CONTENTS

1. INTRODUCTION

- 1.1. Specification
- 1.2. The Manual

2. UPON RECEIPT

- 2.1. Unpacking
- 2.2. Initial Checks
- 2.3. Read Manual
- 2.4. Mounting on Vacuum System
- 2.5. Earth (Ground) Continuity
- 2.6. Electrical Connections
- 2.7. Mounting of Control Unit
 - 2.7.1. Cable Length
 - 2.7.2. Free Standing
 - 2.7.3. Rack Mounting

3. _ CONTROL UNIT

- 3.1. Front Panel Controls
- 3.2. Rear Panel Facilities
- 3.3. Internal Layout

4. RF/HEAD AMPLIFIER

- 4.1. General Arrangement
- 4.2. RF and DC Generator
- 4.3. Head Amplifier

5. QUADRUPOLE

- 5.1. Ion Source
- 5.2. Quadrupole Filter
- 5.3. Detector
 - 5.3.1. Multiplier Detector

6. OPERATION

- 6.1. Switch On and Manual Operation6.1.1. Initial Settings6.1.2. Manual Operating Sequence
- 6.2. Scanning Mode
- 6.3. Override
- 6.4. Check Emission
- 6.5. Degas
- 6.6. Zeroing Amplifier
- 6.7. Operation with electron multiplier6.7.1. Change to multiplier mode6.7.2. To pre-set multiplier gain6.7.3. Multiplier life
- 6.8. Calibration, Sensitivity, Resolution6.8.1. Calibration of Pressure ranges6.8.2. Resolution
- 6.9. Non-Absolute Pressures
- 6.10. Use with Ancillary Equipment6.10.1. Recorder6.10.2. Oscilloscope
 - 6.10.3. Peak Programmer
- 6.11. Operation at High Pressures
- 6.12. Bakeout
- 6.13. Use as a Leak Detector
- 6.14. Relative Sensitivities of Various Gases
- 6.15. Cracking Patterns
- 6.16. Interpretation of Spectra

WARRANTY

Products of Spectrum Scientific Limited are warranted to be free from all defects in material and workmanship. The liability of Spectrum Scientific under this guarantee is limited to servicing, adjusting or replacing defective parts. The guarantee is effective from the date of despatch to the original purchaser for one year, when:

The product is returned to the factory or supplier, transportation charges prepaid, and undamaged by the failure to provide sufficient packaging.

The product appears, to Spectrum Scientific's satisfaction to be defective through no fault of the user.

The equipment has been operated in accordance with the instructions and advice detailed in the appropriate operating instruction manual or any other advice which may be provided by Spectrum Scientific.

The guarantee does not apply to:-

- (1) consumable items,
- (2) components which are outside the component manufacturer's usual guarantee period,
- (3) filaments.
- (4) vacuum feedthroughs,
 - (5) electron multipliers (if fitted).

WARNING

HIGH VOLTAGES EXIST IN VARIOUS PARTS OF THIS INSTRUMENT.

GREAT CARE SHOULD BE EXERCISED WHEN OPERATING WITH THE PROTECTIVE COVERS REMOVED.

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FIGURES

| 3. | Front | Panel |
|----|-------|-------|
| | | |

- 3.1. Input/Output Socket Connections and Power Connections, and Trip Socket Connections.
- 3.2a. Rear Panel
- 3.3. Internal Layout of Control
- 4. RF/Head Amplifier Top Plate
- 5. Quadrupole Head
- 6.a. Analyser Pin Connections
- 6.b. Analyser Ion Source Component List
- 6.c. Analyser Component List
- 7.1a. Main Board Schematic
- 7.2a. Multiplier Schematic
- 7.3b. RF and DC Generator
- 7.4b. Pre-amplifier

TABLES

- 6.14. Relative Sensitivities of some Gases
- 6.15. Simplified Cracking Pattern Tables
- 6.17. Common Residuals and Contaminants

INTRODUCTION

The Spectramass 100 range are small partial pressure analysers of the quadrupole type suitable for residual gas analysis, vacuum problem solving, leak detection, process control and many other similar applications.

The instrument, which is of similar size to most ionisation gauges, has been designed for versatility and has degas, scanning and multiple trip facilities as standard items.

The mass filter is a 4 inch long quadrupole using % inch diameter stainless steel rods and the complete instrument is designed to the following specification:

1.1. Specification

Mass range: 2-100amu.

Resolution: better than 20% valley.

Minimum detectable partial pressure: 1×10^{-11} torr. (4x10 torr for multiplier version)

Minimum detectable total pressure: $5x10^{-12}_{12}$ torr.

(2x10⁻¹³torr for multiplier version)

Maximum operating pressure: 10^{-4} torr (Faraday plate)

Bakeout temperature: 350°C maximum.

Degas power: 2.5 watts (by electron bombardment).

Scanning Speed: 0.1 to 300 seconds in 1,3,10 steps.

Emission: 1mA nominal.

Recorder/Oscilloscope output: 0-10volt o/p.

Timebase output: 0-9volt.

Timebase input programme: 0-10volt.

Power requirements: 115/230volt+or-10% 50/60Hz,60 watts.

The above is the basic specification, but for optimum performance under specific conditions it is sometimes desirable to operate outside the standard specification; for details of this please read the appropriate section of this manual.

Note: The above figures assume an ambient temperature of 25°C for all parts of the instrument. For advice on operation above 40°C please contact the manufacturer.

1.2. The Manual

We earnestly request that you study this manual thoroughly before proceeding to assemble or operate the instrument.

In our experience, bad relations between manufacturer and customer are more likely to develop over the next few hours than at any other time.

Thoroughly reading the manual will not necessarily prevent all problems but it will certainly minimise them. Should any difficulties still remain then communication between us will be a good deal simpler thanks to our mutual understanding of the instrument.

2. UPON RECEIPT.

Please carry out the following procedures.

2.1. Unpacking.

If you have ordered a complete instrument then you should have received the following items:

- (a) a manual (probably the one you are now reading)
- (b) control unit
- (c) quadrupole head (in protective shields)
- (d) RF/Head amplifier
- (e) mains (power) cable
- (f) signal cable for 9-way D connector
- (g) trip cable for 9-way D connector
- (h) 2 spare power fuses
- (i) If dual detector HTK cable
- (j) BNC connectors

If you note any shortages, please check the packing carefully and inform the manufacturer (or agent/supplier) immediately.

2.2. Initial Checks

Please check for any signs of physical damage which may have occurred in shipment. If all is well then:

- (a) Remove cover from control unit (4 small chromium plated screws).
- (b) Check security of all connectors and plug-in components.
- (c) Replace cover.
- (d) RF/Head amplifier. It is unlikely that anything unusual will have happened to this unit, but should you feel the need to check it internally please follow the dismantling procedure laid out in section 8.2.3.
- (e) Quadrupole Head. The quadrupole will have been shipped protected by two aluminium protective covers which are held in place by two screws. Remove these covers carefully and check the quadrupole for obvious loose connections or damage.

2.3. Read Manual

Please read the manual at this stage as requested in section 1.2. before proceeding with the installation of the instrument.

2.4. Mounting on Vacuum System

The vacuum flange on the quadrupole head is a sexless copper gasket UHV flange of 2% inch outside diameter, which is compatible with those supplied by the majority of UHV manufacturers (e.g. the Varian 'Conflat' range).

The seal can be made using either a copper gasket or an appropriate rubber or viton gasket, depending on the nature of the vacuum system in question.

If a compatible flange is not available on your vacuum system it may be necessary to obtain an adaptor flange. If you should require advice on this point please contact your supplier.

It is <u>essential</u> that the vacuum port used should have a minimum inside diameter of 30mm (1%") and an axial clearance of 125mm (5").

It is <u>desirable</u> that you avoid mounting the head in line of sight with other ion sources (e.g. ion gauges) to prevent crosstalk.

It is <u>preferable</u> to mount the head with spigot on the central post of the ten way electrical feedthrough to the right of centre (when viewed from outside vacuum system) so that the RF/Head amplifier hangs vertically downwards. Note the space you will need below your vacuum port to give sufficient clearance for mounting the RF/Head amplifier and its fixed cable.

It is <u>important</u> that a check be made that all 10 pins on the vacuum feedthrough are electrically isolated from the vacuum port, and that there are no electrical shorts between pins, except those used for the filament.

2.5. Earth Continuity

It is essential that a good EARTH (Ground) CONTINUITY exists between the RF/Head amplifier, the quadrupole head and the vacuum port on which the head is mounted, otherwise considerable signal noise may be experienced or in extreme cases the amplifier may be completely unstable giving maximum meter reading.

2.6. Electrical Connections

- (a) Check that the mains (power) voltage selector on the rear of the instrument is selected for your mains supply.
 - (b) For colour coding of mains (power) cable see figure 3.2b.
 - (c) $\sim_{\mathbb{R}}$ Ensure that the POWER switch is in the OFF (out) position.
 - (d) Plug the RF/Head amplifier cable into rear of control and plug onto quadrupole head. Take care to position the cables such that no undue stress is exerted on the RF/Head amplifier and quadrupole head. Otherwise irrepairable damage can be caused to the 10-pin vacuum feedthrough.
 - (e) Plug in high voltage cable (BNC/BNC) between RF/Head and control unit (on dual detector instruments).
 - (f) Plug in mains cable.

The instrument is now ready to operate as a self-contained unit and you may proceed as suggested in the remainder of this manual.

When you require to interface Spectramass with other equipment please refer especially to the following sections:

TRIP FACILITIES

Section

RECORDER/OSCILLOSCOPE

Section

EXTERNAL TIME BASE

on an oscilloscope or peak

programmer Section.

2.7. Mounting of Control Unit

2.7.1. Cable Length

The standard cables supplied are 2 metres in length. Thus the control unit will have to be mounted within this distance from the quadrupole head. If your unit has been adapted for longer cables (up to 100 metres) then please read the additional instructions supplied.

2.7.2. Free Standing

The control unit is designed as a free standing unit and should be placed on a suitable bench or table.

2.7.3 Rack Mounting

The control unit can be rack mounted using the rack mounting ears supplied.

When rack mounting, ensure adequate ventilation is provided above and below the unit.

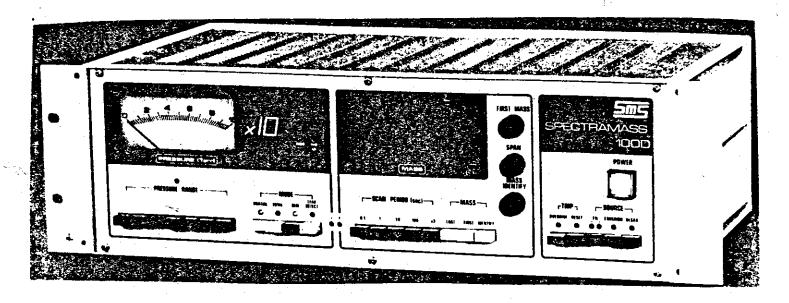


FIGURE 3

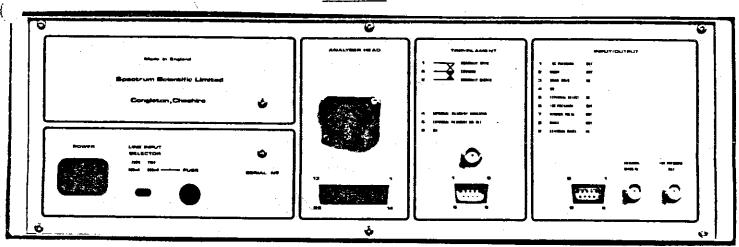


FIGURE 3.2a.

3.1. Front Panel Controls

Figure 3 refers.

This section contains a description of the front panel controls. For operating sequence please refer to section 6.

POWER

Push for on. Switches on mains supply and power to all circuits. Switch illuminates to indicate power on and fuse intact. The unit automatically goes into TRIPPED mode indicated by audible alarm and flashing LED. Press TRIP RESET to activate unit.

EMISSION

Push for on. Switches on filament current. LED indicates when lit that filament current is flowing. If LED is not indicating when emission switch is on, then either filament failure has occurred or circuit has tripped (in this case FILAMENT LED will be flashing).

DEGAS

Push for on. LED indicates power on. Provides 2.5 watts of electron bombardment to outgas source cage. For detailed operation see section 6.6.

TRIP

The trip will operate when the pressure exceeds 1.2 full scale on any range of TOTAL or PARTIAL PRESSURE, unless overridden. TRIPPED STATE indicated by flashing RESET LED and audible alarm. For details of external trip facilities see section 3.2. Rear Panel Facilities.

OVERRIDE

Push for on. All trip facilities (internal and external) are overridden when LED is indicating.

RESET

Push momentarily to reset trip. If the trip resets then LED will go out.

When this switch is pushed and <u>held on</u> the source current (i.e. EMISSION) is indicated on the PRESSURE meter (approx. 30% FSD). See Section 6.4. for full details of this facility.

MODE

Press either PARTIAL or TOTAL to select the required operating mode. LEDs indicate which mode the instrument is in. In TOTAL PRESSURE mode the DC resolution voltage to the quadrupole rods is switched off and ions of all masses have stable trajectories through the quadrupole filter.

PRESSURE RANGE SWITCHES Seven interlocking switches with LED indication of pressure range. Range factors automatically adjusted on switching from Faraday to SEM detector.

ZERO(Z)

Multi-turn potentiometer accessible through the front panel. Under normal conditions of operation

it is unlikely that this control needs adjusting, except when operating at maximum sensitivity. An additional 'coarse zero' is located in the RF/Head amplifier (see Section 4.3.). For details of when it needs adjusting refer to Section 6.7. and Section 8.3.4. for how.

PRESSURE METER The value read from this meter, when multiplied by the range selected, gives the reading of the pressure in TORR (Nitrogen equivalent).

> E.g. A meter reading of 6.5 with a range setting of 10^{-7} indicates a partial pressure of 6.5×10^{-7} torr.

MASS METER

Reads MASS directly in AMU in both manual and scanning modes.

MASS

Pressing FIRST or LAST will display on the mass meter either first or last mass of scan. Also selection of either of these functions will terminate the scan. (See SCAN PERIOD below)

FIRST MASS CONTROL A 10-turn helipot which enables the first mass of a scan to be adjusted to any position between 2 and 100 amu. The FIRST MASS control can be used for manual operation if desired.

SPAN CONTROL

Again a 10-turn helipot. This control sets the width of a scan, i.e. the difference between FIRST and LAST MASSES. Subsequent adjustment of the FIRST MASS control alters both first and last masses, thus maintaining a constant SCAN WINDOW. When used with LAST MASS, the last mass of a scan is easily set.

SCAN PERIOD

Scan times of 0.1, 1, 10 and 100 seconds may be selected by operation of the appropriately marked switches and times of 0.3, 3, 30, and 300 seconds by the additional selection of x3.

These are the times taken to sweep the scan window selected, irrespective of the number of masses covered.

E.g. if first mass is set to 10 amu, last mass is

at 60 and scan time is 100 seconds, then
50 amu will be scanned in 100 seconds,
if first mass is 10 amu, last mass is 15 amu
and scan time is 100 seconds, then 5 amu
will be scanned in 100 seconds.

DIGITAL INSTRUMENTS ONLY

FILAMENT 1/FILAMENT 2 Enables front panel selection of first or second filament.

LEAK DETECT

Leak detect mode is selected by pressing this button.

The unit is automatically tuned to m/e 4 (Helium) and a frequency modulated audio signal is activated.

Other masses may be selected for leak detection using the front panel preset (X).

SEM SWITCH Depressing this switch selects electron multiplier detection system and switches on the high voltage power supply and inverts the main amplifier signal.

SEM CAL Provides facility to adjust high voltage on the multiplier, thereby accurately setting the gain to provide direct calibration in torr.

MASS MARKER Provides split baseline on +ve pressure signal on the input/output 'D' connector with mass readout of intersected peak (mainly used in conjunction with oscilloscope display).

3.2. Rear Panel Facilities

Figure 3.2a refers.

This section contains a description of the facilities available.

Please refer to figures 3.2b for detailed pin connections and colour coding of the standard cables supplied with the instrument.

NOTE: When using either cable please ensure flying leads are terminated before connecting to central unit.

TRIP CABLE

| PIN | The ga | | |
|-----|--------|-------------------------|--|
| 1. | RED | Normally open Contact | |
| 2. | GREEN | Normally closed Contact | |
| 3. | BLUE | Wiper | |
| | · | | |
| | | SIGNAL CABLE | |
| PIN | | | |
| 1. | YELLOW | (-ve pressure signal) | |

3. WHITE (Screen O volt)

4. BLUE (+ve pressure and split baseline, mass identify)5. GREEN Flyback pulse

(Ramp)

POWER CABLE

1. Brown - Live

RED

2.

2. Blue - Neutral

3. Green/Yellow - Earth

POWER

3-pin power socket for mains input.

FUSE

Fuses are 20mm cartridge of the anti-surge type.

(Mains)

For 220 to 240 volts use 630mA.

For 110 to 120 volts use 1.25A.

VOLTAGE SELECTOR

115/230 \pm 5% slide switch recessed to avoid, accidental change.

TRIP

When Spectramass trips as described in Section 3.1 (TRIP), the following facilities are available to command external circuits or equipment:

- (a) Closing contacts rated up to 240 volts at 1 amp (resistive load).
- (b) Opening contacts (rated as above).
- (c) External filament indicator.
- (d) External filament control ON/OFF.
- (e) O volt reference for use with (c) or (d) above.

ANALYSER HEAD

This socket receives the 25-pin plug on the cable from the RF/Head amplifier.

SIGNAL SOCKET

The following input/output signals are available on this socket:

- (a) A fixed voltage ramp of approx. 0-9 volt during scanning to drive oscilloscope x-timebase.
- (b) A negative pulse during flyback for blanking purposes.
- (c) A positive voltage proportional to mass selected for use with an X-Y recorder.
- (d) Both positive and negative signals proportional to pressure for Y-axis on recorder or oscilloscope.
- (e) Incoming signal approx. 0-10 volt to program mass selected.
- (f) Scan hold control.
- (g) External select input for mass programming.

Note: Only the +ve pressure signal on pin 6 includes the split baseline identify signal.

υţ

BNC CONNECTORS 1. +ve pressure signal

- 2. External mass input
- 3. E.H.T. connector to RF Head for multiplier instruments.

3.3. Internal Layout

The general layout of the various printed circuit boards, major components and pre-set controls is shown in figure 3.3.

4. RF/HEAD AMPLIFIER

4.1. General Arrangement

The compact HEAD unit contains both the RF and DC generators and the signal pre-amplifier. It plugs directly into the quadrupole head and no other fitting or electrical connection is involved.

The unit has only two possible adjustment points and these are accessible from the outside (see below).

However, if you require to gain access, please follow the procedure outlined in section 8.2.3.

4.2. RF and DC Generator

This is the board on which is mounted the 10-pin valve base and the RF transformer.

One pre-set type potentiometer, the RESOLUTION control, is mounted on this board and is accessible through a hole in the top plate (see figure 4). It may be adjusted using the trimming tool provided.

For details of when and how to adjust please see sections 6.8.2. and 8.3.5.

4.3. Head Amplifier

One pre-set potentiometer, the AMPLIFIER BALANCE ('coarse zero') is mounted on this board. It is accessible through a hole in the top plate (see figure 4) and may be adjusted with the trimming tool provided.

For details of when to adjust see section 6.7.

For details of how to adjust see section 8.3.4.

5. QUADRUPOLE HEAD

Figure 5 refers.

As in all mass spectrometers there are three principal components:

- 1. The ion source for production of ions that are representative of the gases in its environment and the focusing and injection of these ions into the mass analyser.
- 2. The mass analyser itself which separates ions of different mass to charge ratio and presents these selected ions to the detector.
- 3. The detector which collects the separated ions. Provision is made to eliminate other stray particles.

5.1. Ion Source

Ions are produced in the source by electron bombardment.

Electrons produced from the hot filament are accelerated towards the source cage and pass through it at an energy level of 70eV. The electrons may make several excursions through the source cage because of fields set up by the electron repeller before being collected by the source cage itself. This has the effect of building up a high electron density in the centre of the cage.

The resulting ions are extracted from the source region by means of the focus electrode. This electrode, in conjunction with the entrance electrode, collimates the ion beam into the mass analyser.

5.2. Quadrupole Filter

The mass analyser is of the quadrupole type. It consists of four 4 inch long stainless steel cylindrical rods of % inch diameter. They are mounted symmetrically about the axis of the filter and are supported from the end plates by means of high purity alumina ceramics.

Opposite poles are connected together and antiphase RF and DC voltages are applied to the rod pairs. By selection of the appropriate voltages, ions of the required m/e ratio are made to have stable trajectories through the quadrupole filter. All other ions will have unstable trajectories and will be collected and neutralised on the quadrupole rods.

CAUTION

In order to obtain the precise alignment of the quadrupole rods which is essential to satisfactory performance, specialised jigs are used for assembly. It is therefore recommended that no attempt be made to dismantle the mass filter or remove it from the collector region.

The ion source however can be dismantled.

The procedures which are permissible for maintenance and cleaning are outlined in section 9.

5.3. Detector

Ions are collected on a Faraday plate collector. • This is surrounded by an electron suppressor which repels electrons originating from the source which have drifted down the axis of the filter at low electric field levels.

5.3.1. Detector-multiplier instruments

In addition to the Faraday detector, this instrument is fitted with an off-axis electron multiplier. When a high voltage is applied to the electron multiplier, ions will pass directly into the front face at high energy giving rise to the emission of secondary electrons. These electrons will be attracted through the walls of the multiplier. Many collisions will occur before the electrons finally reach the rear surface. The resulting electron current can be more than 1000 times greater than the ion current entering the electron multiplier.

6. OPERATION

6.1. Switch On and Manual Operation

This section is intended to describe the normal procedure and assumes that Spectramass is being used by itself to monitor vacuum conditions and is not interfaced to any ancillary equipment. It is also assumed that the analyser has been mounted on a suitably operating vacuum system.

6.1.1. Initial Settings Before Switch On

- (a) All switches OFF (i.e. out) position except:
 - (i) PRESSURE RANGE switch 10⁻⁵torr selected.
 - (ii) FIRST MASS selected.
 - (iii) TOTAL switch selected.
- (b) On electron multiplier instruments
 - (i) Multiplier off (i.e. out)follow instruction for Faraday plate operation.
- (c) If cables have been connected as described in section 2.6. then:

6.1.2. Manual Operating Sequence

- (a) Push POWER. POWER and TOTAL LEDs will light, indicating that power is applied to all circuits. Trip reset LED will flash and audible alarm will sound.
- (b) Press TRIP RESET. LED should stop flashing and audible alarm should stop sounding.
- (c) When the quadrupole has been pumped sufficiently for pressure to be below 10⁻⁴torr (check on an alternative gauge if fitted) then:

(d) Switch on FILAMENT. FILAMENT LED should light, indicating that filament current is flowing.

This lamp flashing indicates either filament failure (beginner's luck) or circuit tripped, in which case TRIP LED (beside RESET button) should be flashing. If the latter, depress RESET button momentarily. If the instrument remains tripped DOUBLE CHECK that the pressure is below 10⁻⁴torr.

Assuming EMISSION LED stays on then:

- (e) Select successively lower PRESSURE RANGE switches until TOTAL PRESSURE can be read on the meter.
- (f) Depress PARTIAL switch. The partial pressure LED will light and the instrument is now in the partial pressure mode.
- (g) Depress FIRST MASS switch and dial up mass required. Any individual mass may now be monitored by using this control as a manual mass selector, the corresponding partial pressure now being read on the pressure meter with an appropriate PRESSURE RANGE switch selected.

6.2. Scanning Mode

For scanning operation proceed as in 6.1. above except that in step (g) you should set FIRST MASS to that required as the beginning of the scan then:

- (a) Depress LAST MASS switch and using SPAN control, set the desired value of last mass as indicated on the MASS METER.
- (b) Select SCAN PERIOD required by pressing appropriate switch(es).
- (c) The instrument will now scan repeatedly between the two masses selected.
- (d) To stop scan, select FIRST MASS (or LAST MASS).

6.3. Override

This switch may be depressed to prevent unnecessary tripping.

It is especially useful when investigating small partial pressure peaks in close proximity to major peaks when operating in the PARTIAL PRESSURE mode.

6.4. Check Emission

With the pressure switch at any setting, SOURCE CURRENT can be indicated on the PRESSURE meter by pressing the RESET button and holding down. The reading should be between 25% and 30% of full scale deflection. (3mA full scale)

6.5. Degas

The filament in normal operation radiantly heats a region of the source. If this region has been heavily contaminated by previous exposure, serious outgassing problems can occur. The degas facility is incorporated to raise this region to a higher temperature to remove such contamination before normal operation of the quadrupole.

As, by intention, large quantities of gas are released during outgassing, care should be taken to avoid this process at times when the work being carried out in the vacuum system might prove susceptible to contamination.

From the point of view of the instrument care should be taken:

- (a) To degas only when pressure can be maintained at 10⁻⁶ torr or below, otherwise filament life will be shortened and failure may occur.
- (b) To <u>restrict</u> degassing time to no greater than <u>ten minutes</u>.

 Two or three will normally suffice.

NOTE: Pressure readings cannot be taken when the instrument is in the 'DEGAS' mode.

6.6. Zeroing Amplifier

Normally, zeroing of the amplifier should not be required. If it does become necessary insert screwdriver through the hole in the front panel and adjust zero with the 10^{-10} PRESSURE RANGE switch selected and the filament switched off.

If you are unable to zero with this control then the amplifier output may be out of balance and the procedure laid out in section 8.3.4. should be carried out.

6.7. Operation with Electron Multiplier

The multiplier fitted to this instrument is a small channel plate mounted off-axis at the rear of the quadrupole filter. Its purpose is to extend the detection capability of the instrument in the partial pressure mode when operating at low total pressures.

6.7.1. To change to electron multiplier mode

- (a) Depress SEM button.
- (b) LED indicator will light. This button switches on the power supply to the electron multiplier and changes the amplifier polarity. If a partial pressure peak is being displayed during this operation a change in peak amplitude will be observed.

6.7.2. To pre-set multiplier gain

The multiplier gain has been factory preset to a factor of approximately 100. If it is required to alter the gain, the following procedure should be followed:

- (a) Adjust manual scan control in Faraday plate mode and select small partial pressure peak.
- (b) Observe pressure meter reading.

- (c) Switch to multiplier mode.
- (d) Reduce amplifier gain by depressing next PRESSURE RANGE button (i.e. if in Faraday mode 10^{-8} torr button selected then depress 10^{-7} torr button).
- (e) Rotate 'CAL' control until same meter reading obtained: multiplier now has a gain of 10.
- (f) Repeat steps 4 and 5 to achieve a gain of 100. ... and so

A new multiplier will have a gain of greater than 1000 when 'CAL' control is set to maximum.

If 'DEGAS' or 'TOTAL PRESSURE' is selected when the instrument is in the multiplier mode, the high voltage to the multiplier will automatically switch off and the LED indicator light will flash. Because the amplifier is in the inverted state a negative pressure reading will result. Switching off either of these functions will cause the multiplier supply to automatically reset.

6.7.3. Multiplier Life

To obtain the maximum possible life from the electron multiplier it is important to observe the following rules:

- (a) Avoid operation of the multiplier above 10^{-6} torr.
- (b) Avoid using unnecessarily high multiplier gain.
- (c) Avoid sitting on high partial pressure peaks for prolonged periods of time with high multiplier gain settings.
- (d) Avoid high bake-out temperatures (300°C recommended maximum).

In the event of the multiplier gain falling after a long period of use, or due to excessive operating current and/or high bake-out temperature, the gain can ususally be recovered by exposing the analyser head to atmosphere for several hours.

6.8. Calibration, Sensitivity, Resolution

6.8.1. Calibration of Pressure Ranges

Numerous tests and adjustments are carried out on the unit during manufacture to ensure that it meets its stated specification. These adjustments include many which we do not outline in this manual as they are pre-set prior to despatch.

However, it is worth going into a little detail on the subject of calibration so that the user will appreciate the extent to which he may rely on the unit as a 'gauge' and also some of the limitations.

Briefly summarised, the procedure is this:

- (a) Parameters are optimised so that SENSITIVITY is greater than specification at standard RESOLUTION, yet always ensuring that PEAK SHAPE, MASS RANGE, DISCRIMINATION, SIGNAL TO NOISE and the relationship between TOTAL and PARTIAL PRESSURE (nominally a factor of 10) are satisfactory.
- (b) Calibration of TOTAL PRESSURE is checked against a high quality Bayard-Alpert ionisation gauge and adjusted accordingly.
- (c) This will result in PARTIAL PRESSURE SENSITIVITY being too high. This is adjusted (when operating in a high nitrogen background) by increasing the RESOLUTION until SENSITIVITY is reduced to a suitable level.

Thus both total and partial pressure will be in accordance with a good ion gauge (see section 6.9.) and resolution will be better than specified (i.e. > 20% valley).

Section 8.3.5. will add to your confusion on this subject.

6.8.2. Resolution

On all quadrupoles resolution may be altered (generally made worse) by alteration of the RF/DC ratio. The unit is no exception, but as we expect most customers to be interested in vacuum partial pressure (in torr, not arbitrary units), we calibrate as above and then hide the resolution control.

It is however accessible on the RF/Head amplifier and can be adjusted with the trimming tool provided.

It can be useful in some instances to:

- (a) De-resolve and increase sensitivity, e.g. for leak detection.
- (b) Increase resolution with consequent loss in sensitivity, e.g. if carrying out a high pressure sputtering procedure.

6.9. Non-Absolute Pressures

Please bear in mind that no quadrupole or other mass spectrometer or any vacuum gauge relying on ionisation phenomena is capable of absolute pressure measurement. Accuracy is rarely quoted, but the most expensive (hence best?) ionisation gauges are at best only capable of indicating pressure to $^{-50\%}_{+100\%}$. That is a pressure indication of say 2×10^{-7} torr probably lies somewhere in the range 1×10^{-7} to 4×10^{-7} . What is more, this is the likely pressure at the gauge (not necessarily the same as the rest

However, providing you recognise that all such measurements have their limitations, you will get a great deal more information from a partial pressure gauge, especially if you make intelligent use of the known information on:

of the system) and only if the gas being measured is 100% nitrogen.

- (a) relative sensitivities of various gases, section 6.15.
- (b) Cracking patterns, section 6.16.

6.10. Use with Ancillary Equipment

6.10.1. Recorder

The most used means of obtaining a visual display of the partial pressure spectrum is with the use of a strip chart recorder.

The signal output from a standard instrument is approximately 10 volts for full scale deflection of the pressure meter.

These signals may be attenuated to give signals down to 100mV by means of a simple divider network.

Alternatively, an X-Y recorder may be employed with the X signal (approximately +0.1volt per AMU i.e. approximately 10 volts for 100 amu) being taken from the signal output socket.

SCAN SPEEDS For <u>normal</u> recording, optimum traces will be obtained if scan speeds of the order of 1 AMU per second or slower are utilised.

6.10.2. Oscilloscope (Faraday Plate Instruments)

The desirability of being able to view the spectrum from any mass spectrometer on a CRT (i.e. oscilloscope, visual display monitor, etc.) is very high. However, it must be borne in mind that the unit is fitted with a Faraday plate type of collector, and that to maintain a sensible dynamic range with sensible noise levels, it is fitted with an amplifier of limited response time.

It is therefore <u>necessary</u> to use a oscilloscope with a <u>long</u> persistence display.

When using an oscilloscope it is essential to use a common time base for both the scope and Spectramass. (One can set up a scope display using an oscilloscope's internal time base and driving the unit from its own SCAN, but one's patience is likely to be very quickly exhausted!) Instead, either drive the oscilloscope from the unit or drive the unit from the oscilloscope using the TIME BASE INPUT or OUTPUT facilities as described under X-Y recorder in section 6.10.1. above.

A blanking pulse is provided (see figure 3.2b) for blanking out the CRT during flyback.

SCANNING SPEEDS

It is not recommended that scan speeds of greater than 10 AMU per second are used otherwise severe peak distortion, due to limited response time, will result. Improvement in the response and hence display when using the model 100D fitted with an electron multiplier can be obtained by increasing

the gain using the 'CAL' control. A lower gain setting of the amplifier would have to be used with allowance made for calibration observed.

6.10.3. Peak Programmer

For normal scanning from an external source a 0-10 volt linear ramp is required.

Alternatively, a sequence of stepped voltages may be provided to allow the instrument to jump from peak to peak. Such pulses are normally provided from a PEAK PROGRAMMER.

Requirements of the peak programmer are:

- (a) The ability to provide signals in the range of 0-10 volts in a sequential fashion.
- (b) Ability to hold that voltage for a predetermined length of time (programmed) to an accuracy and stability of better than 10mV (i.e. < 0.1 AMU) for sitting on peak tops.
 - Or ability to jump to predetermined voltages quickly and then to ramp up slowly so that individual peaks or small groups may be monitored.
- (c) Sequence time must be in line with the speed of response as described elsewhere.

If it is desired to output the signals from the various channels onto, say, a multipen recorder then suitable demultiplexing (sample and hold) facilities must also be provided.

6.11. Operation at High Pressure

The unit has been designed to operate up to 10^{-3} torr but certain words of caution are in order regarding such operation.

(a) Filament Life. When operating in the pressure range 10^{-6} to 10^{-4} torr TOTAL PRESSURE filament life will inevitably be shortened particularly if the instrument is frequently used at high pressure and this is especially true where 'active' gases

are present. Two examples of this are:

- (i) High partial pressures of hydrocarbons can give rise to surface deposits which act as insulating layers on the source cage and source electrodes. This may lead to loss in sensitivity due to changes in ion focusing conditions, and in severe cases unstable or even total loss of electron emission. As the source is emission stablilised, filament burn-out can occur because filament power will rise to try to compensate for these effects.
- (ii) High pressures of water vapour accelerate filament erosion and the formation of hot spots. If these vacuum conditions persist the process tends to be cumulative, resulting in premature filament failure.
- (b) Special filaments. Special filaments which are more suitable for high pressure work can be provided.
- (c) <u>Contamination</u>. A high rate of contamination will almost inevitably occur at higher pressures and will necessitate more frequent cleaning by the user (or reconditioning by the manufacturer). See section 8.

6.12. Bakeout

The quadrupole head (with the RF/Head amplifier removed) is fully bakeable to 350°C, although 250°C is usually sufficient for most applications. Normal vacuum/UHV practice regarding rate of rise of temperature and adequate vacuum should be observed.

6.13. Use as a Leak Detector

One of the more important uses of partial pressure analysers is as leak detectors.

In the first place, the presence of the unit in the vacuum system will enable the user to determine whether a higher than normal pressure condition is due to a leak or to some other cause, e.g. severe outgassing, high water vapour, etc.

I it is decided that a leak might be present, a systematic search using a probe gas will be necessary. The following notes might prove useful:

SEARCH GAS: An advantage of a partial pressure guage is that many gases which are readily available in the laboratory or plant can be utilişed, always providing they offer no health hazard. Commonly available gases include:

HYDROGEN

(Mass 2) Not frequently used. Explosive in air. Sometimes present in large quantities in the vacuum system so sensitivity a problem.

HELIUM

(Mass 4) By far the best! but more expensive. Reasonable sensitivity. Very high penetrating power due to low molecular size. Rarely present in the vacuum system.

METHANE

(Mass 16) Useful. Present in coal gas (town gas) which is often readily available. Inflammable.

NEON

(Mass 20) Could be used. Not generally readily available. Can be masked by doubly charged argon.

NITROGEN

(Mass 28) Virtually useless because of 80% presence in air, therefore no substitution takes place. Also could be large CO peak at 28.

PROPANE

(Mass 29) Similar to methane.

OXYGEN

(Mass 32) Little use. Again high content in air. However if a large 32 exists it can sometimes be used by observing the fall of the peak when a leak is covered by another gas or even a liquid, e.g. organic solvent. Emergency use only.

ARGON

(Mass 40) Next most useful after helium. Often available, less expensive. Leaks can be masked by relatively large concentration in air (1%).

BUTANE

(Mass 43) Similar to methane.

CARBON DIOXIDE (Mass 44) Can be used, though there is often a fairly high background of CO_2 so rather limited. RESOLUTION AND SENSITIVITY: It is often advantageous to deresolve the instrument

- (a) to increase sensitivity
- (b) to broaden the peaks thus making them easier to sit on for prolonged periods.

FINE PROBE Use a fine probe such as a piece of glass or metal capillary tubing or a piece of glass tubing drawn to a point.

BAGGING For an elusive leak it is sometimes useful to bag sections of a vacuum system systematically using polythene bags and adhesive tape.

TIME CONSTANT Do not work too quickly when looking for small leaks as the time constant of leaks can on occasions be remarkably long (up to many minutes in the worst instances).

FLOATING GAS Remember that the gas you are using may drift away to other parts of the system, thus causing confusion. Remember also that gases lighter than air rise, e.g. helium, and vice versa, e.g. argon.

MAGNITUDE It is very difficult to determine the absolute numerical magnitude of a leak without knowing the exact pumping speed at the mass spectrometer and carrying out a detailed calibration.

REMEMBER that the analyser may be well upstream of the leak and with a well-pumped system sensitivity may be reduced. Throttle the pump if possible, but ensure that the pressure keeps to a reasonable level.

6.14. Relative Sensitivities of Various Gases

The sensitivity of most vacuum gases is quoted for dry nitrogen. Other gases (or vapours) ionise either more or less readily, thus equal pressures of different gases will give rise to different ion currents.

It is therefore useful to know the sensitivity of a particular gas relative to nitrogen (on which the instrument was calibrated) so that more meaningful partial pressures may be determined. To obtain the true partial pressure of any particular gas, divide the apparent (nitrogen) pressure by the Relative Sensitivity (RS) viz:

Gas partial pressure = Indicated partial pressure
Relative sensitivity

We have listed here the relative sensitivities of the more common gases:

Table 6.14. Relative Sensitivities of Some Gases

Gas H_2 He CH_4 H_2O Ne CO N_2 O_2 A CO_2 Relative Sensitivity 0.7 0.23 1.08 1.17 0.24 1.09 1.00 0.62 1.16 0.9

6.15. Cracking Patterns

The process of ionisation used in the analyser (and most similar instruments) is that of bombardment with electrons at 70eV. As well as giving rise to simple singly ionised molecules, other factors lead to more complicated spectra.

These factors include:

MULTIPLE IONISATION For example, argon (mass 40) will principally be singly ionised by A^{\dagger} which will give a peak in the spectrum at m/e 40. A small percentage (\Longrightarrow 11%) will be doubly ionised to $A^{\dagger+}$, giving a peak at m/e = 40/2 i.e. 20.

FRAGMENTATION Molecules, as well as being ionised, will also to a lesser extent be broken up into smaller fragments.

For example, water will give a peak at 18, plus peaks at 17 (HO), 16(O) and 2 (H₂).

ISOTOPES An additional complication is the naturally occurring isotopes of various elements which are present in the molecule in question.

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These factors, when combined, lead to relatively complex spectral patterns called cracking patterns. A good example of this is CARBON DIOXIDE, for which the principal peaks are:

| m/e | % of largest peak | Cause |
|------------|-------------------|--------------------------------------------------------------------------------|
| _46 | 0.4 | Isotope peak, singly ionised $^{12}C_{\cdot}^{16}O_{\cdot}^{18}O_{\cdot}^{18}$ |
| 45 | 1.1 | Isotope peak, singly ionised $^{13}C^{16}O_2$ |
| | - | (plus 12c17o16o) |
| 44 | 100 | Principal peak, due to $^{12}c^{16}O_2$ |
| | | singly ionised |
| 28 | 11.4 | CO ⁺ cracked from CO ₂ |
| 22 | 1.2 | CO_2^{++} doubly ionised (m/e=44/2=22) |
| 16 | 8.5 | O ⁺ cracked from CO ₂ |
| 12 | 6 | C ⁺ cracked from CO ₂ |

In order to assist you in interpreting spectra we have drawn up a simplified CRACKING PATTERN table (table 6.16.) showing the principal peaks of a number of gases commonly encountered in vacuum work.

<u>Please note</u> that the figures are rounded to the nearest per cent and that the peaks of less than 0.5% have been omitted.

6.16. Interpretation of Spectra

- (a) Plot out the spectrum, preferably on a recorder.
- (b) Identify the principal peaks; see table 6.16 for the more likely ones (remember the mass scale of Spectramass is linear which will help).
- (c) To obtain a clearer indication of individual partial pressures, take into account:
 - (i) Peaks due to cracking pattern of larger molecules (and ignore/eliminate them) table 6.16.
 - (ii) Relative sensitivity, table 6.15.
- (d) Further advice on the interpretation of mass spectra will be found in leaflet SMS Q/13.

Table 6.17 - Common Residuals and Contaminants

| Main Peak | Related Peaks | Peak Height Ratio | Residual | Possible Cause | |
|----------------|------------------------------|-------------------------|----------------------|--------------------------------------------------------------------------------------------------------------------------------------------------------------------|--|
| 2 | 4 | - | Hydrogen | Common residual in most vacuum systems. Caused by cracked water or any hydrogen compounds especially in ion pumped systems. | |
| 4 | - | - | Helium | Sometimes present in ion pumped systems especially if used for leak detection. Not pumped by sorption pumps. Helium diffuses through some glasses. | |
| 12 | - | - | Carbon | Cracked carbon compounds. Not present alone. | |
| 16 | 1 5 | 10/8 | Methane | Common residual in oil diffusion pumped systems. Can be present in ion pumped systems (prior to bakeout) due to ion pump cracking heavier hydrocarbons. | |
| 18 | 17 | 4/1 | Water | Often the largest peak in unbaked systems. Can be removed by baking or use of cold trap. | |
| 20 | 19 | - | HF | Sometimes seen if hydrofluoric acid used in chemical cleaning. | |
| 20 | 22 | 10/1 | Neon | Sometimes seen in sorption pumped UHV systems. | |
| 28 | 12 | 100/4 | Carbon Monoxide | Common residual. Also caused by hydro- carbon cracking on tungsten filaments. | |
| 28 | 14 | 100/7 | Nitrogen | Common residual. 80% of air leak? Look for oxygen/argon. | |
| 32 | 16 | 10/1 | Oxygen | Common residual, 20% of air. Not always seen in well baked systems even though air leak present. | |
| 35 | 37 | 3/1 | Chlorine | Caused by cracked chlorine compounds, e.g. HCl, trichloroethylene. | |
| 40 | 20 | 10/1 | Argon | Fairly common. Argon is 1% of air so can often confirm an air leak. Present also in ion pumped systems due to low pumping speed. | |
| .43 | | <u>-</u> | Acetone | Common cleaning fluid Also present other hydro- | |
| 45 | - | - | Isopropyl Alcohol | Common cleaning fluid carbons so check cracking patterns. | |
| 44 | 28 | 10/1 | Carbon Dioxide | Common residual in UHV systems. | |
| 43 55 69 | 41 and 57 and 71 | 2/l Varies | Hydro- carbons | Caused by pump oils (diffusion and rotary) and many other hydrocarbons. Classic contamination. N.B. Characteristic groups with 14 AMU spacings = CH ₂ . | |

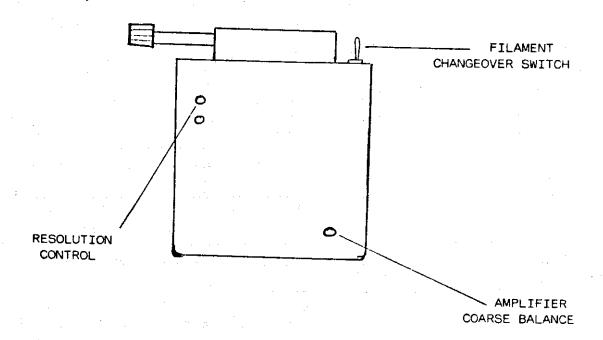
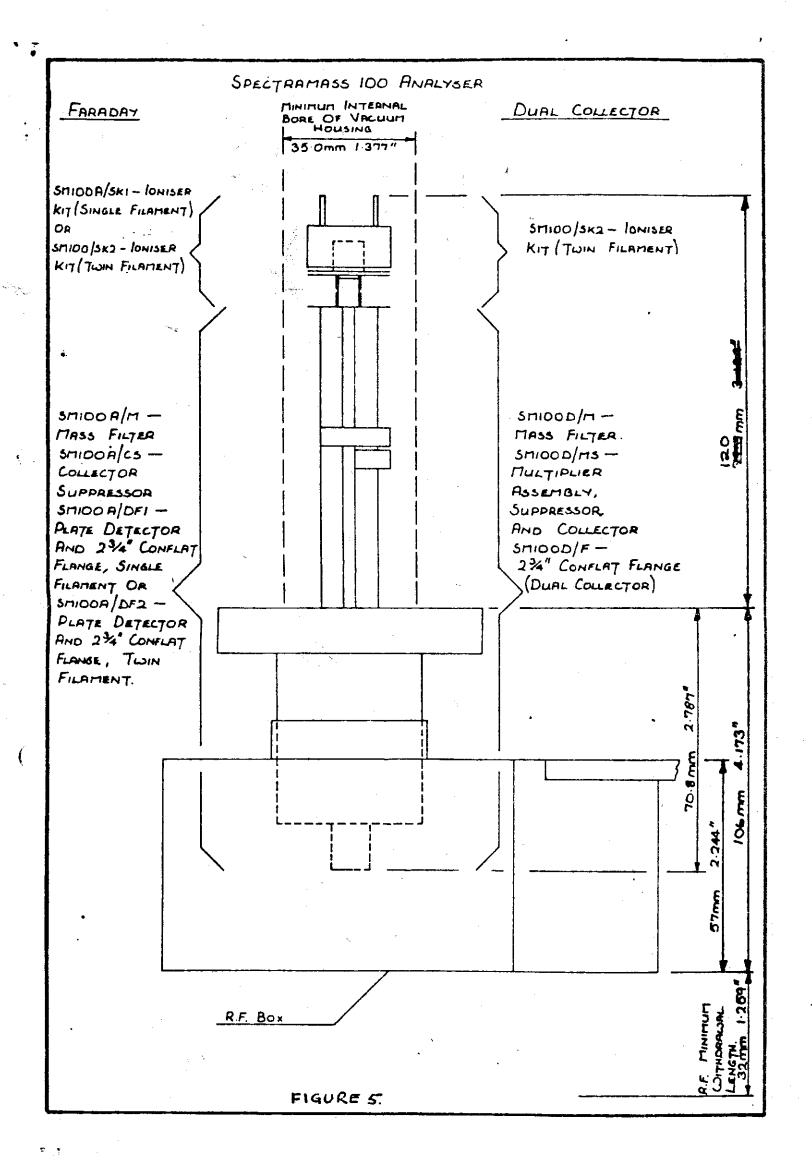
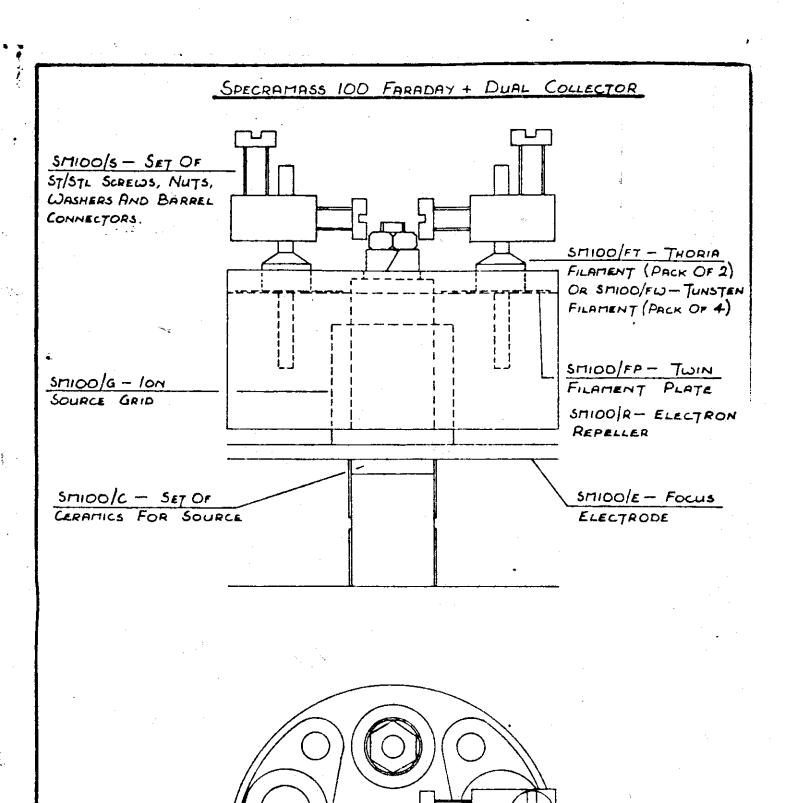
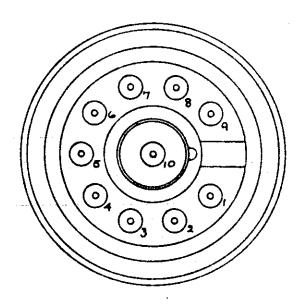


FIG.4 - VIEW OF TOP OF RF HEAD.





ANALYSER PIN CONFIGURATION



FARADAY

- SOURCE
- R.F.
- R.F.
- FILAMENT 2
- SUPPRESSOR
- ENTRANCE FOCUS

- N/C
- COLLECTOR

DUAL

- SOURCE
- R.F.
- R.F.
- FILAMENT 2
- SUPPRESSOR
- 6. ENTRANCE FOCUS, ELECTRON REPELLER
- FILAMENT COMMON (ELECTRON REPELLER) T. FILAMENT COMMON (ELECTRON REPELLER)
- FILAMENT 1
- 8. FILAMENT 1
- 9. MULTIPLIER H.T. SUPPLY
- 10. COLLECTOR

| SPECTRAMASS 100 | | | | |
|------------------------------------------------------------|--------------|--------------------------------------------|--|--|
| FARADAY | | DUAL COLLECTOR | | |
| SMIODA/SKI - TONISER KIT (SINGLE FILAMENT) OR SMIDDO/SK2 - | ## | SMIDO/SK2 - IONISER KIT | | |
| IONISER KIT (TWIN FILAMENT) | | (Wild I CALLEA) | | |
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| SMIDOR/M - MASS FILTER | | SMIDOD/M - MASS FILTER | | |
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| | | | | |
| SMIODA/CS - COLLECTOR SUPPRESSOR | | SMIOOD/MS - MULTIPUE RSSEMBLY, SUPPRESSOR | | |
| COLLECTOR SUPPRESSOR | | AND COLLECTOR. | | |
| | | | | |
| | | | | |
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| SMIDOR OFI - PLATE DETECTOR | | SMIDOD - 23/4" CONFLAT | | |
| AND 23/4" CONFLAT FLANGE, SINGLE FILAMENT OR SMIODAIDF2 | | FLANGE (DUAL COLLECTOR) | | |
| SINGLE FILAMENT OR SMIOOR DF2 - PLATE DETECTOR AND 23/4" | | | | |
| CONFLAT FLANGE, TWIN | | | | |

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