

# DSFP GC-MS AMDIS AND FIELD PORTABLE SAMPLERS

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# WHY DSFP GC-MS?

- Complementing DSFP-LC-MS,
- Faster and precise identification of selected (target) compounds
- Easy to set up required library (Norman SusDat – GC amenable compounds)
- Retention index (RI):
  - Improving confidence in identification of a detected compound
  - A location tool - retention window
- Part of AMDIS

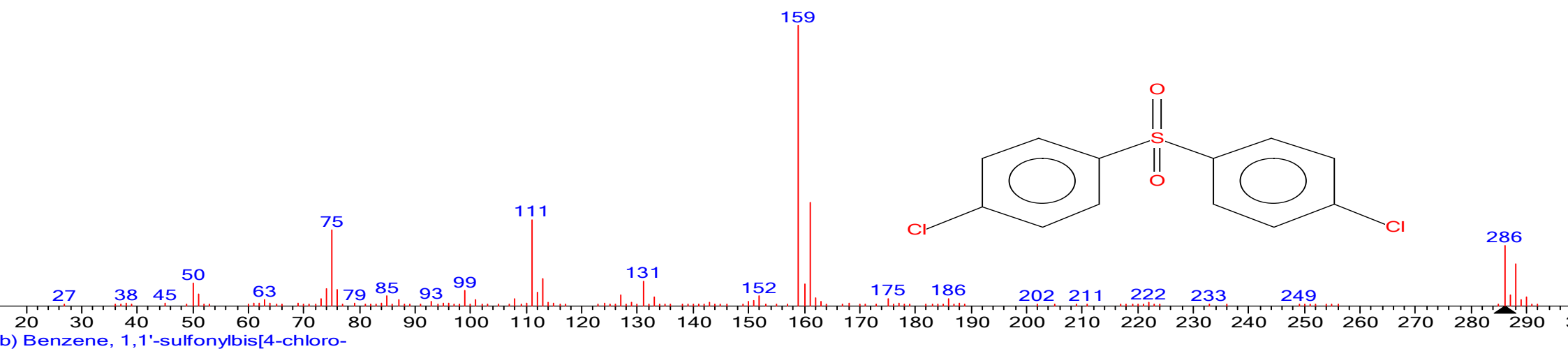
# EXAMPLE - BIS(4-CHLOROPHENYL) SULFONE

Target: Black Sea water samples – XL-SPE- EMBLAS 2017

Procedure:

EI-SCAN of alkanes mixture for RI calculation

EI-SCAN of a real sample



# EXAMPLE - BIS(4-CHLOROPHENYL) SULFONE

Create RI Calibration Data File (CAL):

- RT - actual RT of the deconvoluted peak of injected alkanes,
- RI - RI of injected alkanes,
- Name - name of the injected alkane with its MW

Create Target Library (MSL) - **bis(4-chlorophenyl) sulfone**:

- Name, formula, CAS #,
- RI – from NIST or from a real measurement,
- 10 most intensive fragments from NIST

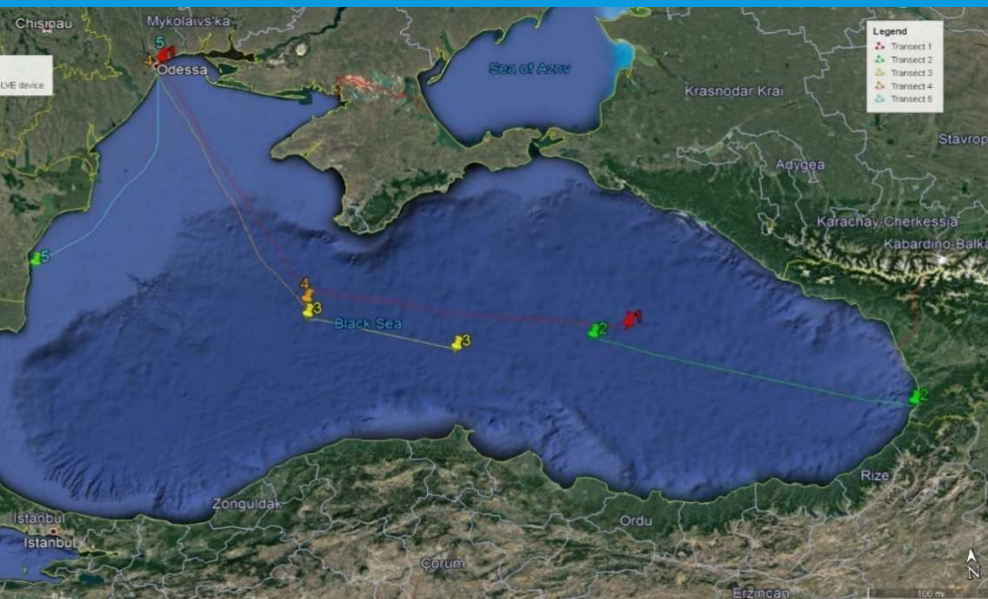
```
NAME: Benzene, 1,1'-sulfonylbis[4-chloro-  
FORM: C12H8Cl2O2S  
CASNO: 80-07-9  
RI: 2231  
RW:  
RT:  
SOURCE: NIST 17  
NUM PEAKS: 10  
(159 999) (161 367) (111 307) (75 269) (286  
(288 149) (113 95) (131 88) (50 80) (160
```

# EXAMPLE - BIS(4-CHLOROPHENYL) SULFONE

Search

The same procedure can be performed for more suspect compounds

Used for RI and 10 most abundant ions from NIST to create user library – a part of NORMAN SusDat

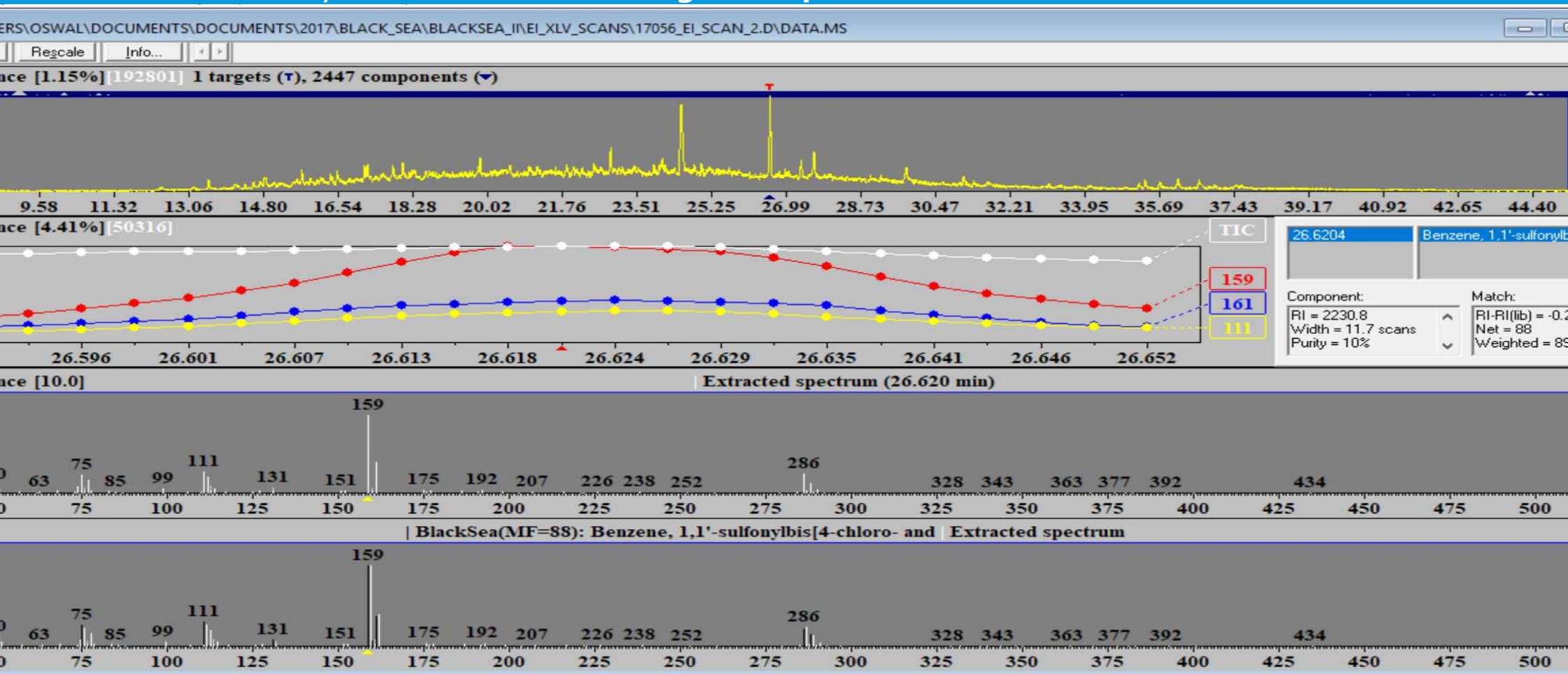


BlackSea\_compounds - Poznámkový blok

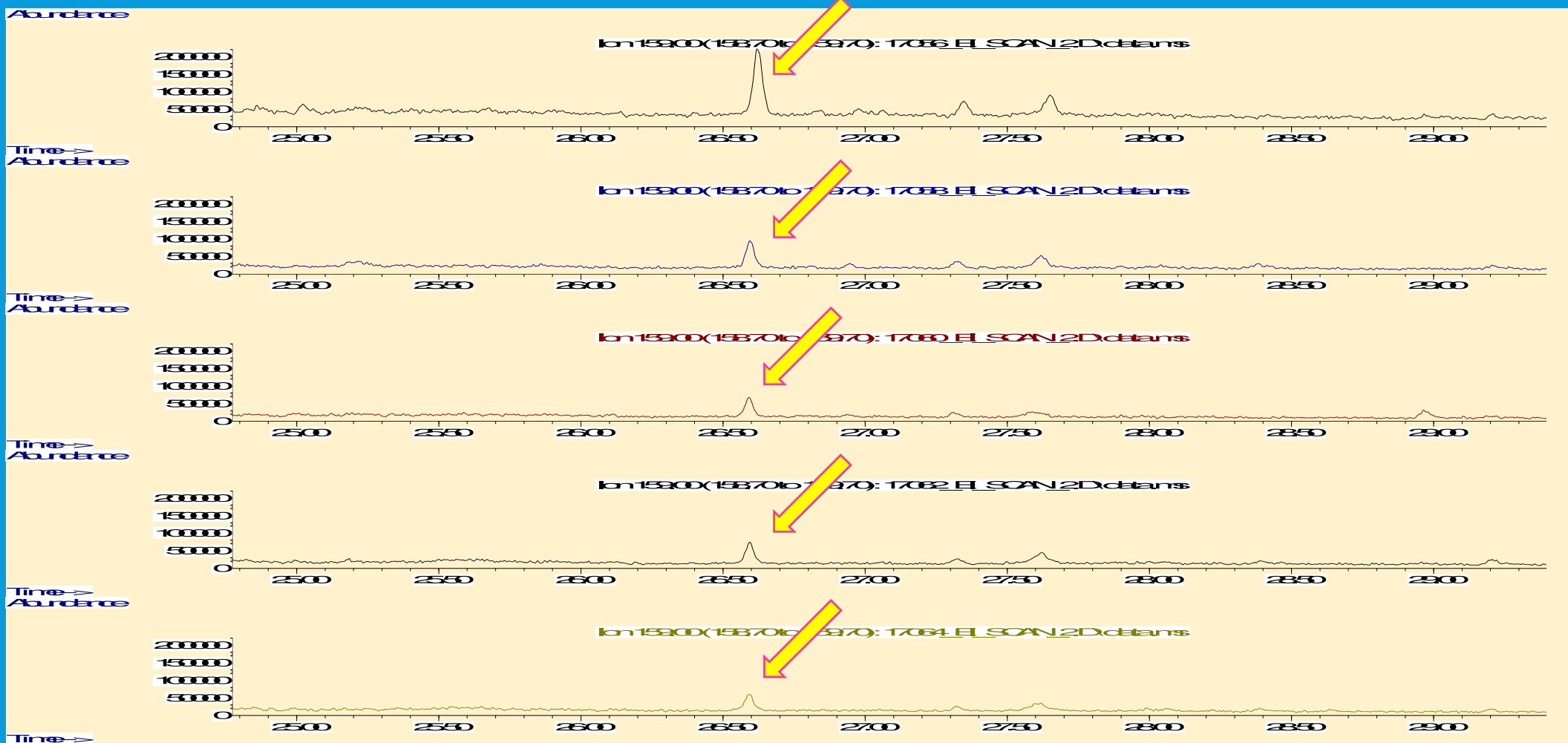
Súbor	Úpravy	Formát	Zobrazit	Pomocník						
(136	86)	(91	81)	(58	64)	(45	62)	(54	54)	
NAME: Benzene, 1,1'-sulfonylbis[4-chloro-										
FORM: C12H8Cl2O2S										
CASNO: 80-07-9										
RI: 2231										
RW:										
RT:										
SOURCE: NIST 17										
NUM PEAKS: 10										
(159	999)	(161	367)	(111	307)	(75	269)	(286	213)	
(288	149)	(113	95)	(131	88)	(50	80)	(160	76)	
NAME: Atrazine										
FORM: C8H14ClN5										
CASNO: 1912-24-9										
RI: 1763										
RW:										
RT:										
SOURCE: NIST 17										
NUM PEAKS: 10										
(200	999)	(215	615)	(58	378)	(173	353)	(202	336)	
(43	218)	(217	213)	(68	194)	(44	175)	(71	162)	

# EXAMPLE - BIS(4-CHLOROPHENYL) SULFONE

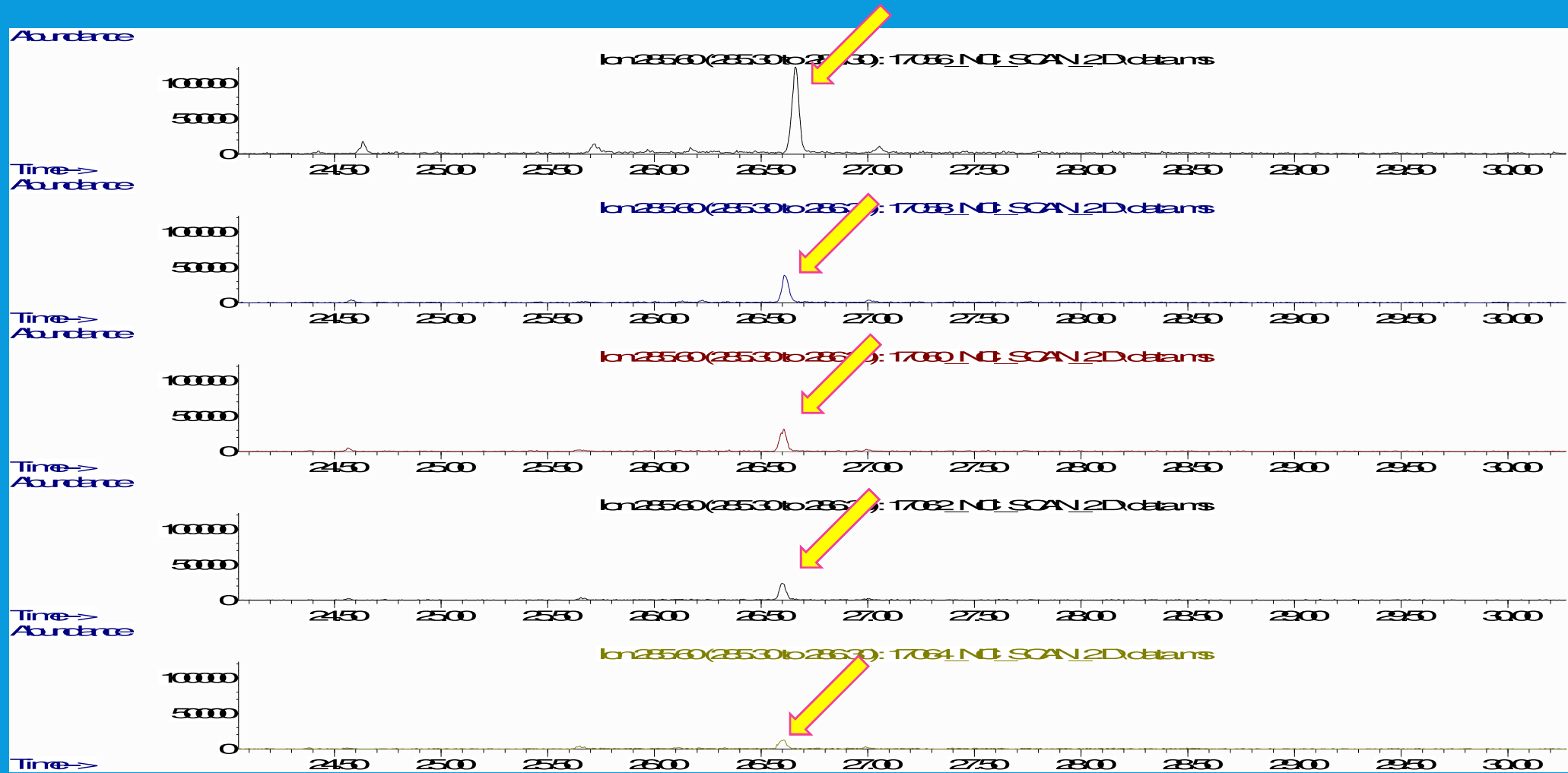
Annotation: AMDIS – Analyse GC/MS chromatogram – positive hit



# EXAMPLE - BIS(4-CHLOROPHENYL) SULFONE



# EXAMPLE - BIS(4-CHLOROPHENYL) SULFONE





# Mariani box - portable field sampler



## *Characteristics:*

- Flexibility, versatility, easy to use.
- Samples directly on-site.
- Easily portable on site: only 6 Kg.
- Target volume: from 1 to 20 L.
- 10 to 20 fold increase of LoQ.
- Reduction of storage facilities volume.
- Increased sample stability on SPE support compared to liquid sample.
- Inexpensive.

- Delivery conditioned filters ready for sample extraction
- Pick-up
- Sample elution in JRC lab. facilities
- LC-MS/MS and GC/MS analysis



# Prototype of portable field sampler - Vacuum Accumulator System



**Sample Volume:** 100ml to 50L

**Volume accuracy:** +/- 3% or better (uses discrete measurement by counting filling cycles of known fixed volume accumulator)

**Sample Flow Rate Control:** Direct-feedback control from liquid flow meter

**Sample Flow Rate Range:** 25 to 300 ml/min

**Flow Rate Accuracy:** +/- 10%

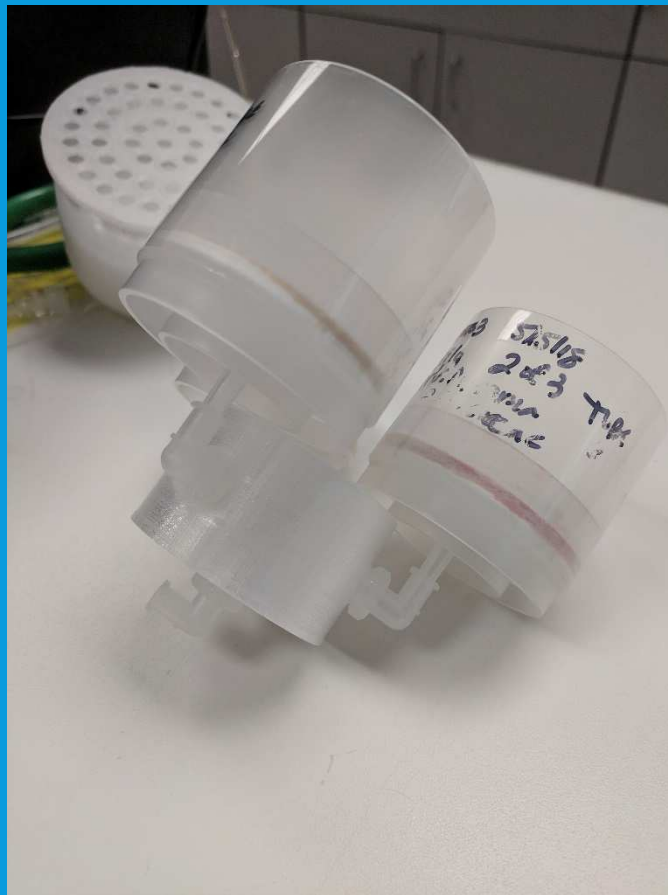
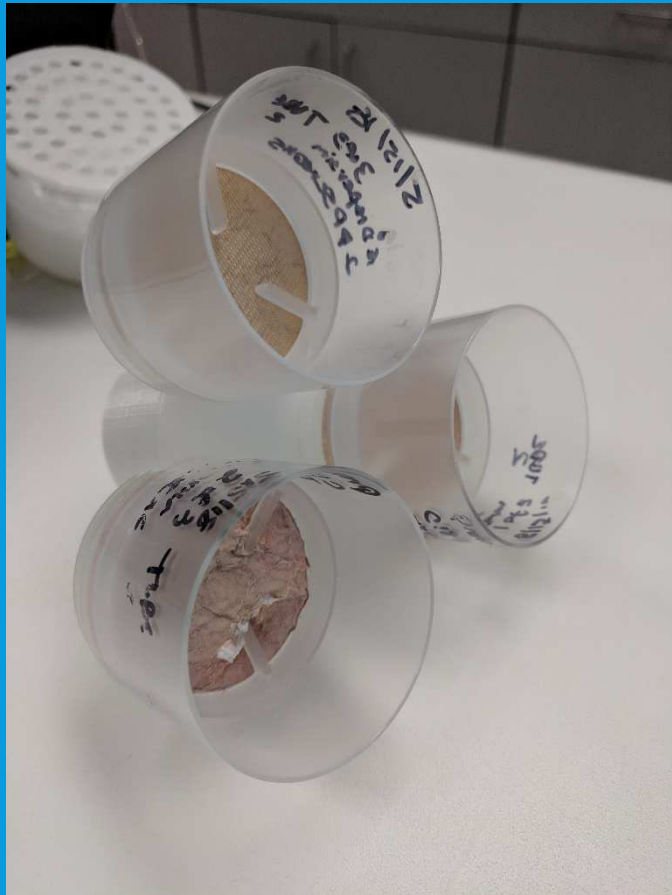
**Suction Lift Capability:** up to 5 m vertical

**Suction Tube Length:** 15 m

**Exhaust Tube Length:** 15 m

**Weight:** < 10 kg (without battery)

# Multiple SPE Disk Sampling



# Activation and extraction protocol

## Membrane filter activation:

- ethyl acetate, methanol and Milli-Q

## Storage:

- at -20°C

## Elution:

- two types of extracts - LC (methanol), GC (ethylacetate)
- LC – reconstitution with LC mobile phase compatible solvents
- GC – reconstitution with GC solvents