DIP Direct Inlet Probe for GC-MS

DIP-5975 MSD

DIP-7000 QQQ
mit Dual-PAL

MSD Direct Inlet Probe

Push rod:

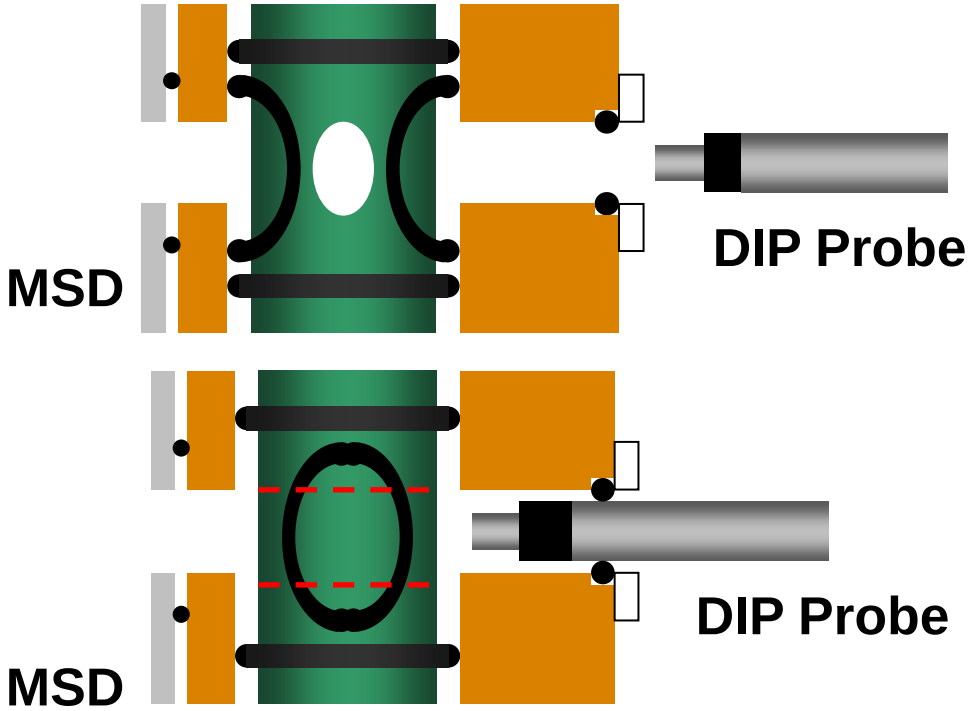
- integrated heating element and cooling device
- sample vessel is located at the tip of the push rod

Control panel:

- a **microprocessor** enables automatic sample transfer into the ion source and
- **heating** and **cooling** of the probe tip at ramp rates

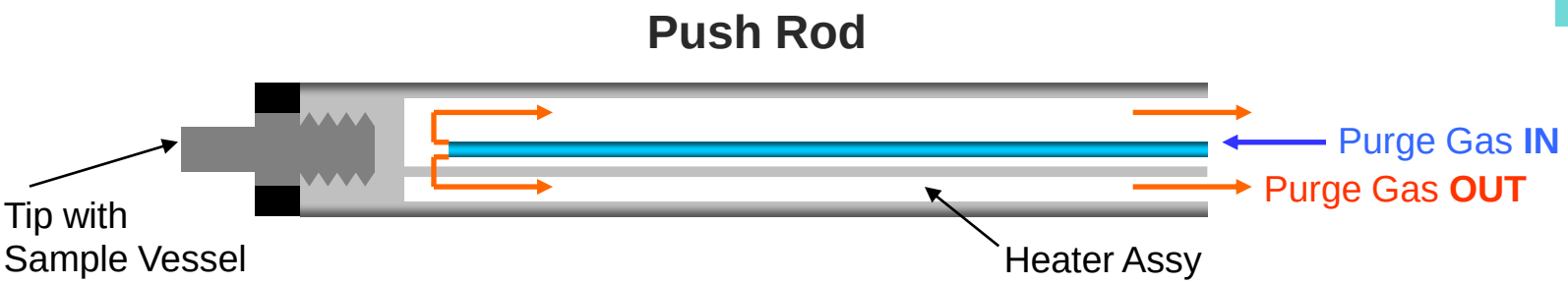
Sealing Mechanism

➔ sample is transferred into the vacuum of the ionization chamber via a **vacuum-lock assembly:**

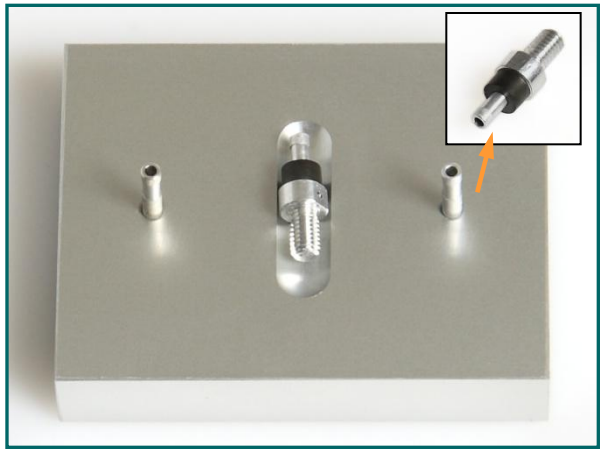


➔ vacuum lock assy with <u>marginal dead volume</u> **:**
no additional pump for the vacuum lock required
(as it is common practice for competitive products)

Details of the Probe



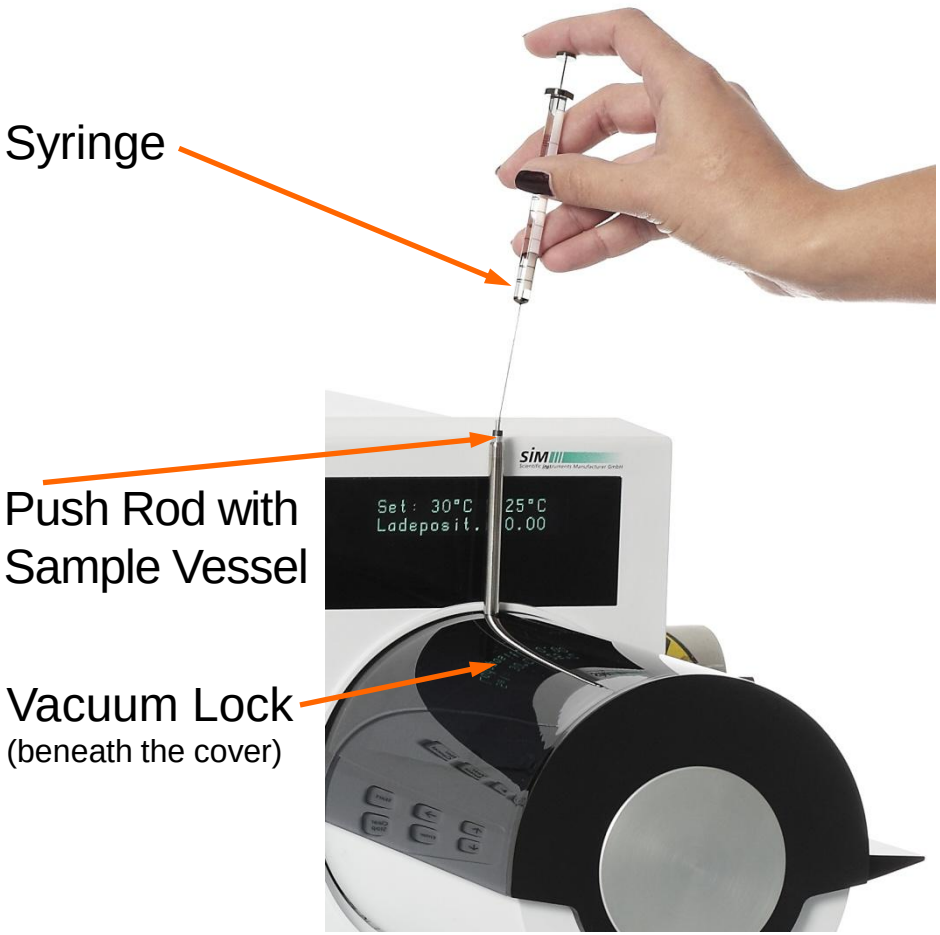
Liquid Sample Tip
(filled with a syringe)



Solid Sample Tip
with aluminium
sample tubes

Push Rod with
body (above),
purge gas line (middle)
heater assembly (below)

Sample Insertion (I)

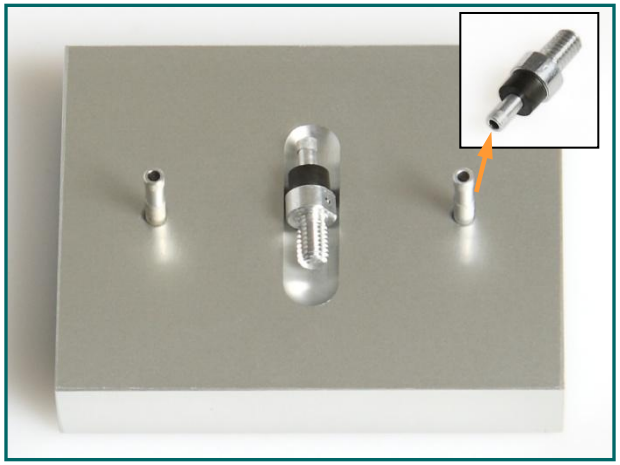


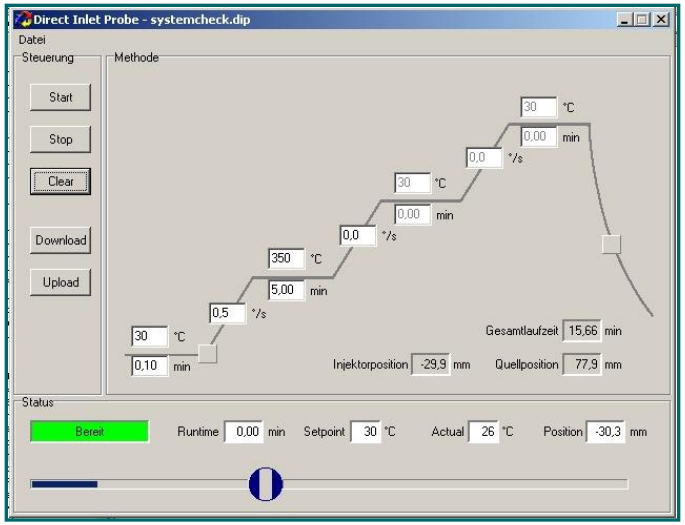
1. Sample (liquid/dissolved) is placed into the sample vessel of the probe tip with a syringe.

2. Push rod is automatically introduced into the high vacuum of the MS up to the ion source funnel (step by step through the vacuum lock).

3. When the probe tip is placed directly in front of the ion source funnel, the tip is heated corresponding to the temperature program.

Sample Insertion (II)





solid samples:

- change tip of the push rod for liquid samples against the solid sample tip
- fill sample tube and put it into the solid sample tip with special tweezers

temperature program:

- user-friendly DIP software enables feeding the temperature values directly into the temperature profile
- the status bar shows the course of the run (last line: plot of the push rod together with valve position)

Multifunctionality of the GC/MS-DIP-System

- GC/MS DIP System equipped with **TwinPAL**:

  1 arm with microliter syringe for **liquid sampling (GC or DIP)** and

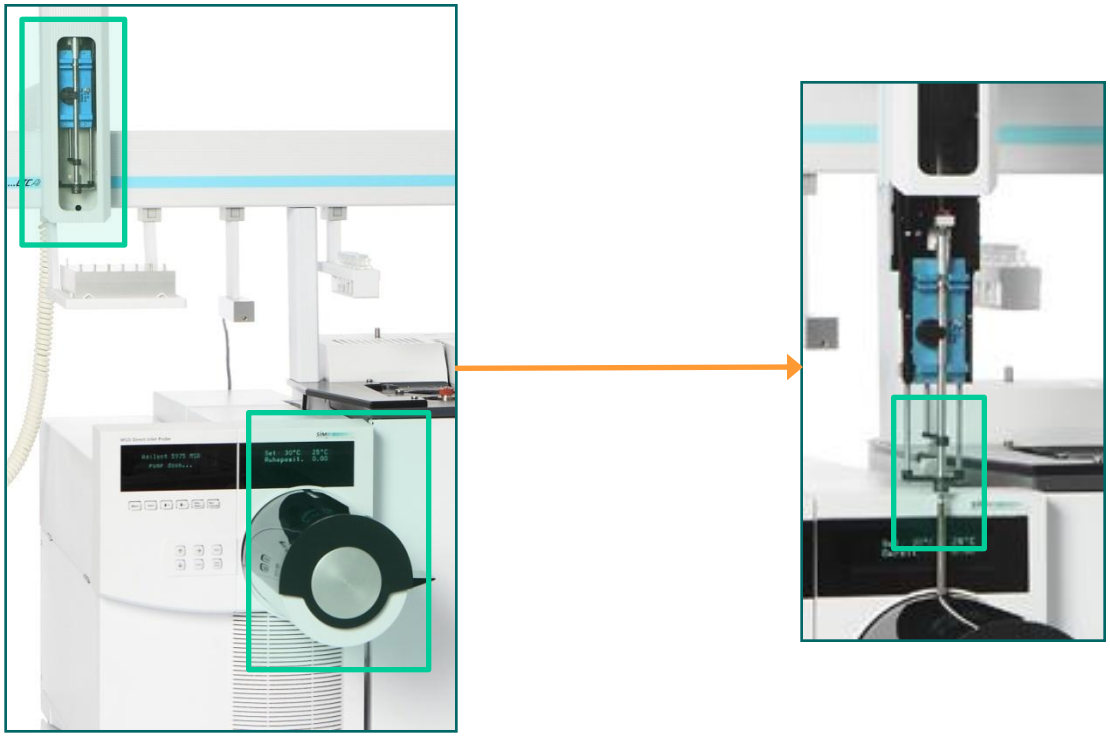
  1 arm with tube-gripper for **solid sampling (DIP)**

➔ GC/MS and DIP/MS runs in series

➔ particularly suited for **screening methods**

➔ effective and time-saving, no hardware changes are necessary

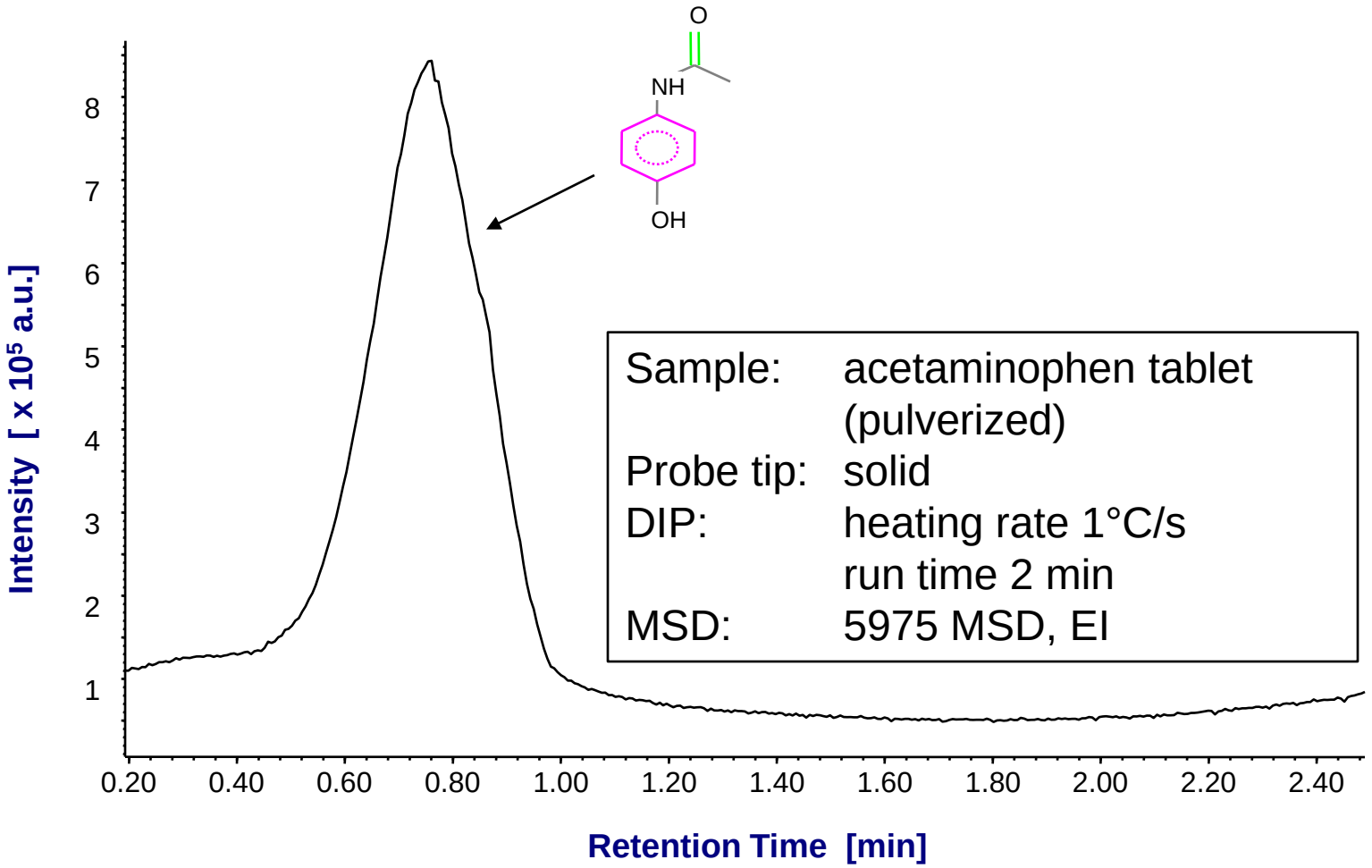
Automated Analysis with PAL Autosampler



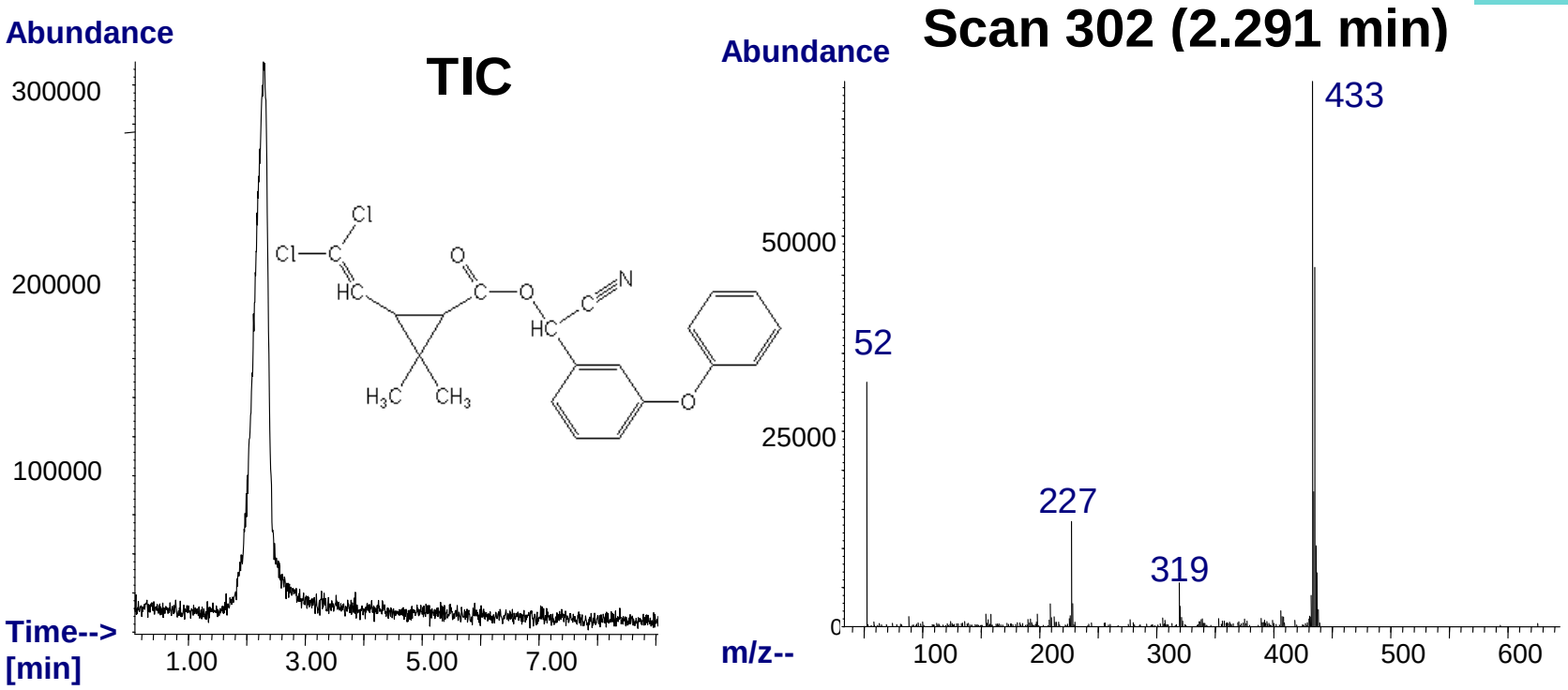
- **liquid samples**: PAL with µl-syringe
  Option: thermostatted flow cell➔ online analysis

- **solid samples:** PAL with **tube gripper** to put the sample tube into the tip of the push rod

Solid Sample (EI): Tablet with Acetaminophen (Paracetamol)



Solid Sample (CI) : Cypermethrin



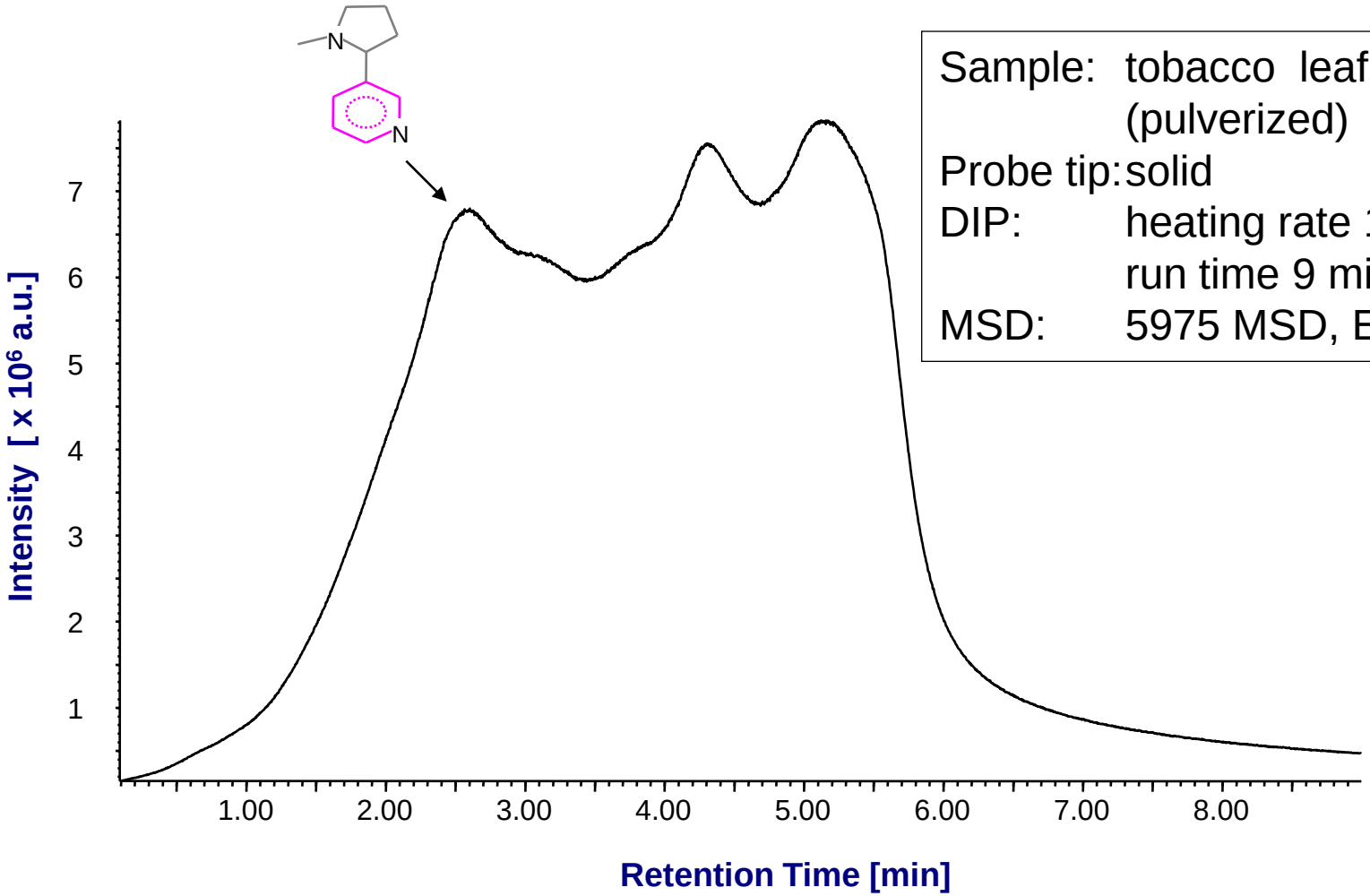
Sample: pyrethroid cypermethrin (solid), M = 416.3 g/mol

Probe tip: solid

DIP: heating rate 2°C/s, run time 5 min

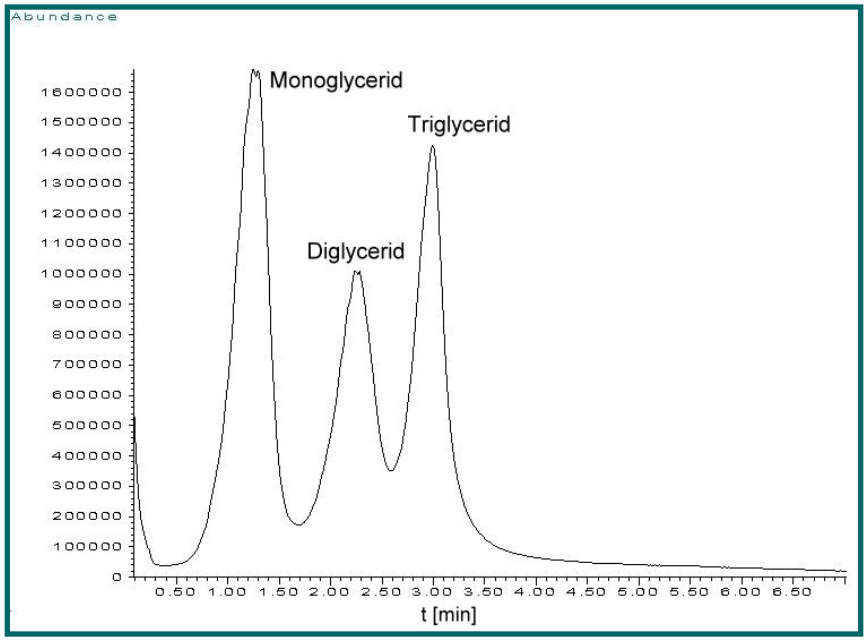
MSD: 5975 MSD, PCI mode with NH_3

Solid Sample (EI): Tobacco Leaf



Sample:	tobacco leaf (pulverized)
Probe tip:	solid
DIP:	heating rate 1°C/s
	run time 9 min
MSD:	5975 MSD, EI

TIC of a Glyceride Mixture



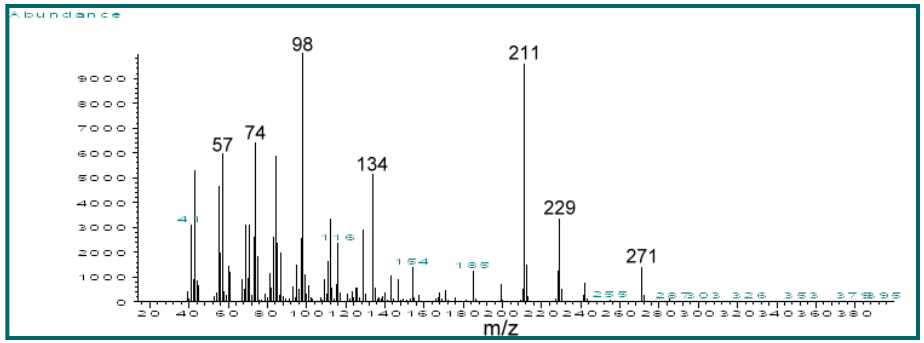
- excellent temperature control and heat transmission at the probe tip

➔ thermal separation of the glyceride mixture:
monoglycerid (130 °C)
diglycerid (190 °C)
triglycerid (230 °C)

Sample:	glycerides of myristic acid (C14:0)
Probe tip:	liquid
DIP:	heating rate: 1 °C/s (50 - 350 °C)
	run time: 6 min
MSD :	5973 MSD, EI

Interpretation of MS Data (example diglyceride)

a (DIP)

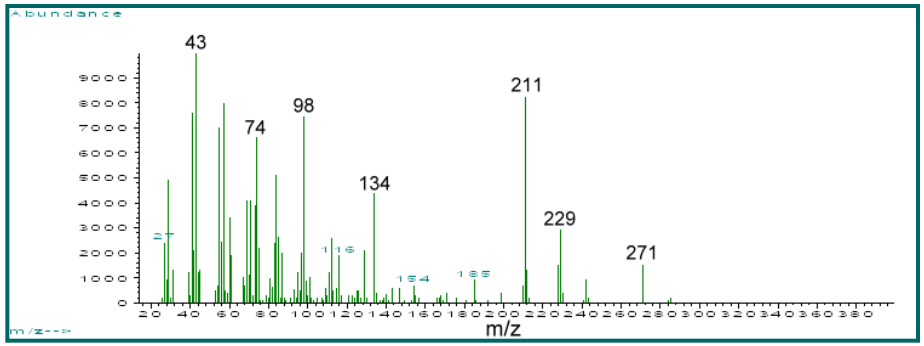


- mass spectrum of **myristic acid (C14:0) diglyceride**

  a) scanned via MSD- DIP System (upper fig.)

  b) result of the NIST library search (lower fig.)

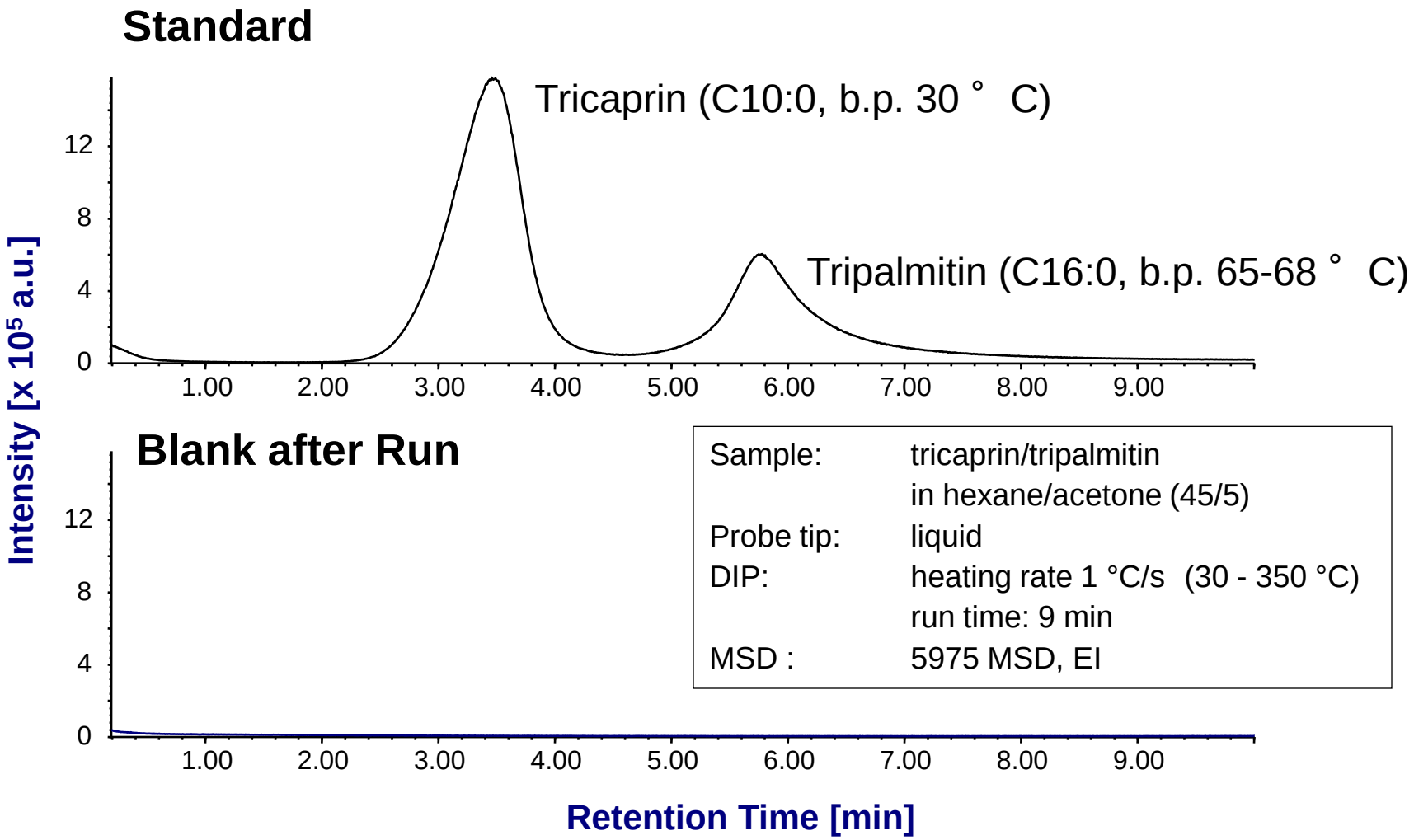
b (NIST)



➔ all tools of the MS software may be applied

➔ identification of analytes by common library search

EI-Standard: Triglycerides (1.5 µg each)



Sample:	tricaprin/tripalmitin in hexane/acetone (45/5)
Probe tip:	liquid
DIP:	heating rate 1 °C/s (30 - 350 °C) run time: 9 min
MSD :	5975 MSD, EI

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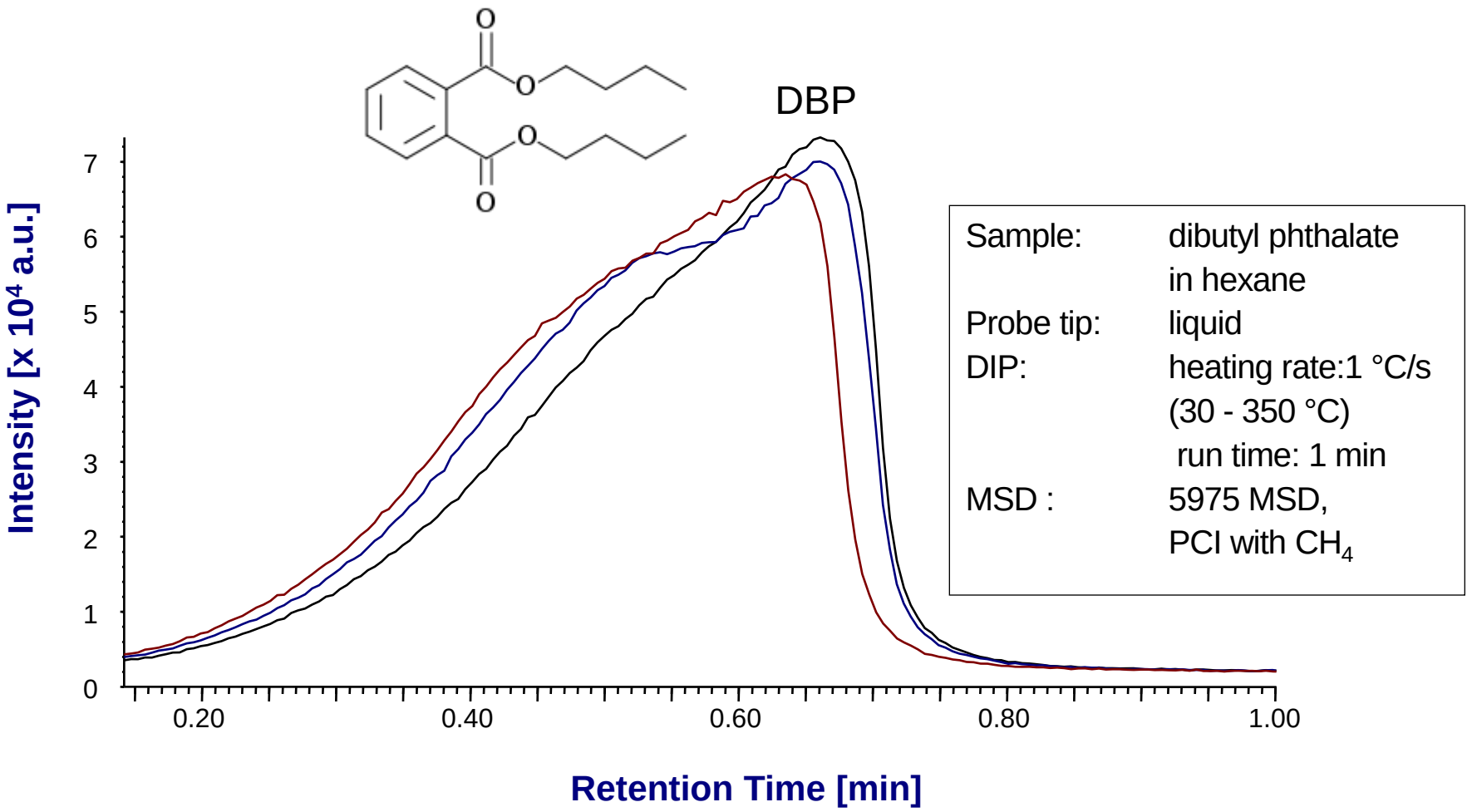
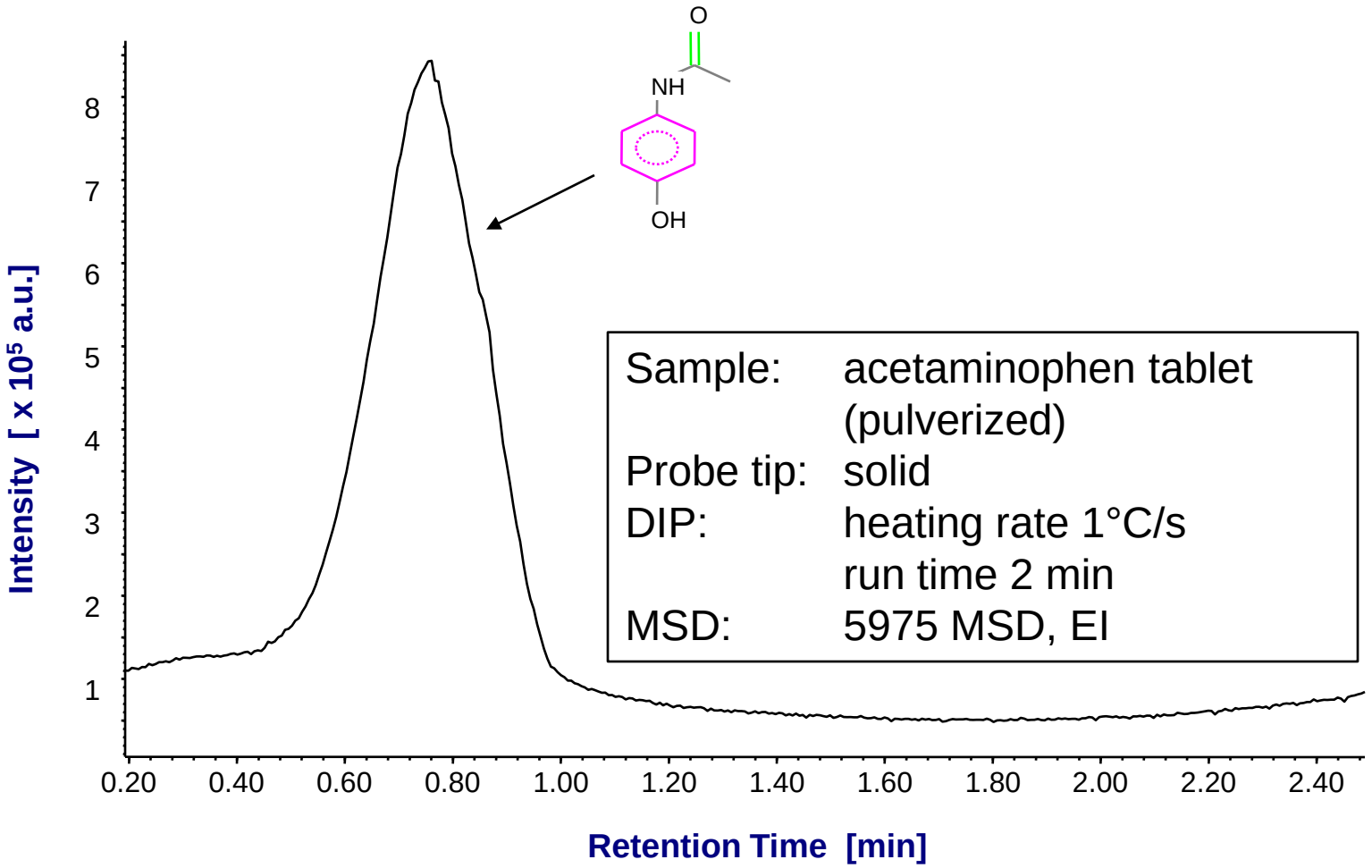
CI-Standard: Dibutyl Phthalate (DBP)

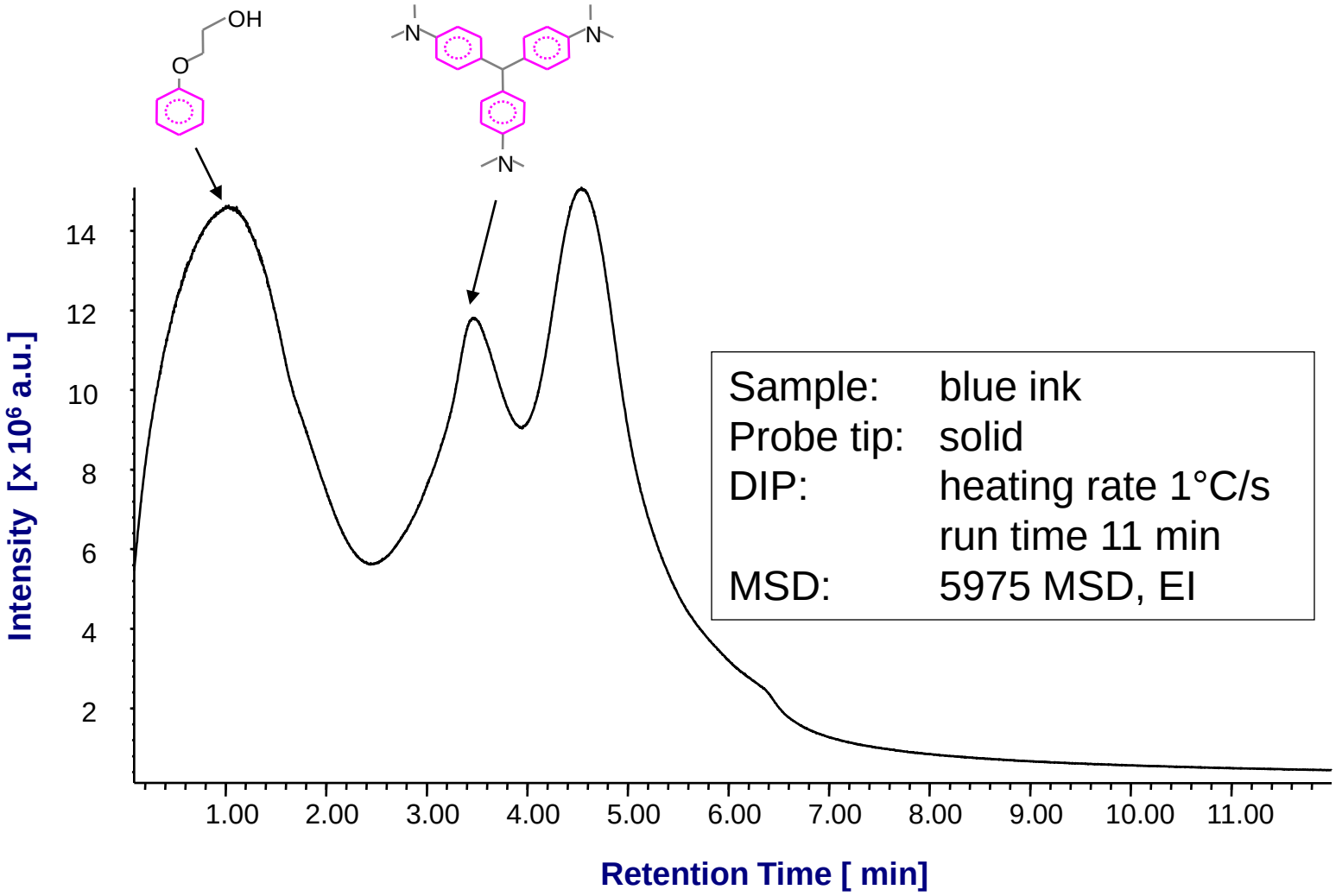


Sample:	dibutyl phthalate in hexane
Probe tip:	liquid
DIP:	heating rate:1 °C/s (30 - 350 °C) run time: 1 min
MSD :	5975 MSD, PCI with CH_4

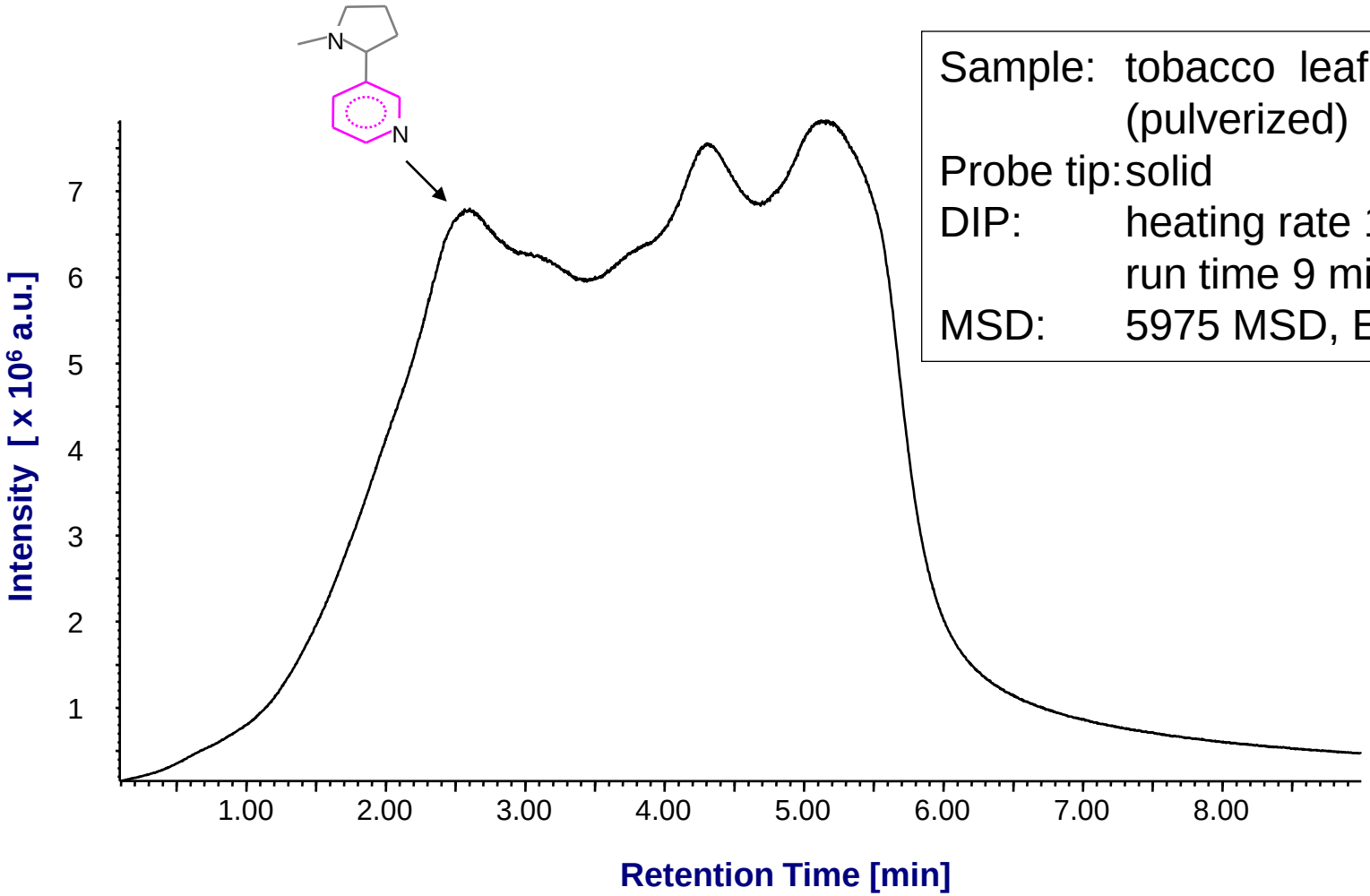
Solid Sample (EI): Tablet with Acetaminophen (Paracetamol)



Solid Sample (EI): Blue Ink (Ball-point pen)

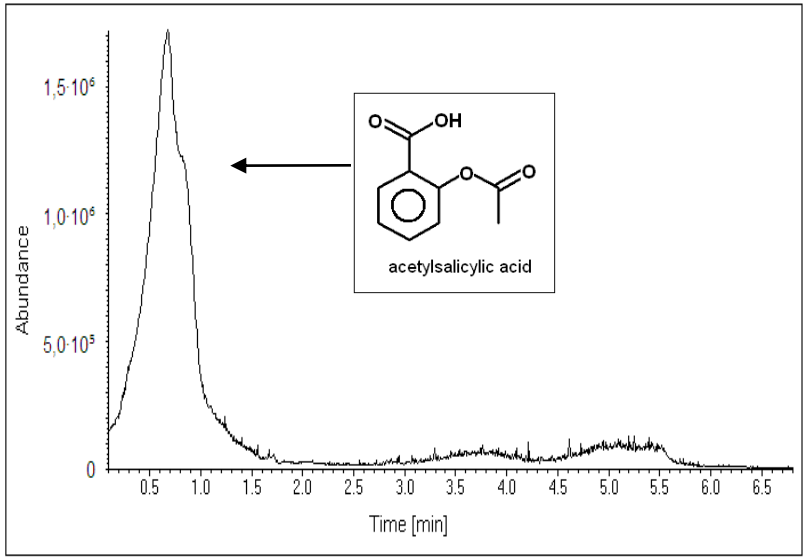


Solid Sample (EI): Tobacco Leaf



Sample:	tobacco leaf (pulverized)
Probe tip:	solid
DIP:	heating rate 1°C/s
	run time 9 min
MSD:	5975 MSD, EI

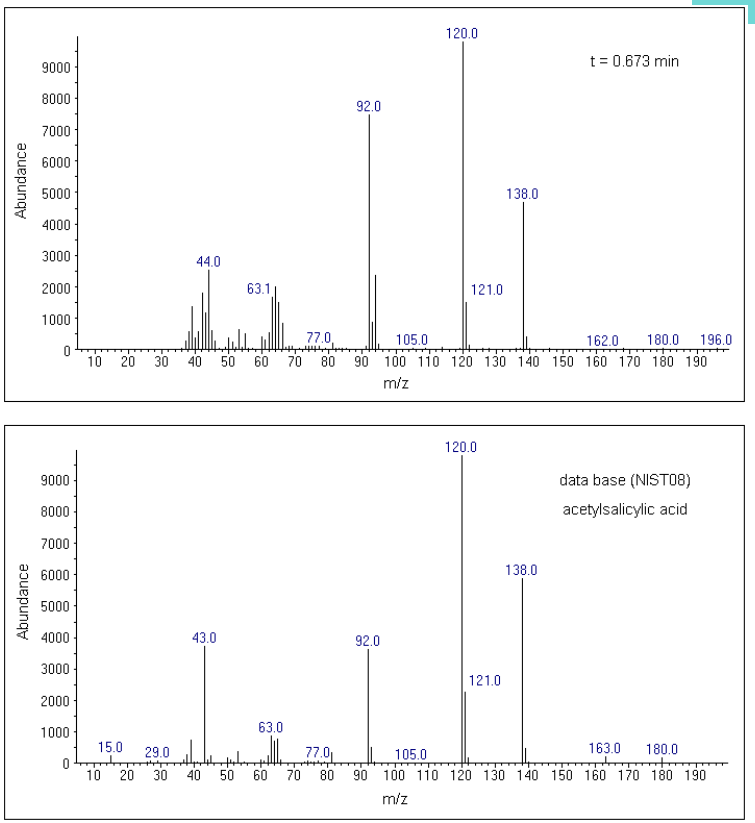
DIP-Application: Pharmaceuticals
Aspirin–Tablet (EI, Solid Sample)



Vaporization Curve of Aspirin-Tablet

MS	Agilent 5975 MSD, EI mode
MS Temp	280 °C (Source), 150 °C (Quad), 280 °C (Transfer Line)
Full Scan	33-600 m/z
DIP	solid tip
DIP Temp	40°C, 0.5°C/s to 350 °C

➔ acetylsalicylic acid can't be vaporized without decomposition, GC/MS analysis needs derivatization



MS spectrum at 0.673 min (top)
and
reference spectrum (NIST 08) of **acetylsalicylic acid** (M = 180.16 g/mol)

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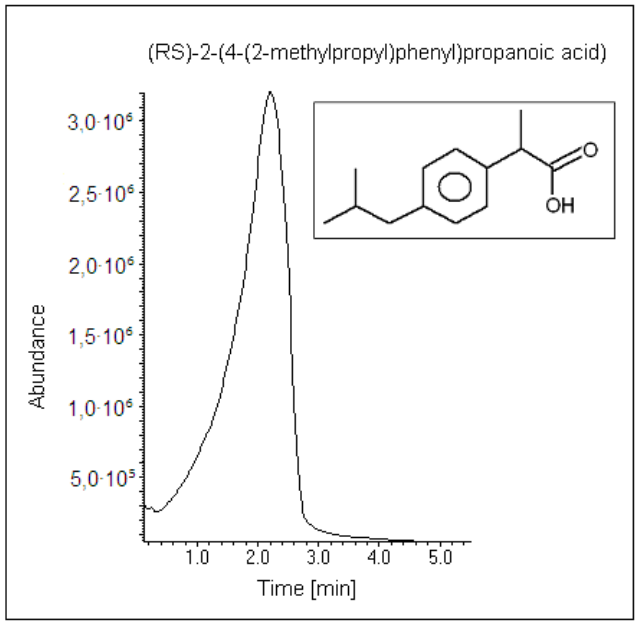
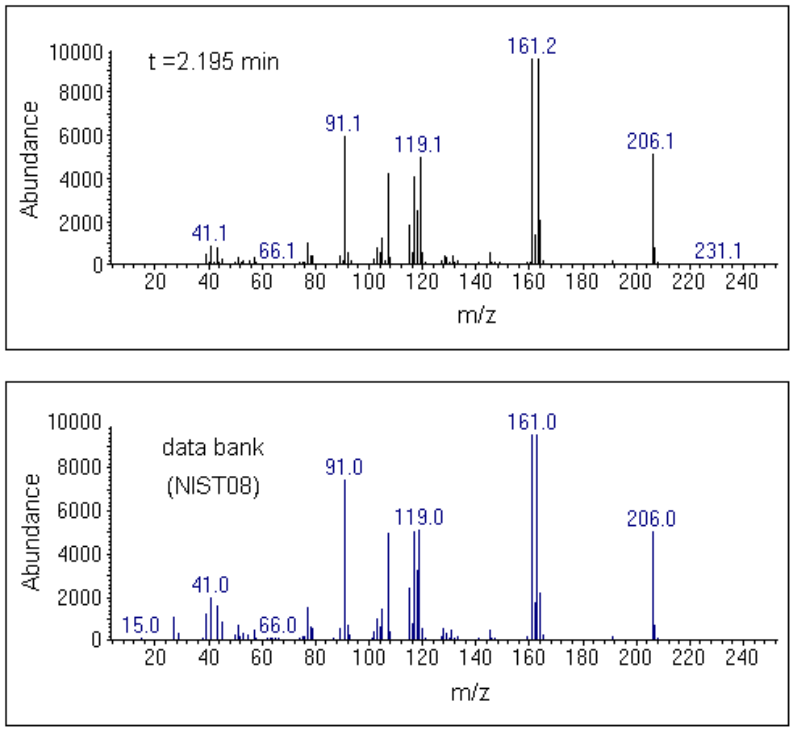
DIP-Application: Pharmaceuticals
Ibuprofen-Tablet (EI, Solid Sample)



Vaporization Curve of Ibuprofen-Tablet

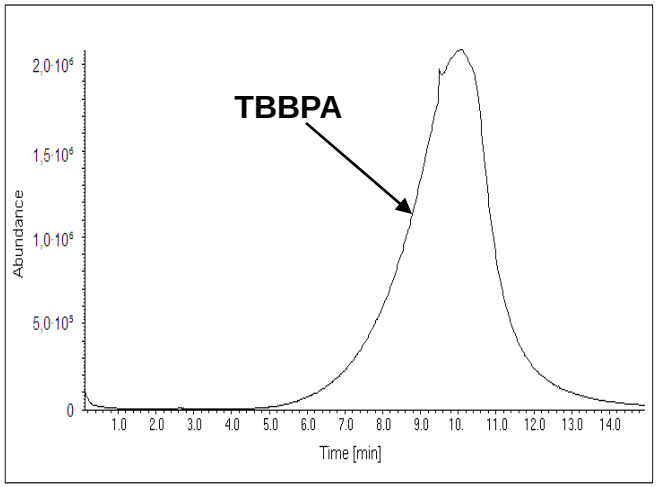
MS	Agilent 5975 MSD, EI mode
MS Temp	280 °C (Source), 150 °C (Quad), 280 °C (Transfer Line)
Full Scan	33-1000 m/z
DIP	solid tip, CO_2-cooling
DIP Temp	5°C, 0.2°C/s to 60 °C, 0.5 °C/s to 400 °C



MS spectrum at 2.195 min (top)
and
reference spectrum (NIST 08) of
Ibuprofen (M = 206.28 g/mol)

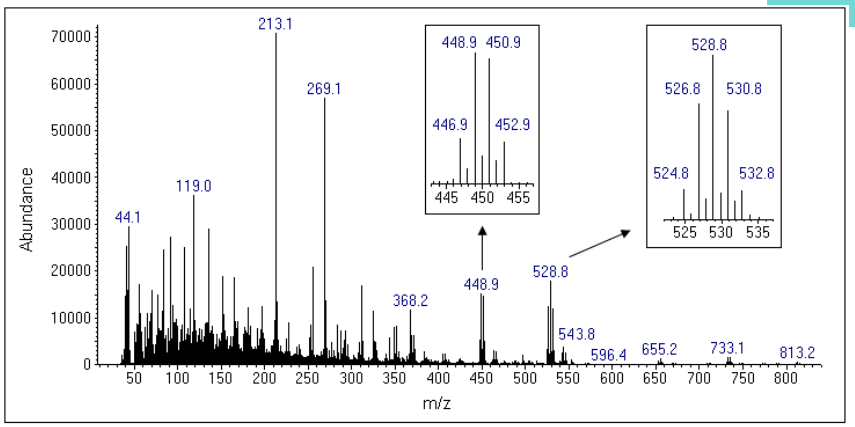
➔ usual GC/MS analysis of ibuprofen: TMS derivate

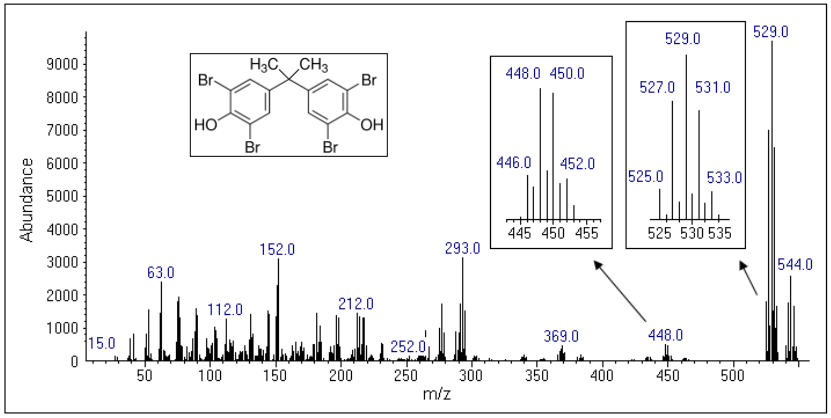
DIP-Application: Brominated Flame Retardant in Printed Circuit Board (EI, Solid Sample)



Vaporization Curve of Printed Circuit Board (chippings)

MS	Agilent 5975 MSD, EI mode
MS Temp	280 °C (Source), 150 °C (Quad), 280 °C (Transfer Line)
Full Scan	33-1000 m/z
DIP	solid tip
DIP Temp	40°C, 0.5 °C/s to 350 °C

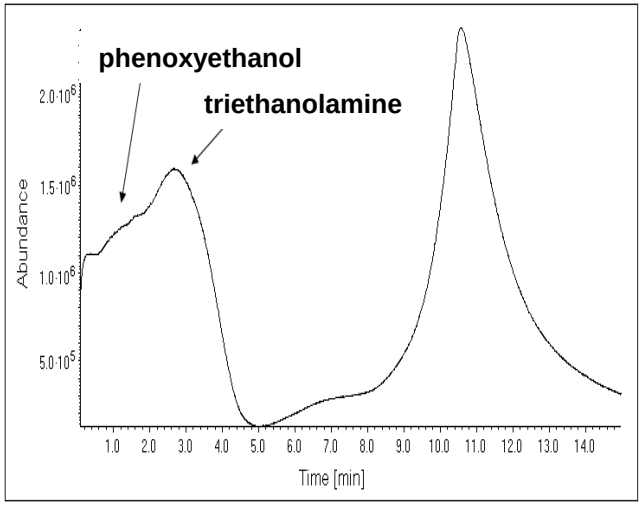




➔ TBBPA:
mainly used flame retardant for printed circuit boards instead of banned PBDEs

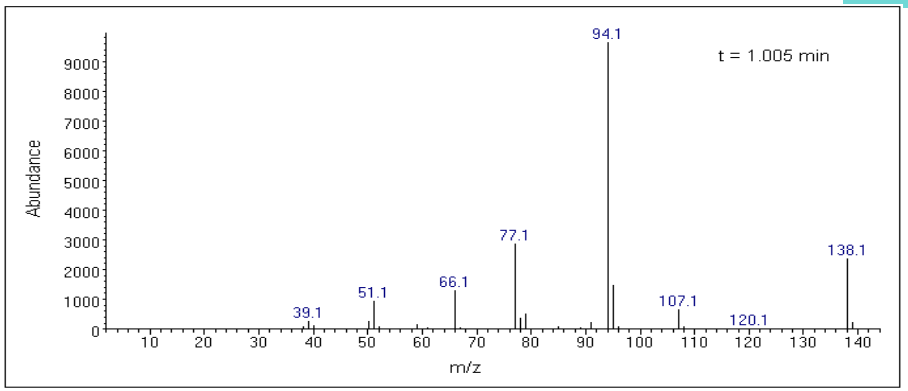
MS spectrum at 9.191 min (top)
and
reference spectrum (NIST 08) of
Tetrabromobisphenol A
(TBBPA, M = 543.88 g/mol)
see typical isotope distribution pattern for bromine substitution

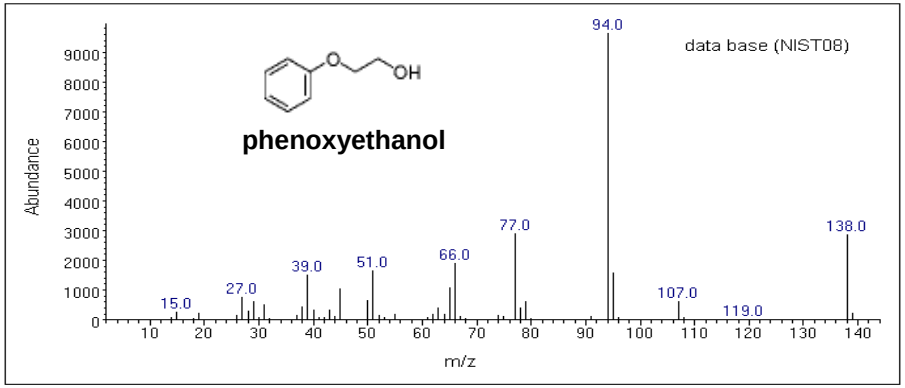
DIP-Application: Cosmetics
Additives in Hair Gel (I) (EI, Solid Sample)



Vaporization Curve of Hair Gel

MS	Agilent 5975 MSD, EI mode
MS Temp.	280 °C (Source), 150 °C (Quad), 280 °C (Transfer Line)
Full Scan	33-800 m/z
DIP	solid tip
DIP Temp.	40°C, 0.5 °C/s to 350 °C





MS spectrum at 1.005 min (top)
and
reference spectrum (NIST 08) of **phenoxyethanol** (M = 138.16 g/mol)

➔ phenoxyethanol is used as **preservative**
(functional components of hair gel: cationic polymers)

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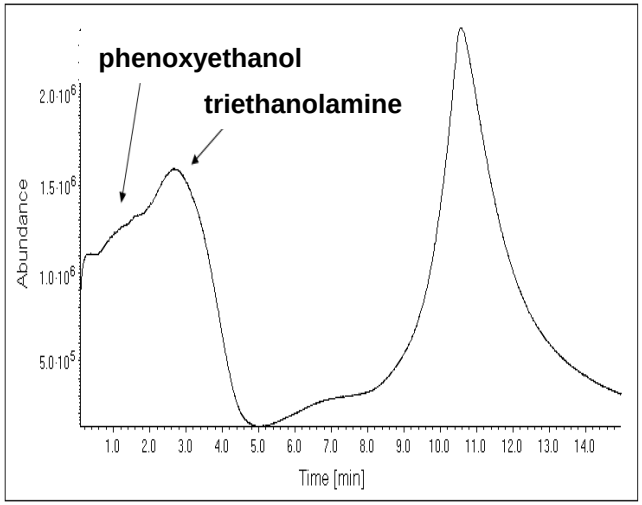
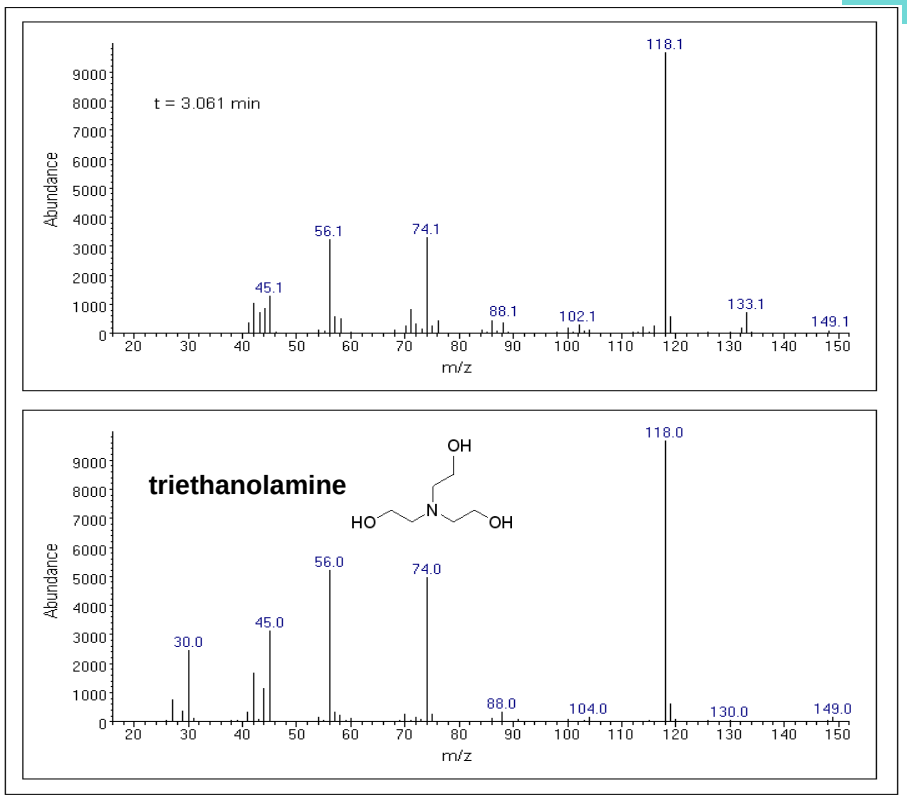
DIP-Application: Cosmetics
Additives in Hair Gel (II) (EI, Solid Sample)



Vaporization Curve of Hair Gel

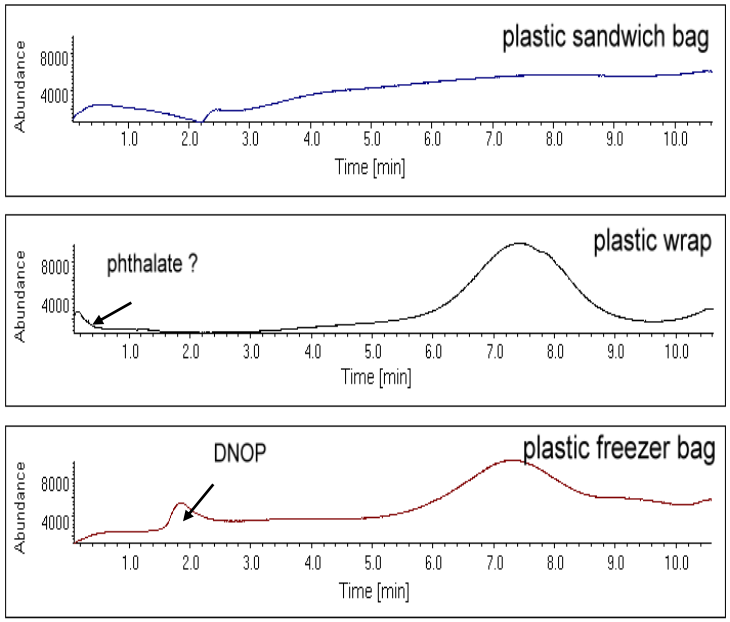
MS	Agilent 5975 MSD, EI mode
MS Temp.	280 °C (Source), 150 °C (Quad), 280 °C (Transfer Line)
Full Scan	33-800 m/z
DIP	solid tip
DIP Temp.	40°C, 0.5 °C/s to 350 °C

➔ triethanolamine is used as **pH-balancer**



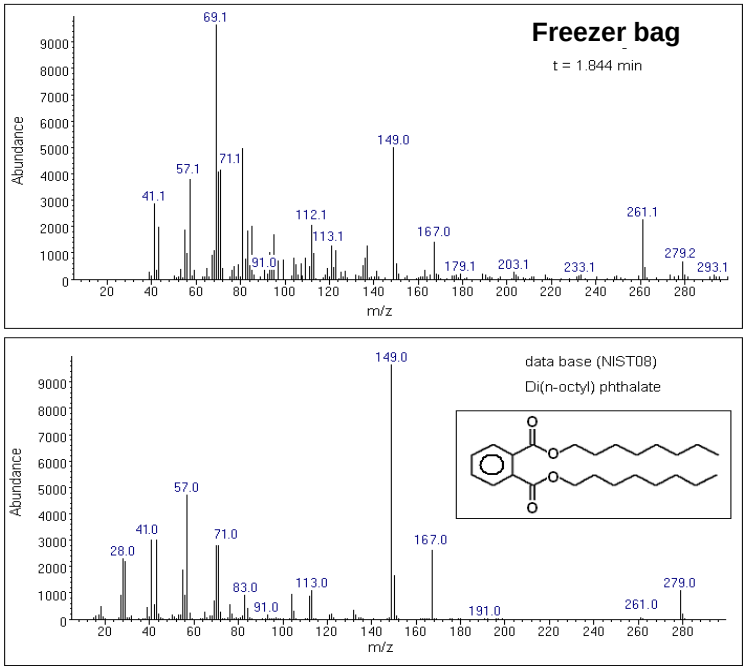
MS spectrum at 3.061 min (top)
and
reference spectrum (NIST 08) of **triethanolamine** (M = 149.19 g/mol)

DIP-Application: Food Packaging
Phthalates (EI, Solid Sample)



Vaporization Curves of 3 food packaging

MS	Agilent 5975 MSD, EI mode
MS Temp.	280 °C (Source), 150 °C (Quad), 280 °C (Transfer Line)
Full Scan	33-800 m/z (bags), 33-600 m/z (wrap)
DIP	solid tip
DIP Temp.	40°C, 0.5 °C/s to 350 °C

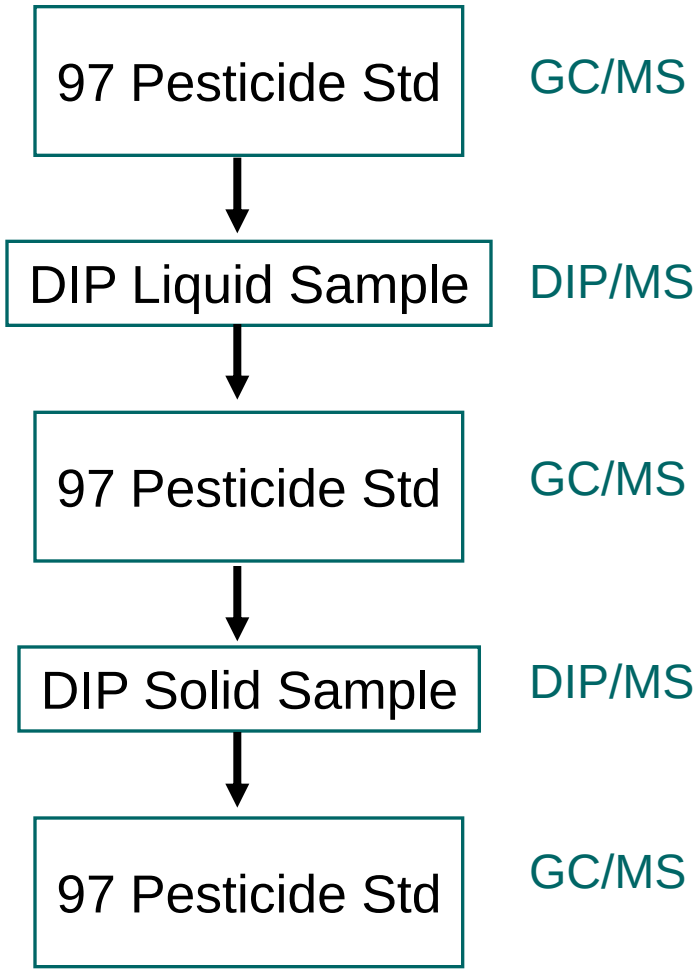


MS spectrum at 1.944 min (plastic freezer bag)
and
reference spectrum (NIST 08) of
di(n-octyl)phthalate (DNOP, M = 390 g/mol)

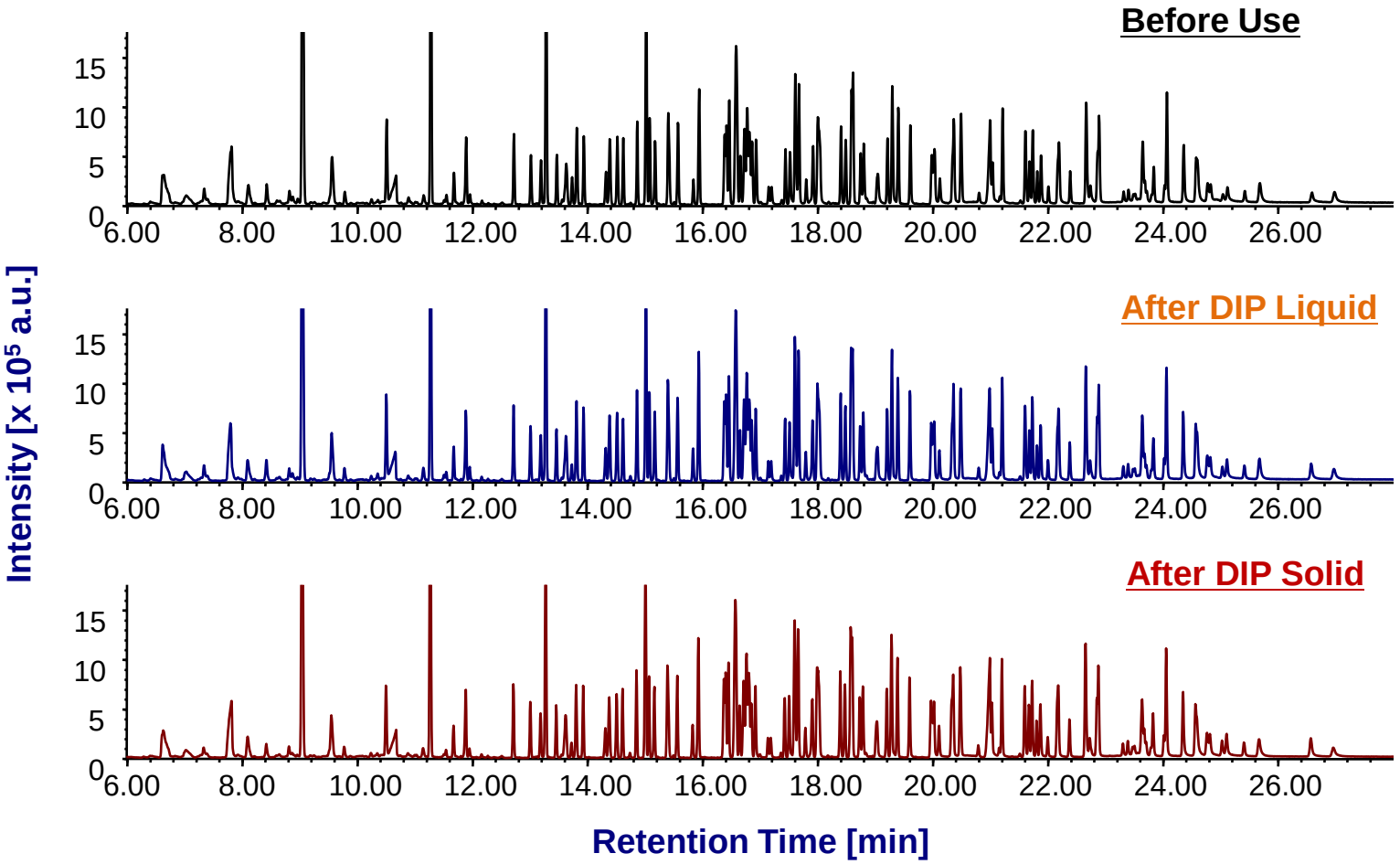
➔ **freezer bag** contains phthalates:
DNOP (see ref. spectrum),
fragment ion 293 gives indication for
di-n-nonyl phthalate (DINP, M = 418 g/mol)

Sensitivity Test

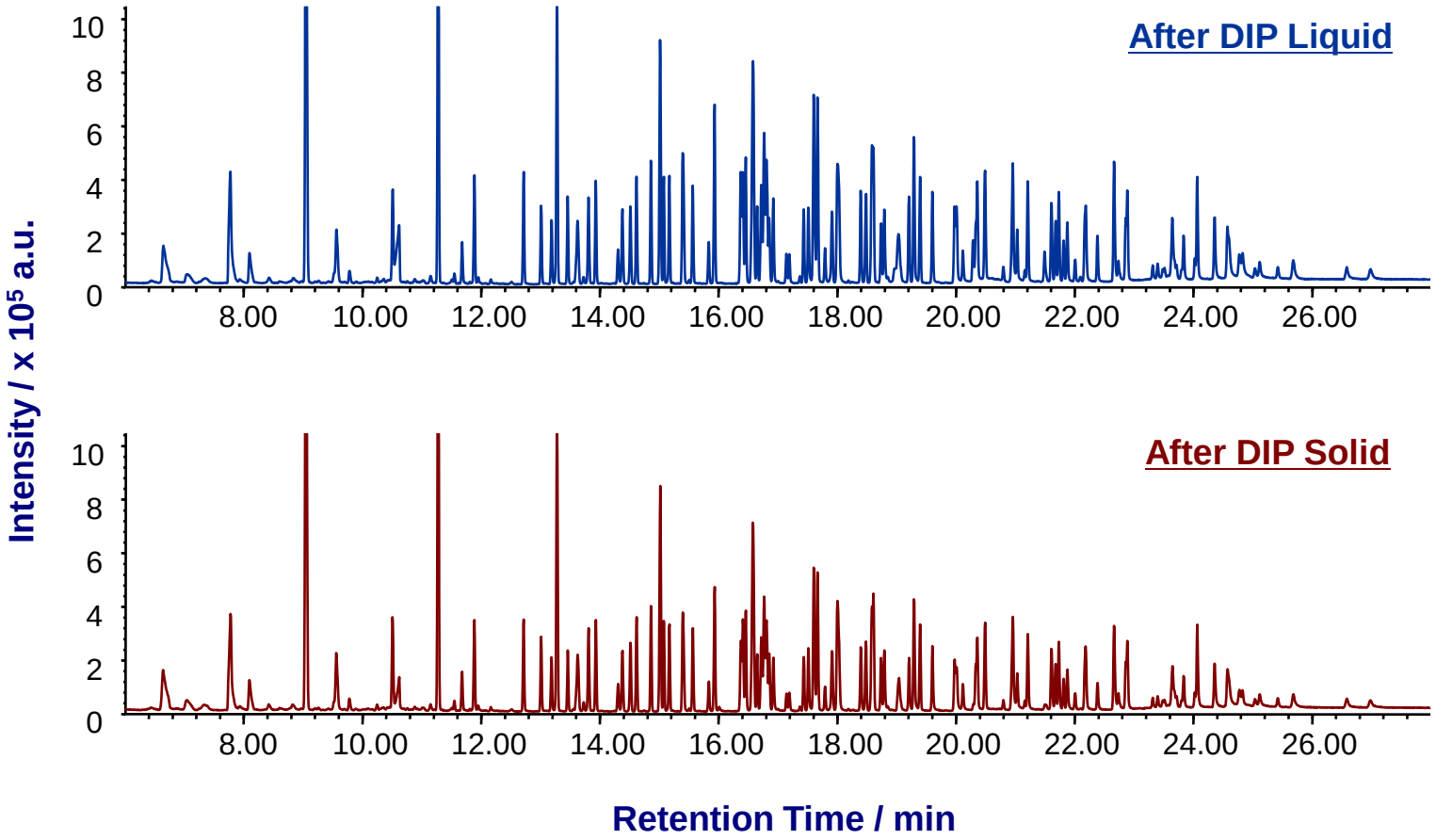
Does DIP measurement influence GC/MS sensitivity?



Sensitivity Test:
GC/MS: TICs of Pesticides at 5 ng



Sensitivity Test:
GC/MS: TICs of Pesticides at 1 ng



DIP Features

- **enhancement** of the MSD application range:
  analysis of low volatile samples (liquid and solid) without preceding chromatographic separation
  → MS as well as GC/MS
- **short analysis time** for mass spectra:
  ideal for high throughput quality control analysis (screening) in EI and CI mode of the 5973/5975 MSD and 7000 Triple Q
- **quick changeover** from GC/MS to DIP-MS:
  it is not necessary to uncouple the GC/MS interface
- **ease of use**:
  microprocessor controlled introduction of the sample into the high vacuum minimizes the change of accidental venting the MS
- **extensive data analysis**
  using the tools of MS ChemStation and MassHunter software
- **possibility of automation** and **online analysis** by combination with a PAL autosampler

DIP Specifications

➢ Requirement:	GC/MS System with Standard or Performance Turbo Pump
➢ Modes:	DIP/MS and GC/MS without uncoupling the GC/MS interface
➢ Sampling:	Probe tip for liquid and solid samples
➢ Temperature Range:	RT up to 400 °C
➢ DIP Temperature Control:	0.1 °C -2 °C/s, max. 3 ramps subsequent cooling with pressurized air
➢ Control system:	DIP software to control the system by PC or via control panel of the DIP
➢ Analysis of mass spectra:	MS ChemStation, MassHunter
➢ Options:	• CO_2-Cooling Kit, • combination with PAL autosampler for automation and online-analysis • SIM Control software for adaptive sequences

SIM
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