Lotus Consulting presents:

Micro Sulfur System (MSS)

The Micro Sulfur System from Lotus Consulting provides impressive detection of trace volatile sulfur compounds in ambient air samples. The system features the Scion 456 Gas Chromatograph with built-in high performance sample concentrator and a sensitive pulsed flame photometric detector (PFPD). This system handles both pressurized canisters and Tedlar bags without hardware changes.

The fully automated system is designed to detect volatile sulfur compounds. Samples are loaded through an optional 16-position automated sampler and trapped onto a low-volume cold trap with a mass flow controller (MFC) setting the sample size. All sample lines are Silcosteel treated for inertness. A proprietary trap material permits full recovery of the very reactive sulfur gases.

Detection of sulfur compounds in ambient air is very difficult due to the interaction of these chemicals with the common materials normally employed in gas chromatographic analyses. For example, hydrogen sulfide at low levels is nearly completely lost when exposed to most metal and glass surfaces. Special precautions are required to ensure full recovery.

In addition, usual sulfur detectors, such as flame photometric or chemilluminescence, are not sensitive enough to detect ambient levels of sulfur components via direct injections of 1-ml samples. Large volume samples must be concentrated into a small volume to enhance detection, especially with the flame photometric detector, as its response is proportional to the square of the concentration. To enhance response with this detector by a factor of ten, the concentration volume loaded must be increased 100 times. All of these operations utilize nearly all of the powerful and comprehensive capabilities of the Scion 456 and Workstation. Such a complex analyzer requires assurances that the data is valid, that the system is fully functional and easy to use, and that the ultimate performance is achievable.

SOLUTION TO DIFFICULT ANALYTICAL PROBLEM…

Clean System Blanks
- No thermal breakdown products from trap material
- Maximum temperature limit of 450 °C
- All valves are heated; limited to 225 °C max
- Cryogenic cleansing of purge gas – vented after each cycle

Elimination of Interfering Artifacts
- Inert trap standard
- No thermal breakdown that would yield interfering peaks
- Trap temperature limit to 450 °C

Sharp Chromatographic Peaks
- Minimum distance from trap to column (~15 cm)
- Cryofocus trap isolation during trap heating
- Columns attached directly to final valve
  - minimum effect of extra-column volumes at critical chromatographic point
- Trap volume – ~600 microliters

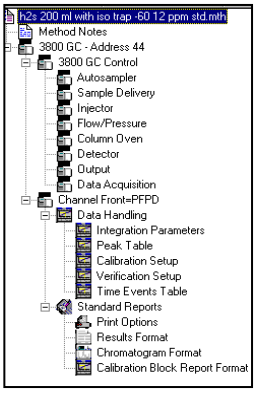
Accurate Measure of Sample Volume
- Sample flow to vent just before trapping
  - Stabilizes MFC
  - Sweeps sample lines with new sample
- Volume-measuring flow path swept with nitrogen prior to trap heating
- Accurate volumes from 5 ml to 2400 ml
- Sample loading independent of canister pressure

Minimal Carry-over
- Traps continuously purged with nitrogen when sample not loading
- Sample lines swept to vent with new sample just before trapping
- Carry-over « 0.1 %

Retention Time Reproducibility
- True electronic **flow** control - not pressure control
- Reproducible + accurate control of column oven temperature
  - proportional control (PID)
  - platinum probe (RTD)
- Typical RT reproducibility - < 0.03 minutes

Quantitation Reproducibility
- Column leaks detected with pressure monitoring
- Measuring flow path swept with nitrogen prior to trap heating
- Typical area reproducibility - < 3 %

Simplified Method Execution
Single run method contains all operating parameters, including cold traps ("Injector"), valve actuations ("Sample Delivery"), compound table, computation entries and report formatting. Simply activating this single method sets up the complete system, to minimize operator errors.



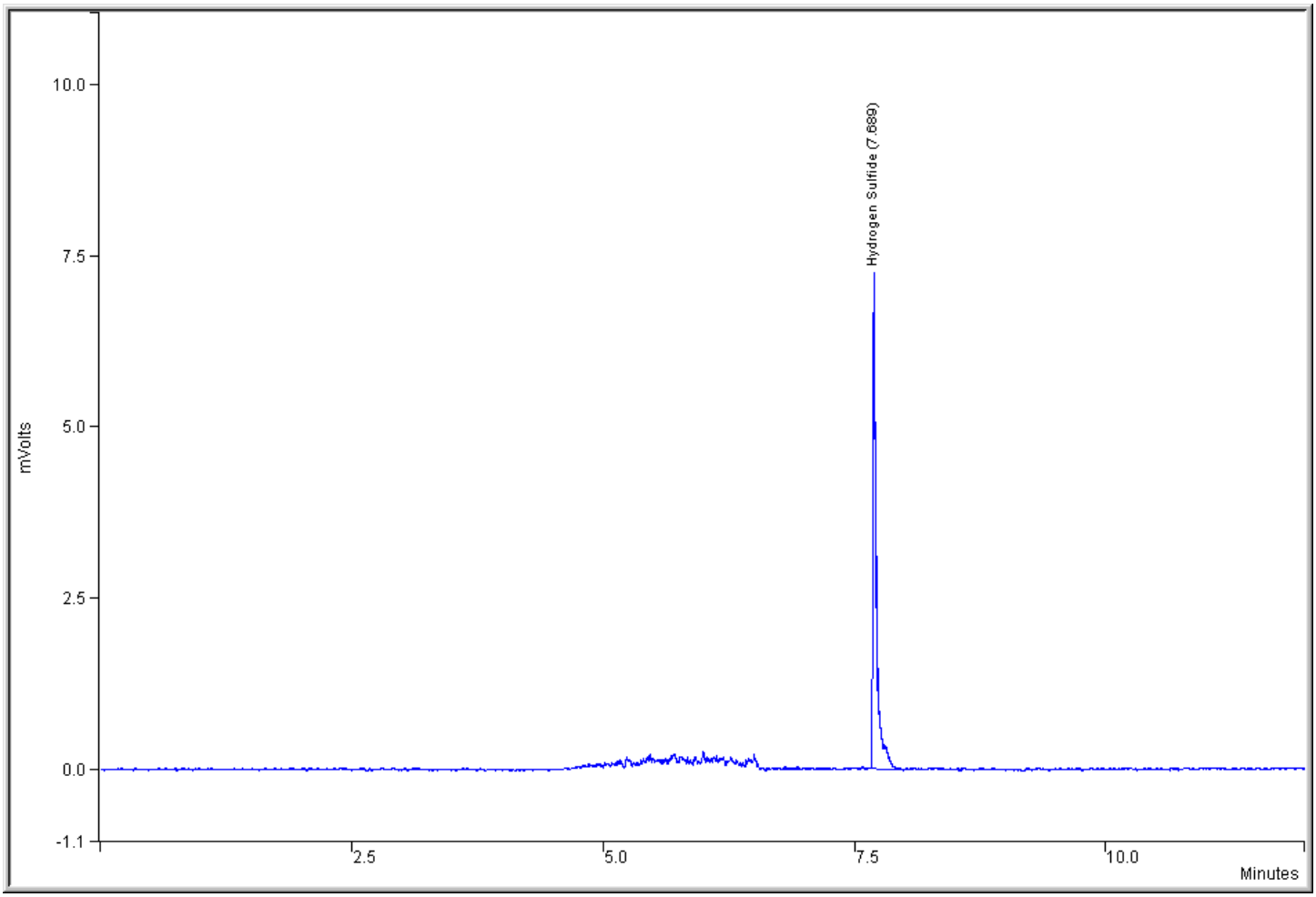
Monitoring of Operations
- True electronic **flow** control for columns – not pressure control
  - Generated backpressure becomes a diagnostic
  - Flow remains constant throughout run without computations/fudging
- Both analog gauges and digital displays for column pressure
- Full electronic flow control of all detector gases
- Flows/pressures documented in results report
- User-specified temperature limits for all thermal zones
- Visual indication of sample loading
- Complete system status with developing chromatograms on one screen

Data Processing
- Single stored data file contains raw chromatographic data, final report, complete run method stream position, run log and error messages
- Data collection, report generation, system control operate in Win 7.
- No overprinting of retention times on chromatogram
- Edit/lock calibration coefficients
- View/edit calibration curves
- Batching printing of reports from Windows Explorer
- Multi-level security with passwords
- Peak names to 39 characters; first 12 printed in reports

Data Integrity
- No overwriting of data files
- Operator cannot change Sample ID after collection
- Operator cannot change Date/Time of injection
- Cannot alter log files after collection
- Cannot change sample notes after collection
- All calibration data is archived with every raw data file
- Message log contains compete listing of system operations
- Instrument run log documents operating conditions
- File names can be labeled automatically with sample ID, injection date/time and module source as variables
- File names can be 255 characters long

Options
- Addition of other detectors, such as Flame Ionization Detectors with simultaneous detection through a separate sampling stream
- Pressure station to bring canisters above atmospheric pressure for proper loadings
- Automatic insertion into SampleList of pressure station readings before and after pressurization for dilution corrections
- Canister cleaner with single high capacity, non-oil pump

Performance

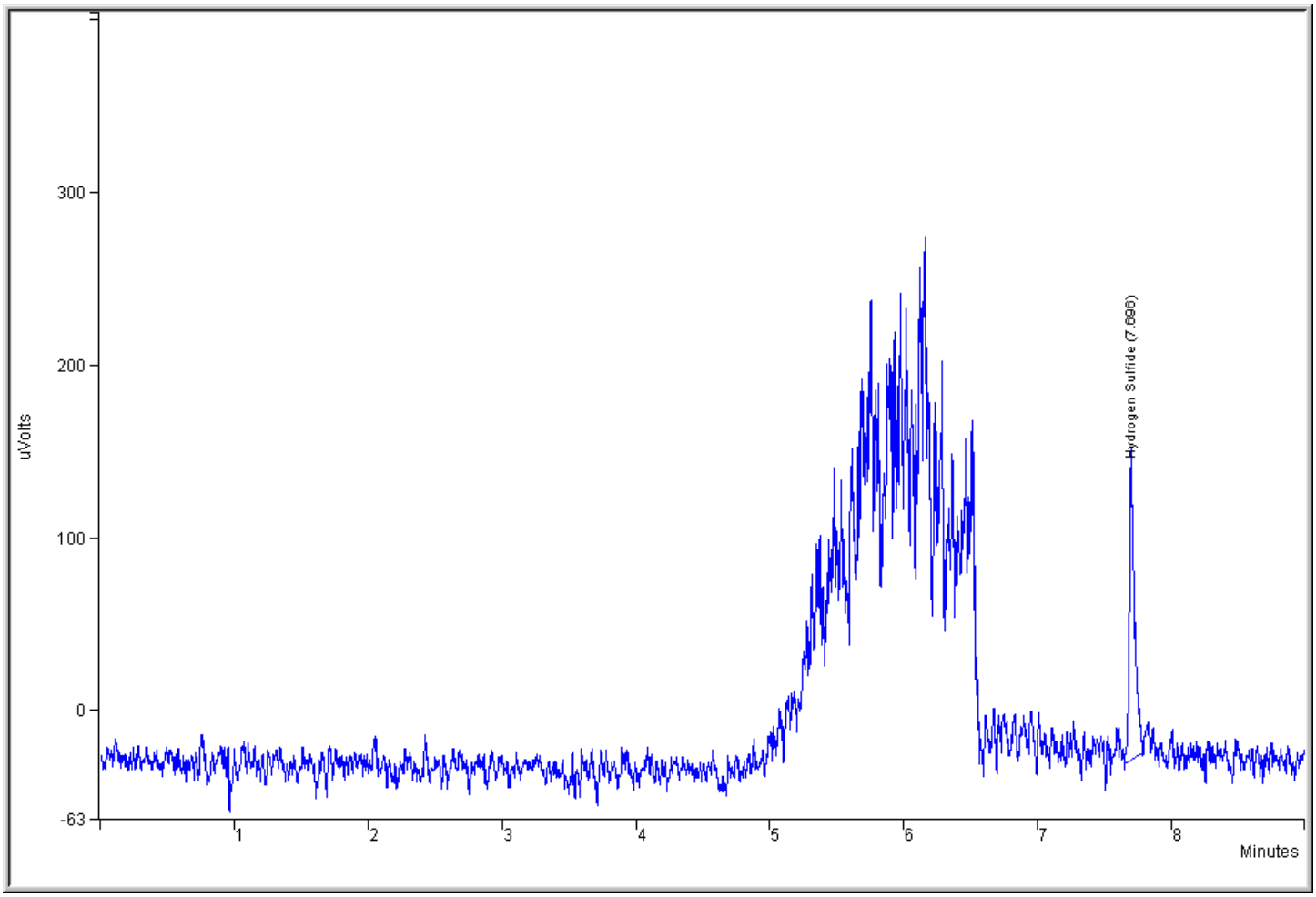


Typical Chromatogram of 1.5 ppb Hydrogen Sulfide Standard in Nitrogen

Sample volume basis: 200 ml.
Sample is 25 ml loading of 12 ppb standard.
Column: Agilent Chrompak CP-Sil 5 CB, 0.53 mm ID, 30 meters, 5 µ film.
Column temperature: 30 ºC. Column flow: 10 ml/min.
Trap: -160 ºC, hold for 6.1 minutes, ramp to 200 ºC at 200 ºC/min.
Detector temperature: 200 ºC.
Detector range: 10.
Sampling portion of chromatogram: 0 to 6.5 minutes
Chromatography starts at 6.5 minutes.

Instrument – Scion 456 Gas Chromatograph
Detector – Pulsed Flame Photometric Detector (PFPD) with DEFC
Sampling System – Cryogenic Concentrator with Fully Automated Valves for unattended measurements

…and More Performance



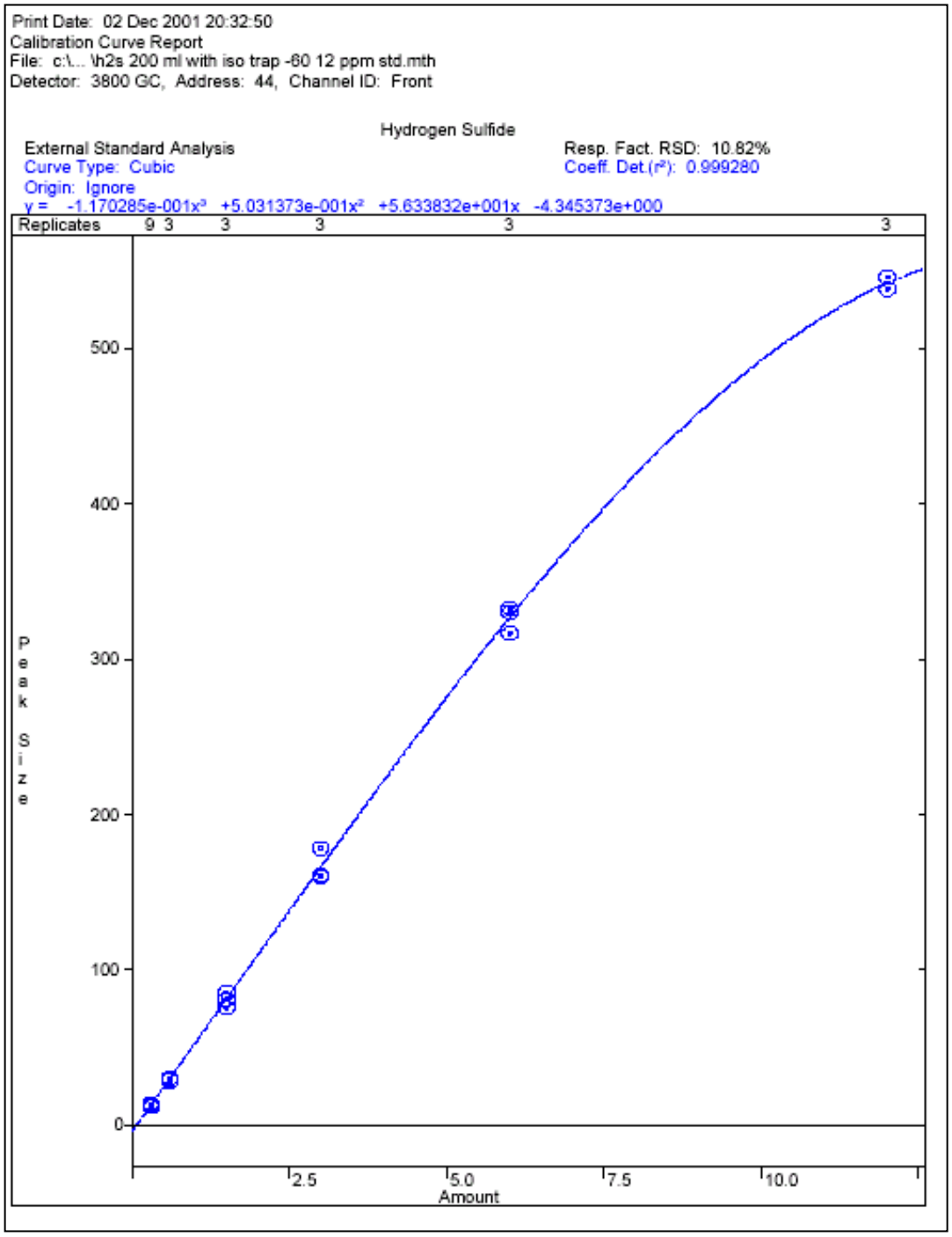
Chromatogram of
0.3 ppb Hydrogen Sulfide Standard in Nitrogen

Conditions are same as above, except sample is 5 ml loading of 12 ppb standard.

Peak Height (µV)	Square Root of Peak Height
163	12.77
181	13.45
160	12.65
178	13.34
149	12.21
163	12.78
164	12.81
151	12.29
160	12.65
Average Square Root of Peak Height	12.77
3 X RSD	1.24
Concentration Equivalent to 3 X RSD	.029 ppb V/V

Measured Detection Limit – 0.03 ppb V/V

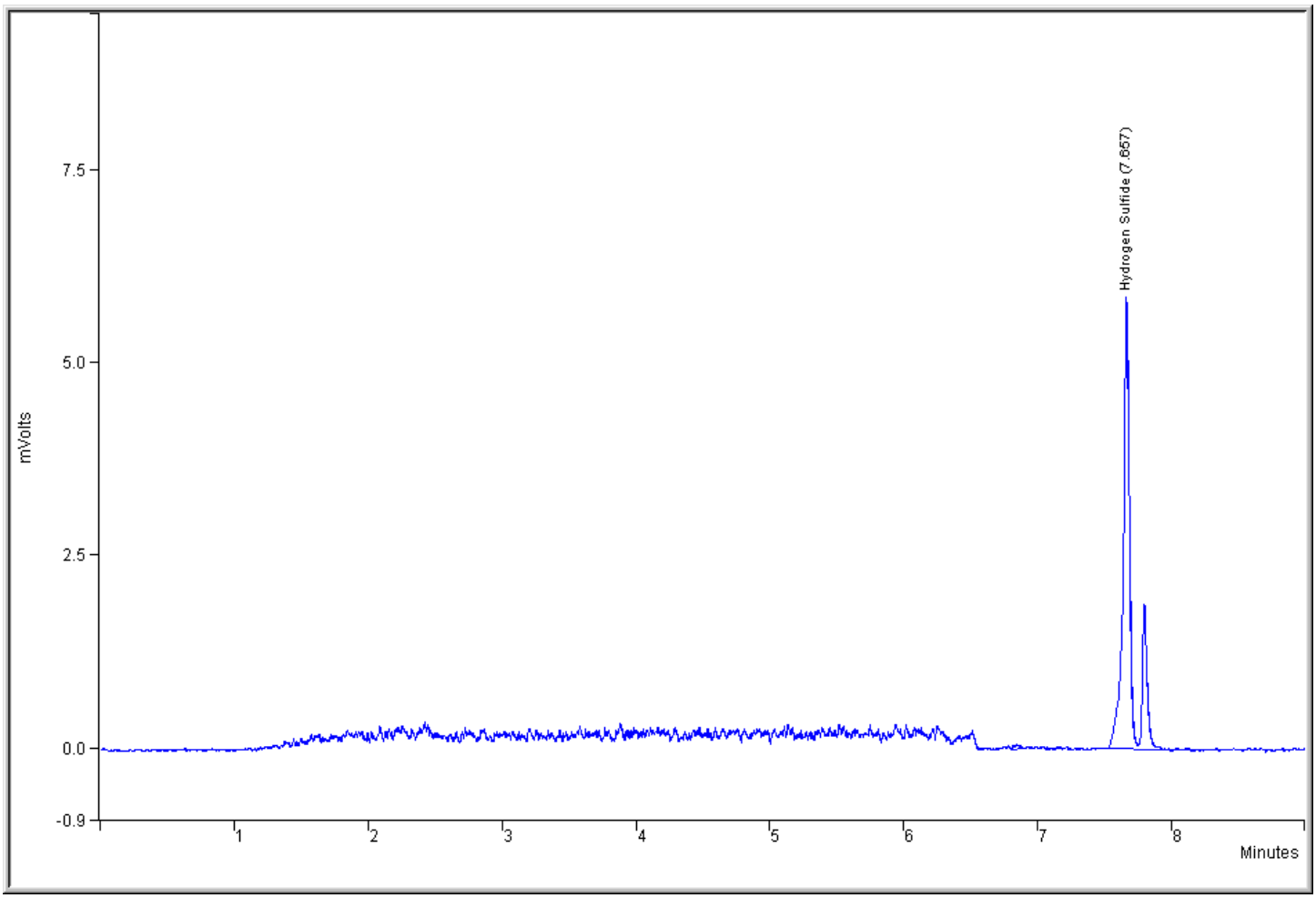
(3 X standard deviation of 9 runs at 0.3 ppb)



GREAT LINEARITY

A nice advantage of Mass Flow Controllers is that they can be used to generate multiple level calibrations, all based on a single standard. As the sample flow to the cold trap remains constant, the sampling time is varied to yield the different concentrations. Levels displayed above were analyzed in triplicate, except lowest level was repeated nine times.

The calibration graph above illustrates the linearity from 0.3 ppb to 12 ppb by setting the MFC to 25 ml/min and varying the sampling time from 0.2 minutes to 8 minutes.

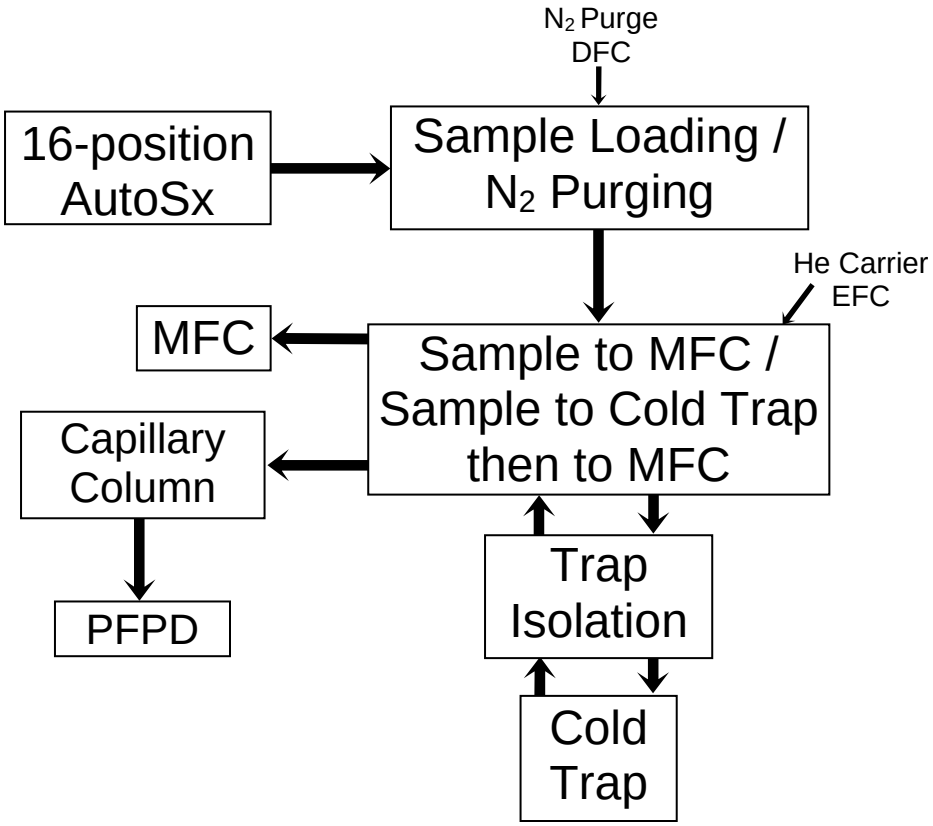


Chromatogram of Room Air with 200 ml volume injected

Conditions are same as above, except sample is 200 ml loading of ambient air.
Unidentified peak adjacent to Hydrogen Sulfide has not been confirmed, but is potentially COS.

Measured concentration of Hydrogen Sulfide in Room Air is 1.45 ppb V/V ± 0.01 ppb V/V

System Diagram



System Specifications

Concentrator Trap

- Temperature range: -196 °C to 450 °C
- Maximum heating rate: >300 °C/minute
- Maximum cooling rate: typically >400 °C/minute
- Temperature stability: < 2 °C after 1 minute stabilization
- Temperature overshoot: max. <10 °C, typically <5 °C
- Trap Cryogen usage: < 2 liters per sample
- Trap internal volume: ~600 microliters
- Trap material: proprietary, inert to sulfur compounds
- All trap settings controlled/monitored through GC with platinum probe (RTD) and proportional controller (PID)
- Programmable in 5 temperature steps with holds

Automated Sampler

- Standard: 16-position
- Micro-electric actuation, self-aligning
- Independently controlled valve oven
- Maximum temperature limit: 225 °C
- Sample position selected through workstation's sample list
- Position documented in final report and archived with data
- Sample lines heated through control of system

Valving

- Fully automated under time-programmable control of GC
- Valves mounted in heated enclosures
- Micro-electric actuation, easy realignment
- Valco Valve Series CWE; max temperature: 225 °C
- Valves can be turned on/off 21 separate event times within single method

Sampling

- Sample loading volume user-selectable through workstation from 2.5 ml to 1600 ml
- Samples in canisters or Tedlar bags can be handled without hardware changes
- Loaded sample volume independent of canister pressure

System Performance

- Detection limit: < 0.030 ppb V/V with 200 ml sample volume
- Typical area reproducibility - < 3 %
- Carry-over « 0.1 %
- Typical RT reproducibility - < 0.03 minutes

Pneumatics

- Column flow employs true Electronic Flow Controller (EFC), not pressure control with computed flow
- Temperature-sensitive flow elements maintained at 45 °C
- Flows automatically adjusted for atmospheric pressure

Column Oven

- Temperature range: -99 °C to 450 °C
- Temperature program rate: 0.1 °C/min to 100 °C/min
- Oven cool-down: 400 °C to 50 °C in 4.5 minutes without cryogen
- Programmable in 7 temperature steps with holds
- Coolant timeout to preserve cryogen when system idle
- Negative temperature programming to save coolant during sample loading

General

- GC display - Touch Screen [Very Thin Film Transistor (VTFT) and Wide Video Graphics Array (WVGA) with resolution 800 x 480, 9" (23 cm) diagonal size] eliminates keyboard "buttons", for more robust operations
- Ethernet communications between GC and Workstation
- Line voltage for GC: 120 V, 20 amperes

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