



Fast and Micro Gas Chromatography - Recent Advances from the Winery to the Refinery to the Wellhead

a Symposium Preview

Dr. Carl Rechsteiner
CRechsteiner Consulting, LLC





What enables significant advancement in Fast, Micro and Ultracompact Gas Chromatography?

Answer:

Broad Commercial Utility and Acceptance

The Prerequisites for Commercial Significance

- *Significantly Differentiating Invention*
- *Validation of the Technology by Industrial Users*
- *Application Capability Serving a Broad Industrial User Set*
- *Commercial Deployment in Routine Use*
- *Validation of the Economic Impact*
- *Replication throughout the Various Enterprises*



Objectives for the Symposium

- *To inform attendees of users' success*
- *To allow users' to share their experience*
- *To brainstorm about future advancements*
- *To enjoy some refreshments together at the end!*



Differentiating Intellectual Property

Fast Micro Gas Chromatograph System

US Patent 8414832



US08414832B1

(12) **United States Patent**
Roques et al.

(10) Patent No.: **US 8,414,832 B1**
(45) Date of Patent: **Apr. 9, 2013**

(54) **FAST MICRO GAS CHROMATOGRAPH SYSTEM**

(76) Inventors: **Ned Roques, Lewisburg, WV (US); John Crandall, Lewisburg, WV (US)**

(*) Notice: Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 889 days.

(21) Appl. No.: **12/555,783**

(22) Filed: **Sep. 8, 2009**

Related U.S. Application Data

(60) Provisional application No. 61/095,075, filed on Sep. 8, 2008.

(51) Int. Cl. **G01N 30/02** (2006.01)
G01N 30/04 (2006.01)

(52) U.S. Cl. **422/89, 73/23.39, 73/23.4, 96/102, 96/106**

(58) **Field of Classification Search** 422/70, 422/89, 73/23.39, 23.4, 61.53, 61.57, 61.58, 95/87, 96/102, 106, 210/198.2
See application file for complete search history.

(56) **References Cited**

U.S. PATENT DOCUMENTS

| | | | |
|---------------|---------|------------------|-------|
| 4,096,908 A * | 6/1979 | Lamy | 16504 |
| 4,872,334 A | 10/1989 | Watanabe | |
| 5,028,243 A | 7/1991 | Ralby | |
| 5,068,322 A | 9/1991 | Hilber et al. | |
| 5,092,156 A | 3/1992 | Mikolajczyk | |
| 5,099,343 A | 3/1992 | Rosenthal et al. | |
| 5,108,785 A | 4/1992 | Rosenthal et al. | 9518 |
| 5,114,439 A | 3/1992 | Yoon et al. | |
| 5,300,758 A | 4/1994 | Rosenthal et al. | |
| 5,310,681 A | 5/1994 | Rosenthal et al. | |
| 5,551,278 A | 9/1996 | Rosenthal et al. | |
| 5,611,846 A | 3/1997 | Ovren | |

FOREIGN PATENT DOCUMENTS

| | | |
|----|-------------------|--------|
| WO | WO 92/0748 | 6/1992 |
| WO | WO 2008/030131 A1 | 3/2008 |

* cited by examiner

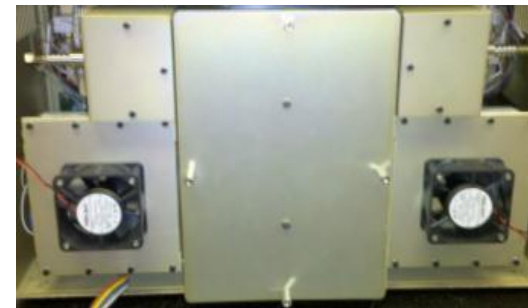
Primary Examiner—Jan Ludlow
(74) *Attorney, Agent, or Firm*—Sheldon H Parker, Esq.

(57) **ABSTRACT**
The invention is a chromatography apparatus which comprises at least one capillary column, which has a coil assembly of column material and a small diameter wire coated with an electrically insulating high temperature material encased within a high temperature sheath. The small diameter wire is at least one electrically conductive element co-linear with the column material. Also provided is means for directly resistively heating the at least one capillary column, and means for controlling the temperature of the capillary column. Additionally, the apparatus includes an oxygen gas containing inlet, a hydrogen inlet, an analyte port and a flame region, oxygen delivery means for delivering oxygen through the oxygen inlet to the flame region, a hydrogen and analyte delivery system for delivering hydrogen and analyte to the flame region, and a detector arranged to detect flame emission.

21 Claims, 8 Drawing Sheets

Trans-Configurable Modular Chromatographic Assembly

US Patent 8336366



US08336366B2

(12) **United States Patent**
Roques et al.

(10) Patent No.: **US 8,336,366 B2**
(45) Date of Patent: **Dec. 25, 2012**

(54) **TRANS-CONFIGURABLE MODULAR CHROMATOGRAPHIC ASSEMBLY**

(75) Inventors: **Ned Roques, Lewisburg, WV (US); John Crandall, Lewisburg, WV (US)**

(73) Assignee: **Falcon Analytical, Lewisburg, WV (US)**

(*) Notice: Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 365 days.

(21) Appl. No.: **12/817,137**

(22) Filed: **Jun. 16, 2010**

(65) **Prior Publication Data**
US 2010/0256922 A1 Oct. 7, 2010

Related U.S. Application Data

(63) Continuation-in-part of application No. 12/555,783, filed on Sep. 8, 2009.

(60) Provisional application No. 61/095,075, filed on Sep. 8, 2008.

(51) Int. Cl. **G01N 30/02** (2006.01)

(52) U.S. Cl. **73/23.39**

(58) **Field of Classification Search** 73/23.39
See application file for complete search history.

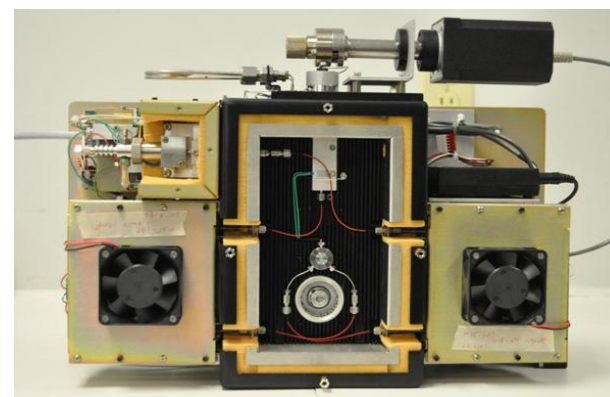
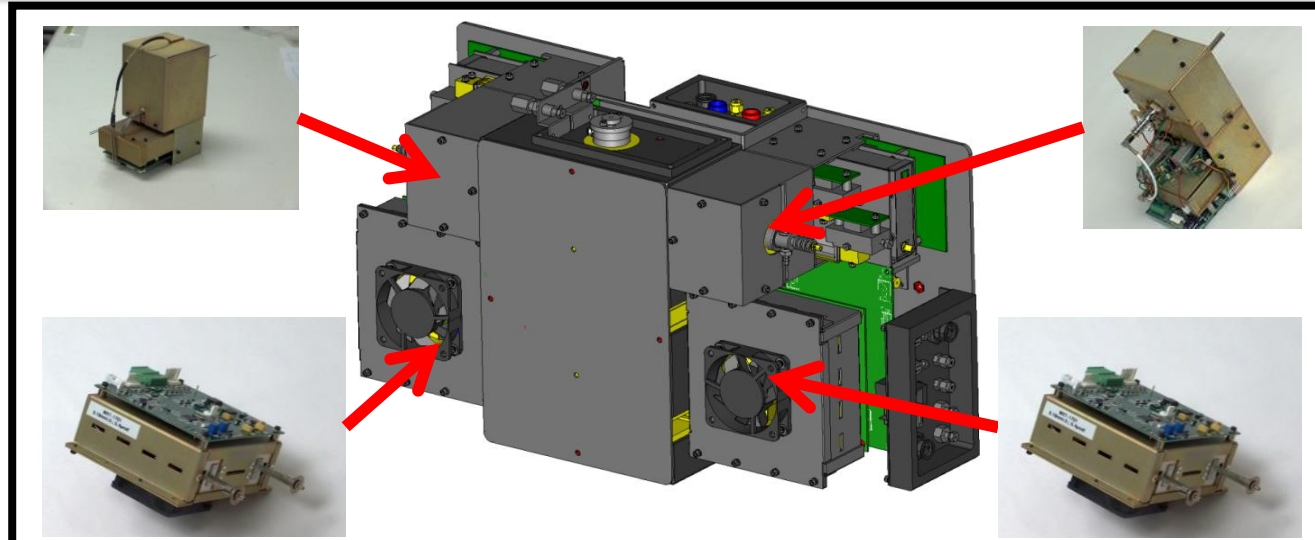
(56) **References Cited**

U.S. PATENT DOCUMENTS

| | | | |
|---------------|--------|------------------|----------|
| 5,611,846 A * | 3/1997 | Ovren et al. | 96/102 |
| 5,868,178 A * | 9/1998 | Rosenthal et al. | 73/23.39 |
| 6,071,408 A * | 6/2000 | Allington et al. | 2108/34 |

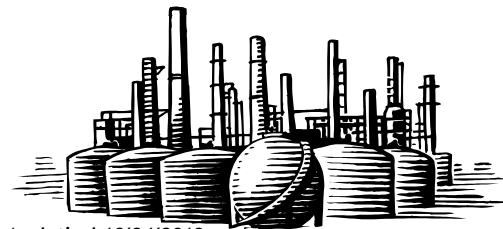
15 Claims, 9 Drawing Sheets

Trans-Configurable MODULAR Chromatographic Assembly



But What about Industrial Validation?

- “Boiling Range Distribution of Petroleum Distillates With Final Boiling Points up to 535° C by Ultra Fast Gas Chromatography (UF GC)” authors Bostic, DiSanzo, Lubkowitz in publication now
- Interlaboratory Study to begin Q4, 2013... for the **new ASTM D-7798** method.
- Here are current results demonstrating conformance with the existing D-2887 requirements.



Boiling Point Repeatability & Reproducibility

| Rep # | 0.50% | 5.00% | 10.00% | 15.00% | 20.00% | 25.00% | 30.00% | 35.00% | 40.00% | 45.00% | 50.00% | 55.00% | 60.00% | 65.00% | 70.00% | 75.00% | 80.00% | 85.00% | 90.00% | 95.00% | 99.50% |
|-------------------|--------------|--------------|--------------|--------------|--------------|--------------|--------------|--------------|--------------|--------------|--------------|--------------|--------------|--------------|--------------|--------------|--------------|--------------|--------------|--------------|--------------|
| 1 | 241.3 | 304.6 | 349.1 | 394.8 | 436.5 | 471.3 | 500.0 | 527.3 | 553.5 | 577.5 | 594.6 | 610.7 | 629.3 | 648.7 | 668.6 | 690.1 | 712.8 | 737.2 | 765.3 | 804.4 | 885.6 |
| 2 | 240.5 | 304.4 | 349.1 | 394.9 | 436.8 | 471.3 | 500.3 | 527.7 | 553.6 | 577.7 | 595.0 | 611.1 | 629.7 | 649.3 | 669.1 | 690.6 | 713.3 | 737.7 | 766.1 | 805.3 | 886.9 |
| 3 | 241.0 | 304.4 | 349.2 | 394.7 | 436.8 | 471.3 | 500.5 | 527.8 | 553.5 | 577.5 | 594.6 | 610.7 | 629.1 | 648.8 | 668.5 | 690.3 | 712.8 | 737.0 | 765.3 | 804.6 | 885.7 |
| 4 | 240.5 | 304.5 | 349.1 | 394.9 | 437.0 | 471.4 | 500.4 | 527.7 | 553.7 | 577.6 | 594.7 | 610.9 | 629.3 | 648.9 | 668.6 | 690.5 | 712.9 | 737.2 | 765.7 | 804.9 | 888.8 |
| 5 | 240.9 | 304.4 | 349.3 | 395.0 | 437.1 | 471.6 | 500.4 | 527.7 | 553.9 | 577.6 | 594.8 | 610.7 | 629.3 | 648.7 | 668.6 | 690.2 | 712.6 | 737.0 | 765.5 | 804.9 | 886.2 |
| 6 | 240.6 | 304.3 | 349.0 | 394.6 | 436.7 | 471.2 | 500.2 | 527.3 | 553.4 | 577.3 | 594.4 | 610.5 | 629.0 | 648.7 | 668.4 | 690.0 | 712.6 | 736.8 | 765.2 | 804.7 | 887.6 |
| 7 | 240.7 | 304.4 | 349.2 | 394.8 | 436.7 | 471.2 | 500.0 | 527.3 | 553.3 | 577.4 | 594.5 | 610.4 | 629.0 | 648.5 | 668.3 | 689.8 | 712.4 | 736.7 | 765.0 | 804.0 | 886.8 |
| 8 | 239.5 | 304.1 | 349.1 | 395.1 | 437.3 | 471.6 | 500.4 | 527.5 | 553.4 | 577.3 | 594.6 | 610.4 | 628.9 | 648.5 | 668.3 | 689.9 | 712.3 | 736.6 | 765.1 | 804.4 | 885.5 |
| 9 | 240.5 | 304.5 | 349.3 | 394.9 | 436.9 | 471.5 | 500.5 | 527.6 | 553.6 | 577.3 | 594.6 | 610.5 | 629.1 | 648.7 | 668.7 | 690.4 | 713.0 | 737.2 | 765.4 | 804.4 | 885.8 |
| 10 | 240.8 | 304.6 | 349.4 | 395.1 | 437.3 | 471.8 | 500.8 | 528.0 | 553.8 | 577.6 | 595.0 | 611.1 | 629.5 | 649.2 | 668.9 | 690.5 | 713.1 | 737.2 | 765.3 | 804.7 | 887.7 |
| 11 | 240.8 | 304.4 | 349.4 | 394.8 | 437.1 | 471.7 | 500.7 | 527.8 | 554.0 | 577.7 | 595.0 | 611.1 | 629.7 | 649.3 | 668.9 | 690.4 | 712.8 | 737.0 | 765.1 | 804.4 | 885.4 |
| 12 | 240.9 | 304.5 | 349.1 | 394.9 | 437.0 | 471.5 | 500.4 | 527.6 | 553.4 | 577.4 | 594.6 | 610.4 | 629.1 | 648.5 | 668.3 | 689.8 | 712.4 | 736.6 | 764.7 | 803.8 | 885.0 |
| 13 | 241.0 | 304.6 | 349.4 | 395.3 | 437.3 | 472.0 | 500.9 | 528.1 | 554.0 | 577.6 | 594.8 | 610.5 | 629.0 | 648.5 | 668.3 | 689.8 | 712.4 | 736.8 | 764.9 | 804.0 | 885.4 |
| 14 | 241.0 | 304.5 | 349.1 | 394.9 | 436.8 | 471.4 | 500.5 | 527.8 | 553.8 | 577.7 | 595.0 | 611.0 | 629.6 | 649.0 | 668.8 | 690.5 | 713.0 | 737.4 | 766.0 | 805.2 | 886.7 |
| 15 | 240.7 | 304.5 | 349.4 | 395.2 | 437.6 | 472.1 | 501.1 | 528.1 | 553.8 | 577.5 | 594.7 | 610.7 | 629.0 | 648.9 | 668.6 | 690.4 | 712.9 | 737.4 | 765.7 | 805.4 | 888.4 |
| AVE | 240.7 | 304.5 | 349.2 | 394.9 | 437.0 | 471.5 | 500.5 | 527.7 | 553.6 | 577.5 | 594.7 | 610.7 | 629.2 | 648.8 | 668.6 | 690.2 | 712.7 | 737.1 | 765.3 | 804.6 | 886.5 |
| SDEV | 0.39 | 0.12 | 0.13 | 0.19 | 0.28 | 0.27 | 0.29 | 0.24 | 0.22 | 0.14 | 0.20 | 0.25 | 0.25 | 0.27 | 0.24 | 0.27 | 0.30 | 0.31 | 0.39 | 0.47 | 1.13 |
| RSD | 0.16% | 0.04% | 0.04% | 0.05% | 0.07% | 0.06% | 0.06% | 0.05% | 0.04% | 0.02% | 0.03% | 0.04% | 0.04% | 0.04% | 0.04% | 0.04% | 0.04% | 0.04% | 0.05% | 0.06% | 0.13% |
| Consensus | 239 | 304 | 349 | 393 | 435 | 469 | 499 | 526 | 552 | 576 | 594 | 610 | 629 | 649 | 669 | 690 | 712 | 736 | 764 | 803 | 887 |
| Difference | 1.71 | 0.45 | 0.21 | 1.94 | 1.99 | 2.53 | 1.47 | 1.69 | 1.64 | 1.52 | 0.73 | 0.72 | 0.24 | -0.19 | -0.41 | 0.22 | 0.75 | 1.06 | 1.35 | 1.59 | -0.50 |

Initial BP = 241°F

Final BP = 886°F

Ave. Sdev = 0.3°F

Ave. RSD = 0.05%

Ave. Difference from Consensus Value = 1.0°F



Broad Applicability - the Winery

- **Rapid “Dilute-and-Shoot” Automated Analysis of Ethanol and Fusel Oils in Wine and Distilled Spirits**



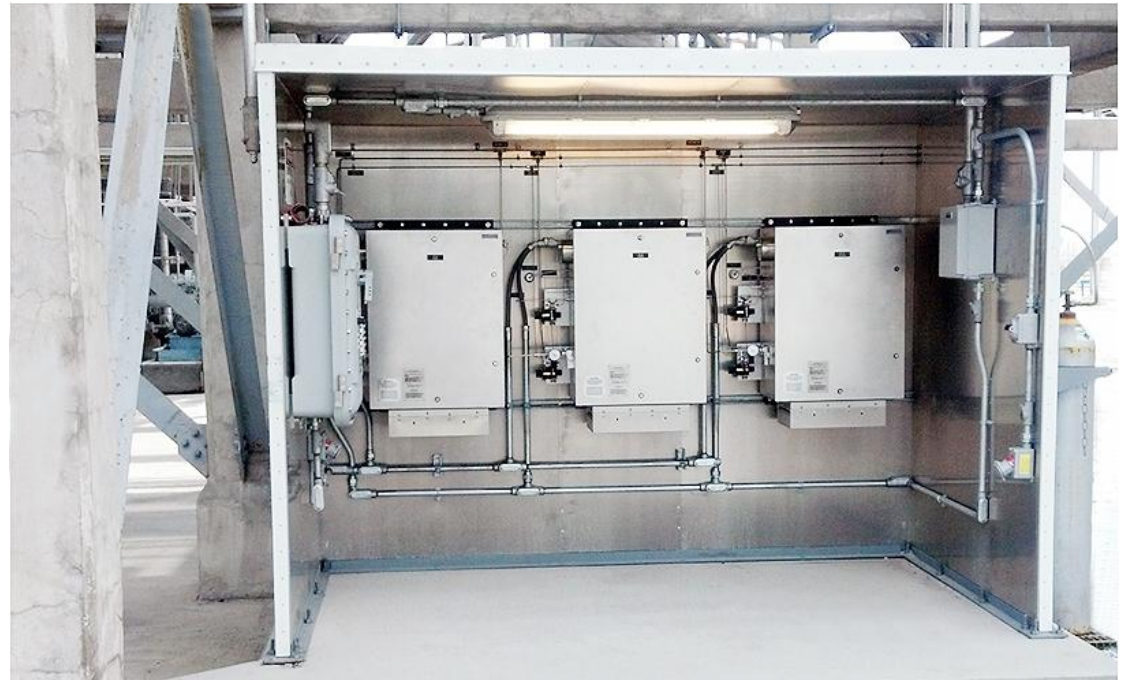
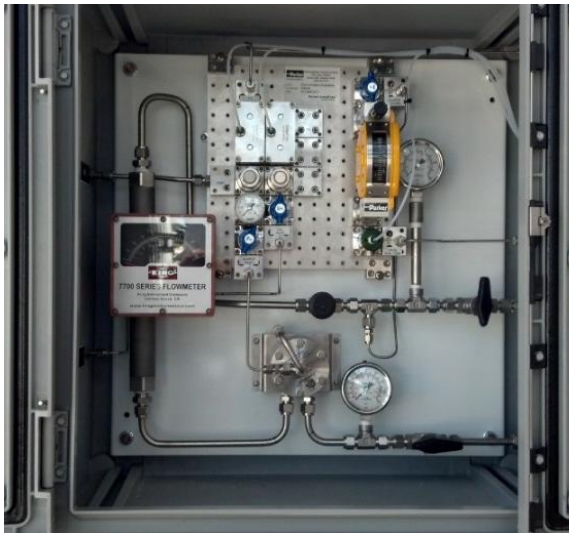
Broad Applicability - Oleo Chemicals

- *Improved Batch Process Throughput with Fast, Automated Food Grade Fatty Acid Endpoint Analysis*



Broad Applicability - Online Petrochemicals

- *Online Process Control using Modular Fluid Delivery and Fast & Micro Process Gas Chromatography: From the Sample Point to the DCS Connection*



Broad Applicability - Refineries

- *Refinery Support from the R&D Laboratory Perspective using Fast & Micro Gas Chromatography*

Refinery/Extended Natural
Gas Analysis



Simulated Distillation/
Boiling Point Distribution

Broad Applicability - Refineries

- **Fully Automated Fast & Micro Gas Chromatography for UltraFast Fuel Characterizations from C5 to C44: Significance & Performance Employing the New ASTM D-7798 Method**

ASTM D-7798/D-2887
Simulated Distillation



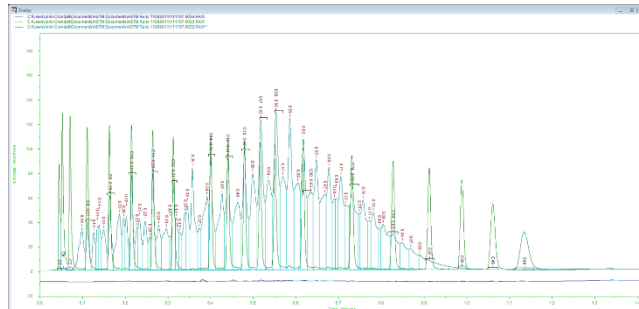
Broad Applicability - Transportable

- *Environmental Assessments using Fast & Micro Gas Chromatography in the Transportable World*

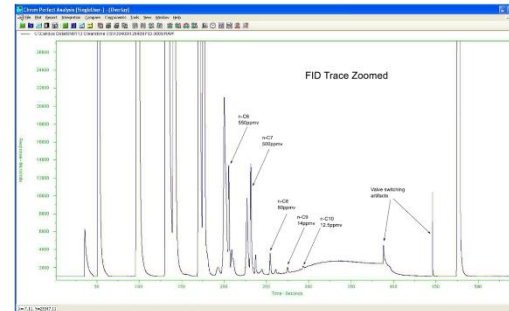


Broad Applicability - Upstream

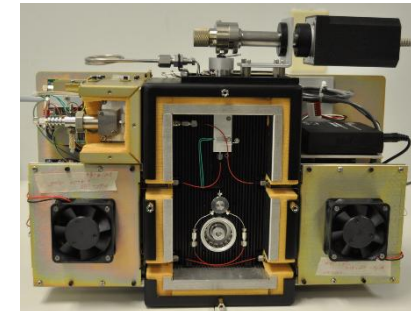
- **Fast & Micro Gas Chromatography and Its Use in Advanced Automated Technologies in the Upstream World**



Oil



Gas

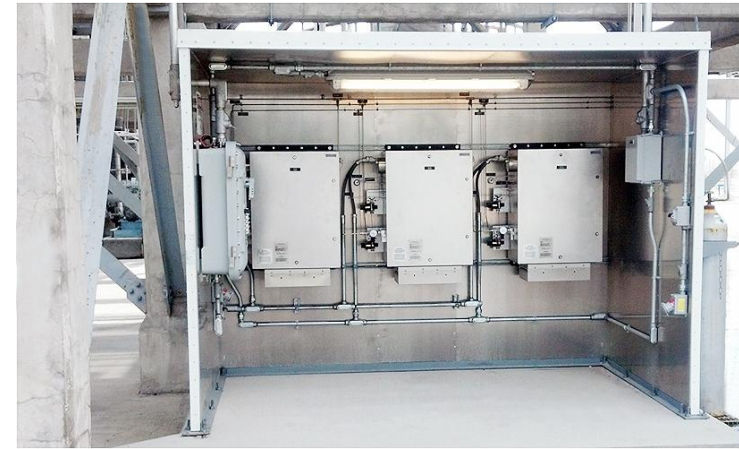


Transportable/Automated

The Supporting Cast

- *Recent Advances in Chromatography Data Systems Software: A More Complete Solution from the Winery to the Refinery to the Wellhead*
- *Advanced Data Processing Requirements for Fast Chromatography: Automation, Automation, Automation*
- *What Does It All Mean: Technological and Economic Impact of Fast & Micro Gas Chromatography*
- *Fast and Micro Gas Chromatography - A User Group Discussion*





Questions?

Let's get started!