#### Denton Research Group's Advanced Detector Technology

• Optical Spectroscopy

#### • Raman Spectroscopy

- Atomic Emission Spectroscopy
- Molecular Fluorescence Spectroscopy
- Array Infrared Spectroscopy
- Imaging Spectroscopy
- Mass Spectrometry
  - Magnetic Sector
  - Ion Mobility
  - Secondary Ion Mass Spectrometry
  - Quadrupole
  - Time of Flight
- X-Ray Techniques
  - Diffraction
  - Fluorescence
  - X-Ray Photoelectron Spectroscopy

# Present Ion Detector Technology – Faraday Cups



•Gain is stable and precisely known (gain=1)

•Bandwidth is consistent with use in sector-based mass spectrometry

•Useful for I<sub>ion</sub> ≥ 10<sup>-15</sup> amp (1 ion/sec = 1.6 × 10<sup>-19</sup> amps)

•Implies that one needs about 6250 ions/sec for detection by Faraday cup

**Operates at any pressure !** 



## **Photomultiplier Tube**





#### **Ion Detection with "Multiplication"**



Today in Mass Spectrometry Array Detectors are not Utilized!!!

## Mattauch-Herzog Mass Spectrometer Geometry



## Mattauch-Herzog Mass Spectrometer Geometry









## Mattauch-Herzog Mass Spectrometer Geometry



#### Mattauch-Herzog Mass Spectrograph



### Mass Spectrum made from several Mass Windows



Conditions: 10 ppb multi elemental solutions, 5.011 ms Integration, 100 Reads, 20 replicates, 10.022 s total integration time, 1 ADU =  $\sim$ 2.4 counts

#### **Isotope Ratio Precision**





**Concentration (pptr)** 



#### Variable gain CTIA electronics





#### DM0003 Layout





#### **Detection Limit**

2 electrons of read noise with NDRO

# 6 <u>IONS</u>!

& we are still optimizing it !!

Keep tuned

## Issues to be Addressed (1) Pixel Spacing

Smallest practical using Wire Bonding Connections is  $\approx 50$  microns









Bump Bonding Allows Connections to as Small as a Few Microns

Ion Collection fingers



## Monolithic Collection Electrodes

Electrode "fingers" Fabricated On IC Die with Amplifiers

CMOS die

- Chip layout measures 6410um x 1950um.
- There are 29 bonding pads on 150um pitch.
- o 48 Detector array is 5mm x 25um.





#### DM0015 Block Diagram / Floor Plan











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LabVIEW



File Edit Operate Tools Browse Window Help



#### **Rare Earth Elements**



10 ppb multi-elemental solution, 0.5 s Integration Time








## Cycloid MS Equipped with an Array



#### B field up out of plane



### **Copper block for mounting array into Monitor Instruments Cycloidal Mass Spectrometer**

Technology Currently Deployed for Explosives (IED)

Ion Mobility Spectrometers (IMS) Require particles ~ micrograms

> Swipe Tests "Puffers" blow particles loose

> > These can **NOT** "smell" explosives !!!

Major suppliers Smith's Detection General Electric Security Many , many others

## **New Ion Detectors**

**Capable of Very High Sensitivity** 

**Operate at High Pressure (no upper pressure limit )** 

**Sufficient Frequency Response (currently to 1 MHz)** 

Ability To Operate in High Background Noise Environments

Inexpensive ( in production ) Multi Gain Levels – Allows implementation of Automatic Gain Control – Important when Deployed in High Background Environments



## **Chemical Identification Based Upon Ion Mobility**



Note: The analyzer is operated at or near atmospheric pressure

# **Development of A Handheld IMS** for Explosive Detection

### New interlocking 8 ring microIMS





25pg TNT -1825VDC Emco PS, 90C 100/50 ml/min Cl-/Air Filament, inj 125C. Filters OFF Resolution 84. Run a baseline noise 0.005 S/N=30 Detection Limit = 2.5 pg







Blank, Cl-/air 110C, Lid ON - same STD Det, fan on Base noise level 0.002-0.0025



### Blank, Cl-/air 110C, Lid OFF - same STD Det, fan ON Base noise level 0.040-0.050 (ie 20X worse)



# Subtracting Environmental Noise with Independent Amplifiers

- Internal amplifier noise is uncorrelated
- Amplifier noise from both preamps adds
- Clipping can occur



## **Differential Ion Detector**



without noise

# True Differential Input Amplifier

(1) Noise is canceled in input stage(before high gain amplification)

(2) True correlated subtraction

(3) Balanced FETs help ensure equal subtraction
(4) Noise cannot limit amplifier



Pad 1

- Chip layout measures 1795um x 2695um.
- Cut die size can be up to 2000um x 2900um.
- Bonding pads have 150um pitch.





Blank, Hot, Lid ON – same Differential Det, Cl-/Air, 90C Base noise level 0.003-0.004



Blank, Hot, Lid OFF – same Differential Det, Cl-/Air, 90C Base noise level 0.0035-0.004 (no noise degradation)



12.5pg TNT 96C, 25V Injector Block, 0.85A Filament, 1200us Pulse, B.C. FIRW On



5pg TNT 95 °C 25V Injector Block 0.85A Filament, 1200 us Pulse B.C. FIRW on Detection limit 400 fgm

4 hours @ 0.5 w Rechargeable Wireless capable Vapor sampling



Version 4.0 future 1.5 X 2 X 4 in. 4 to 6 hours

First field prototype for Fall 2006 Version 2.0 3x3x9"

Version 4.0 is where we want to go for our customer -2 years out.

## TNT Vapor Analysis

No background correction
Vapor emanating from glass surface exposed to 9 ppb TNT vapor
Non-optimized system



# **RDX Vapor Analysis**

No background correction
Vapor emanating from glass surface exposed to 4 ppt RDX vapor
Non-optimized system



Development of a Ultra High Sensitive Explosive Detection System

#### New System for Achieving parts per Quadtrillion Detection Limits



High Volume Ion Source - high ion flux from low concentration vapor pressure explosives

Relative Large Diameter Analyzer Tube - minimize spacecharge effects ie. higher resolution

Ion funnels - provide ion compression between stages

## Atmospheric pressure DC Ion Funnel





### **Vehicle & Checkpoint Based Explosive Detection System**

Progress






Signal Detector Pin Location

> Reference Detector Pin Location

### Aperture Grid & Detector But



### Version I - For laboratory evaluation

## Standoff Testing

**Note - Currently funding is for other applications** 

### NOT Explosives or Improvised Explosive Devices !!

(The System must be modified for optimal Explosive Detection)





#### Cmpd.A 90 feet down the hall (open vial) 11-30-06, low $gain = \pm 100$

\_ 8 ×





#### Cmpd.A 90 feet down the hall (vial closed for 20 minutes) 11-30-06, low gain = $\div$ 100





100 mg TNT @ 135 ft, 12-16-08





#### 100 mg RDX 50 °C @ 70feet





100 mg RDX 24 deg C w/ ring fan on at top; distance 5'



### Sample Capped

### Remember

Until three months ago there was NO funding for explosive detection with the super sensitive Instrument.

## Field Tests

- Initial field tests using compounds selected for customer applications (Not Explosives) conducted 12-15- 2007
- Outside tests reported conducted on 4-1-2008 & 4-3-2008
- Inside tests reported conducted 4-4-2008
- Proposed modifications for additional sensitivity have not yet been incorporated !!















### One of many other scenarios suitable for the Super Sensitive Explosive Detection Instrument's Deployment





### On a larger scale highly portable check points can be implemented using tents etc. etc.



#### **Vehicle based Instrument hunts IED's**



Escalators , elevators or even corridors provide excellent opportunities for real time vapor phase screening



# Raman Spectroscopy The "Awakened Giant" of Molecular Spectroscopy

Modern Raman Instrumentation

Stable

Rugged

Compact

Inexpensive ?

## Basic Raman Spectrometer Block Diagram



# **Raman Scattering Intensity**

$$\Phi_{\rm R} \propto \sigma(v_{\rm ex}) v_{\rm ex}^4 E_0 n_{\rm i} e^{-E_{\rm i}/kT}$$

 $\Phi_{\mathbf{R}} \equiv \mathbf{Raman} \mathbf{scattering} \mathbf{intensity}$ 

 $\sigma(v_{ex}) \equiv$  Raman cross section (analyte dependent)

 $v_{ex}^4$  = Frequency of excitation energy (4th POWER)

 $E_0 \equiv Energy of source irradiance$ 

 $n_i \equiv$  Number density of state i

 $e^{-E_i/kT} \equiv$  Boltzmann factor for state i

Number of available scattering centers

#### First Excited Electronic State



V<sub>0</sub>
#### First Excited Electronic State



#### What Wavelength Should be Used?

### Problems Associated with Laser Wavelength

- Fluorescence
- $v^4$
- Self absorption
- Laser availability
- Detector availability

# Raman Spectral Range Dependence on Excitation $\lambda$



## 785 nm

- 0 3500 cm<sup>-1</sup> = 785 1100 nm
- Shot noise limited detection with a CCD
- Few electronic absorption bands at or below 785 nm
- No significant NIR bands above 1100 nm
- Inexpensive, durable diode lasers
- $v^4$

#### **Raman Spectrum of a Carrot**



#### **Beta**-Carotene



## Beta-Carotene in Plastic Bottle



#### **Omnic Library Match Results**



Partnering with BaySpec's which is a successful manufacturer of Telecom Optics including Holographic Optical & Dispersive Elements







- Holographic optics enable large volume production of high efficient, rugged, low noise, low cost gratings
- High efficiency, compact spectrographs
- Transmission gratings enable Spectral Imaging capabilities

#### BaySpec's VPG based Holographic Optical & Dispersive Elements



- Holographic optics enable large volume production of high efficient, rugged, low noise, low cost gratings
- High efficiency, compact spectrographs and Notch filters
- Transmission gratings enable Spectral Imaging capabilities

## VPG<sup>TM</sup> – Volume Phase Gratings

- Class 100 manufacturing facility at BaySpec
- Widely used in spectroscopic instrumentation, military/defense head-up display, telecom, medical diagnostics, and other demanding applications
- Works in Transmission mode
- Wide wavelength range, 300-3000nm
- High and equal P&S diffraction, Peak diffraction efficiency >98%
- Polarization effects minimal, PDL <0.1dB
- Ruggedized, no affected by shock or vibration
- Fast production cycle of 1-2 days allows for easy customization
- Athermal design feasibility
- Low cost, large qty production capability







#### Miniature Spectral Engine-Design and Design Diagram ~1% AR coating (optional) Input Cover Grating AR coating (optional) **To Host** detector ontro DSP Array Memory Electronics

#### Major Reliability Benefits:

- No moving parts
- Rugged Volume phase gratings
- Solid state electronics
- Hermetic seal of individual units



### Smaller than a Blackberry

## Handheld 1064 nm Raman For Substance Identification

- Specifications
  - Range:  $200 \text{ to } 3000 \text{ cm}^{-1}$
  - Resolution:  $5-7 \text{ cm}^{-1}$
  - Acquisition Time: 10 sec
  - Size: Handheld
  - Weight:  $\sim 1 \text{ lb}$
  - Power Consumption: 10 W peak

#### NIR Spectroscopy/Imaging With Focal Plane Arrays



•Ideal NIR sensor will have high QE between 900-1700 nm, high sensitivity, high dynamic range.

# Raman Spectral Range Dependence on Excitation $\lambda$



## **Richard Reid**



### 22nd December 2001 "Shoebomber"







"New directions in science are launched by new tools much more often then by new concepts. The effect of a concept-driven revolution is to explain old things in new ways. The effect of a tool-driven revolution is to discover new things that have to be explained."

> Freeman Dyson, Imagined Worlds as quoted by Dudley Herschbach, Nobel Laureate in Chemistry



## Previous Record A/BMS

- Qualifying 267.555 (430.496 KPH)
- Return 260.459 MPH (on 5 cylinders)
  (419.079 KPH)

 Average 264.007 (424.787KPH) Also hold AA/BMS Record @ 254.135 MPH (408.903KPH)







Down Run 283.908 MPH (456.808 KPH) Blew motor Caught on Fire could not make return run!

Are we having fun yet?

## We were Just WAITING until this August !

I had regeared the car for

316 MPH (508KPH)

(assuming no slippage)

(for this Aug. 18-24)





## Bonneville 2008

- Down Run
- QUALIFIED @ 292.241 MPH (470.261 kPH )
- Exit speed 301.138 MPH (484.531 kPH )
- Record Run 305.726 MPH (491.913 kPH)
- Exit speed 310.398 MPH (499.430 kPH)
- Record 298.983 MPH (481.064 kPH)



