New Person Portable Gas Chromatograph for Harsh Environments – Design Criteria, and Verification Testing

The 8th Harsh-Environment Mass Spectrometry Workshop

Saint Petersburg Beach, Florida

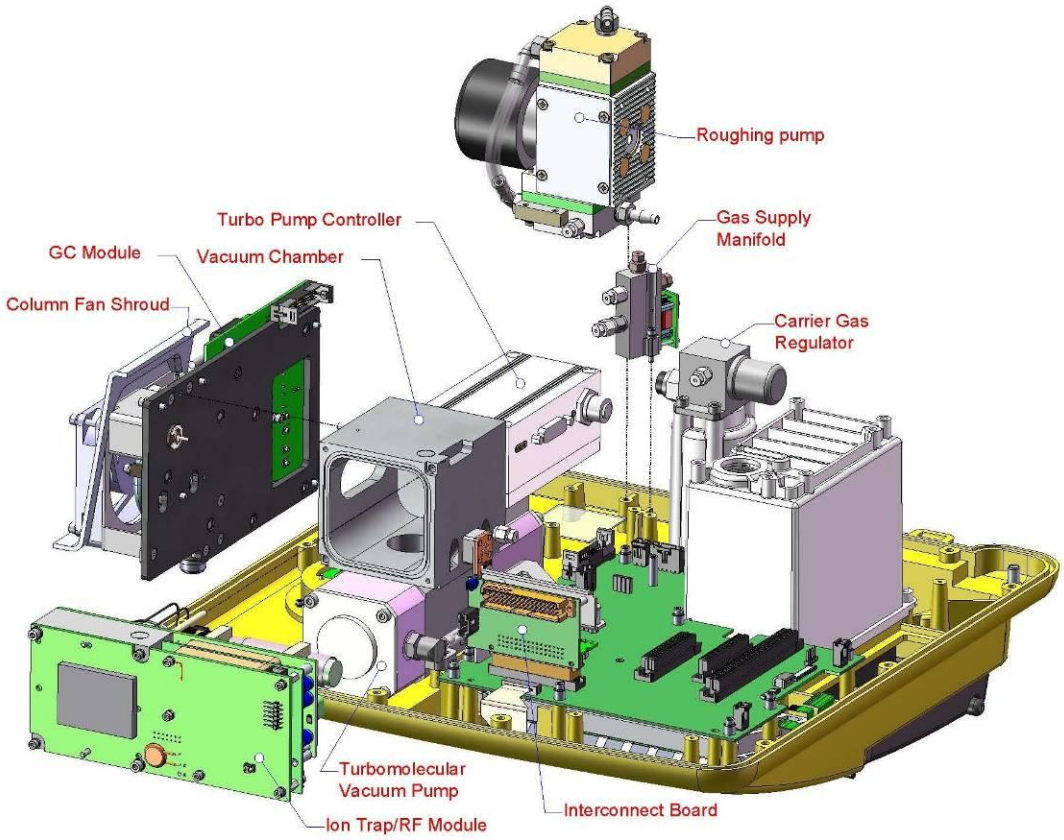
September 20, 2011

Design Criteria – Hand Portable

- Physical
  - Size <14 ½” x 18 ½” x 8”
  - Weight < 30lbs (currently 32lbs)
  - Withstand shock and vibration “Rough Handling”
- Environmental
  - Operation in Harsh Environments
    - Ambient temperature from 0^{o}C to 45^{o}C
    - High humidity and rain
    - Impervious to sand and dust
    - Ability to decontaminate with bleach or other chemicals
- Analytical Performance
  - Rapid startup
  - Gas Chromatograph capable of handling a broad range of analytes
  - High performance standards = “benchtop”

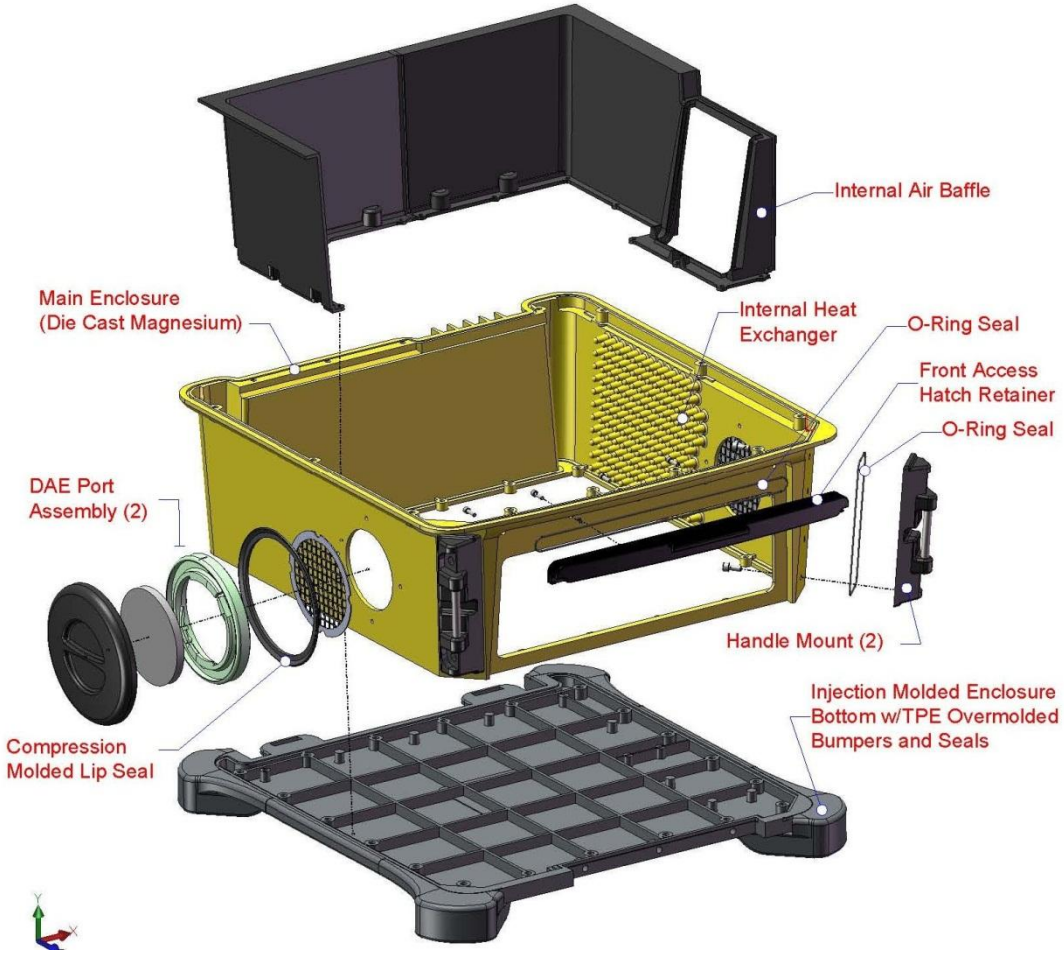
Physical Requirements

- Base System
  - Top Plate die cast magnesium to reduce weight
  - Elimination of cables positive interlock used on all connections
  - Power Management
    - Single rechargeable battery
  - Efficient two stage pumping system
    - Turbo
    - Diaphragm



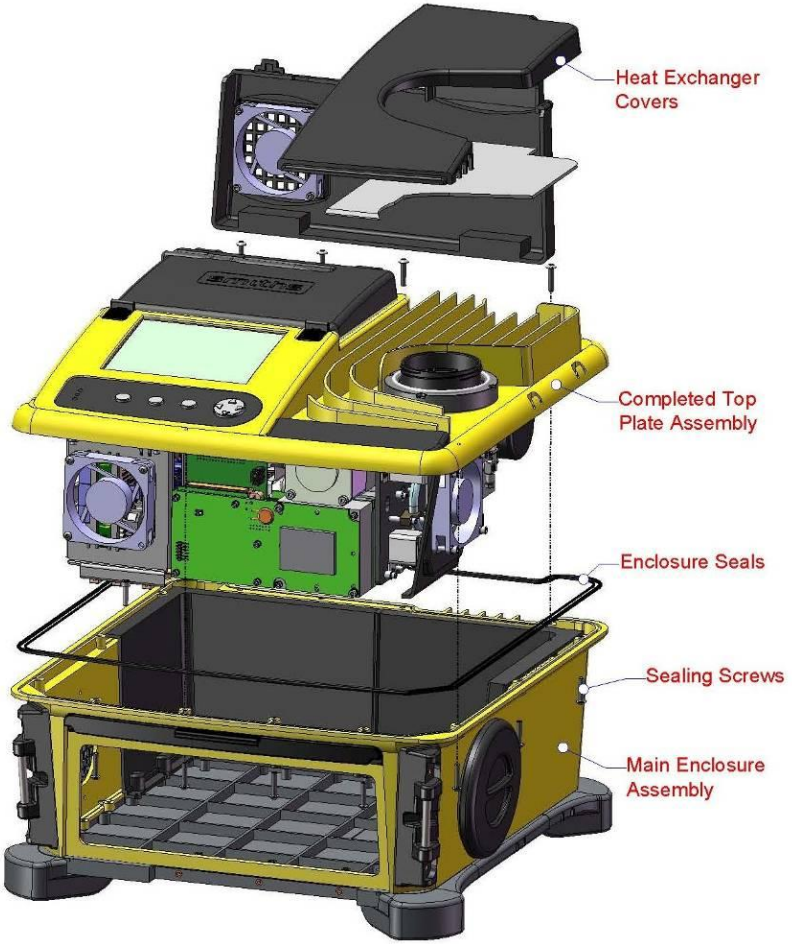
Physical Requirements

- Enclosure
  - Die cast magnesium to reduce weight
  - Integrally cast internal heat exchanger improves heat transfer
  - Internal air baffle interfaces with column fan shroud to control column cooling air source
  - EMI coating on inner surface of enclosure bottom

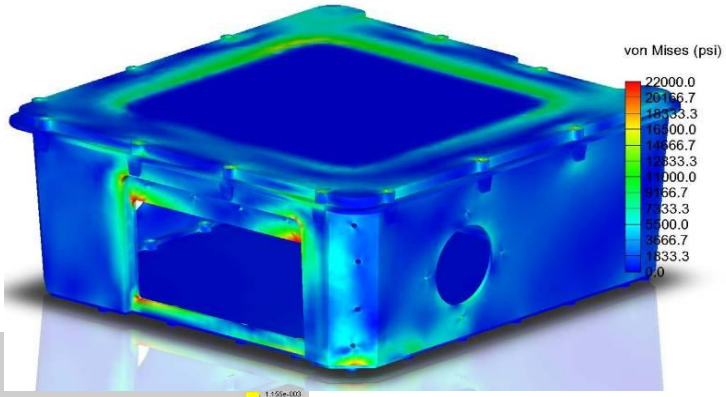


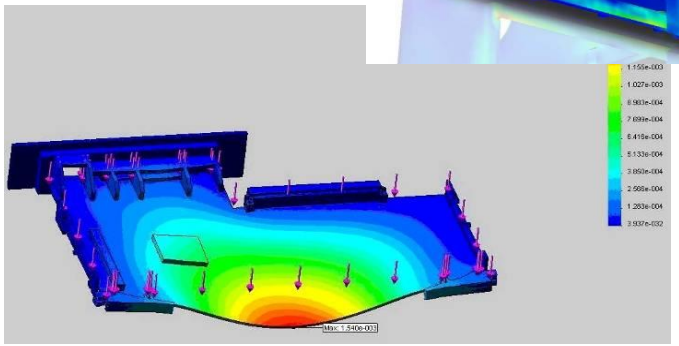
Environmental

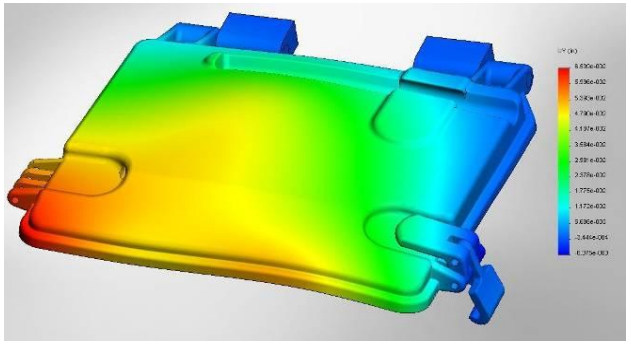
- Enclosure
  - Sealed
    - DECON capable
    - Dust
    - Sand
  - Shock
    - Bumpers
    - Mounting
  - Efficient heat transfer



Mechanical System Analysis



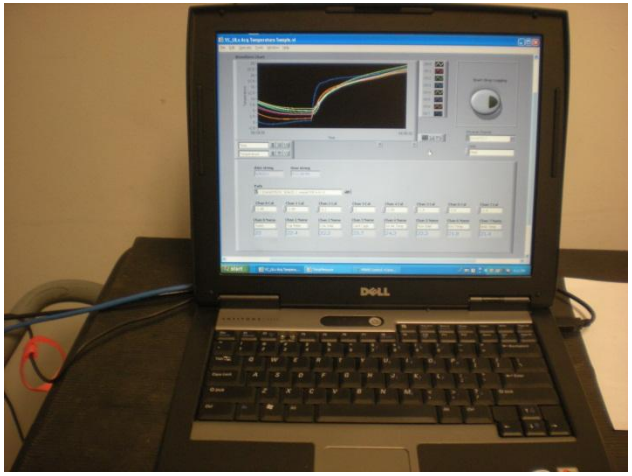


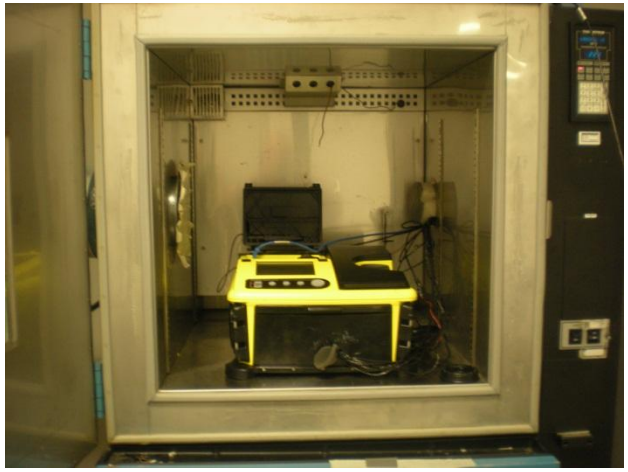


- Stress, deflection, and vibration analysis performed on many components to insure structural integrity and appropriate deflections. (Solidworks Simulation FEA package)
- Used to complement manual calculations where the complexity of the problem dictates.

Physical and Environmental Testing

- Thermal
  - Successful operation from 0°C to 45°C
    - AC/DC and Battery Power
    - RTD's placed throughout system
    - Heat Chamber test conducted for continuous operation





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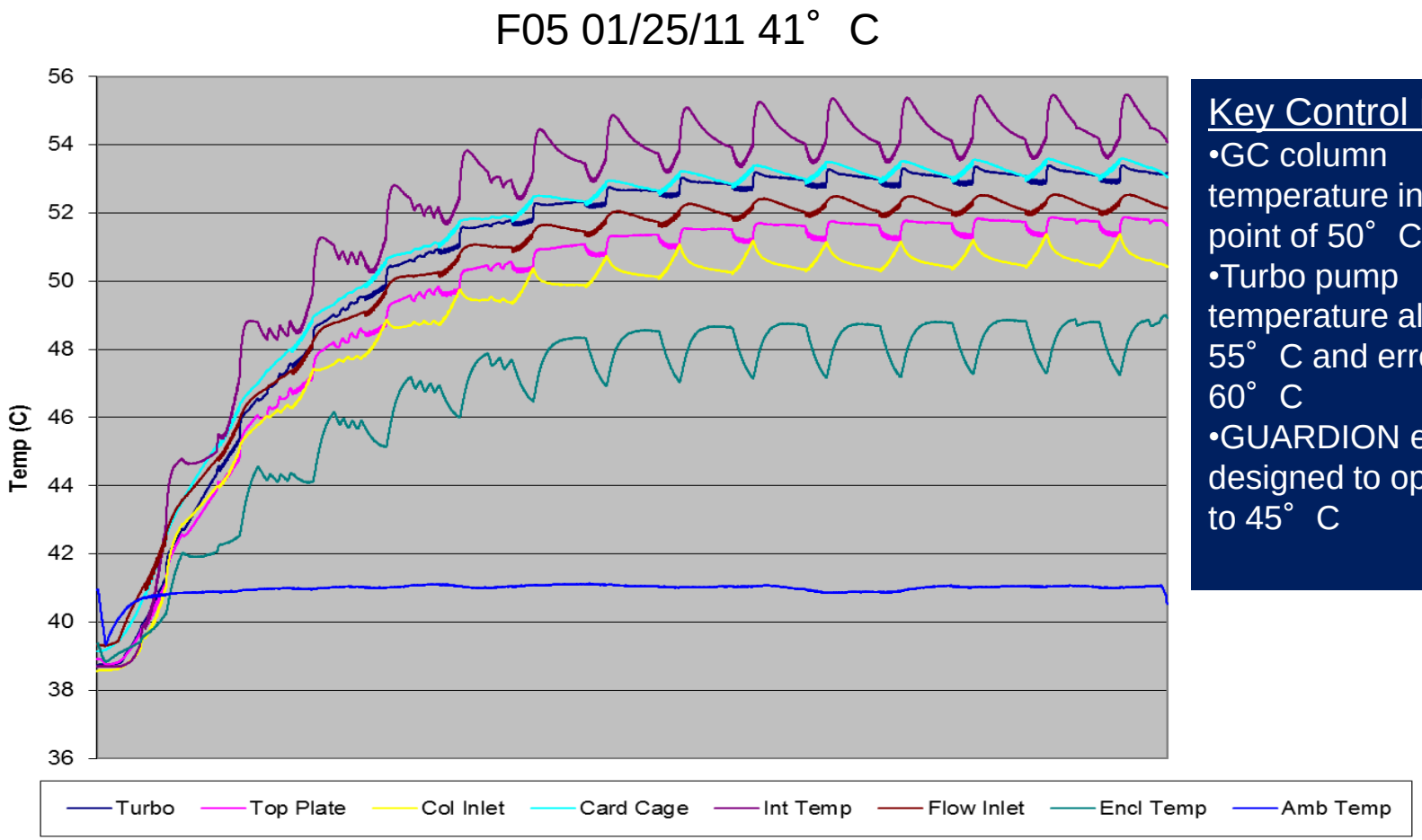
Enclosure Thermal Performance



Key Control Points

- GC column temperature initial set-point of 50° C
- Turbo pump temperature alarms at 55° C and errors out at 60° C
- GUARDION enclosure designed to operate up to 45° C

Physical and Environmental Testing

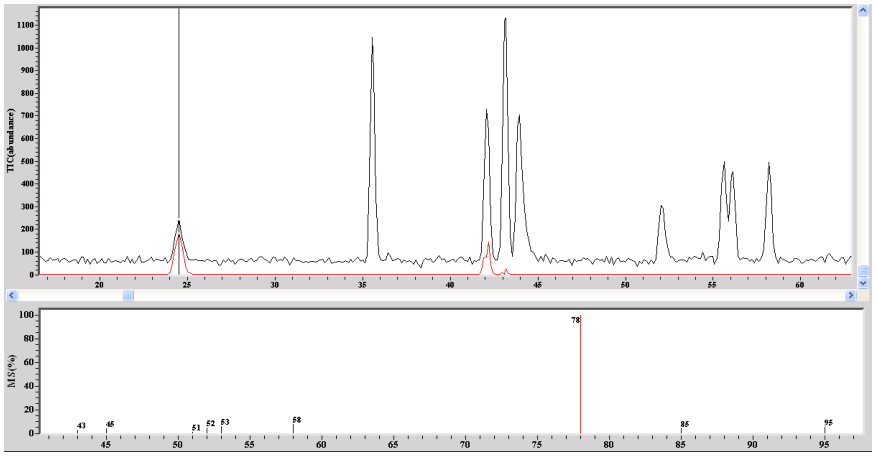
- Drop testing
  - 16.85” (maximum height) all 6 faces and all 8 corners outside of protective case.
  - 3 feet inside transit case, all 6 faces, and 2 corners
- Vibration
  - 30 mins @ 10.5, 60, and 152.5Hz each (outside of protective case).
  - Vibrated on three axis at each frequency 30 min.
- Sealing
  - All environmental seals tested \by submerging in water (~4” depth) for 30 minute duration
- Decon/Chemical resistance
  - System external coating tested for chemical resistance to:
    - Acetone
    - Methanol
    - Methylene Chloride
    - Gasoline
    - Ammonia Solution
    - Motor Oil
    - Bleach
    - Sea Water
    - Windex

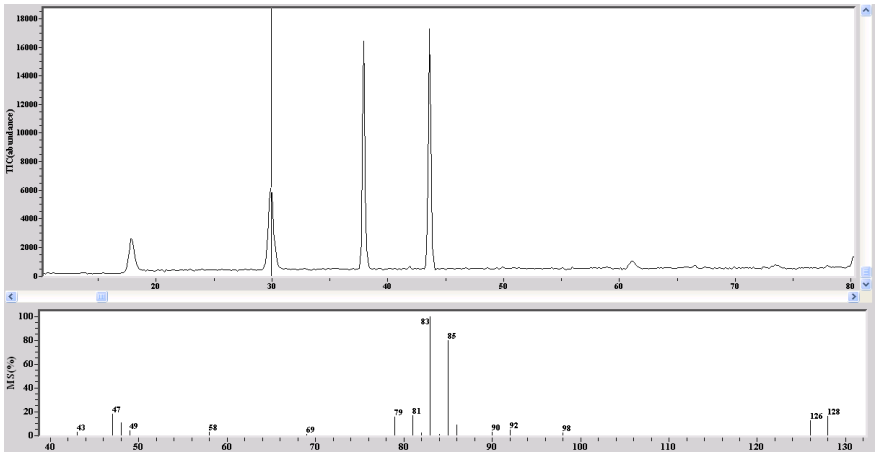
Analytical Performance Criteria

- System
  - Startup time
  - Battery Life
  - Carrier gas consumption
  - Sensitivity
  - Dynamic Range
  - Reliability
- Chromatographic
  - Retention time reproducibility
  - Peak Shape
  - Carryover
  - Boiling point range (VOC &SVOC)
- Mass Analyzer
  - Mass Range 45 - 500
  - Spectral Quality
  - Resolution
  - Scan Speed
  - Mass Calibration Stability

System

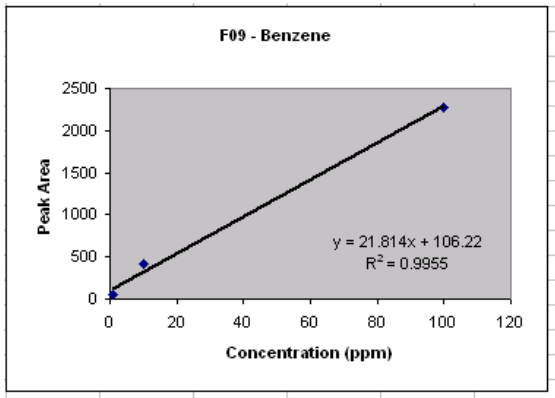
- Startup < 4 minutes at 8 ºC, 22 ºC,35 ºC
  - AC/DC
  - Battery
- Battery Life (includes startup)
  - ≤ 25 runs up to two Hours at maximum data collection rate
- Carrier gas consumption ~ 150 + runs/cylinder
- Sensitivity (1ppm Benzene in Air and 500 ppb Trihalomethanes in water)

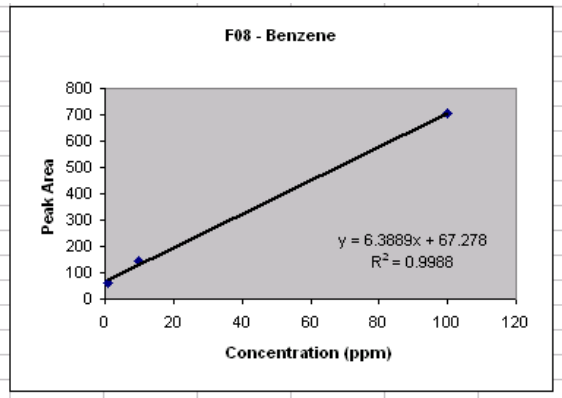


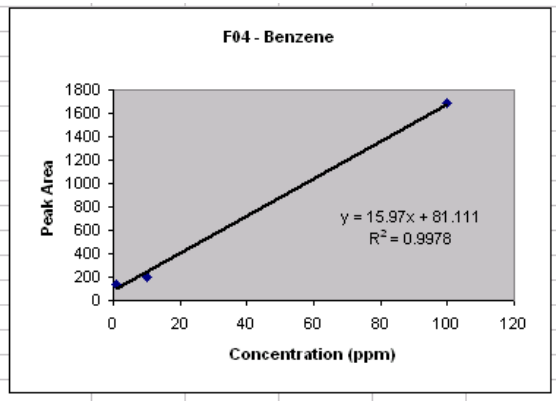


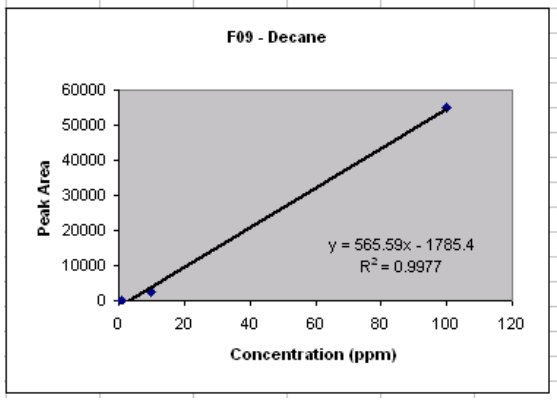
System

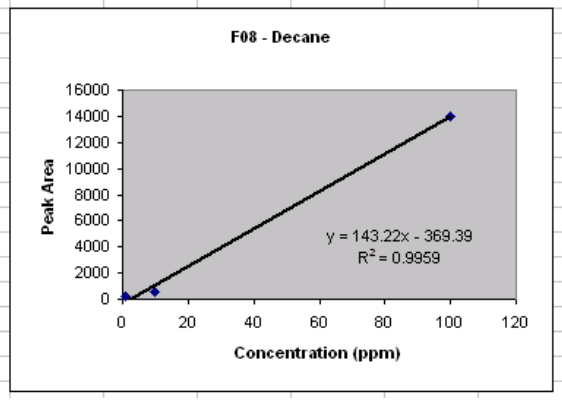
- Dynamic Range (3 instruments/2 compounds)
  - 2 Orders of magnitude (can be extended by sampling mode)

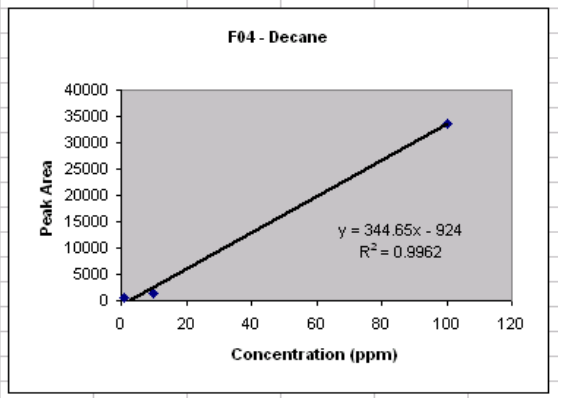






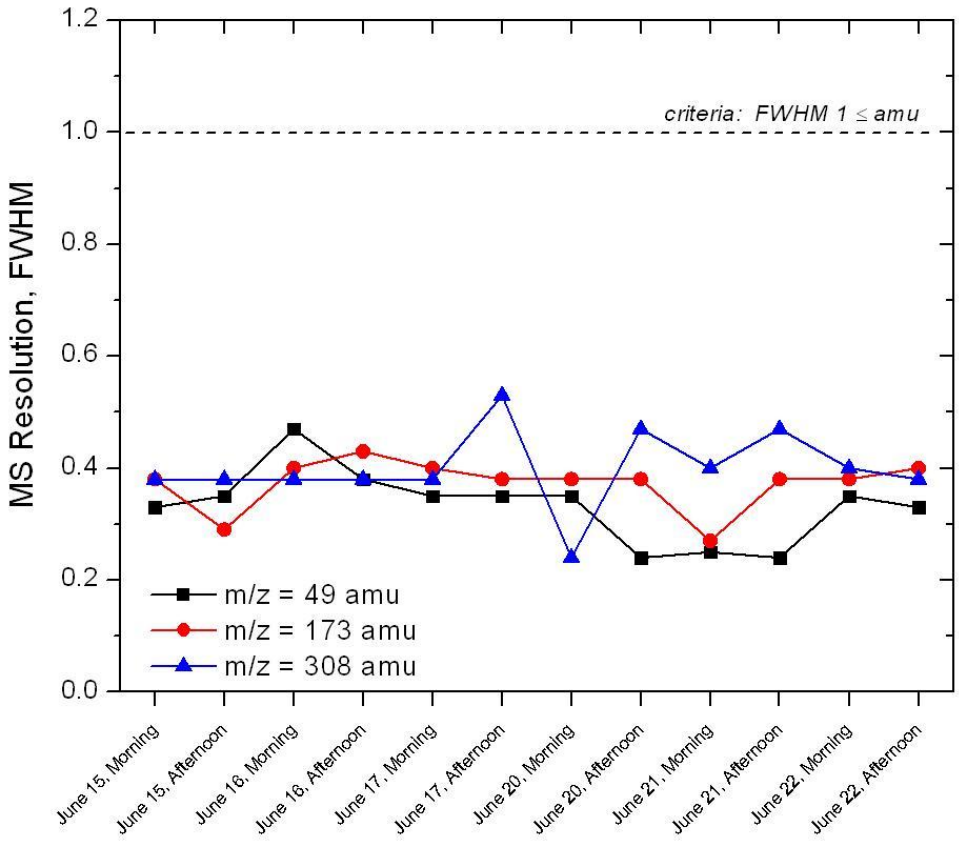


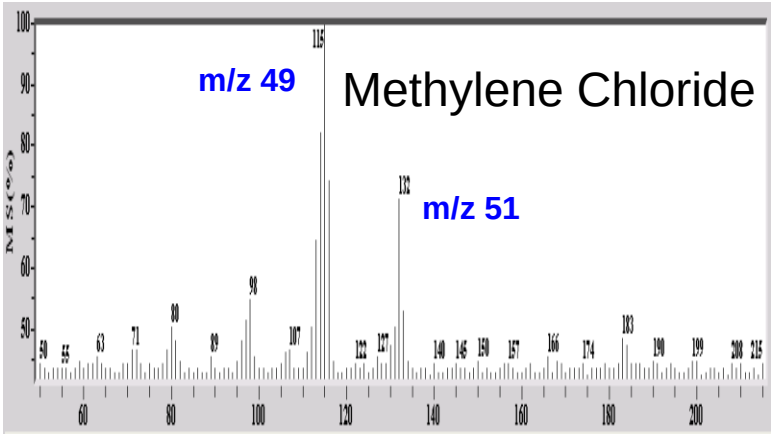


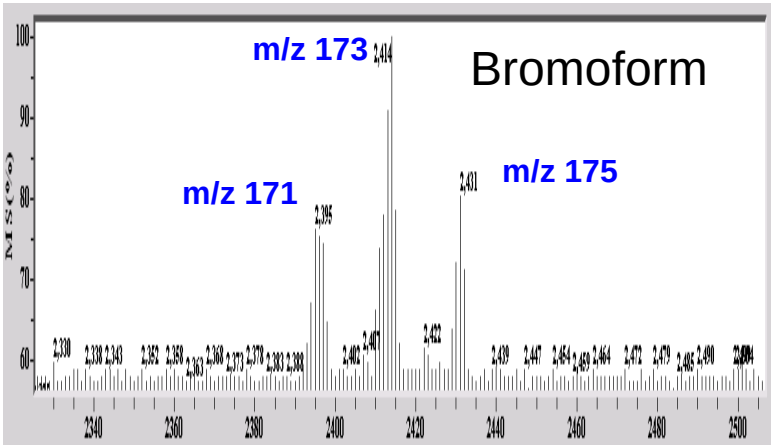


System - # system > 750 runs, sensitivity, mass range, resolution

- Reliability/Longevity







Chromatographic

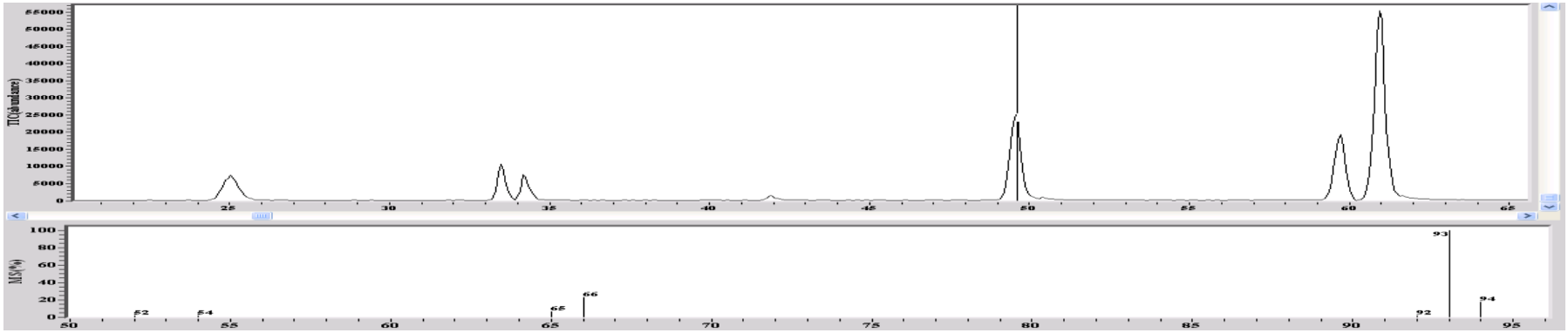
- Retention Time - 3 system > 80 runs, across 3 systems

No.	Analyte	F04		F08		F09	
		RT Avg	*%RSD*	*RT Avg*	*%RSD*	*RT Avg*	*%RSD*
1	*Methylene Chloride*	12.532	**1.594**	13.576	**1.726**	12.013	**2.141**
2	*MTBE*	14.541	**1.571**	15.955	**2.048**	14.016	**2.052**
3	*Methylcyclohexane*	32.050	**0.724**	31.077	**2.583**	32.237	**1.091**
4	*Toluene d8*	35.564	**0.731**	34.464	**2.024**	35.678	**1.005**
5	*Perchloroethylene*	39.982	**0.891**	38.621	**1.418**	38.805	**0.943**
6	*Bromopentafluorobenzene*	43.993	**1.034**	42.743	**2.107**	41.388	**0.878**
7	*Bromoform*	47.217	**1.203**	45.882	**0.654**	43.701	**0.779**
8	*DBTFB*	67.216	**1.476**	65.639	**2.400**	61.830	**0.397**
9	*Methyl Salicylate*	73.331	**1.560**	71.874	**0.248**	67.301	**0.340**
10	*Tetrabromoethane*	81.042	**1.508**	79.139	**0.257**	74.209	**0.306**
11	*Pentadecane*	93.095	**1.590**	92.188	**0.281**	86.654	**0.263**

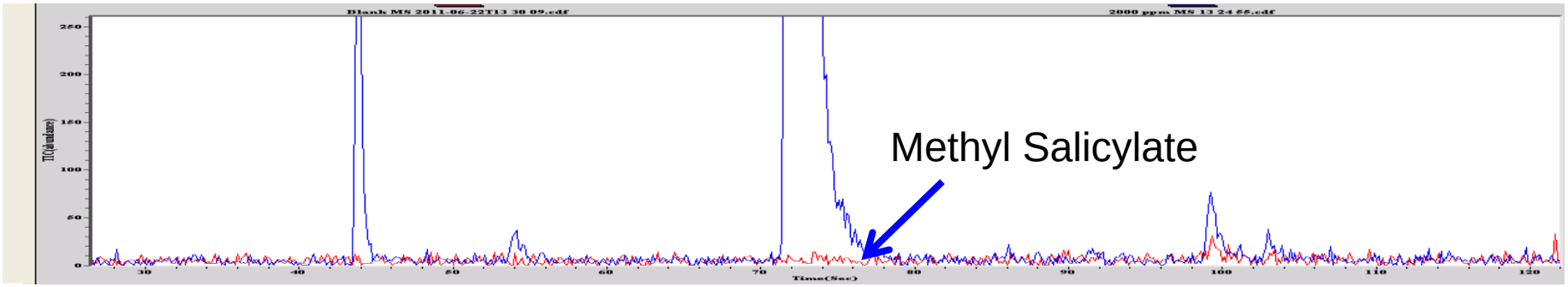
No.	Analyte	Avg. RT	% RSD
1	*Methylene Chloride*	12.707	**6.26**
2	*MTBE*	14.837	**6.76**
3	*Methylcyclohexane*	31.788	**1.96**
4	*Toluene d8*	35.235	**1.90**
5	*Perchloroethylene*	39.136	**1.89**
6	*Bromopentafluorobenzene*	42.708	**3.05**
7	*Bromoform*	45.600	**3.89**
8	*DBTFB*	64.895	**4.27**
9	*Methyl Salicylate*	70.835	**4.44**
10	*Tetrabromoethane*	78.130	**4.51**
11	*Pentadecane*	90.646	**3.85**

Chromatographic

- Peak Shape
  - Benzene, MIBK, Pyridine, Aniline, Nitrobenzene,Triethylphosphate
  - EPA method 537 peak asymmetry factor (A_s) is defined at 10% peak height and must fall within the range of 0.80 – 1.50.

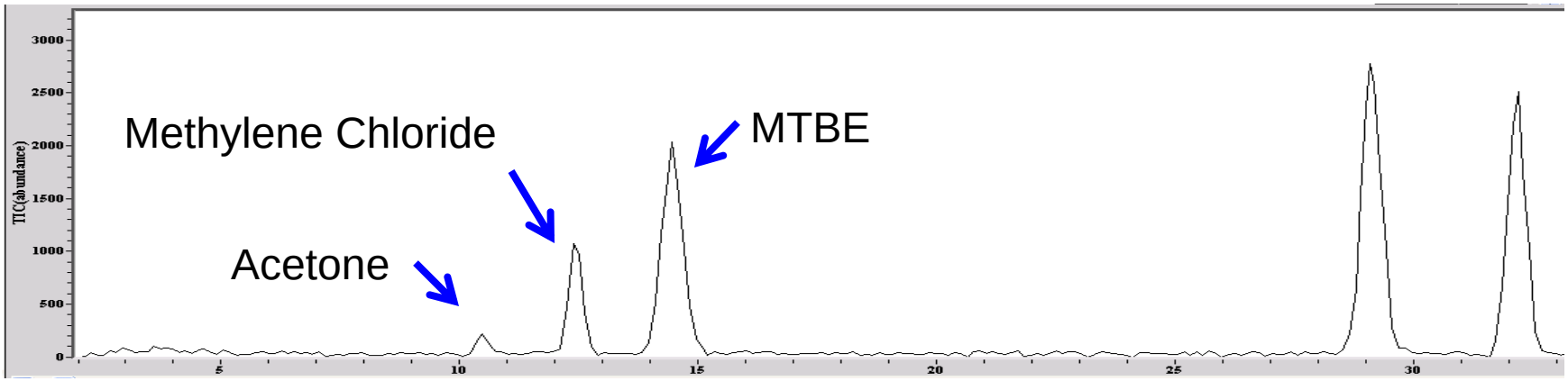


- Carryover < 1.5% at 2000ppm

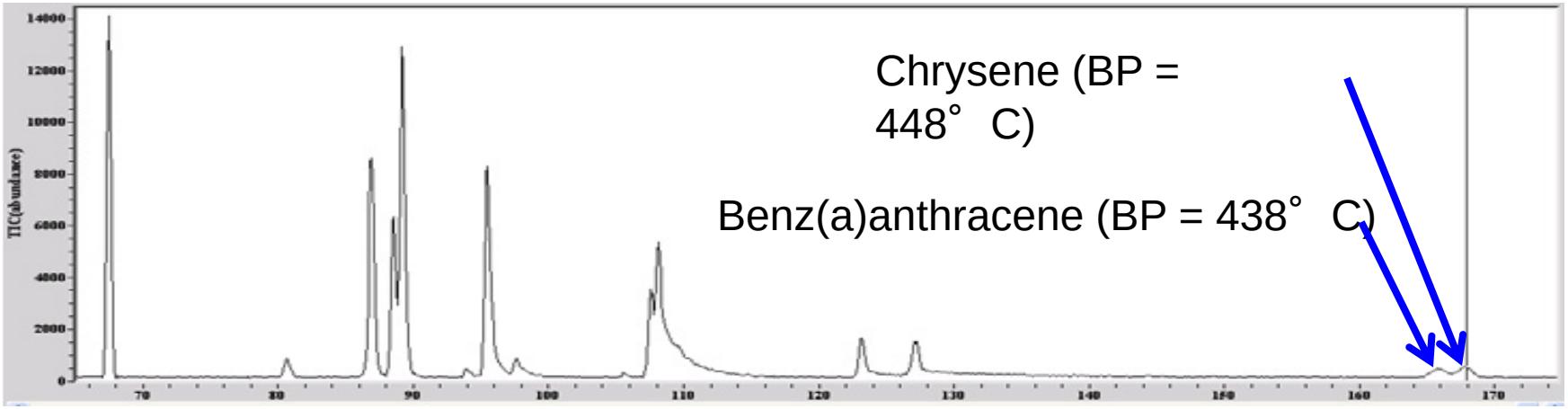


Chromatographic

- Low boiling point separations

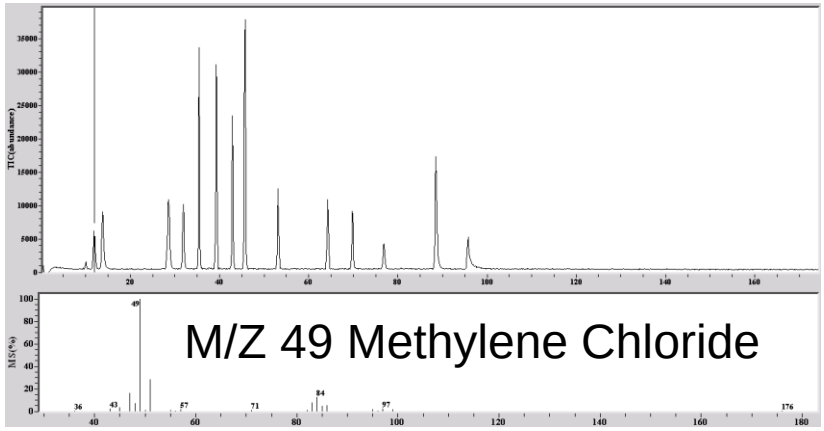


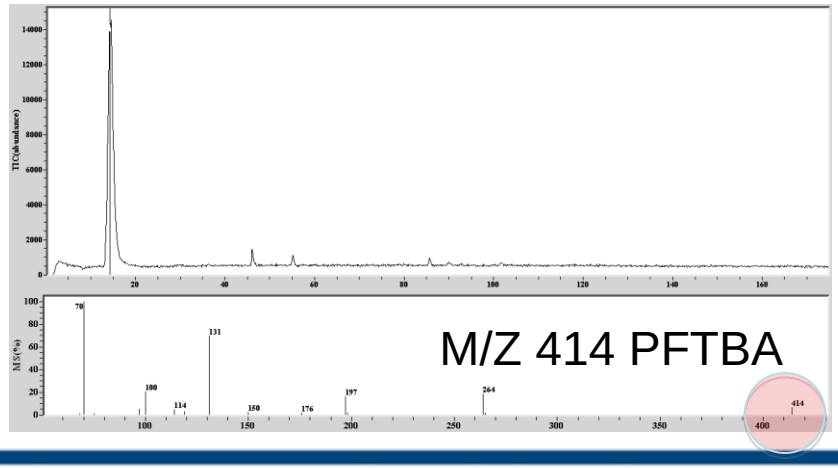
- High boiling point performance



Mass Analyzer

- Mass Range 45 – 500
  - Demonstrated to m/z 442 using DFTPP





- Spectral Quality
  - Three consecutive days 8 samples per day at 1 hour intervals
  - Mass fragments for Perchloroethylene

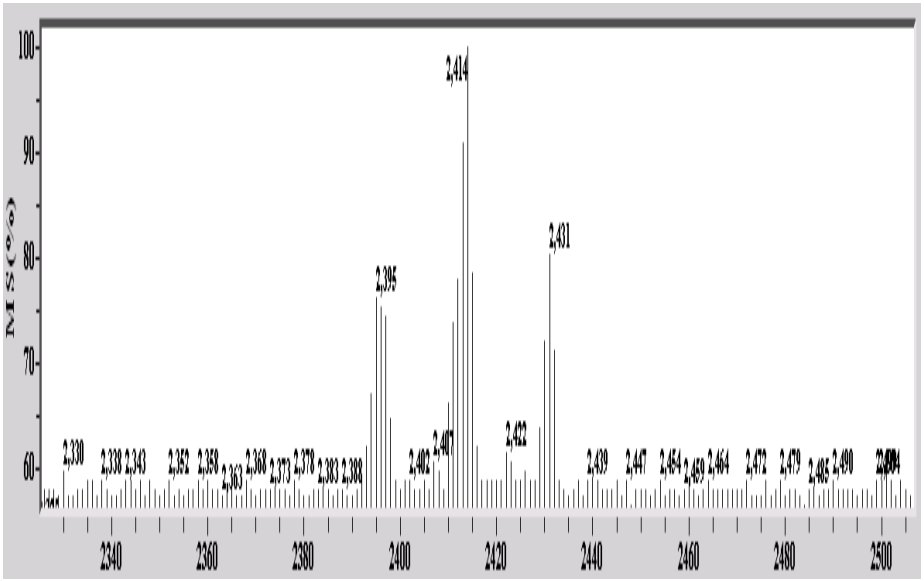
Mass	Relative Abundance
166	110 – 140 % of 164
168	40 – 70% of 164
170	5 – 20% of 164
172	0 – 5% of 164
165,167,169	< 3% of 164

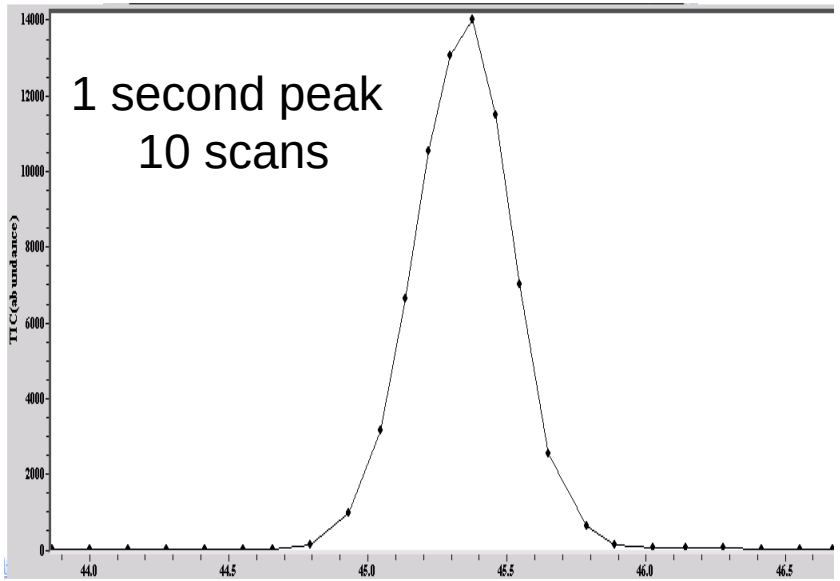
Bromoform

Mass	Relative Abundance
171	35 – 60% of 171
175	35 – 60% of 173
172,174,176	< 3% of 173

Mass Analyzer

- Resolution < 1 amu FWHM across the mass range, evaluated twice per day for the period of the evaluation m/z 49, 173 (shown), 308
- Scan Speed > 5 scans/second

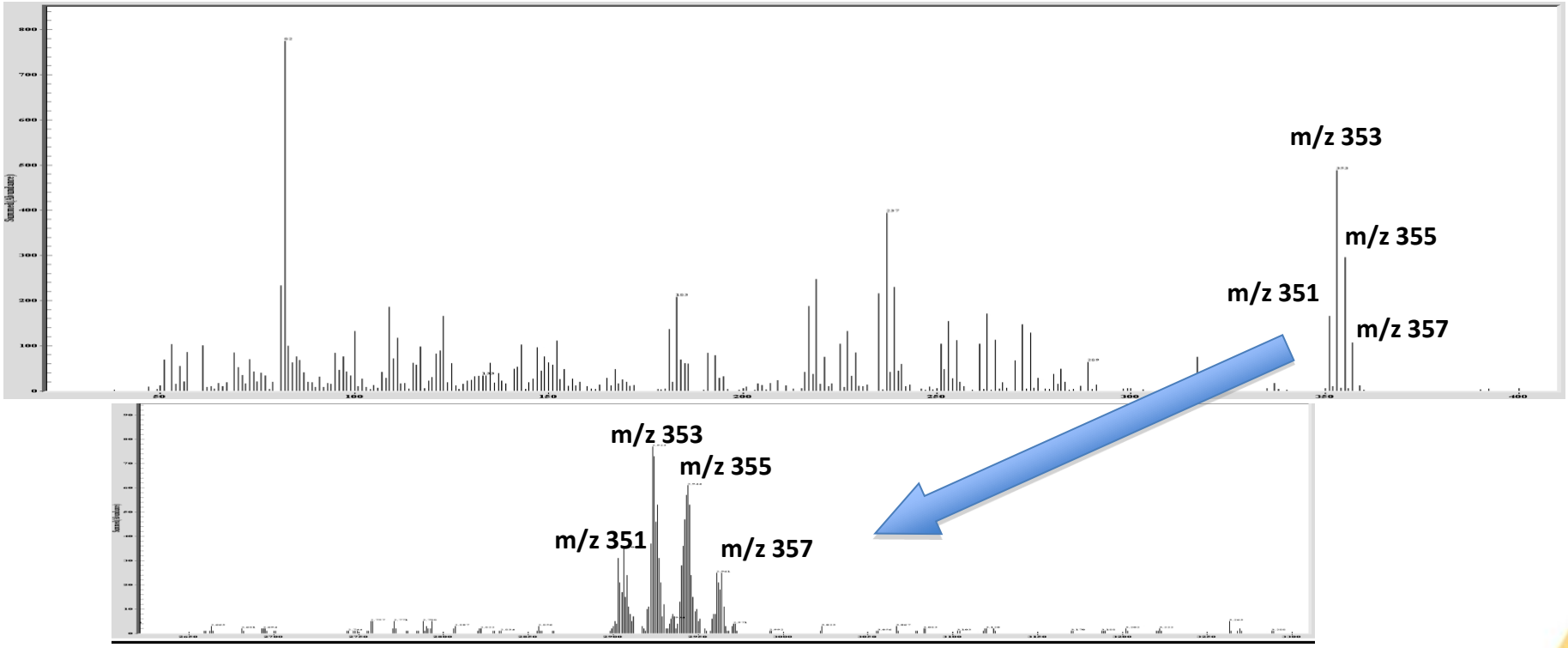




High Mass Range Resolution

Heptachlor Epoxide

Fragment Cluster at m/z 353



Mass Analyzer

- Mass Calibration Stability
  - Monitored daily at 22°C during the test period M/Z 49,100, 173, 308 during the test period

F04 - DAY 2 at ambient conditions											
	Fragment	Cal. Pt	Hr 1	Hr 2	Hr 3	Hr 4	Hr 5	Hr 6	Hr 7	Hr 8	Pass/Fail?
			7:39	*8:41*	*9:48*	*10:53*	*12:00*	*1:01*	*1:58*	*3:06*	
Methylene Chloride	***49***	***116***	115	115	115	115	115	115	116	117	Pass
Toluene d8	***100***	***555***	554	554	554	555	554	554	555	555	Pass
Bromoform	***173***	***1204***	1204	1203	1203	1203	1203	1202	1203	1204	Pass
DBTFB	***308***	***2419***	2419	2419	2419	2418	2418	2418	2418	2420	Pass

  - Ambient temperature shift of 15°C, 20°C to 35°C

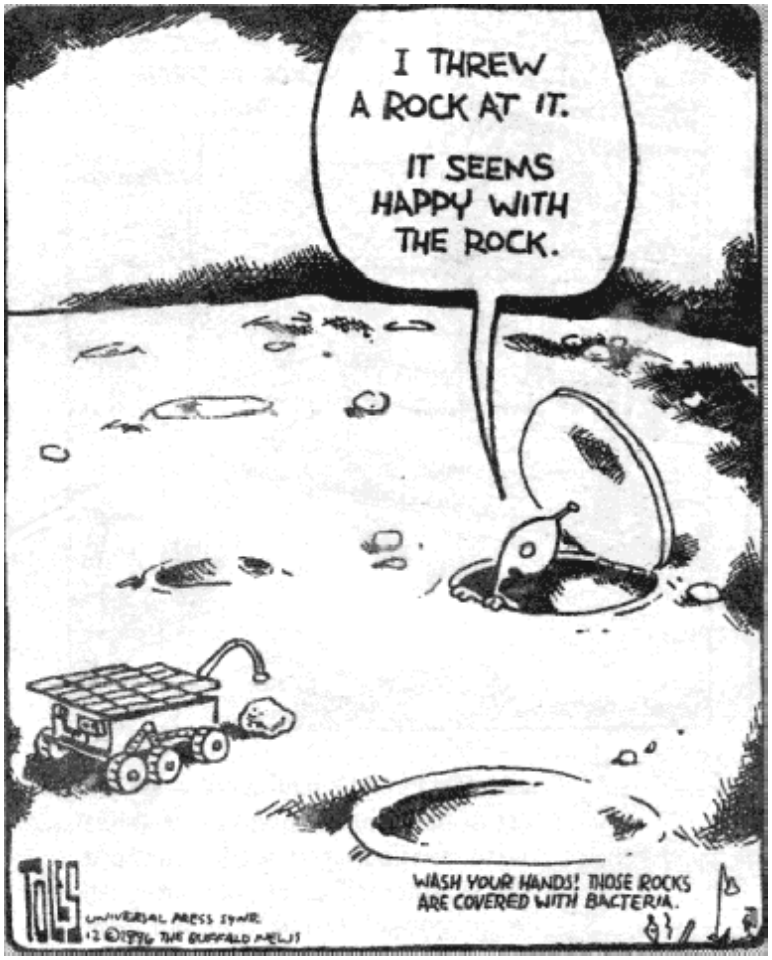
F04 - Average deployment (June 30)							
	Fragment	Cal. Pt.	Hr 1	Hr 2	Hr 3	Hr 4	Pass/Fail?
		24.2C	*20.2C*	*25.1C*	*30.2C*	*35.2C*	
Methylene Chloride	***49***	***114***	114	115	114	116	Pass
Toluene-d8	***100***	***554***	554	554	553	555	Pass
Bromoform	***173***	***1200***	1200	1200	1200	1202	Pass
DBTFB	***308***	***2413***	2413	2414	2413	2417	Pass

Summary

- Extensive Mechanical, Environmental and Analytical performance test were carried out to verify the design and operation of a Hand Portable GC-MS. The design goal was to produce a system that was capable of operation by users at all skill levels in Harsh Environments. Example environments include all climate zones from Alaska to the Utah desert, at drill rig sites, hazardous waste sites for rapid environmental site assessment, and use by deployed troops and first responders around the world .
- Additional third party testing is scheduled to validate internal test

Low Pressure (Altitude)(Intsrument, Instrument in shipping case, & Accessories Shipping Case (populated))	MIL-STD-810G (500.5)
High Temperature (Operational)	MIL-STD-810G (501.5)
High Temperature (Storage)	MIL-STD-810G (501.5)
Low Temperature (Operational)	MIL-STD-810G (502.5)
Low Temperature (Storage)	MIL-STD-810G (502.5)
Temperature Shock	MIL-STD-810G (503.5)
Contamination by Fluids	MIL-STD-810G (504.1)
Rain	MIL-STD-810G (506.5)
Humidity	MIL-STD-810G (507.5)
Sand and Dust	MIL-STD-810G (510.5)
Explosive Atmosphere	MIL-STD-810G (511.5)
Acceleration	MIL-STD-810G (513.6)
Vibration	MIL-STD-810G (514.6)
Shock (In shipping case)	MIL-STD-810G (516.6)
Shock (Outside of shipping case)	MIL-STD-810G (516.6)
Discrete component shock/drop (GC module, trap module) in shipping case	MIL-STD-810G (516.6)

Sorry, not qualified for a MARS mission…yet!



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