A Presentation by OI Analytical

# VOC Cycle Times Using the Eclipse Purge-and-Trap Sample Concentrator


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# VOC System Cycle Time

- The overall VOC system cycle time is composed of three individual cycle times:
  - *Autosampler*
  - *Purge-and-trap (P&T)*
  - *Gas chromatograph*
- The rate-limiting step is the component with the longest individual cycle time
- For best results, balance the cycle times of the three individual components

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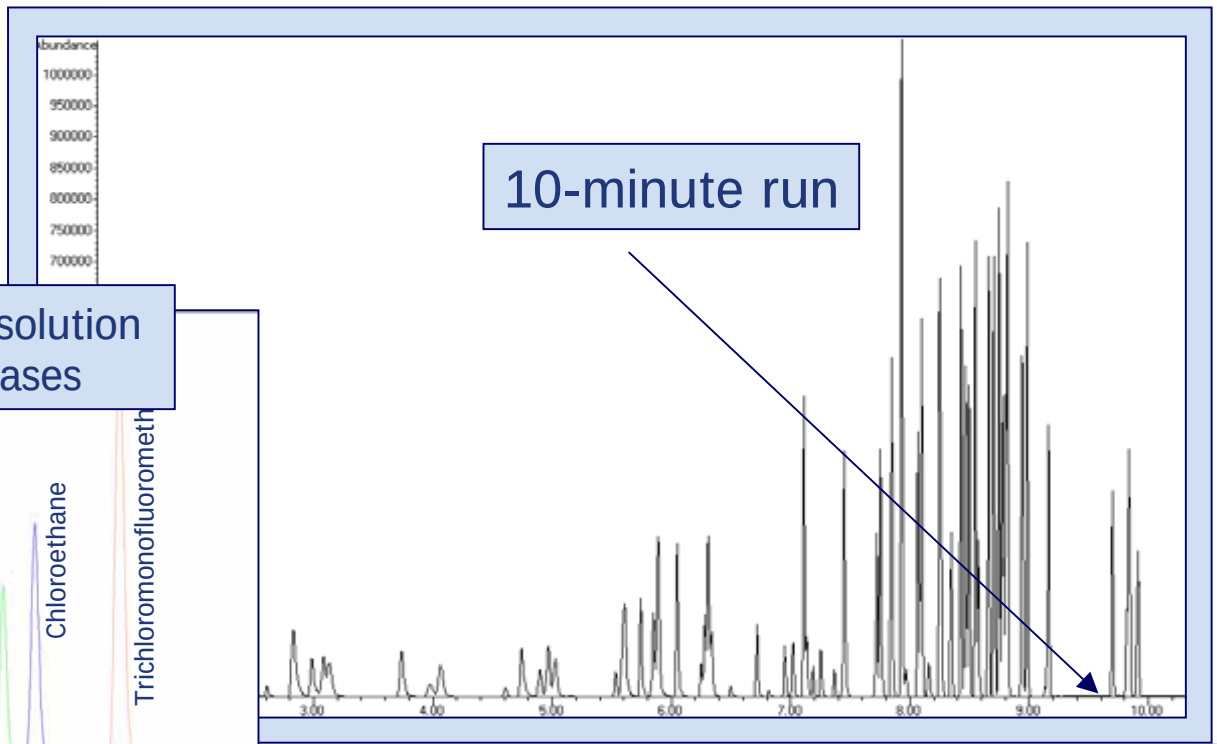
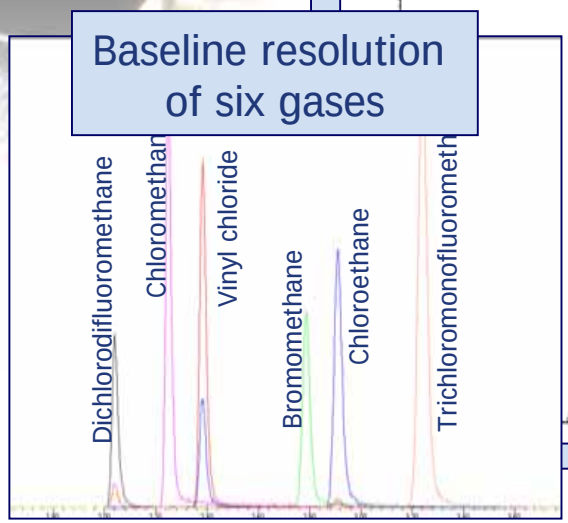
# GC Cycle Time

- GC cycle time is determined by:
  - *Choice of detector (MS vs. PID/ELCD)*
  - *Choice of column (20, 30, 60, or 105 meters)*
  - *Oven program*
  - *Cool down time*
- Fastest GC cycle time achieved with:
  - *MS detector*
  - *20- or 30-meter column*
  - *Balanced ramp rate and, cool laboratory temperatures*
- GC cycle time of 16 to 20 minutes is easily achievable using these conditions

# GC Run Time of ~10 Minutes

- Method 524.2 calibration standard, 86 compounds
- DB-VRX, 20 m x 0.18 mm I.D. x 1.0-µm film
- 10-min GC run + cool down = 16-min GC cycle

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# P&T Cycle Time

- P&T cycle time is the sum of several individual steps
- Some times can vary, others cannot

| | |
|---|---|
| Purge time | Fixed at 11 minutes |
| Dry-purge | Varies from 0 to 6 minutes |
| Desorb preheat | Varies from 0 to ½ minutes |
| Desorb | Varies from ½ to 4 minutes |
| Bake | Varies from 2 to 10+ minutes |
| Cool down | Usually about 2–3 minutes |

# Eliminate the Dry Purge Step

- With the Eclipse (and the Model 4560) eliminate the dry purge step entirely
- Remove $H_2O$ during desorb using the patented Water Management Fitting (WMF)

| Purge time | 11 minutes |
|---|---|
| Dry-purge | Not necessary |
| WMF cool down | ½ minute |
| Desorb preheat | |
| Desorb | |
| Bake | |
| Cool down | 2 minutes |

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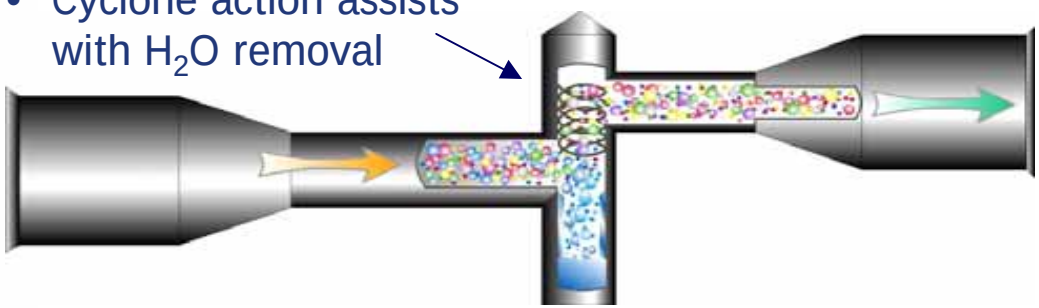
# Eliminate the Dry Purge Step

- Reduce the P&T cycle time by as much as six minutes by completely eliminating the dry purge step
- In the Eclipse, remove water during the desorb state using OI Analytical's patented Water Management Fitting

- Cyclone action assists with $H_2O$ removal

From Trap



To GC

- Reservoir at ambient
- $H_2O$ is cold trapped

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# Shorten the Desorb Time

- With the Eclipse (and Model 4560) direct resistive trap heating allows shortened desorb times
- No loss of performance with 100% recovery

| | |
|---|---|
| Purge time | 11 minutes |
| Dry-purge | Not necessary |
| WMF cool down | ½ minute |
| Desorb preheat | Not necessary |
| Desorb | ½ minute |
| Bake | |
| Cool down | 2 minutes |

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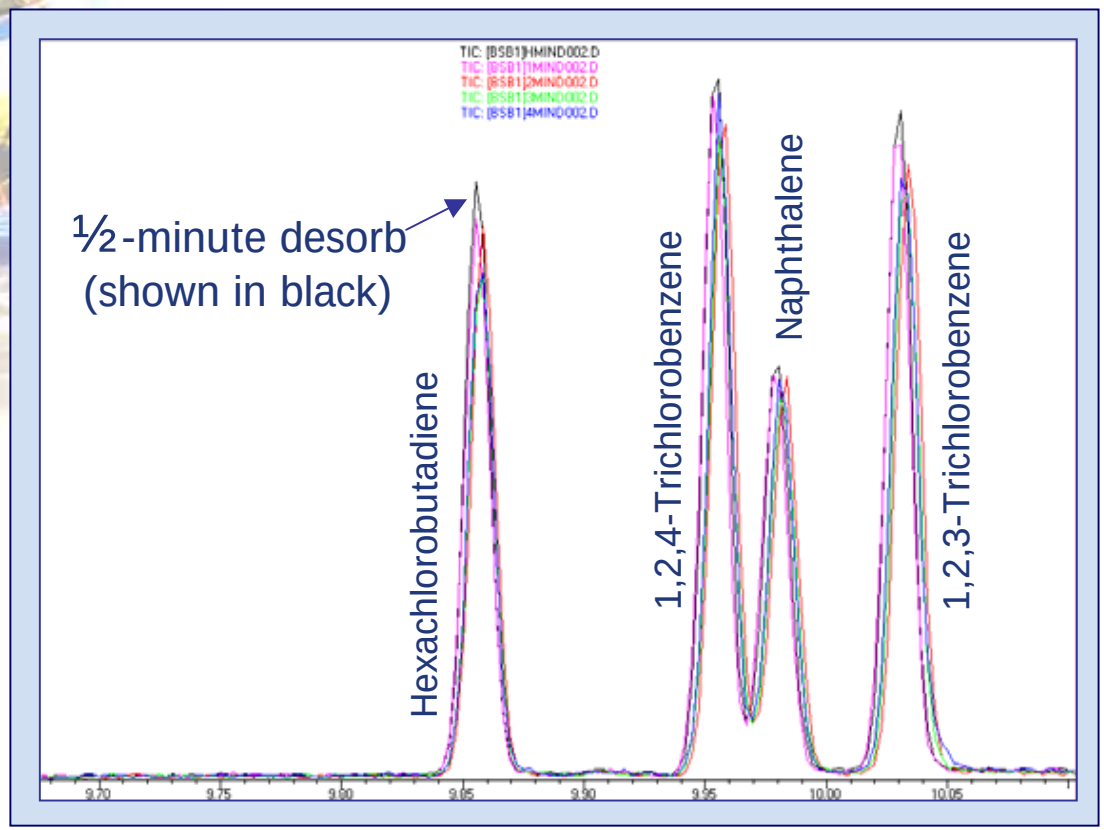
# High Recovery with Short Desorb

Overlaid chromatograms of the four heaviest peaks using desorb times of 4, 3, 2, 1, and ½ minutes




The four heaviest compounds had 100% recovery with a ½-minute desorb

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# Shorten the Bake Time

- With the Eclipse (and Model 4560) direct resistive trap heating allows bake times of two minutes
- No need for excessive gas flows that can compromise trap performance

| | |
|---|---|
| Purge time | 11 minutes |
| Dry-purge | Not necessary |
| WMF cool down | ½ minute |
| Desorb preheat | Not necessary |
| Desorb | ½ minute |
| Bake | 2 to 6 minutes |
| Cool down | 2 minutes |

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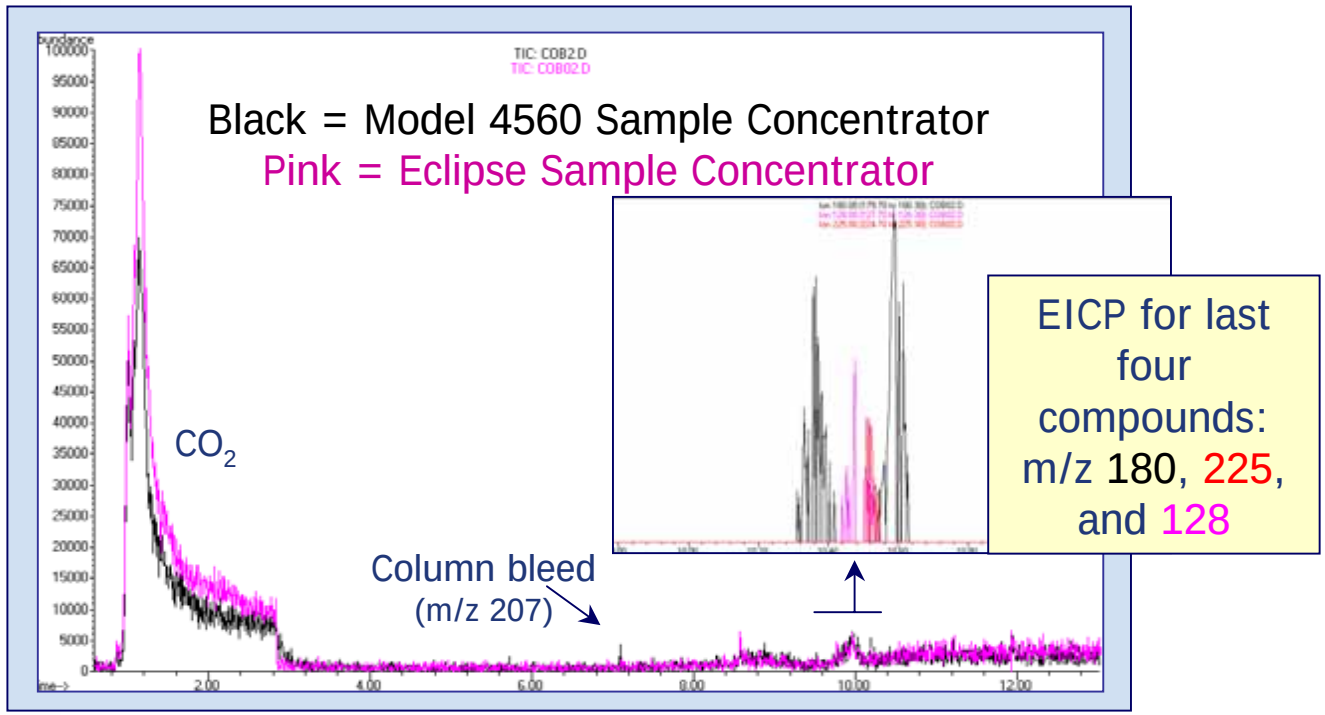
# Low Trap Carryover with Short Bake

- Trap carryover after a 100-ppb standard
- #10 trap, 11-minute purge (40 °C), 2-minute bake




Note: Factors other than bake time can contribute to carryover

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# P&T Cycle Time

- With the patented features of the Eclipse, easily achieve cycle times of 16 to 20 minutes

| | |
|---|---|
| Purge time | 11 minutes |
| Dry-purge | Not necessary |
| WMF cool down | ½ minute |
| Desorb preheat | Not necessary |
| Desorb | ½ minute |
| Bake | 2 to 6 minutes |
| Cool down | 2 minutes |
| Total Cycle Time | 16–20 minutes |

Note: bake time is usually extended to balance with the autosampler cycle time

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# A Note About Carryover

- Trap can be reconditioned in two minutes for samples with low concentrations and few matrix interferences (e.g., drinking water by Method 524.2)
- Other potential sources of carryover must be considered when developing a method:
  - *Sample type and presence of matrix interferences*
  - *Concentration level of analytes*
  - *GC column reconditioning*
  - *Type of autosampler used*
  - *Rinse/flush sample pathway*
  - *P&T bake time*

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# Autosampler Cycle Time

- Autosampler cycle time is determined by:
  - *Type of autosampler*
  - *Sample size*
  - *Number of rinses*
  - *Other user-programmable features*

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# Autosampler Cycle Times

- Examples of autosampler cycle times using two autosamplers and varying conditions

| | Model 4551A Water Only | | Model 4552 Water Mode | |
|---|---|---|---|---|
| Sample/Rinse Volume | Cycle Time (min) | Runs per 12-hr tune | Cycle Time (min) | Runs per 12-hr tune |
| 5-mL + 1 rinse | 19.0 | 38 | 19.5 | 37 |
| 5-mL + 2 rinses | 22.5 | 32 | 23.0 | 31 |
| 25-mL + 1 rinse | 21.5 | 33 | 38.0 | 19 |
| 25-mL + 2 rinses | 26.0 | 27 | 49.0 | 14 |
| 4552 Soil Mode: Soil sample + 5-mL water (for stnds transfer) and ½-min preheat | | | 16.5 | 43 |

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# Best VOC Cycle Times


+

=

- 19-minute cycle time
- 38 samples per 12-hour tune

Waters Only


+

+

=

- 16.5-minute cycle time
- 43 samples per 12-hour tune

Soils & Waters

Note: Configurations that use one autosampler feeding two P&T/GC/MS systems lose twice the productivity when the autosampler fails or requires maintenance

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# Conclusions

- GC cycle time of 16 to 20 minutes is achievable
  - *Using a MS and 20- or 30-meter column*
- Using patented features of the Eclipse a P&T cycle time of 16 to 20 minutes is also achievable
- The autosampler is often the rate-limiting step, but can be matched to the GC and P&T cycles
- Overall VOC system cycle times between 16.5 and 49 minutes depending on the autosampler

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Application Note Number 1932

For full details on this and other P&T applications, please visit us at:

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