Basic GC-MS Training

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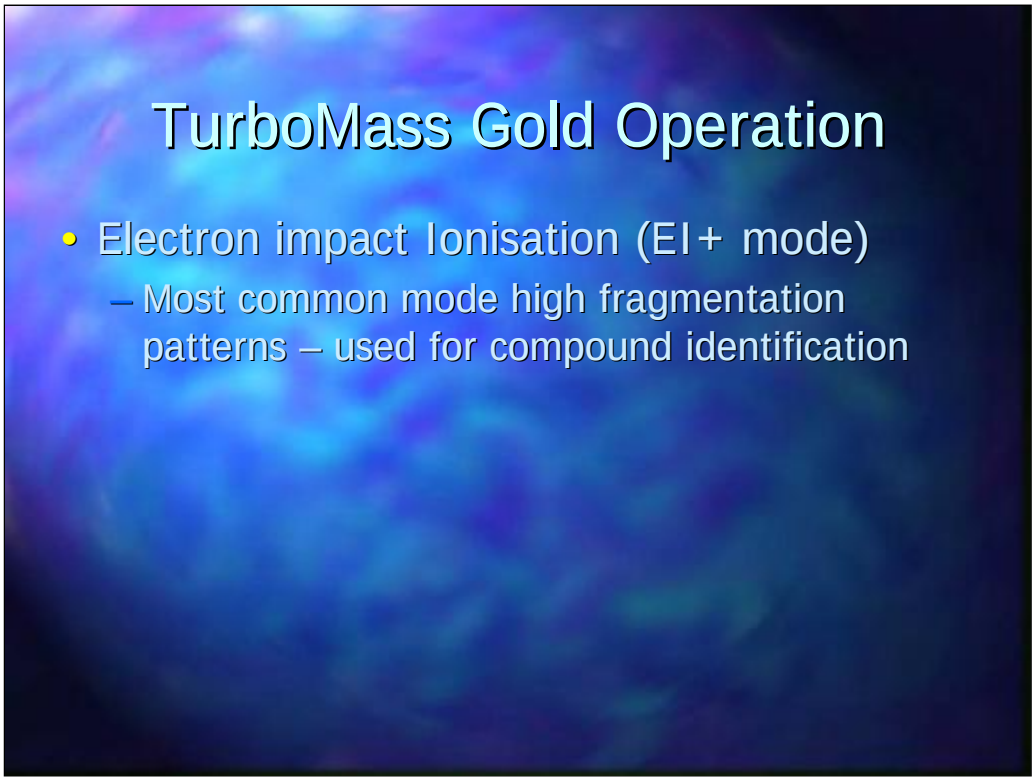
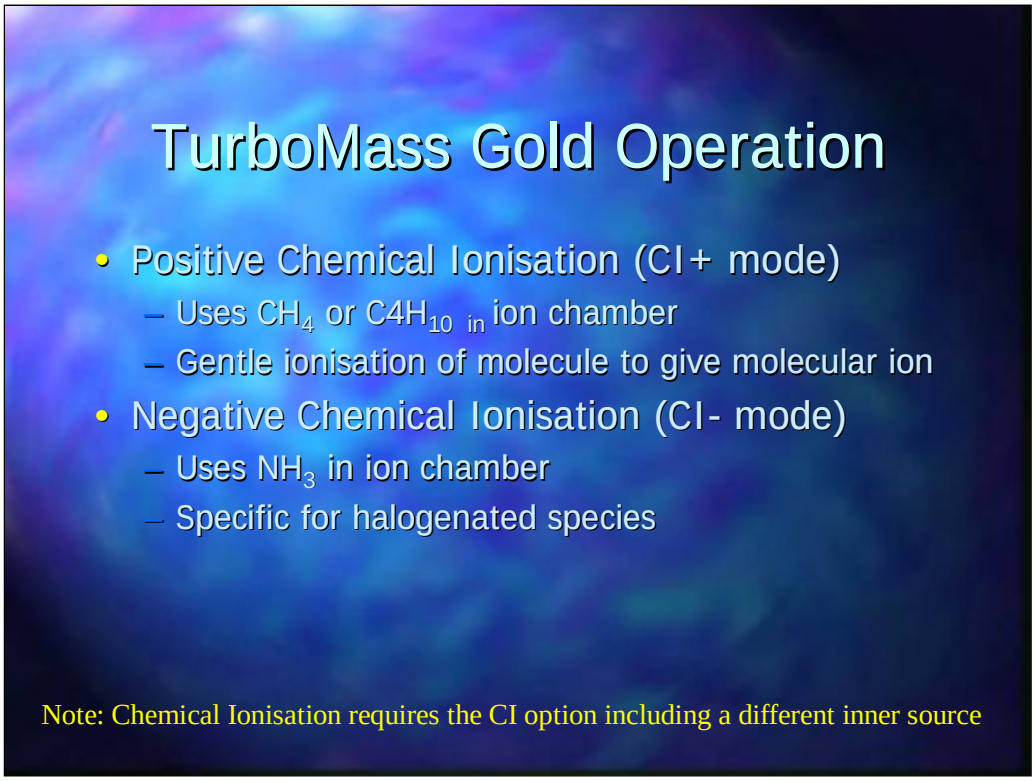
TurboMass Gold Operating Range

- Mass Range 1.5 – 1200 Daltons
- Scan Speed up to 6200 Daltons/s
- High Speed Turbomolecular pump 250L/s
  - Carrier gas flows as high as 5ml/min



Electron Impact ionisation (EI) is the most common form of ionisation used in mass spectroscopy. The sample molecules are bombarded with high energy electrons (70 eV) to fragment the molecule and to give positive ions. Usually a high degree of fragmentation takes place and the fragmented ions are recorded according to their mass to charge ratio (m/z). Often the charge is +1 and so m/z represents the mass of the positive ion. However the charge can be higher in which case the m/z value will not represent the mass of the fragment.

EI is used for identification purposes where the mass spectra are compared to mass spectra available in commercial libraries such as NIST or Wiley.

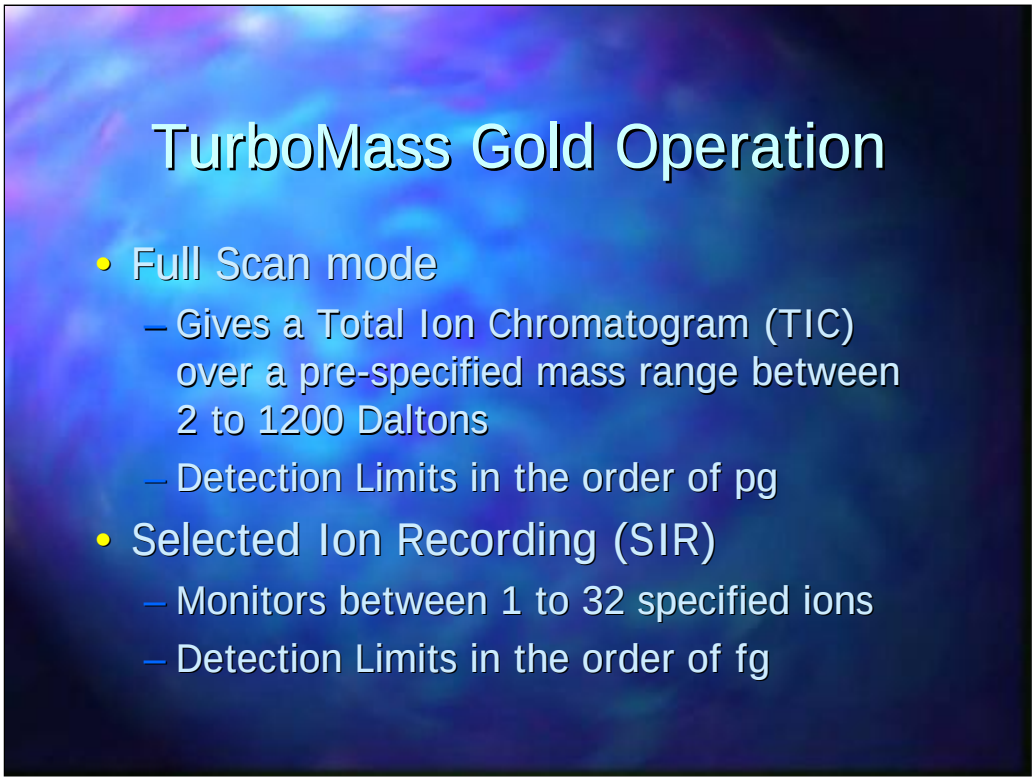


Chemical Ionisation (CI) requires a special option fitted to TurboMass Gold which also includes a modified inner source.

With CI an ionising gas is introduced into the ion source. Electrons collide with the ionising gas molecules which in turn ionise the sample molecules. Two types of chemical ionisation are possible CI+ or CI-

CI+ is a softer form of ionisation where very little fragmentation occurs and so is generally used to identify the molecular ion and hence give an indication of the molecular weight of the sample. The ionisation gases used are usually methane or butane

CI- tends to ionise only halogenated compounds and so gives a specific detection for halogenated compounds. The ionising gas used for CI- is ammonia



Full scan mode allows the user to specify the mass range to be scanned to allow identification of unknown species. All the masses that are scanned are added together to give a total ion abundance for each scan to give a total ion chromatogram.

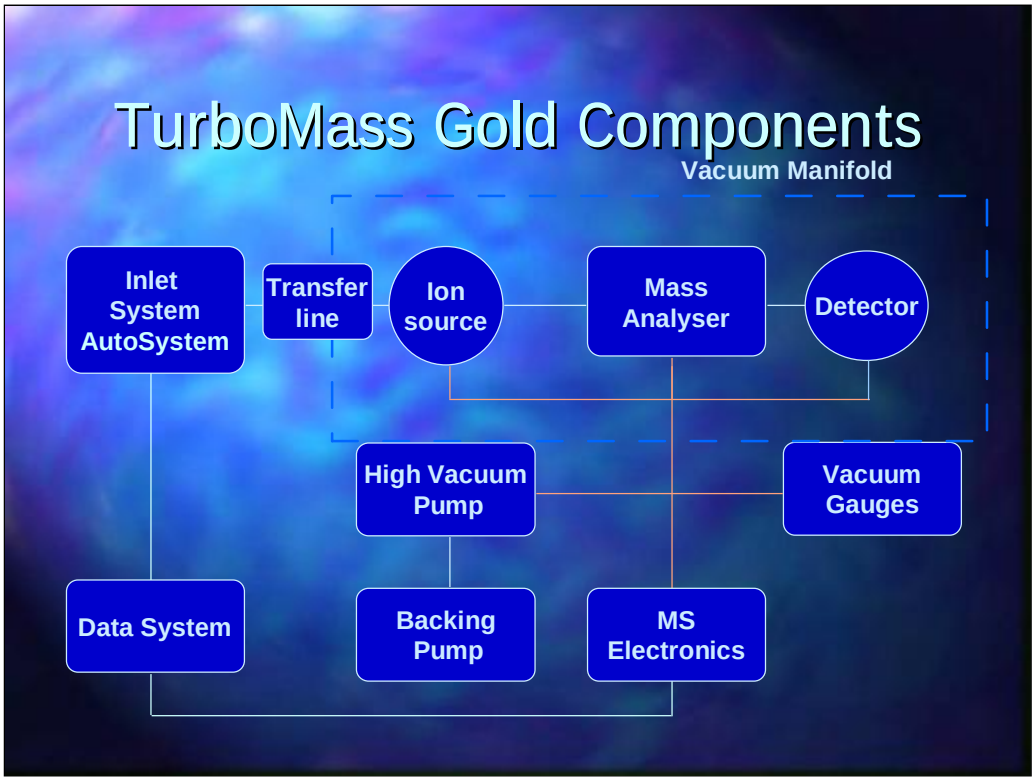
A chromatogram of individual ions, or any combination of ions, from a full scan may be examined.

Detection limits in the order of picogrammes are obtainable in full scan mode.

Selected Ion Recording (SIR) allows up to 32 specific ions to be monitored.

SIR generally gives better signal to noise ratios and so has a better detection limit compared to Full scan mode. Detection limits in the order of femtogrammes are common.

TurboMass Gold may be programmed to collect both Full Scan and SIR chromatograms simultaneously.



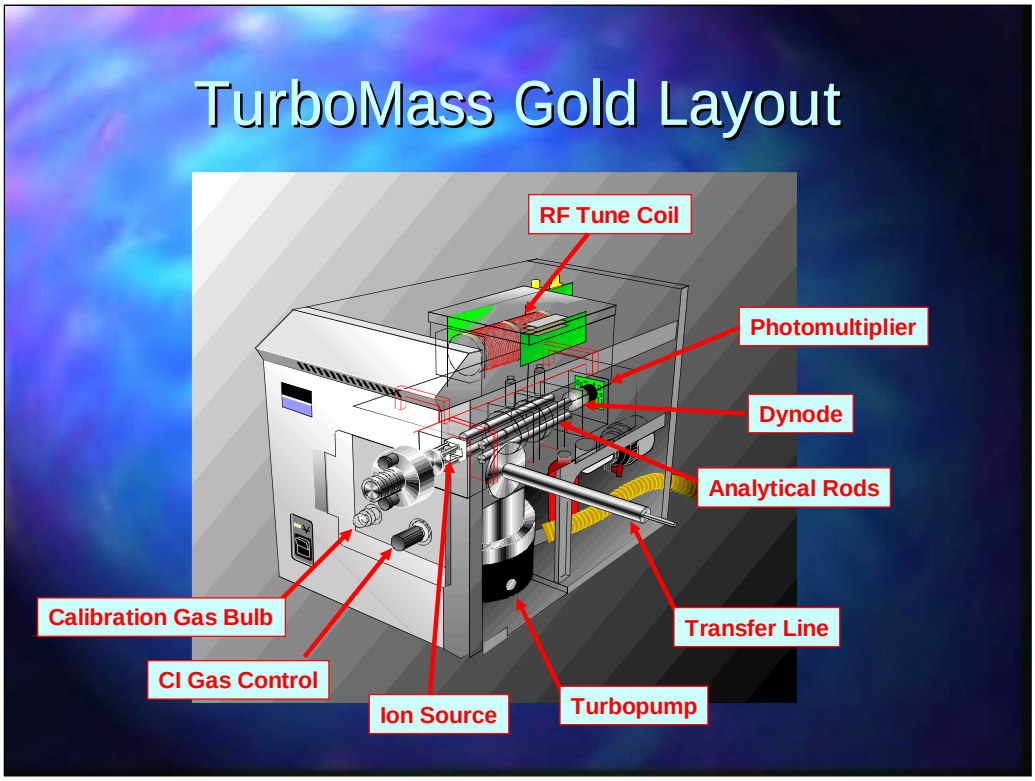
A schematic of the main components in the TurboMass Gold system is shown here.

The Data System (TurboMass Gold software) controls both the AutoSystem GC conditions and the TurboMass Gold conditions and also collects, stores and analyses the data obtained from TurboMass Gold

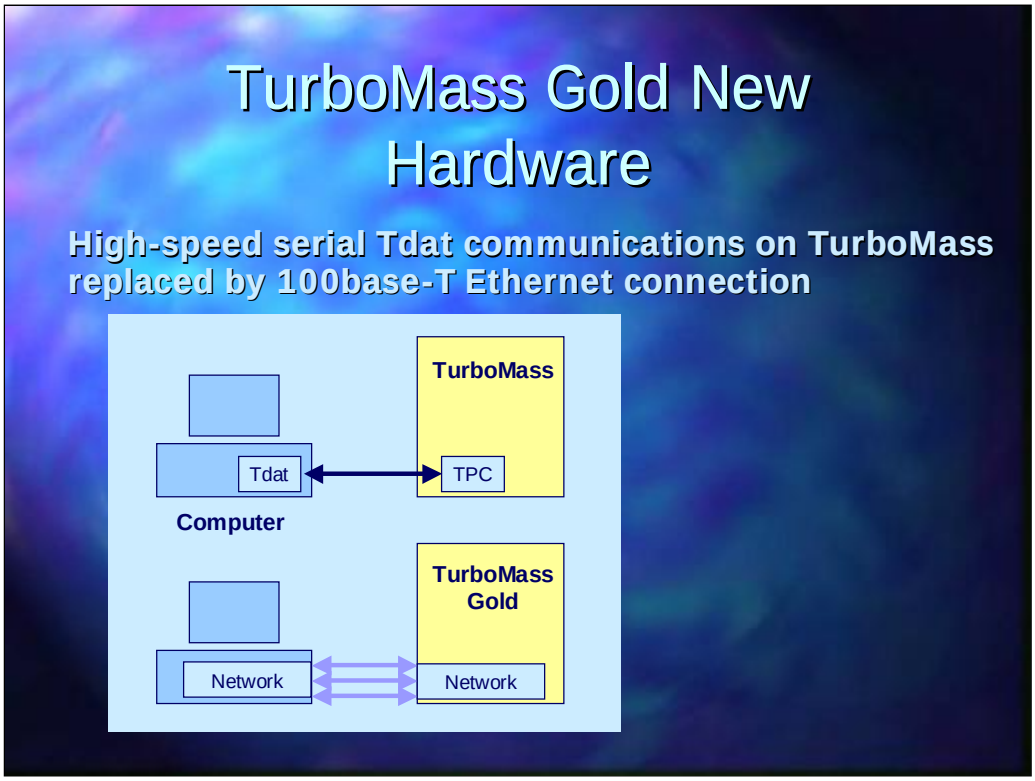
A Transfer Line connects the column outlet to the ion source. The transfer line is part of the TurboMass Gold vacuum manifold as is the Ion Source, Mass Analyser (quadrapoles) and the Detector (photomultiplier)

A high capacity turbomolecular pump, backed by a roughing pump are used to obtain a vacuum in the order of 1 to 2 x 10^{-5} torr.

The vacuum is measured by the vacuum gauges.



The principal components to TurboMass Gold are shown in this slide



TurboMass Gold incorporates 2 computers, 1 built into the hardware and 1 which is used as the user interface and used to control the GC. The 2 computers are connected by means of an Ethernet connection which gives extremely fast data transfer. This allows mass spectra to be scanned at 6200Daltons/s

TurboMass Gold Performance

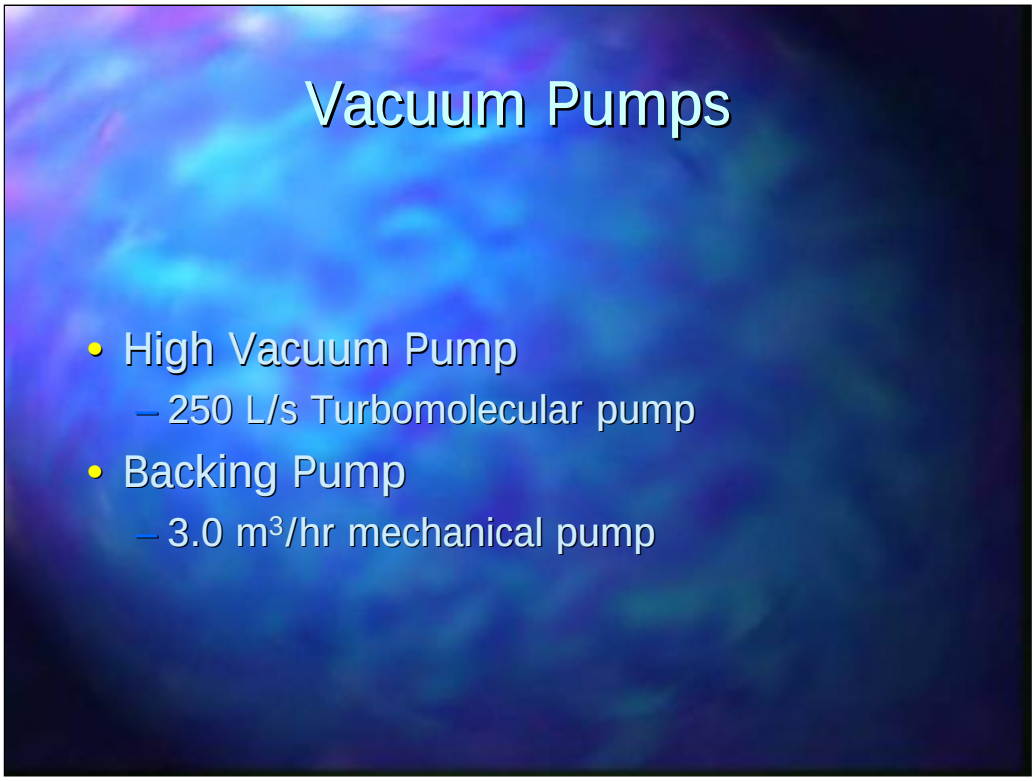
- Faster data acquisition than TurboMass
  - x3 improvement - 200kHz on new A-to-D vs 66kHz on TPU processor
- Faster data transfer rate (Ethernet)
  - 100mps vs 2mps on high speed serial Tdat
- Faster scan speed
  - more computing horsepower and higher data transfer rate
  - up to 60 scans/sec depending on mass range
  - yields 6200 Da/sec typ on 500amu range

GC Transferline

- Temperature controlled by Mass Spectrometer
- Alarm condition on GC will shut off transfer line heater
- Vacuum insulated inner tube
- 350 °C upper limit

The transfer line connects the AutoSystem GC to TurboMass Gold and guides the column directly to the ion source. The temperature of the transfer line is controlled from the Tune File in the TurboMass Gold software (see Tuning & Mass Calibration section) and is part of the vacuum system of TurboMass Gold.

If an alarm condition occurs on the AutoSystem GC, such as loss of carrier gas, the transfer line heater will be turned off .



The vacuum is achieved by using 2 pumps. The backing pump is a mechanical pump that will achieve a vacuum in the order of 1×10^{-4} torr. A Turbomolecular pump is used to achieve the every high vacuum, in the order of 1×10^{-5} torr that is required for mass spectroscopy

250 L/s Turbomolecular Pump

- Software controlled pump down
- Hardware controlled vent valve
  - vents when pump slows to 50% full speed
- Maintenance free

The Turbomolecular pump can pump up to 250L/s. This allows carrier gas flows of up to 5ml/min of helium and can still maintain a vacuum around 2.5 x 10-5 torr.

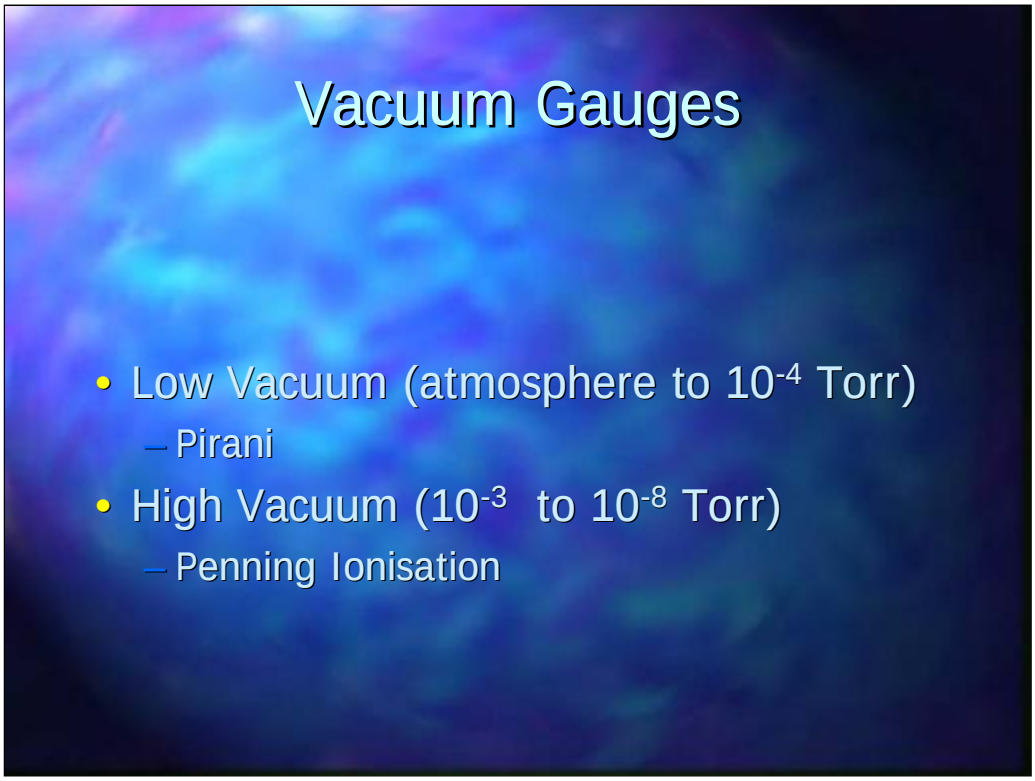
The pump is controlled by the software and will automatically turn on when the vacuum has reached approximately 1 x10-4 torr.

When the pump is turned off the vacuum is maintained until the pump speed has reached 50% of its maximum. At this point a valve is opened to vent the system fully.

Backing Pump

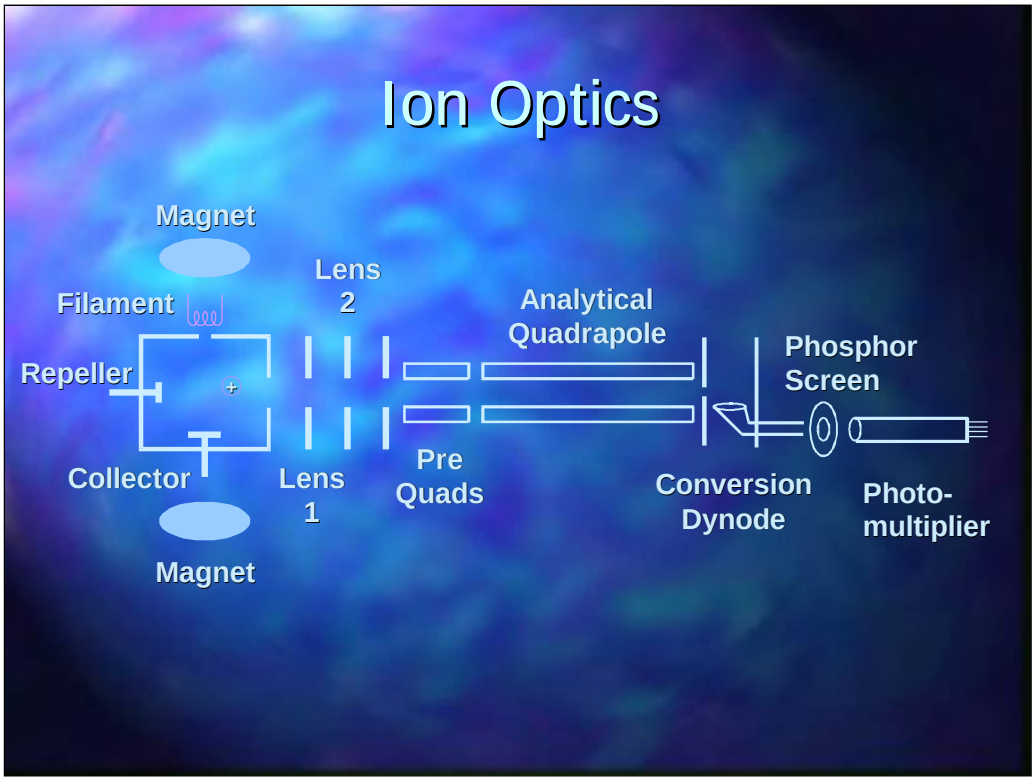
- Rotary Vane Pump
- High vacuum mode
  - 2×10^{-3} Torr
- Matched to turbo pump

The backing pump is a rotary vane oil pump which is matched to the turbomolecular pump. It is an oil filled pump and the oil must be maintained at the specified levels. The oil should be changed every 6 months.



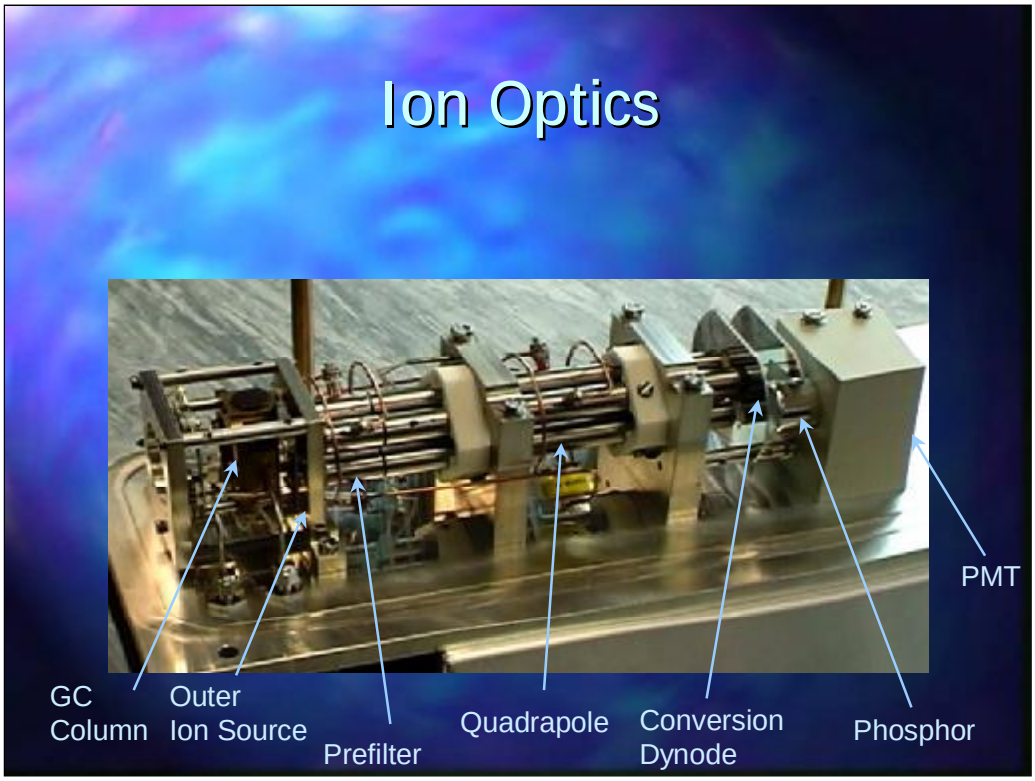
TurboMass Gold has 2 vacuum gauges. The Pirani gauge operates at low vacuums and is used during pump-down to check that the system is leak tight.

When the vacuum reaches 1 x 10-4 torr the Pirani gauge has reached its limit and at this point the Penning Ionisation gauge is used. This will measure the vacuum at the normal operating pressure of TurboMass Gold and monitors the presence of very small leaks.



This shows a schematic of the Ion Optics in TurboMass Gold. To the left is the ion source. The GC column enters the source in the direction that the schematic is viewed. Electrons released from the filament collide with the sample molecules eluted from the column in the ion chamber. The positive ions generated by the electron impacts are drawn out of the chamber by Lens 1, focussed by Lens 2 and directed to the analytical quadrapole where they are separated according to their mass. The analytical quadrapole is protected by pre-quads, short rods, similar to the analytical quadrapoles, which protect the analytical quadrapoles from contamination.

As the positive ions leave the quadrapoles they are collected by the conversion dynode which generates electrons. The electrons then hit a phosphor screen to release photons which are then measured by the photomultiplier. The off-axis conversion dynode and the phosphor screen prevent any contamination reaching the photomultiplier thus enhancing it life.

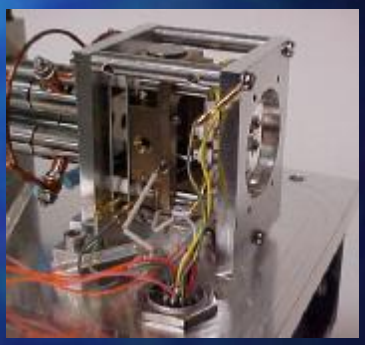


This photograph shows the manifold assembly, including the outer source, prequads, quadrapoles,conversion dynode, phosphor screen and photomultiplier

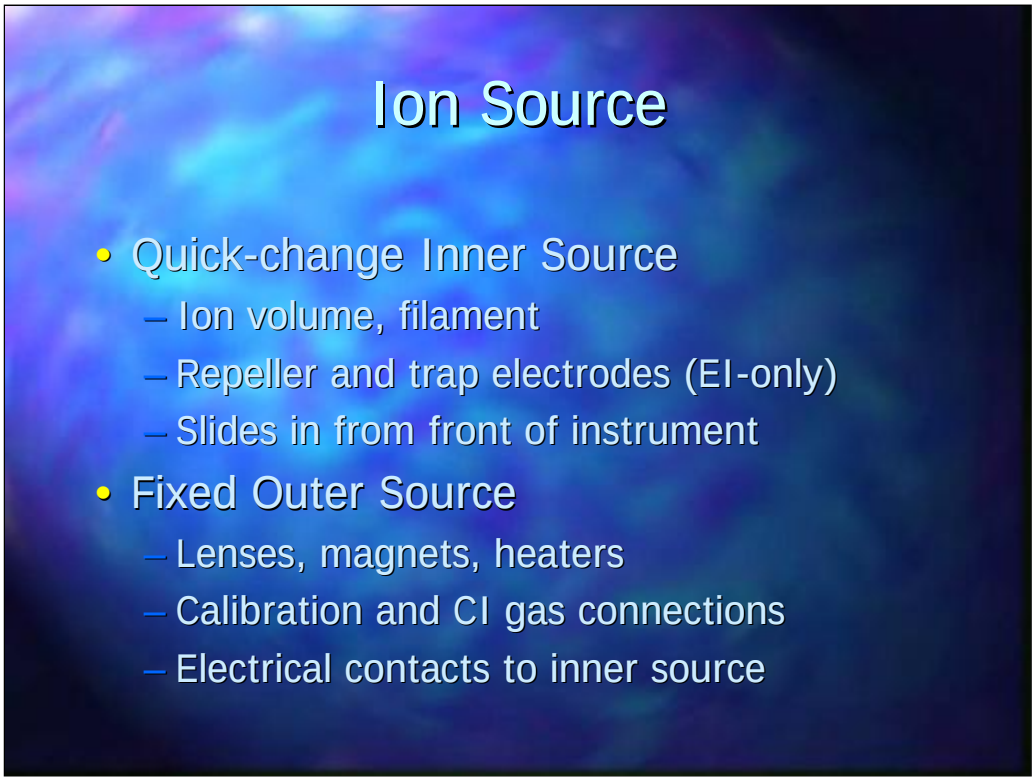
TurboMass Gold Analyzer Enhancements

Improvements for Stability and Sensitivity

- Refined cleaning process for inner and outer source and rod set
  - reduce noise from contaminants



A new cleaning procedure has been introduced with the TurboMass Gold for the inner and outer source during its assembly. This has improved the stability and sensitivity of the system



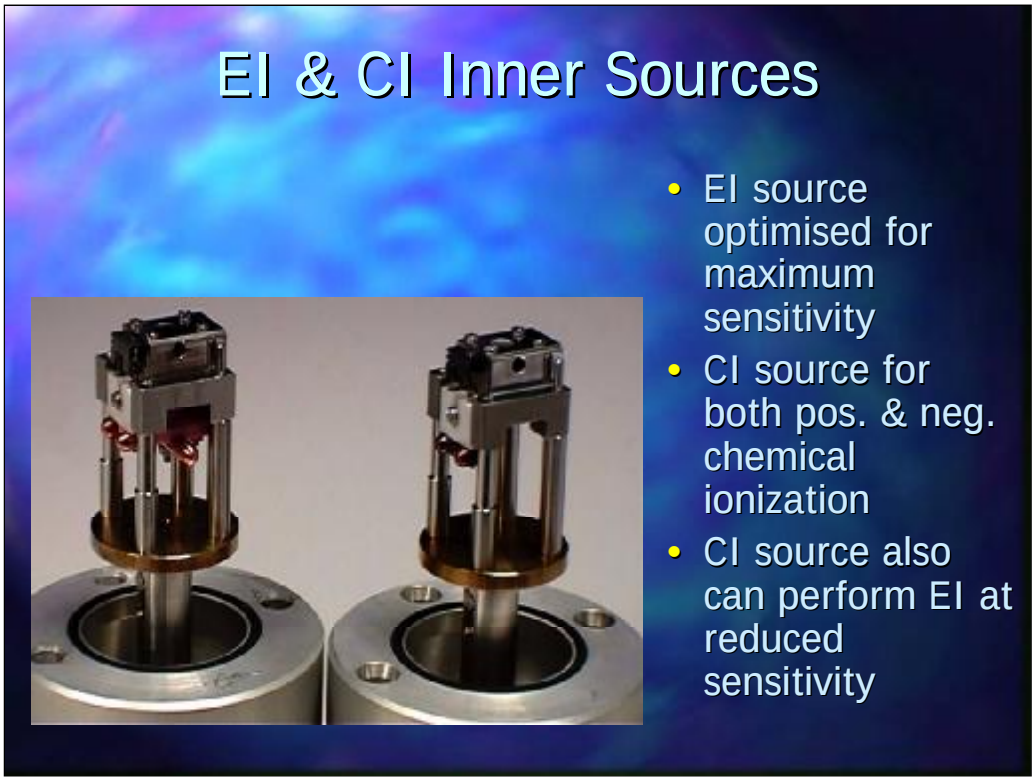
The Ion source itself consists of two parts, the outer source which is non-removable and contains the lenses, magnets, heaters and gas connections and the inner source, which is user removable, containing the filament, repeller and trap electrodes and ion volume.

Quick Change Inner Source

- No tools needed
- Simply unscrew two knurled nuts for complete removal
- Wiper Contacts automatically make & break all electrical connections
- Change source & re-stabilise in less than 15 min

The inner source may be removed and serviced by the user. No tools are required to remove the source, it is simply held in by two knurled nuts. Electrical contacts to between the inner and outer sources are made using wiper contacts which automatically make 7 break when the source is installed and removed.

The low ion volume, combined with the high pump rate achieved with the turbomolecular pump mean that sources can be changed and the system re-stabilised in around 15 minutes.



Two inner sources are available. The EI source is optimised for maximum sensitivity for EI+ analysis. A CI source is used for chemical ionisation and may be used in both the CI+ and CI- modes. The ion chamber is designed to maintain the ionisation gas in the ion chamber. It can also be used for EI analysis but with a much reduced sensitivity compared with the EI source.

Inner Source Filament Assembly

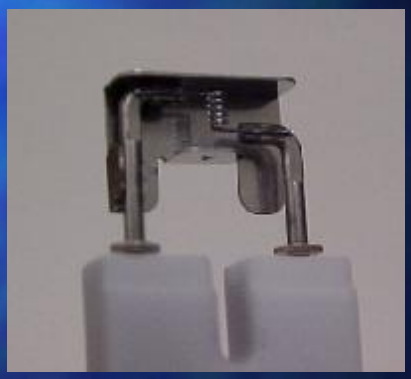
- Compact rugged assembly
- Easy change - single screw removal
- Single Filament for Optimum positioning
- Auto aligning
- Electrical wiper contacts eliminate cable connections

The Filament is part of the inner source and is held in place with a single screw. If a new filament needs to be installed it is a simple operation and the new filament automatically aligns when fitted. The TurboMass gold also has an extended shield over the filament that reduces stray light from the filament and reduces noise

TurboMass Gold Rhenium Filament

New 0.20mm diameter wire rhenium filament

- **runs at lower current than TurboMass filament**
- **better stability for certain specific applications**



The TurboMass Gold uses a new rhenium filament. This runs at a lower current (around 3.5a) than the TurboMass tungsten filament resulting in better stability and lower noise.

TurboMass Gold Analyzer Enhancements

Improvements for Stability and Sensitivity

- Refined cleaning process for inner and outer source and rod set
  - reduce noise from contaminants
- Replace aluminium dynode with gold-plated copper
  - eliminate oxidation

The aluminium dynode that was used in the TurboMass system has been replaced with a gold plated copper dynode to eliminate the possibility of surface oxidation. This, combined with new cleaning procedures for the inner and outer sources and the quadrapole assembly has resulted in a much more stable and higher sensitivity system.

TurboMass Gold Summary

- EI, CI+ and CI- modes (only one CI source)
- EI possible with CI source (reduced sensitivity)
- Quick & Simple Source changeover
- Full Scan, or SIR, in all modes
- SIMULTANEOUS FullScan & SIR data collection
- Picogram (Full Scan) & sub-picogram (SIR) sensitivity
- Extensive Mass Range (1.5 to 1200 Da)
- Fast scanning (6200 Da / sec)
- Compatible with all fused silica capillary columns