Thermal desorption inletting for portable mass spectrometers

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Outline

- Background
- Planning a new instrument for surface sampling
  - Collection of samples
  - Sample separation
- Thermal desorption on a portable mass spectrometer
  - Experimental results
- Future work
- Conclusions

Background

- HEMS 2005
  - “A field portable thermal desorption time-of-flight mass spectrometer”
  - Dr Matthew Brookes

- Thermal desorption directly into a membrane inlet without sample separation on a modified Kore MS200 portable mass spectrometer

Future goal

- A man portable mass spectrometer that can analyse trace contamination from dirty surfaces in seconds
- What type of instrument do we need?
- Sample collection
  - Direct surface analysis
  - Wipe
    - Thermal desorption
- Sample separation
  - IMS
  - GC

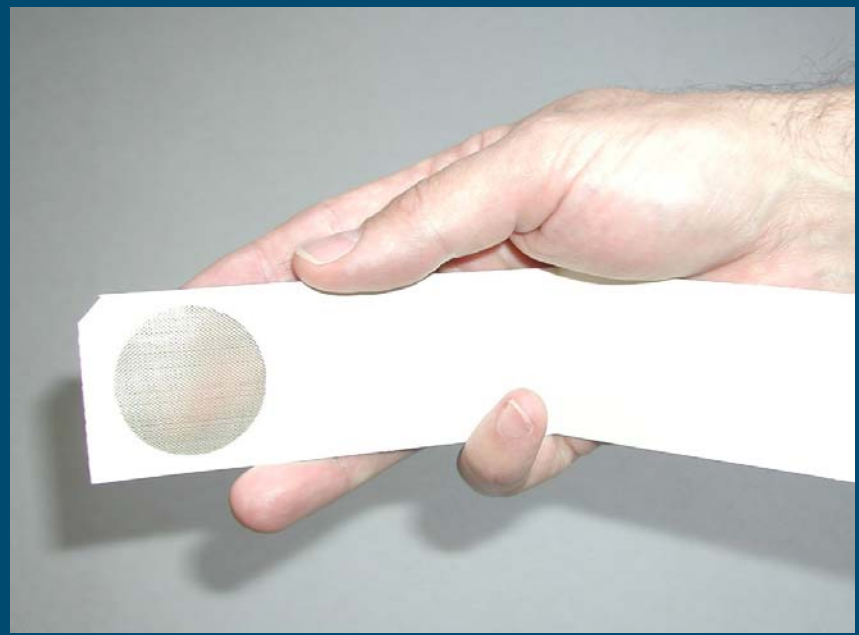
Direct analysis of surfaces

- DESI or DART type experiments
- No need for a wipe
  - However, a wipe can be analysed in the same way as any other surface
- Ambient temperature
  - Less breakdown of thermally labile compounds
- Soft ionisation
  - Clear simple mass spectra

Direct analysis of surfaces

- The spectra from dirty surfaces are complex
  - Separation is needed
    - Tandem MS could overcome this problem
  - If separation is required then you may need to desorb into a chromatographic device before ionisation
- EI spectra are useful for library searching of unknowns
- Difficult to sample large or irregularly shaped items
  - Small analysis area
  - Wipe is low tech but fast and relatively efficient

Collection of contamination from surfaces using a wipe

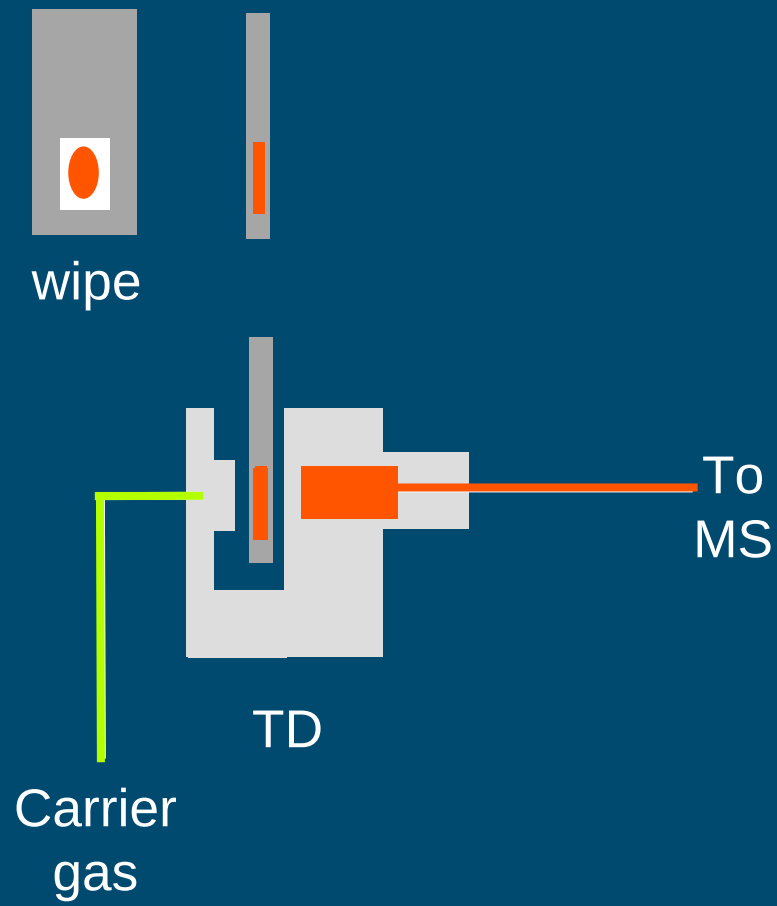


Why a wipe?

- Currently most efficient method of collection
- Versatile
  - Collect from a variety of surfaces of different shapes, sizes, materials and textures
  - Dry or wet
- Cost effective
  - Currently it is cost and time efficient to have one high value portable mass spectrometer and make many sample collections with low cost wipes than several high value instruments with operators making direct analyses

Removal of material from the wipe

- Have to remove the material trapped on the wipe
- Heat and flush from the surface with a gas flow
  - Thermal desorption



Why thermal desorption (TD)

- Advantages
  - Rapid
  - Minimum sample preparation
  - Efficient sample introduction for broad range of chemicals
    - The method of choice for IMS devices etc

- Disadvantages
  - Thermally fragile compounds can be destroyed
  - No intrinsic specificity or separation
    - Low resolution if ramped

Sample separation

- TD directly into the mass spectrometer as in the MS200
- There can be a complex mixture of contaminants on surfaces
- Selective ionisation
  - Some compounds will not be detected
- Soft ionisation
  - The overlaid spectra of several unknown compounds can be difficult to interpret
- Sample separation is needed

Sample separation options

- Ion mobility spectrometry (IMS)
  - Very very fast (milliseconds)
  - Small (handheld and palm size devices commercially available)
  - Low power consumption
  - Sensitive
  - Has been coupled to MS successfully
- Gas Chromatography (GC)
  - Fast (from seconds to minutes)
  - Portable GC/MS commercially available

IMS/MS

- Differential pumping required for IMS/MS
- The amount of vacuum pumping required depends on
  - The pressure of the MS ($1x10^{-5}$ Torr)
  - The pressure of the IMS (760 Torr)
  - The orifice size between the IMS and MS ($1mm^2 = 1 \times 10^{-6}\ m^2$)

IMS pressure **x** orifice area **x** speed of sound = MS pressure **x** pumping speed

$$760 \times 1 \times 10^{-6} \times 200 = 1x10^{-5} \times PS$$

$$PS = 15\ 200\ m^3/s = 15\ 200\ 000\ L/s$$

Estimate of the Speed of sound in a choked viscous gas = 200 m/s

IMS/MS

- Reducing IMS pressure and increasing mass spectral pressure

$$\text{IMS pressure} \times \text{orifice area} \times \text{speed of sound} = \text{MS pressure} \times \text{pumping speed}$$

$$1 \times 1 \times 10^{-6} \times 200 = 1 \times 10^{-3} \times PS$$

$$PS = 0.2\ m^3/s = 200\ L/s$$

- Reducing IMS pressure can reduce separating power
  - Sample has to be let in which leads to extra vacuum pumping
    - Sensitivity drop
- Mass spectral pressure below operational limits for TOF
  - Possible with an ion trap
- Can reduce orifice size but this reduces sensitivity

IMS/MS conclusions

- The vacuum pumping needed for a highly sensitive instrument is too large for a truly portable instrument

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- Others are working on solving this problem
  - This could be overcome if the orifice is made to selectively admit ions over neutral carrier gas molecules
- We are interested in portable IMS/MS if you have the solutions!

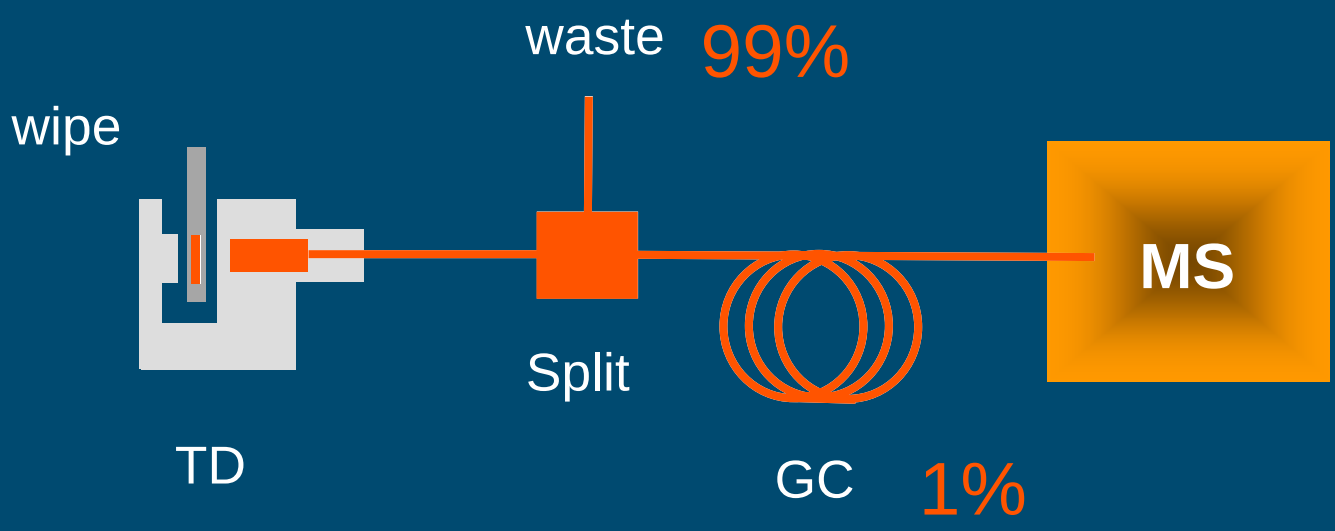
GC/MS

- Advantages
  - Well known technique
  - More orthogonal separation than IMS
  - Portable GC/MS instruments are available
- Disadvantages
  - Thermally labile compounds can be destroyed or are difficult to analyse
  - Slow

TD/GC of a wipe on a portable MS

- Thermally desorb material from a wipe into the GC
- High gas flows needed to efficiently flush off the sample in a short time
- GC/MS can only accept a low flow rate
  - If you can wait - use a low flow over a long period
  - Split sample and through away excess gas flow
  - Trap sample on a cold spot

TD/GC on a portable MS

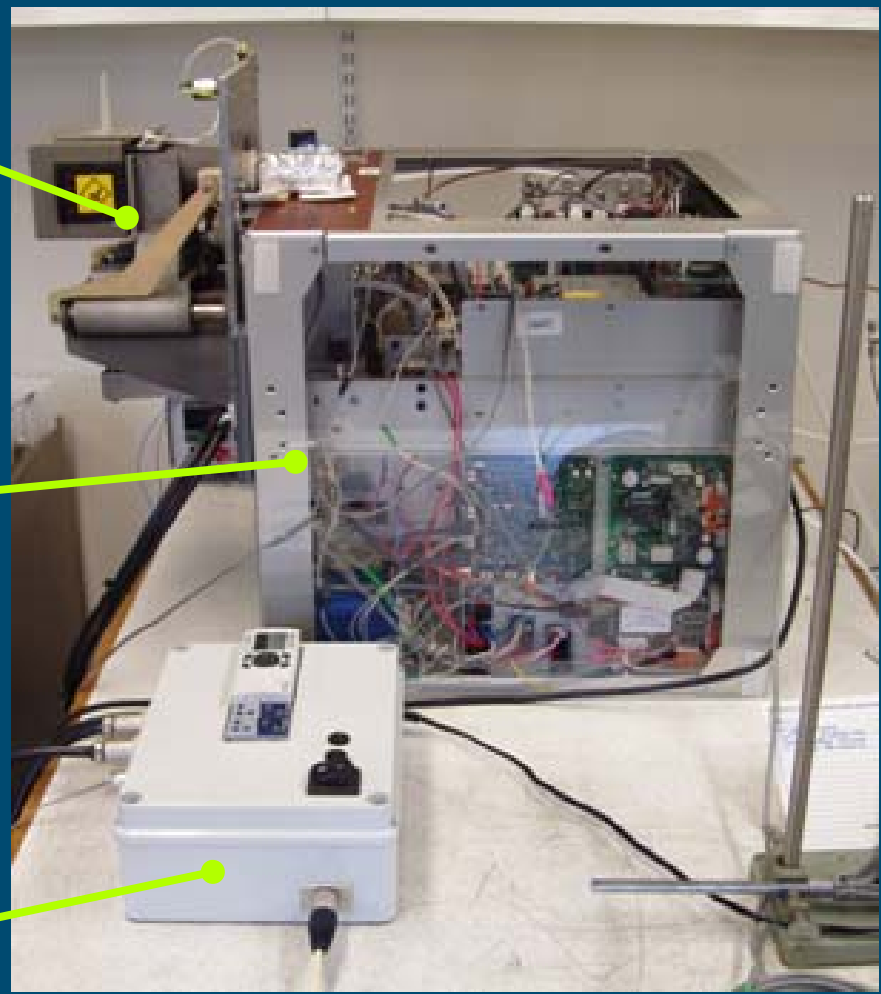


TD = 100 ml/min

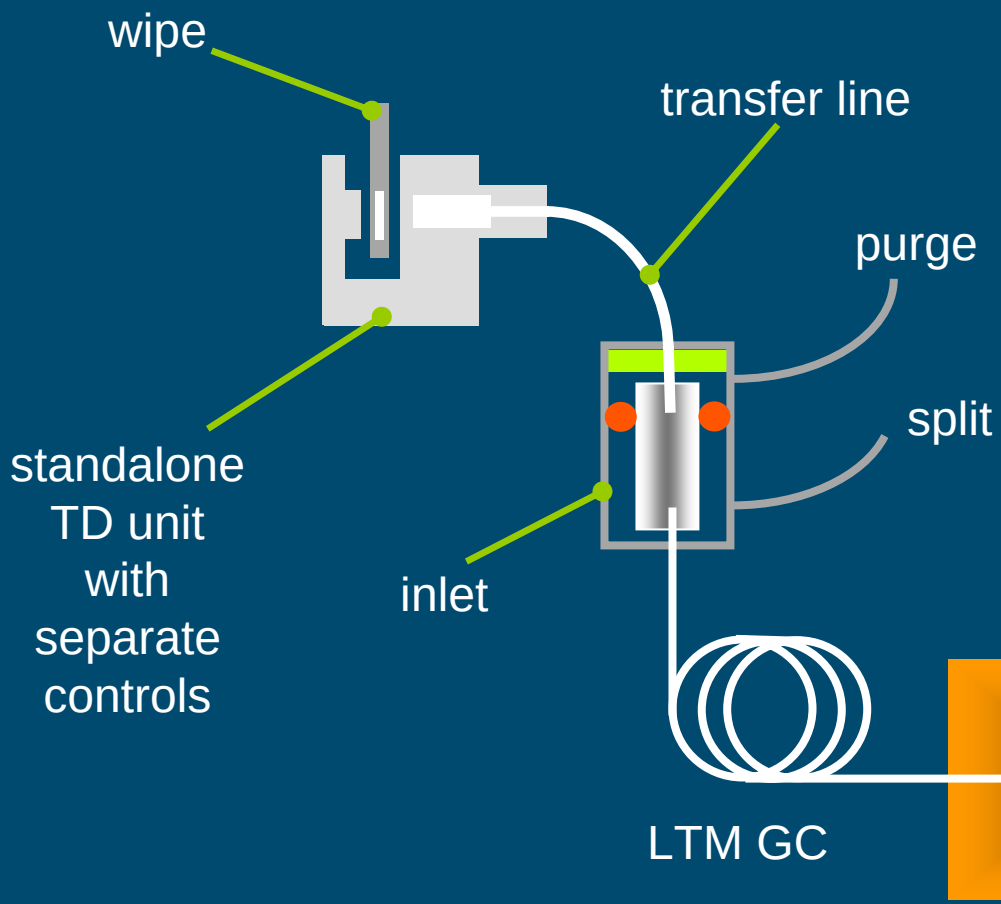
GC = 1ml/min

Wipe TD coupled to a portable GC/MS

- A wipe TD from a commercially available equipment coupled to a Griffin 400 GC ion trap mass spectrometer



Schematic of TD/GC interface



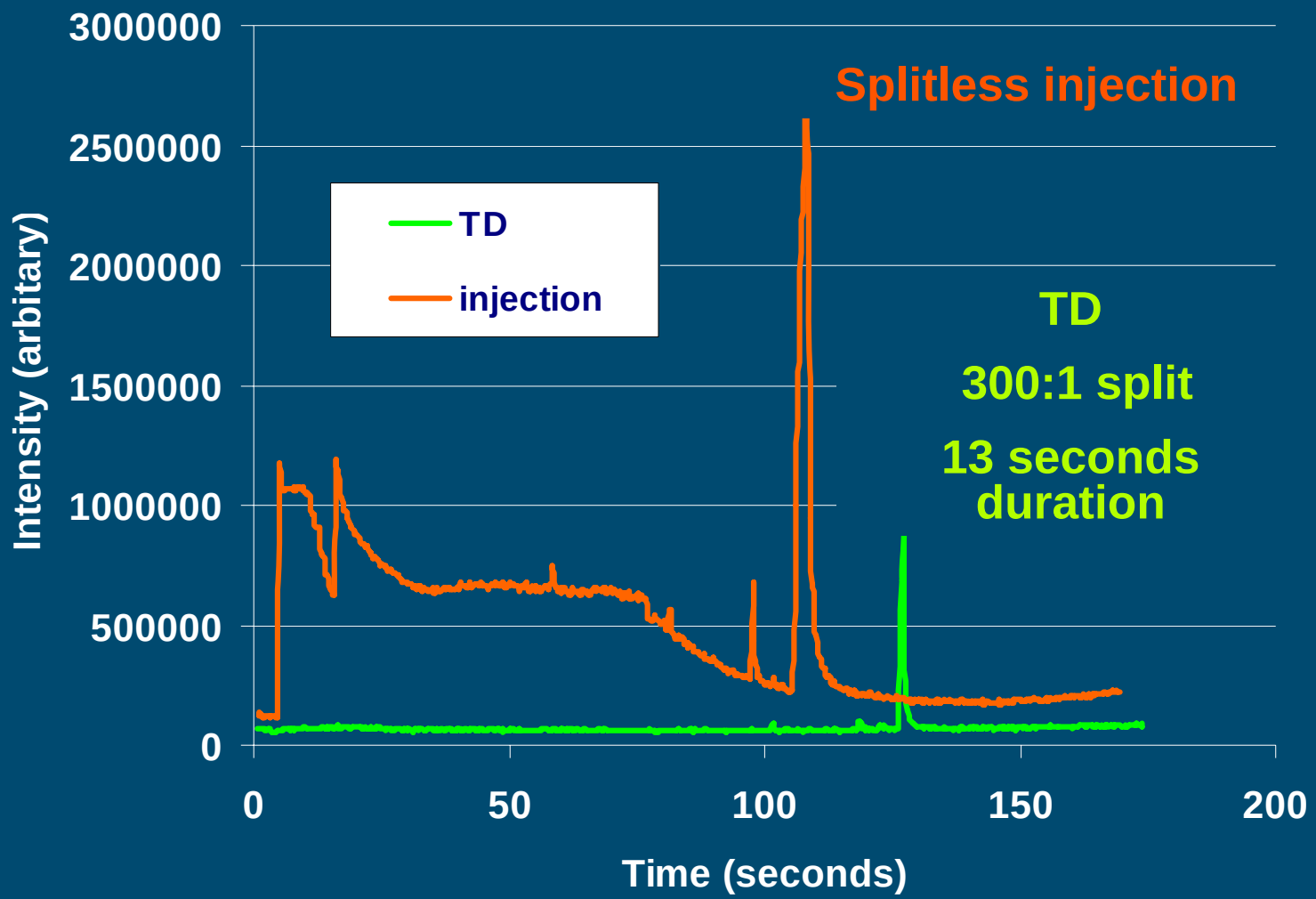
TD backing pressure = 40 psi

Injector pressure = 20 psi

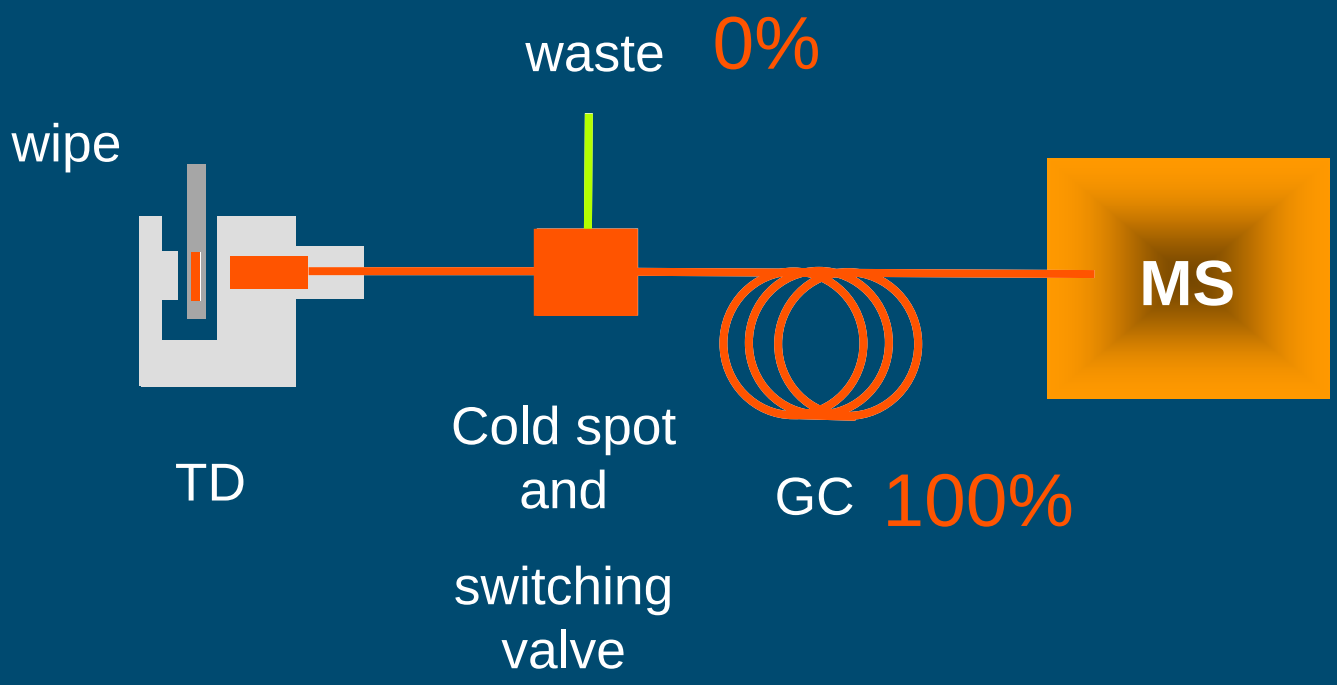
TD temp 170ºC

Split up to 300:1

Overlaid chronograms of 1000ng of DNT injected and thermally desorbed into the GC/MS



TD/GC on a portable MS with a cold spot



TD = 100 ml/min

GC = 1ml/min

TD testing summary

- Produced stand-alone TD inlet
  - Can connect to any standard GC with a septum inlet
  - The gas flow through the injector can rise up to 300 ml/min for 13 seconds
- The signal recorded is better than would be expected from the split ratio
- Next stage is to try a cold spot system
- Reduce power requirements for a portable instrument

Future work

- Working on a fast TD/GC/MS portable time-of-flight instrument
- TOF will enable very fast mass spectral sampling rate for rapid separation

Conclusions

- Wipe collection and TD provide advantages for sampling surface contamination
- Separation after TD is needed for many applications
  - GC at present or possibly IMS in the future
- Working on a portable TD/GC/MS
- Dstl is open to collaboration and discussion with companies and academics to try and solve the problems and improve solutions

Any questions?

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