

# A Novel FTIR-GC/FTIR Detection Method as Applied to Process Monitoring of Carbon Dioxide Purity

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# Carbon Dioxide Purity

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- **Electronic grade (N5.0)**

- CO<sub>2</sub> for semiconductor FABs (chip cleaning, litho)
- Reactor refrigerants
- Laser gas

- **Impurity levels (SEMI)**

<i>Impurity</i>	<i>Maximum Acceptable Level</i>
Nitrogen	4 ppm
Oxygen	1 ppm
Methane	0.5 ppm
Moisture	2 ppm

*(for specialty cases, some VOCs are at ppb levels)*

# Carbon Dioxide Purity (cont'd)

- **Beverage grade (N3.0)**
  - CO<sub>2</sub> for carbonation of soft drinks and beers
- **Impurity levels (International Society of Beverage Technologists) ISBT**

<i>Impurity</i>	<i>Maximum Acceptable Level</i>
Carbon Monoxide	4 ppm
Oxygen	30 ppm
Volatile HCs	50 ppm
Moisture	20 ppm
Phosphine	300 ppb
Nitrogen Oxides	2.5 ppm
Acetaldehyde	200 ppb
Aromatic HCs	20 ppb
Sulfur Dioxide	1 ppm
Total Reduced Sulfur	100 ppb
Ammonia	2.5 ppm

# Electronic Grade Monitoring

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- **Need low detection limits (ppb to ppt)**
  - Some sacrifice has to be made
  - Preconcentrate (Thermal Desorption Tubes)
  
- **MAX™**
  - GC/FTIR

# What is MAX™?

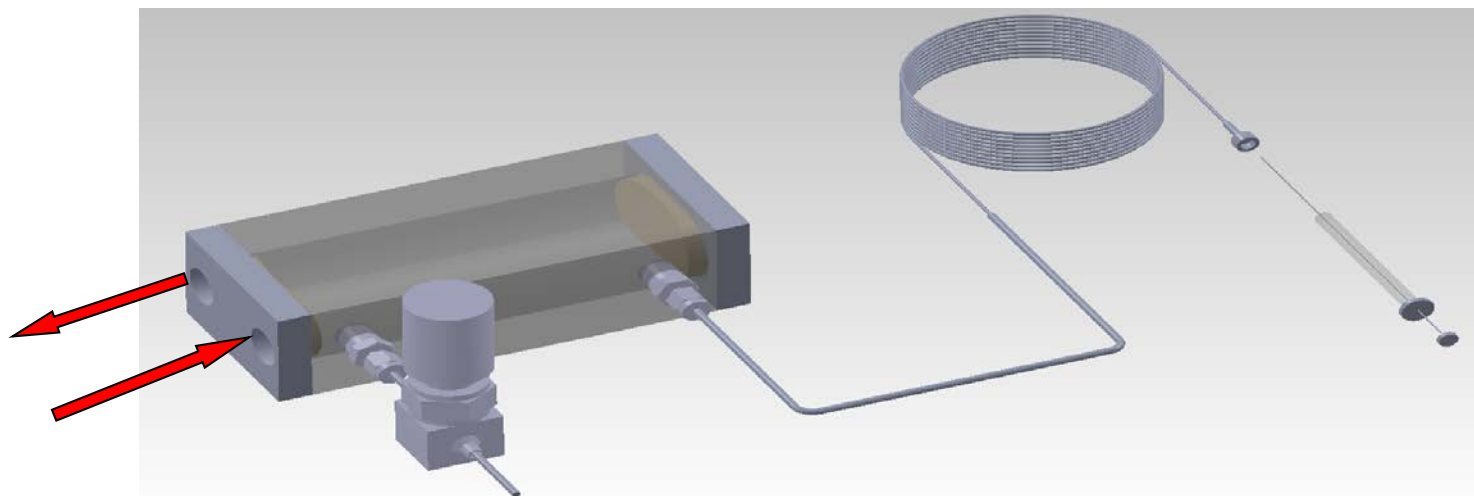
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## MAX is a Combination of GC and FTIR Gas Analyzer

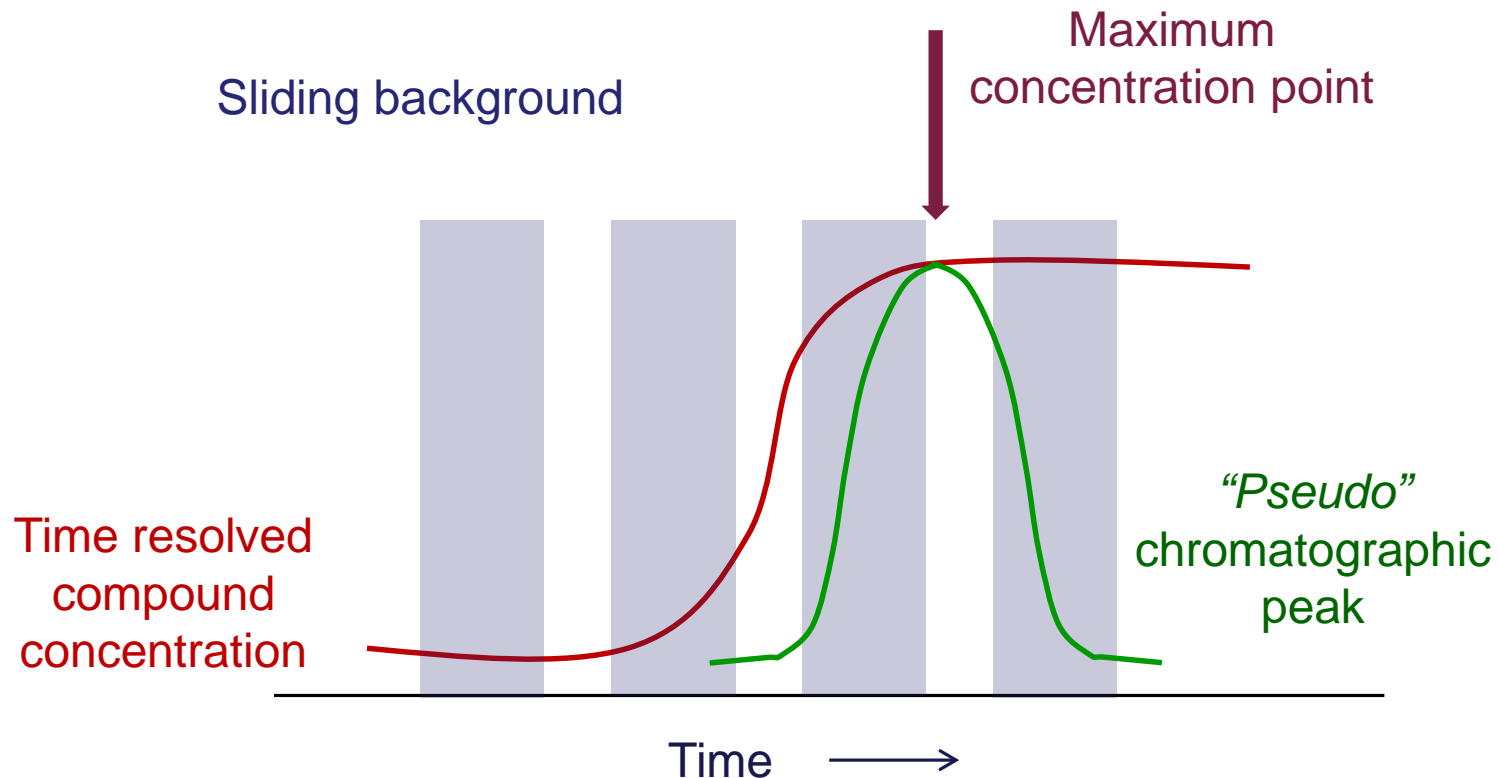
- **MAX uses a “large volume” multiple path IR gas cell**
  - Not the small “light-pipe” normally utilized in GC-FTIR products
  - Samples eluting from GC are collected into the gas cell (i.e., integrated)
  - Sensitivity is enhanced because the gas is measured once it has completely eluted from the GC. Think integration and signal averaging.
  - Previous eluting peaks do not interfere because they become part of the background spectrum over time
  - Sensitivities in sub ng have been achieved
  - Concentrations from % to ppt can be measured in the same sample

# How does MAX™ Work?

- Sample collected on thermal desorption tubes desorbed and passed directly onto chromatographic column (right)
- GC separates components, which enter/fill sealed multiple pass gas cell (left)
- The gas is then probed by an infrared beam (red arrows in and out)
- MAX™ integrates and then measures each fully eluted compound



# MAX™ Compound Determination



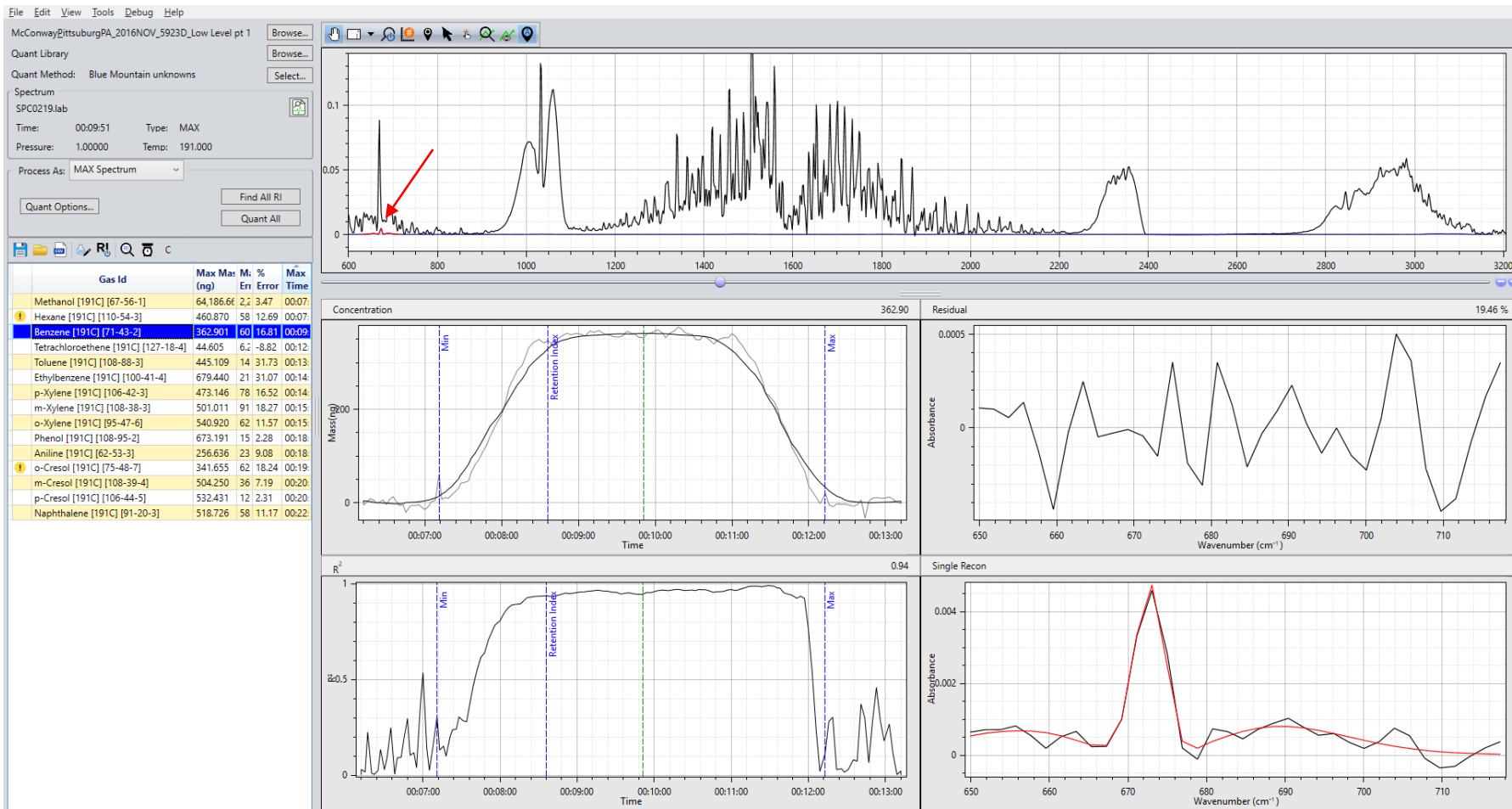
# Advantages of MAX™

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- **Absorption spectroscopy so calibrations are constant**
  - User never has to calibrate the system
  - Different GCs and separation devices have no affect on calibration (no splitting)
  - Significant spectral information so 20+ co-eluting compounds can be measured
  - Can use a second dimension (retention indices) for improving detections
- **No He or turbo pumps required**
  - Improved reliability and can be up and running during first day of install
  - Sample cannot damage instrument
- **Sensitivity increases with longer retention times**
  - Later eluting compounds normally lose sensitivity
    - With MAX, their detection limits are actually reduced due to longer integration times
  - Parts-per-trillion detection limits have been demonstrated on VOCs and SVOCs



# Typical MAX results



# Beverage Grade Monitoring

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- **Speed can't be sacrificed (>10 min analysis time)**
  - Measure for International Society of Beverage Technologists (ISBT) standard impurities
- **MAX™- Bev**
  - FTIR (real time analysis – 5 sec)
  - Increased pressure to increase sensitivity
    - Wouldn't help with GC/MS (have to increase split)
  - TRS analyzer (oxidizing oven)

# Prism's – MAX™ Bev Technology

- **MAX Bev Technology**
  - Two Technology Instrument
    - FTIR Gas Analysis
    - Sulfur Analyzer
  - Automated Gas Handling System
    - Multiple inputs associated with plant
      - Internal (Filters) / External – truck terminal
  - Output
    - 20+ species (cf. typical 4 or 5 species)
    - Inorganics/organics simultaneously

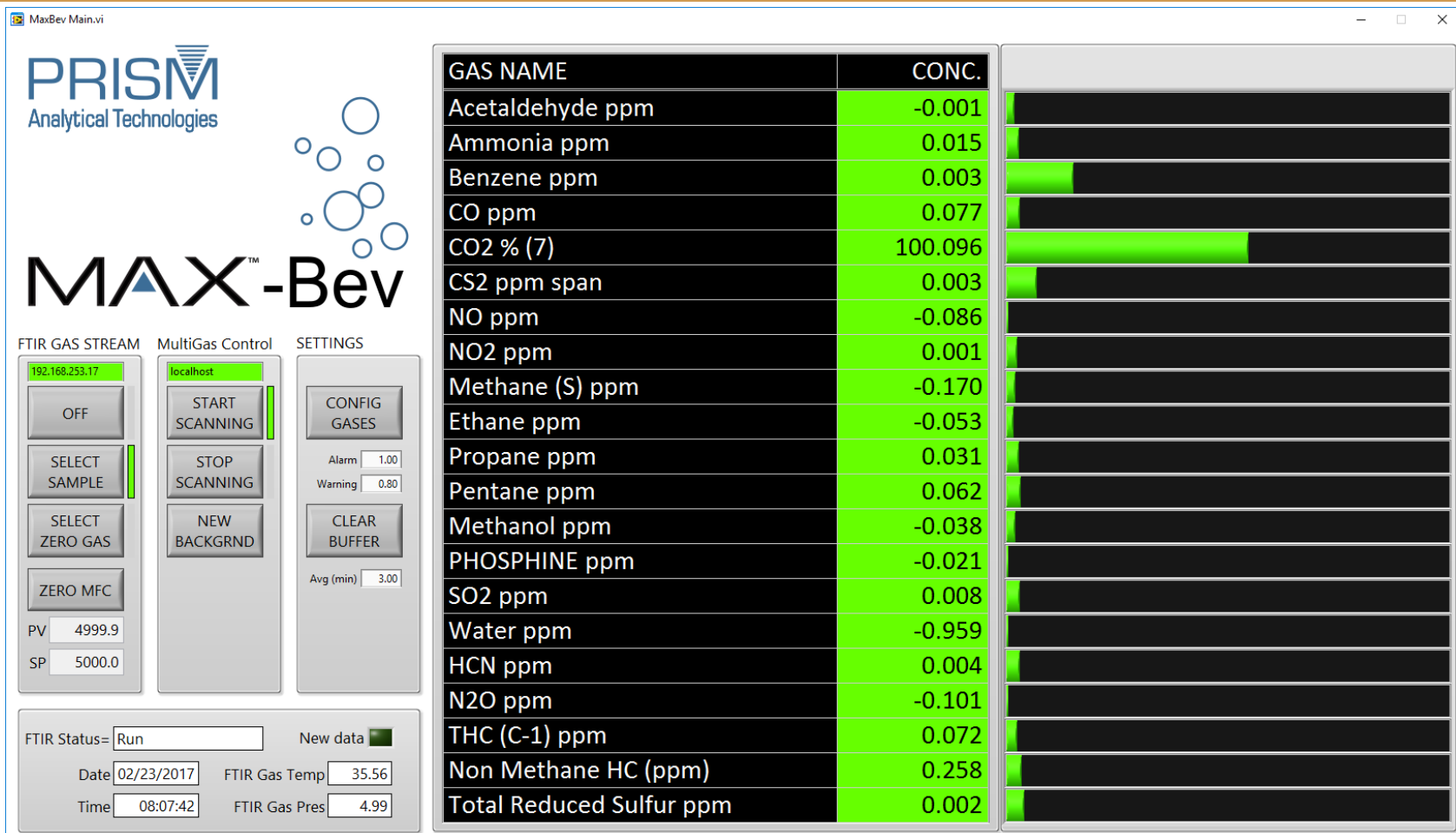


# Prism's – MAX™ Bev Technology

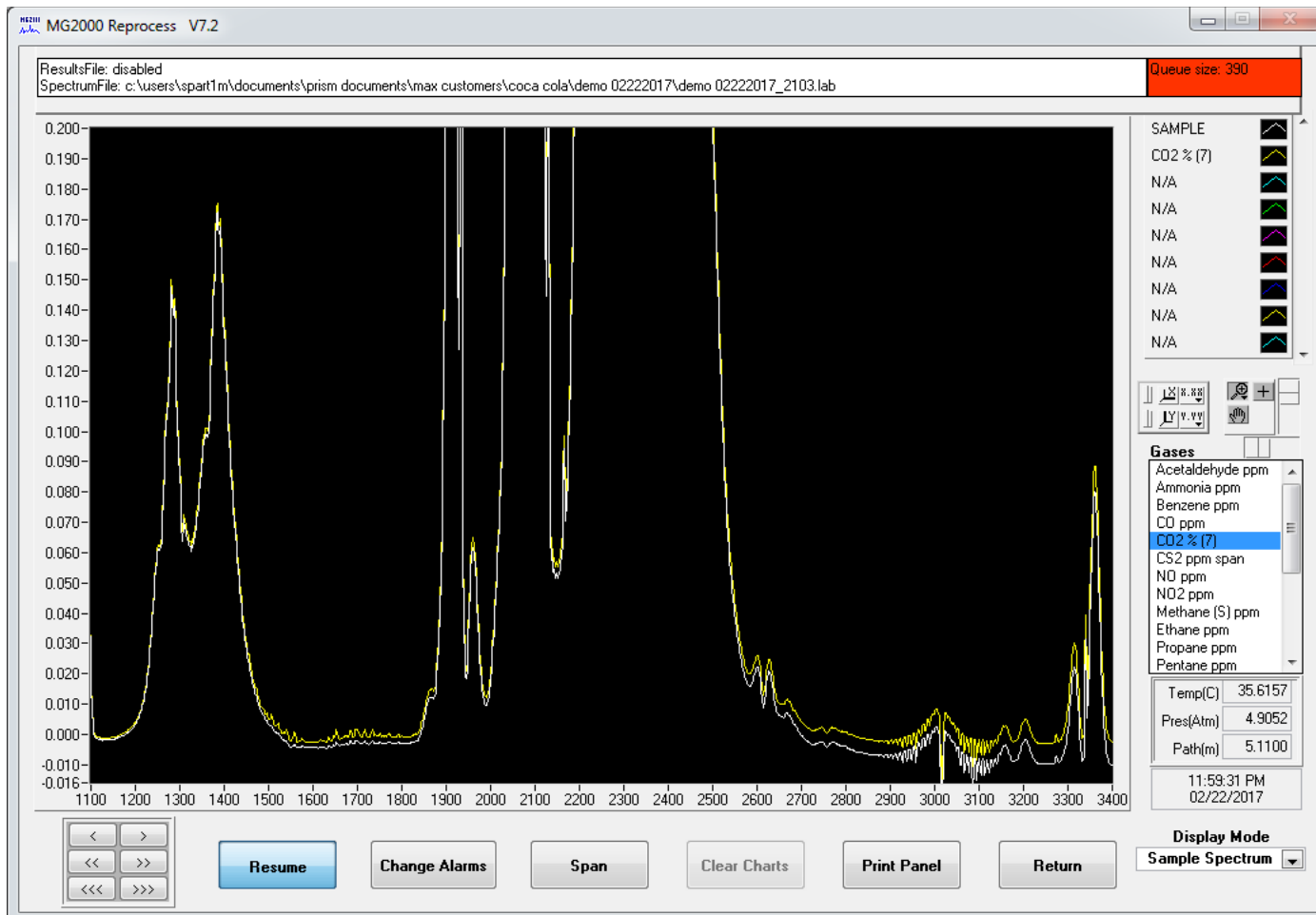
- **MAX Bev Technology**
  - FTIR Gas Analyzer
    - Specialized detector
    - Specialized pressurized system
    - Constant calibrations between instruments
  - Sulfur Analyzer
    - Total sulfur measurement
    - UV Fluorescence (SO<sub>2</sub>)
    - Permeation Bench for calibration monitoring
  - MAX Bev Gas Delivery System
    - 5,000 sccm MFC
    - Gas cell back pressure
    - Gas switching valves



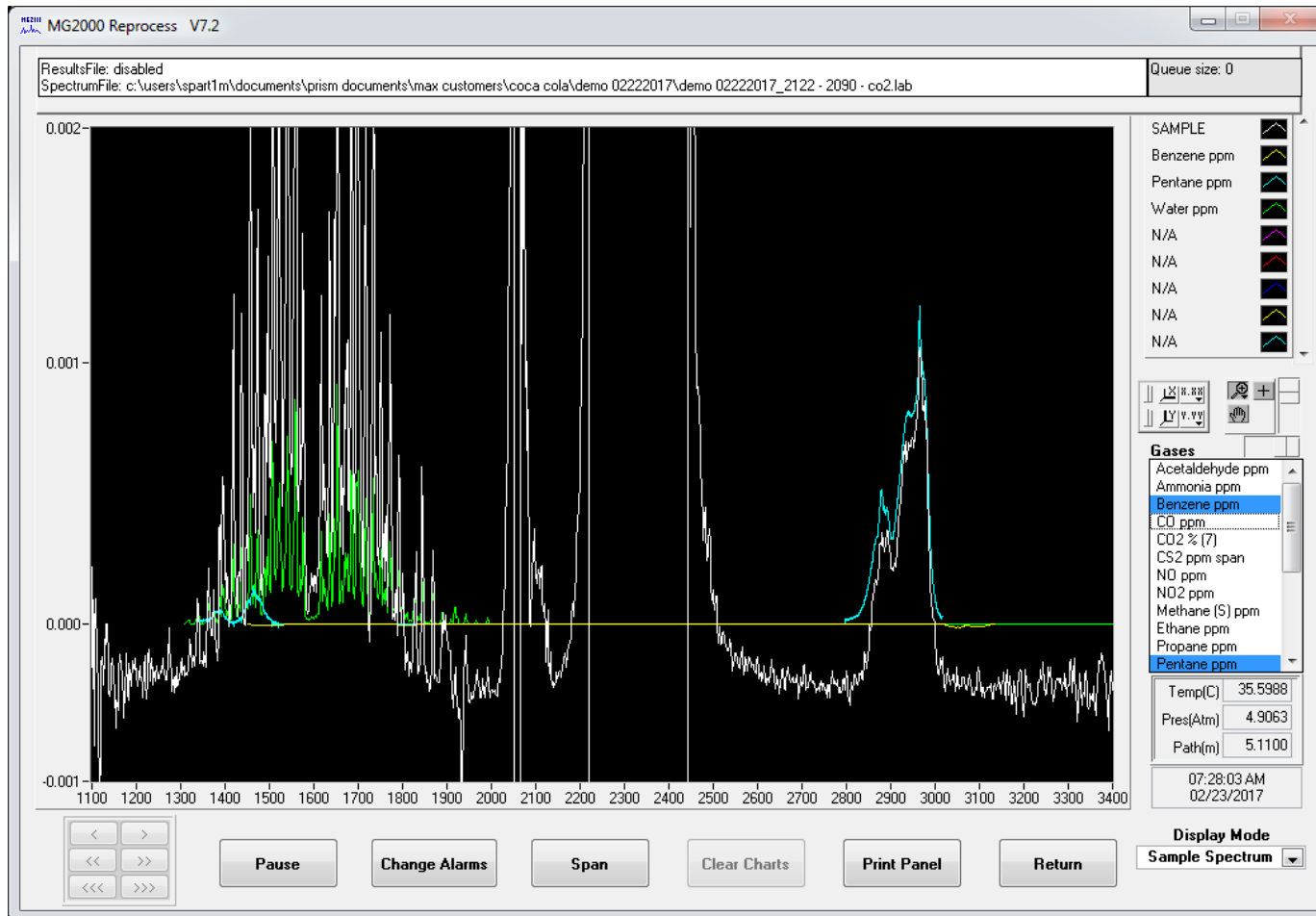
# Prism's – MAX™ Bev Technology



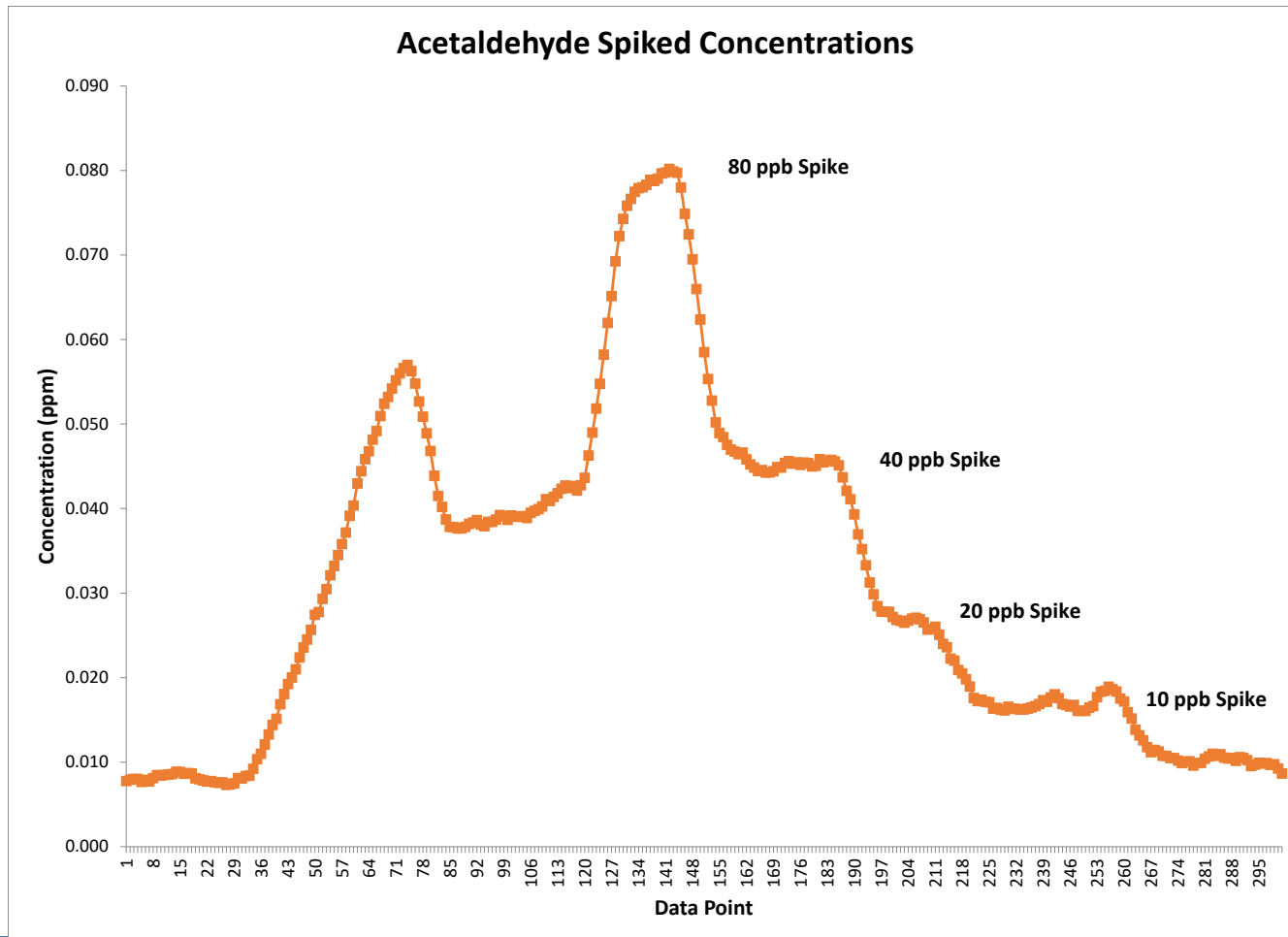
# Prism's – MAX™ Bev Technology



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# Questions?

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**See Prism at Booth #1145**