# OPERATING INSTRUCTIONS UV LAMP

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# OPERATING INSTRUCTIONS ULTRA VIOLET SOURCE

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## INSTALLING THE UV LAMP

## DESCRIPTION

The UV source is a gas discharge lamp in which the discharge is confined by a quartz capillary tube. External fan cooling may be employed if required on to a shielded copper cooling disc attached to the anode feedthrough. Two stage differential pumping is employed so that the analyser can remain under good vacuum whilst the lamp is operating.

The gas supplied to the lamp must be as pure as possible in order to obtain good results, particularly when using the He 11 line at 40.8 eV. It may take some time (several hours of operation) for the gas supply lines to become sufficiently clean for proper operation in a new installation.

The UV lamp power supply provides 5kV for starting the discharge and a 1kV current stabilised supply variable from 20 to 50mA to run the lamp. The lamp voltage is determined by the discharge characteristics of the lamp itself.

#### FITTING THE LAMP

The lamp is mounted on a FC38 flange. A bellows unit and three studs permits the lamp to be aligned for maximum signal.

FC919 flanges connect gas inlet, rough pumping and diffusion pumping lines. These are all copper gasket seals using 4mm cap-head bolts, nuts and washers. The rough vacuum differential pumping port can be identified by it's smaller diameter tubulation. Access to the quartz capillary tube can be obtained by removing the ceramic feedthrough flange and gas inlet flange from the lamp while the pumping lines remain untouched.

#### STARTING THE LAMP

Connect the lamp power supply to the sockets provided on the lamp and switch on the supply. As the regulator valve warms up, 1kV should be indicated on the voltmeter.

Open the valves to the differential pumping and admit 1 lb per square inch of lamp gas into the inlet valve feed line via a suitable regulator.

Slowly open the leak valve to admit gas to the lamp.

Observe the pressure in the lamp roughing line. The lamp should start when the pressure in the roughing line is about 0.5 mb. Once the lamps has started reduce the gas pressure to 0.1 mb in the roughing line. The leak valve should be adjusted very slowly since there is a considerable time lag before steady pressures are indicated due to the small pumping speeds through the capillaries. Allow the lamp to run for a minimum of 15 minutes to degas itself.

If after bakeout or a period of operation from the lamp is difficult to start, observe the lamp (if possible) and slowly vary the gas pressure until the lamp starts to flash at regular short intervals. Do not cool the lamp and ensure that the current control on the lamp supply is set for maximum.

The lamp will start if left for a short period under these conditions.

If there is still no sign of a discharge, it is possible that the initiating 5kV supply is low or faulty. Measure this potential with an electrostatic voltmeter since the circuit employs a voltage multiplier with limited current capability to produce this potential. The reading should be about 5.5kV with the supply disconnected from the lamp.

CARE SHOULD BE TAKEN IN MAKING THIS MEASUREMENT SINCE THE MAIN SUPPLY IS ALSO AT 1kV WITH 50mA CAPABILITY.

With the gas supply turned off this should be the potential on the anode unless leakage exists in the lamp electrodes.

If this is substantially lower than 1kV, then a spark coil should be applied to the anode with the normal supply disconnected and the gas flow turned on to the normal start pressure. A discharge should be seen to occur and when the supply is reconnected the lamp will start.

If no discharge can be observed then there is too great a build up of sputtered material and the lamp should be dis-assembled for cleaning of the quartz capillary tube or the fitting of a new one.

#### LAMP OPERATION

It is difficult to give precise figures of lamp pressures and voltages since the quality of the gas state of the lamp electrodes can be quite different from time to time and from system to system.

#### RUNNNING

The lamp is designed to operate as a HeI source at 21.21eV with high photon yield and long term stable operation.

In addition the NeI line at 16.8eV is even more readily obtained since Ne is more easily ionized.

Additional lines are always produced in the gas discharge but these are of relatively low intensity. However, by reduing the gas pressure the relative proportion of HeI to HeII and NeI to NeII can be changed and the lamp will generate usable photon yields of HeII at 40.8eV and NeII at 26.9eV.

Typical figures for operating normally as a HeI lamp are roughing line 0.5 to 0.1 mb diffusion pump lines at 2 to 5 x  $10^{-6}$  mb and the main chamber in the low  $10^{-8}$  mb range. As the gas pressure is reduced the lamp discharge will change colour from the yellowish pink to greenish blue, indicating that a significant yield of HeII is being obtained.

Pressure in this mode may be from 0.005 to 0.008 mb in the roughing line, 1 to  $4 \times 10^{-7}$  mb in the diffusion pump line and the analyser chamber from  $5 \times 10^{-9}$  mb to  $9 \times 10^{-9}$  mb. In the mode the lamp may be difficult to keep operating and any attempt to lower the current from 50mA may well result in the lamp extinguishing.

In general the lamp performs better after operating for a few hours as it cleans up and will run at a much lower pressure than it does initially.

### ALIGNMENT

The lamp body is mounted on a spherical joint with three studs which may be adjusted to direct the beam at the sample.

Adjustment is made on a trial and error basis by inserting an Allen key into a hole in one or other of the three nuts and using it as a lever to turn the nut. A clean evaporated film of silver is a suitable sample to set up on. The sharp Fermi edge on silver is also useful for calibration. The rising background observed when the lamp is misaligned may be attributed to secondary electron production when the photon beam strikes parts of the analyser electrostatic shield. When the sample is contaminated by carbon and oxygen containing outgassing produces, the valence bands of these elements interfere.

### SWITCHING THE LAMP OFF

Switch off the power supply with the lamp still running to discharge the power supply.

Close the MD6 leak valve to shut off the lamp gas supply. Close both lamp pumping valves CR38 and CR14.

UPS OPTION MANUAL

REV. 2

JUNE 14, 1990

# **UPS OPTION KIT- 18840**

INTRODUCTION: This kit describes a set of optional hardware and software that may be added to the SSI M-Probe ESCA product to enable it to perform ultra violet photoelectron spectroscopy in the energy range of 0 to 30 ev. The option involves adding a Helium plasma lamp with gas bottle fittings and cold trap along with the lamp power supply. The option is intended to operate with the 8724 Extended Range Power Supply, but the standard 8701B power supply may be readily modified for the application as well. Extreme care must be paid to the pumping/vacuum system, since surface contamination can substantially degrade the spectra of the desired component. Vacuums better then 1 x 10 -9 Torr are necessary for this application and one should preferably substitute a 550 liter/sec Turbo pump with a liquid nitrogen cooled titanium sublimator for the standard Cryo pump generally supplied with the ESCA system. A full system bakeout at 200°C is essential prior to performing UPS. Great care must be taken to insure that the vicinity of the sample area is demagnetized to a level below 100 milligauss. On the standard M-Probe the UPS pass energy is implemented into the "RESOLUTION 1" mode. To perform UPS spectroscopy it is necessary to have a modified software package as well, which allows for setting the V1 voltage into the required range and for proper labelling of the spectra. The retardation voltage is also programmed to scan in one direction only. The remaining three modes are set to 25, 50, & 100 ev. Other pass energies are optionally available. It is necessary to have an ion sputtering gun to clean up the sample surface prior to taking silver witness spectra. The performance specifications for UPS spectroscopy on silver are as follows.

# TABLE I -PERFORMANCE SPECIFICATIONS -UPS

Test Sample Silver Unscanned Count Rate\* >50 kcnts/sec on Val. Band <0.12 ev (20 to 80%) Fermi Edge Resolution Helium I line Radiation Source Pass energy 5 ev Kinetic Energy Range 0 to 35 ev  $< 10^{-9}$  Torr Pressure Required 350 to the lens axis Sample Angle 50 Volts DC Max. Sample Bias Voltage

\* Count Rate depends a great deal on the discharge lamp used, pressure, and sample surface conditions. These rates are cited for source inspection at SSI on SSI specified equipment.

# MATERIALS LIST: The following materials are required.

UV Lamp Source (VG model 22-101)
UV Lamp Power Supply (VG model 232)
Helium Gas (99.999%)
Argon Supply Gas (Lab grade)
Helium Filter & Cold Trap
Poly bucket (Thermos)
Software diskettes (3)

HP Vectra vs. 1.20 or

HP 310, 320 Basic vs. 8.51

Silver Specimen

8701B-UPS or 8724 Spectrometer Power Supply Vacuum Plumbing Hardware:

2.62 Conflat Tee

2.62 Conflat Nipple

1.33 Flexible UHV Hosing -12" (2)

1.33 Flexible UHV Hosing -30"

1.33 Conflat Tees (2)

1.33 Conflat Manual Valves (2)

1.33 UHV Conflat to 1" quick-fit adapter

1.00 quick-fit Tee

1.00 x 18" quick-fit flexible tubing (3)

Pirani Gauge (with controller)

Turbo Pump Mounting Bracket

Ion Gauge (with controller)

Turbo Molecular Pump (25 l/s)

Turbo Pump Controller

Mechanical Fore-pump

Gas Plumbing Items:

0.25" stainless steel tubing -6 ft.

0.25" gas valves with Swagelok fittings (2)

1.33" Conflat Leak valve

1.33" Conflat to .25" tubing adapter

**UPS Option Manual** 

# **INSTALLATION INSTRUCTIONS:**

- 1. Verify proper operation of the ESCA system prior to any installation. Correct any defects that prevent the system from meeting its performance specifications.
- 2. Turn power off and let the analytical chamber up to atomsphere in the usual manner after a 30 minute cool down waiting period..
- 3. Install the UV lamp into the right-most port (see figure 1) over the sample manipulator on the top hat.
- 4. Install the lamp's differential pumping hardware as shown in figure 1. Keep the run to the Turbo pump as short as possible (preferably to under 12").
- 5. Mount the Helium Cold trap and filter to the back of the ESCA system.
- Connect up the Helium gas bottle to the gas supply tube running to the cold trap-filter assembly using the .25 inch stainless tubing.
- 7. Connect the Lamp to the leak valve as shown in figure 1.
- 8. Connect the first of the two differential pumping lines to the lamp per figure 1. It is wise to insert a Tee in this line and connect an ion gauge in the line to monitor the lamp pressure.
- 9. Mount the Turbo-molecular pump within 12" of the lamp and connect it to the lamp through a 1.33" conflat manual valve.
- 10. Connect the second differential pump line between the mechanical pump and the rearmost pump port on the lamp, again through a 1.33" manual valve. A UHV tee must be installed between the mechanical pump and the Turbo pump to accomodate this line. It is also advisable to connect a Pirani gauge to this line to monitor the pressure.
- 11. Connect the insulated sample stub to pin H on the vacuum feedthrough. Connect up the sample bias supply and check for continuity to the sample stub.
- 12. Check all fittings and restore the vacuum.
- 13. In the usual manner re-verify that ESCA performs normally.
- 14. Replace the 8701 power supply with the 8701B-UPS (or with the 8724) power supply.
- 15. Load in the UPS software package in accordance with the software upgrade instructions.

OPERATOR INSTRUCTIONS: The UPS option is tuned to analyse electron kinetic energies from the sample that are in the range of 0 to ≈50 ev. These electrons are generated using the ultraviolet lamp which typically produces HeI radiation. To achieve the optimum energy resolution while maintaining reasonable count rates the spectrometer has been set up with 5 ev pass energy. The UPS software is a simple extension to the ESCA's software where the energy resolution 1 setting has been converted to the UPS 5 ev resolution. Resolution 2,3, & 4 are generally tuned for 25, 50, and 100 ev pass energies, unless the customer has requested a different set of values.

To obtain a UV spectrum introduce the sample into the analytical chamber. The sample should be mounted on an electrically isolated stub, which can be biased from an external 12 volt battery. Be careful to de-gauss the stub to a level below 100 milligauss. Connect the sample to the negative terminal of the 12 volt battery via pin H of the sample chamber's electrical feedthrough connector. Ground the other terminal of the battery to the chamber wall. Locate the sample in the center of the cross hairs of the Wilde-Heerbrugge microscope and adjust the sample's x-y-z position to bring the field of interest into best focus and aligned with the cross hairs.

Turn on the UV lamp in accordance with the manufacturer's instructions. Sometimes these lamps can be hard to "strike" and it is useful to introduce Argon gas flow if this is the case. If this is to be the first attempt at getting a UPS Spectra after installation of the UV Lamp it is advisable to use a silver sample first to aid in alignment of the lamp's discharge tube to the sample measurement location determined above (cross-hair). With the 8701B-UPS turned on, select the resolution 1 mode. Select a non-scanned mode and set the edge of the spectral window to 17 ev. It is now necessary to systematically adjust the "steering" angle of the lamp by means of the adjustment screws at the base of the lamp by the conflat flange to bring the illuminated spot into coincidence with the measurement location. The UV spot will not be visible to the eye. The lamp's angular alignment must be adjusted to achieve best count rate. Check that the 8701B (or 8724) voltages are adjusted to those recorded in Table II. Next sputter-clean the surface of the sample using a high purity gas source to remove surface contamination. Reset the edge of the spectral window to approximately a Binding Energy of 6 ev and accumulate a spectrum in the unscanned mode.

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Adjust the lamp position to give a detector count rate (Silver valence band) of  $\approx$  60,000 counts/sec.

# TABLE II -TYPICAL 8701B-UPS SETUP VOLTAGES

## VOLTAGES FOR THE RESOLUTION 1 -5 EV PASS ENERGY MODE.

V2	=	30.1		Second lens voltage			
VO	=	17.5		Retardation			
VQ	=	20.7		Quadrupole voltage			
V1	=	9.1		Focus Voltage			
VTRIM	=	18.8		Sphere Trim Voltage			
V+	=	15.0		Sphere			
V -	=	20.0		Sphere			
<b>IMAG</b>	=	26.0	ma	Magnet			
B.E.	=	6.5	volts				
VACC = 12.4 volts (sample bias voltage)							
UPS REF = 30.0 (COMPUTER ENTRY)							

To take an actual spectra set up the spectral scan range using the menu tree's ACCUMULATION, SINGLE SCAN selection. Enter UPS in the spot size location (see figure 2). Complete the table entries to scan over the desired region under the conditions of interest. Figure 2 shows the conditions for an ideal scan on silver. To initiate a scan depress the accumulate or alternative soft key to start acquisition (refer to your ESCA operator's manual). The spectral scan for UPS spectroscopy is unidirectional, scanning from low binding energy to high binding energy only. The UPS binding energy "zero" can be adjusted in the table of parameters. Starting at the top menu, select the INSTRUMENT CONTROL soft key followed by INSTRUMENT PARAMETERS. Adjust the UPS binding energy zero by selecting the SET UPS REFERENCE soft key and type in the value desired. (the instrument is factory preset to 30 volts). Figures 3 & 4 show a typical spectrum displayed at two different scale factors. Figure 5 shows the silver spectrum greatly expanded around the Fermi edge. The typical method of measuring the Fermi edge resolution is shown in figure 6. The conventional definition for the Fermi edge resolution is taken to be the 80% to 20% spread as shown in figure 6 (sometimes other definitions are used such as the 10-90% width, the 14 to 86% width etc.). Figure 7 shows a typical instrument "system parameters" set-up for reference.

# **SUPPLEMENTARY DATA:**

Sphere Supply 060050-UPS Schematic Schematic Magnet Supply 060080-UPS 060060-UPS Schematic Offset Supply

ESCA 8.5 Software Release Notes ESCA Vectra v. 1.2 Release Notes

Lamp Data Sheet

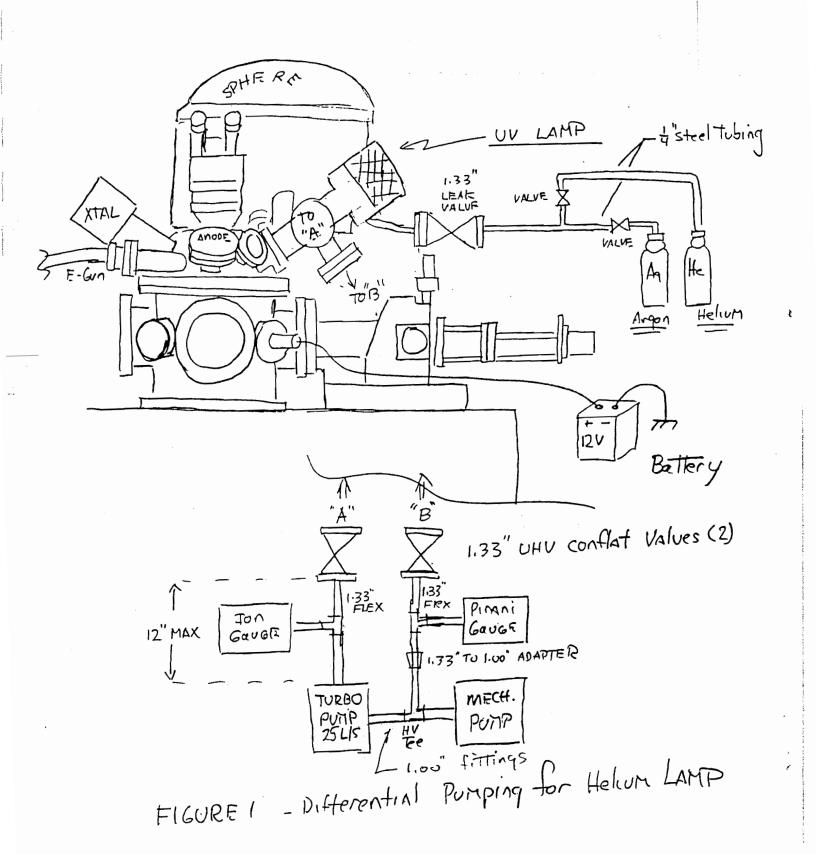


Figure 2- Commands for Initiating UPS

# SINGLE SCAN

File name:	SINGL_SCAN			
Description:	UPS SPECTRUM			
Operator:	MK			
# of groups:	2			
Lower BE:	0.0			
Scan width:	25			
# of scans:	1			
Flood gun:	0.0			
Resolution:	1			
Spot size:	UPS			
Aperture:	None			

START ACCUM :

: QUICK SCAN |FAST STRT SCAN: DISP SPECTRUM
: | \*PREVIOUS MENU \*

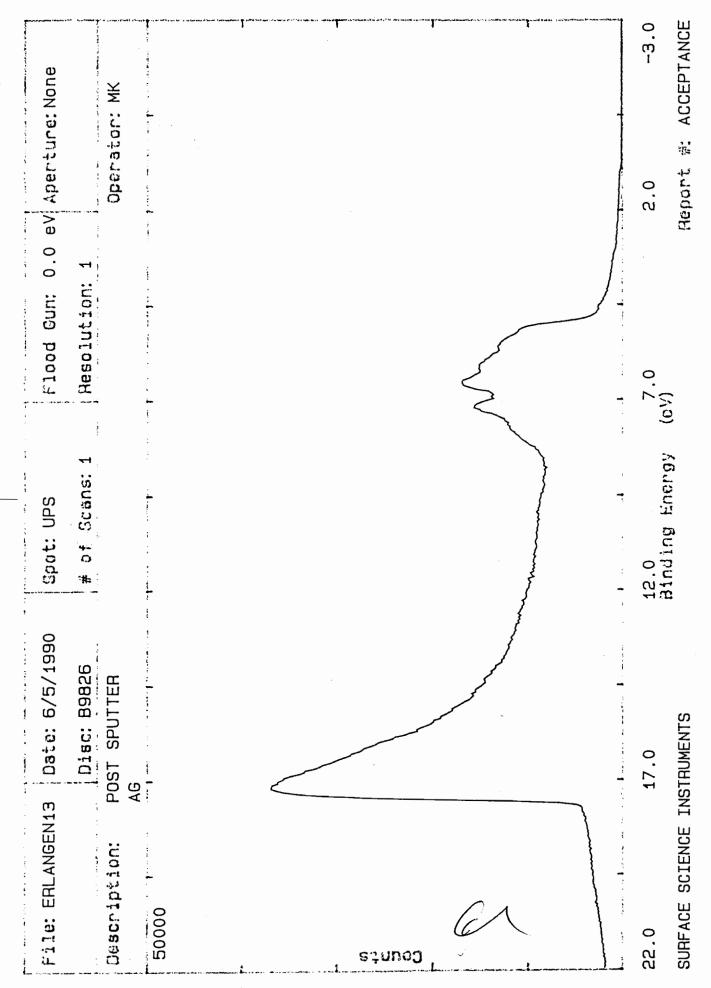
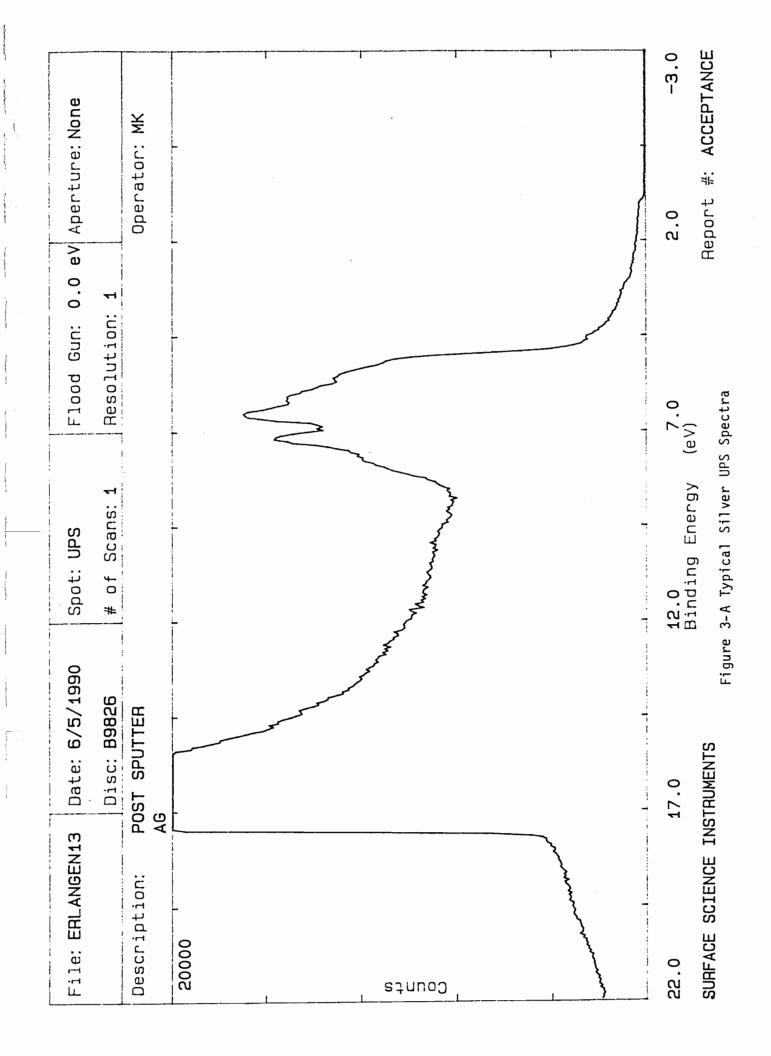
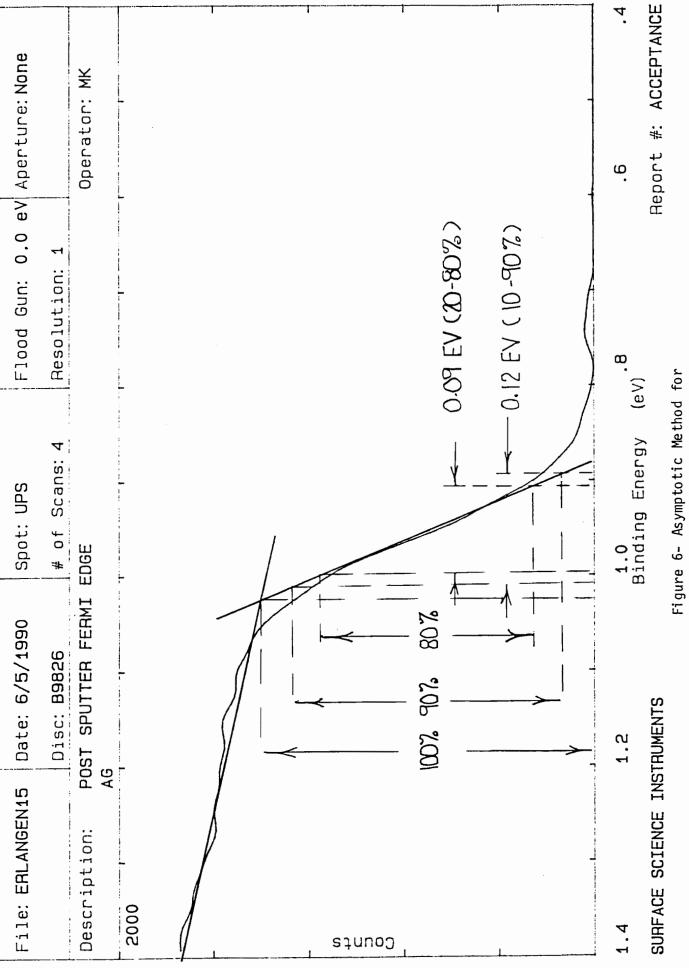


Figure 4- Rescaled UPS Spectra



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Figure 5- Expansion of the Silver UPS Spectrum About the Fermi Edge.



ure 6- Asymptotic Method for Measuring Resolution

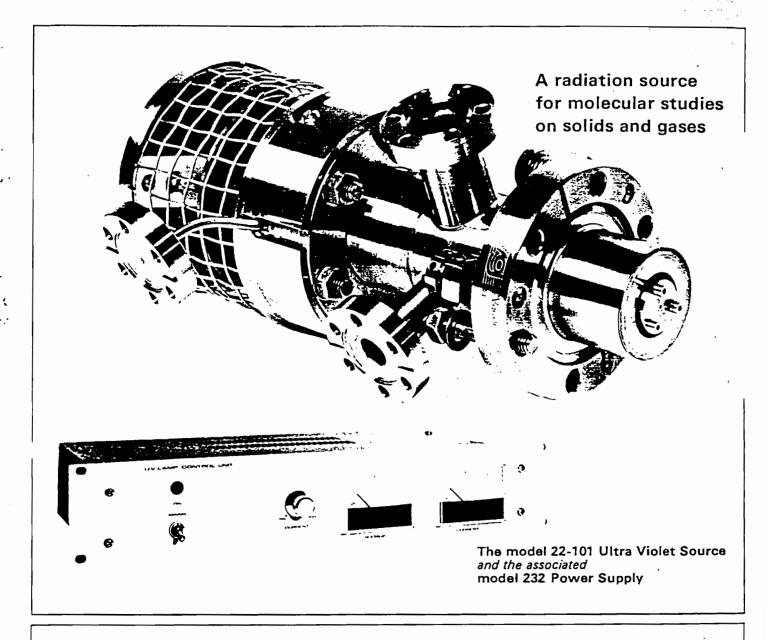
# Figure 7- Typical Systems Parameters for A UPS Modified ESCA.

Ref Energy, Mono (eV): Ref Energy, Non-mono (eV): DAC Calibration: Sens. Factor Exponent:	1100.00	Non- Dete	-mono To	arget: ype:	LINE/SPOT Mg FAST (2412-5) Left: 0 Rig	ht: 0
Resolution:		2	3			
Detector Widths (eV):	.81	3.55	7.11	13.98		
Pass Energies (eV):	5.00	24.51	50.00	100.90		
V1 offsets:	9.50	66.01	179.45	234.00		
V1 slopes:	0.00	.49	.59	.51		
Spot Size:	150 u	300	u	200×750u	400×1000u	
Binding Energy Offset (eV):	0.00	Ø.0	9Ø	0.00	0.00	
Aperture Settings:	inner	ou t	er			
Apenture Type:	7?????	7??	?			
Ref Energy, UPS (eU):	30.00					

SET REF ENERGY SET SPOT TYPE : SET NON-MONO SET DAG FACTOR SET SENS EXPON SET DETECTOR SET SIZ OFFSET: SET APERTURE : SET REF UPS : \*PREVIOUS MENU

# VG SCIENTIFIC

THIS LAWR IS NO CONGER Recommended. Model 22-101
Ultra
Violet
Source



# Features of the model 22-101 U-V Source

■ Produces NeI – 1

NeI -16.8eV(\1\nm)

HeI - 21-2eV

NeII - 26·9eV

HeII - 40.8eV

- 3° Tilt for alignment
- Double differential pumping \*
- Self-starting cold-cathode discharge
- Fully demountable discharge chamber

- Compact construction 70mm OD flange mounting
- Fully bakable to 200°C
- High intensity windowless design
- Air-cooled anode fan provided
- Provision for user fitment of filter
- Design service for custom installations

The model 22-101 ultra violet source was originally developed for use on the VG Scientific ESCA3 Photoelectron Spectrometer, large numbers of which are in use throughout the world.

The source is now available as an accessory for work on both solids and gases. It is connected to the system via a 2%" OD standard copper gasket flange and is provided with two 1.33" OD miniature copper gasket sealed flanged ports, to which the differential pumping system is attached. The discharge conditions can be adjusted to optimise the intensity of HeI to HeII or NeI to NeII and useful intensities of HeIII have been reported by users.

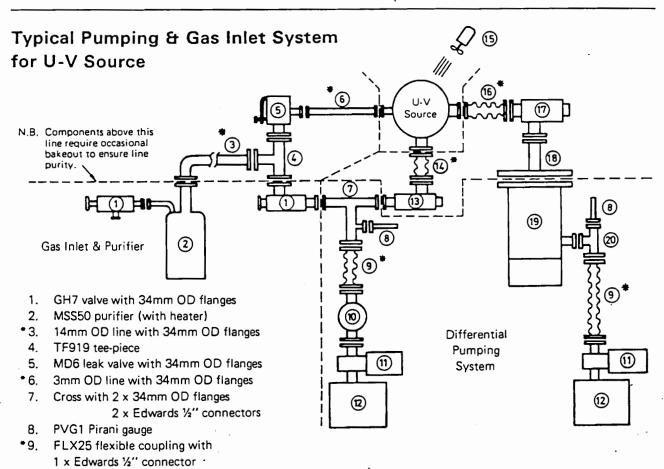
The lamp is windowless and, with the correct pumping system, can be operated with a spectrometer vessel pressure less than  $5 \times 10^{-9}$  torr for HeI and less than  $10^{-9}$  torr for HeII.

The model 232 power supply provides a 5kV current limited voltage for start up and a 1kV 20 to 50mA current stabilised supply to maintain the discharge.

Each lamp is individually tested to a specified photoelectron count rate and its performance, when fitted to an ESCA3 or other complete instrument manufactured by VG Scientific, is guaranteed. The effect of instrumental configuration is such that it is not possible to guarantee precise performance when the model 22-101 source is supplied as a component.

The pumping requirements of the U-V source are illustrated below. The use of u.h.v. compatible components (with occasional bakeout) and a gas purification system minimises the level of contaminant gases in the discharge region thus maintaining spectral purity.

Components available from VG Scientific in standard form are listed — those marked with an asterisk can be supplied to dimensions specified by you.



- \*16. FLX25 flexible coupling with 1 x 34mm OD flange
  - 1 x 70mm OD flange
  - 17. CR38 valve
  - FC38 to Edwards EO2 inlet adaptor
  - Edwards EO2 diffusion pump fitted with CB63 baffle
  - 20. Edwards 1/2" tee-piece

- RT1 sorption trap
- Edwards ½" magnetic isolation valve with
   ½"/1" adaptor
- 12. Edwards ED50 rotary pump

1 x Edwards 1" connector

- 13. CR14 valve
- 14. FLX919 flexible connector with 34mm flanges
- Cooling fan