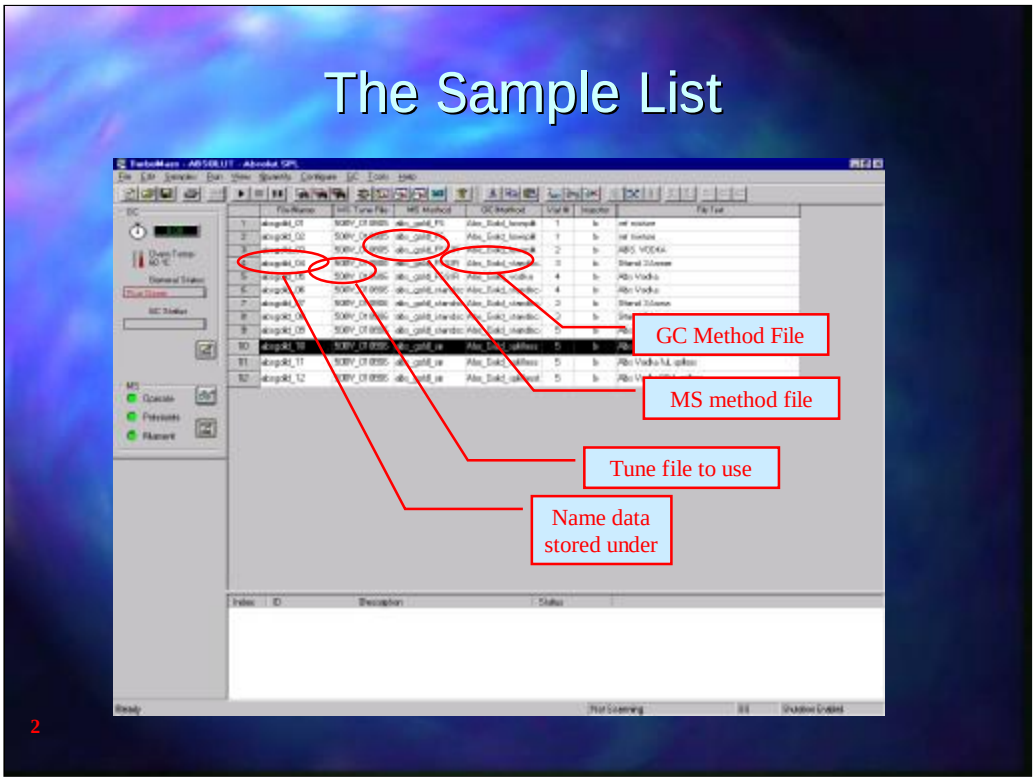

Running a sample &
Component Identification
1



The sample list consists of a number of rows and columns, each row representing a separate analytical run. Each project may contain any number of sample lists. The columns displayed may be edited by clicking with the right mouse in the grey area at the top of any column. Typical column headers include:-

File Name – The name that the data collected will be stored

MS Tune File – Can be used to control sensitivity with the PM Voltage

MS Method – Used to collect total ion chromatograms (TIC) and/or Selected ion recording chromatograms(SIR)

GC Method – Control of the AutoSystem GC

Vial Number – For the AutoSystem GC Autosampler

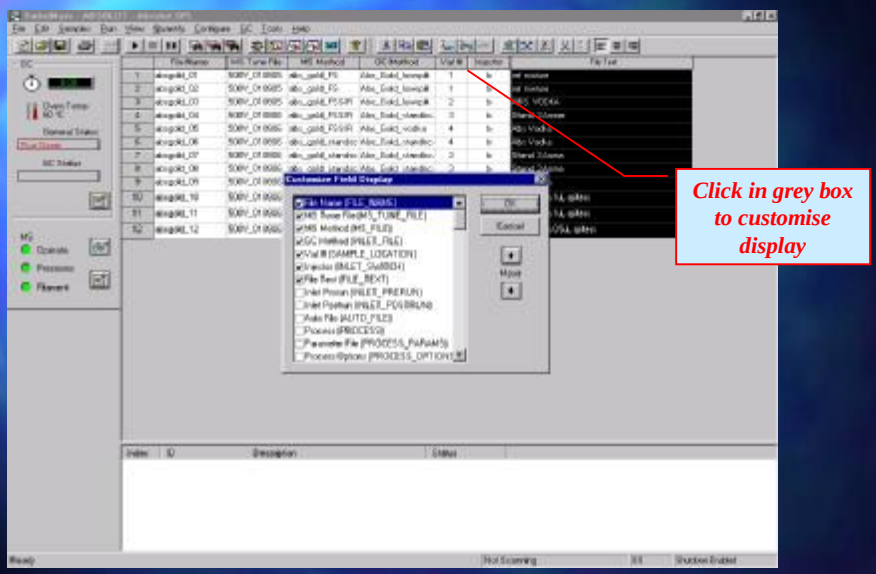
Injection Site – On the AutoSystem

Sample Type – Sample or Calibrant

Concentrations- For Calibrants

And many others

Customising the Sample List



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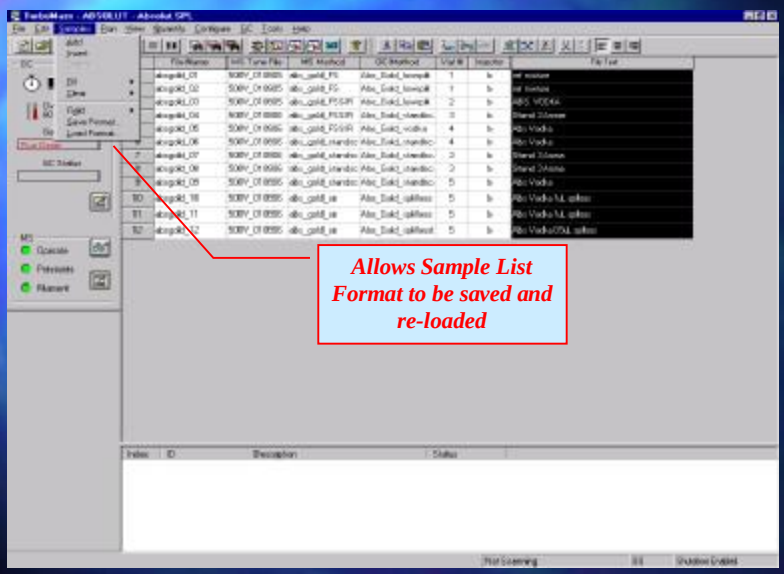
The sample list may be customised by clicking with the right mouse button when the pointer is in any one of the grey “header” boxes at the top of each column

Customising the Sample List



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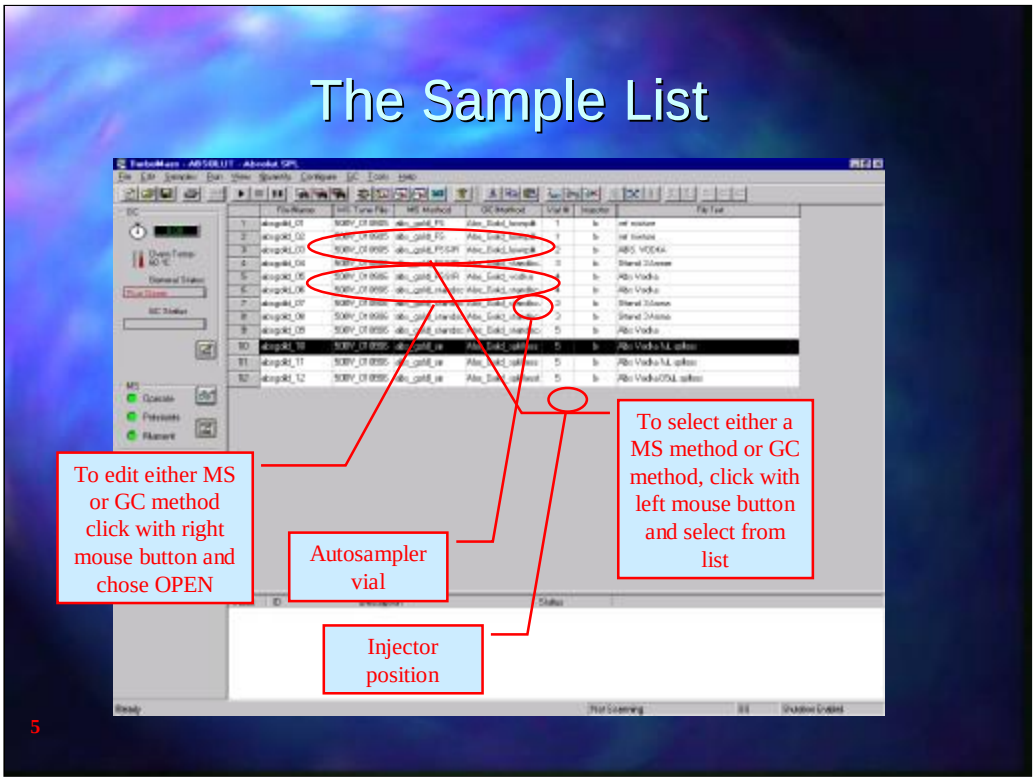
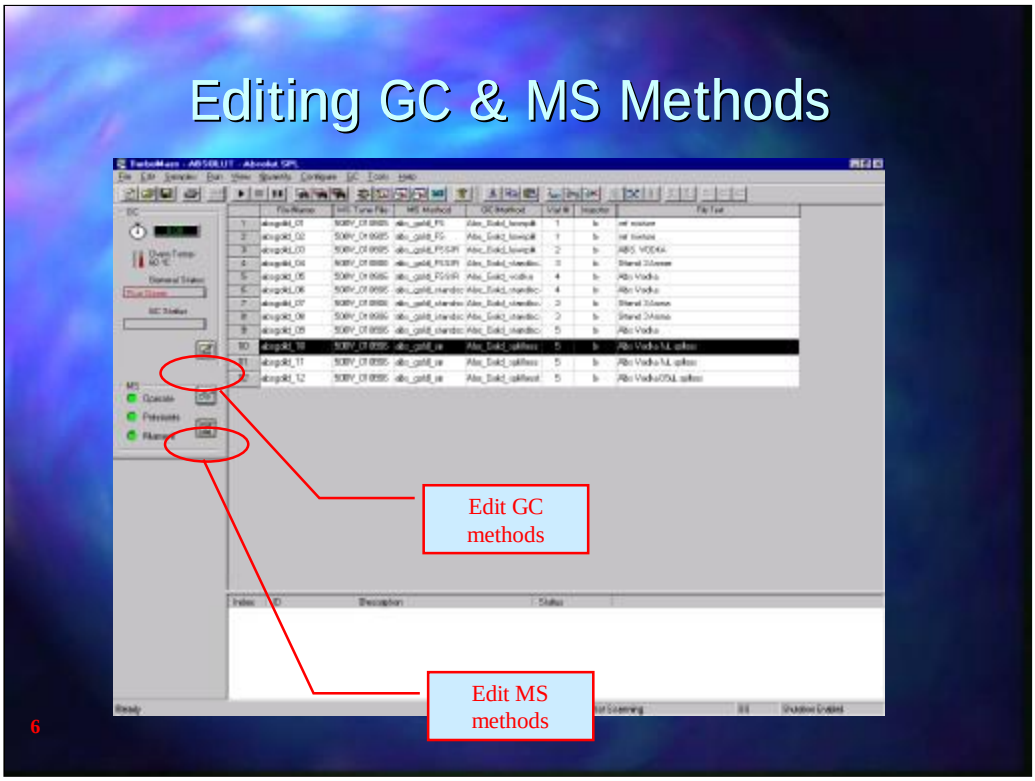
The format for the sample list may be saved using the SAMPLES, SAVE FORMAT. The sample list format files are automatically saved to the C:\turbomass directory. Similarly, LOAD FORMAT will recall a saved sample list format.



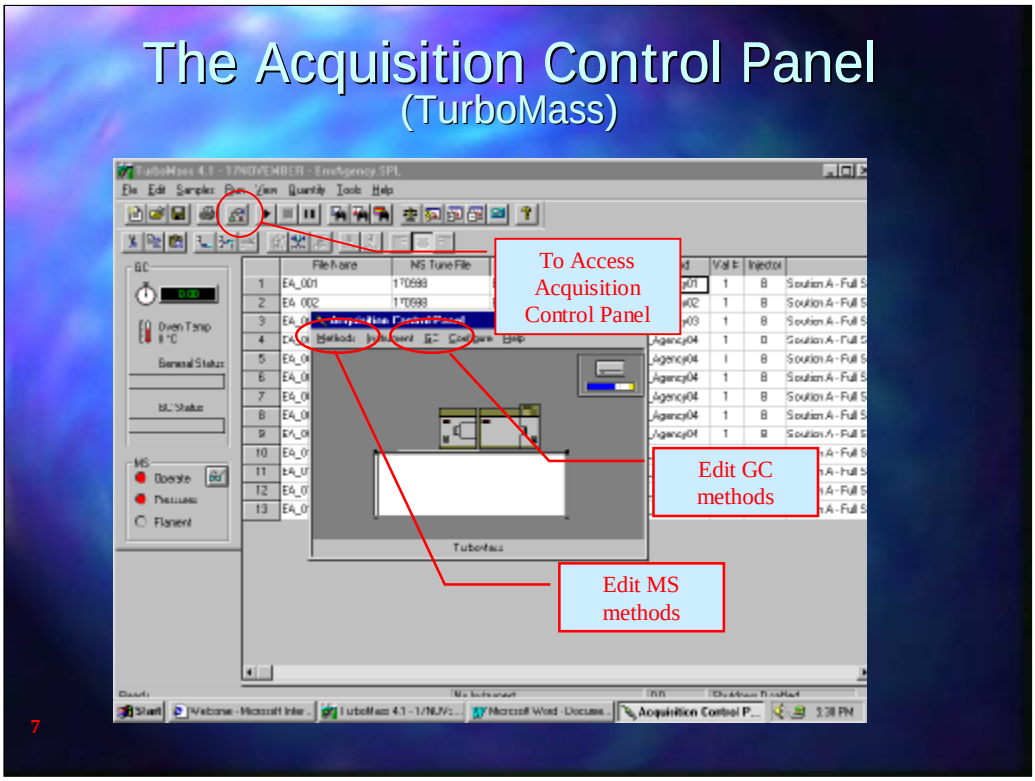
The GC method and the MS method may be accessed directly from the sample list for editing purposes by pointing to desired cell and clicking with the right mouse button and selecting open.

A list of all the available methods is obtained by clicking with the left mouse button to bring up a drop down list.

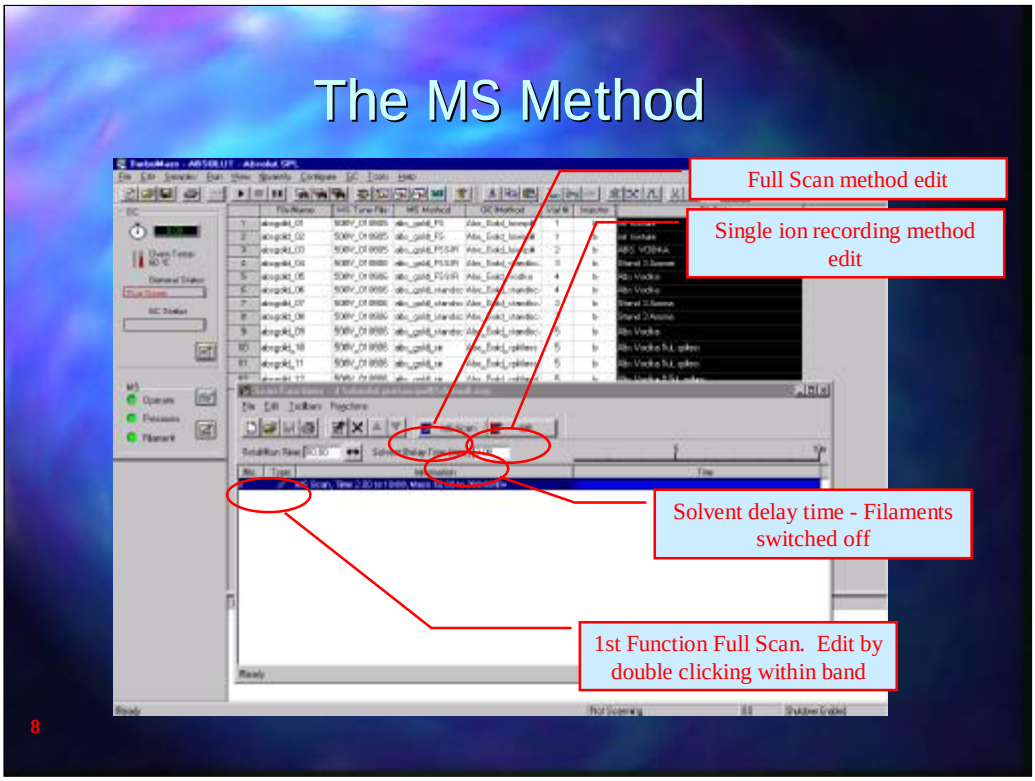
As new rows are added the file name and the vial number are automatically incremented by one. If the original file name contained only text the number 1 will automatically be added when a new row is added and then be incremented when further rows are added.



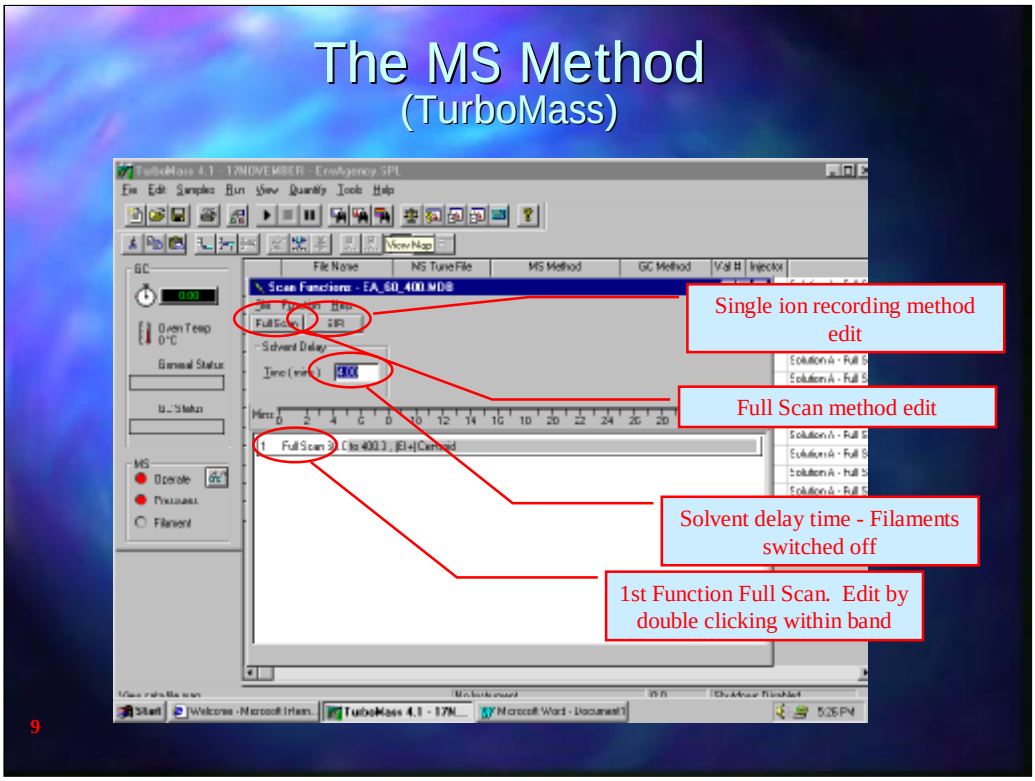
Both MS Methods and GC methods can also be opened from the Sample List Page by clicking on the button. The upper button, in the GC Status box, will open the GC method editor,whilst the lower button, in the MS Status Box, will open the MS method editor. The GC status also shows the oven temperature and status of the GC whilst the MS status box give the status of the vacuum and filament, green for OK, red for a poor vacuum or filament failure and blank when the filament is turned off.



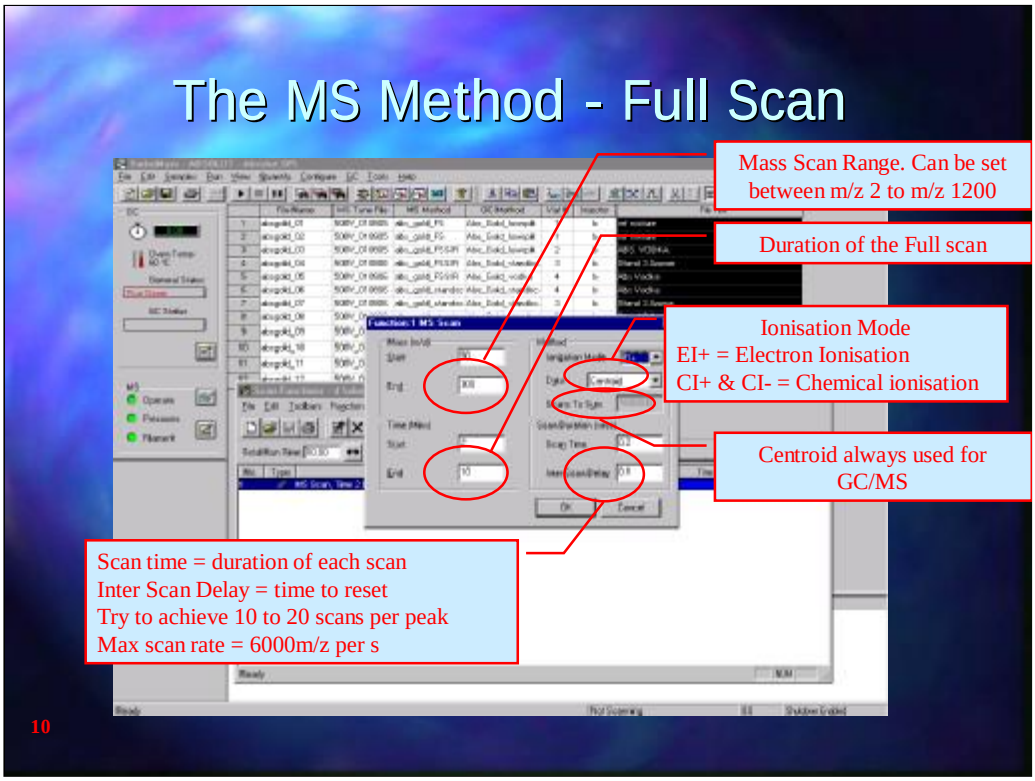
Both MS Methods and GC methods can also be opened from the Acquisition Control Panel. The Acquisition Control panel is accessed by clicking on the  button.



The MS method will always contain one Full Scan function. This is known as function 1. To edit function 1 point the mouse anywhere inside the box and double click. Further functions can be added using the buttons marked Full Scan for further traces or SIR for a selected ion recording scan. Functions may be active at different times of the GC analysis, for example using a SIR to improve the sensitivity at a time when a component is known to elute, or at the same time. The window labelled Solvent Delay Time is a delay time before the filaments are switched on to allow large volumes of solvent to pass through the system.



The MS method will always contain one Total Ion Chromatogram (TIC) as a default, known as function 1. To edit function 1 point the mouse anywhere inside the box and double click. Further functions can be added using the buttons marked Full Scan for further TIC traces or SIR for a selected ion scan. Functions may be active at different times of the GC analysis, for example using a SIR to improve the sensitivity at a time when a component is known to elute, or at the same time. The window labelled Time is a delay time before the filaments are switched on.



The Full Scan mode collects a Total Ion Chromatogram (TIC).

The time period over which the TIC is to be collected are entered by Start Time and End Time in the Retention Window. Mass determines the starting and ending masses for the Scan.

Ionisation Mode determines how the sample molecules will be ionised.

EI+ - Ionised by electron bombardment. The molecule is highly fragmented and is typically used for identification with libraries such as NIST and Wiley.

CI- - Chemical Ionisation of the molecule in the presence of an ionising gas such as methane or butane. A much softer ionisation process with very little fragmentation of the molecule. Usually used to determine the molecular ion and hence the molecular weight of the molecule.

CI+ - Chemical ionisation using NH_3 as the ionising gas. Specific for halogenated species

Data Always set to Centroid for GC/MS work.

Repeats is only active when more than one function is used and determines how many full scans are performed before it moves on to the next function

Scan Duration The time period for each scan. This needs to be set with some care as it determines the number of scans across each peak. For good quantitation this needs to be set to obtain at least 15 scans across the width of the peak and with an 0.25mm id column peak widths of about 3 seconds are possible.

The Inter-Scan delay is the period between each scan and needs to be taken into consideration when calculating the number of scans across a peak.

Effect of Scan Time on m/z 50 to m/z 60 ratio

(TurboMass Gold only)

Start Mass	End Mass	Mass Range	Scan Time (s)	Inter-scan Delay (s)	Scan Rate (Da/s)	50/69 (%)
48	547	500	0.08	0.020	6238	4.08
48	547	500	0.08	0.015	6238	4.49
48	547	500	0.08	0.010	6238	0.75
48	247	200	0.04	0.015	4975	4.56
48	247	200	0.04	0.010	4975	4.38
48	247	200	0.04	0.005	4975	0.65
48	247	200	0.03	0.015	6633	4.33
48	247	200	0.03	0.010	6633	4.20
48	247	200	0.03	0.005	6633	0.72

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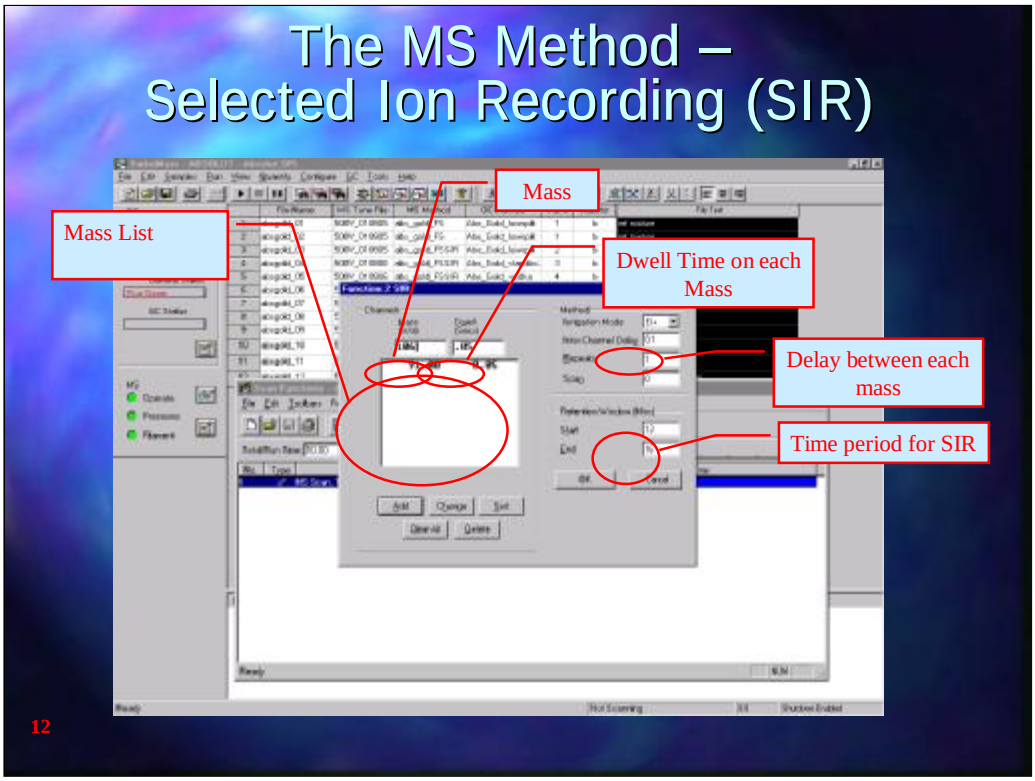
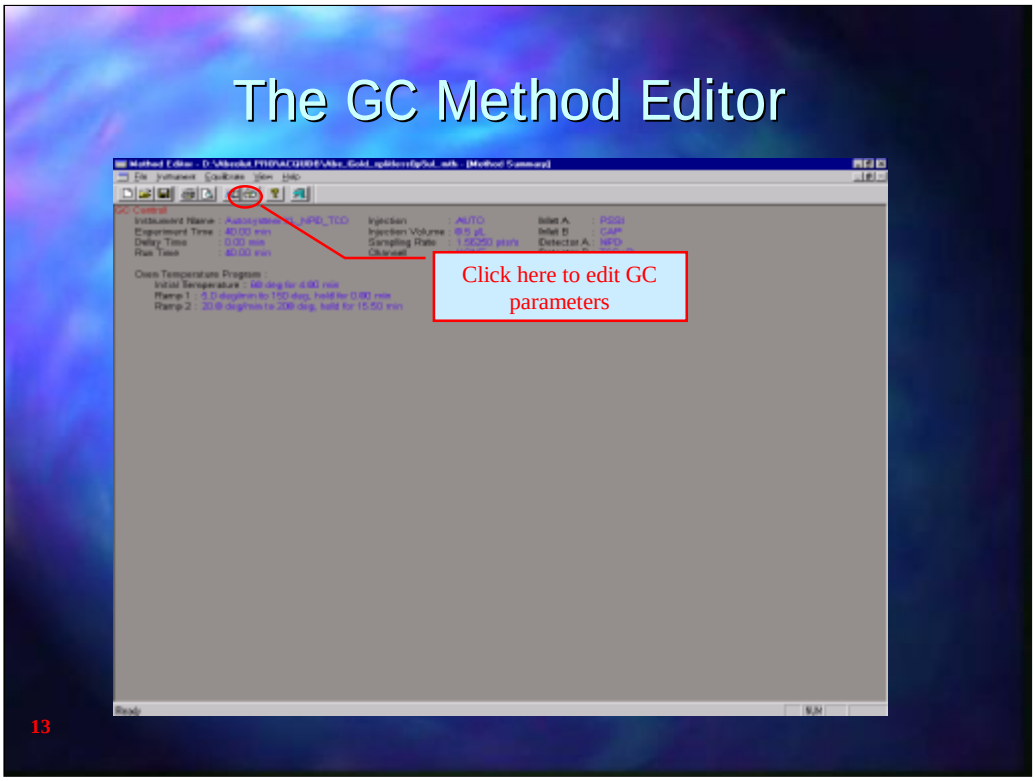
This slide shows the effects of varying Scan times and Inter Scan delay times by measuring the ratio of m/z 50 and m/z 69 for the reference gas. The abundance of m/z 50 should be around 4 to 4.5% of the abundance of m/z 69. As can be seen from this data there are significant errors in the m/z 50 / m/z 69 ratio with an Inter-scan delay of 0.010 seconds for a mass range of 500 and at an Inter-scan delay of 0.005 seconds for a mass range of 200. This technique may be used to determine the optimum scan time and Inter-scan delay if there is any doubt.



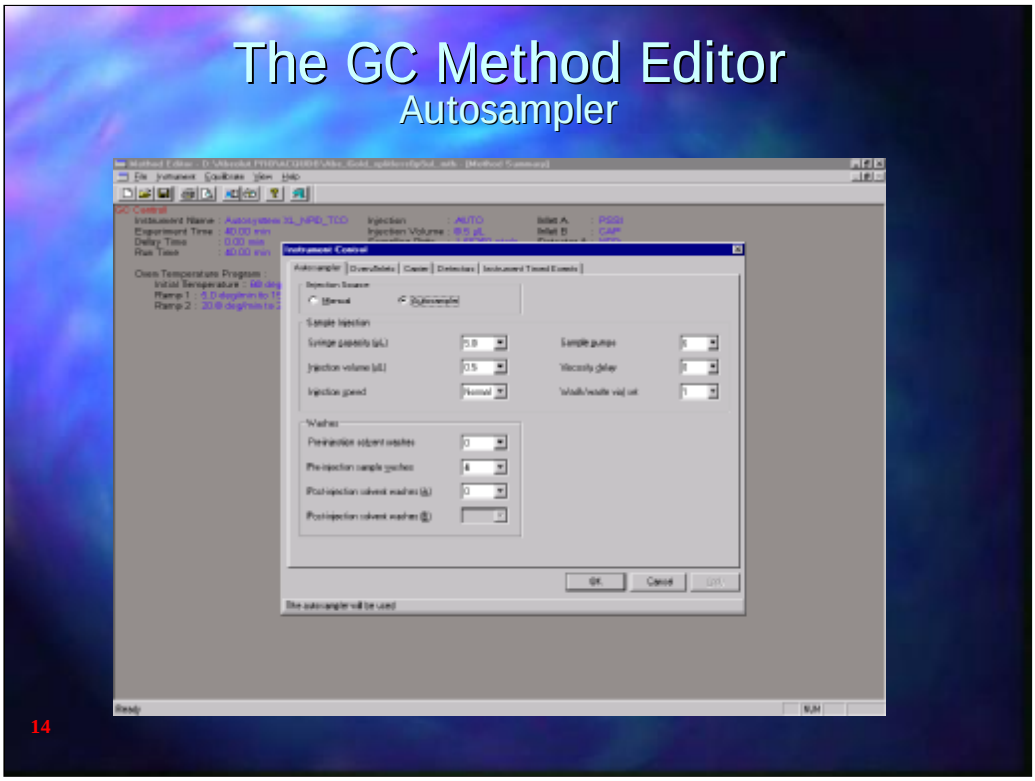
Selected Ion Recording is used for monitoring a single ion or a few selected ions rather than a full a Full Scan. Selected ion recording will give an improved sensitivity over Full Scan and depending on which ion or ions are chosen can be around 50 time more sensitive than Full Scan.

Mass determines which ions are monitored

Dwell is the length of time spent on each ion. This, along with the Inter-Channel delay. Although Selected Ion recording only monitors a few ions, no more than 10, the chromatogram obtained from an SIR function is still referred to as a Total Ion chromatogram (TIC) al be it from less than 10 ions



The GC Method Editor top page is shown above. To edit the GC parameters click on the  button.



The 1st page in the GC Method Editor controls the Autosampler but does not specify which vials to inject. These are specified in the Sample List. This page will not be displayed if an autosampler is not installed on the GC.

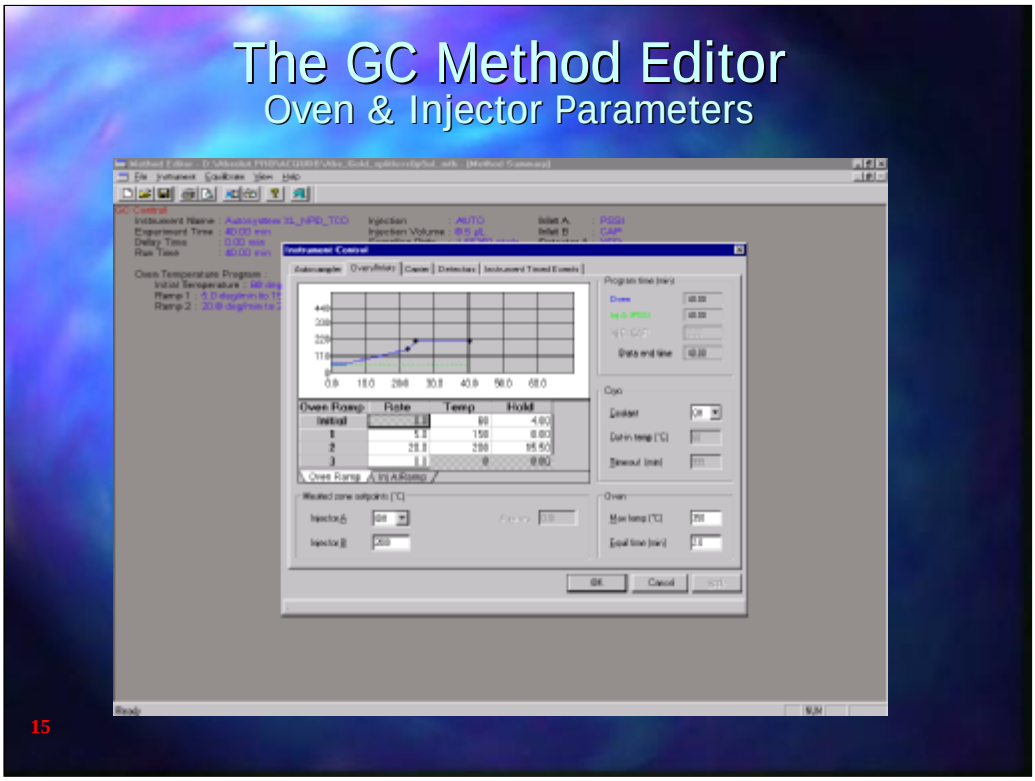
The Syringe capacity must match with the syringe that is installed in the autosampler and the injection volume determines how much will be injected.

Three injection speeds are available; Normal is similar to a manual injection. Fast injects the sample in 200ms. It can only be used for flash vaporisation and gives a very narrow sample band. It also minimises discrimination effects seen with wide boiling range samples. Do **NOT** use with syringes that have an 0.47mm needle.

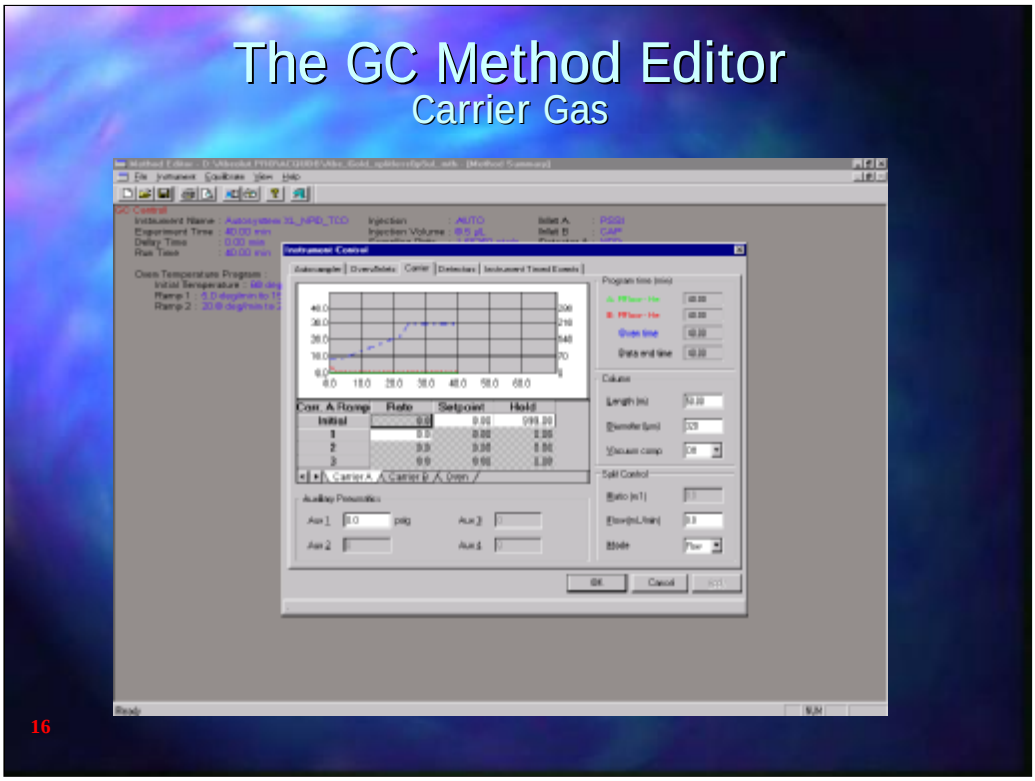
Sample pumps is the number of times that the plunger is operated during syringe filling to ensure that there are no air bubbles in the syringe.

Viscosity delay is entered in seconds and is a wait period at the top of each plunger stroke to allow viscous samples to “catch up” with the plunger.

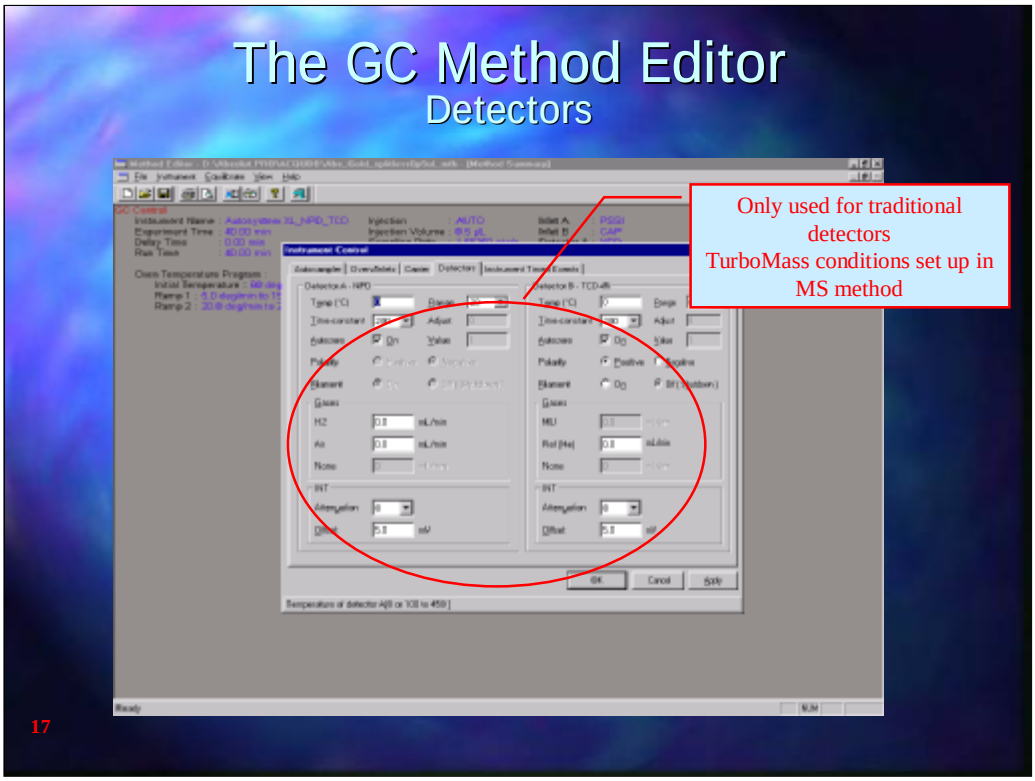
There are 2 Wash/waste sets. 1uses wash and waste vials 1 and 2; 2 uses wash and waste vials 3 and 4. With solvent washes the first half of the washes will be from the odd numbered vial and the second half from the even number vial. The odd numbered waste vial will receive waste from the sample washes and the even numbered vial will receive waste from the solvent washes.



The Oven/Inlets page gives control of the oven and injector temperatures. Note that if a Programmable split/splitless injector is installed the temperature programme is entered by accessing the Injector A and/or the Injector B tabs situated behind the Oven Ramp tab. A maximum oven temperature, to protect the column, and an equilibration time, to allow the column to equilibrate with the GC oven temperature after cooldown after a temperature programme are also entered on this page



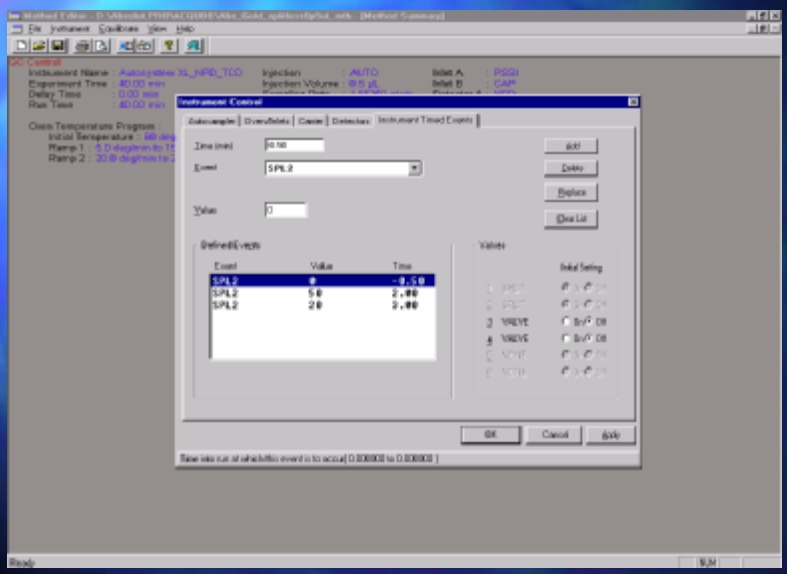
The Carrier page controls the carrier gas programme. The display will depend on how the pneumatics have been configured on the GC, whether it is set for pressure control (psi or kPa), flow control (ml/min) or gas velocity control (cm/s). The primary gas control is a pressure regulator and if either flow control or gas velocity control have been selected it is essential to enter the correct column length and internal diameter (µm)! to enable the software to calculate the correct pressure to achieve the desired value. As the column outlet will be in the ion source and under vacuum it is essential that Vacuum compensation is also turned **ON**.



The Detector page in the GC method is only for control of the conventional GC detectors, FID, ECD etc and so would not normally be used with TurboMass.

The GC Method Editor

Timed Events



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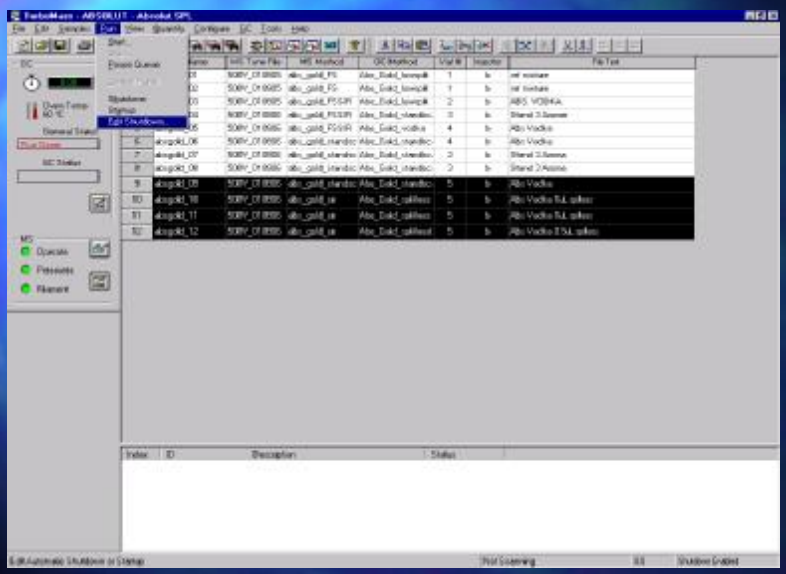
The Timed events section allows for parameters such as split flow rate etc to be changed during the analysis

Editing Shutdown Procedures



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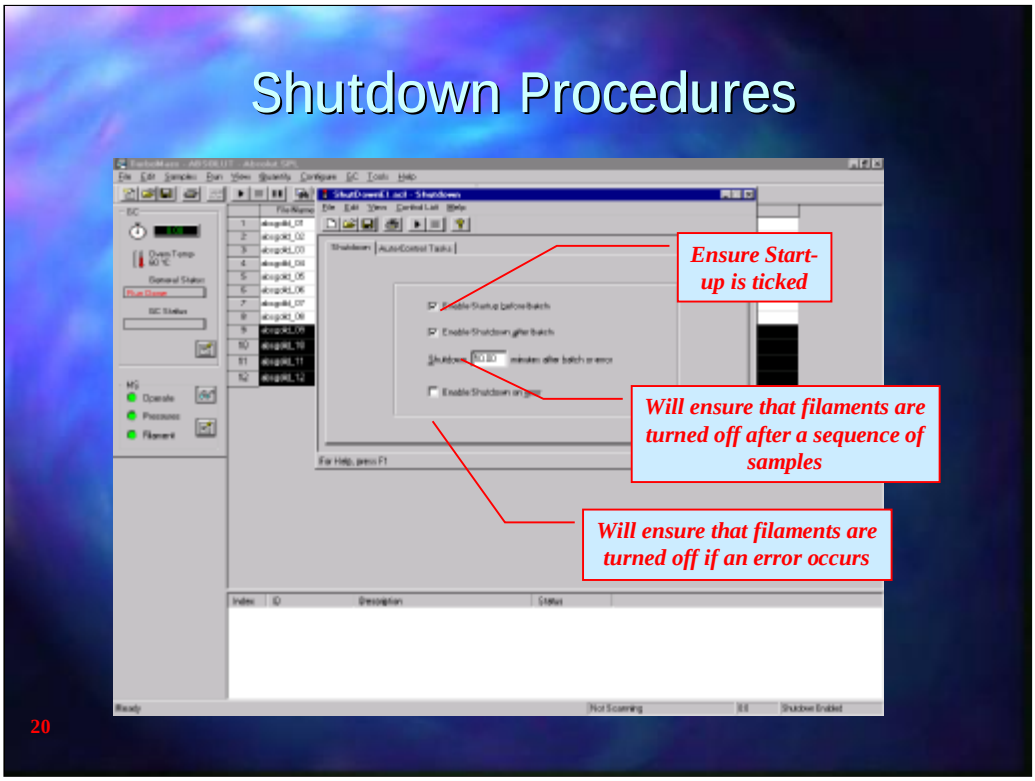
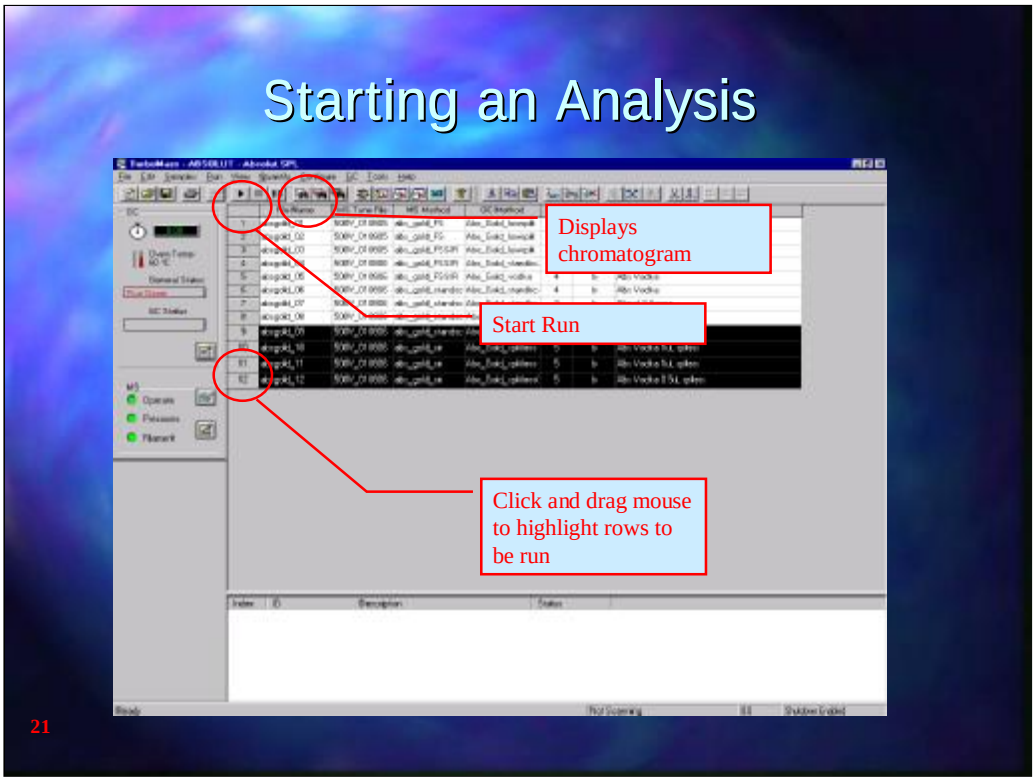
Before setting up a sequence of samples for analysis it is necessary to ensure that the SHUTDOWN procedure has been set up correctly. This is accessed from the SAMPLES drop down menu under EDIT SHUTDOWN.



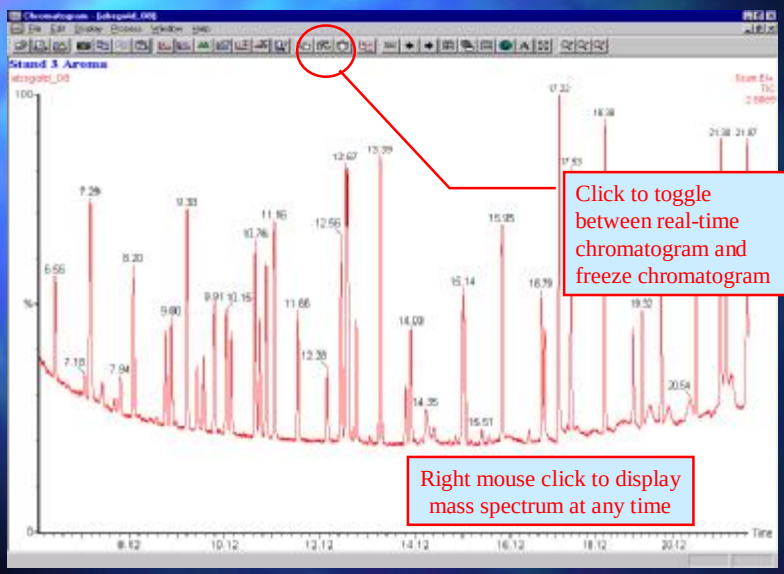
During Standby the filament is normally turned off to prolong its life. When a sequence of analyses start it is necessary to turn the filament on. This is set up in the Edit Shut down window and it is essential that Enable Startup before Batch is ticked. Also Enable Shut down after batch should be ticked to turn the filaments off at the end of a batch of samples. A delay time can also be entered to keep the filament on for a short time in case there are more samples to be run. It is also a wise precaution to tick the Enable shut down on Error so that the filaments are turned off if an error occurs in the system.



To start an analysis highlight the analyses to be performed by clicking and dragging the mouse over the row numbers (the grey cells) to be analysed. Then click the  button.

The mass chromatogram can be displayed at any time by clicking on the  button

The Chromatogram Page



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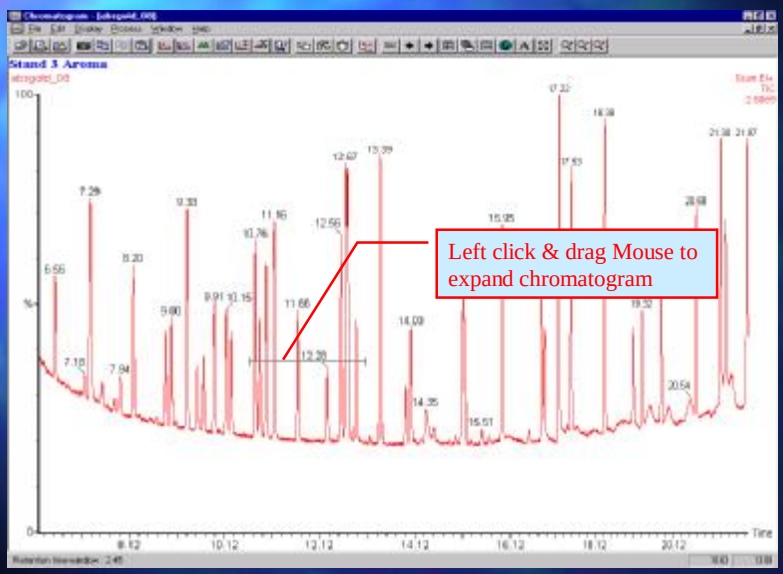
To view the chromatogram developing click on the button. When it is highlighted the display will be updated continually. If however, the display is expanded it is better to turn the update mode off by clicking on the same button as the display will automatically rescale if the trace reaches the end of the window when update is turned on.

Clicking with the right mouse anywhere in the display will automatically bring up the mass spectrum for the scan at the point in time.

The Chromatogram Page



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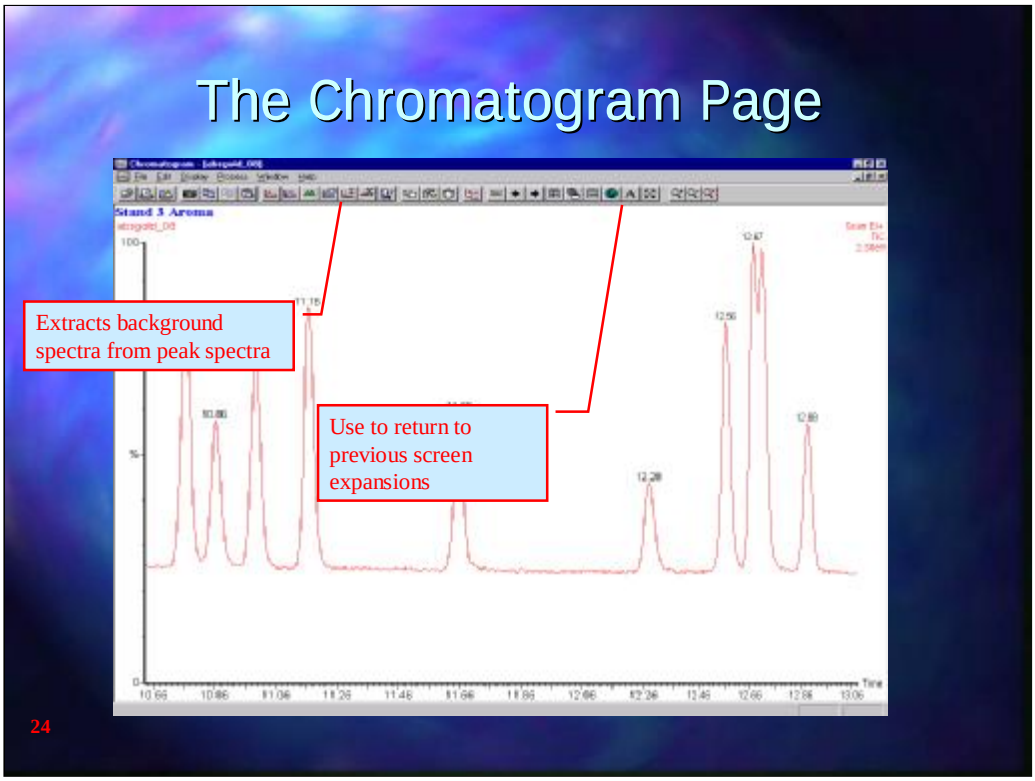
The chromatogram may be expanded by clicking and dragging with the mouse over the portion of the chromatogram to be viewed. The chromatogram may be expanded horizontally, vertically or a combination of both. A black line or box will show the potion that will be displayed after expansion

Note: It is better to have the Real time update turned off before expanding the chromatogram.

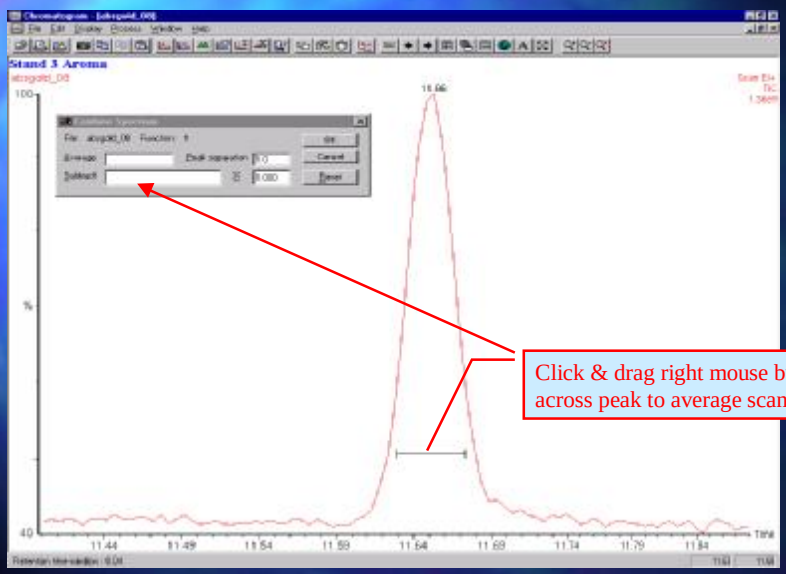


To return to the previous chromatogram display click the  button.

To obtain a mass spectrum of a component peak it is best to subtract the background from the column etc. from the peak. This may be done by using the  button. This allows both individual scans to be combined, to give a truer representation of the mass spectrum, and background to be subtracted.

The Chromatogram Page

Obtaining a mass spectrum



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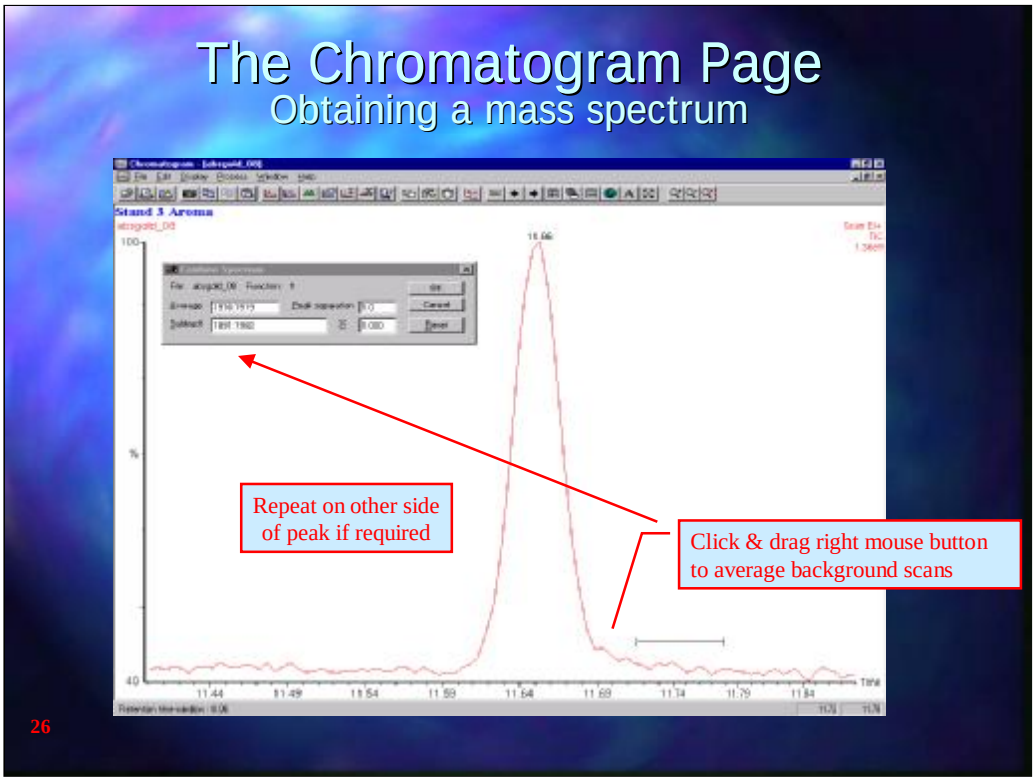
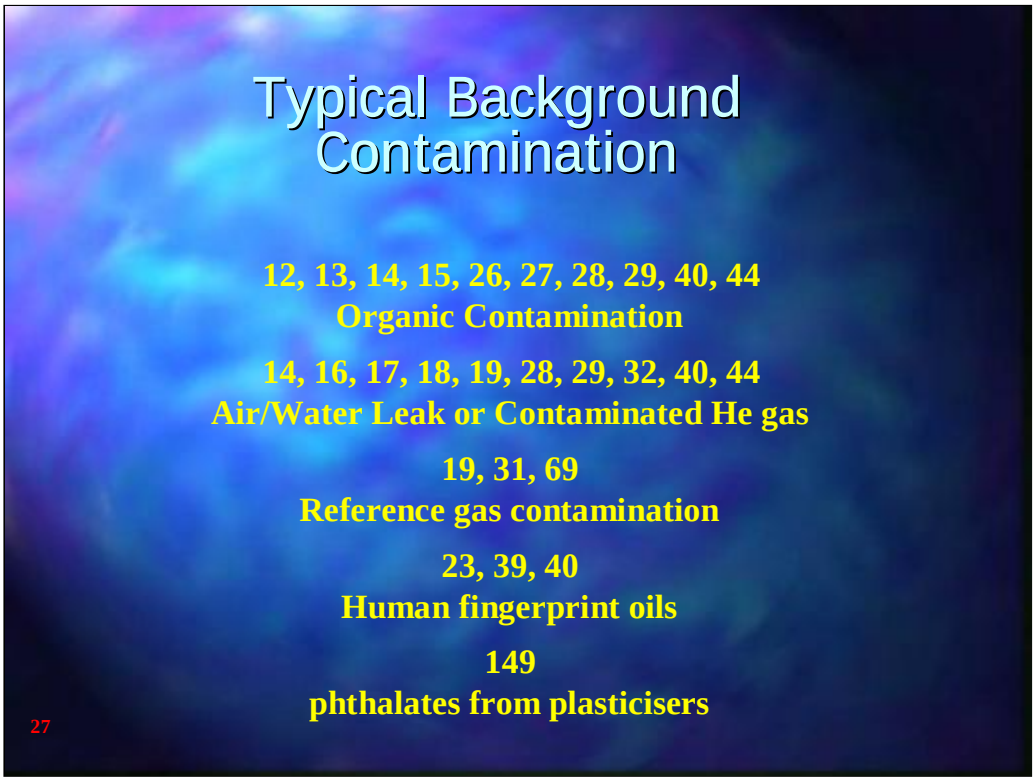
It is usually best to expand the chromatogram first so that the peak(s) of interest can be seen in detail. Click on  to bring up the Combine window. Click and drag with the Right mouse button across the peak to combine the scans obtained when the component was eluting from the column

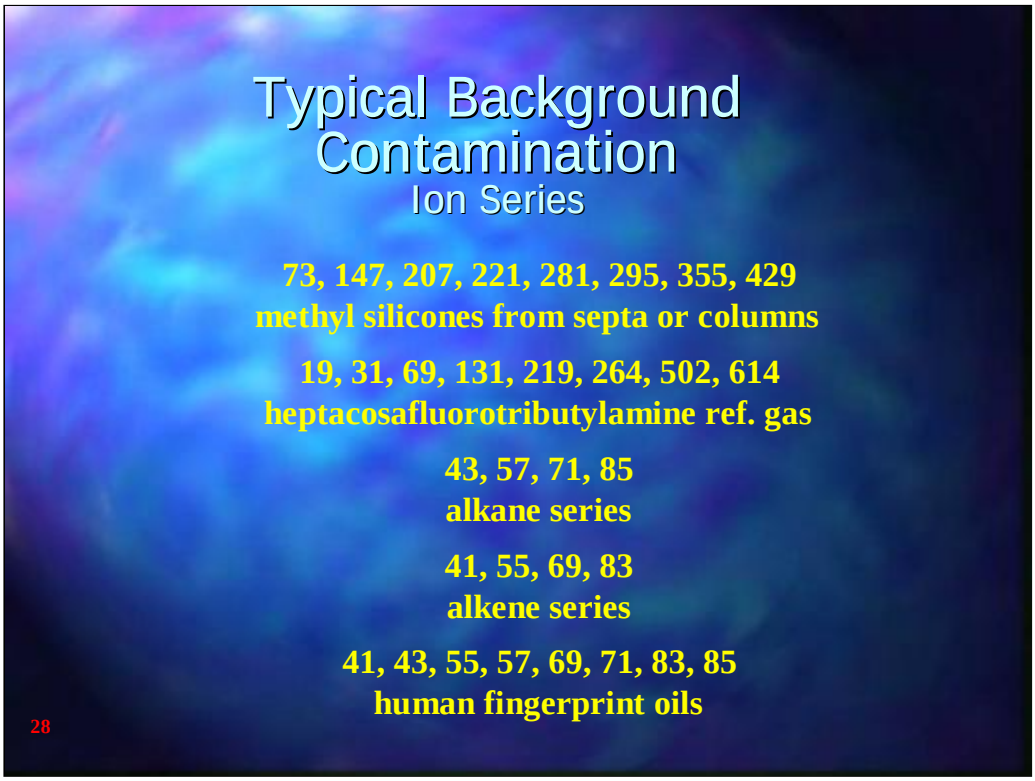


Now click and drag with the right mouse button across a portion of the baseline close to the peak. The scans that the mouse was dragged across will be combined and entered into the subtract box. This may be repeated on the other side of the peak so that the baseline on both sides of the peak will be subtract from the mass spectrum of the peak.



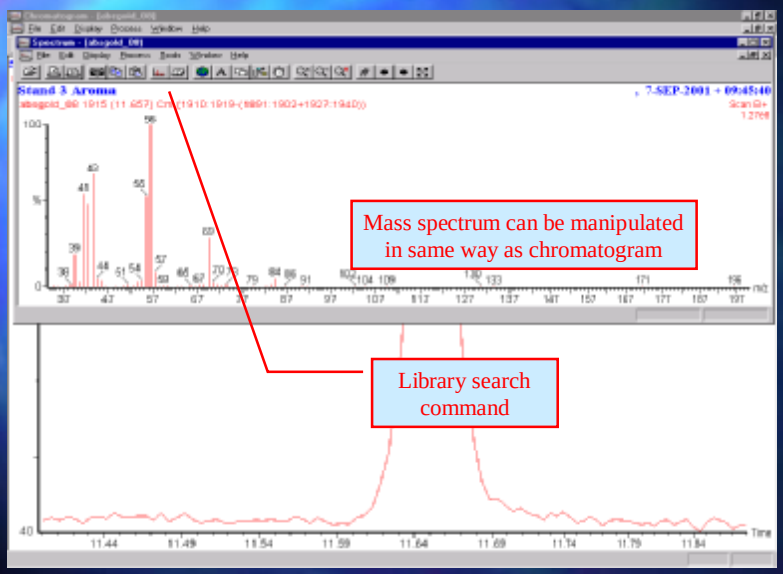
The reason that the background needs to be subtracted is that there will be some ions present that that are derived from system contamination from the total GC system. These need to be subtracted from the peak to obtain a better representation of the mass spectrum that is due to the component under investigation.

Some ions typically seen in GC/MS systems and their causes are listed here.



Some components commonly associated with GC/MS background contamination show characteristic ion series, which are listed here.

The Mass Spectrum



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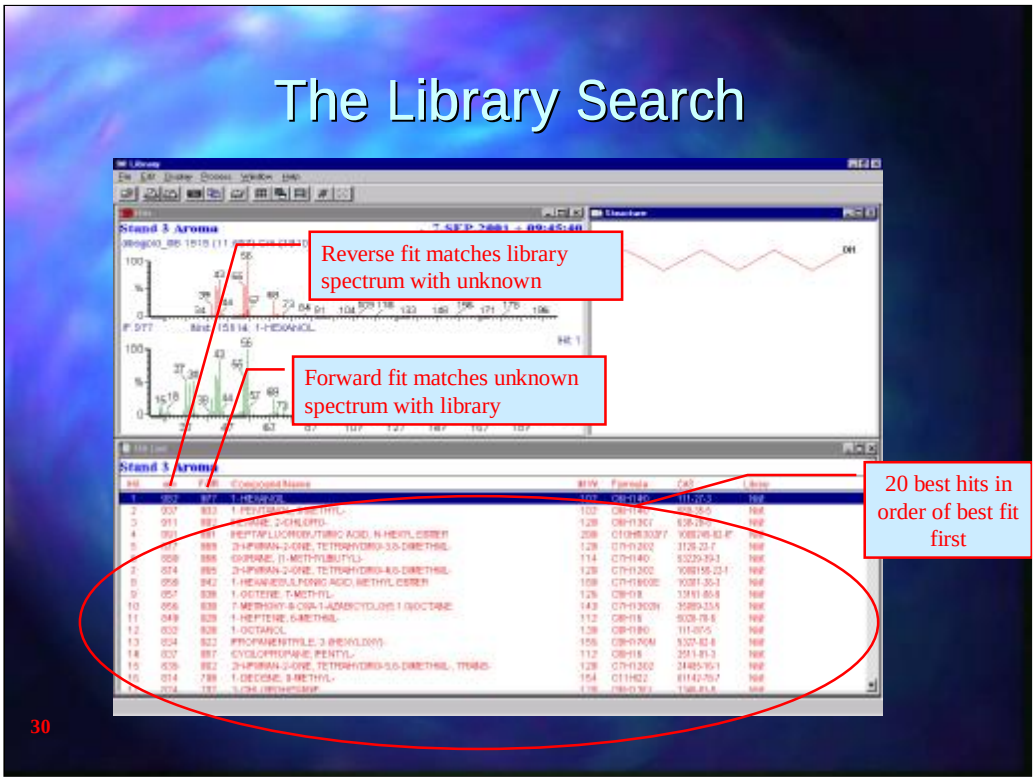
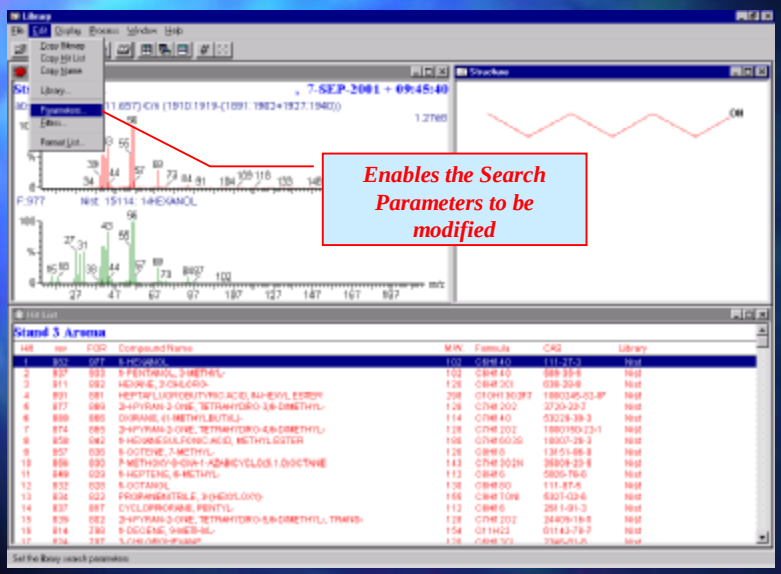
When OK in the combine spectra window has been clicked the software will combine the spectra obtained across the peak and subtract the combined spectra from the background to produce a “corrected” spectrum. A library search can then be performed by clicking on the  button.



The library search will listed the 20 closest “hits” found in the library to the spectrum from the analysis. The hits are (usually) arranged according to best Reverse Fit match first and Forward Fit match second. The Reverse fit is a measure of how well the ions in the library spectrum match with ions in the sample spectrum whilst the Forward Fit compares ions in the sample spectrum with those in the library spectrum. Both give the match a number out of 1000.

Library Search Parameters



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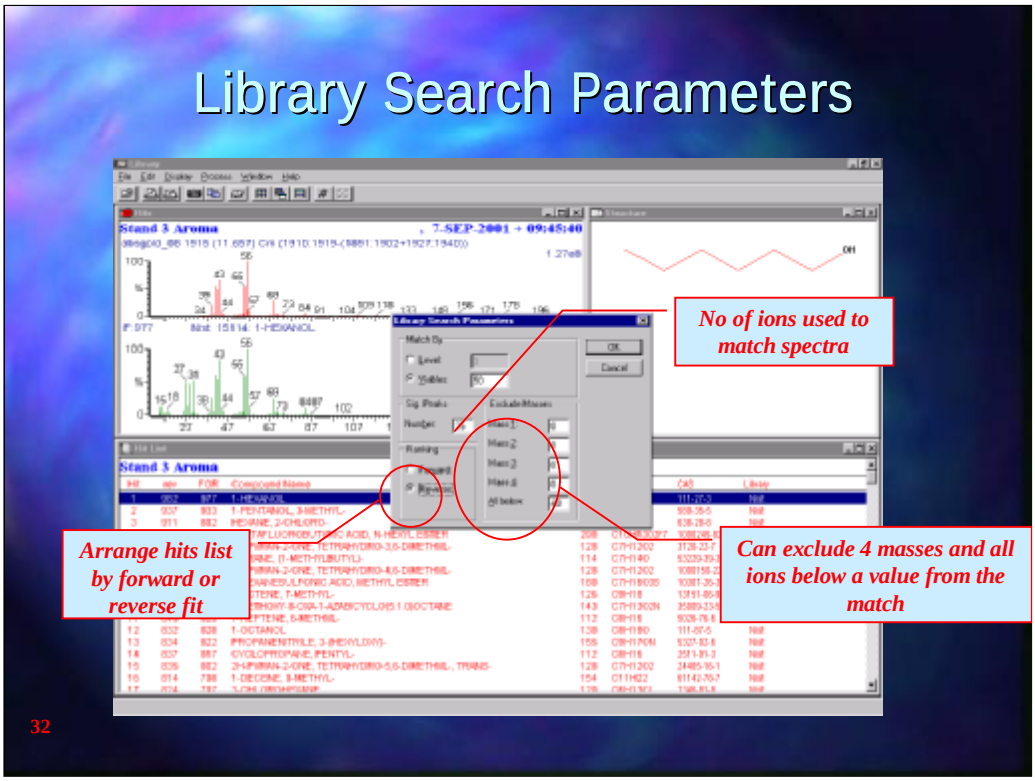
Occasionally the library search may not be match spectra very well. This is usually caused by stray ions from the background, because the library spectra have been scanned from a much lower mass number or because the spectra not not have sufficient ions of a high enough intensity to make a good comparison. The Library Search Parameters may be fine tuned to enable a better match to be achieved. They are accessed from the EDIT drop down menu



The Search Parameters that have the largest effect on the search routines are:-

Sig(nificant) peaks Refers to the number of ions used to compare the unknown spectra with the library spectra. The default value is 35 but if a the spectrum shows very few significant ions then the background noise ions will also be used in the comparison.

Exclude Masses Allows 4 background masses to be excluded from the library comparison. Can also exclude all masses below a given value. When the MS method has been set up to start the scan from a certain mass that mass should be entered here.