RESTEK Pure Chromatography

Exploration of New Low-Pressure GC Columns for Food and Environmental Emerging Contaminants

Jessi Collier[1], Jana Hepner[1], Whitney Dudek-Salisbury[1], Kristi Sellers[1], Jaap de Zeeuw[1], Chris English[1]

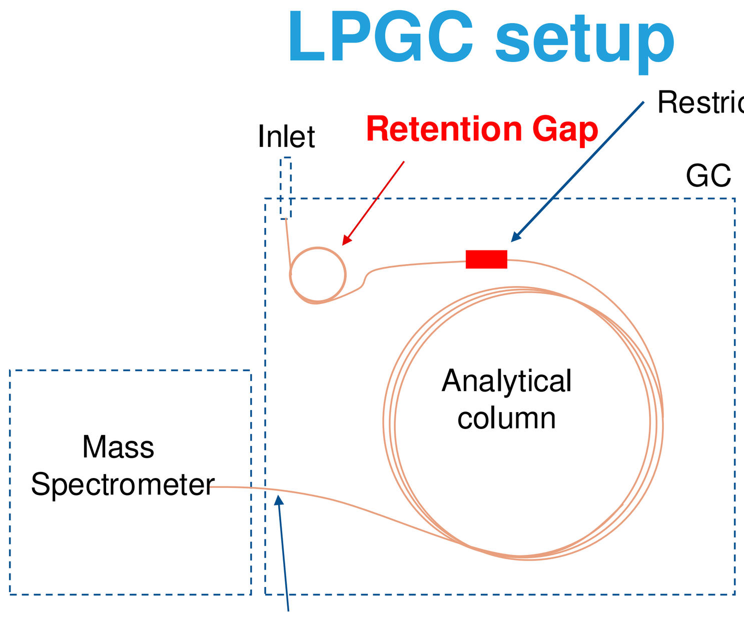
[1]**Restek Corporation,** 110 Benner Circle; Bellefonte, PA 16823, USA; jessi.collier@restek.com

Introduction

The Low-Pressure GC (LPGC technique) has been successfully used in the past for pesticide residues' analysis. However, the technique is very versatile, and it allows for other applications, especially if different column phases are used. So far, the majority of the applications have been using the "5"-type phase (95% dimethylpolysiloxane, 5% diphenyl polymer). To expand on the previous applications, four additional column phases were selected (cyanopropylphenyl dimethylpolysiloxane; 50% dimethylsiloxane, 50% diphenyl; 65% dimethylsiloxane, 35% diphenyl; and trifluoropropylmethyl polysiloxane phases) to analyze various food and environmental contaminants, such as nitrosamines, alkylfurans, phthalates, arylamines and fluorotelomer alcohols.

The LPGC techniques provided significant reduction in run times (up to 3.3x faster runs) and helium consumption reduction (up to 81% less helium used), while keeping an acceptable resolution.

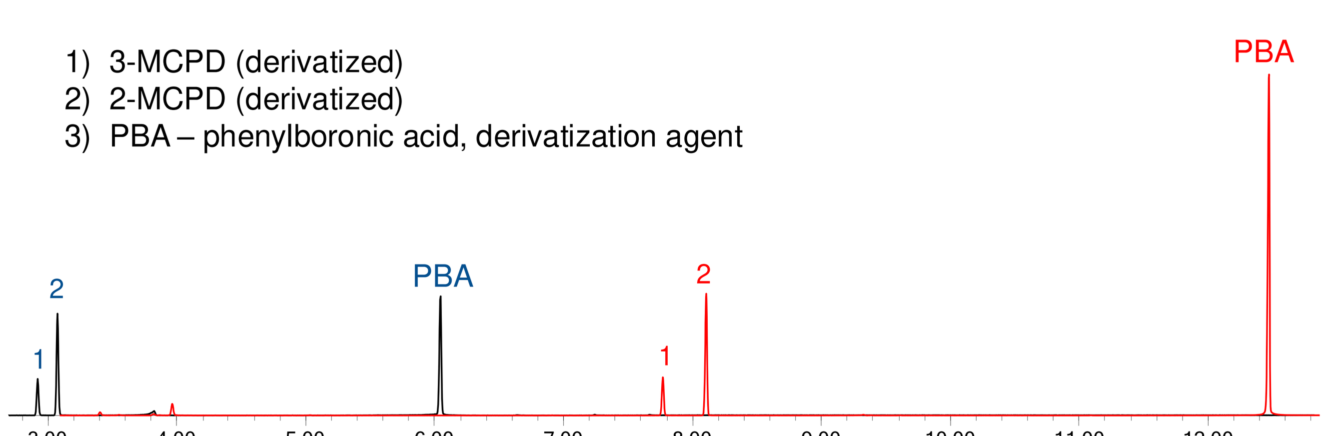
LPGC setup



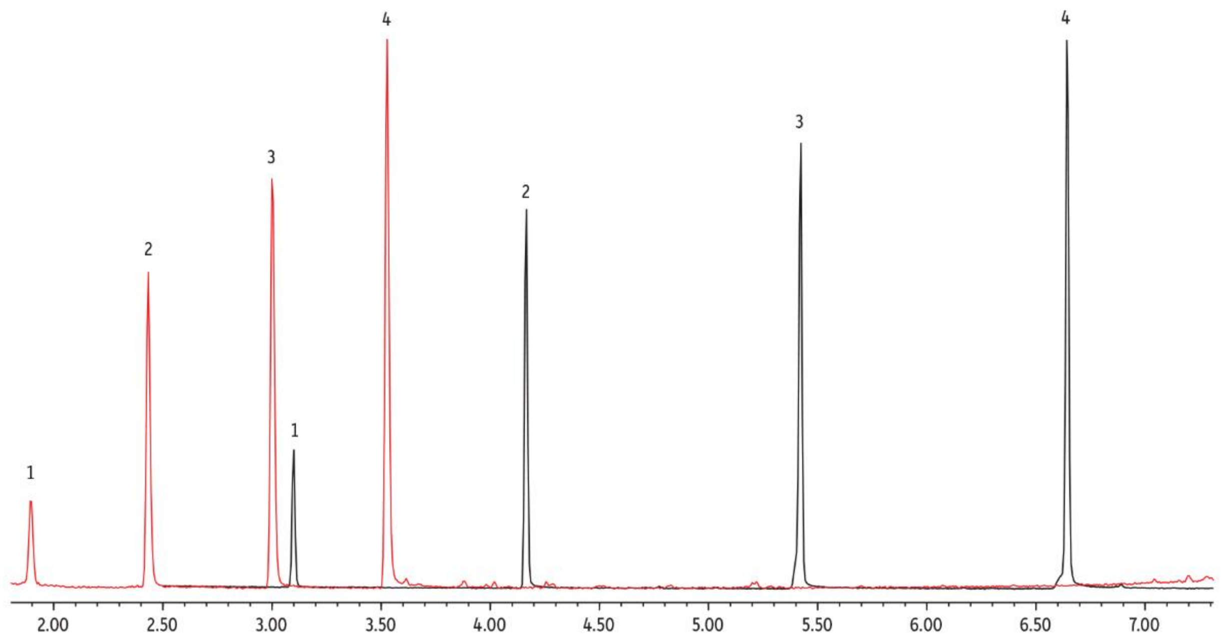
Column set is delivered pre-connected in the box
Only extra consumable needed is 0.8 mm vespel/graphite ferrule for MS transfer line

LPGC Rxi-17Sil MS Application - MCPDs

1) 3-MCPD (derivatized)
2) 2-MCPD (derivatized)
3) PBA – phenylboronic acid, derivatization agent



LPGC Rtx-200 – Fluorotelomer Alcohols (FTOHs)

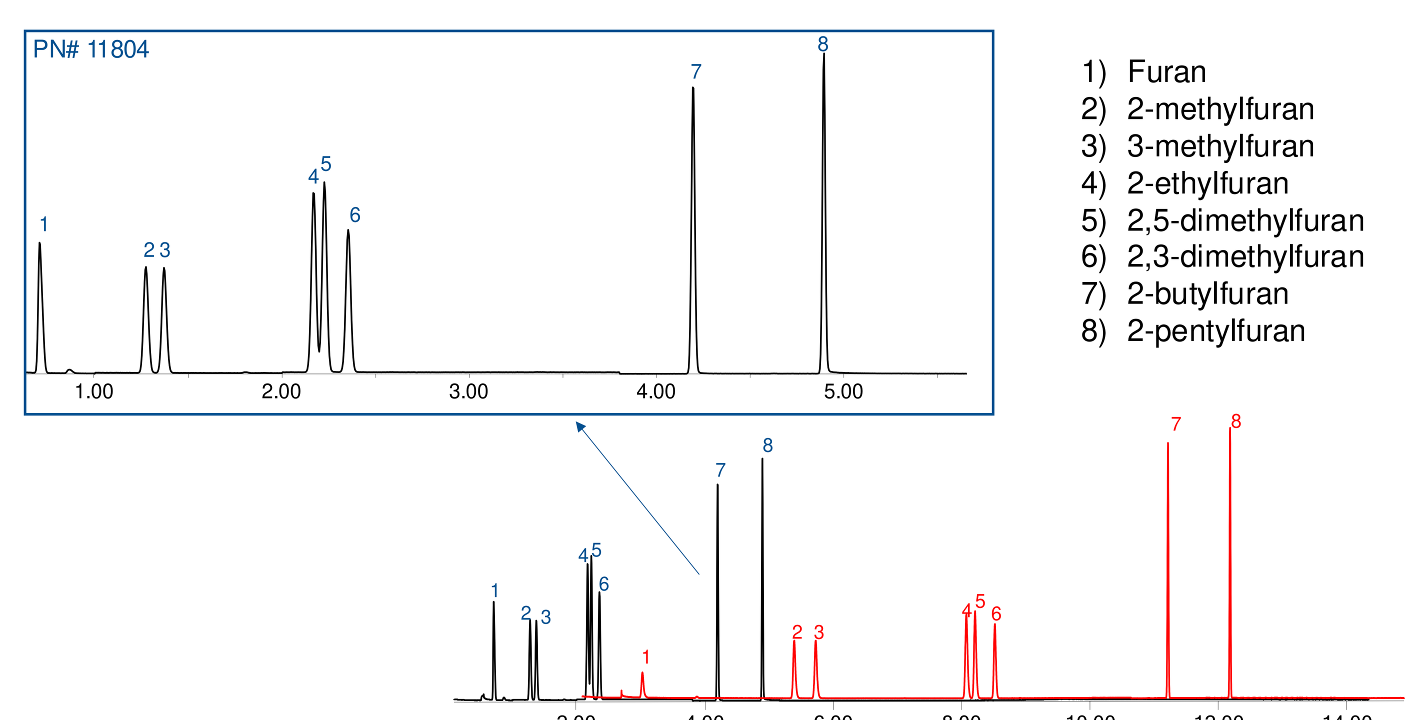


1) 4:2 FTOH (2-perfluorobutyl alcohol)
2) 6:2 FTOH (2-perfluorohexyl alcohol)
3) 8:2 FTOH (2-perfluorooctyl alcohol)
4) 10:2 FTOH (2-perfluorodecyl alcohol)

Advantages of LPGC

- **Fast analysis with short 0.53 mm or 0.32 mm capillaries**
  - **Short analysis times**
  - **Increased sensitivity**
  - **Potentially higher capacity**
- **Peak width enough for any type of MS**
- **Lower elution temperatures**
- **Elution at 50-80°C lower temperatures**
  - **Lower bleed**
  - **Standard injection techniques, high volume injections**
- **All stationary phase chemistries**

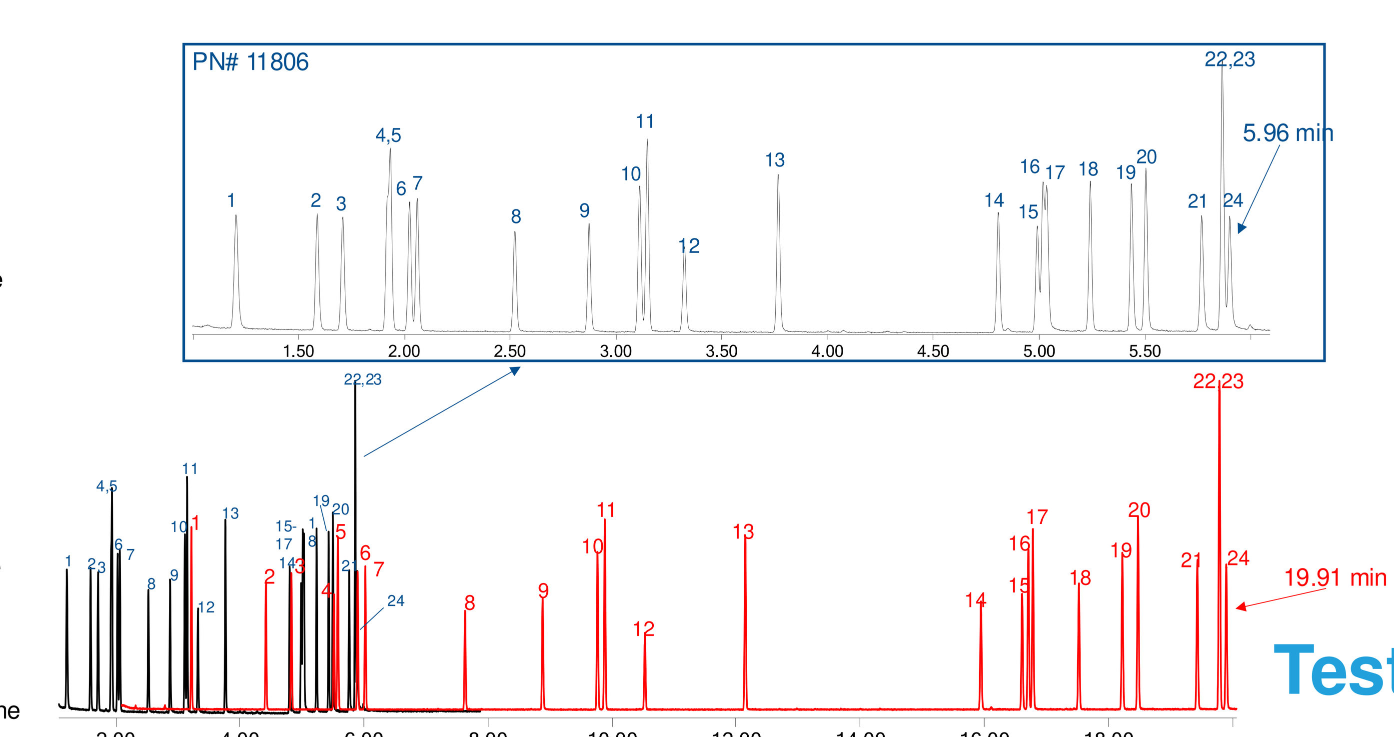
LPGC Rxi-624Sil MS Application #2 - Alkylfurans



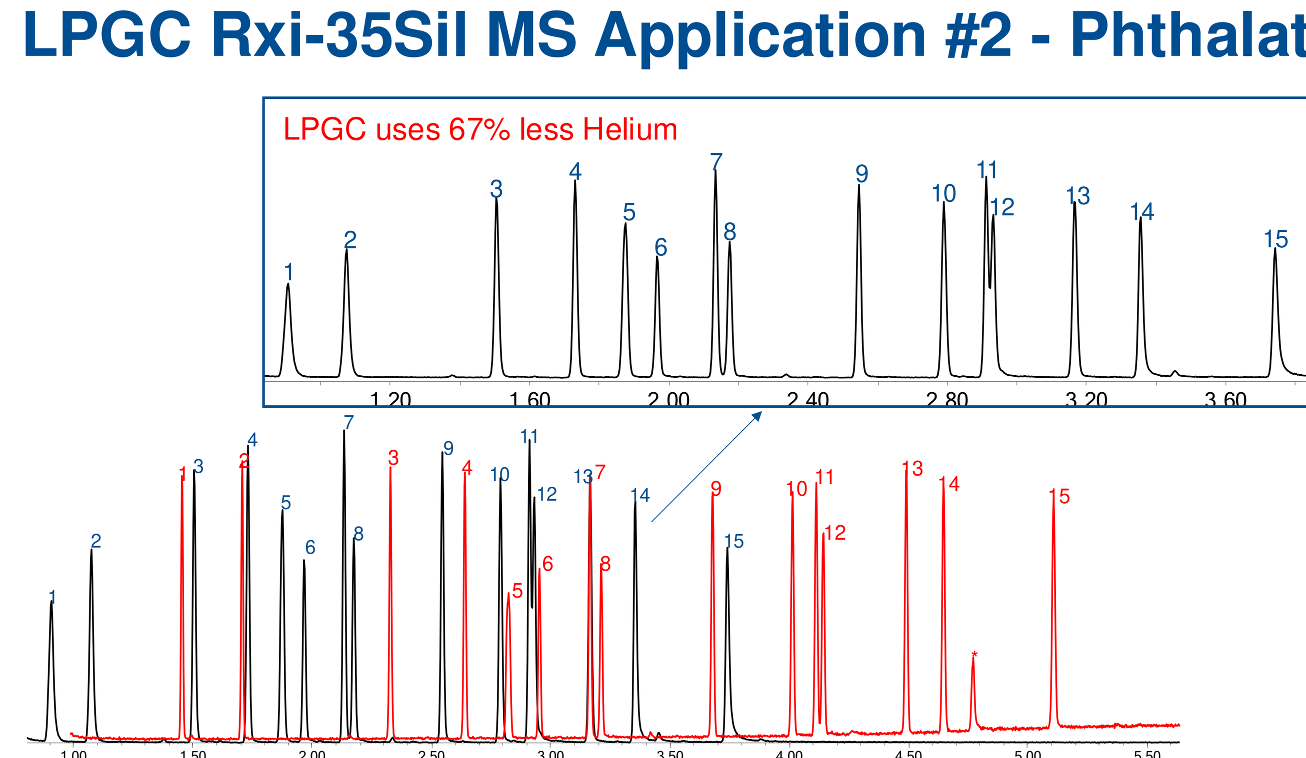
1) Furan
2) 2-methylfuran
3) 3-methylfuran
4) 2-ethylfuran
5) 2,5-dimethylfuran
6) 2,3-dimethylfuran
7) 2-butylfuran
8) 2-pentylfuran

LPGC Rxi-35Sil MS Application #1 - Arylamines

1) o-Toluidine
2) o-Anisidine
3) 4-Chloroaniline
4) p-Cresidine
5) 2,4,5-Trimethylaniline
6) 3-Chloro-o-toluidine
7) 4-Chloro-o-toluidine
8) 2,4-Diaminotoluene
9) 2,4-Diaminoanisole
10) 2-Naphthylamine
11) 2-Aminobiphenyl
12) 2-Amino-4-nitrotoluene
13) 4-Aminobiphenyl
14) p-Aminoazobenzene
15) 4,4'-Oxydianiline
16) 4,4'-Diaminodiphenylmethane
17) Benzidine
18) o-Aminoazotoluene
19) 3,3'-Dimethyl-4,4'-diaminodiphenylmethane
20) 3,3'-Dimethylbenzidine
21) 4,4'-Thiodianiline
22) 3,3'-Dichlorobenzidine
23) 4,4'-Methylenebis (2-chloroaniline)
24) 3,3'-Dimethoxybenzidine

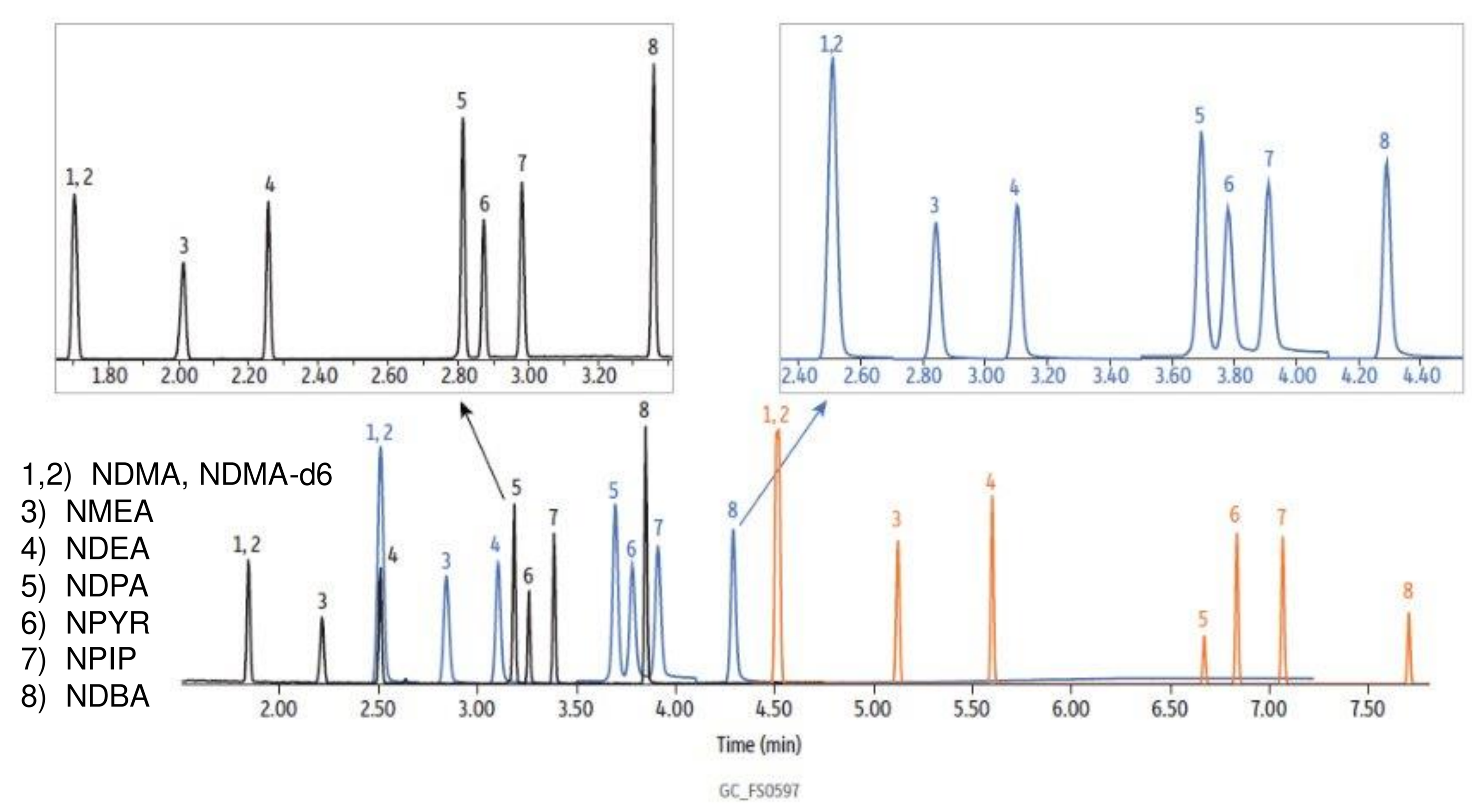


LPGC Rxi-35Sil MS Application #2 - Phthalates



1) Dimethyl phthalate
2) Diethyl phthalate
3) Diisobutyl phthalate
4) Di-n-butyl phthalate
5) Bis(2-methoxyethyl) phthalate
6) Bis[4-methyl-2-pentyl] phthalate isomers
7) Di-n-pentyl phthalate
8) Bis(2-ethoxyethyl) phthalate
9) Di-n-hexyl phthalate
10) Butyl benzyl phthalate
11) Bis(2-ethylhexyl) phthalate
12) Bis(2-butoxyethyl) phthalate
13) Dicyclohexyl phthalate
14) Di-n-octyl phthalate
15) Dinonyl phthalate

LPGC Rxi-624Sil MS Application #1 - Nitrosamines



1,2) NDMA, NDMA-d6
3) NMEA
4) NDEA
5) NDPA
6) NPYR
7) NPIP
8) NDBA

Limitations of LPGC
Strategies for Addressing Shortcomings

- **Loss of theoretical plates (compared to conventional column)**
  - **Can be mitigated by selective detection by MS**
- **Greater potential for leaks**
  - **Pre-connected set**
- **More complicated to cut analytical column**
  - **Less need to cut column**
- **Need for MS instrument**
- **Fast oven heating needed**
  - **120 V instruments might need an accelerator**

Can I use LPGC for my application?
Questions to ask yourself

- **Is MS a suitable detector?**
  - **Vacuum is needed**
- **What kind of column is the conventional method using?**
  - **In general, most conventional methods with 30 m column can be translated to LPGC method**
- **Are there isobars to resolve?**
  - **What level of resolution is needed?**
  - **What is the resolution in the conventional method?**
    - **If it is above 2.0, usually we can get peaks resolved**

Tested Applications Background

- **Both LPGC and conventional column runs were acquired**
- **The last eluting compound was used to calculate both the time reduction and helium savings**
- **The initial and final temperatures were held constant**
  - **Temperature ramps were held below 35°C/min**
    - **Better times/helium savings are possible with rapid heating oven (220-240 V and/or with accelerator)**
- **Leaks were tested using the instrument's leak test**
  - **Leak-free column set has less than 5% nitrogen or water present in the MS**

Conclusions

- **LPGC offering has been expanded**
  - **Analysis up to 3.3x faster**
    - **Limited by the temperature program**
  - **Up to 81% less helium used**
- **No need to change instrumentation**
  - **Now compatible with all instruments**