

2011/12 **Environmental Products**

- **some Features . . . Restek products**
- **Overview presentation**

Outline

- Semivolatile organic compounds
- Volatile organic compounds
- Pesticide
- Air sampling

Semivolatile Overview

- Method Summary:
 - Semi-volatile compounds are extracted from sample matrix using solvent (methylene chloride). The extract is concentrated and an aliquot is typically analyzed by GC/MS.
- Compound lists include US EPA Methods 8270, 625, & 525
- Products offered:
 - GC Columns (Rxi-5Sil MS)
 - Reference Standards
 - Inlet Supplies (liners, inlet seals, septa)
 - Sample prep products (extraction disks, ASE Parts)
 - Autosampler Vials & Syringes

What Compounds are Considered Semi-Volatiles?

- Solvent extractable (over 100 compounds)
- Wide variety of compound classes
- pH adjustment for base-neutral & acidic compounds

pH = 2

pH = 12

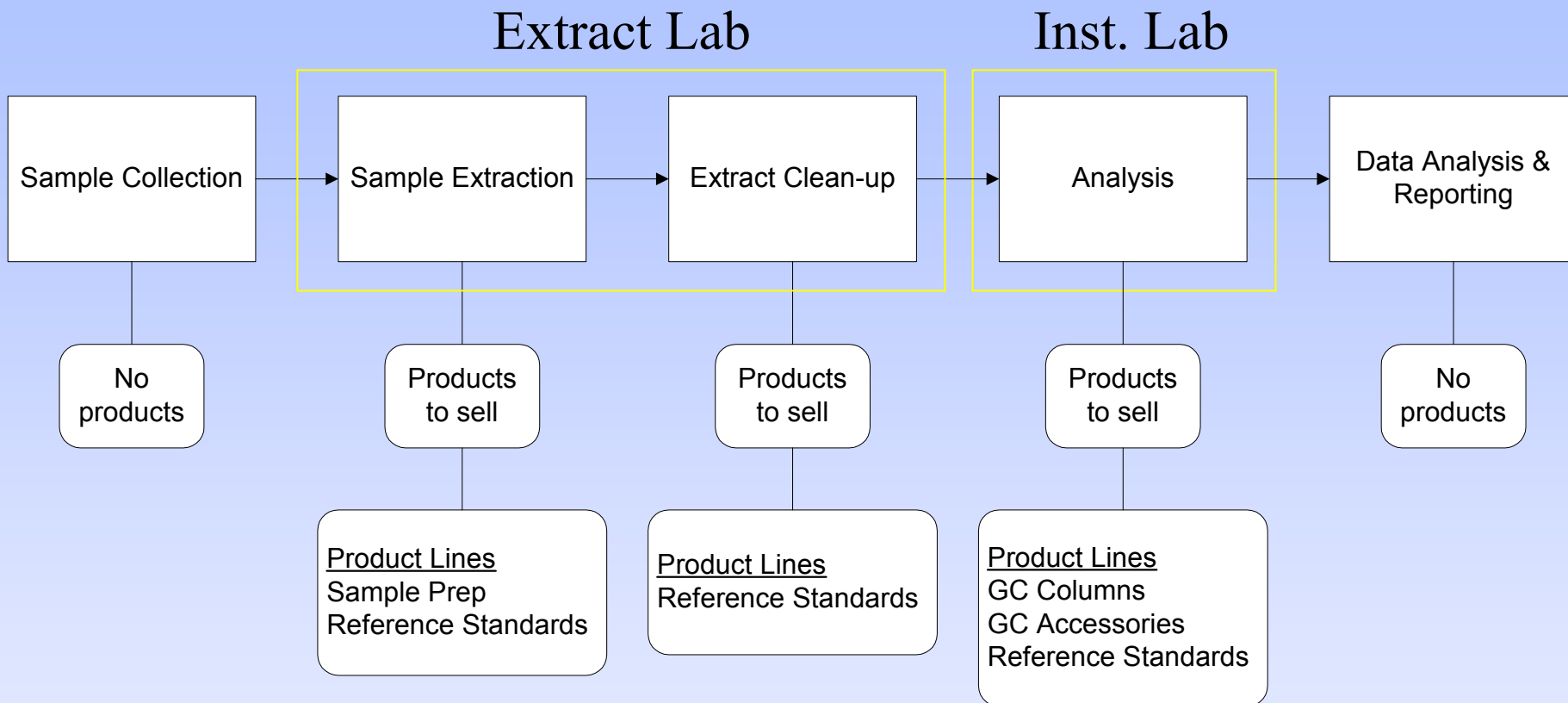


Phenols

PAHs, phthalates, pesticides,
chlorinated hydrocarbons

Nitrosamines,
anilines

Sample Process for Semi-Volatiles



Semivolatiles Products

- best available column
 - Rxi-5Sil MS columns (best in-class for inertness, bleed, resolution of benzo(b&k)fluoranthene, and reproducibility)
 - Calibration mixes
 - Mega Mixes (75 compounds in one ampoule)
 - Surrogate & matrix spikes
 - Custom standards
 - Sky Inlet Liners
 - Single gooseneck w/ wool
 - Flip Inlet Seals (more reliable seal to eliminate injection port leaks and double the use)
 - Leak detector
- other products:
 - Other instrument supplies
 - Vials & Syringes

Semivolatiles Analytical Solution

- GC Column

- 30m x 0.25mm x 0.25µm Rxi-5Sil MS
 - Excellent choice for capacity and ruggedness
 - Flow matches well with MS pumping capacity
 - 5m or 10m Integra Guard columns available

- Injection port set-up

- New Sky Inlet Liners
 - 4mm ID single gooseneck liner w/ wool
- New optimized deactivation results in more inert liner

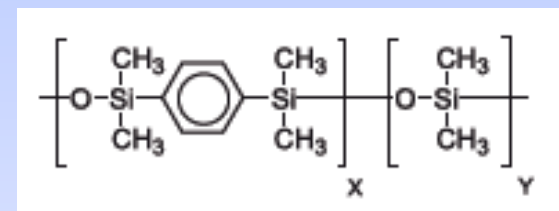


Rxi-5SiI MS

GC Column Technology



- Highest inertness
- Lowest bleed
- Exceptional reproducibility



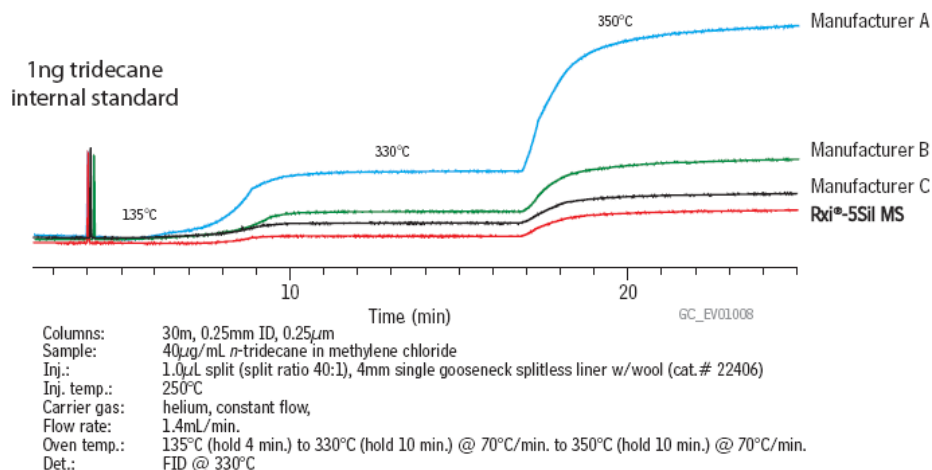
Rxi-5SiI MS

- Combination of features are what is important for environmental analysis of semivolatile organic compounds

Rxi-5Sil MS Column

- Low-bleed
 - Very important for MS, especially ion trap
 - But most vendors have low-bleed columns
 - Rxi is even better
 - DB-5ms, ZB-5ms, VF-5ms

Figure 1 Rxi®-5Sil MS columns outperform competitors, producing lower bleed at 350°C than any other column on the market.



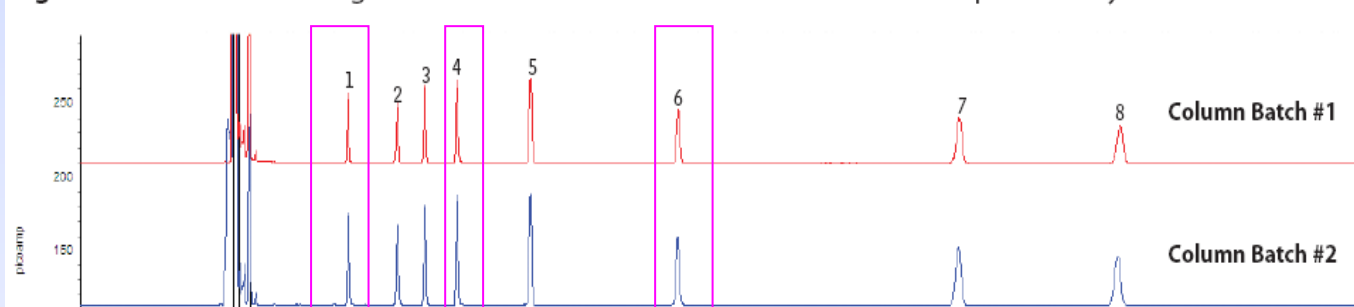
Rxi-5Si1 MS Column

Reproducibility

- Customers want the same chromatography every time they install new column
- Critical parameters of column:
 - Bleed: **Signal to noise**
 - Film thickness: **Absolute retention time**
 - Plates/meter: **Peak sharpness**
 - Selectivity: **Peak relative retention times**
 - Inertness: **Peak response of active compounds**

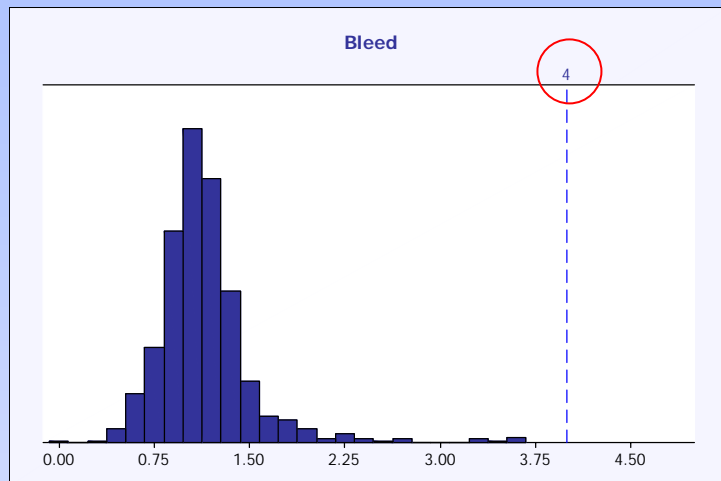
1. 1,6-hexanediol
2. 4-chlorophenol
3. methyl nonanoate
4. 1-decylamine
5. tridecane
6. 1-undecanol
7. acenaphthylene
8. pentadecane

Figure 6 Rxi® columns are engineered to assure column-to-column and lot-to-lot reproducibility.



Rxi-5Sil MS Column Reproducibility

(Rxi-5Sil MS, 30m x 0.25mm, 0.25um)



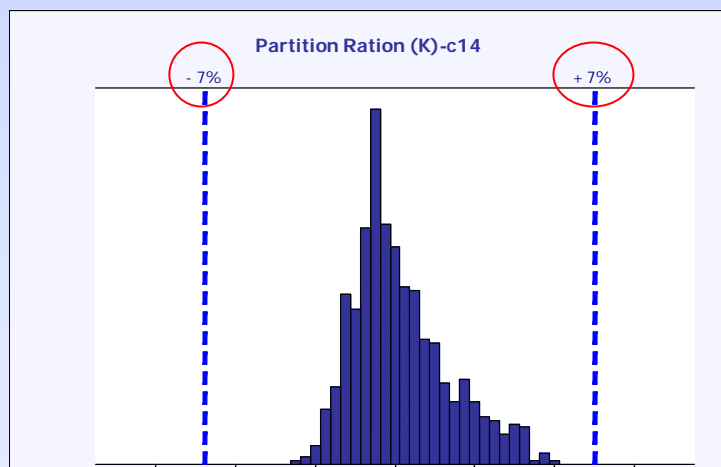
Bleed

Mean: 1.1

Std dev: 0.38

Ind. Std: 4pA

All columns manufactured
in a years time



Partition Coefficient (k)

Mean: 5.6

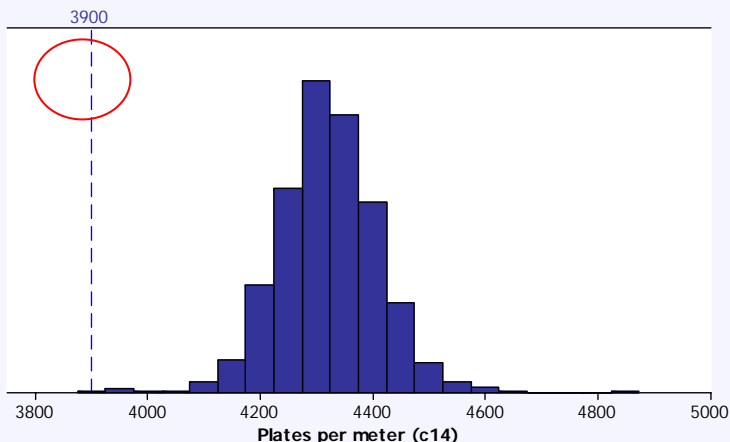
Std dev: 0.09

Ind. Std: +/- 7%

Rxi-5Sil MS Column Reproducibility

(Rxi-5Sil MS, 30m x 0.25mm, 0.25um)

Plates per meter (c14)



Plates/meter

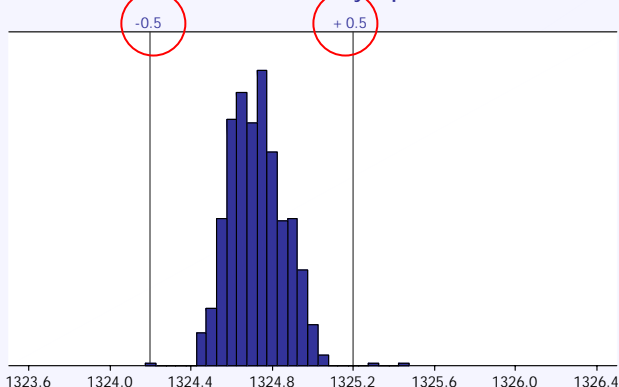
Mean: 4321

Std dev: 91

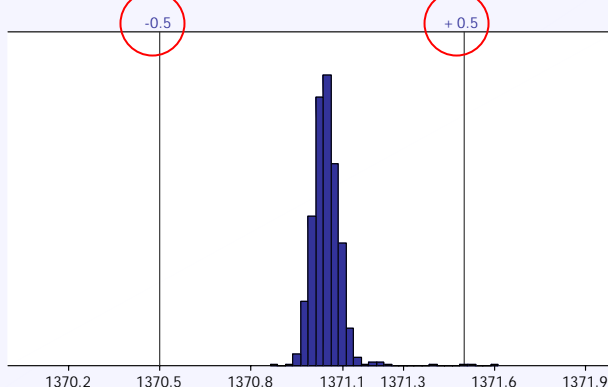
Ind. Std: 3900

All columns manufactured
in a years time

Retention Index 1-Methylnaphthalene



Retention Index 1-Undecanol



Retention Indices (RI)

Methylnaphthalene

Mean: 1324.7

Std dev: 0.13

1-Undecanol

Mean: 1371.0

Std dev: 0.05

Ind. Std: +/-5%

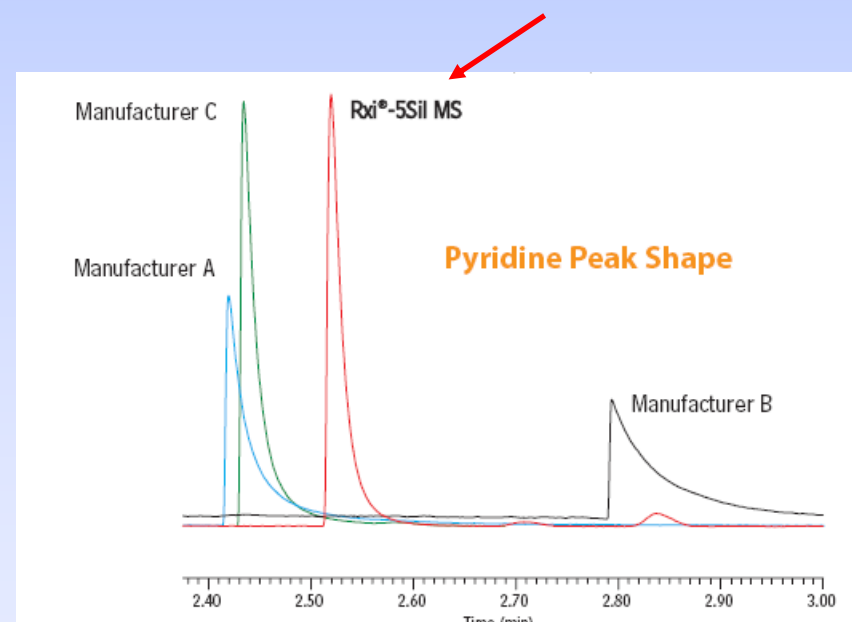
Rxi-5Sil MS Column

- Inertness!
 - An IMPORTANT selling feature for the Rxi-5Sil MS and semivolatile analyses
- Semivolatiles includes a wide range of acids, bases, neutrals, polar compounds
 - Pyridine; 2,4-dinitrophenol; etc.

Table I Rxi®-5Sil MS columns produce high response factors for both basic and acidic compounds.

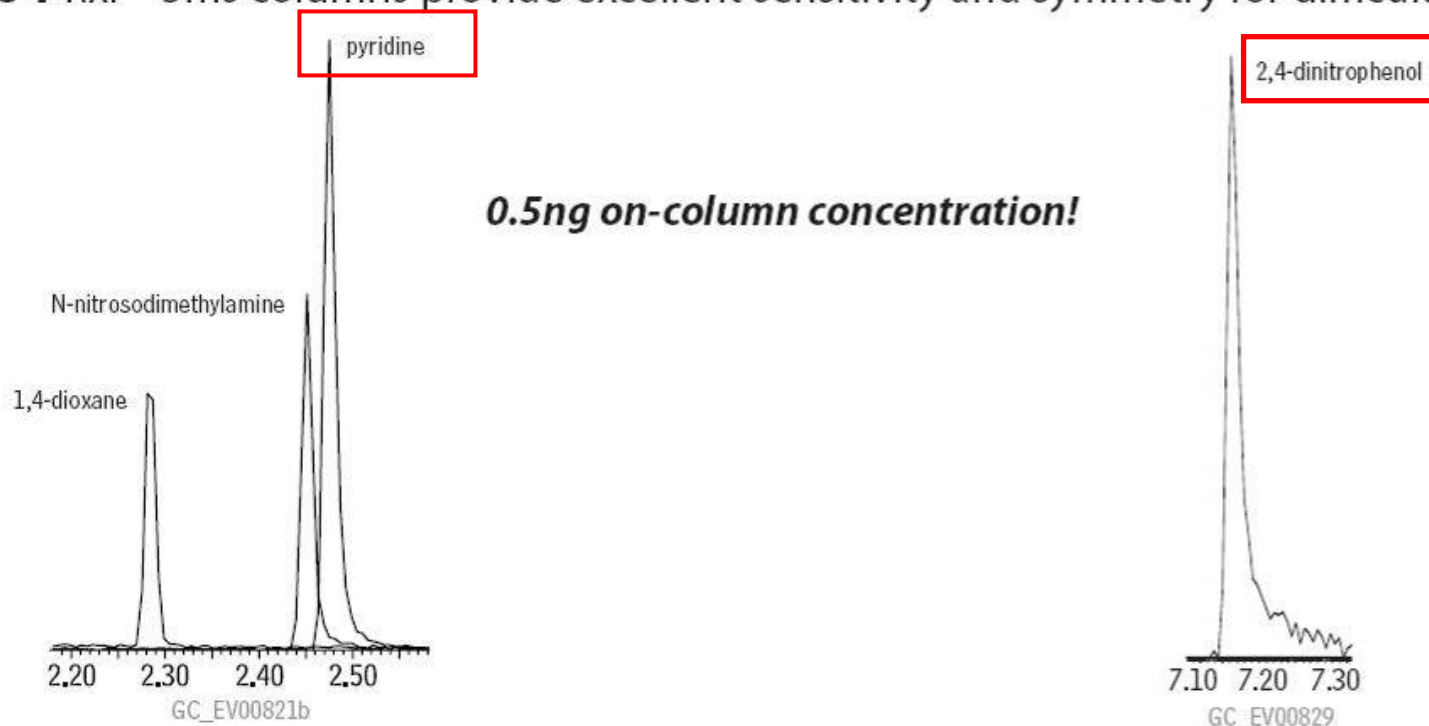
	2,4-dinitrophenol (average RF)	pyridine (average RF)
Rxi®-5Sil MS	0.24	0.74
Manufacturer A	0.20	0.63
Manufacturer B	0.22	0.64
Manufacturer C	0.24	0.65

Response factors are based on phenanthrene. (n=7)



High Inertness of Rxi for Semivolatile Base and Acid at Low Level

Figure 1 Rxi™-5ms columns provide excellent sensitivity and symmetry for difficult compounds.

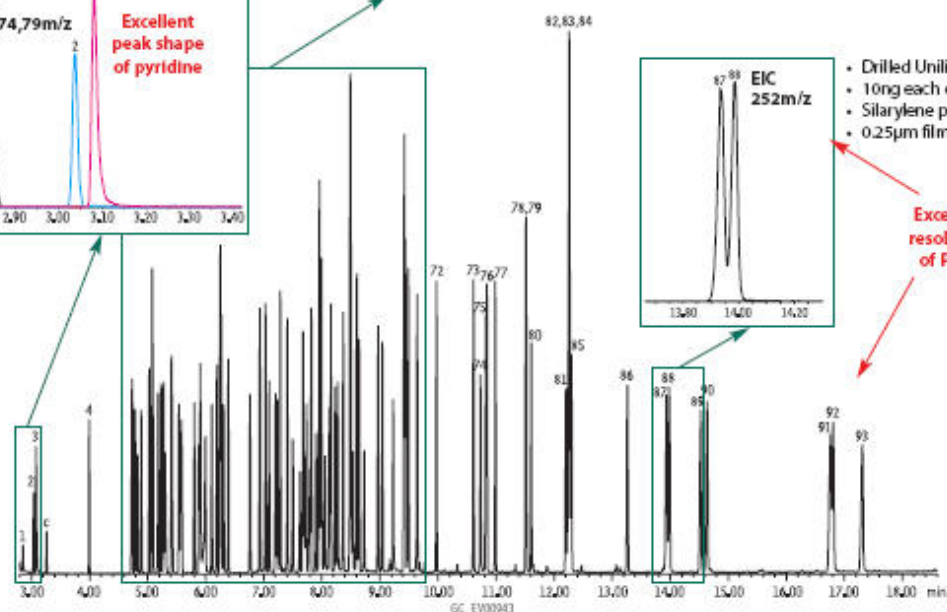
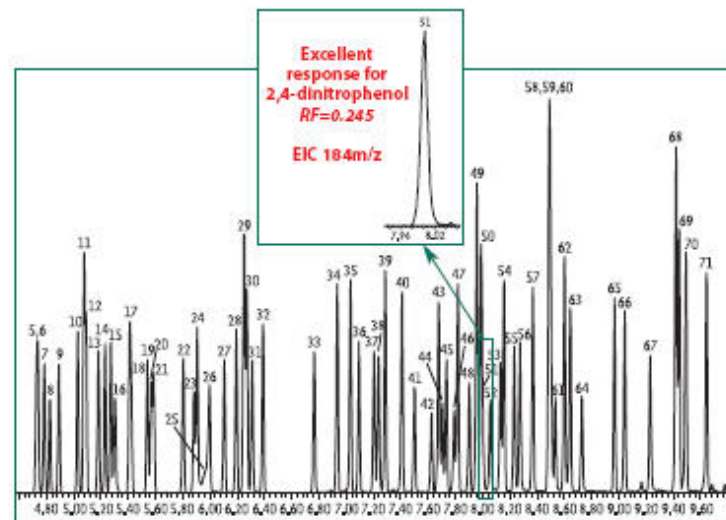
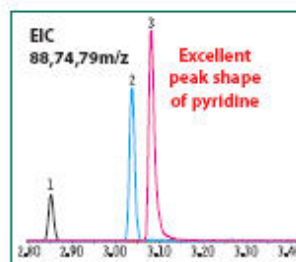


Semivolatile Analysis Rxi-5Sil MS 30m x 0.25mm, 0.25um

10ng on-column

Semivolatile Organics US EPA Method 8270 Rxi®-5Sil MS

Column: Rxi®-5Sil MS, 30m, 0.25mm ID,
0.25um (cat.# 13623)
Sample: US EPA Method 8270 Mix, 1µL of
10µg/mL (IS 40µg/mL)
8270 MegaMix® (cat.# 31850)
Benzoic Acid (cat.# 31879)
8270 Benzodioxin Mix (cat.# 31852)
Acid Surrogate Mix (4/89 SOW) (cat.# 31025)
Revised B7N Surrogate Mix (cat.# 31857)
1,4-Dioxane (cat.# 31853)
SV Intermal Standard Mix (cat.# 31206)
1µL (10ng on-column concentration),
4mm Drilled Unliner® (hole near bottom)
inlet liner (cat.# 20756),
pulsed splitless: pulse 25psi @ 0.2 min.,
60mL/min. @ 0.15 min.,
250°C/min.
Inj. temp.: 250°C
Carrier gas: helium, constant flow
Flow rate: 1.2mL/min.
Oven temp.: 40°C (hold 1.0 min.) to 280°C @ 25°C/min.
to 320°C @ 5°C/min. (hold 1 min.)
MS
Det.: Transfer line
temp: 280°C
Scan range: 35-550amu
Ionization: EI
Mode: scan



- Drilled Unliner® liner,
- 10ng each compound
- Silarylene phase
- 0.25µm film thickness

Rxi-5Sil MS

30m x 0.25mm, 0.25um

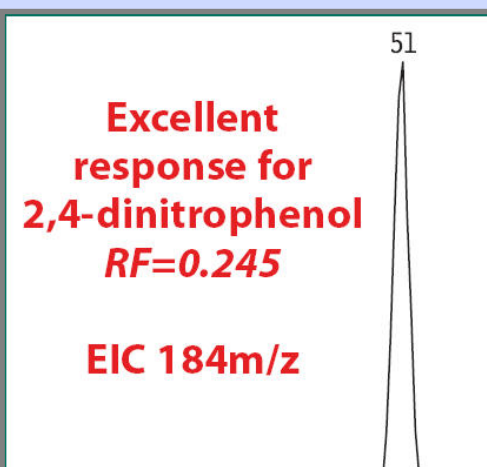
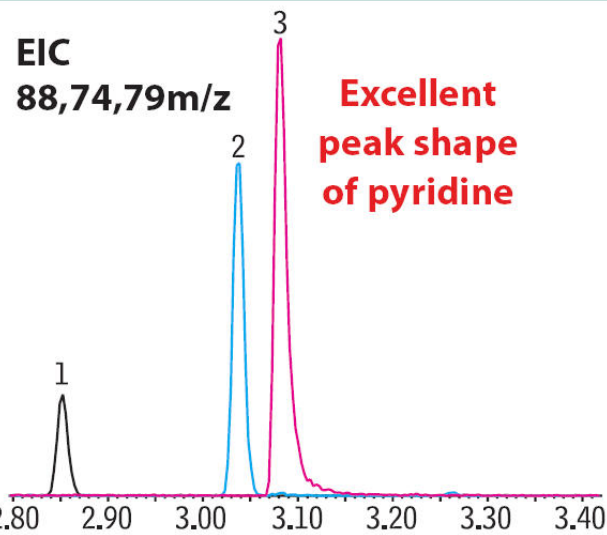
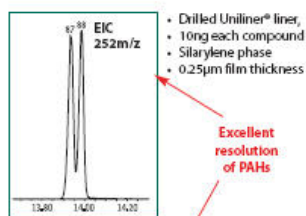
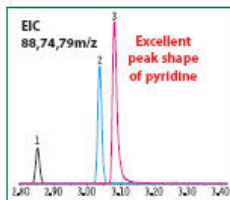
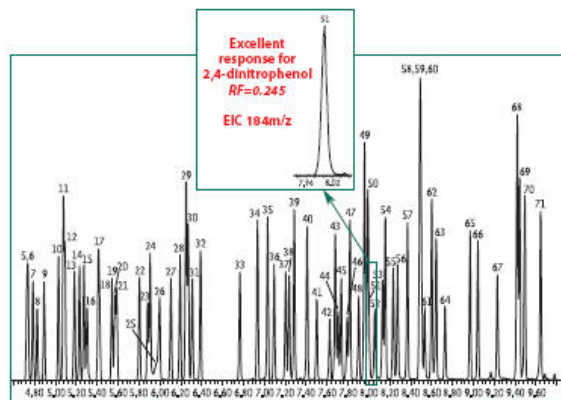
10ng on-column

Semivolatiles Organics

US EPA Method 8270

Rxi®-5Sil MS

Column: Rxi®-5Sil MS, 30m, 0.25mm ID,
0.25um (cat.# 13623)
Sample: US EPA Method 8270 Mix, 1µL of
10µg/mL (5 40µg/mL)
8270 MegaMix® (cat.# 31850)
Benzoic Acid (cat.# 31879)
8270 Benzidines Mix (cat.# 31852)
Acid Surrogate Mix (4/B SOW) (cat.# 31025)
Revised B/N Surrogate Mix (cat.# 31857)
1,4-Dioxane (cat.# 31853)
SV Internal Standard Mix (cat.# 31206)
Inj: 1.0µL (10ng on-column concentration),
4mm Drilled Uniliner® (hole near bottom)
inlet liner (cat.# 20750),
pulsed splitless: pulse 25psi @ 0.2 min.,
60mL/min. @ 0.15 min.
Inj. temp.: 250°C
Carrier gas: helium, constant flow
Flow rate: 1.2mL/min.
Oven temp.: 40°C (hold 1.0 min.) to 280°C/min.
to 220°C @ 5°C/min. (hold 1 min.)
MS
Transfer line
temp.: 280°C
Scan range: 35-550amu
Ionization: EI
Mode: scan



High inertness on Rxi-5Sil MS

RESTEK

CHROMALYTIC +61(0)3 9762 2034
ECHnology Pty Ltd

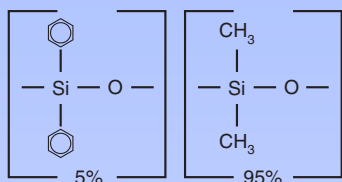
Australian Distributors
Importers & Manufacturers
www.chromtech.net.au

12/13

Website NEW : www.chromalytic.com.au E-mail : info@chromtech.net.au Tel: 03 9762 2034 . . . in AUSTRALIA

2011/16

Rxi-5ms and Rxi-5Sil MS columns are not the same

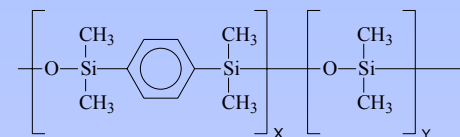


Rxi-5ms

(5% Diphenyl-PDMS)

Rxi-5Sil MS

(Silarylene-stabilized equivalent of
5% diphenyl-PDMS)



Restek

Rtx-5, Rtx-5ms

Rxi-5Sil MS

Agilent

HP-5, HP-5ms, DB-5,
Ultra-2

DB-5ms

Varian

CP-Sil 8 CB

VF-5ms, CP-Sil 8 CB LB/MS

Alltech

AT-5

AT-5ms

Supelco

Equity-5

MDN-12, SLB-5ms

SGE

BP-5

BPX-5

Phenomenex

ZB-5

ZB-5ms

Machery-Nagel

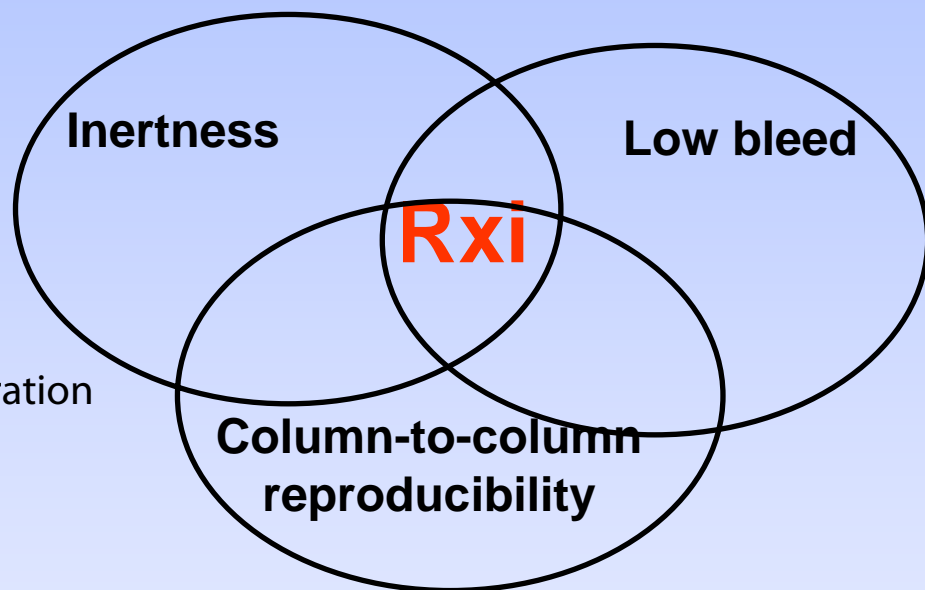
Optima-5

Optima-5ms

Value to the chromatographer

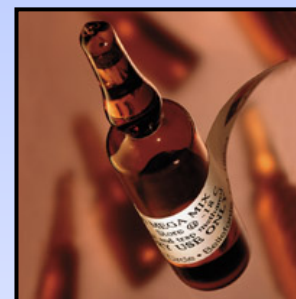


- The combination of column inertness, low bleed and column-to-column reproducibility is what makes the Rxi-5Sil MS valuable to analytical chemists
- Ultra inert column
 - Reduced tailing of active compounds
 - Higher response factors
 - More accurate data from better integration
- Low bleed
 - Higher signal to noise
 - Lower detection limits
- Column-to-column reproducibility
 - Reduced down time from column exchange



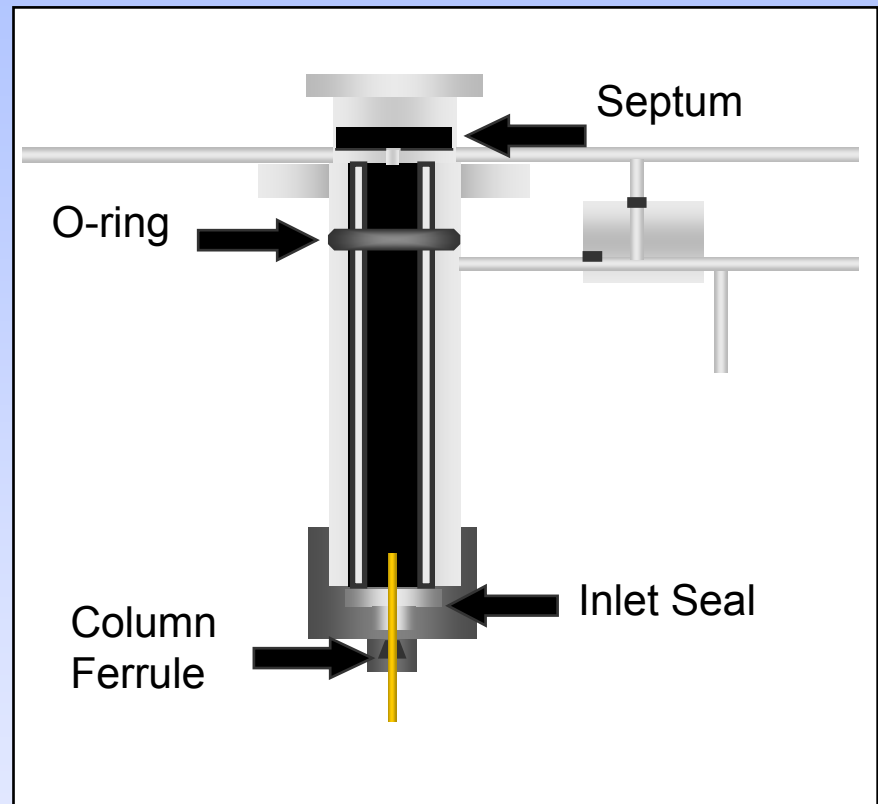
Semivolatile Standards

- Analytical reference materials
 - Up to 76 compound MegaMix®
 - Additional compounds available
 - Matrix spike solutions
 - Internal standards
 - Surrogate standards



Injection Ports

- Regular maintenance required
 - Septum
 - O-ring
 - Liner
 - Inlet seal
 - Column ferrule
- Why?
 - Build up of nonvolatile material in liner and beginning of column causes tailing of late eluting compounds (PAHs), active sites for acids (PCP, 2,4-dinitrophenol)




Injection Ports

- Septum

- 3 types
 - IceBlue (250°C)
 - Thermolite (340°C)
 - BTO (400°C)
- Recommend Thermolite

- O-rings

- Max. Temp: 350°C



IceBlue® Septa

Thermolite® Septa

BTO® Septa


Septa for Agilent GCs Preconditioned and ready to use!

Septum Diameter	25-pk.	50-pk.	100-pk.
Thermolite Septa (usable to 340°C inlet temp.)			
5mm (3/16")	27120	27121	27122
9.5mm (3/8")	27135	27136	27137
10mm	27138	27139	27140
11mm (7/16")	27141	27142	27143

High Temperature O-Rings for Agilent GCs

NEW!

- Usable to 350 °C inlet temperature.
- Fits split (6.3 mm OD) and splitless (6.5 mm OD) liners.



Description	Max. temp.	10-pk. cat.#	25-pk. cat.#
High Temperature O-Rings for Agilent GCs	350°C	23403	23404

Injection Ports

• Sky Inlet Liners

- Many geometry of liners used
- Recommend single gooseneck w/ wool
 - Inert for active compound
 - Wool traps nonvolatile materials



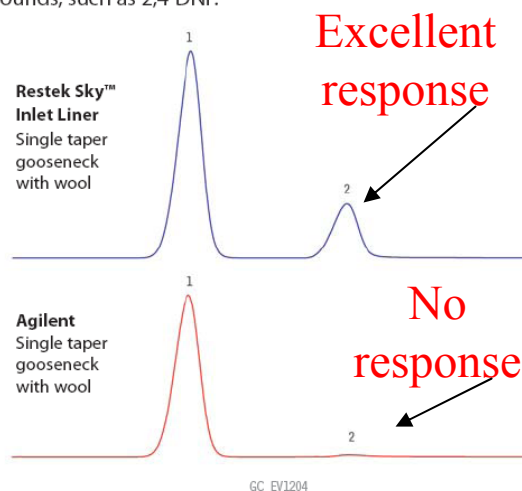
Wool

Gooseneck at bottom of liner



2,4-dinitrophenol response

Figure 2 The state-of-the-art deactivation used for Sky™ liners with wool results in higher responses for active acid compounds, such as 2,4-DNP.



Excellent response

No response

Increase the response of active analytes with inert Sky™ inlet liners!

	2,4-DNP
	Response Factor
Restek	0.28
Agilent	0.02

Peaks
1. Acenaphthene (IS)
2. 2,4-DNP

10ng injected

Column Rxi®-5Sil MS, 15 m, 0.25 mm ID, 0.25 µm (cat.# 13620);
Sample 10 µg/mL each 2,4-dinitrophenol and acenaphthene standard in methylene chloride; Injection Inj. Vol.: 1 µL splitless (hold 1 min.);
Liner: Comparison of Sky™ Single Taper Gooseneck Inlet Liner with Wool (cat.# 23303.5) and Agilent Single Taper Gooseneck Inlet Liner with Wool (cat.# 5062-3587); Inj. Temp.: 250 °C.

Injection Port

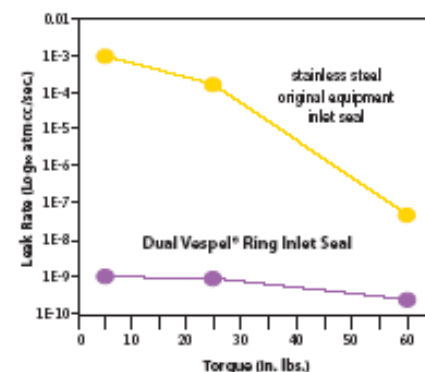
- Inlet seals

- Recommend Gold Flip Seal
- Double use
- Gold plating is inert for active compounds

- Note: Do need adaptor, 1 time purchase



Figure 1 The Dual Vespel® Ring Inlet Seal achieves leak-tight seals even at low torque, reducing the chance of leak-related problems.



Easy to seal

Feature	Benefit
Reversible design.	Allows twice as many uses as other seals, at the same cost.
Vespel® ring embedded in bottom surface.	Eliminates need for a washer.
Vespel® ring embedded in top surface.	Very little torque required to make seal—reduces operator variability.
Lower leak rate compared to OEM metal inlet seals.	Less detector noise.
Prevents oxygen from permeating the carrier gas.	Increases column lifetime.
Gold or Siltek Treated seals.	Reduces breakdown and adsorption of compounds, maximizing component transfer to GC column.

Sample Extraction for Semi-Volatiles

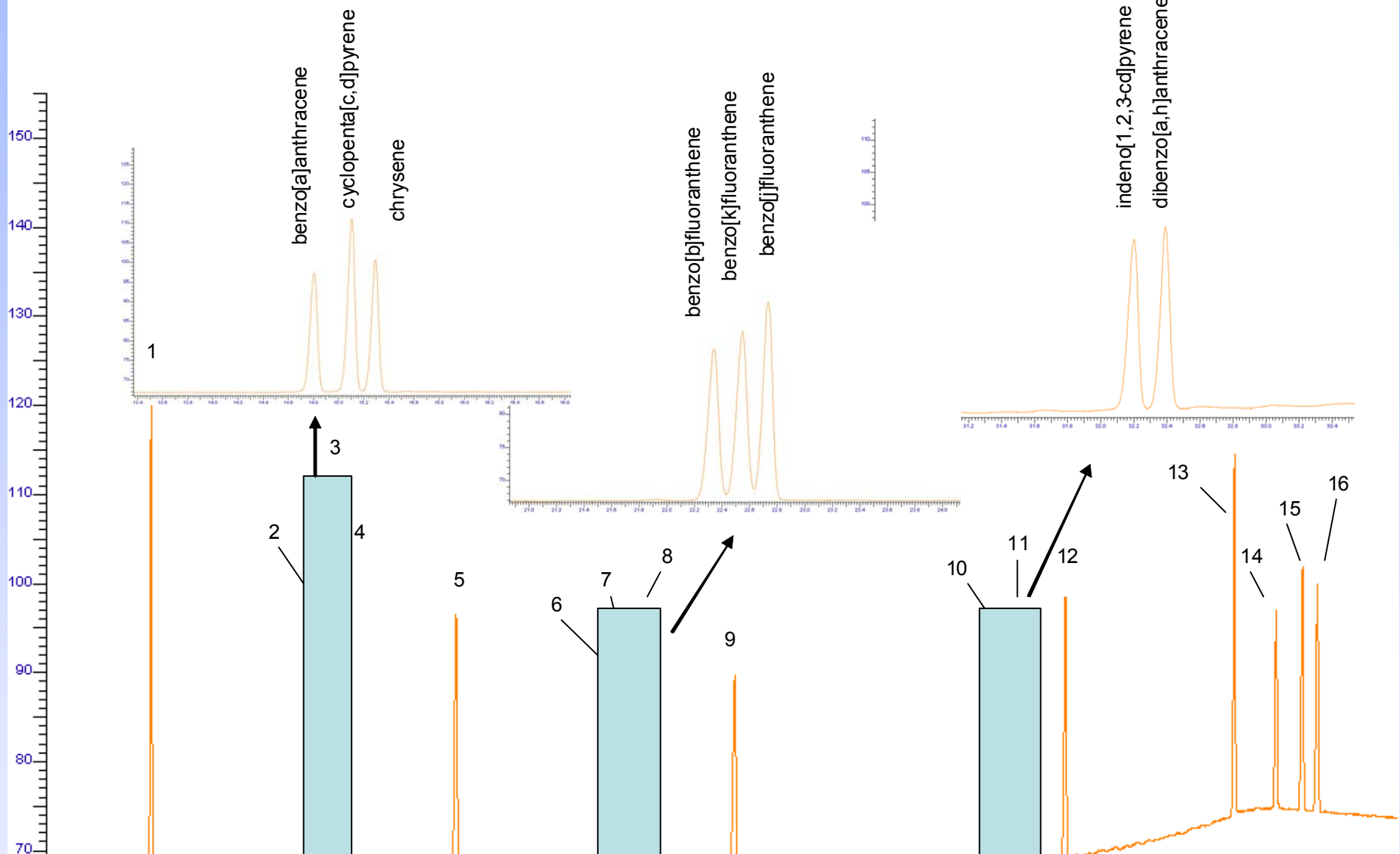
- Extraction Disks
 - 47mm C18, C8, and DVB
 - Drinking water methods
- ASE Replacement Parts
 - Models 200 & 300
 - Cells, PEEK Washers, Filters, tools
 - Used with Dionex automated solvent extractor
 - Soil extraction
- Surrogate & matrix spikes
 - Added to samples prior to extraction
 - Higher concentration & larger package size for lower spike per sample cost
 - QA documentation available
 - On-line data pack



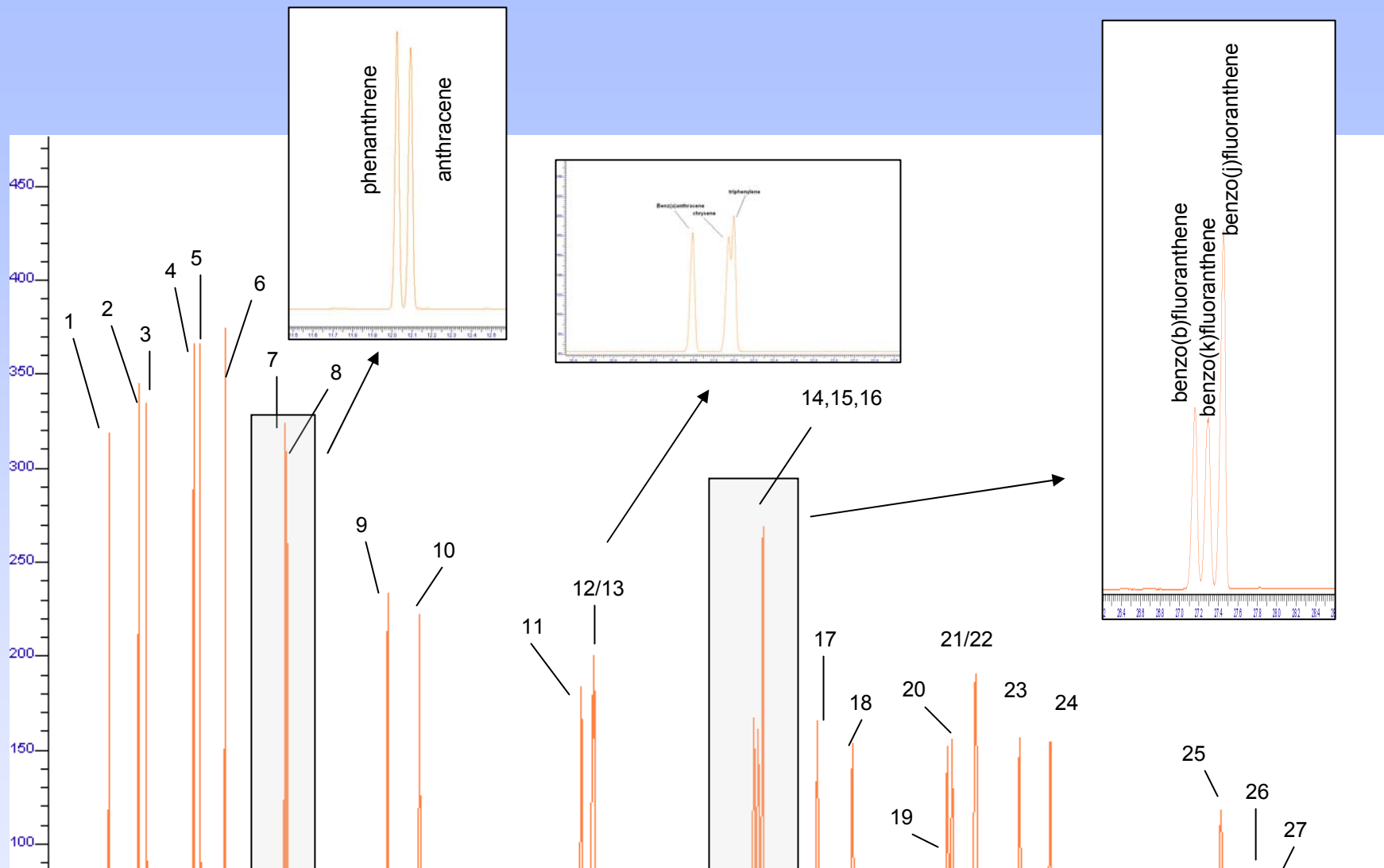
Polycyclic Aromatic Hydrocarbons (PAHs)

- Rxi-17Sil MS
 - High max temp @ 360°C
 - Column dimension: 30m x 0.25mm, 0.25um
- Competitor columns
 - DB-EUPAH (340°C)
 - Varian Select PAH (350°C)
 - Column dimensions:
 - Columns “down coated”
 - 30m x 0.25mm, 0.15um
 - 20m x 0.15mm, 0.1um
 - We are investigating thinner coating affects

EU 15 + 1 PAHs using an Rxi-17sil ms (20m x 0.18mm x 0.18μm)



PAHs using an Rxi-17Sil MS (30m x 0.25mm x 0.25 μ m)



General Chromatography Supplies

- Ferrules



- Ferrule removal kit



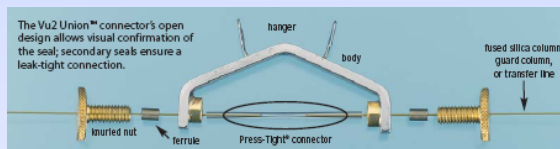
- Gas generators



Leak detector



Super clean gas filters



Column connectors



Scoring wafers

Volatile Analysis

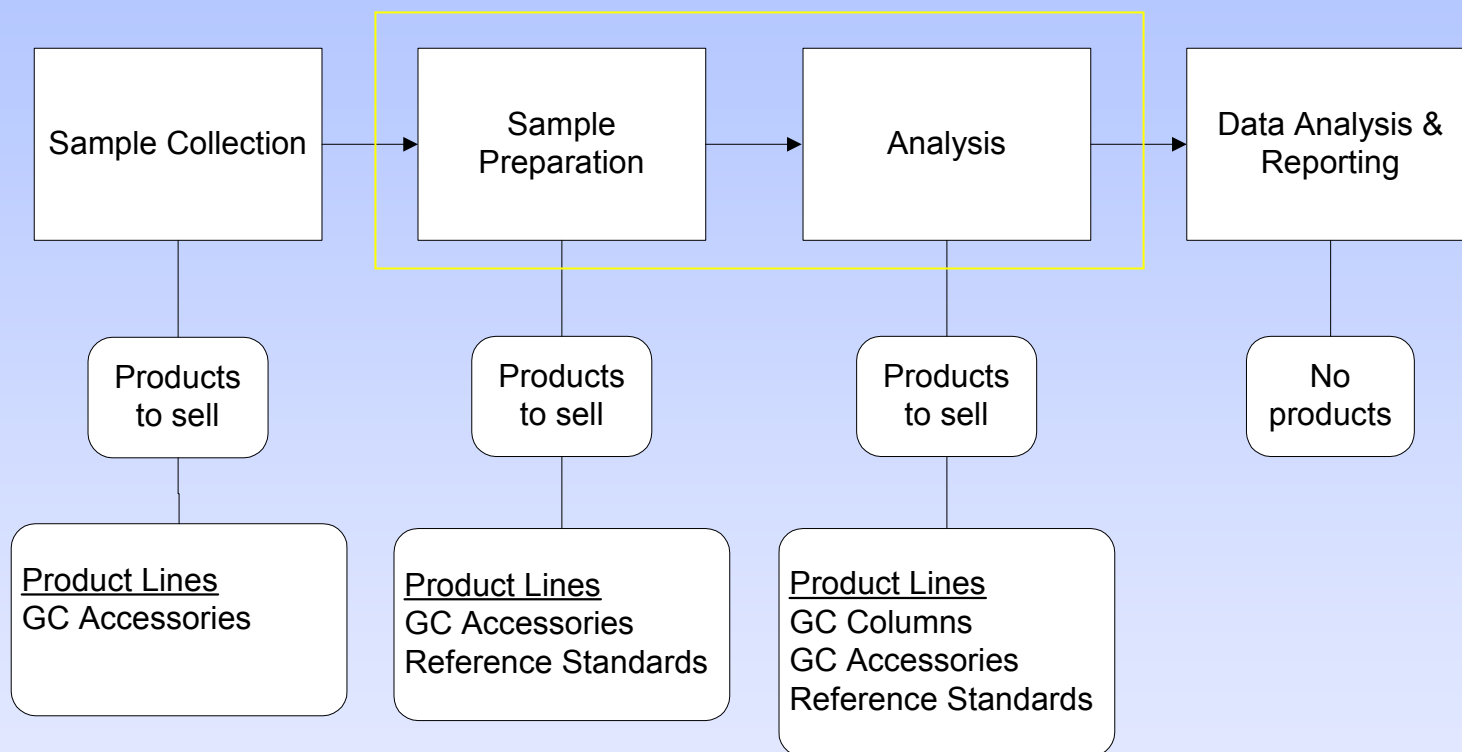
- How laboratory is set-up
- Analysis of volatile compounds
 - Rtx-VMS
 - Rxi-624Sil MS
- Instrument maintenance

Volatiles Overview

- Method Summary:
 - Volatile compounds are extracted from sample matrix using a Purge & Trap or headspace concentrator. Analysis is typically done by GC/MS, but GC with selective detectors are also used.
- Compound lists to reference: US EPA Methods 8260, 624, & 502.2
 - http://www.epa.gov/epawaste/hazard/testmethods/sw846/online/8_series.htm
- Products offered:
 - GC Columns (Rtx-VMS, Rxi-624Sil MS)
 - Reference Standards
 - Purge & Trap replacement parts (sparger tubes, transfer lines)
 - Pre-cleaned VOA Vials for sample collection

Volatiles Overview

Inst. Lab



Products for the Volatiles Lab:

- best available Columns:
 - Rtx-VMS columns (best overall separation, fast analysis time)
 - Mega Mixes (fewest ampoules and lowest overall cost for standards)
 - NEW Column phase: Rxi-624Sil MS
- other products:
 - Purge & Trap Supplies
 - VOA Vials, etc.

Analysis for Volatiles

GC Columns

- Rtx-VMS
 - First choice for complex volatiles analysis
 - Offers most complete separation
 - Unique phase exclusive to Restek
 - Specifically designed to separate over 100 compounds
 - Optimized analysis time under 12 minutes
 - Available in 0.18, 0.25, 0.32, 0.45, & 0.53mm ID
 - GC/MS Recommend 30m x 0.25mm x 1.4df

Analysis for Volatiles

GC Columns – Rtx-VMS

- Available in 0.18, 0.25, 0.32, 0.45, & 0.53mm ID
 - GC/MS *30m x 0.25mm x 1.4df*
 - Selectivity not column length key factor
 - 60m columns have 2x bleed (2x cost to customer)
 - 0.25mm ID better with water/methanol vs. 0.18mm
 - GC *75m x 0.45mm x 2.55df*
60m x 0.32mm x 1.80df
 - Good capacity & excellent sensitivity

Analysis for Volatiles

Other GC Columns for Volatiles

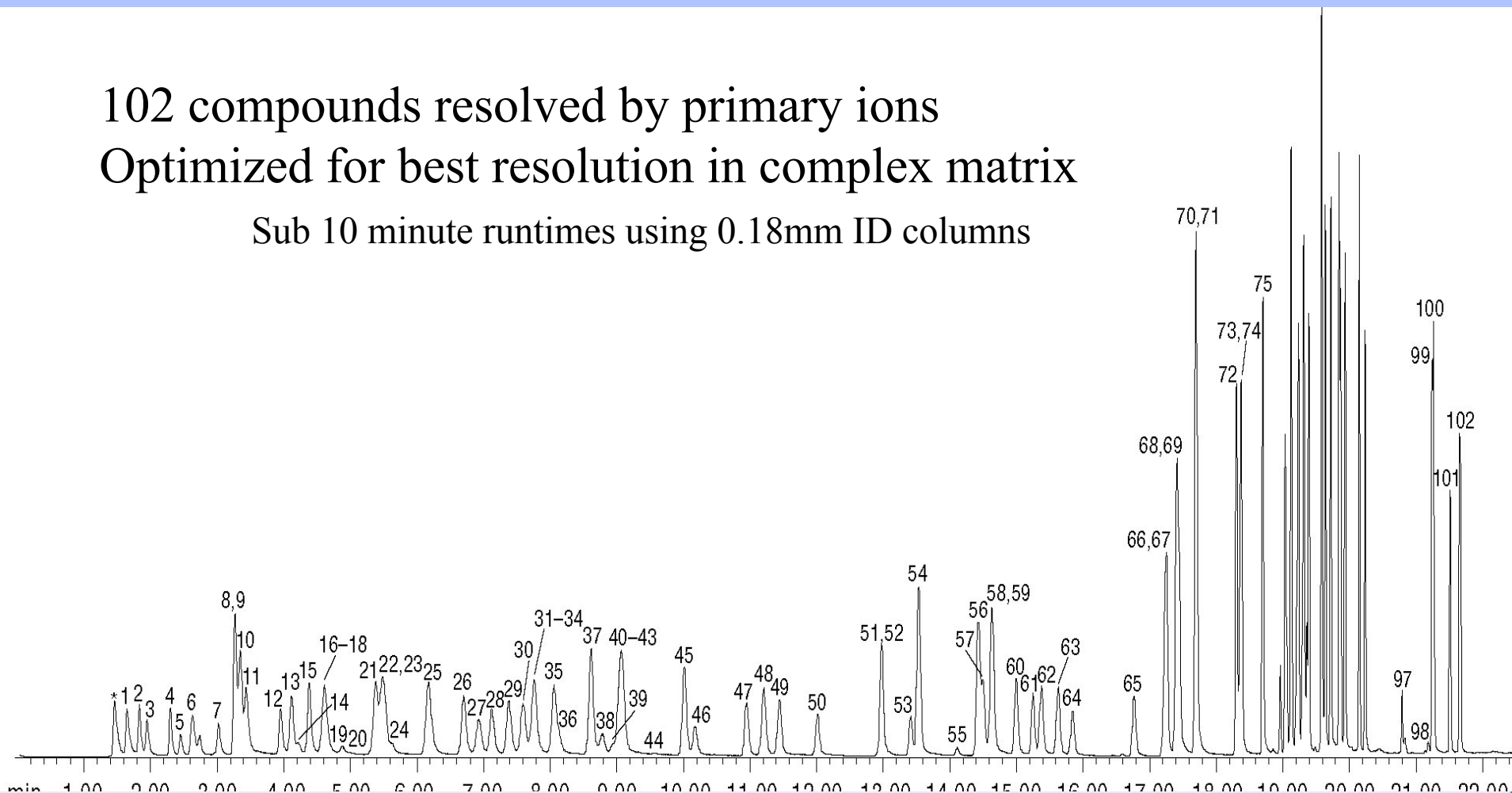
- Competitive Columns
 - DB-VRX, DB-624, DB-502.2, DB-VRX (Agilent/J&W)
 - HP-VOC
 - ZB-624 (Phenomenex)
 - SPB-624 & Vocol (Supelco)
 - VF-624ms & CP Select 624 CB (Varian)
- Other Restek Volatiles Columns
 - Rxi-624Sil MS
 - Rtx-502.2
 - Rtx-VRX
 - Rtx-Volatiles

Rtx[®]-VMS 30m x 0.25mm x 1.4df

102 compounds resolved by primary ions

Optimized for best resolution in complex matrix

Sub 10 minute runtimes using 0.18mm ID columns

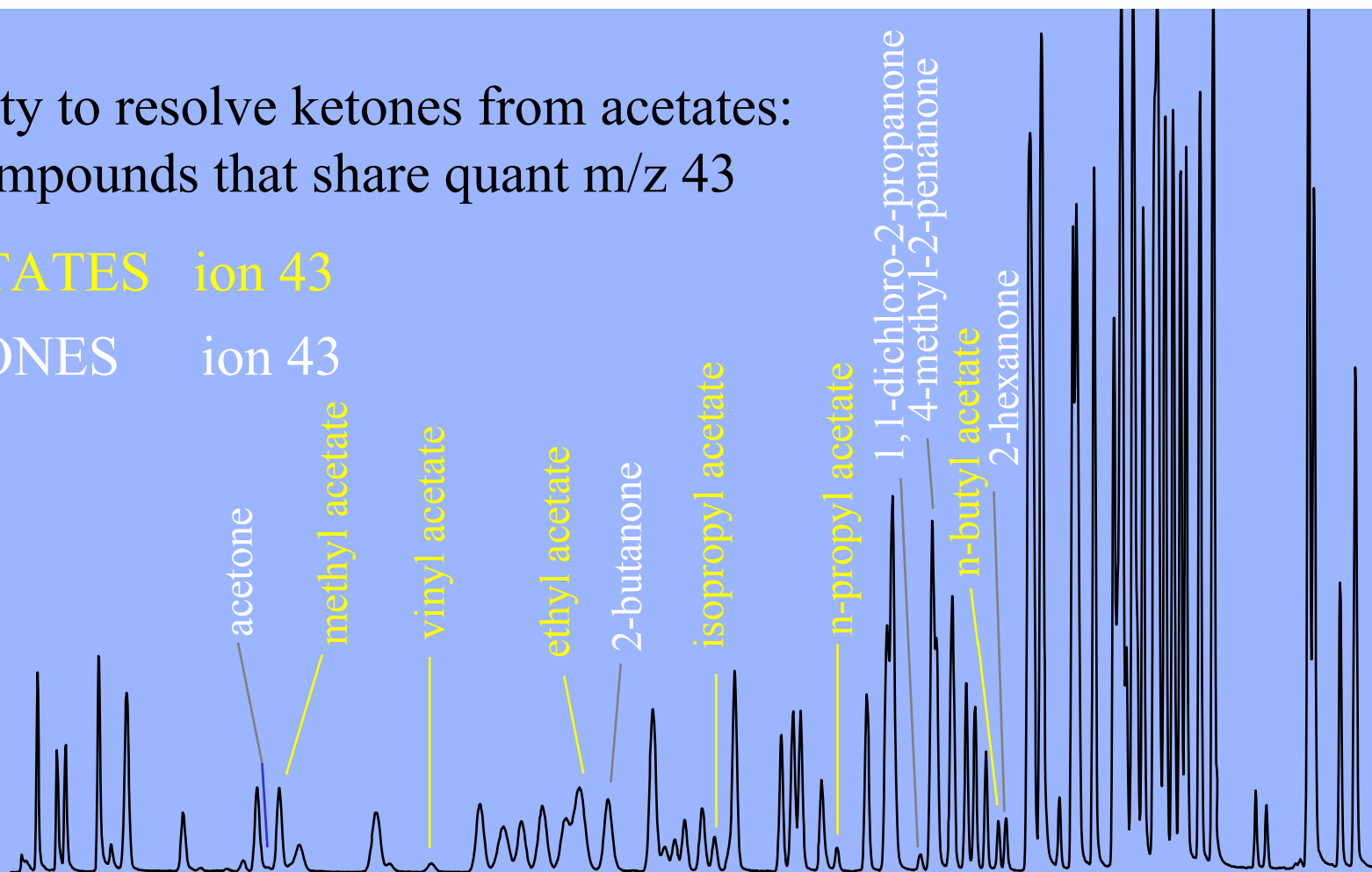


Rtx[®]-VMS

Ability to resolve ketones from acetates:
compounds that share quant m/z 43

ACETATES ion 43

KETONES ion 43



Rtx-VMS advantages over other phases

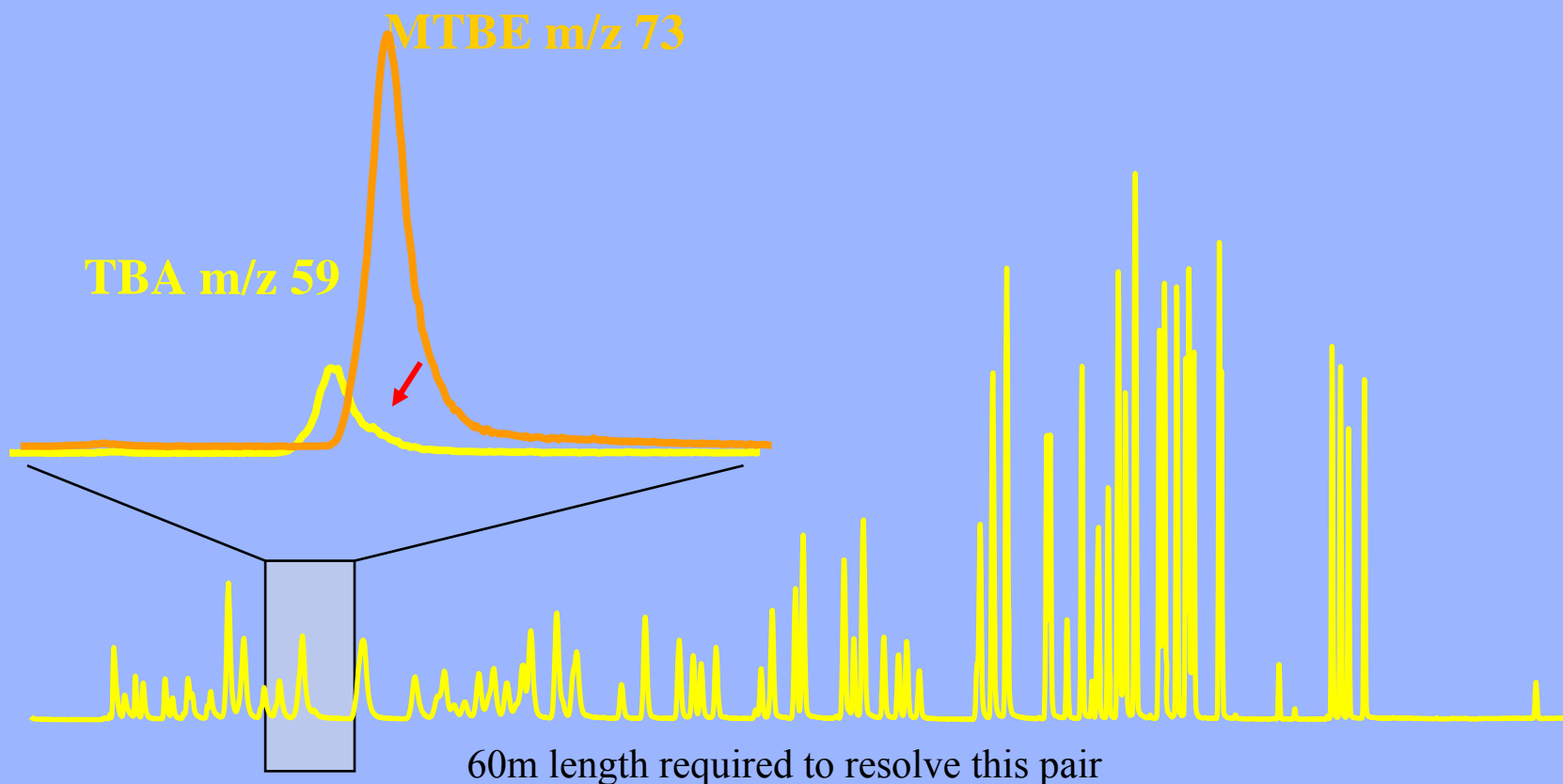
- “624 type phases”
 - Resolution of 2/4-chlorotoluene
 - TBA & MTBE resolution (m/z 59)
 - J&W type: Benzene & 12DCA (m/z 62)
 - GC: C12DCE & 22DCPropane
 - **Rxi-624Sil MS designed not to have these disadvantages**
- “phenyl type phases”

Includes HP-VOC, Rtx-Volatiles, Rtx-502.2, VOCOL

 - Poor resolution of bromomethane & chloroethane
 - Water/Methanol: results in poor gas peak shape
 - Plus: Generally lower bleed & withstand small leaks
- “VRX type phases”
 - Chlorobenzene-D5 (IS) / 1112TCA resolution
 - Bleed similar to 624, VMS
 - GC: Poor resolution of the trihalomethanes

624 Phase Disadvantage

MTBE's minor m/z 59 ion contributes to area of TBA

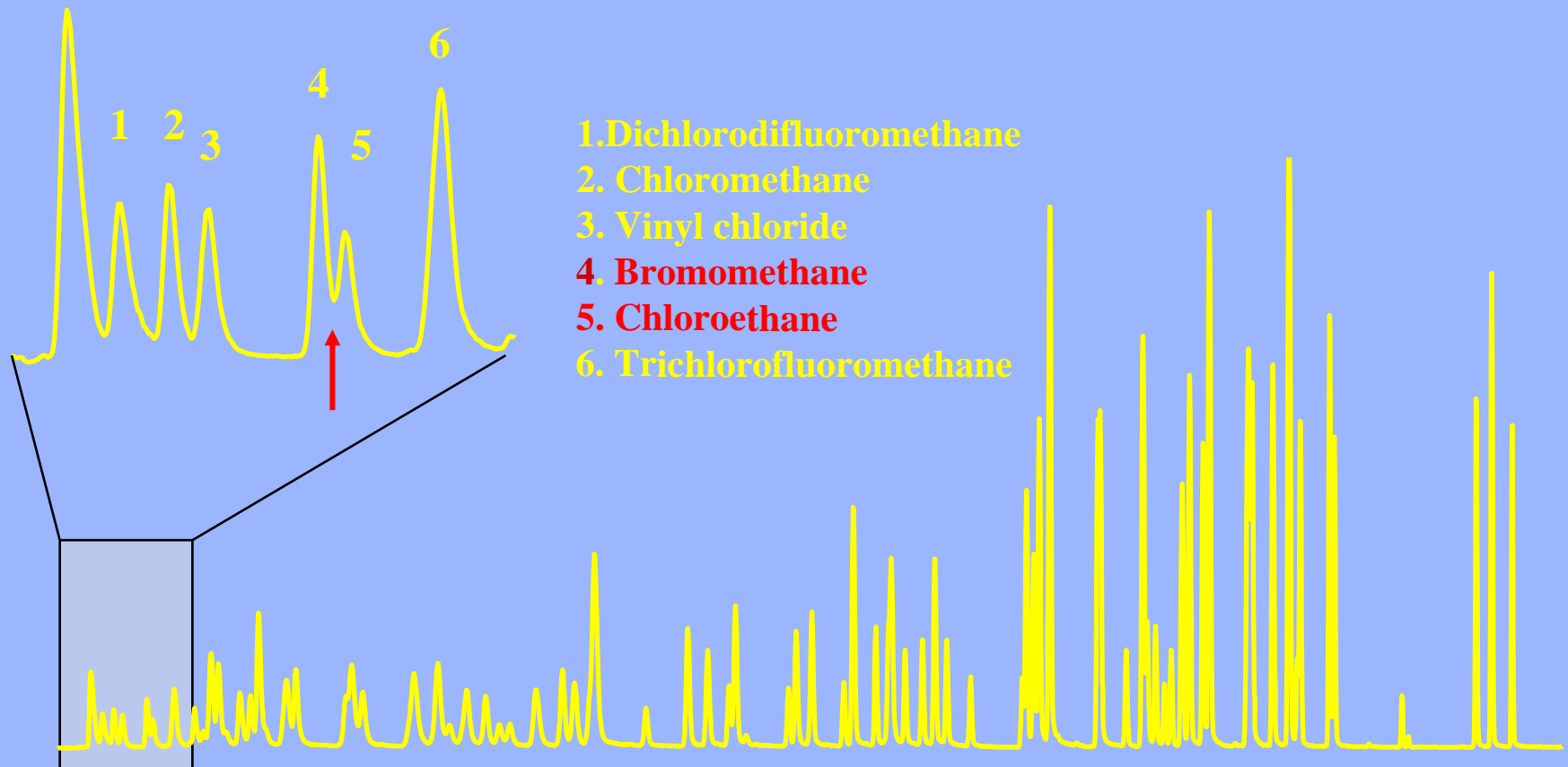


Rtx-Volatiles (phenyl type phase)

All phenyl type phases have this disadvantage

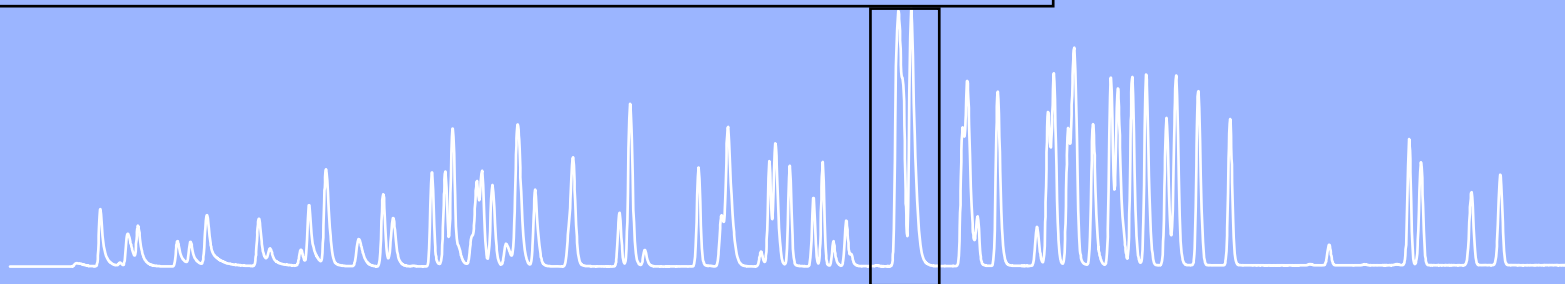
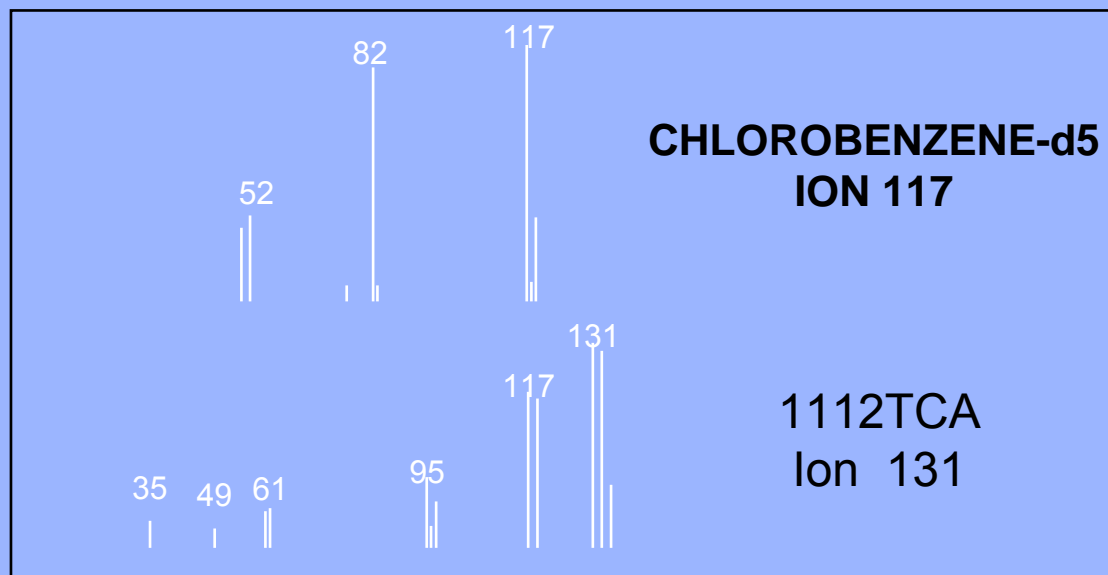
VOA Methods Require 90% Resolution of the Gases

:



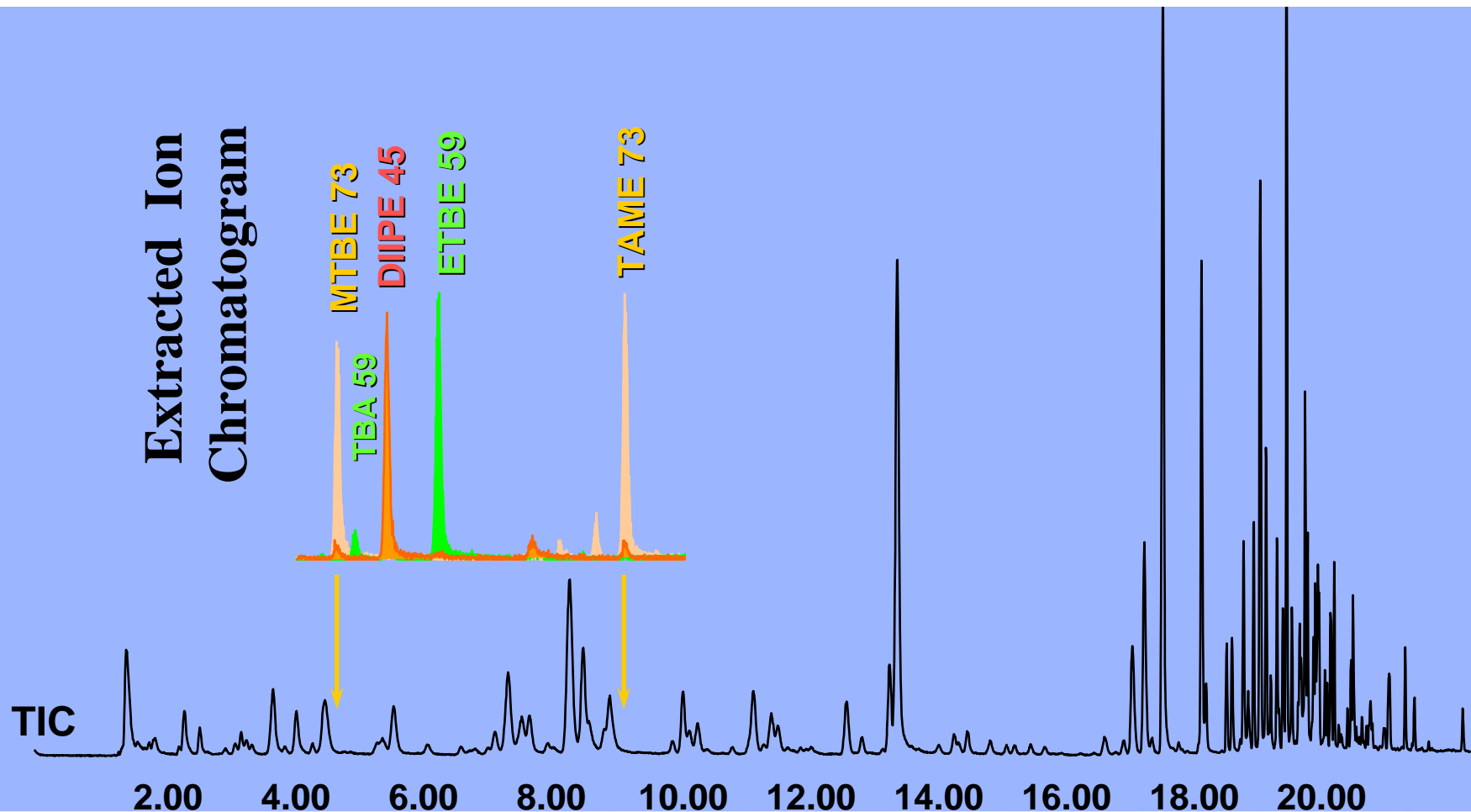
VRX Phase Disadvantage

Coelution of Internal Standard (IS) and Target Compound,
where primary ion for IS is m/z 117



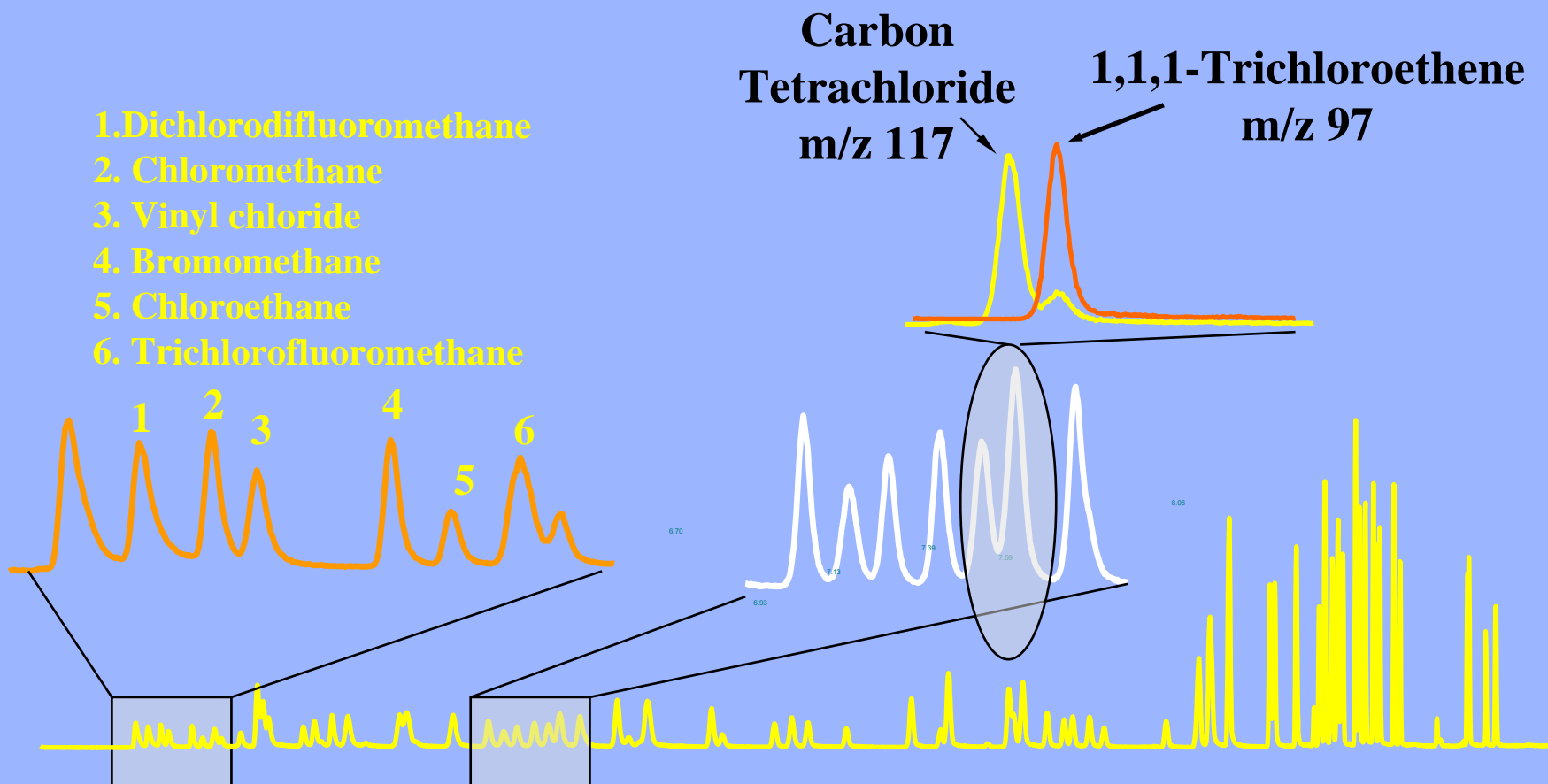
Customers may be able to use m/z 82 ion

Rtx-VMS Quantify Target Compounds in a High Concentration Gasoline Matrix: 1ppm Non-Oxygenated Gasoline Standard Spiked with 5ppb Oxygenates



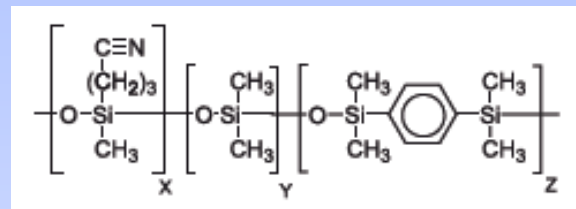
Rtx-VMS Critical Resolutions

1. Dichlorodifluoromethane
2. Chloromethane
3. Vinyl chloride
4. Bromomethane
5. Chloroethane
6. Trichlorofluoromethane

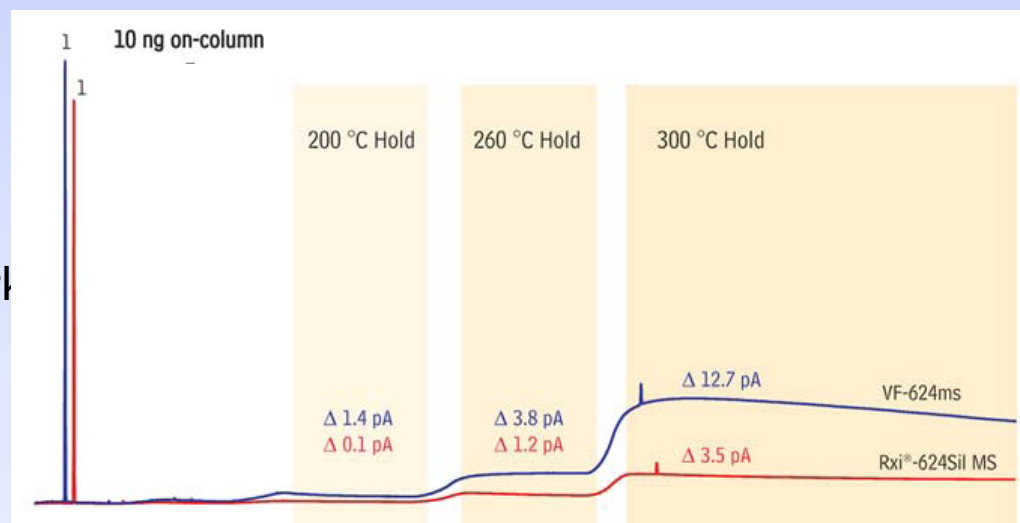


Volatiles Analysis with **NEW** Rxi-624Sil MS

- New Rxi-624Sil MS
 - Silarylene in backbone of polymer
 - Selectivity similar to DB-625, VF-624ms, ZB-624

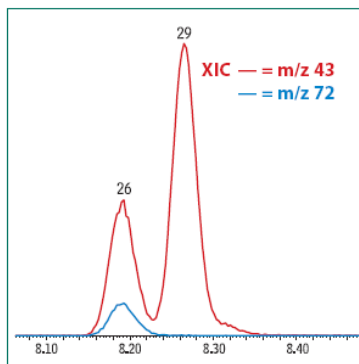


- What is different?
 - Max. temp of 320C
 - Lowest bleed column on market



Volatile Analysis – Rxi-624Sil MS

- 30m x 0.25mm, 1.4 μ m
- Resolves 2-butanol & ethyl acetate
- Less than 17 minute analysis time
- Complete Pro EzGC library for modeling

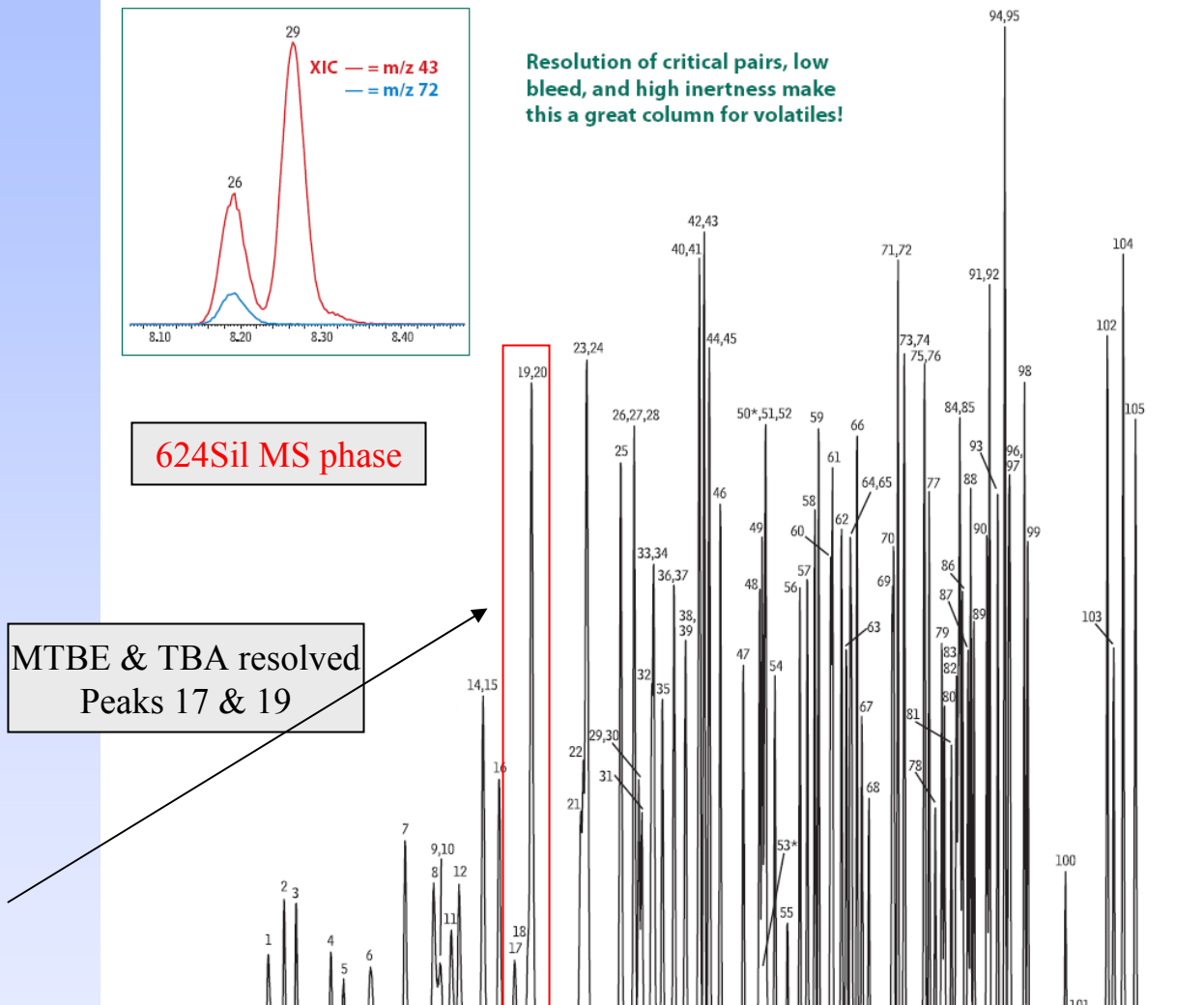
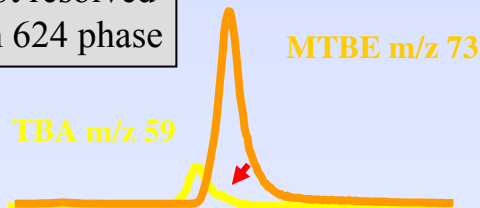


Resolution of critical pairs, low bleed, and high inertness make this a great column for volatiles!

624Sil MS phase

MTBE & TBA resolved
Peaks 17 & 19

Not resolved
on 624 phase



Standards for Volatiles

- Mixtures available for all volatile meth
 - Mega Mixes (502.2, 624, & 8260 compound list)
 - Less expensive, fewer ampoule alternative
 - Reduces cost and preparation time
 - Designed for maximum stability
 - Individual Components
 - Excellent Selection of Oxygenates & alcohol standards
 - Surrogate & Internal Standards
 - QA Documentation Available on all stock products
 - On-line data packs
- Custom Mixtures Available
 - Designed for customer specific requirements
 - Hybrid quotes (Mega Mix plus lab specific compounds)



Sample Preparation for Volatiles

- Vials

- Used for making dilutions
- Soil analysis



- Headspace vials

- Syringes

- Used for making dilution
- Spiking standards



5mL
10mL
25mL
50mL
100mL

*Order needles separately.

- Standards

- Surrogates
- Matrix spikes



Other Products for Volatiles

- **Purge & Trap Supplies**

- Spargers for Tekmar systems
- Silcosteel Transfer Lines
- Moisture Control Bypass Lines for Tekmar



- **PID Lamps**



- **ELCD Nickel reaction tubes & other “things”**



General Chromatography Supplies

- Ferrules



- Ferrule removal kit



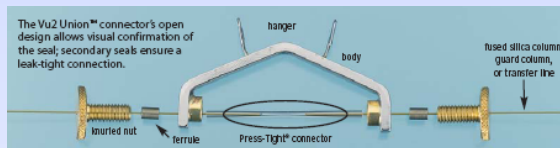
- Gas generators



Leak detector



Super clean gas filters



Column connectors



Scoring wafers

Products for the Volatiles Lab

Best available column:

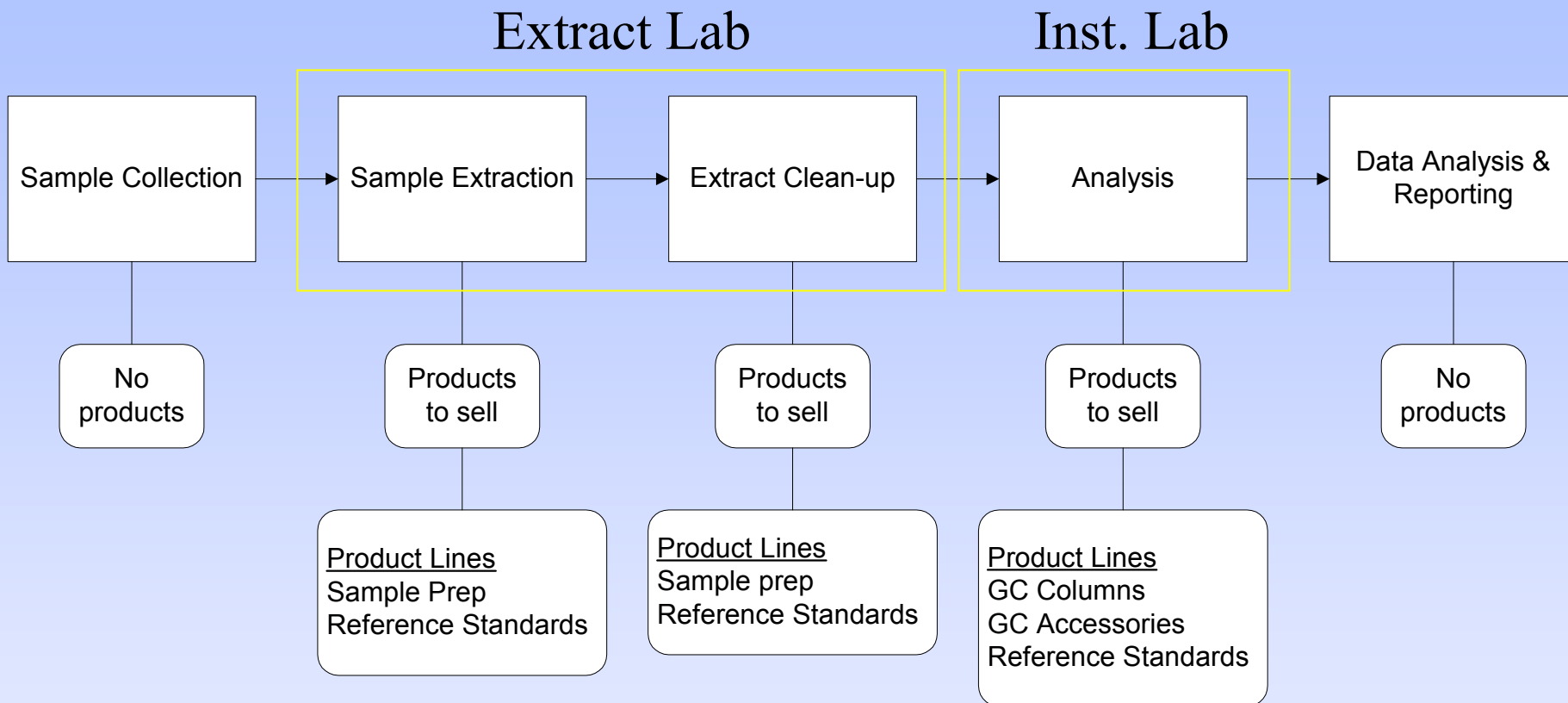
- Rtx-VMS columns (best overall separation, fast analysis time)
- Now have direct replacement for DB-624, ZB-624 or ZB-624ms with the Rxi-624Sil MS
- Mega Mixes (fewest ampoules and lowest overall cost for standards)
- other products:
 - Purge & Trap Supplies
 - VOA Vials, etc.

Pesticide Analysis

Chlorinated Pesticide Overview

- Method Summary:
 - Pesticides are extracted from sample matrix using an appropriate solvent. The extract is concentrated and cleaned to remove interferences. An aliquot of the extract is typically analyzed by GC with MS detection.
- Products offered:
 - GC Columns (Rxi-5Sil MS)
 - Reference Standards
 - Inlet Supplies (liners, inlet seals, septa)
 - Sample prep products (SPE Tubes, Adsorbents, ASE Parts)
 - Autosampler Vials & Syringes

Sample Process for Pesticides

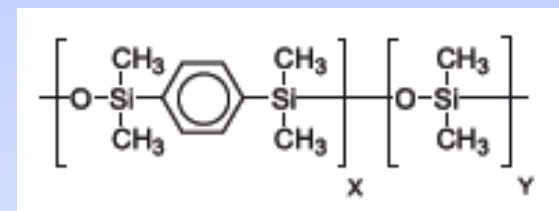


Rxi-5SiI MS

GC Column Technology



- Highest inertness
- Lowest bleed
- Exceptional reproducibility



Rxi-5SiI MS

- Combination of features are what is important for environmental analysis of pesticides

Pesticide Analysis by GC/MS

Figure 1. Total Ion Chromatogram of Pesticides @ 1ppm using Rxi-5sil ms

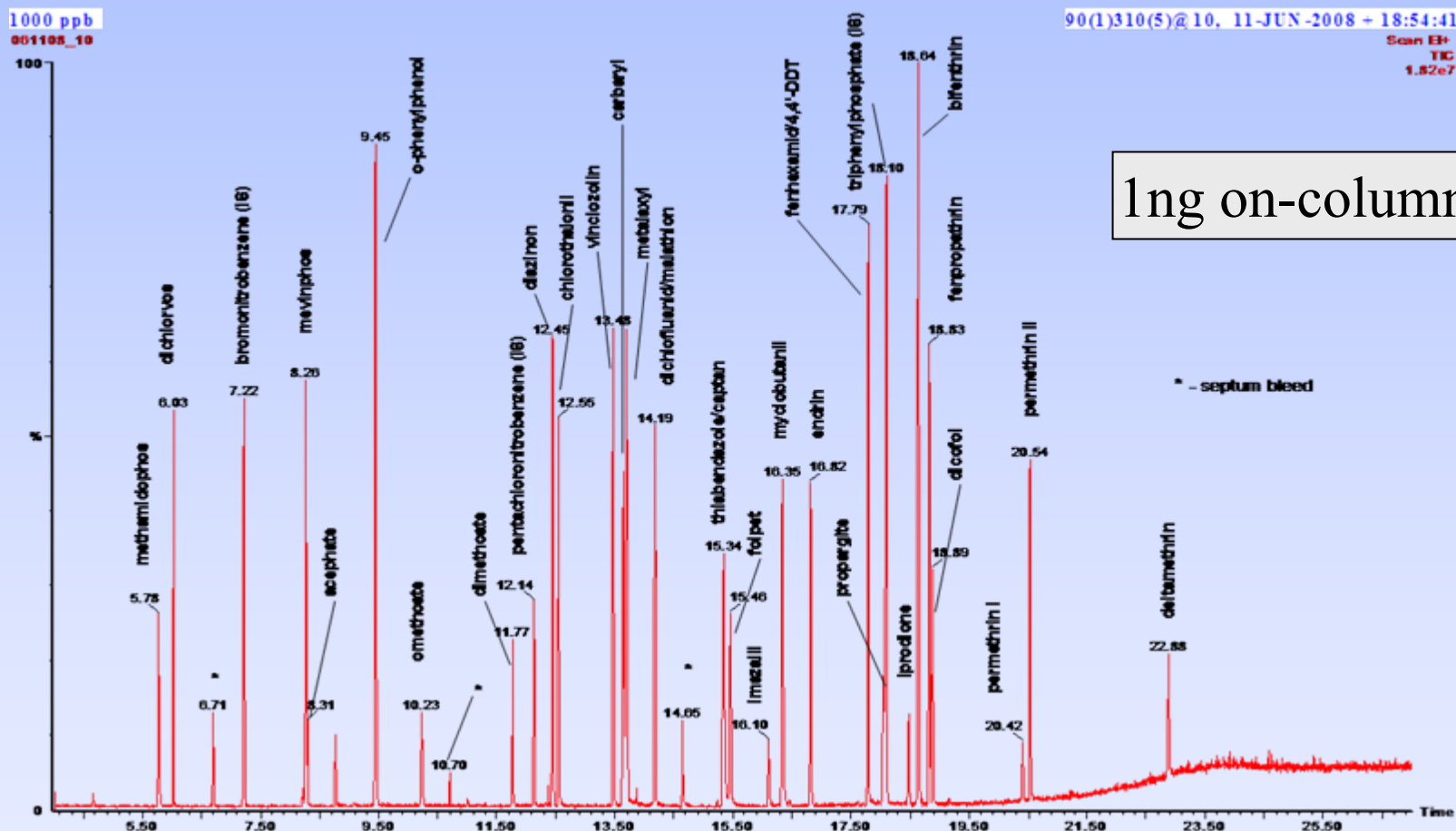


Table 1. MS Acquisition Parameters Rxi-5sil ms

Column: 30m x 0.25mm x 0.25µm (cat.# 13623) serial # 880676

Instrument: Perkin Elmer Clarus 500 GC/MS

Conditions: Oven program: 90°C (1 min.) → 310°C (5 min.) @ 10°C/min.

Inj: 250°C 0.5µL; 1.5 ml/min. constant flow - He; Splitless injection with Uniliner

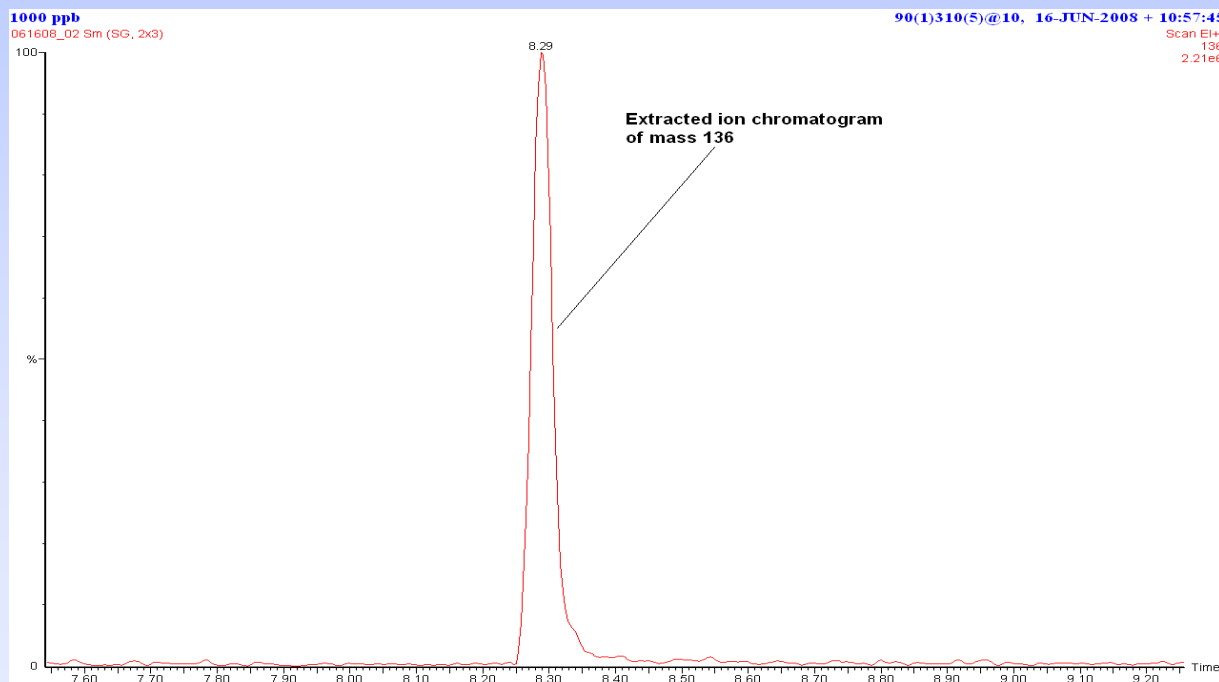
Det: Interface: 300°C; Source: 280°C; SIR mode

Compound	rt	Quantr	Qual 1	Qual 2	CAS#	MW	func#	start	end	IS	r ² (0.01 - 1 ppm)
methamidophos	5.77	141	95	94	10265-92-6	141	1	5.67	5.87	BNB	0.997
dichlorvos	6.02	185	79	109	62-73-7	220	2	5.92	6.12	BNB	0.998
bromonitrobenzene (IS)	7.21	203	201	157	577-19-5	201	3	7.11	7.31	IS	—
mevinphos	8.26	192	127	109	7786-34-7	224	4	8.16	8.36	BNB	0.995
acephate	8.30	136	95	94	30560-19-1	183	5	8.20	8.40	BNB	0.982
o-phenylphenol	9.44	170	169	141	90-43-7	170	6	9.34	9.54	BNB	0.997
omethoate	10.23	156	110	109	1113-02-6	213	7	10.13	10.33	BNB	0.976
dimethoate	11.77	125	143	93	60-51-5	229	8	11.67	11.87	BNB	0.981
pentachloronitrobenzene (IS)	12.13	295	249	237	82-68-8	293	9	12.03	12.23	IS	—
diazinon	12.45	179	304	137	333-41-5	304	10	12.35	12.55	PCNB	0.994
chlorothalonil	12.55	266	264	268	1897-45-6	264	11	12.45	12.65	PCNB	0.983
vinclozin	13.48	285	198	212	50471-44-8	285	12	13.38	13.58	PCNB	0.998
carbaryl	13.65	144	116	115	63-25-2	237	13	13.55	13.75	PCNB	0.996
metalaxyl	13.69	206	160	132	57837-19-1	279	14	13.59	13.79	PCNB	0.997
dichlofuanid	14.17	123	167	224	1085-98-9	332	15	14.07	14.27	PCNB	0.954
malathion	14.19	173	125	127	121-75-5	330	16	14.09	14.29	PCNB	0.992
thiabendazole I	15.34	201	202	174	148-79-8	201	17	15.24	15.44	PCNB	0.958
captan	15.34	79	119	149	133-06-2	299	18	15.24	15.44	PCNB	0.987
folpet	15.46	260	130	104	133-07-3	295	19	15.36	15.56	PCNB	0.964
imazalil	16.10	215	175	173	35554-44-0	296	20	16.00	16.20	PCNB	0.982
myclobutanil	16.34	206	179	150	88671-89-0	289	21	16.24	16.44	PCNB	0.973
endrin	16.82	265	279	317	72-20-8	378	22	16.72	16.92	PCNB	0.989
fenhexamid	17.79	177	179	97	126833-17-8	302	23	17.69	17.89	PCNB	0.969
4,4'-DDT	17.79	237	235	165	50-29-3	352	24	17.94	18.14	PCNB	0.998
propargite	18.04	173	150	135	2312-35-8	350	25	17.94	18.14	PCNB	0.999
triphenylphosphate (IS)	18.09	325	215	326	115-86-6	326	26	17.99	18.19	IS	—
iprodione	18.47	314	316	187	36734-19-7	329	27	18.37	18.57	TPP	0.991
bifenthrin	18.64	181	166	165	82657-04-3	423	28	18.54	18.74	TPP	0.998
fenpropathrin	18.82	265	208	181	39515-41-8	349	29	18.72	18.92	TPP	0.985
dicofof	18.89	139	251	253	115-32-2	368	30	18.79	18.99	TPP	0.788
permethrin I	20.41	183	165	163	52645-53-1	390	31	20.31	20.64	TPP	0.998
permethrin II	20.54	183	163	165	52645-53-1	390	31	20.31	20.64	TPP	0.995
deltamethrin	22.87	253	251	181	52918-63-5	505	32	22.77	22.97	TPP	0.995

Acephate @ 1ng on-column

- Very sensitive to active sites in the injection port and analytical column

Figure 3. EIC of acephate @ 1 ppm



Injection Ports

- Activity from heated inlet systems
 - Some pesticide compounds more active than others
 - Endrin & DDT are 2 compounds sensitive to inlet activity
- How to prevent activity
 - Sky Inlet liners
 - Flip Seals
 - Replacement parts

Injection Ports

• Sky Inlet Liners

- Many geometry of liners used
- Recommend single gooseneck w/ wool



- Inert for active compound
- Wool traps nonvolatile materials

Wool

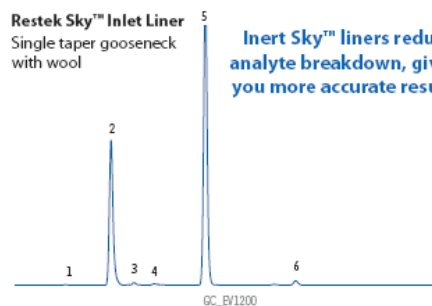
Gooseneck at bottom of liner



Figure 1 Endrin and DDT breakdown is significantly reduced with Sky™ liners, demonstrating higher inertness.

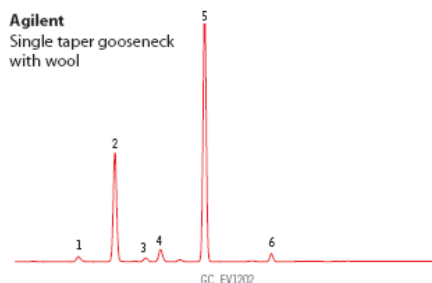
Restek Sky™ Inlet Liner
Single taper gooseneck with wool

Inert Sky™ liners reduce analyte breakdown, giving you more accurate results.



	% Breakdown	
	endrin	DDT
Restek	4.8	1.3
Agilent	16.5	4.2

Agilent
Single taper gooseneck with wool



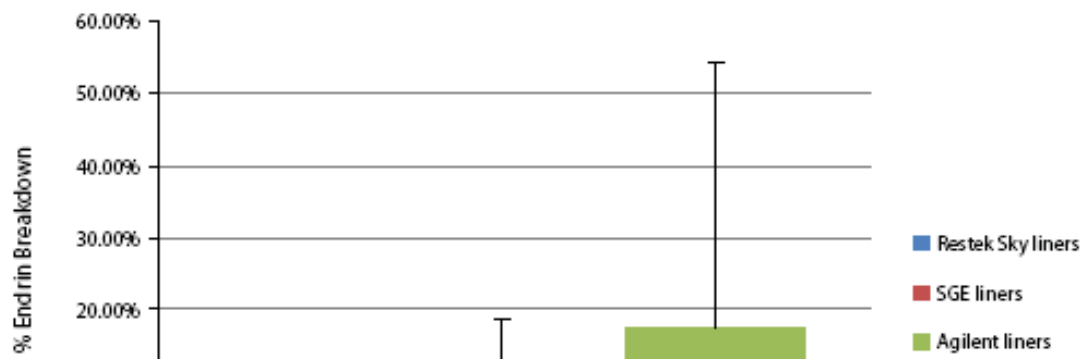
Peaks
1. DDE*
2. Endrin
3. DDD*
4. Endrin aldehyde*
5. DDT
6. Endrin ketone*
*breakdown products

Column Rxi™-5Sil MS, 15 m, 0.25 mm ID, 0.25 µm (cat.# 13620); Sample endrin (50 ng/mL) and DDT (100 ng/mL) in hexane; Injection Inj. Vol.: 1 µL splitless (hold 0.75 min.); Liner: Comparison of Sky™ Single Taper Gooseneck Liner with Wool (cat.# 23303.5) and Agilent Single Taper Gooseneck Liner with Wool (cat.# 5062-3587); Inj. Temp.: 250 °C.

Variation of Inlet Liners

- Due to activity of some of the pesticide compounds variation from liner to liner is important
- The Sky Inlet Liner deactivation has the least variation of liners in the market

Figure 4 Sky™ inlet liners from Restek consistently show less endrin breakdown than comparable liners from other sources.



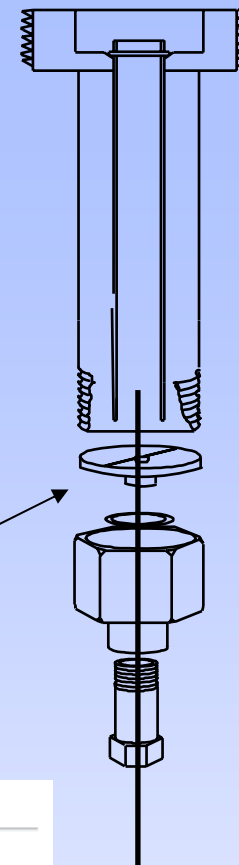
Injection Port

- Inlet seals

- Recommend Gold Flip Seal
- Double use
- Gold plating is inert for active compounds
- Note: Do need adaptor, 1 time purchase



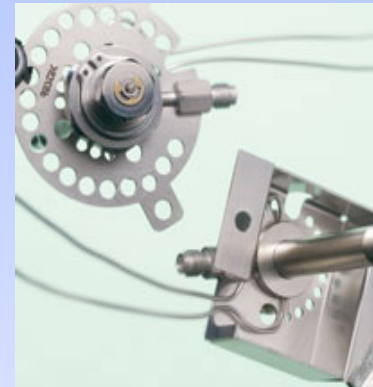
Inlet seal



Feature	Benefit
Reversible design.	Allows twice as many uses as other seals, at the same cost.
Vespel® ring embedded in bottom surface.	Eliminates need for a washer.
Vespel® ring embedded in top surface.	Very little torque required to make seal—reduces operator variability.
Lower leak rate compared to OEM metal inlet seals.	Less detector noise.
Prevents oxygen from permeating the carrier gas.	Increases column lifetime.
Gold or Siltek Treated seals.	Reduces breakdown and adsorption of compounds, maximizing component transfer to GC column.

Injection Port

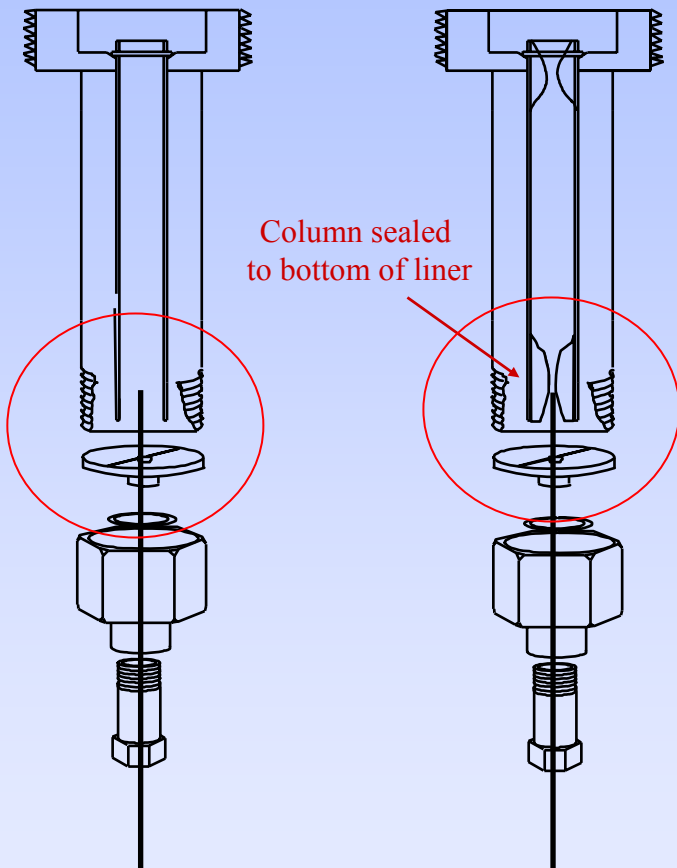
- Replacement Injection Ports
 - EZ Twist Top
 - Not available in Germany, Japan
- Other instrument replacement parts offered
 - Support Agilent, Shimadzu, Perkin Elmer, Thermo



For Most Inert Inlet System

Gooseneck Liners

Drilled Uniliner



Affect of Drilled Uniliner

- Very inert sample pathway
- “Funneling” all sample to column
- Most effective inlet system
- No inlet seal exposure

DI Liners for Agilent GCs
(for 0.25/0.32/0.53mm ID Columns)



Drilled Uniliner (hole near bottom)



Drilled Uniliner (hole near bottom) w/Wool

Consumable Products for Pesticides

- Autosampler Vials

- Screw Cap
- Crimp Top
- Certified Vials – New



- Autosampler Syringes

- SGE
- Hamilton



Sample Extraction for Pesticides

- Extraction Disks
 - 47mm C18, C8, and DVB
 - Drinking water methods
- ASE Replacement Parts
 - Models 200 & 300
 - Cells, PEEK Washers, Filters, tools
 - Used with Dionex automated solvent extractor
 - Soil extraction
- Surrogate & matrix spikes
 - Added to samples prior to extraction
 - Higher concentration & larger package size for lower spike per sample cost
 - QA documentation available
 - On-line data pack



SPE Products for Specific Compounds

Description	Applications	Tube Volume, Bed Weight	qty.	cat.#
EPH Fractionation	Extraction of hexane-extractable petroleum hydrocarbons from soil and waste samples. Specially treated to reduce contaminants and increase capacity. Silica packing.	20mL, 5g	20-pk.	26065
EPA Methods 521 & 522	For use in EPA Method 521: Nitrosamines in Drinking Water and EPA Method 522 for 1,4-Dioxane in Drinking Water. Activated charcoal packing.	6mL, 2g	30-pk.	26032
EPA Method 548.1	Extraction of endosulfan from aqueous samples. Weak anion exchange resin (BioRex 5) packing.	6mL	30-pk.	26063
Ultra Quat SPE	For use in HPLC analysis of paraquat/diquat, as an alternative to EPA 549.2. For an HPLC column developed specifically for this application, see page 189.	6mL, 500mg	30-pk.	25499
EPA Method 8321 (AH SPE)	For use in HPLC analysis of phenoxy acid herbicides.	6mL, 500mg	30-pk.	26029
Organo Tin	High-capacity clean-up of butyl and phenyl tin compounds from soil, water, and biota. Mixed bed.	60mL	16-pk.	24049
Drug Prep I	Suitable for acidic, basic, and neutral drugs. Mixed-mode hydrophobic and ion exchange sites provide selective, reproducible extraction of biological samples containing therapeutic or illegal drugs.	3mL, 200mg	50-pk.	26044
		10mL, 200mg	50-pk.	26045
Drug Prep II	Extraction of THCA from biological samples. Copolymeric anion exchange sites provide selective, reproducible extraction of THCA from urine.	3mL, 200mg	50-pk.	26046
		10mL, 200mg	50-pk.	26047
GHB	Extraction of γ -hydroxybutyric acid from biological samples. Provides reproducible extraction of GHB from urine, blood, or tissue without conversion to GBL (γ -butyrolactone).	10mL, 200mg	50-pk.	26208
RDX	Extraction of explosive compounds (similar to EPA Method 8095 and 8330 list) from water samples.	6mL, 500mg	30-pk.	26093

Extract Clean-up for Pesticides

- Typically clean-up is performed

- Florisil & Silica gel tubes
- CarboPrep



- Bulk Adsorbents

- Florisil PR
- Activated Copper
- Sodium Sulfate



Reference Materials for Pesticides

- Mixtures available for all US EPA Methods
 - Pesticide Mixes
 - QA documentation available on all stock products
 - On-line data-pack
 - Individual Pesticides
 - Surrogate & Internal Standards
- Custom Mixtures Available
 - Designed for customer specific compound lists



Strategy for Pesticides

Summary

- **Some Product considerations :** -
 - Rxi-5Sil MS columns
 - 30m x 0.25mm, 0.25um
 - Sky Inlet Liners
 - First suggestion is single gooseneck w/ wool
 - Cyclo double gooseneck
 - Drilled Uniliner for maximum transfer of compounds and inertness
 - Calibration mixes
 - Multi component mixes
 - Surrogate & matrix spikes
 - Custom mixes
 - Flip Seal (Reversible Dual Vespel Inlet Seal)
 - CarboPrep SPE tubes for cleaning extracts
 - Chlorinated and organophosphorus pesticides evaluated
- - Other instrument supplies
 - Sample Prep products
 - Vials & Syringes

General Chromatography Supplies

- Ferrules



- Ferrule removal kit



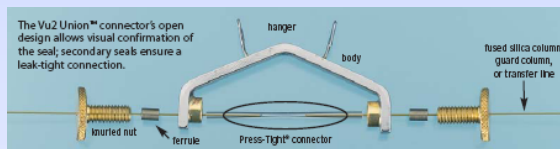
- Gas generators



Leak detector



Super clean gas filters



Column connectors



Scoring wafers

AIR SAMPLING



Australian Distributors
Importers & Manufacturers
www.chromtech.net.au

12/13

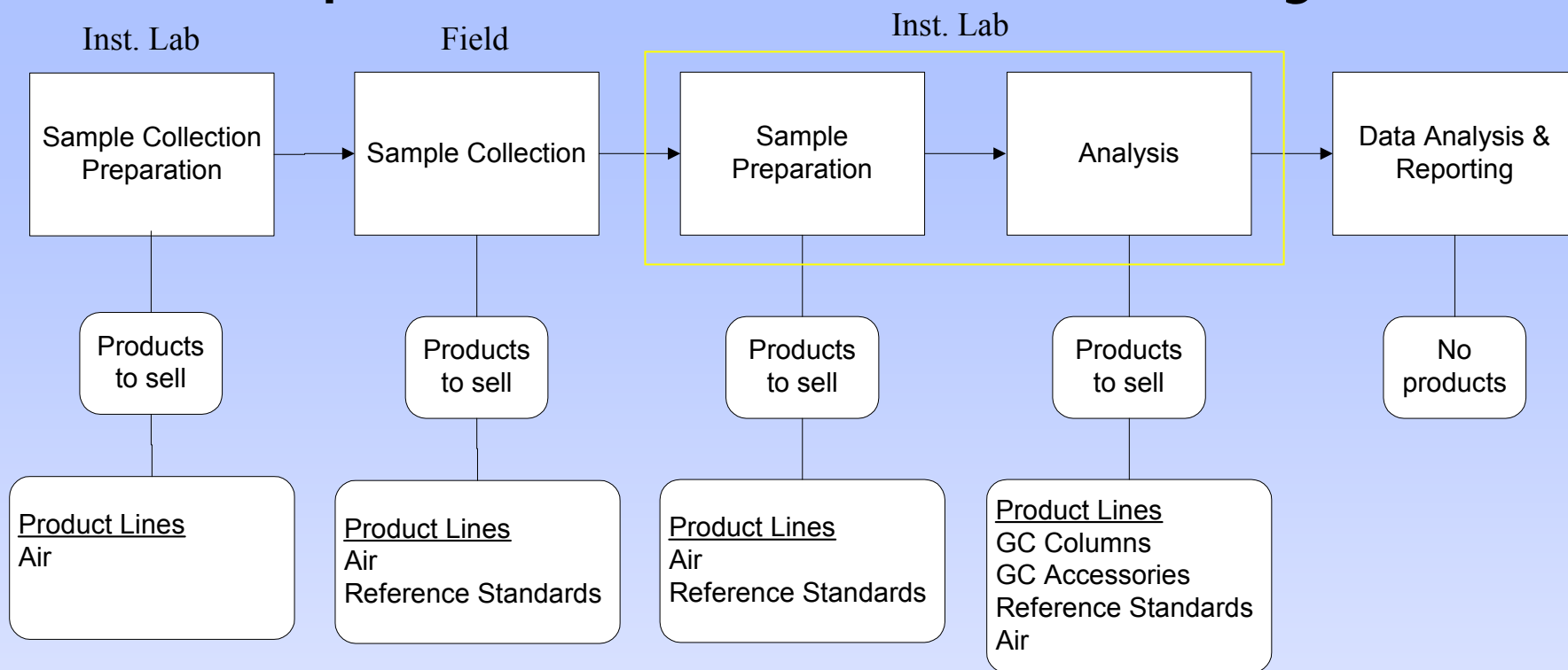
Website NEW : www.chromalytic.com.au E-mail : info@chromtech.net.au Tel: 03 9762 2034 . . . in AUSTRALIA

2011/71

Air Monitoring Overview

- Method Summary:
 - Volatile organic contaminants in air samples are collected in canisters or on thermal desorption tubes. The collected volatile compounds are transferred to a GC or GC/MS system using a sample concentrator or thermal desorption system.
 - Semi-volatile contaminants are collected on adsorbents which are extracted using an appropriate solvent. The solvent is concentrated and an aliquot of the extract is typically analyzed by GC with a selective detector, or by GC/MS.
- Methods: TO-13 (SV), TO-15 & TO-17 (VOCs)
- Products offered:
 - Canisters (Silcocans & TO-Cans)
 - Passive sampling Kits
 - Sample Timer
 - Thermal Desorption Tubes
 - Gas Sampling Bags
 - Gas Standards
 - Semi-volatile sampling products

Sample Process for Air Analysis



Three sampling techniques supported

1. Canisters (Volatiles)
2. Thermal desorption tubes (Volatiles)
3. High volume sampling (semi-volatiles)

Products for Air Labs

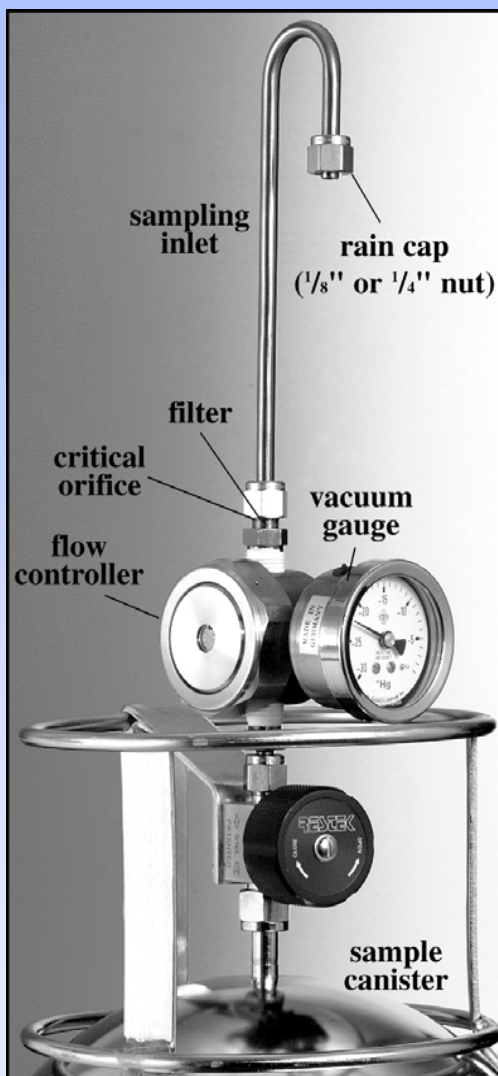
- Canisters
 - Large inventory for fast Delivery
 - Available Siltek coated and electro-polished
 - 3 valve options
- Passive sampling kits
 - Available Siltek coated and stainless steel
 - Replaceable orifices
- Ultra-Clean Resin
- Sampling Bags
- Thermal Desorption Tubes
- Gas Standards

Sample Collection Preparation (Canisters)

- Air canister heating jacket
 - Heats canister and valve
 - Mini Cans can be cleaned the same way
 - Although there are better systems on the market
 - Sampling kit
 - Heat with flow
- Humidification chamber
 - Need to use humidified nitrogen for best cleaning results



Passive Sampling w/Canisters



- No sampling pump required
- Time-integrated sampling using flow controller
- Multiple orifices for broad range of flow rates
 - 0.5 – 350 cc/min

Sample Collection - Canisters



- “Gold Standard” for ambient air sampling of VOCs
 - Whole air sampling
- Variety of sizes
 - 1, 3.2, 6 and 15L
- Grab & time-integrated sampling
 - No sampling pump
- Valve options now available
 - Swagelok SS4H same as Entech
- Large inventory for fast delivery



Sample Collection – Canisters

Timer

- Automated valve open/close for grab and time-integrated sampling
 - 12 timed events
 - Manual operation also available
- Long battery life – months
- Recharge with USB port on PC
- ¼" inlet/outlet compatible with any canister and flow controller



Sample Preparation - Canisters

- Canister comes into lab under vacuum
 - Nitrogen is added to pressurize sample
 - Need to measure vacuum and pressure to determine sample volume
- Ashcroft Test Gauges



Analysis

- GC Columns
 - Rxi-1ms, 60m x 0.32mm, 3um
- Standards
 - TO-14, TO-15, and Ozone Precursor Standards
 - DIN EN ISO 16017
 - 12 month stability
 - Includes Certificate of Analysis
 - PI Marked Cylinders (Scotty)
 - Meets EU Requirements
 - 110L size
 - CGA 180



In nitrogen, 104 liters @ 1,800psi
1ppm cat. # 34400 (ea.)
100ppb cat. # 34421 (ea.)
In nitrogen, 110 liters @ 1,800psi (PI-marked Cylinder)
1ppm cat. # 34400-PI (ea.)
100ppb cat. # 34421-PI (ea.)

cylinder design

Spectra 104L Cylinders:

Aluminum construction.

Size: 8 x 24 cm.

Volume/Pressure:

104 liters of gas

@ 1,800psi

CGA-180

outlet fitting.

Weight:

1.5 lbs./0.7 kg



Sample Collection – Mini Canisters

- 400cc & 1000cc
- Electro-polished & Sulfinert treated
- Both quick-connect fitting and diaphragm valve
- Uses: indoor air, personal, emergency response, soil gas sampling
- Miniature sampling kit components now available
 - Shorter sampling inlet
 - Smaller gauge



Sample Collection Preparation (Thermal Desorption Tubes)

- Alternative technique to canisters
- SS & glass tubes compatible with Perkin Elmer and Markes instruments
- Active (requires sampling pump) and passive sampling modes
- Written in several methods
 - EPA TO-17, NIOSH 2549, ASTM D6196
 - Widely accepted technique in Europe, particularly for personal monitoring (DIN EN ISO 16017)
- Major competitors
 - Perkin Elmer, Supelco, SKC
- Pre-cleaned tubes available
 - Pricey!!! (\$585 vs \$950)
- Do not offer tube cleaning system



Sample Collection – TDU Tubes

- Need pumps, orifice, etc.
 - We do not offer at this time
 - Looking into



Sample Collection Preparation (Gas Sampling Bags)

- ALTEF Gas Sampling Bags

- Replacement for Tedlar bags
- SS & polypropylene septum/valve fitting
- Inert
- Sizes from 0.5 to 100 liters



- Cali-5-Bond™ Bags (New!)

- Opaque for light sensitive compounds
- 5-layer inert construction
- Rugged, reusable
- Hose barb valve with separate septum port
- Commonly used for emission sampling & petroleum industry



Semivolatile Air Sampling (XAD2 & PUFs)

- XAD2
 - Precleaned material
- XAD2 – Not cleaned
 - 1kg quantities
- Polyurathane Foam (PUFs)
 - XAD2 – Soxhlet extractor
 - Tubes – heat with flow
- Do not offer equipment for re-use

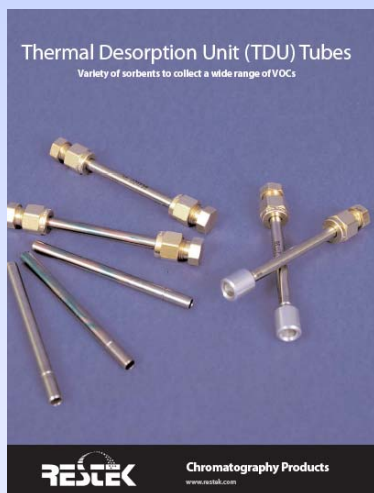
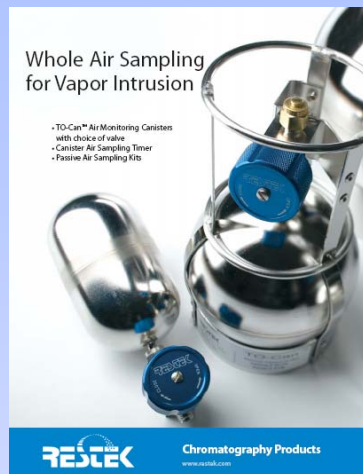


Other Air Sampling Products

- Large volume syringes
- Custom standards
- Carrying boxes/cases
 - Use for shipping



Product Literature



RESTEK

CHROMalytic +61(0)3 9762 2034
ECHnology Pty Ltd
Website NEW : www.chromalytic.com.au E-mail : info@chromtech.net.au Tel: 03 9762 2034 . . . in AUSTRALIA

Australian Distributors
Importers & Manufacturers
www.chromtech.net.au

12/13

2011/87

Competitive Positioning for Selling into the Air Lab

- - Canisters
 - Large inventory for fast Delivery
 - Available Siltek coated and electro-polished
 - 3 valve options
 - Passive sampling kits
 - Available Siltek coated and stainless steel
 - Replaceable orifices
 - Ultra-Clean Resin
- - Thermal Desorption Tubes
 - Sampling Bags
 - Gas Standards
- Diverse product line offering many options for VOC and SVOC sampling