4th Harsh Environments Mass Spectrometry Workshop, 9th October 2003

Field Portable MS for Explosive Detection

David Groves

Detection Department,

Dstl Fort Halstead, Sevenoaks, Kent, UK

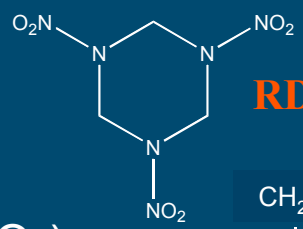
DMGroves@dstl.gov.uk, +44 (0) 1959 892448

Introduction

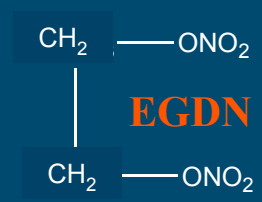
- Explosives detection
  - Why MS for explosive detection
- CITMS
  - pMSED
  - FE
  - GAT ‘Minotaur’
- ‘TOF’
  - Dynatronics ‘Isostach’

Detection of Explosives

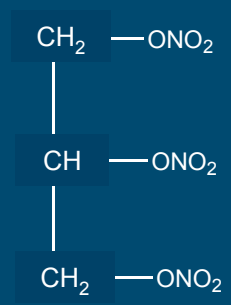
- Traditional organic explosives
  - Nitramines ($N\text{-}NO_2$)
  - Nitrate esters ($C\text{-}ONO_2$)
  - Nitroaromatics ($Ar\text{-}NO_2$)
- Taggants
  - DMNB



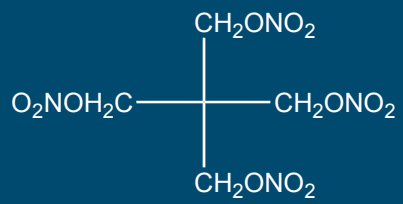
RDX



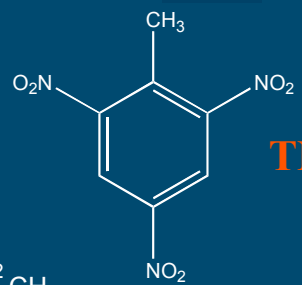
EGDN



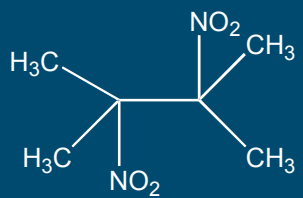
NG



PETN



TNT



DMNB

Challenges of Explosive Detection

- Small molecules
- Wide range of vapour pressures
- Readily condense & 'sticky'
  - heated inlets/systems
- Prone to thermal decomposition
- Extensive fragmentation in EI
  - often only 46 *m/z* ions observed
- High electron affinity
  - negative ionisation gives selectivity
    - nCI, APCI, ASGSI, e monochromators

Compound	V_{sat} @ 25 C pg/l
EGDN	6.4×10^{8}
DMNB	1.6×10^{7}
NG	5.4×10^{6}
DNT	2.2×10^{6}
TNT	8.4×10^{4}
PETN	1.7×10^{2}
RDX	2.9×10^{1}

Cylindrical Ion Traps (CIT)

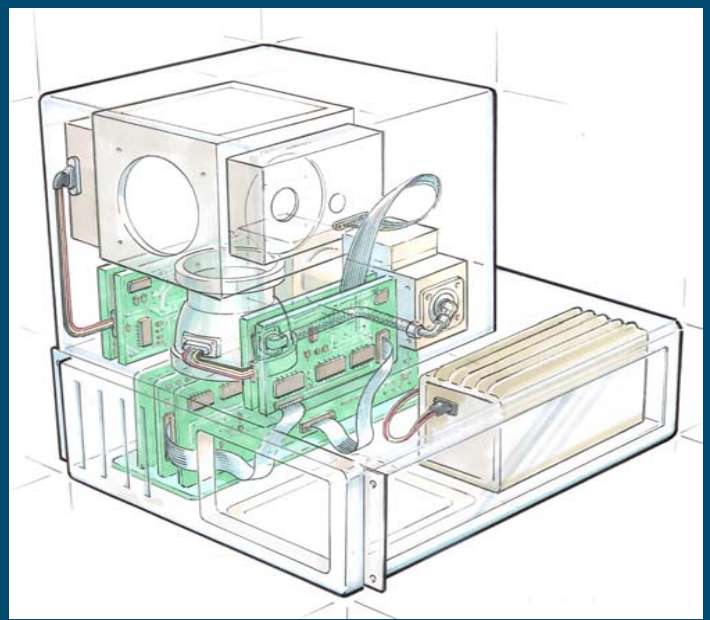
- Cylindrical Ion traps (CIT)
  - cylindrical ring electrode, planar endcaps
    - easier and cheaper to manufacture and miniaturise
    - lower voltage requirements
- CITs can easily form arrays
  - reduce space charge
  - increase sensitivity over single CIT
  - different size CITs in one array to trap different mass ranges
    - reducing electronics and power requirements

Dstl CIT systems

- 2 ‘Clone’ CIT systems assembled

- GC-CIT

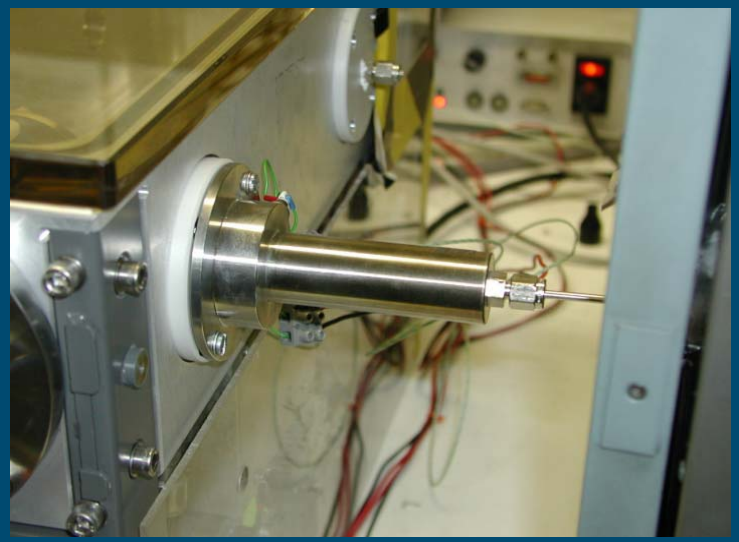
- pMSED



GC- CITMS

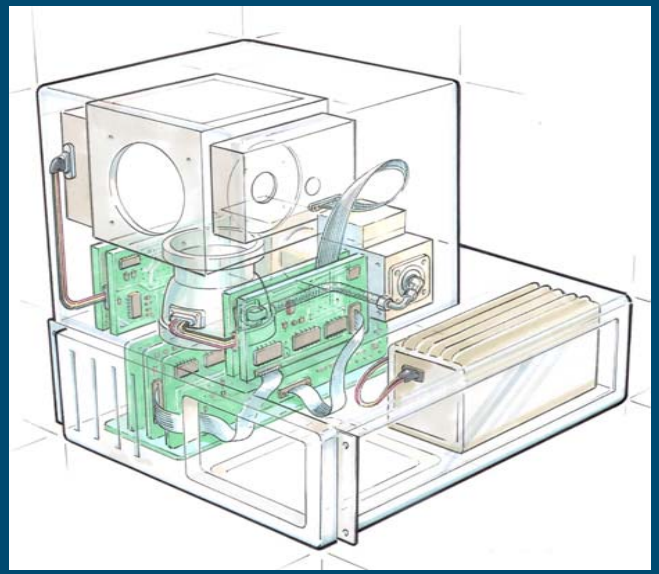
- Assembled to provide reproducible & quantifiable sample introduction

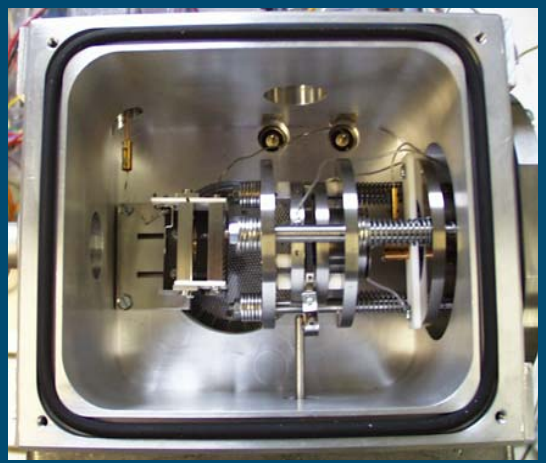
- Custom built heated GC interface based on a transfer line for a MS800.



Portable MSED

- In-house designed manifold (15 x 18 cm)
- Entire instrument mounted in 19” rack box



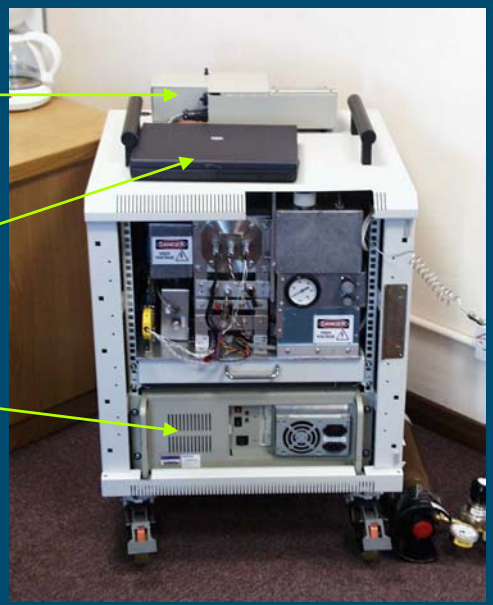


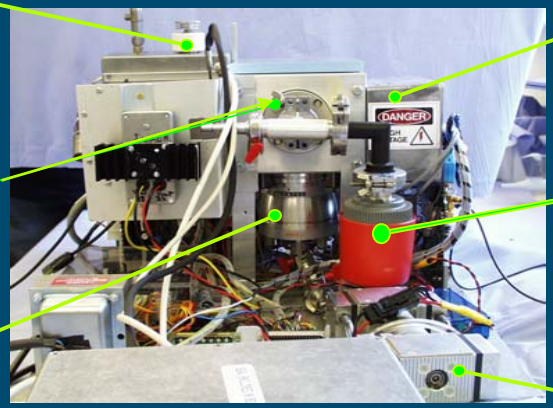
System Description

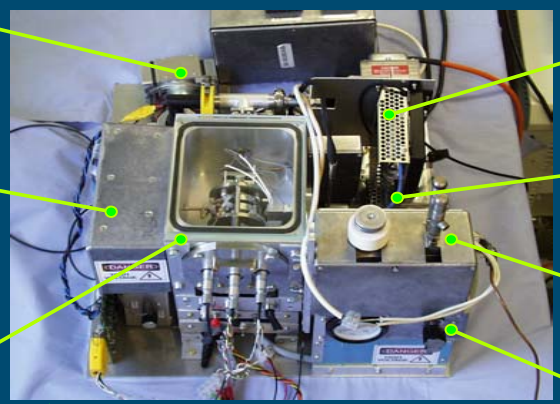
- CIT [1]: r_0 = 2.5 mm, z_0 =2.35, endcap orifice 2mm ∅, sealed with Teflon spacers, heated
- Vacuum system: Alcatel ATH31 TMP and KNF 813 diaphragm
- Pressure: 6 x 10^{-4} Torr in trap (estimate), 6 x 10^{-5} Torr in manifold
- Ionisation source: Filament
- Detector: K & M model 7707mh3
- Rf trapping signal: approx. 2MHz, in house tuned LC tank circuit
- Control software: Teledyne 3DQ
- GCs: Carlo Erba HRGC 5300 and Fisons AS800

[1] Badman, E.R., Cooks R.G. *Anal. Chem.* **2000,** 72 5079-5086

pMSED



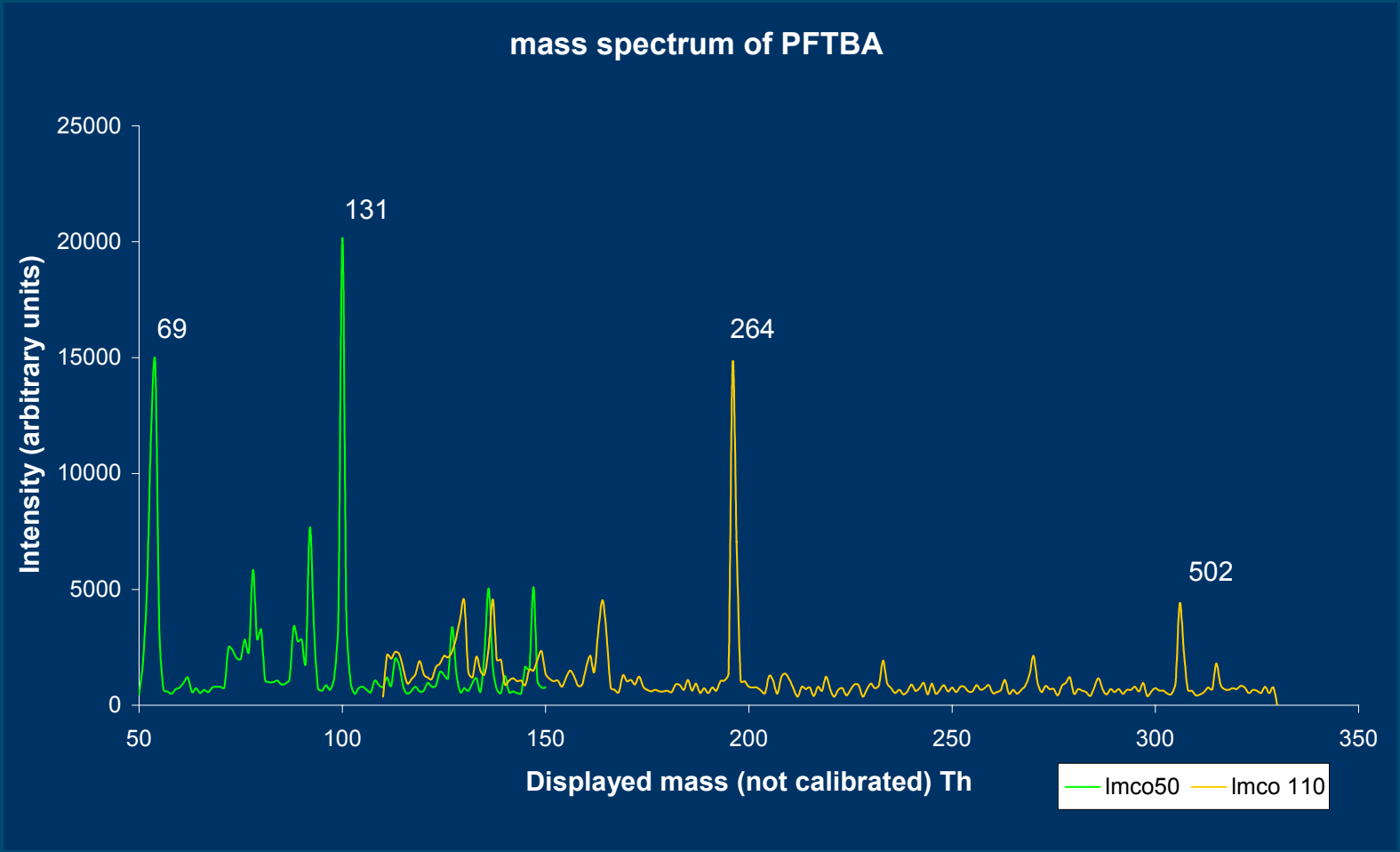




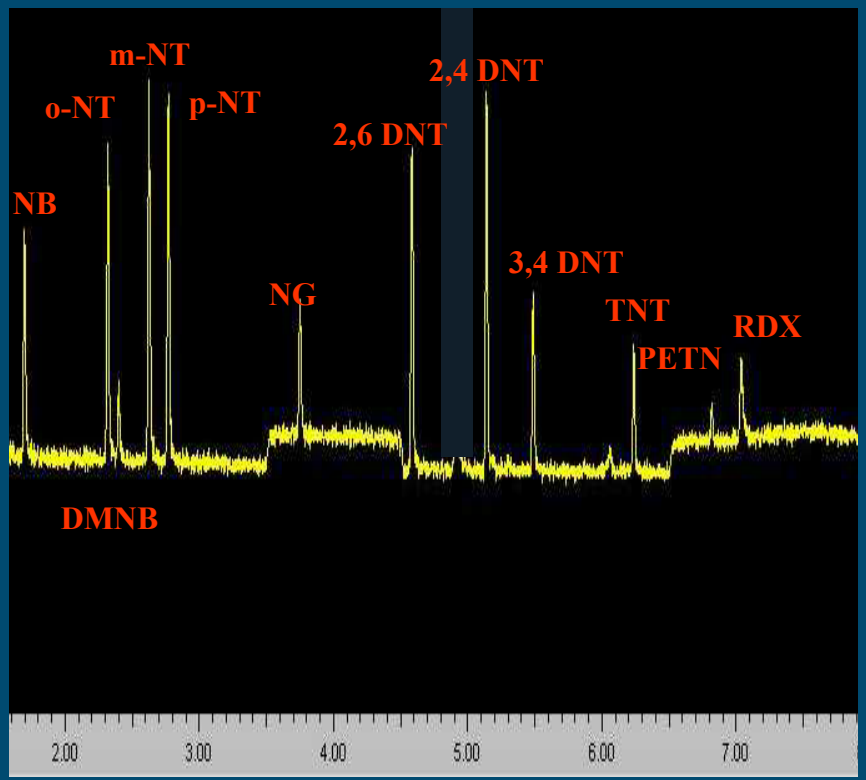
System Performance

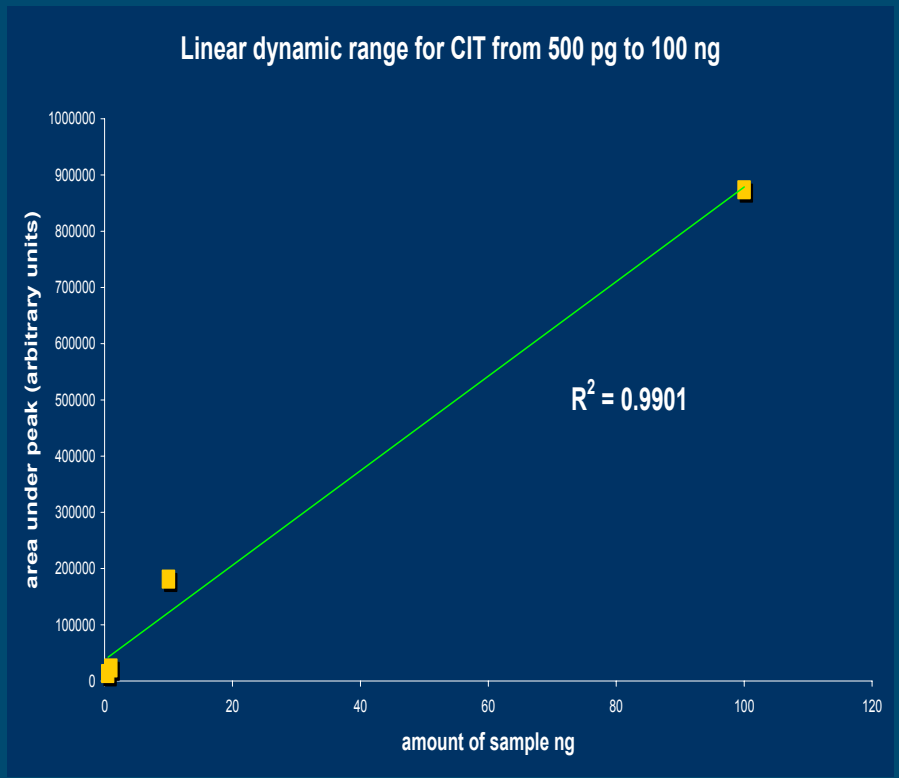
- EI spectra obtained for: Full range of organic explosives
- Mass range: ~ 40 - 550 amu
- Resolution: 2 amu
- MS/MS efficiency: Ca 45 % for n-butylbenzene
- Dynamic range: 10^3

Mass Range

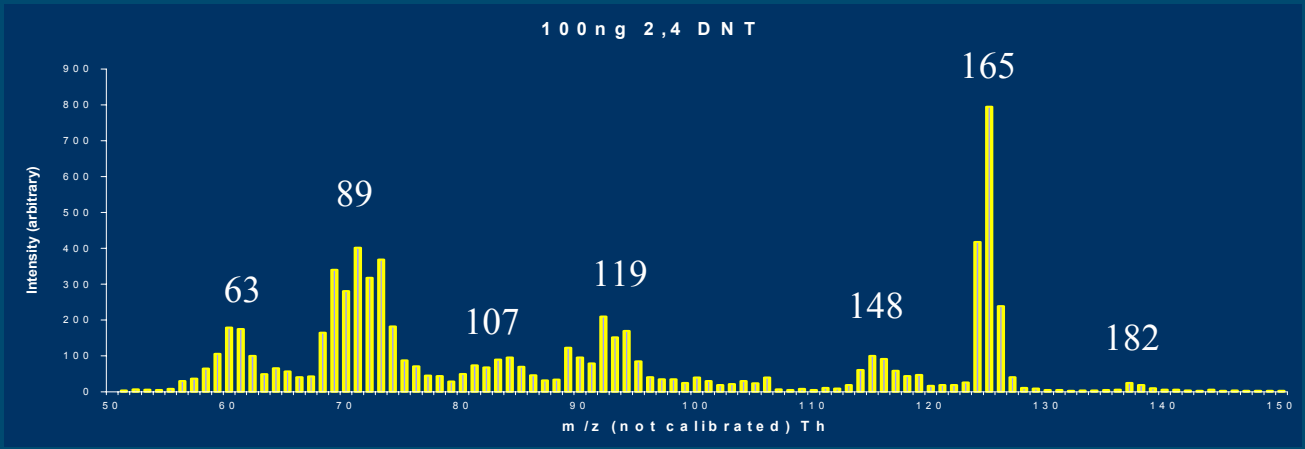


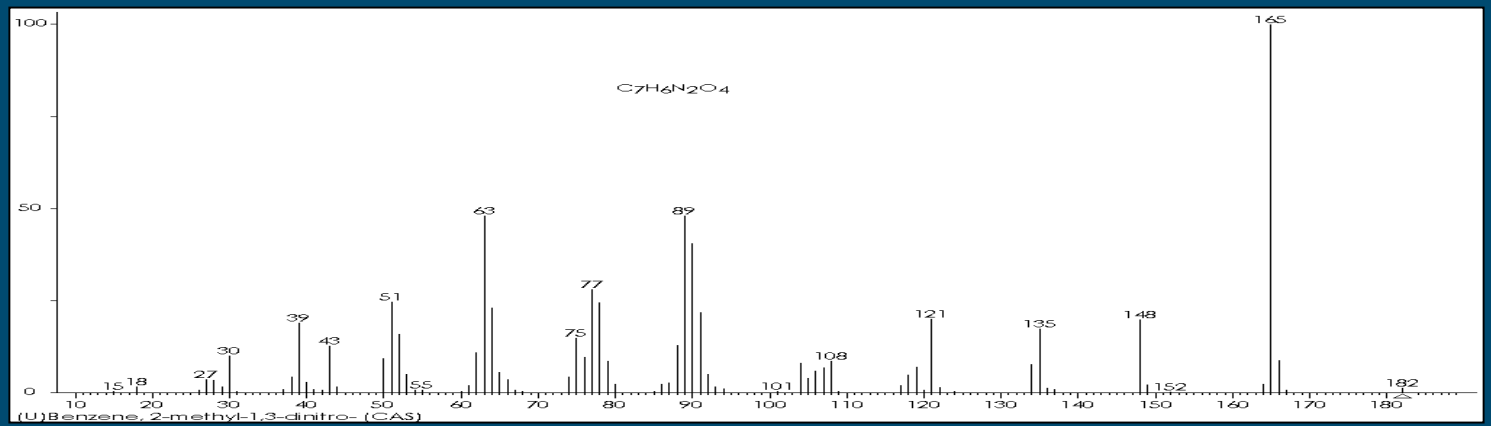
GC TIC chromatograph





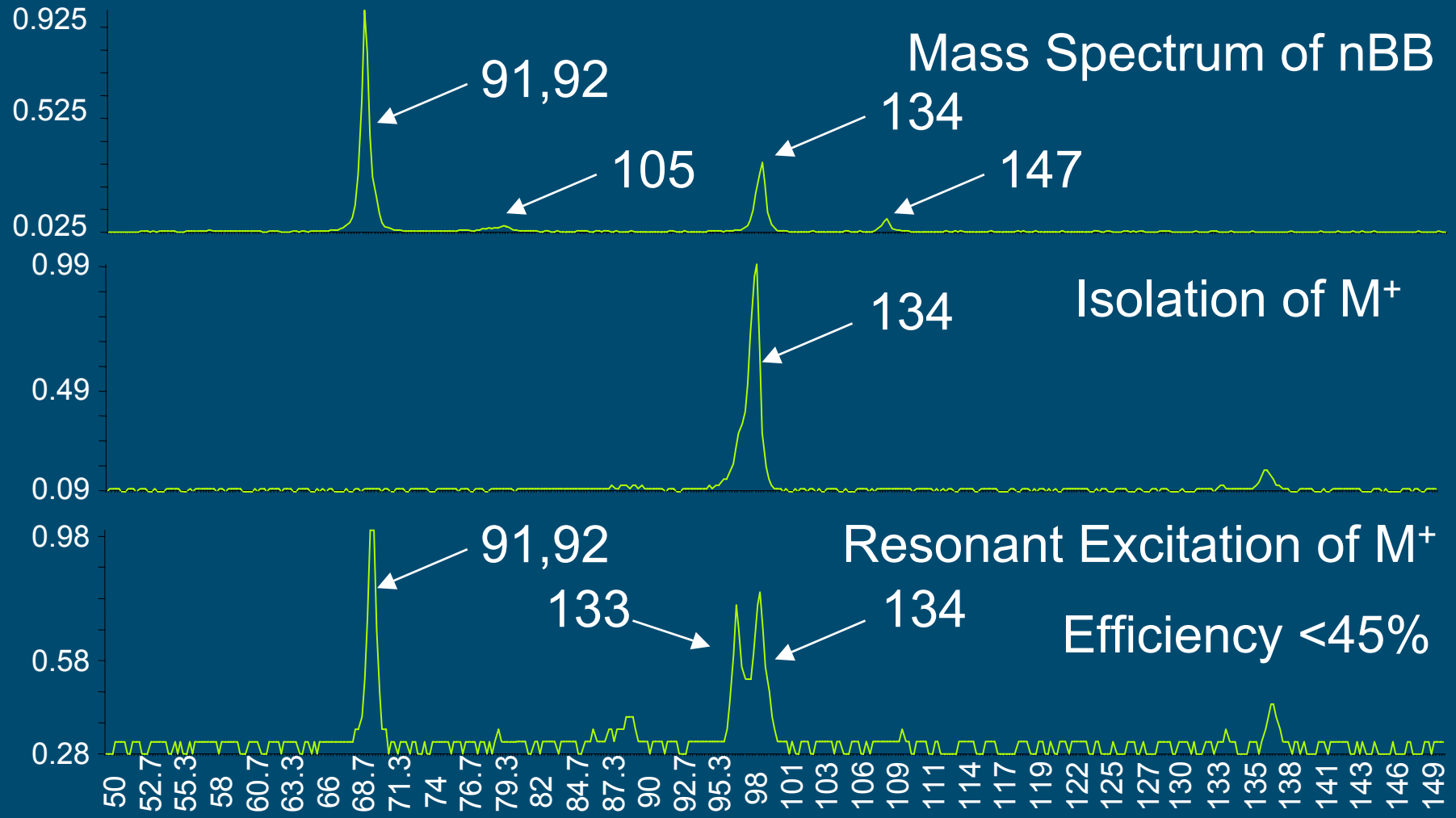
2,4 DNT EI Spectrum





Dstl is part of the Ministry of Defence

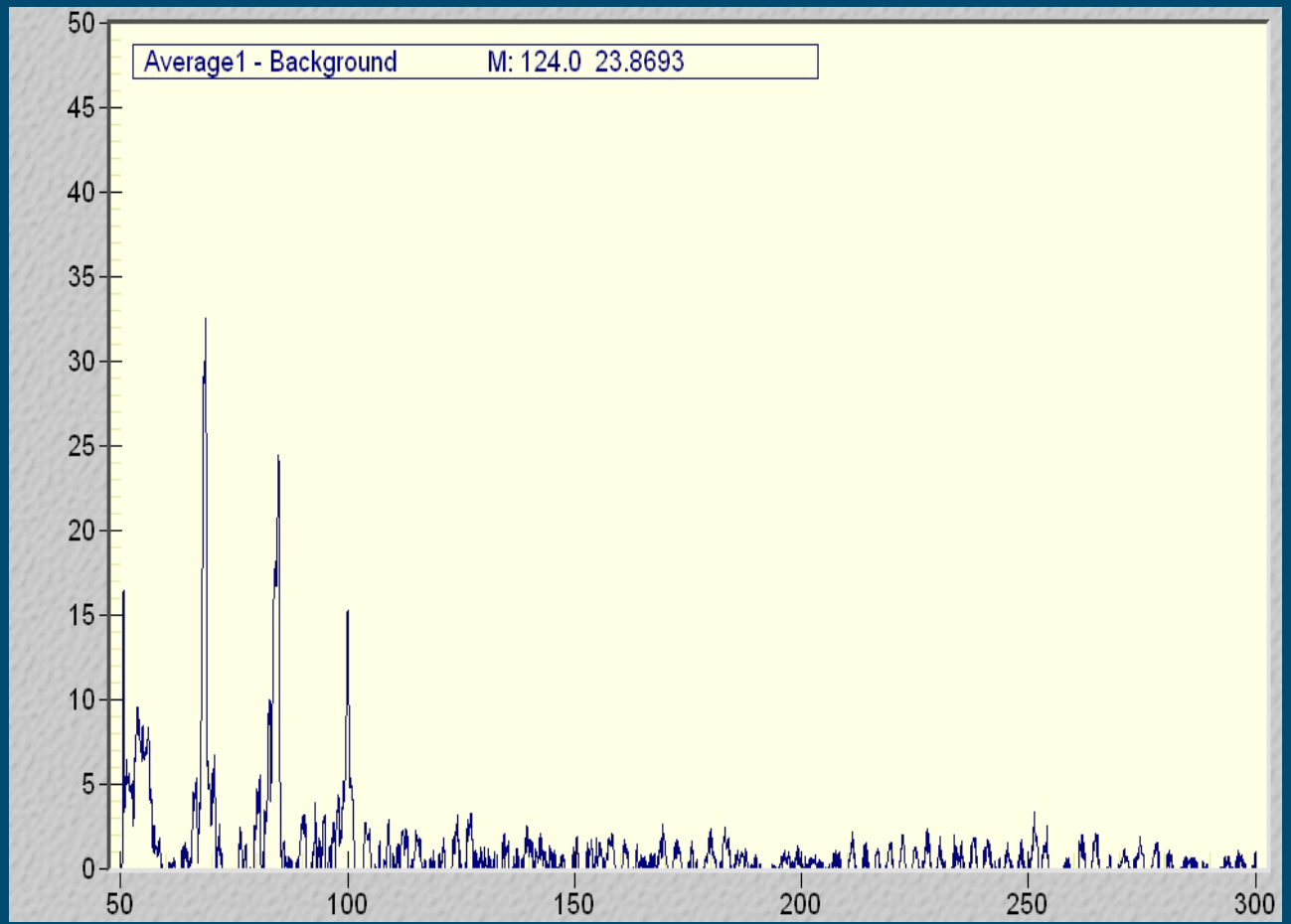
MS/MS of nBB



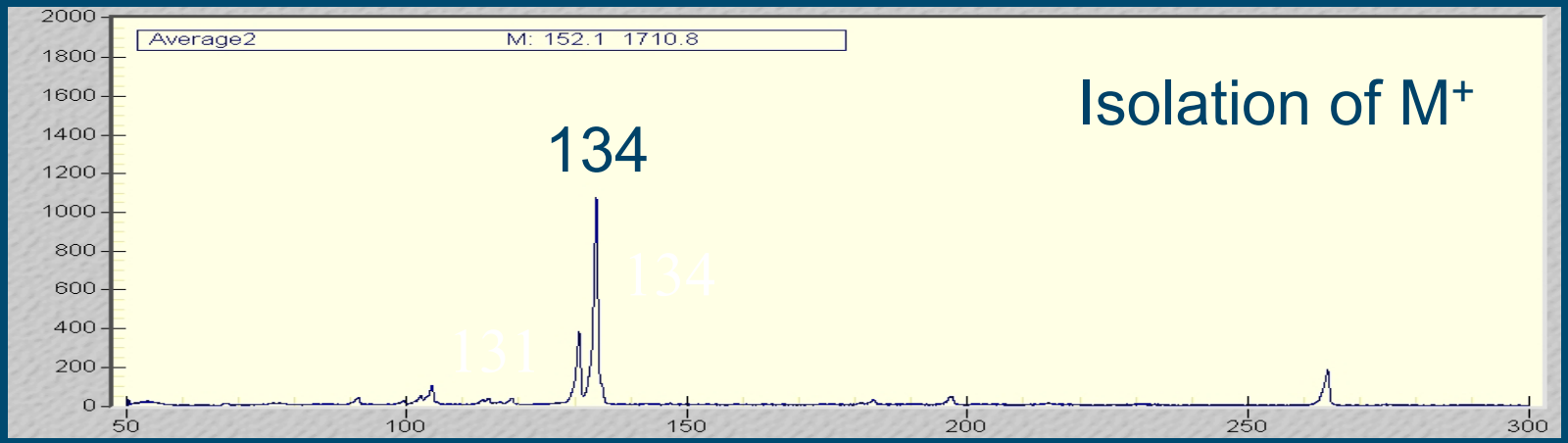
GAT ‘Minotaur’

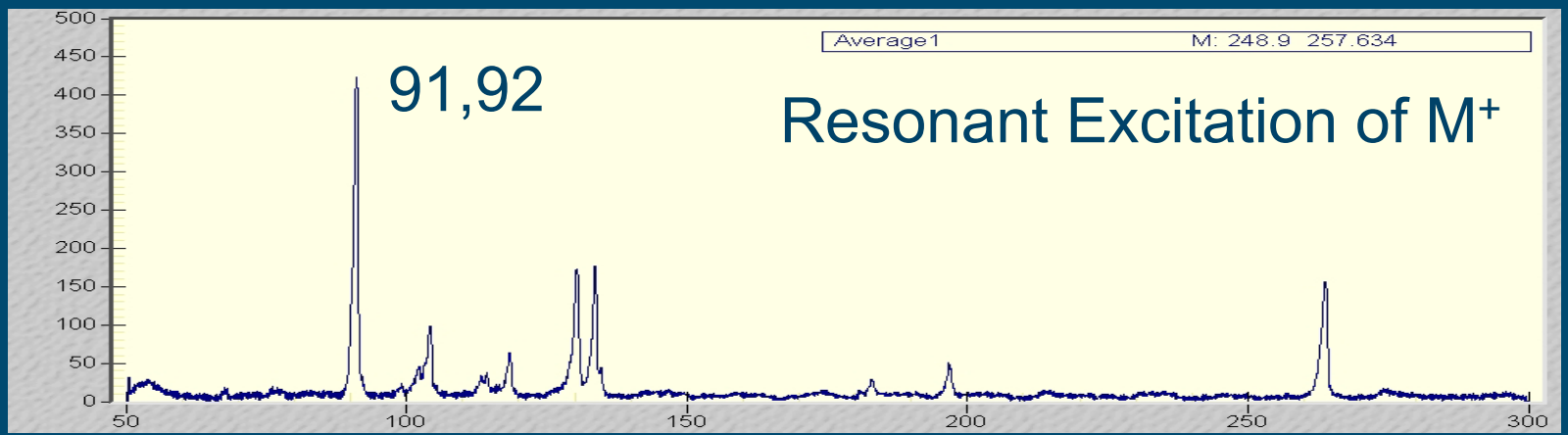
- Progression from pMSED
  - offers advanced, lower power, more flexible electronics and software in battery powered, field portable equipment
  - recently taken delivery of first instrument
  - act as prototype platform

EI Spectrum of DMNB



MS/MS of nBB

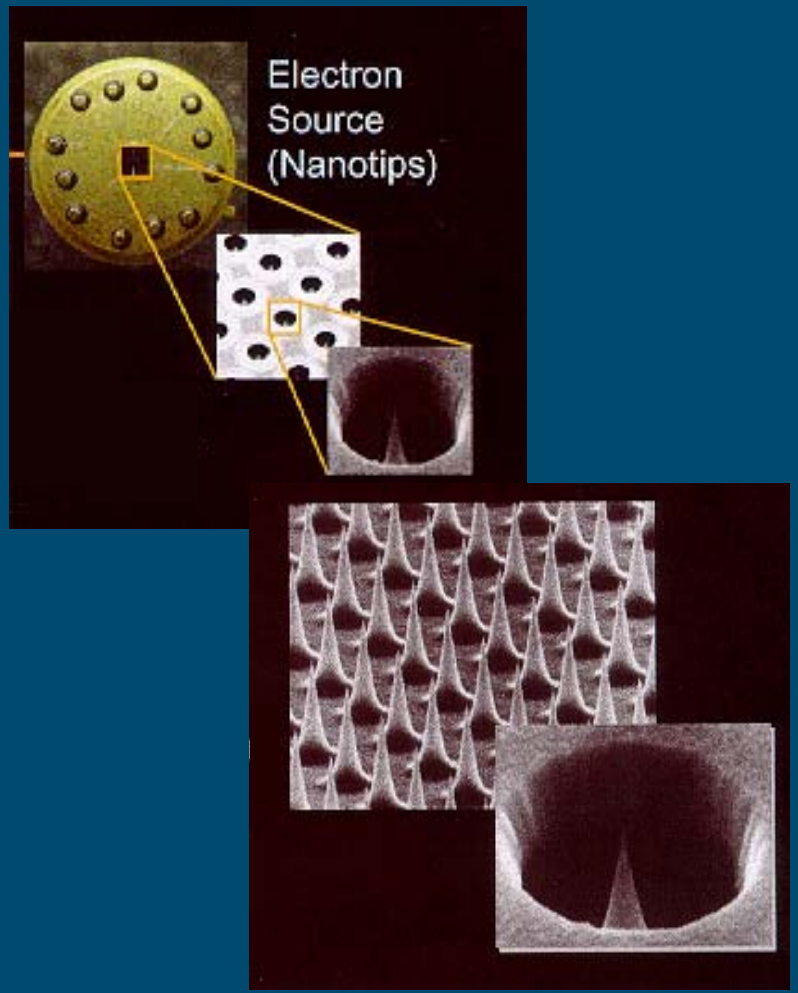




Field Emitters

- Developed by Central Microstructures Facility, Rutherford Appleton Laboratory for space instruments

- Arrays of single crystal silicon field emitters with integrated gates

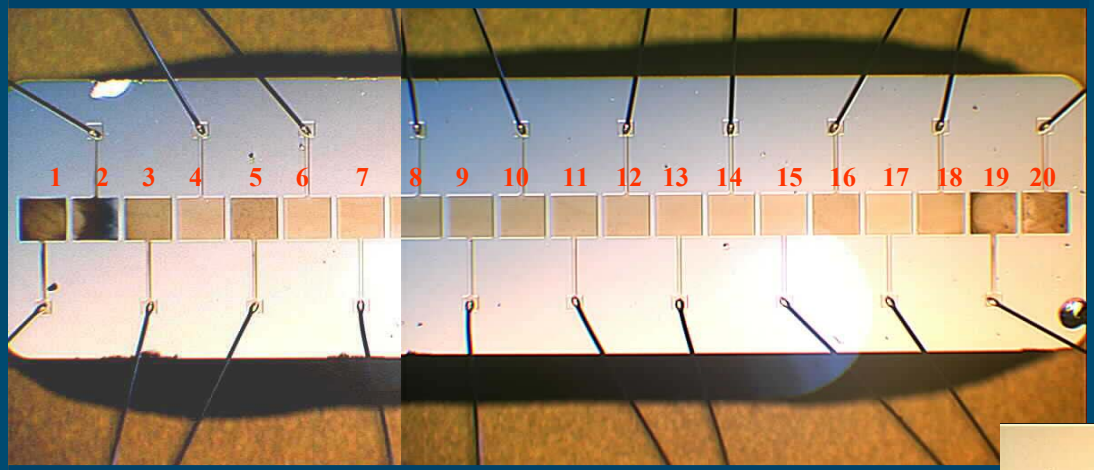


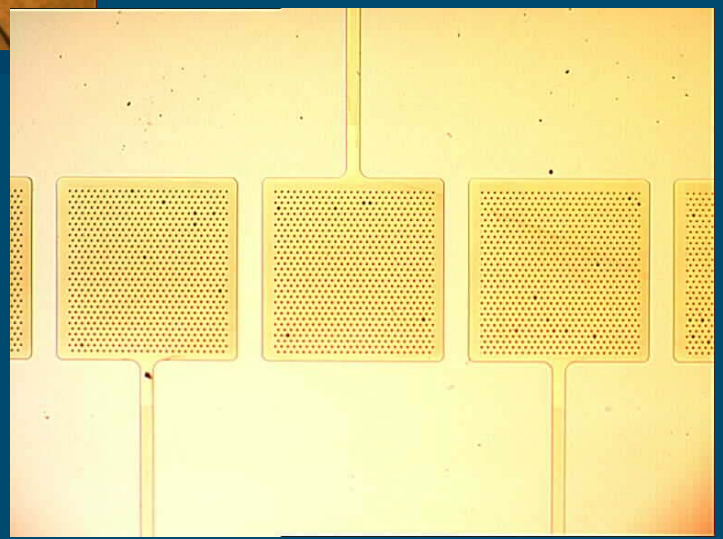
Proceedings 3rd Round Table Micro/Nano Technologies for Space ESTEC, 15-19 May 2000

Specification

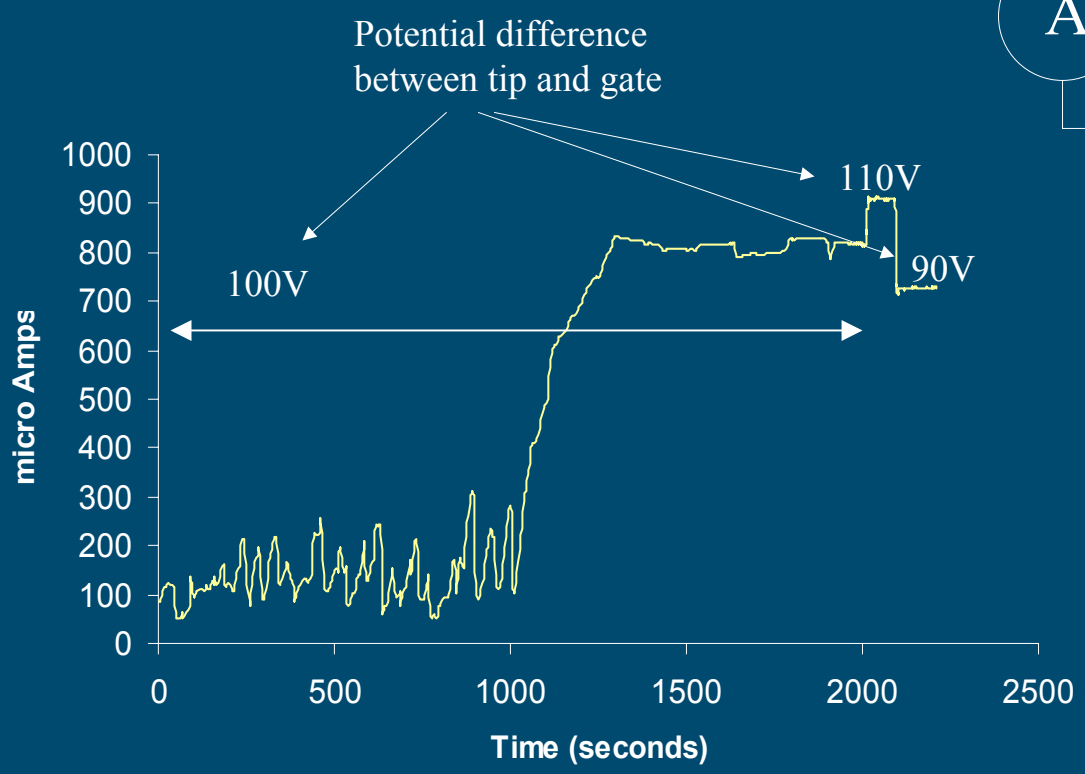
- Dimensions: 2.2 μm high, 10 nm tip radius, 7 μm pitch
- Extraction voltage: 50 - 100 V, breakdown > 500 V
- Array: 25 x 35 emitters per array, 20 arrays, 17500 emitters per chip
- Emission current: 100 μA, stability ±1% using constant current source electronics
- Pulse mode: extraction gate switching, 1-2 ms pulse widths

RAL Field Emitters





Emission Current Measurement

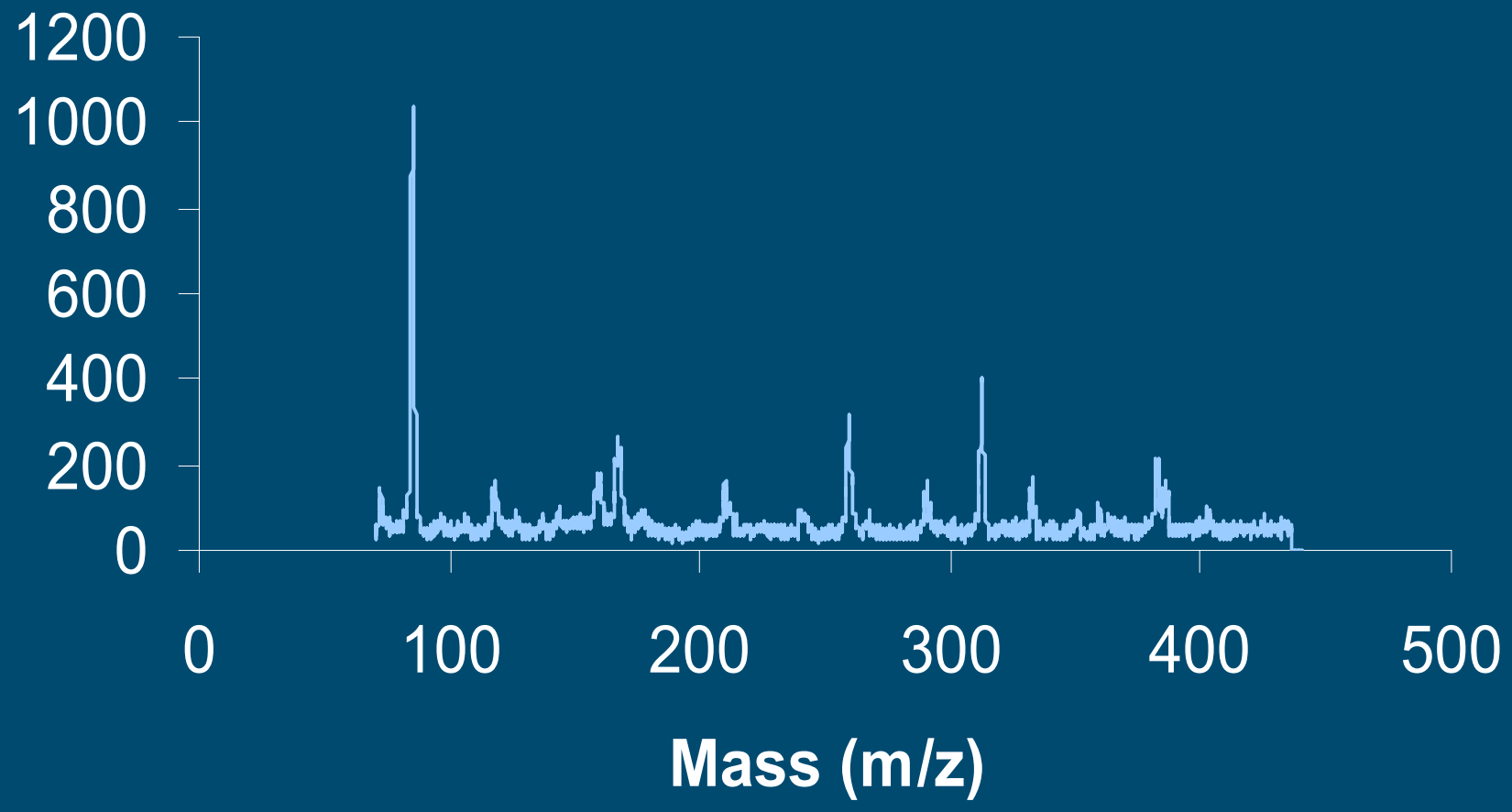


Current recorded on PC.

Chips must be run-in before use.

Graph shows change in emission characteristic during run-in period from unstable early emission to stable, high current emission.

Mass Spectrum of FC43



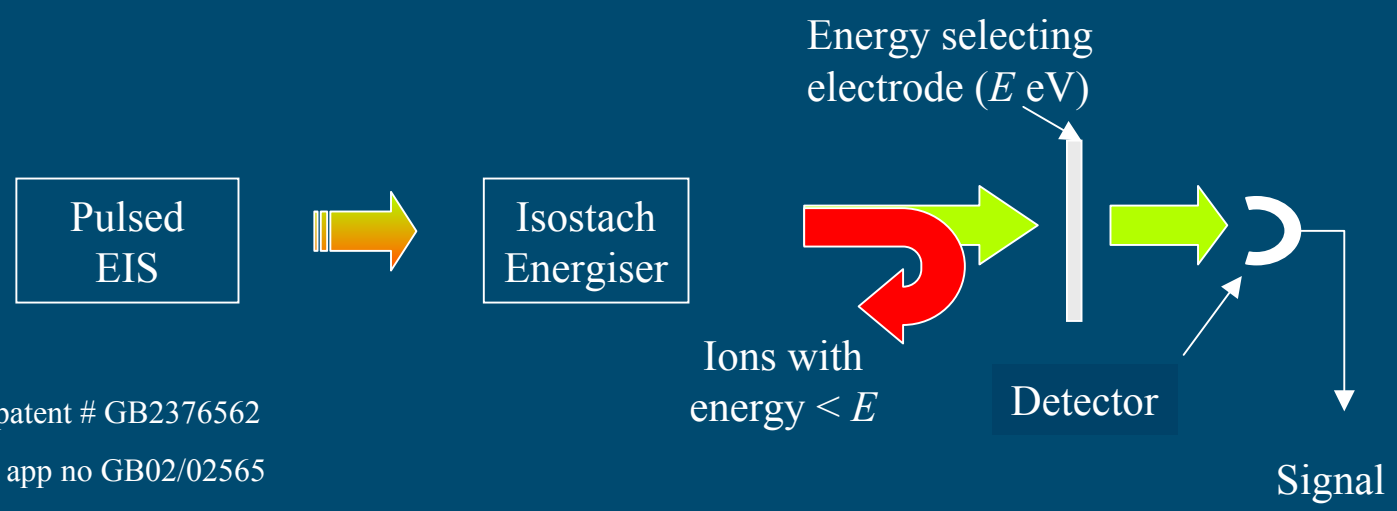
Characterisation

- Underway

- 3 issues to be investigated
  - Lifetimes
    - application specific
  - Poisoning by organic compounds
    - spectra of FC43 obtained
  - Pressure tolerance
    - spectra collected at 10^{-5} Torr

Dynatronics '*Isostach*' MS

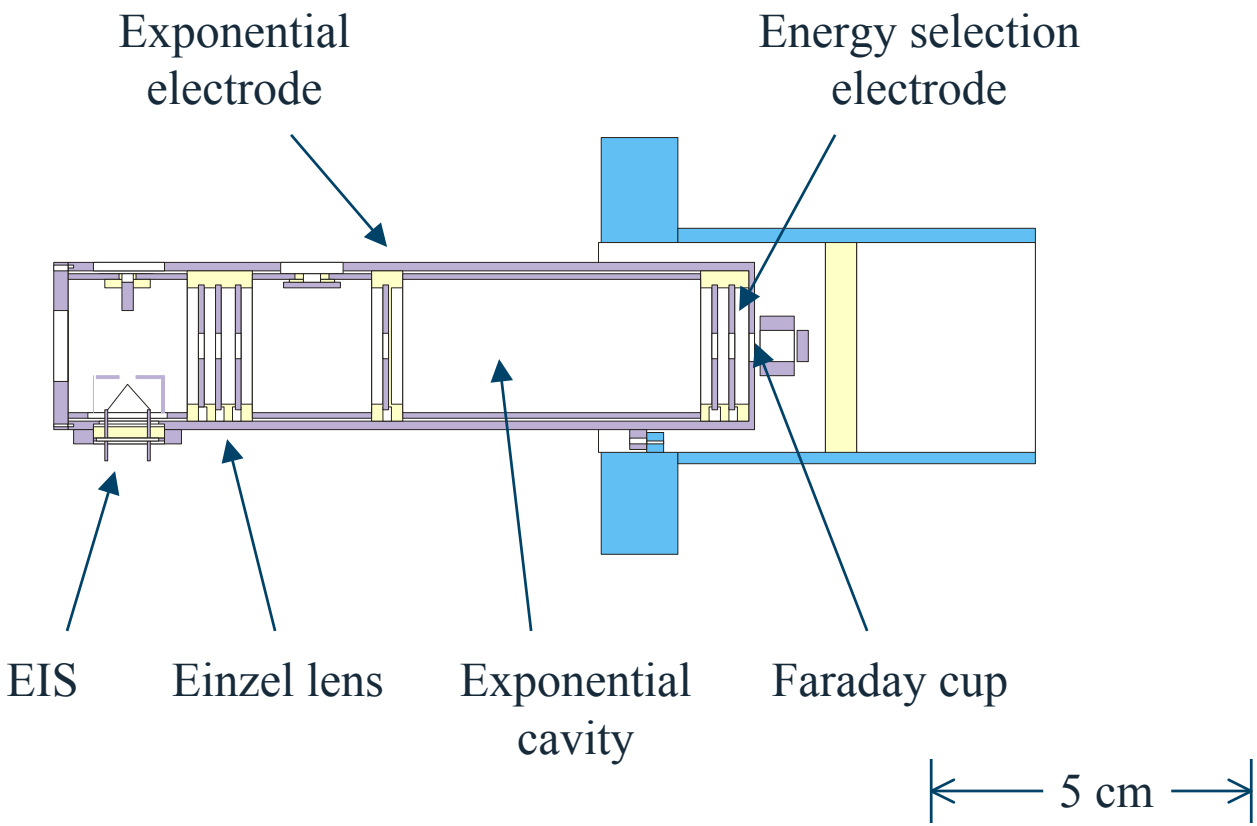
- Patented[1] novel MS in which ions produced have same ***velocity*** (rather than ***same energy*** as in TOF)
  - different ions have different energy, discriminated by energy analyser
  - offers 'TOF' like performance in compact instrument



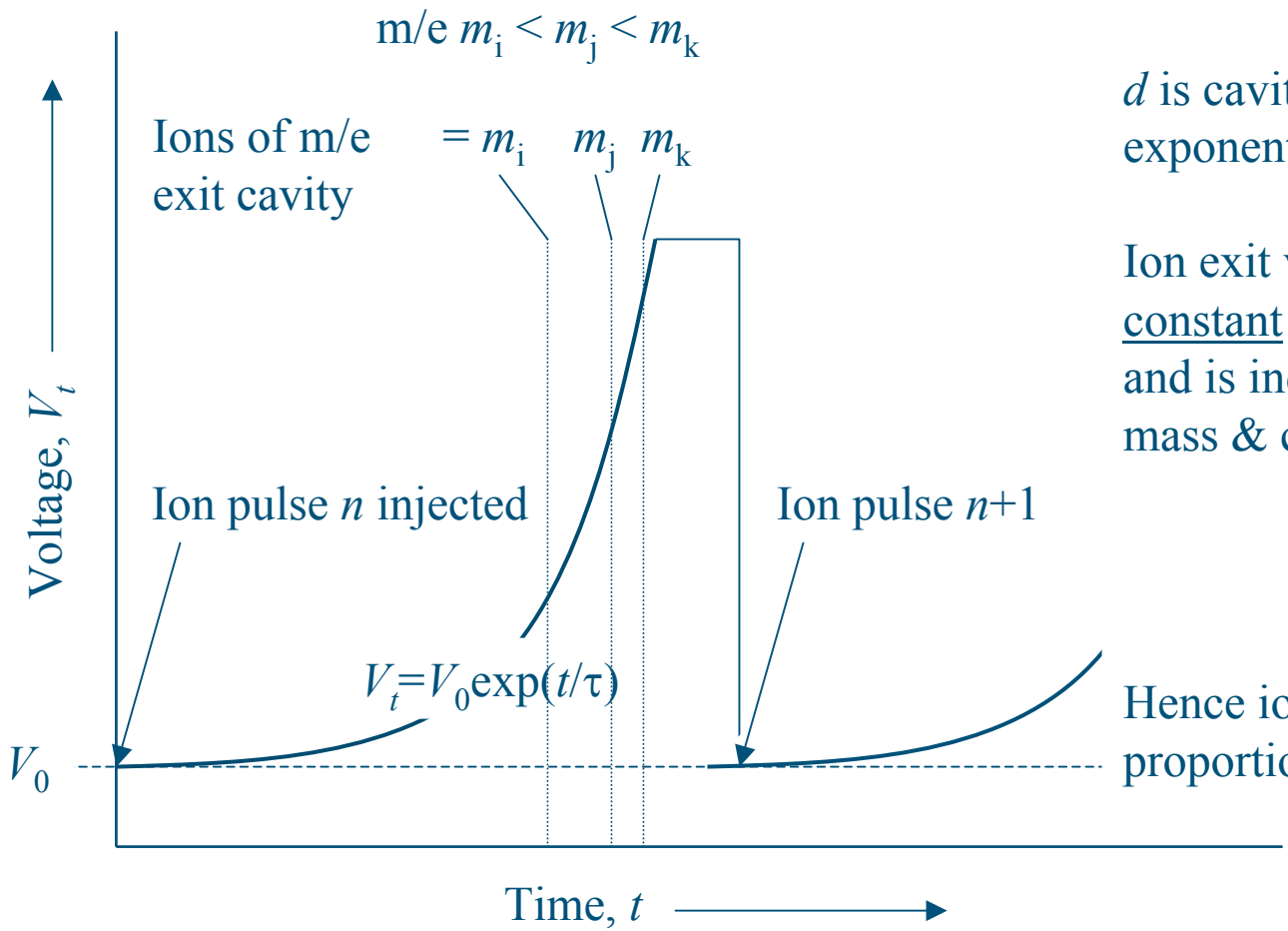
UK patent # GB2376562

PCT app no GB02/02565

'Isostach' MS Layout



Theory



d is cavity length, τ is exponential time constant.

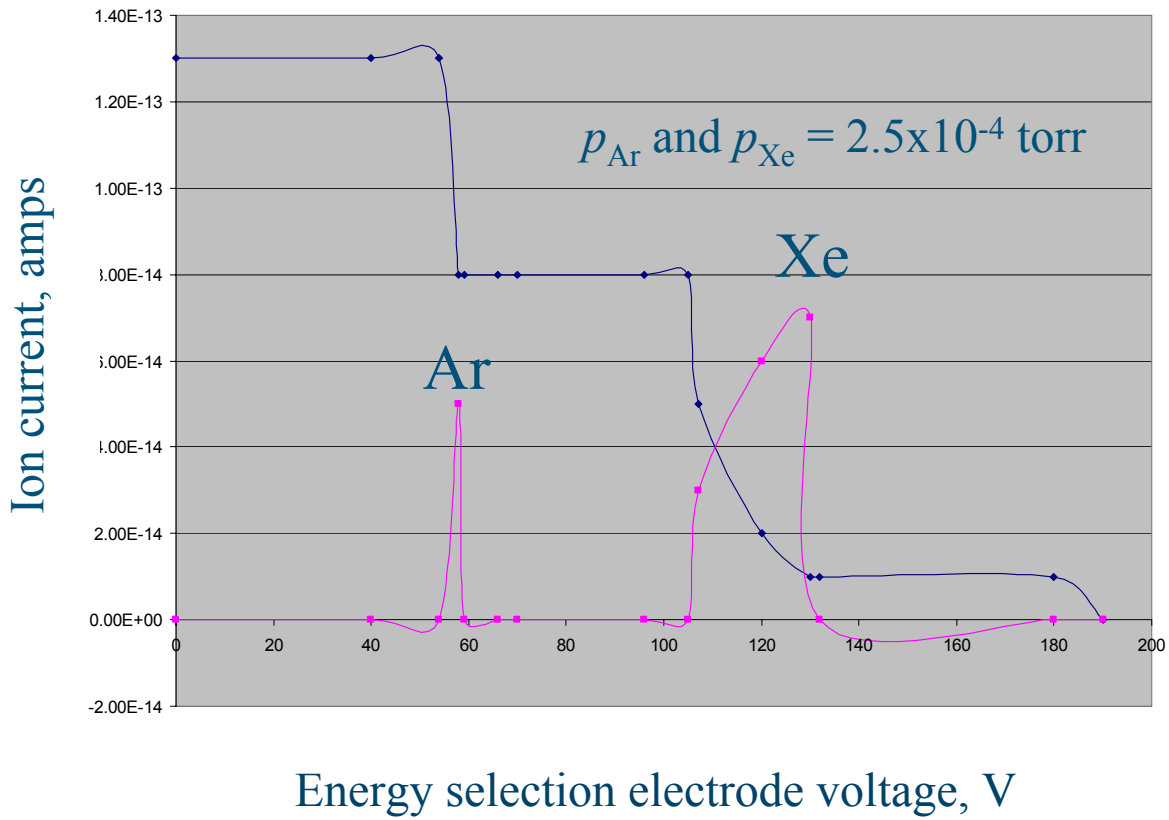
Ion exit velocity, v, is <u>constant</u> for fixed d and τ and is independent of ion mass & charge:

$$v = d/\tau$$

Hence ion energy is proportional to ion mass.

Results

- First MS obtained for Ar and Xe (with broad energy spread EIS)



Summary

- Assembled two ‘clone’ CIT systems
- Characterised CITMS for explosive analysis
  - obtained first EI CIT spectra of explosives
  - quantified performance for explosive analysis
  - demonstrated MS/MS
- Explored benefits/limitations of CITs

Acknowledgements

- This work was funded by the UK MoD
- Dstl EDG team
- Purdue University
  - Graham Cooks Research Group, especially
    - Andrew Guymon
    - Chris Doerge
- Griffin Analytical Technologies
  - Garth Patterson
  - Dennis Barker
  - Mitch Wells

Acknowledgements

- RAL
  - Barry Kent
- Dynatronics
  - Brian Webb
  - Don Young
  - Brian Holden