

Low Cost Solution for Accelerating Gas Chromatographic Separations

Gary Stidsen, Stephen J. MacDonald, Frank Dorman and Brad Rightnour

Restek Corporation
www.restekcorp.com



Desires of GC Analysts

- Higher Sample Throughput
 - Lowers cost/sample
 - Increases sample capacity
 - Fewer instruments to accomplish same workload
- Better Resolution
 - Can allow for shorter run times
 - Improves quantitation
 - Can allow for analysis of very complex matrices

Methods to Improve Speed and/or Resolution “high-end” technology

- Fast GC/Flash GC
 - Short, narrow I.d. columns
 - Ballistic heating (resistive, microwave)
- Multicolumn GC
 - Bertsch, Guichon, Giddings
- Comprehensive 2D-GC
 - Begun by John Phillips – Southern Illinois Univ.
- Stop-Flow GC
 - Richard Sacks – Univ. of Michigan

Methods to Improve Speed and/or Resolution “lower-end” technology

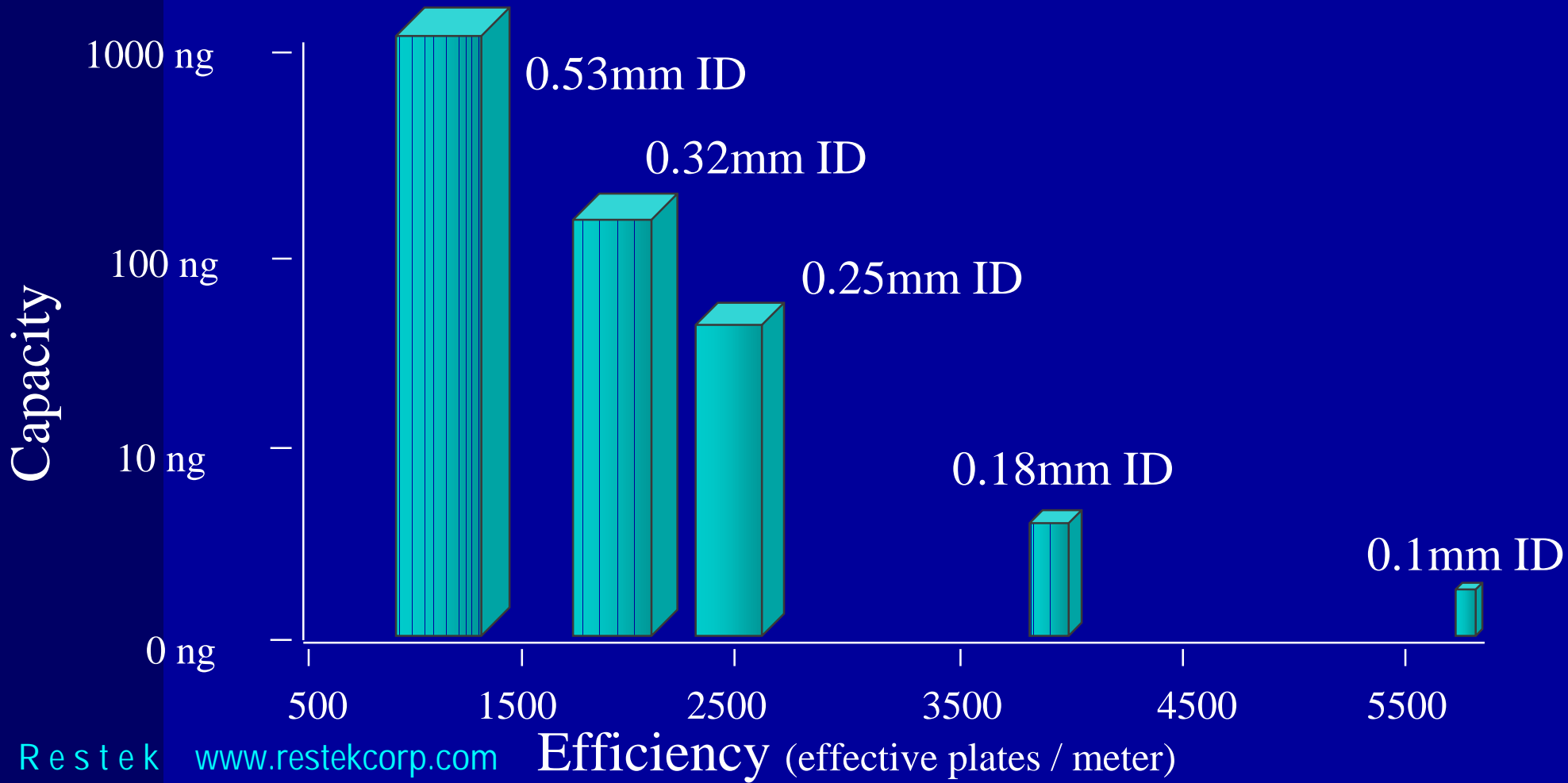
- Tuning Stationary Phase Selectivity
 - Design column to achieve specific separation
 - Users can send retention data for optimization
- Physical Parameter Optimization
 - Pro EZ-GC Software allows user optimization
- Hardware Modification
 - GC Racer allows common existing instrumentation to achieve increased temperature ramp rates

“Fast” GC Techniques

- How fast is fast?
 - 100 C/min, 100 C/sec ?
- How fast is necessary?
 - Partially depends on column dimensions
- Does the technique require different columns?
 - “caged” columns for resistive heating
 - Microwave heated columns
 - Narrow-bore columns

Column Selection

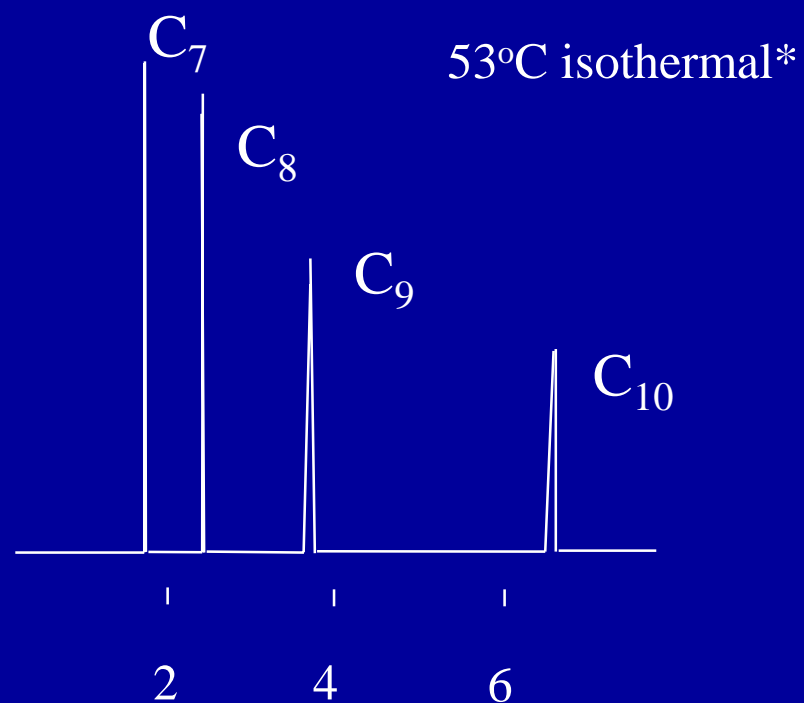
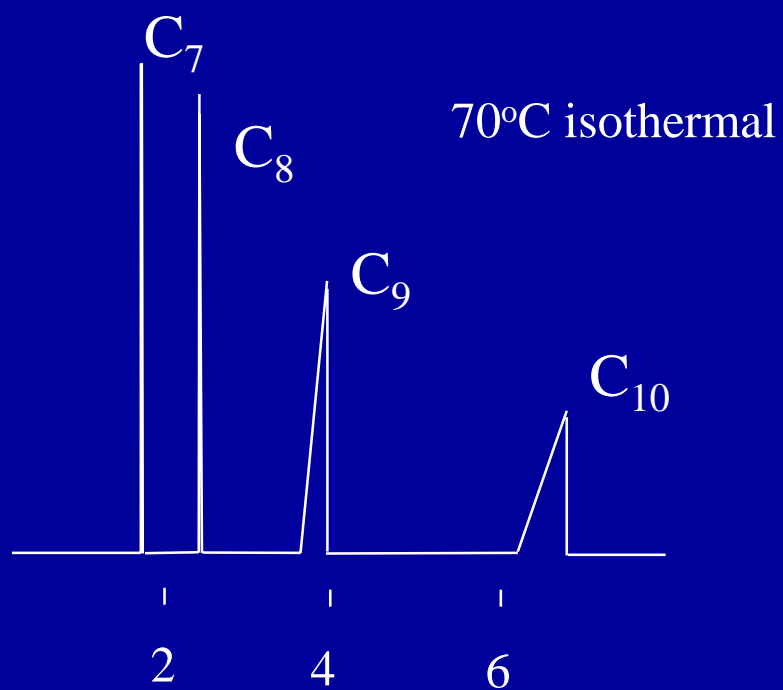
Typical Column Capacity vs. Efficiency



Effect of ID on Capacity

Rtx-1: 15m, 0.25mm ID, 0.25 μ m

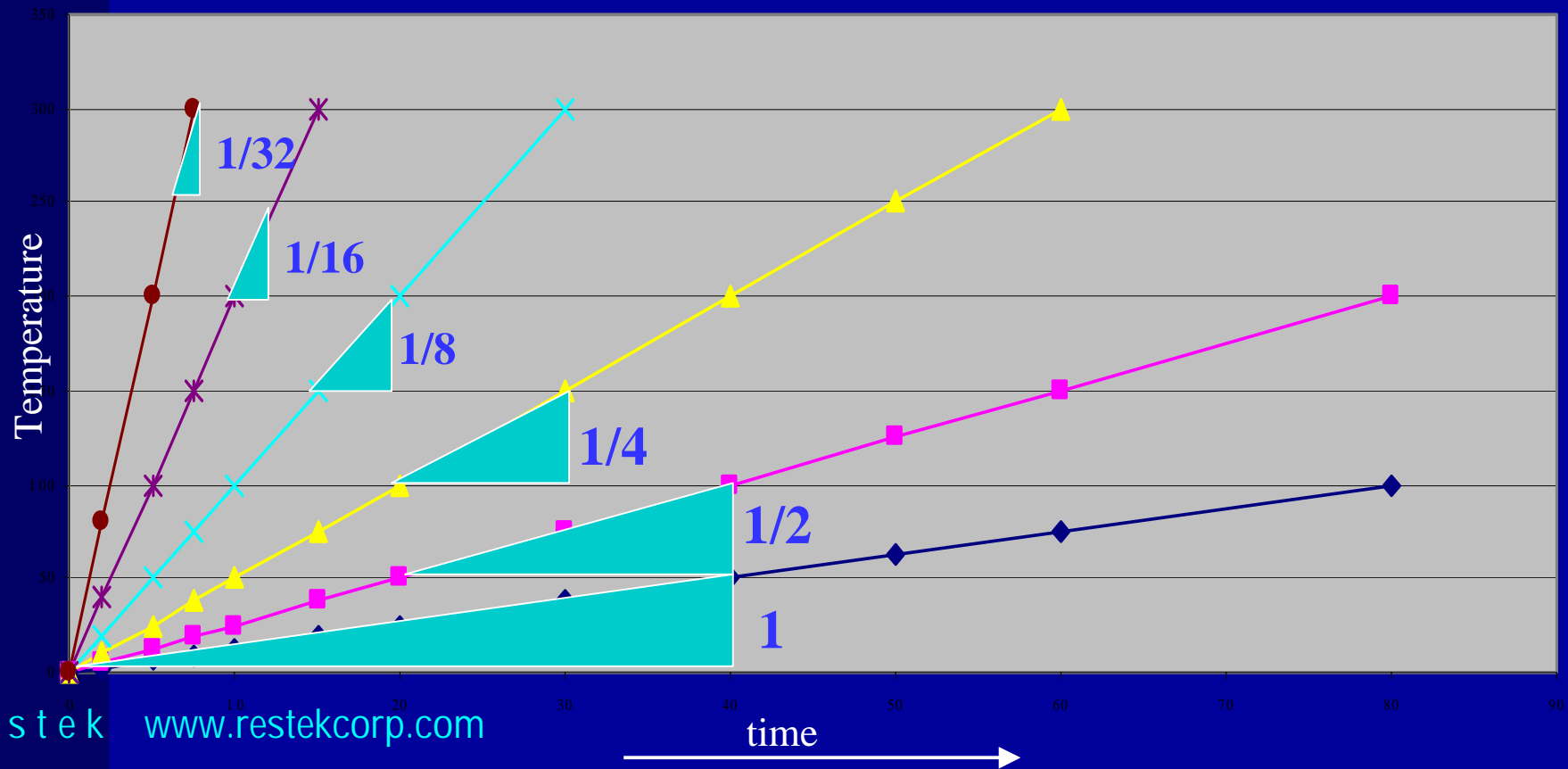
15m, 0.53mm ID, 0.25 μ m



Restek www.restek.com *run at different temperatures to keep k values similar

Effect of Temperature Programming Rate on Number of Theoretical Plates:

Area of the triangle Represents relative number of theoretical plates at different heating rates.



GC Racer

- Interfaces to existing GC's
 - 5890 A model available
 - 5890 Series 2 model available
 - 6890 Series available
- Operates using existing GC control
 - No software or firmware
- Allows for maximum ramp rates up to 440 C
- Can allow for 2-5 times speed enhancement for most methods



GC Racer Heater Installed in an Agilent 5890

Restek www.restekcorp.com



GC Racer Installed on an Agilent 5890

Restek www.restekcorp.com

Versatility of HSGC Techniques

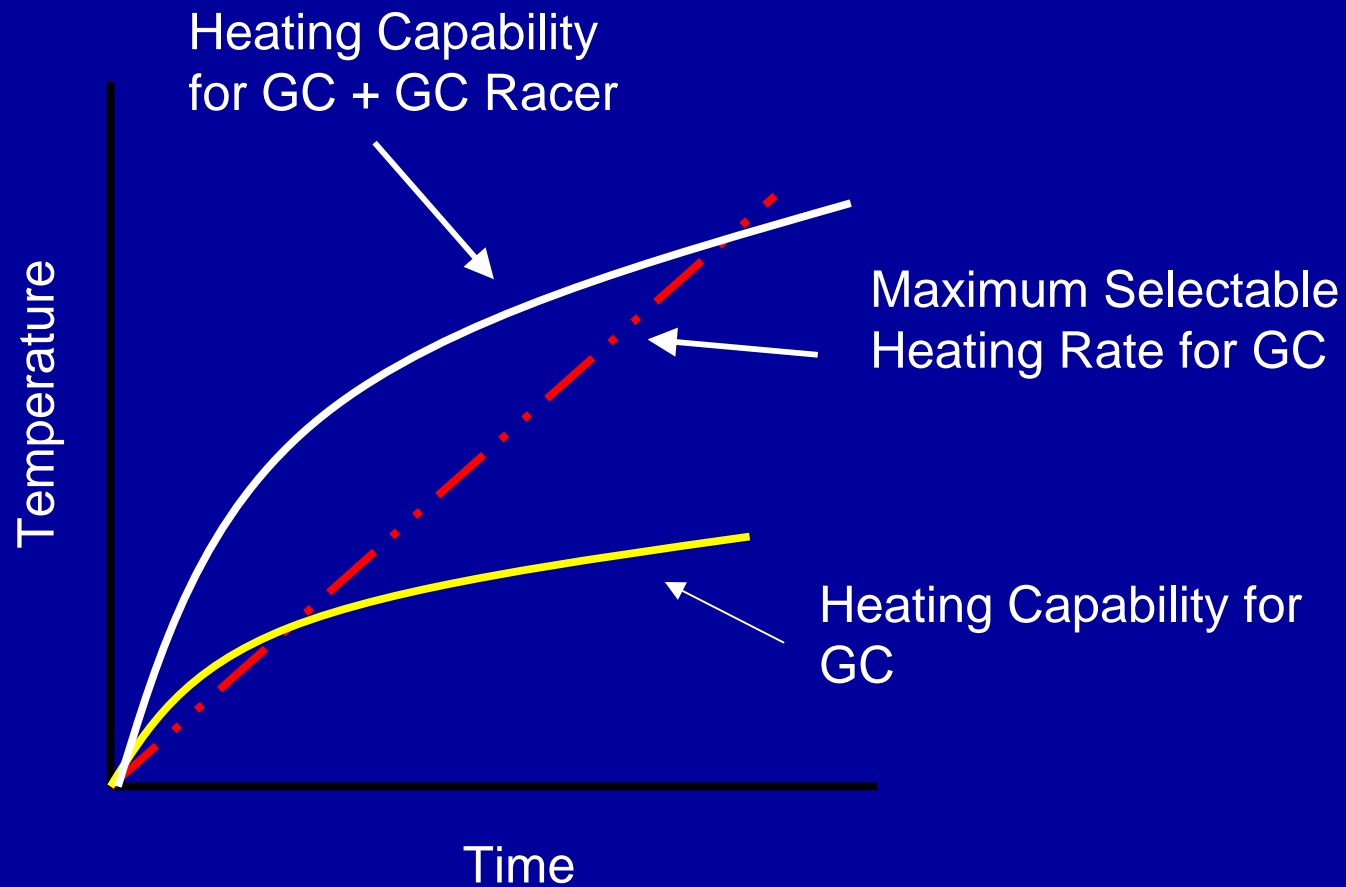
HSGC Technique	GC Racer	Flash	Micro Bore	Others
<u>Versatility Factor</u>				
Injectors	All	Split/splitless	Most	?
Guard Columns	Yes	No	Yes	Yes
Retention Gap	Yes	No	Yes	Yes
Columns	All	5 or 10 m	< 0.1 mm i.d.	Small i.d. ⁽¹⁾
Sample Capacity	Full Range	Med - Low	Low	??
Temp Programming	Yes	Yes	Yes	Yes
Detectors	All	Most	All	?
EPC	Yes	No	High Pressure	All
RT Locking	Yes	No	Yes	Yes
Validation	None	Required	None	Required

Simplicity of HSGC Techniques

HSGC Technique	GC Racer	Flash	Micro Bore	Others
<u>Simplicity Factor</u>				
Installation	User	Professional	Not	Professional ?
Column changing	Plug and Play		Applicable	
Column Purchase	Standard	Specialized	Standard	Standard?
User Training	Any Source	Sole Source	Any Source	?
Software	None	Yes	None	Yes
Bench space	None	Yes	None	Yes
Added Operational Requirements	No	Yes	No	??

Affordability of HSGC Techniques

HSGC Technique	GC Racer	Flash	Micro Bore	Others
<u>Cost Factor</u>				?
Purchase Price	3,800	20,000	400	
Installation Cost	0	3,000	0	
Maintenance Cost	0	??	0	
Training Cost	0	included in installation cost	0	
Number of Columns	GC dependent (1 – 4)	1	1	



GC Racer: Allows use of fast heating rates throughout entire temperature range

Retention time

Versatility of Zip GC Racer

<u>Versatility Factor</u>		
Injectors	All	S/SS, Direct, PTV, SPME, 3 RD Party
Columns	All	Every size, every manufacturer, every length - Fused silica, metal, packed.
Guard Columns	Yes	
Retention Gap	Yes	
Sample Capacity		
Temp Programming		From microbore to packed column capacity HP 5890: 70 °C/min up to 350 °C. HP 6890: 70 °C/min up to 400 °C.
Detectors	Full Range	FID, MSD, FPD, PID, TCD, HID, ... every make, model, manufacturer.
EPC	All	original EPC and third party EPC accessories
RT Locking	Yes	Seamless addition to existing system

Simplicity of Zip GC Racer

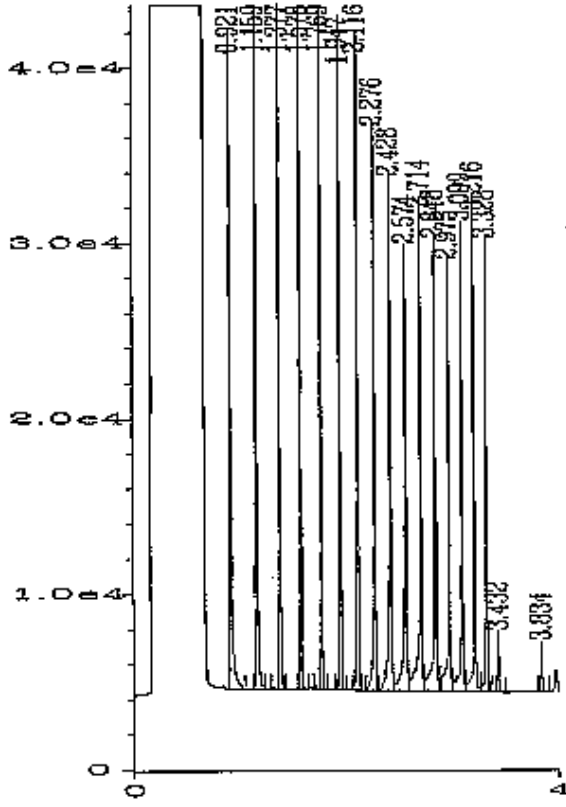
Simplicity Factor

Installation	Simple; 3 plug in connections. Plug and Play
Column changing	Standard method, no special columns, interfaces, or tools.
Column Purchase	User continues to buy from vendor of choice.
User Training	None
Software	None
Bench space	None
Added Operational Requirements	None

Ruggedness of Zip GC Racer

The Zip GC Racer is based on the same heating technology used by GC manufacturers for the past 25+ years. Most GCs go to the junkyard without ever having oven failures. MTBF for GC oven heaters is a long, long, very long time.

Alpha Site	U Mass			
Beta Sites			Woods Hole Environmental Group	Restek Corp
Host GC	HP 5890 Series II		HP 5890	HP 5890
Application	Tenn/Miss DRO		Extractable HC	SimDis PCB's
Comments	Blew a few fuses – nobody got hurt.			
Problems encountered	Typical Design Issues		None	None



=====
 Area Percent Report
 =====

a File Name : C:\HPCHEM\1\DATA\MARCH19\001F0403.D
 Operator : SteveMac Page Number : 1
 Instrument : ANALYZER1 Vial Number : 1
 Sample Name : Tenn/Miss DRO Injection Number : 3
 Run Time Bar Code : Sequence Line : 4
 Acquired on : 21 Oct 00 11:26 AM Instrument Method: TENNDRO.MTH
 Report Created on: 24 Oct 00 06:03 PM Analysis Method : TENNDRO.MTH

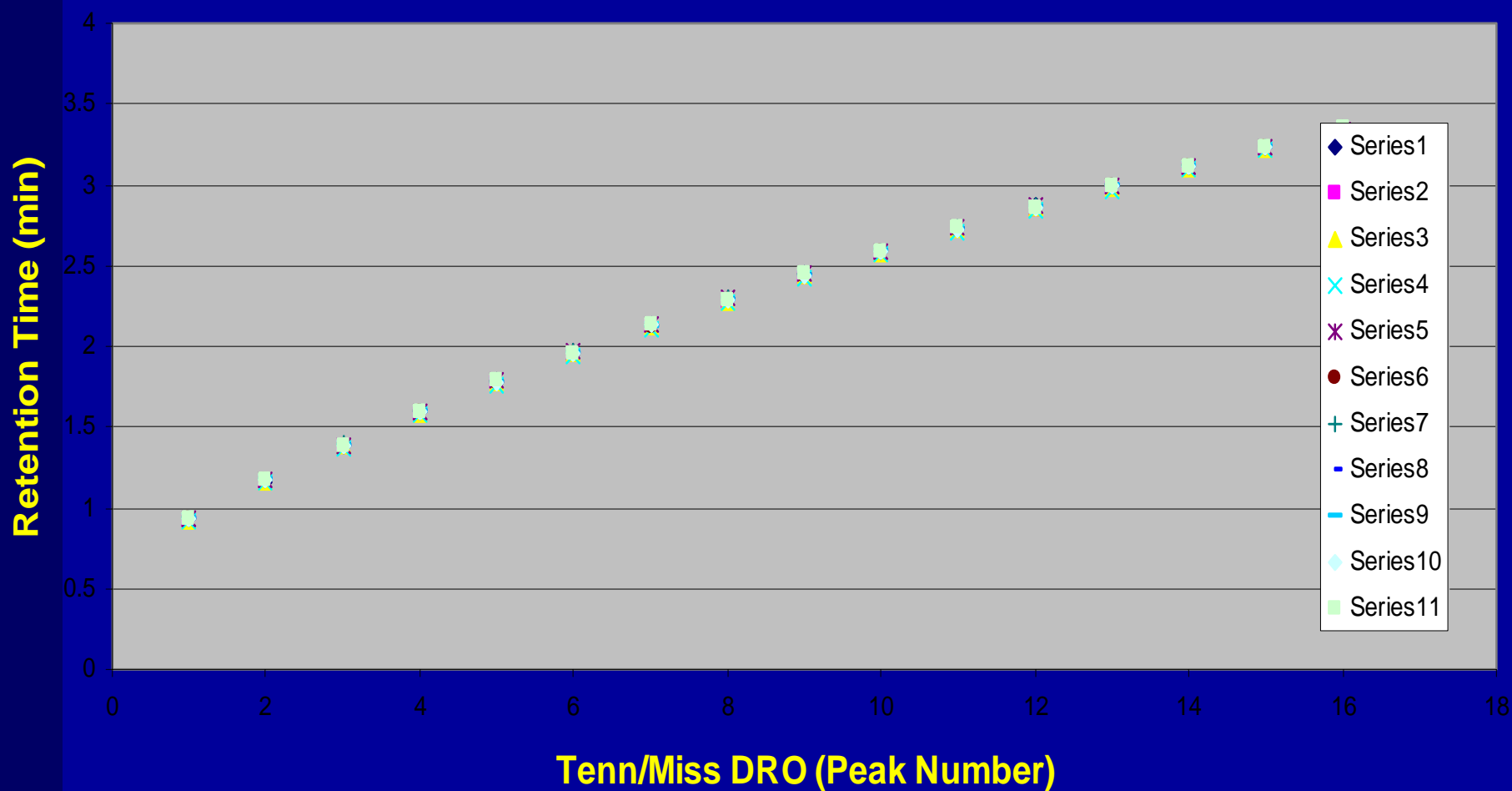
=====
 Sig. 1 in C:\HPCHEM\1\DATA\MARCH19\001F0403.D
 =====

pk#	Ret Time	Area	Height	Type	Width	Area %
1	0.921	34658	37521	BB	0.028	7.1855
2	1.159	35141	41700	BB	0.021	7.2856
3	1.377	34297	47435	BB	0.024	7.1105
4	1.578	30035	43098	BB	0.018	6.2270
5	1.769	32638	36410	BB	0.020	6.7665
6	1.947	31864	39014	BB	0.022	6.6061
7	2.116	34325	39851	BB	0.019	7.1164
8	2.276	30390	30819	BV	0.038	6.3006
9	2.428	33151	28803	VV	0.019	6.8729
10	2.574	27481	25768	VV	0.022	5.6975
11	2.714	30435	28265	VV	0.020	6.3099
12	2.848	27168	25436	VV	0.020	5.6325
13	2.975	24715	24750	VV	0.017	5.1240
14	3.099	23861	26293	VV	0.021	4.9469
15	3.216	25866	26421	VV	0.015	5.3626
16	3.328	21451	25677	VB	0.025	4.4473
17	3.432	2475	3450	BB	0.017	0.5130
18	3.834	2388	2889	BB	0.021	0.4951

Tenn/Miss DRO Mix with GC Racer

Temp Program: 50°C (0.33 min hold), 70°C/min to 300°C, hold 0.1 min

GC Racer RT Stability over 7 Days/900 Runs

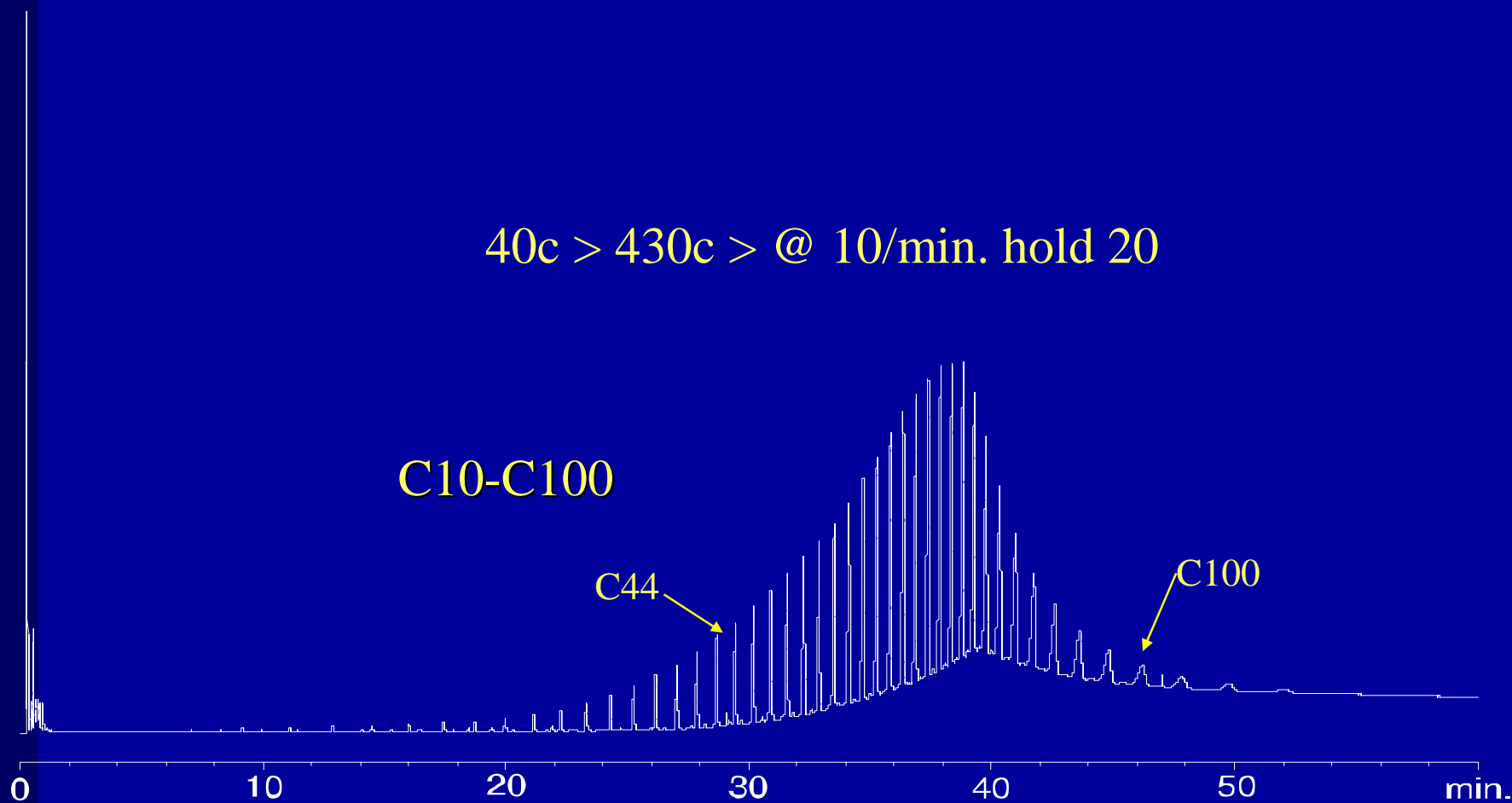


Polywax 1000

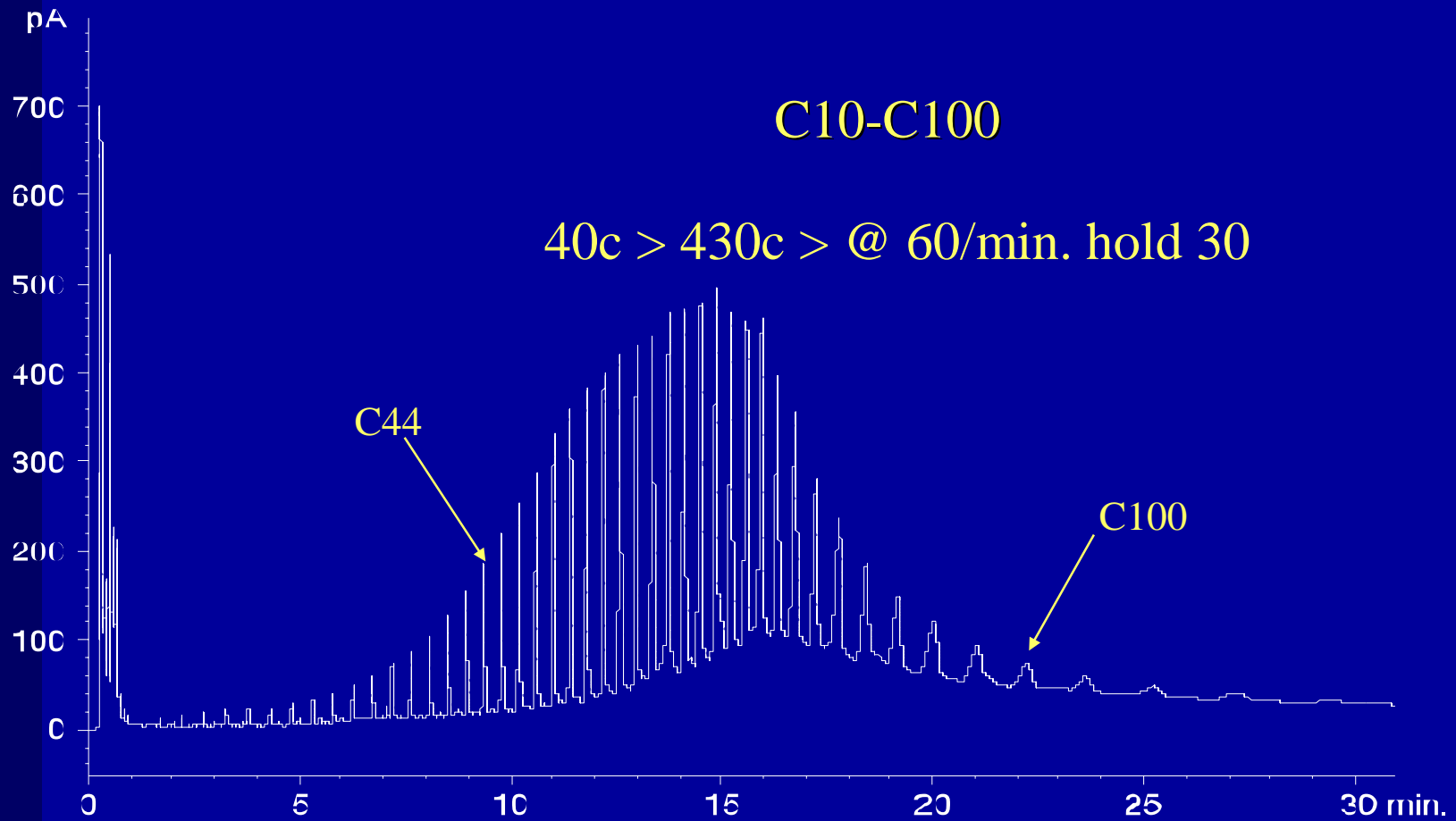
- Polyethylene (even numbers) with average molecular weight of 1000 daltons
- Requires high temperature for elution of homologous series
- Good candidate for faster technique

MXT-1HT Polywax 1000 C₁₀-C₁₀₀

40c > 430c > @ 10/min. hold 20



Polywax 1000 $60^{\circ}\text{C}/\text{minute}$ ramp rate



C10-C100

40c > 430c > @ 60/min. hold 30

C44

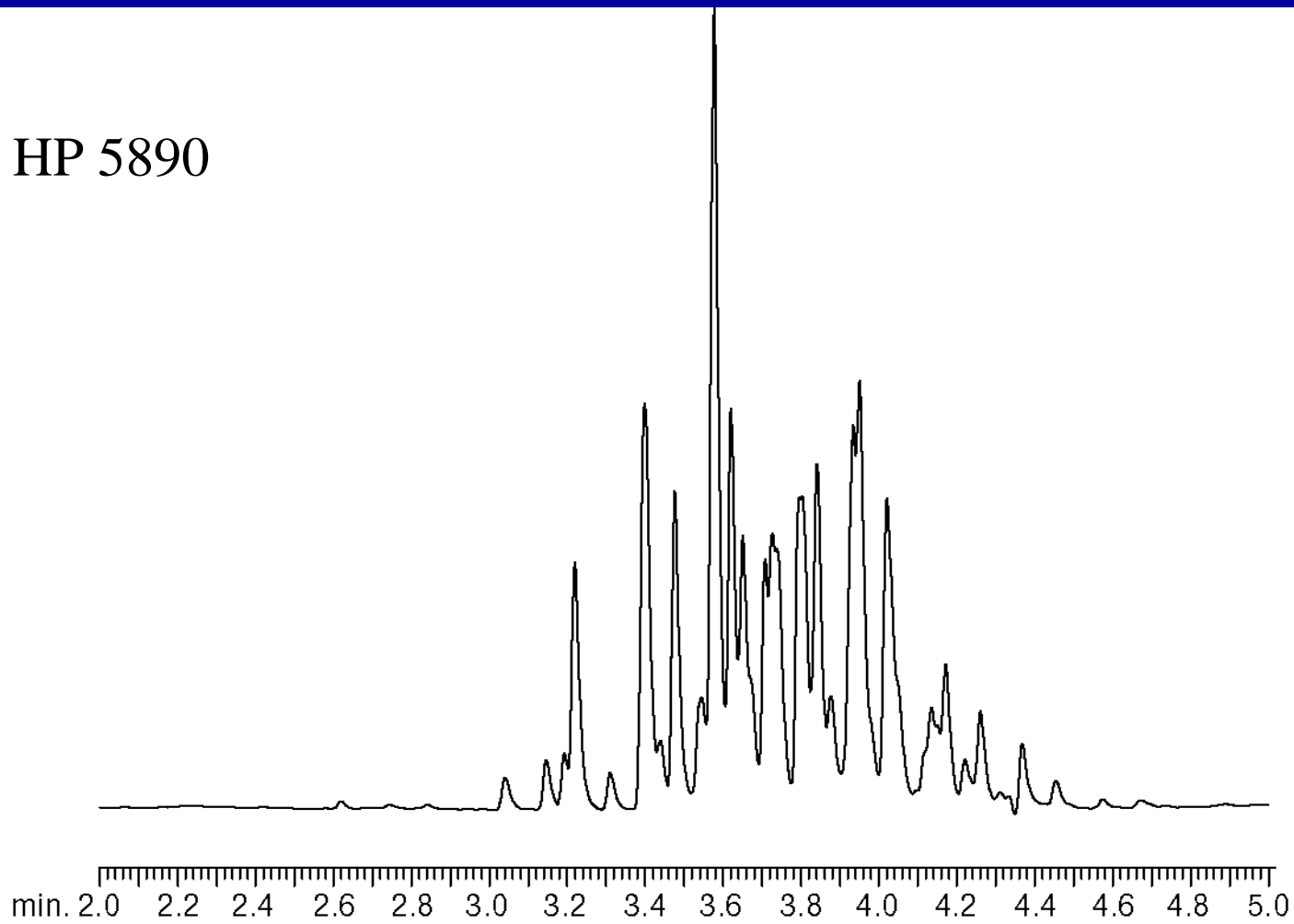
C100

PCB Aroclors

- Screening or analysis of PCB Aroclors can be performed quickly since complete separation is not a factor
- One of the most common tests required at remediation sites or for waste oil disposal
- Good candidate for accelerated separation

Rtx-5 15 m X 0.32 mm I.d. X 0.50 um d.f.

HP 5890



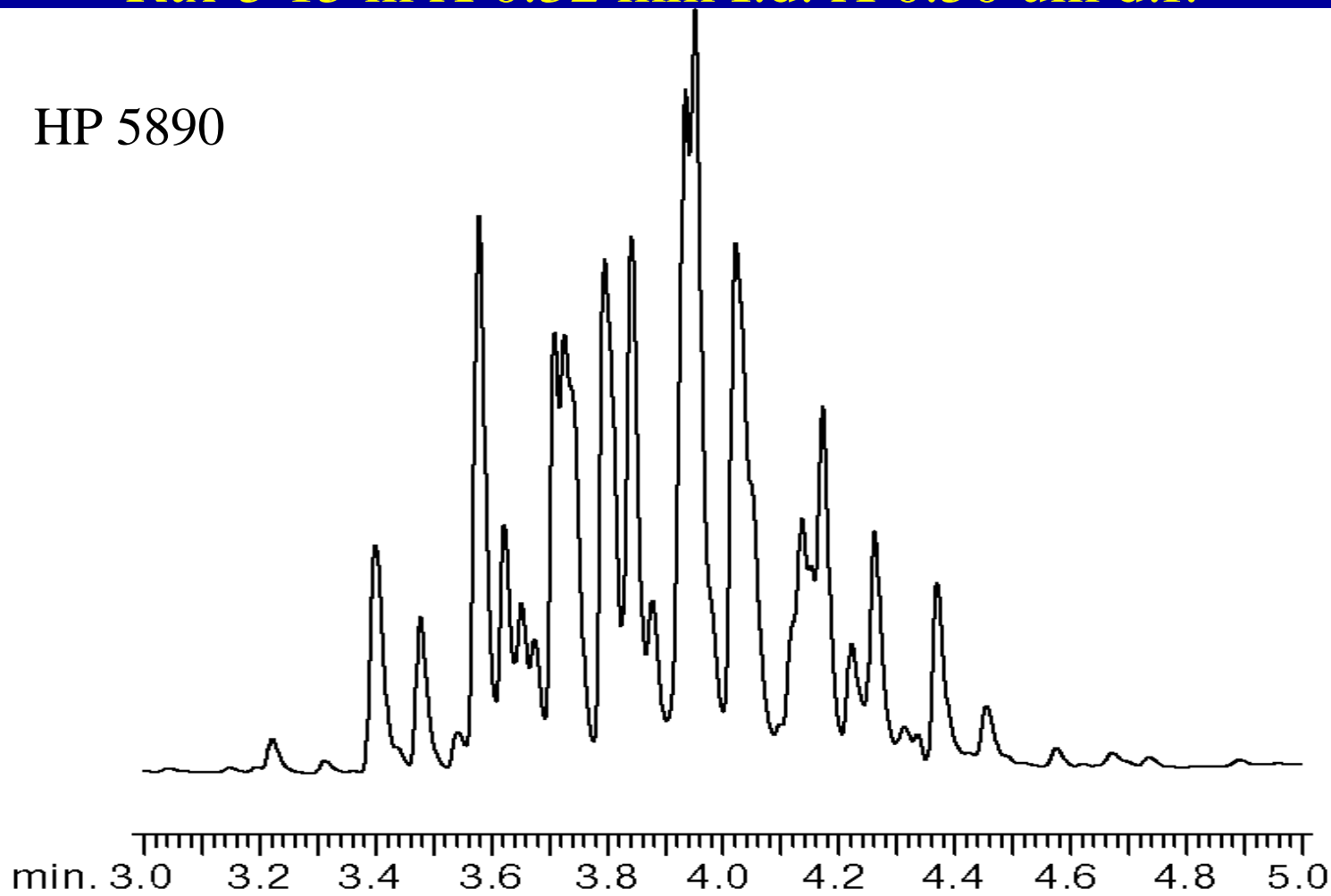
Aroclor 1242 with Zip Racer 110 C (Hold 1 min) to
300 C at 60 C/min (hold 5 min)

Restek

www.restekcorp.com

Rtx-5 15 m X 0.32 mm I.d. X 0.50 μ m d.f.

HP 5890



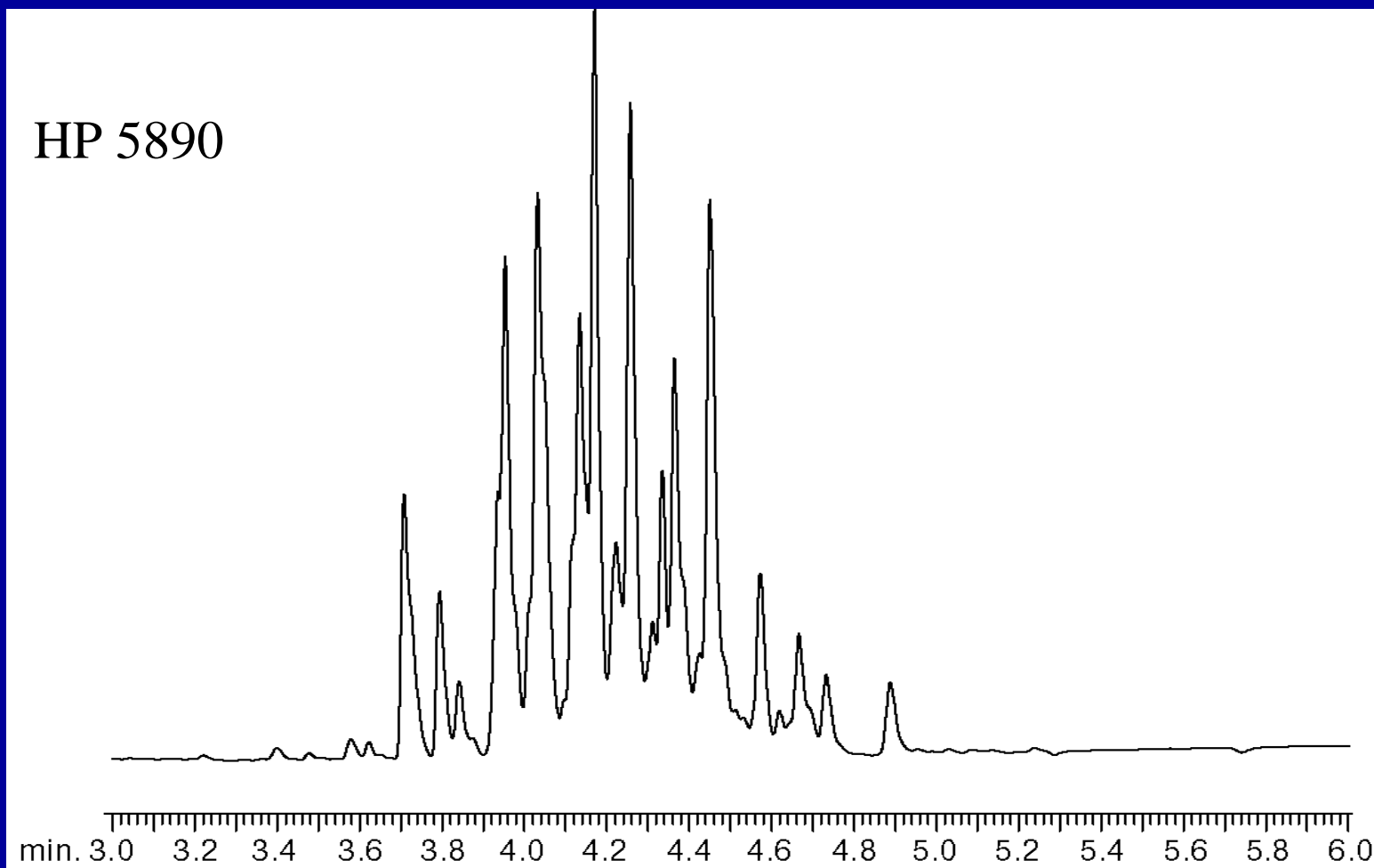
Aroclor 1248 with Zip Racer 110 C (Hold 1 min) to 300 C at
60C/min (hold 5 min)

Restek

www.restekcorp.com

Rtx-5 15 m X 0.32 mm I.d. X 0.50 um d.f.

HP 5890



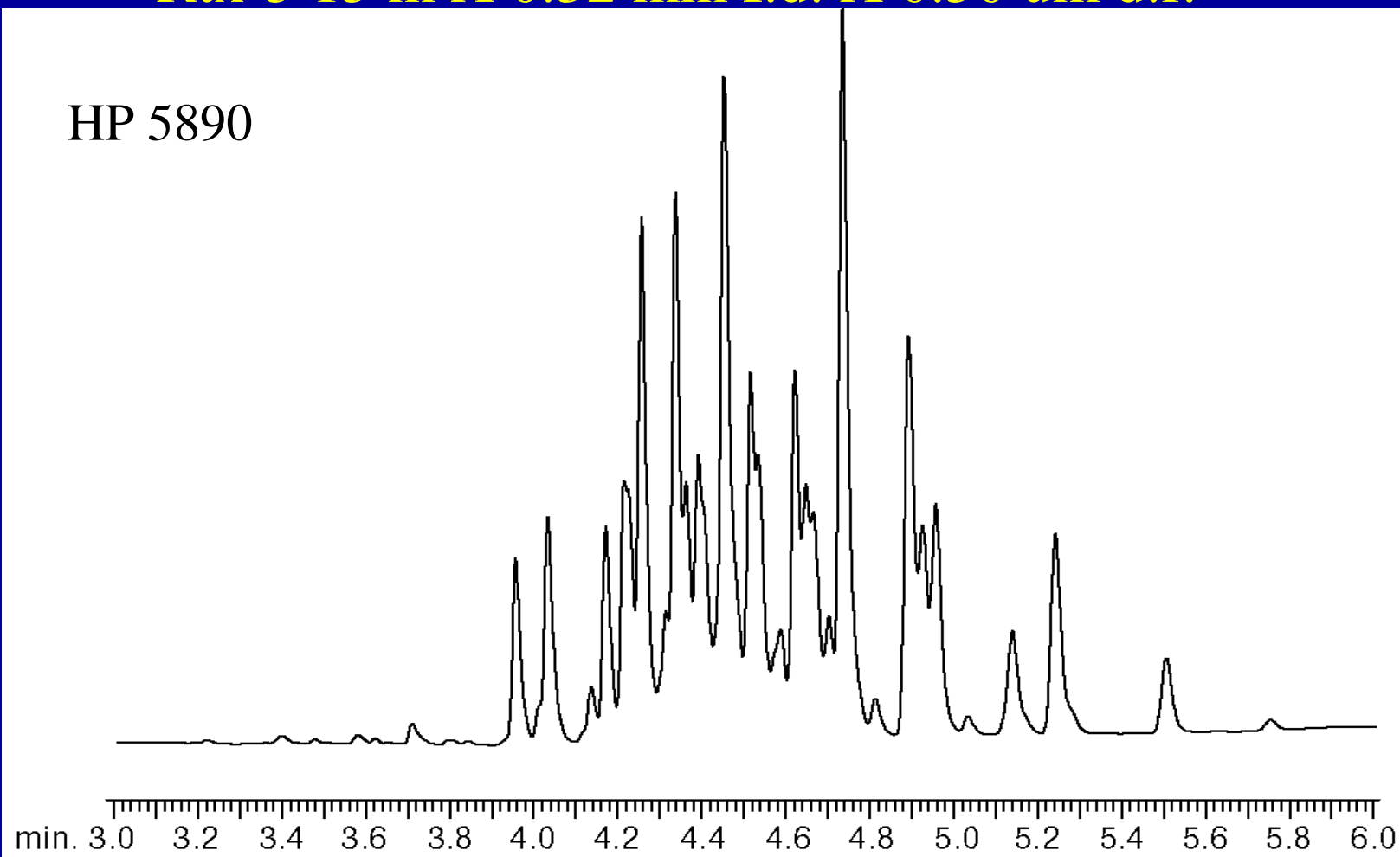
Aroclor 1254 with Zip Racer 110 C (Hold 1 min) to 300 C at
60C/min (hold 5 min)

Restek

www.restek.com

Rtx-5 15 m X 0.32 mm I.d. X 0.50 μ m d.f.

HP 5890



Aroclor 1260 with Zip Racer 110 C (Hold 1 min) to 300 C at
60C/min (hold 5 min)

Restek

www.restekcorp.com

Affordability of Zip GC Racer

Purchase Price	Small fraction of the cost of existing technology.			
Installation Cost	User installed in less than 30 min.			
Maintenance Cost	No Preventative Maintenance Required			
Training Cost	None - No Training Required			
Number of Columns	GC dependent; 1 or 2			

Summary

For the Routine User:

- GC Racer is simple to install
- No training necessary
- Allows improvement in oven ramp rates
 - Many methods can benefit
- No re-qualification necessary
- No software to learn
- Uses existing GC instrumentation