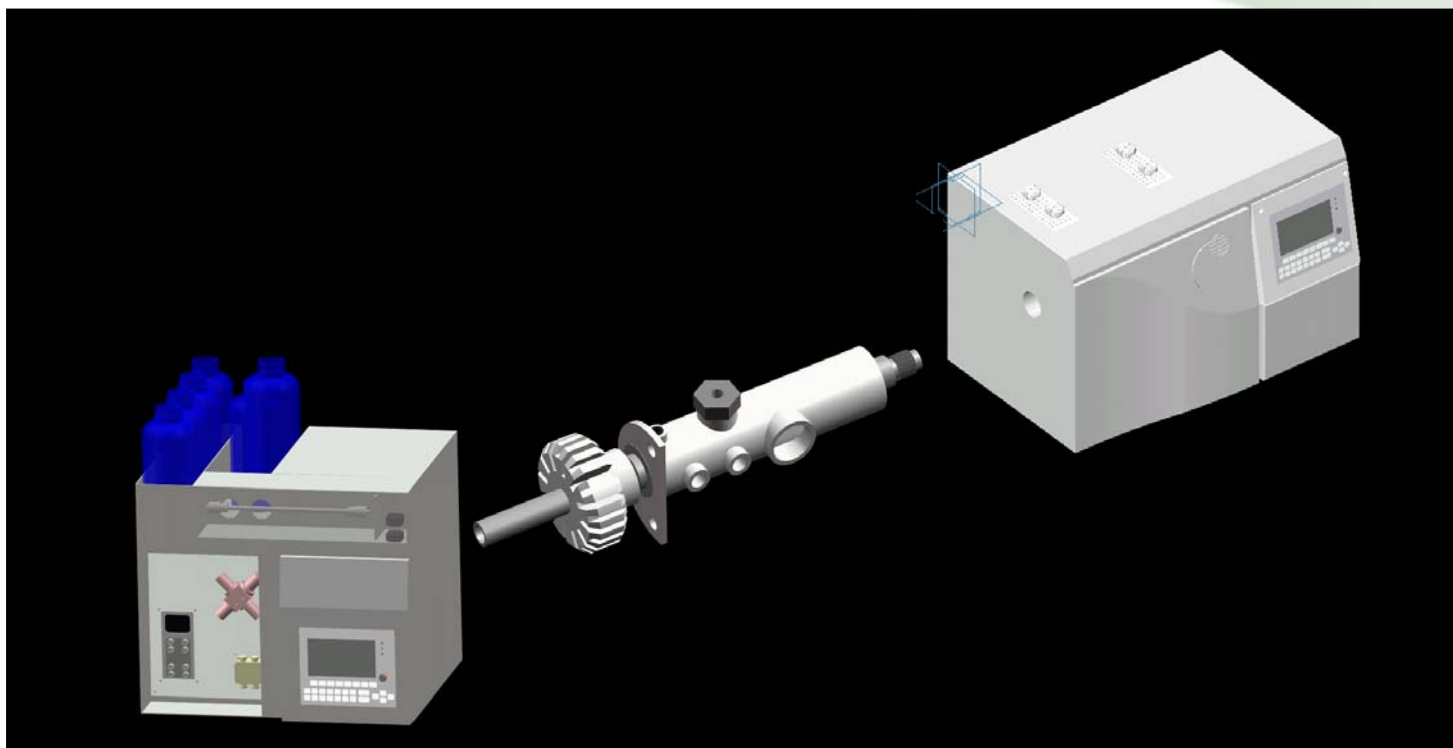


Automated clean-up and analysis of complex samples by on-line LC-GC using the KONIK K2 system



KONIK HPLC-HRGC K2 SYSTEM

Large Volume Injection in GC On-Line coupling LC-GC



SAMPLE PREPARATION METHODS

- Liquid-Liquid Extraction
- Soxhlet Extraction
- Column Chromatography
- Microwave Extraction
- Solid Phase Extraction
- Supercritical Fluid Extraction
- ...

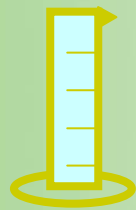
- Organic Solvents
- Time-consuming
- Low Recovery
- Internal Standard required



Olive Oil Sample



Extraction



Concentration



HRGC



Olive Oil Sample



HPLC



HRGC

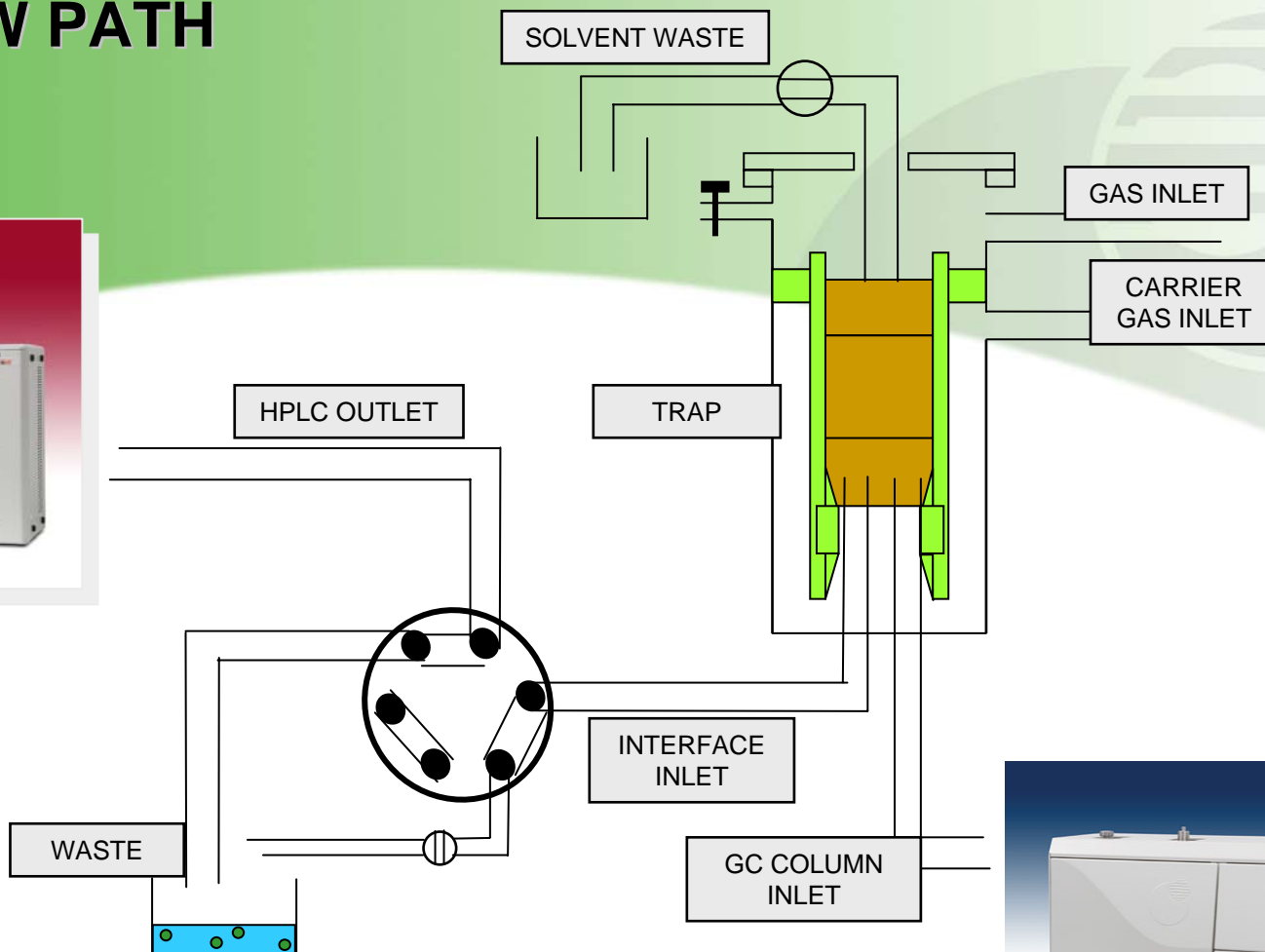


Olive Oil Sample

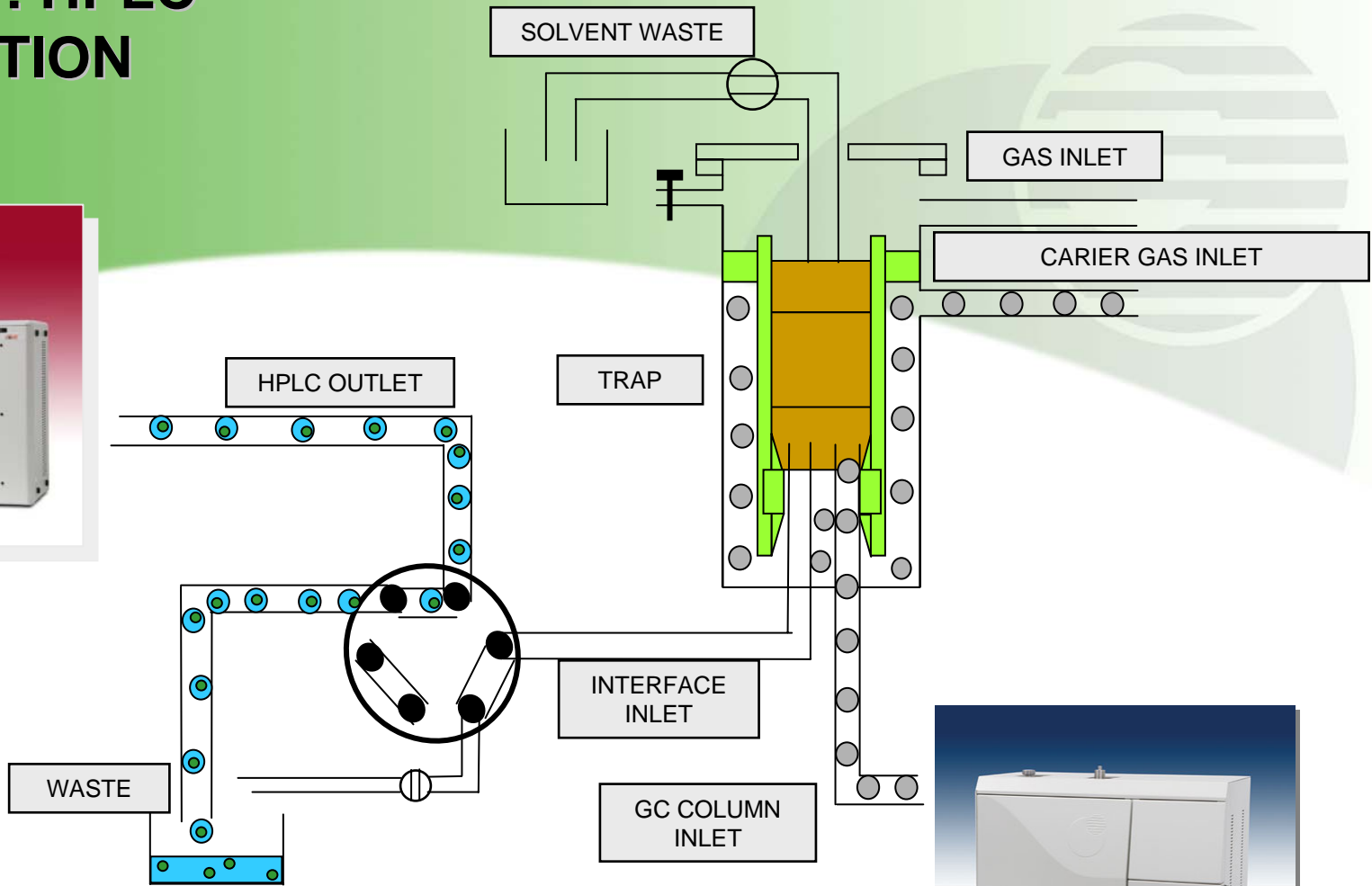


K2

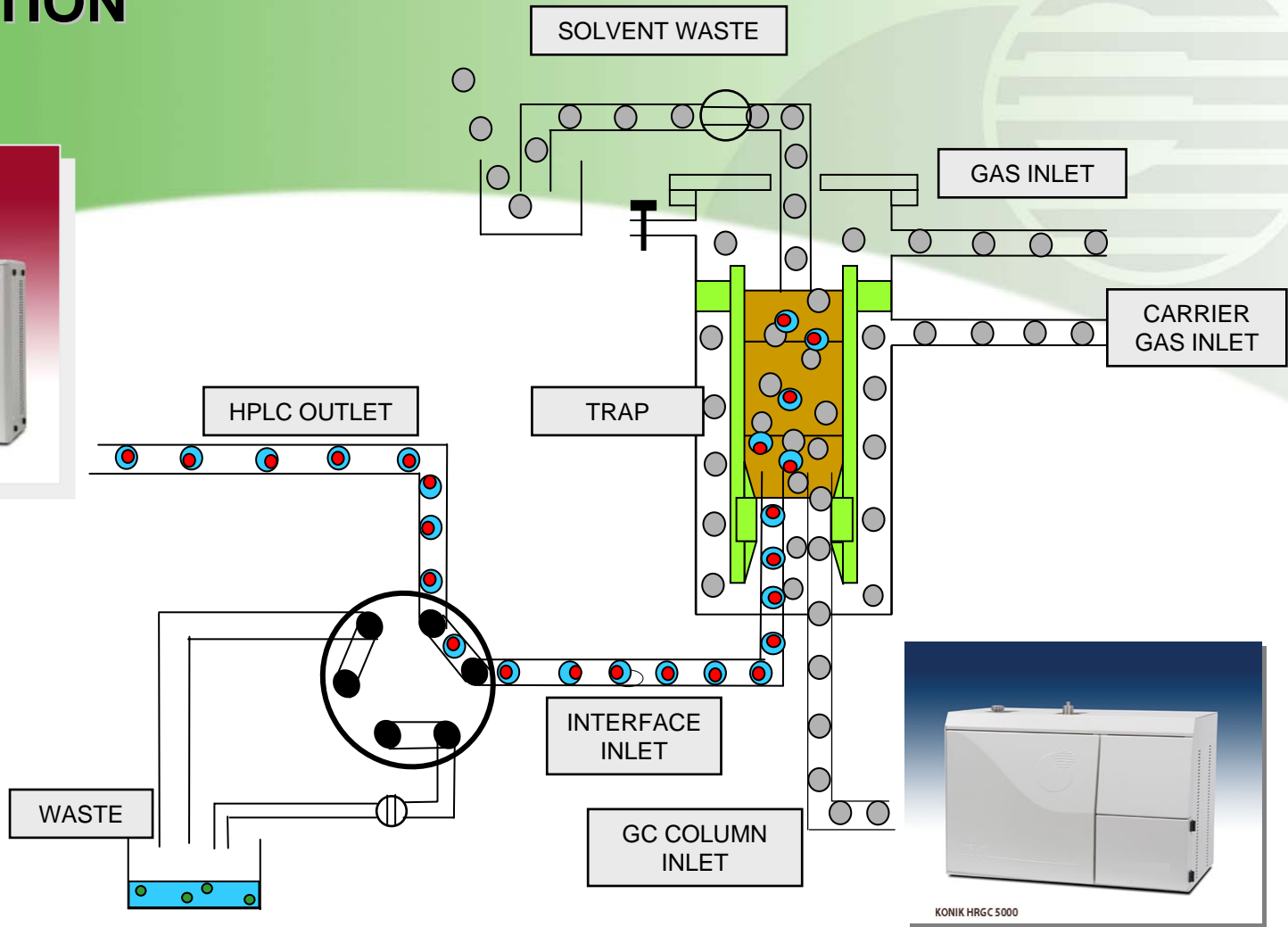
K2 FLOW PATH



1st STEP: HPLC SEPARATION



2nd STEP: ABSORPTION



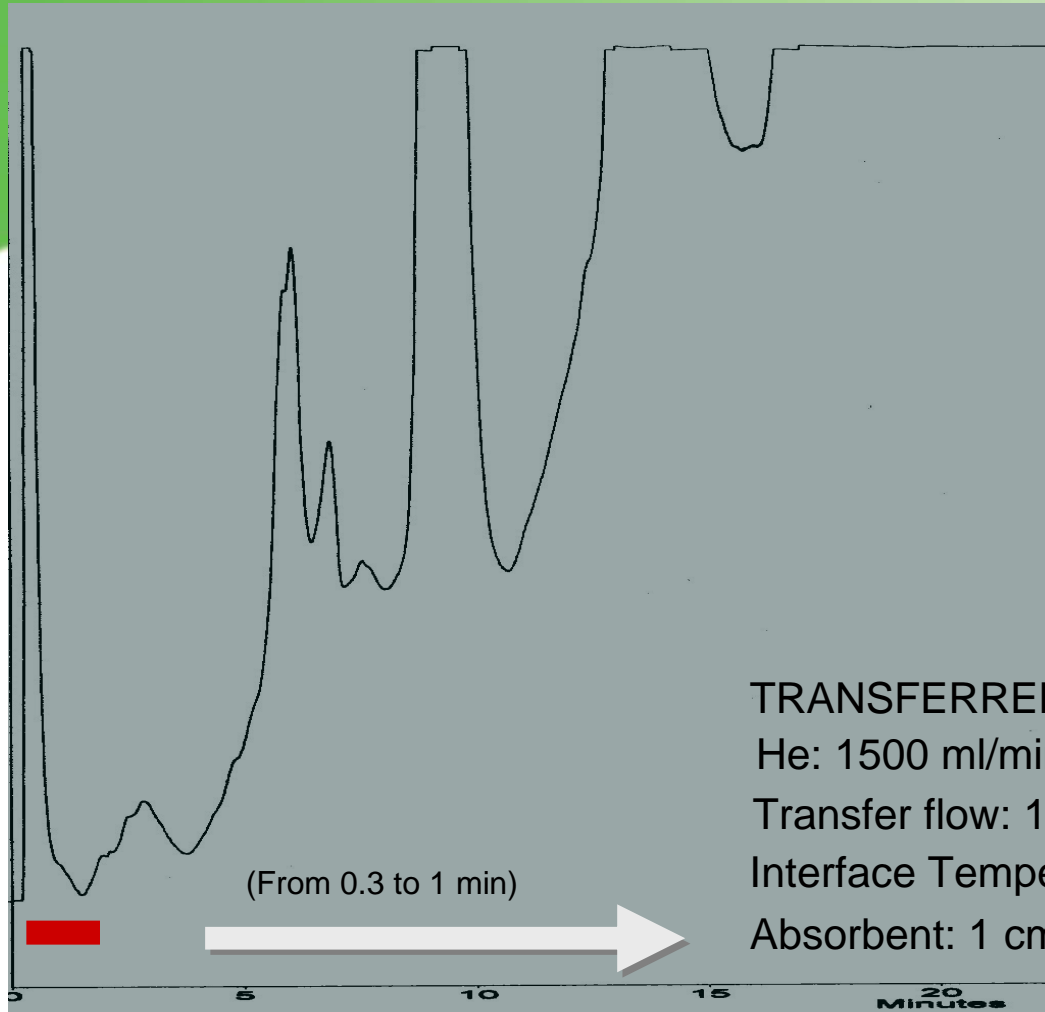
HPLC+HRGC K2 APPLICATIONS

- ANALYSIS OF PESTICIDE RESIDUE IN OLIVE OIL.
- ANALYSIS OF PAH IN MINERAL OIL.
- ANALYSIS OF FREE FATTY ACIDS IN PLANT EXTRACTS.
- DETERMINATION OF HAZARDOUS PESTICIDES IN URINE.
- ANALYSIS OF PCBs IN TRANSFORMER OIL.

HPLC+HRGC K2 APPLICATIONS

- **PITTCON 01 (New Orleans)**: REVERSED HPLC-HRGC COUPLING VIA A PATENTED THROUGH OVEN TRANSFER ADSORPTION DESORPTION (TOTAD[®]) INTERFACE . APPLICATION TO THE FAST, DIRECT AND RELIABLE ANALYSIS OF PESTICIDE RESIDUE IN OLIVE OIL.

PESTICIDE RESIDUES IN OLIVE OIL



HPLC CONDITIONS

Sample: 20 μ l olive oil fortified with
1 μ g/ml

Column: C4 (50 x 4.6 mm ID)

Flow: 2 ml/min CH₃OH/H₂O gradient

UV-VIS: 205 nm

TRANSFERRED VOLUME: 2800 μ l

He: 1500 ml/min

Transfer flow: 100 μ l/min

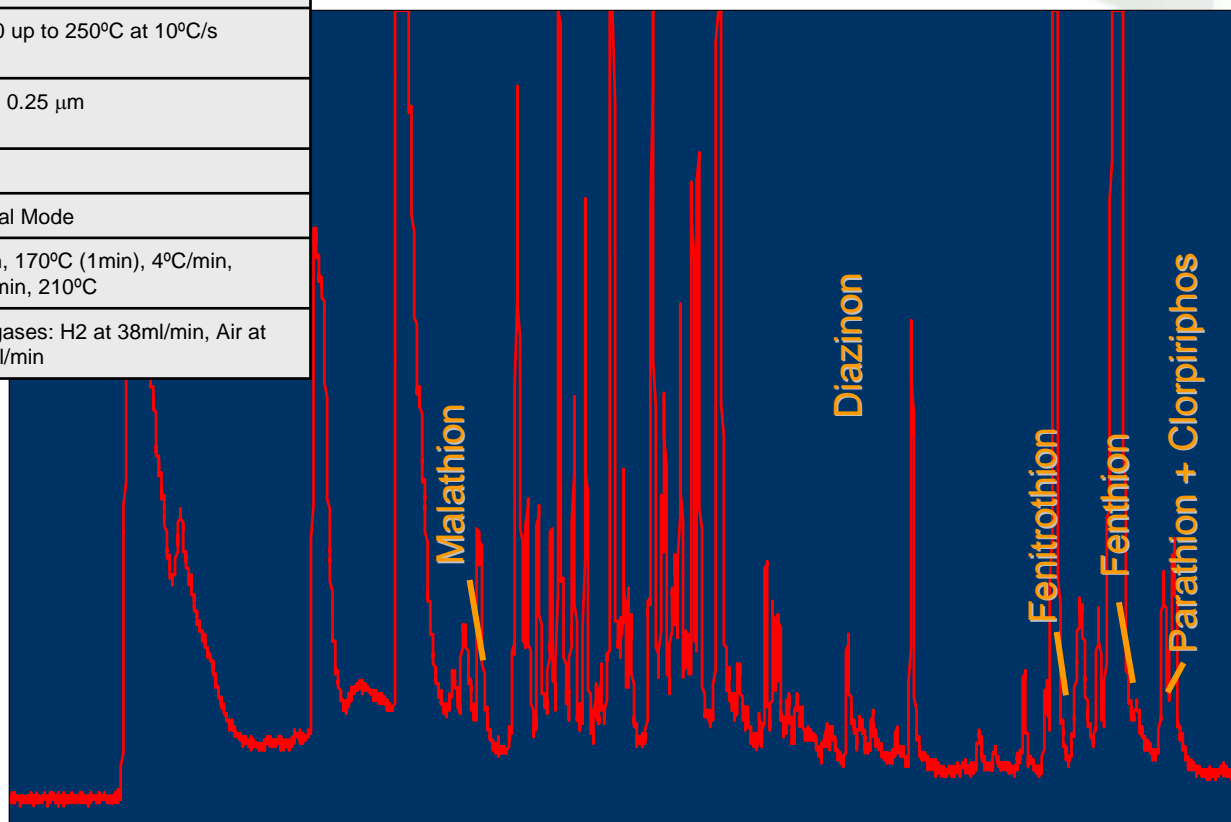
Interface Temperature: 70 °C

Absorbent: 1 cm Tenax TA

PESTICIDE RESIDUES IN OLIVE OIL



SAMPLE	1 ppm fortified olive oil
HPLC CONDITIONS	Column: C4 (50 x 4.6 mm ID)
	Mobile Phase: CH ₃ OH:H ₂ O gradient; cleaning: 100% IPA
	Flow: 2 ml/min
	Volume.: 20 µl
INTERFACE CONDITIONS	Flow: 0.1 ml/min (He: 1500 ml/min)
	Absorbent: 1 cm TENAX TA
	Temp: 70°C (absorption); 100 up to 250°C at 10°C/s (desorption)
HRGC CONDITIONS	GC Column: KAP-5, 30m, 0.32mm, 0.25 µm
	Carrier: Helium at 1.8 ml/min
	Injector: 70°C, Inj.: Conventional Mode
	Oven: 40°C (1min), 20°C/min, 170°C (1min), 4°C/min, 190°C (10min), 10°C/min, 210°C
	Detector: FID, 250°C, detector gases: H ₂ at 38ml/min, Air at 220ml/min, He at 25ml/min



PESTICIDE RESIDUES IN OLIVE OIL

PRECISION (n=5)

Pesticide	Rt RSD	Area RSD
Malathion	0.084	14.2
Diazinon	0.065	9.0
Fenitrothion	0.077	13.0
Fenthion	0.064	14.8
Parathion + Chlorpirifhes	0.054	11.6

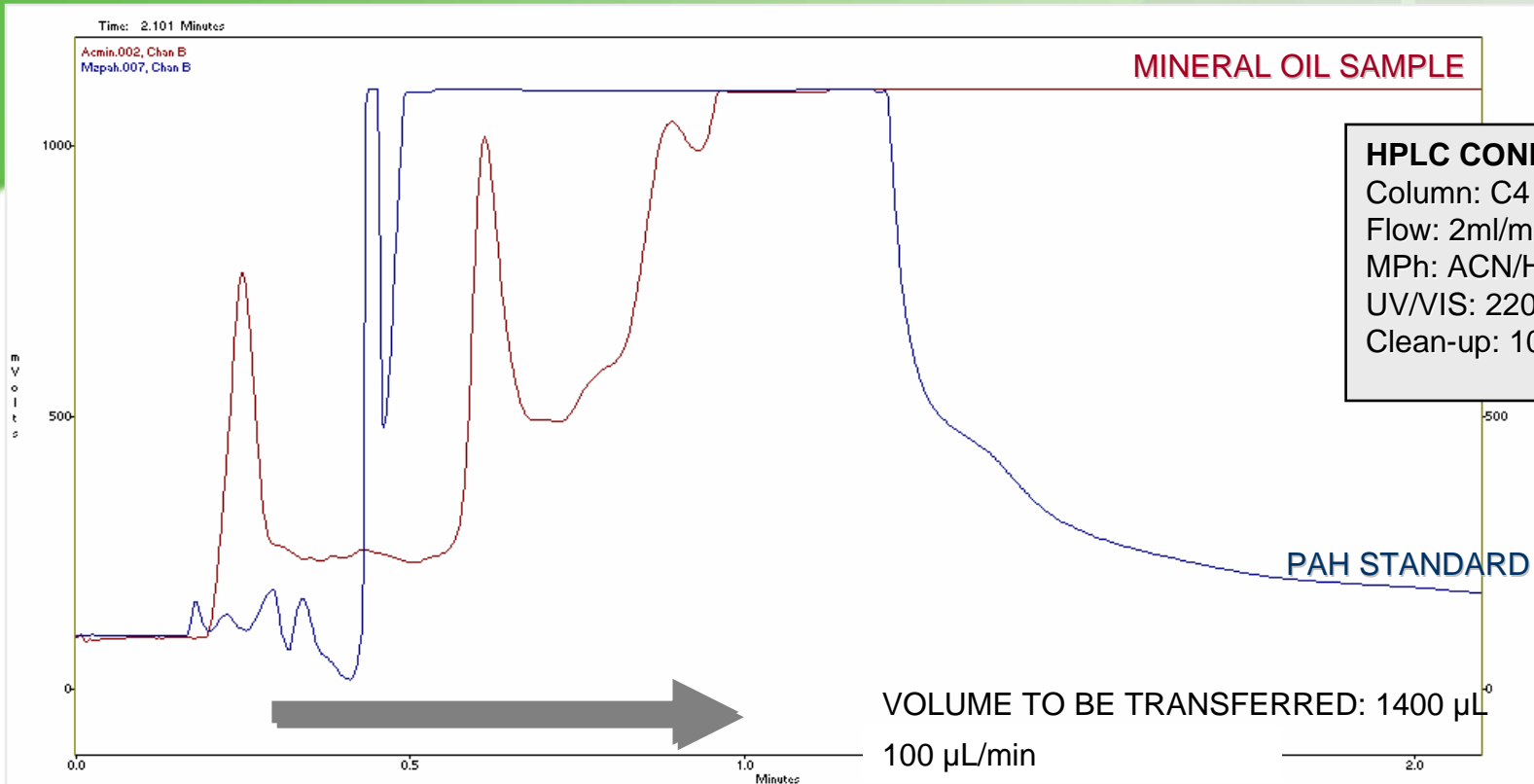
DETECTION LIMITS ($\mu\text{g/ml}$) (S/N: 5)

Pesticide	MDL ($\mu\text{g/ml}$)
Malathion	0.31
Diazinon	0.072
Fenitrothion	0.13
Fenthion	0.14
Parathion	0.35
Chlorpirifhes	0.41

HPLC+HRGC K2 APPLICATIONS

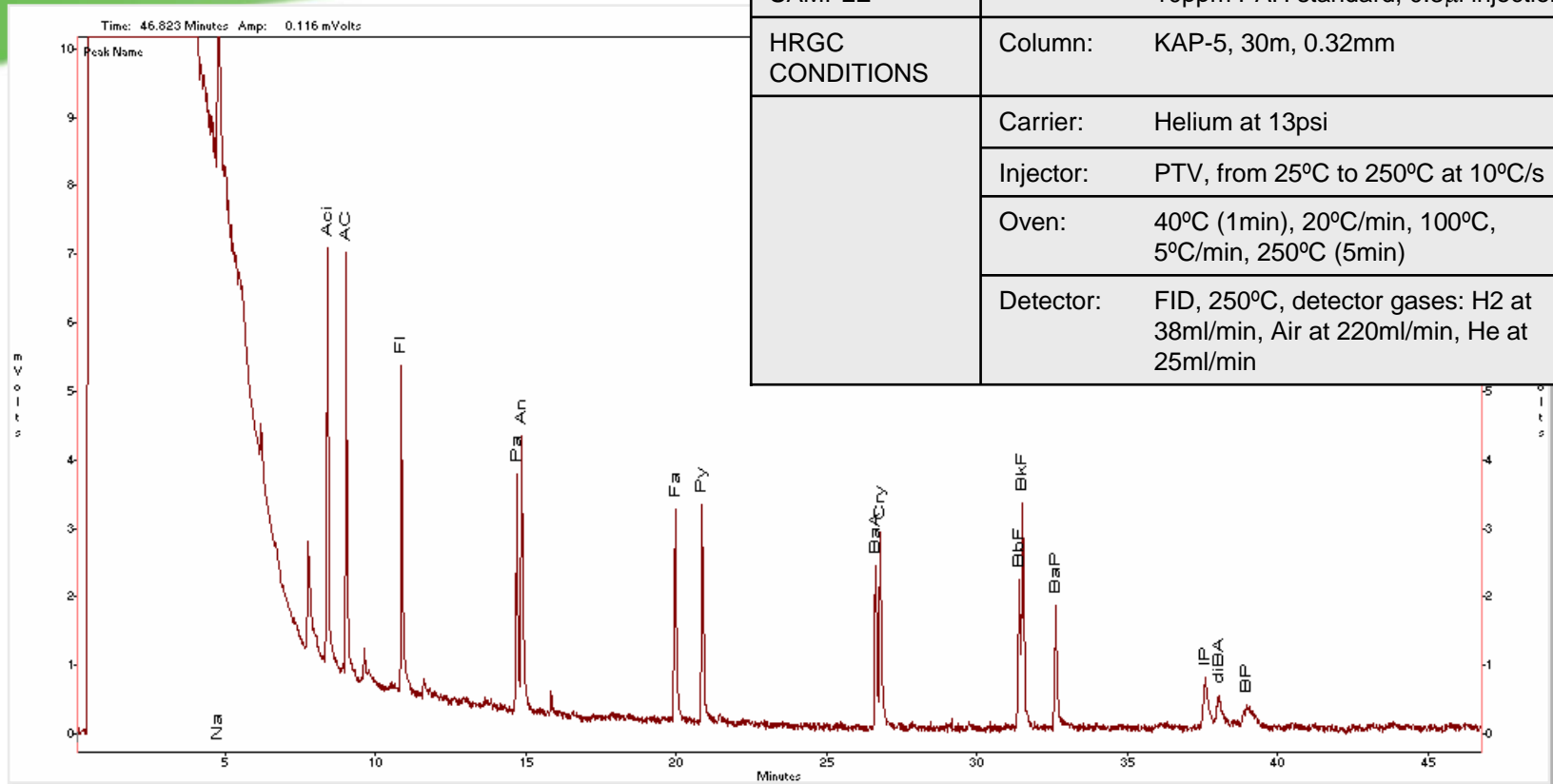
- **PITTCON 03 (New Orleans)**: REVERSED HPLC-HRGC COUPLING VIA A PATENTED THROUGH OVEN TRANSFER ADSORPTION DESORPTION (TOTAD[®]) INTERFACE . APPLICATION TO THE FAST, DIRECT AND RELIABLE ANALYSIS OF PETROLEUM FRACTIONS.

PAH ANALYSIS IN MINERAL OIL



PAH ANALYSIS IN MINERAL OIL

PAH STANDARD

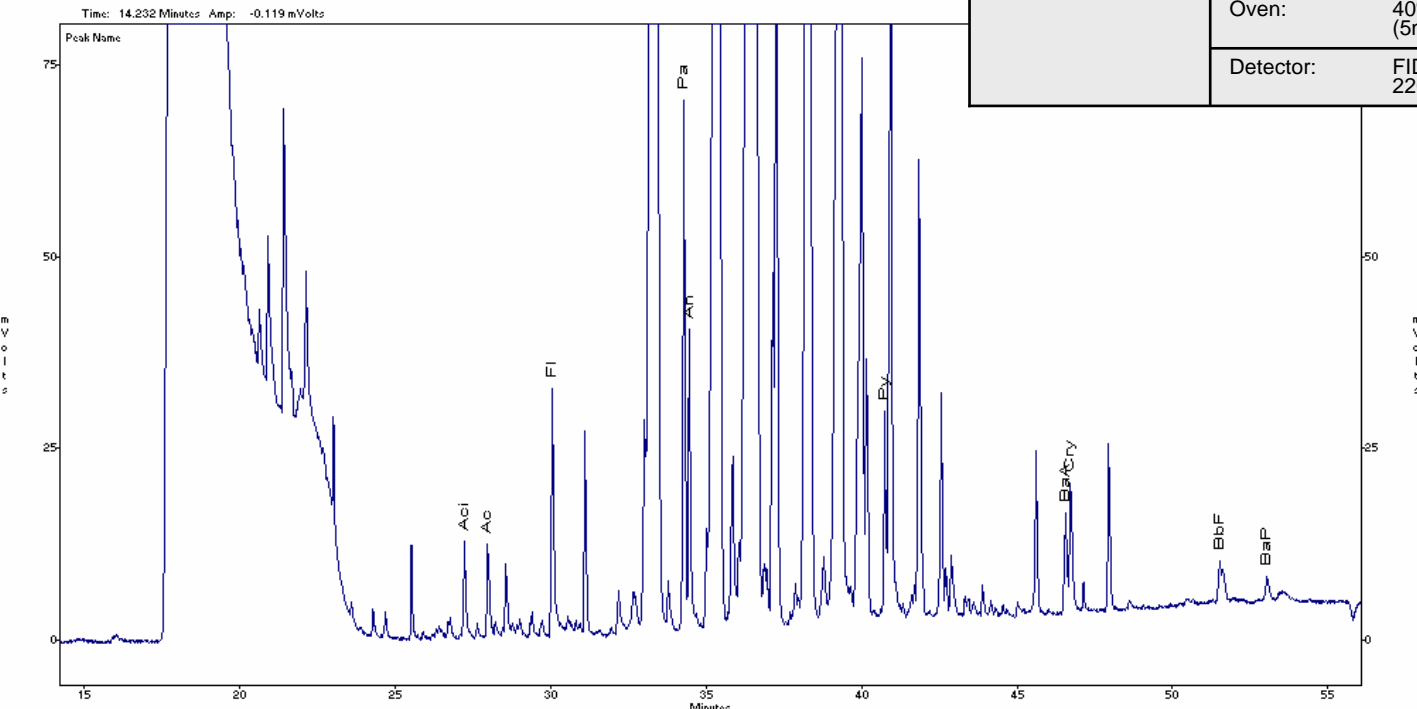


SAMPLE	10ppm PAH standard, 0.3µl injection
HRGC CONDITIONS	Column: KAP-5, 30m, 0.32mm
	Carrier: Helium at 13psi
	Injector: PTV, from 25°C to 250°C at 10°C/s
	Oven: 40°C (1min), 20°C/min, 100°C, 5°C/min, 250°C (5min)
	Detector: FID, 250°C, detector gases: H2 at 38ml/min, Air at 220ml/min, He at 25ml/min

PAH ANALYSIS IN MINERAL OIL

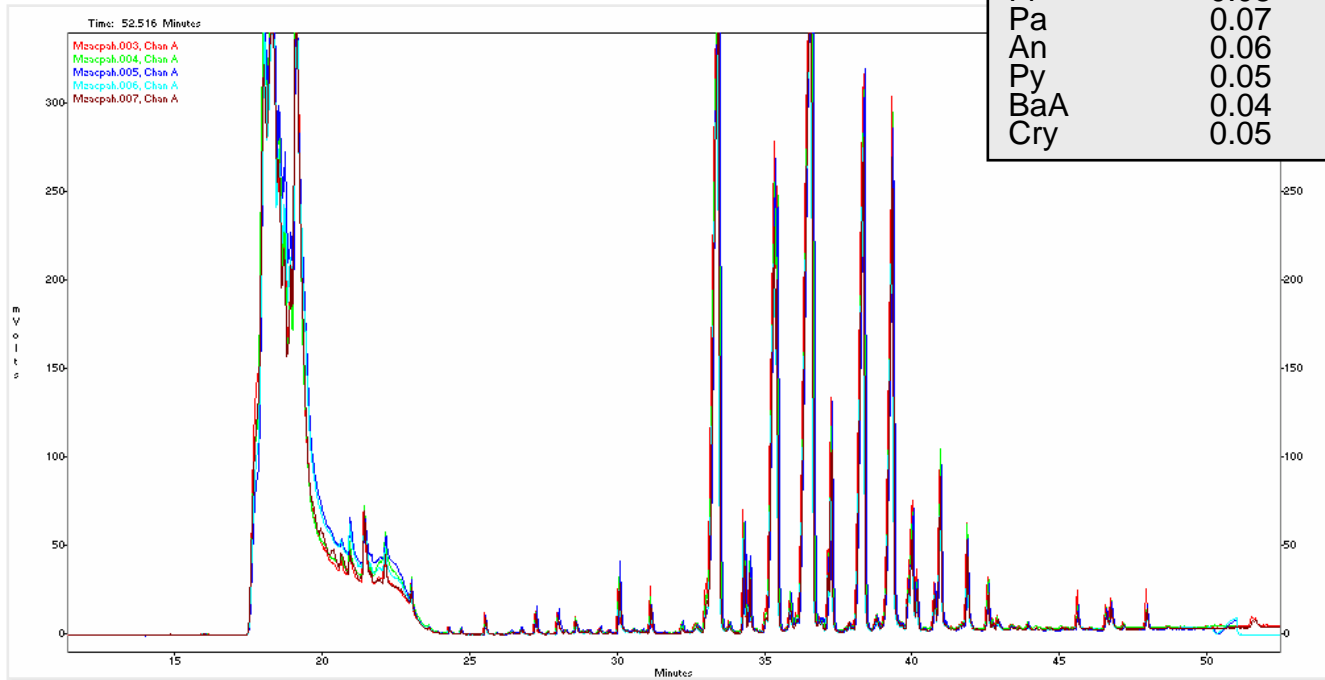
SAMPLE	Mineral oil 10ppm PAH spiked, 20µl injection
HPLC CONDITIONS	Column: C4 (50 x 4.6 mm ID)
	MPh: 70:30 ACN:H ₂ O; clean-up: 100% IPA
	Flow: 2ml/min
	Injected Volume: 20µl
INTERFACE CONDITIONS	Flow: 0.1ml/min
	Absorbent: 1cm TENAX TA
	Temp: 100°C (absorption); 100 up to 250°C at 10°C/s (desorption)
HRGC CONDITIONS	Column: KAP-5, 30m, 0.32mm
	Carrier: Helium at 13psi
	Injector: 250°C, Inj. : Conventional Mode
	Oven: 40°C (1min), 20°C/min, 100°C, 5°C/min, 250°C (5min)
	Detector: FID, 250°C, detector gases: H ₂ at 38ml/min, Air at 220ml/min, He at 25ml/min

FORTIFIED MINERAL OIL



PAH ANALYSIS IN MINERAL OIL

PRECISION (n=5)

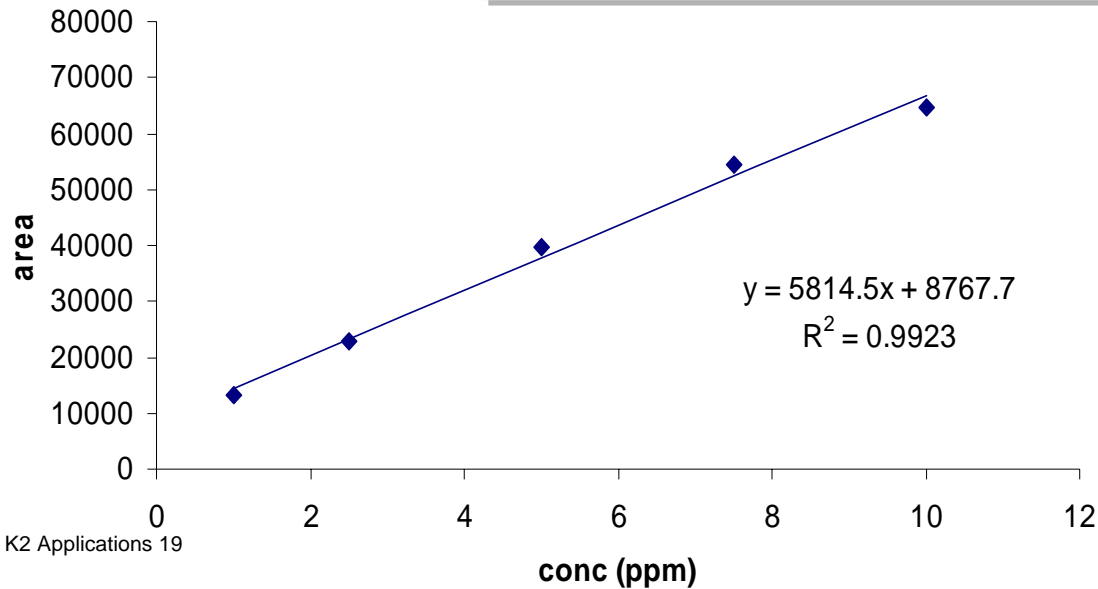
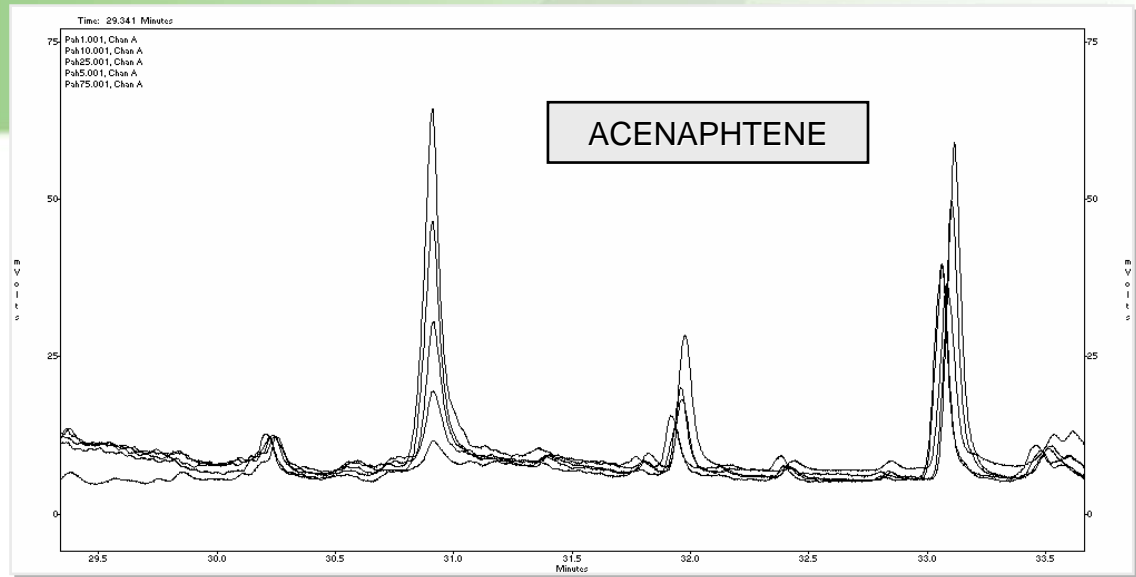


PAH	Rt RSD	Area RSD
Aci	0.07	10.2
Ace	0.08	16.0
Fl	0.08	16.0
Pa	0.07	13.4
An	0.06	11.3
Py	0.05	15.5
BaA	0.04	15.8
Cry	0.05	15.2

PAH ANALYSIS IN MINERAL OIL

LINEARITY

PAH	R ²
Aci	0.97
Ace	0.99
Fl	0.96
Pa	0.98
An	0.99
BaA	0.97
Cry	0.98



PAH ANALYSIS IN MINERAL OIL

DETECTION LIMITS ($\mu\text{g/ml}$) (S/N=5)

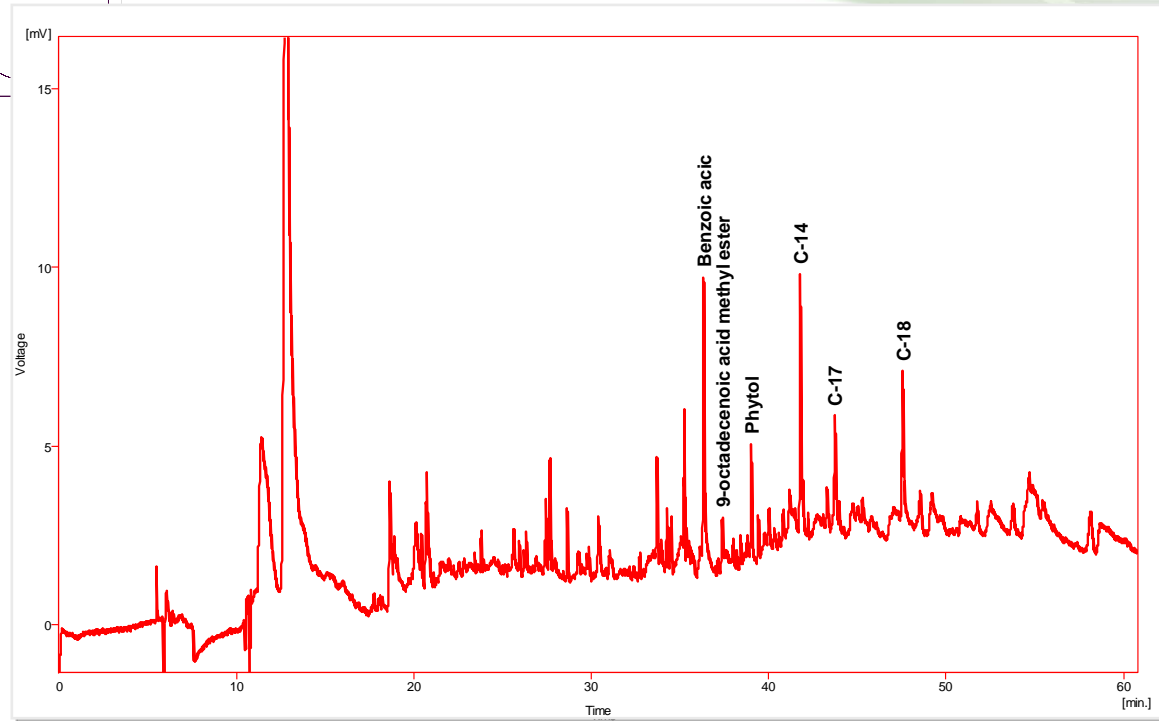
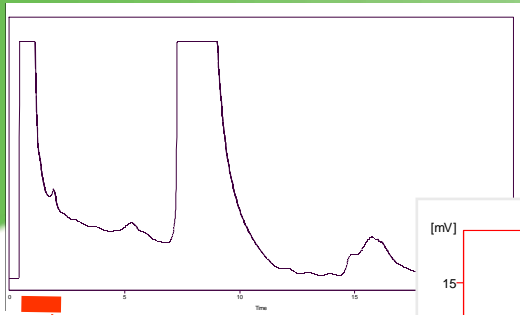
PAH	MDL ($\mu\text{g/ml}$)
Aci	0.27
Ace	0.29
Fl	0.20
Pa	0.05
An	0.12
BaA	0.22
Cry	0.19
BbF	0.47
BaP	0.70

HPLC+HRGC K2 APPLICATIONS

- **JAI 05 (Barcelona):** ANALYTICAL CHARACTERIZATION OF AN ACTIVE *Erythroxylum minutifolium* EXTRACT. ANALYSIS OF FREE FATTY ACIDS BY ON-LINE HPLC-HRGC USING THE K2 SYSTEM

FATTY ACIDS IN A PLANT EXTRACT

HPLC Chromatogram – UV-VIS detection

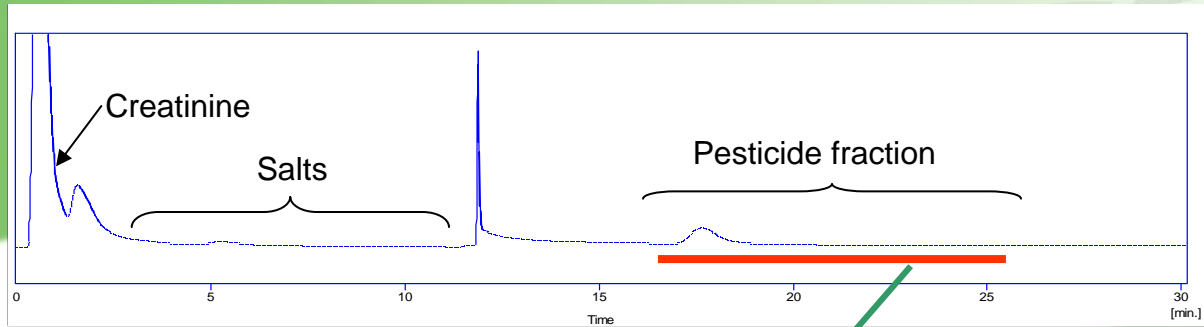


HRGC Chromatogram – FID detection

HPLC+HRGC K2 APPLICATIONS

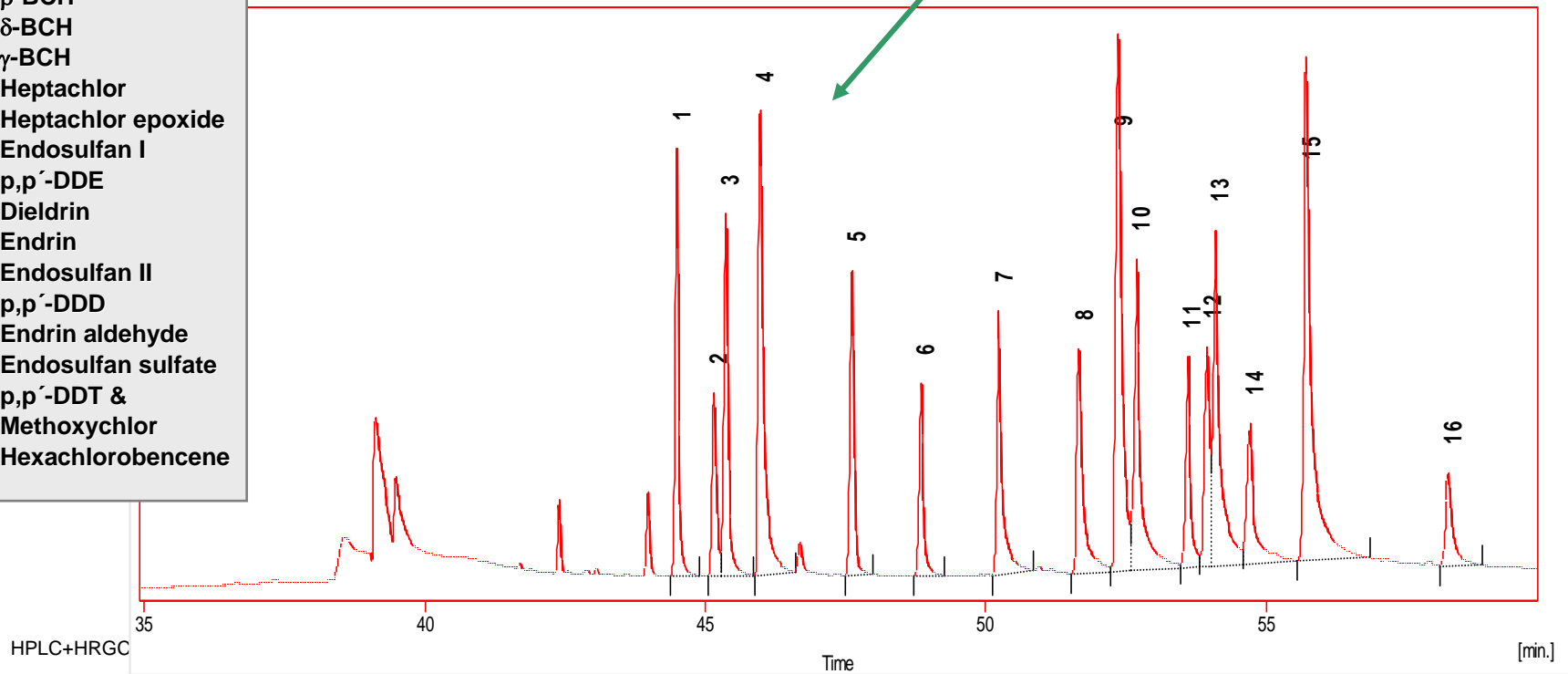
- **EXTECH 06 (York):** AUTOMATED ON-LINE RPLC-GC FOR THE DETERMINATION OF HAZARDOUS PESTICIDES IN URINE

PESTICIDES IN URINE SAMPLES



HPLC Chromatogram – UV-VIS detection

1. α -BCH
2. β -BCH
3. δ -BCH
4. γ -BCH
5. Heptachlor
6. Heptachlor epoxide
7. Endosulfan I
8. p,p'-DDE
9. Dieldrin
10. Endrin
11. Endosulfan II
12. p,p'-DDD
13. Endrin aldehyde
14. Endosulfan sulfate
15. p,p'-DDT & Methoxychlor
16. Hexachlorobencene



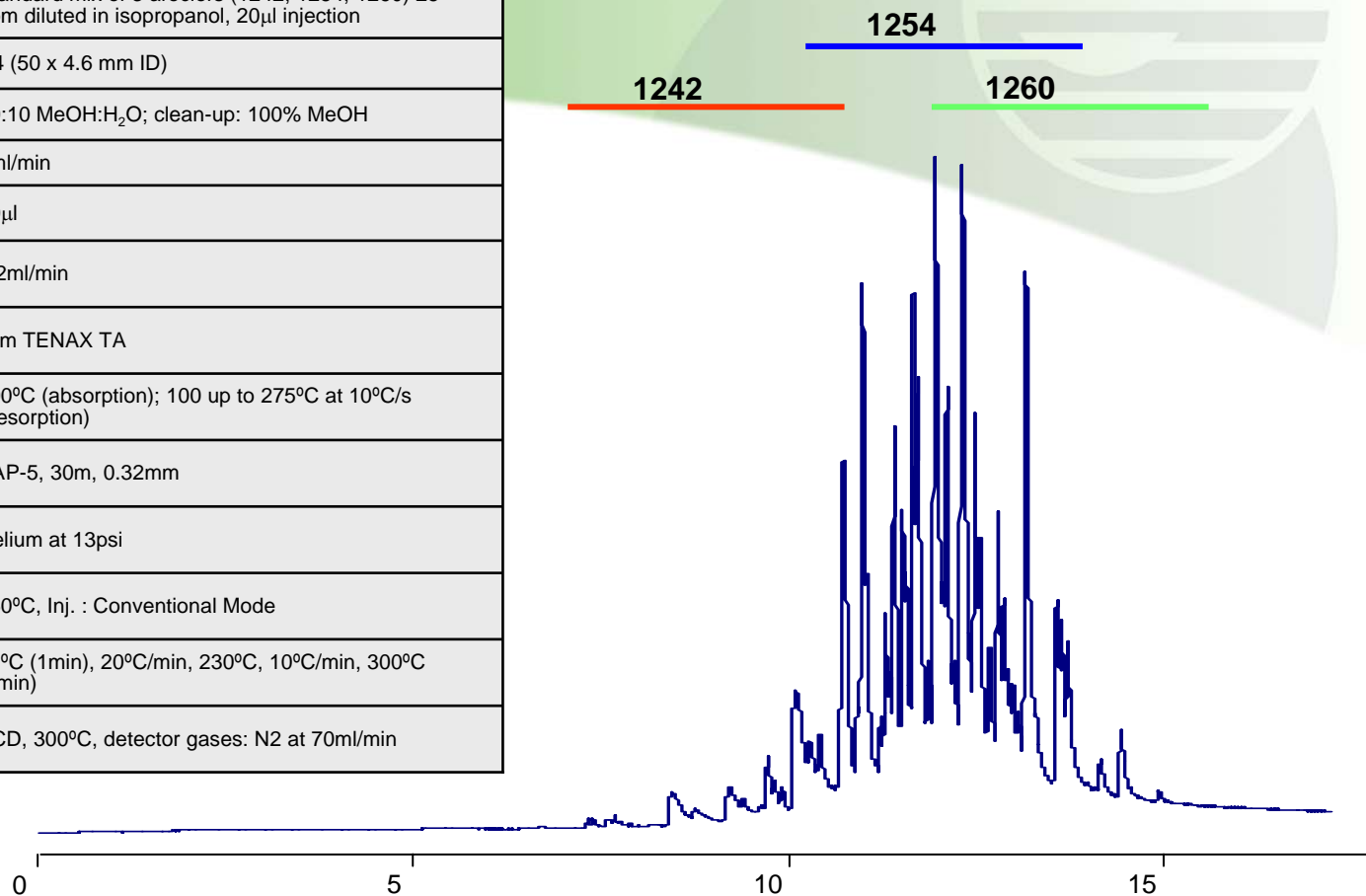
HRGC Chromatogram – ECD detection

HPLC+HRGC K2 APPLICATIONS

- **PITTCON 06 (Orlando):** DETERMINATION OF POLICHLORINATED BYPHENYLS (PCBs) IN TRANSFORMED OIL BY ON-LINE REVERSED PHASE LC-GC USING THE TOTAD[®] (THROUGH OVEN TRANSFER ADSORPTION DESORPTION) INTERFACE

PCBs IN TRANSFORMER OIL

SAMPLE	Standard mix of 3 aroclors (1242, 1254, 1260) 25 ppm diluted in isopropanol, 20µl injection
HPLC CONDITIONS	Column: C4 (50 x 4.6 mm ID)
	MPh: 90:10 MeOH:H ₂ O; clean-up: 100% MeOH
	Flow: 1ml/min
	Vol: 20µl
INTERFACE CONDITIONS	Flow: 0.2ml/min
	Absorbent: 1cm TENAX TA
	Temp: 100°C (absorption); 100 up to 275°C at 10°C/s (desorption)
HRGC CONDITIONS	Column: KAP-5, 30m, 0.32mm
	Carrier: Helium at 13psi
	Injector: 250°C, Inj. : Conventional Mode
	Oven: 40°C (1min), 20°C/min, 230°C, 10°C/min, 300°C (5min)
	Detector: ECD, 300°C, detector gases: N ₂ at 70ml/min

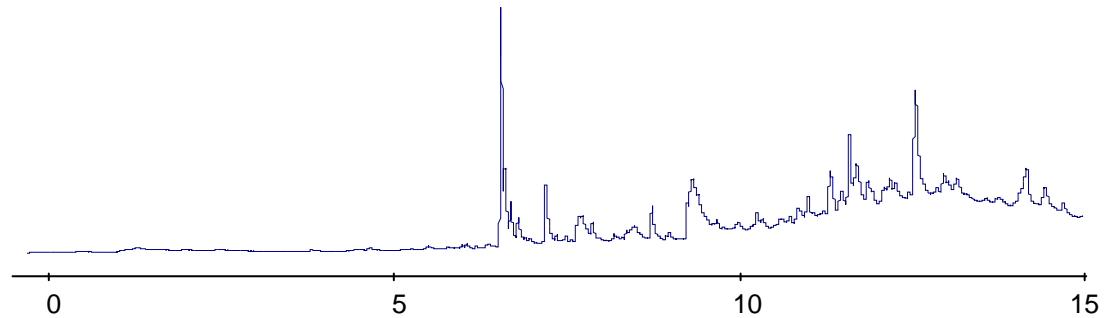


PCBs IN TRANSFORMER OIL

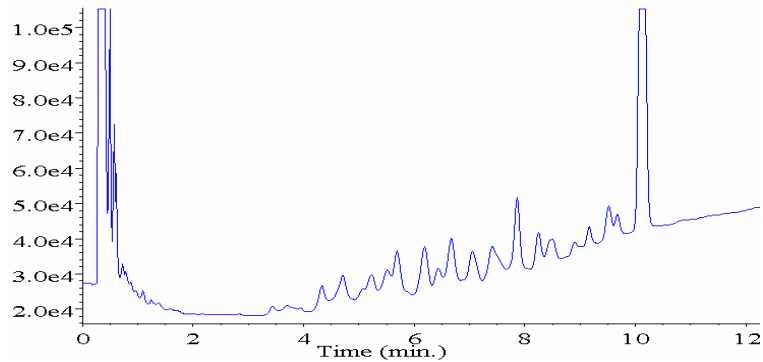
PRECISION (n=5)

PCBs	RSD (area)
Aroclor 1242	9,2
Aroclor 1254	5,8
Aroclor 1260	6,1

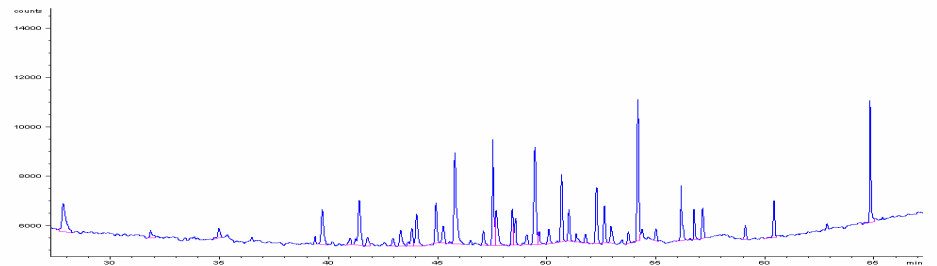
(a) K2 system



(b) ASTM D4059-00



(c) European Norm EN-61619



HPLC+HRGC K2

- Sample Preparation simplification
- Totally automated
- Less analysis time
- Less solvent consumption
- Allows quick and easy current analytical methods adjustment and new methods development
- Guarantees sample integrity while increasing recovery and facilitates quantitation
- Universal or selective detection. Detection limits improvement.
- Qualitative confirmation for routine analysis thanks to the dual retention time

A big amount of analytes can be analyzed in complex samples with the combination of:

- Any HPLC and HRGC column
- Any HPLC solvent
- Any absorbent trap and media

New methods development:

- Food: Pesticides, Fatty Acids, Sterols, Alcohols, Aromas, Vitamins, ...
- Water and environmental: pesticides, PCBs, Dioxins, BTX, detergents, ...
- Petroleum: Aliphatics, Aromatics, Naphtenes, Poliolephinics, PONA, PIANA,
- Metabolites in urine and plasma: pharmacology, clinical chemistry, early diagnosis,
- Natural products and flavors: essential oils, bioactives, ...
- Toxicology: industrial hygiene, abuse drugs,
- Pharma: analgesics, antibiotics, ...