KONIK GROUP

Khromatography® + SpeKtrometry®
Analytical Solutions + Control

AN EVALUATION AND COMPARISON OF THREE DIFERENT SAMPLING PROCEDURES: STATIC HEAD SPACE, DINAMIC HEAD SPACE (PURGE & TRAP), AND SOLID PHASE MICROEXTRACTION IN THE INNOVATIVE MULTIMODE AUTOSAMPLER KONIK K-MAS5.

ROGER GIBERT
SERVICE & TECHNICAL SUPPORT DIRECTOR
KONIK-TECH
BARCELONA, SPAIN

SAMPLE PREPARATION TECHNIQUES

- EXTRACTION AND DIRECT INJECTION
- STATIC HEAD SPACE (HS)
- DYNAMIC HEAD SPACE - PURGE & TRAP (P&T)
- SOLID PHASE MICROEXTRACTION (SPμ E)
- SPμE + HS

MULTIMODE AUTOSAMPLER

KONIK K-MAS5:

+ 5 OPERATIONAL MODES

COMPOUNDS

- BTEX
- HALOMETHANES

MATRIXES

- DRINKING WATER
- SURFACE WATER
- GROUND WATER
- MUNICIPAL DISCHARGES
- INDUSTRIAL DISCHARGES

KONIK GROUP

Khromatography® + SpeKtrometry®
Analytical Solutions + Control

	EPA (ENVIRONMENTAL PROTECTION AGENCY)				WHO (WORLD HEALTH ORGANIZATION)		
	METHODS	TECHNIQUE	RANGE	MDL	TECHNIQUE	RANGE	MDL
BENZENE TOLUENE ETHYLBENZENE o,p,m- XYLENE	502.2 524.2 602 624 1624	P & T - GC - ELCD, MS, PID	0.02 - 200 µg/L	0.01 - 0.2 µg/L	P & T - GC - MS, PID	0.02 - 1500 µg/L	0.2 µg/L
CHLOROFORM DIBROMOCHLOROMETHANE BROMODICHLOROMETHANE BROMOFORM	502.2 524.2 624 1624 551 551.1 601	P & T; Direct Inj. - GC - ECD		(MS: <25 µg/L)	Direct Inj. - GC - ECD		0.02-1.0 µg/L

ANALYTICAL CONDITIONS

- SAMPLE: BTEX water spiked

- HRGC: HRGC KONIK 4000B SERIES

COLUMN:	J&W DB-624, 30 m, 0.53 mm, 3 μm
CARRIER:	Helium, ct. Flow 4 ml/min
INJECTOR:	250ºC, inj. Mode: conventional injector
OVEN:	35ºC (1min); 10ºC/min; 200ºC (1min)
DETECTOR:	FID, 250 ºC: detector gases: H2 at 38ml/min, Air at 220ml/min

- AUTOSAMPLER: KONIK K-MAS5 MULTIMODE AUTOSAMPLER (HRGC, HS, P&T and SPμE mode)

DIRECT INJECTION

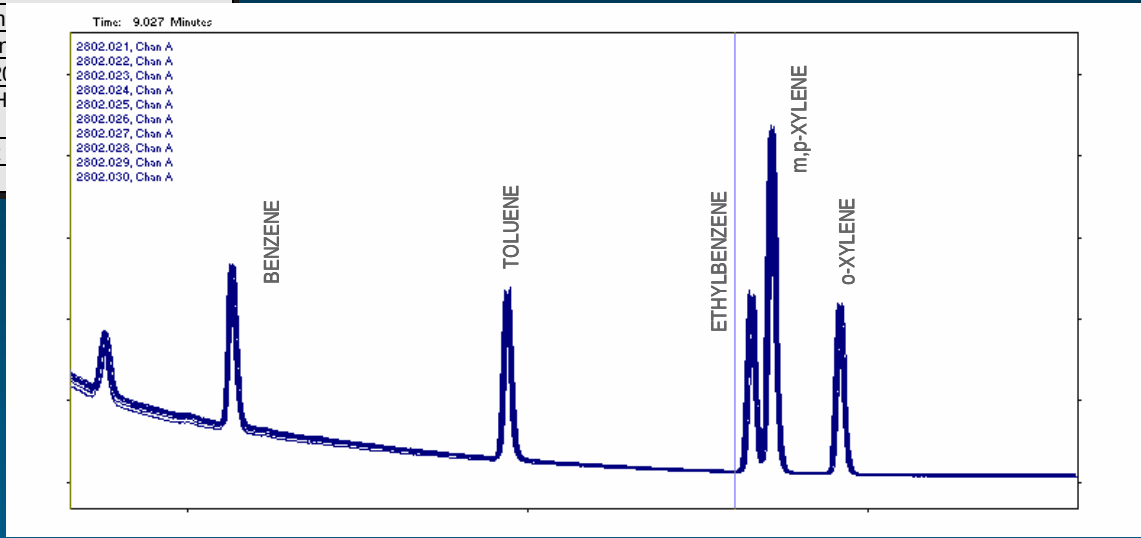
- √ High injection repeatability
- √ Sample preparation step: Liquid - Liquid Extraction with a solvent and injection of Xµl of the extract
- √ Solvent use
- √ Poor recovery (need of preparation sample internal standard)
- √ Poor sensitivity

KONIK GROUP

Khromatography® + SpeKtrometry®
Analytical Solutions + Control

DIRECT INJECTION

SAMPLE		6ppm BTEX (in pentane)
GC	Column:	J&W- DB-624, 30m, 0.53mm, 3µm (ref.: 125-1334)
	Carrier:	He; constant Flow 4ml/m
	Injector:	250ºC, inj. mode: conver
	Oven:	35ºC (min); 10ml/min; 2(
	Detector:	250ºC; detector gases: H 220ml/min
Autosampler:	Tray:	105vials of 2ml; ambient
	Inj. volum	1ul



	Rt (min)	RSD Rt (n=10)	RSD Area (n=10)	MDL (ppm)
BENZENE	5.329	0.29	1.9	1.7
TOLUENE	7.347	0.21	2.3	0.5
ETHYLBENZENE	9.144	0.17	2.1	0.4
p,m- XYLENE	9.293	0.15	2.3	0.2
o-XYLENE	9.793	0.15	2.2	0.4

STATIC HEADSPACE

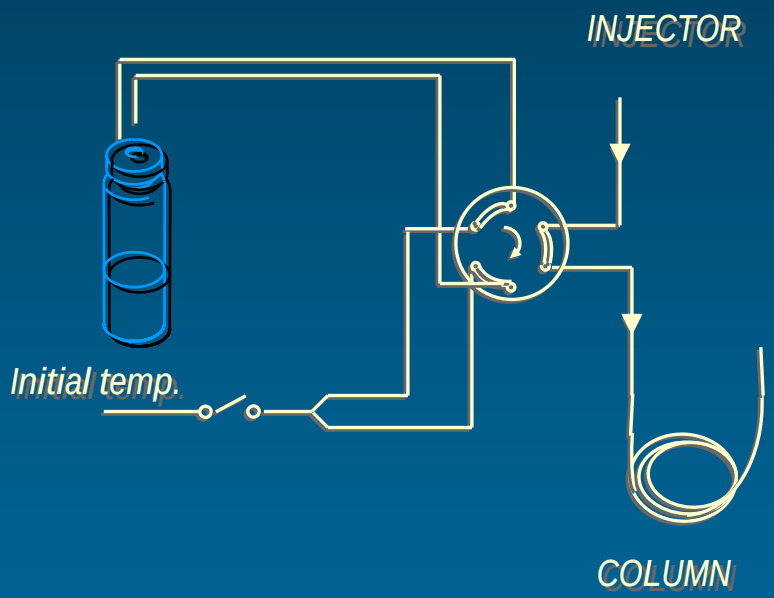
- √ No sample preparation step
- √ Minimum analysis time (with intelligent time programming)
- √ Solvent Saving
- √ Need the use of internal standard (difficult to obtain an standard matrix)

KONIK GROUP

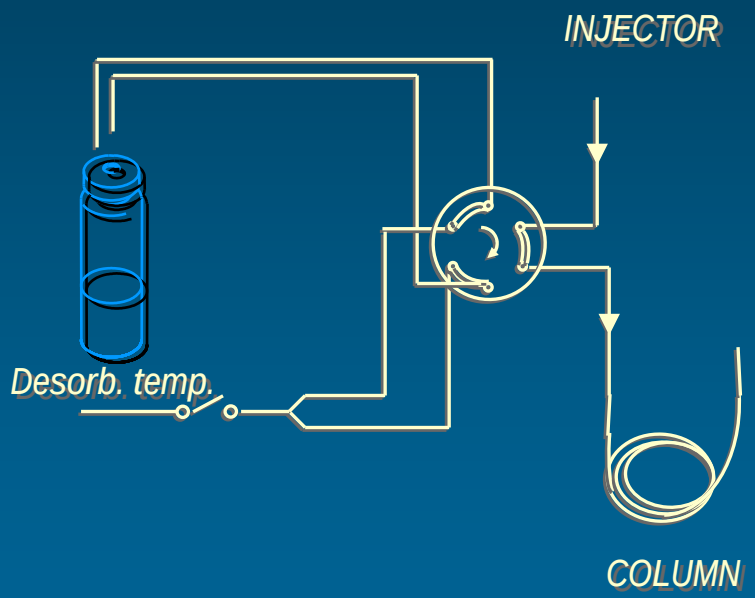
Khromatography® + SpeKtrometry®
Analytical Solutions + Control

STATIC HEADSPACE

1. Initial



2. Desorbtion

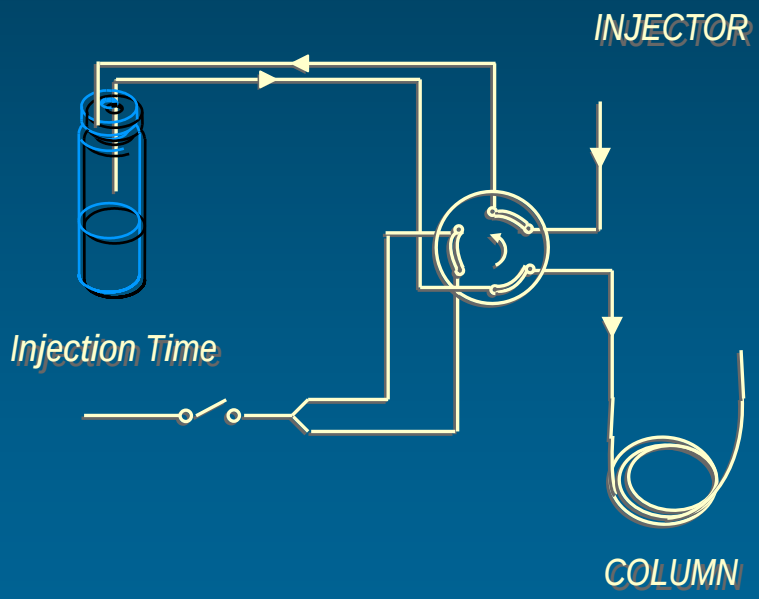


KONIK GROUP

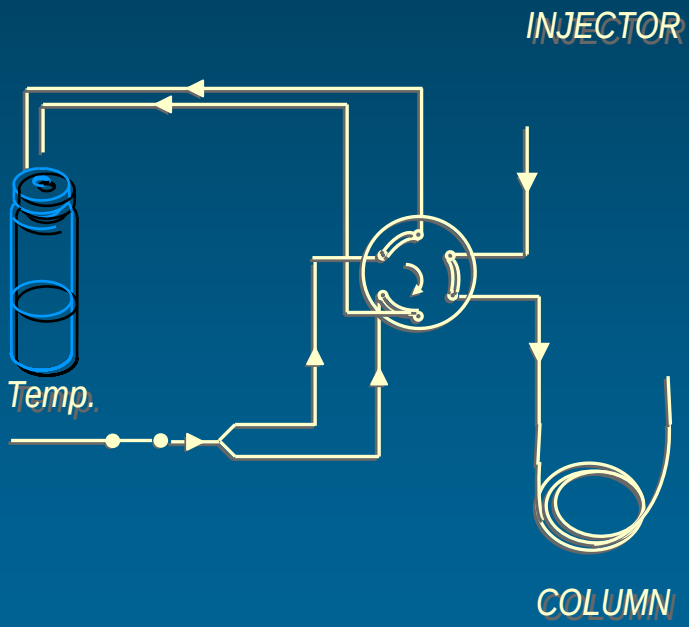
Khromatography® + SpeKtrometry®
Analytical Solutions + Control

STATIC HEADSPACE

3. Injection



4. Cleaning

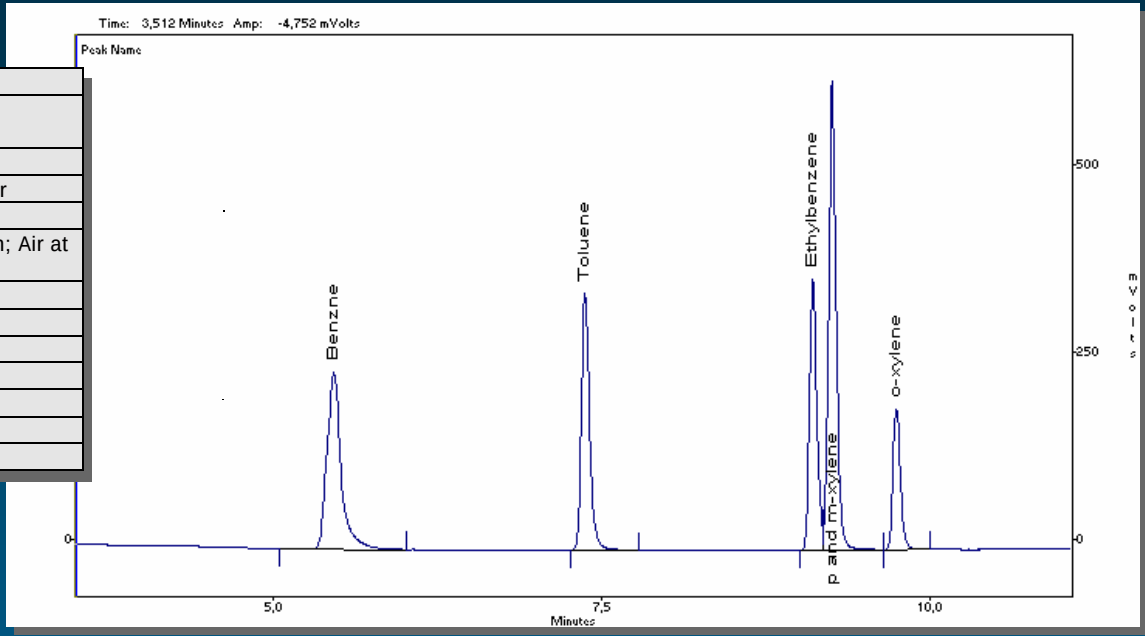


KONIK GROUP

Khromatography® + SpeKtrometry®
Analytical Solutions + Control

STATIC HEADSPACE

SAMPLE		0.5 ppm BTEX
GC	Column:	J&W- DB-624, 30m, 0.53mm, 3µm (ref.: 125-1334)
	Carrier:	He; constant Flow 4 ml/min
	Injector:	250°C, inj. mode: conventional injector
	Oven:	35°C (1min); 10°C/min; 200°C (1min)
	Detector:	250°C; detector gases: H_2 at 38ml/min; Air at 220ml/min
HeadSpace:	Tray:	32vials of 10ml; ambient temperature
	T desorb:	80°C
	T line:	160°C
	T valve:	160°C
	t desorb:	10min
	t inj:	20s
	Cleaning gas:	He 8psi



	Rt (min)	RSD Rt (n=10)	RSD Area (n=10)	MDL (ppb)
BENZENE	5.523	0.30	6.8	0.8
TOLUENE	7.455	0.29	9.5	1.5
ETHYLBENZENE	9.210	0.22	9.6	4.5
p,m- XYLENE	9.356	0.22	5.9	3.5
o- XYLENE	9.855	0.21	10.4	6.9

DYNAMIC HEAD SPACE - PURGE & TRAP

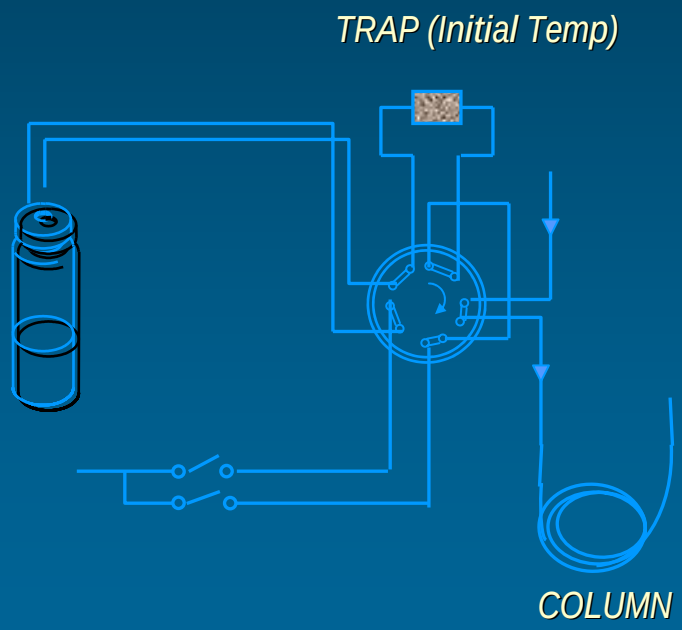
- √ No sample preparation step
- √ Solvent Saving
- √ Low MDL
- √ High Selectivity depending on trap
- √ Need the use of internal standard (difficult to obtain an standard matrix)

KONIK GROUP

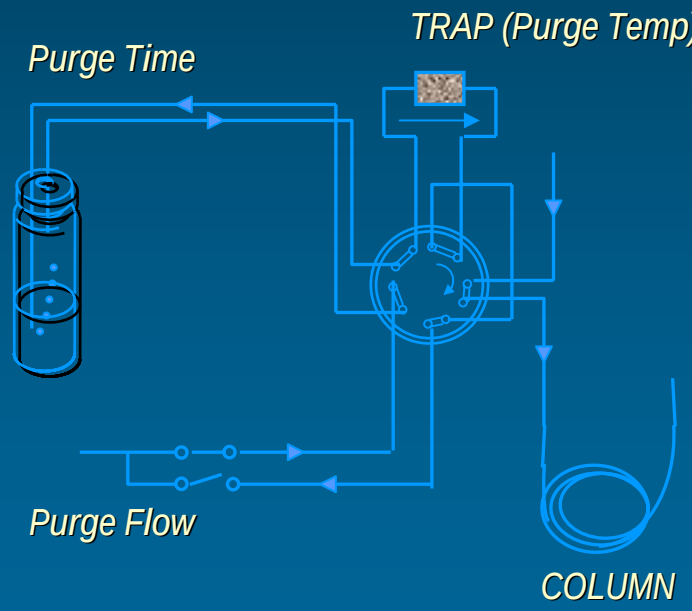
Khromatography® + SpeKtrometry®
Analytical Solutions + Control

DYNAMIC HEAD SPACE - PURGE & TRAP

1. Initial

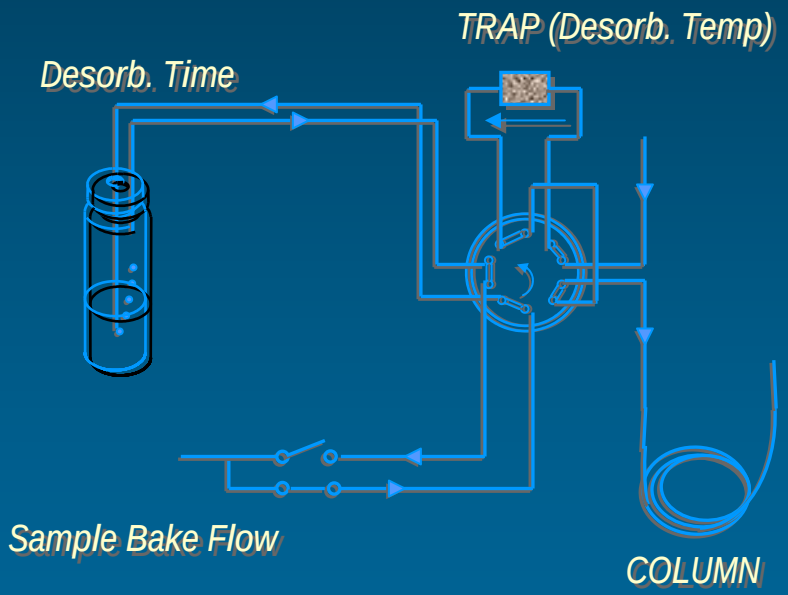


2. Purge

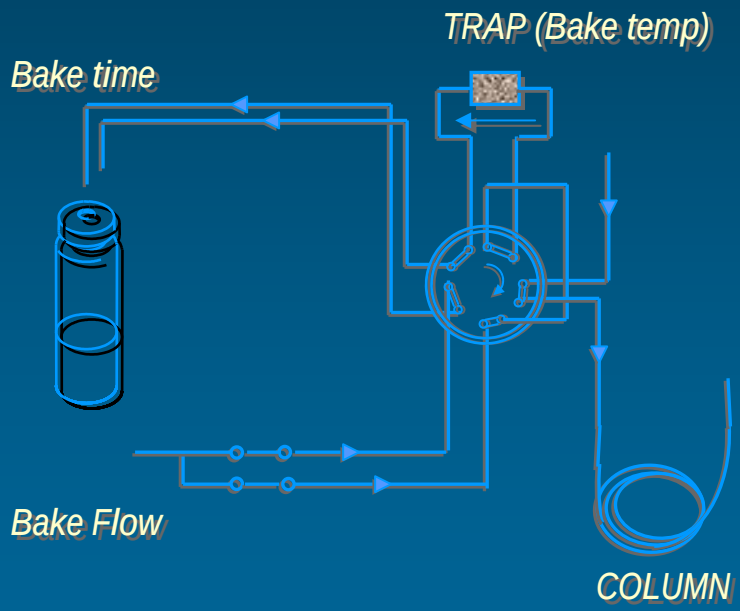


DYNAMIC HEAD SPACE - PURGE & TRAP

3. Desorbtion & Vent



4. Cleaning

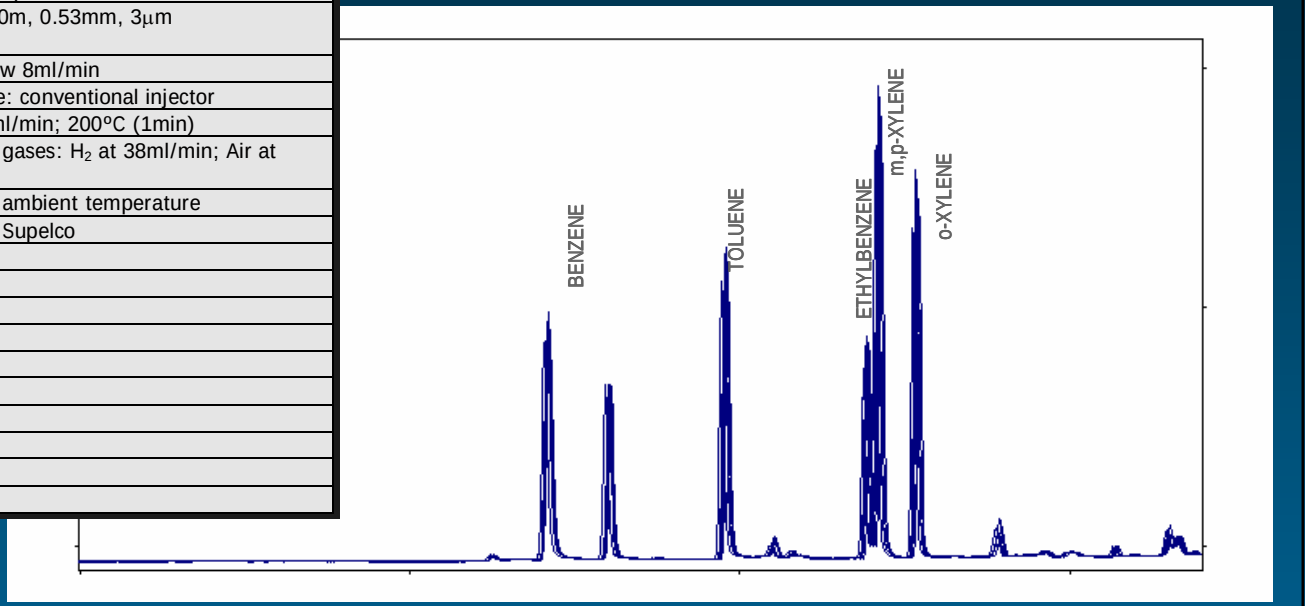


KONIK GROUP

Khromatography® + SpeKtrometry®
Analytical Solutions + Control

DYNAMIC HEAD SPACE - PURGE & TRAP

SAMPLE		6ppb BTEX (n=10)
GC	Column:	J&W- DB-624, 30m, 0.53mm, 3µm (ref.: 125-1334)
	Carrier:	He; constant Flow 8ml/min
	Injector:	250°C, inj. mode: conventional injector
	Oven:	35°C (min); 10ml/min; 200°C (1min)
	Detector:	250°C; detector gases: H_2 at 38ml/min; Air at 220ml/min
Purge & Trap:	Tray:	32vials of 20ml; ambient temperature
	Trap:	BTEX Trap from Supelco
	T trap initial:	25°C
	T purge:	25°C / 20min
	Purge Flow:	40 ml/min
	T predesorb:	180°C
	T desorb:	200°C
	T line:	200°C
	T valve:	200°C
	t desorb:	1min
	Cleaning Temp:	220°C / 10min
	Cleaning gas:	He 40ml/min

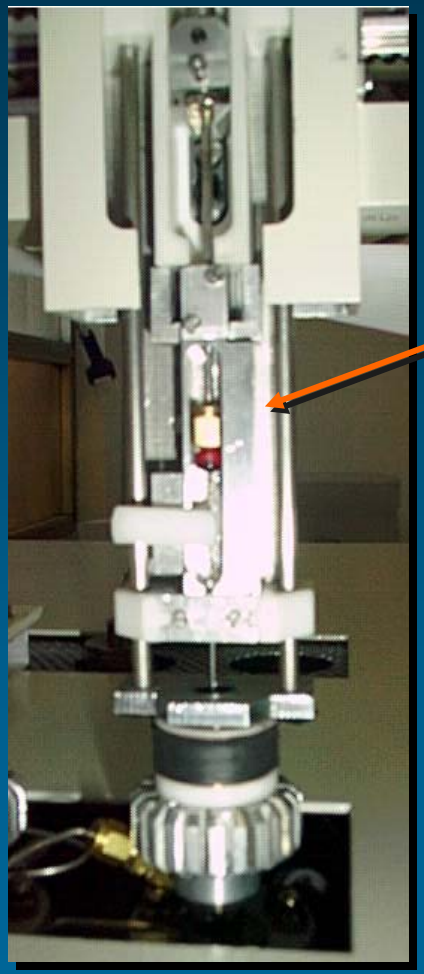


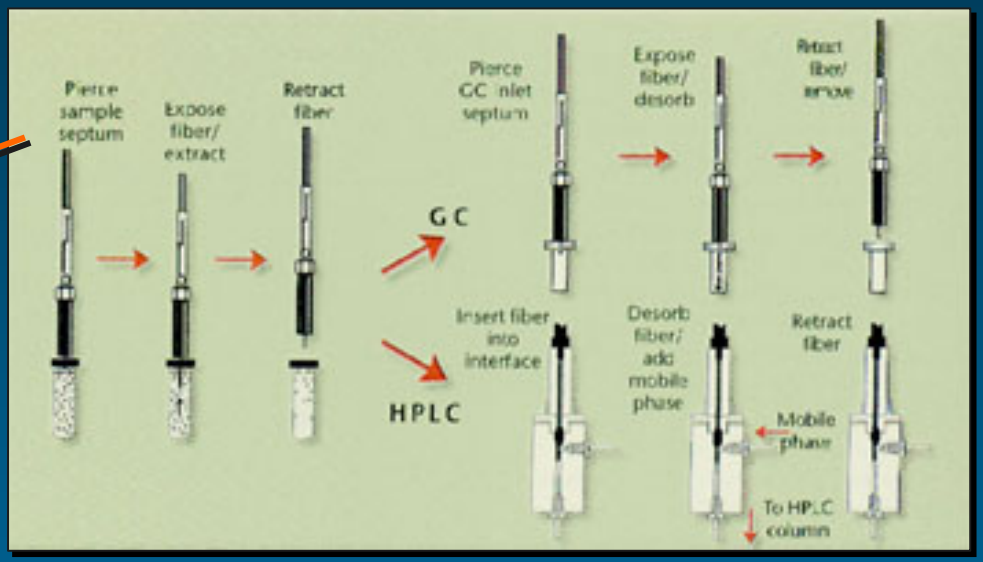
	Rt (min)	RSD Rt (n=10)	RSD Area (n=10)	MDL (ppt)
BENZENE	5.123	0.39	2.7	60
TOLUENE	7.803	0.32	3.3	8
ETHYLBENZENE	9.953	0.26	6.5	30
p,m- XYLENE	10.129	0.26	6.5	20
o-XYLENE	10.690	0.24	5.9	30

SOLID PHASE MICROEXTRACTION (SPμE)

- √ Easy and Reliable Extraction Concentration Method
- √ Solvent Saving
- √ High Selectivity depending on extracting fiber

SOLID PHASE MICROEXTRACTION (SPμE)



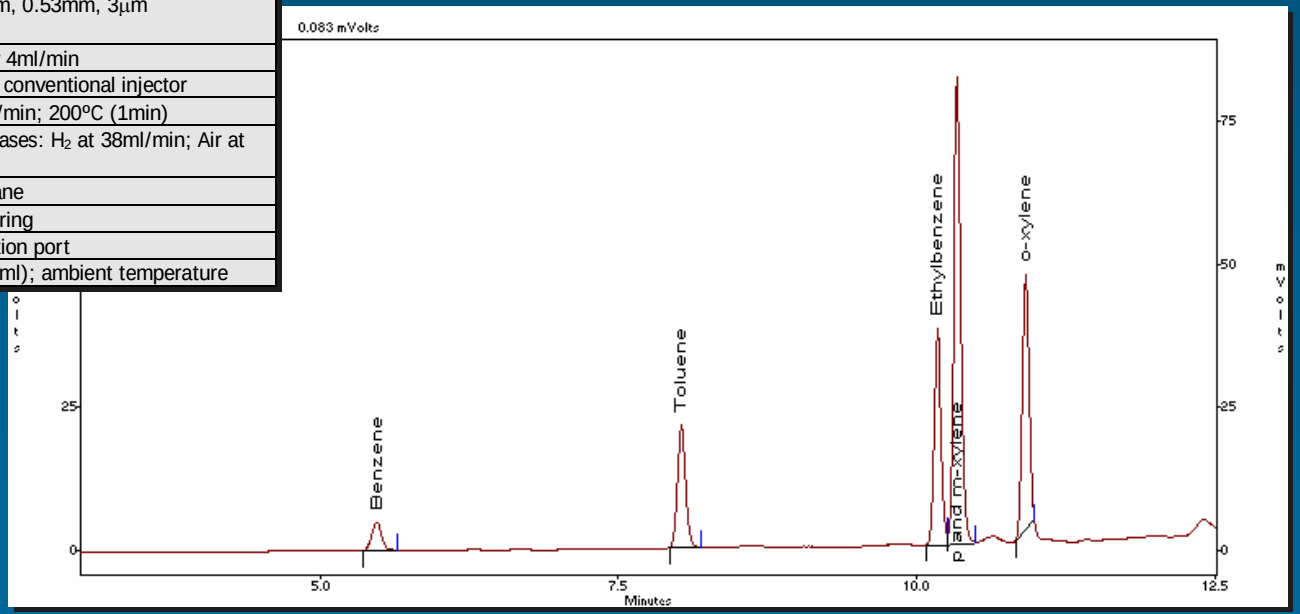


FROM SUPELCO CATALOG

SOLID PHASE MICROEXTRACTION (SPμE)

	Rt (min)	RSD Rt (n=10)	RSD Area (n=10)	MDL (ppb)
BENZENE	5.495	0.31	8.3	4.7
TOLUENE	8.040	0.25	11.2	1.0
ETHYLBENZENE	10.187	0.22	11.0	0.6
p,m- XYLENE	10.353	0.19	7.5	0.3
o-XYLENE	10.929	0.28	12.4	0.5

SAMPLE		6ppb BTEX (n=10)
GC	Column:	J&W- DB-624, 30m, 0.53mm, 3μm (ref.: 125-1334)
	Carrier:	He; constant Flow 4ml/min
	Injector:	250°C, inj. mode: conventional injector
	Oven:	35°C (min); 10ml/min; 200°C (1min)
	Detector:	250°C; detector gases: H_2 at 38ml/min; Air at 220ml/min
Purge & Trap:	Fiber:	Polydimethylsiloxane
	Tadsor.:	80°C; 10 min; stirring
	tdesorb.:	5 min in the injection port
	Tray:	32vials of 10ml (2ml); ambient temperature



CONCLUSIONS

TOLUENE	DIRECT INJ.	HS	P & T	SPµE	SPµE - HS
Rt	7.347	7.455	7.803	8.040	-------
RSD Rt	0.21	0.29	0.32	0.25	------
RSD AREA	2.3	9.5	3.3	11.2	------
MDL (ppb)	500	1.5	0.008	1.0	------
Recovery	√	√	√	√	√
Analysis Time	10 min + prep. Step	10 min	31 min	20 min	20 min
Solvent Use	Yes	No	No	No	No