

# Positive Ion APCI LC/MS Analysis of Enlite Herbicides

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# Agricultural Spraying of Herbicides



# Enlite Background



## DuPont™ Enlite®

herbicide

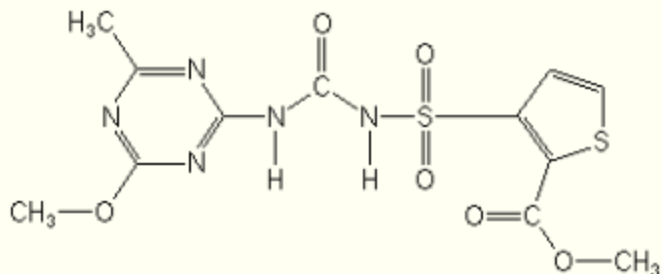
### *Dispersible Granules*

<b>Active Ingredients</b>	<b>By Weight</b>
Chlorimuron ethyl Ethyl 2-[[[(4-chloro-6-methoxypyrimidin-2-yl)amino]carbonyl]amino]sulfonyl]benzoate	2.85%
Flumioxazin 2-[7-fluor-3,4-dihydro-3oxo-4-(2-propynyl)-2H-1,4-benzoxazin-6-yl]-4,5,6,7-tetrahydro-1H-isoindole-1,3(2H)-dione	36.21%
Thifensulfuron methyl Methyl 3-[[[(4-methoxy-6-methyl-1,3,5-triazin-2-yl)amino]carbonyl]amino]sulfonyl]-2-thiophenecarboxylate	8.80%
<b>Other Ingredients</b>	52.14%
<b>TOTAL</b>	100.00%

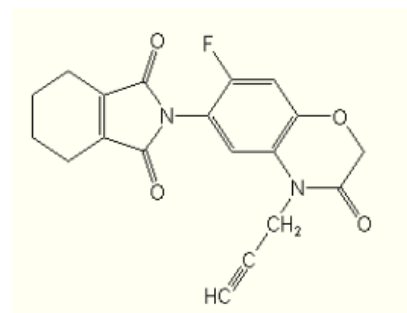
### DUPONT™ ENLITE® Highlights

- ENLITE® provides selective burndown, and preemergence weed control in soybeans. Use rate is 2.8 ounces per acre.
- When ENLITE® is applied according to instructions on this label, it will control many broadleaf weeds and provide partial control of annual grasses.
- ENLITE® has two modes of action and rapidly inhibits the growth of susceptible weed species.

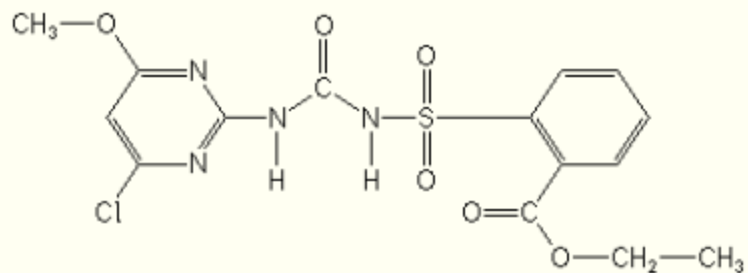
# Chemical Structures of Enlite Pre Emergent Herbicides



Thifensulfuron-methyl  
MW=387.39; (M+H)<sup>+</sup> = m/z 388.3



Flumioxazin  
MW=354.34; (M+H)<sup>+</sup> = m/z 355.34



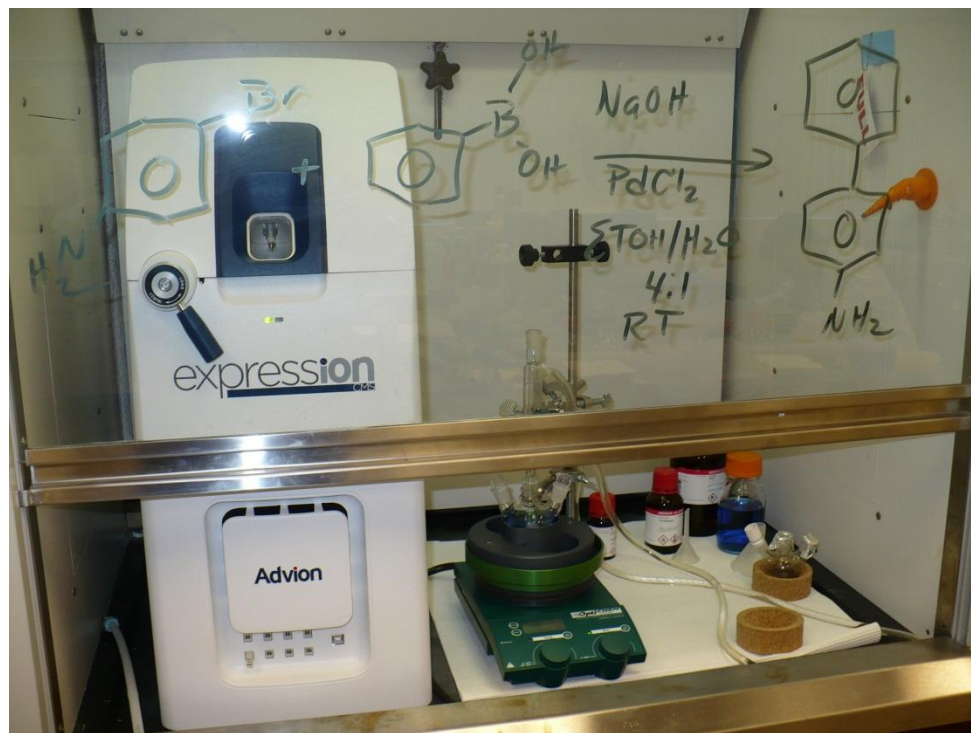
Chlorimuron-ethyl  
MW=414.35; (M+H)<sup>+</sup> = m/z 415.3

# Experimental

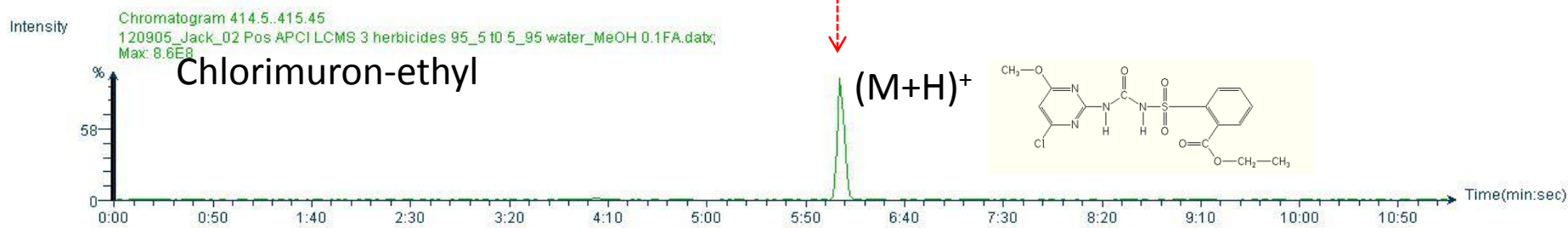
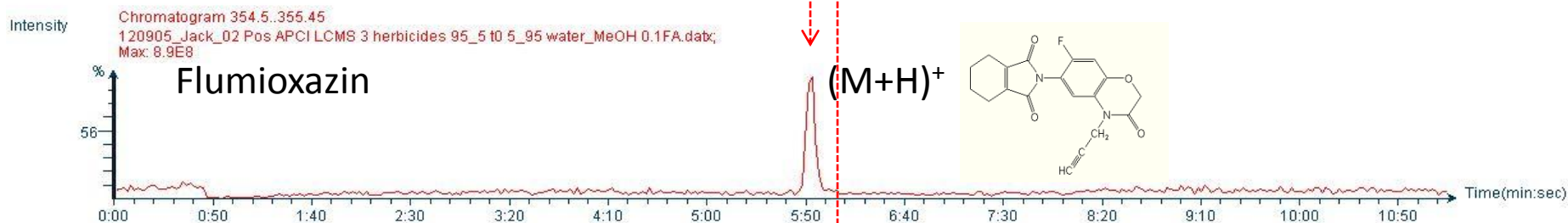
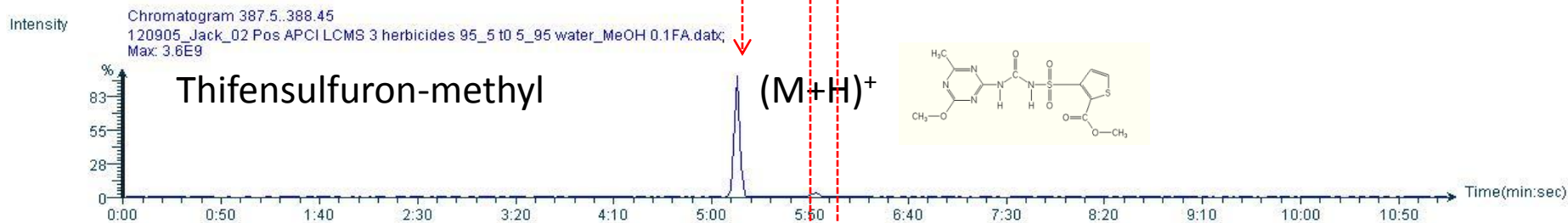
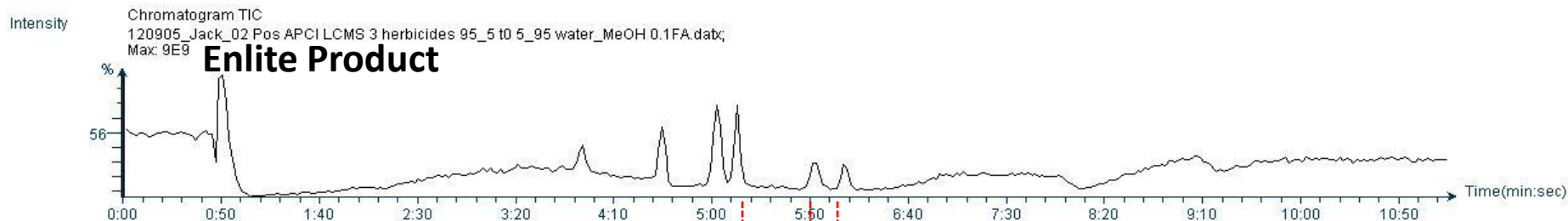
- Mass Spectrometry
  - expression compact mass spectrometer
  - ESI and APCI ion source
  - Positive and Negative ions
  - Full-scan or Selected Ion Monitoring (SIM)
- HPLC Conditions
  - Column: Phenomenex Luna 2.0 x 50 mm C-18(2), 5 microns
  - Mobile phase: water/MeOH with 0.1% formic acid
  - Flow: 200 uL/min
  - Gradient: 95/5 isocratic for 1 min then to 5/95 over 5 min, isocratic for 1 min with 1 min recycle
- Soil extraction
  - Slurry extract with MeOH, filter and concentrate
  - Directly inject for full-scan or SIM LC/MS analysis



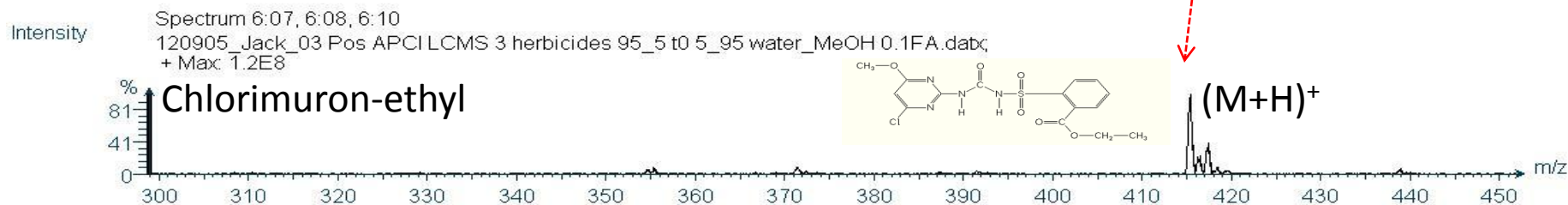
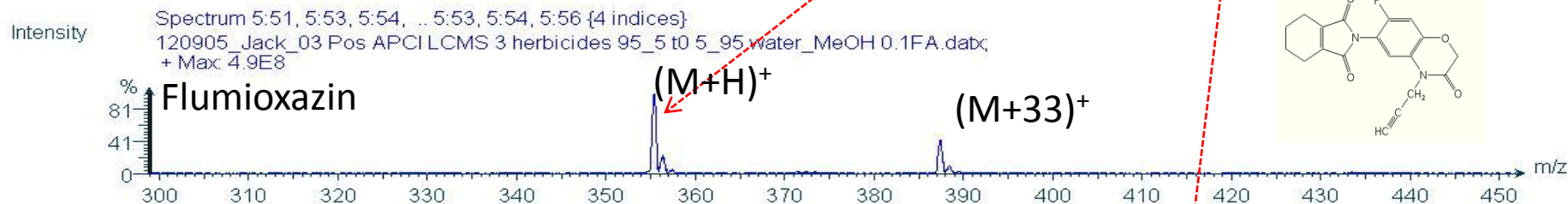
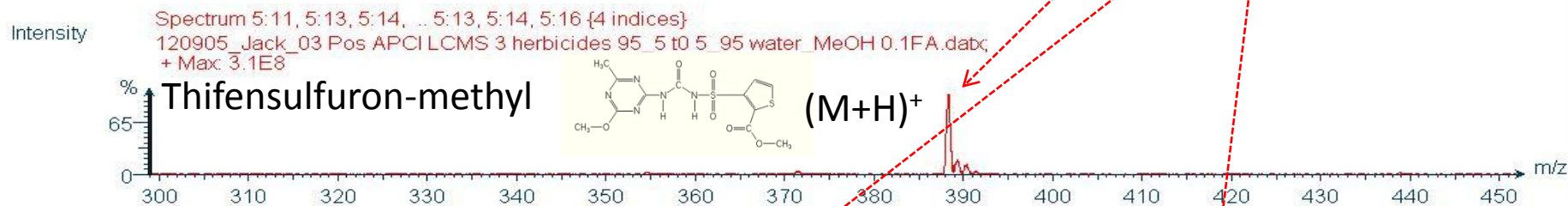
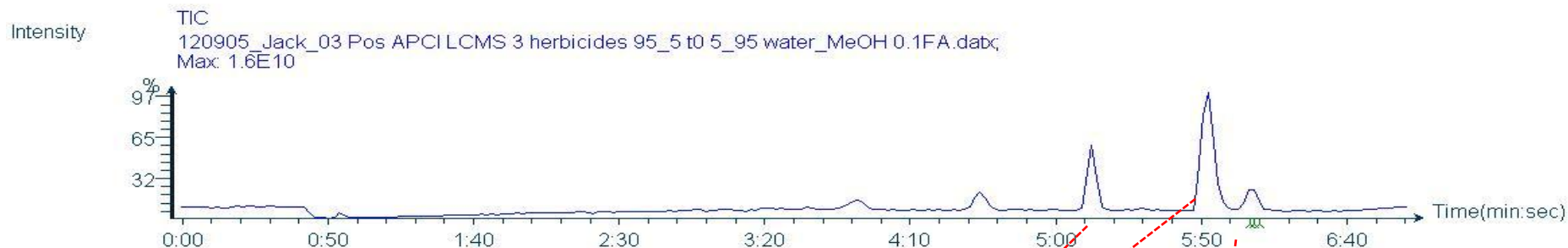
# LC/MS Adjacent or in the Hood



# Pos APCI Full-Scan LC/MS of Enlite Pre Emergent Herbicide

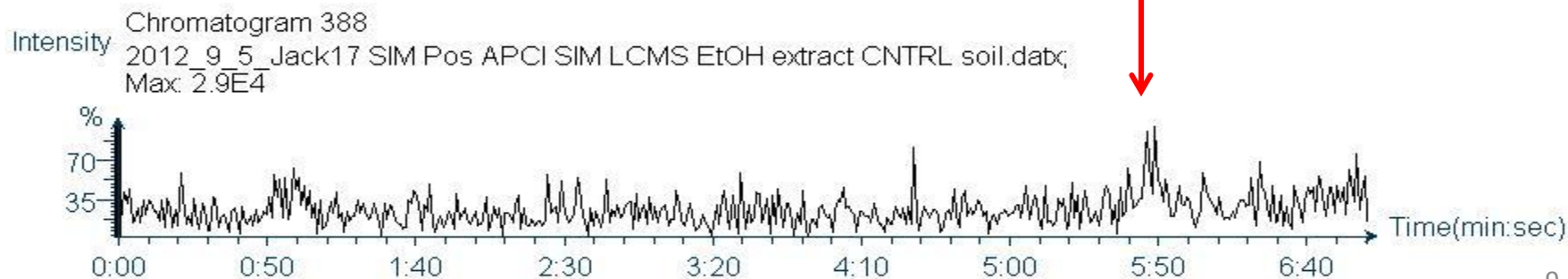
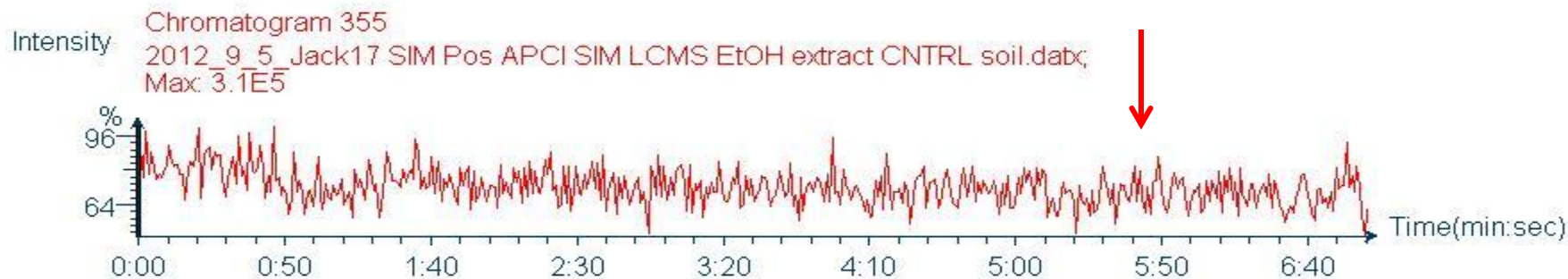
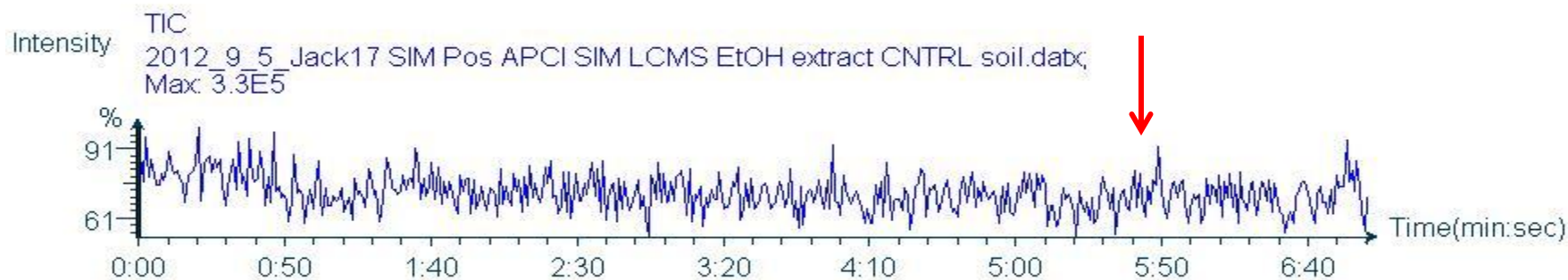


# Pos APCI LC/MS Enlite Herbicides

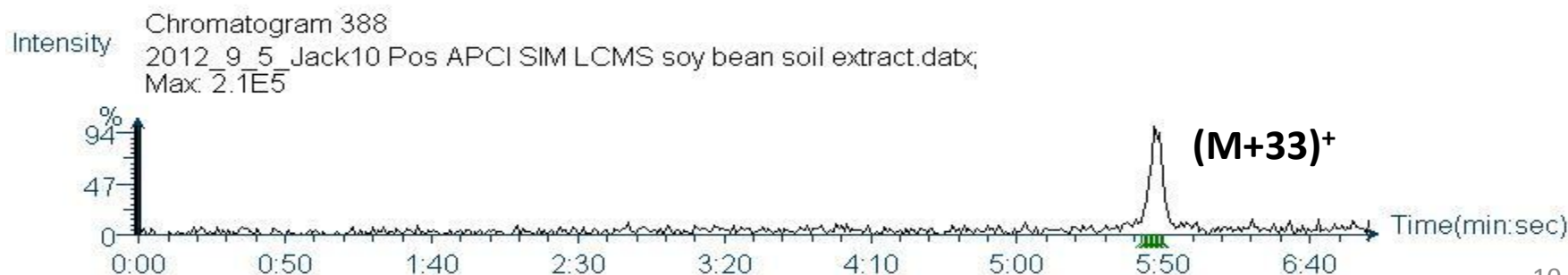
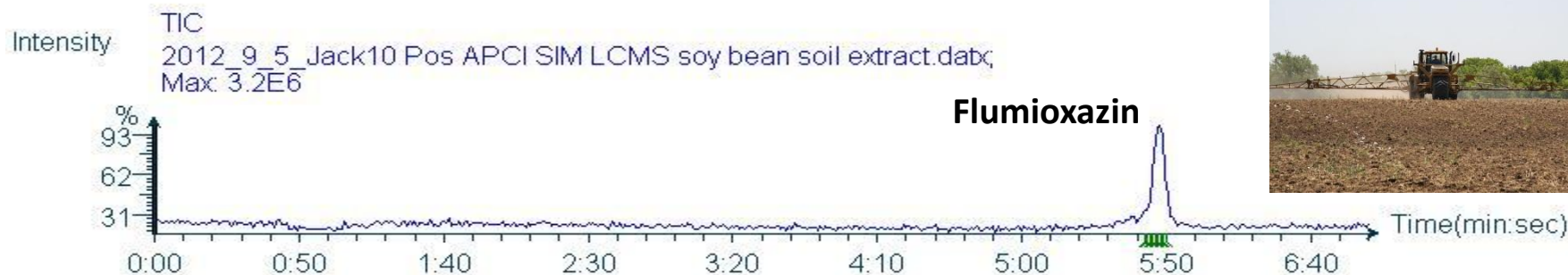




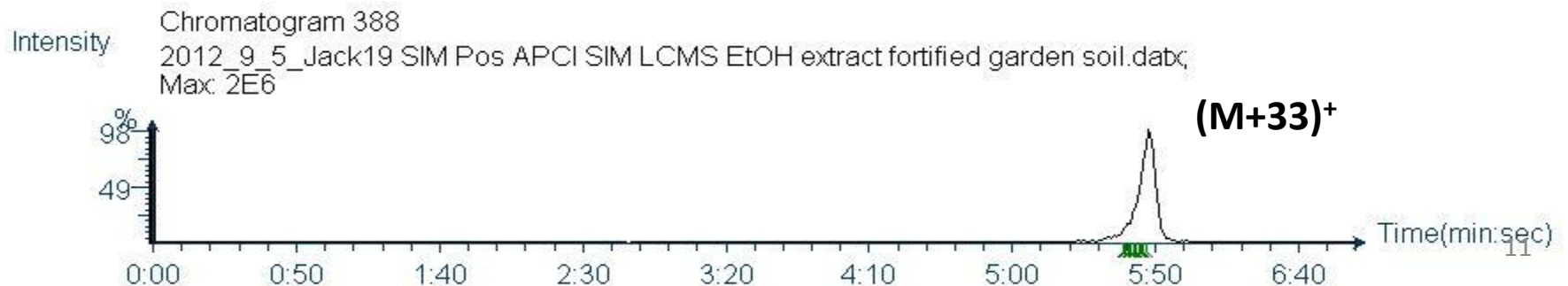
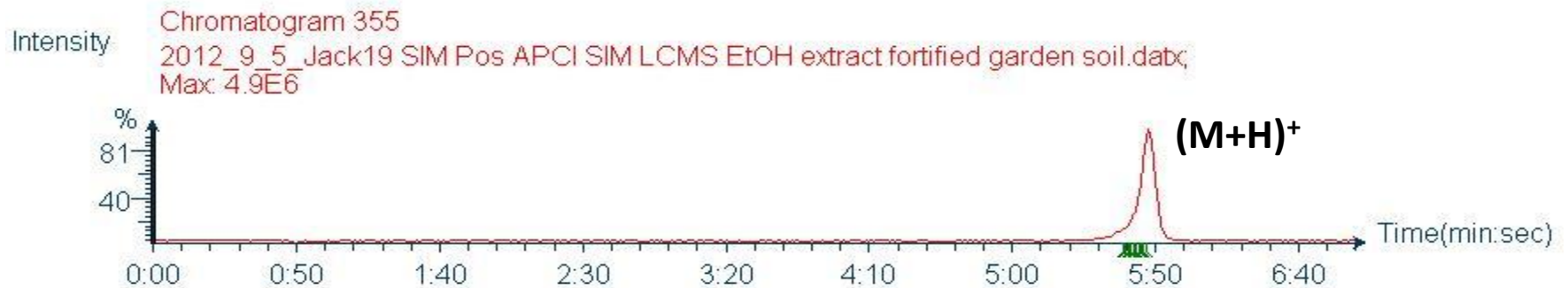
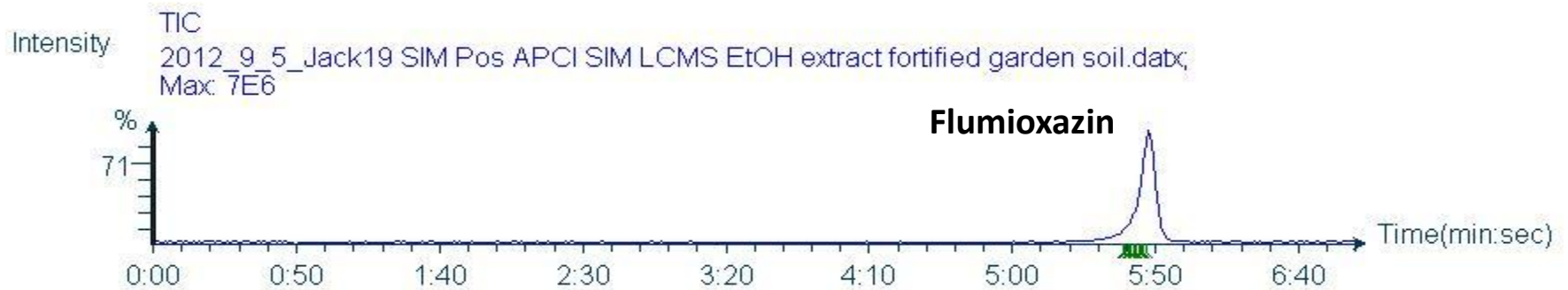
# Pos APCI SIM LC/MS Negative Control Garden Soil Extract



# Pos APCI SIM LC/MS Enlite Sprayed Soy Bean Soil Extract (10 ppb)



# Pos APCI SIM LC/MS of Extract from Fortified Control Garden Soil (20 ppb)



# Summary Enlite from Soil

- Three pre emergence herbicides in Enlite soil readily detected by Pos APCI LC/MS
- Flumioxizin readily detected in soy bean soil sprayed with Enlite, but the other two lower level herbicides were not detected
  - Only Flumioxizin was detected from extracts of fortified control soil sprayed with Enlite.
- It appears thifensulfuran methyl and chlormuron ethyl are not readily extracted from soil using the simple methanol extraction employed.

# **APCI LC/MS Determination of Malathion in Skim Milk**

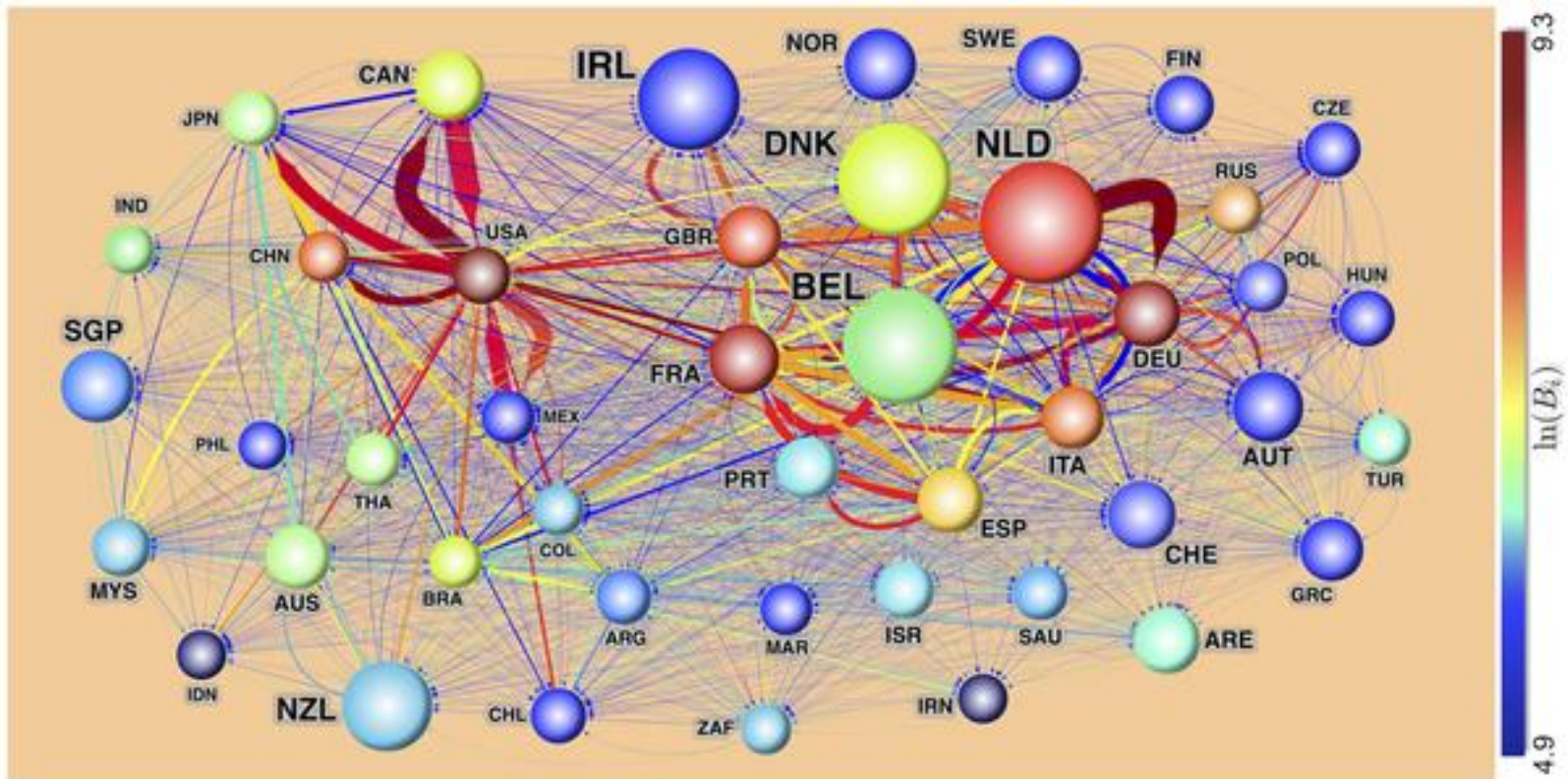
**Sleuthing the Adulteration of our Foods**

Jack Henion

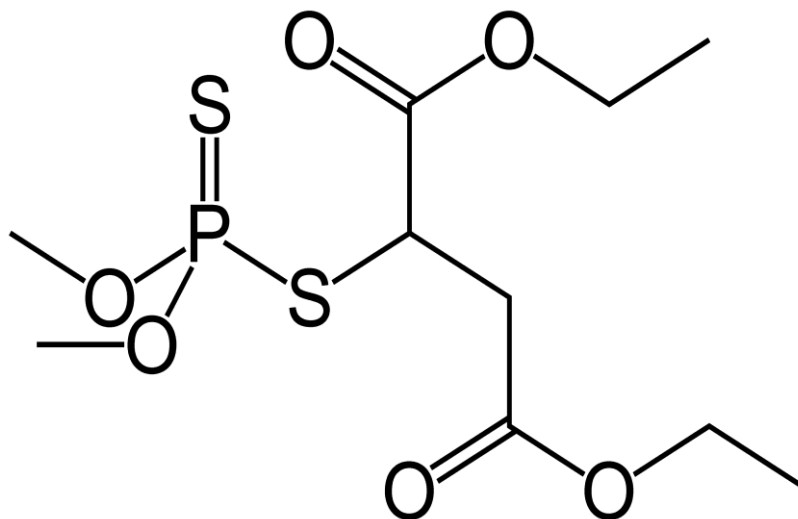
5 October 2012



# International agro-food trade network (IFTN), 2007

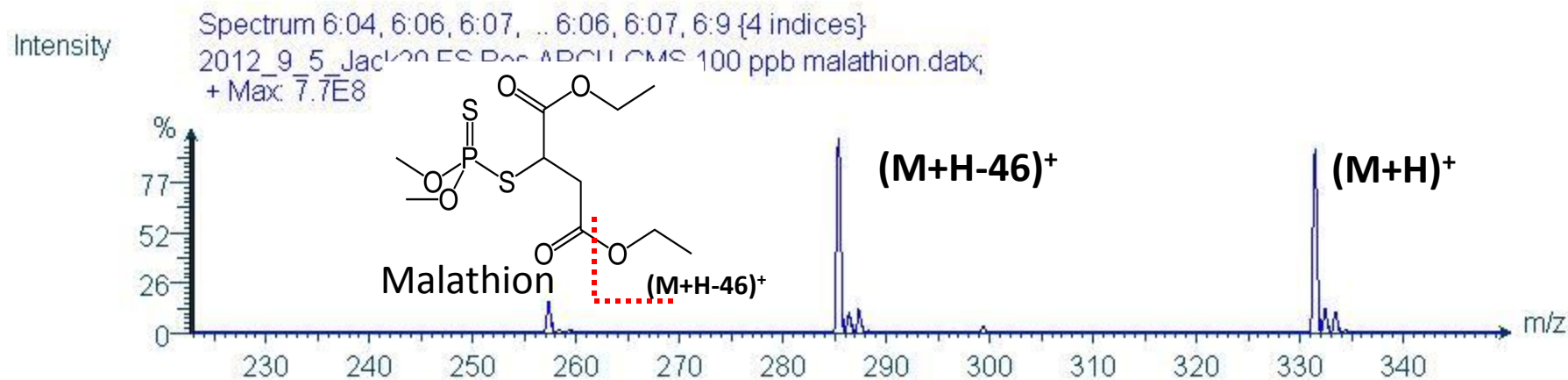
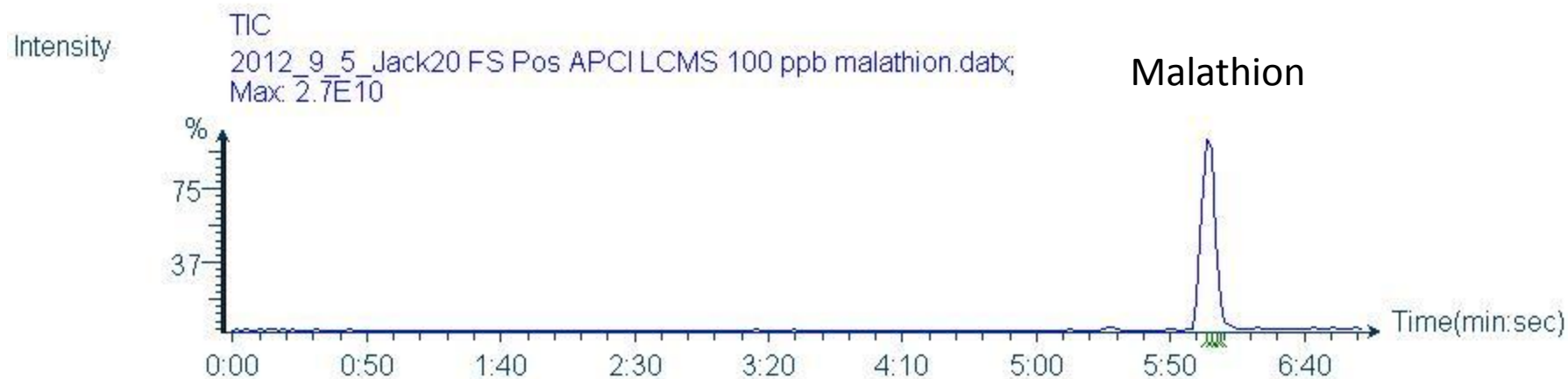


# Malathion Chemical Structure



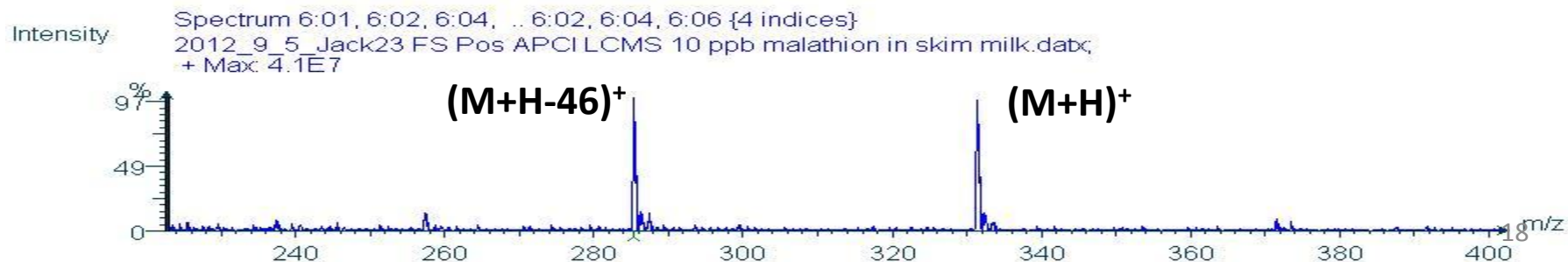
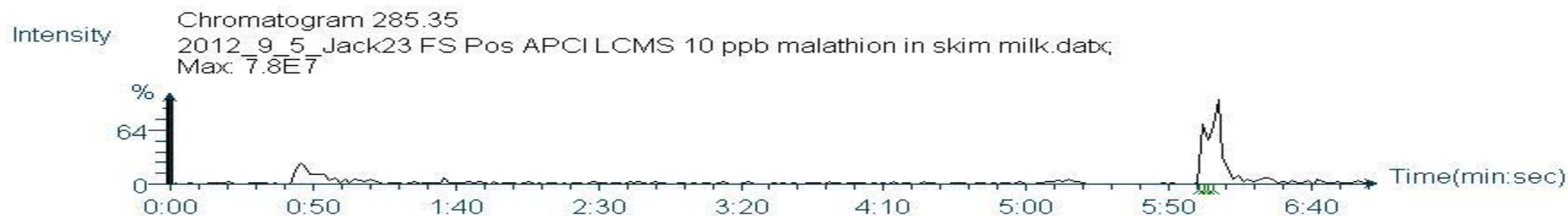
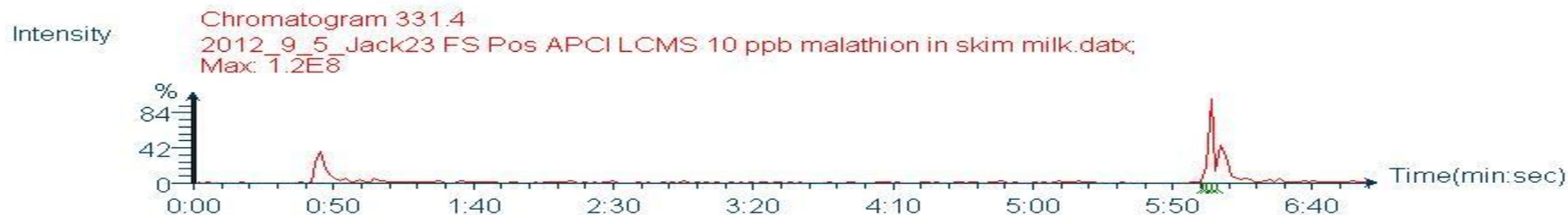
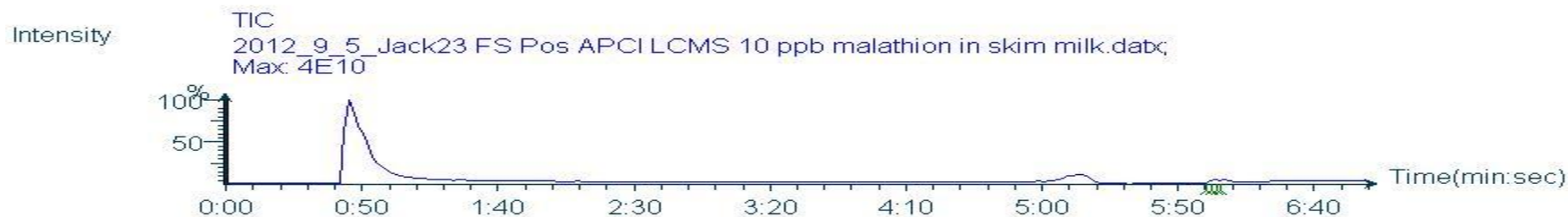
**MW= 330.1; (M+H)<sup>+</sup> = 331.1**

# Pos APCI LC/MS Malathion



# Pos APCI Full Scan LC/MS

## 10 ppb Malathion in Skim Milk



# Summary

- There must be better ways to do sample preparation for food matrices!
- And with good sample prep better analyses occur.
- **It is human nature to resist change. Let us embrace new ways of doing things if they can be shown to be better, faster and easier**



**Thank you!**