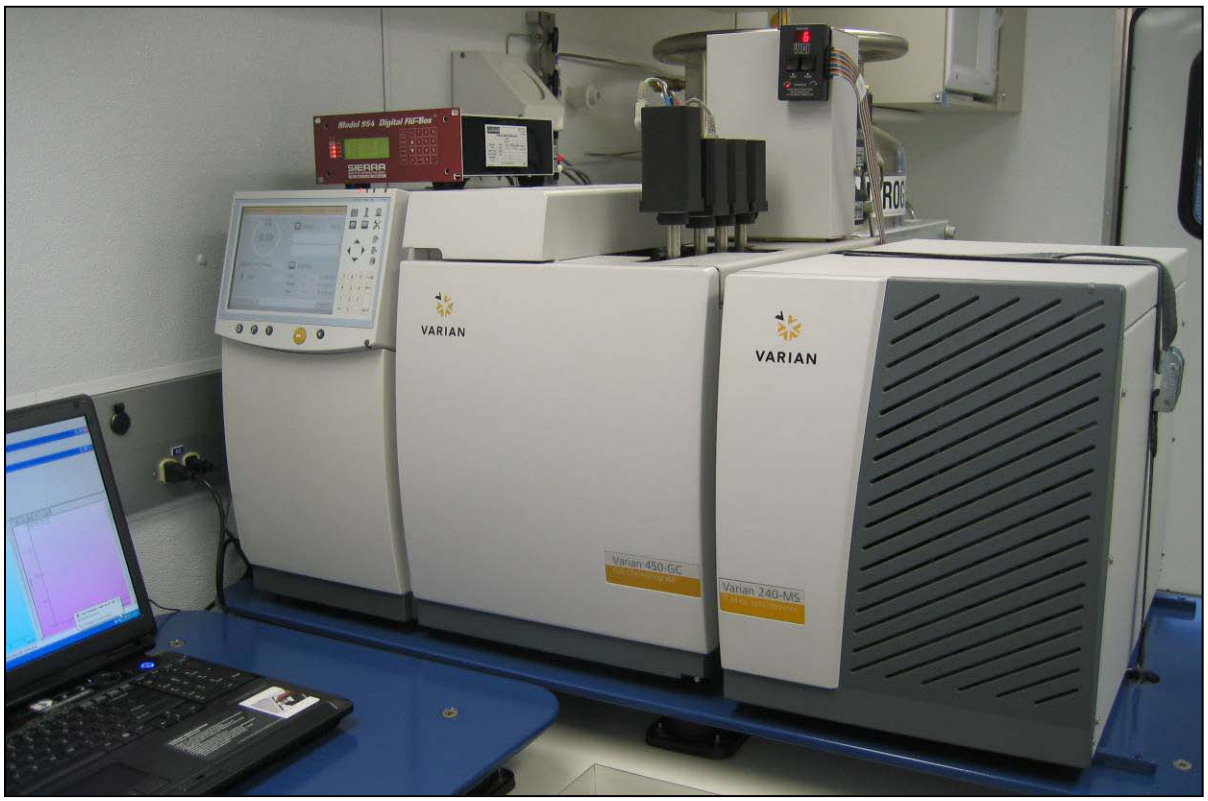
Lotus Consulting presents:

Ultra Trace Toxics System - 240 (UTTS - 240)

The Ultra Trace Toxics System - 240 from Lotus Consulting provides impressive separations and detection of trace volatile organics in ambient air samples. The system features the Varian 450 Gas Chromatograph with built-in high performance sample concentrator and the Varian 240 Ion Trap Mass Spectrometer. This system handles both pressurized canisters and Tedlar bags without hardware changes. And the system meets the exacting requirements for the US EPA TO-15 implementation for speciation of toxic compounds and the California Air Resources Board SOP No. MLD052 for determination of toxic organic compounds in ambient air.

The fully automated system is designed to completely resolve nearly all volatile organics from Freons to Hexachlorobutadiene to levels typically below 0.008 ppb V/V (300 ml injection). Samples are loaded through a 16-position automated sampler and trapped onto a low-volume adsorbent trap, with a mass flow controller (MFC) setting the sample size. A secondary cryofocus trap reduces the sample components to a smaller volume for injection into the column.

Detection of toxic organic compounds in ambient air is undoubtedly one of the most difficult analyses in gas chromatography. Samples must be concentrated into a small volume to enhance detection. A very large number of possible organics (>300) must be “fully” resolved to avoid improperly assigning concentrations from overlapping peaks. Identification and detection is facilitated with the extremely sensitive Varian 240 MS. The system involves an adsorbent trap, a cold trap, at least 4 automated valves, 16- or 31-position automated sampler, and one workstation. All of these operations utilize nearly all of the powerful and comprehensive capabilities of the Varian 450, Varian 240 MS and Varian 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.



SOLUTIONS TO DIFFICULT ANALYTICAL PROBLEMS…

Clean System Blanks

- Empty tubing or carbon adsorbents for trap
  - No thermal breakdown products
  - Maximum temperature limit of 400 °C
- All valves are heated; limited to 225 °C max
- Cryogenic cleansing of purge gas – vented after each cycle

Efficient Recovery of Light-End Components

- Area reproducibility for Ethane - < 2 %
- Accurate control of trap temperature (< ±2 °C) over complete temperature range
  - Self-calibrating platinum probe (RTD)
- Stable control of trap temperature (< ±2 °C)
  - Proportional controller
  - Close contact between heater, cryogen and trap
  - Silver-soldered connection of trap to mandrill
- Efficient trapping of Ethane on empty tubing and glass beads at –172°C

Full Recovery of "Heavy" Compounds

- All sample lines heated – no cold spots
- Smooth and inert sample lines – electroformed nickel
- Trap desorbing temperatures to 450 °C
- Effective release (>90 % of C_{12}; >80 % of C_{13}) from empty tubing or glass beads trap at 200 °C
- Maximum trap heating rate – 300 °C/min

Elimination of Interfering Artifacts

- Multi-bed carbon adsorbent trap and cryofocus trap standard
- No thermal breakdown of trap adsorbents that would yield interfering hydrocarbons (i.e. Benzene with Tenax)
- No reaction with NO_x that would yield interfering hydrocarbons (Ethene)
- Trap temperature limit to 450 °C

Sharp Chromatographic Peaks

- Minimum distance from cryofocus 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:
  - Adsorbent trap – ~600 microliters
  - Empty tubing - ~90 microliters
- diCl diF Methane peakwidth$_{½\ height}$: < 6 sec

Accurate Measure of Sample Volume

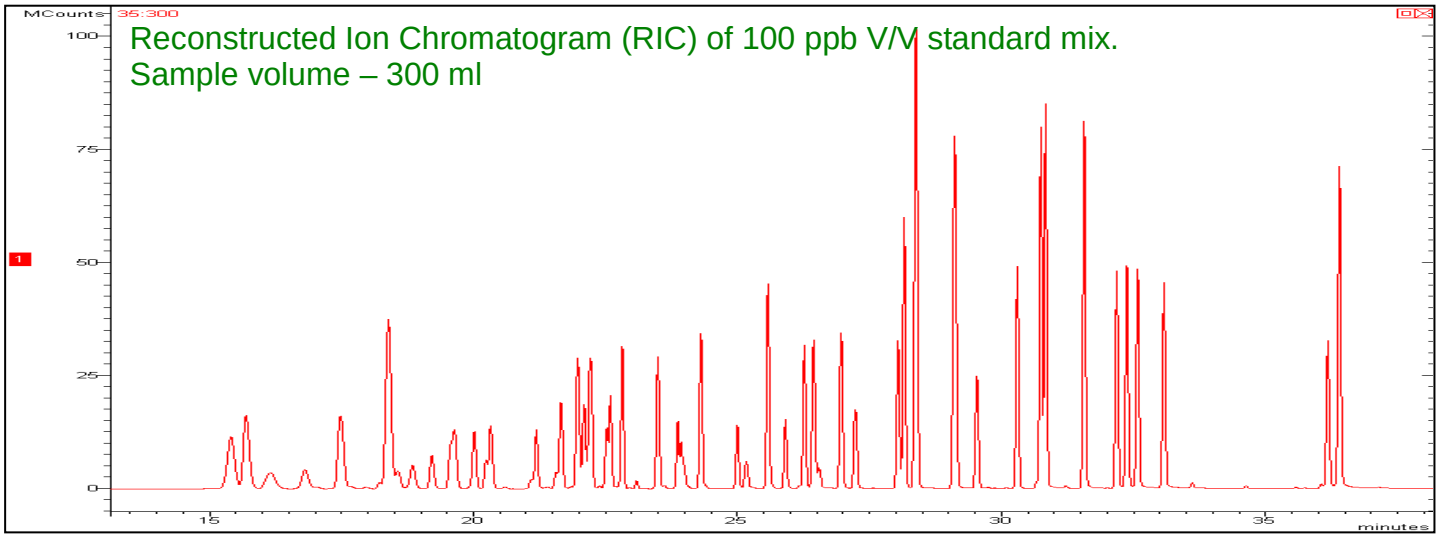
- Sample flow to vent just before trapping
  - Stabilizes MFC
  - Sweeps sample lines with new sample
- Volume-measuring flow path swept with nitrogen prior to trap heating
- Accurate volumes from 1 ml to 2400 ml
- Sample pressure can be below atmospheric and still maintain proper loading
- Sample loading independent of canister pressure

High Concentrations of CO_2?

- Mass Flow Controller (MFC) not accurate with major portions of CO_2
  - MFC usually calibrated for air; cannot handle gas mixtures properly
  - 50% CO_2 yields **double** the sample volume!!
- Optional fixed volume sample loop to 400 ml
  - Multiple loadings to trap for larger volumes
  - Full recovery of all hydrocarbons
  - Accurate measure of sample volume - even mixtures
- CO2 normally elutes prior to first toxic and does not interfere spectrally

Water Treatment

- Multi-bed carbon absorbents are hydrophobic and do not trap water at ambient temperatures and are effective traps for all components on TO15 list
  - Allows full recovery of both light-ends and heavies
  - Effectively handles water-saturated samples
  - Dry purge step time-programmable



…AND MORE SOLUTIONS

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 %

Proper Introduction of Surrogate/ Internal Standard

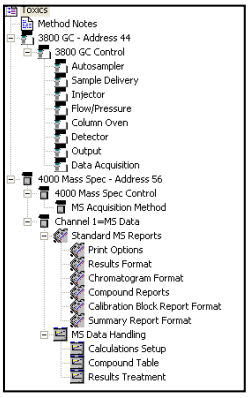
- Fixed volume sample loop
- Loop comes to atmosphere before injection
- Loaded onto trap as trap is purged with nitrogen

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
- 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

Simplified Method Execution

- Single run method contains **all** operating parameters for both GC and MS, 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.



Data Processing

- User can easily view both the developing chromatogram and MS spectra in real time
- Workstation can perform library searches on peaks in developing chromatogram for immediate confirmation of peak ID
- Single stored data file contains raw chromatographic data, final report, complete run method (including GC, MS, trap parameters), stream position, run log and error messages
- Data collection, report generation, system control, custom report and StarFinder operate in Window XP
- View/edit calibration curves
- Batching printing of reports from Windows Explorer
- Multi-level security with passwords
- Peak names to 40 characters

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 with sample ID, injection date/time, method used and module source as variables
- File names can be up to 255 characters long

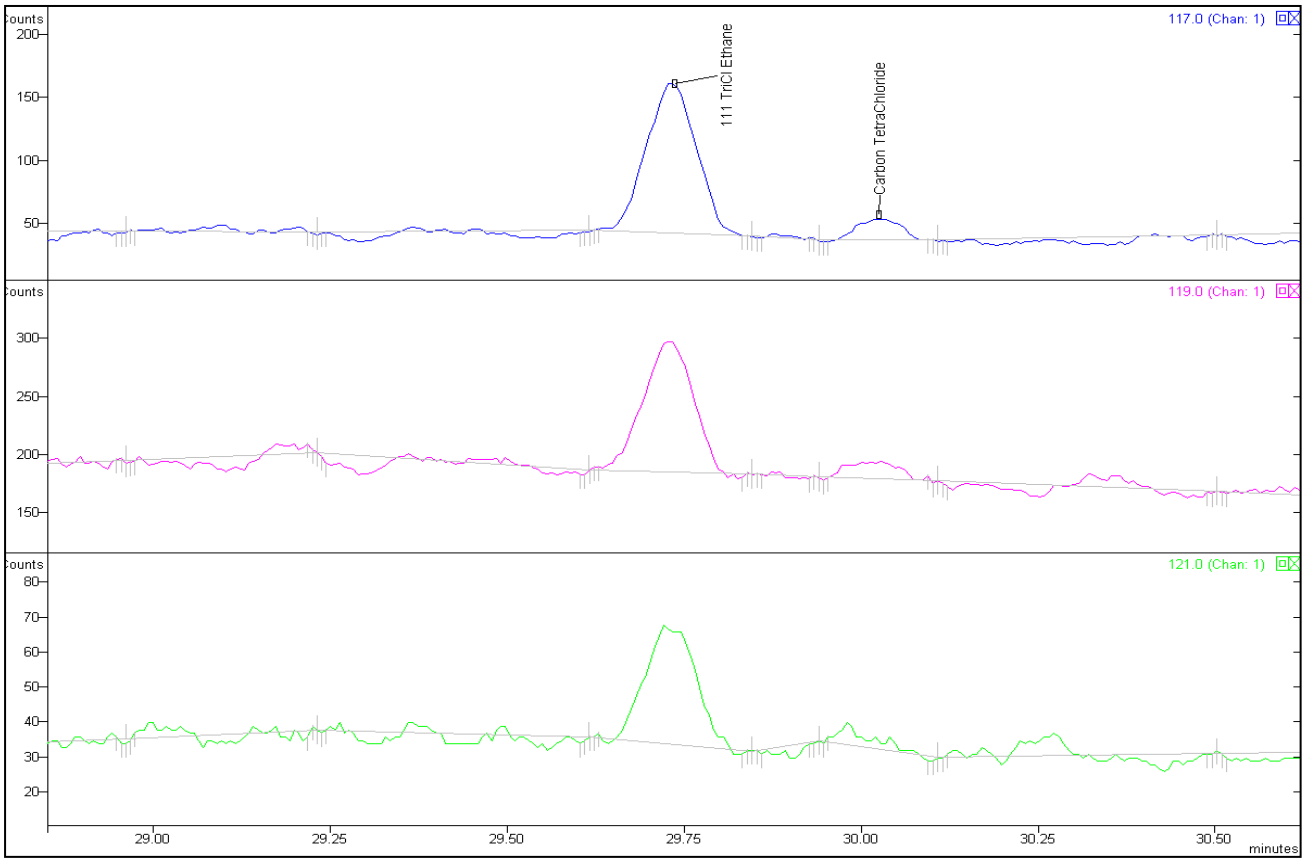
Options

- Fixed volume sample loop for proper handling of high CO_2 samples
- Addition of other detectors, such as Flame Ionization Detectors (for quantitation of hydrocarbons, especially the very light ones, including ethane, ethene, ethyne, propane, propene and propyne) and Pulsed Flame Photometric Detector (for sulfur compounds)
- Pressure station to bring canisters above atmospheric pressure to proper loadings
- Automatic insertion into SampleList of pressure station readings before and after pressurization for dilution corrections
- 10 position Canister cleaner with single high capacity, non-oil pump

Performance

EXTREME SENSITIVITY

Sensitivity is demonstrated with this chromatogram of Carbon Tetrachloride (right). This NIST component concentration is 930 ppQ V/V (0.0009 ppb)! Loaded sample volume is equivalent to just 150 ml. Displayed is the quantitation ion 117, 119 and 121 m/z only for the region at the elution of Carbon Tetrachloride and 111 TriChloroEthane.



The Varian Ion Trap is inherently more sensitive than conventional linear quadrupole mass spectrometers because nearly all of the sample ions are held within the ion trap until ejected out by the varying RF field when the proper mass is selected. For a quadrupole, only ions of a particular mass are allowed to pass through the quad for just a few microseconds as the RF field ramps through the mass range, often with only 0.1% of the original ions reaching the detector. More importantly, higher mass ions have lower transfer efficiencies.

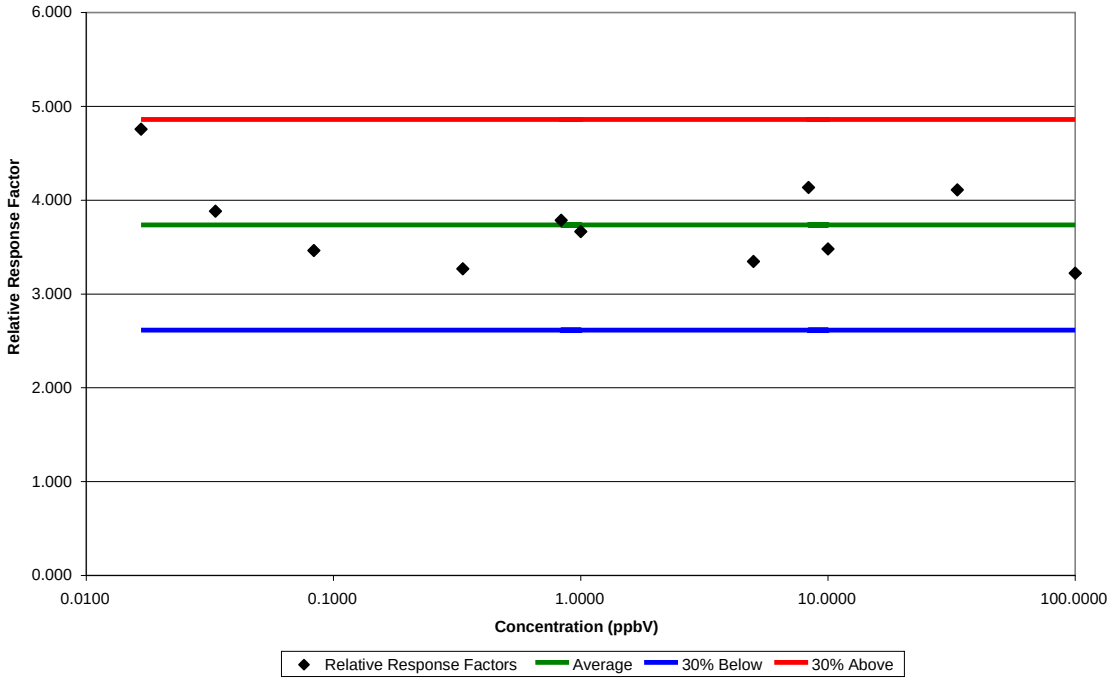
The Varian 240 Ion Trap takes the next step in achieving enhanced performance by transferring more ions to the detector through a physical change in the electrode design and a new patented ejecting waveform. Its detector is mounted 90^{o} to the exit of the trap so that neutrals and stray electrical charges end up going straight and miss the bend, while the ions of interest make the curve, resulting in more desirable signal and less noise. The linear range is also extended with these improvements.

Single Ion Monitoring (SIM) is frequently used to make quadrupoles perform closer to ion traps in sensitivity. By monitoring only one mass, we end up losing the full spectrum and identification with NIST library is no longer possible. With ion traps, since all ions are held anyway, Selected Ion Storage (SIS) offers no great enhancement and we can still use library searches with the full scan, even at extremely low levels.

…and More Performance

GREAT LINEAR RANGE
< 6,600

Bromodichloromethane
from 0.016 ppbV/V to 100 ppbV/V



A nice advantage of Mass Flow Controllers for sample loading is that we can use them to generate multiple level calibrations, all based on a single standard or just a few standards. As the sample flow to the cold trap remains constant, the sampling time is varied to yield the different concentrations.

The graph above illustrates the linearity from 0.016 ppb to 100 ppb by setting the MFC to 50 ml/min and varying the sampling time from 0.1 minutes to 6 minutes for three standards of 100 ppb V, 10 ppbV and 1 ppbV.

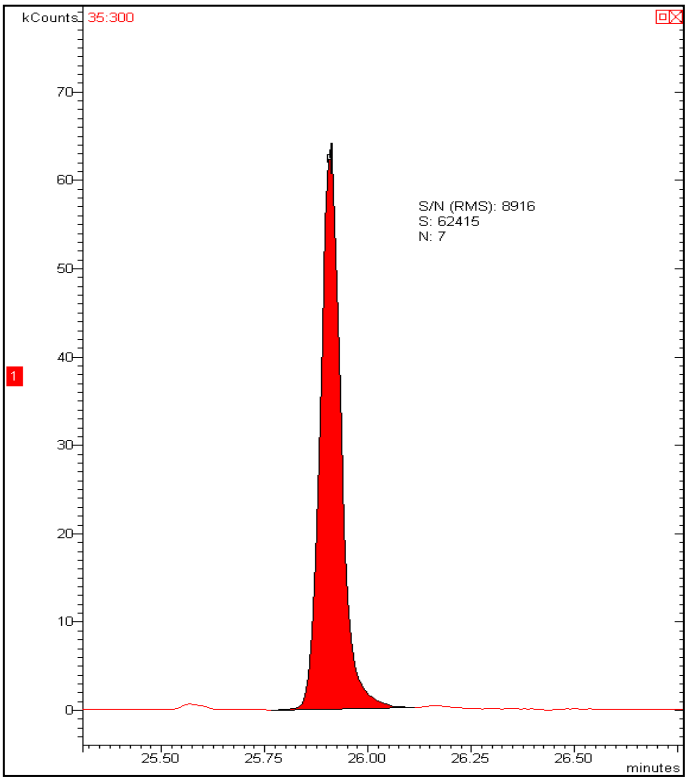
...and Even More Performance

TYPICAL DETECTION LIMITS
for Selected Compounds
(300 ml sample, full scan)

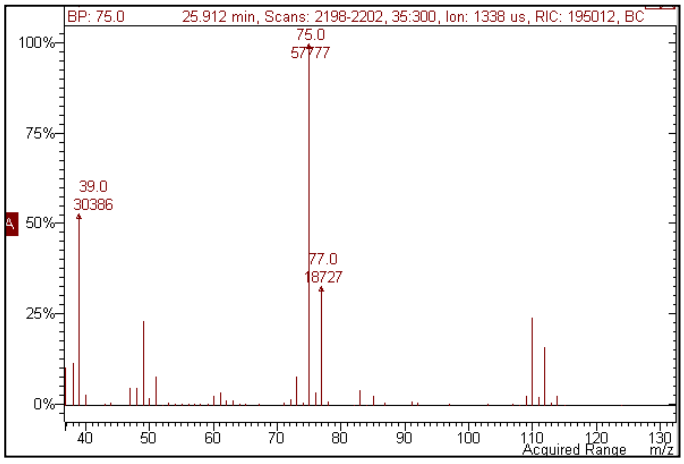
Compound	Quant Ion	Detection Limit[1]	Standard Concentration
diCl diF Methane	85	0.008 ppb V/V	0.028 ppb V/V
Vinyl Cl (Cl Ethene)	62	0.012 ppb V/V	0.030 ppb V/V
CCl3F	101	0.008 ppb V/V	0.030 ppb V/V
13Butadiene	54	0.006 ppb V/V	0.024 ppb V/V
Bromomethane	94	0.006 ppb V/V	0.031 ppb V/V
diCl Methane	49	0.008 ppb V/V	0.064 ppb V/V
12diCl Ethane	62	0.007 ppb V/V	0.024 ppb V/V
Chloroform	83	0.006 ppb V/V	0.025 ppb V/V
111triCl Ethane	97	0.006 ppb V/V	0.024 ppb V/V
Benzene	78	0.006 ppb V/V	0.026 ppb V/V
12diCl Propane	76	0.004 ppb V/V	0.014 ppb V/V
c13diCl Propene	75	0.009 ppb V/V	0.028 ppb V/V
Toluene	91	0.006 ppb V/V	0.032 ppb V/V
t13diCl Propene	75	0.004 ppb V/V	0.030 ppb V/V
TetraCl Ethene	166	0.005 ppb V/V	0.031 ppb V/V
ClBenzene	112	0.010 ppb V/V	0.031 ppb V/V
EtBenzene	106	0.009 ppb V/V	0.035 ppb V/V
m/pXylene	106	0.013 ppb V/V	0.069 ppb V/V
Styrene	104	0.008 ppb V/V	0.022 ppb V/V
oXylene	106	0.008 ppb V/V	0.040 ppb V/V
13diCl Benzene	146	0.006 ppb V/V	0.029 ppb V/V
14diCl Benzene	146	0.006 ppb V/V	0.029 ppb V/V
12diCl Benzene	146	0.007 ppb V/V	0.031 ppb V/V
HexaChloro Butadiene	225	0.009 ppb V/V	0.041 ppb V/V

[1] Detection Limit is 3X Std Dev at or near this level.
Standard employed to generate detection limits;
Sample flow rate – 50 ml/min
.Sampling time – 0.05 min
Sample Volume Injected – 5 milliliter

RADICAL SIGNAL-TO-NOISE

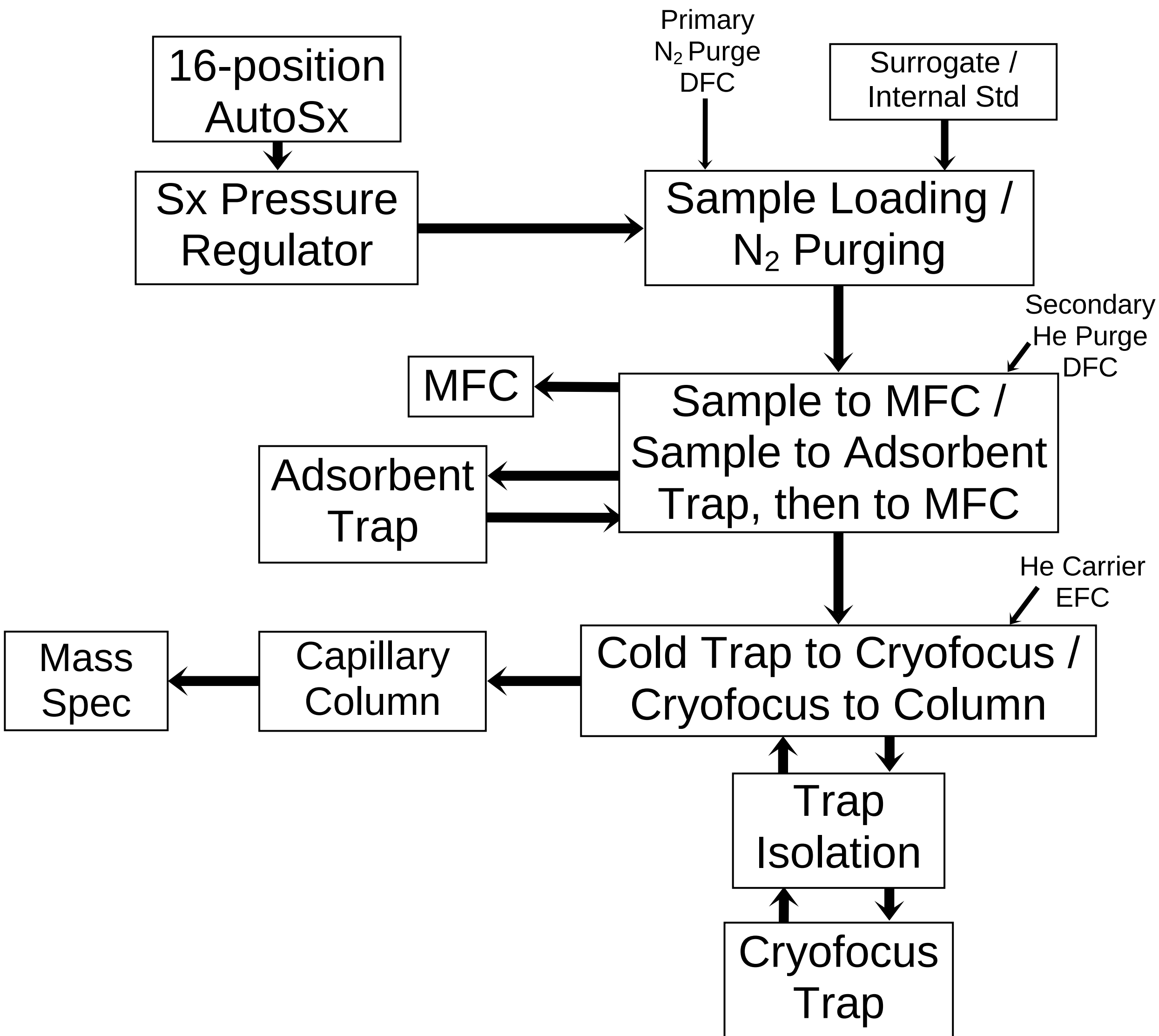


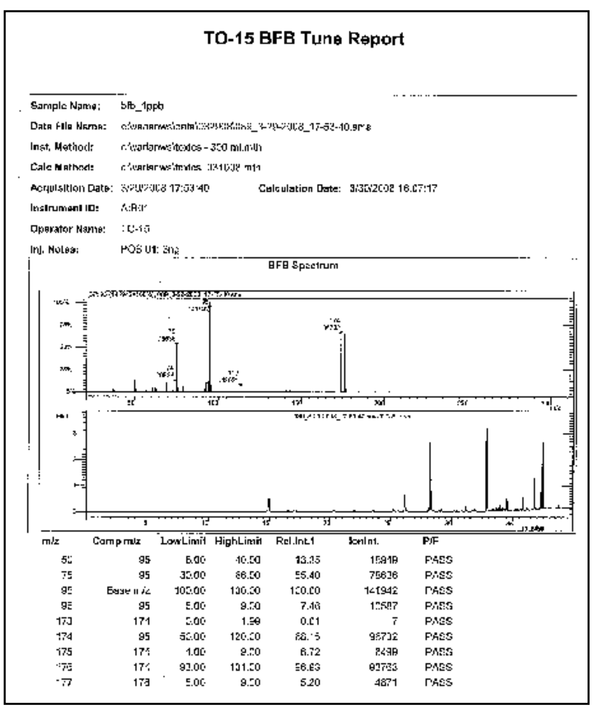
Just 300 ml sample loading of 0.030 ppb V/V trans-1,3 Dichloropropene yields a superb signal-to-noise ratio and still see full scan. Effective trapping and remarkable performance of the Varian 240 team together to provide this achievement.



Spectrum of 0.030 ppbV/V trans-1,3 Dichloropropene

System Diagram





TO-15 BFB Tune Report

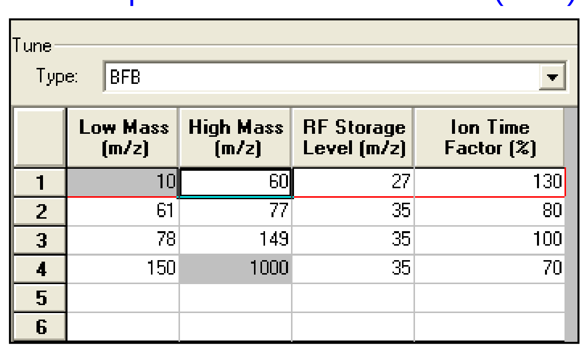
Sample Name: bfb_1ppb
Data File Name: c:/varianws/data/...-2008_17-52-40.sms
Inst. Method: c:/varianws/todos - 300 ml.mth
Calc Method: c:/varianws/todos ...
Acquisition Date: 3/29/2008 17:53:40 Calculation Date: 3/30/2008 16:07:17
Instrument ID: AirBr
Operator Name: TO-15
Inj. Notes: POS 01: 3ng

BFB Spectrum

m/z	Comp m/z	LowLimit	HighLimit	Rel.Int.1	IonInt.	P/F
50	95	8.00	40.00	13.35	15919	PASS
75	95	30.00	66.00	55.40	78636	PASS
95	Base m/z	100.00	100.00	100.00	141942	PASS
96	95	5.00	9.00	7.46	10587	PASS
173	174	0.00	1.99	0.01	7	PASS
174	95	50.00	120.00	68.15	96732	PASS
175	174	4.00	9.00	8.72	8438	PASS
176	174	93.00	101.00	96.83	92733	PASS
177	176	5.00	9.00	5.20	4871	PASS

BFB Tune Compliance

Ion traps are inherently very gentle and effective at ionizating target analytes and they generate spectra that emphasize the heavier ions, often the ones that uniquely characterize the compound. Without modifications to the mass responses, these native spectra would not fully match NIST libraries from other MS instrument types. To allow a perfect tune to p-Bromofluoro-benzene (BFB) criteria, specific mass regions can be scaled down (or up) through adjustments to Ion Time Factors as a function of mass, as shown to the right.



Tune

Type: BFB

	Low Mass (m/z)	High Mass (m/z)	RF Storage Level (m/z)	Ion Time Factor (%)
1	10	60	27	130
2	61	77	35	80
3	78	149	35	100
4	150	1000	35	70
5				
6				

System Specifications

Concentrator Traps

- 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: < 4 liters per sample
- Trap internal volume: ~90 microliters – cryofocus
  ~600 microliters – adsorbent trap
- 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 Series CWE; maximum temperature: 225 °C
- Valves can be turned on/off 21 separate event times within single method
- Automatic addition of surrogate/internal standard

Sampling

- Sample loading volume user-seletcable through workstation from 5 ml to 1600 ml
- Samples in canisters or Tedlar bags can be handled without hardware changes
- Loaded sample volume independent of canister pressure
- Optional fixed loop sampling for high CO_2 samples

System Performance

- Detection limit: < 0.004 ppb V/V to < 0.021 ppb V/V (compound dependent) with 300 ml sample volume under full scan
- Area reproducibility typically < 2 %
- 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 or vacuum

Column Oven

- Temperature range: -99 °C to 450 °C with LN_2 cryogen
- 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

Mass Spectrometer

- Quadrupole Ion Trap Design
- Mass range: 10 to 1000 u, in 0.1 u increments programmable throughout the analysis
- Scan rate: 10,000 u/second
- Resolution: better than unit mass (with 10% valley)
- Ionization modes: Electron impact and Selected Ion Storage with programmable control from one mode to another within single analysis
- Internal ionization for superior low mass detection
- Ion gauge and foreline gauge included
- Independent manifold heater of ion trap to 250 °C
- Turbomolecular pumping rate: 230 L/sec
- Sensitivity: 200 fg Octafluornapthalene produces a peak with S/N (RMS) 20:1 or greater

General

- GC keyboard 11 lines and 35 characters/line for ease of programming and monitoring
- Ethernet communications between GC and Workstation
- USB communications between MS and Workstation
- Instrument width: 43 inches, including concentrator
- Line voltage for GC: 120 V, 20 amperes;
  for MS: 120V, 15 amperes

Lotus Consulting
310/569-0128 Fax 714/898-7461
email ebramstoncook@msn.com

5781 Campo Walk
Long Beach, California 90803