Hyphenation of Thermogravimetric Analyzers (TGA) with FTIR, MS and GC-MS Instruments

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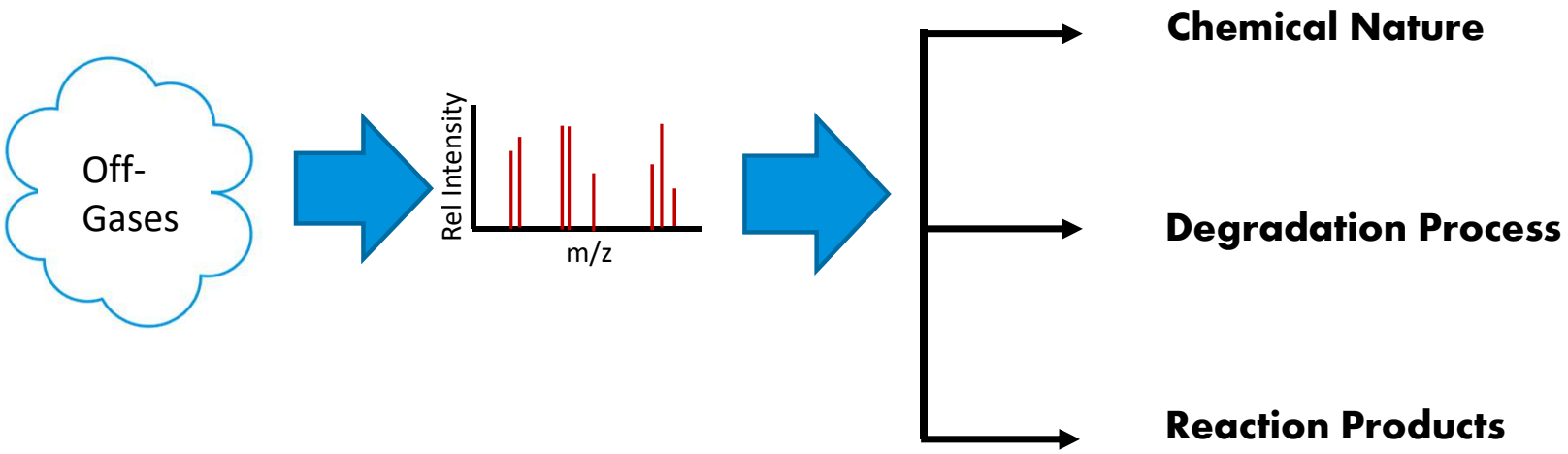
The Motivation for Hyphenation

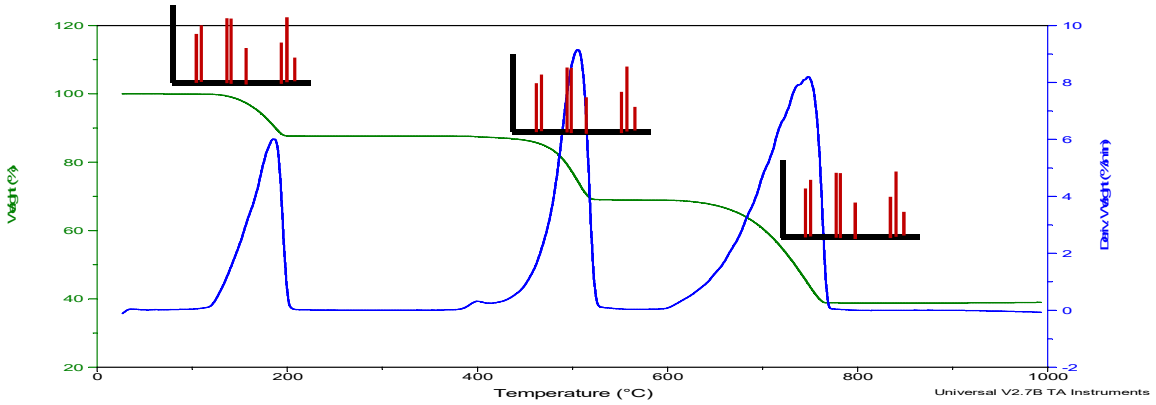
In the vast majority of TGA experiments, gases are evolved. This represents information that in most cases is ejected out of the roof of the building!



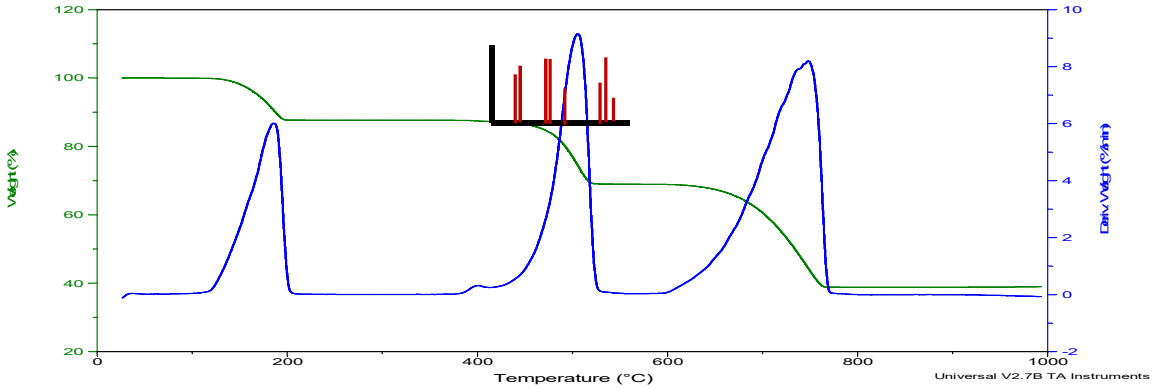
Requires a heated transfer line so that the off-gases remain in the vapor state as they are transferred from the TGA to the off-gas analysis equipment.

Which Technique is Best?

Continuous versus Non-continuous Spectra Collection



Continuous: Multiple spectra obtained over time during expt.

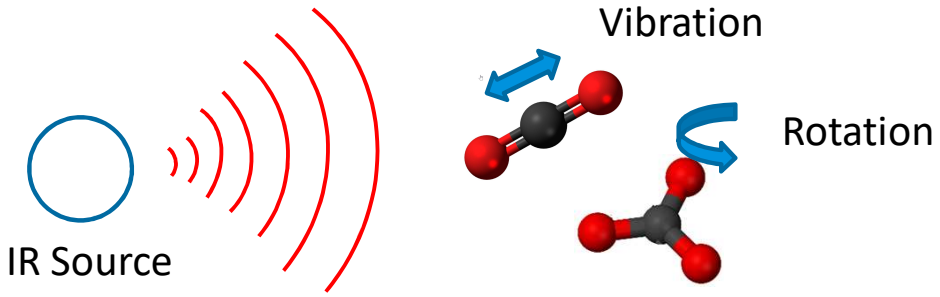


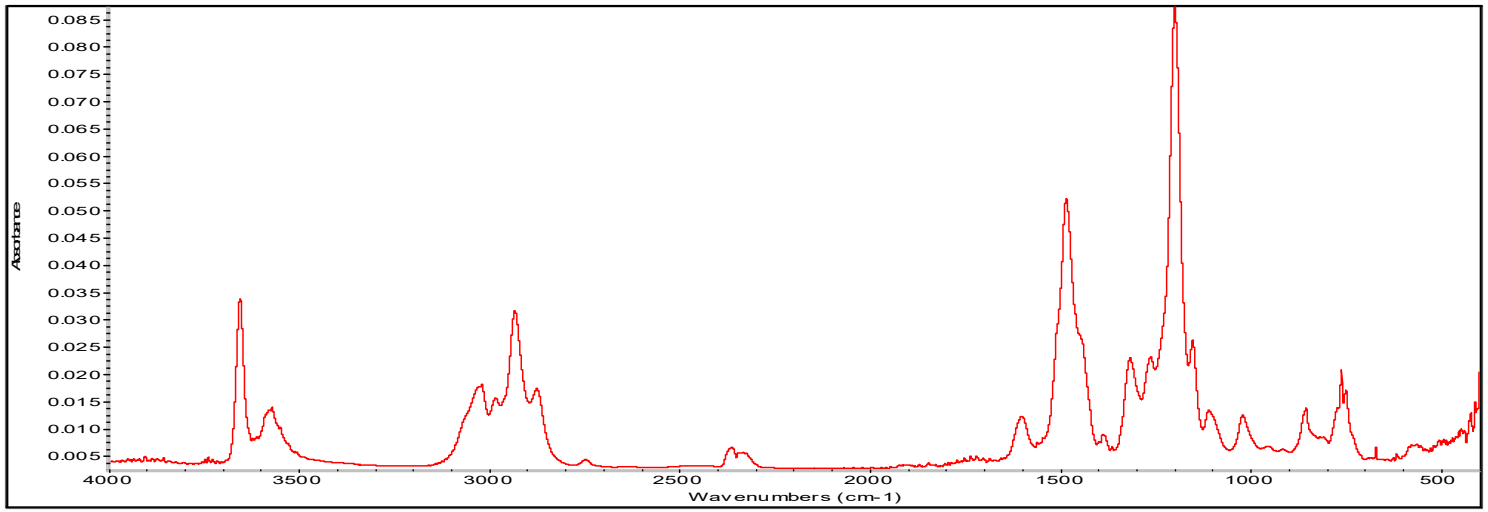
Non-Continuous: One spectrum obtained at only one time during expt.

Off-Gases Typically Analyzed

- Sometimes the gas or vapor of the substance under investigation.
- Sometimes reaction products of the substance under investigation.
- Sometimes degradation products of the substance under investigation.

Types of Hyphenation: Infrared Spectrometry





TGA - FTIR

FTIR (Fourier Transform Infrared Spectroscopy):

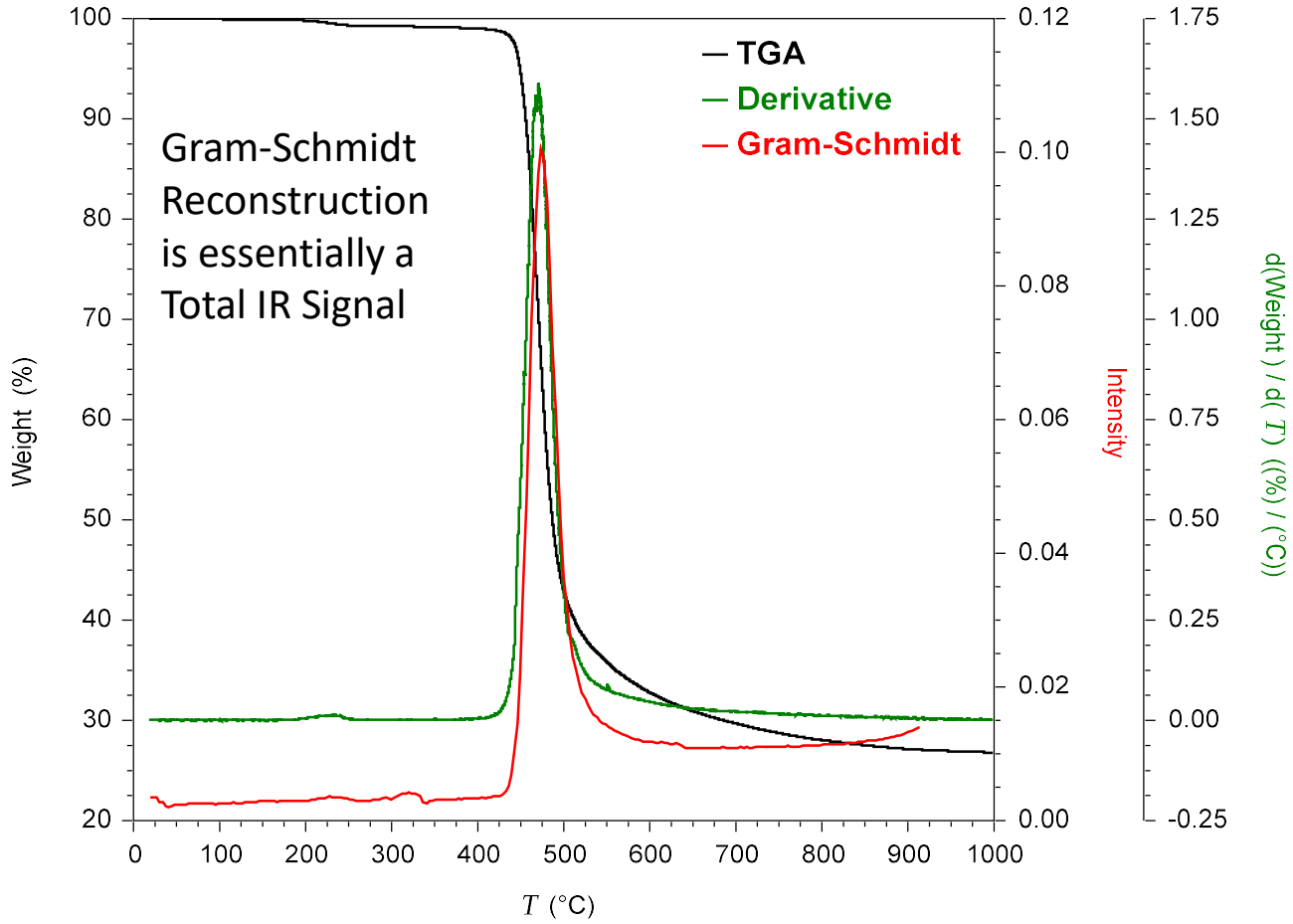
Advantages:

- Continuous method
- Easy Spectral Subtraction
- Library searches are straightforward / deconvolution possible

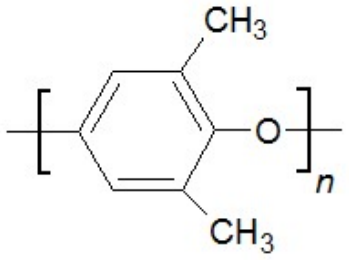
Disadvantages:

- No detection of gases lacking a dipole moment

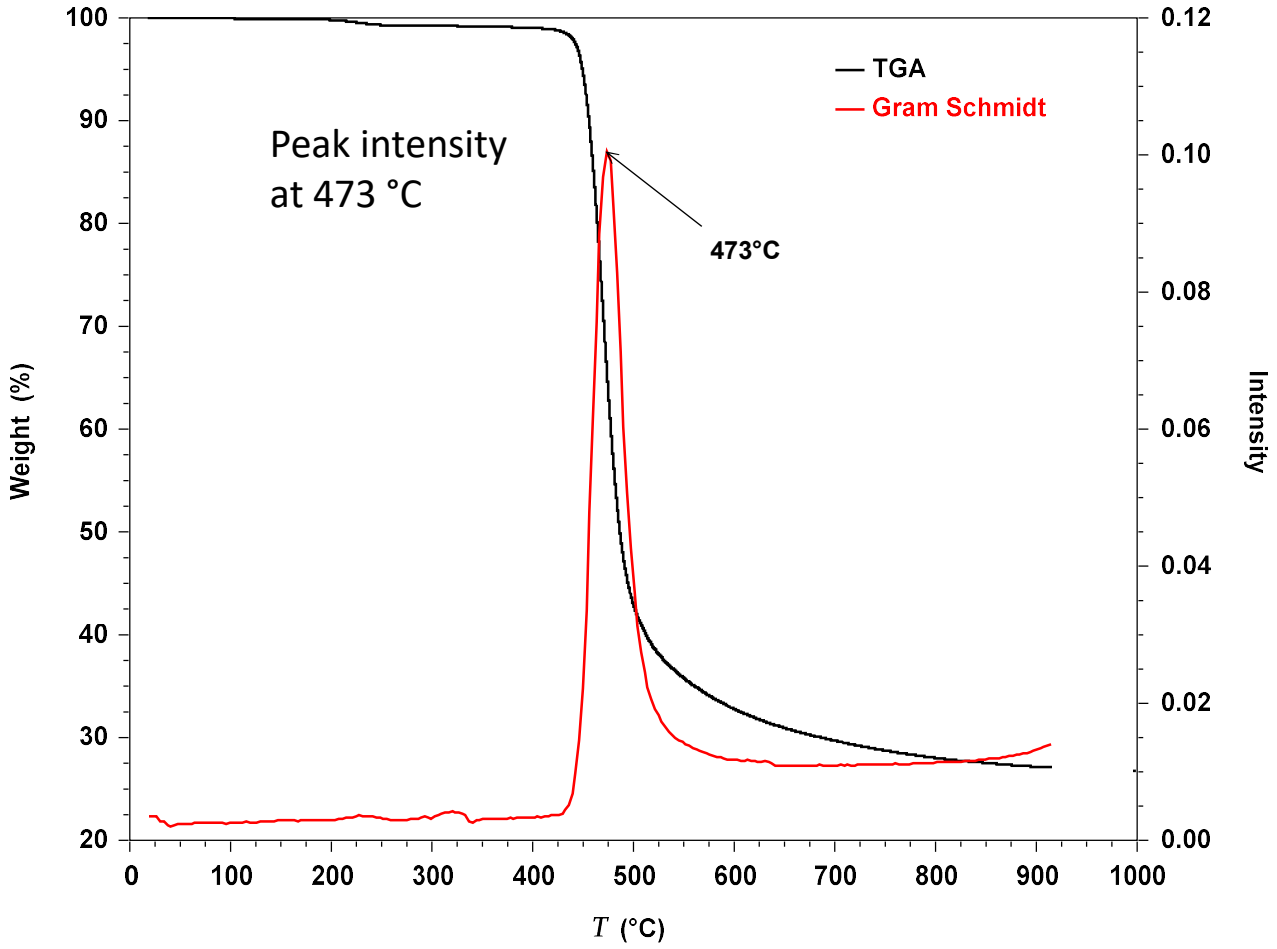
TGA-FTIR: Analysis of Polyphenylene Oxide



Engineering polymer
-Heat resistant
-Good tensile properties



TGA-FTIR: Analysis of Polyphenylene Oxide



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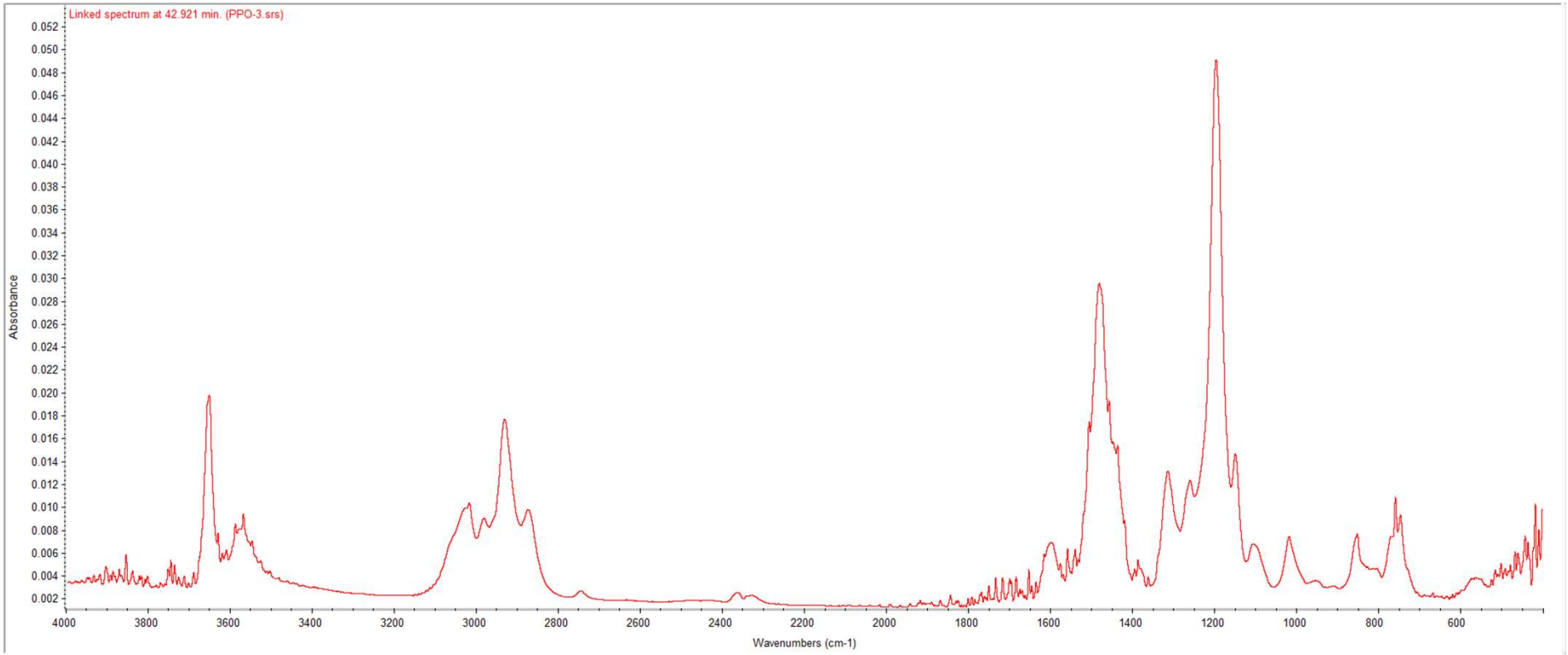

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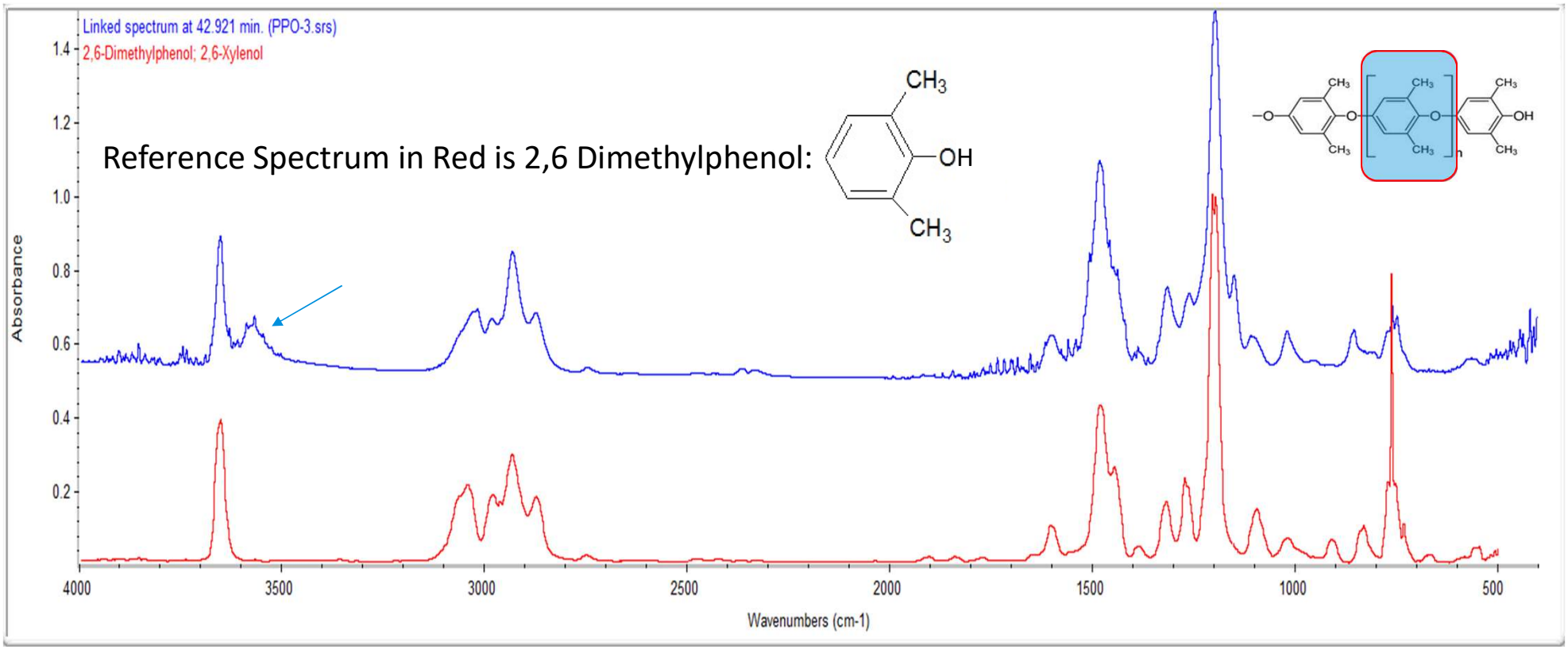
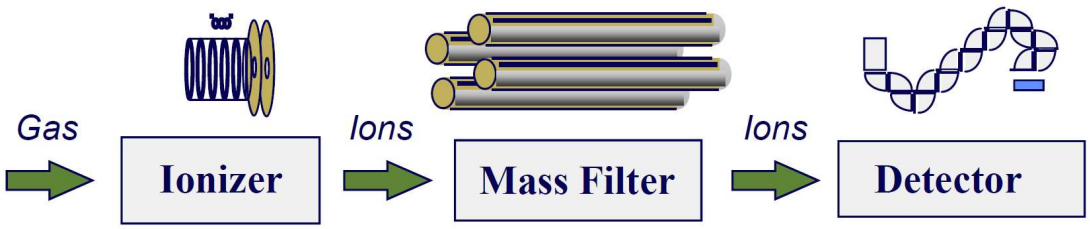


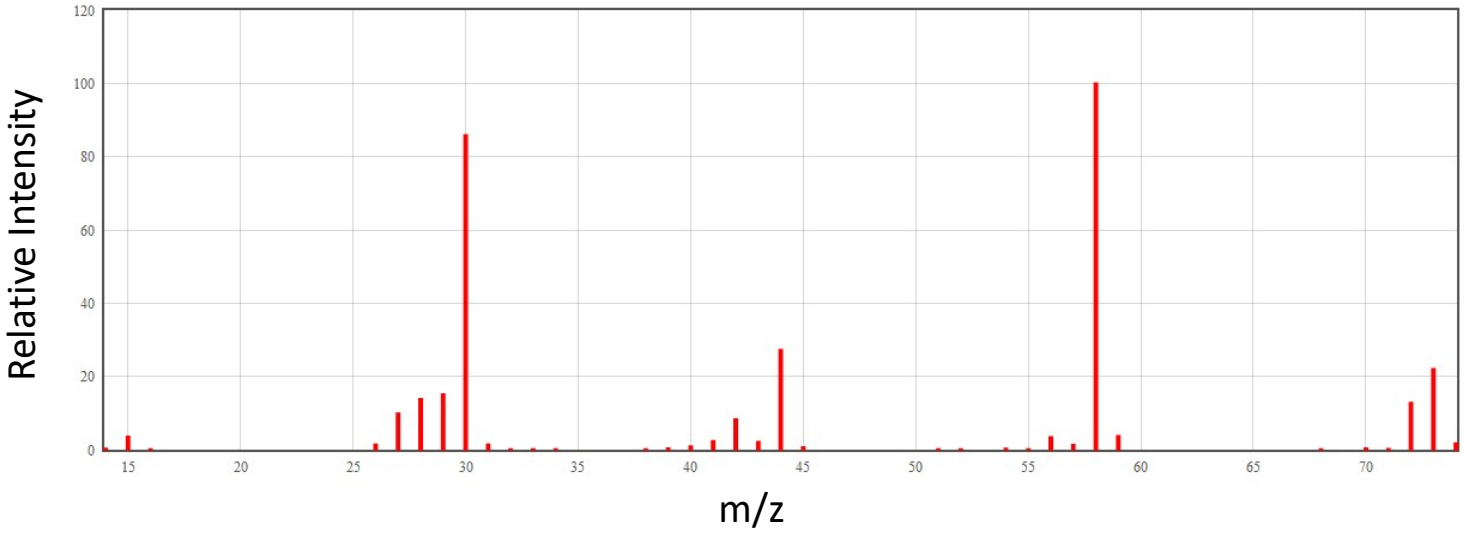
And Now a Word About Library Searches:

- Libraries give the best single spectra match to the data presented.
- …But if multiple components are being emitted during a single weight loss event, the spectra will be superimposed upon each other possibly leading to difficulties.
- The existence of searchable libraries does not relieve the analytical chemist from critically analyzing the search results.
- ThermoNicolet FTIR software can attempt to deconvolute a spectrum to a maximum of four components. Demonstration can be found in this TA Instrument webinar:

https://www.tainstruments.com/evolved-gas-analysis-tgaftir/

Types of Hyphenation: Mass Spectrometry





TGA-Mass Spectroscopy

Advantages:

- Continuous method
- Higher sensitivity than IR Spectroscopy.
- Measures non-IR absorbing gases.
- Rapid response (gases drawn into capillary).

Disadvantage:

- Cannot distinguish between molecules with similar molecular weights. (e.g. N_2 and CO)

The Discovery Mass Spectrometer (DMS)

- Benchtop, unit resolution quadrupole mass spec designed and optimized for evolved gas analysis (EGA)

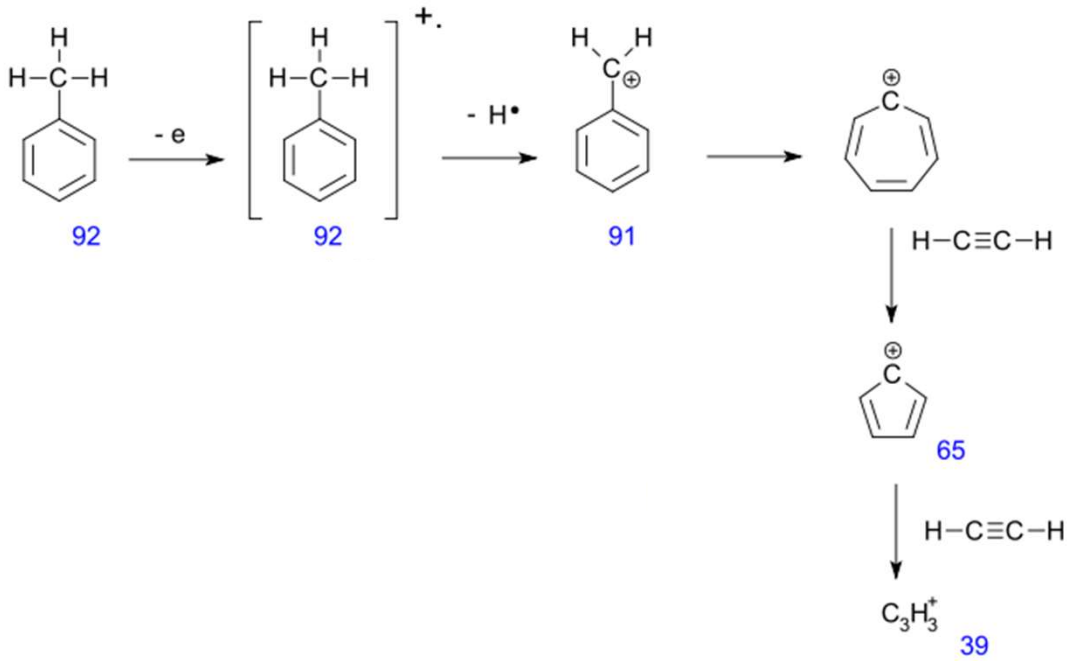
- Quadrupole detection system includes…
  - a closed ion source
  - a quadrupole mass filter assembly
  - 1-300 amu range
  - dual detector system (Faraday and Secondary Electron Multiplier)

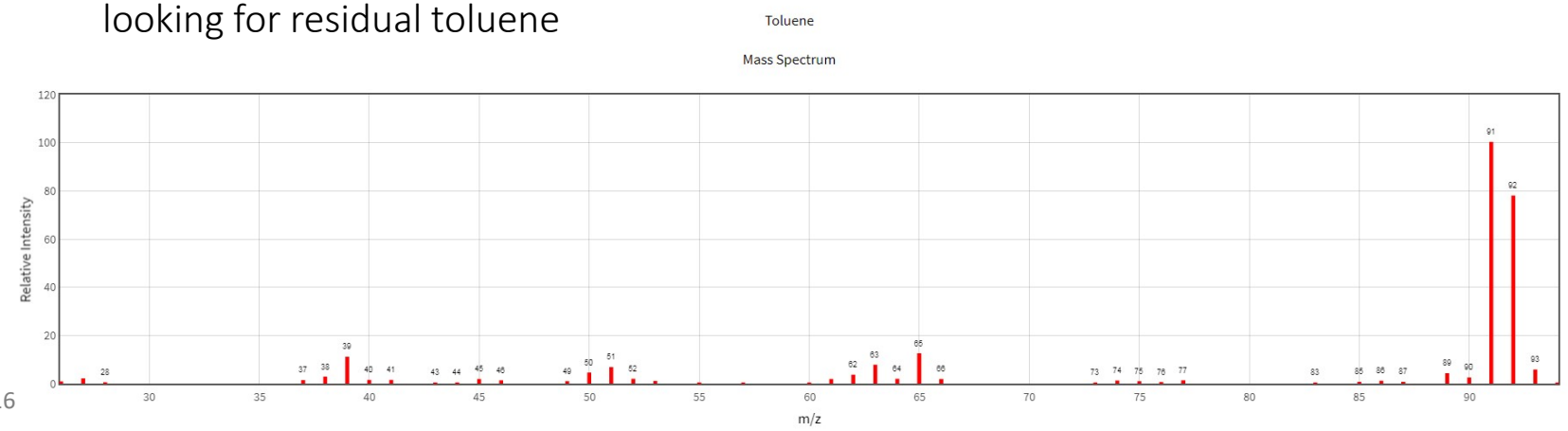
IR Furnace

EGA Furnace

TGA/MS: Experiments

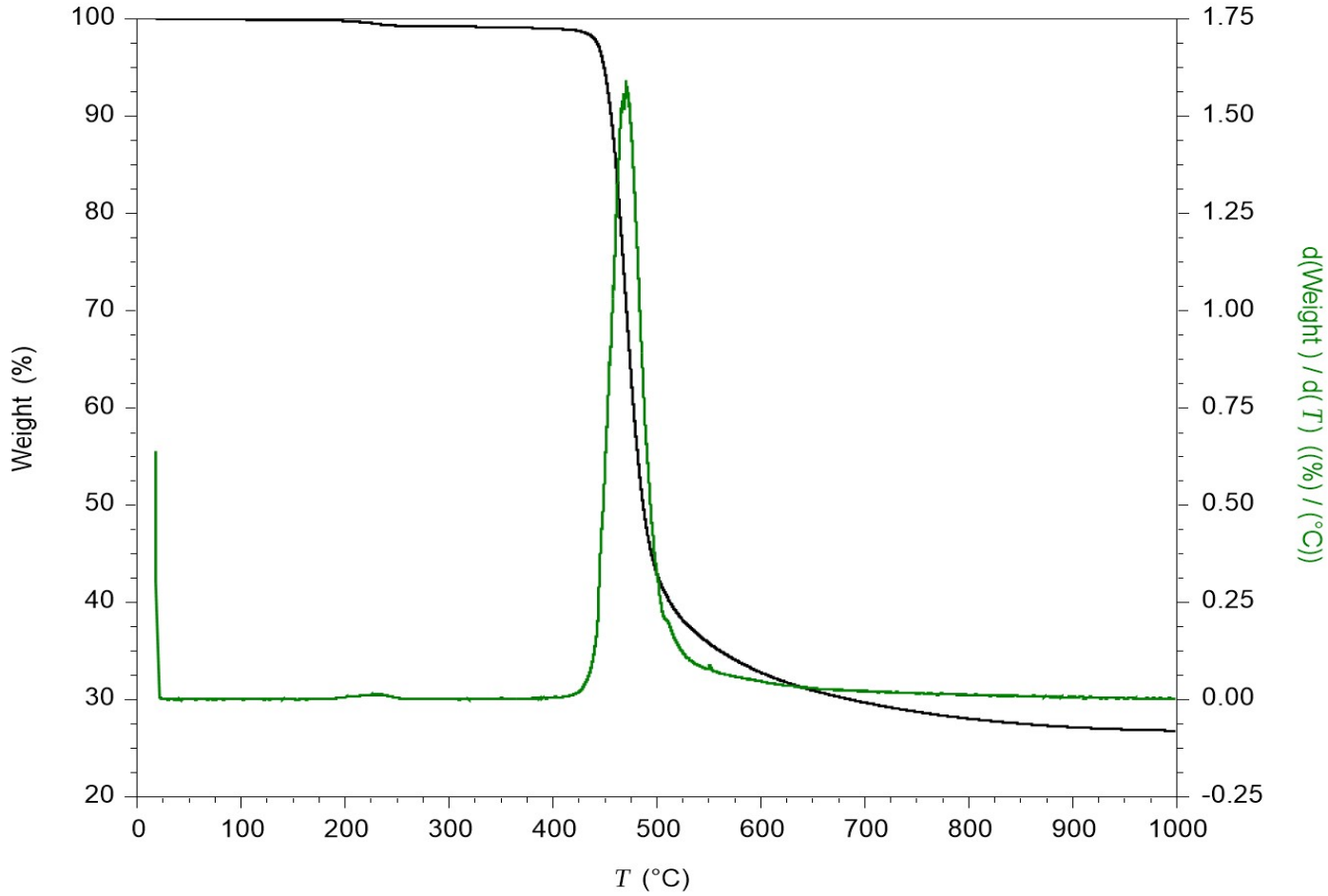
- Barchart
  - Scan across specified ion range - m/z 1 to m/z 300
  - Typically used as first approach for an unknown compound
- Peak Jump
  - Scan specific ions
  - Example, scan m/z 91, 65, 51, 39 if you are looking for residual toluene



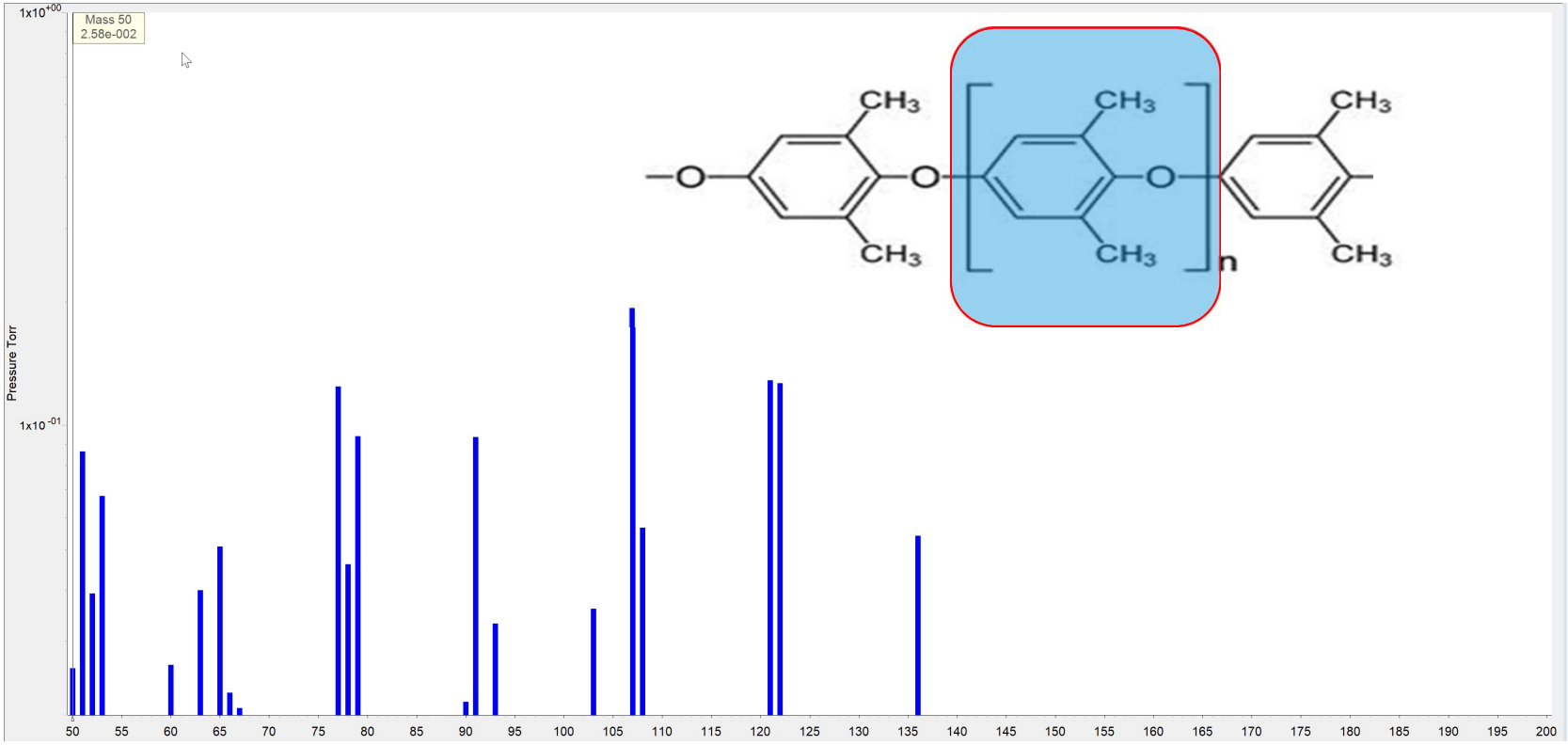


Reference: NIST MS Library

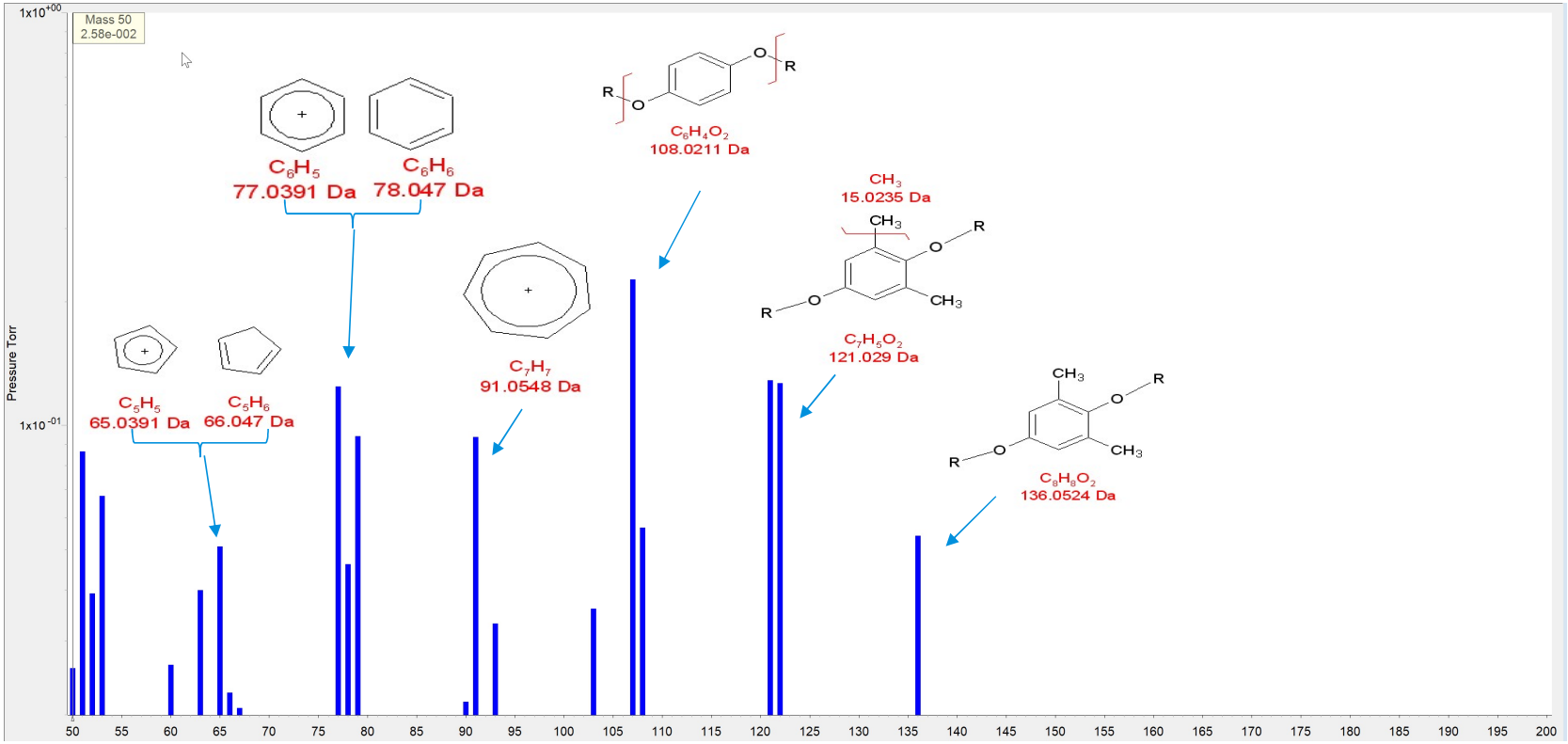
TGA : Analysis of Polyphenylene Oxide



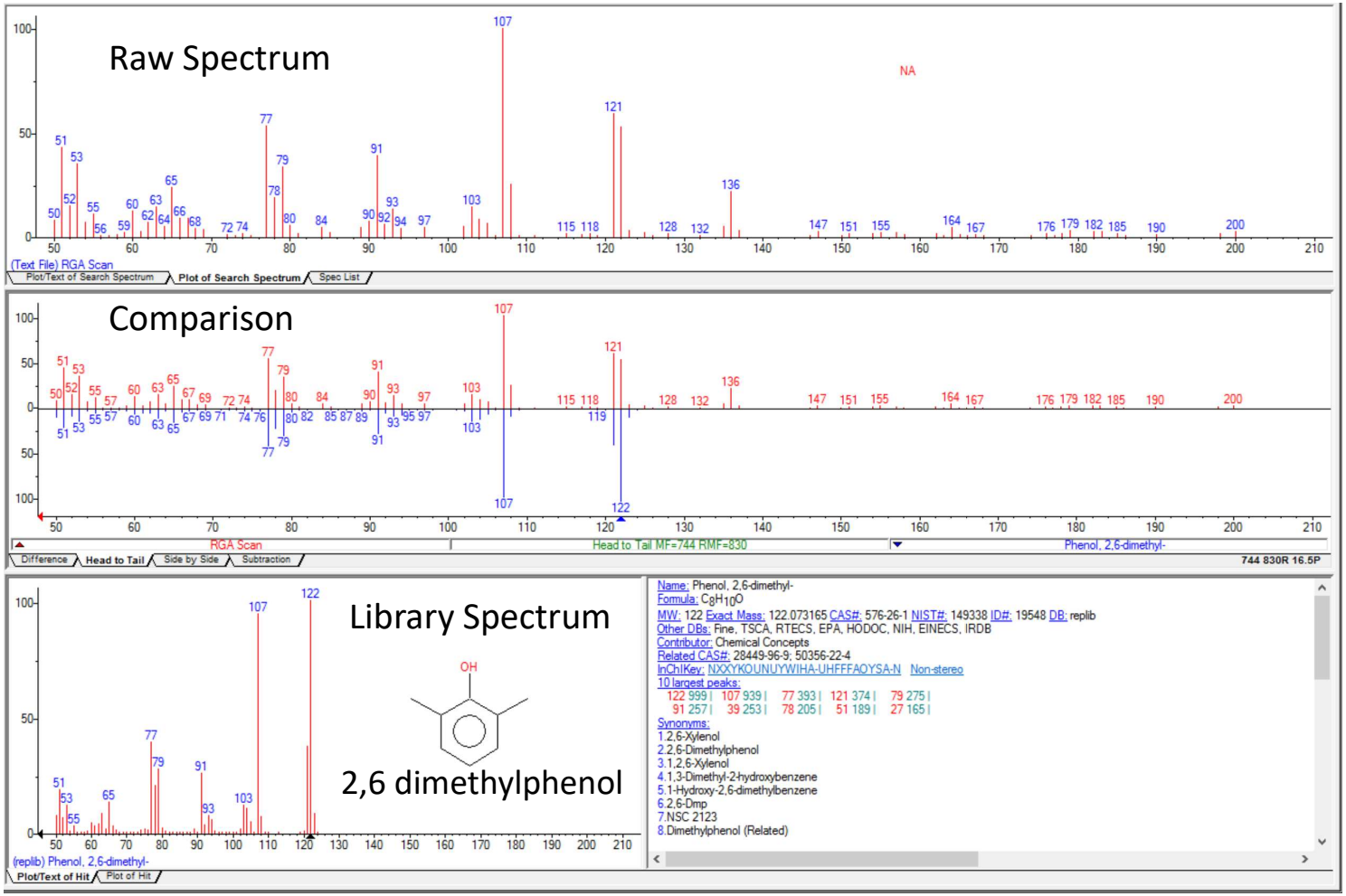
TGA MS: Polyphenylene Oxide (PPO)



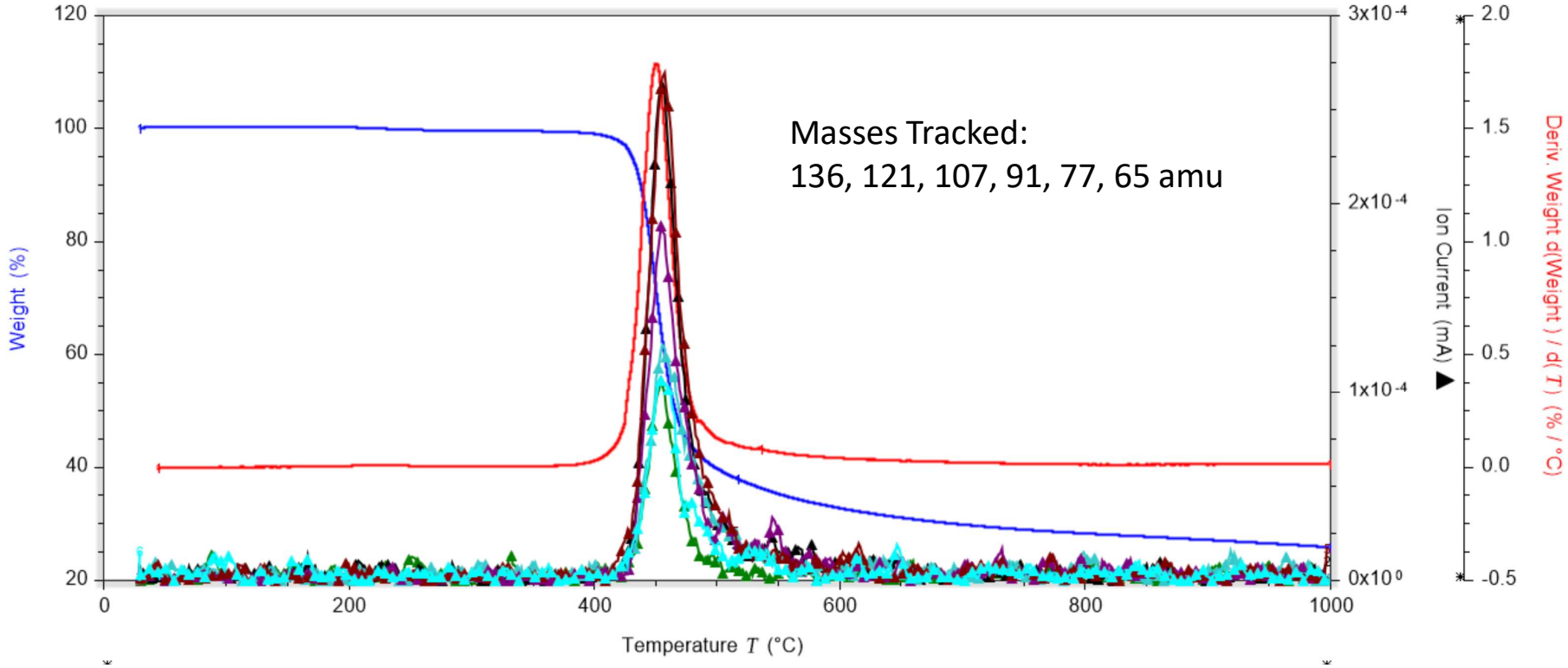
TGA-MS: Polyphenylene Oxide (PPO)



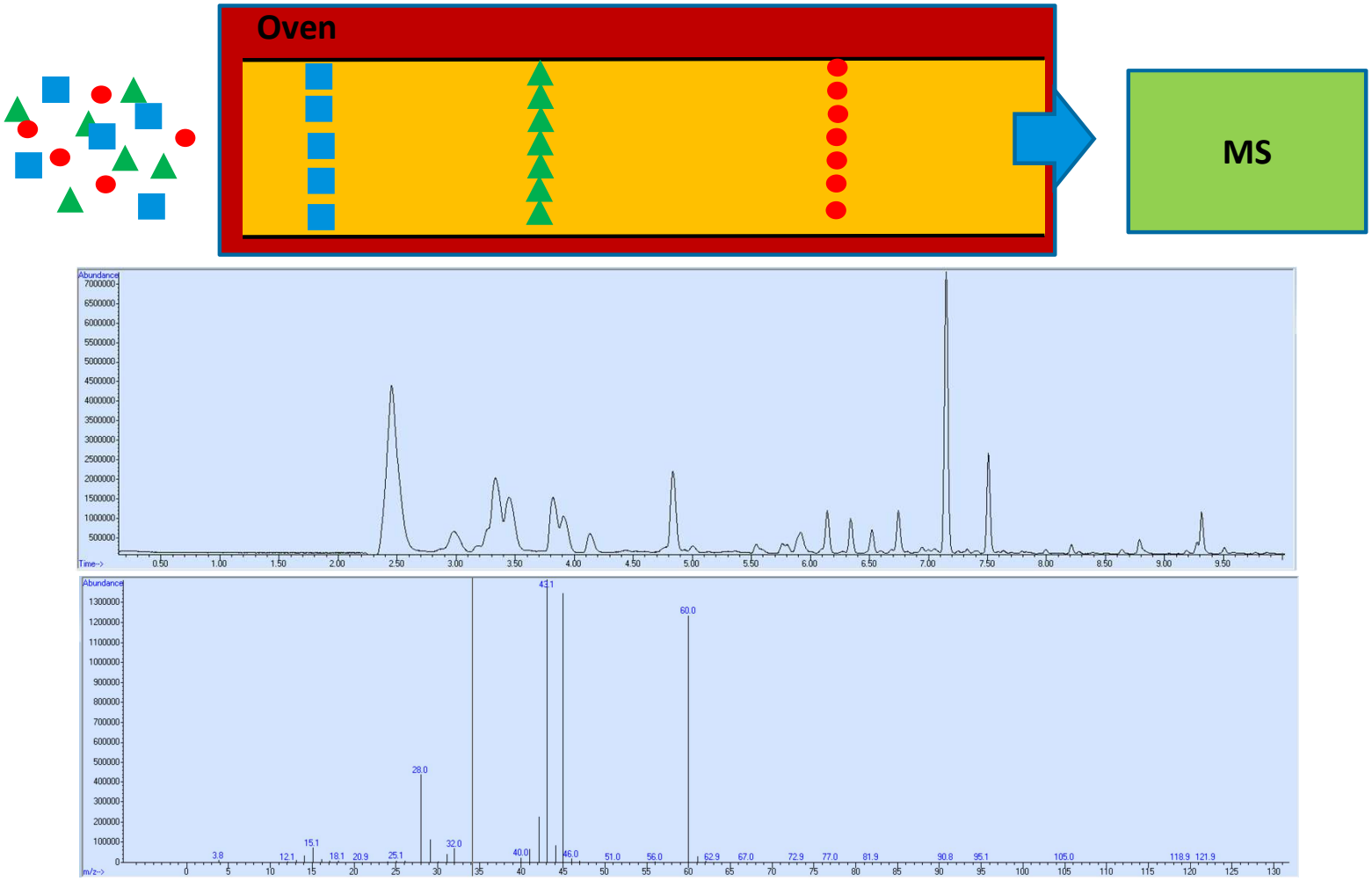
NIST Library Search Results



TGA-MS: Polyphenylene Oxide (PPO)



Types of Hyphenation: Gas Chromatography / Mass Spectrometry



TGA – GC/MS

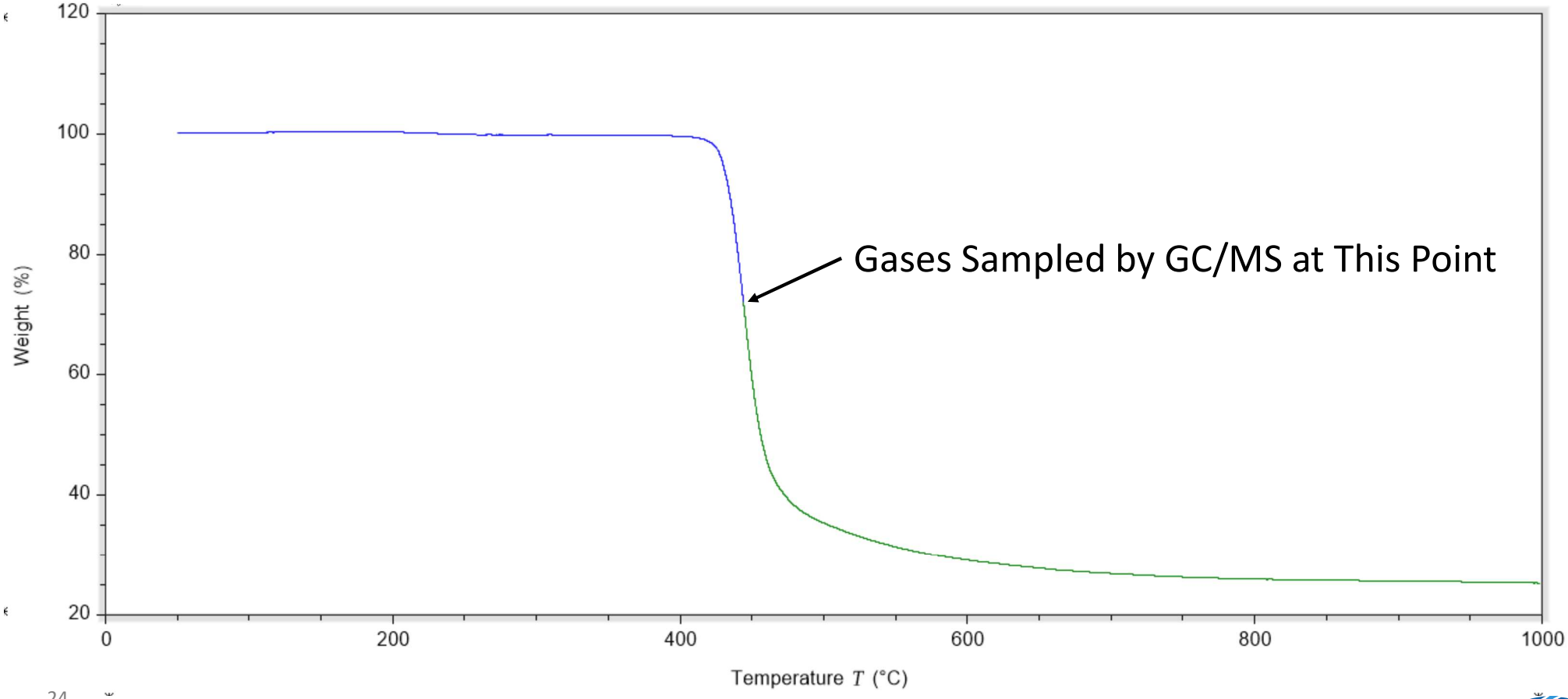
Advantages:

- Chemical Separation
- Easy library searching

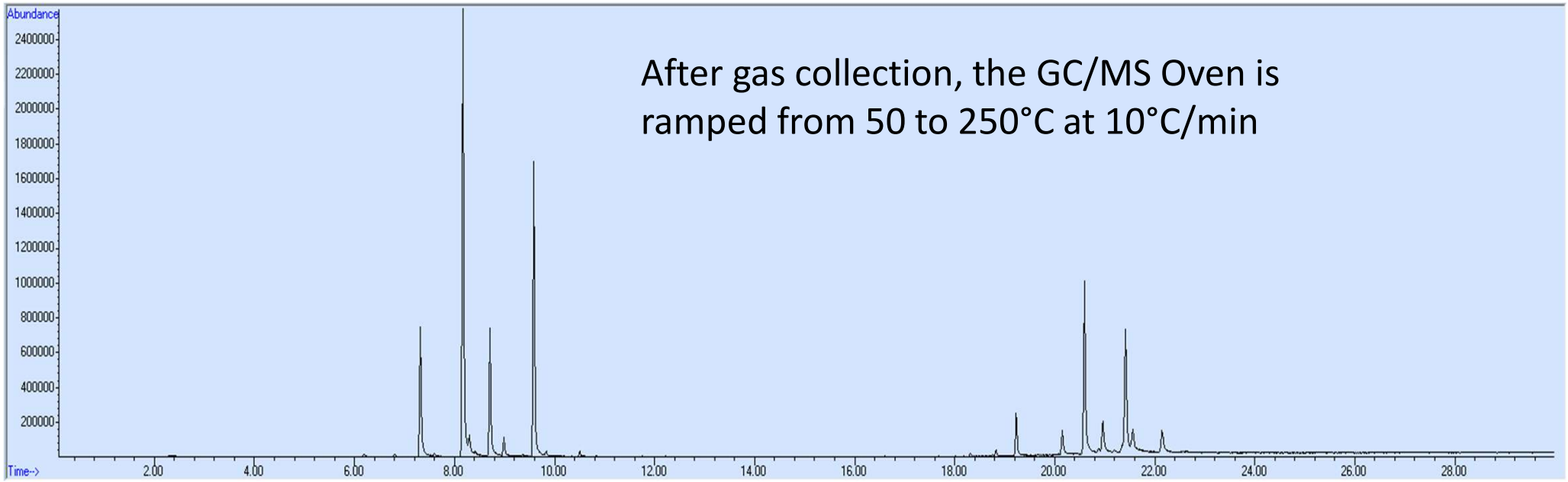
Disadvantages:

- Typically time consuming – not a continuous measurement

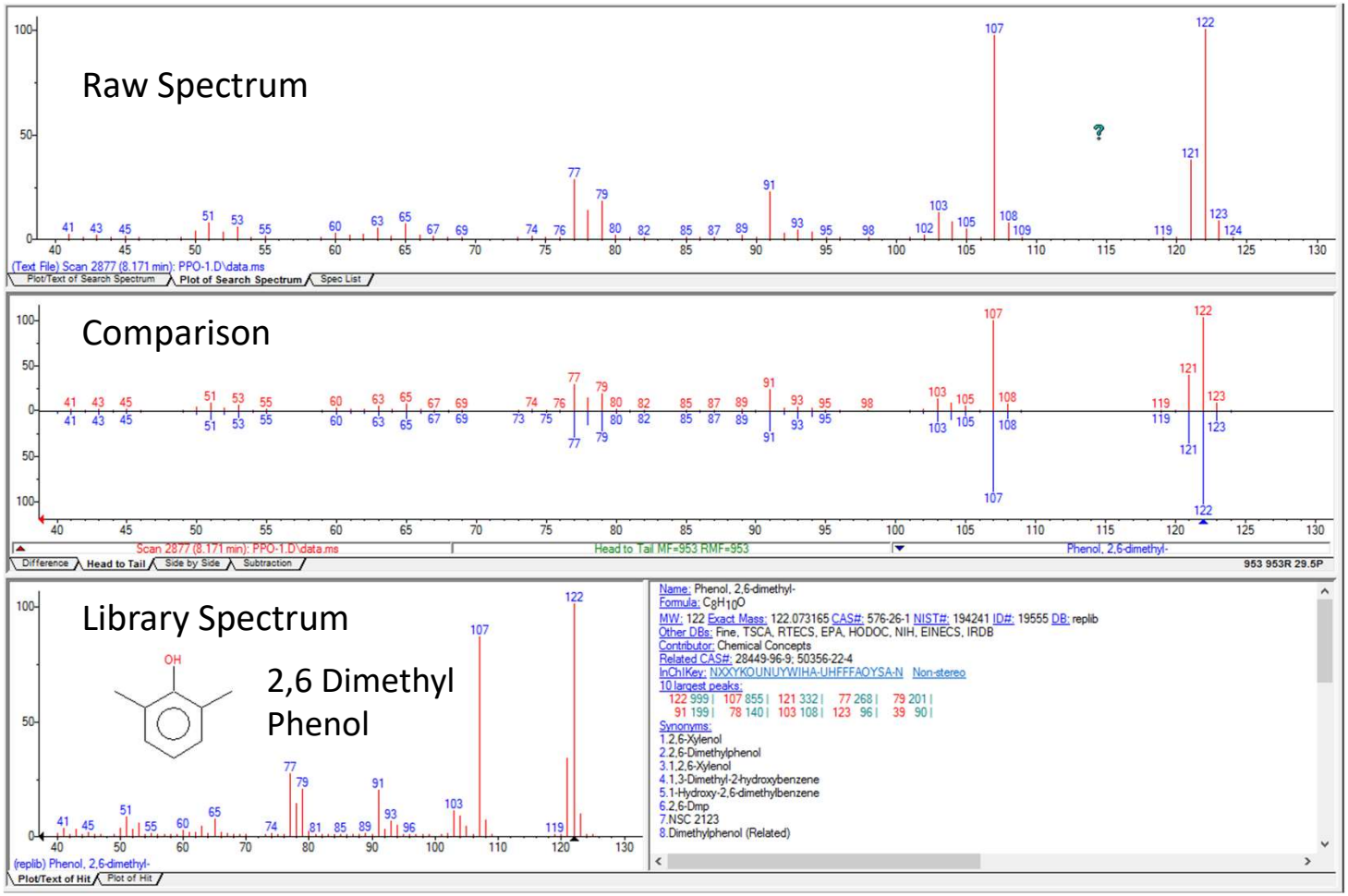
Anatomy of a GC/MS Run: Polyphenylene Oxide



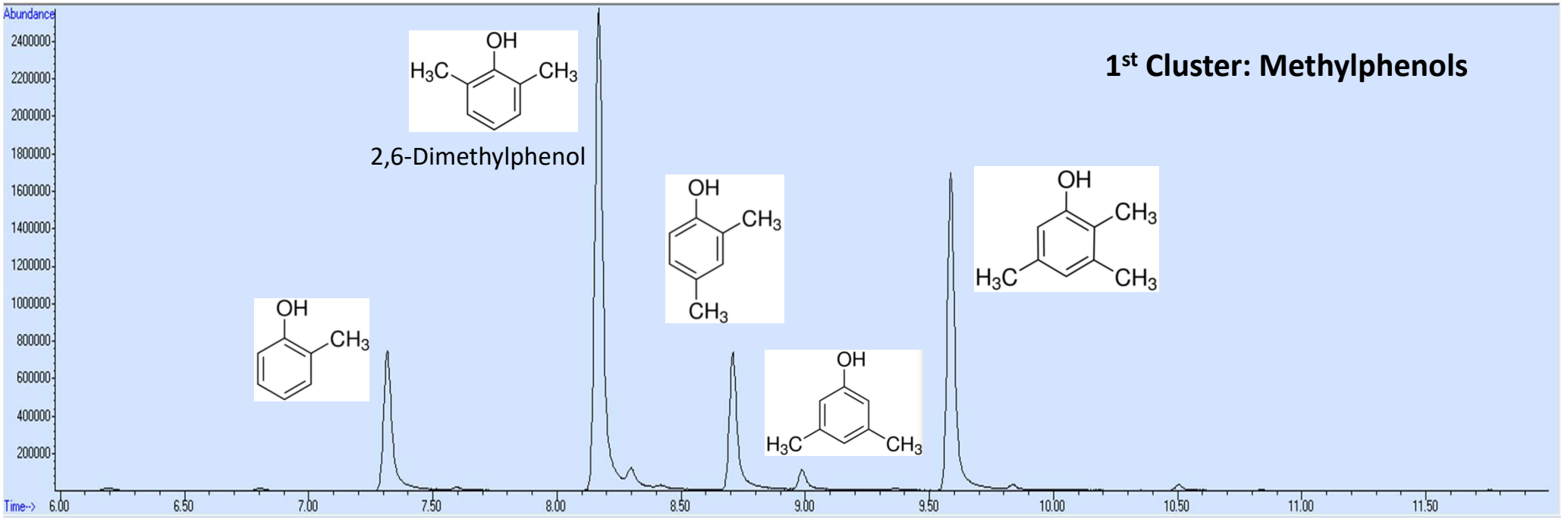
TGA-GC/MS: Analysis of Polyphenylene Oxide



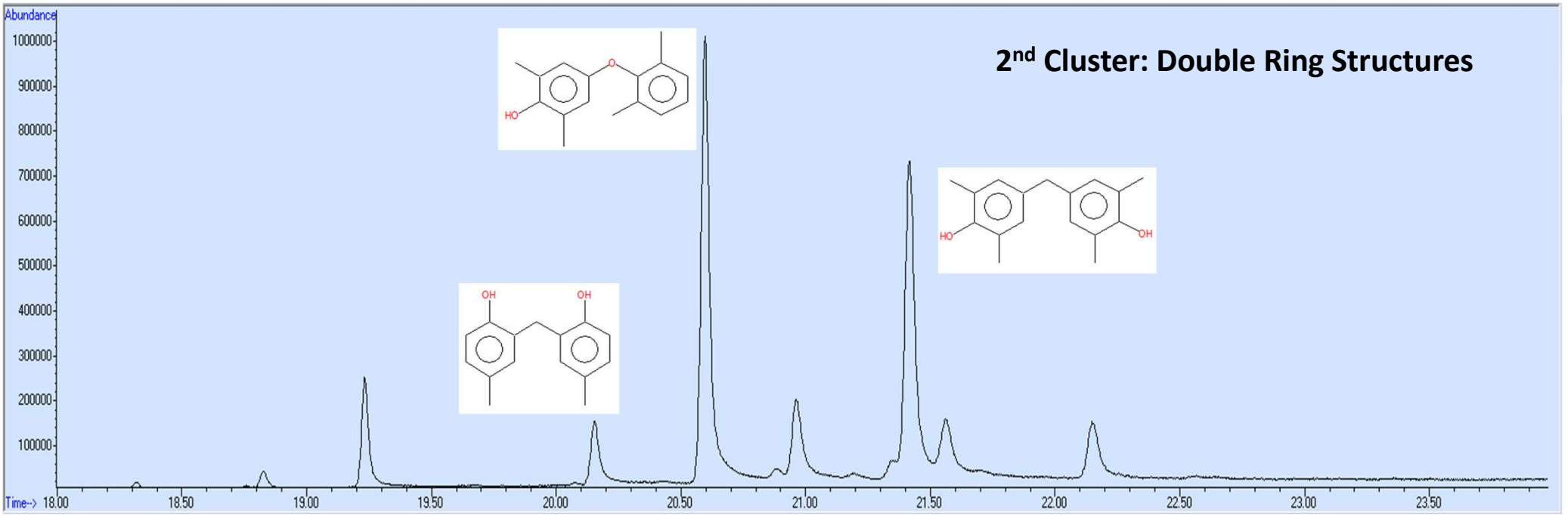
GC/MS Library Search; Largest Peak



TGA-GC/MS: Analysis of Polyphenylene Oxide



TGA-GC/MS: Analysis of Polyphenylene Oxide



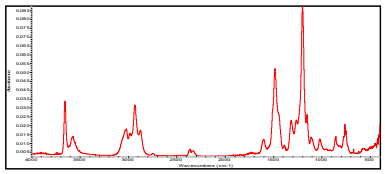
Evolve Gas Analysis – TGA Multiple Hyphenation

GC/MS **FTIR** **TGA**

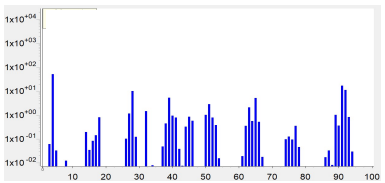
Linked Spectrometers

Linking an FTIR with a GC/MS

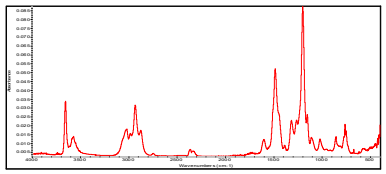
FTIR

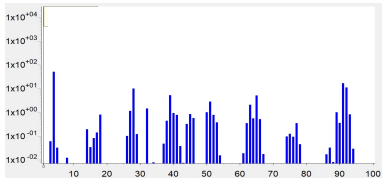


MS

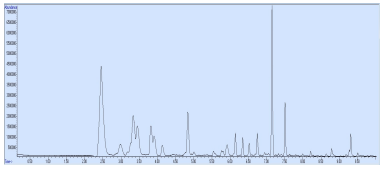


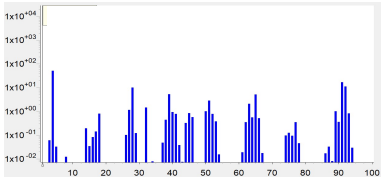
FTIR/MS



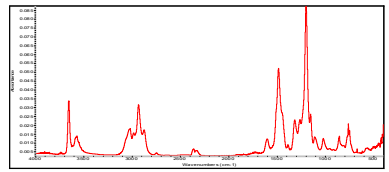


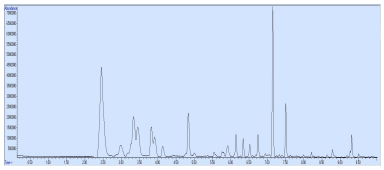
GC/MS

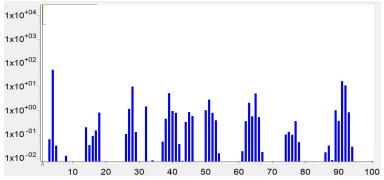




FTIR/GC/MS







Conclusions

- Hyphenation to a spectrometer allows for the chemical analysis of the off-gas products.
- Multiple hyphenation may allow for more complete set of spectral information.
- TA Instruments horizontal flow TGAs and SDTs provide superior TGA data for pairing with spectroscopy data.