Chin-Kai Meng, Ph.D.
Chemical Solutions Business Unit
June 28, 2000

Fast GC/MS Screening of 567 Pesticides and Suspected Endocrine Disrupters in Complex Matrices

11:00 am EST
816-650-0621
Chairperson: Lisa Lloyd
Chin-Kai Meng, Ph.D.
Chemical Solutions Business Unit
June 28, 2000

Fast GC/MS Screening of 567 Pesticides and Suspected Endocrine Disrupters in Complex Matrices

Starts in FIVE minutes
816-650-0621
Chairperson: Lisa Lloyd

Agilent Technologies
Inventing the HP Way
Chin-Kai Meng, Ph.D.
Chemical Solutions Business Unit
June 28, 2000

Fast GC/MS Screening of 567 Pesticides and Suspected Endocrine Disrupters in Complex Matrices

Starts in ONE minute
816-650-0621
Chairperson: Lisa Lloyd
Audio Check

• The audio portion of today’s e-Seminar has just begun. To access, please dial 1-816-650-0621, Chairperson Lisa Lloyd.

• If you have dialed in and cannot hear the audio, please press *0 and you will be connected to the operator.

• If you have audio, but the speaker is talking too quietly or too quickly, please send a chat message.
Why a Pesticide Screening Method?

- More than 700 pesticides used in the world
- International commerce requires more screening
- Increasing health concerns -- especially children
- Most current methods are for ~100 pesticides
Typical GC-MS Screening Approach

Full Scan Library Search
What are the Disadvantages of Using the Library Search

• If the peak in the total ion chromatogram (TIC) was not integrated (due to poor peak shape or poor resolution), no library search result will be given.

• Overlapping compounds will not be identified properly.

• Compounds with similar fragmentation patterns (for example, isomers, alkanes) may not be properly identified.
Compound Identification

- Retention Time Locking
  -
  -

Gives MS library a 3rd dimension -- the retention time.
Retention Time Locking

Retention Time Locking:
- Initial run: 4.296 psi
- Cut 1 meter off: 4.064 psi
- First lock: 4.314 psi
- Second lock: 4.297 psi

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RTL Compensates for Differences in Column Length

10 meters were cut from the column - then it was re-locked

- Column cut
- Original column
- 50 m
- 60 m
Translate FID Method to MSD Method

### GC Method Translation

<table>
<thead>
<tr>
<th>Criterion:</th>
<th>Translate Only</th>
<th>Best Efficiency</th>
<th>Fast Analysis</th>
<th>None</th>
<th>Speed gain: 1.00001</th>
</tr>
</thead>
</table>

<table>
<thead>
<tr>
<th>Column</th>
<th>Original Method</th>
<th>Translated Method</th>
</tr>
</thead>
<tbody>
<tr>
<td>Length, m</td>
<td>30</td>
<td>30</td>
</tr>
<tr>
<td>Internal Diameter, μm</td>
<td>250.0</td>
<td>250.0</td>
</tr>
<tr>
<td>Film Thickness, μm</td>
<td>0.25</td>
<td>0.25</td>
</tr>
<tr>
<td>Phase Ratio</td>
<td>250.0</td>
<td>250.0</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Carrier Gas</th>
<th>Helium</th>
<th>Helium</th>
</tr>
</thead>
<tbody>
<tr>
<td>Enter one Setpoint Head Pressure, psi</td>
<td>18.2</td>
<td>8.443</td>
</tr>
<tr>
<td>Flow Rate, mL/min</td>
<td>1.4678</td>
<td>0.9073</td>
</tr>
<tr>
<td>Outlet Velocity, cm/sec</td>
<td>59.27</td>
<td>34.90</td>
</tr>
<tr>
<td>Average Velocity, cm/sec</td>
<td>34.90</td>
<td>1.43265</td>
</tr>
<tr>
<td>Hold-up Time, min</td>
<td>1.43267</td>
<td>Very large</td>
</tr>
<tr>
<td>Outlet Pressure (absolute), psi</td>
<td>14.696</td>
<td>0</td>
</tr>
<tr>
<td>Ambient Pressure (absolute), psi</td>
<td>14.696</td>
<td>14.696</td>
</tr>
</tbody>
</table>

---

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Slide 11
RT-Locked GC-FID vs. GC-MSD

80°C (9 min)
5°C/min to 150°C

Column Head Pressure

GC-FID
18.2 psi

GC-MSD
7.9 psi

Column exit at 14.7 psi
Column exit at ~0.0 psi
Universal Pesticide Method for GC, GC/AED, GC/MSD

For HP-5MS columns  30m x 0.25mm x 0.25µm

Flexible injection schemes:
  COC, LVI - PTV, Split/Splitless

Any detector:
  MSD, AED, ECD, NPD, FPD

Total run time = 41.87 min
4X faster, done in 10.5 min
Break Number 1

To ask a question:
Press *1 on your phone
OR
use chat

(If we don’t get to your question today,
everyone will receive a follow-up e-mail with
all questions and answers.)
Compound Identification

- Retention Time Locking
- Spectral Database Search

Agilent Pesticide & Endocrine Disrupter Library

- Locked to the pesticide method
- Contains 567 pesticides and suspected endocrine disrupters of interest worldwide
- Every entry has:
  - Retention Time
  - A Target Ion and three Qualifier Ions (and ion ratios)
  - Full scan spectrum
After a sample is run, ion currents are extracted for each database entry in a time window and integrated.
GC/MS Screener Report

<table>
<thead>
<tr>
<th>Compound</th>
<th>Retention Time</th>
<th>Mass</th>
<th>Molar Response</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dichlorvos</td>
<td>5.831</td>
<td>0.017</td>
<td>28045217</td>
</tr>
<tr>
<td>Mevinphos</td>
<td>7.595</td>
<td>0.013</td>
<td>28783747</td>
</tr>
<tr>
<td>Butylate</td>
<td>7.607</td>
<td>0.023</td>
<td>27294146</td>
</tr>
<tr>
<td>Vernolate</td>
<td>7.806</td>
<td>0.021</td>
<td>18046998</td>
</tr>
<tr>
<td>Dibrom (naled)</td>
<td>11.221</td>
<td>0.200</td>
<td>9059531</td>
</tr>
<tr>
<td>2,3,5-Trimethacarb</td>
<td>11.202</td>
<td>0.061</td>
<td>2775 121.91.135</td>
</tr>
<tr>
<td>Ethalfluralin</td>
<td>11.283</td>
<td>0.008</td>
<td>4696400</td>
</tr>
<tr>
<td>Trifluralin</td>
<td>11.637</td>
<td>0.007</td>
<td>19794785</td>
</tr>
<tr>
<td>Prometon</td>
<td>12.990</td>
<td>0.011</td>
<td>13502280</td>
</tr>
<tr>
<td>Chlorbufam</td>
<td>13.098</td>
<td>0.067</td>
<td>15818 39.53.118</td>
</tr>
<tr>
<td>Dimethipin</td>
<td>13.159</td>
<td>0.005</td>
<td>15438792</td>
</tr>
</tbody>
</table>

Report shows details of each compound in the database and is user defined.
200 µL Spiked Green Bell Pepper Extract, PTV

Sample courtesy of Marc Engel, State of Florida

50 µL x 4 @ 10 ppb

50 µL x 4 @ 1 ppb
### GC/MS Results for Analysis of Green Bell Pepper Extract
(Spiked with 48 Components)

<table>
<thead>
<tr>
<th></th>
<th>200 µL PTV</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>1 ppb</td>
</tr>
<tr>
<td>Total reported compounds</td>
<td>82</td>
</tr>
<tr>
<td>Identified (x on screen report)</td>
<td>41</td>
</tr>
<tr>
<td>Identified (?) on screen report</td>
<td>6</td>
</tr>
<tr>
<td>Unidentified</td>
<td>1</td>
</tr>
<tr>
<td>Other confirmed compounds (x)</td>
<td>6</td>
</tr>
<tr>
<td>Others with “?”</td>
<td>29</td>
</tr>
</tbody>
</table>
Banana Skin Study (Acetone Wash)

200 µL injection
**Banana Skin Study (Acetone Wash)**

- **200 µL injection**
- **Imazalil**, ~ 400 ng
- **Thiabendazole**, ~ 200 ng
- **Chlorpyrifos**, ~ 2 ng

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Slide 22
Break Number 2

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(If we don’t get to your question today, everyone will receive a follow-up e-mail with all questions and answers.)
Compound Identification

- Retention Time Locking
- Spectral Database Search
- Visual Confirmation
RTL Screener Results:
A “hit”
RTL Screener Results: Possible “hit”
RTL Screener Results: False

- 5.604 Methamidophos
- Dichlorvos
- 6.248 Alidochlor
- 2,3,5-Trichlorophen
- 6.778 2,6-Dichlorobenzene
- 6.873 Nicotine
- 2,4,6-Trichlorophen
- 6.813 EPTC
- 6.873 Dichlorid
- 2,4,5-Trichlorophen
- Phenoxyacetic acid
- 7.113 Biphenyl
- Propamocarb
- 7.155 2-Phenoxypropio
- 7.406 3,5-Dichloroanilin

- Scan 610 (6.675 min): 0401003.D
- m/z -> 43 79 101 129 151 172 203 243 291 327

- Ion Exp% Act%
  - 172.00 47.90 18.70#
  - 124.00 29.40 3.51#
  - 56.00 23.10 26.53
Time Scaling

PCB 1 (1x)

PCB 1 (4x)
| 1  | 5.814 | Dichlorovos          | 15 | 19.227 | Chlorpyrifos        |
| 2  | 7.582 | Mevinphos           | 16 | 23.858 | Dieldrin            |
| 3  | 7.785 | Vernolate            | 17 | 24.010 | p,p'-DDE            |
| 4  | 11.202| Dibrom (naled)      | 18 | 27.373 | Hexazinone          |
| 5  | 11.275| Ethalfluralin       | 19 | 27.720 | Propargite          |
| 6  | 11.629| Trifluralin         | 20 | 29.721 | Leptophos           |
| 7  | 13.001| Prometron           | 21 | 29.826 | Mirex               |
| 8  | 13.168| Atrazine/BHC beta isomer | 22 | 32.696 | Cypermethrin I     |
| 9  | 13.445| Lindane             | 23 | 32.851 | Cypermethrin II    |
| 10 | 14.783| Chlorothalonil      | 24 | 32.972 | Cypermethrin III   |
| 11 | 16.607| Methyl Chlorpyrifos | 25 | 33.026 | Cypermethrin IV    |
| 12 | 16.782| Heptachlor          | 26 | 34.287 | Fluvalinate         |
| 13 | 18.348| Bromocil            | 27 | 34.695 | Fluvalinate-tau-II |
| 14 | 18.799| Malathion           | 28 | 34.823 | Fluvalinate-tau-I  |

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Slide 29
Screen capture showing the method translation (MTL) software data entry used in a 4X speed gain translation.
RTL Makes Scaling Easy and Precise

Libraries with RTs can also be scaled according to the analysis for fast screening!
Resolution Loss With Increased Speed

- **1X 250 µm**
  - Original Method

- **2.5X 250 µm**

- **4X 250 µm**
Summary

• RTL can produce the same RT in every GC system using your method.
  ➢ AED, MSD, FPD, NPD, ECD, etc.
  ➢ Split/splitless, PTV, on-column, etc.
• The MS RTLPEST library (567 pesticides & endocrine disrupters) contains locked RT and compound mass spectra. The library can be time-scaled.
  ➢ Retention time, spectral, and visual confirmation
• User can build in-house library for screening
Break Number 3

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(If we don’t get to your question today,
everyone will receive a follow-up e-mail with
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Wrap-up Questions

Thanks for attending. We hope to see you at a future Agilent e-Seminar. Our list of topics keeps growing, so visit often.

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