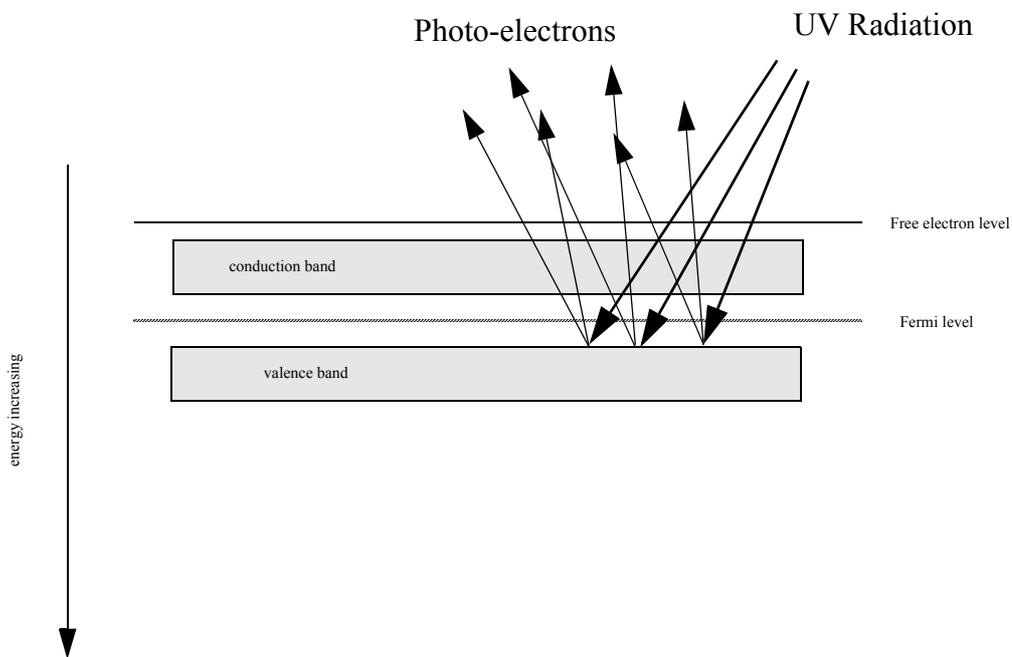




AXIS



UPS System OPERATING MANUAL



UPS System
OPERATING MANUAL

Ultraviolet Photoelectron Spectroscopy System

Operating Manual

© Kratos Analytical Ltd., 2002

Catalogue Number 39-287

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SAFETY NOTICE

Operation Warning

Kratos Analytical continuously monitors all safety aspects involved with the operation of its equipment. As with all complex instruments, operator safety is dependent on correct and sensible use of all the systems. Provided that the instructions set out in the various sections of this Manual are followed, and the general warnings set out below are heeded, any AXIS can be properly operated without significant danger.

For older systems with manual control of the High Voltage power supply please take note of the following.

Please ensure that the High Voltage Supply is reduced to 0kV before selecting the U.V. source mode from the emission stabiliser unit. This will prevent arcing during the switch over and prolong the lifetime of the High Voltage changeover relay which is located within the U.V. source supply

High Voltage Warning

In common with many other sophisticated instruments, potentially lethal voltages are present on various parts of this equipment. Consequently, the instrument must never be operated with any of the safety covers removed. If access is required to any part of the equipment normally protected by safety covers, the instrument must be isolated from the mains supply before the covers are removed. Many of the instrument functions are computer controlled. Do not assume that a voltage indicated by the computer, particularly zero volts, actually appears at any point.

Always check!.

The UV Source and supply unit are operated at High Voltages, typically 6kV d.c. Before any service work is carried out on the system ensure that the High Voltage supply is switched off and isolated from the U.V. Source supply unit.

Radiation Hazard

An intense flux of UV-radiation is generated from the Kratos UV source and continued direct exposure at close proximity is harmful to the skin and to eyes. The Kratos U.V. lamp incorporates a U.V. filtering viewing glass window which reduces the strength of the UV light to a safe level enabling direct eye inspection of the UV lamp discharge.

However, if any modifications are undertaken by the user, the need to maintain an adequate level of shielding must be borne in mind.

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1.1 Introduction.

The accessory allows the AXIS electron spectrometer to be used for ultra-violet photoelectron spectroscopy (UPS) of solids. It consists of a double differentially pumped ultra-violet light source compatible with UHV, appropriate pumping lines and gas admission systems and suitable provision for a discharge voltage to be applied to the source. In addition a divider network to extend the voltage range of the analyser and lens supplies is provided.

The lamp can produce He(I) and He(II) resonance lines when fed with pure helium gas. Other gases can be used to provide other UV resonance lines (e.g. Ne(I), Ne(II)) as required. The lamp does not require forced cooling and typically runs at a power of 20 - 25 Watts.

Provision is made for biasing suitable specimens. Insulating specimens can generally be run with the aid of the spectrometer charge neutraliser.

The various components of the accessory are described and full running conditions for the source are given.

The acquisition conditions for both He(I) and He(II) spectra are explained in detail as are the optimum running conditions of the source for producing He(I) and He(II) UV radiations.

Numerous examples of spectra from metals and insulators are given.

In addition there are details on cleaning the source and as well as a section describing the origin and nomenclature of UV spectral lines.

2.1 A Brief Introduction to the Components

The UPS accessory is made up of the following parts. A UV source, a suitable pumping system for the source and for the admission of helium gas (or other gases if desired), some means of providing a discharge voltage to the source and, if not provided, a suitable upgrade to the software for operating the UV source and obtaining UPS spectra.

2.2 The UV Source (Sometimes Referred to as the UV Lamp)

The UV source is a differentially pumped high voltage discharge chamber. Helium, or another suitable gas, is fed into the discharge region where a high voltage initiates and maintains a discharge. The discharge produces excited states of helium atoms and helium ions and the de-excitation of these excited states produces various UV lines of which the strongest is the He(I) series of resonance lines followed by the He(II) series of resonance lines. The He(I) lines originate from excited atoms of helium whilst the He(II) lines originate from excited singly charged helium ions.

Details of how the lines originate and their nomenclature is given in “Origin and nomenclature of Helium Resonance Lines” on page 47.

A partially collimated beam of the UV radiation is directed at the specimen by means of a capillary system. The capillary also forms part of the differential pumping of the source.

A schematic cross-section of the UV source is given in Figure 1.

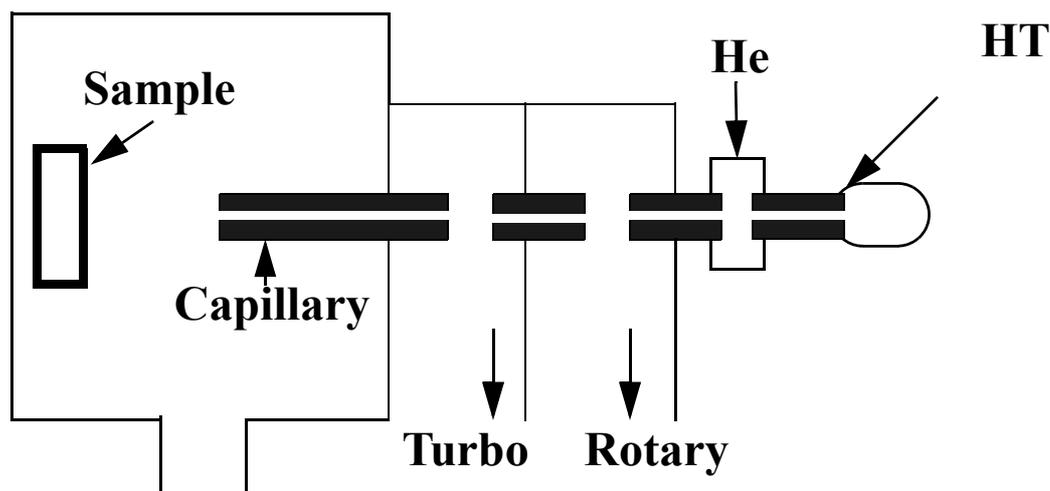


Figure 1. UV lamp schematic

2.3 Pumping and Gas Admission Systems.

The gas handling and vacuum schematic of the UV source configured into a standard Axis HSi, Axis Ultra or Axis 165 fitted with an STC (sample treatment chamber) pumped with a 160 l/s or faster turbomolecular pump is shown in Figure 2. In this system the fine differential pumping for the UV source is provided by the Turbomolecular pump used to pump the STC. As will be seen in the operating section of this manual care has to be taken to override the computer controlled vacuum system.

It may well be that a specially designed system has been configured. Figure 3. shows a preferred system in which the differential pumping turbo pump is separate from the pumping of the STC.

In the standard configuration the UV lamp is connected to the Sample Analysis Chamber (SAC) by a 70mm conflat flange. The lamp has two stages of differential pumping., the first stage is rough pumped and the second stage fine pumped via the STC/lock turbo pump. This ensures that most of the source gas is taken away and minimises gas flow into the SAC thus maintaining a good base pressure.

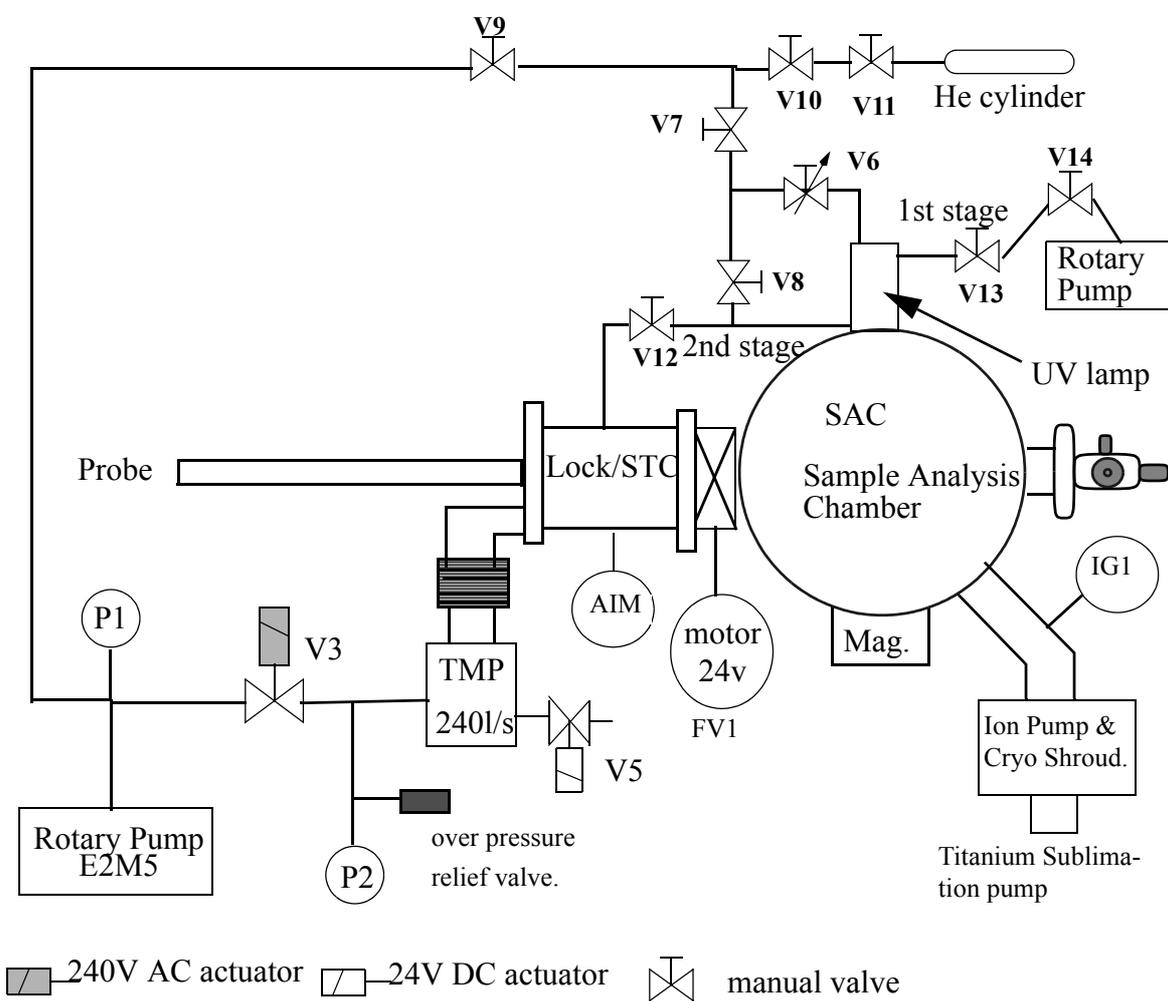
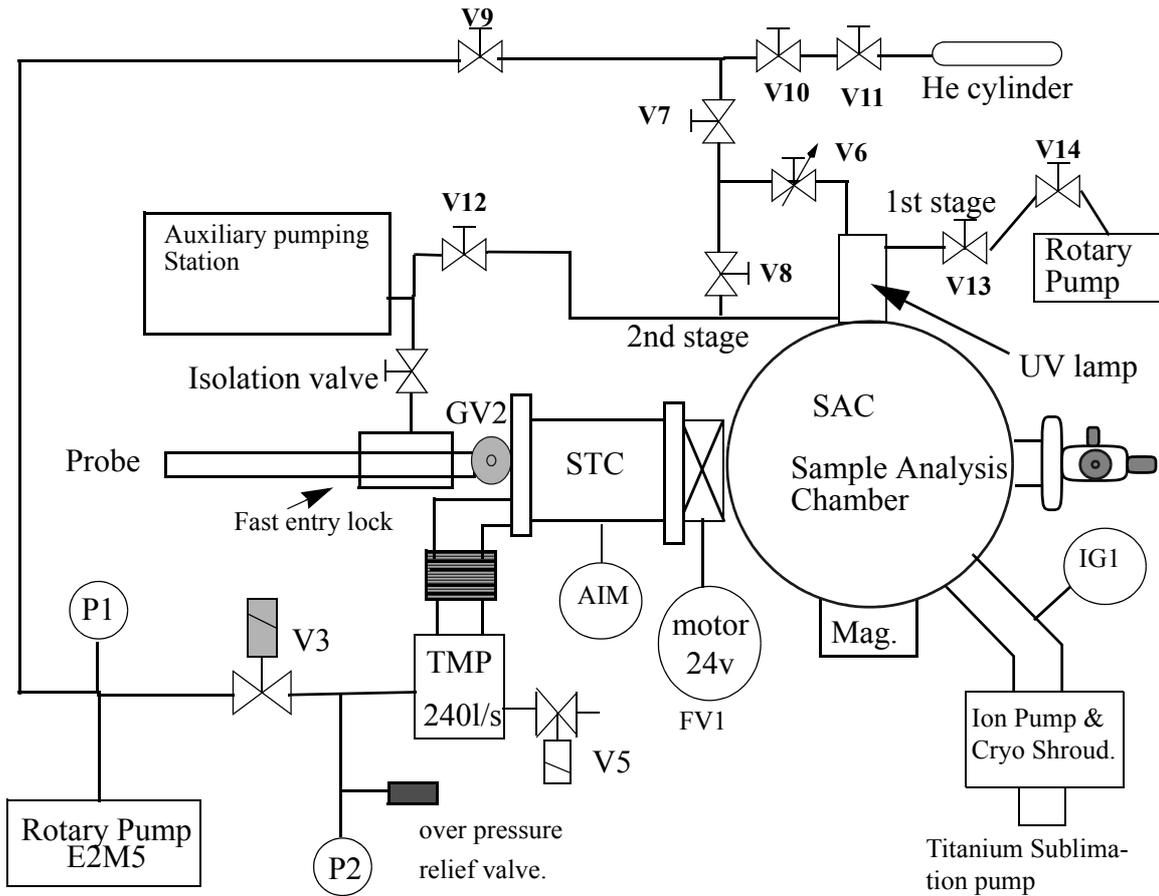


Figure 2. AXIS Ultra/165/HSi Pumping Configuration.



 240V AC actuator
  24V DC actuator
  manual valve

Figure 3. A specially configured vacuum system for the UPS accessory.

The valving of the gas admittance system allows the helium line to be evacuated before the helium gas is admitted to it. Repetitive evacuation and filling ensures that the lamp is provided with pure dry helium.

When the source is first installed the source and the evacuated helium lines will be baked with heater tapes (provided) to ensure that no volatiles are retained by the walls of the stainless steel gas introduction system.

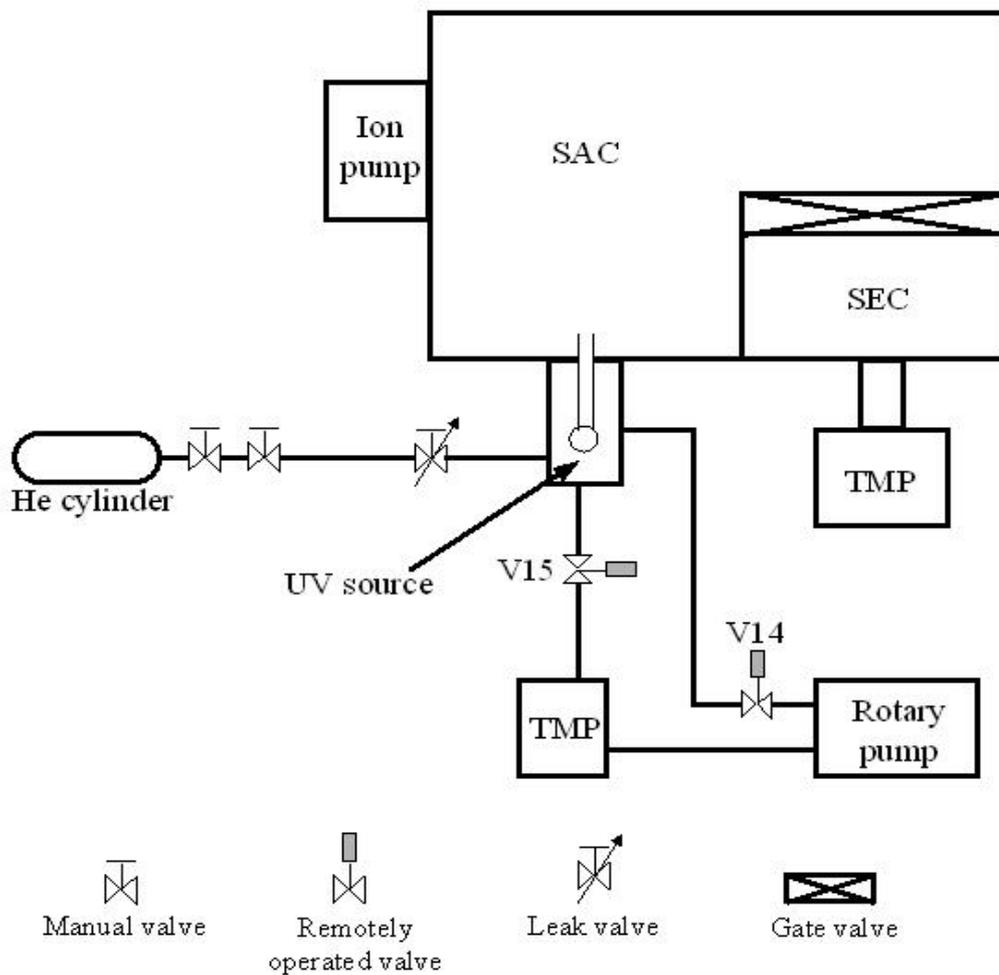


Figure 4. NOVA vacuum system configuration for the UPS source

The vacuum system of the NOVA is different to the other AXIS machines. The UPS source is attached directly to the SAC. The UV source is differentially pumped by a second turbo pump incorporated for this purpose. This pumping system also allows the

gas lines to be flushed with helium to remove all other gases after the gas line has been connected (or reconnected).

2.4 Power Supplies for the UV Source

The Kratos UV source is invariably fitted as an accessory to an existing XPS electron spectrometer. In order to provide the accessory at an economical cost the power for the source is derived from the X-ray EHT unit.

The most recent series of Kratos photoelectron spectrometers (the Axis HSi, the Axis 165 and the Axis Ultra) are fitted with a fully computer controlled unit manufactured by Start Spellman. This unit is fitted with an output which can be used to directly drive the UV source. The source has to be run in a current limited mode and this will be discussed in the appropriate section. The Start Spellman unit is controlled from the work station and the voltage and current limit to the source are set via the station.

Where the UV accessory is fitted to an earlier Kratos or other photoelectron spectrometer then a high power series resistance network is placed in series with the source to control the current through it. This high power series resistance network is provided in an enclosure which also contains a high voltage switch. This series network has to be used in conjunction with an emission stabiliser - which is standard with the Kratos series of electron spectrometers. The high voltage units used for driving the X-ray source (and hence the UV source) are usually those manufactured by Glassman or Wallis.

The different configurations are shown schematically in Figure 6. Option 1 shows the preferred option - the Start Spellman unit, option 2 shows the use of the series resistor network used in conjunction with the X-ray supply and the emission stabiliser. Option 3 in Figure 6. covers all third party UV lamp power supplies where the customer has provided his own UV source and/or his own supply to the source. Other sources and supplies will be briefly be mentioned in section 2.8 on page 11.

2.5 NOVA UV source control unit

The NOVA uses a different PSU to the other axis spectrometers. It has a dedicated PSU to control the UV source. The HV supply to the UV source is controlled automatically from within the unit. The emission is controlled via a dial on the front of the unit.



Figure 5. NOVA UV source control unit.

2.6 The “Divide by Ten” Unit.

X-ray photoelectron spectrometers are generally designed to operate up to 1500eV (with an $AlK\alpha$ X-ray monochromator) or up to 3000eV or greater (for $AgL\alpha$ X-radiation or electron excited Auger electron spectroscopy (AES)). This means that analyser and lens supplies have to operate up to these voltages or even higher. Considering that such high voltages have to be stable and that small step sizes are necessary for obtaining viable spectra it will be appreciated that obtaining stable low voltages (<5 volts) with a 25mV step size presents a considerable design problem. One way of overcoming these not insignificant difficulties is to take higher voltages and to “divide them by ten” with a suitable resistor potentiometric divider. Thus if a voltage of 5 volts is required to drive a lens element in UPS mode then the software will operate the supply to give an output of 50 volts which will then be reduced to 5 volts by the “divide by ten” unit. The unit is shown schematically as part of Figure 6. configured for the AXIS Ultra. Other AXIS products are very similar with only the lens labels being different.

The divider is switched in and out whenever the UPS lens mode is selected and the currently selected technique is XPS. It is done automatically under computer control. The divide by ten unit also contains a variable (manually set via a potentiometer on the rear panel of the unit) bias supply of up to -10 volts (nominal) which can be applied to conducting specimens to cause a shift upward in kinetic energy in the spectrum if required. This is particularly useful if it is wished to study the onset of the secondary electron cascade where electrons leaving the solid have near zero energy.

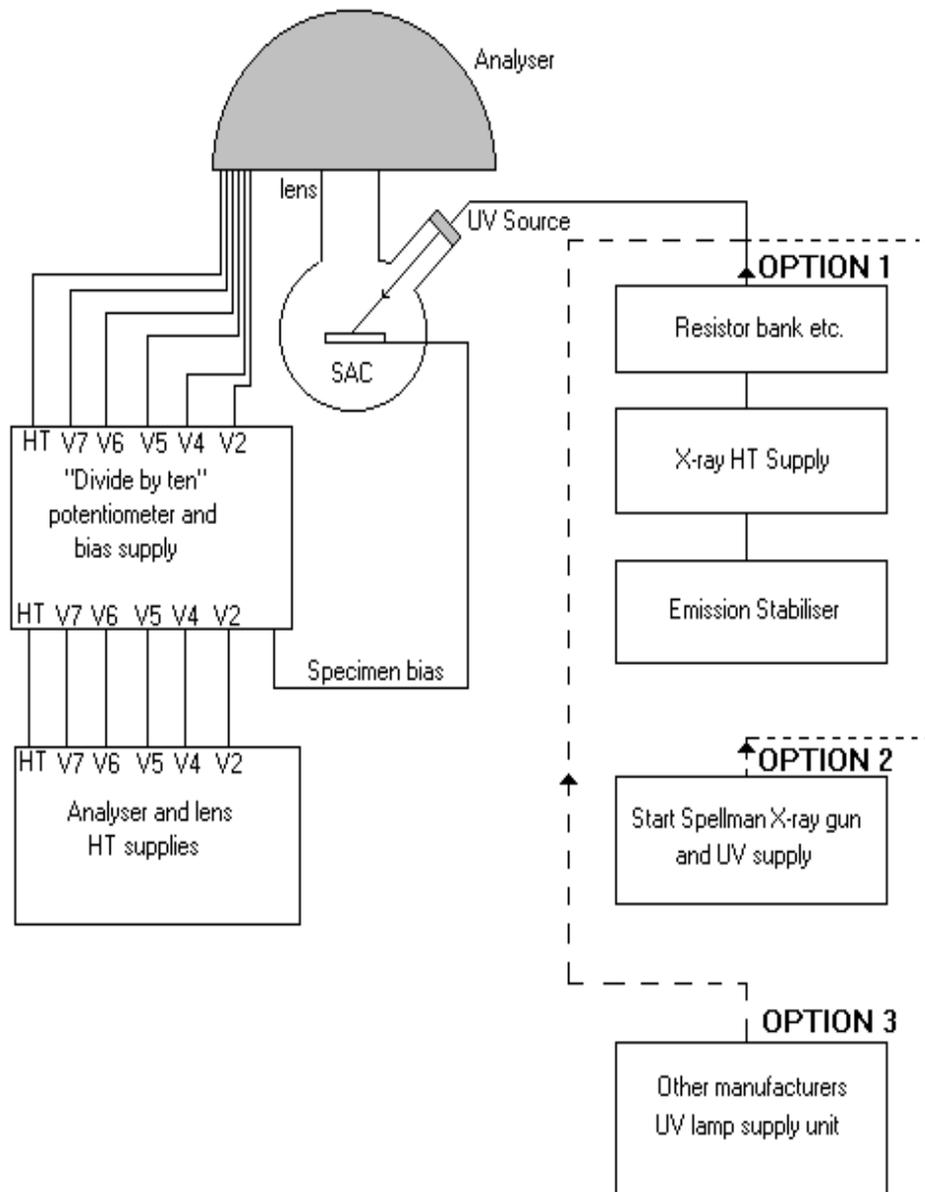


Figure 6. Arrangement of Divide by 10 Unit and Power Supply Options

2.7 Software

The most recent of Kratos electron spectrometers will be fitted with “Vision2” software which should contain the necessary software to run the UPS accessory. All that is required is that the configuration file is accessed and the appropriate units etc. activated.

For customers with earlier software Kratos engineers should be consulted at the time of ordering or purchasing of the accessory to ensure that the software is adequate for UPS work or if any upgrading is necessary

2.8 Other UV Sources and Supplies.

Where another manufacturers UV source and/or supply is fitted to a Kratos spectrometer care should be taken to ensure that it is adequately pumped and that the fitting of it will not compromise the performance of the spectrometer. A Kratos engineer should be consulted when it is proposed to fit such as system.

Kratos can accept no responsibility for the performance and lifetime of the source supply. The appropriate manufacturers literature should be followed at all times. See also the notes on the high count rates encountered in UPS work - see section 4.5 on page 26.

If the UV accessory is to function correctly it is essential that the pumping system is efficient, is clean, and is easily maintained in a clean condition. Similarly it is necessary to ensure that a pure gas is delivered to the source. The recommended operation of the pumping system and the gas admittance system is described in this section of the manual.

3.1 Overview of the standard vacuum system

The system has been shown schematically in Figure 2. Two stages of differential pumping are provided - the fine pumping stage utilising the STC turbo pump, the rough pumping stage utilising a trapped rotary pump (provided). The gas lines from the regulator to the leak valve can be roughed out with the main instrument rotary pump and then pumped out with a fine vacuum opening the leak valve, but having V10 and V7 (a shut off valve so that the fine vacuum does not “see” the regulator) shut - the valve configuration for fine pumping the gas lines will be given in the baking section.

The helium gas should be very pure - high purity laboratory grade (99.999% purity). The regulator should be free of greases and other volatiles.

3.2 Overview of the NOVA pumping system

While the NOVA has a different pumping arrangement to that shown in Figure 2, the principle is the same. The UPS source is provided with two stage differential pumping. Rough pumping via a trapped rotary pump, and fine pumping via dedicated turbo pump. The lines from the regulator to the UV source can be pumped through the UV source roughly with the rotary pump first and then with the turbo pump. The pumping for the UV source on a NOVA is shown in Figure 4.

3.3 Nova vacuum control unit

The UPS vacuum control menu on the vacuum control unit can be accessed from the main menu, see Figure 7. First press the ‘Custom’ button, this takes you to a screen that

looks like Figure 8. If the 'UPS' option is then chosen the 'UPS control menu' is displayed, see Figure 9. Please note that all vacuum gauges were turned off when the photos for these figures were taken, this is not normally the case.



Figure 7. Vacuum control unit 'Main menu'



Figure 8. Vacuum control unit 'CE options menu'



Figure 9. Vacuum control unit 'UPS control menu'

3.4 Baking the source and the gas lines

On a new installation the source and the gas lines should be baked out using heater tapes and the main spectrometer should be baked at the same time.

The sequence for preparing the system for baking is as follows:

1. Ensure gas bottle is connected up and connections are tight.
2. Open fine pumping valve V12 if not already open
3. Bring up the vacuum control window in Vision and close the turbo backing valve V3.
4. With valves V6, V10, V12, and V13 closed, manually open the UPS roughing valve V9 located near the gas inlets to rough pump the gas lines.
5. Monitor the pressure on the ion gauge control unit for Pirani P1. When the pressure is better than 10-2 torr open valve V7 and wait until the pressure on P1 is better than 10-2 torr again.
6. Close the roughing valve V9.
7. From Vision re-open the turbo backing valve.

8. Open gas line fine pumping valve V8 and allow the gas line to fine pump for about 20 minutes or until the STC/lock pressure is seen to be better than 10^{-7} torr under these conditions.
9. Open the leak valve fully.
10. The baking can now be turned on for both the UPS system and for the whole instrument.

3.5 NOVA - Baking the source and gas lines

The UV source should be baked whenever the chamber is baked. However it is not necessary to bake the gas supply lines unless they have been exposed to air or otherwise become contaminated.

If the gas lines are not to be baked then the UPS source should be left under fine pumping via the dedicated turbo pump. To achieve this the UV source leak valve should be closed, V14 should be closed and V15 open.

If the UPS gas supply lines require baking then the procedure described below should be followed.

WARNING - This procedure will expose the SEC and SAC to the contamination that is in the gas supply lines.

The SEC/SAC valve must be open and both chambers should be under vacuum.

1. Close valve V15 (fine pumping).
2. Open valve V14 (rough pumping).
3. Monitor the UPS backing pressure via the UPS vacuum gauge reading. Wait for the UPS pressure to recover to less than 5×10^{-2} torr.
4. Slowly open the leak valve to allow the UPS gas lines to be rough pumped. Ensure that the SAC chamber pressure does not rise above 10^{-4} torr.
5. Once the leak valve is fully open, close V14 and open V15.

3.6 Procedure prior to admitting the gas (flushing the lines)

This follows the above procedure (section 3.4) very closely until the last steps.

1. Ensure gas bottle is connected up and connections are tight.
2. Open fine pumping valve V12 if not already open
3. Bring up the vacuum control window in Vision and close the turbo backing valve.
4. With valves V6, V10, V12, and V13 closed, manually open the UPS roughing valve V9 located near the gas inlets.
5. Monitor the pressure on the ion gauge control unit for Pirani P1. When the pressure is better than 10⁻² torr open valve V7 and wait until the pressure on P1 is better than 10⁻² torr again.
6. Close the roughing valve V9.
7. From Vision re-open the turbo backing valve.
8. Open gas line fine pumping valve V8 and allow the gas line to fine pump for about 20 minutes or until the STC/lock pressure is seen to be better than 10⁻⁷torr under these conditions.
9. Open the roughing valve V13 whilst monitoring the STC pressure. The pressure is expected to rise to about 10⁻⁶ torr.
10. Open the shut off valve to the gas supply and admit the gas.
11. It may be necessary to repeat the above procedure more than once to ensure the gas line is fully purged and free from contamination.

3.7 NOVA - Flushing the lines

1. Follow the entire procedure described in section 3.5 for the NOVA
2. Allow the gas lines to pump under fine vacuum for at least 20 min.
3. Close the leak valve.
4. Close valve V15
5. Open valve V14
6. Fill gas line from regulator to leak valve with gas.
7. Slowly open the leak valve to allow the UPS gas lines to be rough pumped. Ensure that the SAC chamber pressure does not rise above 10⁻⁴ torr.
5. Once the leak valve is fully open, close V14 and open V15.
6. It may be necessary to repeat the above procedure (from step 2) more than once to ensure the gas line is fully purged and free from contamination.

Pumping, baking and cleaning the source

In this section there will be a small degree of overlap with the previous section - principally concerning starting and optimising the source. Once the source is running then details are given of suitable acquisition conditions for the appropriate UPS spectra.

Silver metal is used as a sample in most of the examples as this sample is widely used in XPS for sensitivity and calibration checks.

Obtaining spectra from insulating samples will be described in the latter part of this section.

4.1 Setting up the UV Source Running Conditions

It is assumed that the source is being differentially pumped and that the leak valve has not been opened although the helium lines will be full of helium at regulator pressure.

On the *manual* menu (Figure 10.) set the X-ray gun panel so that the UV source (Anode:UV) is selected - if the voltage has been set at a value greater than 6kV then the workstation will give an audible alarm and a visible “cross” on the voltage window. This serves to remind the operator that the voltage should be set at 6kV when the UV source is selected. Having set the Anode HT to 6kV the current can then be set to 20mA. The source can be switched “on” - in the UV condition there is no need to go to the “standby” condition. It is important when running the system in UV mode that the Start Spellman EHT unit is set in the current limited mode rather than the voltage limited mode which is used in XPS. The middle button on the left hand side of the Start Spellman unit front panel should be in the “in” position and illuminated when in the current limited mode.

The leak valve on the source can now be carefully opened - as the pressure increases then the lamp will attempt to strike. Initially it will “flicker” - oscillating between on and off. As the pressure is further increased the lamp will stabilise and give a steady output. The current shown in the current read back will stabilise at 20mA whilst the

voltage read back will drop to $\sim 0.5\text{kV}$ (~ 1 to 2kV with later lamps incorporating a larger ballast resistor in the lamp). It may be possible to turn back the leak valve a little way without the lamp extinguishing. If the lamp has been run previously with helium then it will give a “peachy” coloured discharge almost immediately. If it is the first time that the lamp has been run for a while then the discharge may be more “bluish white” due to impurities from the lines and from outgassing components in the source itself.

ENSURE THAT THE UV SOURCE IS FITTED WITH A UV GUARD WINDOW BEFORE STARTING ANY OBSERVATIONS OF THE COLOUR OF THE DISCHARGE

4.2 Using the UV source on a NOVA

It is assumed that the source is being differentially pumped and that the leak valve has not been opened although the helium lines will be full of helium at regulator pressure.

The PSU for the NOVA is different to the other axis machines. Once the PSU is turned on, HV is applied to the UV source. The dial on the front of the PSU is used to control the MAXIMUM allowed emission current. The UV source will provide the maximum emission current possible up to the maximum allowed by the setting of the emission current control dial. The emission current is affected by the gas pressure within the UV source, therefore maximum emission may not be achieved if the gas pressure is too low.

If the dial is turned to maximum a maximum emission current of 40 mA is set. However the PSU limits the actual maximum emission current to approximately 37 mA.

Before operating the UV source the SEC/SAC valve must be opened. When gas is let into the UV source the chamber pressure rises considerably. It is necessary to ensure the SEC/SAC valve is open at all times while gas is in the UV source. This allows the SEC turbo pump to also pump the SAC. If the SEC/SAC valve is closed the pressure in the SAC will rise uncontrollably.

To start the UV source, turn the PSU on and set the emission current dial to between 5 and 10 on the scale. There must be pure dry helium (or other gas) behind the leak valve. Slowly open the leak valve. As the pressure rises the lamp will attempt to strike. Initially the lamp may flicker on and off. Continue to raise the pressure in the source until a steady discharge is observed. Set the emission current limit to the desired value

(the pressure may need to be increased further to achieve this). If the lamp has been used previously with helium then it will give a “peachy” coloured discharge almost immediately. If it is the first time that the lamp has been run for a while then the discharge may be more “bluish white” due to impurities from the lines and from outgassing components in the source itself.

ENSURE THAT THE UV SOURCE IS FITTED WITH A UV GUARD WINDOW BEFORE STARTING ANY OBSERVATIONS OF THE COLOUR OF THE DISCHARGE.

The pressure reading in the SAC can be used to provide an indirect indication of the pressure in the UV source. CAREFULLY the pressure reading in the SAC will tend to lag behind the pressure in the source when the pressure is changed. It is necessary to let the pressure in the SAC stabilise to get a meaningful indication of the pressure in the UV source.

A pressure of approximately 2×10^{-6} torr in the SAC should be sufficient to allow the maximum of approximately 37 mA emission current to be obtained for He I emission.

A pressure of $2 - 4 \times 10^{-7}$ torr should be used to maximise the He II/He I ratio when He I radiation is required. The maximum emission current attainable at this low pressure should be approximately 20 - 25 mA. The colour of the discharge changes from “peachy” to blue when in the He II operating region.

NOTE. The UV source will require a pressure of approximately 6×10^{-7} torr (SAC reading) to strike initially. The pressure may then be reduced if required without the discharge going out.

4.3 Setting up an acquisition run and obtaining a spectrum

Having established that the lamp is running with a peachy colour and that it is stable we can now obtain a He(I) excited UPS spectrum. Using the manual window, in the lens mode ensure that “UPS” is selected and set the resolution to pass energy 5eV. Kratos recommends that only pass energies 5 and 10eV are used with the UPS system. These pass energies give both good sensitivity and energy resolution.

Make sure that on the energy region bar the reference is He(I). If values in the energy region have incorrect binding energies appropriate to He(I) then the work station will give an audible bleep to indicate that the binding energies need changing - note that the system is binding energy sensitive rather than kinetic energy sensitive. In addition the system will give audible warnings and will also flash up warning messages if the kinetic energy is too low. The lowest kinetic energy that can be selected depends on the pass energy selected, on the number and configuration of the electron multipliers, and on the work function of the electron spectrometer.

For a work function of 4.22eV and a pass energy of 5eV then with all 8 multipliers selected then the minimum kinetic energy that can be inserted in the energy region window is 4.9eV. For the same conditions but with only number 1 multiplier on then the minimum value of kinetic energy is 3.6eV. If a sensible value is chosen and the warning message remains the message can be deleted by “pressing” the “Active” button in the energy region.

For the first run only one multiplier should be used - for reasons which we shall go into in some detail shortly. One of the first 4 multipliers should be used e.g. multiplier #3.

With He(I) selected let the energy region be from 4eV to 24eV kinetic energy, choose a step size of 0.05eV and a dwell time of 100msec. Select one scan and make sure the active button is “depressed”. See Figure 10. where the above conditions are set up in the manual control panel.

For the rest of this section the description is made with reference to an 8 channel AXIS Ultra detector system. The same instructions apply to 3, 5 or 8 channel systems (AXIS HS, HSi and 165 respectively) but in these cases it is recommended that the centre detector i.e. 2, 3, and 5 respectively be used where the instruction is to use a single detector or more or all the detectors for higher kinetic energy work.

4.4 Spectrometers with channelplate and delay line detectors

For the NOVA and any other machine that uses a channelplate and delay line detector combination instead of a series of discrete electron multipliers. The lowest kinetic energy that can be selected for an acquisition is limited by the number of detectors chosen, the work function and the PE.

Above it is recommended to only use 1 multiplier for the first wide scan. This is not

Obtaining UPS Spectra

4

necessary when using a detector with a channelplate and delay line detector. This is discussed in more detail in section 4.10 .

Obtaining UPS Spectra

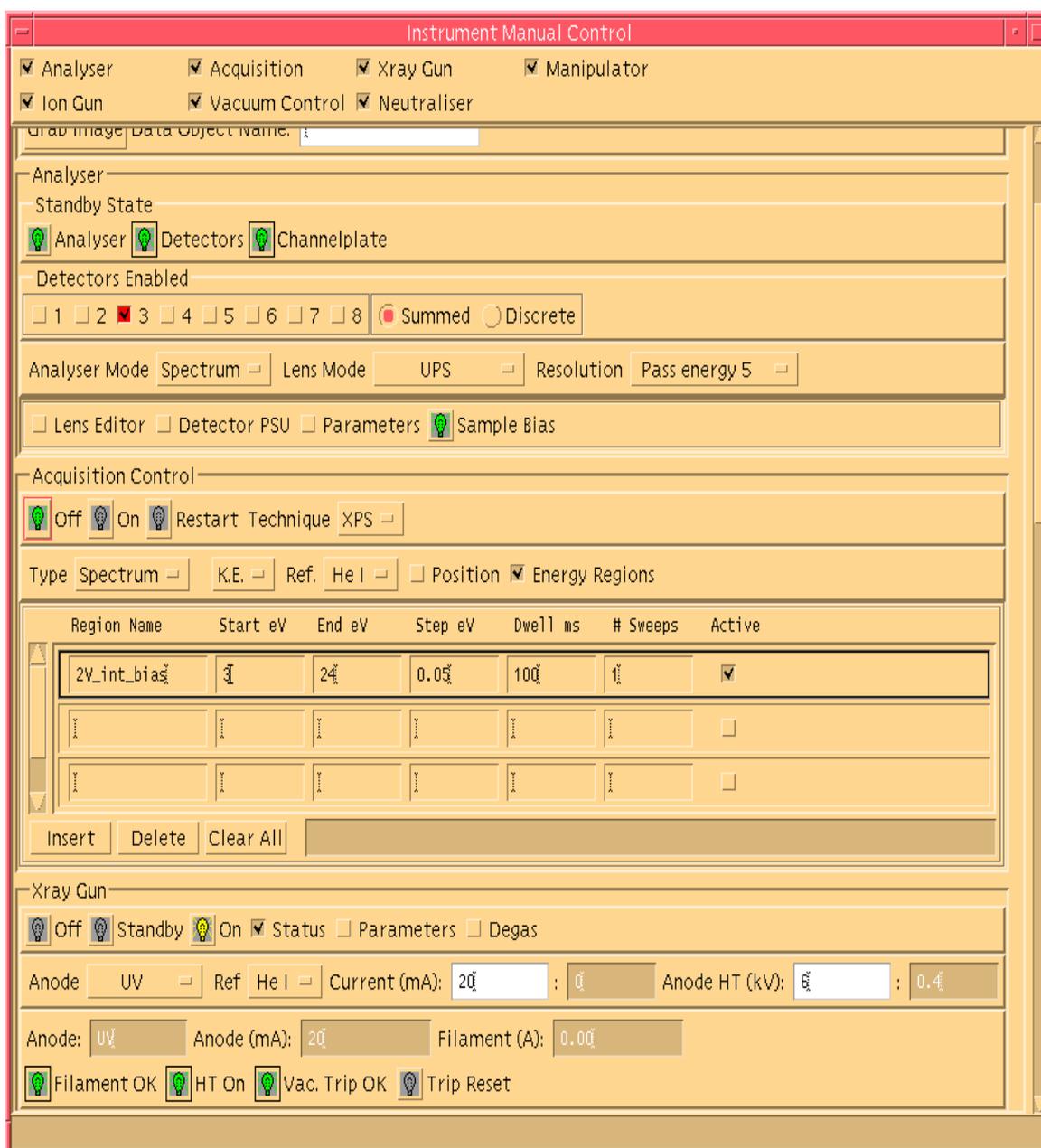


Figure 10. Manual Window Setup for UPS Operation (Note for later lamps with in built ballast resistor the Anode Ht readback will read up to ~2kV depending on lamp operating current)

The “on” button on the acquisition control panel can now be depressed and the scan will start. Note that the technique selected on the acquisition panel is “XPS” - there is no UPS option. The real time display should display a spectrum similar to that shown in Figure 11.

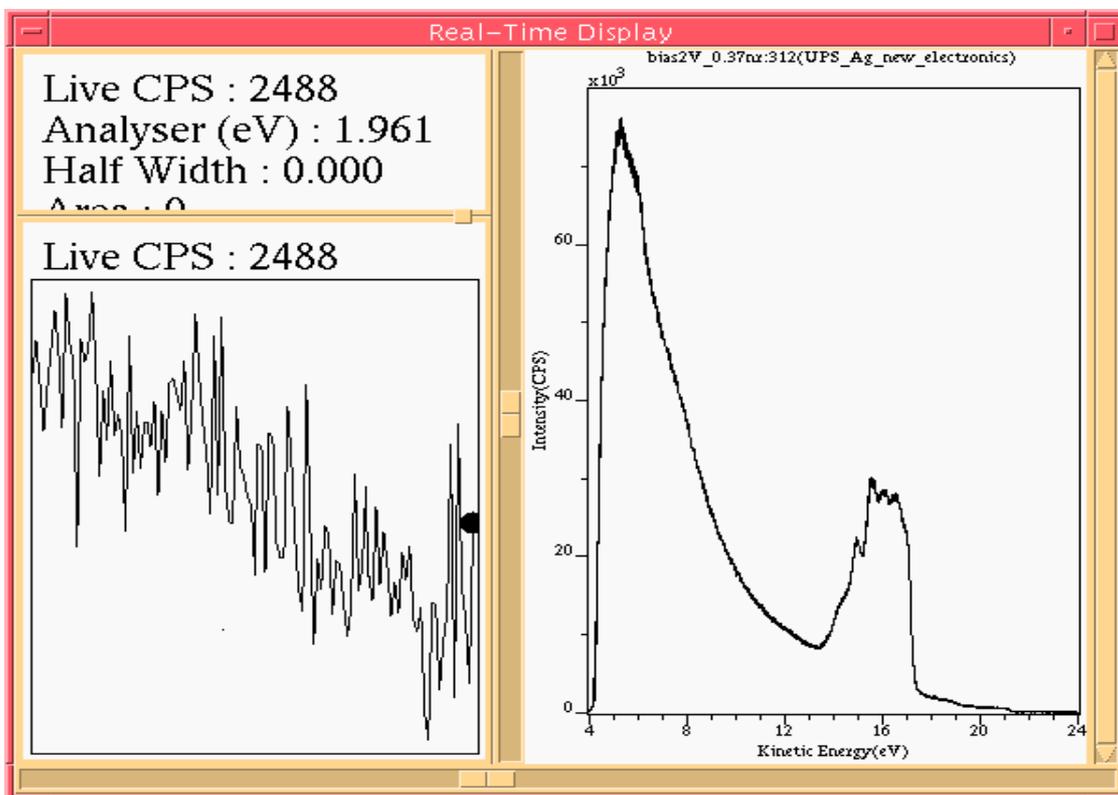


Figure 11. Real Time Display

Ensure that the spectrum is similar to that of clean silver - dirty silver will exhibit a less well resolved spectrum - for full details see section 5.1 on page 37 where the silver spectrum is discussed.

Once a clean silver spectrum has been obtained then the running conditions can be optimised. This is best done by noting where an intense part of the silver valence band

occurs on the kinetic energy scale (or binding energy scale depending on your preference) - say 16eV on the K.E. scale.

Go back to the energy region in the manual panel and now set the region so that it has a centre of 16eV, a width of 10eV, a step of 0.05eV, a dwell time of 50msec and 1 sweep. Restart the acquisition and check that the centre of the scan corresponds to an intense part of the valence band. This being the case then set the width of the scan to 0 volts and reduce the dwell time to 50msec. In the lower left hand window of the real time display the intensity of the valence band is now displayed as a function of time. Now is the time to optimise any parameters that may need adjusting. For instance the current in the UV source can be increased 30 mA and an increase in intensity should be seen. Other parameters such as the sample height, the source pressure can be adjusted to maximise intensity. Care should be taken when altering source pressure to ensure that the pressure does not become high enough to load the vacuum system or low enough to cause the lamp to become unstable or even to extinguish.

When the intensity has been optimised re-run a spectrum to ensure that the form of the spectrum is still good. Check that the Fermi edge can be seen and that it is occurring at a kinetic energy of 21.21eV. Acquire a spectrum into a data set using the combination of the manual system and a data set set up in the manager panel as outlined in the main spectrometer Instruction Manual.

When satisfied with the form of the spectrum the system can be set up to produce and acquire He(I) excited UPS spectra.

IF OPERATING A VISION 1 SYSTEM THEN SIMILAR RUNNING CONDITIONS CAN BE SET UP. THERE WILL BE NO WARNING MESSAGES REGARDING THE LOWEST PERMISSIBLE KINETIC ENERGIES.

4.5 CAUTION!! Restriction of the count rate during acquisition.

UV sources, particularly when running in the He(I) condition have high intensities and can cause very high count rates to be recorded in the UPS spectrum. If the count rate

PER ELECTRON MULTIPLIER is in excess of 1 million cps then the lifetime of the multiplier can be seriously compromised. It is suggested that under such conditions the selected area aperture and/or the iris is reduced to cut down the count rate in the spectrum.

Another situation where extremely high count rates can be recorded is when studying insulating sample with the charge neutraliser - particularly when recording UPS spectra excited by He(I). The charge neutraliser in the Axis series of photoelectron spectrometers (Axis HSi, Axis 165, Axis Ultra, Axis NOVA) is a source of thermionic electrons channelled back to the specimen by the action of the electrostatic and magnetic fields in the specimen vicinity. However some of the electrons can be scattered back and some reflected by the specimen to produce a narrow (in terms of energy) peak of low kinetic energy which generally coincides with the on-set of electron emission from the sample. The peak from the charge neutraliser can be very intense and apertures should be reduced as far as possible as is compatible with obtaining the He(I) induced UPS spectrum. It is also worthwhile attempting to run the charge neutraliser with reduced filament current (reduced temperature). Reducing the gain of the multiplier may ameliorate conditions also but it does not reduce the number of electrons striking the front of the multiplier.

4.6 Channelplate detectors

For The NOVA and any other machine that utilises a channelplate detector. The lifetime of the channelplate can also be seriously compromised by very high count rates. If the total count rate (with maximum detectors) is in excess of 10 million cps then it is recommended that under such conditions the selected area aperture and/or the iris is reduced to cut down the count rate in the spectrum.

4.7 Producing and acquiring He(II) UPS spectra

We shall assume that the source is running with a “peachy” coloured discharge and is capable of producing good He(I) spectra. The manual mode window is assumed to be running but now the reference should be set for He(II) and the scanned energy range

should be from 20eV to 44eV (kinetic energy) - the scan step should be 0.05eV, the dwell time in the order of 100msec; it should be set for 1 sweep and the pass energy should be 5 or 10eV (lens mode UPS). It is permissible to set more multipliers when acquiring data at the higher kinetic energies associated with He(II) excitation. Run with multipliers 1-4 (inclusive) in operation - BUT ENSURE THAT COUNT RATES ARE NOT EXCESSIVE - see notes in section 4.5 on page 26.

A spectrum may be seen depending on whether the source is producing any He(II) of significant intensity.

Keep observing the scanned spectral region in the real time display whilst gradually adjusting the leak valve to the source so as to reduce the helium flow. This will cause the pressure in the source to fall and the colour of the discharge will gradually turn from a “peachy” colour and start to become a lilac/violet colour. Further reduction of pressure will cause the discharge to become blue. The anode cavity of the source will be filled with a blue mist - this is the He(II) operating region and a He(II) excited UPS spectrum of Ag should be seen in the real time display. The form of the Ag He(II) UPS spectrum is shown in Figure 12. As in the case of He(I) the system can be optimised as outlined in section 4.3 on page 21. However it should be noted that maximum production of He(II) is obtained when the source is very near extinction and more care and patience will be required to get the best out of the source. The source has a bigger hysteresis in the He(II) mode - i.e. if the source does extinguish then the pressure will have to be significantly increased to cause the source to re-ignite. Then the gradual reduction of pressure will have to be repeated. In He(I) mode the source tends to be more “forgiving”. If this sounds tedious it is not really so - experience will soon be acquired as to how to rapidly set the source up for good He(II) production.

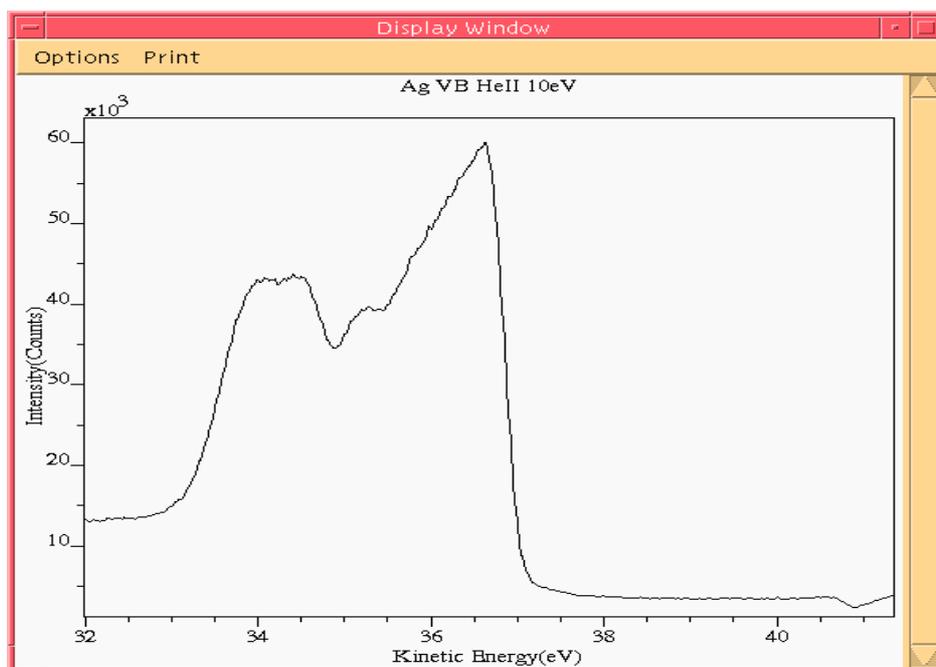


Figure 12. He(II) spectrum of Ag

Once the system has been optimised then acquire spectra in the same way as for He(I) spectra (allowing for different kinetic energies of course!) and examine them and compare them with those in sections 5 and 7 of this manual.

4.8 Checking the He(II) to He(I) ratio

A quick way of checking that the source is running efficiently in the He(II) mode is to measure the ratio of He(II) to He(I) under these conditions. A scan can be set up to cover both the He(I) and He(II) regions or, alternatively, two separate scans can be set up and the Ag valance band spectra as excited by He(I) and He(II) compared - all other conditions being equal - i.e. same pass energy, same number of multipliers etc. etc. The intensity of the He(II) UPS spectrum of the Ag valance band is typically $\sim 20\%$ of the He(I) excited valance band. PLEASE ensure that excessive count rates are not experienced by the electron multipliers.

4.9 Notes on the number of multipliers to use during acquisitions

It will not have escaped the readers notice that it was suggested that only one multiplier was used during the acquisition of the He(I) excited UPS spectrum - see section 4.3 on page 21

The reason for this is that at very low kinetic energies it is almost impossible to make the spectral response for each multiplier the same - stray fields round the specimen plus a widely dispersed set of discrete multipliers make for significantly different paths for very low energy electrons - say 1eV electrons. It is such electrons which are seen at the on-set of electron emission and to obtain consistent results for this very low energy region it is suggested that only one or two multipliers are used. The different spectral responses can be seen by putting a number of multipliers on in “discrete” mode - at low energies their responses will differ - at higher energies their responses become identical. Depending on the energy range you wish to study examine the region with the multipliers in discrete mode and then decide how many multipliers to use.

4.10 Using a channelplate detector

The channelplate detection system is nominally split into 127 detectors. If a smaller number of detectors is specified in the software then the detection area towards the edges of the channelplate (closer to the hemispheres) is not used. For this reason it is not possible to select any given individual area of the channelplate. Therefore it is not possible to independently compare the contributions from different parts of the channelplate. However for the reasons stated above, it may be beneficial when detecting very low energy electrons to reduce the number of detectors used. It is suggested that approximately 20 detectors should be suitable to obtain consistent results when detecting very low energy electrons.

4.11 Applying sample bias volts to the sample

The ability to apply bias to certain samples can be advantageous in UPS. The “divide by ten” unit has an adjustable approx. -1 to -10 volts bias supply incorporated. The voltage setting is made via the potentiometer on the rear panel of the unit. The use of the sample bias can be selected by pressing the Sample Bias button (shown in Figure 13.) which goes green when the sample bias is switched in. There is also an indicator

light on the divide by 10 unit to show when the bias is applied - *this only lights when a non-zero bias voltage is entered into the “sample bias” parameters text box and an acquisition is running*. Note also that the bias is applied via the sample current connection and that when applied the sample current readout is not connected.

When the parameters button is activated a display drops down with a window for the bias volts. One enters the actual bias volts that are applied in this window - this means that the bias voltage has to be measured either with a calibrated voltmeter or by measuring the shift in a spectrum. Only positive values can be placed in the window and these correspond to the negative bias volts applied. For instance on a test instrument the actual bias voltage was -2.01 volts - this meant that a value of 2.01 must be inserted in the bias window. This corrects the binding energy and kinetic energy scales on the displayed spectrum for the bias voltage i.e. the energy scale will read correctly without the operator correcting for the effect of the bias. Random values can be inserted to activate the bias - if one wishes to measure the bias by the shift in the spectrum this is the way to proceed but obviously the energy scale will be incorrect until the accurate value of the bias has been determined and inserted.

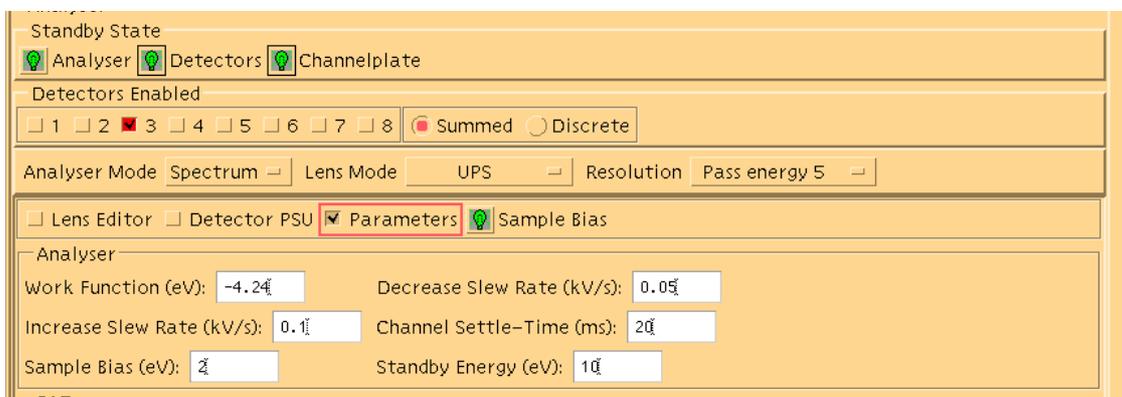


Figure 13. Sample Bias Setup.

4.12 Taking UPS spectra in the automatic acquisition mode.

It will be appreciated from the discussion above that the UV lamp can't be turned on a off in the same way as the x-ray sources on the instrument. It is assumed that if the automatic mode is to be used that the running conditions for the lamp will have been set up in the manual mode and that the automatic mode is to be used to acquire spectra from perhaps a number of different samples with given lamp operating conditions. In

Obtaining UPS Spectra

in this case the standby control should be set to “leave on” from the first acquisition through all the runs envisaged in the experiment. If sample bias has been selected in the manual window this is applied in the automatic mode also.

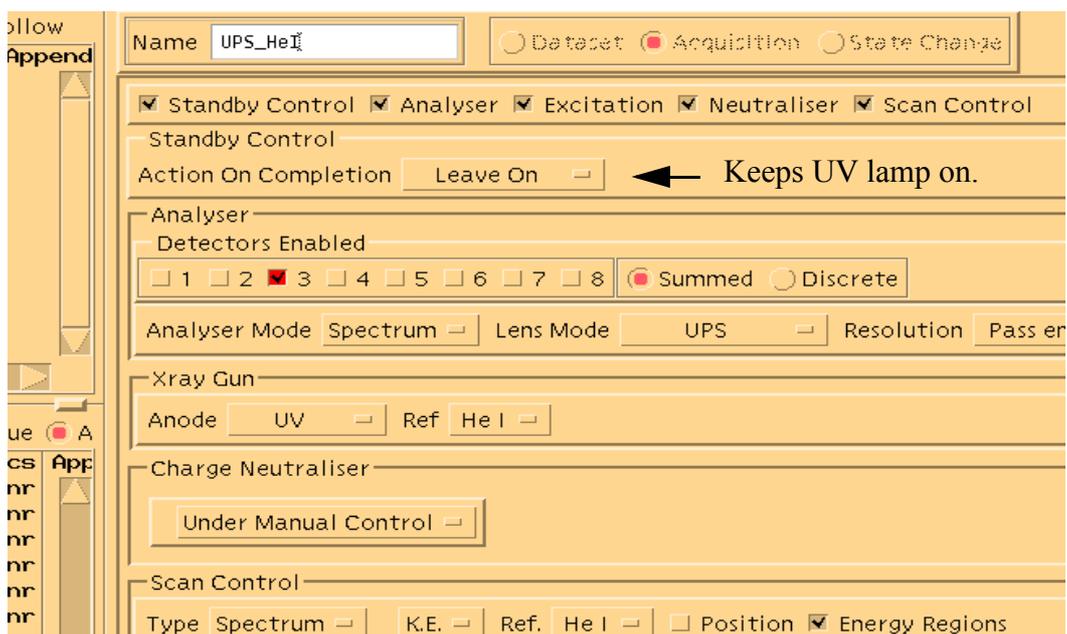


Figure 14. Manager Standby control

In the case of the NOVA or where a lamp from a third party is used then the operation of the lamp is independent of the instrument control system and no considerations on the instrument state at the end of an acquisition sequence need to be made.

4.13 Obtaining UPS spectra from insulating materials.

Insulating samples tend to charge up under UV radiation as the photo ejected electrons tend to cause the sample to acquire a positive charge - just as in XPS. The charging has a profound effect on the low voltage photoelectrons and can cause the spectrum to be distorted or lost.

Very thin films of insulators can often be recorded as enough photoconductivity can be induced in the layer to maintain a constant near earth potential at the specimen surface. In this way the spectrum of a thin film of a liquid insulator has been recorded - this example is discussed in the Examples section (section 5 on page 37) of this manual.

Figure 15. shows the He(I) UPS spectrum of a heavily argon ion etched sample of gallium arsenide (GaAs). Figure 16. shows the effect of a very thin layer of a polymer overlaying the GaAs substrate. The polymer in question was polymethyl methacrylate (PMMA) and the XPS spectrum shows both the spectrum details of PMMA and of GaAs - in the He(I) UPS spectrum it is seen how the apparent valence band has been modified by this thin overlayer - no charging was experienced but as the thickness of PMMA was increased the spectrum showed some charging.

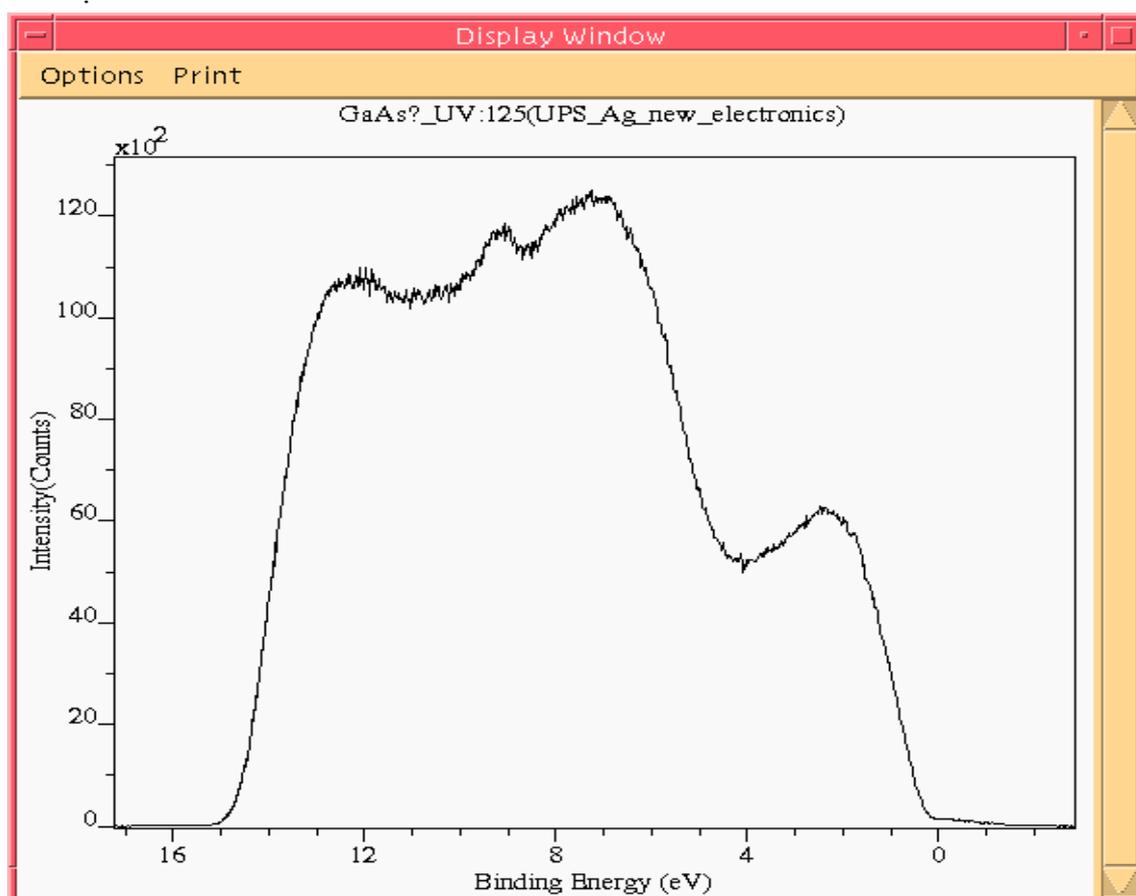


Figure 15. He(I) UPS spectra of heavily etched GaAs

He(I) excited UPS spectra tend to occur on a background of secondary electrons - as we have seen for the Ag metal spectrum and can be seen for Cu and Au samples in section 5.1 on page 37.

Samples which have a relatively weak valence band i.e. the cross sections are low and the number of photoelectrons are relatively low this background can tend to hide some of the spectrum. The usual approach to eliminate charging is to illuminate the sample with low energy electrons to compensate for the loss of photoelectrons - and the Kratos series of photoelectron spectrometers are fitted with a very effective form of neutraliser which electrostatically and magnetically channels the neutraliser electrons to exactly where they are needed on the surface to effect neutralisation.

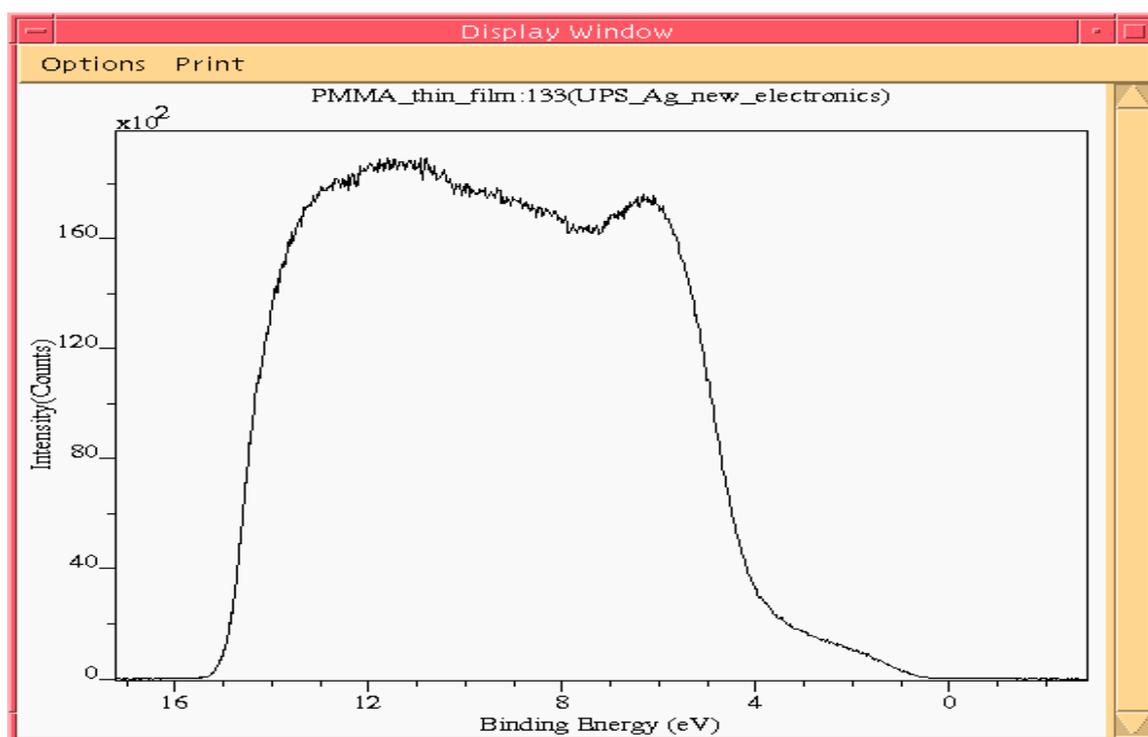


Figure 16. Effect of thin film of PMMA (see text) overlaying the GaAs sample of Figure 15.

It is found that this system works well with He(II) excited UPS spectra but is much more critical to set up when attempting to effect neutralisation during the acquisition of He(I). Indeed bulk specimens of polymer have been effectively neutralised in the He(II) excited mode. An example is provided by a thick layer of the liquid insulator previously mentioned - details of which are given in section 5.3 on page 43. Bulk polymers such as PMMA and PTFE have also been neutralised in this way.

The difficulty experienced with the neutraliser in the He(I) mode is that some of the neutraliser electrons are reflected/scattered from the specimen and cause a high intensity peak which overlaps the region where the secondary electron emission on-set occurs. This obscures the on-set and can also produce a very intense peak of neutraliser electrons which can damage the multiplier(s) - see notes in section 4.5 on page 26 of this manual. Reducing the temperature of the neutraliser filament can reduce the number of electrons reaching the detectors but can make the neutraliser less efficient as a neutraliser.

4

Obtaining UPS Spectra

In this section examples will be given of He(I) and He(II) excited UPS spectra from various materials. Some of the materials will be familiar and easily obtained for those who wish to repeat the work to check out the UPS system. In particular the spectra from copper, silver and gold should be easy to repeat - the form of the spectra should indicate that the lens functions are set up correctly as well as indicating that the surfaces are clean and that the UV radiation is “pure”.

Insulating samples have been studied and some of the results are presented in section 5.3. Obtaining good UPS spectra from insulators, especially bulk insulators, presents a number of problems and these will be discussed in the section. A number of approaches have been used, some successful, some less successful. Kratos welcomes any feedback that customers might have regarding the examination of insulating material by UPS - the field of polymers where quite an extensive data base of XPS spectra of the valence band is available and is a field of special interest.

5.1 Spectra from the coinage metals (Cu, Ag and Au)

The He(I) spectra of copper(Cu), Silver(Ag) and Gold(Au) are frequently used to check that the UPS system is operating correctly - e.g. a) that the UV source is producing a “pure” He(I) line (or “pure” He(I) mixed with “pure” He(II)), b) that the sample surface is clean and c) that the UV source is running in a stable manner. In addition it should be possible to discern the on-set of electron emission, the main features of the UV induced spectrum and the Fermi edge of the metal.

As has been mentioned already silver metal is a useful sample on which to test out the UV system - just as it is for checking out the XPS mode of the electron spectrometer.

An example of the He(I) excited UPS spectrum of silver(Ag) metal is shown in Figure 17. . The main features of the spectrum (on-set of electron emission, the detailed valence band and the Fermi edge are all clearly marked). Notice how the valence band “sits” on the falling background of secondary electrons. The spectrum also contains information from the other, weaker, He(I) lines and the evaluation of this information is discussed in some detail in section 7.

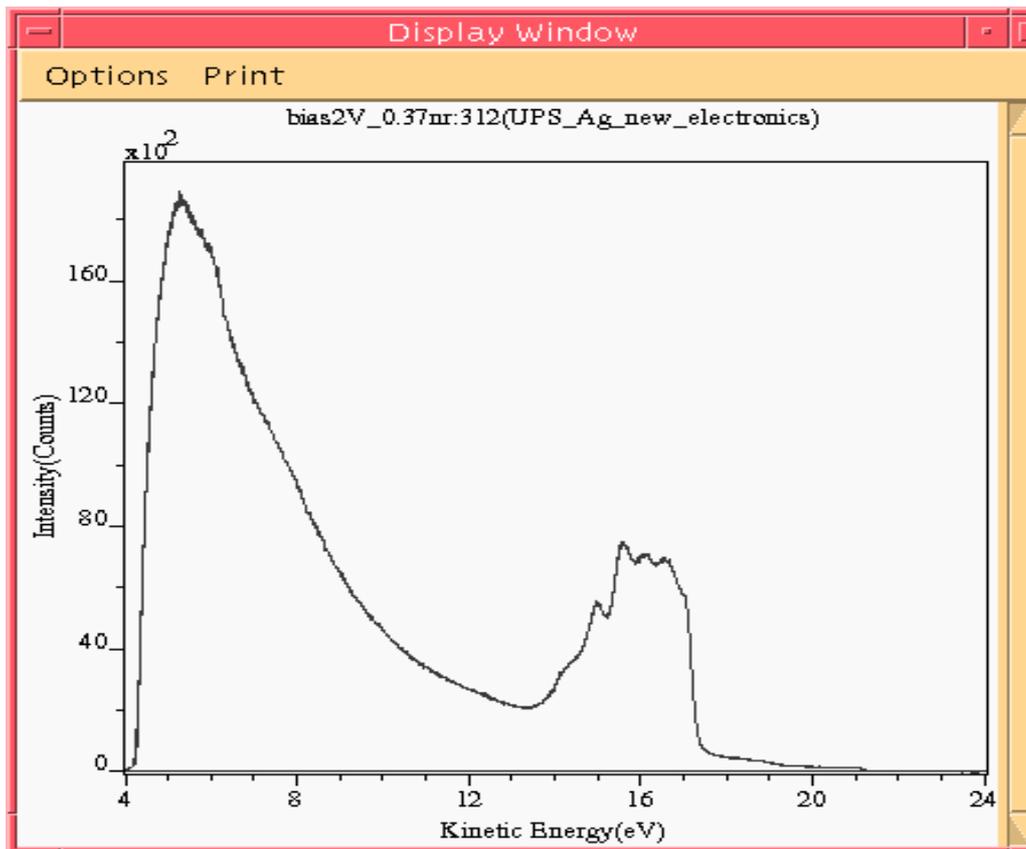


Figure 17. "Clean" Ag

If the sample is "dirty" then the spectrum shows a lack of definition of the "fingers" of the valence band detail and the start of the valence band detail is not sharp - Figure 18. shows the He(I) excited UPS spectrum from a dirty Ag sample. The sample represented by Figure 18. was examined by XPS and the intensity of the C1s line (carbon being the contamination on the sample) was only 1% of the Ag3d(5/2) line illustrating the sensitivity of the He(I) excited spectrum to contamination.

Examples of He(I) and He(II) UPS Spectra.

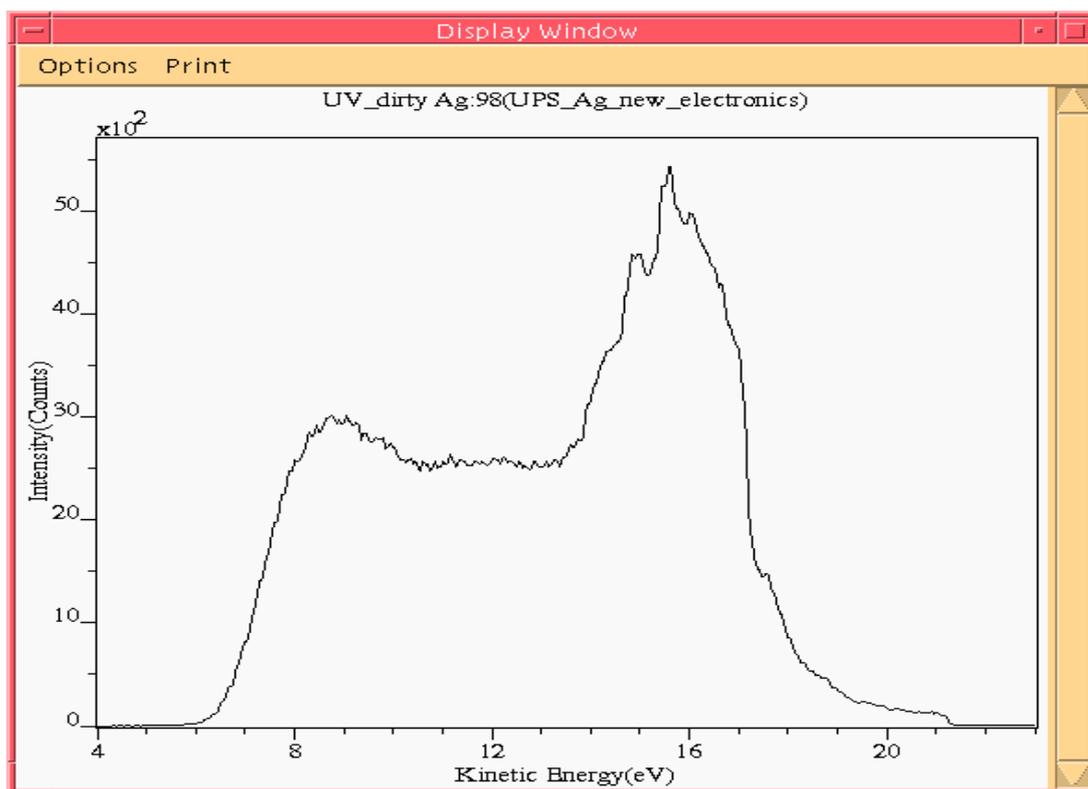


Figure 18. "Dirty" Ag

The photoelectric cross-sections of the various electron levels making up the VB can change quite rapidly with low energy excitation and this is illustrated by the He(II) excited UPS spectrum of Ag metal which is shown in Figure 19. Note the overall change in form of the valence band. He(II) excited UPS spectra are not sitting on a significant background of secondary electrons and quite often the Fermi edge is obscured by the effect of He(II) "satellites" - see section 6.1 on page 47.

Examples of He(I) and He(II) UPS Spectra.

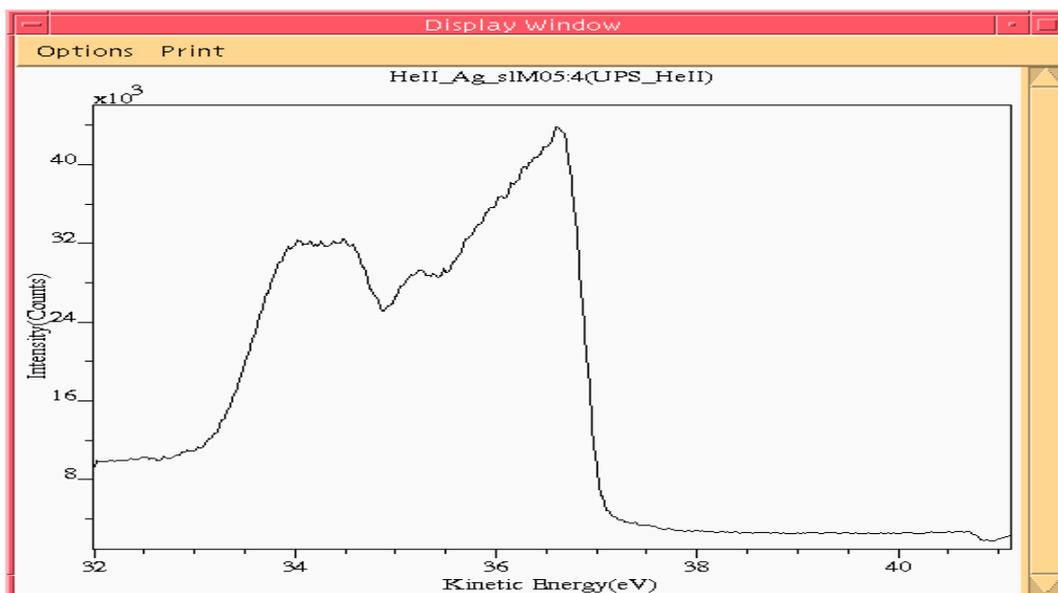


Figure 19. He(II) spectrum of Ag.

The He(I) excited UPS spectra of gold(Au) metal and copper(Cu) metal are shown in Figure 20.

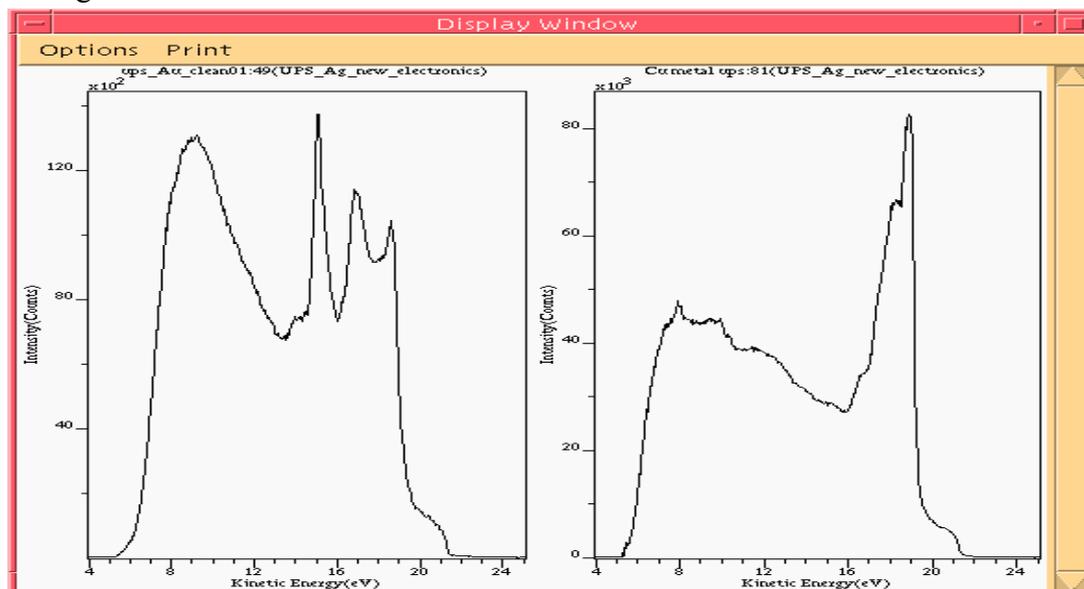


Figure 20.

5.2 Angular Variation of UV Photo-electron Spectra of Solids.

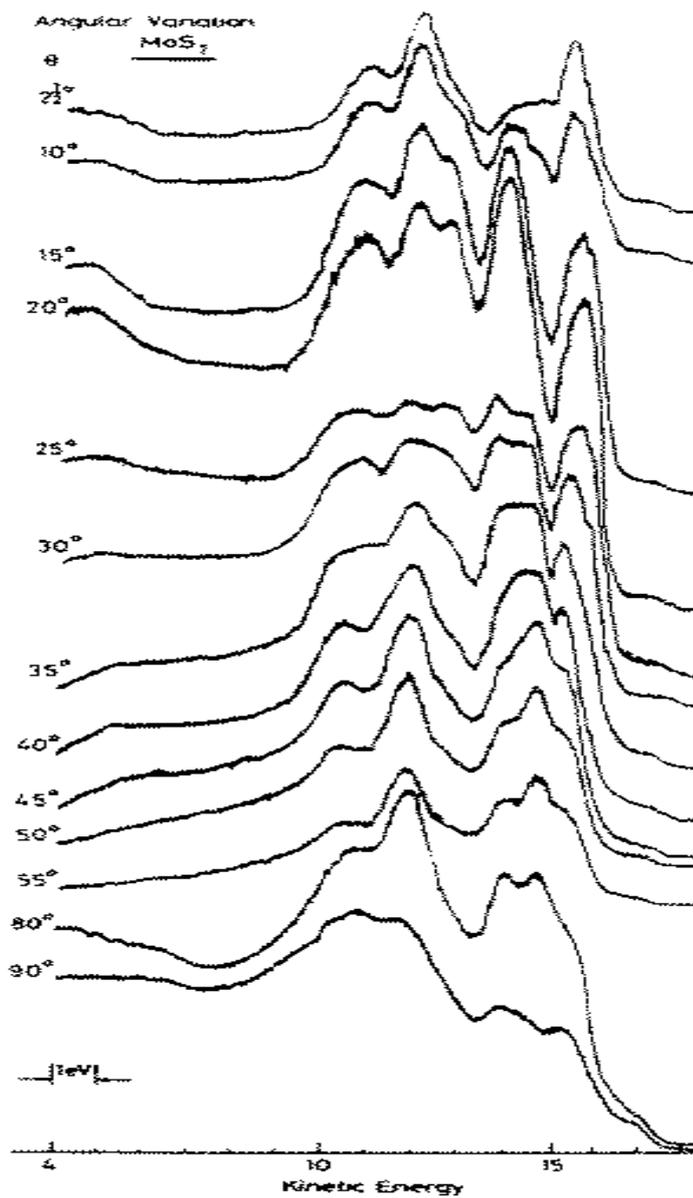
It has been observed that valence band spectra can be influenced by the angle of incidence of low energy photons, for example He I at 21.2eV, upon the sample surface. It is possible to deduce the character of valence bands from the spectra, because symmetrical bands remain comparatively constant in intensity and asymmetrical bands change in intensity as the angle is varied.

This is illustrated by the spectra recorded on the single crystal of molybdenum disulphide (see Figure 21.) which was cleaved to allow only the basal plane to be irradiated while the angle was varied.

The interpretation of these spectra is reported by Thomas et al(1). The maximum shown at approximately 10eV on the kinetic energy scale remains relatively constant in intensity as the angle is varied. It is possible that this peak relates to the deepest lying valence band, arising from the spherically symmetrical 3s orbitals of free sulphur, although there is some doubt about this interpretation(1). The angular dependence of peaks located in the range 11eV to 16eV indicates electrons arising from bands derived from S3p, Mo4d, Mo5s, and Mo5p orbitals. The individual peaks represent bands formed by the bonding of S3p and Mo hybridized valence orbitals.

Reference (1). The Band Structure of Molybdenum Disulphide from the Angular Variation of UV Stimulated Photo-electric Emission, J.M. Thomas, R.H. Williams, M. Barber and N. Alford. Chemical Physics Letters, Vol. 17, No.1, p.142-144, November, 1972.

Examples of He(I) and He(II) UPS Spectra.

Figure 21. Spectra from Molybdenum Disulphide (MoS₂) at Varying Angles of Incidence

5.3 He(I) and He(II) induced UPS spectra from insulators

The production of photoelectrons caused by UV illumination of the sample causes insulating samples to charge up to a positive potential causing distortions in the UPS spectrum and, in many cases, making acquisition of the UPS spectrum impossible. To overcome this problem a “charge neutraliser” is fitted to the electron spectrometer. The charge neutraliser is a low energy source of electrons and the low energy electrons are allowed to land on the specimen so as to prevent the sample acquiring a positive charge. The flux of electrons reaching the sample can be controlled by altering the running parameters of the charge neutraliser. The parameters under control are the filament current(temperature), the filament bias and the charge balance plate. Details of the charge neutraliser operation will be found in the main instrument manual.

It is not always necessary to use the charge neutraliser to obtain UPS spectra of insulators - for instance if the insulating material is prepared in the form of a thin film on a conducting substrate. It would seem that with this type of specimen enough photoconductivity is induced in the sample to maintain it at the substrate potential. Figure 22. shows the He(I) UPS spectrum of an insulating organic liquid specimen - the sample containing carbon, oxygen, fluorine, nitrogen and phosphorus (as shown by the XPS spectrum) and having a very low vapour pressure allowing it to be studied under UHV conditions. The sample is a useful one for checking the quantitative performance of the spectrometer when working in XPS and the author has nicknamed the sample as “Lorenzo’s Oil” because of its valuable properties for quantification.

The He(II) excited spectrum of Lorenzo’s Oil is shown in Figure 24. A comparison of the He(I), He(II) UPS spectra and the AlKalpha(mono) XPS spectrum of the valence band is given in section Figure 24. on page 45 (I W Drummond - unpublished work). It will be seen that some correlation seems to exist between the three spectra.

Examples of He(I) and He(II) UPS Spectra.

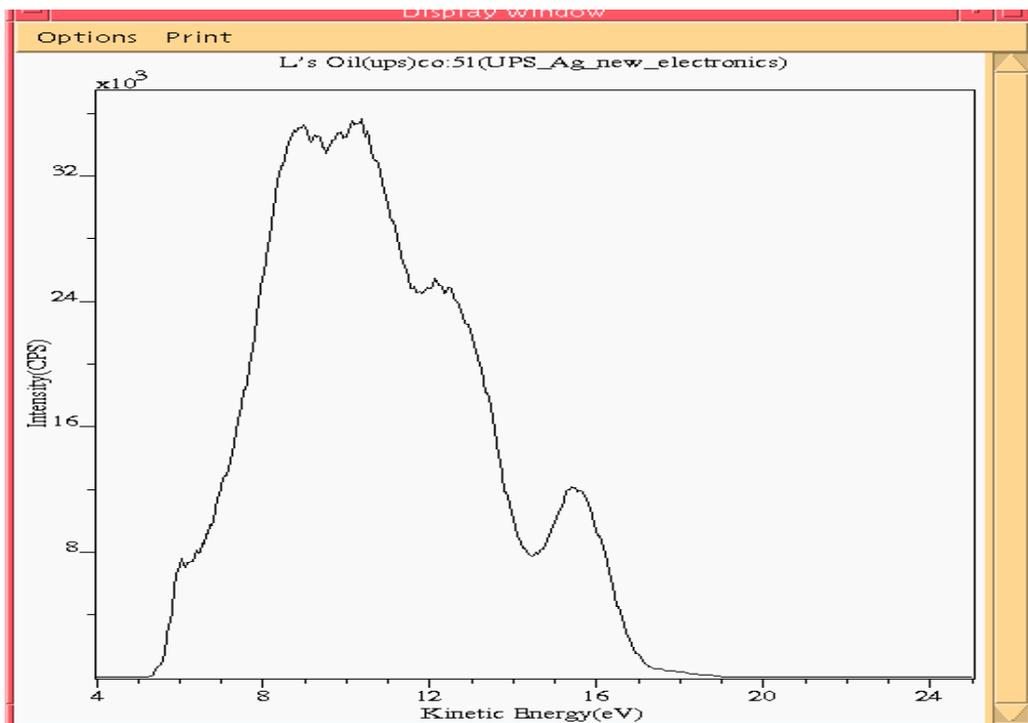
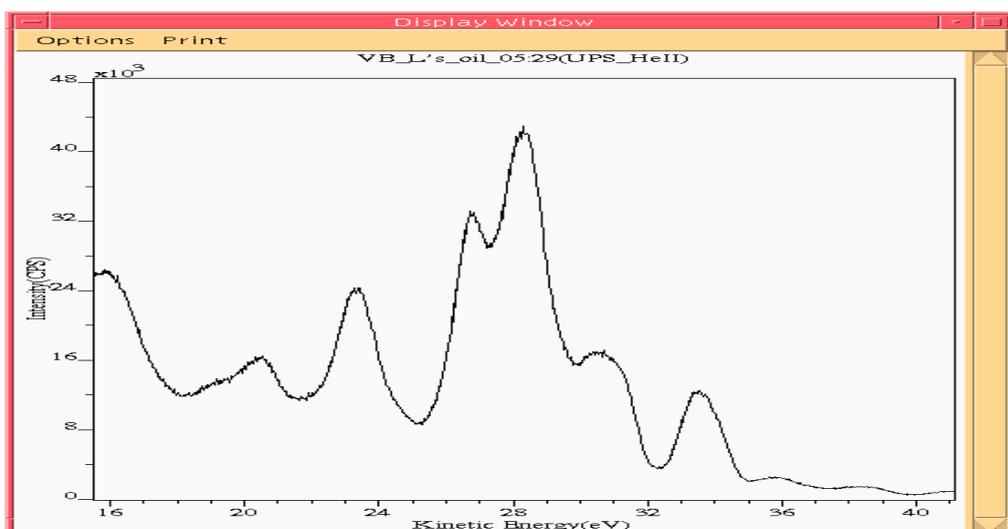


Figure 22.

Figure 23.



Examples of He(I) and He(II) UPS Spectra.

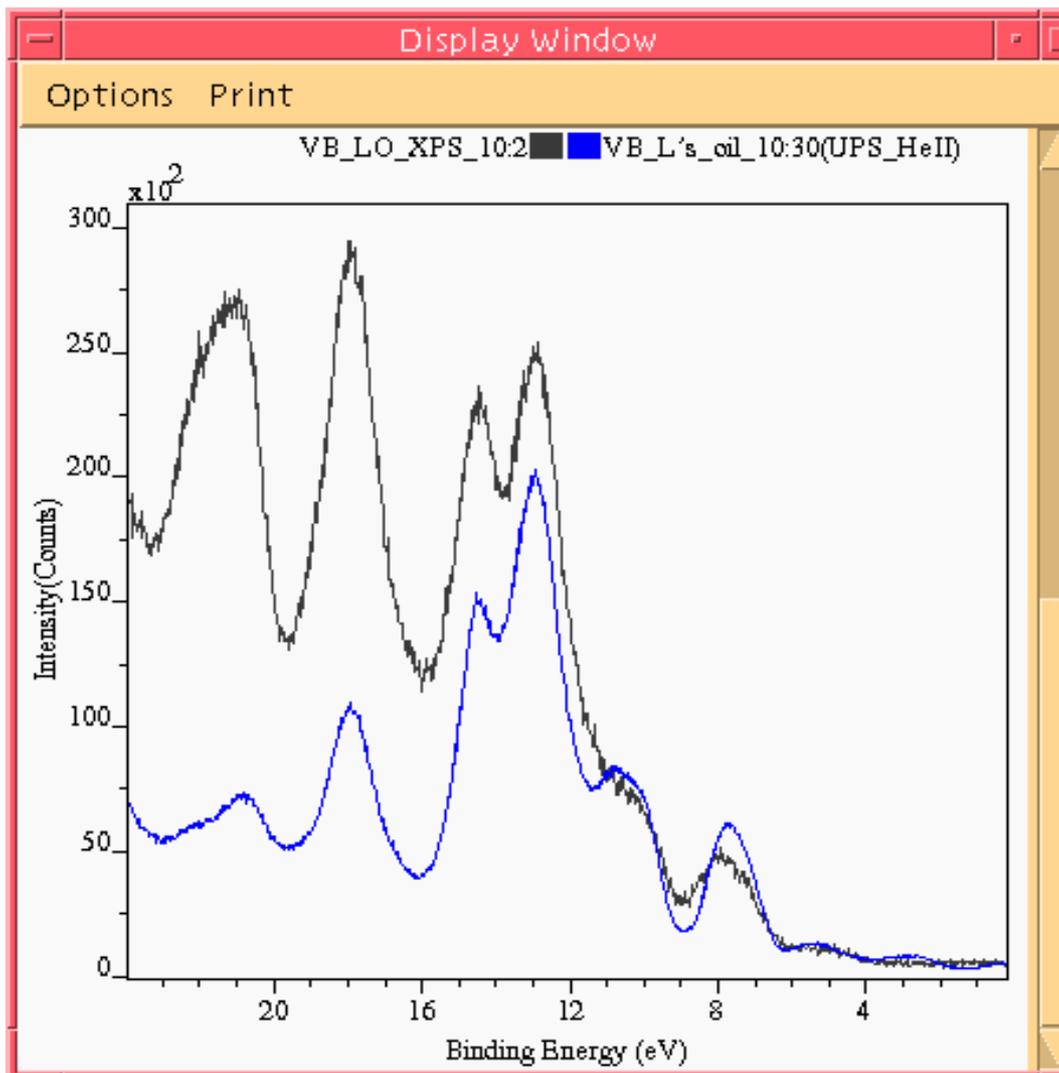


Figure 24.

Two points should be made about studying insulators with He(I) radiation. If the charge neutraliser is used it will be found that the neutraliser electrons are scattered and reflected from the specimen and these electrons will mask the on-set of electron

Examples of He(I) and He(II) UPS Spectra.

emission from the sample. Thus there are limitations on the amount of information that can be gleaned from the specimen. The scattered/reflected electron peak is also very intense (counts can exceed many millions of counts per second) and it may be advisable to aperture down the electron spectrometer and to use only one detector electron multiplier running at reduced gain to observe it without damaging the multiplier(s). Polymer samples also have valence bands which extend over more than 21eV and it may be more convenient to study the valence band of the polymer using He(II) radiation. The He(II) spectrum is also not complicated by the presence of the electron peak from the charge neutraliser and also has the advantage of not sitting on a significant secondary electron background which can obscure details in weak valence bands.

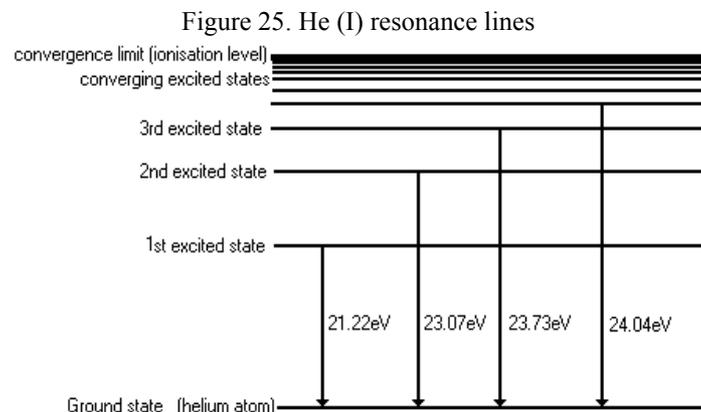
Origin and nomenclature of Helium Resonance Lines 6

A brief explanation of the origin of the helium resonance lines is given. It will be seen that there are “satellites” associated with the principal lines and these can be observed in the UPS spectra albeit of low intensity. The intensity of the lines is dependant on the running conditions of the lamp and a method of determining the contribution of the satellites is outlined.

6.1 Origin of the He(I) resonance lines

He(I) resonance lines arise from electron transitions from excited states to the ground state of the helium atom i.e. He* to He(1s). The transition from the lowest excited state to the ground level is known as the raie ultime: this is the strongest transition and has an energy of 21.21eV. This is the line which we generally refer to as He(I) radiation. It is however accompanied by transitions from higher excited states to the ground level i.e. He** to He(1s). All these “satellite” resonance lines which converge to the first ionisation potential of He at 24.587eV. Transitions may occur between excited levels, if they are permitted, but these are not resonance lines and the relative intensities are difficult to determine.

The origin of the lines is shown schematically in Figure 25. together with some of the energies of the first few lines. Similar information is given in Table 1: .



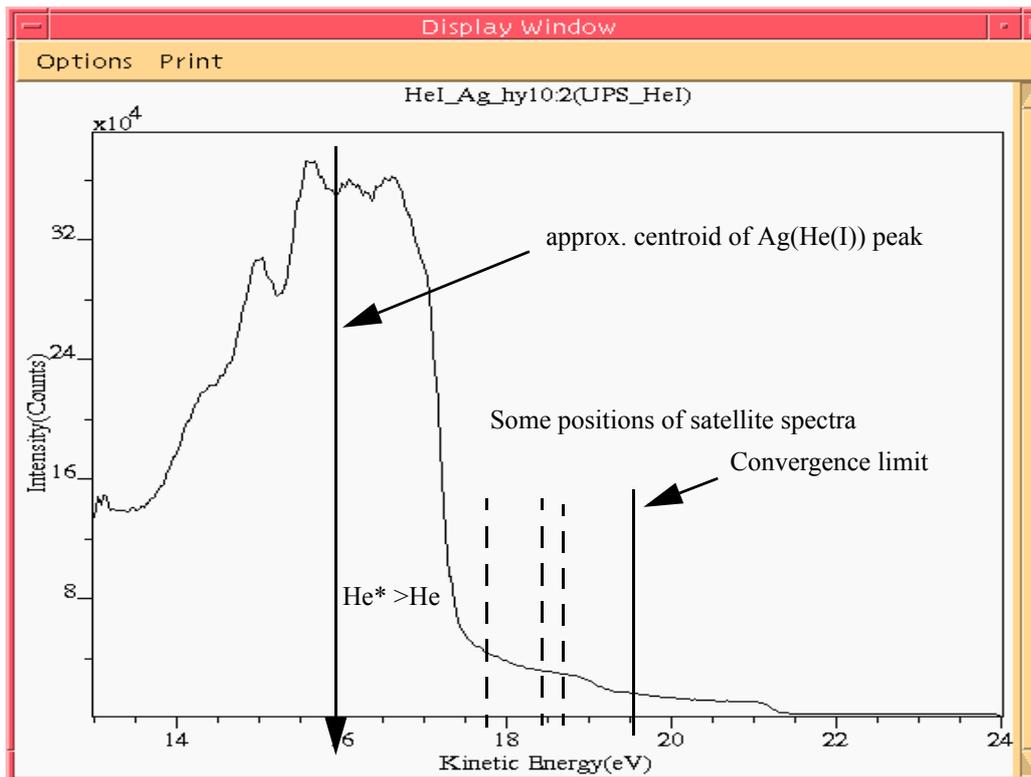


Figure 26.

The position of the centroid of the valence band for each of the lines is shown schematically in Figure 26. . It will be seen that the intensity of the satellites must be relatively low for no great disturbance of the spectrum is noted although there is a “shelf” at ~19eV K.E. The series converges at ~ 19.5 eV (K.E.) and the satellite of the main valence band will not interfere with the Fermi edge at 21.21eV.

6.2 The relative intensities of the “satellite” He(I) lines

It is possible to get a “handle” on the relative intensities of the satellites of He(I) by examining the Fermi edges induced by the different satellites - 3 Fermi edges are clearly seen in Figure 27. , the most intense step corresponding to the main He(I) line and the other two to the first and second satellites of the main line. Measurements made on the

Origin and nomenclature of Helium Resonance Lines

Fermi edges in this case, which seem typical of He(I) running conditions, indicate that the ratio of the main line to the first two satellites is 100:2.4:0.6.

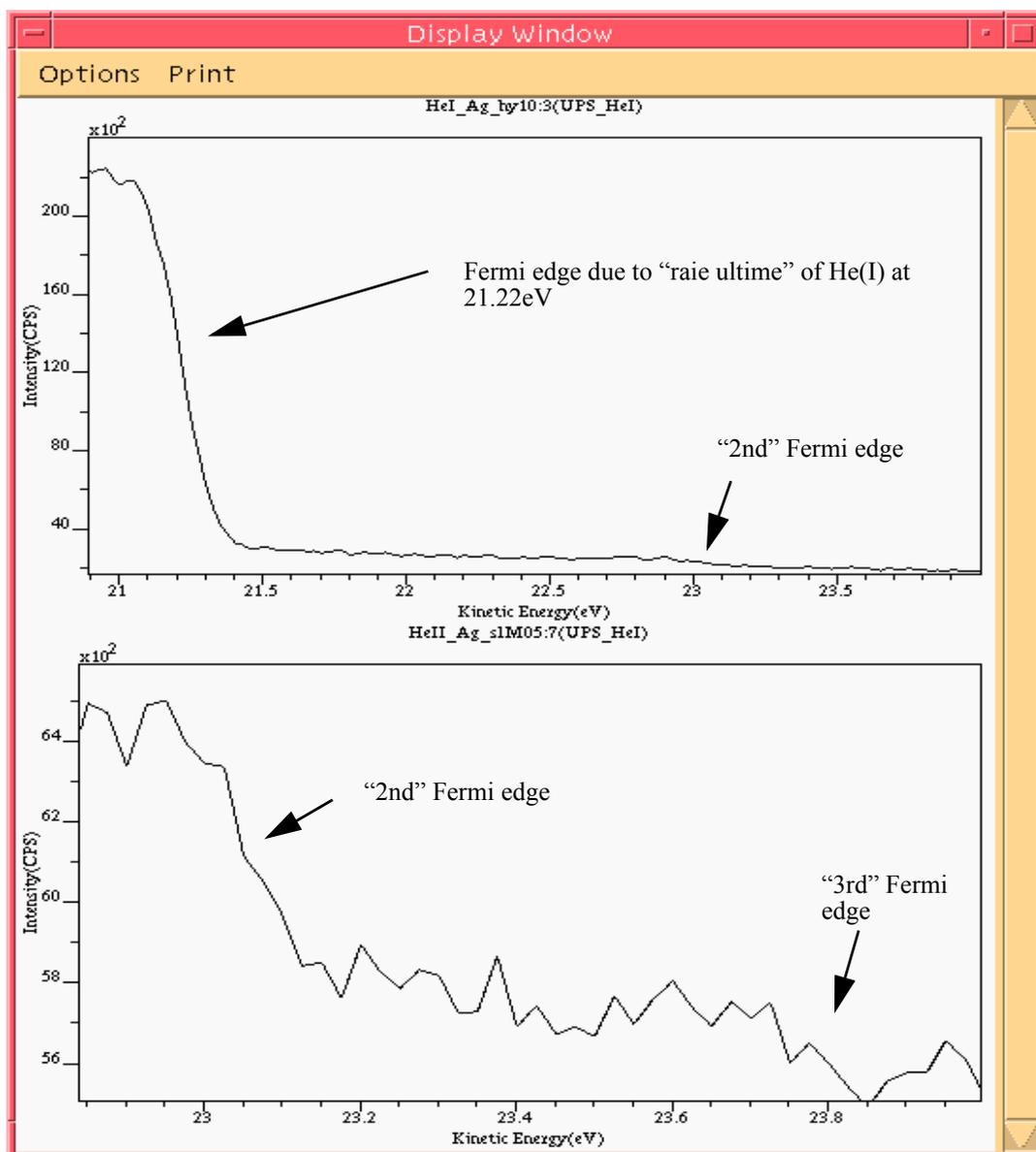


Figure 27. Fermi edges due to He(I) satellites

Obviously the accuracy of the above method depends on the transmission function of the instrument being uniform or at least known - it can be checked to a certain extent by applying different bias voltages to the sample (within limits). It also assumes that the cross-section near to the Fermi edge is not varying rapidly with the energy of the satellites. It is not felt that these errors will be too large and in any event the method will show how the satellite intensities change with the running conditions of the source.

6.3 The origin of the He(II) lines

In the case of He(II) UV radiation the ground level for He(II) is the ground state of the single charged positive helium ion (He(1s)) and once again there is a series of resonance lines representing transitions from the higher excited levels to the ground state of the ion as shown in Figure 28. and as tabulated in table 7.2. In this case the series converge at the second ionisation potential of the helium atom (54.416eV).

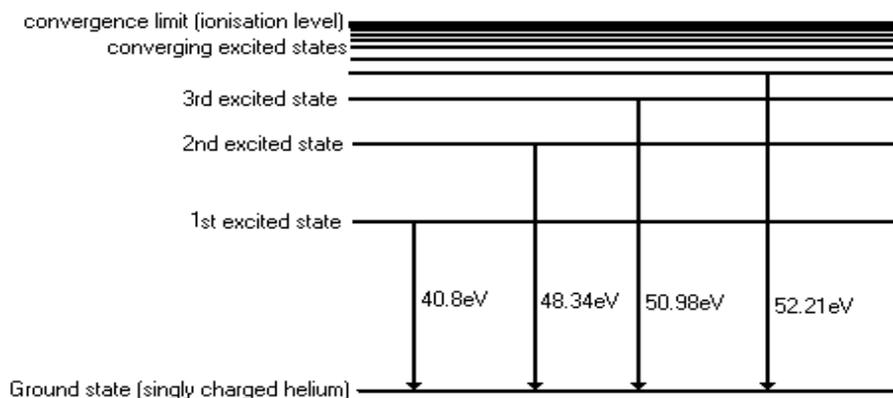


Figure 28. He (II) resonance lines

6.4 The intensity of the He(II) satellites

The helium(II) resonance series has energies of 40.8eV (the standard He(II) line - the raie ultime), 48.35eV, 50.98eV, 52.21eV and so on up to ~54.42eV.

The effect on the Ag metal valence band spectrum excited by He(II) is shown in Figure 29. where it will be seen that the energy differences between satellites is greater than in the case of He(I) and the “satellite” spectra “spill over” the Fermi edge excited by the He(II) raie ultime at 40.8eV - we can see where the Fermi edge is but we cannot make any resolution measurement on it. The first satellite is seen to have an intensity of ~ 7% of the main line. The satellite structures are shown in some detail in Figure 29. Figure 30. and Figure 31.

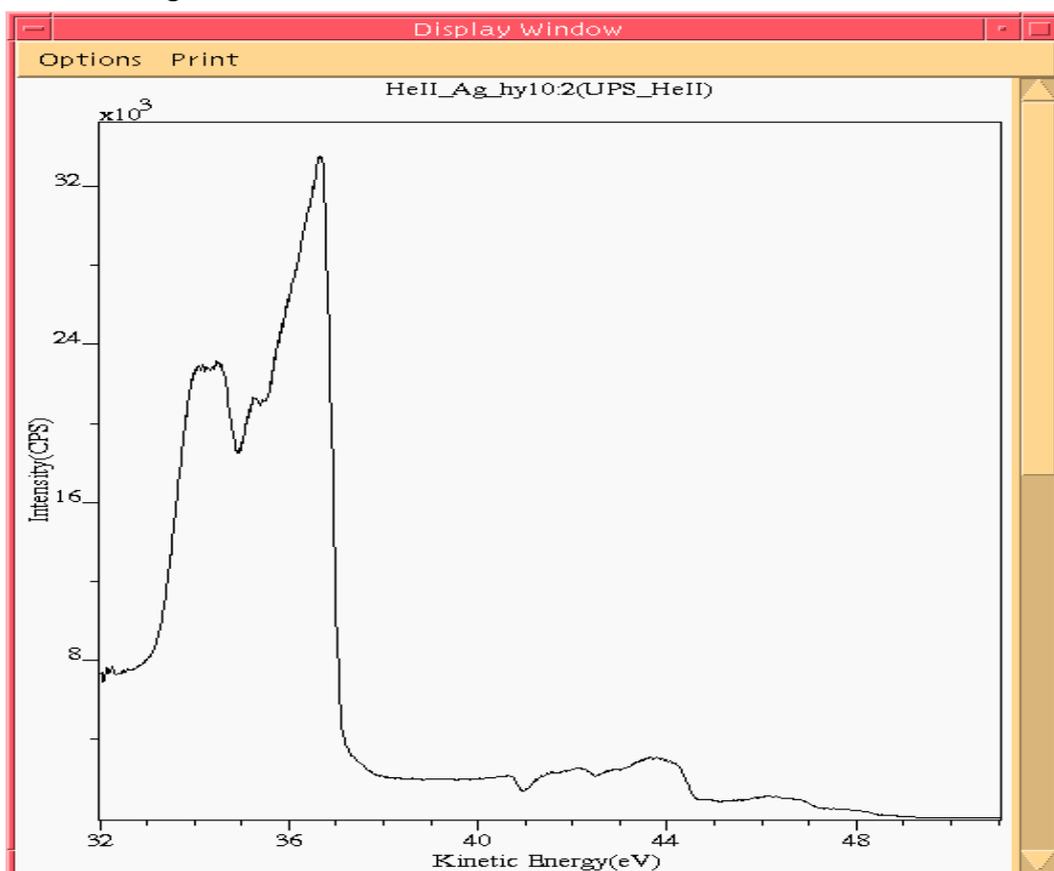


Figure 29. Ag valence band with He(II)

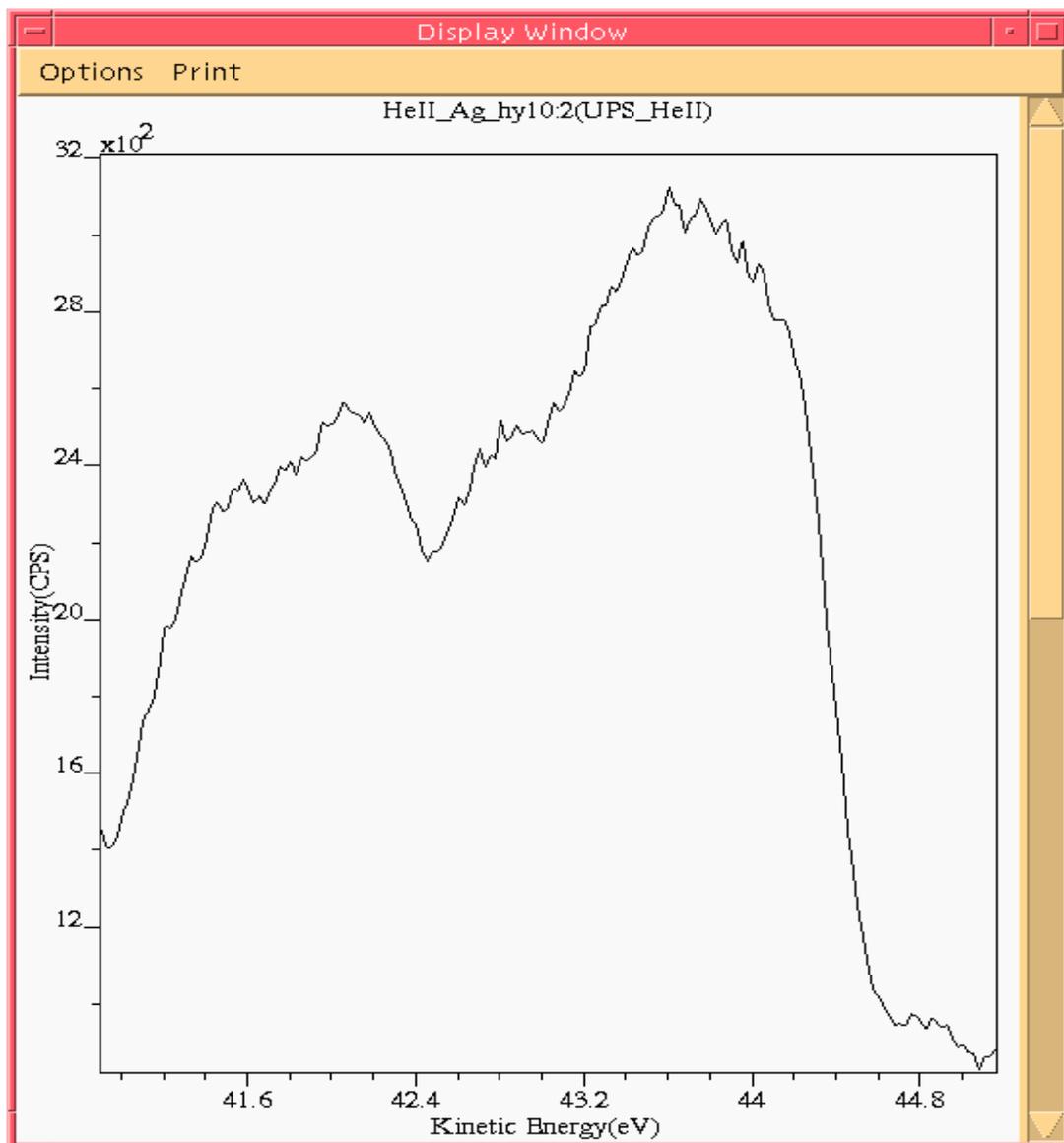


Figure 30. First He(II) satellite

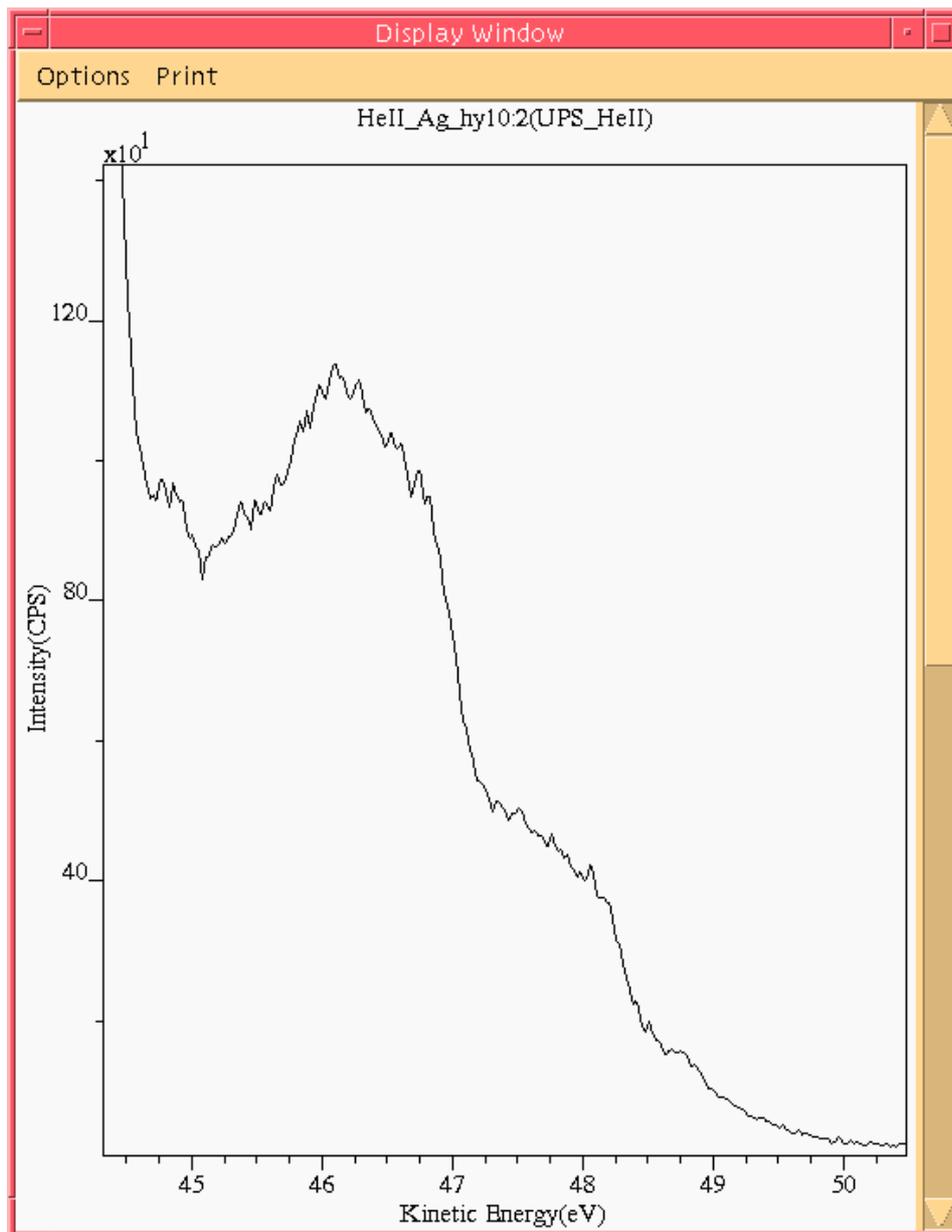


Figure 31. Further He(II) satellites

Table 1: Satellites of He(I) i.e. He* He(1s²) ground state

wavelength(A)	Energy(eV)	Energy difference from main line(eV)	Delta adj lines(eV)
584.334	21.22		
537.39	23.07	1.85	1.85
522.419	23.73	2.51	0.66
515.711	24.04	2.82	0.31

Table 2: Satellites of He(II) i.e. He⁺* He⁺(1s¹) ground state

wavelength(A)	Energy(eV)	Energy difference from main line(eV)	Delta adj lines(eV)
303.78	40.8		
256.32	48.35	7.54	7.54
243.03	50.98	10.18	2.64
237.33	52.21	11.41	1.23
234.35	52.9	12.1	0.69

N.B there may be some small differences between the energies given above and other sources - these result in different values for the conversion from wavelength to energy.

7.1 Maintenance of the UV Lamp

In time, some internal parts of the lamp in the cathode region will become covered with a black deposit and reduce the radiation output from the lamp. When this occurs the discharge section of the lamp may be removed as follows:

Switch OFF all power to the lamp and ensure that the lamp is safe..

Close V13, V7, V8, V12.

Vent the instrument SAC - refer to the main Instrument Operator's Manual.

In the following notes the bracketed numbers are items shown on the UV lamp-assembly drawing (BC6189AA).

Disconnect the HT cable. Remove the perforated cover taking care not to strain the internal wire connections.

Remove the lamp flange (10,2) from the body of the lamp.

No other parts may be removed from the internal structure of the lamp.

Scrape off any heavy deposit from the silica tube and aluminium cathode.

The bore of the silica tube can be cleaned by threading through a length of multi stranded wire. This is better than using a single thick wire. Ensure no particulate matter is pushed into the closed end of the lamp..

Under no circumstances attempt to clean this part of the lamp using a solvent of any kind.

NOTE:

Further dismantling of the lamp should not be attempted otherwise the lamp anode-to-cathode insulation will be lost. Inspect the light path down the central bore of the double- pumping stage. If cleaning is indicated, then the lamp should be returned to Kratos for service.

