

**Welcome to our E-Seminar:**

# **Pesticide Analysis in a Complex Matrix using a 2D GC/MS System**

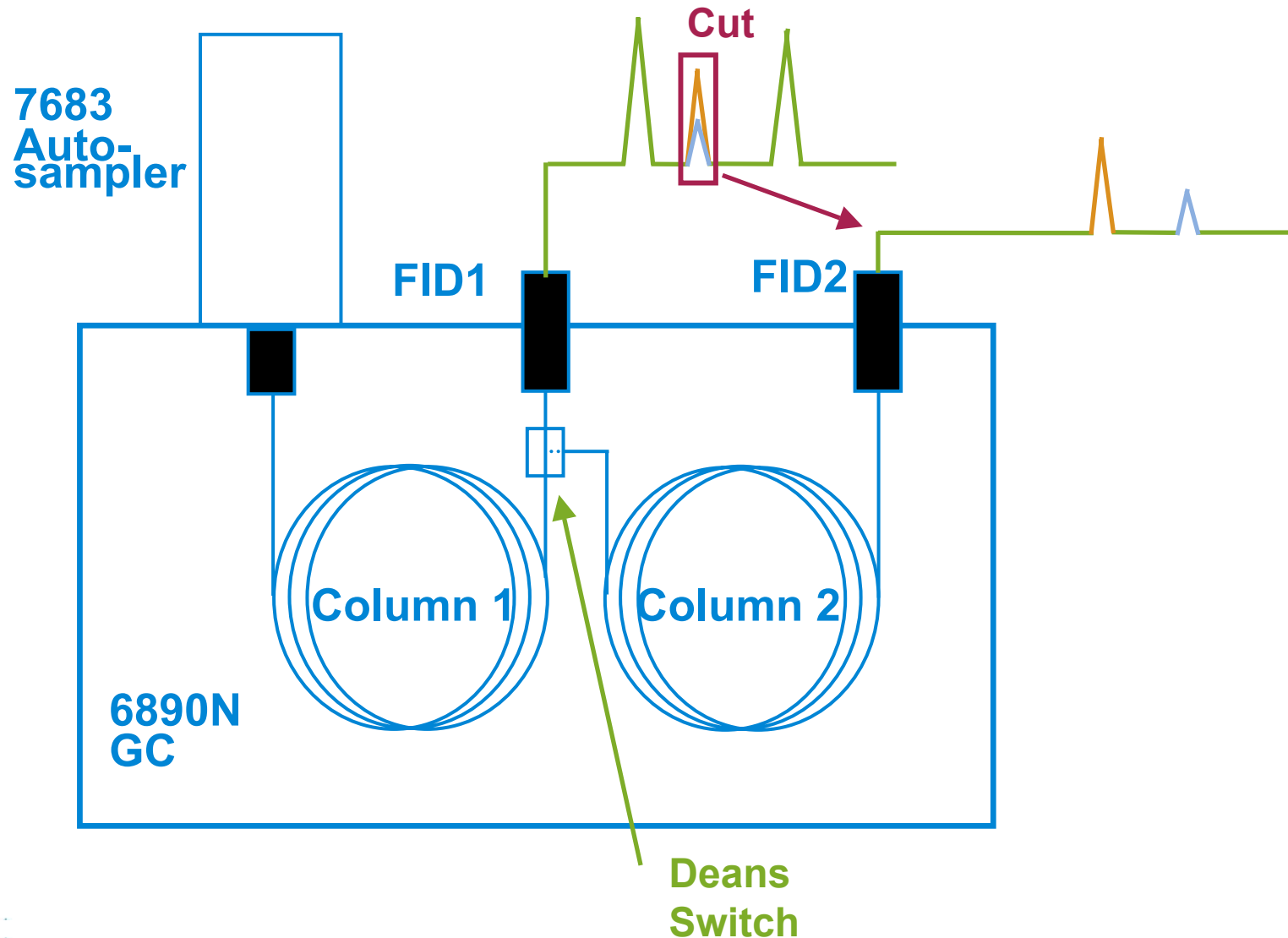


# Multidimensional (2-D) GC

- Very old (>25 yrs) but powerful separation technique
- Based on cutting peak(s) from one GC column onto another with stationary phase of different selectivity
- Compounds that co-elute with analyte on first column separate from analyte on second column
- Example pairs of complimentary phases:
  - HP-5MS (low polarity) with DB-17MS (polar)
  - HP-5MS (low polarity) with Cyclosil (chiral)



# “Simplified” 2-Dimensional GC

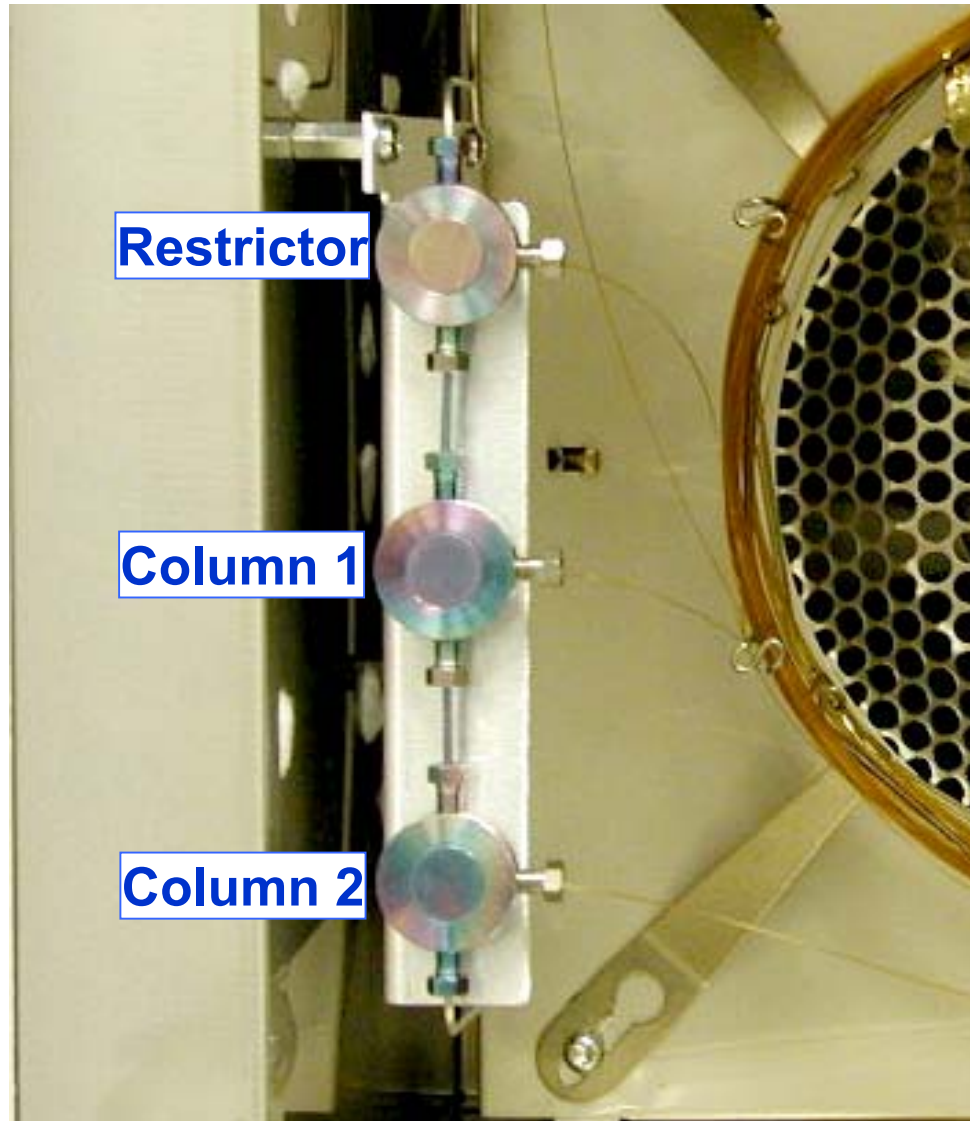


# Why 2-D GC? What's Changed?

- **6890 Simplified 2-D GC systems are much easier to use**
  - Column connections are easier, zero dead volume, inert, and reliable
  - Balancing gas flows done with EPC and Flow Calculator
  - Retention time drift greatly reduced with modern oven and EPC
  - Inertness problems with switch hardware eliminated with surface coatings
  - Because RT control is so tight and the switch is so quick, multiple ovens and cryo focusing devices can often be avoided



# Deans Hardware



Restrictor

Column 1

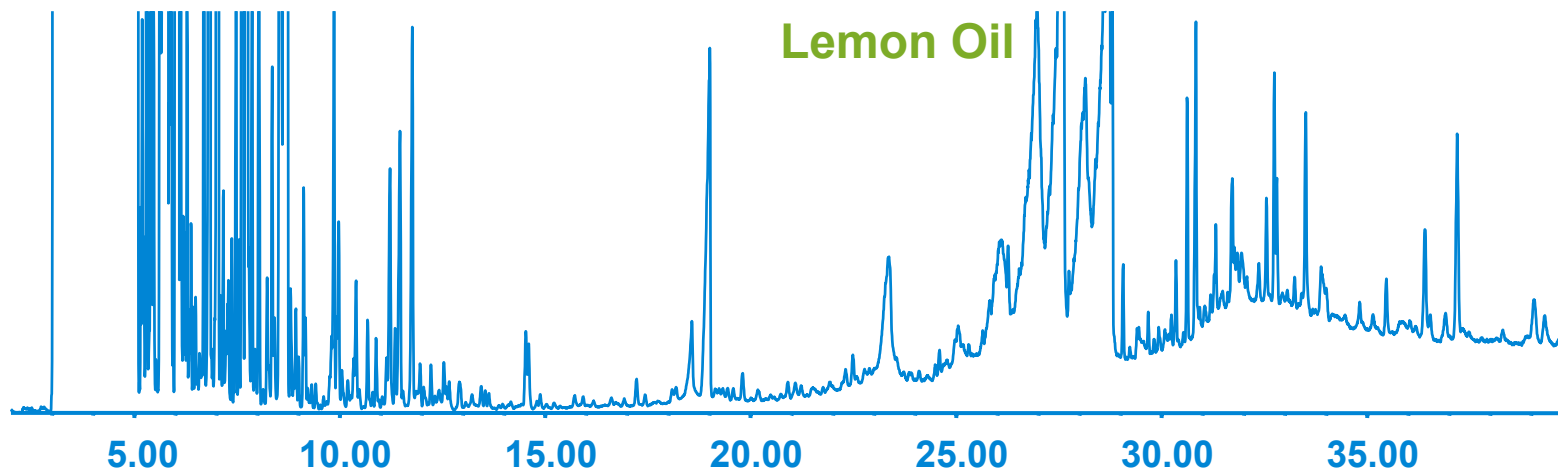
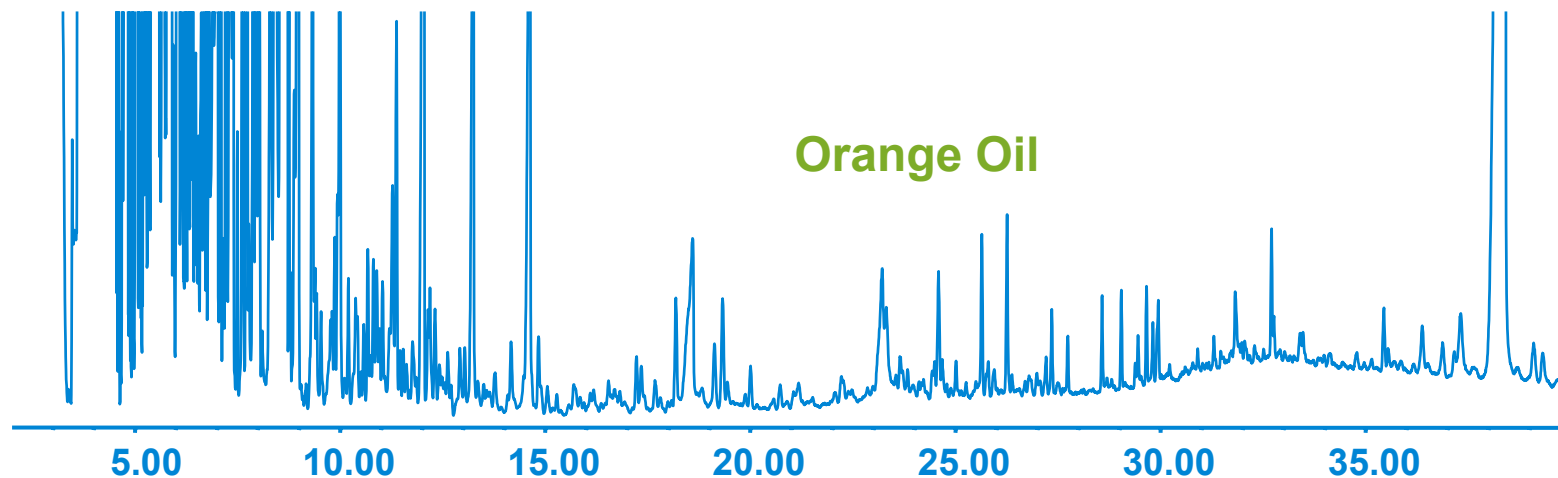
Column 2

# Problems with OP Pesticides

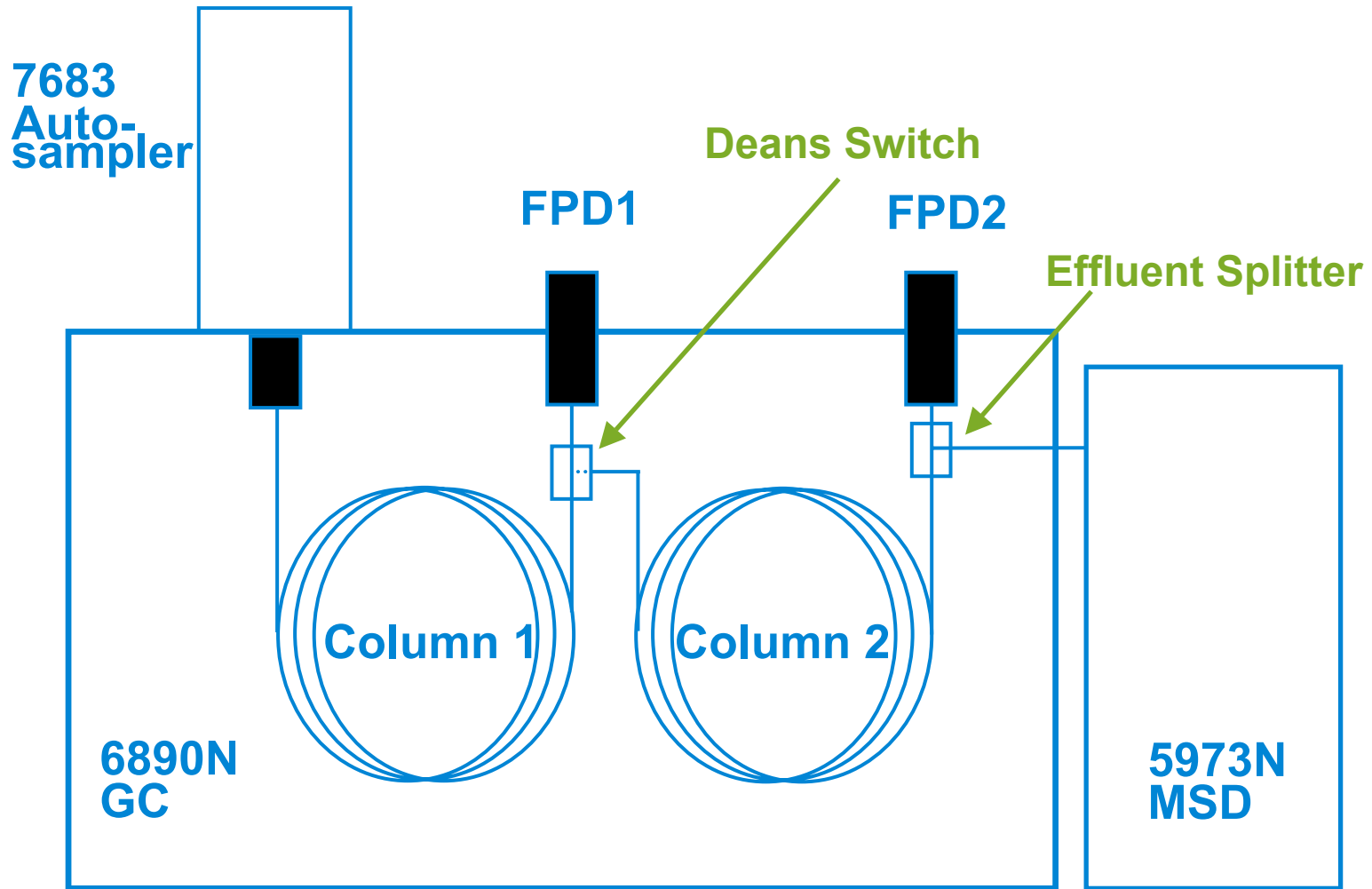
- Organophosphorus (OP) pesticides are often present in citrus oils and need to be measured
- Citrus oil matrix is very complex and causes serious chromatographic interferences with identification and confirmation of OPs
- GC screening for OP compounds with phosphorus selective detector (FPD or AED) is useful, but offers limited identification and confirmation capability
- GC-MS would be desirable for identification and confirmation, but oil matrix interferences cause problems with spectral interferences



# Orange Oil GC-MS TIC (split 10:1)



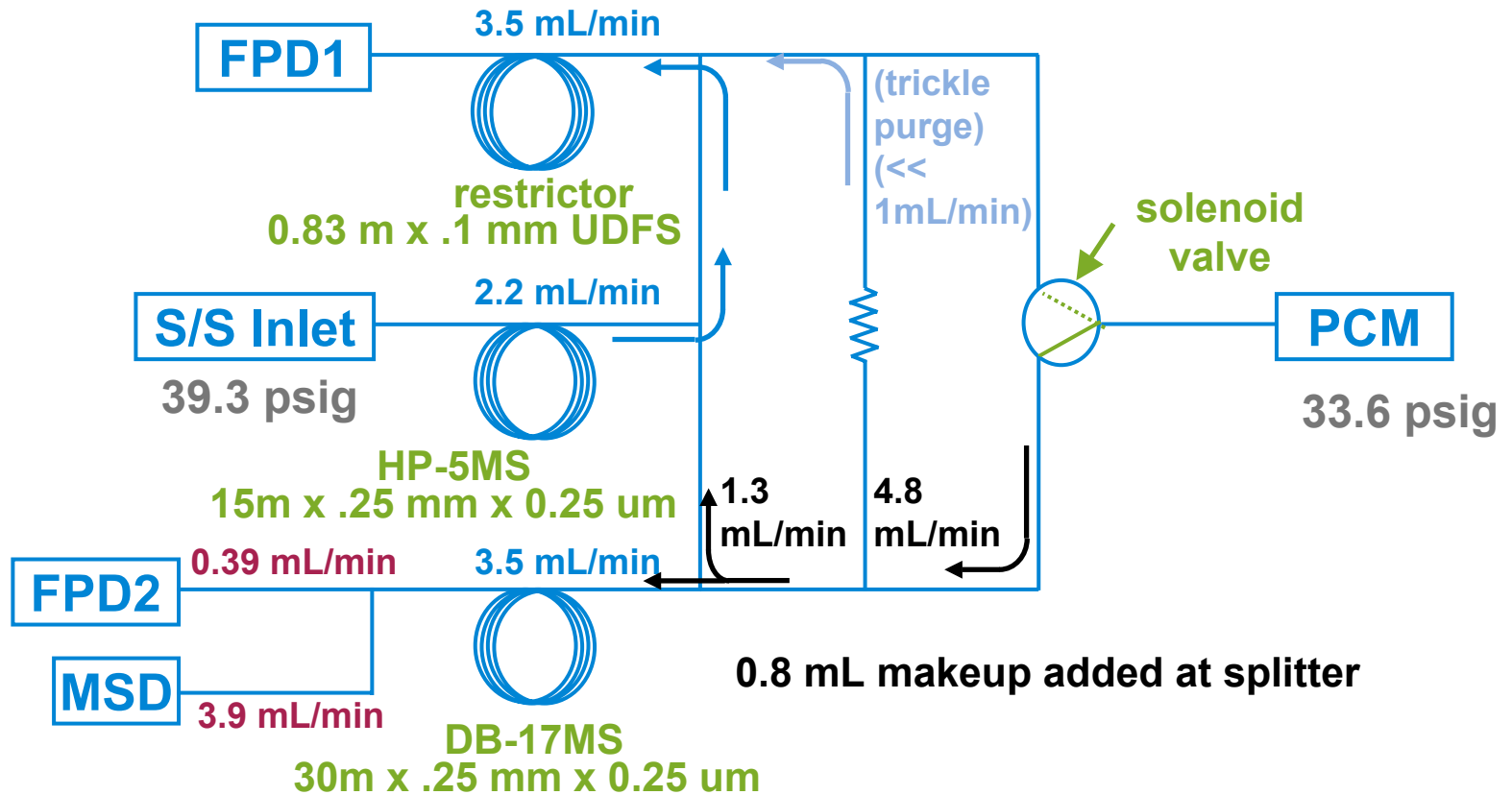
# System Configuration





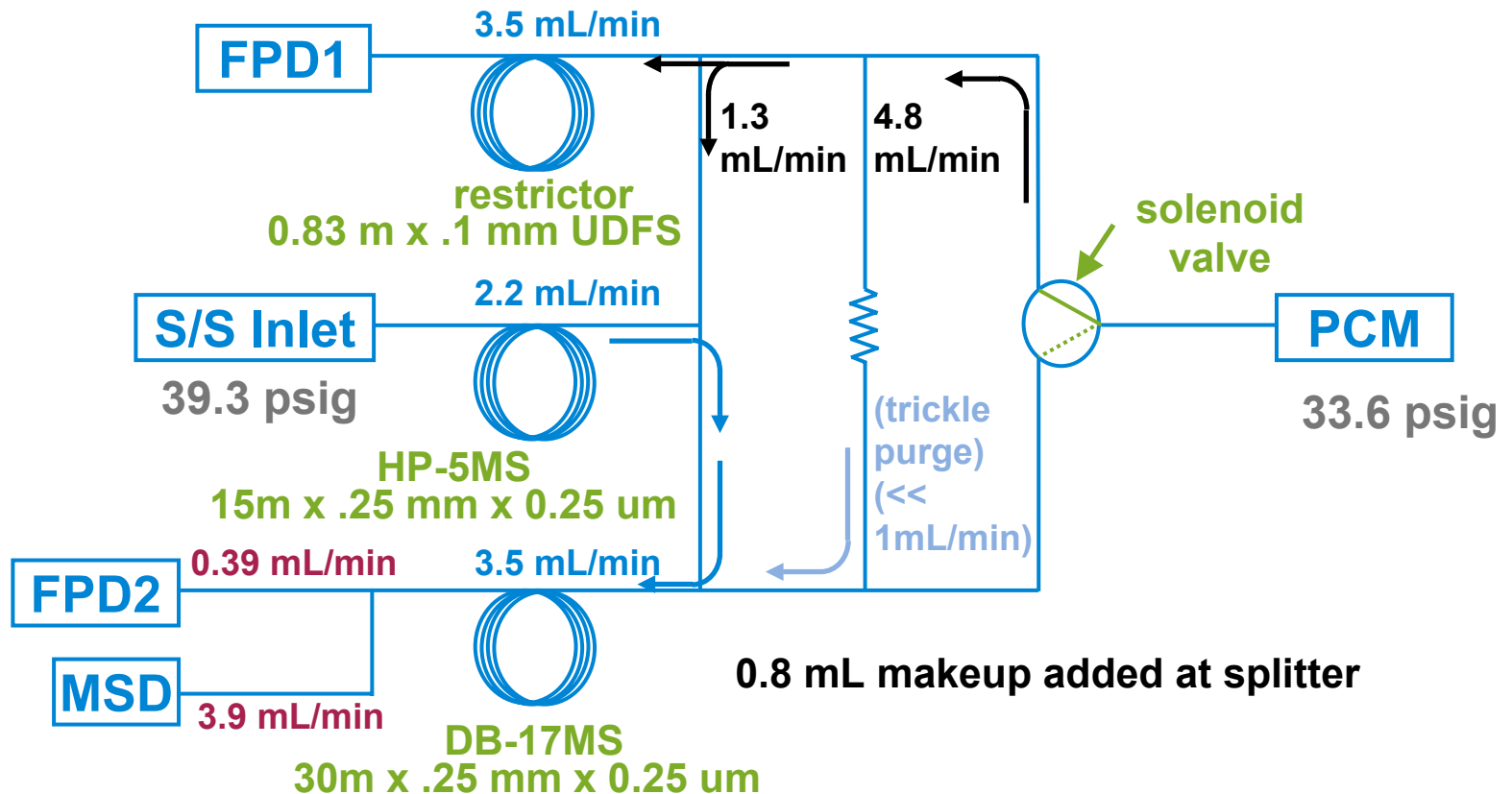
# OP Pesticides, Deans Setup, not Cutting

Effluent from second column split between MSD and FPD in 10:1 ratio



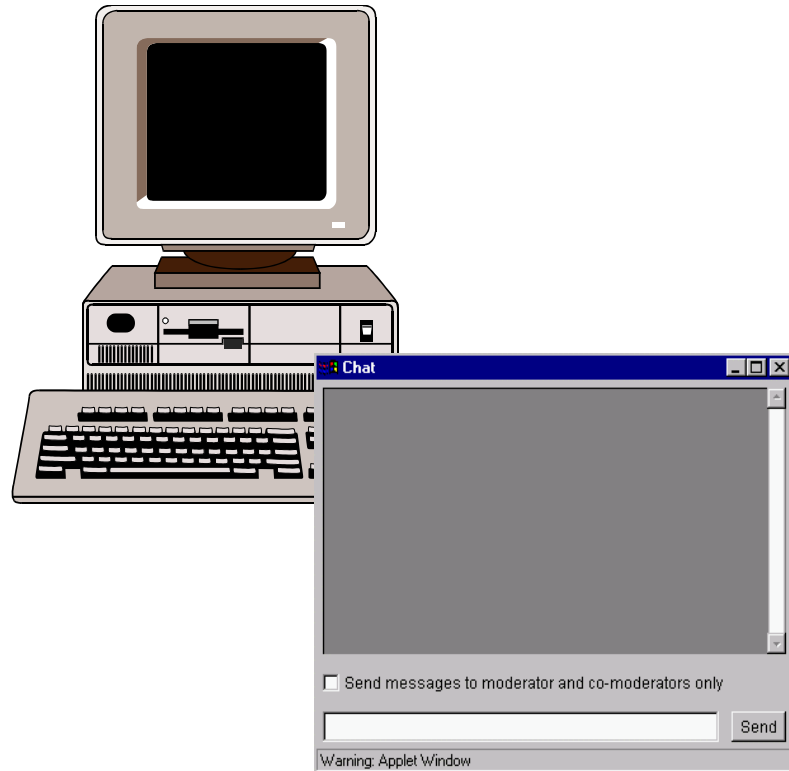
# OP Pesticides, Deans Setup, Cutting

Effluent from second column split between MSD and FPD in 10:1 ratio



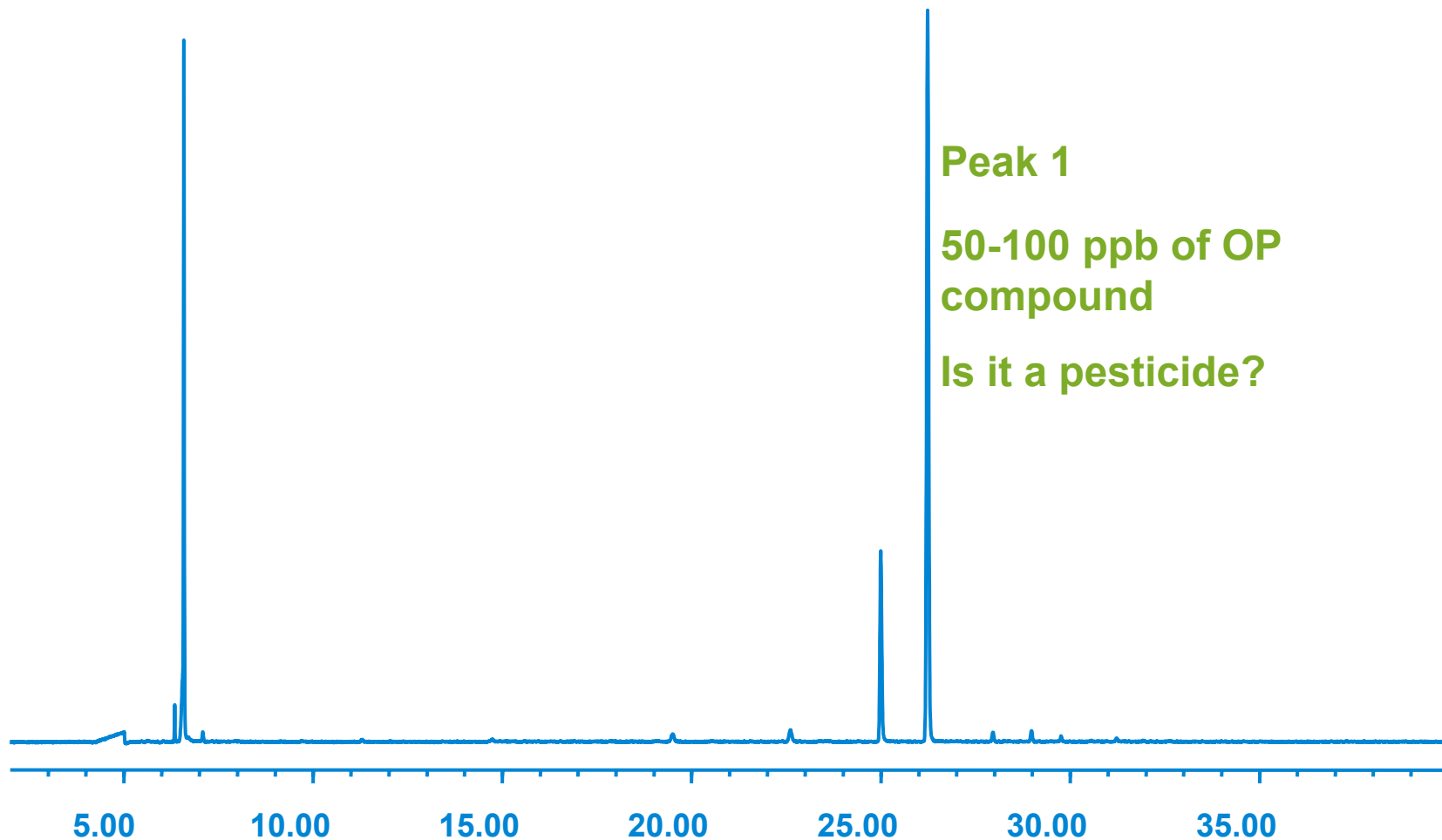
# Break Number 1

Please type your question into the Chat Box at any time during the presentation.

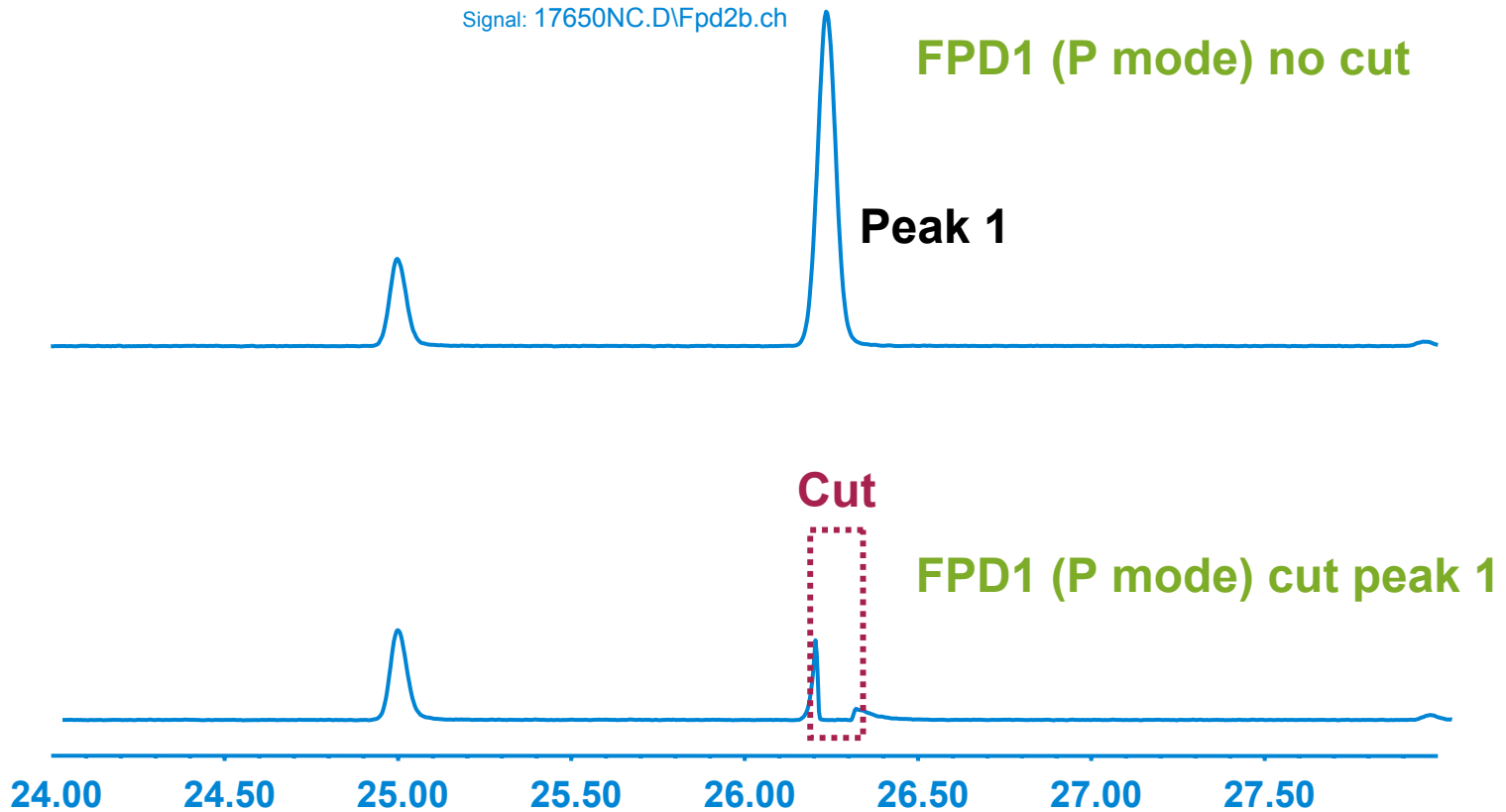


# FPD1 Screen of Orange Oil A

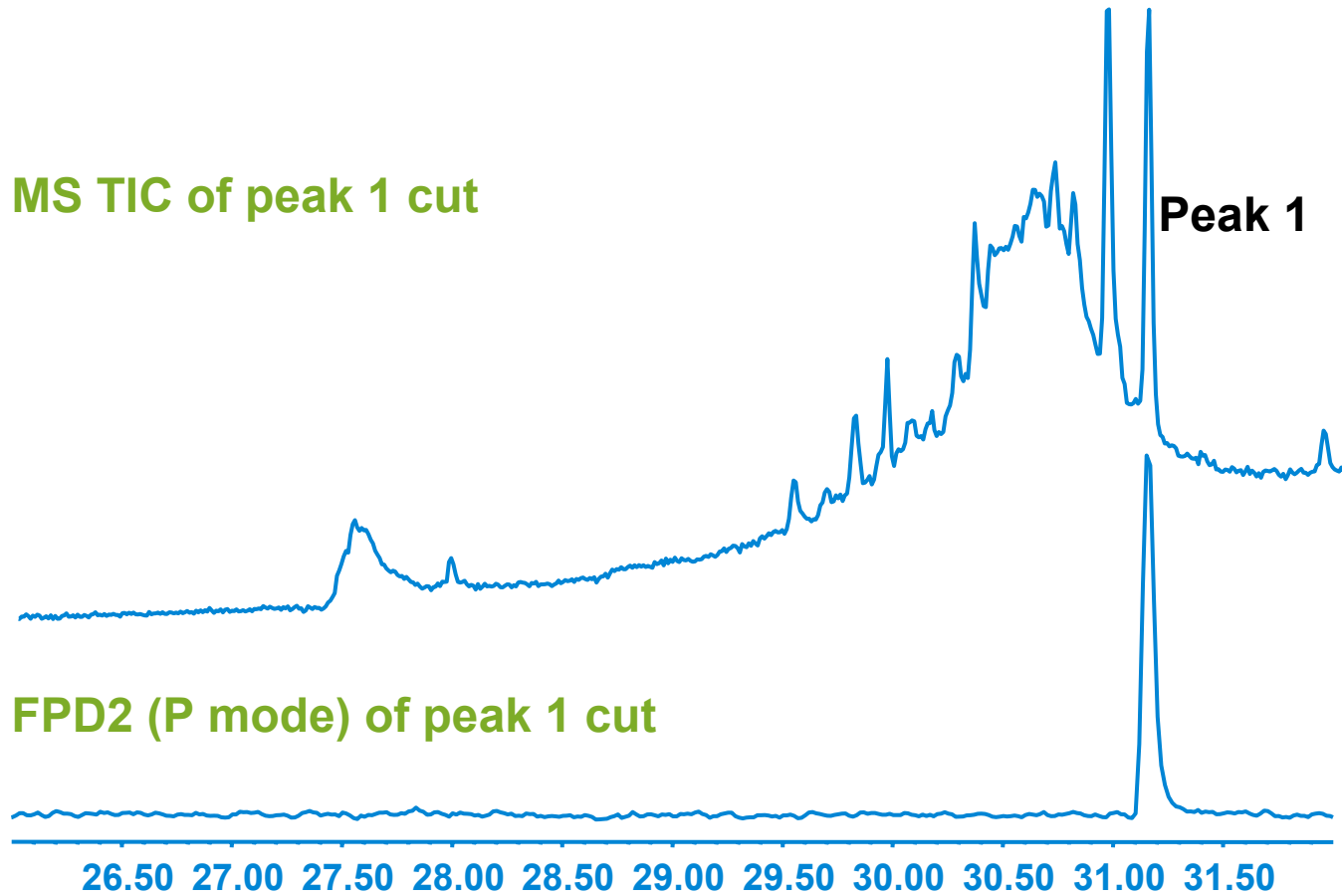
Signal: 17650NC.D\Fpd2b.ch



# FPD1 Screen of Orange Oil A

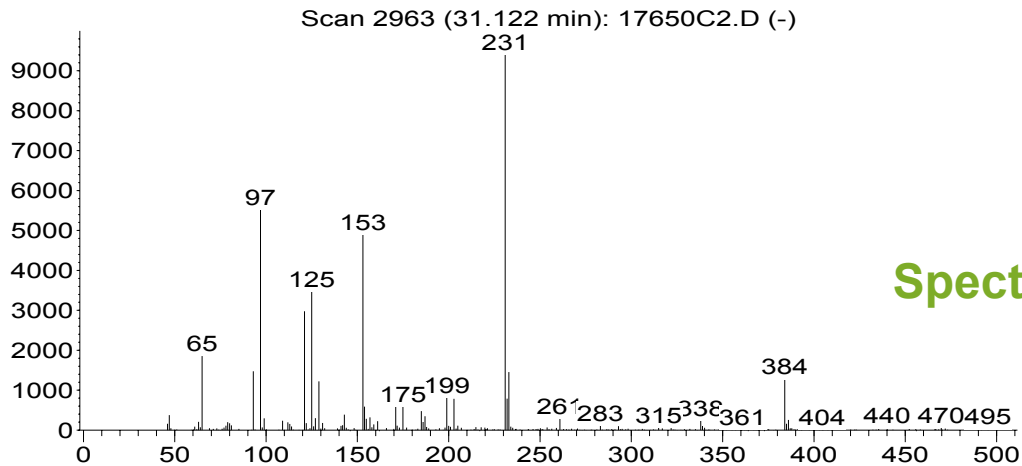


# Peak 1 Cut Results



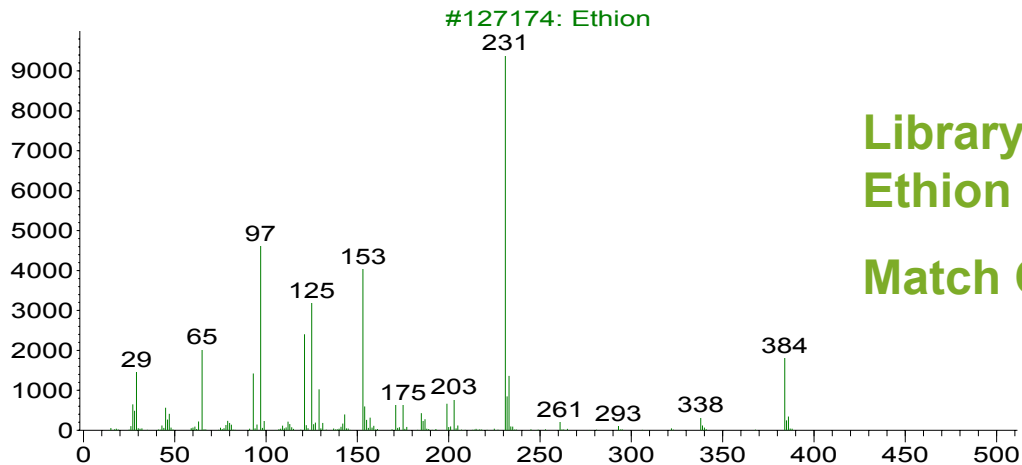
# Spectrum and Search of Peak 1

Abundance



Spectrum of peak 1

m/z-->  
Abundance



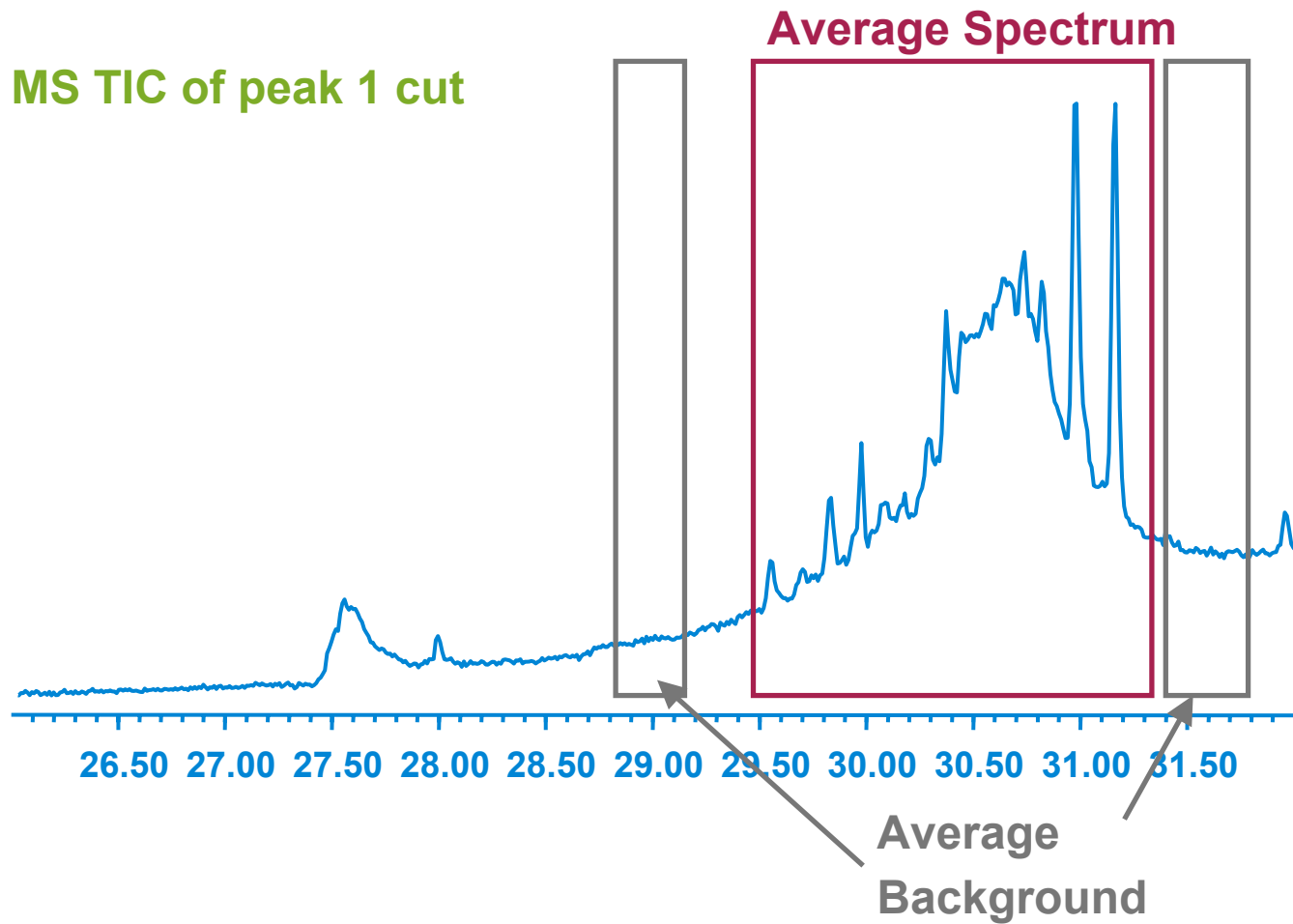
Library Match Spectrum:  
Ethion

Match Quality 99%

m/z-->



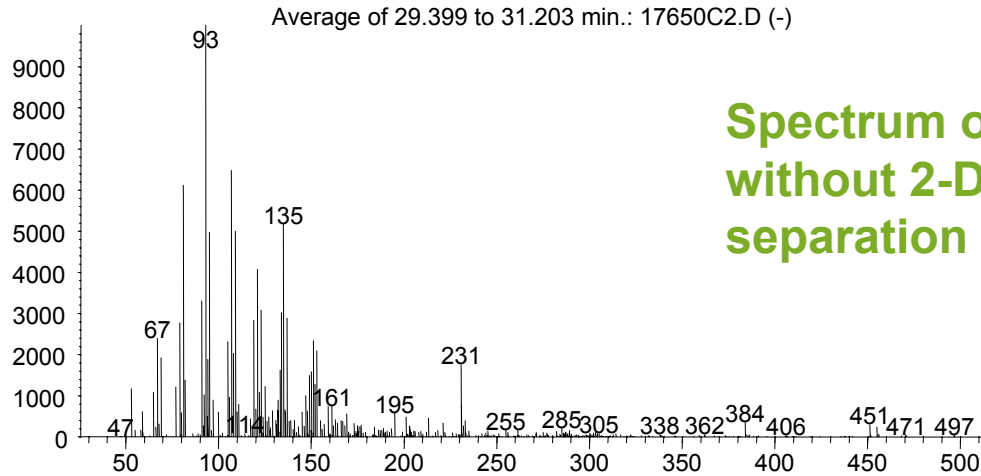
# Spectrum If 2-D Not Used





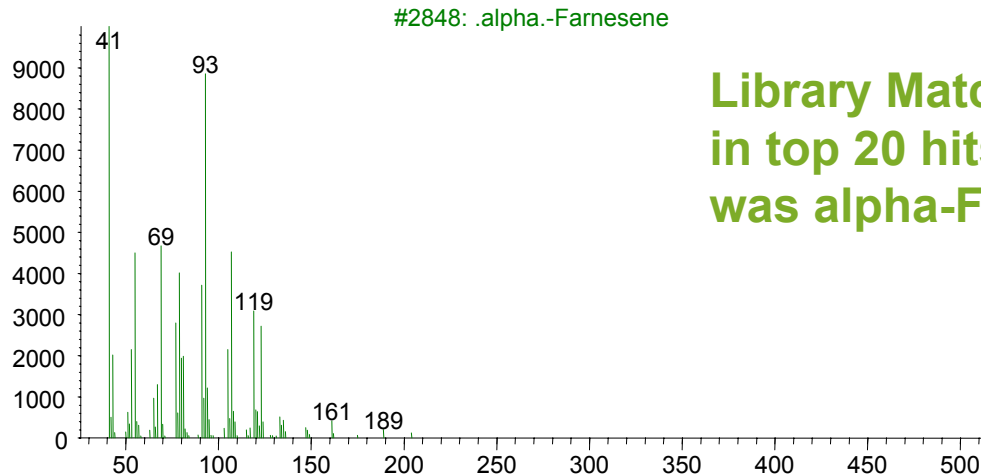
# Spectrum Unusable Without 2-D

Abundance



**Spectrum of peak 1  
without 2-D  
separation**

m/z-->  
Abundance

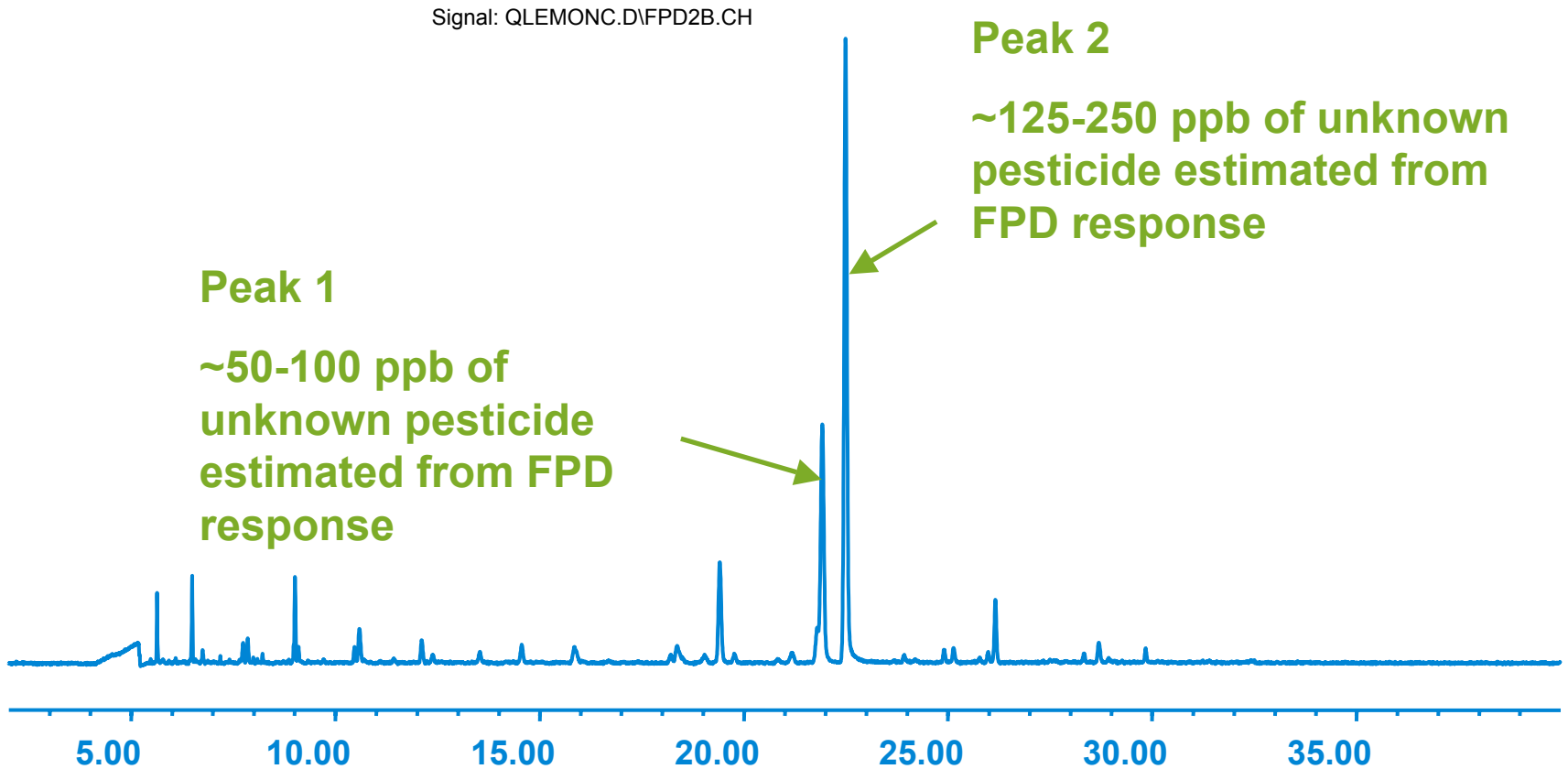


**Library Match: Ethion not listed  
in top 20 hits. Best match (30%)  
was alpha-Farnesene**

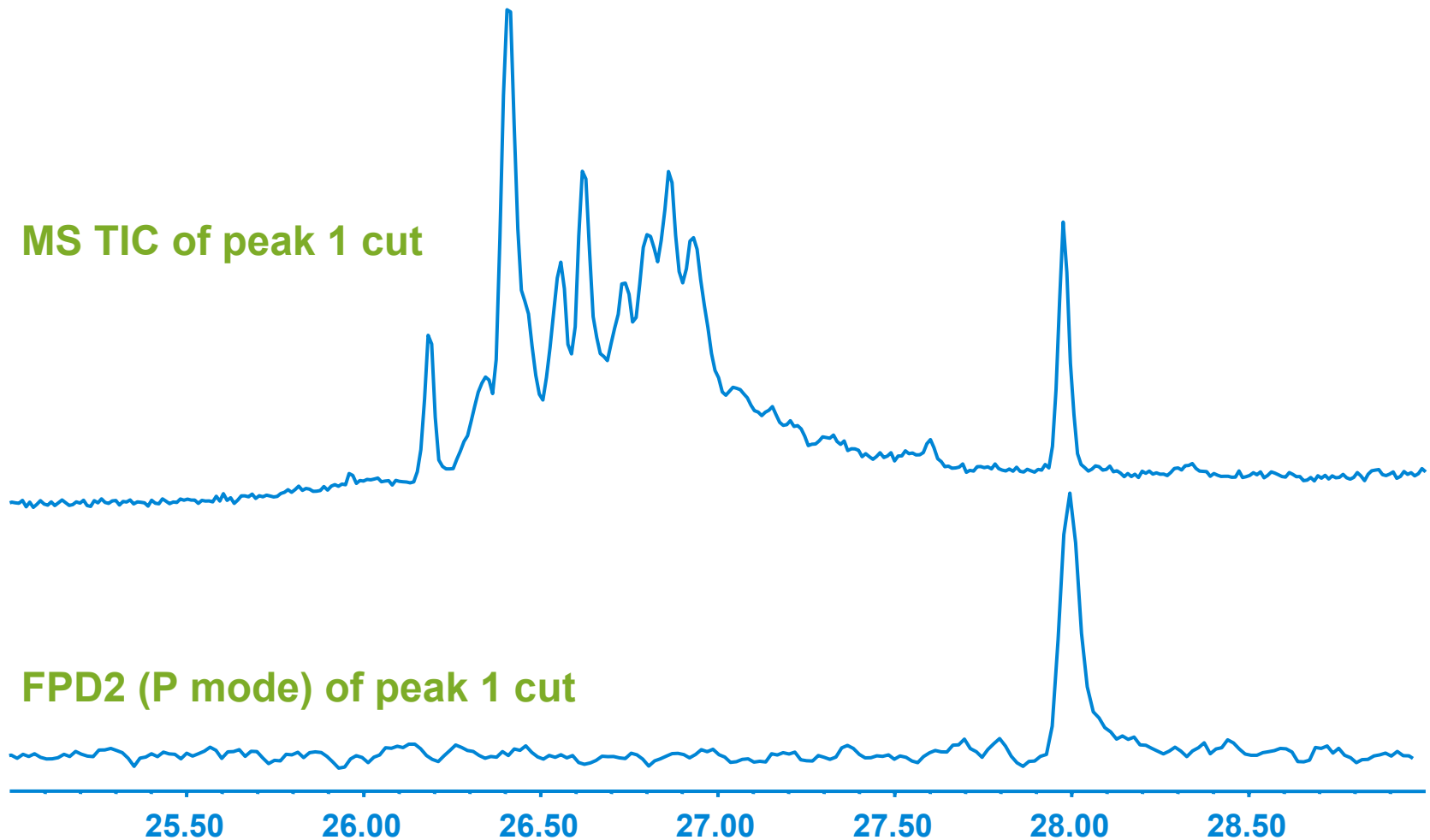
m/z-->



# FPD1 Screen of Lemon Oil A

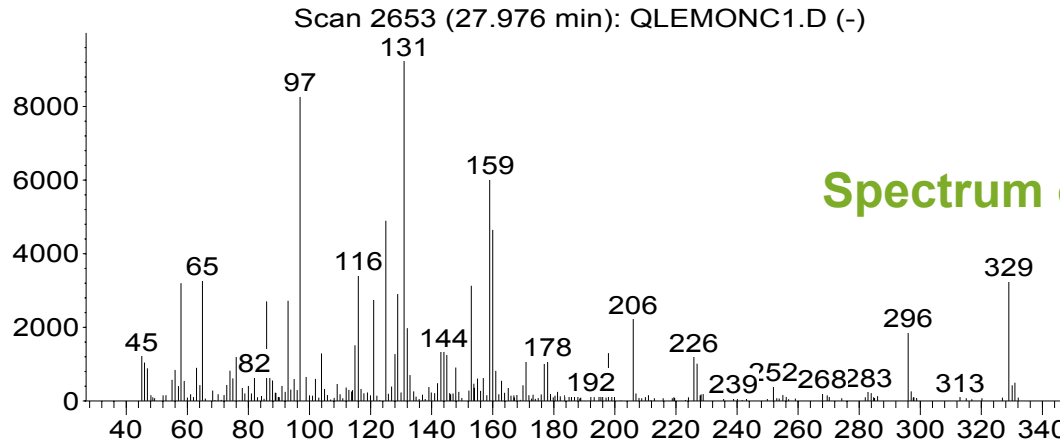


# Lemon Oil, Peak 1 Cut Results



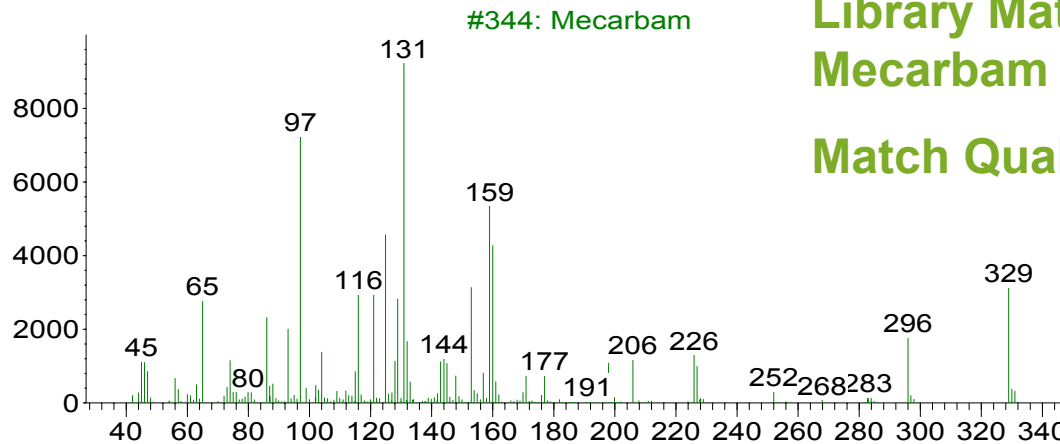
# Peak 1 is Mecarbam

Abundance



Spectrum of peak 1

m/z-->  
Abundance



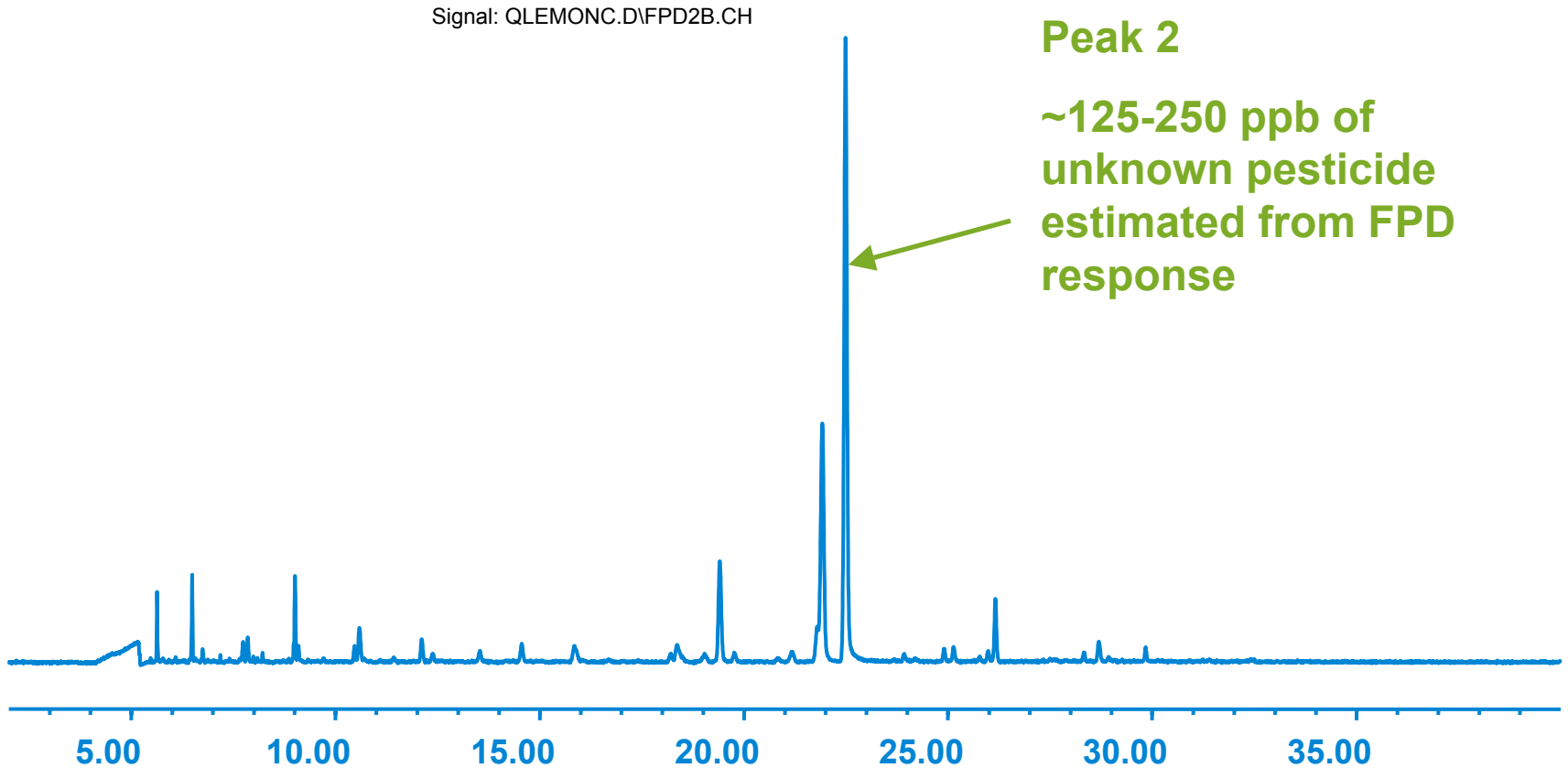
Library Match Spectrum:  
Mecarbam

Match Quality 99%

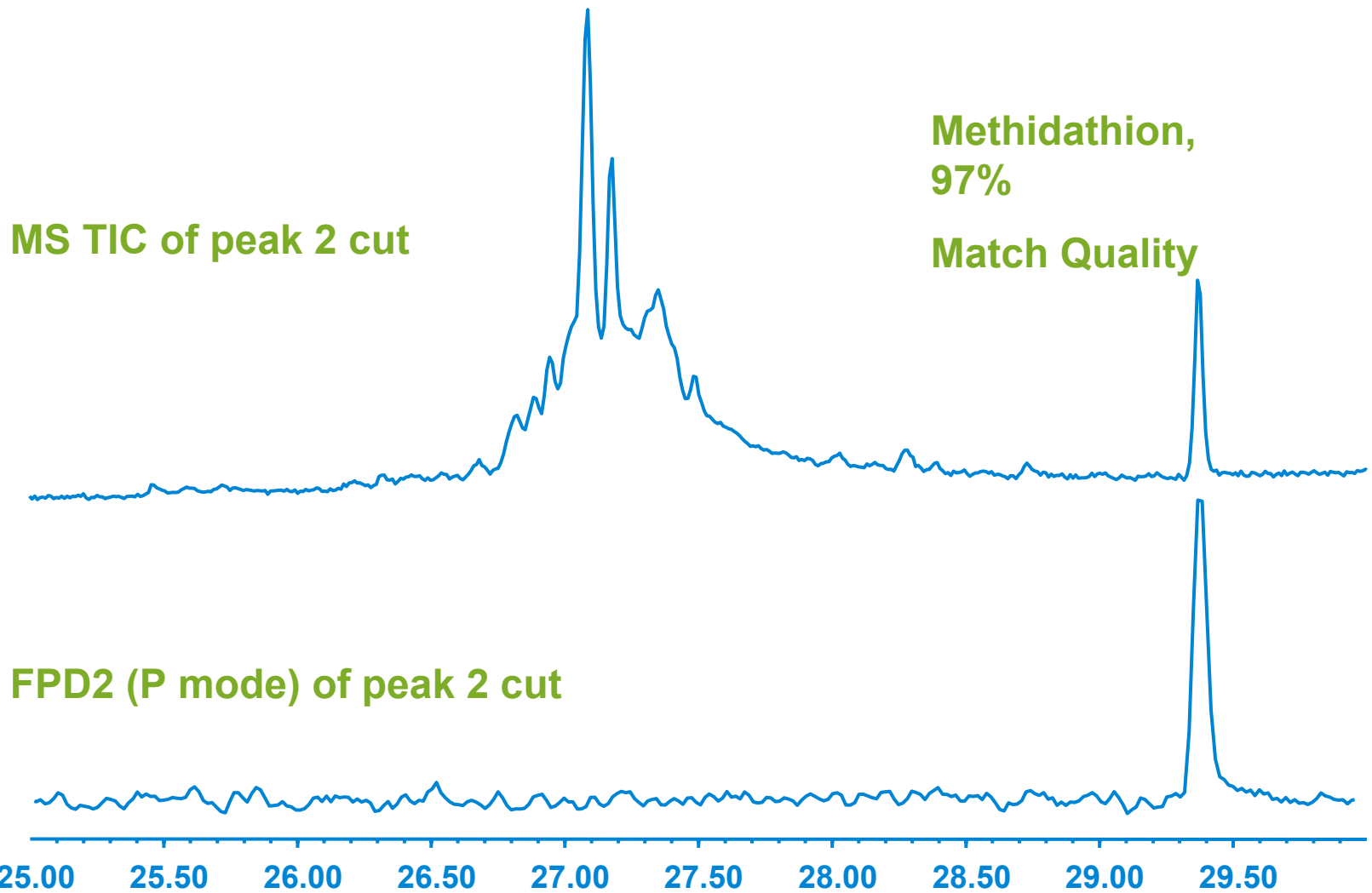
m/z-->



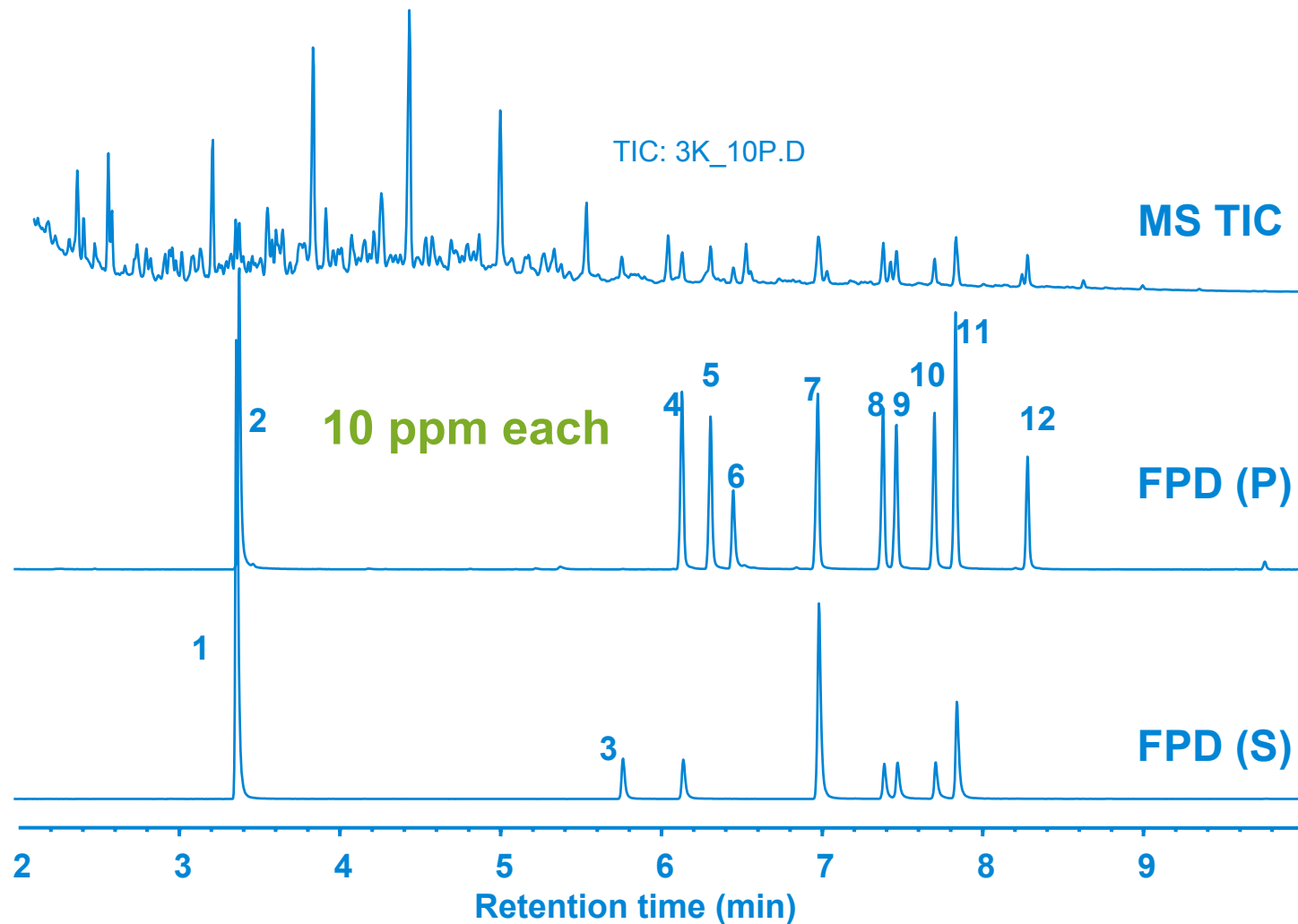
# FPD1 Screen of Lemon Oil A



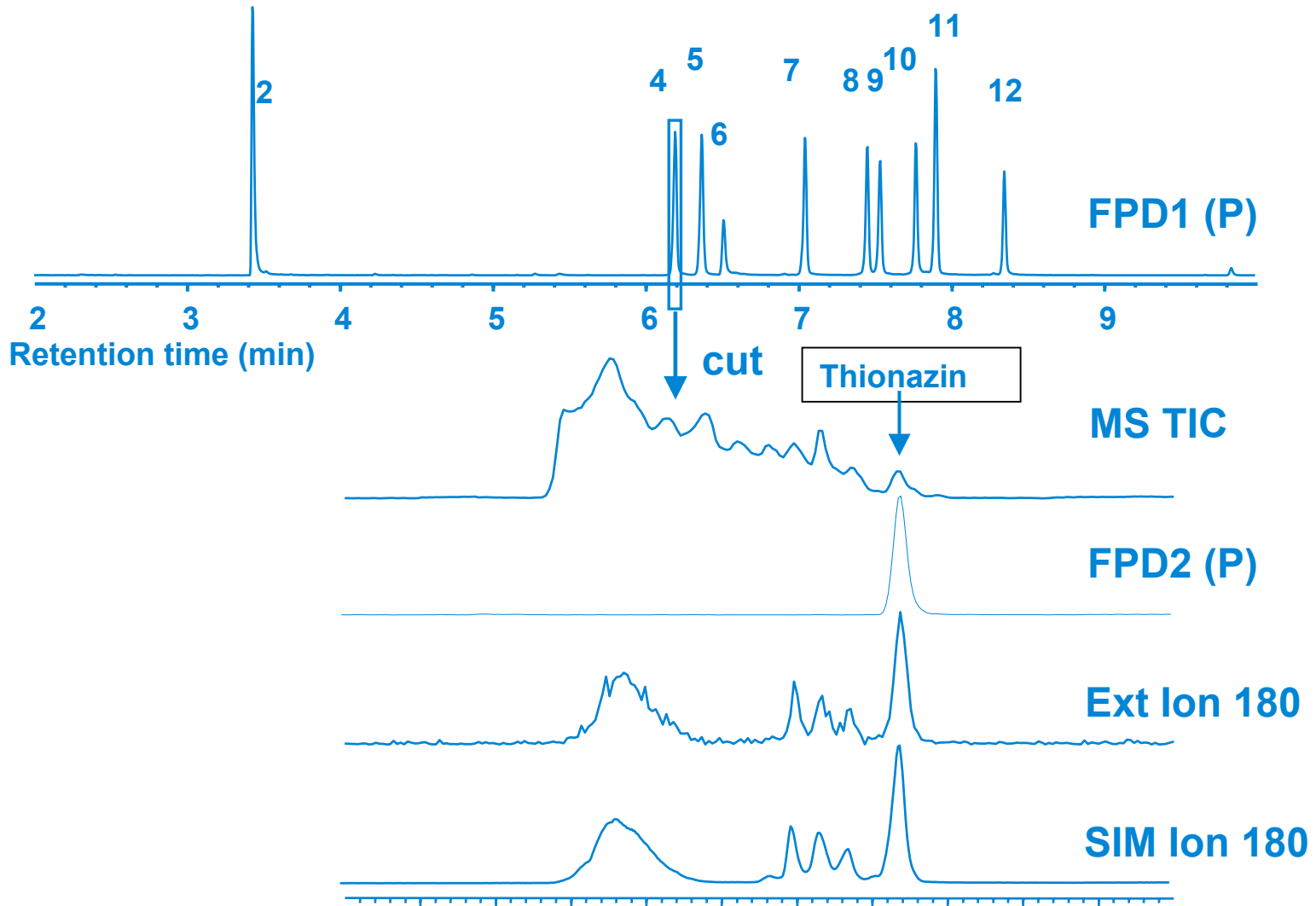
# Lemon Oil, Peak 2 Cut Results



# 12 Organophosphorus/sulfur pesticides in 3000 ppm diesel



# Cut 1 pesticide from 3000 ppm diesel





# Summary

- Developments in GC hardware in recent years have made Deans switch systems easier to construct, use, and maintain.
- Single oven, non-cryo systems can often solve the problem
- 2-D GC is a powerful tool that can be used by itself or combined with other selective techniques to solve difficult separation problems.

