

Jim McCurry
Chemical Solutions Business Unit
September 5, 2000

**Qualitative GC - Can Retention Time Now
Replace Retention Index?**

11:00 am EST
816-650-0789
Chairperson: Bill Berry



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Jim McCurry
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**Qualitative GC - Can Retention Time Now
Replace Retention Index?**

Starts in FIVE minutes
816-650-0789
Chairperson: Bill Berry



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Starts in ONE minute
816-650-0789
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Audio Check

- **The audio portion of today's e-Seminar has just begun. To access, please dial 1-816-650-0789, Chairperson Bill Berry.**
- **If you have dialed in and cannot hear the audio, please press *0 and you will be connected to the operator.**
- **If you have audio, but the speaker is talking too quietly or too quickly, please send a chat message.**



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GC Retention Time Precision

You can achieve excellent retention time precision when you use the Agilent 6890 and 6850 with today's capillary columns

Why?

- Advances in electronic pneumatics control (EPC)
- GC temperature control and pressure compensation
- Advanced column manufacturing processes



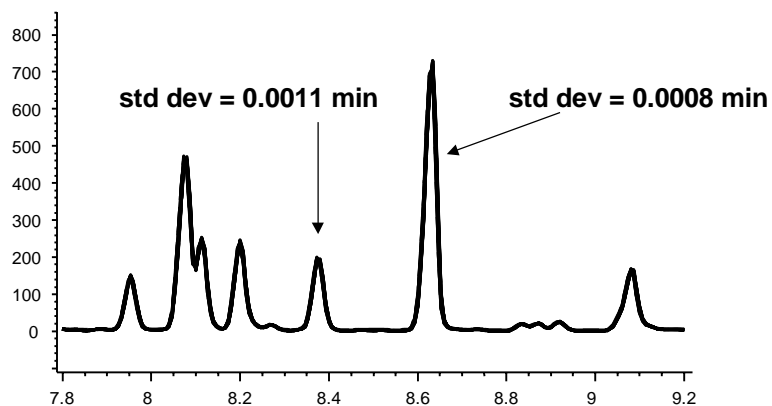
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RT Precision in a Single GC Setup

15 Runs Over 5 Days



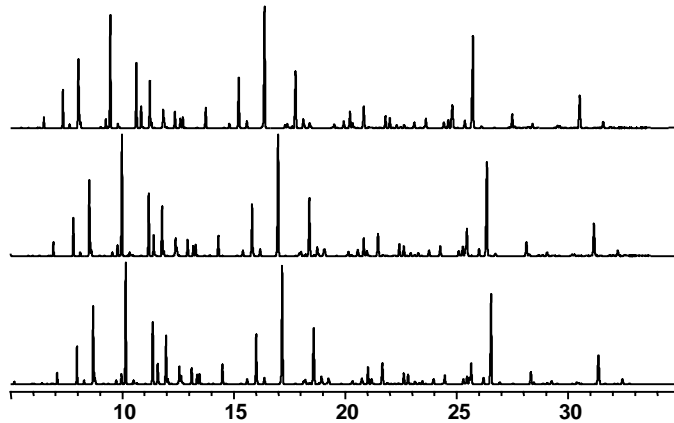
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Fragrance Sample on 3 GC Systems

Inlet P = 14.11 psi



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RT Reproducibility of Fragrance Sample

Inlet P = 14.11 psi

Retention Time (min)			Std Dev (min)	RSD	Range (min)
GC System 1	GC System 2	GC System 3			
7.343	7.803	7.971	0.325	4.219	0.628
9.465	9.981	10.155	0.359	3.637	0.690
12.361	12.929	13.113	0.392	3.062	0.752
15.226	15.823	16.010	0.409	2.610	0.784
20.212	20.837	21.027	0.426	2.061	0.815
21.805	22.435	22.631	0.432	1.936	0.826
24.431	25.093	25.302	0.455	1.823	0.871
24.631	25.273	25.464	0.436	1.737	0.833
24.814	25.460	25.650	0.438	1.732	0.836
28.400	29.060	29.264	0.452	1.562	0.864
30.514	31.163	31.366	0.445	1.435	0.852
Avg			0.415	2.347	0.796



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Traditional Approach - Retention Indices (RI)

- Enhances comparison of GC data collected on different GC systems
- Uses a homologous series as a retention time reference
 - n-alkanes or n-ethyl esters are a common index reference standard
- Linear retention index calculations for temperature-programmed runs has special requirements:
 - exact adherence to oven temperature, column dimensions, liquid-phase chemistry, and pneumatics control
 - slight variations in conditions can reduce effectiveness of linear RI calculations



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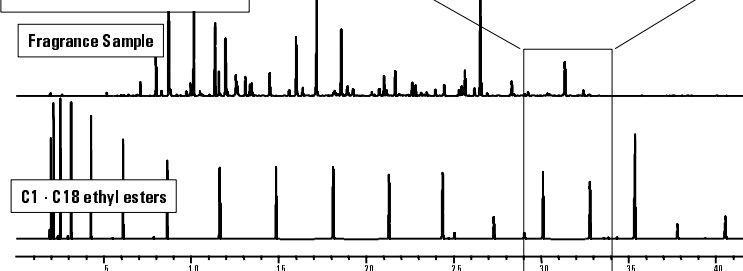
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Linear Retention Index Calculation

$$RI_a = 100N + 100n(t_{Ra} - t_{RN}) / (t_{R(N+n)} - t_{RN})$$

Where:

N = carbon number of lower ester
n = difference in carbon number of the esters that bracket the compound
 t_{Ra} = retention time of compound
 t_{RN} = retention time of lower ester
 $t_{R(N+n)}$ = retention time of higher ester



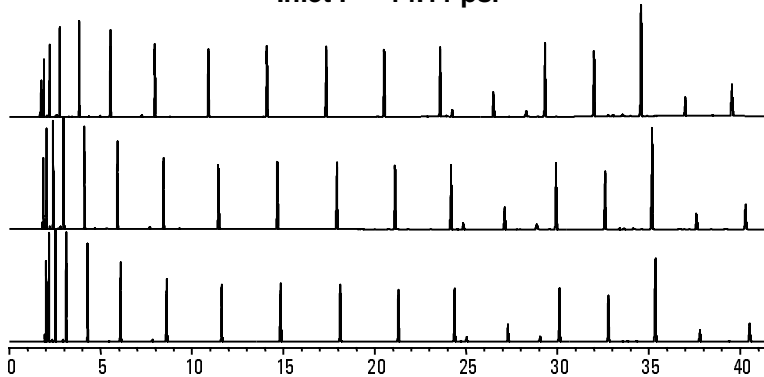
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RI Reference on 3 Different GCs

Normal C1 - C18 ethyl esters
Inlet P = 14.11 psi



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RT Reproducibility of RI Reference

Ethyl Ester	Retention Time (min)			Std Dev (min)	RSD
	GC System 1	GC System 2	GC System 3		
C1	1.731	1.881	1.991	0.131	6.988
C2	1.882	2.045	2.162	0.141	6.929
C3	2.192	2.381	2.511	0.160	6.793
C4	2.736	2.996	3.118	0.195	6.614
C5	3.806	4.111	4.270	0.236	5.804
C6	5.529	5.932	6.096	0.292	4.986
C7	7.957	8.449	8.619	0.344	4.122
C8	10.902	11.454	11.631	0.380	3.356
C9	14.097	14.683	14.863	0.401	2.753
C10	17.342	17.945	18.127	0.411	2.308
C11	20.518	21.127	21.310	0.415	1.976
C12	23.587	24.197	24.382	0.416	1.729
C13	26.505	27.121	27.309	0.421	1.559
C14	29.330	29.942	30.124	0.416	1.396
C15	32.009	32.620	32.803	0.416	1.280
C16	34.592	35.198	35.376	0.411	1.172
C17	37.005	37.622	37.811	0.422	1.125
C18	39.558	40.313	40.544	0.516	1.285
			Avg	0.340	3.454



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RI Improves Comparison of Data

Retention Index			Std Dev	RSD	Range
GC System 1	GC System 2	GC System 3			
674.13	674.35	674.30	0.12	0.02	0.22
750.65	750.98	750.98	0.19	0.03	0.33
845.18	845.68	845.85	0.35	0.04	0.67
934.27	934.93	935.14	0.45	0.05	0.87
1089.96	1090.89	1091.10	0.61	0.06	1.14
1141.60	1142.61	1142.99	0.72	0.06	1.39
1228.51	1230.63	1231.45	1.52	0.12	2.94
1235.42	1236.79	1237.00	0.86	0.07	1.58
1241.72	1243.19	1243.34	0.90	0.07	1.62
1366.74	1368.74	1369.45	1.41	0.10	2.71
1443.93	1445.58	1446.35	1.24	0.09	2.42
Avg			0.76	0.06	1.44



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Break Number 1

To ask a question:
Press *1 on your phone

OR
use chat



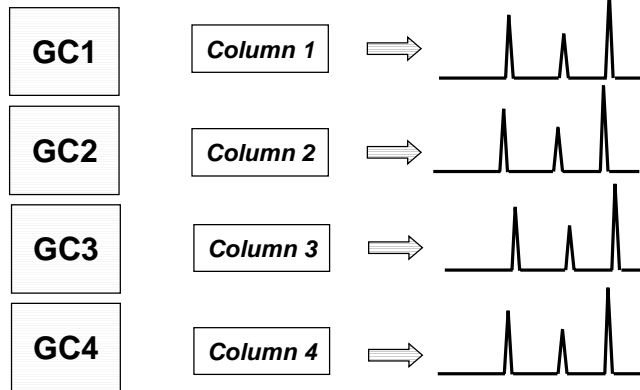
(If we don't get to your question today, everyone will receive a follow-up e-mail with all questions and answers.)



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Optimizing the Method's RT Precision

Retention times vary from GC to GC by as much as 0.5 min. ...



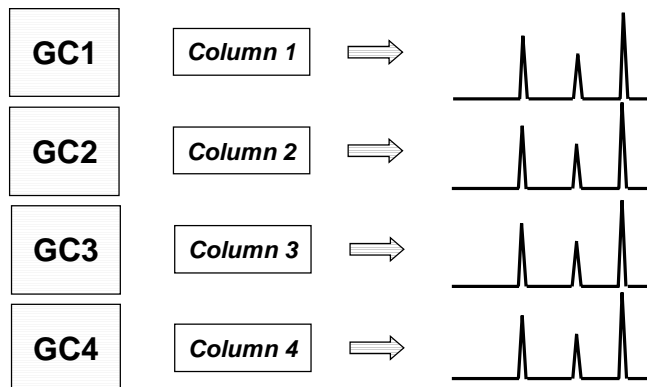
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Retention Time Locking (RTL)

Retention time variation between GC systems now less than 0.03 min with RTL ...



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What is Retention Time Locking?

The ability to precisely match chromatographic retention times in any system to those in another chromatographic system with the same nominal column.



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How is Retention Time Locking Done?

By Adjusting Column Head Pressure via

EPC

Interactive Agilent ChemStation RTL software



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RTL Method Development

Choosing A Target Compound

- Perform ALL method development work first
 - appropriate column(s)
 - GC conditions
 - required resolution
 - required sensitivity
- Target Compound Requirements
 - well resolved
 - easily detected
 - middle of run
 - not close to oven temperature transitions



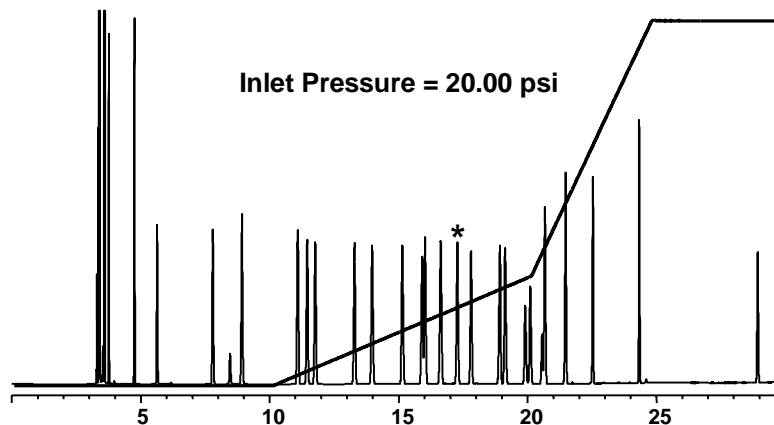
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RTL Method Development

Choosing A Target Compound



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Done Once for a Method - *FOREVER*

- Make five runs at five different inlet pressures:
 - ➔ target -20%
 - ➔ target -10%
 - ➔ target
 - ➔ target +10%
 - ➔ target +20%
- Determine RT of your target compound in each chromatogram
- Enter into RTL software



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Retention Time Locking Calibration

	Pressure	Ret Time
Run 1	16	19.084
Run 2	18	17.766
Run 3	20	16.633
Run 4	22	15.641
Run 5	24	14.756

Pressure Units:

Desired Ret Time:

Min relock pressure:

Max relock pressure:

Column:

Compound Name:

Locking a GC System

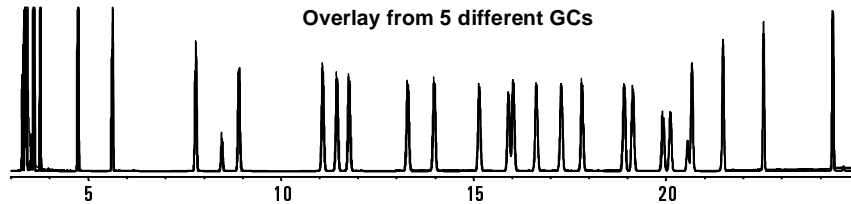
1. Make one (or two) runs at nominal method pressure.
2. Determine RT of target compound.
3. New locking pressure is calculated by software.
4. Make run to confirm locked RTs.

[Re]Lock current method

Retention time: Enter current retention time of:
5 pk @ ca. 6 min.
 Minutes
Then select button 'Update Method' to calculate a new pressure and enter it in the method.

Method Information:
Current Method: RTL_20.M
Column:
Pressure used: psi
Desired RT: 6.147 Minutes
 psi

RT Precision Between GCs with RTL



GC	inlet P (psi)	Retention Time (min.)			
		benzene	cumene	t-butylbenzene *	α -methylstyrene
6890#1	20.00	5.581	13.097	16.423	21.011
6890#2	20.04	5.572	13.097	16.424	21.003
6850#1	19.64	5.518	13.089	16.420	20.976
6850#2	19.52	5.576	13.110	16.426	20.975
6850#3	19.51	5.572	13.098	16.426	20.994
	std. dev.	0.026	0.008	0.002	0.016
	range	0.063	0.021	0.006	0.036

* target peak used for RTL calibration



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Can RTL Replace Retention Indices?

Some Advantages to Using RTL

- RTL uses only one peak to calibrate RT vs. pressure
- RTL calibration only needs to be done once per method
- No special calculations required
- Do not need to estimate if missing RI standard
- Time is an easier concept to grasp
- Graphically easier to compare data between systems
- Easier to review historic data



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Locking a Fragrance Sample

Retention Time Locking Calibration

Run	Pressure	Ret Time
Run 1	11.29	26.028
Run 2	12.69	25.137
Run 3	14.11	24.382
Run 4	15.52	23.723
Run 5	16.93	23.142

Pressure Units:

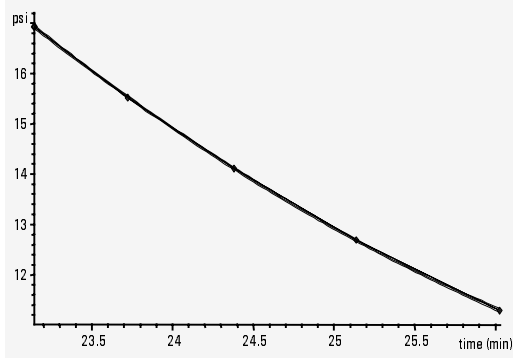
Desired Ret Time:

Min relock pressure:

Max relock pressure:

Column:

Compound Name:

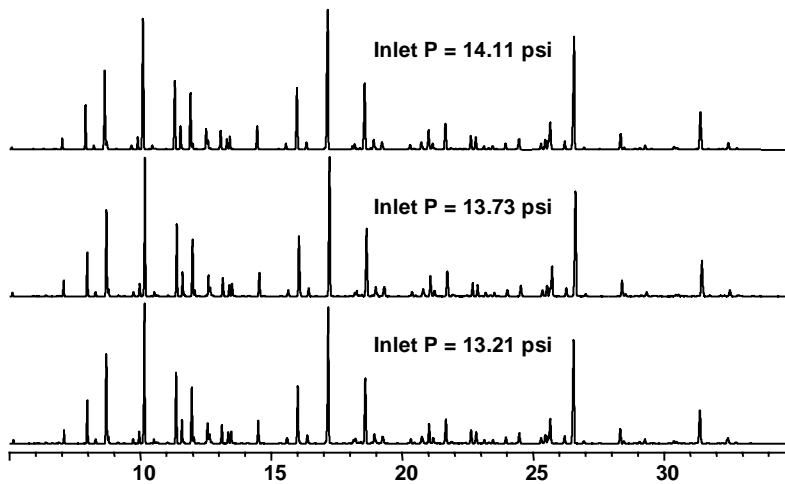


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A Fragrance Sample After RTL



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10x Better RT Precision With RTL

Retention Times After RTL					
GC System 1	GC System 2	GC System 3	Std Dev	RSD	Range (Min)
7.909	7.971	7.975	0.037	0.465	0.066
10.102	10.176	10.159	0.039	0.382	0.074
13.065	13.148	13.118	0.042	0.321	0.083
15.977	16.059	16.015	0.041	0.256	0.082
21.008	21.082	21.033	0.038	0.179	0.074
22.618	22.688	22.637	0.036	0.160	0.070
25.298	25.359	25.309	0.033	0.128	0.061
25.582	25.532	25.470	0.056	0.220	0.112
25.650	25.719	25.658	0.038	0.147	0.069
29.276	29.335	29.270	0.036	0.123	0.065
31.388	31.447	31.374	0.039	0.123	0.073
Avg			0.039	0.228	0.075

vs. 2.347
without RTL!



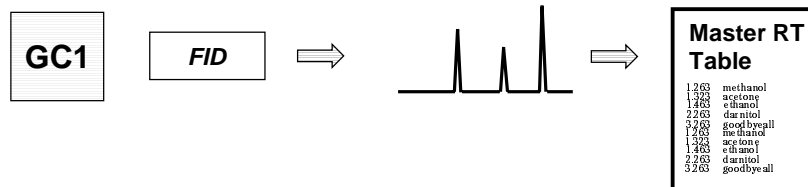
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Build a Retention Time Table

Record retention times under RT-Locked conditions...

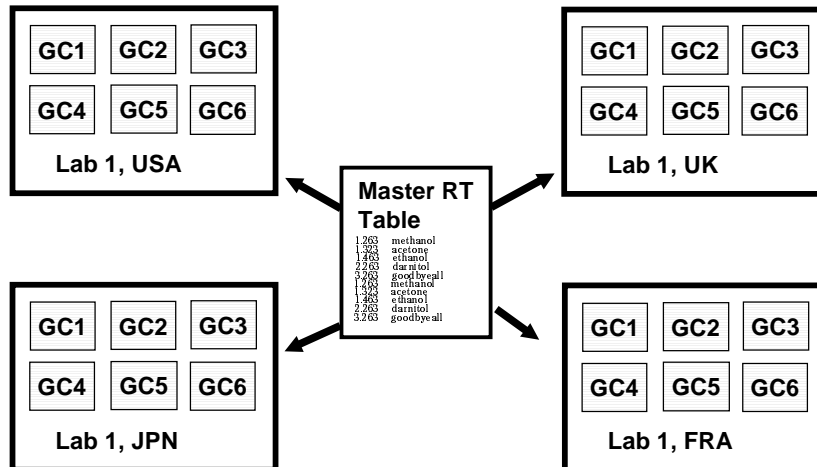


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Use RT Tables Worldwide



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Combining RTL with RI

- RTL uses only one peak in the reference standard to calibrate RT vs. pressure
- After RTL, the RTs of all the reference peaks on all GCs will be the same
- RTL can improve the precision of linear retention index calculations
 - reduces errors contributed by variations in column dimensions



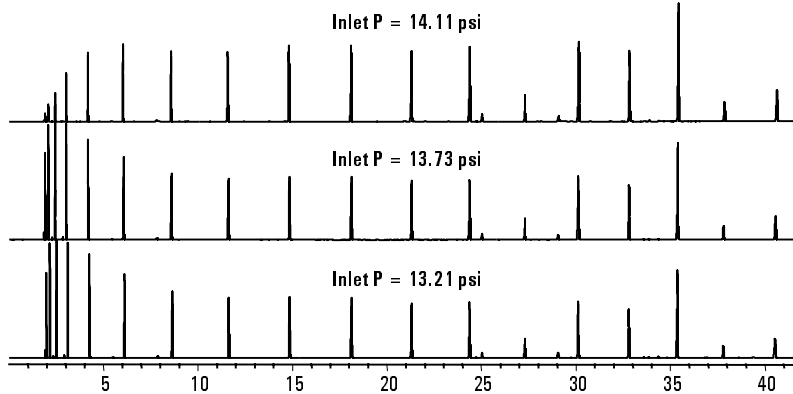
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RI Reference After RTL

Normal C1 - C18 ethyl esters



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If You Still Need Retention Indices...

RTL Can Improve RI Precision

Retention Index After RTL					
GC System 1	GC System 2	GC System 3	Std Dev	RSD	Range
674.29	674.93	674.32	0.36	0.05	0.64
751.01	751.63	750.99	0.36	0.05	0.64
845.64	846.36	845.84	0.37	0.04	0.72
935.01	935.65	935.12	0.34	0.04	0.64
1090.71	1091.14	1091.03	0.22	0.02	0.43
1142.56	1143.00	1142.92	0.23	0.02	0.44
1230.94	1231.26	1231.39	0.23	0.02	0.45
1236.49	1237.17	1236.88	0.34	0.03	0.68
1242.96	1243.55	1243.32	0.30	0.02	0.59
1368.85	1369.38	1369.38	0.31	0.02	0.53
1445.68	1446.00	1446.29	0.31	0.02	0.61
Avg			0.31	0.03	0.58

vs. 0.06
without RTL!



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Summary

No RTL or RI RSD = 2.35%

RI Only: RSD = 0.06%

RTL Only: RSD = 0.23%

RTL, RI Combination: RSD = 0.03%



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Wrap-up Questions

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