



ADVANCEMENTS IN MICRO GAS CHROMATOGRAPHY (GC)

Fast Analysis of C1 to C8 hydrocarbons for mud logging applications within 2 minutes using a temperature programmable Micro GC Fusion®

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03/12/15 – 11:05 a.m.

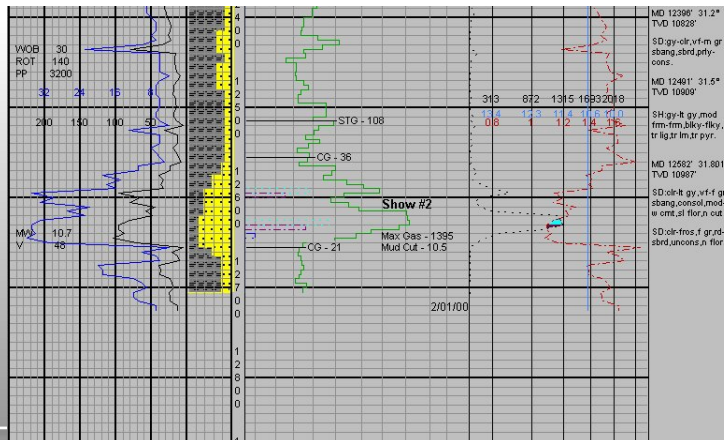
PITTCON 2015

Introduction

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- Gas Sampling at the Well Site
- Gas Chromatography (GC) for Hydrocarbon Analysis
- Instrumentation
- Instrument Features
- Methods and Data
 - C1-C5 in 34 seconds
 - C1-C8 in 2 minutes
- On-Site Tips for Successful Analysis
- Future Development

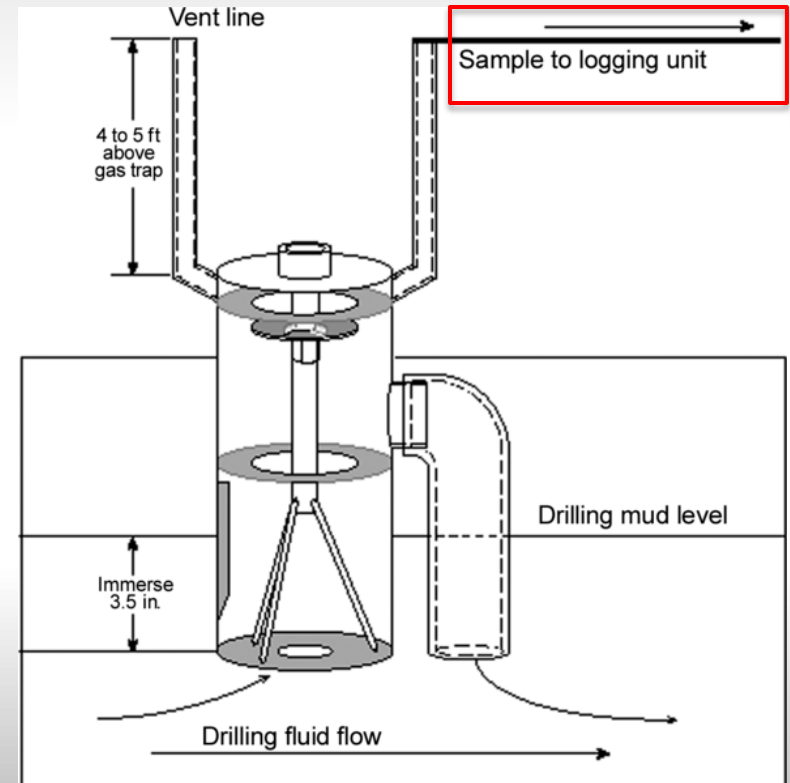
Mud Logging Introduction

- Mud logging is the creation of detailed reports of a borehole on a well site during active drilling
- Liquid, or “mud”, is used as the drilling fluid to bring gas, formation fluids, and rock cuttings to the surface for analysis
- A mud log provides key geographical information, including lithology, rate of penetration, and **hydrocarbon content**
- The concentration of C1-C8 compounds is critical and must be performed on a continuous, 24-7 basis



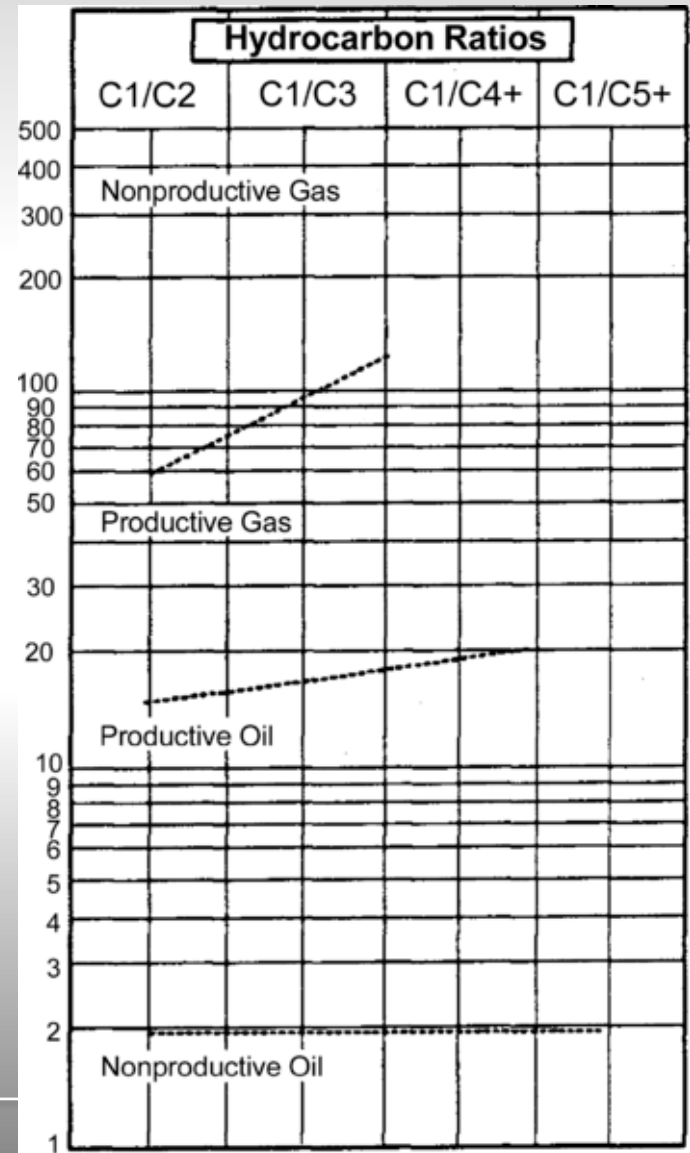
Gas Sampling at the Well Site

- A gas trap allows the mud to separate between the gas and liquid components
- The head space gas then goes to the gas analyzer
- Gas chromatography (GC) is often used to analyze the gas sample



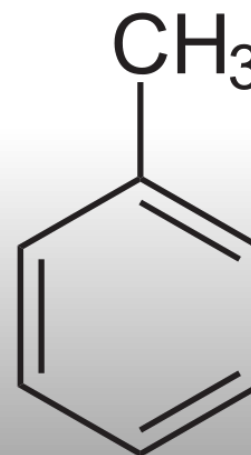
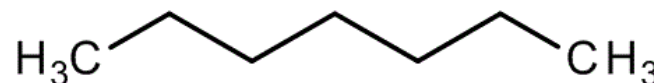
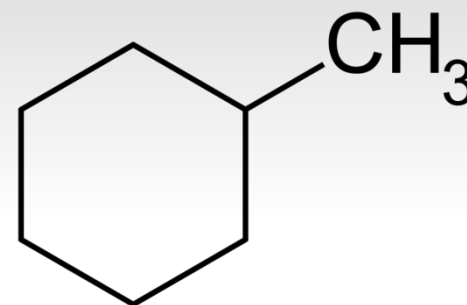
Gas Chromatography for Hydrocarbon Analysis

- GC has been used for mud logging for decades to characterize reservoir fluid composition
- Each reservoir is composed of different amounts of hydrocarbon species
- The amount and ratios of compounds help identify zones of producible oil or gas
- Other calculations are used for wetness (W_h), balance (B_h), and character (C_h)



Gas Chromatography for Hydrocarbon Analysis

- C1-C5 compounds are of main importance; however, analysis of heavier hydrocarbons is often desired
- Heavier hydrocarbons, such as methylcyclohexane and C7 help indicate the presence of liquid phases



Instrumentation

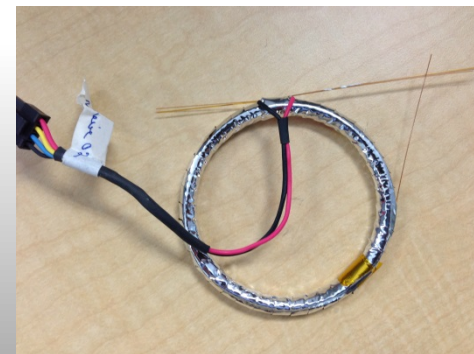
- Small, transportable GC with microelectromechanical systems (MEMS) technology
- Successor to the 3000 Micro GC
- Gas only analysis
- Thermal conductivity detector (TCD)



Micro GC Fusion

Instrument Features

- Temperature programmable columns
 - Minimizes carryover
 - Allows for more components to elute on a single column
 - Increases sample throughput
- Modular configuration
 - Each module contains an injector, column, and TCD
 - Easy module exchange in the field
- Web-based user interface
 - Can run on any operating system/platform
 - Can be operated from the front panel display
- Integrated sample conditioner (optional)
 - Reduces of sample pressures from up to 1000 psi down to approximately 5 psi
 - Filters out particulates
 - Heats the sample to 100 °C



Isothermal Method – C1-C5

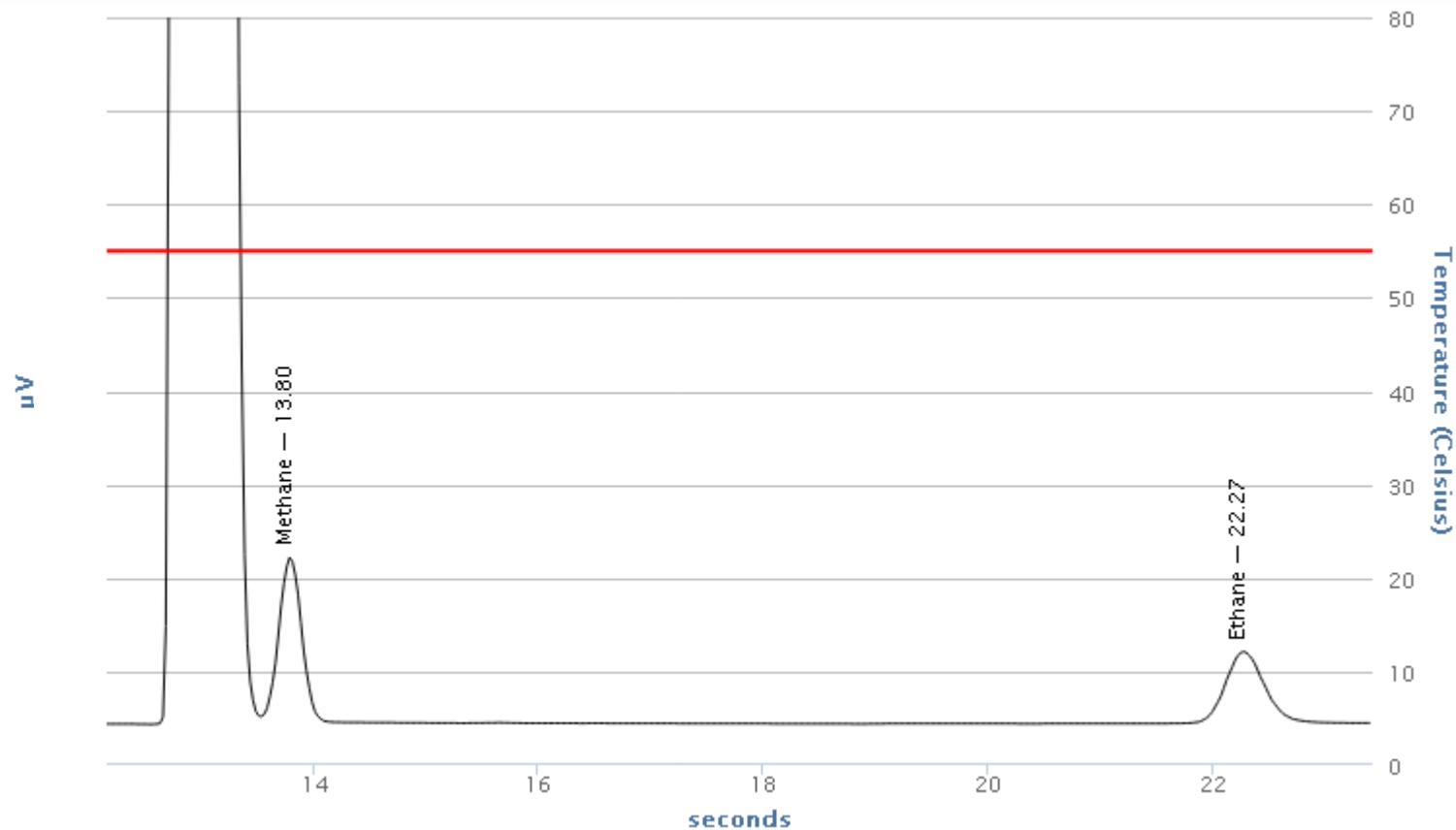
- 2 module system
 - Module A – 8m RT-Q-Bond, variable volume injector
 - Module B – 10m Rxi-1ms, variable volume injector
- A method was designed to run continuously, using **isothermal operation**
- Total cycle time was **34 seconds**

Calibration Standard

| Component | Amount (%) |
|-----------|------------|
| Nitrogen | 99.191 |
| Methane | 0.200 |
| Ethane | 0.101 |
| Propane | 0.100 |
| iC4 | 0.102 |
| nC4 | 0.103 |
| iC5 | 0.102 |
| nC5 | 0.101 |

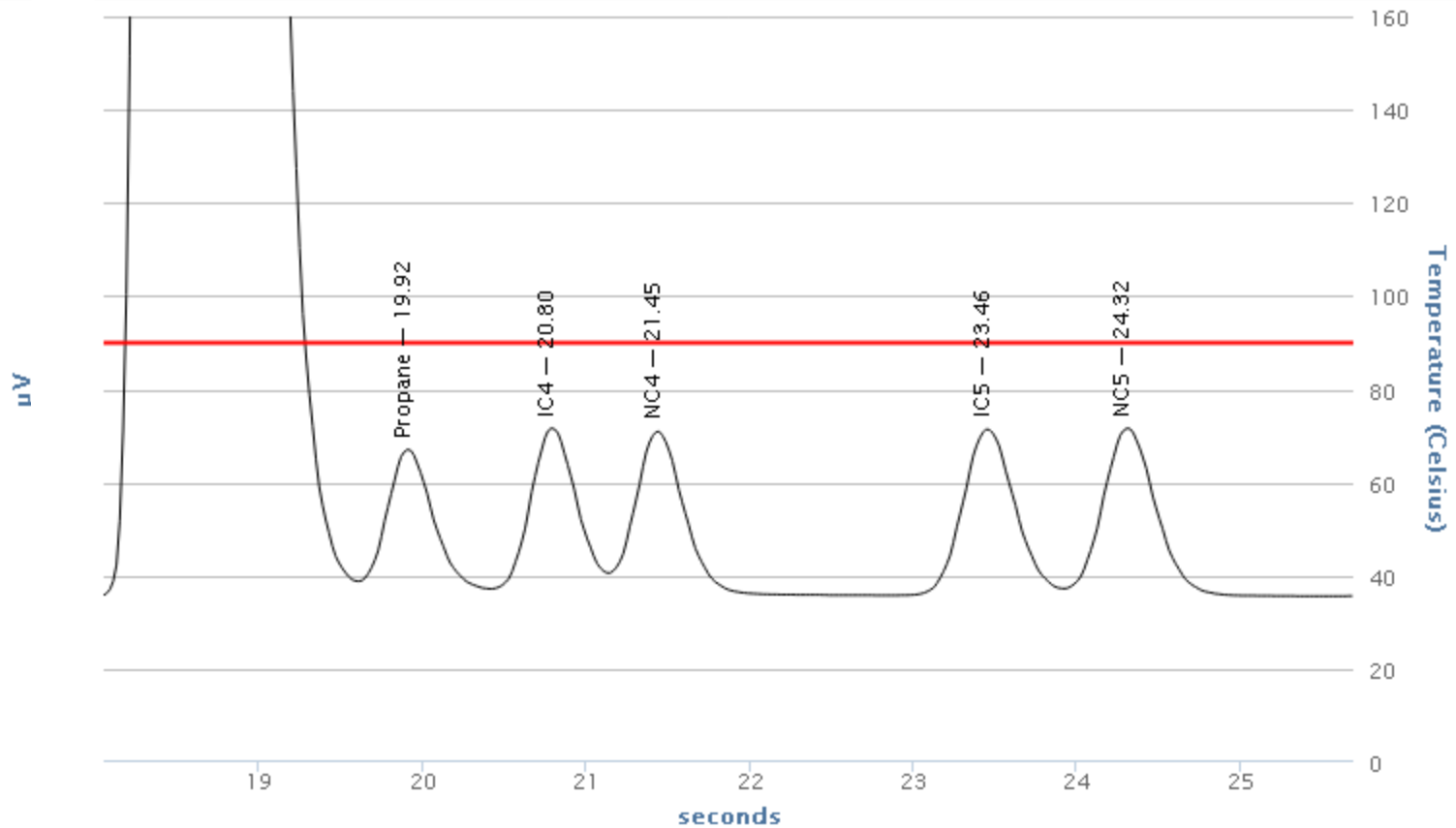
Chromatograms

- Module A: RT-Q-Bond
- Initial parameters used:
 - 55°C, 37 psi, 15 ms injection time, 5 second pump, Helium carrier gas



Chromatograms

- Module B: Rxi-1ms
- Initial parameters used:
 - 90°C, 33 psi, 15 ms injection time, 5 second pump, Helium carrier gas

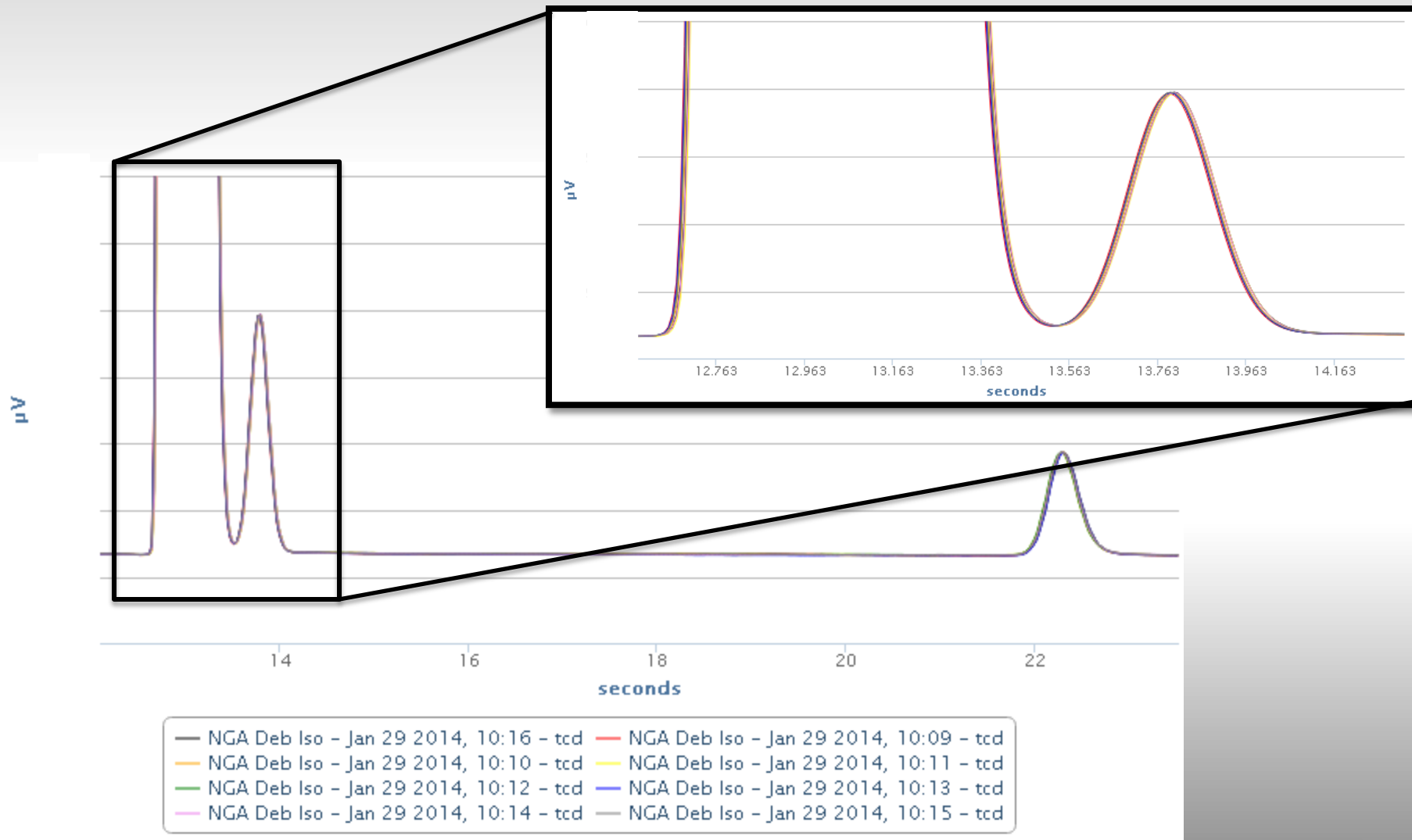


Repeatability

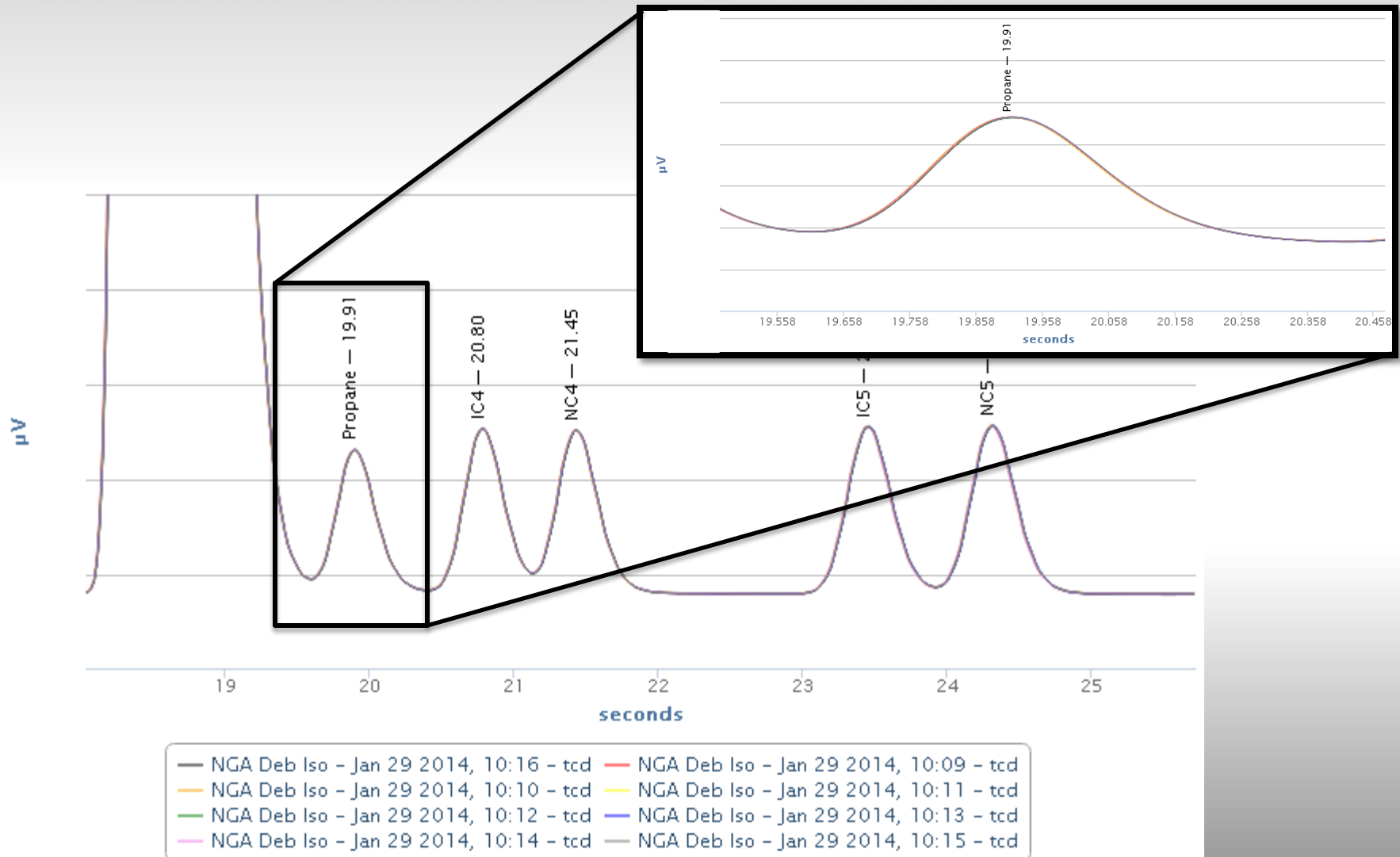
- Exceptional repeatability
 - Less than 0.1 %RSD for retention time
 - Less than 0.6 %RSD for area

| 1/29/2014 | Last 10 runs (10:09 a.m. to 10:16 a.m.) | |
|-----------|---|---------|
| | Area %RSD | RT %RSD |
| Nitrogen | 0.09 | 0.037 |
| Methane | 0.57 | 0.041 |
| Ethane | 0.51 | 0.049 |
| Propane | 0.05 | 0.000 |
| iC4 | 0.33 | 0.025 |
| nC4 | 0.46 | 0.024 |
| iC5 | 0.39 | 0.000 |
| nC5 | 0.33 | 0.021 |

10 Runs Overlaid – RT-Q-Bond

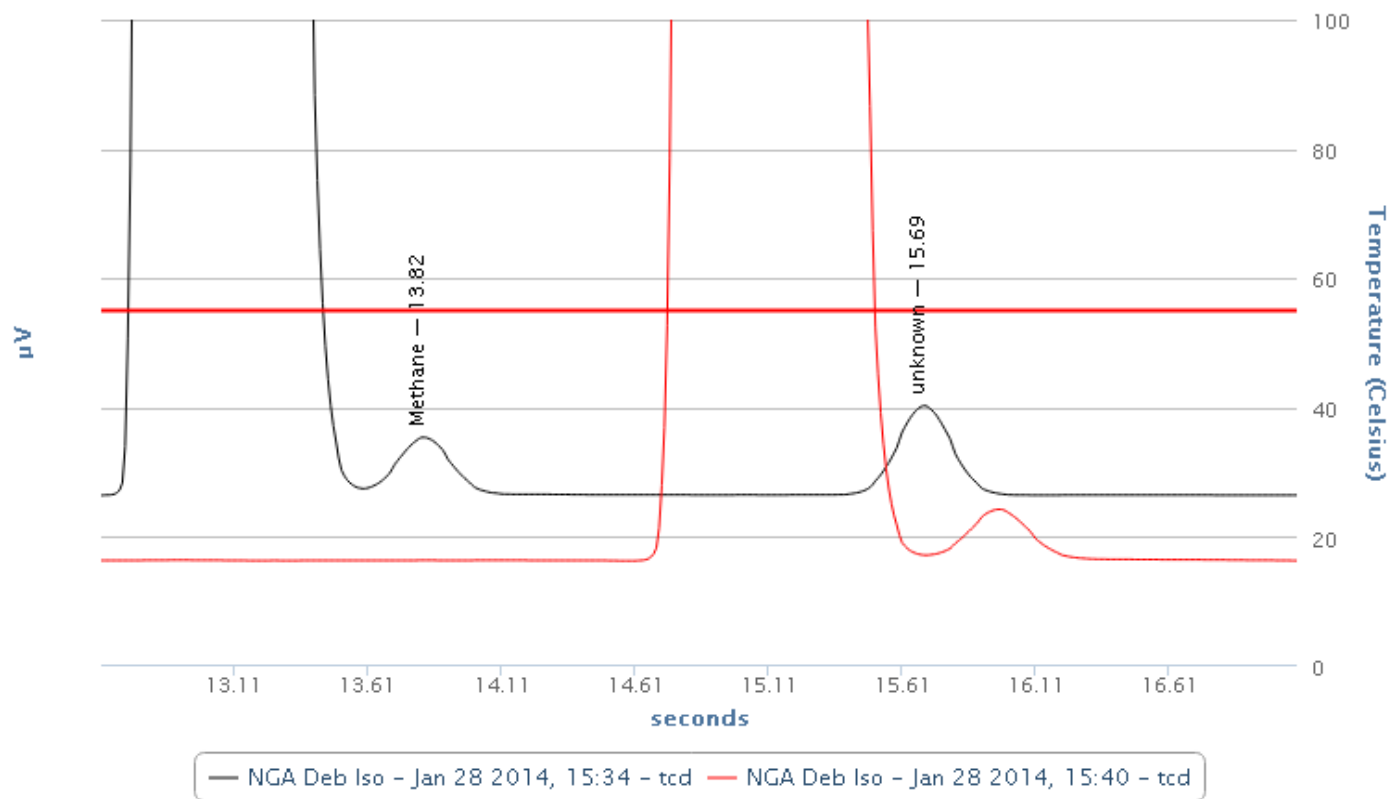


10 Runs Overlaid – Rxi-1ms



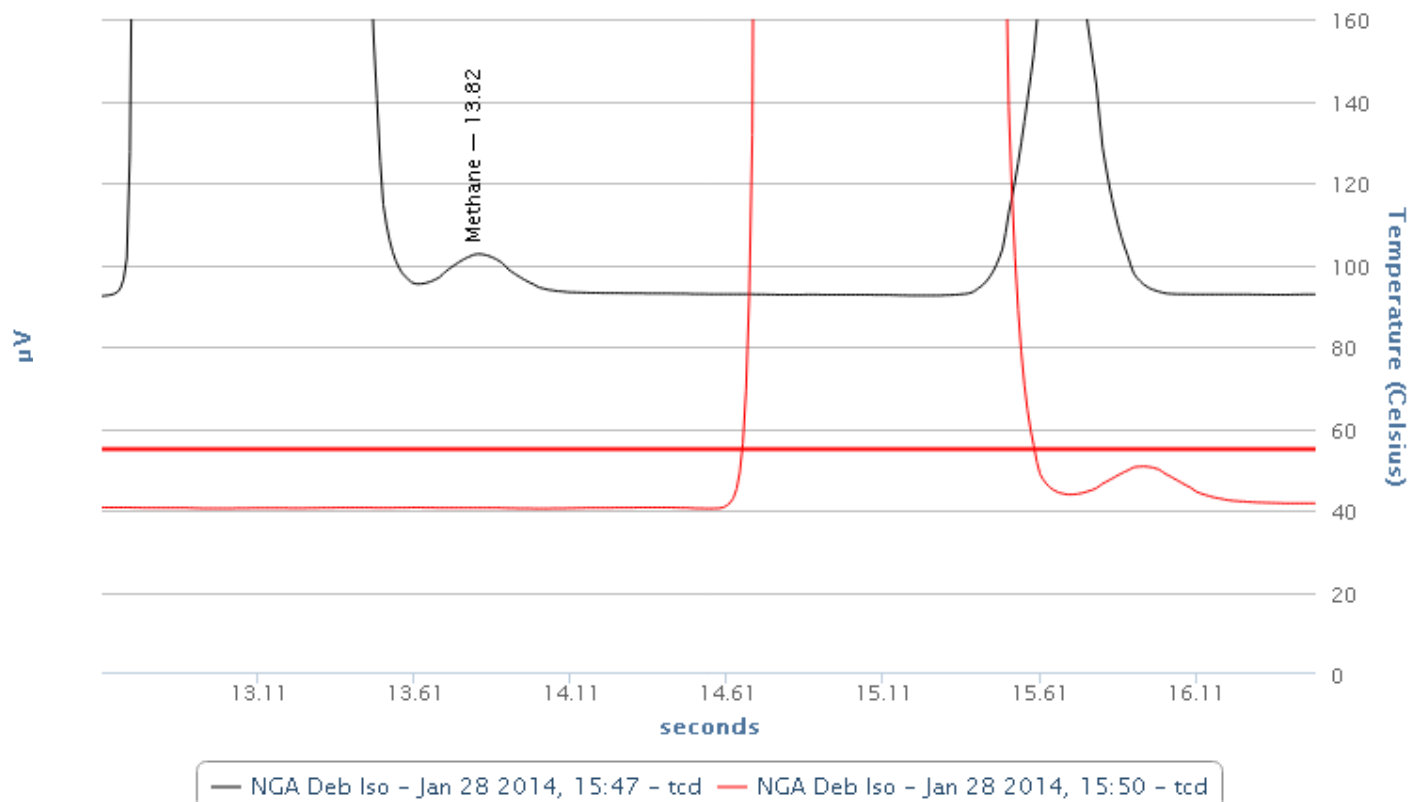
500 ppm Methane

- Module A: RT-Q-Bond - 37 psi and 32 psi
- Syringe dilution
- Reported concentration 0.051%



100 ppm Methane

- Module A: RT-Q-Bond - 37 psi and 32 psi
- Syringe dilution
- Reported concentration 0.014%



Temperature Ramped Method – C1-C8

- 2 module system
 - Module A – 8m RT-Q-Bond, variable volume injector
 - Module B – 10m Rxi-1ms, variable volume injector
- A method was designed to run continuously, using temperature programming
- Total cycle time was approximately 2 minutes

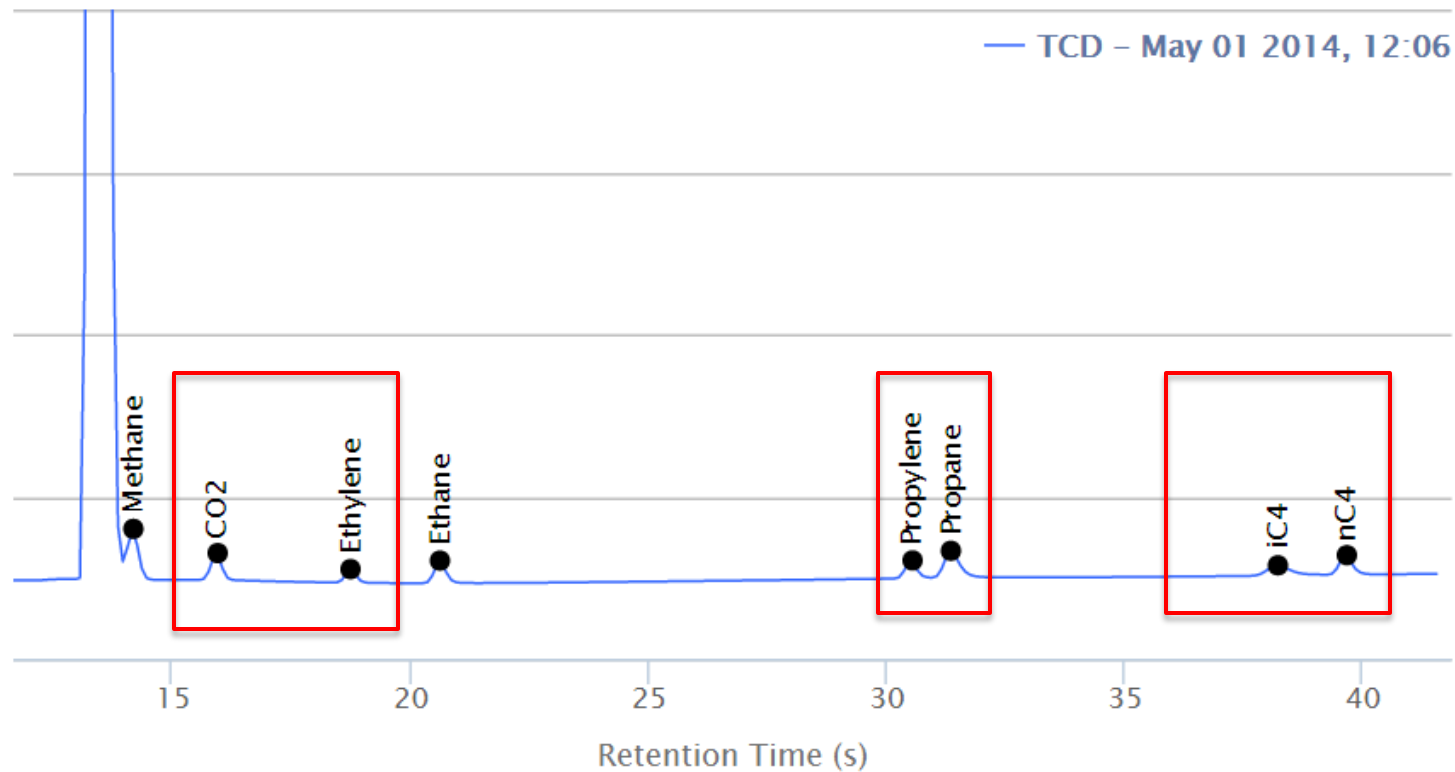
Extended Calibration Standard

| Component | Amount (%) |
|-----------------|------------|
| Nitrogen | 98.054 |
| Methane | 0.5 |
| CO ₂ | 0.197 |
| Ethylene | 0.095 |
| Ethane | 0.2 |
| Propylene | 0.103 |
| Propane | 0.197 |
| iC ₄ | 0.098 |
| nC ₄ | 0.098 |
| iC ₅ | 0.102 |
| nC ₅ | 0.102 |

| Component | Amount (%) |
|-------------------|------------|
| nC ₆ | 0.12 |
| Benzene | 0.021 |
| Cyclohexane | 0.032 |
| nC ₇ | 0.01 |
| Methylcyclohexane | 0.01 |
| Toluene | 0.011 |
| nC ₈ | 0.02 |

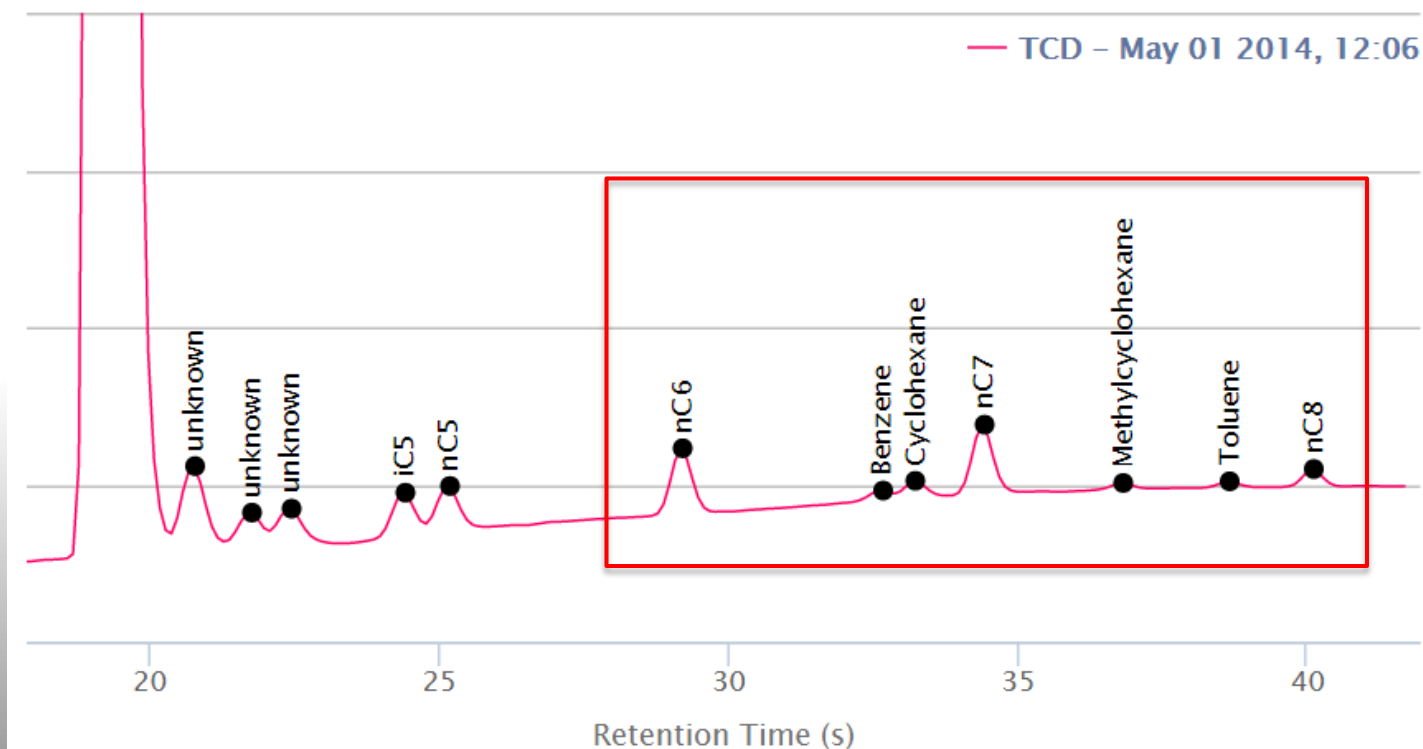
Chromatograms

- Module A: RT-Q-Bond
 - 55°C(15s hold)→220°C(10s hold)
 - 7°C/s, 35 psi, 30 ms injection time, Helium carrier gas



Chromatograms

- Module B: Rxi-1ms
 - 75°C(9s hold) → 220°C(12s hold)
 - 7°C/s, 33 psi, 80ms injection time, Helium carrier gas

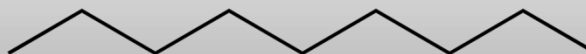
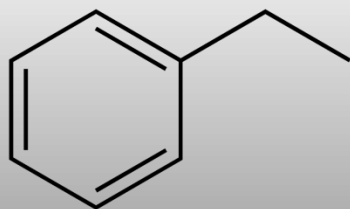


On-Site Tips for Successful Analysis

- Power
 - Access to stable power
 - Surge protector, UPS
- Heated transfer lines
 - To keep heavier hydrocarbons from condensing
- Gas trap stability
- Water traps, filters

Future Developments

- Method was optimized for speed
 - Aggressive nature of the ramp may not be optimized for instrument stability
- Future developments will include:
 - Backflush injectors to prevent carryover
 - Different combination of columns types/lengths
 - Analysis of ethylbenzene, nC9 with the same cycle time

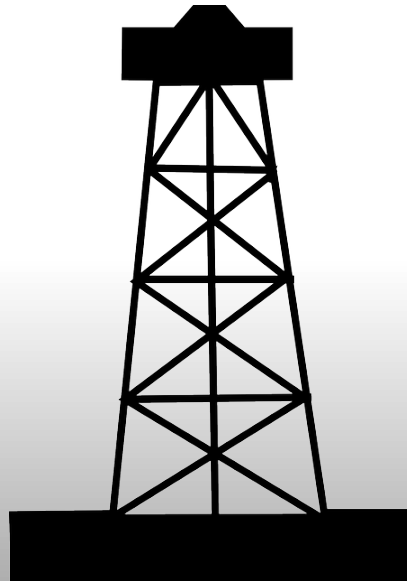


Conclusion

- Using isothermal operation, C1-C5 compounds can be analyzed within 34 seconds using Micro GC Fusion with excellent repeatability
- Using new rapid temperature ramping, C1-C8 compounds can be analyzed within 2 minute cycle time
 - Additional compounds, such as CO₂, ethylene, and propylene were also analyzed
- Future developments will focus on optimizing the configuration of Micro GC Fusion and the corresponding methods for the mud logging industry

References

- http://petrowiki.org/Formation_evaluation_during_mud_logging
- http://petrowiki.org/Mud_logging
- http://www.ppdm.org/wiki/index.php/Well_Operations_Reference_guide



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Questions?

