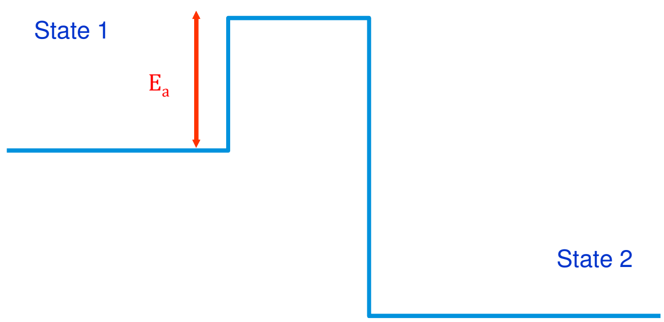
Kinetic Analysis

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



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Introduction: Decomposition Kinetics

Thermal decomposition kinetics can be represented by the following generic kinetic equation:

$$\frac{d\alpha}{dt} = f(\alpha)k(T), \text{ where:}$$

α - conversion fraction (normalized weight on a TGA curve)

$\frac{d\alpha}{dt}$ - rate of conversion (rate of weight loss on a TGA curve)

$f(\alpha)$ - rate function dependant upon conversion fraction

$k(T)$ - rate function dependant upon temperature

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Introduction: Decomposition Kinetics

Almost without exception this is taken to be the Arrhenius equation, so:

$$\frac{d\alpha}{dt} = f(\alpha)Ze^{-E_a/RT}$$

α - conversion fraction (normalized weight on a TGA curve)

$\frac{d\alpha}{dt}$ - rate of conversion (rate of weight loss on a TGA curve)

$f(\alpha)$ - rate function dependant upon conversion fraction

Z - pre-exponent factor

E_a - activation energy

R - gas constant

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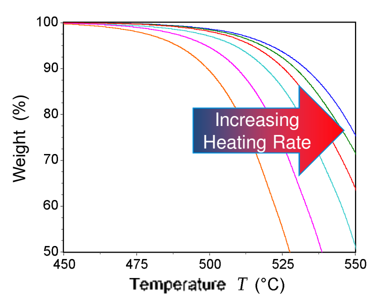
Decomposition Kinetics by TGA

- Traditional methods include isothermal and constant heating rate techniques.
- ASTM E1641
  - Based on method of Flynn and Wall – Polymer Letters, 19, 323 (1966). Requires collection of multiple curves at multiple heating rates.



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Introduction: Decomposition Kinetics

Heating rate:

$$\beta = \frac{dT}{dt}$$

Thus

$$\frac{d\alpha}{dT} = \frac{Z}{\beta} e^{-E_a/RT} f(\alpha)$$

For a single heating rate:

$$ln\left(\frac{d\alpha}{dT}\frac{\beta}{f(\alpha)}\right) = ln(Z) - E_a/RT$$

At a select α conversion

$$\left[\frac{d\ ln\ (d\ \alpha/dt)}{dT^{-1}}\right]_{\alpha} = -E_a/R$$

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Recipe for Activation Energy by ASTM E1641. Polymer Decomposition

- Run TGA experiment on polymer at 4-6 different heating rates
- Obtain a temperature at an isoconversional point – for example 10% weight loss for each heating rate
- Plot the ln of the heating rate (β) versus **1/T** (temperature units must be in Kelvin)
- Slope of the line is ($-E_a/R$). Multiply the slope of the line by –(8.314×10^{-3}) to obtain the activation energy in kJ/mol.

TA Instruments Application Note TA251
Blaine, R. L. and Hahn, B. K., "Obtaining Kinetic Parameters by Modulated Thermogravimetry," J. Therm. Anal., Vol. 54, 1998, p. 694.

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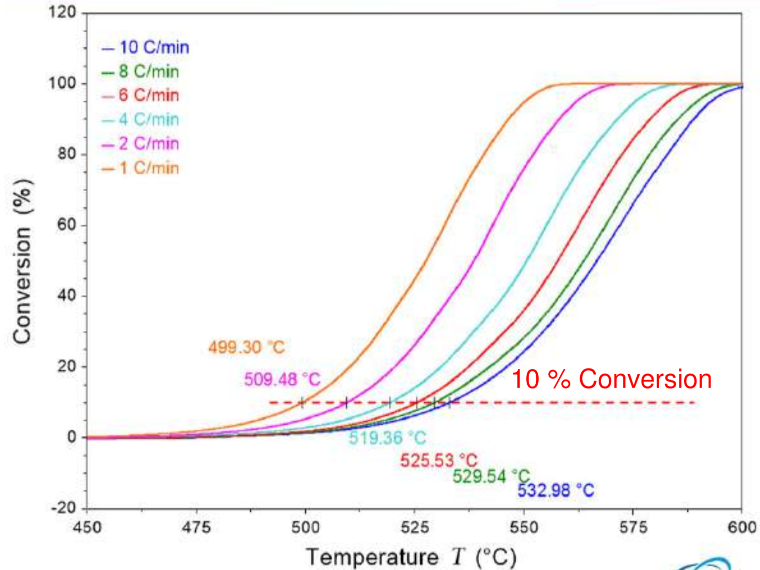
PTFE Decomposition by ASTM E1641



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PTFE Decomposition by ASTM E1641

Heating Rate (β) ℃ / min	ln (β)	Temperature at 10 % Weight Loss (℃)	1/T (K)
10	2.303	532.98	0.001240
8	2.079	529.54	0.001246
6	1.792	525.53	0.001252
4	1.386	519.36	0.001262
2	0.693	509.48	0.001278
1	0.000	499.30	0.001295
Intercept of ln(β) vs 1/T	55.25		
Slope of ln(β) vs 1/T	-42685		
Activation Energy Ea (kJ/mol)	354.9		

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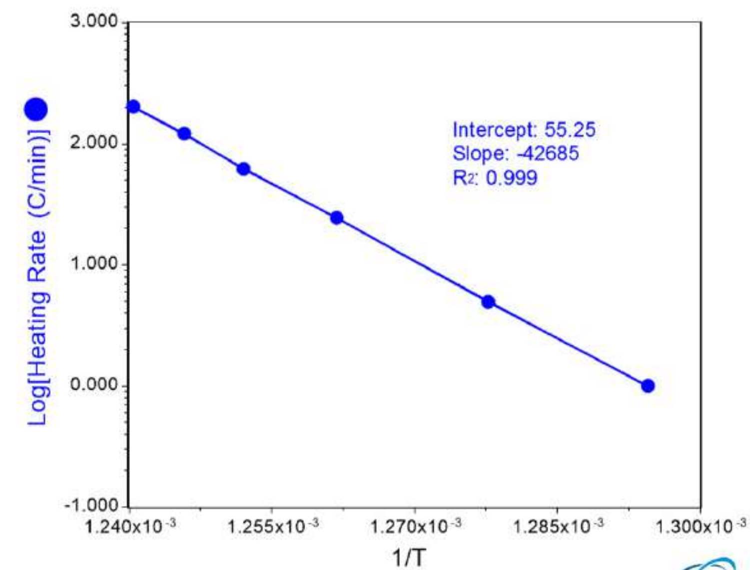
PTFE Decomposition by ASTM E1641



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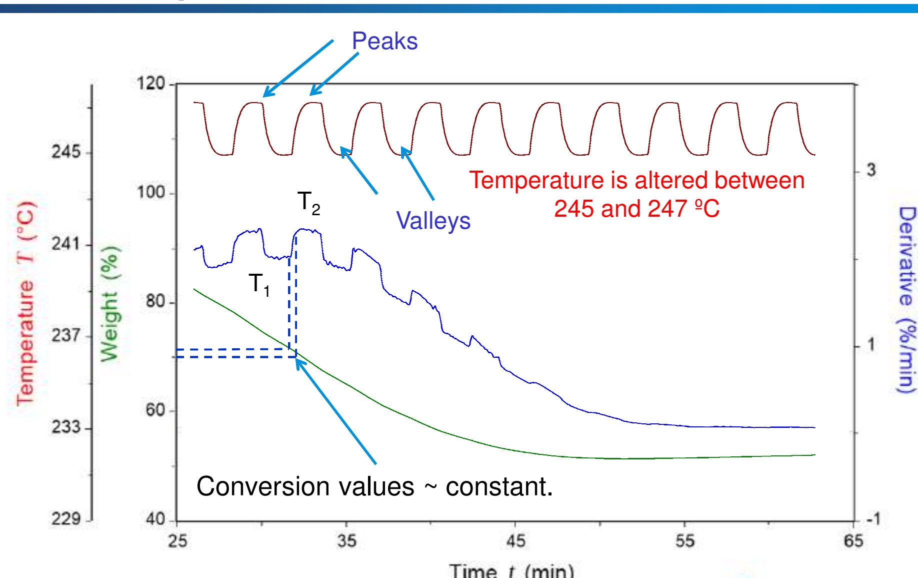
Decomposition Kinetics



94 *J.H. Flynn, Thermal Analysis, R.F. Schwenker and P.D. Garn, Eds., Academic Press, Budapest, **2**, 1969, 1111-1123 TAINSTRUMENTS.COM

Solution for E_a Assuming Constant Conversion

From the general rate equation at each temperature T_1 and T_2 :

$(d\alpha/dt)_{T_1} = f(\alpha)Ze^{-E_a/RT_1}$ and $(d\alpha/dt)_{T_2} = f(\alpha)Ze^{-E_a/RT_2}$

Assuming f(α) is the same (constant conversion values), take the ratio and solve for E_a:

$$(d\alpha_1/d\alpha_2) = e^{-E_a/R(1/T_1-1/T_2)}$$

$$E_a = \frac{R\ ln(d\alpha_1/d\alpha_2)}{(1/T_2 - 1/T_1)}$$

This method is called the Factor Jump Method.

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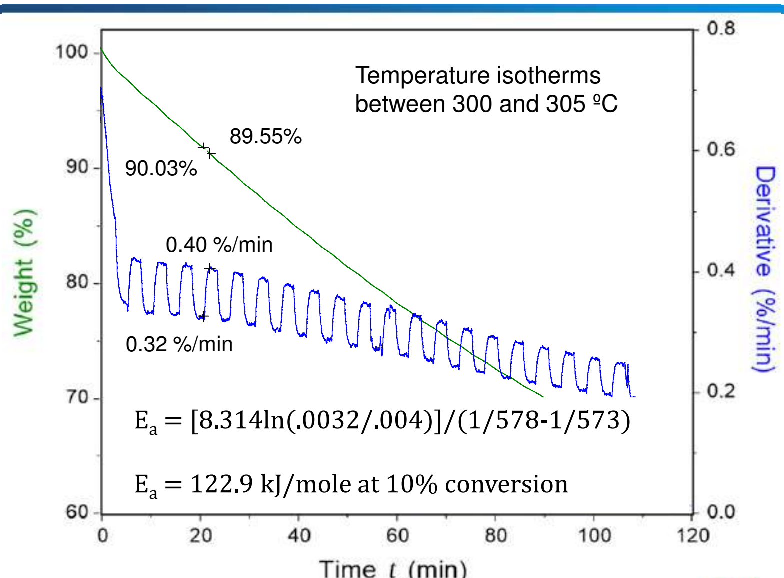
PS: Temperature Step Change Experiment



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Proceed from Step Change to Sinusoidal Change in Temperature

For sinusoidal temperature:

$$T = T_i + T_a \sin(\omega t)$$

T_i - isothermal temperature
T_a - modulation amplitude

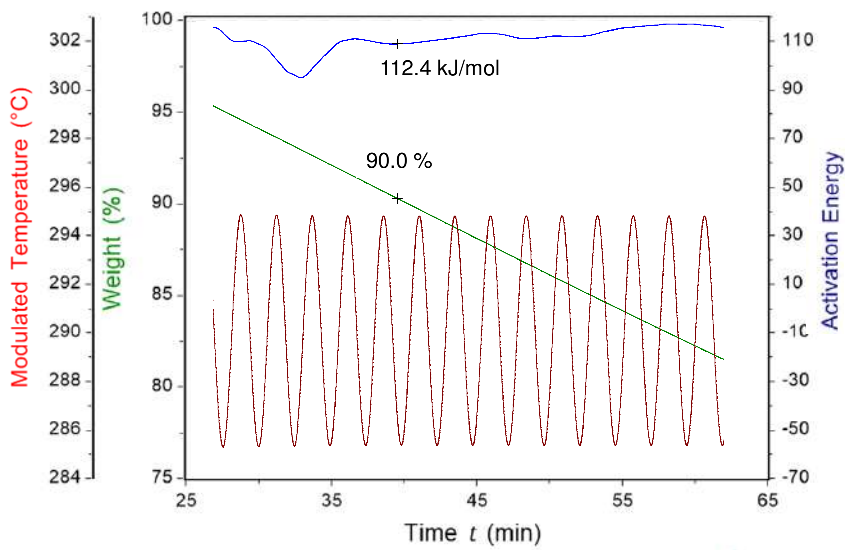
For peak and valley temperatures:

$$T_P = T_i + T_a \quad T_V = T_i - T_a$$

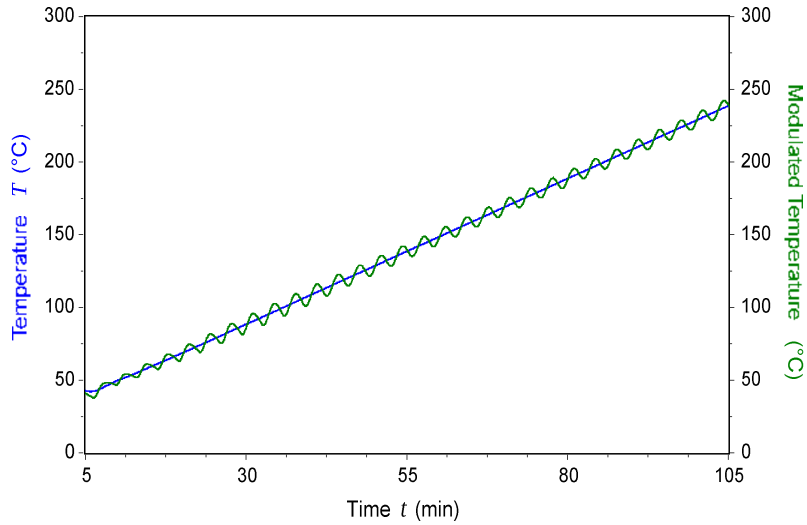
After substitution and rearrangement

$$E_a = R\, ln(d\alpha_1/d\alpha_2) \frac{(T_i^2 - T_a^2)}{2T_a}$$

PS: Quasi-Isothermal Experiment @ 290°C



Extension to Modulated Temperature Ramping



Advantages of MTGA™

- One run needed to obtain activation energy.
- Activation energy is a signal in the data file.
- Comparable with Flynn-Wall method for calculated Ea with the benefit of the reduced time.
- Method works under quasi-isothermal or ramping conditions.
- Activation energy is obtained as a continuous curve and so can be manipulated numerous ways. For example, it can be plotted as a function of conversion.
- Can be combined with Hi-Res™ to speed up experiments and more accurately handle multiple weight loss events.

Setting Up MTGA™ Experiment

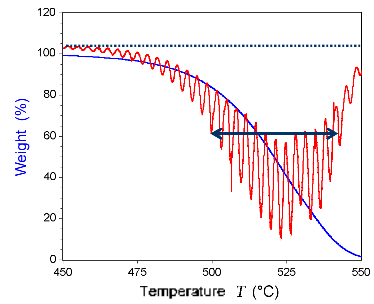
- Three parameters must be defined:
  - heating rate
  - modulation period
  - modulation amplitude
- What is the effect of these parameters on the data? What are the optimum values?

General Guidelines

- Higher amplitudes improve signal/noise and increase precision, but require longer periods to make sure the sample is remaining in equilibrium.
- Longer periods ensure equilibrium, but require slower ramp rates so the minimum 5 cycles per transition can be obtained.
- A scouting run at 10 °C/min is useful to determine width of transitions.
- Range of consistent results:
  - Period = 200-300 s (practical range: 100 – 500s)
  - Amplitude = 3-5 °C (practical range: 1 – 10 °C)
  - Ramp Rate = 1 °C/min (practical range: 0.5 – 2 °C/min)

MTGA™ – Typical Values

- Modulation period – 200 seconds.
- Amplitude – 4-5°C.
- Heating rate – 1 to 2 °C/min.
- Plot derivative of weight loss and calculate the width at half height of the derivative weight loss peak.
  - Need at least 5 modulation cycles across this region.



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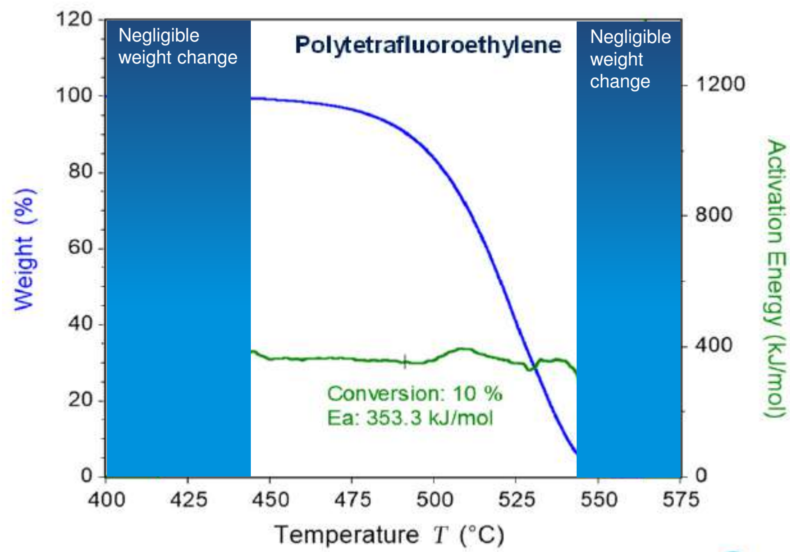
PTFE: Modulated Ramping Experiment



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MTGA Precision: PTFE

Conversion (%)	#1	#2	#3	#4	#5	Average (kJ/mol)	Standard Deviation (kJ/mol)	Relative Stnd Dev (%)
1	333.0	342.4	335.8	345.9	340	339.42	5.14	1.51
2	334.9	329.3	330.8	330.8	327.7	330.70	2.67	0.81
5	319.3	322.6	323.7	319.7	323.1	321.68	2.03	0.63
10	313.1	314	311.9	316.2	318.6	314.76	2.66	0.85

Repeatability = 2.8X Standard Deviation

C.G. Slough The Accuracy, Repeatability, and Reproducibility of Activation Energy Values Measured by Modulated Thermogravimetry. J of Testing and Evaluation V. 42, N. 6 (2014)

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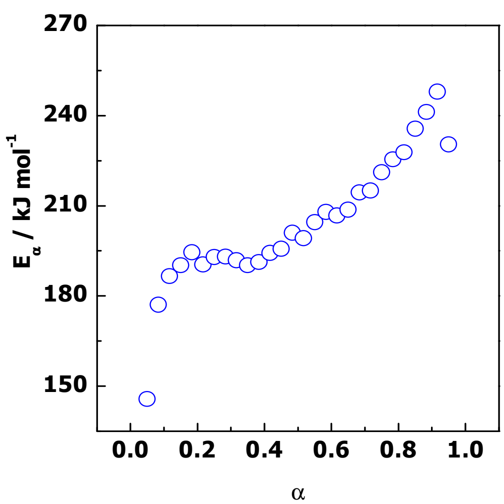
Decomposition of PP by Flynn-Wall Method



Atmosphere	Ea* / kJ mol^{-1}
N_2	244
N_2	216
N_2	214
N_2	160
N_2	115 – 200
N_2	130 – 200
N_2	230
Vacuum	257
Ar	98, 328

* Data obtained from multiple publications

Thermal Analysis of Polymers: Fundamentals and Applications. Chapter 3. Edited by by J. Menczel and R. B. Prime (2009)

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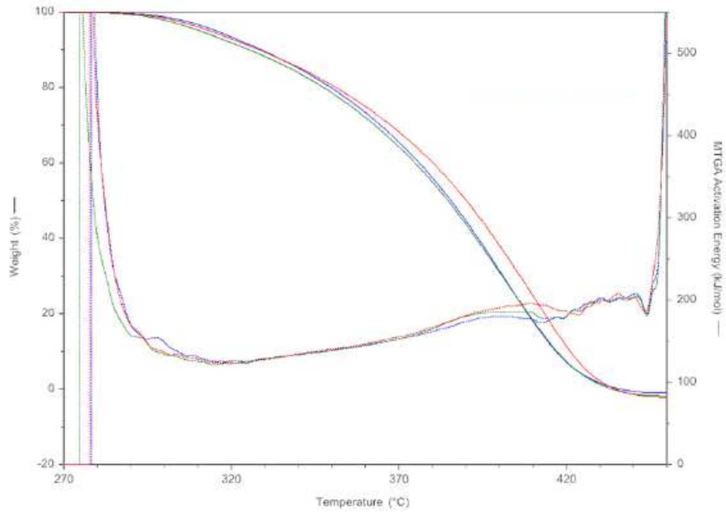
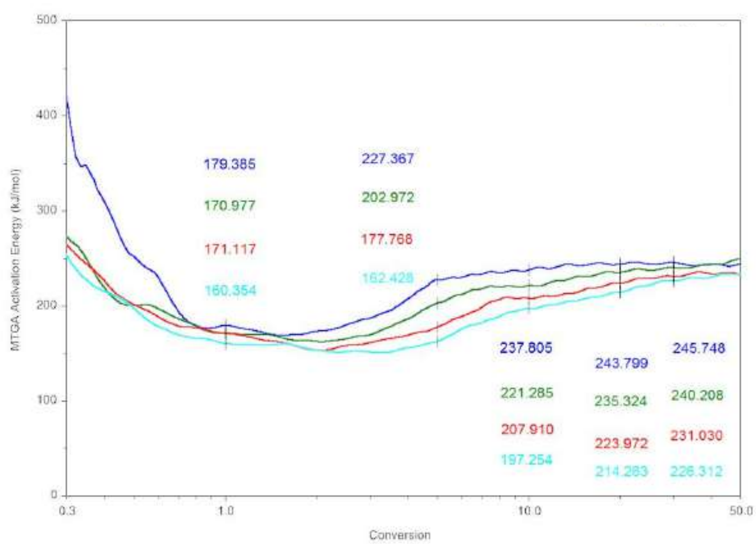
PP: Modulated Ramping Experiment

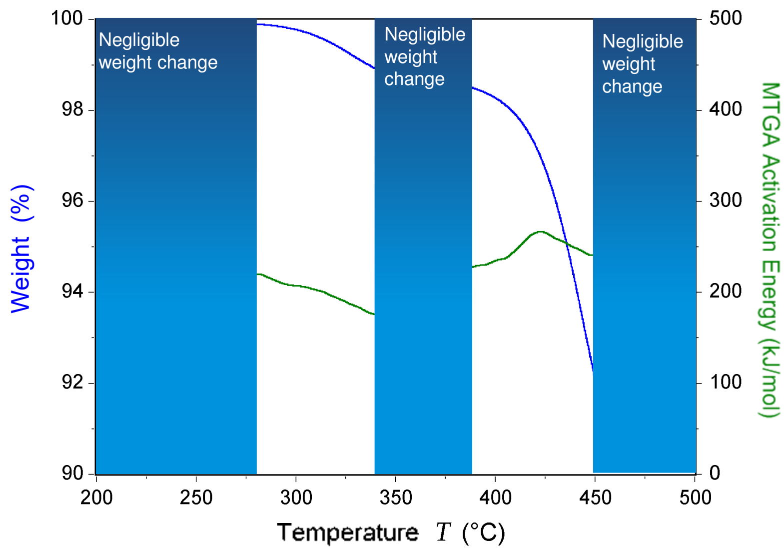


Literature: E_a = 232 ± 27 kJ/mol (12 % standard deviation)
This study: E_a = 200 ± 19 kJ/mol (standard deviation 9.5 %)

PP: Modulated Ramping Experiment



MTGA™ - 40% EVA



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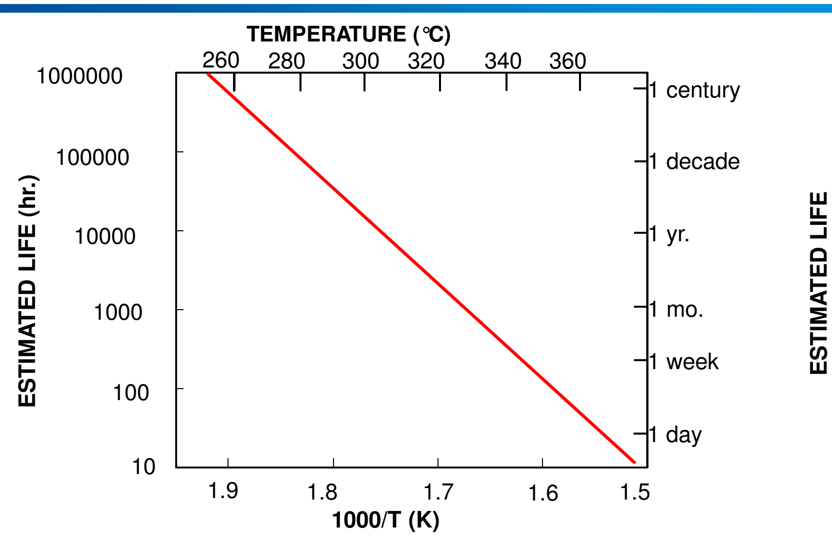
TGA Kinetics - Estimated Lifetime



TA Instruments Application Note TA125
Estimation of Polymer Lifetime by TGA Decomposition Kinetics

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Concluding remarks

- ASTM committee E37 on Thermal Methods is currently developing a standard for kinetics by either the Factor Jump method or MTGA
- Modulated TGA is a comprehensive method to obtain an activation energy of decomposition in a single run
- Data by MTGA is comparable to that of the Flynn-Wall method
- It can be a powerful tool to compare energetics of similar systems (for example, composites of close compositions, polymers with additives)

A Practical Approach to Thermal Analysis Thermogravimetric Analysis

- An Overview of Thermogravimetric Analysis (TGA)
- Methods to improve resolution of complex TGA weight loss profiles – An introduction to HiRes™ TGA and Stepwise Isothermal TGA
- **Determining Decomposition Activation Energy by Modulated TGA**
- Evolve Gas Analysis – An Introduction to TGA-Mass Spectrometry

Modulated TGA Technology

A New Approach for Obtaining Kinetic Parameters

Decomposition Kinetics

- Based on Arrhenius Equation
- Many models in the literature
- Includes isothermal and constant heating rate methods.
- TA Specialty Library – will model lifetimes based on method of Toop
- 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.
- Modulated TGA requires only one run instead of multiple at different heating rates.

Simple Polymer Decomposition

$$\phi = A\exp\left(\frac{-\Delta E}{RT}\right)$$

$$\ln\phi = A - \left(\frac{\Delta E}{RT}\right)$$

where

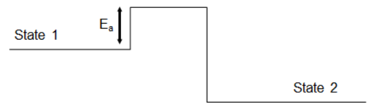
ϕ = heating rate (°C / min)

A = pre - exponential

ΔE = Activation Energy (kJ / mol)

T = temperature (K)

R = Universal Gas Constant



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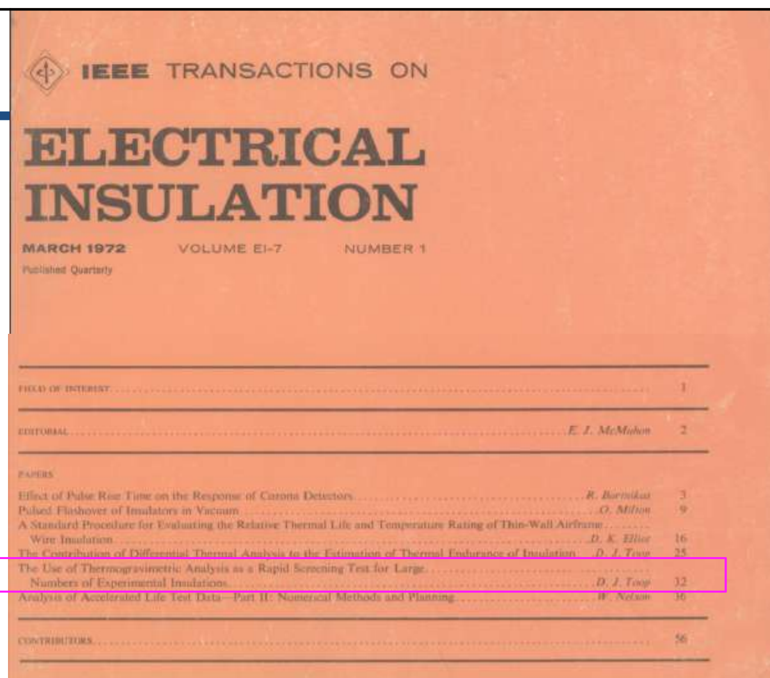

IEEE TRANSACTIONS ON

ELECTRICAL INSULATION

MARCH 1972 VOLUME EI-7 NUMBER 1

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FIELD OF INTEREST 1

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A Standard Procedure for Evaluating the Relative Thermal Life and Temperature Rating of Thin-Wall Airframe Wire Insulation ..... D. K. Elliot 16
The Contribution of Differential Thermal Analysis to the Estimation of Thermal Endurance of Insulation ..... D. J. Toop 25
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CONTRIBUTORS 56

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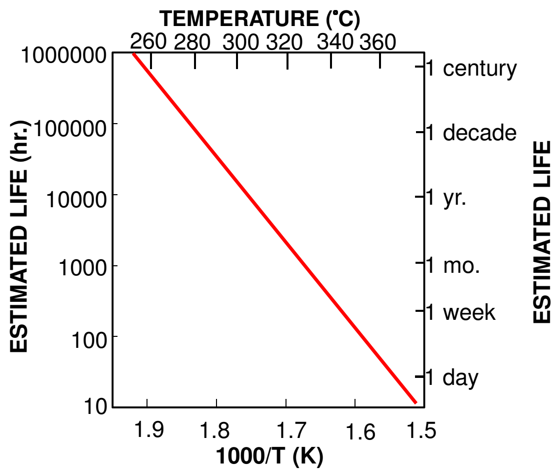
Lifetimes Method by Toop



$$T_f = \frac{E/R}{\ln t_f - \ln\left[\frac{E}{\beta R} \cdot P(X_f)\right]}$$

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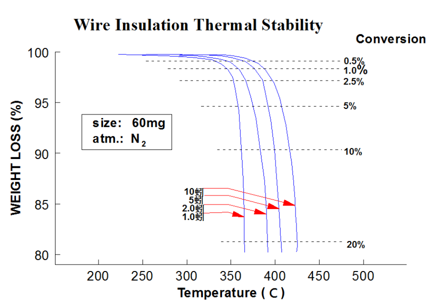
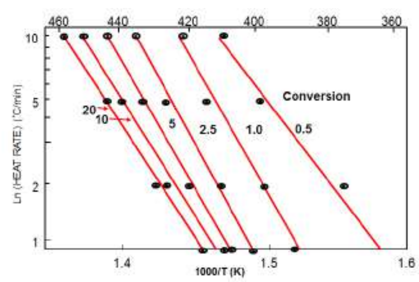
問題：如何決定正確的ΔE？





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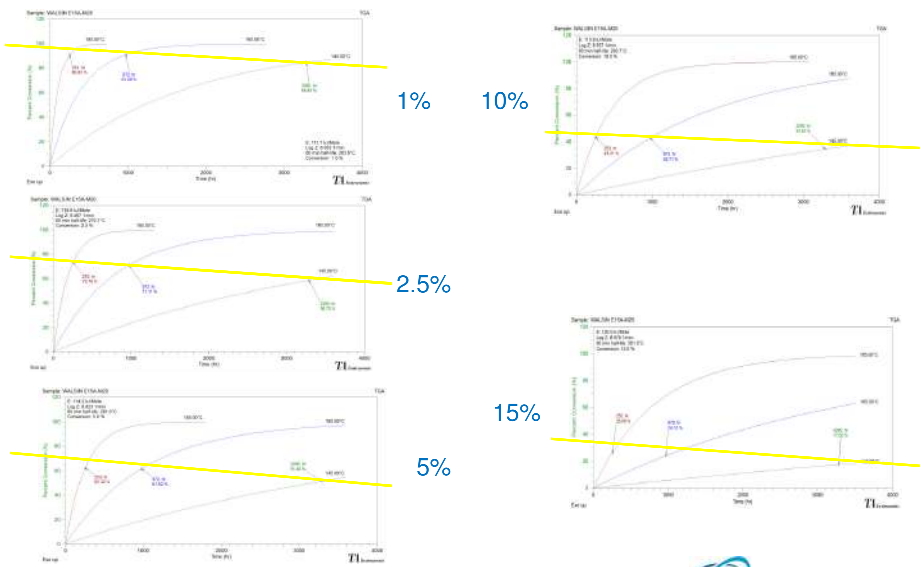
實例 : Wire Insulation Thermal Stability



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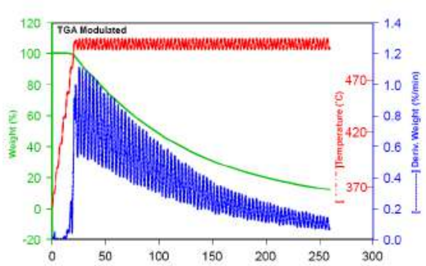
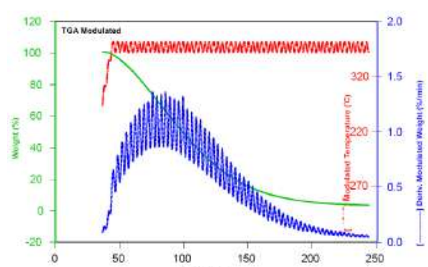
Quasi-Isothermal MTGA :定溫下分解活化能不必然固定

PTFE



PS



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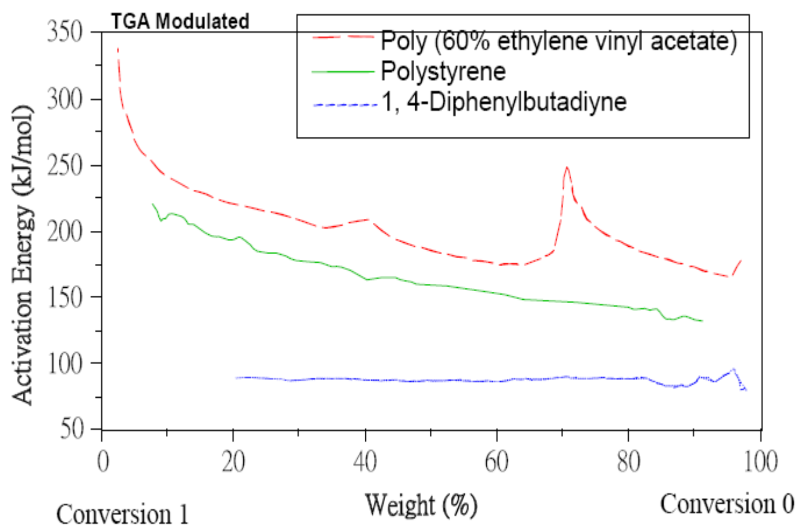
MTGA：分解活化能在過程中不必然是固定的



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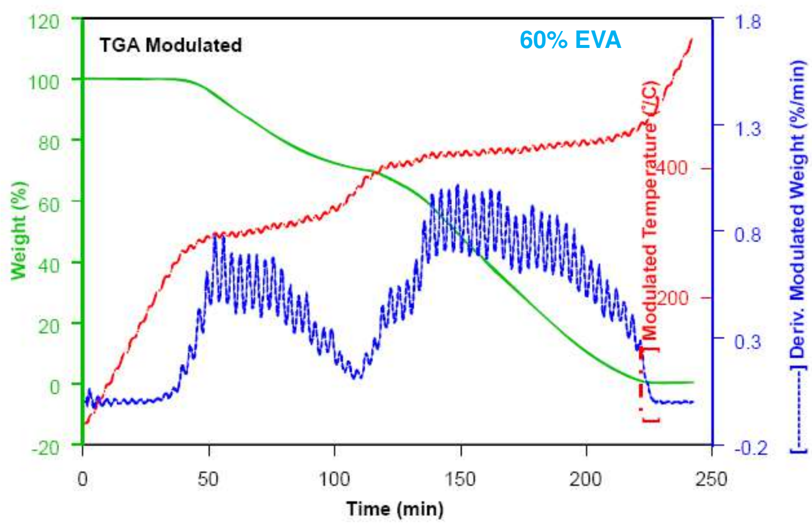
MTGA：可結合Hi-Res TGA (Dynamic Heating Rate)



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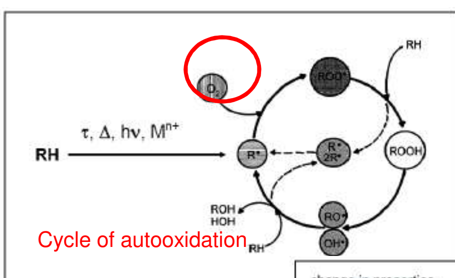
MTGA案例研討

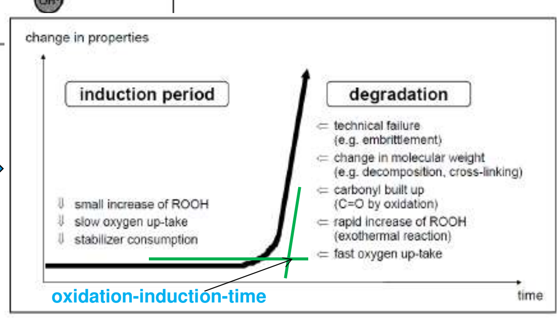
電線電纜熱壽命預測

材料之熱氧劣化

例子：壓延加工，室內或機箱內使用



Change of properties after the induction period.



失效品質項目之選擇與加速老化實驗

Criteria	PE, PP	PS	PMMA	PVC	POM	PA	PET	PC
embrittlement	XX					X		
yellowing	X	XX	X	XX	X	X		XX
tensile strength and elongation	X	X	X	X	X	XX	XX	
impact strength	X	XX	XX	X	X	X	X	X
bending strength	X	X						
intrinsic viscosity						X		X
loss in weight					XX			

XX = main criterion, X = auxiliary criterion

$$\log t_e = a + b \times \left(\frac{1}{T}\right)$$

te = time up to the appearance of a defined property change in the material
- e.g. half-time up to 50% reduction of a property (tensile strength and elongation, ...)
- e.g. embrittlement time by oven ageing or oxidation induction time by DSC at a given temperature

T = absolute temperature in Kelvin
a = axis intercept
b = angular coefficient

Accelerated oven ageing

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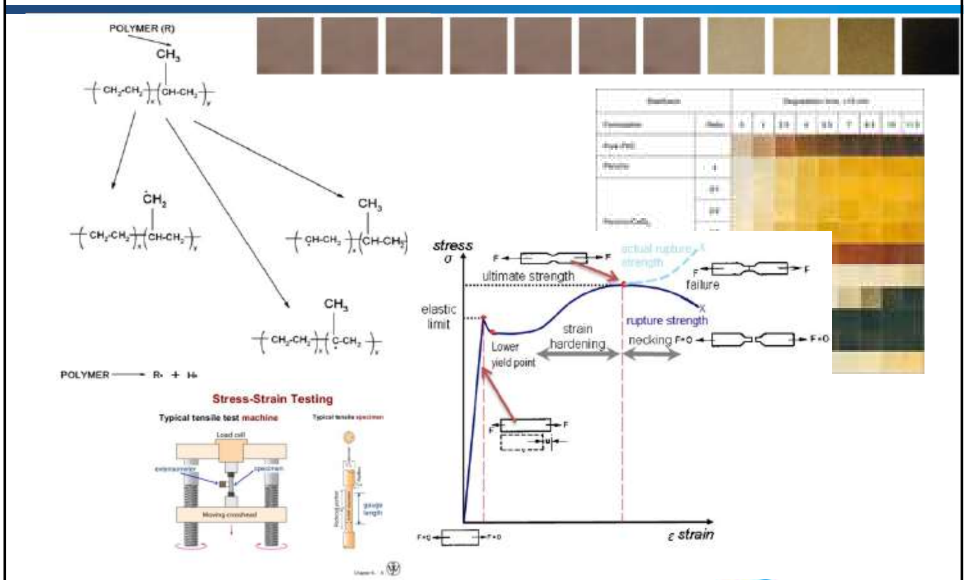
Accelerated oven ageing

Still spend too much time!



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由已知案例，尋求更快速的解決方案

accelerated environmental conditions

SAFETY AGENCY STANDARDS

- UL 62
- UL 1581

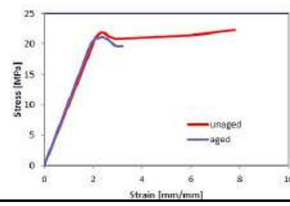
4.5.11 Thermal Stability

A minimum of three tensile specimens shall be prepared in accordance with ASTM D 3032. The specimens shall be aged in an air-circulating oven for the time and temperature specified in the applicable specification sheet. Upon removal from the aging oven, the specimens shall be conditioned at 20 ± 5℃ for a minimum of four hours, and then tested for elongation and tensile strength per 4.5.7.

→ 120C > 20,000hr

AIR-OVEN AGING

Specimen Rubdown Machine



185C-253hr
165C-972hr
145C, 3287hr

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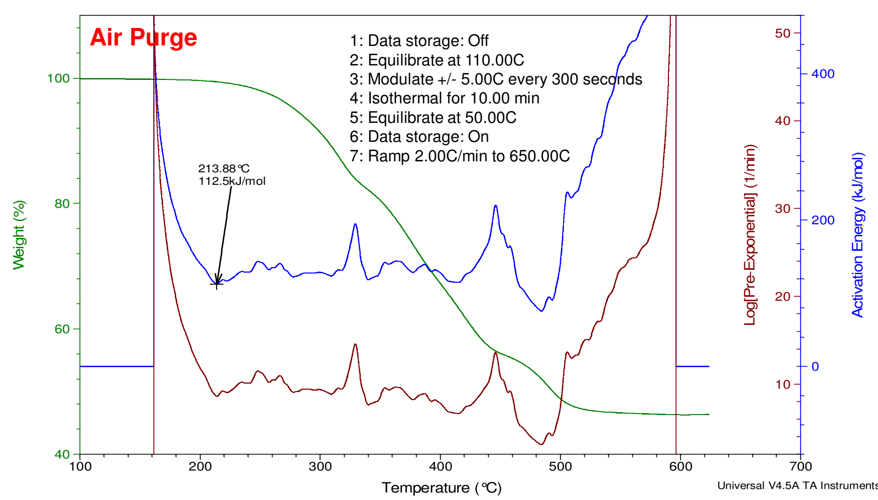
利用 Modulated TGA 解答

Sample: [redacted]
Size: 52.5760 mg
Method: Modulated - Standard
Comment: 185C-253hr, 165C-972hr, 145C-3287hr

TGA

File: D:...\[redacted]
Operator: Rico
Run Date: 30-Jun-2015 18:43
Instrument: TGA Q500 V20.13 Build 39



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如何由活化能預測失效時間

$$\ln t_f = \frac{E}{RT_f} + \ln\left[\frac{E}{\beta R} \bullet P(X_f)\right]$$

Where:

t_f	= Estimated Time to Failure (min)
E	= Activation Energy (J/mol)
T_f	= Failure Temperature (K)
R	= Gas Constant (8.134 J/mol K)
$P(X_f)$	= A function whose values depend on E at the failure temperature.
T_c	= Temperature for 5% Loss at β (K)
β	= Heating Rate (°C/min)

This value is then used to select a value for log P(Xf) from the numerical integration table given in Toop.s paper. The numerical value for P(Xf) can then be calculated by taking the antilogarithm.

$$-\log P(x') = -\log\left[\frac{1}{x'e^{x'}} - \int_{x'}^{\infty} \frac{dx}{xe^{x}}\right]$$

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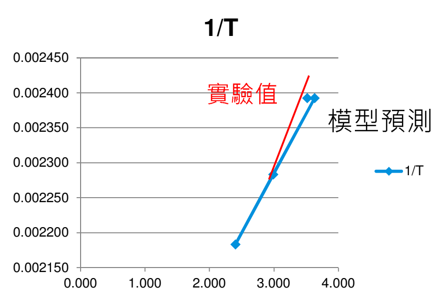
如何由活化能預測失效時間

已知結果 → 185C-253hr, 165C-972hr, 145C, 3287hr

T,C	T,K	te,hr	log(te)	1/T
145	418	3287	3.517	0.002392
145	418	4275	3.631	0.002392
165	438	972	2.988	0.002283
185	458	253	2.403	0.002183



Note : 120C > 20,000hr

t_f	hr	t_f	min	$\ln t_f$	$P(X_f)$	Ea/RT_f	$Ea/\beta R$	β	$C\cdot min^{-1}$	Ea	$kJ\cdot mol^{-1}$	R	$J\cdot K^{-1}\cdot mol^{-1}$	T_f	K	T_f	C
253		15180		9.63	3.32E-13	29.543	6765.320	2		112.5		8.314		458		185	
975		58497		10.98	3.32E-13	30.892	6765.320	2		112.5		8.314		438		165	
4275		256481		12.45	3.32E-13	32.370	6765.320	2		112.5		8.314		418		145	
33511		2010643		14.51	3.32E-13	34.429	6765.320	2		112.5		8.314		393		120	

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PART IV
Evolved Gas Analysis: Introduction to TGA/MS

Evolved Gas Analysis

- Evolved Gas Analyses (EGA) are exciting techniques for today's analytical chemist.
- Sometimes referred to as 'hyphenated' techniques, they commonly combine TGA with FTIR (TGA/FTIR), mass spectrometry (TGA/MS) and gas chromatography and mass spectrometry (TGA/GC-MS).
- For TGA/MS, the sample is introduced by vaporizing in the TGA and introducing the sample gas into the mass spectrometer via a heated stainless steel capillary to the MS inlet orifice or molecular leak.

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Examples of TGA/MS Data Presentation

- Representative Barchart Mass Spectral Scan
  - Mass Spectral Scan 1 – 200 AMU (m/z)
- TGA/MS Data Presentation
  - Weight Lose Data vs Temperature
  - Mass Spectral Data vs Temperature

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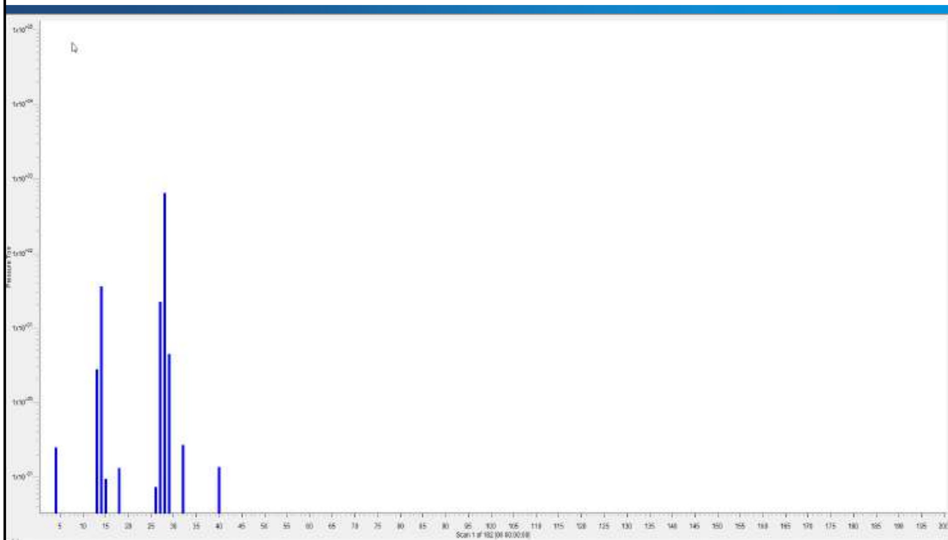
Simplified Bar Chart Display: Background for Polyphenylene Oxide at Start of Experiment



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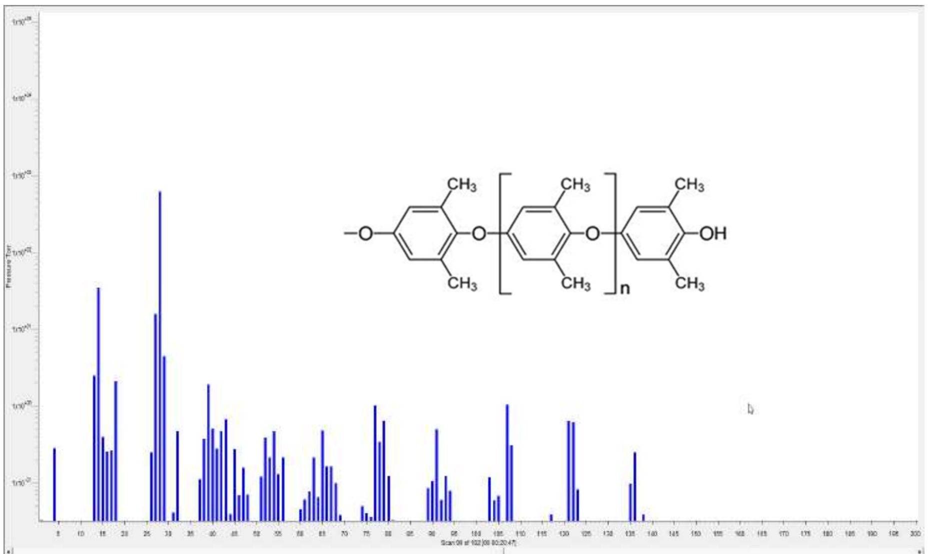
Typical Raw Data: m/z vs Partial Pressure for Polyphenylene Oxide



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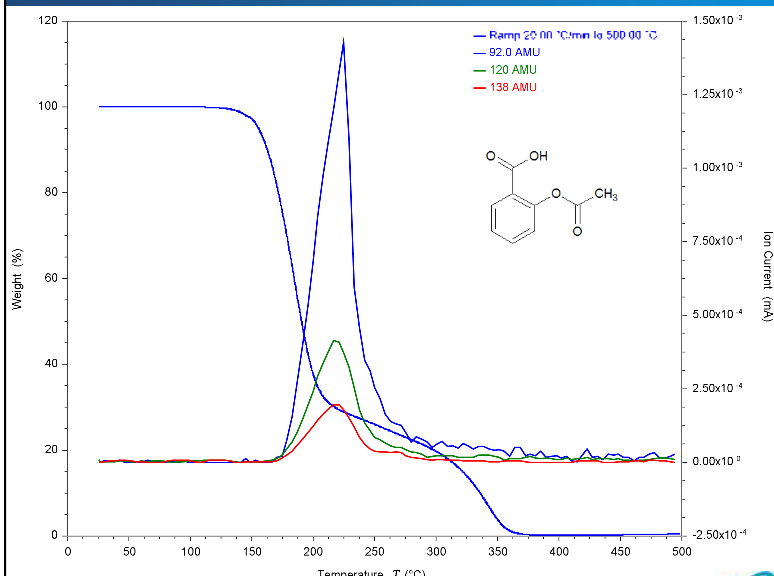
Example: Typical TGA/MS Data Presentation



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The Discovery Mass Spectrometer (DMS)

- Benchtop, unit resolution quadrupole mass spec designed and optimized for evolved gas analysis (EGA)
- Quadrupole detection system includes…
  - a closed ion source
  - a quadrupole mass filter assembly
  - dual detector system (Faraday and Secondary Electron Multiplier)

  …ensuring excellent sensitivity from ppb to percent concentrations

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DMS System Overview

Heated SS Capillary Inlet Custom Interfaced to TGA Exhaust
- Inlet consists of a SS capillary tube with a 300 ℃ heater assembly

Dry Vacuum Pumps
- Hydrocarbon-free vacuum system
- 70 l/s wide range turbo molecular pump
- 4 headed, higher compression, diaphragm backing pump

Integrated Pressure Gauge
- Independent pressure measurement
- Trip signal to protect analyzer

EM Thermal trip
- Protects the electron multiplier from damage if operated above 80 ˚C

24 Volt operation
- Mains supply independent
- Longer diaphragm lifetime on the pump, due to cooler operation

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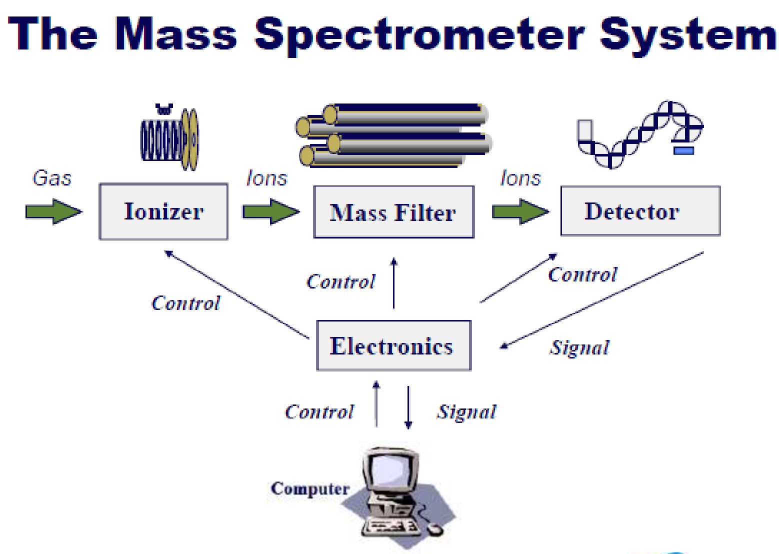
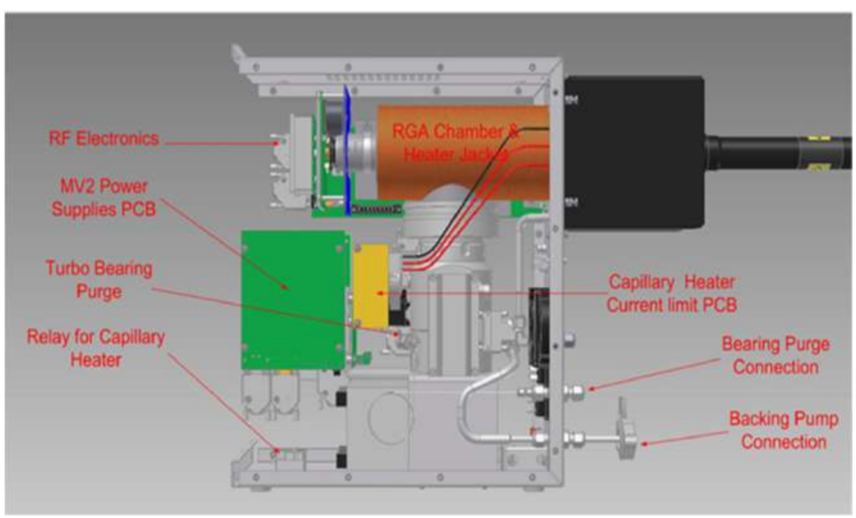
A Quadrupole Mass Spectrometer

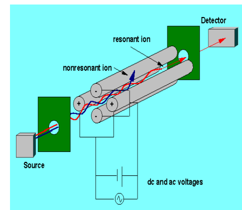


Discovery Mass Spectrometer layout

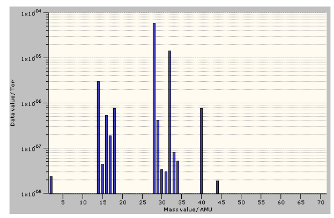


What is a Quadrupole Mass Spectrometer?

A Quadrupole Mass Analyzer



- Ionizes gas molecules and atoms
  - Electron impact knocks off an electron and fragments molecules forming positive ions
- Sorts by the mass/charge (m/z) ratio
- Measures the ion current
- Displays ion current vs. m/z ratio
- When calibrated against inlet pressure - can display partial pressure vs. m/z ratio



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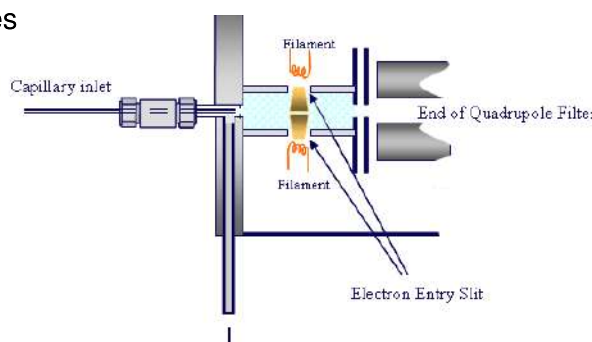
Ion source design

Closed Ion Source

- Significantly improves detection capability compared with open ion source designs
- Eliminates system background interference & maximizes sample peak intensity



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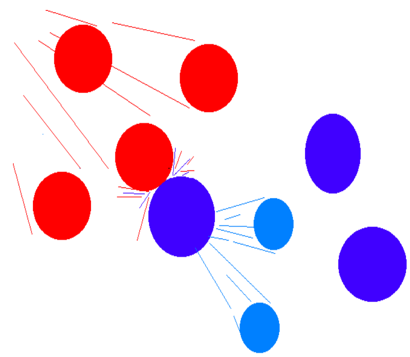
What is a Mass Spectrometer?

Ionization Process

- Electrons are emitted from hot filaments and accelerated toward the source cage via an electrical bias voltage
- The fast moving electrons collide with gas molecules, dislodging electrons, thus ionizing them
- An ion is an atom or group of atoms which have become charged, positively or negatively, through losing or gaining electrons



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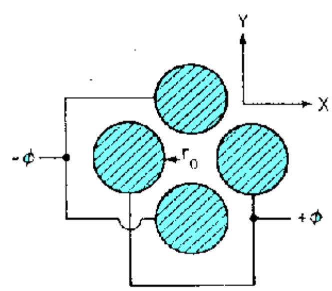
What is a Mass Spectrometer?

The Quadrupole Mass Filter Assembly

- The quadruple array is composed of four precision stainless steel rods
- RF power is applied to all rods with - DC to one pair and +DC to the opposing pair
- This creates a complex electrical field
- By varying the voltage, electrical field can be controller



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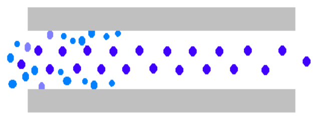
How a Quadrupole Mass Spec Filter Works



- The electrical field is varied by ramping the RF/DC voltages in a 6:1 ratio
- As ramp increases, ions with an appropriate m/z will oscillate in a stable three dimensional trajectory through the poles
- Ions of incorrect m/z will oscillate out of control and collide with the poles

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Ion Detector

- Faraday Cup
  - Ion current collected directly and fed to amplifier
  - Basic sensitivity is measured at the Faraday cup
- Electron Multiplier
  - Continuous Dynode
    - Microchannel Plate
  - Gain– ratio of output with multiplier to Faraday cup current (no multiplier)

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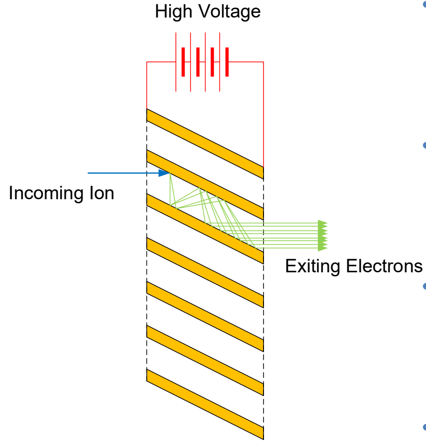
Micro-Channel Plate Electron Multiplier

Active surface: PbO-B_2O_3 glass



High-resistivity surface with a high secondary emission coefficient for electrons

- MCP is a plate made of small glass tubes of approximately 10 micron diameter
- Incoming ions cause emission of electrons from the surface of the tubes
- The ratio between incoming ions and exiting electrons is the multiplier gain
- MCP can give a gain of up to 3000

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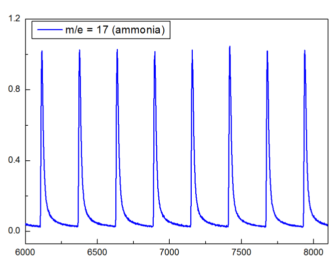
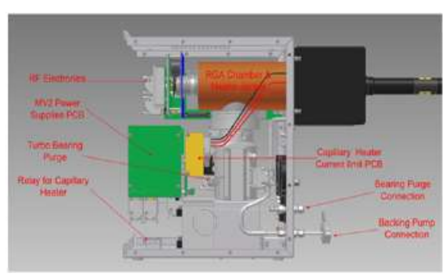
Heater and Inlet design

- Capillary and inlet materials minimize ad-/desorption effects even for reactive and corrosive species
- Consistent heating across the whole inlet and the spectrometer



Reproducibility of short NH3 pulses



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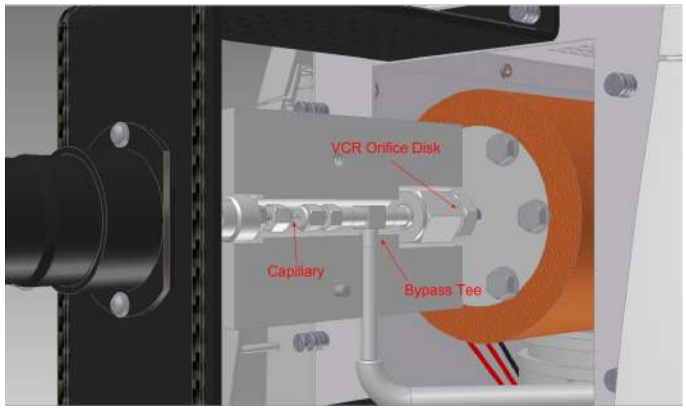
Heater and Inlet design



- Orifice disk is located inside standard VRC coupling
- Capillary is sealed inside the 1/16” Swagelok fitting

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Summary of DMS System

- 1-300 AMU Quadruple Mass Filter
- Compact Overall System Design
- Easy Maintenance
  - Capillary Inlet and Pressure Reduce Orifice
- High Sensitivity Detector
- High Speed Data Collection
- Resistant to Corrosive Gasses
- Integrated Control Via TGA Digital Trigger

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Specific Benefits of Discovery Mass Spectrometer

TA Discovery Mass Spectrometer offers a number of specific advantages:

- Rugged design
- Easy maintenance
- Excellent sensitivity
- Neat sample analysis – no need for solvents
- Ease of use – operation software features simple experimental design
- Automatic synchronization of TGA collection data including support of the TGA autosampler
- Powerful TRIOS software for TGA/MS data analysis
- Worldwide support
- Expert local knowledge

Applications

- Polymers
- Residual Solvent
- Pharmaceuticals
- Biomass
- Soil
- Excellent scouting instrument for GC/MS or HPLC/MS as a first pass for unknown or samples that may be fairly 'dirty'.
- Many others!

TGA/MS: Experiments

- Experiments are called 'recipes'
- Barchart
  - Scan across specified ion range - m/z 1 to m/z 300
  - Typically used as first approach for an unknown compound
- Peak Jump
  - Scan specific ions
  - Example, scan m/z 91, 65, 51, 39 if you are looking for residual toluene

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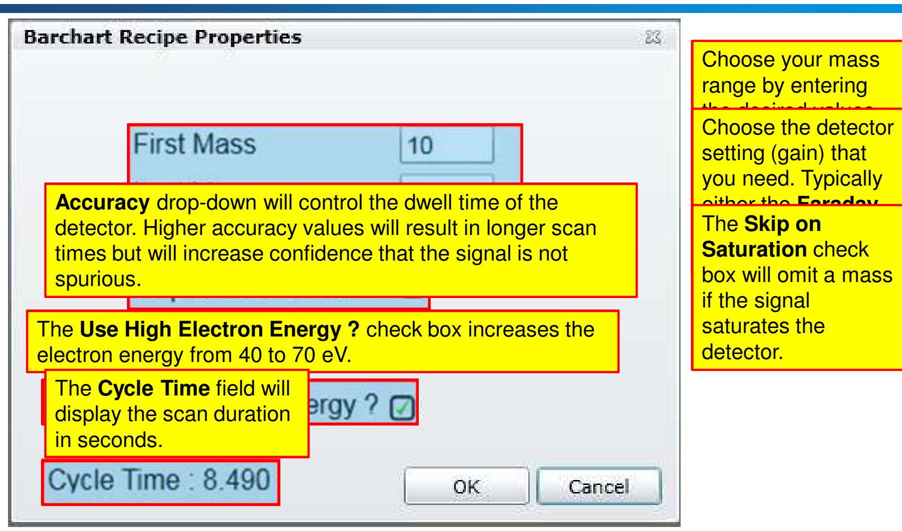
Basic System Operation: Selection of Parameters for Barchart Recipe



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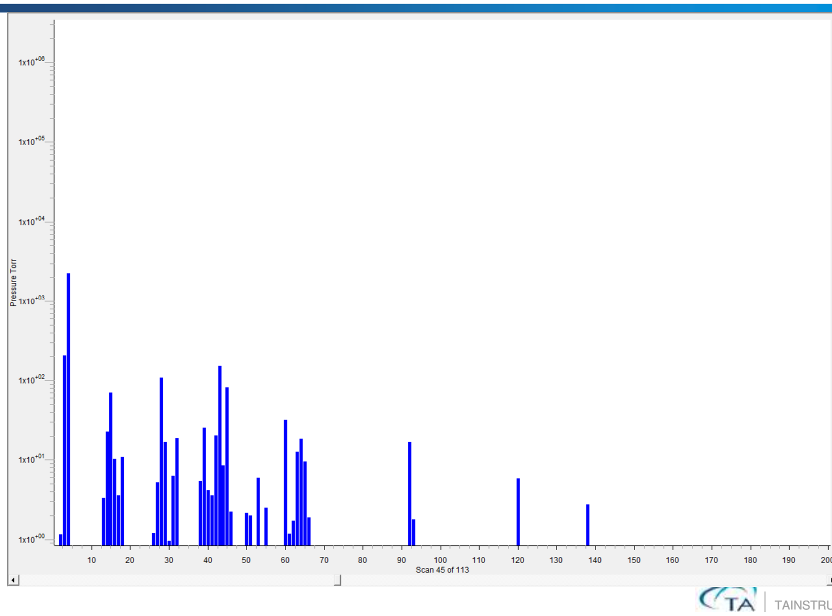
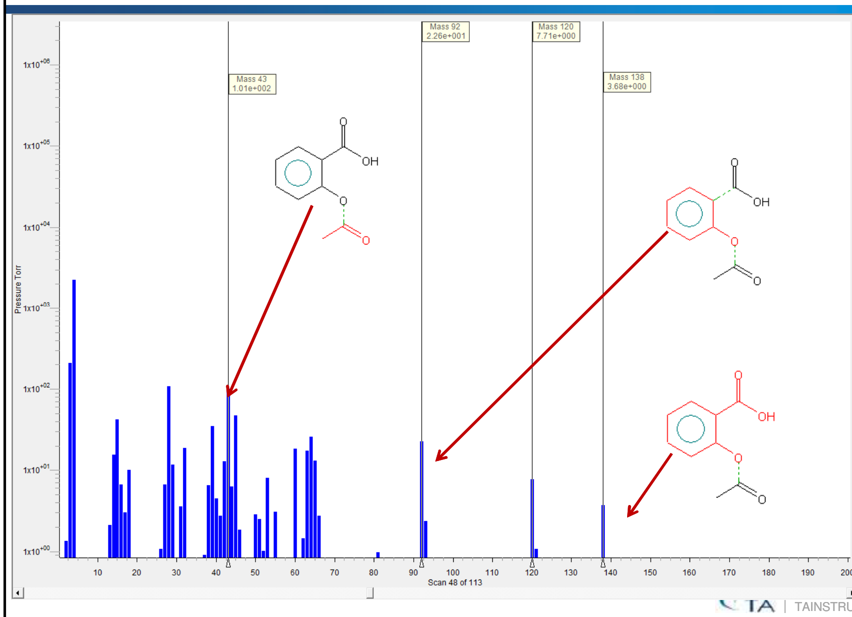
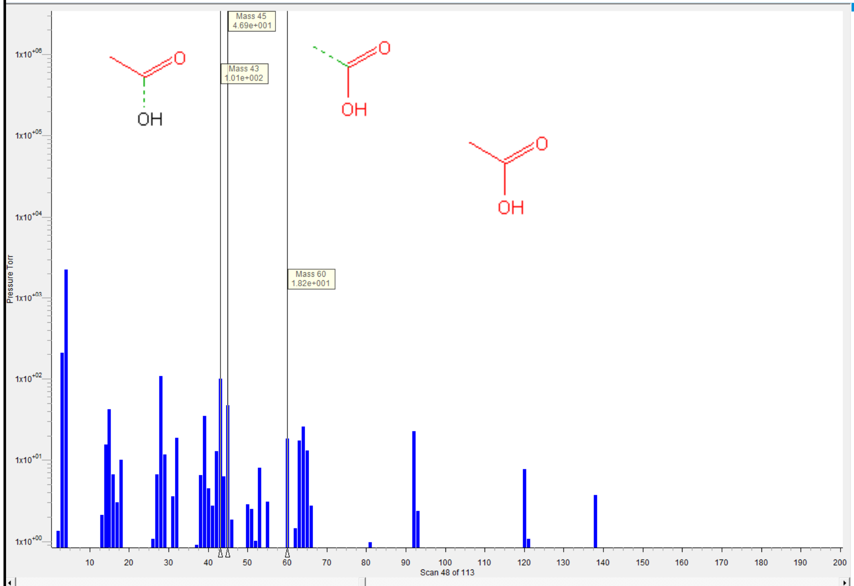
Example: Aspirin



Example: Aspirin



Example: Aspirin



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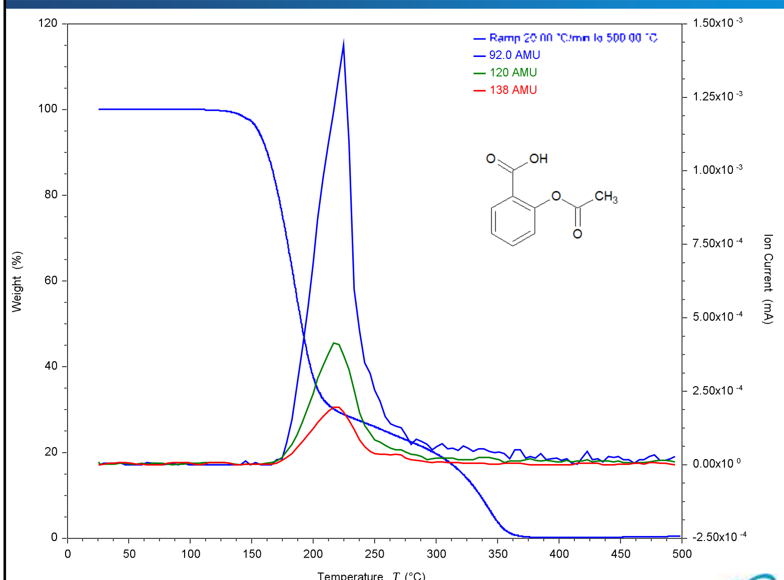
Example: Aspirin



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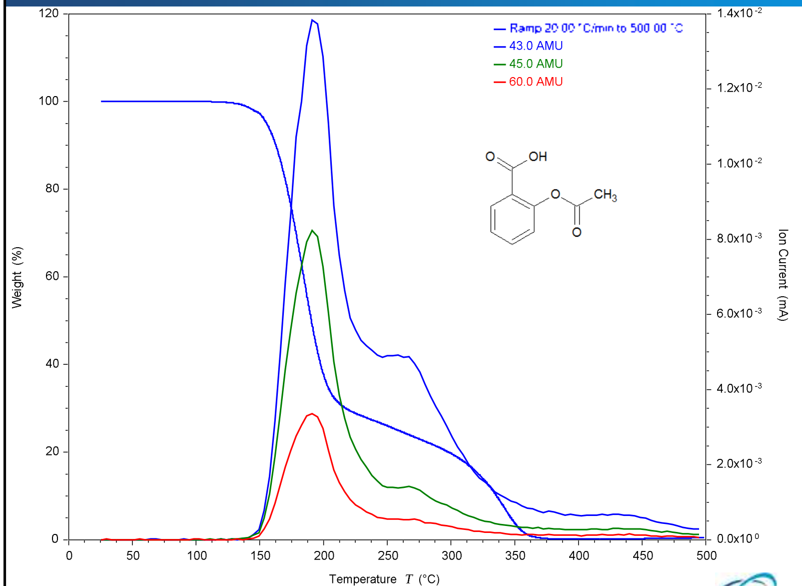
Example: Aspirin



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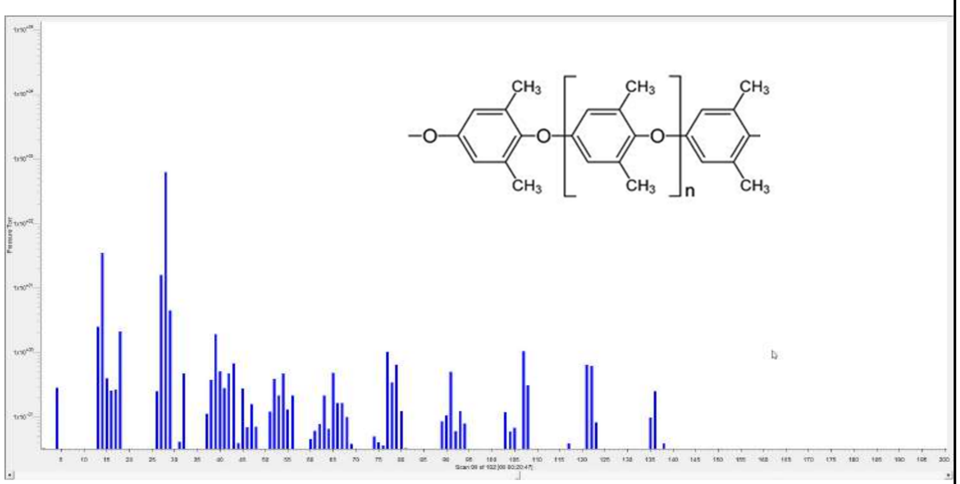
Example: Polyphenylene Oxide (PPO)



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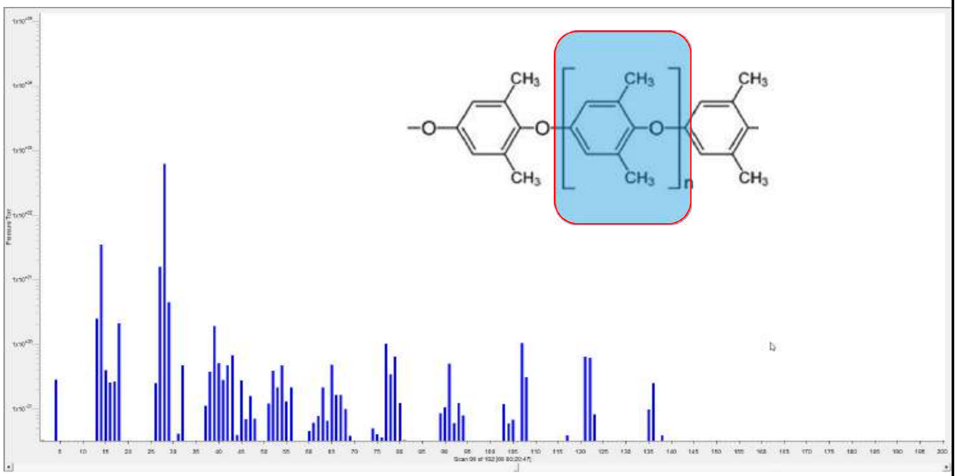
Example: Polyphenylene Oxide (PPO)



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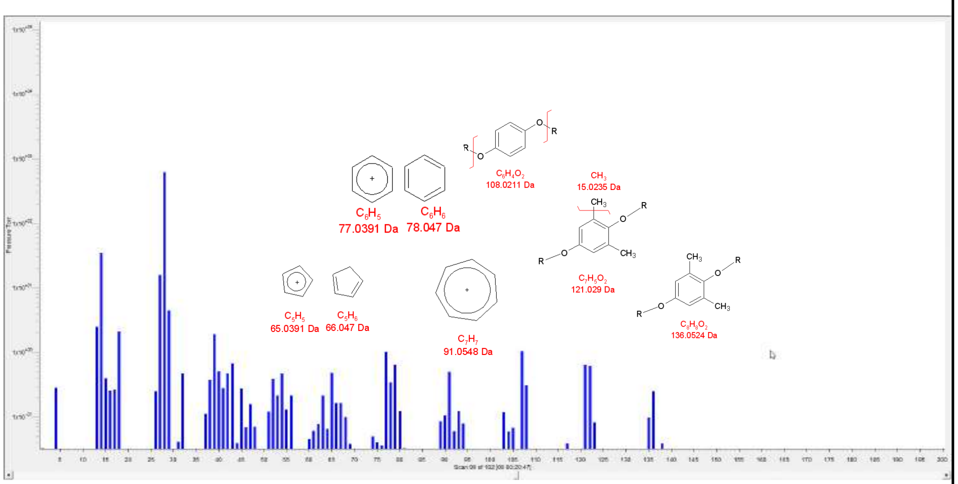
Example: Polyphenylene Oxide (PPO)



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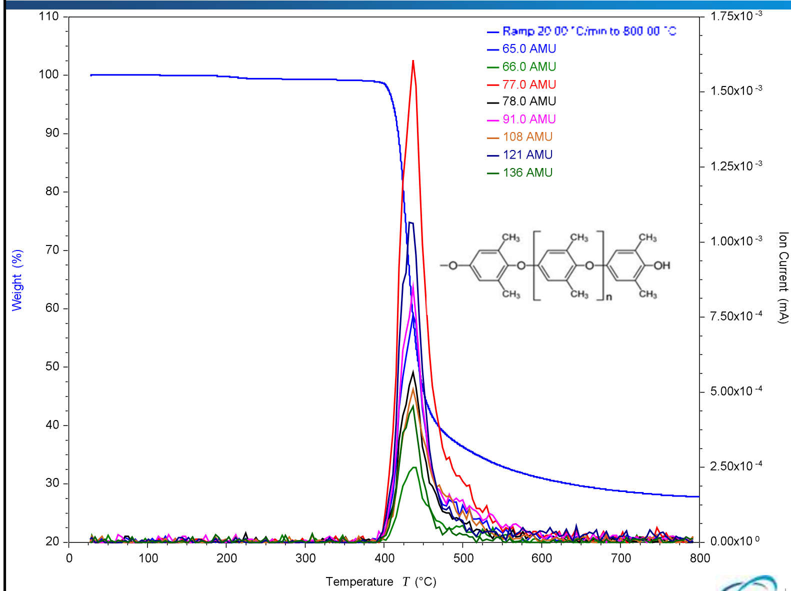
Example: Polyphenylene Oxide (PPO)



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Experimental Design: Some Considerations

- Barchart experiment is a good place to start especially for unknowns.
- Peak Jump is very useful for ‘known’ samples; for example, monitoring residual solvent, reaction products, contaminants, etc.
- Start with small sample masses: 2-3 mg and increase if needed.
- Purge gas
  - Use High Purity (may still contain air)
  - He, N2, Ar, Air
- Monitor background before and after run.

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Experimental Design: Some Considerations

- An isothermal before starting the heating ramp is effective for obtaining clean data showing minimal atmospheric changes as the furnace closes.
- For volatile samples, a DSC pinhole pan will often minimize the loss of sample during the isothermal.

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Data Reduction

- Many different approaches, find what works for you.
- TRIOS software is well suited for working up TGA/MS data and easily generating high quality reports.
- ChemSketch is also quite useful.

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Summary

- TGA/MS is powerful tandem and excellent addition to the analytical chemist's tools.
- High quality data is easy to obtain. Powerful software makes data reduction and reporting easy.
- Instrument is easy to operate and easy to maintain.
- Excellent analytical tool for 'difficult' samples

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Thank You

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