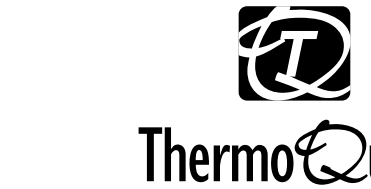

Finnigan TRACE MS

[Including Voyager and MD Series]

Hardware Manual

Revision B
FM101555

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P.R. China: ThermoQuest China • Suite 912-916, Ping An Mansion. • No. 23, Jin Rong Street • Xicheng District • Beijing 100032 • [86] (010) 6621 0839
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Finnigan TRACE MS Hardware Manual
Revision B
P/N FM101555

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	Strongly Agree	Agree	Neutral	Disagree	Strongly Disagree
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The instructions are easy to follow.	1	2	3	4	5
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The technical information is easy to understand.	1	2	3	4	5
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Crewe Road
Wythenshawe
Manchester M23 9BE
UK
Tel: + 44 (161) 946 1060
Fax: + 44 (161) 998 9882
Web: www.finnigan.co.uk

EU DECLARATION OF CONFORMITY

The EU Directives covered by this Declaration

89/336/EEC	Electromagnetic Compatibility Directive, amended by 92/31/EEC & 93/68/EEC
73/23/EEC	Low Voltage Equipment Directive, amended by 93/68/EEC
93/68/EEC	CE Marking Directive

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The Finnigan TRACE MS series (formerly MD series) of mass spectrometers for GC/MS.

The Basis on which Conformity is being Declared

The products identified above comply with the EU directive 89/336/EEC by meeting the following standards:

EN55022 Class A:1994	Limits and methods of measurement of radio interference characteristics of information technology equipment.
EN50082-1:1992	Electromagnetic compatibility - Generic immunity standard. Part 1. Residential, commercial and light industry.
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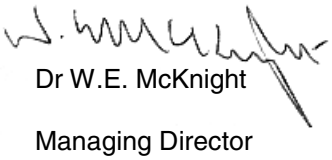
Signed: 
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Authority: Managing Director

Date: May 1999

ATTENTION!

The attention of the purchaser, installer and user is drawn to the special measures and limitations to use which must be observed when the product is taken into service to maintain compliance with the above directives. Details of these measures are contained in the User Manual.

ISO 9001
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Read This First

Welcome to the Finnigan TRACE MS system!

The **TRACE MS Hardware Manual** contains a description of the modes of operation and principal hardware components of your TRACE MS system. In addition, this manual provides step-by-step instructions for the cleaning and routine maintenance of your system.

> **Note.** The information contained within this manual can also be applied to the Voyager and MD Series of mass spectrometers. However, the reader should be aware that all text, examples and Figures refer only to the Finnigan TRACE MS for the purpose of simplification.

The **TRACE MS Hardware Manual** includes the following chapters:

Chapter 1: Introducing GC/MS and TRACE MS provides a general introduction to GC/MS technology and techniques, and also introduces the TRACE MS system.

Chapter 2: Choosing, Installing and Maintaining a GC Column describes how to choose, install and maintain a GC column.

Chapter 3: Changing MS Ionization Modes describes how to change the ion source: removing the existing source and installing a new one.

Chapter 4: Maintaining the System provides details of the maintenance tasks which must be carried out to keep the TRACE MS system in its optimum condition.

Chapter 5: Troubleshooting is designed to help you diagnose and resolve problems that may occur from time-to-time with the TRACE MS system.

Chapter 6: Shutting Down and Restarting the System describes how to shut down the TRACE MS system for a temporary period (up to 2 weeks) or for a longer period. It also describes how to restart the system following a long-term shut down.

Chapter 7: Consumables and Spare Parts lists the TRACE MS system consumables and replaceable parts.

Changes to the Manual and Online Help

To suggest changes to this manual or the online Help, please send your comments to:

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You are encouraged to report errors or omissions in the text or index.
Thank you!

Abbreviations

The following abbreviations are used in this and other TRACE MS manuals and in the online Help.

A	ampere
ac	alternating current
ADC	analog-to-digital converter
amu	atomic mass unit
AP	acquisition processor
APCI	atmospheric pressure chemical ionization
API	atmospheric pressure ionization
ASCII	American Standard Code for Information Interchange
b	bit
B	byte (8 b)
baud rate	data transmission speed in events per second
°C	degrees Celsius
cfm	cubic feet per minute
CD	compact disc
CD-ROM	compact disc read-only memory
CI	chemical ionization
CIF	Carriage, Insurance and Freight Paid to
CIP	Carriage and Insurance Paid to
cm	centimeter
cm^3	cubic centimeter
CPU	central processing unit (of a computer)
CRM	consecutive reaction monitoring
CSE	customer support engineer
<Ctrl>	control key on the terminal keyboard
d	depth
Da	dalton
DAC	digital-to-analog converter
dc	direct current
DDS	direct digital synthesizer
DS	data system
DSP	digital signal processor

EI	electron ionization
<Enter>	<Enter> key on the terminal keyboard
EMC	electromagnetic compatibility
ESD	electrostatic discharge
ESI	electrospray ionization
eV	electron volt
f	femto (10^{-15})
°F	degrees Fahrenheit
FID	Flame Ionization Detector
FOB	Free on Board
FPD	Flame Photometric Detector
ft	foot
FTP	file transfer protocol
g	gram
G	giga (10^{9})
GC	gas chromatograph
GC/MS	gas chromatograph / mass spectrometer
GND	electrical ground
GPIB	general-purpose interface bus
GUI	graphical user interface
h	height
h	hour
HPLC	high-performance liquid chromatograph
HV	high voltage
Hz	hertz (cycles per second)
ICIS™	Interactive Chemical Information System
ICL™	Instrument Control Language™
IEC	International Electrotechnical Commission
IEEE	Institute of Electrical and Electronics Engineers
in.	inch
I/O	input/output
k	kilo (10^{3}, 1000)
K	kilo (2^{10}, 1024)
kg	kilogram

l	length
L	liter
LAN	local area network
lb	pound
LC	liquid chromatograph
LC/MS	liquid chromatograph / mass spectrometer
LED	light-emitting diode
m	meter
m	milli (10^{-3})
M	mega (10^{6})
M+	molecular ion
µ	micro (10^{-6})
min	minute
mL	milliliter
mm	millimeter
MS	scan power: MS^1
MS/MS	scan power: MS^2
MS^n	scan power: MS^n, n = 1 through 10
m/z	mass-to-charge ratio
n	nano (10^{-9})
NCBI	National Center for Biotechnology Information (USA)
NIST	National Institute of Standards and Technology
Ω	ohm
p	pico (10^{-12})
Pa	pascal
PC	personal computer
PCB	printed circuit board
PID	Photo Ionization Detector
PMD	Photo Multiplier Detector
PMT	Photo Multiplier Tube
P/N	part number
P/P	peak-to-peak voltage
ppm	parts per million
psig	pounds per square inch, gauge

RAM	random access memory
<Return>	<Return> key on the terminal keyboard
RF	radio frequency
RMS	root mean square
ROM	read-only memory
RS232	industry standard for serial communications
s	second
SCSI	small computer system interface
SIM	selected ion monitoring
solids probe	direct insertion probe
TIC	total ion current
TCP/IP	transmission control protocol / Internet protocol
Torr	torr
u	atomic mass unit
URL	uniform resource locator
V	volt
V ac	volts alternating current
V dc	volts direct current
VGA	Video Graphics Array
w	width
W	Watt
WWW	World Wide Web

Note. Exponents are written as superscripts. In the corresponding online Help, exponents are written with a caret (^) or with *e* notation because of design constraints in the online Help. For example:

MS^n (in this manual)	MS^n (in the online Help)
10^5 (in this manual)	10^5 (in the online Help)

Typographical Conventions

Typographical conventions have been established for ThermoQuest manuals for the following:

- Data input
- Notes, Cautions, and WARNINGS
- Topic headings

Data Input

Throughout this manual, the following conventions indicate data input and output via the computer:

- Prompts and messages displayed on the screen are represented in this manual by capitalizing the initial letter of each word and italicizing each word.
- Input that is to be entered by keyboard or buttons that are to be clicked on by the mouse is represented in **bold face letters**. (Titles of topics, chapters, and manuals also appear in bold face letters.)
- For brevity, expressions such as "choose **File | Directories**" are used rather than "pull down the File menu and choose Directories."
- Any command enclosed in angle brackets < > represents a single keystroke. For example, "press **<F1>**" means press the key labeled *F1*.
- Any command that requires pressing two or more keys simultaneously is shown with a hyphen connecting the keys. For example, "press **<Shift>-<F1>"** means depress and hold the <Shift> key and then press the <F1> key.

Notes, Cautions, and Warnings

Notes, Cautions, and WARNINGS are displayed in boxes such as the one below.

> **Note.** Boxes such as this are used to display Notes, Cautions, and WARNINGS.

A ***Note*** contains information that can affect the quality of your data. In addition, notes often contain information that you may need if you are having trouble.

A ***Caution*** contains information necessary to protect your instrument from damage.

A ***WARNING*** describes hazards to human beings.

Topic Headings

The following headings are used to show the organization of topics within a chapter:

Chapter 1
Chapter Name

1.1 Second Level Topics

Third Level Topics

Fourth Level Topics

Fifth Level Topics

Reply Card

TRACE MS manuals contain a Reader Survey card located at the front of each manual.

A message on the Reader Survey card asks the user to fill out and return the card after he or she has had an opportunity to use the manual. The Reader Survey card has two functions. Firstly, it allows the user to tell ThermoQuest what he or she does and doesn't like about the manual. Secondly, when the user returns the card, he or she is registered and placed on the ThermoQuest mailing list. Once registered, the user will receive ThermoQuest's newsletter *Analytical News* and will be notified of events of interest, such as user meetings.

Chapter 1
Introducing GC/MS and TRACE MS

Contents **1-i**

1.1 What is GC/MS? 1-1

1.2 Overview of GC 1-2

1.3 Overview of MS 1-4
- Ionization Techniques 1-5
  - Electron Ionization (EI) 1-5
  - Chemical Ionization 1-6
- Mass Analysis and Detection 1-8

1.4 GC/MS Data Systems 1-10
- Xcalibur 1-11
  - The Server 1-13
  - The Tune Window 1-14

1.1 What is GC/MS?

GC/MS is a highly effective analytical technique. In this technique, the power of gas chromatography (GC) to separate compounds from a complex mixture is coupled with the ability of mass spectroscopy (MS) to identify those compounds (qualitative analysis), and accurately determine the amounts present (quantitative analysis).

The chromatographic and mass spectral data is recorded by a computerized data system, which can also be used to analyze the information obtained and control the various instruments.

GC/MS is a very sensitive technique and can be used to identify samples containing femtogram quantities of compounds. It can be used to analyze samples from a wide range of sources and is routinely used in a variety of areas, for example, environmental agencies, medical and forensic laboratories, and the control boards for athletics and horse racing.

1.2 Overview of GC

Gas chromatography (GC) separates complex mixtures of compounds on the basis of their relative affinities for a mobile gaseous phase and a stationary liquid phase.

In standard split/splitless injection, a small amount of sample material is injected into the instrument and is vaporized in a heated zone at the top of a narrow capillary column (the GC column).

The sample vapor is then carried through the column (a thin tube of flexible bonded silica of between 15 and 200 meters long) in a stream of gas (typically Helium); this forms the mobile phase. The inner wall of the column is coated with a liquid; that is the stationary phase.

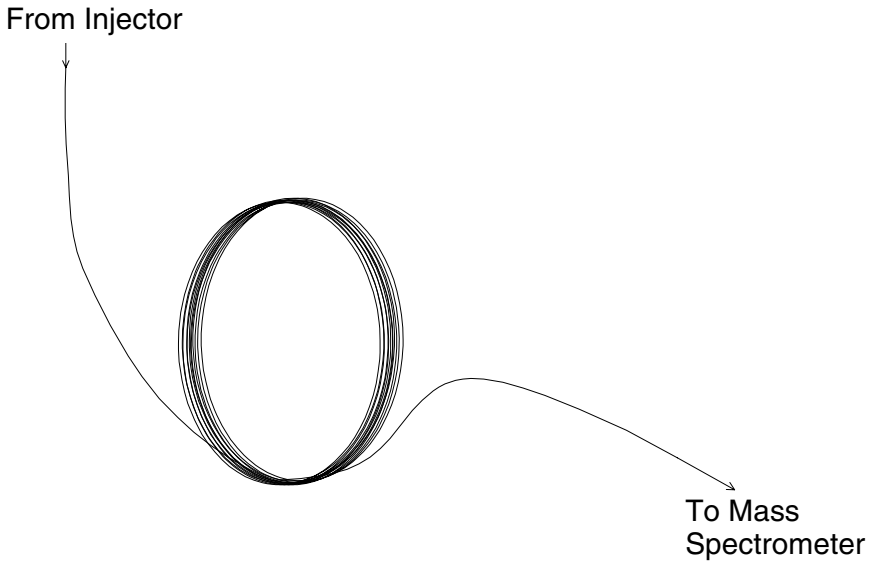

Figure 1-1. Gas chromatography column

Individual compounds travel along the column at different rates, and as a result, emerge from the end of the column at different times; that is, they become separated on the column.

The length of time an individual compound takes to move through the column, (that is, its retention time) depends upon its solubility in both the mobile and stationary phases. A compound that is more soluble in the stationary phase than in the gaseous phase tends to be “held” on the column and progresses more slowly than one that is more soluble in the gaseous phase than the stationary phase.

A typical chromatogram trace is shown below.

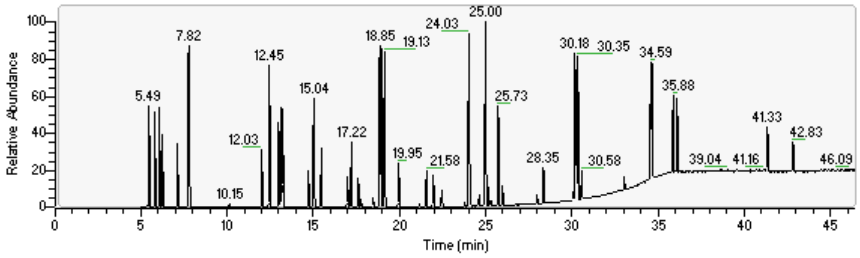

Figure 1-2. Typical chromatogram trace

In a GC/MS system, compounds emerging (eluting) from the end of the column are passed into the mass spectrometer for identification and further analysis.

1.3 Overview of MS

In mass spectroscopy (MS), various techniques are used to ionize and, in some cases, fragment ions of the compound under investigation. The molecular ion and ion fragments then pass into an analyzer, that separates them and measures both their mass-to-charge ratio and intensity.

The pattern of fragmentation for a given compound generates a characteristic spectrum or chemical fingerprint that enables identification.

In contrast to gas chromatography, a mass spectrometer is not well suited to dealing with mixtures of compounds. The mass spectrum for a mixture of compounds would be a summation of all the spectra of the individual components in the mixture.

In GC/MS, the compounds in the sample are separated on the GC column that terminates in the source, so that each compound elutes, in sequence, directly into the source as shown in Figure 1-3.

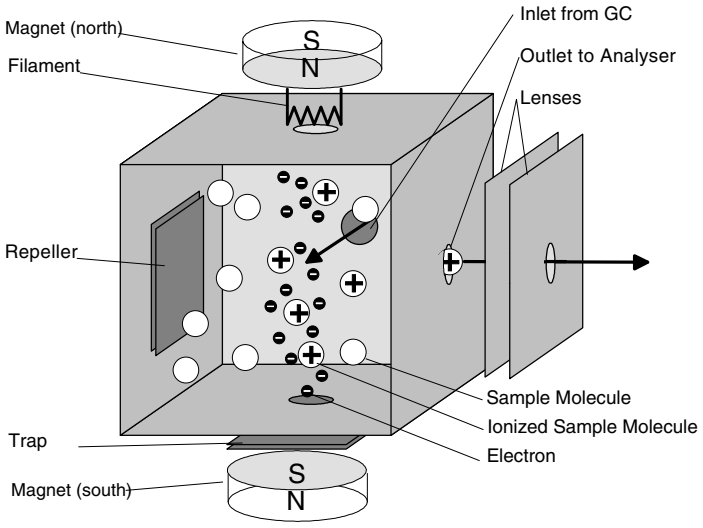

Figure 1-3. Ionization of compounds

Ionizing electrons are emitted from the filament and are kept in a tight beam (collimated) by the trap and the two magnets. The ion fragments created in the ionization process are forced out of the source and into the analyzer by electrostatic forces, applied by the repeller and the first lens. The lenses also act to focus the ions leaving the source, before they enter the analyzer.

Ionization Techniques

MS uses three major ionization techniques:

- Electron ionization (EI)
- Chemical ionization (CI+ and CI-)

Each technique is best suited to a particular range of applications.

Electron Ionization (EI)

In electron ionization (EI), the filament emits a beam of electrons that both ionize and fragment the sample molecules, as shown below.

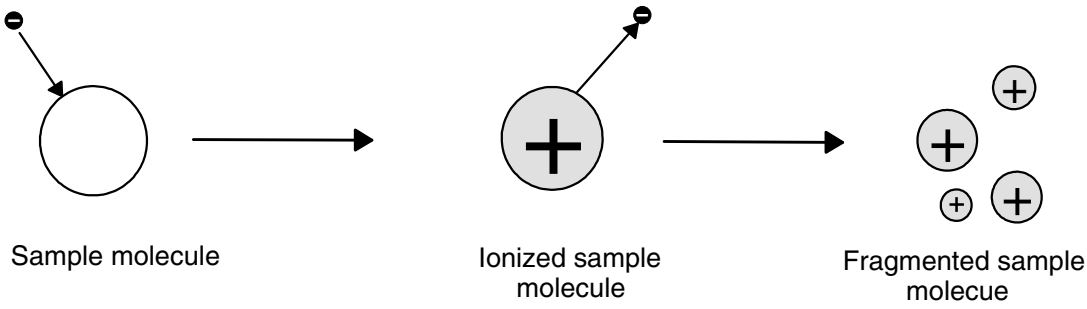

Figure 1-4. Electron ionization

EI is the most commonly used of the ionization modes, because the pattern of fragmentation of the sample molecules is consistent and forms a unique chemical spectrum. This spectrum includes the molecular ion and a range of predictable fragments.

An EI spectrum serves as a chemical fingerprint and can be compared against libraries of known spectra to determine the identity of the sample compound.

A typical EI spectrum is shown below.

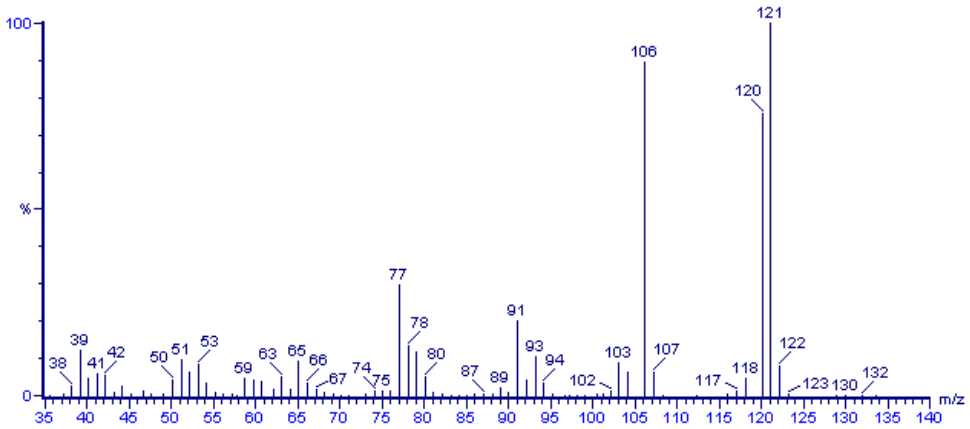

Figure 1-5. Typical EI spectrum

In this example, the peaks at 77, 91, and 106 represent ions produced by the fragmentation of the compound.

EI is used mainly for the identification of compounds, for quantitative analysis and for determining chemical structures.

Chemical Ionization

In certain cases, EI fragments the sample compound too much and this can make data identification difficult. In such circumstances, chemical ionization represents a useful, softer, alternative in which the principal ionization product is the molecular ion rather than ionized fragments.

In chemical ionization, a relatively high concentration of a reagent gas (usually ammonia, methane or isobutane) is introduced into the source, and the sample under investigation represents only a small fraction of the total number of molecules present. Thus, the electrons in the source beam interact with molecules of the reagent gas to form reagent gas ions.

There are two types of chemical ionization reaction: CI+ and CI-.

CI+ involves the direct interaction between reagent gas ions and the sample molecules, as shown below.

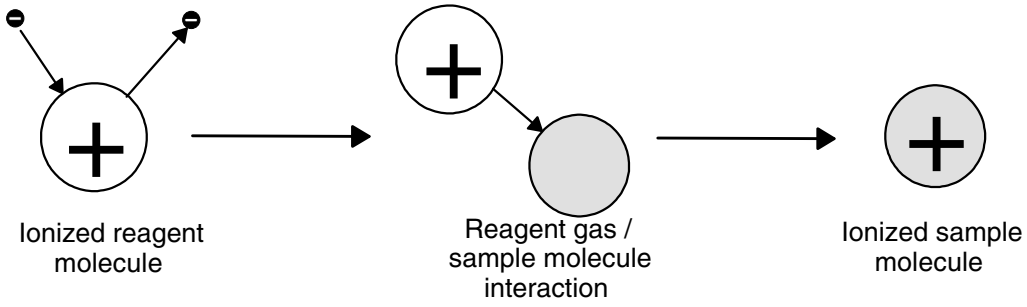

Figure 1-6. CI+ ionization reaction

CI- involves the capture of electrons that have been slowed down by the reagent gas.

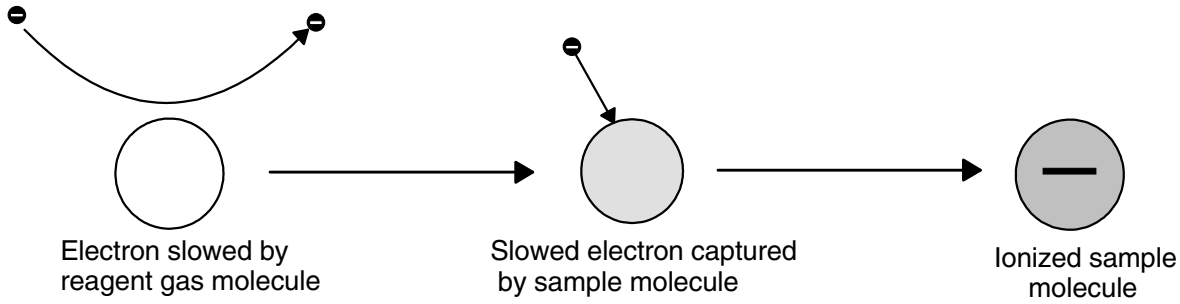

Figure 1-7. CI- ionization reaction

Both chemical ionization reactions take place simultaneously in the source, but the instrument detects only positive ions in CI+ and negative ions in CI- operation.

A typical chemical ionization spectrum is shown below.

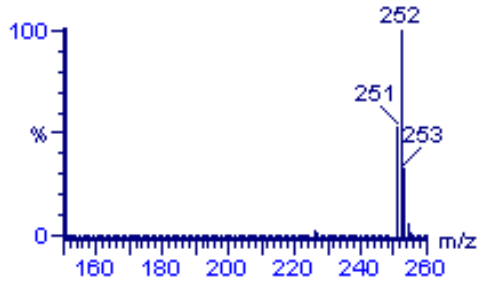

Figure 1-8. Typical CI spectrum

Because chemical ionization is a much "softer" technique, fragmentation of the molecule does not occur to the same extent as in EI. The three peaks shown in the example spectrum represent an isotope mixture of the molecular ion.

Chemical ionization is often used where molecular mass information is required, complementing the spectral data provided by EI.

Mass Analysis and Detection

Once ions leave the ion source, they pass into a mass analyzer. The function of the analyzer is to separate the ions and to measure their masses. (In fact, what is really measured is the mass-to-charge ratio (m/z) for each ion. However, because in most cases the charge is 1, the m/z value can be taken as being equivalent to the ion mass.)

In each short time interval, ions of a particular mass are allowed to pass through the analyzer whereupon they are counted by the detector. In the next short time interval, ions of a different mass are allowed to pass through the analyzer and again the detector counts how many ions are present. In this way, the analyzer scans through a large range of masses.

In GC/MS, scanning speed is important, because components that have been separated on the GC column may all elute in a short time interval.

In addition, the TRACE MS can be used to monitor selected ion masses. Selected Ion Monitoring (SIM) is typically used to look for ions that are characteristic of a target compound or family of compounds.

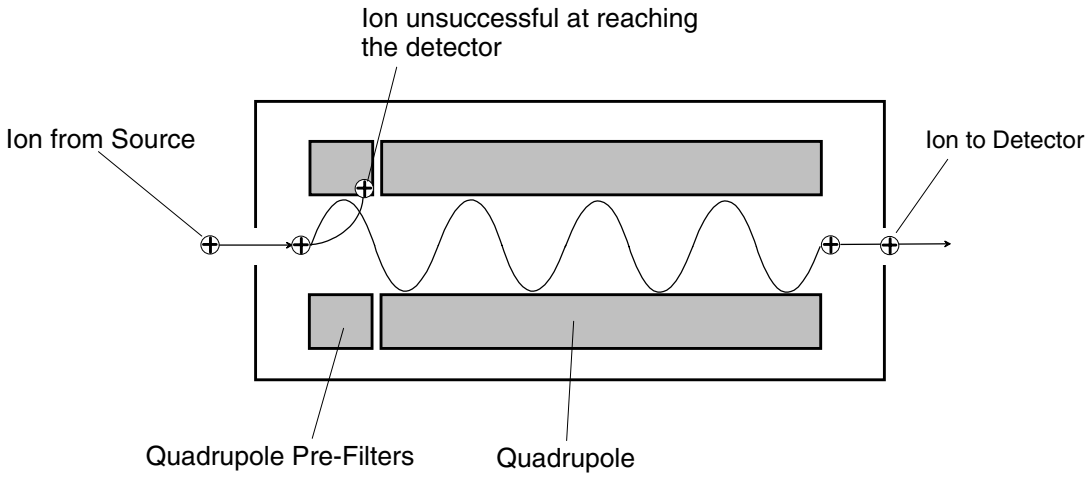

Figure 1-9. Quadrupole analyzer

The analyzer consists of four rods. DC and RF voltages are applied to each rod, creating a continuously varying electric field along the length of the analyzer. Once in this field, ions are accelerated down the analyzer towards the detector.

The varying electric field is precisely controlled so that, during each stage of a scan, ions of one particular mass-to-charge ratio pass down the length of the analyzer. Ions with any other mass-to-charge value impact on the quadrupole rods (see Figure 1-9).

The efficiency of the quadrupole is impaired after a build up of ions impacting on them. Therefore, a set of pre-filters is added to the quadrupole to remove as many of the ions that would have impacted on the main quadrupole as possible. This increases the time needed between routine cleaning of the rods.

The majority of ion intensity is concentrated in the low mass region. When the analyzer is set to pass higher masses, the majority of low mass ions will be collected on the pre-filters.

1.4 GC/MS Data Systems

A typical GC/MS scanning experiment generates a Total Ion Current (TIC) chromatogram. This is made up of a very large number of mass spectra. The instrument is set to scan throughout the period during which compounds elute from the column. The TIC is a summation of the total ion intensity in each spectrum.

The chromatogram produced is similar to that from other GC detectors. With the TRACE MS however, the data system software, Xcalibur™, is capable of viewing each spectrum making up the TIC, producing filtered mass chromatograms, and library-searching these chemical fingerprints to produce both qualitative and quantitative information as required.

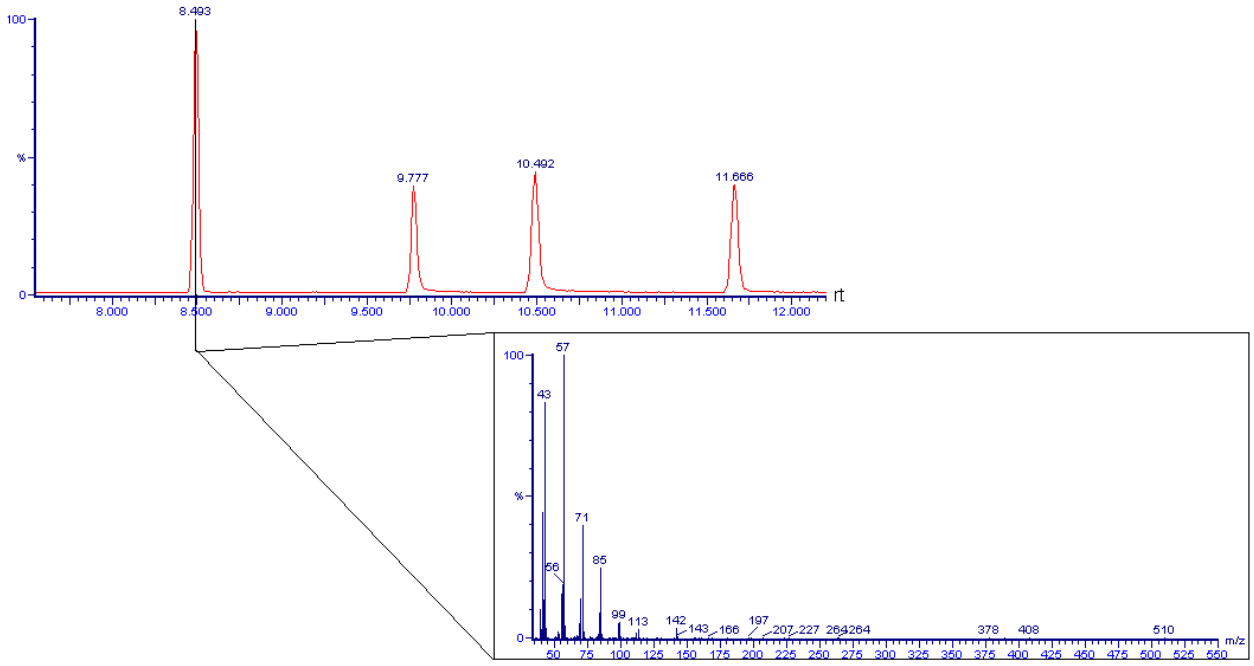

Figure 1-10. Relationship between a chromatogram and a mass spectrum

Figure 1-10 shows how a mass spectrum relates to a single point on a chromatogram trace. All other points on the trace also have a related mass spectrum.

To handle all the data that is provided during an experiment, a highly efficient computerized data system is required.

Xcalibur

The data system, Xcalibur, has complete control of the TRACE MS system and runs on a Microsoft® Windows NT® platform. For a more detailed introduction to the Xcalibur software and its usage, refer to **TRACE MS Getting Started**.

When Xcalibur is run, the Xcalibur Home page, shown below, is displayed. The Home Page provides a "road map" of the data system.

Figure 1-11. Xcalibur Home page

The icons shown on the road map provide access to all the Xcalibur modules, including:

- Instrument Setup

  Use this module to configure the TRACE MS system and attached instruments (such as the GC and autosamplers), prior to the acquisition of data.

- Processing Setup

  Use this module to specify parameters for the processing, reporting and manipulation of data acquired from samples.

- Sequence Setup

  Use this module to specify details of the samples to be examined and to control the acquisition of data by the system.

- Qual Browser

  Use this module to examine and manipulate acquired data, to determine what compounds are present within a GC peak. This involves matching the mass spectrum of your GC peak against the mass spectra held in the National Institute of Standards and Technology (NIST) library, or other libraries.

- Quan Browser

  Use this module to manipulate acquired data to obtain an accurate determination of the amounts of individual compounds present in a sample.

- Library Browser

  Use this module to perform more extensive searches of the NIST library, and to create your own library of spectra.

To access Xcalibur, double-click on the Xcalibur short-cut icon shown on the Windows NT® desktop.

Figure 1-12. Xcalibur desktop icon

The Server

The TRACE MS Server provides facilities for controlling key aspects of the TRACE MS hardware, in particular pumping-down and venting.

The Server is displayed as an icon in the Windows Taskbar. It is shown in Figure 1-13 just to the left of the time display.

Figure 1-13. The taskbar showing Xcalibur Home Page and Server

The Server is shown as three lights:

- The green, top light represents the Vacuum status.
- The red, middle light represents the Operate status.
- The yellow, bottom light represents the Reference Gas status.

Table 1-1. Server light status meanings

Server light status	Meaning
Dull	Off
Flashing (vacuum light only)	Instrument is pumping down (vacuum light only)
Bright	On

When all lights are on, The TRACE MS is under vacuum, in Operate mode, and with the reference gas on.

The Tune Window

The Tune Window can be opened by double-clicking on the Tune desktop shortcut.

Figure 1-14. Tune Window desktop icon

The Tune Window allows you to optimize the operation of the ion source. Both autotuning and manual tuning are available. For a more detailed discussion on tuning the TRACE MS, refer to **TRACE MS Getting Started**.

Chapter 2

Installing and Maintaining a GC Column

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2.1 Introduction

A good GC method provides optimum separation of chromatographic peaks while minimizing unwanted properties such as tailing or peak-broadening. This improves the chances of a successful library search for an unknown compound, or a positive identification of a trace component.

Chromatographic performance depends on a number of factors:

- Column selection
- Injector setup and use
- Column installation and maintenance
- Choice of oven temperature and flow/pressure programs

When selecting a GC column, it is important to keep in mind the maximum flow rates recommended for your TRACE MS turbomolecular pumping configuration (see Table 2-1). The TRACE MS is fitted with one of three different pumping configurations, specified at the time of purchase. These are as follows:

- 70 L/s single pumped system
- 250 L/s single pumped system
- 250 L/s differentially pumped system

Table 2-1. Maximum flow rates recommended for each TRACE MS pumping configuration

	Turbomolecular Pumping Configuration		
	70 L/s (single)	**250 L/s (single)**	**250 L/s (differential)**
Maximum Flow Rates mL/min	2*	5*	8*

* The recommended flow rates assume Helium to be the carrier gas.

The following pages in this chapter describe injector setup and use, and how to install and maintain a GC column.

2.2 Injector Setup and Use

There are two main types of injectors: split/splitless and on-column.

Split/Splitless Injector

The split/splitless (or vaporizing) injector operates at about 250 °C. At this relatively high temperature, an injected sample is rapidly vaporized into the carrier gas flow in the injector liner. The sample's progress through the GC depends on the mode of operation chosen for the analysis: split or splitless.

Split Mode

In split mode, the injector divides the sample between the column and bottom (or split) valve. The ratio of sample vented through the split vent to that transferred to the column is termed the *split ratio*. Typically the split ratio is about 100 (about 1% of the sample is passed on to the column). This lends the technique to the analysis of large, rather than trace, concentrations. Split mode is valuable in the analysis of volatile or gaseous compounds that would otherwise be difficult to focus.

Due to the brevity of the sample transfer, the column receives the sample in a sharp band. This allows high start temperatures or even isothermal oven programs, without compromising component separation.

Peak shape is directly related to the injector performance. Sample injection must be quick and smooth with no delay between the piercing of the septum and the depression of the plunger. Hesitant injections result in split peaks or broad peaks.

Splitless Mode

In splitless mode, the split (bottom) valve on the injector is closed for the splitless duration, so the entire sample is transferred onto the column. The process typically takes about 1 min to achieve 98% transfer because of the low flow rate of the capillary column and relatively large dead volume in the injector (see Figure 2-1).

This transfer mechanism results in the analytes arriving on the column over an extended period. Without any other effects, this would result in broad peaks for all the analytes.

In practice, the peaks are sharpened by the chromatography system. An initially cool oven serves as a cold trap, collecting the analytes at the column entrance. A temperature ramp then evaporates the analytes and brings them through the column as sharp peaks.

The technique is well suited to trace (10 fg to 1 ng per microliter) analyses because most of the sample is passed to the column. To focus volatile compounds, a cryogenically cooled GC oven, or a column phase with high retention, is necessary.

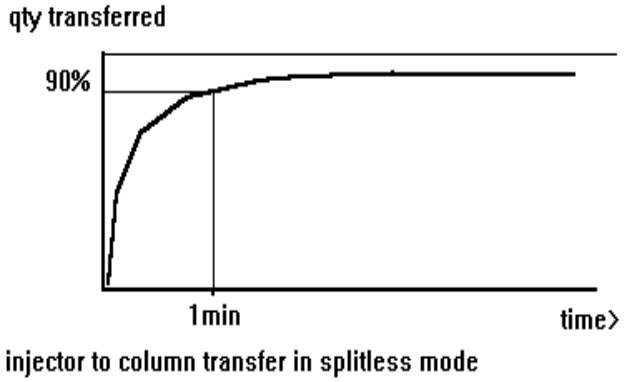


Figure 2-1. Amount of sample transferred to column against time in a splitless injection

The Solvent Effect

The solvent effect is a phenomenon useful for focusing analytes that are too volatile for focusing by the column phase and oven temperature. It relies on the analyte being slightly less volatile than the solvent.

If the oven temperature is about 20 °C lower than the boiling point of the solvent, the re-condensing solvent forms a liquid film inside the GC column. This film serves as a highly retentive stationary phase. Analytes remain in the evaporating liquid film until it is saturated. During the oven temperature ramp, the analytes vaporize in a sharp band.

The injection technique and timing can have an effect on peak shape. Short injections may result in a partial evaporation from the syringe needle and give the potential for a distillation effect. More volatile components may be transferred more completely than less volatile components.

You can remedy this effect (termed needle discrimination) in two ways:

- Leave the needle in the injector for a few seconds after depressing the plunger. This completes the evaporation process for all analytes.
- Fill the syringe with a solvent “plug” before taking up the sample. During the injection, the solvent plug washes the sample out of the syringe and needle.

Splitless Duration

In splitless mode, the carrier gas never completely sweeps the sample from the liner because of the low flow rate and the effects of dead volumes, mixing and diffusion in the injector body (see Figure 2-1). Any remaining solvent could give rise to a significant tailing signal as it is purged.

To avoid this effect, ensure that the split valve opens at a preset time after the injection. The split flow then sweeps any remaining solvent out of the injector. Choose the timing so that the injector-to-column transfer has proceeded almost to completion by the time the injector is purged (typically one minute).

Septum Purge

Air diffuses into the injector through holes made in the septum by the syringe needle. This can affect the operation of the MS detector and may harm the column phase if it is sensitive to oxygen.

The Septum Purge is an additional vent providing a flow of helium under the septum and out of the injector body. This flow simply removes any septum bleed vapors from the injector.

Liners

In a splitless injection, the sample spends a considerable amount of time in the injector. An inert liner positioned inside the body of the injector minimizes the breakdown of compounds sensitive to catalytic decomposition on metal inlet parts.

The choice of liner depends upon the application and the volume of solvent you want to inject. If you are analyzing particularly “dirty” samples, pack the liner with wool or fused silica beads to trap non-volatile residue and prevent column contamination. Liners may are also be deactivated using a high temperature silanization process.

There are two types of tapered splitless liners available from both CE Instruments and Restek (the Restek liners, as supplied with the TRACE MS, are pre-silanized and very inert):

- 3 mm ID (internal volume 0.60 cm^3)
- 5 mm ID (internal volume 1.67 cm^3)

Solvent Expansion Volumes

You must limit the injected sample volume so that its vapor is contained within the liner. Problems such as split or broad peaks, can occur if the solvent and sample vapor expands beyond the injector into gas lines and the column.

Table 2-2 shows the vapor volumes at 250 °C resulting from 1 µL injections of four common solvents. In particular, this shows the relatively large vapor volume of water. If you are using water as a solvent, make sure you check the volume of your injected sample.

Table 2-2. Vapor volumes (mL) from 1 µL injections at 250 °C

Solvent	Head Pressure [psig]		
	5	10	15
Hexane	0.245	0.196	0.163
Methanol	0.792	0.628	0.525
Methylene chloride	0.500	0.399	0.332
Water	1.776	1.418	1.179

Calculate expansion volumes using the following formula:

Solvent expansion volume = nRT/P

Where:

n = number of moles of solvent [volume (mL) × density (g/mL) / molecular weight (g/mole)]

T = injector temperature [K]

P = column head pressure (atm) + 1 atm

R = gas law constant = 82.06 cm^3

Note. When calculating expansion volumes, remember that the average 10 µL syringe contains approximately 0.8 µL of sample in the needle. This is important in both calculating the volume and in using the solvent flush injection technique.

On-column Injectors

With an on-column injector, the syringe needle is inserted about 3 cm inside the capillary column. An injection deposits the entire sample, as a liquid, directly onto the column.

You should adjust the temperature of the injector body and the section of column receiving the sample to about 20 °C below the boiling point of the solvent. This ensures that the solvent effect operates, and that the sample does not boil during the injection stage. If solvent boiling occurs, the sample sputters down the column, resulting in split peaks and/or broad peaks for all components.

On-column injection has similar capabilities to splitless injection. Both techniques:

- Are suited to trace analysis (because the entire sample reaches the chromatography system).
- Are applicable to samples with the same boiling range (because of the generally low oven temperatures).

In comparison with splitless injectors, on-column injectors have some advantages and disadvantages. The advantages are:

- No septum bleed.
- No needle discrimination or sample thermal decomposition because the injector is maintained at a low temperature.
- 100%, and reproducible, syringe-to-column transfer.
- Reduced consumables cost (septa, liners, ferrules).
- Suited to autosampler injection.

The potential disadvantages of on-column injection are:

- Flooding of the front of the column with liquid solvent during every injection. This flooding effect may split peaks, despite careful attention to flow rates, temperature and injection technique. It occurs because of the interaction between the column phase and the solvent.

  For this reason, a pre-column may be used. This is a section of deactivated fused silica, generally with a larger diameter (often 0.53 mm) than the analytical column. It serves as a removable trap or "guard column" for contaminants. The extra volume and lack of phase in the injector region also improves peak shape.

- Thermally labile contaminants, trapped by the liner in a split/splitless injector, are deposited directly onto the column. This increases the need for frequent column maintenance. To preserve peak shape, you may need to regularly remove the front end of the column. The pre-column can also be used to address this problem; removal of sections of it periodically will not affect retention times of target compounds.

2.3 Installing a Column

The following pages describe how to:

- Install capillary and wide bore columns.
- Connect a capillary column to a split/splitless injector.
- Connect a capillary column to an on-column injector.
- Connect the large volume injection system T-piece.
- Prepare a capillary column.
- Leak checking an installed capillary column.
- Connecting a column to the TRACE MS - GC interface.

Installing Capillary and Wide Bore Columns

The capillary and wide bore capillary columns should be positioned inside the oven on the column support with the column ends correctly aligned with the capillary injector and capillary detector base bodies. Wide bore capillary columns may also be installed into the packed and purged packed injector.

On-column injectors with autosamplers require a wide bore pre-column. Pre-columns help prevent contamination of the analytical column.

Using the Correct Fittings

To connect a capillary column to the injector and detector base body, proper column ferrules and retaining nuts are required.

Column Ferrules

Graphite and graphitized Vespel® ferrules are commonly used for column connections.

- Stainless steel encapsulated graphite ferrules connect the capillary column to the S/SL injector and to the detector base body. These can be re-used many times.
- Graphitized Vespel ferrules are used *only* to connect capillary columns to on-column injectors.

> **Caution.** Over-tightening compression ferrules does not necessarily produce a stronger, leak-free joint. Too much pressure can cause a leak in the joint, making it very difficult to reseal when changing columns.

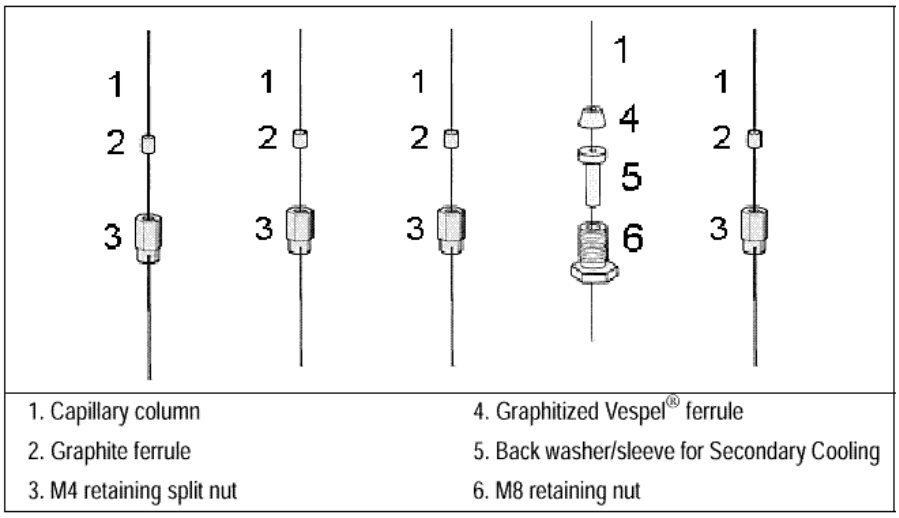
The table below lists the ferrules to use according to the pre-column and capillary column internal diameter. Ferrules that are the wrong size cause leaks and contamination.

Capillary Column	Graphite Ferrules	Graphitized Vespel Ferrules
0.2 mm ID	0.25 mm ID	0.25 mm ID
0.25 mm ID	0.35 mm ID	0.35 mm ID
0.32 mm ID	0.45 mm ID	0.45 mm ID
0.53 mm ID	0.8 mm ID	0.8 mm ID

Retaining Nuts

M4 retaining nuts are used to connect capillary columns to injector and detector base bodies. The nuts are split to allow easy installation and removal from the column. On-column injectors require a dedicated M8 retaining nut.

Figure 2-2 shows when to use graphite and graphitized Vespel ferrules and retaining nuts according to injectors and detector base bodies.



1. Capillary column
2. Graphite ferrule
3. M4 retaining split nut
4. Graphitized Vespel® ferrule
5. Back washer/sleeve for Secondary Cooling
6. M8 retaining nut

Figure 2-2. Injector and Detector base body connections

Press-fit Connections and Butt Connectors

Glass press-fit connectors are used to couple the fused silica pre-column to the capillary column. Y Press-Fit connections are used for multi-detector configurations.

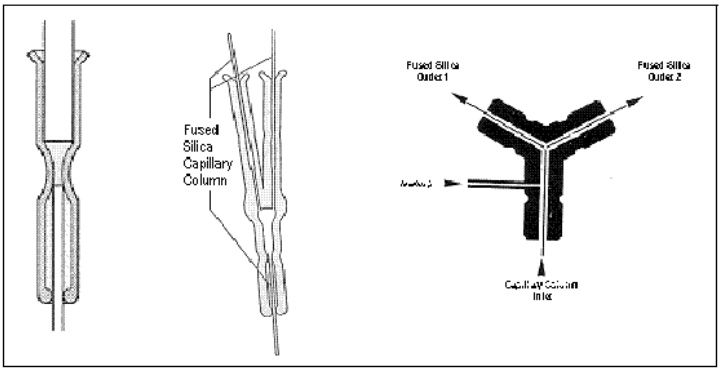


Figure 2-3. Press-fit connections

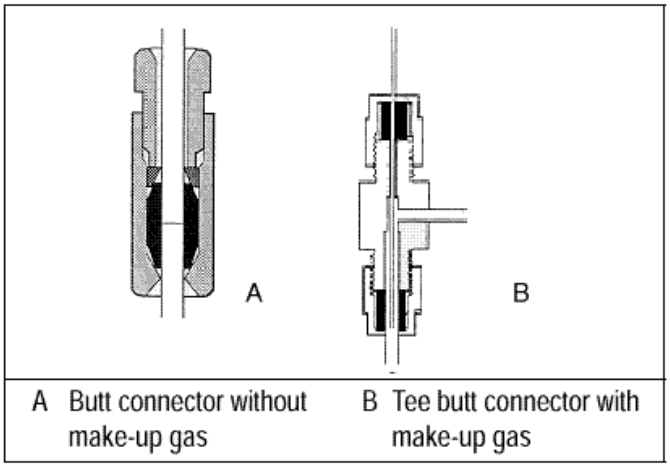


Figure 2-4. Butt connectors

Figure 2-4 part A shows a butt connector with a single Vespel or graphite ferrule used to connect a pre-column to an analytical column with the same inside diameter.

Figure 2-4 part B shows a butt connector with make-up lines used to connect a pre-column, normally wide bore, to an analytical column with a smaller diameter. The make-up line supplies a make-up gas to effectively flush the connection.

> **Note.** Press fit connectors can be used instead of butt connectors in all cases.

Connecting a Capillary Column to a Split/Splitless Injector

Before connecting the column, make sure the injector has been properly assembled and programmed to its operating temperature.

You will need the following materials:

- M4 column retaining nut
- Graphite ferrule with the correct ID depending on the external diameter of the column in use
- Typewriter correction fluid or a felt tip marker
- 6 mm wrench

To connect a capillary column:

1. Insert the graphite ferrule onto the capillary column with the open end facing the end of the column you will connect to the injector. Be careful to avoid damaging the graphite when inserting the column.
2. Cut at least 1 cm from the column end; see **Preparing a Capillary Column**, on page 2-16 for details.
3. Use typewriter correction fluid or a felt tip marker to mark the correct position of the ferrule from the end of the column depending on the injection technique. The correct positions are as follows:
   - 40 mm for split injection.
   - 64 mm for splitless injection.
4. Insert the column about 2 cm into the injector and slide the ferrule on the column up to the injector base, then slide the retaining nut onto the column through the side cut. The retaining nuts have a slotted design that makes them easy to add and remove.
5. Finger tighten the column retaining nut until it starts to grip the column.
6. Adjust the column position so that the correction fluid mark is level with the column retaining nut.
7. Use the 6 mm wrench to tighten the retaining nut with the minimum pressure required to obtain a good seal (¼ to ½ turn).
8. Conduct a leak check of the column installation, as described in the section **Leak Checking an Installed Capillary Column**, on page 2-16.

Connecting a Capillary Column to an On-column Injector

You will need the following materials:

- M8 column retaining nut.
- Backwasher / sleeve for Secondary Cooling.
- Graphitized Vespel ferrule with the correct ID depending on the external diameter of the column in use.
- 10 mm wrench.

Before you begin this procedure, insert the syringe needle into the injector. If you are using a pre-column, connect it to the capillary column using a press-fit or butt connector.

To connect the capillary column:

1. Insert the M8 retaining nut, the secondary cooling sleeve and the Vespel ferrule onto the capillary column (or pre-column, if used). Refer to Figure 2-2 for the correct assembly order.

> **Note.** If the HOT device is used, the integral M8 retaining nut and HOT device are used in place of the standard secondary cooling sleeve.

2. Slide the column onto the needle protruding through the injector into the column oven, then push the column into the injector as far as it will go.
3. Slide the ferrule and retaining nut/secondary cooling sleeve up the column and screw the nut onto the injector, tightening it with a 10 mm wrench until the column is secure. Use the minimum pressure needed to ensure a good seal.
4. Remove the syringe needle and reinsert it. It should slide easily into the column without friction. If not, repeat the column installation procedure.

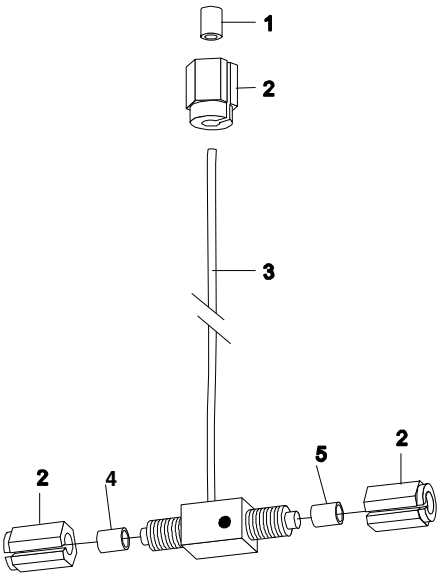
> **Note.** To check that the column positioning in the on-column injector has not blocked the carrier gas path, turn on the carrier gas line. You should hear carrier gas escaping through the syringe needle channel when the injection valve is opened.

5. Conduct a leak check of the column installation, as described in the section **Leak Checking an Installed Capillary Column** on page 2-16.

Connecting the Large Volume Injection System T-Piece

You will need the following materials:

- Uncoret™ 12 meter long, 0.53 mm ID uncoated pre-column as retention gap and 3 m long coated segment (SE-54; 0.45 µm film thickness).
- 0.32 or 0.25 mm ID fused silica capillary column.
- T-connector with M4 column retaining nuts and graphite ferrules, as shown in Figure 2-5.
- 7 mm wrench.
- 10 mm wrench.



1. 1.0 mm Graphite ferrule
2. M4 Retaining nut
3. SVE line metal tube
4. 0.5 mm Graphite ferrule for 0.32 mm ID column
5. 0.8 mm Graphite ferrule for 0.53 mm ID column

Figure 2-5. T-connection assembly

> **Note.** Before starting, insert the AS200 Autosampler syringe needle into the injector.

You should you connect the analytical column to the detector after a leak test of the Solvent Vapor Exit (SVE) system.

Connecting the Uncoret Pre-column

To connect the pre-column:

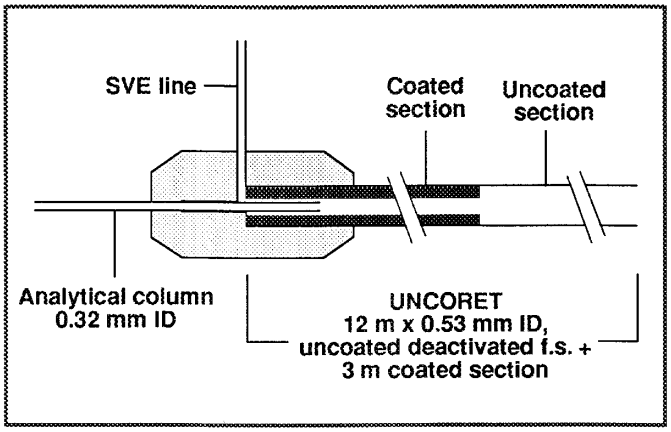
1. Install the Uncoret pre-column in the on-column injector as described in the section **Connecting a Capillary Column to an On-column Injector**, on page 2-11.
2. Insert the 0.8 mm graphite ferrule onto the pre-column with the open end facing the end of the pre-column you will connect to the T-connector. Be careful to avoid damaging the graphite when inserting the column.
3. Cut 1 cm from the pre-column end.
4. Insert the pre-column into the T-connector.
5. Slide the M4 retaining nut on the column through the side cut.
6. Tighten the column retaining nut until it starts to grip the pre-column.

Connecting the Analytical Column

To connect the analytical column:

1. Insert the 0.45 mm graphite ferrule onto the pre-column with the open end facing the end of the pre-column you will connect to the T-piece. Be careful to avoid damaging the graphite when inserting the column.
2. Cut 1 cm from the pre-column end.
3. Insert the analytical column end through the T-connector as shown in Figure 2-6.

4. Slide the M4 retaining nut onto the column through its side cut.
5. Finger tighten the column retaining nut until it starts to grip the column.



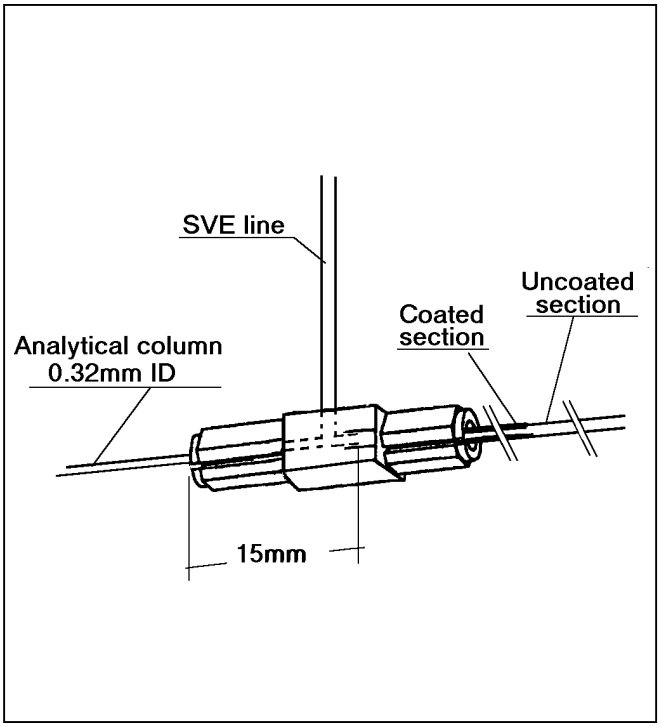


Figure 2-6. Uncoret pre-column to column connection

Connecting the SVE System

To connect the SVE system:

1. Slide the M4 retaining nut on the metal tube.
2. Insert the 1.0 mm graphite ferrule onto the SVE line metal tube with the open end facing the end of the pre-column you will connect to the SVE valve. Be careful to avoid damaging the graphite when inserting the column.
3. Insert the SVE line metal tube into the SVE system.
4. Finger tighten the retaining nut until it starts to grip the SVE system.
5. Use the 6 mm wrench to tighten all the M4 retaining nuts with the minimum pressure required to obtain a good seal (¼ to ½ turn).

The result of this operation is shown in Figure 2-7.

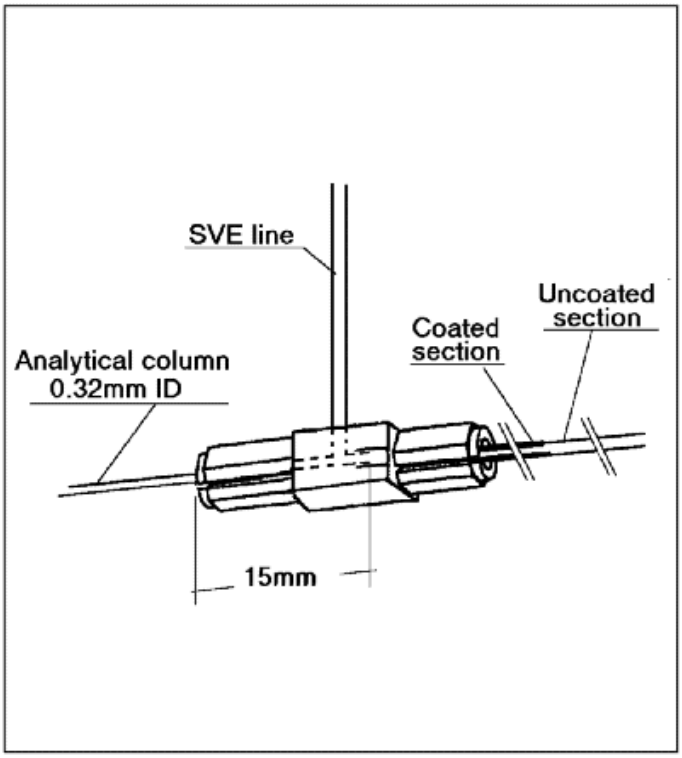


Figure 2-7. Large volume injection T-connection

6. Conduct a leak check of the column installation, as described in the section **Leak Checking an Installed Capillary Column**, on page 2-16.

Preparing a Capillary Column

To obtain a correct cut, use a ceramic scoring wafer (smooth edge) or sapphire scribe.

1. Hold the capillary column between your thumb and index finger, with the column extending past the tip of the index finger.
2. Score the column very gently. Do not apply excessive force that may crush the column end.
3. Snap off the end of the column.
4. Inspect the column end for an even, flat cut.

WARNING. Wear safety glasses to protect your eyes from flying particles while handling, cutting, or installing columns. Use care in handling these columns to avoid accidental injuries to your hands.

Leak Checking an Installed Capillary Column

Before starting this procedure, you must install the column into the injector but not into the detector base body.

You will need a silicone rubber septum of any dimension.

To leak check the installed column:

1. Carefully push the detector end of the capillary column into the rubber septum to seal it.
2. Close off any splitting or purge valves on the injector.
3. Increase the carrier gas pressure to 150–200 kPa and allow the column and injector pressure to stabilize. This can take up to 30 seconds.
4. Reduce the pressure to 50 kPa.
5. Observe the actual pressure. In a leak tight system, the pressure should not drop by more than 1 kPa/min.

If your installed column is leak tight, remove the septum and prepare the end of the column for installation into the detector.

If it is not leak tight, check the tightness of the column ferrule and repeat the leak check procedure.

Connecting a Column to the TRACE MS - GC Interface

To connect a new column to the TRACE MS - GC interface:

1. Right-click on the TRACE MS Server icon in the Windows task-bar. The server menu is displayed.
2. Choose the **Vacuum | Vent** menu option to vent the system.
3. Remove the source, as described in Chapter 3.

WARNING. Wait until the GC oven and the GC interface are cool before proceeding.

4. Inside the GC oven, remove the brass blanking ferrule from the end of the inner-tube in the GC interface.
5. Place a 5/16” stainless steel SwageLok® nut over the column, followed by a graphitized Vespel ferrule, then cut the column using the previously described method, see **Preparing a Capillary Column** on page 2-16.
6. Carefully slide the column into the inner-tube, until it protrudes through the GC interface into the TRACE MS’s vacuum housing.
7. Draw the column back through the interface, until only 1-3 mm remains visible (see Figure 2-8).

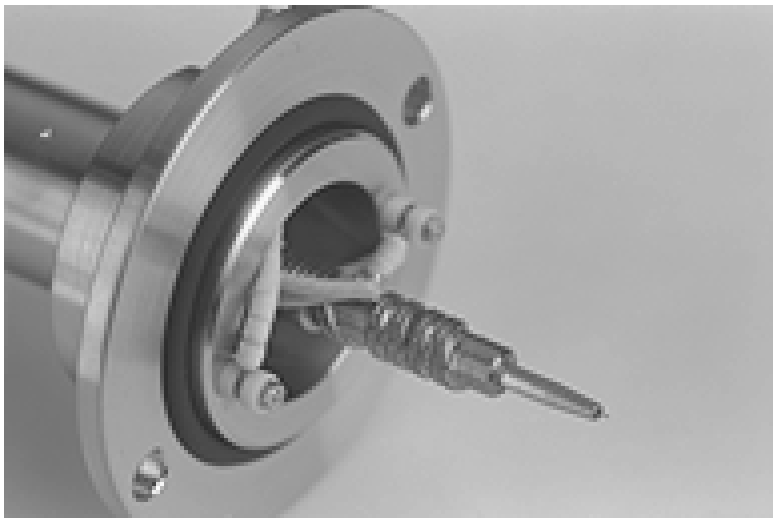


Figure 2-8. GC interface end-plate with 1-3 mm of column protruding

8. Re-install the source.

9. Tighten the nut on the GC side of the interface that holds the column in the glass-lined tube.
10. Right-click on the TRACE MS Server icon in the Windows task-bar and choose the **Vacuum | Pump** menu option to pump-down the system.

2.4 Maintaining a GC Column

Column bleed is the major source of contamination in the MS detector. A number of ions are characteristic of siloxane degradation products (m/z 207, 281, 355). These ions often dominate the base spectrum information of late eluting peaks at high GC temperatures. The appearance of degradation products at low temperatures indicates the need for column maintenance. In extreme cases, you may need to replace the column.

Other common sources of contamination produce ions at m/z 147 (often due to septum breakdown) and m/z 149 (due to phthalates).

To recondition an old or poorly operating GC column:

1. Right-click on the TRACE MS Server icon in the Windows task-bar. The server menu is displayed.
2. Choose the **Vacuum | Vent** menu option to vent the system.
3. Remove the column from the GC interface and hang it on the column hanger.

WARNING. If the TRACE MS has been used recently, the GC interface will be very hot. Wait until it cools before removing the column. Proceed with caution.

4. Blank off the interface with the 5/16” brass blanking nut, supplied in the TRACE MS toolkit.
5. Slowly bring the GC up to the maximum operating temperature of the column and leave it there overnight. See the documentation supplied by the column manufacturer for details.
6. Re-cut the column prior to re-installation in the GC interface.
7. Re-insert the column into the GC interface.
8. Right-click on the TRACE MS Server icon in the Windows task-bar and choose the **Vacuum | Pump** menu option to pump-down the system.

Chapter 3
Changing MS Ionization Modes

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Advantages of CI 3-1

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3.3 Installing a New Source 3-6

3.1 Choosing an Ionization Mode

The TRACE MS comes complete with an EI source as standard. Other ion sources (CI+, CI- and combined) are available as optional extras.

It is not possible to say precisely which mode will be the best for every occasion. Remember that the configuration and choice of the reagent gas can significantly affect sensitivity and performance in CI modes.

> **Note.** CI is not available to TRACE MS systems fitted with the 70L/s turbomolecular pumping configuration.

Identification of Unknown Compounds

In general, you should use EI to identify an unknown compound.

The Library Browser contains the NIST/EPA/NIH Mass Spectral Library with over 108,000 reference EI spectra. There are no commercial library databases of CI spectra, although you can create your own CI spectrum library for use within Xcalibur. See **Xcalibur Getting Productive: Qualitative Analysis** for details.

You may find it useful to apply CI modes as well, particularly in cases where fragmentation is excessive and you want confirmation of the molecular weight. Adduct formation occurs in CI+ mode and this must be taken into account in any molecular weight assessment. You could use a CI confirmed molecular weight to constrain a library search and increase the confidence of a library match.

Advantages of CI

In trace analysis, CI modes may be more appropriate because of the:

- Lower fragmentation.
- Significantly higher sensitivity (orders of magnitude in CI-) for certain classes of compounds (containing double bonds, sulfur, phosphorus, chlorine or bromine).
- Greater discrimination, especially for CI-.

3.2 Removing the Current Source

The following pages describe the procedures for removing the current source from the TRACE MS. The procedures vary with the type of source.

EI Source

To remove an EI source:

1. Loosen the locking screws on the source faceplate by a 1/4 turn.

> **Caution.** Do not fully loosen the locking screws at this stage as this could cause damage to the source and /or to the GC interface.

2. Right-click on the TRACE MS Server icon in the Windows task-bar.

   The server menu is displayed.

3. Choose the **Vacuum | Vent** menu option to vent the system.
4. Withdraw the GC interface.

   Ensure the GC interface is withdrawn by at least 2 cm before attempting to remove the source, to prevent damage to either component.

> **WARNING.** If the TRACE MS has been used recently, the GC interface will be very hot. Wait until it cools before removing it. Proceed with caution.

5. To remove the source:
   a. Disconnect the cable connector by gripping firmly and pulling, and then loosen the two locking screws holding it in place (see Figure 3-1).
   b. Remove the source carefully, taking care not to damage the GC interface.

> **WARNING.** If the TRACE MS has been used recently, the source may be hot. Use caution when handling it.

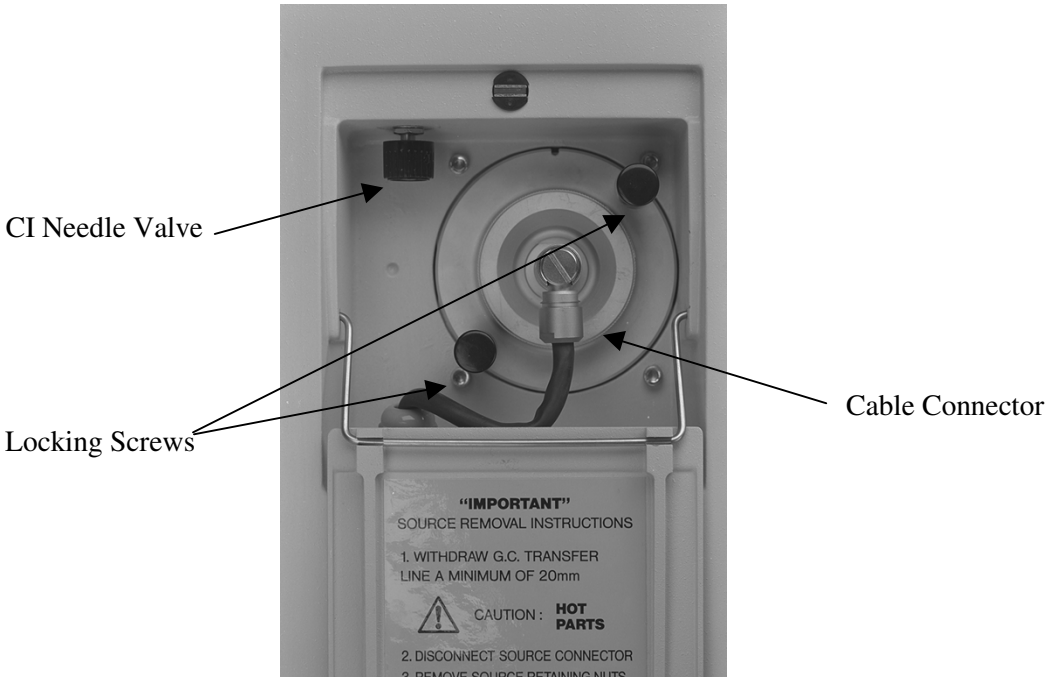

Figure 3-1. Source faceplate

Other Source Types

In changing from CI to EI, you must clear the reagent gas out of the reagent gas line before removing the source. This is done to ensure that no gas escapes when removing the source. This is especially important with harmful gases such as ammonia.

To remove a CI+, CI- or a combined source, follow one of the procedures detailed below, depending on whether your TRACE MS is fitted with digital CI flow control (Xcalibur software) or manual valve CI flow control (CI needle valve):

Digital CI Flow Control

1. Close off the reagent gas cylinder regulator.
2. Double-click on the TRACE MS Tune desktop short-cut to open the Xcalibur Tune Window.

3. Choose **Instrument | Operate** or click on the Toggle Operate toolbar button to switch the instrument into Operate mode.
4. In the TRACE MS Controls bar, set the CI reagent gas flow to about 5 ml/min using the CI (ml/min) text box at the bottom-right of the Controls bar (see Figure 3-2).

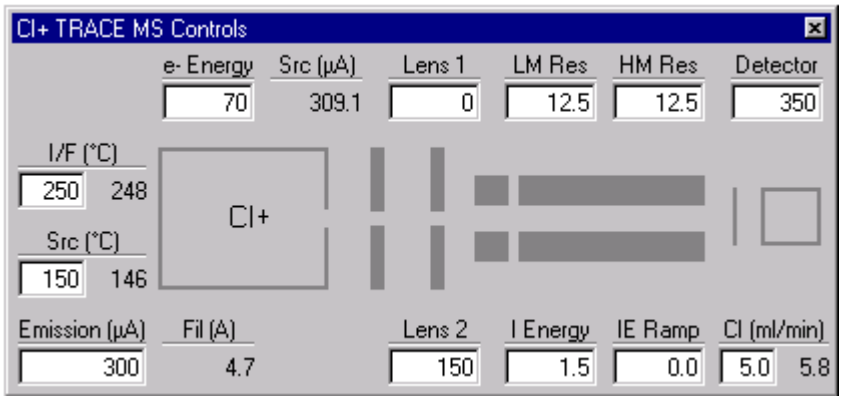

Figure 3-2. TRACE MS Controls bar with CI flow set at 5 ml/min

5. Wait for two or three minutes until all the gas has been removed from the reagent gas line.

6. Choose **Instrument | Operate** or click on the Toggle Operate toolbar button to switch the instrument out of Operate mode.
7. Right-click on the TRACE MS Server icon in the Windows task-bar to display the Server menu.
8. Choose the **Vacuum | Vent** menu option to vent the system.
9. Withdraw the GC interface.

   Ensure the GC interface is withdrawn by at least 2 cm before attempting to remove the source to prevent damage to either component.

WARNING. If the TRACE MS has been used recently, the GC interface will be very hot. Wait until it cools before removing it. Proceed with caution.

10. To remove the source:
    a. Disconnect the cable connector by gripping firmly and pulling, and then loosen the two locking screws holding it in place (see Figure 3-1).
    b. Remove the source carefully, taking care not to damage the GC interface.

WARNING. If the TRACE MS has been used recently, the source may be hot. Use caution when handling it.

Manual CI Needle Valve Flow Control

1. Evacuate the reagent gas line:
   a. Close off the reagent gas cylinder regulator.
   b. Slowly open the CI needle valve in the source flap, stopping whenever the TRACE MS's orange pump light comes on. See Figure 3-1, which shows the position of the valve.

      When the valve is fully open, and all the reagent gas has been evacuated, the pump light changes to steady green.

> **Caution.** The CI needle valve is a precision valve with a hard steel-to-steel closure. Never close it tighter than gentle finger-tightness, otherwise you may cause irreparable damage.

2. Right-click on the TRACE MS Server icon in the Windows task-bar. The server menu is displayed.
3. Choose the **Vacuum | Vent** menu option to vent the system.
4. Withdraw the GC interface.

   Ensure the GC interface is withdrawn by at least 2 cm before attempting to remove the source to prevent damage to either component.

> **WARNING.** If the TRACE MS has been used recently, the GC interface will be very hot. Wait until it cools before removing it. Proceed with caution.

5. To remove the source:
   a. Disconnect the cable connector by gripping firmly and pulling, and then loosen the two locking screws holding it in place (see Figure 3-1).
   b. Remove the source carefully, taking care not to damage the GC interface.

> **WARNING.** If the TRACE MS has been used recently, the source may be hot. Use caution when handling it.

3.3 Installing a New Source

The procedure for installing a new source is the same for all source types.

1. Ensure that all the plumbing needed for the new source is already installed; that is, a reagent gas line for CI and combined sources is in place.

   When the source is correctly inserted, the reference and reagent lines automatically make a sealed connection from the TRACE MS to the source.

2. Ensure that the source is aligned correctly – **with the notch in the baseplate at the top of the source** (see Figure 3-3).

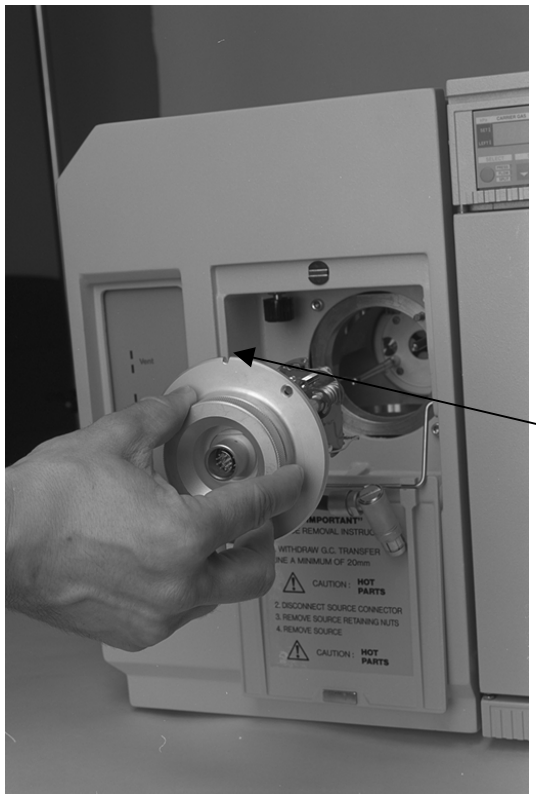

Figure 3-3. Correct alignment of source

3. Introduce the source into the vacuum chamber so that the two guide poles slide into the source arms (see Figure 3-4).

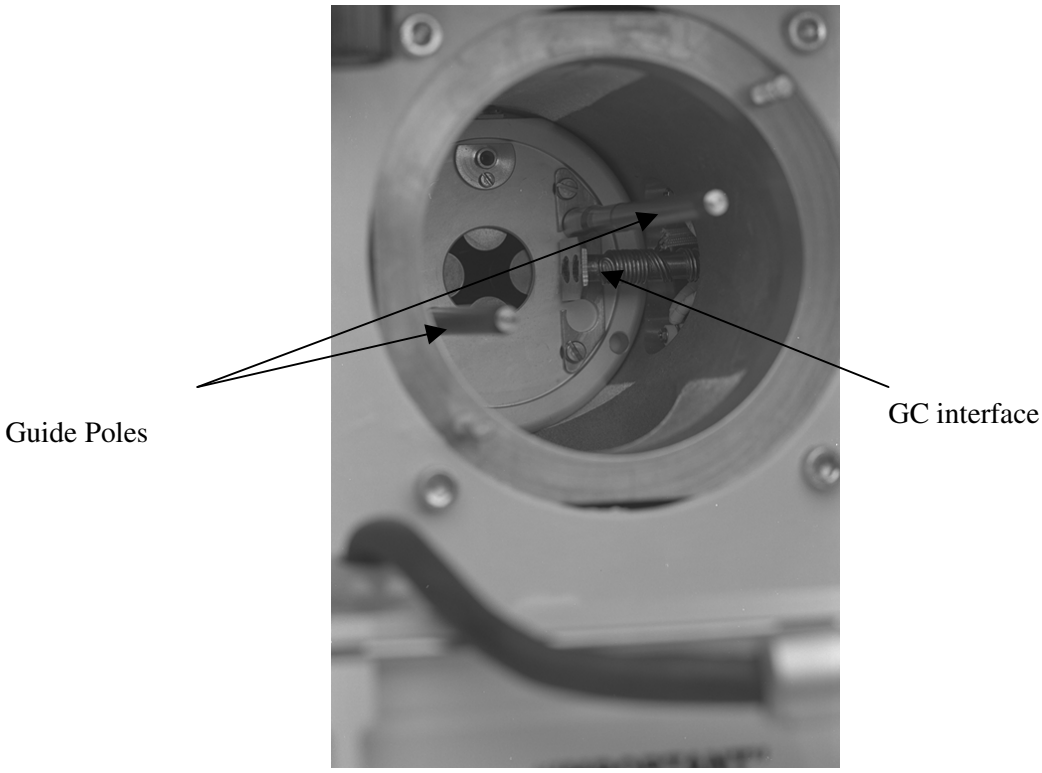

Figure 3-4. Vacuum chamber - showing guide poles and GC interface inner tube withdrawn

> **Caution.** Before inserting the source, ensure that the GC interface inner tube is withdrawn by at least 2 cm from its set position.

4. Gently move the source fully into the vacuum chamber.

   If the source touches the GC interface, stop and retract the GC interface a little further.
5. Tighten the two locking screws on the baseplate of the source.
6. Connect the cable connector.
7. Replace the GC interface inner tube.
8. Right-click on the TRACE MS Server icon in the Windows task-bar.
9. Choose the **Source Type** menu option and then select the option that reflects the new source type you have just installed.
10. Choose the **Vacuum | Pump** option to pump-down the system.

Chapter 4
Maintaining the System

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4.1 Introduction

This chapter provides details of the maintenance tasks you must carry out to keep the TRACE MS system in its optimum condition. These include:

- Scheduled maintenance tasks that should be performed on a regular basis.
- Ad-hoc maintenance tasks that should be performed as and when necessary. These are related to the cleaning of the ion source, the frequency of which depends upon the nature of the samples you work with.

4.2 Routine Maintenance Tasks

The table below identifies the major routine maintenance tasks and indicates how often they should be carried out.

Frequency	Maintenance Task
Weekly	Check and, if necessary, top-up the rotary pump oil level.
Quarterly	Clean the rear panel fan filter.
	Refill the Reference vial.
	Clean or replace the PC fan filter. See your PC hardware manual for details.
Half-yearly	Drain and replace the rotary pump oil.
	Replace the foreline trap pellets.

Checking the Rotary Pump Oil Level

> **Note.** For more information on operating the rotary pump, refer to the **Rotary Pump User Manual**, supplied with the TRACE MS system.

You can carry out this procedure while the instrument is operating.

Follow the steps described below. No specialist equipment or materials are required.

1. Look through the viewing window in the end of the rotary pump (see Figure 4-1).

   The oil level should be between the upper and lower marks next to the window.

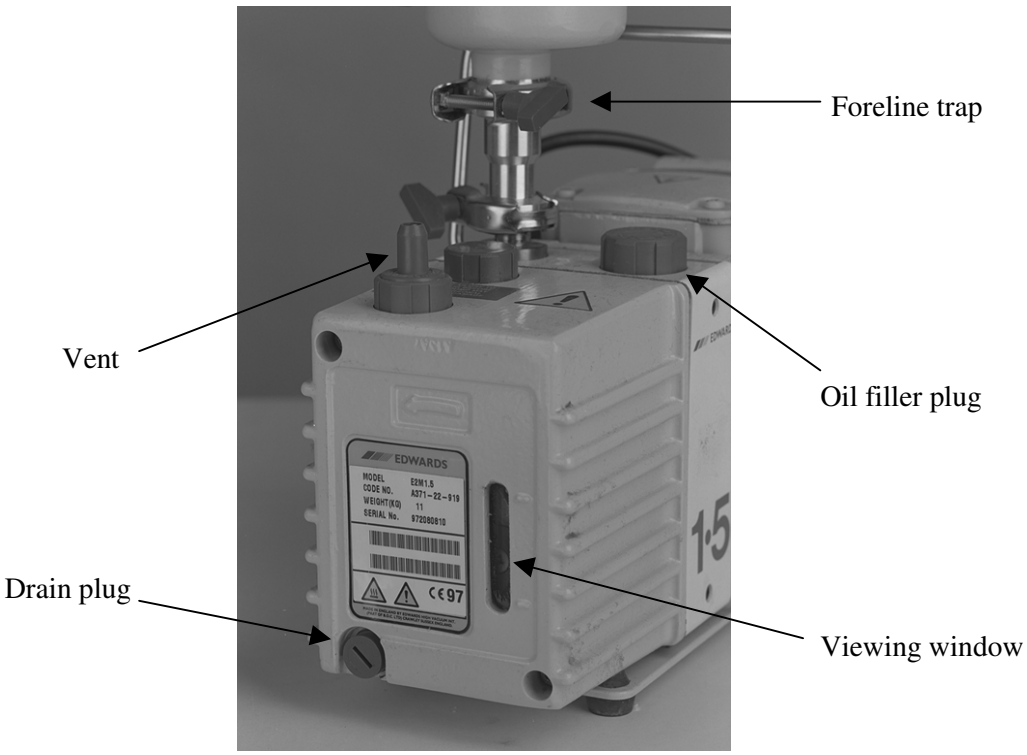

Figure 4-1. Rotary pump

2. If the oil level is near or below the lower mark, add more oil, as described below. However, if a half-yearly service is due, it may be more convenient to drain and replace the oil; see **Changing the Rotary Pump Oil** on page 4-8 for details.

Topping-up the Oil Reservoir

If the oil level in the rotary pump gets low before the scheduled oil change, add more oil to the reservoir.

To top-up the oil reservoir, you will require E2 M1.5 Rotary Pump Oil (P/N 5560018).

Follow the procedure described below.

1. Right-click on the TRACE MS Server icon in the Windows task-bar.

   The server menu is displayed.

2. Choose the **Vacuum | Vent** menu option to vent the system.

WARNING. Do not add oil while the pump is running, This could result in splashing of hot oil.

3. Remove the oil filler plug (see Figure 4-1).
4. Pour oil into the pump until the oil level in the viewing window is close to, but not above the upper mark.
5. Replace the oil filler plug.
6. Right-click on the TRACE MS Server icon in the Windows task-bar and choose the **Vacuum | Pump** option to pump-down the TRACE MS.

Cleaning the Rear Panel Fan Filter

The rear panel fan filter removes dust from the air used to cool the TRACE MS's electronics. The filter consists of a gauze, held in place by a plastic cover. It is the gauze that acts as the filter, trapping dust that would otherwise be drawn into the electronics.

To clean the filter, follow the steps described below. No specialist equipment or materials are required.

1. Remove the plastic cover from the back of the TRACE MS.

   The cover is held in place by friction, and should come free easily with a gentle pull (see Figure 4-2).

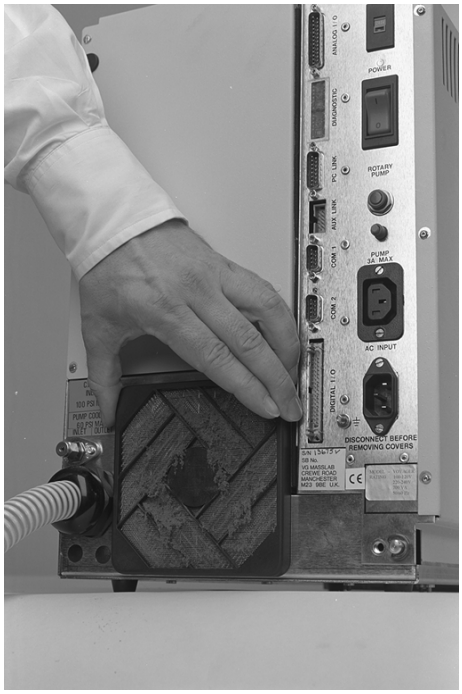

Figure 4-2. Removing the rear fan filter

2. Take the filter out of the room where the TRACE MS is situated.
3. Clean the gauze by removing it and tapping it against a hard surface.

   The filter is quite robust and will not be damaged.
4. Remove any persistent dust with a vacuum cleaner.
5. Replace the gauze and cover.

Refilling the Reference Vial

The reference vial contains a volatile compound (typically PFTBA), that is used during the tuning and calibration of the instrument in EI mode.

The instrument was tested before being shipped from the factory, and the reference vial filled with approximately 400 µL PFTBA. Under normal circumstances, this should last approximately 6 months.

To refill the reference vial, you will require approximately 400µl of reference compound.

Follow the procedure described below.

1. Right-click on the TRACE MS Server icon in the Windows task-bar. The server menu is displayed.
2. Choose the **Vacuum | Vent** menu option to vent the system.
3. Close down Xcalibur.
4. Switch off the TRACE MS, by means of its circuit breaker, and allow at least half an hour for the system to cool.
5. Undo the transit bolt at the back of the digital electronics unit and lower the unit carefully until it rests on the bench (see Figure 4-3).

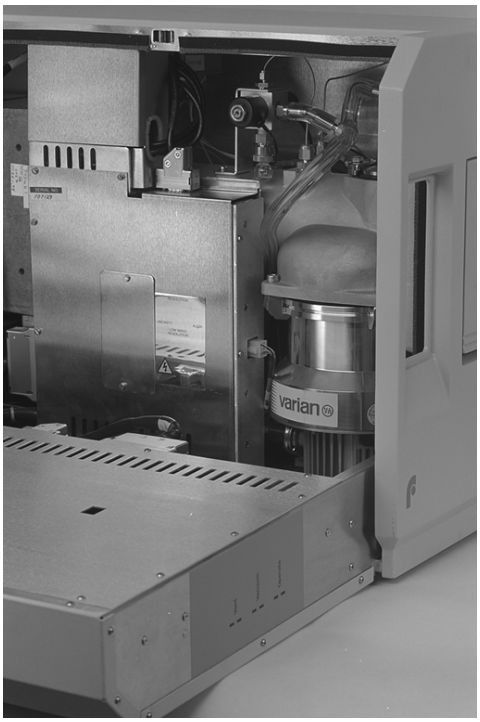

Figure 4-3. Opening the TRACE MS

WARNING. Risk of burns. Solenoid gas valves reach high temperatures in operation.

6. Unscrew the knurled nut (Cajon union) attached to the tube of the reference vial (see Figure 4-4).

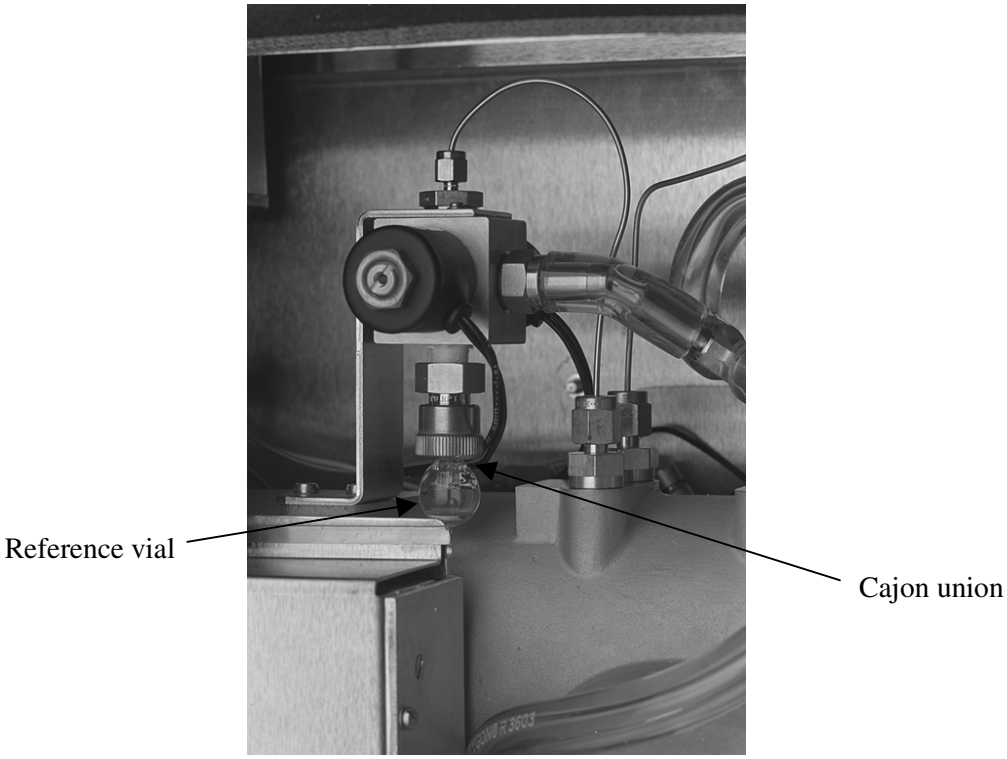

Figure 4-4. The TRACE MS's internal view

7. Remove the vial from the instrument.
8. Top up the vial, using approximately 400 µl (but no more than 0.5 ml) of reference compound.

WARNING. PTFBA, a tertiary amine, is a corrosive compound. Do not let it come into contact with either your skin or the instrument.

9. Replace the vial in the instrument and refit it to the union.
10. Tighten the knurled nut to finger tightness.
11. Close the instrument and replace the transit bolt at the back of the digital electronics unit.
12. Switch on the TRACE MS's power and restart Xcalibur.

13. Right-click on the TRACE MS Server icon in the Windows task-bar.

    The server menu is displayed.

14. Choose the **Vacuum | Pump** option to pump-down the TRACE MS.
15. Click on the TRACE MS Tune desktop short-cut.

    The Tune page is displayed.

16. Choose the **Instrument | Pump Gas** option to remove any air.
17. After about 5 seconds, choose the **Instrument | Pump Gas** option a second time to stop the pumping.

> **Caution.** Do NOT leave the **Pump Gas** option switched on.

Test the operation of the reference inlet by observing the Tune page as you switch the reference gas on and then off again. The peaks should rise normally as the gas is switched on and then fade away when it is switched off.

Changing the Rotary Pump Oil

> **Note.** For more information on operating the rotary pump, refer to the **Rotary Pump User Manual**, supplied with the TRACE MS system.

The rotary pump oil should be replaced at least once every six months. At the same time as you replace the oil, you should also consider replacing the foreline trap pellets; see **Replacing the Foreline Trap Pellets** on page 4-9.

> **WARNING.** Always wear gloves when changing the oil. Avoid contact with the oil because it may contain dissolved residue from analyzed samples. Observe appropriate disposal requirements when discarding the old oil.

To replace the rotary pump oil, you will require E2 M1.5 Rotary Pump Oil (P/N 5560018).

Follow the procedure described below.

1. Right-click on the TRACE MS Server icon in the Windows task-bar.

   The server menu is displayed.

2. Choose the **Vacuum | Vent** menu option to vent the system.
3. Place a container under the drain plug on the rotary pump.
4. Remove the filler plug from the top of the pump (see Figure 4-1).
5. Remove the drain plug to release the oil.
6. Turn on the pump for 2 or 3 seconds using the TRACE MS Server **Vacuum | Pump** menu option.

   The remains of the old oil are displaced from the internal pump cavity.

> **Caution**. Do not allow the pump to run for more than a few seconds without oil.

7. Reinsert the drain plug and fill the pump reservoir up to, but not above, the upper mark.
8. Reinsert the filler plug.
9. Gas ballast the new oil in the pump; for details, refer to the **Rotary Pump User Manual**.
10. Right-click on the TRACE MS Server icon in the Windows task-bar and choose the **Vacuum | Pump** menu option to pump-down the system.

Replacing the Foreline Trap Pellets

> **Note.** For more information on operating the rotary pump, refer to the **Rotary Pump User Manual**, supplied with the TRACE MS system.

Absorbent pellets in the foreline trap help maintain the TRACE MS's high vacuum.

The rotary pump oil should be replaced at least once every six months. At the same time as you replace the oil, you should also consider replacing the foreline trap pellets.

WARNING. Always wear gloves when handling the pellets. Avoid contact with the pellets because they may contain dissolved residue from analyzed samples. Observe appropriate disposal requirements when discarding the old pellets.

To replace the foreline trap pellets, you will require Absorbent Pellets.

Follow the procedure described below.

1. Loosen the foreline trap end cap by a 1/4 turn.
2. Right-click on the TRACE MS Server icon in the Windows task-bar.

   The server menu is displayed.
3. Choose the **Vacuum | Vent** menu option to vent the system.
4. Remove the end cap from the foreline trap to gain access to the inner baskets of pellets (see Figure 4-1).
5. Open a new can of pellets. Bake them in the GC oven for 2 hours at 250 ºC. Allow the pellets to cool and then place them in the foreline trap basket.
6. Reassemble the foreline trap.
7. Right-click on the TRACE MS Server icon in the Windows task-bar and choose the **Vacuum | Pump** menu option to pump-down the system.

4.3 Maintaining the Source

The following pages describe how to:

- Identify different source types
- Clean an ion source
- Replace any worn or damaged components

Identifying Sources

This section describes how to distinguish the various ion sources that can be used with the TRACE MS.

EI Source

The TRACE MS EI source is shown in Figure 4-5.

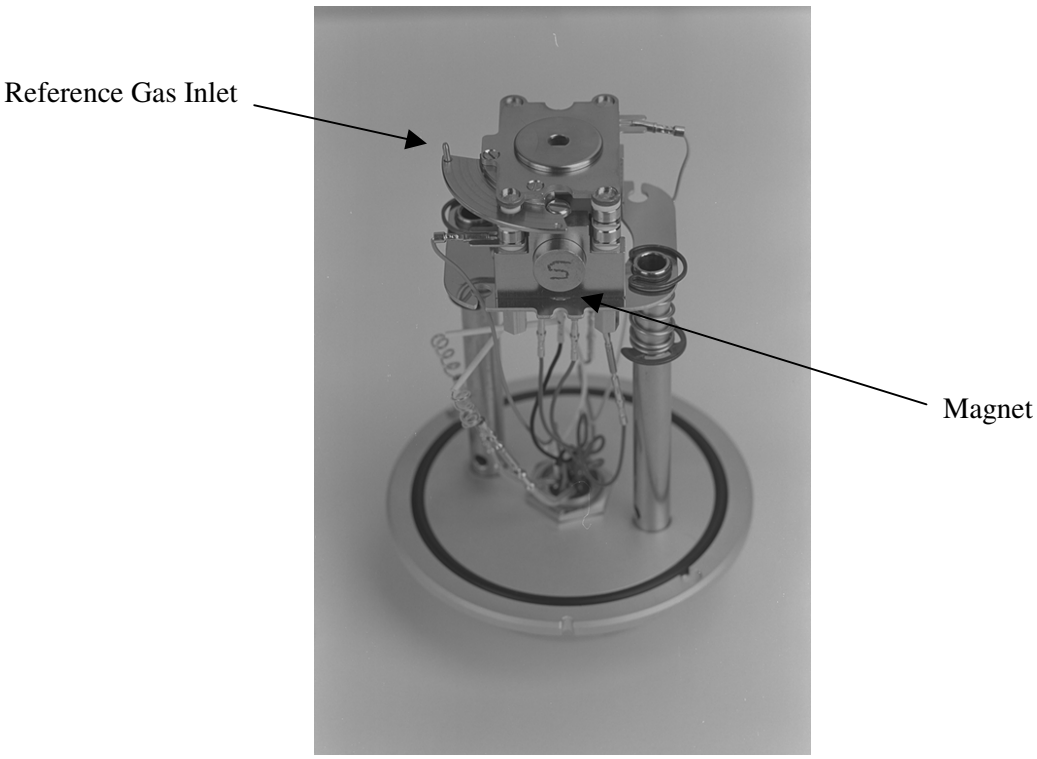

Figure 4-5. The TRACE MS's EI source

The TRACE MS EI source can easily be identified by the absence of a reagent gas inlet line, which is present on CI and combined sources.

The size of the hole in the exit plate is also indicative; it is much larger (5 mm) than the hole in the exit plate of other sources.

The EI source has ten wires running from baseplate to source body:

- Repeller – 1 black wire (female).
- Ion lenses – 1 yellow and 1 green wire (female).
- Source heater – 2 red (male).
- Filament – 1 red and 1 blue (female).
- Trap – 1 white (female).
- Thermocouple – 2 gray (ceramic).

CI+ Source

The TRACE MS CI+ source is shown in Figure 4-6.

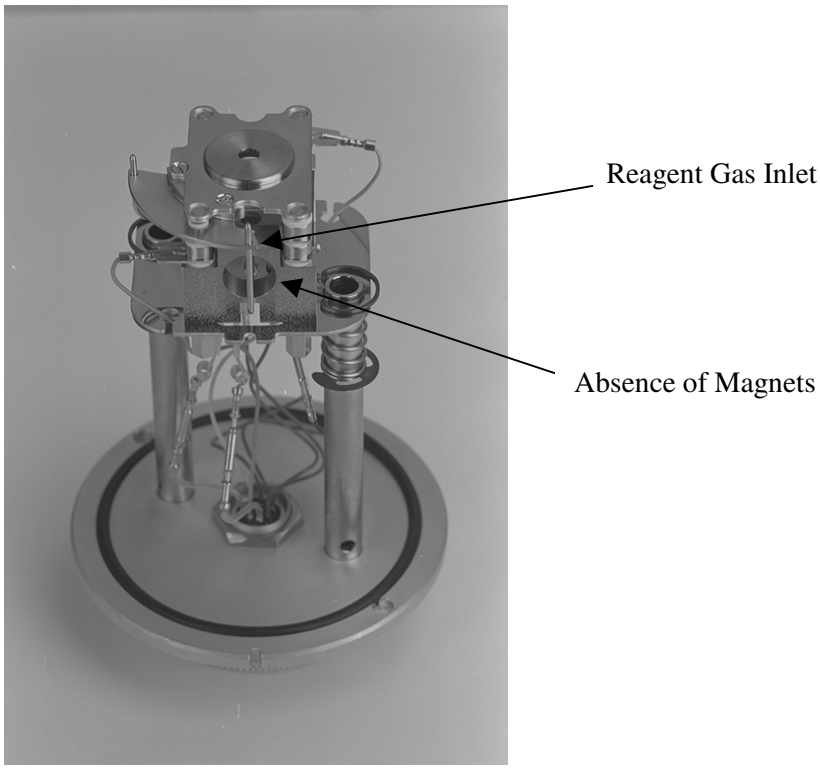

Figure 4-6. The TRACE MS's CI+ source

The CI+ source, in common with the CI- and combined source but in contrast to an EI source, has a reagent gas inlet.

The hole in the exit plate is very small (0.5 mm).

Unlike a CI- source, a CI+ source does not have a repeller, a trap or magnets. The most obvious difference is the absence of magnets. There are also fewer wires on a CI+ source, because of the absence of certain parts.

A CI+ source has eight wires:

- Ion lenses – 1 yellow and 1 green wire (female).
- Source heater – 2 red (male)
- Filament – 1 red and 1 blue (female).
- Thermocouple – 2 gray (ceramic).

CI- Source

The TRACE MS CI- source is shown in Figure 4-7.

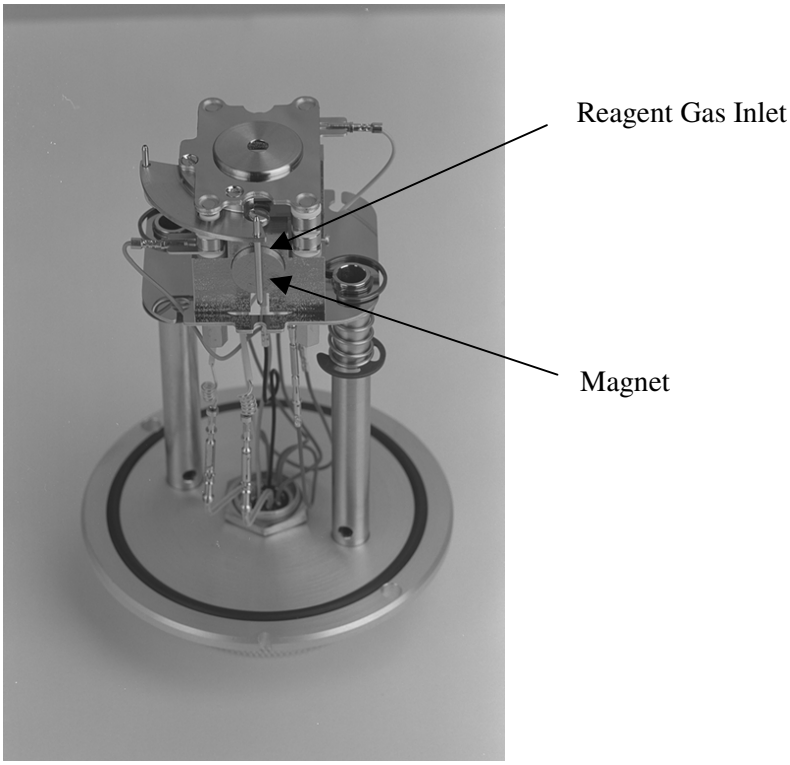

Figure 4-7. The TRACE MS's CI- source

The CI- source has a reagent gas line, magnets and a repeller but does not have a trap. The easiest way to see this is to look at the wires entering the source body.

The hole in the exit plate is very small (1 mm).

A CI- source has nine wires:

- Repeller – 1 black wire (female).
- Ion lenses – 1 yellow and 1 green wire (female).
- Source heater – 2 red (male).
- Filament – 1 red and 1 blue (female).
- Thermocouple – 2 gray (ceramic).

Combined Source

A combined source must acquire in both EI and CI modes, and must have all the features necessary to enable it to operate in both modes. Consequently, a combined source must have a reagent gas inlet, magnets, a repeller and a trap (see Figure 4-8).

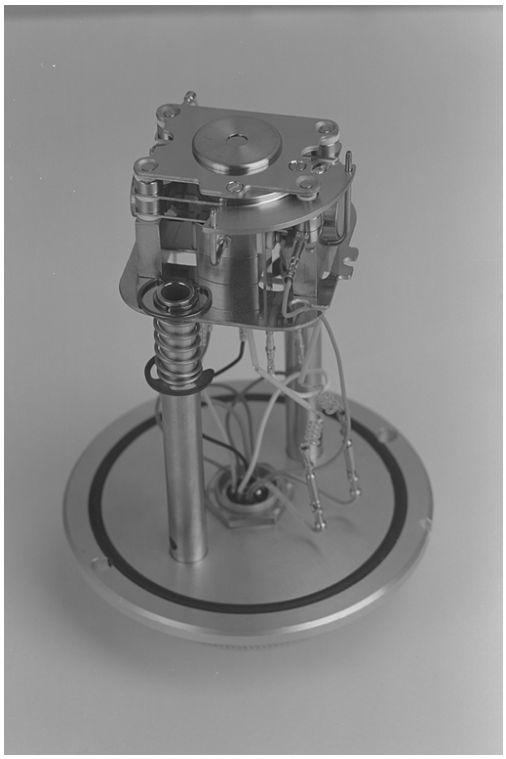

Figure 4-8. The TRACE MS's combined EI/CI source

The hole in the exit plate is very small (1 mm).

To distinguish this source type, first check the source has magnets and a reagent gas inlet. If the source has both of these, count the wires connecting baseplate and source body; a combined source has 10 wires:

- Repeller – 1 black wire (female).
- Ion lenses – 1 yellow and 1 green wire (female).
- Source heater – 2 red (male).
- Filament – 1 red and 1 blue (female).
- Trap – 1 white (female).
- Thermocouple – 2 gray (ceramic).

Dismantling the Source for Cleaning

This section describes how to dismantle the source for cleaning. Some of the steps are also required when you replace source components; see page 4-24 for details.

Follow the steps described below.

> **Caution.** To guard against unwanted contamination from finger grease, you are recommended to wear clean, lint-free gloves during all procedures involving handling of the source.

1. Follow the procedure described in Chapter 3 to remove the source from the instrument.
2. Remove the wires connecting the source by disconnecting the pushpins.
3. Remove the source body from the rest of the assembly by removing the push-on circlips and lifting the body off the base (see Figure 4-9).

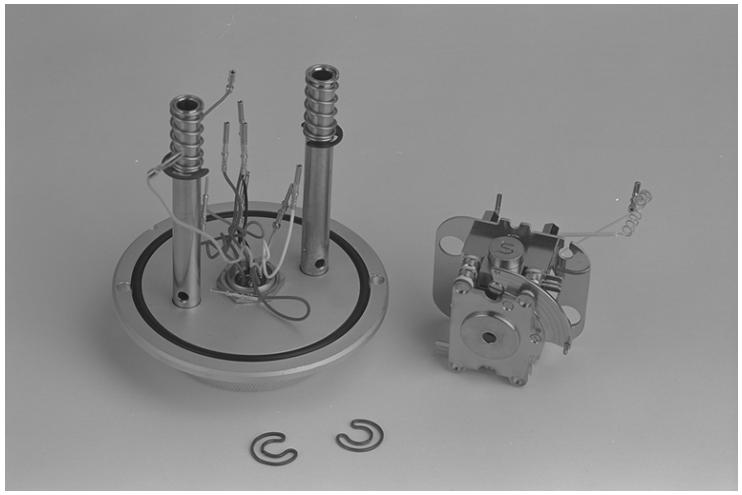

Figure 4-9. Source body removed from the rest of the assembly

4. Remove the four hex nuts and remove the mounting plate (see Figure 4-10).

   In CI and combined sources, the source block comes away with the mounting plate (see Figure 4-11).

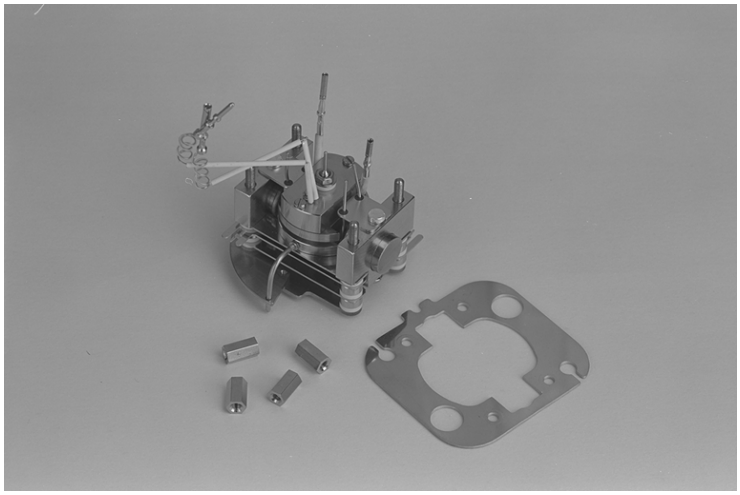

Figure 4-10. Mounting plate removed from body (EI source)

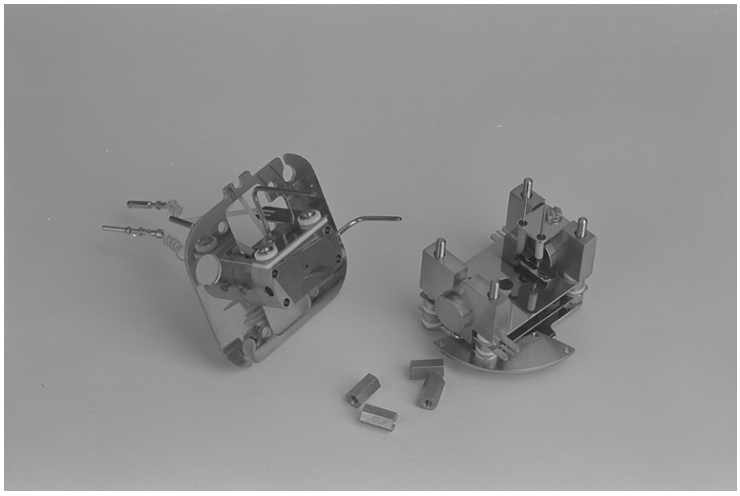

Figure 4-11. Mounting plate removed from body (CI and combined sources)

5. Slide the magnet assemblies off, noting which is top and bottom (see Figure 4-12). The filament is always adjacent to the North Pole.

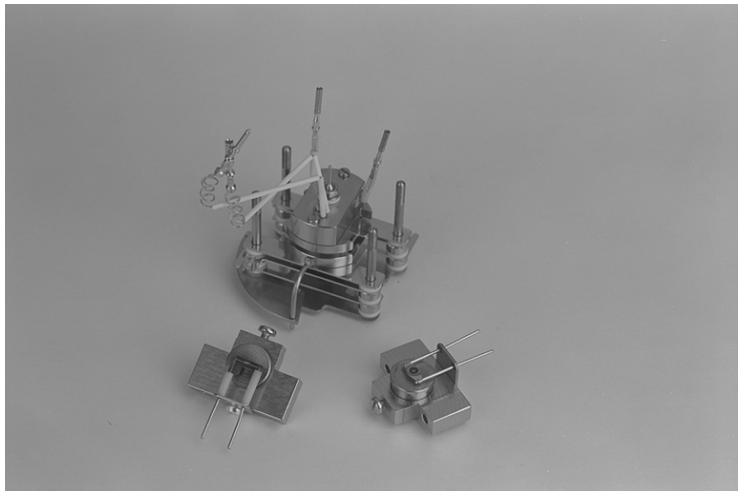

Figure 4-12. Magnet assemblies removed

6. Remove the filament and the trap by removing the fixing screws (see Figure 4-13).

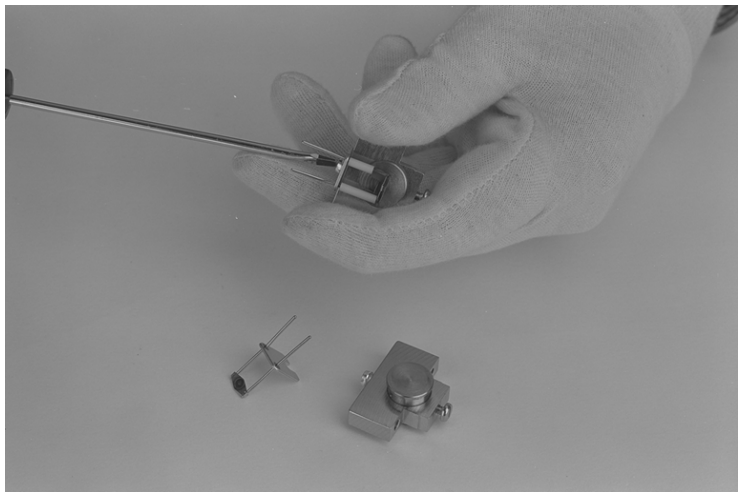

Figure 4-13. Removal of filament and trap

> **Caution.** Handle the filament with particular care and store it safely.

7. For EI sources, remove the source block on the ion exit plate and then remove the exit plate from the block (see Figure 4-14).

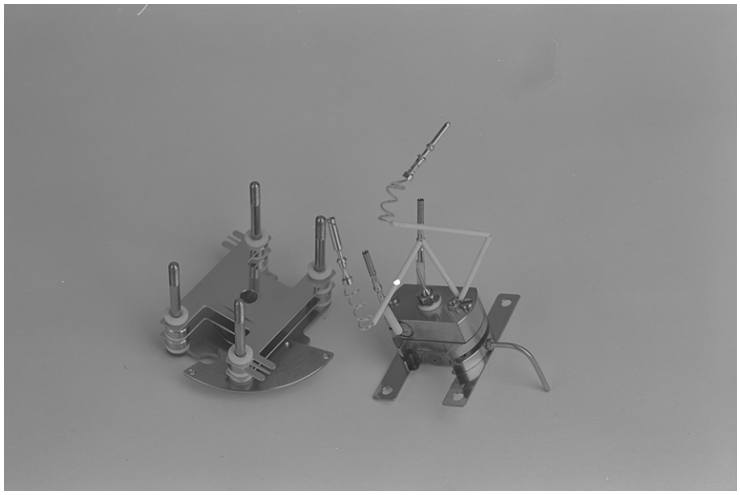

Figure 4-14. Exit plate removed from source block

8. Undo the two remaining screws to separate the ion block from the heater block (see Figure 4-15).

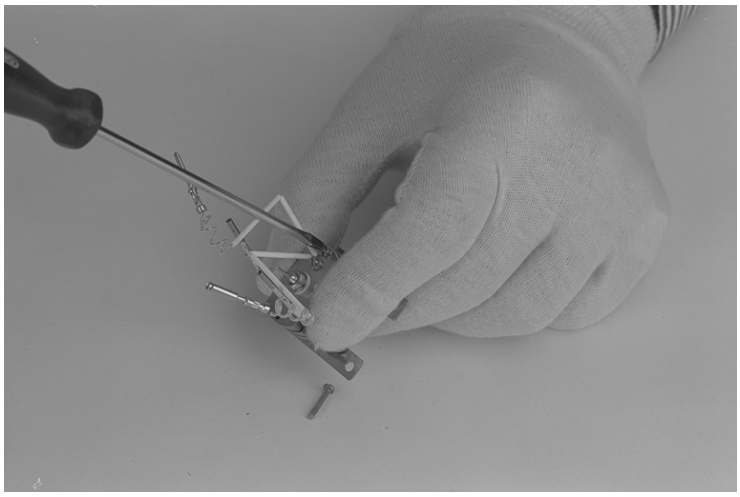

Figure 4-15. Separating the ion block from the heater block

9. Undo the fixing screw and remove the thermocouple (see Figure 4-16).

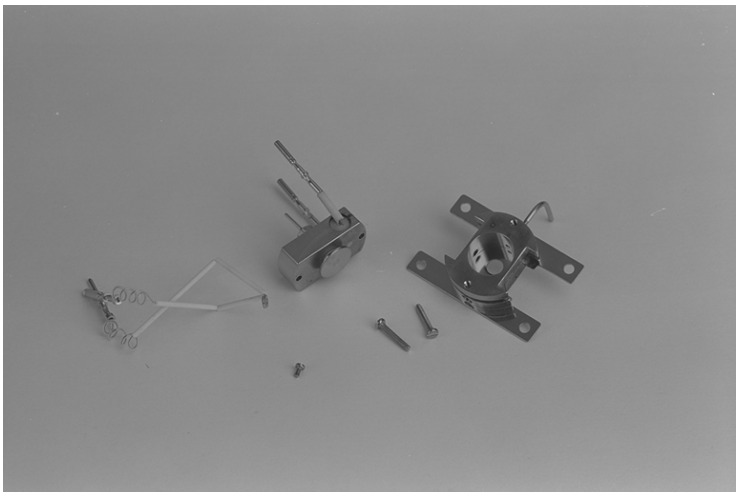

Figure 4-16. Removed thermocouple

> **Caution.** Take care not to bend the arms of the thermocouple.

10. Undo the nut and remove the repeller assembly (see Figure 4-17).

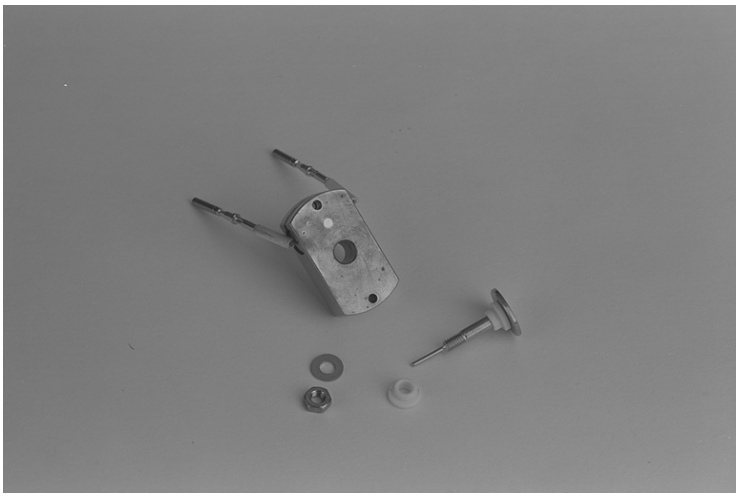

Figure 4-17. Removed repeller assembly

11. Loosen and remove the reference inlet tube and, for CI and combined sources, the reagent gas inlet tube (see Figure 4-18).

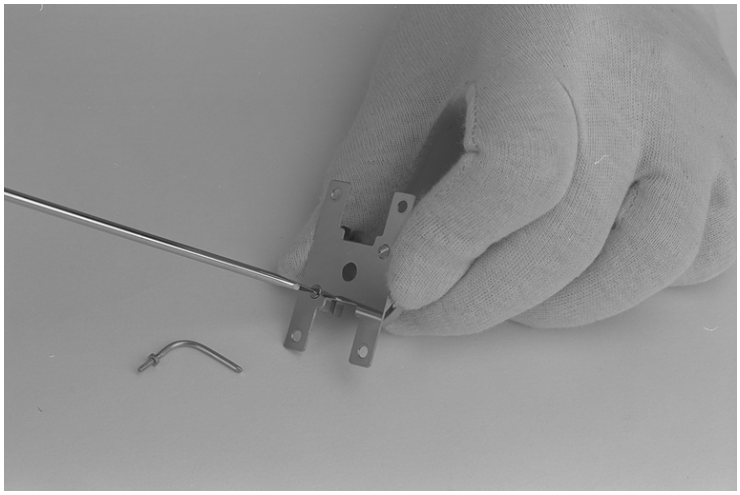

Figure 4-18. Removing gas inlet tube

12. Dismantle the ion lens assembly, noting the relative lens positions and taking care not to damage the ceramics or spacers (see Figure 4-19).

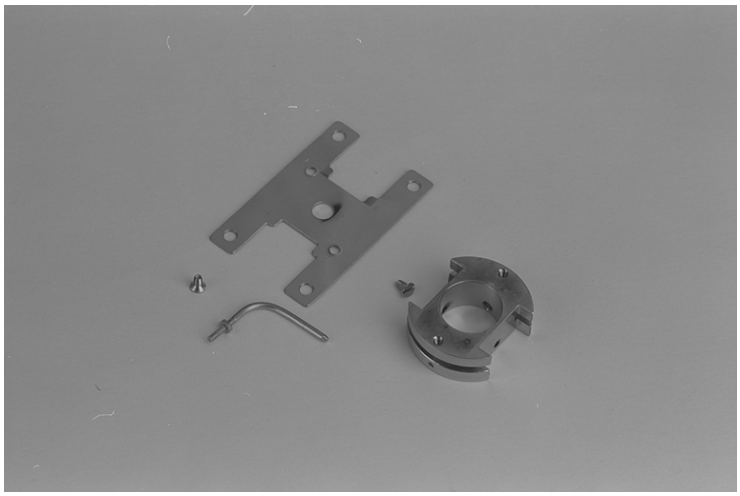

Figure 4-19. Dismantled lens assembly

The ion block, plates and spacers can now be cleaned, by following the procedure described in the following section.

> **Caution.** You are advised **NOT** to remove the source heater elements for routine cleaning, because the constant bending of its legs will eventually lead to fractures. For this reason, ultrasonic cleaning of the repeller block is not recommended.

Cleaning the Source

Only those source components that are exposed to the sample or to the ion beam require regular cleaning. These are the:

- Ion block
- Repeller assembly
- Ion lenses
- Analyzer entry plate

Other parts, exterior to the ion source, do not require regular cleaning.

> **Caution.** The filament should never be cleaned. Ultrasonic cleaning of the repeller block is not recommended.

All metal source components, excepting the repeller block containing the heater element, can be cleaned as described below.

If a component is visibly dirty or burnt, an abrasive cleaner can be used. Suitable abrasives include 6000 grade Micromesh (5010009) and 1200 Micromesh (5010010) or aluminum oxide paste.

1. Rinse the component in clean water.
2. Clean using ultrasonics in methanol.
3. Rinse in a degreasing agent, such as 1,1,1-trichloroethane.

> **WARNING.** When using solvents, all precautions for their safe use and disposal, detailed in the appropriate National Health and Safety regulations, should be followed.

4. Place the cleaned component in a clean beaker and dry in a GC oven at 100 ºC for 30 minutes.

5. The trap should not need regular cleaning, but when necessary it should be removed by disconnecting only the trap wire and removing the holding screw. Clean as above; that is, abrasively where burnt and ultrasonically. On refitting the trap, it is aligned automatically if the mounting plate is already fitted.
6. The repeller block containing the heater element should be cleaned inside, using an abrasive, and then rinsed in methanol.

Replacing Source Components

The following pages describe how to replace source components, including:

- Filament
- Source heater
- Thermocouple

Filament

> **Caution.** Never try to clean the filament: this may cause damage to it.

To replace a source filament:

1. Follow steps 1 to 3 of the procedure for dismantling the source for cleaning; see page 4-16.
2. Remove the filament fixing screw and then take out the old filament (see Figure 4-20).

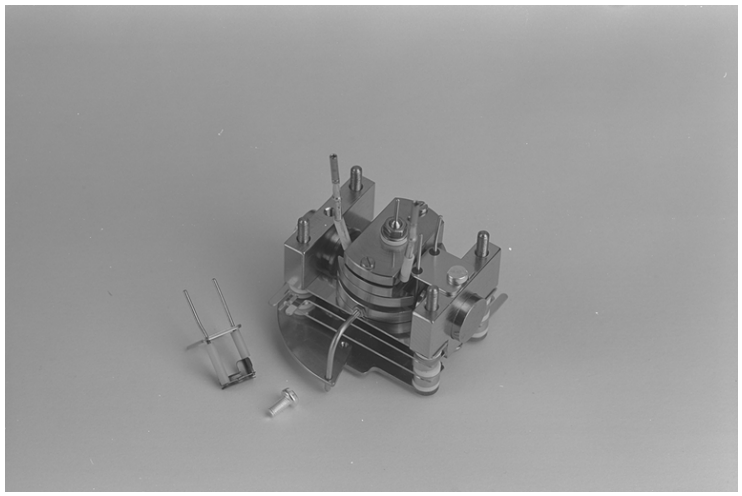

Figure 4-20. Removed filament

3. Carefully fit the replacement filament and refit the fixing screw.

   The new filament self-aligns.

Source Heater

To replace a source heater:

1. Follow steps 1 to 10 of the procedure for dismantling the source for cleaning; see page 4-16.
2. Slacken off the two grub screws holding the heater element (only one on CI and combined sources), and then carefully bend down one leg and retract the used element (see Figure 4-21).

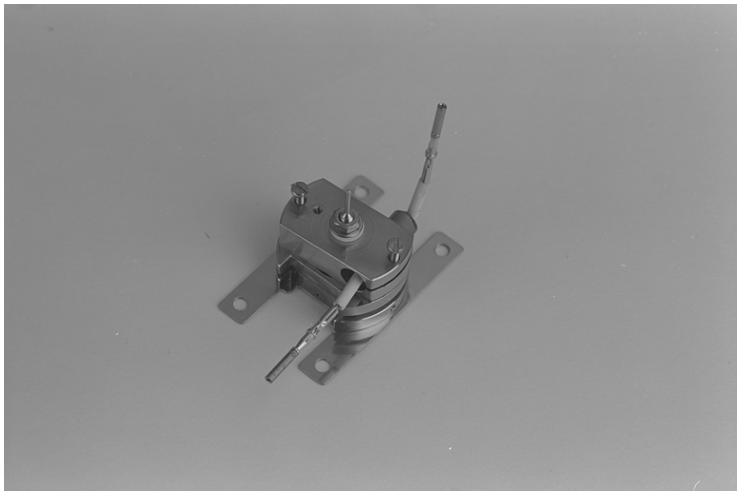

Figure 4-21. Heater with grub screws loosened to allow removal of element

> **Caution.** Always allow the heater to cool before disassembly.

3. Fit a new heater element into the block and carefully bend the leg back up into place.
4. Tighten the grub screw(s) until the element is held firmly in place. Do not over-tighten.

Thermocouple

To replace a source thermocouple:

1. Follow steps 1 to 3 of the procedure for dismantling the source for cleaning; see page 4-16.
2. Remove the thermocouple fixing screw and then take out the old thermocouple (see Figure 4-22).

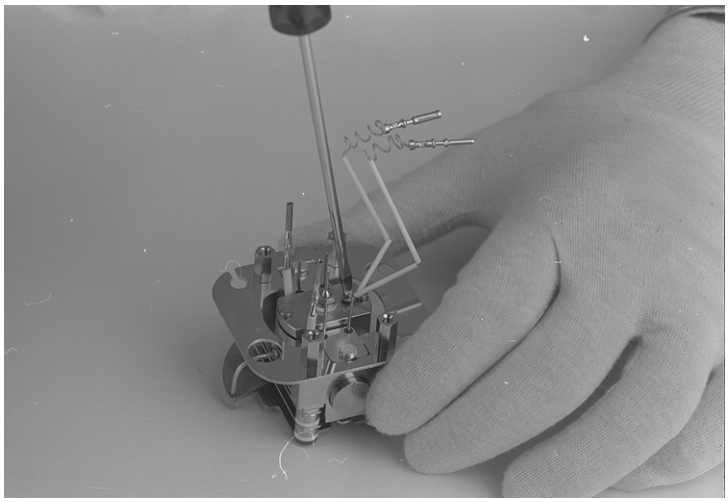

Figure 4-22. Removing the thermocouple

3. Carefully fit the replacement filament and refit the fixing screw.

> **Note.** Only use K0 type thermocouples supplied by ThermoQuest. The connections are keyed by having one male and one female connector.

Reassembling the Source

Reassemble the source following the reverse of the procedure described earlier for disassembling it.

A useful guide to how to assemble EI, CI+ and CI- sources is provided by the animation SOURCE.SS that is included on the CD-ROM supplied with the TRACE MS system. For details of how to run the animation, refer to the read-me file, README.TXT, on the CD.

> **Caution.** Do not over-tighten nuts and screws because this can damage the ceramic spacers.

Chapter 5
Troubleshooting

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5.1 Introduction

This chapter is designed to help you diagnose and resolve problems that may occur from time-to-time with the TRACE MS system.

If you encounter a problem that is not described here, or have a problem that is not resolved by the remedy suggested, contact your local ThermoQuest service representative who will be able to provide additional advice.

This chapter is divided into the following sections:

- Troubleshooting tables.

  The tables are designed to help you identify the precise nature of the problem and the action to be taken.

- Resolving common problems

  This section provides procedures to help you remedy some of the more easily resolved problems you might encounter.

5.2 Troubleshooting Tables

The tables that follow are designed to help you identify problems in the following areas:

- General system problems

  Problems relating to the basic operation of the system; including, for example, breaks in power supply.

- Communications problems

  Problems relating to communications between the various parts of the system and with Xcalibur.

- Spectral problems

  Problems relating to the acquisition of mass spectra.

- Chromatography problems

  Problems relating to gas chromatography.

- Tuning and calibration problems

  Problems relating to tuning and calibration during the setting up of the TRACE MS.

- Source problems

  Problems relating to the operation of the ion source.

The tables describe the symptoms you might observe, the likely cause of the problem, and suggestions on how to remedy it.

General Problems

Symptom	Likely Cause	Remedy
System fails to power-up.	System not plugged in or not switched on at the mains. Mains supply problems.	Plug in; switch on. Check mains supply conforms to the requirements of the TRACE MS system; see **Checking the TRACE MS Power Supply Requirements** on page 5-17 for details.
Fan fails to operate.	Fan is blocked and needs cleaning. Fan is broken.	See Chapter 4 for details. Contact your ThermoQuest service representative.
Rotary pump fails to switch on.	Pump not plugged in or not switched on at the mains	Plug in; switch on. Check circuit breaker.
Turbo pump fails to switch on.	Communications failure. Line extender cable disconnected.	See **Communication Problems** on page 5-5. Reconnect.

Symptom	Likely Cause	Remedy
Pumps switch on but fail to generate vacuum.	Source not inserted correctly.	Remove source and re-insert. See Chapter 3 for details.
	Air is leaking into the system.	Trace and repair the air leak; see **Tracing Air Leaks** on page 5-18 for details.
	Rotary pump foreline trap pellets need replacing.	See Chapter 4 for details.
	Rotary pump oil needs topping-up.	See Chapter 4 for details.
	Rotary pump gas ballast open.	Close ballast.
	Turbo-pump overheating.	Check room temperature / water cooler.
	GC column is broken or is leaking at the interface.	Check the GC column has been installed properly.
		Check the ferrules on the GC interface. See Chapter 2 for details.
	Faulty pirani gauge	Contact your ThermoQuest service representative.
	Column flow rate is too high.	Reduce the flow rate.
	CI gas valve is open or the gas line is leaking.	Check the valve and gas line.

Communication Problems

Symptom	Likely Cause	Remedy
Xcalibur does not communicate with the MS.	Loose line extender cable connection.	Check the connection. Reboot Xcalibur; see **Rebooting** on page 5-20 for details.
	A transient break or fluctuation in mains power supply has halted Xcalibur.	Reboot Xcalibur; see **Rebooting** on page 5-20 for details.
	The Xcalibur software has become corrupted.	Reload the software.
	A malfunction has occurred in the system electronics.	Contact your ThermoQuest service representative.
Xcalibur cannot control the GC and/or autosampler.	Loose RS232 communications cable(s).	Check the connection. Reboot Xcalibur; see **Rebooting** on page 5-20 for details.
	A transient break or fluctuation in mains power supply has halted Xcalibur.	Reboot Xcalibur; see **Rebooting** on page 5-20 for details.
	A malfunction has occurred in the GC or autosampler electronics.	Contact your ThermoQuest service representative.
Xcalibur crashes at the start of data acquisition.	Loose RS232 communications cable(s).	Consult your PC hardware manual and check the connection.
	The Xcalibur software has become corrupted.	Reload the software.
	A malfunction has occurred in the TRACE MS electronics.	Contact your ThermoQuest service representative.

Spectral Problems

Symptom	Likely Cause	Remedy
The spectra show a high level of noise.	The ion source is dirty.	See Chapter 4 for details of how to clean the source.
The spectra are skewed or distorted.	You are scanning too slowly.	Use the Xcalibur Acquisition Manager to increase the scan rate to >6 scans across the peak.
	Unstable or poor tuning.	Re-tune the system. See **TRACE MS Getting Started** for details.
The spectra show incorrect isotope ratios.	Poor calibration.	Re-calibrate the system. See **TRACE MS Getting Started** for details.
	Unstable or poor tuning.	Re-tune the system. See **TRACE MS Getting Started Manual** for details.
	Air is leaking into the system.	See **Tracing Air Leaks** on page 5-18.
	The detector is being overloaded (saturated).	If any peaks are >2x10^6, reduce the concentration of the sample, increase the injector split, or reduce the multiplier or emission tuning values (see **TRACE MS Getting Started** for details).
The total ion current chromatogram (TIC) has a high background.	The ion source is dirty.	See Chapter 4 for details of how to clean the source.
	There is bleeding from the stationary phase of the GC column.	Condition or change the column.
	Air is leaking into the system.	See **Tracing Air Leaks** on page 5-18.
	The carrier gas is of poor quality.	Replace the carrier gas source.
	The sample is dirty.	Clean or re-make the sample.

Symptom	Likely Cause	Remedy
There is no noise on the mass chromatogram.	Acquisition started too soon after tuning.	Wait a short time and then re-try.
	Detector voltage too low.	Increase the voltage on the TRACE MS Tune View. See **TRACE MS Getting Started** for details of how to tune the system.
	Data compression is set “on”.	Switch off data compression on the instrument control panel.
Isotope peaks are missing from the spectrum.	Poor calibration.	See **TRACE MS Getting Started** for details of how to calibrate the system.
	Poor tuning.	See **TRACE MS Getting Started** for details of how to tune the system.
	The ion source is dirty.	See Chapter 4 for details of how to clean the source.
	The sample is too dilute.	Repeat with a more concentrated sample.
	Detector voltage is too low.	Increase the voltage on the TRACE MS Tune View. See **TRACE MS Getting Started** for details of how to tune the system.

Chromatography Problems

Symptom	Likely Cause	Remedy
The retention times are inconsistent.	The GC column is in a poor condition.	Condition or change the column.
	Poor injection.	Repeat, with better injection technique.
	Air is leaking into the system at the injector seal or the carrier gas manifold.	See **Tracing Air Leaks** on page 5-18.
Rising total ion current (TIC) baseline.	The GC column is bleeding.	Condition or change the column.
	Air is leaking into the system.	See **Tracing Air Leaks** on page 5-18.
Chromatograms show discreet high intensity contaminant peaks.	The GC column is bleeding.	Condition or change the column.
	Bleeding is occurring from the septum.	Replace the septum. See Chapter 2 for details.
Chromatogram peaks show “tailing” (that is, sloping on the right hand side).	The injector is not hot enough.	Reset the injector temperature on the GC.
	The interface temperature is not high enough.	Increase the temperature on the GC.
	The carrier gas flow is inadequate.	See your GC user manual for details.
	The injection liner is dirty.	Replace the liner. See Chapter 2 for details.
	The GC column or injector has active sites.	Condition or change the column.

Symptom	Likely Cause	Remedy
The chromatogram peaks are too wide.	The injector is not hot enough.	Increase the injector temperature.
	The sample is overloading the column.	Reduce the amount and/or concentration of the sample.
	Poor GC oven program.	Alter the GC program. See your GC user manual for details.
The chromatogram peaks show an excessive slope on the left hand side.	The sample is overloading the column.	Reduce the amount and/or concentration of the sample.
	Poor injection.	Repeat, with better injection technique.
The chromatograms show flat-topped peaks.	The signal strength exceeds the dynamic range of the detector.	Reduce the amount of the sample.
	The sample is too concentrated.	Repeat with a less concentrated sample.
High baseline.	The sample is dirty.	Clean or re-make the sample.
	Contaminated rinsing solvent.	Replace the solvent.
	Air is leaking into the system.	See **Tracing Air Leaks** on page 5-18.
Baseline falls away slowly from a high initial value.	Purge valve left closed during acquisition.	Alter the GC program. See your GC user manual for details.
	Inadequate purge flow rate.	Increase the purge flow rate.
	Purge left off for too long.	Shorten the purge time.
	Solvent tail peak.	Increase the solvent delay or use a different solvent.
	The pre-filters are dirty	Contact your ThermoQuest service representative.

Symptom	Likely Cause	Remedy
Low sensitivity.	Air is leaking into the system.	Trace and repair the air leak; see **Tracing Air Leaks** on page 5-18 for details.
	The ion source is dirty.	See Chapter 4 for details of how to clean the source.
	The GC column is in a poor condition.	Condition or change the column.
	Incorrect column type.	Change the column.
	The sample injector is dirty.	Clean the injector; replace the liner.
	The ion source temperature is not optimized.	Re-tune the system. See **TRACE MS Getting Started** for details.
	Detector voltage set too low.	Increase the voltage on the TRACE MS Tune View. See **TRACE MS Getting Started** for details of how to tune the system.
	The system is not tuned properly.	See **TRACE MS Getting Started** for details of how to tune the system.
	Poor source filament alignment.	Dis-assemble the source and re-align the filament. See Chapter 4 for details.
	Incorrect column alignment in source.	Re-align the column.
	The pre-filters are dirty.	Contact your ThermoQuest service representative.

Symptom	Likely Cause	Remedy
Poor reproducibility.	The ion source is dirty or badly assembled.	Dis-assemble, clean, and re-assemble the source. See Chapter 4 for details.
	Old or damaged source filament.	Replace the source filament. See Chapter 4 for details.
	Poor calibration.	See **TRACE MS Getting Started** for details of how to calibrate the system.
	Poor tuning.	See **TRACE MS Getting Started** for details of how to tune the system.
	Air is leaking into the system.	See **Tracing Air Leaks** on page 5-18.
	Active sites in the column or liner.	Condition or change the column.
	Incorrect column type.	Change the column.
	Loose connections in the ion source.	Dis-assemble and re-assemble the source. See Chapter 4 for details.
	Poor injection.	Repeat, with improved injection technique.
Poor signal / noise on test standards.	Accidental split injection.	Repeat with splitless injection.
	Column flow rate too high.	Reduce the flow rate.

Tuning and Calibration Problems

Note. In the event of any problems during tuning and calibration, use the TRACE MS tuning diagnostics facilities to obtain further information. See page 5-22 for details.

Symptom	Likely Cause	Remedy
No reference peaks.	The reference vial is empty.	See Chapter 4 for details of how to refill the reference vial.
	Faulty reference solenoid valve.	Contact your ThermoQuest service representative.
	Air is leaking into the system.	See **Tracing Air Leaks** on page 5-18.
	Reference gas pipes are blocked or missing.	Check and clean the source. See Chapter 4 for details.
	There are problems with the quadrupole or pre-filters	Contact your ThermoQuest service representative.
No peaks (or very low intensity peaks) although the source readbacks are correct.	Source lenses poorly aligned.	Dis-assemble the source, as described in Chapter 4, and check the lens alignment with the tool provided.
	Air is leaking into the system.	See **Tracing Air Leaks** on page 5-18.
	Detector voltage too low.	Increase the voltage on the TRACE MS Tune View. See **TRACE MS Getting Started** for details of how to tune the system.
	The ion source is dirty.	See Chapter 4 for details of how to clean the source.
	The pre-filters are dirty.	Contact your ThermoQuest service representative.
	Faulty detector supply.	Contact your ThermoQuest service representative.
	The GC column is broken.	Replace the GC column.

Symptom	Likely Cause	Remedy
High mass peaks (502 and 614 m/z) are missing.	Pre-filters are short-circuiting.	Contact your ThermoQuest service representative.
Tune peaks show signs of breaking up.	Bad connection in the ion source.	Dis-assemble and re-assemble the source. See Chapter 4 for details.
	Pre-filters are short-circuiting.	Contact your ThermoQuest service representative.
	Malfunction in system electronics.	Contact your ThermoQuest service representative.
Changing a tuning parameter has no effect on the peaks.	Source plates not connected or short-circuited.	See **Analyzing Tune Window Readbacks** on page 5-21.
	Malfunction in system electronics.	Contact your ThermoQuest service representative.
Peaks have an irregular shape.	The ion source is dirty.	See Chapter 4 for details of how to clean the source.
	Old or faulty filament.	Replace the source filament. See Chapter 4 for details.
	Poor tuning.	See **TRACE MS Getting Started** for details of how to tune the system.
	Source lenses poorly aligned.	Dis-assemble the source, as described in Chapter 4, and check the lens alignment with the tool provided.
Peaks are shifted from their nominal position.	Poor calibration.	See **TRACE MS Getting Started** for details of how to calibrate the system.
	Malfunction in system electronics.	Contact your ThermoQuest service representative.
Large peaks observed at 18, 28 and/or 32 m/z.	Air is leaking into the system.	See **Tracing Air Leaks** on page 5-18.
	GC carrier gas bottle is nearly empty.	Replace the gas bottle.
	The system is still moist following a change of column or source.	Leave the system in vacuum for a little longer.

Symptom	Likely Cause	Remedy
After changing parameters there is a delay before peaks are changed.	Pre-filters are dirty. The ion source is dirty.	Contact your ThermoQuest service representative. See Chapter 4 for details of how to clean the source.
Excessive ion energy voltage or ion repeller voltage is required.	The ion source is dirty (ion repeller voltage >6.0 V). Pre-filters are dirty (ion energy voltage >3.0 V). Poor tuning.	See Chapter 4 for details of how to clean the source. Contact your ThermoQuest service representative. See **TRACE MS Getting Started** for details of how to tune the system.
The instrument will not calibrate.	Poor tuning. Source temperature is too high or too low. Air is leaking into the system. Electron energy not at 70 eV. The quadrupole is dirty.	See **TRACE MS Getting Started** for details of how to tune the system. Adjust the source temperature. on the TRACE MS Tune View. See **TRACE MS Getting Started** for details of how to tune the system. See **Tracing Air Leaks** on page 5-18. Reset value on the TRACE MS Tune View. See **TRACE MS Getting Started** for details of how to tune the system. Increase the source temperature to 300 °C and leave at that temperature for 2 hours.
Unusual Emission, Filament or Source current readbacks shown on Tune View.	Electrical problem within the source. The source magnets are inserted incorrectly.	See **Analyzing Tune Window Readbacks** on page 5-21. Check the alignment of the magnets: one should have the "N" pole visible; the other, the "S" pole. See Chapter 4 for details of how to dis-assemble and re-assemble the source.

Symptom	Likely Cause	Remedy
The ratio of Source current to Emission current shown on the Tune View is greater than 10:1.	The ion source is dirty.	See Chapter 4 for details of how to clean the source.
	The source filament has deteriorated.	Replace the source filament. See Chapter 4 for details.
	Air is leaking into the system.	See **Tracing Air Leaks** on page 5-18.
	The source filament is poorly aligned.	Dis-assemble and re-assemble the source. See Chapter 4 for details.
	The trap is dirty or has a poor connection.	Clean or replace the trap. See Chapter 4 for details.
	The source magnets are inserted incorrectly.	Check the alignment of the magnets: one should have the "N" pole visible; the other, the "S" pole. See Chapter 4 for details of how to dis-assemble and re-assemble the source.
Readback values are shown in red on the Tune View.	One or more values are out of range.	Run the TRACE MS tuning diagnostics facility. See page 5-22 for details.

Source Problems

> **Note.** Refer to Chapter 4 for information on how to maintain and clean the ion source.

Symptom	Likely Cause	Remedy
Source not heating.	The heater is malfunctioning or is not connected.	Check the heater connection and replace, if necessary. See Chapter 4 for details of how to dis-assemble and re-assemble the source.
	The source thermocouple is faulty.	Replace the thermocouple.
	The source thermocouple is not connected properly.	Re-connect the thermocouple.
Source temperature readback is at maximum (not regulating).	The source thermocouple is faulty.	Replace the thermocouple.
	The source thermocouple is not connected properly.	Re-connect the thermocouple.
Source is becoming discolored, is too hot or is hotter than intended.	The source thermocouple is faulty.	Replace the thermocouple.
	The source thermocouple is not connected properly.	Re-connect the thermocouple.
	The source thermocouple is not connected to the ion block.	Re-connect the thermocouple.
	The source heater is short-circuiting.	See Chapter 4 for details of how to clean the source.

5.3 Resolving Common Problems

The following pages describe how to resolve some of the more easily handled problems.

Checking the TRACE MS Power Supply Requirements

The TRACE MS should be connected to a suitable mains electricity supply. Full details of the power requirements for your TRACE MS can be found in the **TRACE MS Preinstallation Requirements Guide**.

> **Note.** For details of the requirements of other system peripherals, refer to the appropriate manufacturer's specifications.

Tracing Air Leaks

The following pages describe how to trace an air leak, including:

- Confirming an air leak
- Locating the leak

Confirming an Air Leak

To confirm that you have an air leak, set the TRACE MS Tune View to look at the Helium and Nitrogen peaks - m/z 4 and 28 respectively; see **TRACE MS Getting Started** for details of how to tune the system. The relative abundance of helium to nitrogen should be in the order of 10 : 1. A value that differs significantly from this (that is, if the proportion of Nitrogen is much higher than it should be) is confirmation of an air leak in the system.

Locating the Leak

Once you have confirmed the presence of an air leak, you must locate and repair it.

A useful method of locating a leak is to spray likely areas with a solvent, using a syringe or a spray can. If a leak is present, you should be able to detect solvent molecules diffusing into the system by using the TRACE MS Tune View to look for the appropriate mass peak. These peaks should decrease when the leak is sealed.

Freon is a suitable solvent for this purpose; see the side of the can for the molecular weight.

The areas that you should check include:

- Column connections
- Supply plumbing
- GC internal plumbing

Column Connections

If press-fit unions are used on the analytical column, examine these first:

1. Gently tighten the GC interface column 1/16” Swagelok securing nut by a fraction of a turn.
2. Gently tighten the injector column-securing nut by no more than a quarter of a turn.

   Depending on the tightness of the column, the response of the air peak display will be delayed for between ½ and 2 minutes if the leak is here.

> **Caution.** Be wary of over-tightening ferrules. This can reduce their lifespan and risk extruding them, where they might absorb analytes.

Supply Plumbing

Make a particular check in this area if any changes are made to the supply plumbing after commissioning of the instrument.

If the carrier gas cylinder is near the TRACE MS:

1. Close the regulator valve and use a leak-checker (a helium sniffing electronic device would be most useful) to check the cylinder thread and all exposed seals and joints of the regulator body.
2. Set the cylinder regulator to at least 50 psig and repeat the leak checking procedure from the supply to the GC inlet.

If the carrier gas supply comes from a manifold serving a large number of systems, you can only make an effective check for leaks in new plumbing. Once you have done this, examine whether the leak might be from another source. Finally, if necessary, obtain a gas cylinder and plumb it directly into the system.

GC Internal Plumbing

To check for air leaks in the GC column:

1. Connect the carrier gas supply to a single injector and remove the column.
2. Blank off the base of the injector.
3. Fit a new septum.

   Ensure that the septum support plate is in the correct orientation and that the liner is installed correctly.
4. Set the injector to the operating temperature.
5. Close the top and bottom valves, using the GC keypad.
6. Increase the column head pressure to about 50 psig and then close the on/off valve situated below the head pressure regulator.

There should be no noticeable deviation in needle position over a 20 minute period. (You should mark the dial to avoid parallax errors).

If you detect a leak, localize it as follows:

1. Return the column to its normal head pressure.
2. Use a freon spray, as described earlier.

   Even though the plumbing is at a higher pressure than the surroundings, enough freon diffuses into the system to be detectable.

Rebooting Xcalibur

Many of the communications problems that occur with Xcalibur are transient and can be resolved by rebooting.

To re-boot Xcalibur:

1. Try to save any data files that are open.

   Depending upon the nature of the problem, this may not be possible, but you should note that any data that is not saved will be lost.
2. Close down Xcalibur.
3. Exit from the Windows operating system in the usual way, and switch off the PC.

   Consult your PC user documentation for details.
4. Restart the PC, the operating system, and Xcalibur.

Analyzing Tune Window Readbacks

Unusual Emission, Filament and Source readback current values - shown on the TRACE MS Tune View - can be used to diagnose problems within the ion source, as shown in the table below.

Tune Window Readback Values				
Filament (A)	**Source (µA)**	**Emission (µA)**	**Problem**	**Remedy**
0	0	>3000	Trap shorted to ground.	Remove the source and check that the trap is clean (no fibers, swarf, or other items of dust or dirt present).
5 (max)	>3000	0	Trap not connected. Filament shorted to ground on –ve side.	Remove the source and check that the white wire is connected to the trap. Check that the filament leg/connector is not touching any other connector. Check the filament for swarf, fibers, or other items of dust or dirt.
0	0	0	Filament open circuit or not connected.	Check that the filament is intact and that the blue and red wires are connected.
0	>3000	0	Filament shorted to ground on +ve side.	Check that the filament leg/connector is not touching any other connector. Check the filament for swarf, fibers, or other items of dust or dirt.

Running TRACE MS Tuning Diagnostics

In the event of any tuning or calibration problems, use the TRACE MS tuning diagnostics facility to help identify the nature of the problem.

To run the tuning diagnostics:

1. Click on the TRACE MS Tune desktop short-cut.

   The Tune Window is displayed.

2. Choose the **Instrument | Diagnostics** menu option.

   The screen shown below is displayed.

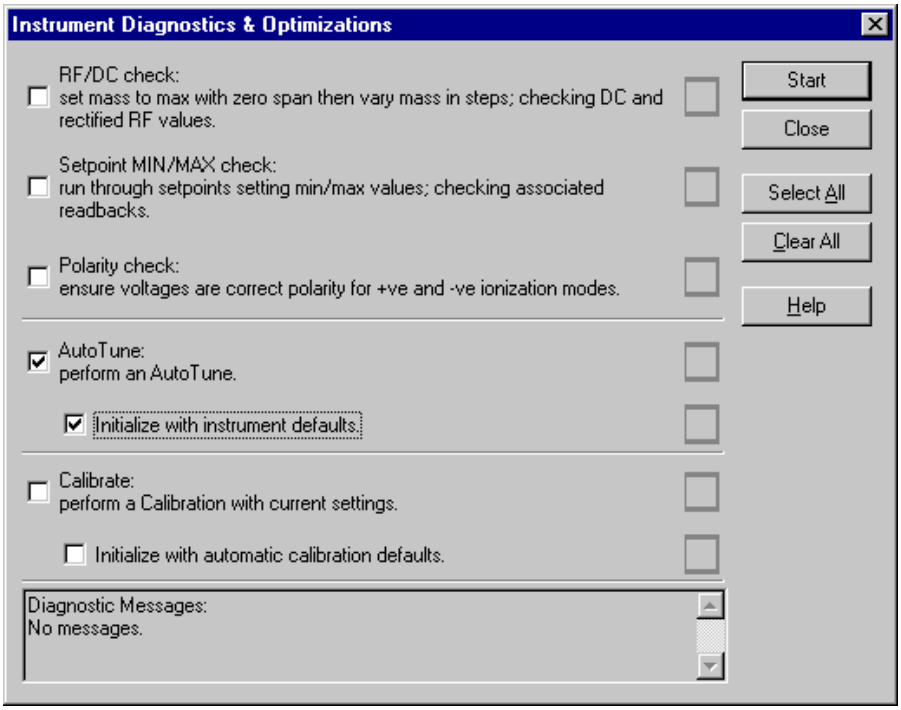

Figure 5-1. The TRACE MS Diagnostics screen

3. Select the boxes alongside the diagnostic tests you want to carry out. Usually, you will want to perform all the diagnostic tests available.
4. Click on **Start**.

The system reports the success ( ) or failure ( ) of its tests (see Figure 5-2).

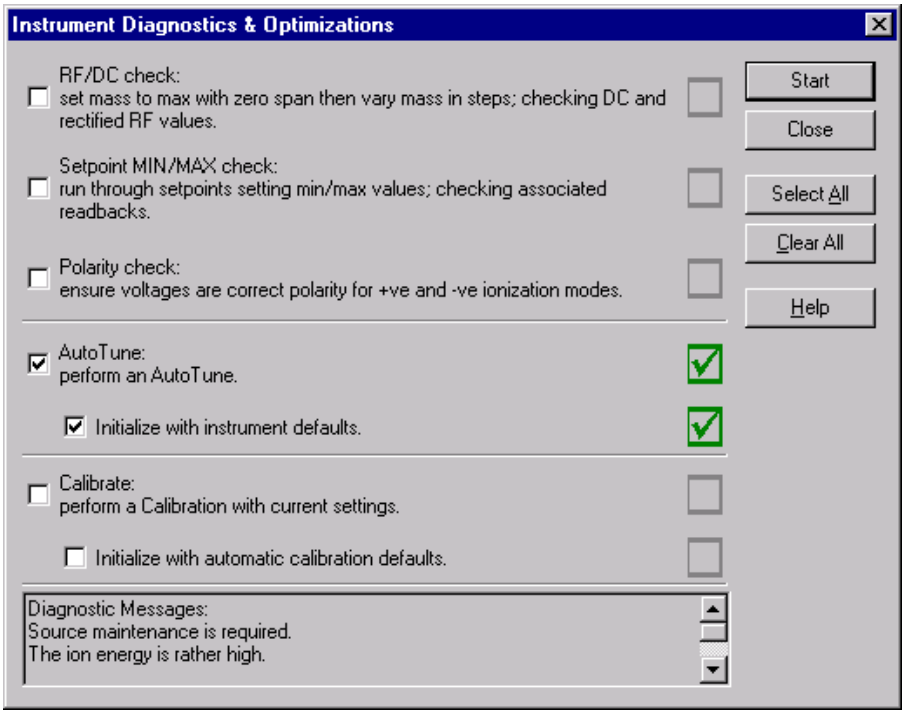

Figure 5-2. Diagnostics results

> **Note.** You may be asked to run the diagnostics facility should you contact ThermoQuest regarding a tuning or calibration problem.

Chapter 6

Shutting Down and Restarting the System

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6.1 Introduction

This chapter provides details of how to shut down the TRACE MS system for a temporary period (up to 2 weeks) or for a longer period.

It also describes how to restart the system following a long-term shut down.

6.2 Temporary Shut Down

If the TRACE MS system is unlikely to be used for a short period, up to 2 weeks, it should not be totally shut down but left in stand-by mode.

To switch the TRACE MS into stand-by mode:

1. Click on the TRACE MS Tune desktop short-cut.
   The Tune Window is displayed.
2. Choose the **Instrument | Operate** option to switch the system out of operating mode, that is, to remove the tick from adjacent to the Operate option.
3. Switch off the PC monitor.
4. If you are using a CI source, close the regulator on the reagent gas cylinder.

The remainder of the system should be left running.

6.3 Long-term Shut Down

If the TRACE MS system is unlikely to be used for an extended period, longer than 2 weeks, follow the steps described below to perform a full shut down.

1. Right-click on the TRACE MS Server icon in the Windows task-bar.

   The server menu is displayed.

2. Choose the **Vacuum | Vent** menu option to vent the system.
3. Exit from Xcalibur and shut down the PC in the usual way.

   Ensure that you have switched off the PC monitor, printer and any other associated hardware.

4. Switch off the instrument and unplug it from the mains.
5. Shut down the GC, following the instructions provided in the documentation supplied with the column.
6. Remove the column and secure the ends to stop air entering it.
7. Close the compressed gas cylinders at the main regulator.
8. If the TRACE MS system is equipped with water cooling, ensure that the water is turned off after the system has cooled.

6.4 Restarting the System

This section describes how to restart the system

Following a long-term shut down, you should:

1. Carry out the visual checks that form the pre switch-on checklist.
2. Follow the system start-up procedure.
3. Pump-down the TRACE MS.

> **Note.** When you have completed these procedures, the system is ready for tuning and calibration; see **TRACE MS Getting Started** for details.

Pre Switch-on Checklist

Before switching on the system after an extended shut down period, a major overhaul or instrument relocation, you should carry out a number of visual checks (see **TRACE MS Getting Connected** for full details).

These include:

Connections check

- Connections to the PC
- GC and MS power cables
- GC and MS communication cables

GC check

- Carrier gas connection to the GC
- Servo-air connection to the GC
- Secondary cooling connection to the GC (if appropriate)
- GC column does not touch the sides of the oven

MS check

- Water cooling connections (if appropriate)
- The MS source is screwed in tight
- Reagent gas connections to the MS (for MS with CI+, CI- or combined source)

System Start-up Procedure

To start up the system, follow the steps described below.

Starting up the GC

1. Activate the GC, following the instructions provided in the documentation supplied with the GC.
2. Wait for the GC to complete its initialization before continuing.

Starting up the TRACE MS

1. Plug in the TRACE MS system and switch it on at the mains.
2. Press the ON switch at the rear of the instrument.

   Ensure that the rotary pump power breaker is depressed.

> **Note.** The TRACE MS vacuum pumps do not start straight away - a command from Xcalibur is required; see below for details.

3. Start up Xcalibur, as described below.
4. Start the gas flow and the water cooler (if appropriate).

Starting up Xcalibur

1. Switch on the PC.
2. Follow the usual procedure for loading the Windows NT operating system.

   See your PC user documentation for details.
3. Double-click on the Xcalibur short-cut icon shown on the Windows desktop (see Figure 6-1).

Figure 6-1. Xcalibur short-cut icon

4. The Xcalibur Home page is displayed.

Pumping-down the TRACE MS

Pumping down is necessary to create a high vacuum within the system.

To pump down the TRACE MS:

1. Right-click on the TRACE MS Server icon in the Windows task-bar.

   The server menu is displayed.

2. Choose **Vacuum | Pump** to pump down the system. It should take less than 10 minutes for the TRACE MS to achieve vacuum, the time being dependent on your pumping configuration. If vacuum has not been reached after 15 minutes, investigate the possibility that air is leaking into the system; refer to the Chapter 5 for details.

   You can monitor the status of the instrument by observing the display panel situated next to the source flap (see Figure 6-2). You can also observe the Server icon display in the Windows taskbar (see Chapter 1).

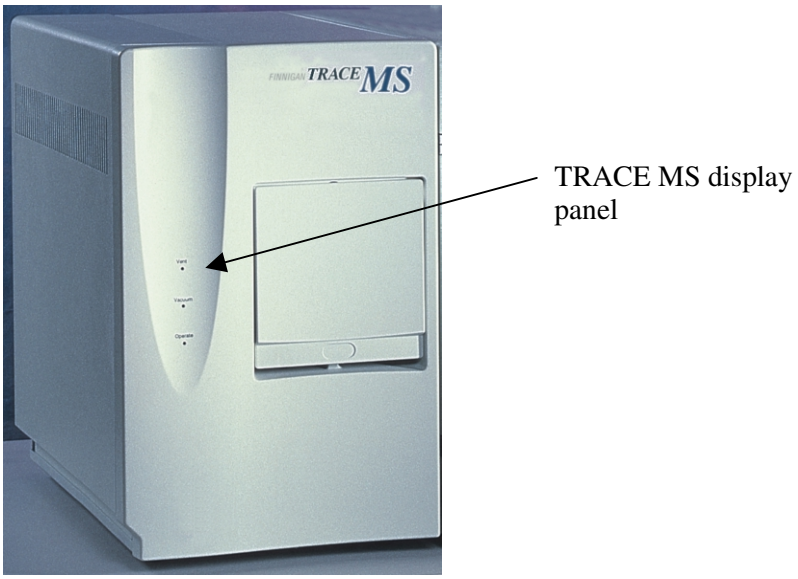

Figure 6-2. TRACE MS display panel

The display panel consists of 6 colored lights, 2 for each of Vent, Vacuum, and Operate. These indicate various vacuum and operating conditions, as shown in Table 6-2.

Table 6-2. TRACE MS status indicator lights

Lights	Flashing Green	Steady Green	Steady Orange
Vent	Not applicable.	At atmospheric pressure.	MS has vented for safety.
Vacuum	Pumping-down – turbomolecular pumps not yet up to speed.	Vacuum achieved.	Turbomolecular pumps up to speed - vacuum not yet achieved.
Operate	Not applicable.	Operate mode on.	Not ready to operate.

3. If you are working with a CI+, CI- or combined source, you should evacuate the reagent gas line. To do this, follow one of the procedures detailed below, depending on whether your TRACE MS is fitted with digital CI flow control (Xcalibur software) or manual valve CI flow control (CI needle valve).

Digital CI Flow Control

1. Click on the TRACE MS Tune desktop short-cut.
   The Tune Window is displayed.

2. Choose **Instrument | Operate** or click on the Toggle Operate toolbar button to switch the instrument into Operate mode.
3. In the TRACE MS Controls bar, set the CI reagent gas flow to about 5 ml/min using the CI (ml/min) text box at the bottom-right of the Controls bar (see Figure 6-3).

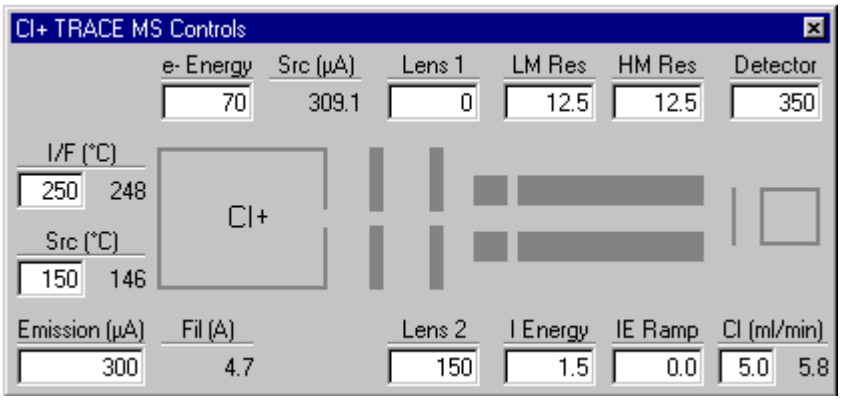

Figure 6-3. TRACE MS Controls bar with CI flow set at 5 ml/min

4. Wait for two or three minutes until all the gas has been removed from the reagent gas line.

5. Choose **Instrument | Operate** or click on the Toggle Operate toolbar button to switch the instrument out of Operate mode.
6. Set the reagent gas regulator to 5 psig before opening the on/off valve of the bottle regulator. The reagent gas line now contains gas at the required pressure.

Manual CI Needle Valve Flow Control

1. Drop the source flap on the front of the TRACE MS and then slowly open the CI needle valve (see Figure 6-4). It is possible that the Vacuum status light, if already green, will turn amber. If this happens, stop opening the valve until it recovers green status again. Repeat the process until the valve is fully open and the vacuum status light is steady green.
2. Close the CI needle valve fully and set the reagent gas regulator to 5 psig before opening the on/off valve of the bottle regulator. The reagent gas line now contains gas at the required pressure.

Figure 6-4. The CI needle valve

Chapter 7
Consumables and Spare Parts

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7.1 EI Source Parts List

Source Block Assembly

Part Number	Description
1132211	M3x6 mm CH HD Silver Plated Screw
1141711	M3 Hexagonal Spacer
5312012	M2x12 mm CH HD ST STL Screw
5312204	M2x4 mm CSK HD Screw
5314016	M3x6 mm CH HD ST STL Screw
5316033	M1.6x3 mm CH HD ST STL Screw
5321011	M1.6 ST STL Nut
7024606	Spring Clip
7024912	Cap Source Magnet
C030A	Filament Assembly
C034M	Source Mounting Plate
C035M	Magnet Housing
C037A	Heater Assembly
C038M	Heater Plate
C039M	Ion Exit Plate
C045M	Repeller
C046M	Lens Spacer 3.5 mm
C112A	Trap Assembly
C119M	Focus Plate
C182M	Source Alignment Tool
C306A	Source Guide Plate
C307A	EI Ion Chamber Sub-Assembly
C308M	Reagent Gas Inlet Tube
C309M	EI/CI Tube Connection Segment
C813M	Heater Gasket
TMAG051	Source Magnet
TSLI050	Source Lens Insulator (10)

Part Number	Description
TTDC001	Thermocouple Ion Block Assembly
C814A	Source Guide Plate

Lower Source Assembly

Part Number	Description
5321004	M3 ST STL Nut
5711125	O-Rings BS235
6366308	E-Type Circlip 3/8”
7024602	Compression Spring
C028M	Source End Flange
C111M	M8x15mm Long Stud
C310A	EI Source Sub-Assembly

7.2 CI+ Source Parts List

Source Block Assembly

Part Number	Description
1132211	M3x6 mm CH HD Silver Plated Screw
1141711	M3 Hexagonal Spacer
5312009	M2x16 mm CH HD ST STL Screw
5312204	M2x4 mm CSK HD Screw
5316006	M1.6x6 mm CH HD Screw
5321011	M1.6 ST STL Nut
7024606	Spring Clip
C030A	Filament Assembly
C037A	Heater Assembly
C046M	Lens Spacer 3.5 mm
C119M	Focus Plate
C306A	Source Guide Plate
C308M	Reagent Gas Inlet Tube
C309M	EI/CI Tube Connection Segment
C315M	CI Source Mounting Plate
C318M	Top Magnet Housing CI
C319M	Filament Magnet Housing CI
C320M	CI Gas Inlet Tube
C323M	Source Alignment Tool CI+
C326A	CI+ Ion Chamber Sub-Assembly
C329M	CI+ Ion Chamber Sub-Assembly
C404M	CI Source Setting JIG Plate
TSLI050	Source Lens Insulator (10)
TTDC001	Thermocouple Ion Block Assembly
C814A	Source Guide Plate

Lower Source Assembly

Part Number	Description
5711125	O-Rings BS235
6366308	E-Type Circlip 3/8”
7024602	Compression Spring
C028M	Source End Flange
C111M	M8x15 mm Long Stud
C229A	CI+ Source Sub-Assembly

7.3 CI- Source Parts List

Source Block Assembly

Part Number	Description
1132211	M3x6 mm CH HD Silver Plated Screw
1141711	M3 Hexagonal Spacer
5312009	M2x16 mm CH HD ST STL Screw
5312204	M2x4 mm CSK HD Screw
5314016	M3x6 mm CH HD ST STL Screw
5316006	M1.6x6 mm CH HD Screw
5316033	M1.6x3 mm CH HD ST STL Screw
5321004	M3 ST STL Nut
5321011	M1.6 ST STL Nut
7024606	Spring Clip
7024912	Cap Source Magnet
C030A	Filament Assembly
C037A	Heater Assembly
C046M	Lens Spacer 3.5 mm
C119M	Focus Plate
C226A	CI- Ion Chamber Sub-Assembly
C306A	Source Guide Plate
C308M	Reagent Gas Inlet Tube
C309M	EI/CI Tube Connection Segment
C315M	CI Source Mounting Plate
C316M	CI- Repeller
C317M	CI- Ion Chamber Base
C318M	Top Magnet Housing CI
C319M	Filament Magnet Housing CI
C320M	CI Gas Inlet Tube
C324M	CI Alignment Tool
C404M	CI Source Setting JIG Plate

Part Number	Description
TMAG051	Source Magnet
TSLI050	Source Lens Insulator (10)
TTDC001	Thermocouple Ion Block Assembly
C814A	Source Guide Plate

Lower Source Assembly

Part Number	Description
5711125	O-RIngs BS235
6366308	E-Type Circlip 3/8”
7024602	Compression Spring
C028M	Source End Flange
C111M	M8x15 mm Long Stud

7.4 Combined Source Parts List

Part Number	Description
1132211	M3x6 mm CH HD Silver Plated Screw
1141711	M3 Hexagonal Spacer
1150705	M2 ST STL Nonmagnetic Washer
5312006	M2x6 mm CH HD Screw
5312012	M2x12 mm CH HD ST STL Screw
5316005	M1.6x5 mm CH HD ST STL Screw
5316033	M1.6x3 mm CH HD ST STL Screw
5321004	M3 ST STL Nut
5321011	M1.6 ST STL Nut
6366308	E-Type Circlip 3/8"
7024602	Compression Spring
7024606	Spring Clip
C028M	Source End Flange
C030A	Filament Assembly
C037A	Heater Assembly
C046M	Lens Spacer 3.5 mm
C111M	M8x15 mm Long Stud
C119M	Focus Plate
C306A	Source Guide Plate Sub-Assembly
C309M	EI/CI Tube Connection Segment
C316M	CI- Repeller
C317M	CI- Ion Chamber Base
C318M	Top Magnet Housing CI
C319M	Filament Magnet Housing CI
C320M	CI Gas Inlet Tube
C589M	Combination Source Mounting Plate
C590M	Combination Source Trap Plate
C591M	Combination Source Trap Insulator
TMAG051	Source Magnet
TSLI050	Source Lens Insulator (10)

Part Number	Description
TSLI051	Source Lens Insulator
TTDC001	Thermocouple Ion Block Assembly
C814A	Source Guide Plate

7.5 GC Interface Parts List

GC 8000 Top

Part Number	Description
6070104	1/8" Vespel Ferrule
6070623	1/16" Blanking Plug
6070628	1/8" Knurled ST STL Nut
C774A	GC Inner Tube Standard

Long

Part Number	Description
6070104	1/8" Vespel Ferrule
6070623	1/16" Blanking Plug
C786A	GC Inner Tube Long

TRACE GC 2000 / Short

Part Number	Description
6070104	1/8" Vespel Ferrule
6070623	1/16" Blanking Plug
C764A	GC Inner Tube Short

7.6 Solids and DCI Probes Parts List

Source and Probe Lock Assembly

Part Number	Description
C692A	Insertion Lock Assembly
C890A	Insertion Lock Spares Kit
5314013	M3×12 mm Long CH HD ST STL Screw
5314018	M3×16 mm Long CH HD ST STL Screw
5711013	O-Ring BS013 Viton
5711018	O-Ring BS011 Viton
5711035	O-Ring BS018 Viton
C851M	Insertion Lock Bush
6366308	E-Type Clip 9.5
7024602	Compression Spring
C713A	Source Sub-Assembly (Solids Probe)
C030A	Filament Assembly
C046M	Spacer (3.5 mm thick)
C119M	Focus Plate
C306A	Source Guide Plate Sub-Assembly
C309M	Connection Segment
C318M	Magnet Housing (top)
C319M	Magnet Housing (filament)
C711A	Probe Source Mounting Assembly
1150705	M2 Flat Washer
5312006	M2×6 mm Long CH HD ST STL Screw
5312012	M2×12 mm Long CH HD ST STL Screw
5316005	M1.6×5 mm Long CH HD ST STL Screw
5316033	M1.6×3 mm Long CH HD ST STL Screw
5321011	M1.6 ST STL Nut

Part Number	Description
C037A	Source Heater Assembly
C308M	Gas Inlet Tube
C320M	Gas Inlet Tube
C589M	Mounting Plate
C590M	Trap Plate
C591M	Trap Insulator
C698A	Ion Block Sub-Assembly (probe)
C712M	Source Extraction Plate (probe)
TTDC001	Thermocouple Assembly
TMAG051	Source Magnet
TSLI050	Source Lens Insulator

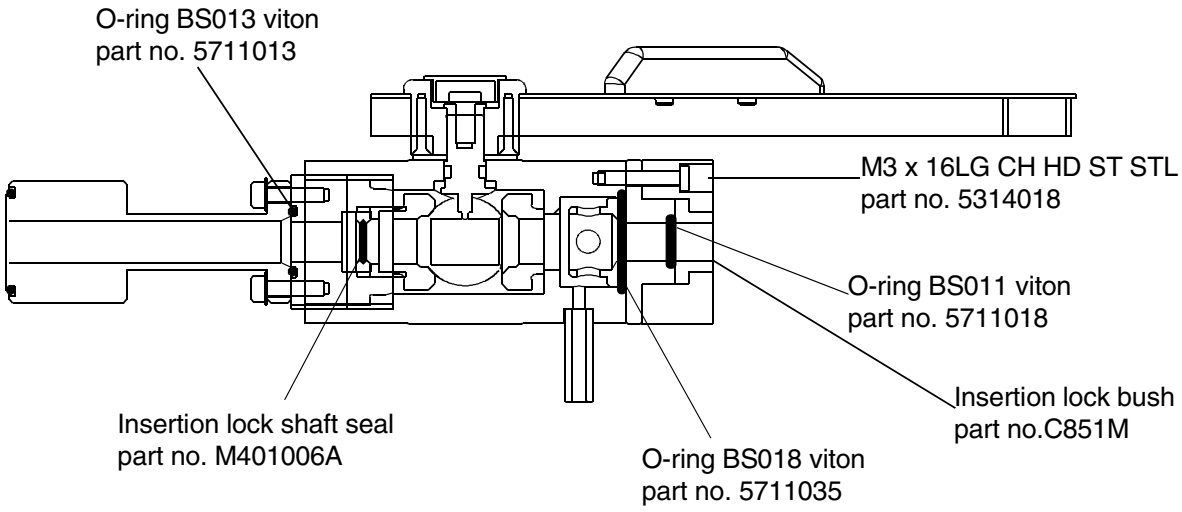

Figure 7-1. Cross-section of probe lock

Note. Parts M401006A, 5711035, 5711018, 5711013 and C851A are all part of the Insertion lock spares kit which has a part number of C890A.

Solids Probe Assembly

Part Number	Description
ML10-005BD1	Sample Cup Grip
TPRB057	Probe Tip - Earthed Source
TPRB058	Probe Sample Holder (deep)
TPRB059	Probe Sample Holder (shallow)
TPRC001	Water-Cooled Probe Cable Assembly

DCI Probe Assembly

Part Number	Description
TPRB009	DCI Probe Tip Assembly
TPRC002	Probe Cable Assembly

Rotary Pump and Control Unit

Part Number	Description
TRPK001	E2M1.5 Rotary Pump 240 V
TRPK001C2	E2M1.5 Rotary Pump 110 V
5560010	Rotary Pump Oil - Ultragrade 19
PDM00117	T10 A fuse for 110 V Rotary Pump (20 mm slow blow fuse)
PDM00118	T6.3 A fuse for 230-240 V Rotary Pump (20 mm slow blow fuse)

7.7 General Spare Parts List

Part Number	Part Name
6270121	Heptacosa
TRPK001	Rotary Pump 240 V
TRPK001C2	Rotary Pump 110 V
5560010	Rotary Pump Oil
6060116	Foreline Trap Alumina Pellets
C773A	Line Extender Cable (3 m)
C384A	GC Cable (GC TOP)

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