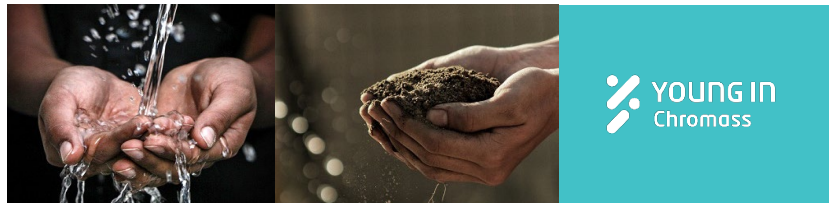


BTEX Analysis Even Better with Dynamic Headspace

- GC Application



Abstract

BTEX (Benzene, Toluene, Ethylbenzene, o,m,p-Xylene) represents a group of volatile organic compounds (VOCs) and exists as gases at atmosphere pressure. Due to its water solubility, it's classified as water contaminant and it's also hazardous to health.

The recovery of VOCs analysis in water differs depending on sample preparation. Its high volatility decreases the recovery with LLE (Liquid/Liquid Extraction) or SPME (Solid Phase Micro Extraction). So, VOCs analysis commonly goes with Headspace or Purge & Trap sample preparation but the U-shaped sparger of Purge & Trap can be easily contaminated when it comes to analyzing waste water including some particles, which finally reduces the accuracy.

So, it's better to prepare the sample in Headspace, especially Dynamic Headspace when analyzing trace level of VOCs or semi-VOCs.

This study shows comparisons between Static Headspace with Dynamic Headspace according to EPA method 8260 Volatile Organic Compounds by GC/MS (Gas Chromatograph/Mass Spectrometer).

Method

In this study, YL6900 GC/MS coupled to ChroZen PAL RSI System including Static Headspace and Dynamic Headspace (ITEX) was configured to conduct the analysis. The change of both injection tools (Static HS and Dynamic HS) was easily done in seconds.

The same GC/MS parameter setting was applied while changing Static HS and Dynamic HS to compare both. The detail of conditions is described in Table 1 & 2.



Fig 1. ChroZen PAL RSI System

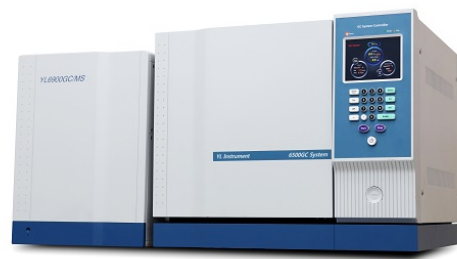


Fig 2. YL 6900 GC/MS

Table 1. HS Condition

| Static HS condition | Dynamic HS condition |
|-----------------------------------|------------------------------------|
| Sample Conditioning @ 80 °C, 6min | Sample Conditioning @ 80 °C, 2 min |
| Syringe Temperature: 85 °C | Extraction Strokes: 10 Strokes |
| Injection: 1mL | Desorption @ 220 °C, 10uL/s |

Table 2. GC/MS conditions

| GC condition | MS condition |
|---|--|
| Column: YL 5ms (30m x 0.25 mm i.d. x 0.25um) | Ion source 200 °C, Transfer Line 250 °C |
| Inlet: split, 1/10 split ratio | Detection: SIM mode |
| Oven temperature program: 40 °C, 5min, 10 °C/min to 150 °C, 2min | SIM: ion monitored: 78, 91 (185 ms dwell times) |

Result

50 ppt of BTEX sample was analyzed and Fig.3 shows each chromatogram with Static HS, Dynamic HS and the overlaid one. The sensitivity difference is clearly shown on the Fig.3 and Table 3.

To validate the data accuracy, 5-point calibration curve was prepared in the range of 0.1~10 µg/L concentration. And the linearity in use of Static HS and Dynamic HS was measured greater than 0.997. [Table 4]

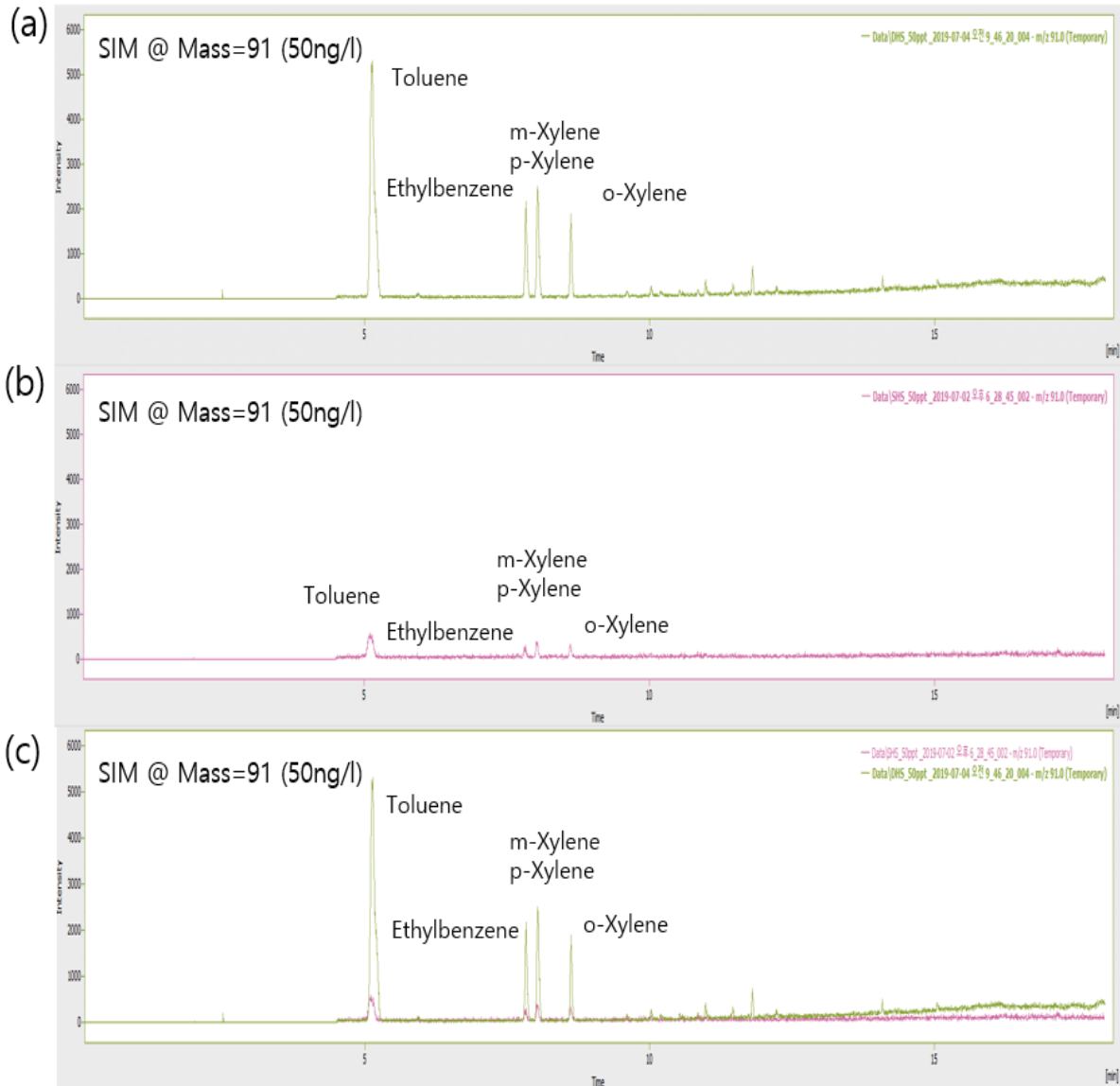


Fig 3. 50ppt of BTEX Chromatogram

(a) Dynamic HS, (b) Static HS, (c) a + b overlay

Table 3. Peak Area of 50 ppt BTEX in Dynamic HS and Static HS

| | Toluene | Ethylbenzene | m,p-Xylene | o-Xylene |
|------------|---------|--------------|------------|----------|
| Dynamic HS | 28930.6 | 5458.1 | 7372.7 | 4437.7 |
| Static HS | 5893.8 | 901.7 | 1650.4 | 1109.1 |

Table 4. Comparison in Calibration

| R ² | Benzene | Toluene | Ethylbenzene | m,p-Xylene | o-Xylene |
|----------------|---------|---------|--------------|------------|----------|
| Static HS | 0.9993 | 0.9991 | 0.9989 | 0.9978 | 0.9986 |
| Dynamic HS | 0.9999 | 0.9985 | 0.9997 | 0.9995 | 0.9995 |

Conclusion

This study is to show the difference of Static HS and Dynamic HS in sensitivity and Dynamic HS (ITEX) results in much higher sensitivity than Static HS because it enables sample enrichment by concentrating analytes on the Tenax Trap. Therefore, it's recommended to use Dynamic HS when it comes to analyze trace level of VOCs.

Reference

- EPA method 8260 (2017)



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Configuration

| item | Description | Part No. | |
|---|--|--|----------------------|
| Oven | 6500 GC Oven System Module | 6501011012 | |
| Inlet | YL6500GC Capillary Inlet Assembly | 6601011023 | |
| Detector | YL6900 MSD for YL6500 GC incl. built-in turbomolecular pump - Single Quadropole - EI source - Turbo pump(240 L/sec) - Include fore pump and spares kit | 6501011440 | |
| Install. Option | Start-up kit includes (Without GC Capillary Column) 1) Nuts and ferrules 1/8" 2) Nuts, 1/4" 3) Vespel ferrule, 1/4" 4) Union Tee, 1/8" 5) Septa, 11 mm, 50/pk 6) Tubing cutter 7) Monkey spanner 8) Wrench 1/2" & 9/16" 9) Wrench 1/2" & 7/16" 10) Wrench 3/8" & 7/16" 11) Wrench 1/4" & 5/16" 12) (+) screw driver 6x100 13) (+) screw driver 5x100 14) (-) screw driver 6x100 15) Leak detection fluid 16) 10 µL syringe 17) Copper tubing, 1/8" | 1601011110 | |
| ChroZen PAL RSI Headspace (Static) | ChroZen PAL RSI 850 system with headspace option consisting of: 1 pc base 850 X/Y unit with PAL Control Board 1 pc RSI-zhead 1 pc terminal (Manual Controller) 1 pc Safety guard 1 pc power and cables 1 pc Gas Purge Line for Headspace Technique built-in 1 pc Trayplate 3 pc Sample Racks for 15 vials of 10/20 mL 1 pc Headspace Syringe Tool for 2.5 mL syringe 1 pc Headspace syringe 2.5 mL (Gauge 23, Point Style AS) 1 pc Agitator Module | 6501011600 | |
| | Mounting Kit for YoungLin 6500 GC | PAL3-Kit-YL6500 | |
| PAL RSI Optional Module (ITEX - Dynamic HS) | PAL3 ITEX Tool, including: 1 pc ITEX Syringe 1.3mL (YL PAL3-SYH-209295) 2 pc ITEX Trap TENAX TA 80/100 mesh (YL PAL3-ITEX-Trap-TXTA100) | PAL3-ITEX-Kit | |
| | ITEX Syringe 1.3mL | PAL3-SYH-209295 | |
| CDS | YL-Clarity software for single instrument of YL GC | 5301011020 | |
| | MS module of YL-Clarity(Library option) | 5301011180 | |
| | Autosampler control of YL-Clarity | 5301011040 | |
| Column | DB-5MS (30m, 0.25 mm, 0.25 µm) | 122-5532 | |
| Acc | PAL System Vial 20CV, 20mL Clear Glass with Label, designed for the YL PAL Autosampler. | 75.5x22.5mm, 1st Class Hydrolytic Glass with flat finish for better sealing, fits ND18 Screw Caps, Pk of 100 Pcs | Vial-20-ND18-CG-100 |
| | PAL System Screw Cap 10CV and 20CV, designed for the YL PAL Autosampler. | ND18 Magnetic, Silver, Silicone white/PTFE blue Septa 1.5mm, Pk of 100 pcs | Cap-ND18-St-SP15-100 |