

SOLUTIONS BY



DETERMINATION OF PAH via GPC with On-line EVAporation and HPLC-FLD Measurement

Determination of PAH via GPC with on-line EVaporation and HPLC-FLD-measurement

Principle of the Method

The extracted samples, i.e. food, are handled automatically by the FREESTYLE GPC. They are processed via the on-line evaporation in the EVaporation module and filled into an HPLC vial ready for injection.

Procedure

The extracted, dried and filtered raw extract is adjusted to 10.0 mL and placed on the FREESTYLE system in a 16 mL vial that is closed with a cap/septum.

The sample is processed on the system using the method shown in the report on page 4.

The description of the process in brief:

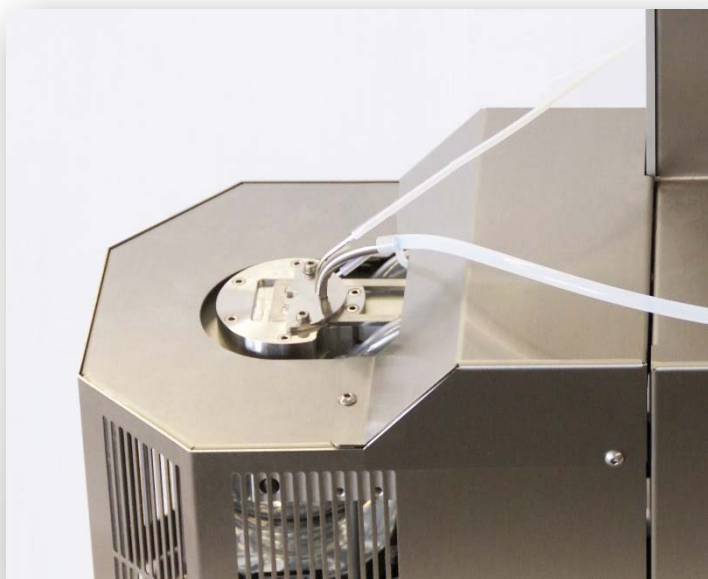
5.0 mL are applied by the calibrated sample loop on the GPC column. The matrix gets separated and the main run with the PAH is already concentrated on-line in the EVaporation module, that means while the process in the GPC is still running. After the evaporation an automated solvent exchange to Acetonitrile/1% 1-Butanol takes place. Finally the EVaporation chamber is rinsed, the concentrate is precisely adjusted to 1.0 mL and filled into an HPLC vial.

The measurement of the analytes happens on Thermo 3000 Ultimate with a fluorescence detector.




Devices and Materials

- | | |
|---|----------------------------|
| 1. FREESTYLE BASIC | P/N 12663 |
| 2. FREESTYLE EVAporation | P/N 13841 |
| 3. FREESTYLE GPC | P/N 12664 |
| 4. GPC-Column | P/N GPC10011 |
| 5. Frame for rack 11933 | P/N 11920 |
| 6. Rack for 16 mL vials | P/N 11933 |
| 7. Frame for rack 11920 | P/N 11915 |
| 8. Rack for GC vials | P/N 11920 |
| 9. 16 mL- vials | P/N V016 (100 pcs/pck) |
| 10. Screw cap for 16 mL vials | P/N V0016-SL (100 pcs/pck) |
| 11. Seals | P/N V0016-D (100 pcs/pck) |
| 12. GC vials | P/N V0001 (100 pcs/pck) |
| 13. Crimp cap for GC-vials with seal | P/N V0001-B (100 pcs/pck) |
| 14. Chiller | P/N 12060, 230 VAC, 50 Hz |
| 15. Ethyl acetate/Cyclohexane for the analysis of organic trace compounds | |
| 16. Aceton p.a. | |
| 17. Acetonitrile p.a. with 1 % 1-Butanol | |
| 18. Standard laboratory glassware and -apparatus | |



On-line connection from GPC directly into the EVAporation chamber – possibility of precise concentration up to 0.2 mL.

Parameterization of the Method on the FREESTYLE-System

|  | |
|--|---|
| LCTech FreeStyle - Report on Methods: GPC -> EVA Date: 10.10.2014 Time: 09:23:02 | |
| Name: PAH_Flex.fmt | |
| GPC - Method: PAH_Flex_GPC.gpc | Online =====> |
| EVA - Method: PAH_Flex_EVA.evp | |
| GPC: Type: | Loop Overfill |
| Source / Input of Sample: | from vial / vials Transfer Speed: 10 ml / min. |
| Volume of Sample Loop: | 5 ml |
| Flow: | 5 ml/min |
| Min. Pressure: | 0.3 bar |
| Max. Pressure: | 12 bar |
| Forerun: | 30 min. |
| Collection Time: | 20 min. |
| Tailing Time: | 0.2 min. |
| UV Recording: | OFF |
| Column: | D25_1.clm |
| Method of Fractioning: | GPC Pump OFF |
| Source vial list: | |
| | Nr.: 1 1 x 5.5 ml --> Type1@18 Sediment position: 0 mm |
| Collection vial list: | |
| | Nr.:1 1 x 240 ml --> Type1@240 Dump: no Online: yes |
| EVA: Temperature water heating | 40 °C Temperature bottom cone 50 °C |
| Sample input: Online from GPC or SPE process | |
| Batch volume = limit from where concentration starts: 5 ml (fix) + Waiting time: 10 min. | |
| Vacuum during GPC online sample input: 200 mbar | |
| Phase 1: Concentrate to level: 1 ml | |
| Vacuum absolute: 180 mbar | |
| Rinsing volume after phase 1: | 5 ml Rinsing steps: 1 x Solvent from Port: 8 (Acetonitrile-1-Butanol) |
| Skip phase 2 | |
| Time control for vacuum process: no | |
| to dryness: no | |
| Nitrogen blow-down: no | |
| Remove Aliquot: no | |
| Solvent exchange: yes | |
| At reach of level: 2 ml | |
| Solvent addition per exchange: | 5 ml Solvent from Port: 8 (Acetonitrile-1-Butanol) |
| Number of solvent exchanges: | 1 End volume after exchange: 0.5 ml |
| Vacuum starts at level: | 180 mbar abs. Gradient of vacuum: -20 mbar/min Vacuum end at level: 100 mbar abs. |
| Rinsing, filling up, mixing and transfer into vials: | |
| Rinsing volume at the end: | 0.5 ml Rinsing steps: 1 x Solvent from Port: 7 (Acetonitrile) |
| Fill up to volume: | <input type="text" value="1 ml"/> Way of mixing: suck up and release |
| Concentrate: into vials | |
| Nr.: | 1 1 [each] Type: Type1@1 ml Volume per vial 1 ml |
| Fill Quantitativ: no | |
| 1. Cleaning cycle | |
| Rinsing volume: | 5 ml Rinsing steps: 1 x Solvent from Port: 9 (Acetone) |
| 2. Cleaning cycle | |
| Rinsing volume: | 5 ml Rinsing steps: 1 x Solvent from Port: 1 (EA/CY 1/1) |

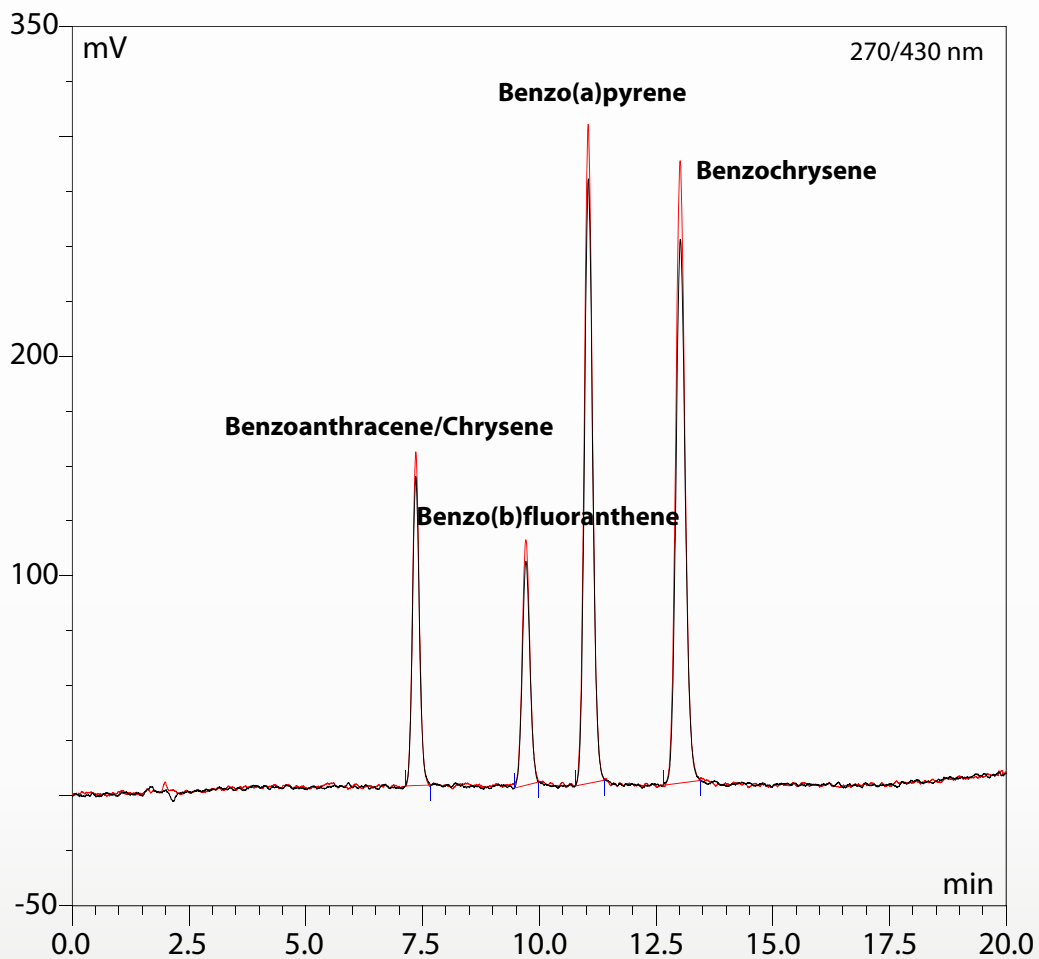
Results

The process time of a sample including solvent exchange and transfer into a GC vial is just 1 h 35 min.

Remark: The process time takes at least 10 minutes less, if the sample is measured in a GC and a solvent exchange isn't necessary

Recovery rates of an exemplary experiment (n=3)

| Description | Recovery Rate |
|--------------------------|---------------|
| Benzoanthracene/Chrysene | 91 ± 4 % |
| Benzo(b)fluoranthene | 90 ± 4 % |
| Benzo(a)pyrene | 85 ± 5 % |
| Benzo(chrysene) | 83 ± 8 % |



The figure shows the overlapping of two fluorescence chromatograms: external PAH standard (red) and a finished sample (black).

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