Evolved Gas Analysis: TGA/FTIR

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EGA Analysis: TGA/FTIR

- Evolved Gas Analysis is a powerful tandem for today's analytical chemist
- Sometimes referred to as 'hyphenated techniques', the two most common combinations with TGA are FTIR and Mass Spectrometry
- In today's discussion, we present the basics of TGA/FTIR analyses.

EGA Analysis: TGA / FTIR; General Experimental Considerations

- No special sample preparation is necessary – Simply run a TGA experiment.
- Normal ramp rates work well (10 °C / min).
- Dynamic heating generally results in better data than an isothermal hold depending on the volatility of the analyte.

General Tips for Obtaining Good Data

EGA ANALYSIS: TGA / FTIR

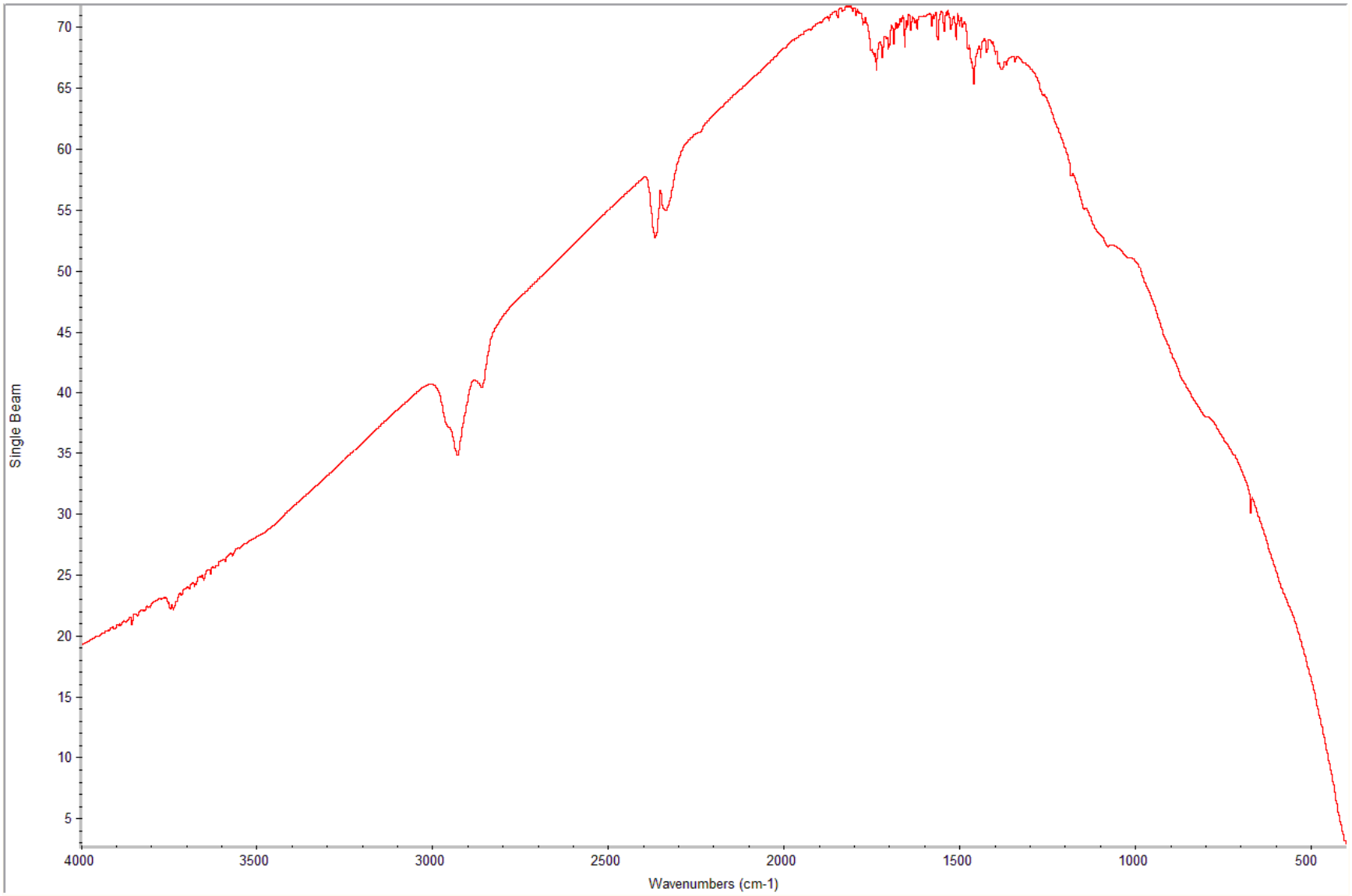
EGA Analysis – TGA/FTIR; General Experimental Considerations

- Be sure the TGA is clean
- Periodically disconnect the spectrometer transfer line and run an isothermal in air or oxygen at 1000 ℃.
- This is especially important if you are running olefinic or oily samples.
- Clean the adapter port, make sure it is not clogged. Any stiff, thin wire will do – an electric guitar string works well. The thin ‘E’ string is good for the EGA adapter on the Discovery a wound ‘D’ string is good for the FTIR transfer line.
- Be careful cleaning the EGA adapter – it is very hot and will give a serious burn. Also be careful running a cleaning wire into the transfer line – it will come out very hot.
- If necessary, follow the procedures in the help menu for more thorough cleaning of the furnace.
- Disconnect or re-route the flow from the adapter when running samples that are not TGA/FTIR samples.

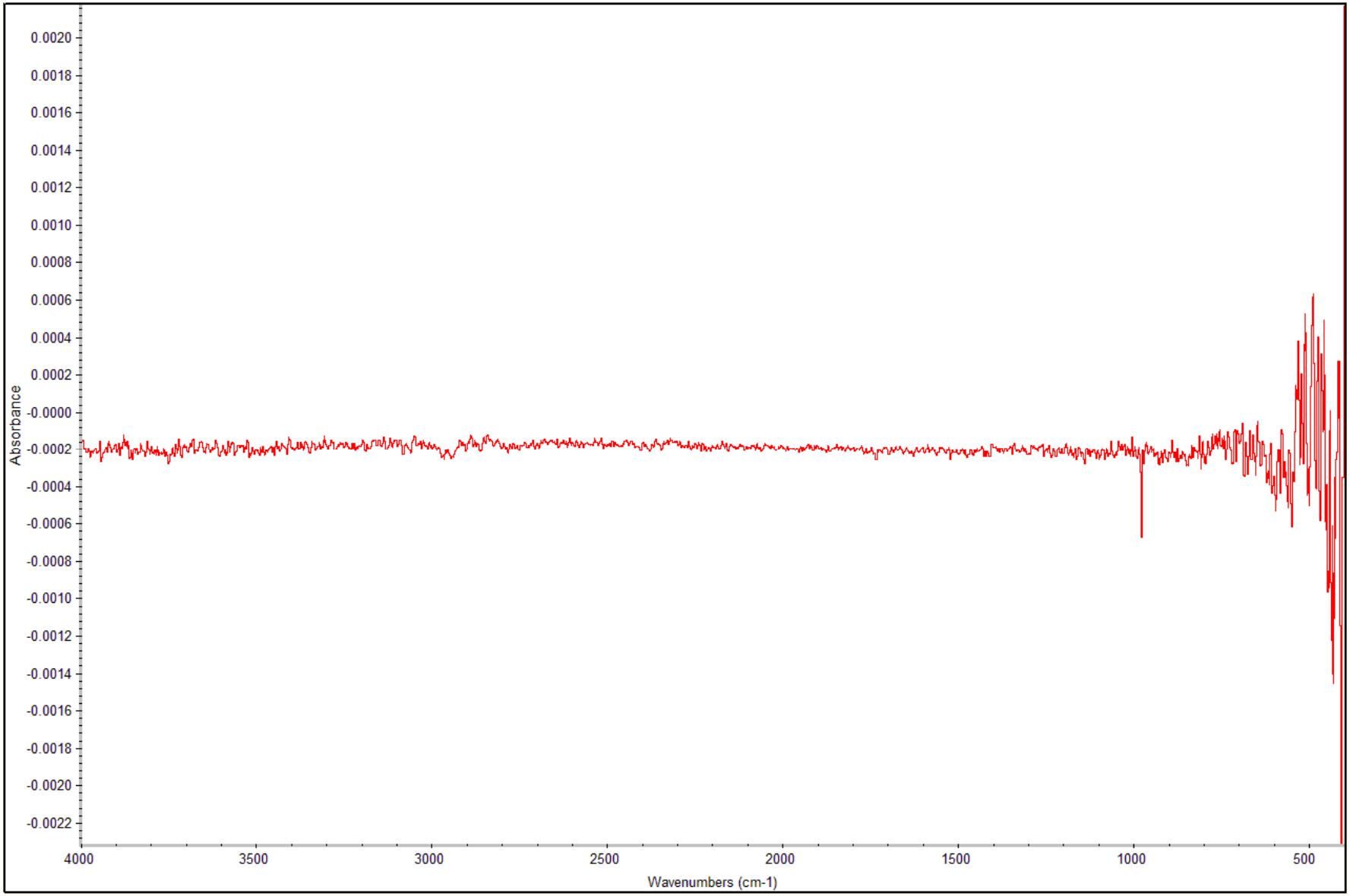
EGA Analysis: TGA/FTIR – Know Your Background

- Begin each experiment by obtaining a single-beam spectrum and a 100% line
  - N.B. – A 100% line is simply a background and absorbance spectrum scan. You should see no evidence of obvious absorbances. Positive absorbances probably mean that residual sample remains, negative absorbances mean that the background is changing and probably improving.
- Check with the spectrometer manufacturer for any information regarding expected signal to noise performance, etc.
- Check the single beam spectrum between runs, be certain there is no residual sample remaining. Run a 100% line if you are not sure.
- Check with the spectrometer manufacturer for cleaning procedures for the gas cell, windows, etc.

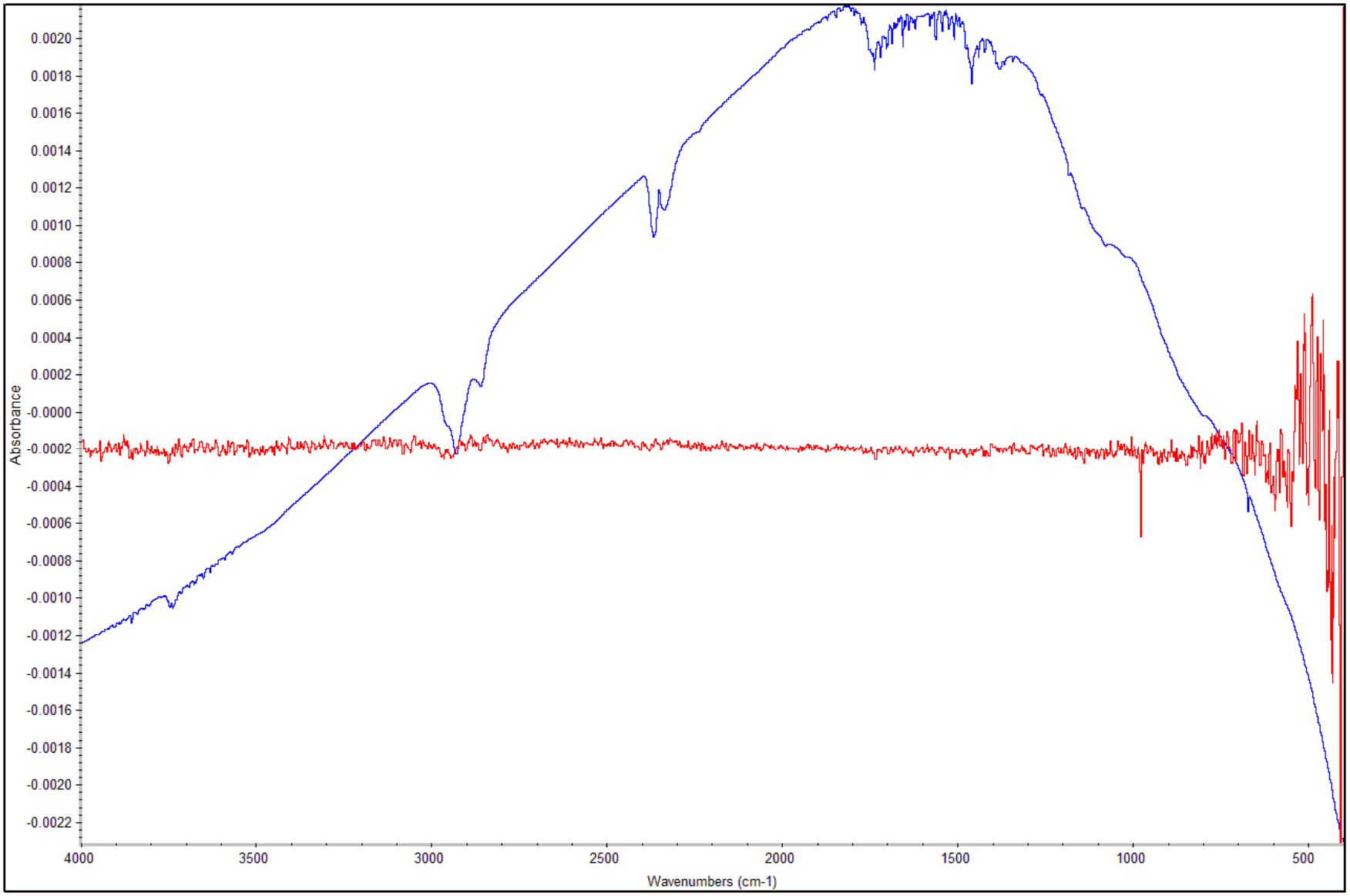
TGA/FTIR – Single Beam Spectrum



TGA/FTIR – 100% Line



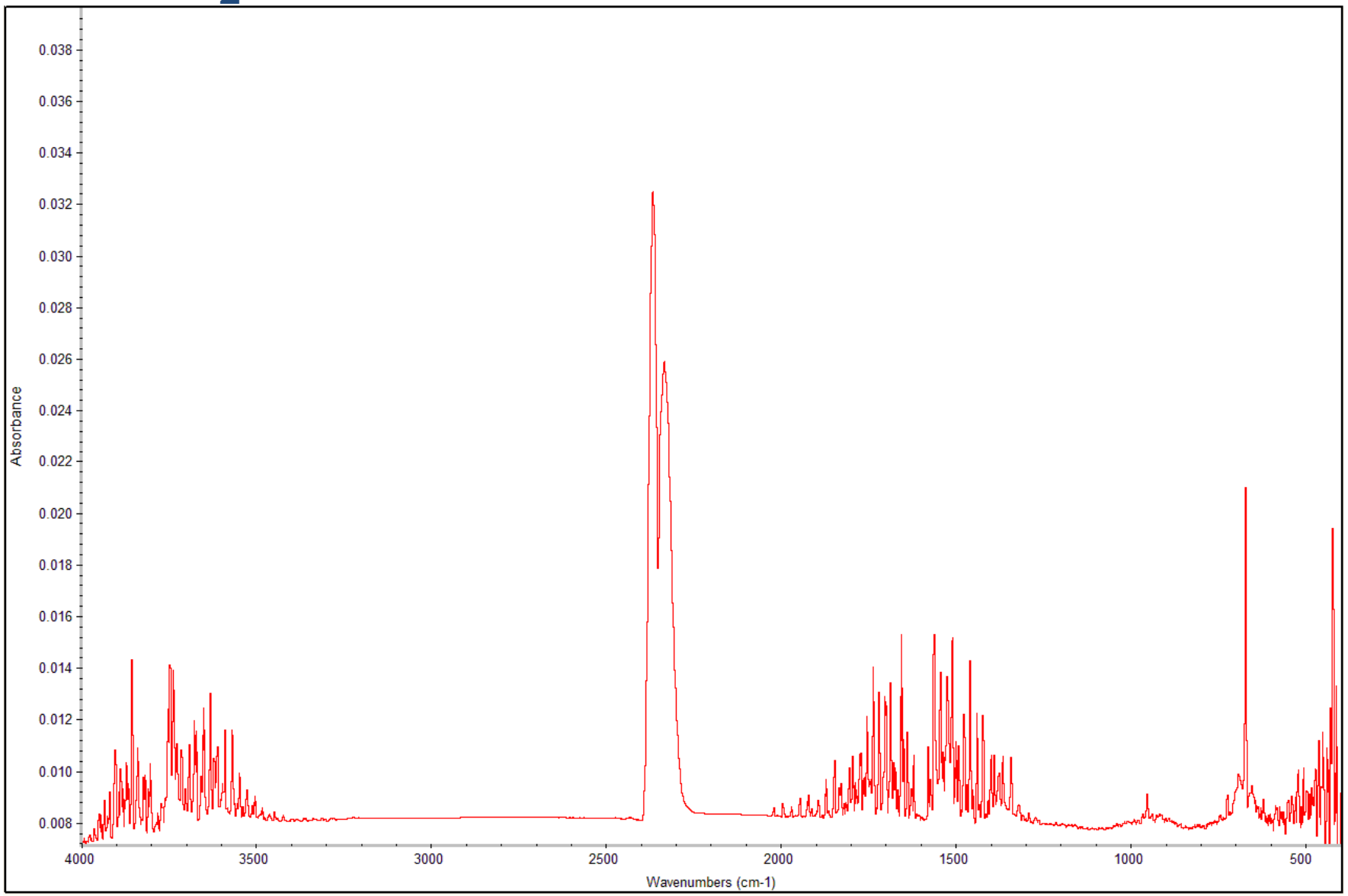
TGA/FTIR – 100% Line / Single Beam Overlay



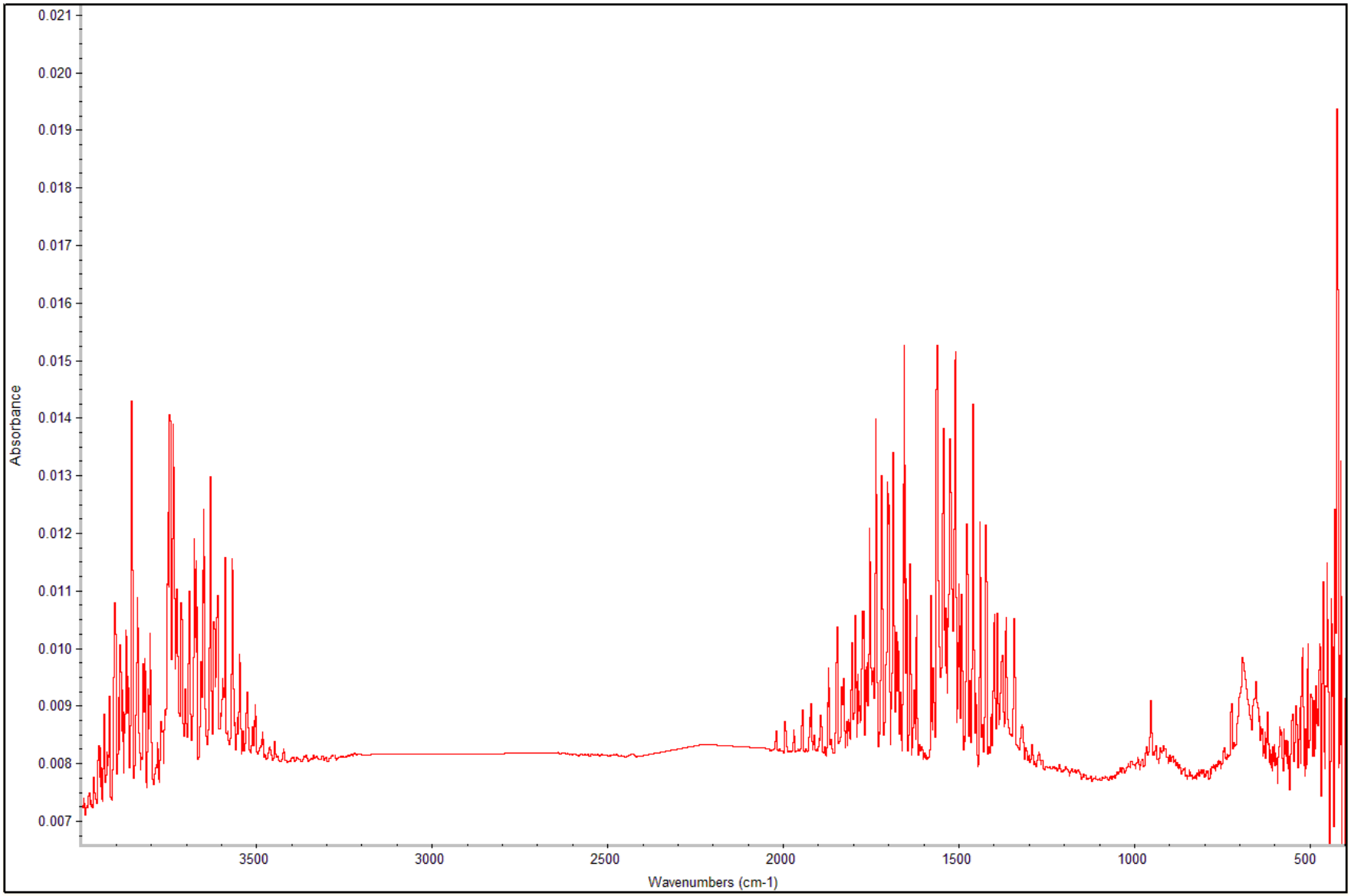
TGA/FTIR – Use Spectral Tools to Your Advantage – i.e. Subtract Interferences

- One of the most common by products of the TGA experiment is water vapor.
- Because of the ubiquitous nature of water vapor, and its spectral properties, it often interferes with making positive identifications of evolved gases.
- Fortunately, there is a simple solution:
  - Collect and store a background spectrum of water vapor
  - Obtain a purged background spectrum and open the furnace and collect the lab atmosphere.
  - Store it and use it to subtract from your sample spectra and also correct for over-subtractions of water that can occur occasionally.
  - FTIR software packages provide for spectral subtractions – check the directions for your particular software.

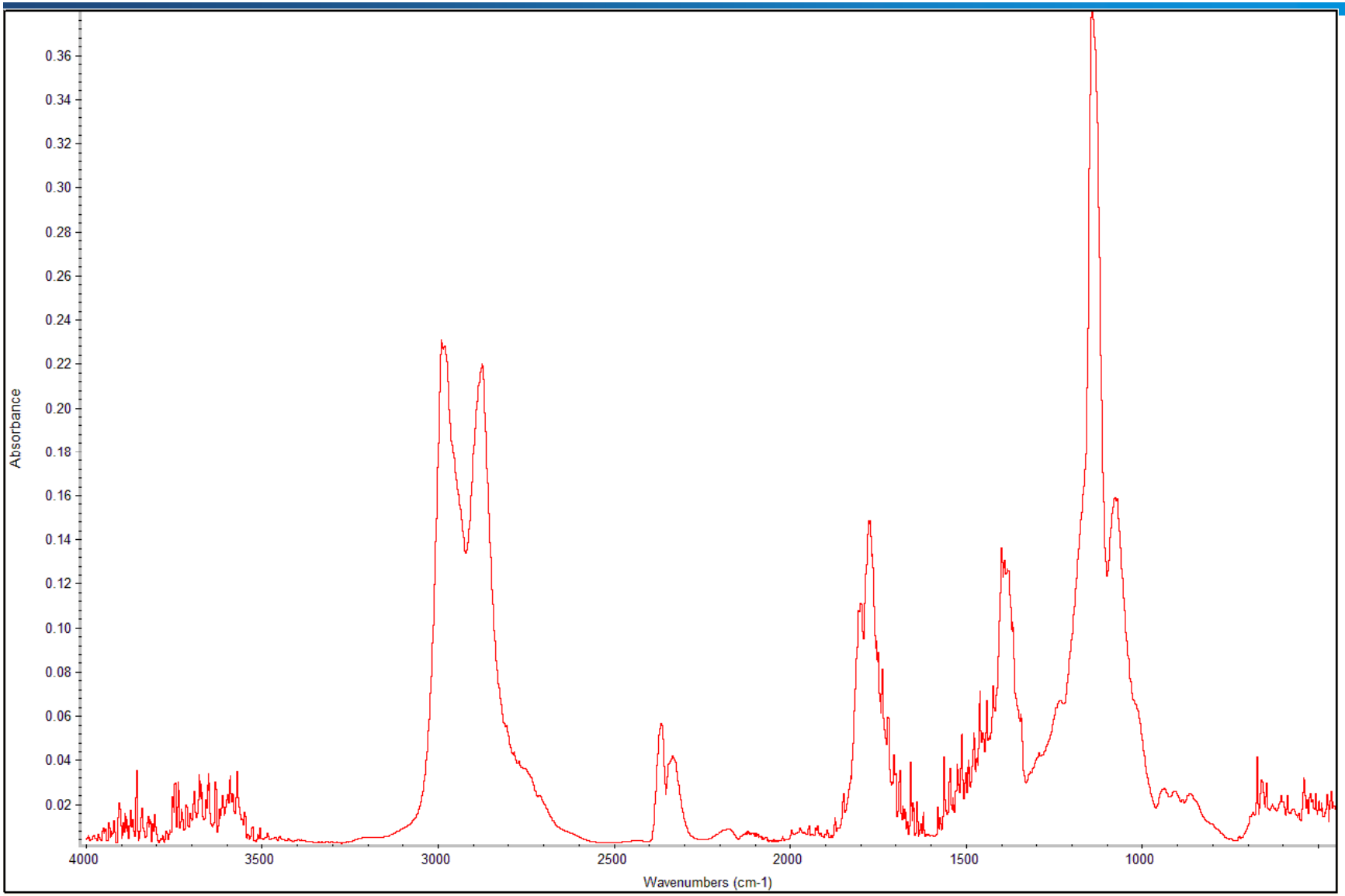
EGA Analysis: TGA/FTIR – Water Vapor and CO_2



TGA/FTIR – Water Vapor Minus CO_2 – Asymmetric Stretching and Wagging Regions Set to a Straight Line.



Example: Unknown Pharmaceutical



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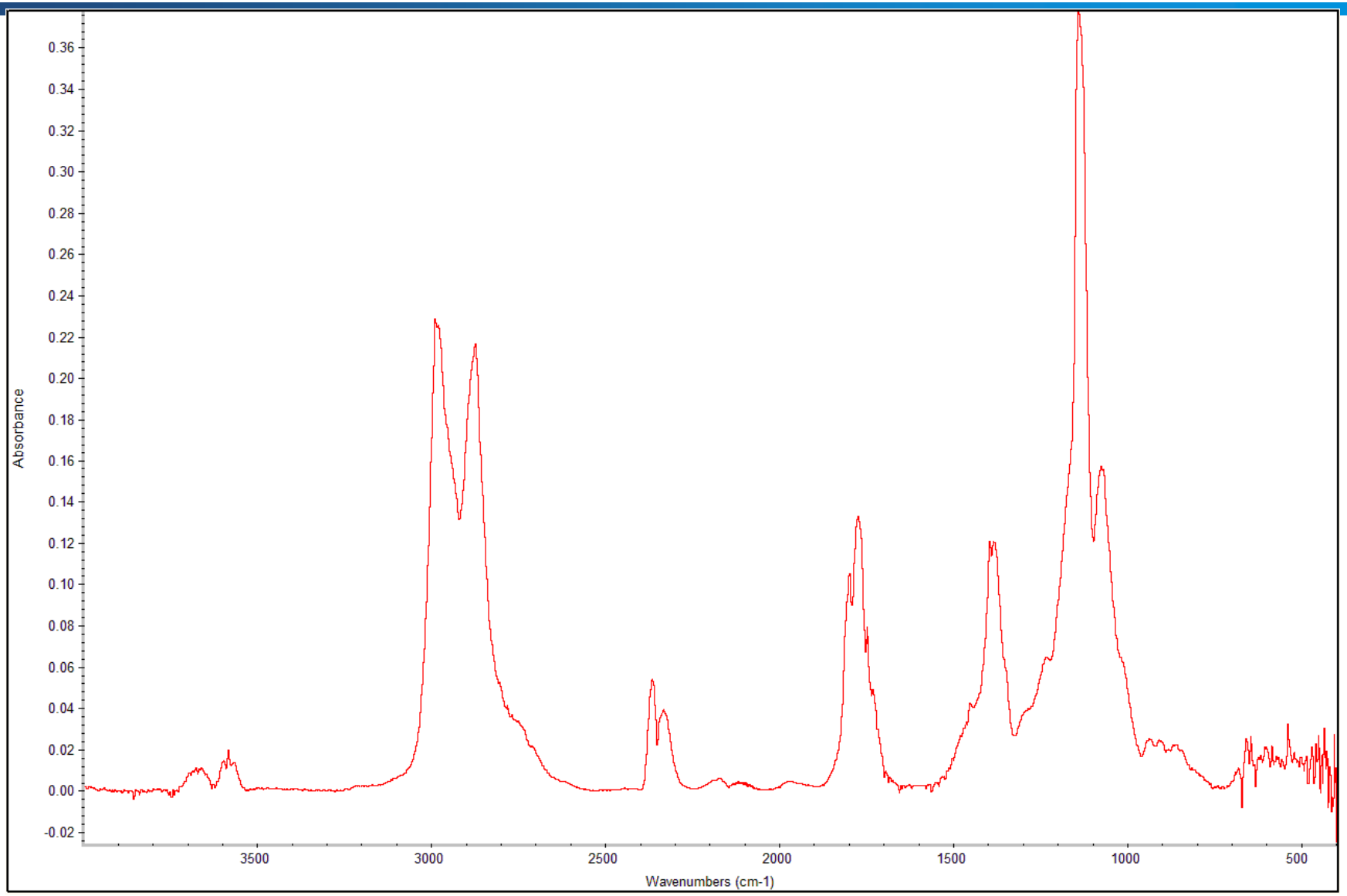
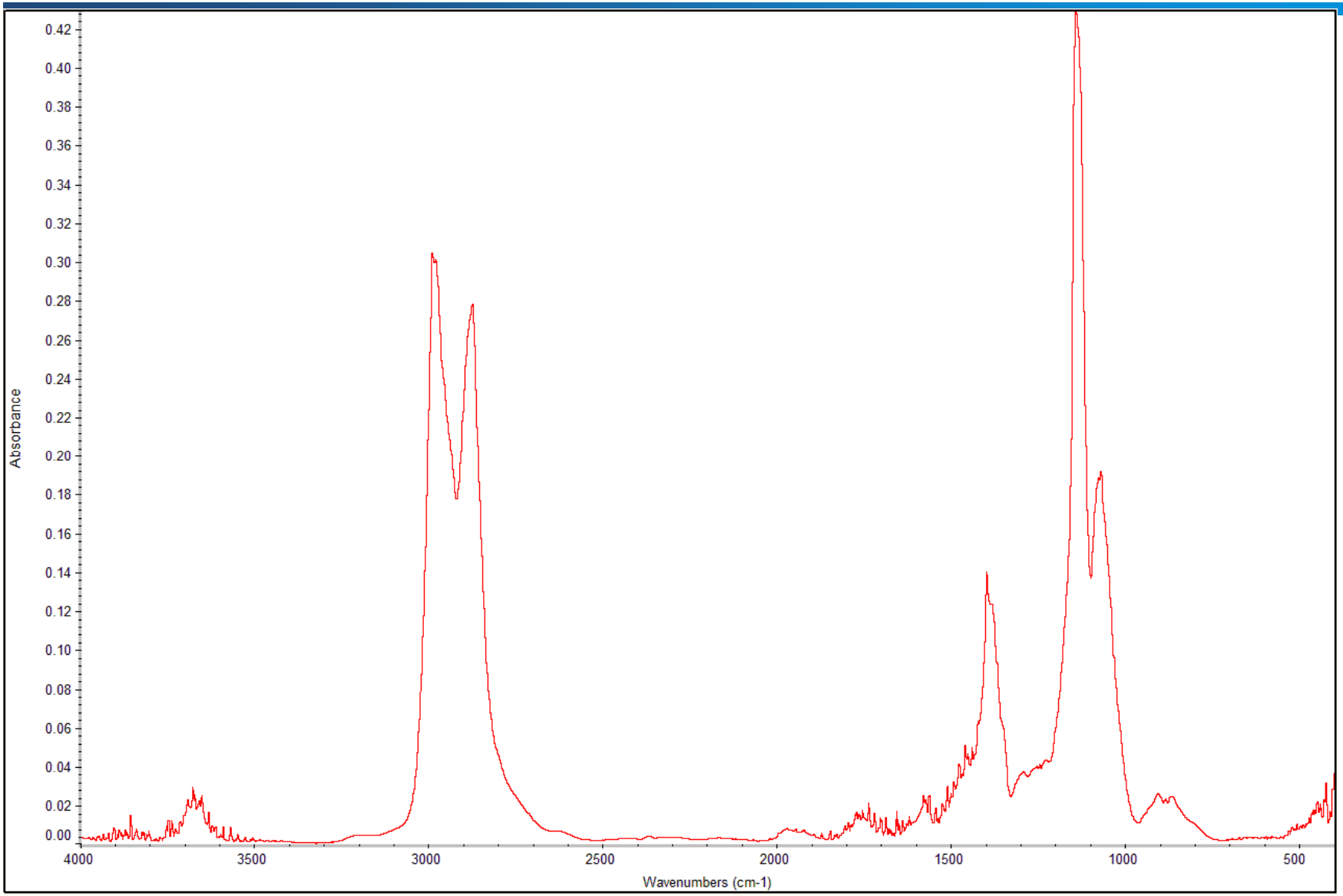


Example: Unknown Pharmaceutical – Higher Temperature



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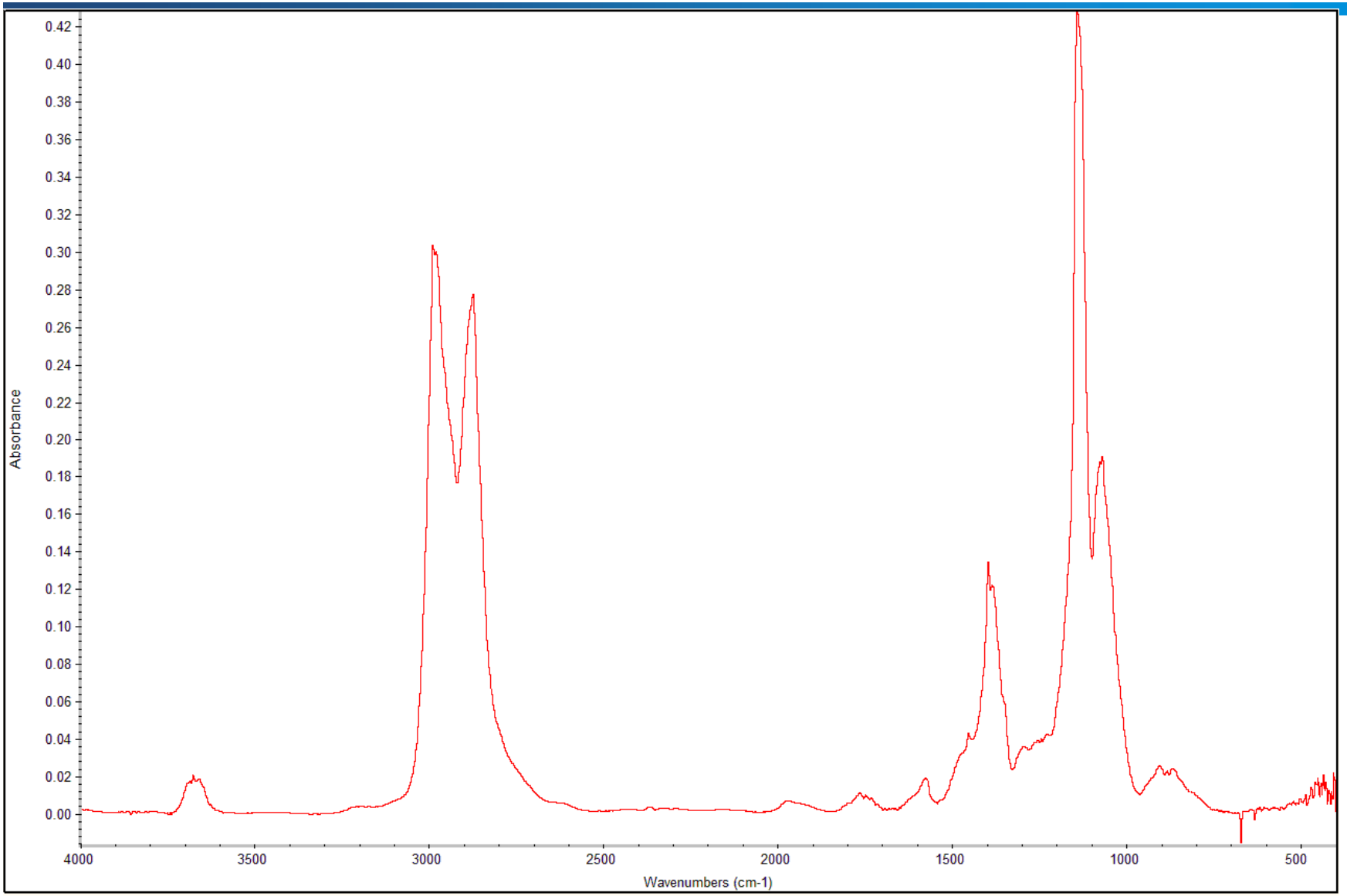
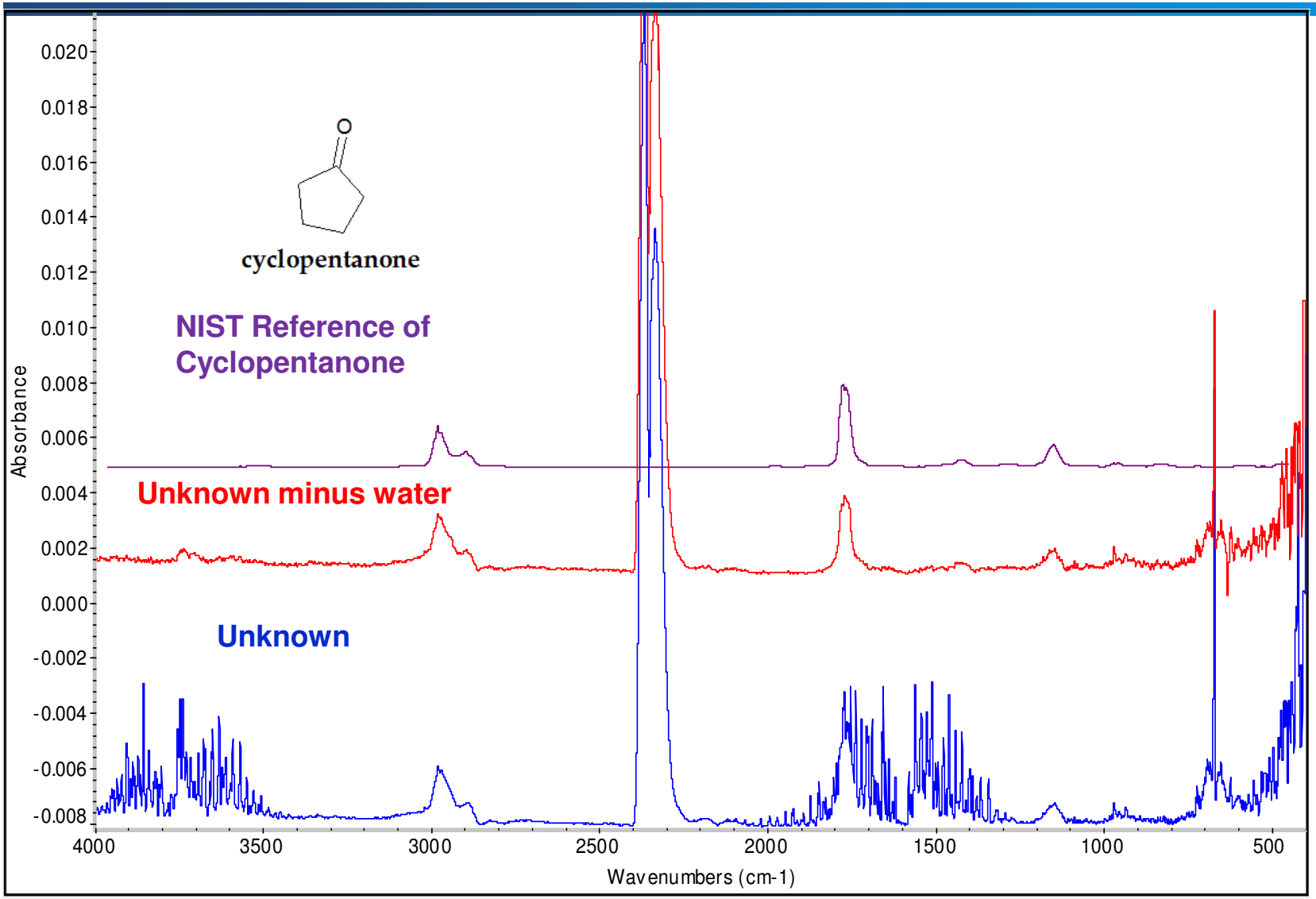
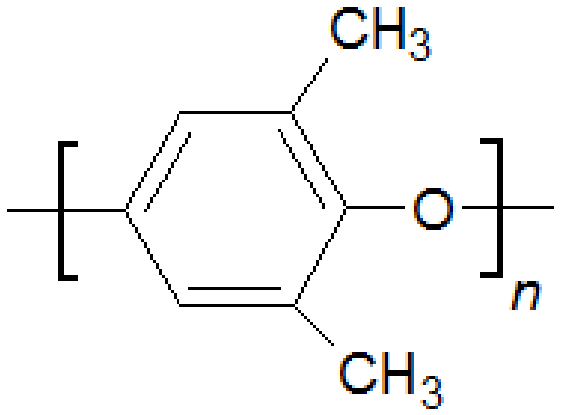


Example – Unknown Decomposition Product



Example: Analysis of Polyphenylene Oxide

- Engineering polymer
  - Heat resistant
  - Good tensile properties
  - High surface energy – paintable



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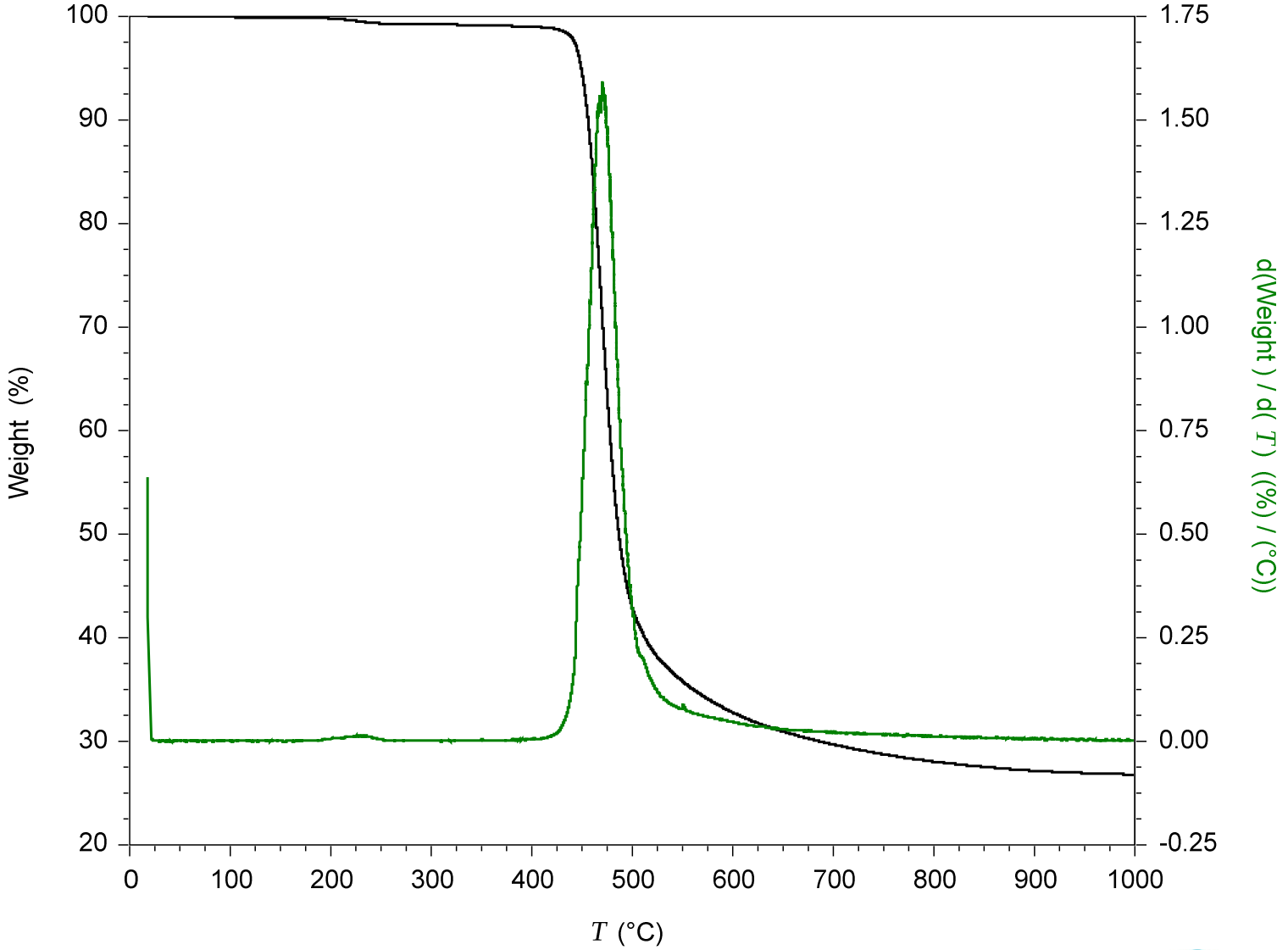

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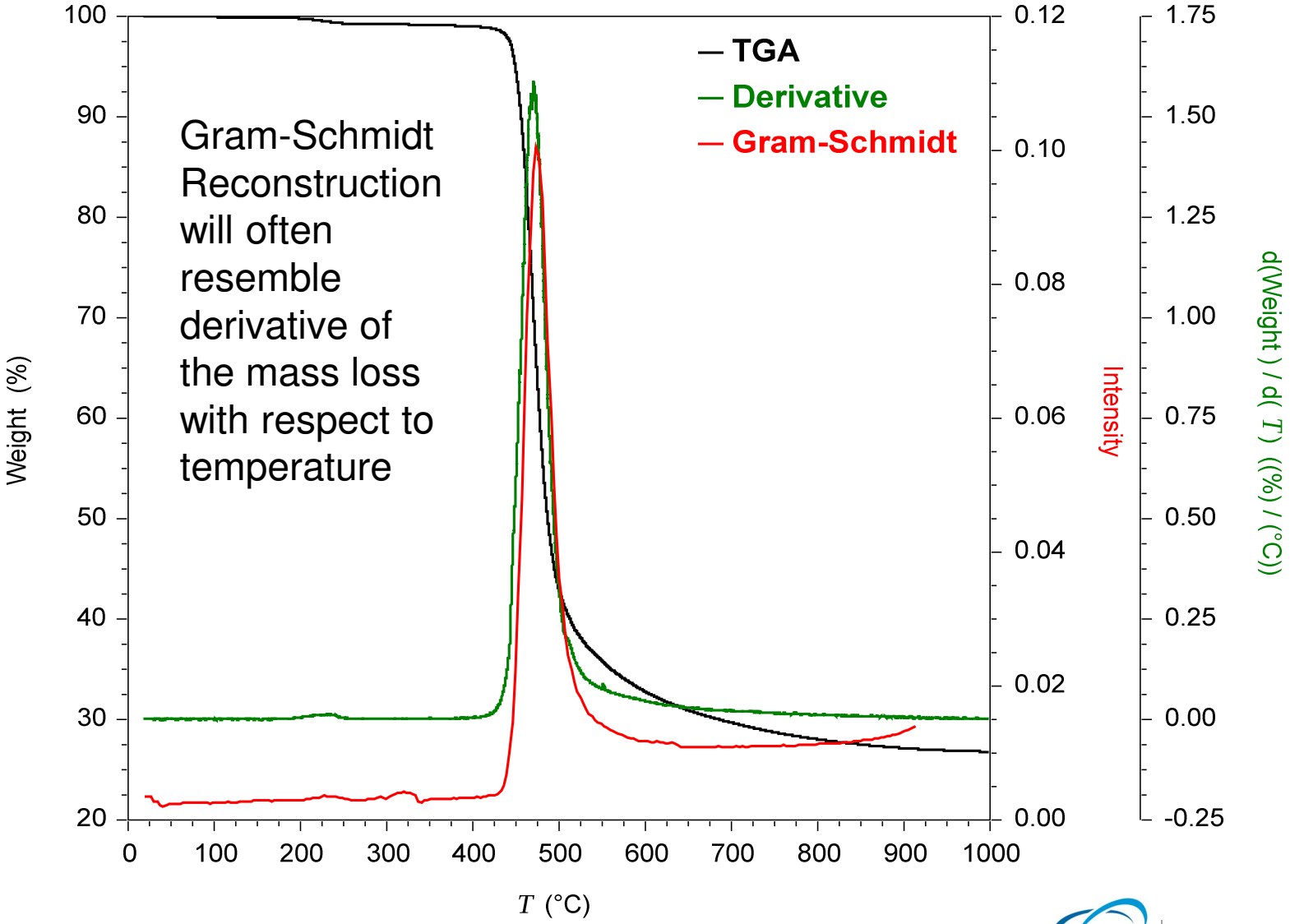

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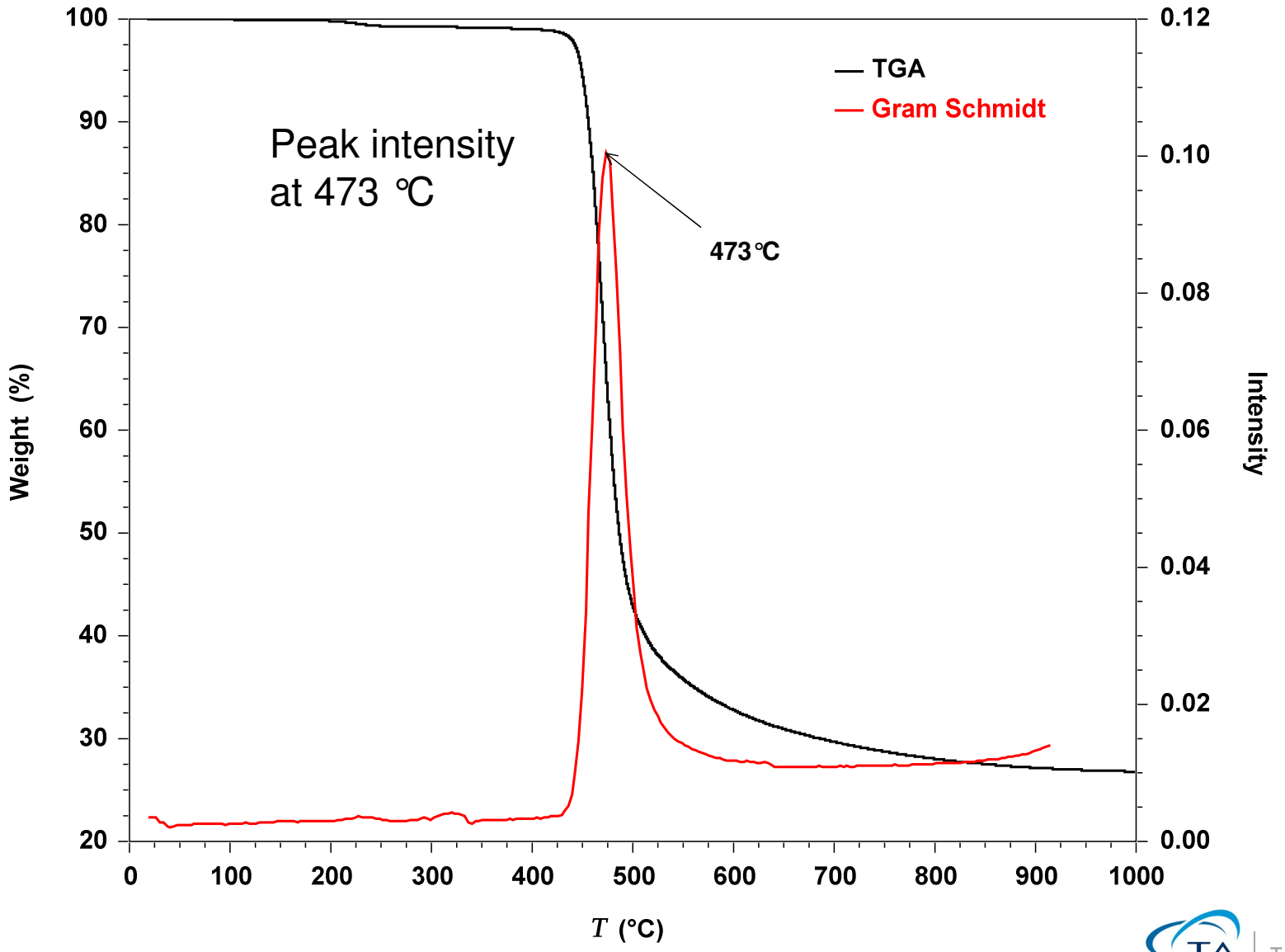

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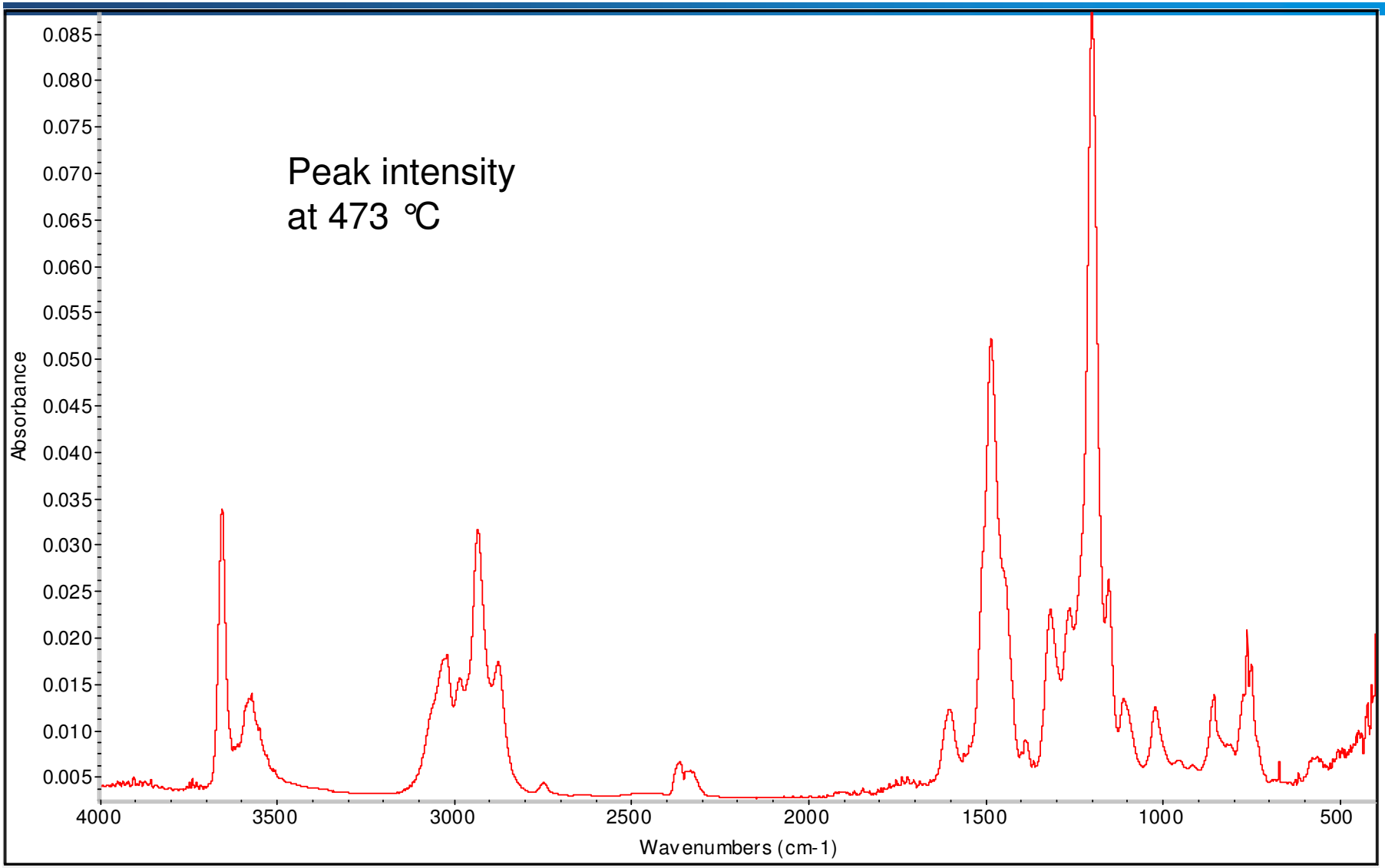

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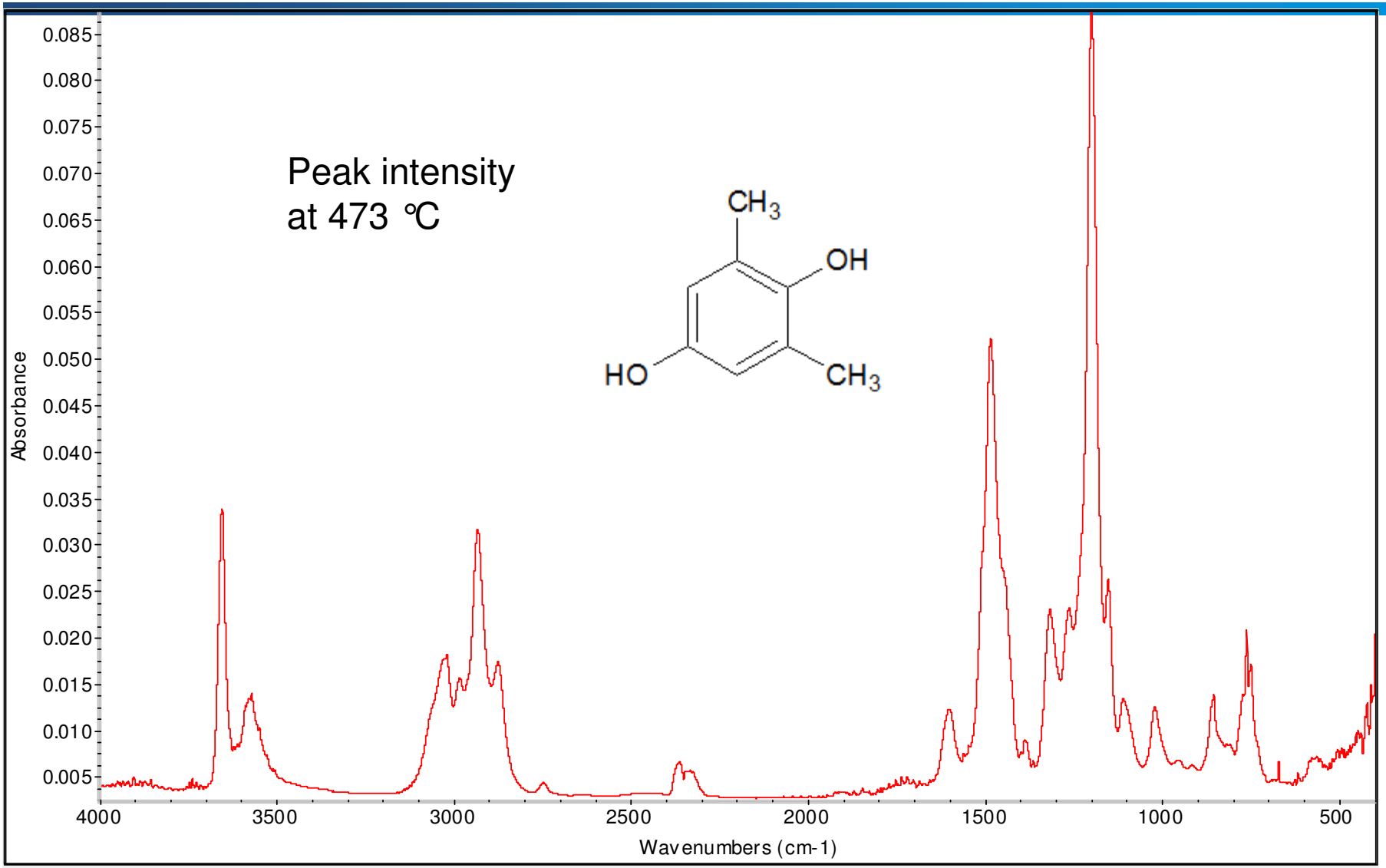

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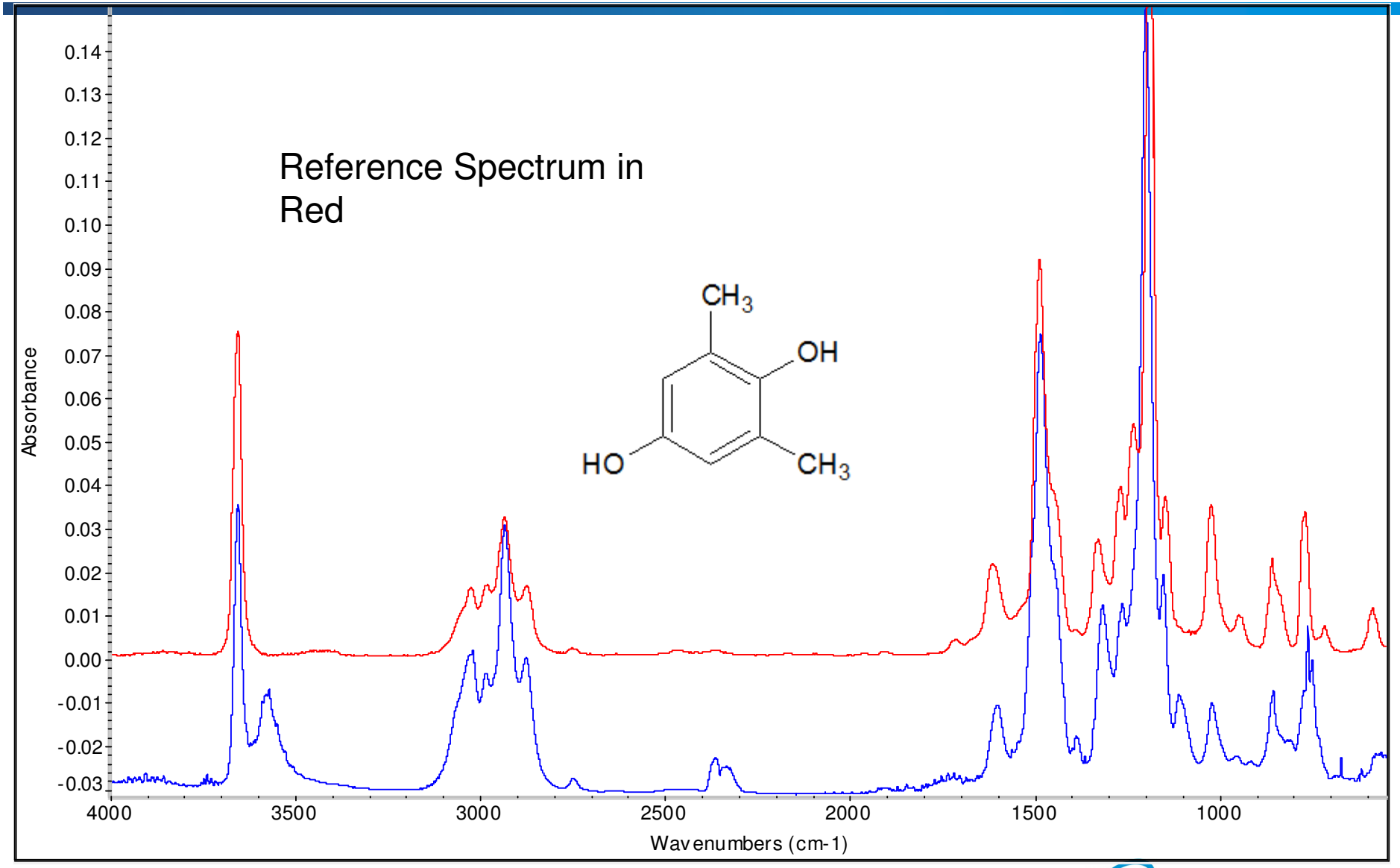

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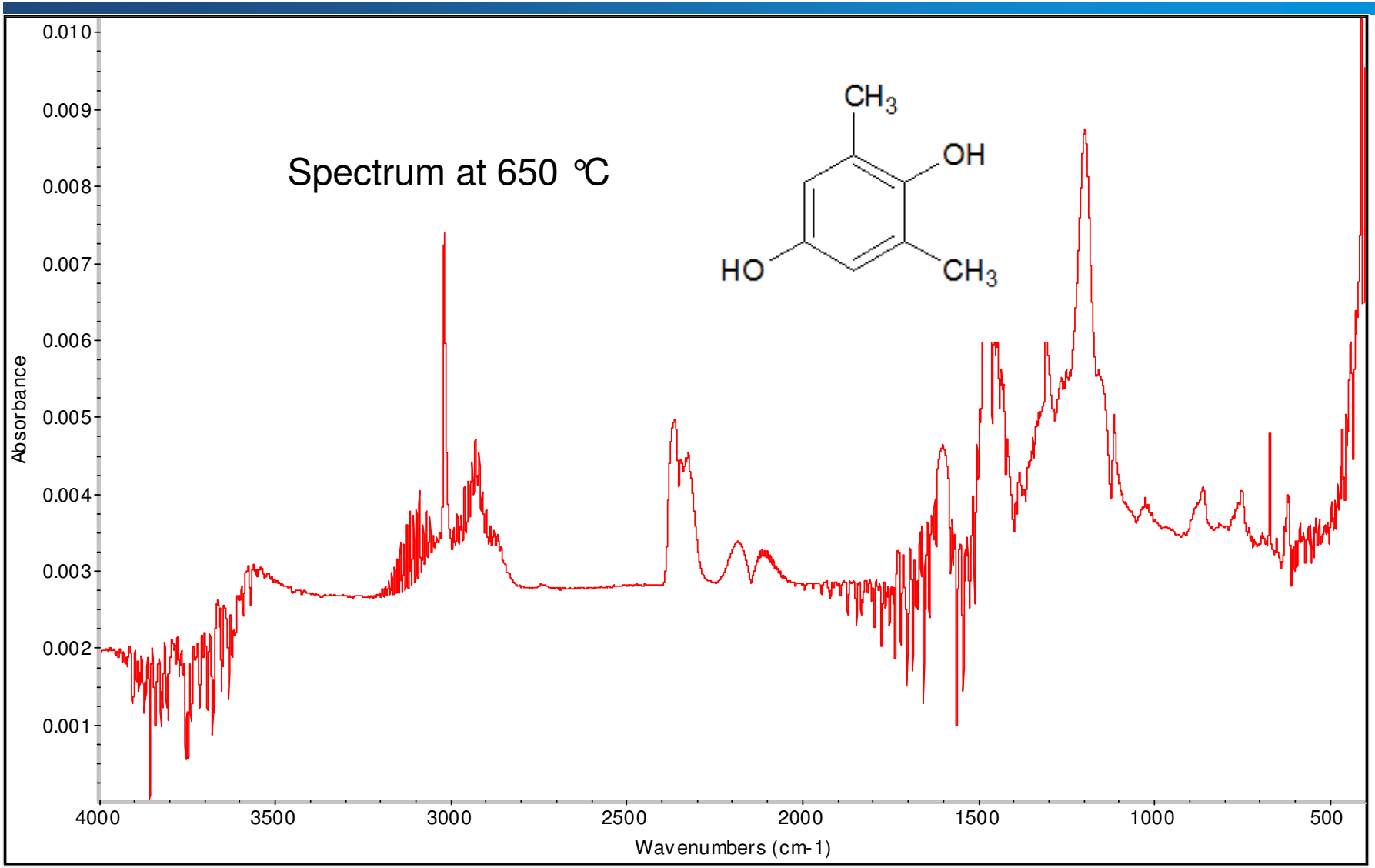

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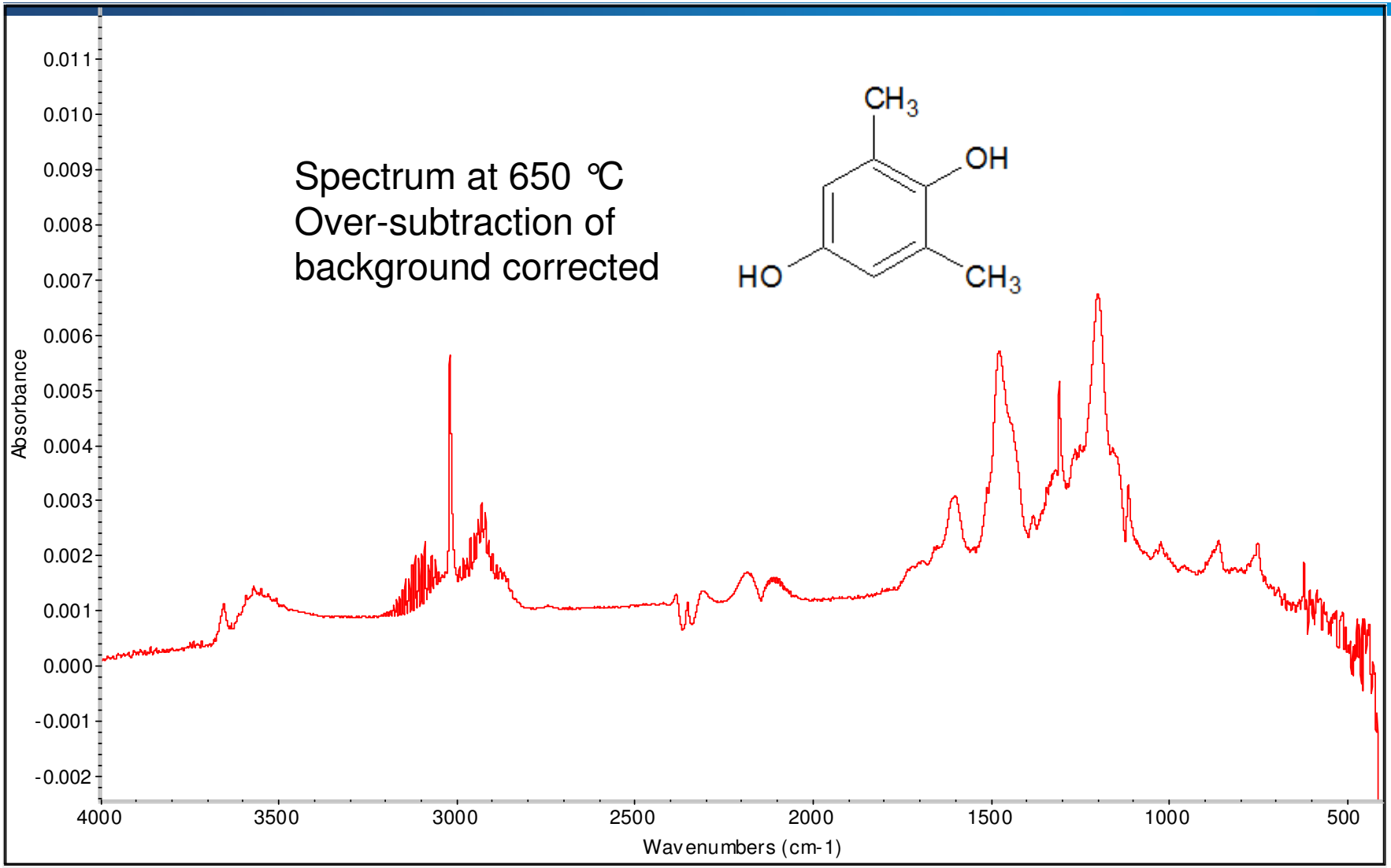

EGA Analysis: TGA/FTIR - Summary

- TGA combined with FTIR is a very powerful analytical tool.
  - Plastics
  - Pharmaceuticals
  - Fuels
  - Biomass
  - Hazardous Decomposition Products
- The search capabilities of ThermoFisher's Mercury and Spectra software make FTIR spectroscopy very accessible to all analytical chemists.
- Simple sample preparation

Thank You

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