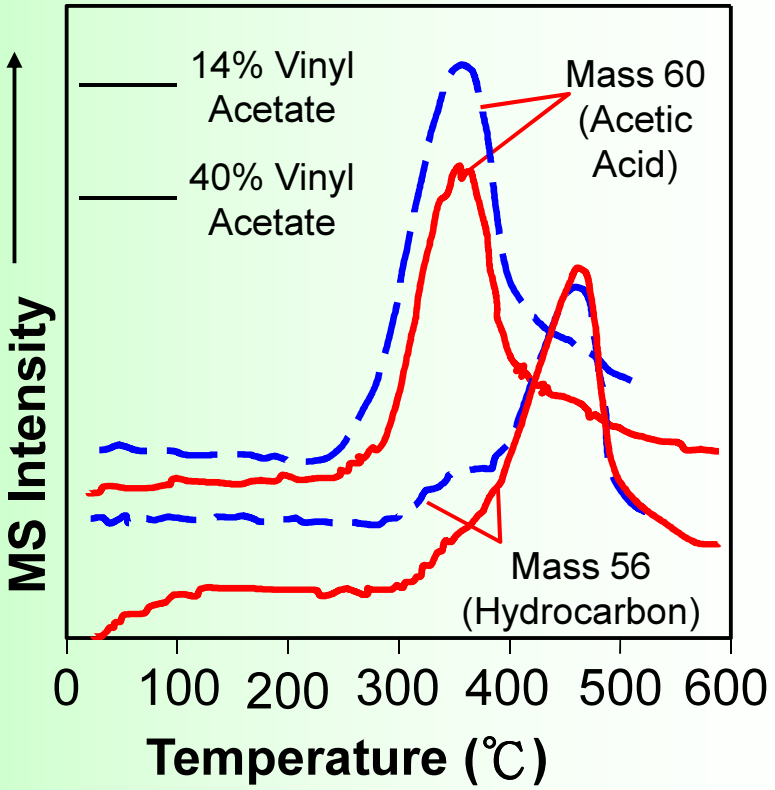


TGA-MS: 成分鑑定的利器
毛細管介面(Capillary Interface)



TGA-MS: 成分鑑定的利器
決定高分子組成(EVA共聚物)





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TGA智慧調變溫控技術

High Resolution TGA(HRTGA)

Stepwise TGA(STGA)

Modulated TGA(MTGA)

傳統TGA –提高解析度的對策與限制

- 降低升溫速率--浪費時間且不見得有效
- 減少樣品量--同時會提高雜訊與誤差
- 提高總表面積--增加製備困難度
- 改變洗滌氣體,如氦氣--浪費錢
- 採用針孔密封盤--只有某些樣品適用

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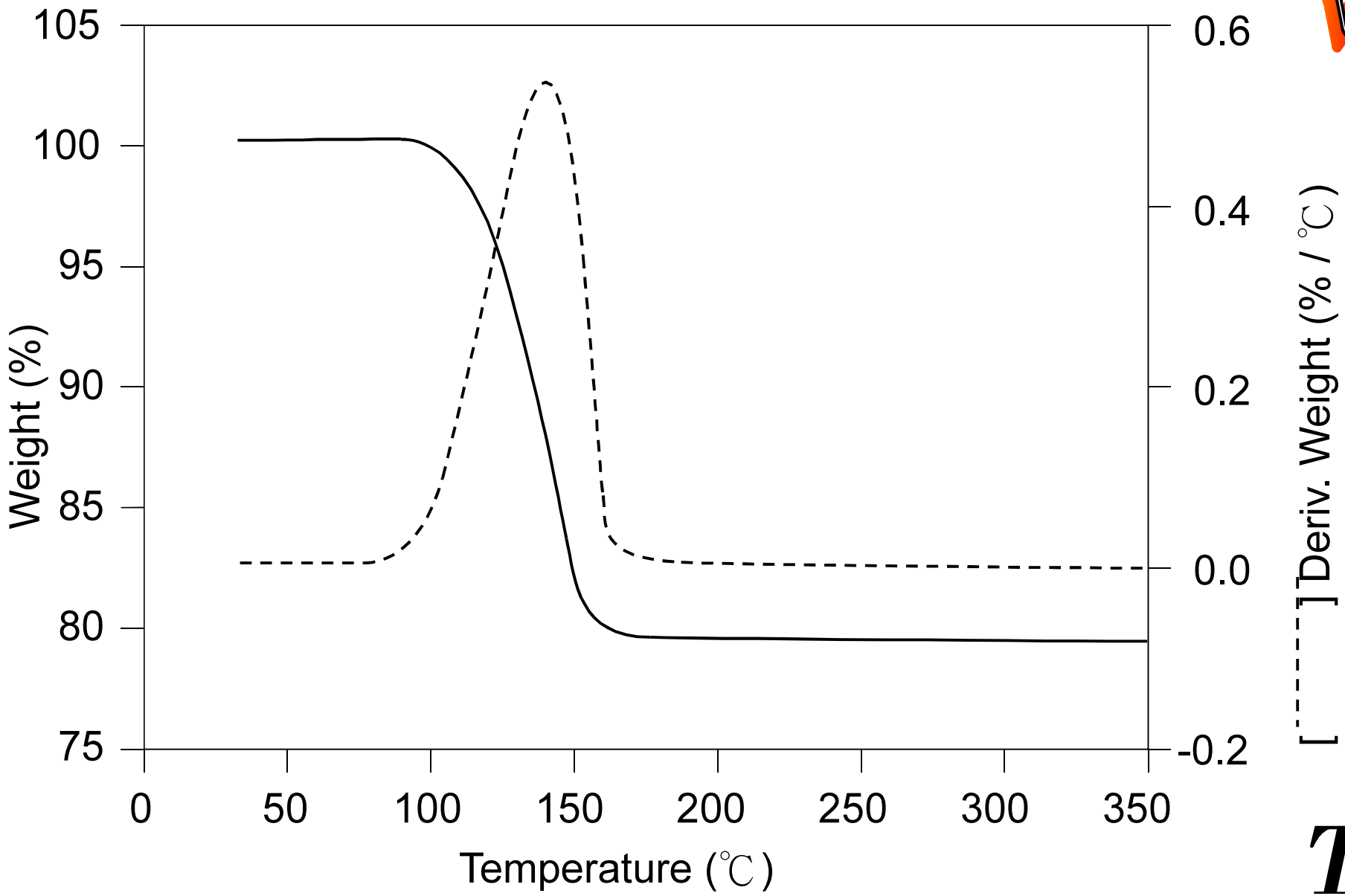
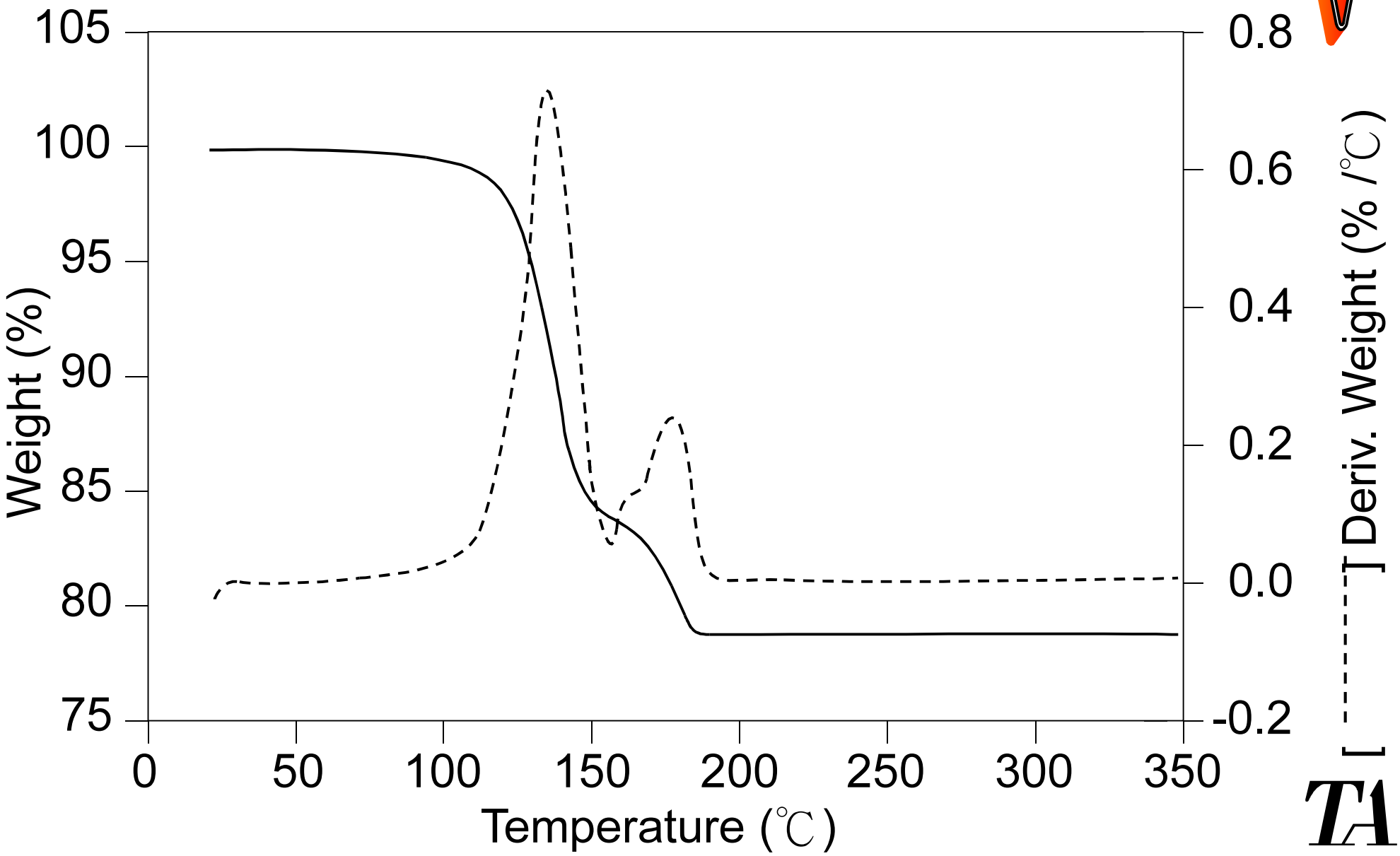
傳統TGA傳統做法 – 二水硫酸鈣(Open Pan)



傳統TGA改善做法 – 二水硫酸鈣 (Pinhole Lid Pan)



Hi-Res TGA –開創提高解析度的另一途徑

導致傳統TGA解析度不彰的原因:

- 複雜的熱掃描圖譜又逢寬闊的失重特性

and/or

- 重疊-前者未去,後者又生

Hi-Res TGA – 結合精確的爐溫控制與智慧型的溫控策略

- 三種手法:
  - **Dynamic (Hi-Res) TGA**
    動態溫控策略
  - **Constant Reaction Rate TGA**
    固定失重速率策略
  - **Step Wise Isothermal TGA**
    步階恆溫與固定升溫速率策略

TA

TGA: Hi-Res TGA –
何謂 Dynamic TGA?

一旦失重速率發生變化時,系統依據使用者指定之實驗條件(最大加熱速率;靈敏度;解析度)自動改變加熱速率; 而此加熱速率的改變係以動態調諧的方式進行,一點都不勉強,得以獲得高解析且避免發生誤判.

TA

Hi-Res TGA - Dynamic Rate

好處看得見

- 輕輕鬆鬆找到適當的實驗條件
- 迅速在寬廣的溫度範圍內尋獲優異的解析度
- 不必要求對樣品事先有充分了解
- 即便一無所知, 仍然可以在短時間內得到相當不錯的解析度

TA

Hi-Res TGA - Dynamic Rate

舉一個溫控程式的例子

1. Sensitivity 2.0
2. Ramp 50℃/min, Res. 5.0 to 1000℃

說明:

50℃/min : Max. ramp rate

Res. 5.0 : Resolution setting (0 to +8.0)

2.0 : Sensitivity setting (1.0-8.0)

1000℃ : Final temperature

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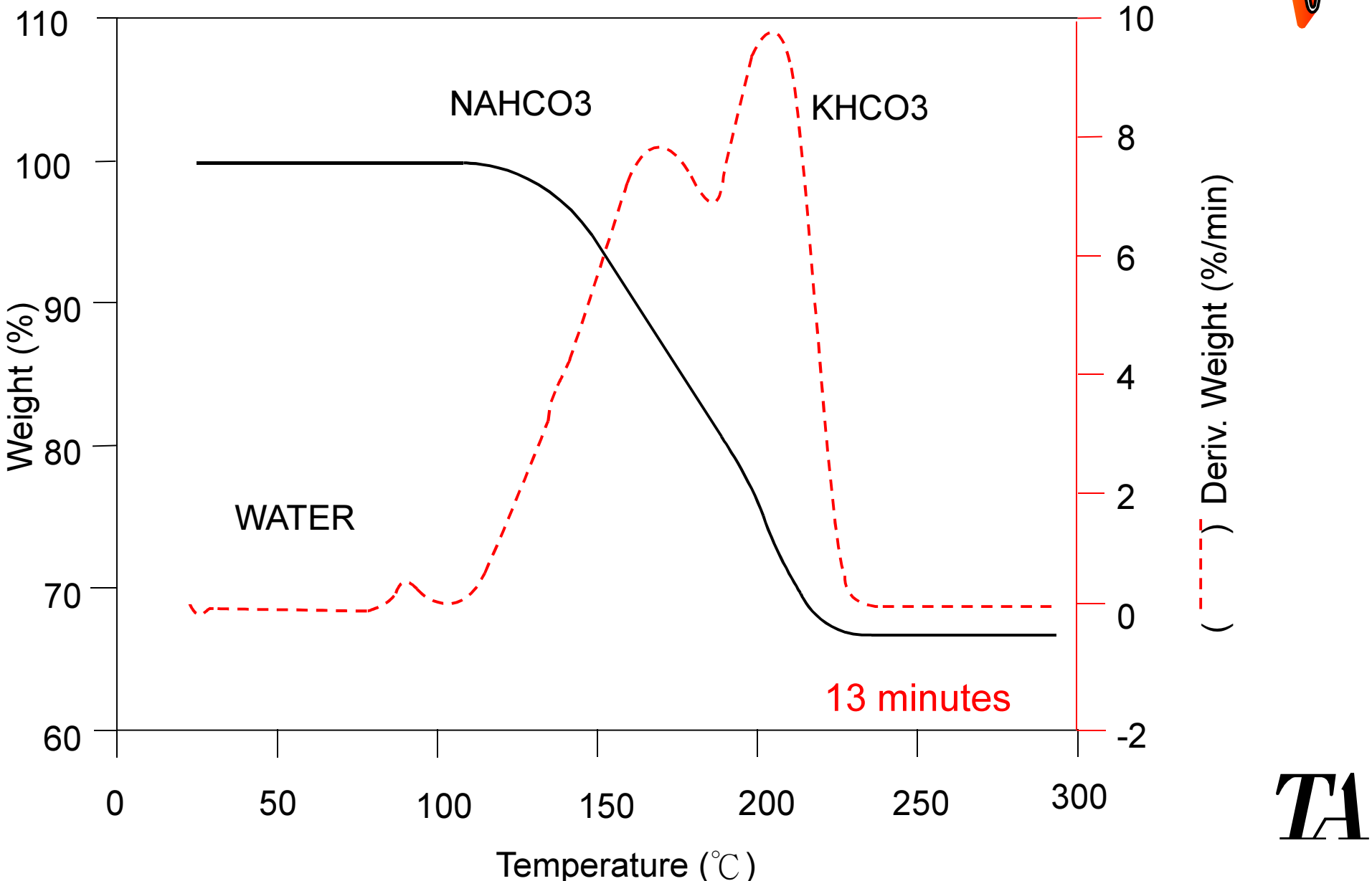
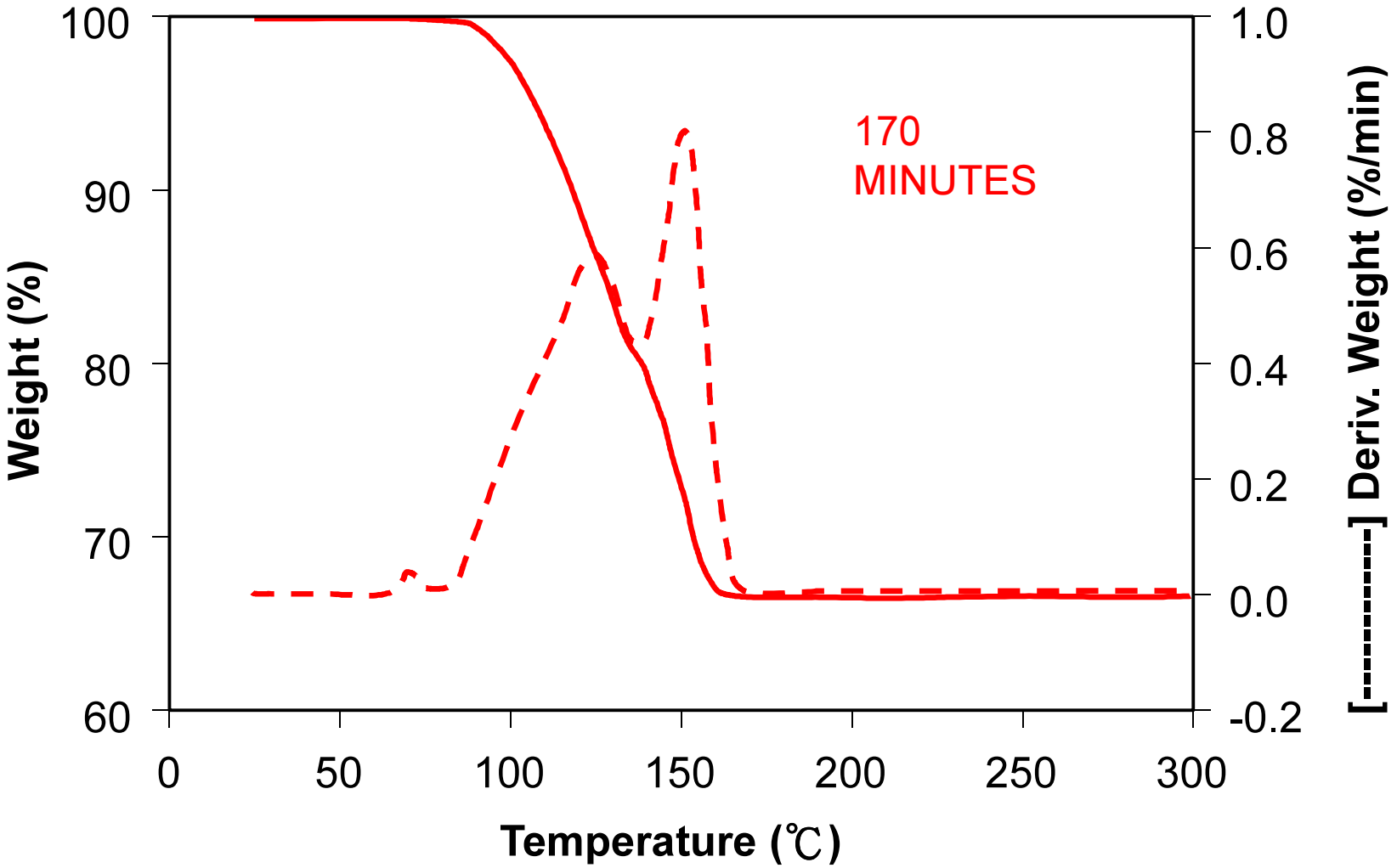
TGA: 分離兩種碳酸鹽類 - Conventional TGA at 20℃/min.



TGA:分離兩種碳酸鹽類 - Conventional TGA at 1℃/min.



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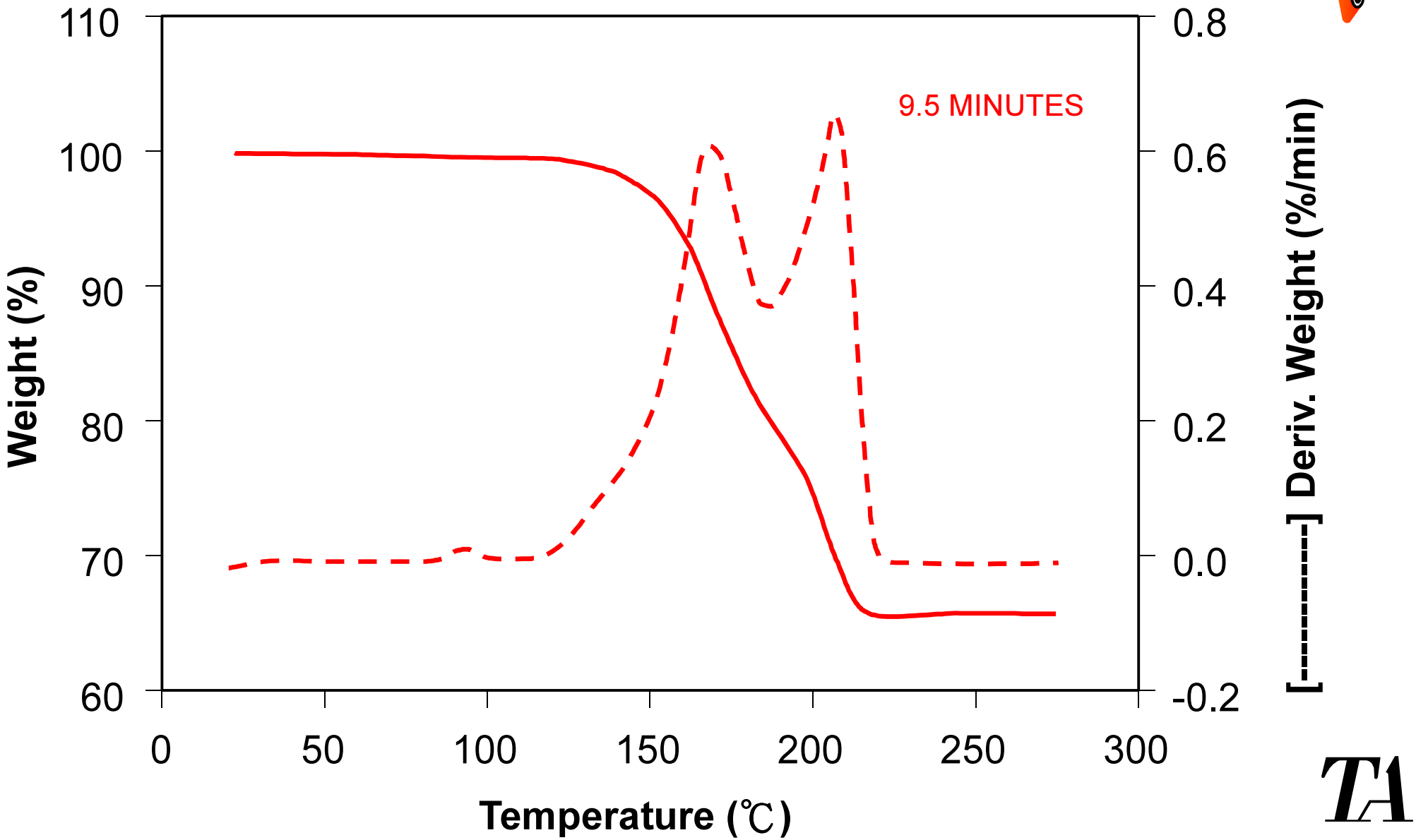
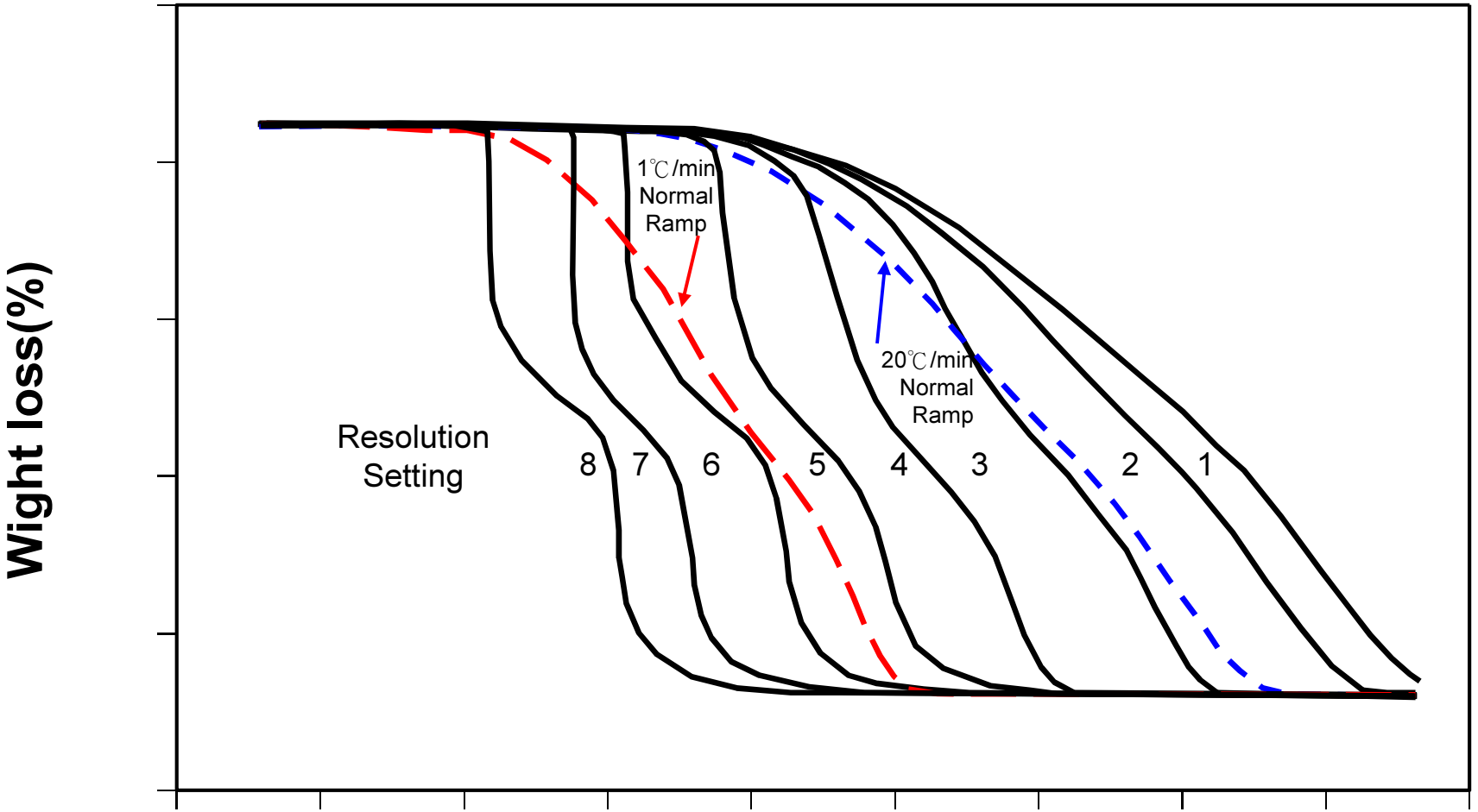
TGA:分離兩種碳酸鹽類 –
Hi-Res TGA (Dynamic Rate)



Hi-Res TGA (Dynamic Rate)比較解析度1-8

樣品:碳酸氫鉀與碳酸氫鈉各半(Air Purge)



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