S P C S

Surface Analysis and Computer Technology

PHOIBOS

Hemispherical Energy Analyzer User Manual

> PHOIBOS 150 PHOIBOS 100

> > 1.2

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User Manual for the energy analyzer and for the energy analyzer

PHOIBOS 150 PHOIBOS 100 as well.

Version 1.2 of the 25.5.99.

SPECS order number for this manual: 78 000 101.

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Chapter 1

Introduction

The SPECS PHOIBOS hemispherical electrostatic energy analyzer allows recording of energy spectra for negative particles (electrons) and positive particles (ions) in the kinetic energy range from 0 eV to 3.0 keV.

The PHOIBOS series of hemispherical analyzers is a range of hemispherical deflectors available in two sizes (100mm and 150 mm mean radii) and an input lens designed to accommodate a wide range of applications.

The analyzer is equipped standard with a single and optional with a multichannel detector with 5 (PHOIBOS100) and 9 (PHOIBOS150) discrete channels. The pulse counting electronics consisting of preamplifier, pulse forming unit and 24 bit counter is supplied together with the analyzer control unit.

All units provide the detection of electron energies between 0-3000 eV with minimum step witdhs of <13 meV. The unit can be upgraded by an additional voltage modules (Hi-RES module , 0-300 eV with minimum step witdhs of <1.3 meV; Super Hi-Res module , 0-300 eV max. range 50 eV with step witdhs < 0.2meV).

Beside the multichannel detection a variable magnification lens are available as option. Due to the new SPECS Slit Orbit mechanism and a Multi Mode Lens the analyzer sampling area and lens acceptance angle area are selectable. Thus the analyzer allowing confined area measurements down to 100-200 μ m area diameter as well as large area investigations accociated with different lens acceptance angles.

All units are completely controled by SPECS software. Features and operation of the software will be described in a separate manual.

Typical use of the PHOIBOS analyzer is in photoelectron spectroscopy (XPS, SSXPS, UPS), Auger electron spectroscopy (AES, SAM) and ion scattering spectroscopy (ISS). The PHOIBOS is bakeable up to 250° C after remove of few connections for the detector and lens supply.

4

Safety Hints

Before any electric or electronic operations please consult "SPECS Safety Instructions" and follow them strictly.

Some adjustments which might have to be carried out according to this manual are hazardous. At each such a point is there a warning label:

Attention!

The tests described in the following have to be performed on the electronic unit with its cover removed. Hazardous voltage are present, therefore only persons with the appropriate training are allowed to do the job. Make all trimmer settings only with a special insulated trimmer screwdriver.

Chapter 2

Description

2.1 System Description

The PHOIBOS analyzer consists of the following mechanical parts:

- analyzer housing,
- internal µ-metal shielding,
- lens system,
- hemispherical analyzer,
- multichannel (MCD) or single detector (SCD)
- Slit Orbit mechanism.

The electronics of the PHOIBOS consists of:

- power supply for PHOIBOS analyzer (HSA 3000)
- preamplifier
- cables with Filter Unit and series resistor for ech channel $(R = 20 M\Omega \text{ divided by the 'number of channels', fixed with the cable)}$

The HSA 3000 unit fully remote controlled.

The electrical connection diagram is given in figure 1.

2.2 Electrical Connections

All devices must be switched off before connecting or removing cables



Figure 1

Connection scheme of the PHOIBOS SCD components





Connection scheme of the PHOIBOS MCD components

The connection to the analyzer and the detector are supplied by 2 multi-pin vacuum feedthroughs, which are designed for high voltages up to 5 kV:

1. A 12-pin feedthrough on a flange DN38CF, mounted to the lens housing (F3 from figure 6), for all voltages of lens electrodes and capacitor electrodes, which is schematically shown in figure 3.

view from atmospheric side



Figure 3

Schematics of the 12-pin HSA feedthrough

2. The detector feedthrough and its pin assignments are shown in figure 4. Mounted on flange F5 from figure 6 (DN 38CF), seen from the atmosphere side.



PIN A:AnodePIN T:Tube (Cathode)PIN F1:Collector F1

Figure 4

Detector schematic connection setup and feedthrough pin booking (view from atmospheric side)

2.3 Basic Spectrometer Description

The PHOIBOS spectrometer consists of a vacuum housing and four major internal components, which are shown in figure 6 and in figure 7. All the parts must be exist within Ultra High Vacuum environment, as particles emitted from the sample surface could collide with the gas particles and so be lost from the study. The internal components are:

- input lens system for receiving charged particles;
- 180° hemispherical analyzer (HSA) with 100 / 150 mm nominal radius for performing spectroscopic energy measurements;
- detector assembly for single particle detection;
- Slit Orbit mechanism with external rotary feedtrough.

The source of primary radiation is dependent upon the technique to be used, but is commonly x-rays or other photons, electrons or ions. Before the particles pass into the hemispherical analyzer they first pass through an electron lens system and a slit. Both the electron lens system and the slits sizes (entrance and exit) have an effect on the spread of energies detected at the analyzer detection system.

The input lens system (figure 6, "Analyzer vacuum housing (PHOIBOS 150)," page 9) includes ten lens stages. For undisturbed imaging quality, the input lens system is grid-free. The lens stages defines the analysis area and angular acceptance by imaging the sample onto the entrance slit.

The particles passing through the lens stages and focused onto the input slit S1 of the hemispherical capacitor. They are retarded in the lens for subsequent energy analysis in the hemispherical capacitor. In addition the lens stage confines the capacitor acceptance angles and area due to the chosen magnification (*Large, Medium, Small*) and by the entrance slit.

The lens system allows three principal magnifications, a *Small* area mode with high lateral magnification, a *Medium* mode lens and a *Large* area mode with low magnification (see "The Lens System" on page 11). This magnification can be selected by the SPECS software.

The angle of acceptance of analysis is constant for *Large* and *Medium* mode, both for electrons and ions, independent of their kinetic energy. For the *Small* mode the acceptance area kept constant. Using *Large* mode it is recommended to use large slits and small pass energies (e.g. 4-20 eV with slit 6 x 20 mm) and vice versa for *Small* (e.g. 30 - 300 eV with slit diam. 1mm).

In the hemispherical capacitor the particles passing through the entrance slit S1 are focused onto the capacitor output plane S2. The radial position of the slit image in plane S2 depends on the kinetic energy of the particles in the capacitor. Particles on the central trajectory possess the nominal pass energy. They are focused to the central radial position. As the radial position of the slit image increases, for fixed capacitor potential, with the particle energy, particles with higher kinetic energy are focused further outside, and particles with lower energy are focused further inside in plane S2. This offers the possibility for multichannel detection, with simultaneous recording of an energy band around the nominal pass energy.

The particles passing through the capacitor output plane S2 are accelerated onto the detector system C. In the multichannel detector the particles are first multiplied using a multiplier arrangement. Each channel is connected to a separate preamplifier mounted outside the vacuum. The preamplifiers are read out by a Multi Channel Detector (MCD) counter interface of the SPECS data acquisition software.

The PHOIBOS system can operate in a Constant Retardation Ratio (CRR) or in a Constant Analyzer Energy (CAE) mode.



Figure 5

Analyzer vacuum housing (PHOIBOS 100)





Analyzer vacuum housing (PHOIBOS 150)



Figure 7

Main components of the spectrometer

| T1 to T10: | electrodes of the lens stage |
|------------------|---|
| S1: | hemispherical capacitor entrance slit |
| S2: | hemispherical capacitor exit plane |
| IH: | inner hemisphere |
| OH: | outer hemisphere |
| r _o : | nominal capacitor radius (150 mm) |
| C1 to C9: | discrete collection, single / multichannel detector (1 / 5 or 9 channels) |
| | multichannel assembly (MCD) |



Figure 8

Analyzer Voltage Principle

| U ₀ | main retardation voltage numerically equal to kinetic energy of the detected particles | | | |
|--------------------|--|-------------------|---------------------------|--|
| Un | slit potential -> numerically equal t | o pass energy | | |
| U _{Anode} | anode potential | UT | main transmission voltage | |
| Ud | detector voltage | U _{conv} | conversion voltage | |
| ГĪТ10 | lens potentials | IH, OH | inner / outer hemisphere | |

2.3.1 The Lens System

A lens system with the variable Slit Orbit mechanism is necessary to

- image the sample plane on the HSA entrance plane
- define the analyzed sample area and the accepted solid angle on a sample,
- accelerate / decelerate the particles with the observed energy to the pass energy.

By the lens stage, the particles emerging from the sample S are imaged onto the entrance slit S1, with the sample being in the focal plane of the lens system, i.e. 40 mm in front of the first lens electrode T1. If S1 has the dimension D1, then by theory the imaged area of the sample has the dimension DS with

$$\mathbf{DS} = \mathbf{D1} / \mathbf{M} \tag{EQ 1}$$

For the PHOIBOS the magnification of the lens stage is selectable to be M = 10, 3 or 1. in the *Small Medium* and *Large* lens mode. The magnification is changed electrically by connecting appropriate voltages to the lens electrodes. The voltages are a function of the spectrometer voltage U_0 , which depend on the particle kinetic energy being analyzed and the analyzer pass energy (see "Hemispherical Analyzer (HSA)" on page 12). U_0 is negative for electrons and positive for ions.

In the lens stage, the particles passing through an intermediate image and will focused onto the input slit S1 of the hemispherical capacitor (figure 7). At S1 the particles have been retarded by the energy difference between the nominal particle kinetic energy E_{kin} and the nominal pass energy E_{pass} .

The PHOIBOS can operate in Constant Retardation Ratio (CRR) and Constant Analyzer Energy (CAE) modes. In constant analyzer energy mode the applied voltage to the hemispheres is defined by equation 3 at page 13. In the constant retardation mode, Epass is given by $E_{pass} = E_{kin} / B$, with the retardation ratio B (Bremsfaktor).

As the trajectories of electrons emitted from the sample are influenced by electrical fields around the sample, T1 has a fixed potential, it is set to ground potential after switching on the power supply.

The actual size of the analyzer sampling area DS is in principle given by equation 1. Due to spherical aberration of the input lens, however, the image in plane of the entrance slit is diffused. The degree of diffusion increases, for fixed magnification, with the input lens acceptance angle. This means that also the viewed area in the focal planes of the input lens system is smeared out with increasing angle, resulting in larger sampling dimensions than given by equation 1. Thus the lens acceptance angle is selectable by the magnification modes to keep the spherical aberration in a well known acceptable value.

A second reason for confining lens acceptance angles are angle resolved measurements, e.g. in tilt experiments or angle resolved photoemission investigations. Confining the lens acceptance angle is also essential in ISS, as the kinetic energy in ISS depends on the scattering angle, and thus peak broadening or double peaks appear with too large lens acceptance angles.

Within the PHOIBOS magnification and angular aperture are selectable. There are 3 different combinations available. The 3 lens settings can be combined with the different possible slit pairs, resulting in 3 time 'number of slit pairs' combinations. The analyzer sampling areas and input lens acceptance angles for these combinations are given in table 1.

| Table 1 | Analyzer sampling areas and input lens |
|---------|--|
| | acceptance angles |

| lens | lateral magnification | max. acceptance angle (degree) | remarks |
|--------|-----------------------|-----------------------------------|-----------------------|
| Small | 10 | ± 8 | AES, ISS ^a |
| Medium | 3 | ±2 | |
| Large | 1 | ±4 | b c |

a. recommended for point sources

b. recommended for large area measurements

c. For large pass energies (>20eV) the peaks become asymmetrically because of overfilling.

2.3.2 Hemispherical Analyzer (HSA)

The hemispherical analyzer (HSA) with a mean radius R_0 (100 mm / 150 mm) performs the spectroscopic energy measurement, due to energy dispersion. Charged particles entering the HSA

through the entrance slit S_1 are deflected to elliptical trajectories by the radial electrical field between the inner hemisphere R_{in} and the outer hemisphere R_{out} . The radii of the PHOIBOS hemispheres are 1.25 R_0 and 0.75 R_0 , respectively. The entrance slit S_1 and exit plane S_2 , are centered on the mean radius R_0 :

$$R_0 = \frac{R_{in} + R_{out}}{2} = 150mm.$$
 (EQ 2)

For a fixed electrical field gradient, only particles with kinetic energies in a certain energy interval are able to pass the full deflection angle from the entrance slit S_1 to the exit plane S_2 . Particles with higher kinetic energy approach the outer hemisphere, whereas particles with lower kinetic energy are deflected toward the inner hemisphere. Those particles which enter the HSA normal to S_1 and move through the hemispheres on the central circular trajectory have the nominal pass energy E_{nass} :

$$E_{pass} = (-q) k\Delta V, \qquad (EQ 3)$$

where q is the charge of the particle, the potential difference $\Delta V = V_{out} - V_{in}$ applied to the hemispheres, k is the calibration constant,

$$k = \frac{R_{in}R_{out}}{2R_0(R_{out} - R_{in})} = 0,9375 \qquad . \tag{EQ 4}$$

These particles reach S_2 at the nominal radial position R_0 .

If the HSA accepts the half angle α in the dispersion direction, the HSA resolution or FWHM (full width at half maximum) of the transmitted line ΔE_{an} is given by

$$\frac{\Delta E_{an}}{E_{pass}} = \frac{S}{2R_0} + \frac{\alpha^2}{4} \qquad (EQ 5)$$

where $S = (S_1 + S_2)/2$. This value is an analyzer constant.

There are additional contributions to the line width observed in the spectrum. For photoemission lines, the main additional contributions are:

a) inherent line width of the atomic level ΔE_{level} (e.g. O 1s, C 1s),

b) natural line width of the characteristic radiation used for excitation ΔE_{photo} (e.g. Mg K α , Al K α). The observed total FWHM_{total} is given by the convolution of the single FWHMs, e.g for gaussian line withhs

$$FWHM_{total} = (\Delta E_{an}^{2} + \Delta E_{level}^{2} + \Delta E_{photo}^{2})^{1/2} = \Delta E .$$
 (EQ 6)

FWHM_{total} is usually specified using a sputter-cleaned silver sample and recording the Ag $3d_{5/2}$ level, after linear background substraction. For Mg K α excitation, the resolution at low HSA pass energies for the Ag $3d_{5/2}$ level is found to be

$$FWHM_{MgK\alpha} = 0.8eV. \tag{EQ 7}$$

In most practical work, a resolution of 0.9 eV is usually sufficient for high resolution investigations. For higher instrumental resolution, it is possible to use monochromatized X-radiation for excitation, e.g. mainly monochromatized Al K α radiation. Then the background is usually taken at 10 eV on the

high kinetic energy side of the Ag $3d_{5/2}$ level. For monochromatized Al K α radiation and for the Ag $3d_{5/2}$ level, the extreme resolution is found to be

$$FWHM_{extreme} = 0.44 \text{ eV}$$
 (EQ 8)

For attaining the extreme resolution of 0.44 eV, FWHM X-ray has to be strongly restricted, by utilizing only a small part of the X-ray monochromator, at the expense of a strong loss in intensity. In practical work, a resolution of 0.65 eV is usually sufficient for high resolution investigations with monochromatized Al K α excitation.

For monochromatized radiation, FWHM_{total} is sometimes specified recording the Si $2p_{3/2}$ level instead of the Ag 3d5/2 level, which results in smaller values of FWHM_{extreme}. due to the narrower inherent line width of the Si 2p level.

The integral signal intensity I of the measured particles (the area under the peak with a background subtracted) is proportional to product of the accepted solid angle Ω_S , the accepted sample area A_S and the HSA resolution ΔE_{an} :

$$I \sim \Delta E_{an} \Omega_S A_S = \Delta E_{an} \Omega_0 A_0 \frac{E_{pass}}{E_{kin}} \sim \frac{E_{pass}^2}{E_{kin}}, \qquad (EQ 9)$$

where Ω_0 and A_0 are the values of the acceptances for the HSA. They are analyzer constants. The equation results from Liouville's theorem¹.

The analyzer can be operated in two different modes: a) Constant Retardation Ratio (CRR), the retardation ratio B is defined as

$$B = \frac{E_{kin}}{E_{pass}}.$$
 (EQ 10)

In this mode all particles are decelerated with this same fixed factor. Therefore the pass energy is proportional to the kinetic energy. The intensity is increasing with the kinetic energy

$$I \sim E_{kin}, \qquad (EQ \ 11)$$

while the energy resolution is decreasing.

b) Constant Analyzer Energy (CAE), E_{pass} and ΔE_{an} in according to equation 5 are adjustable constants. The signals of all particles, independent of the kinetic energy, are measured with the same resolution. The intensity is decreasing with the kinetic energy:

$$I \sim \frac{1}{E_{kin}} \,. \tag{EQ 12}$$

^{1.} For more informations there are some excellent publications on analyzers. We recommend two of them:

K. D. Sevier, Low Energy Electron Spectrometry, Wiley-Interscience, 1972

D. Roy and D. Tremblay, Design of Electron Spectrometers, Rep. Prog. Phys. 53, 1621-1674,1990

The two modes are both generally possible for all kinds of measurements. There are some applications where one of them is traditionally preferred. The CRR mode is mostly used in AES, ISS and is convenient for the measurement of a survey spectrum. The CAE mode is mainly used in XPS and UPS when detailed information is needed and the resolution should not be dependent on the energy.

Besides, when E $_{\rm kin}$ is a constant, by measuring the same peak with a different pass energy, it follows that

$$I \sim E_{pass}^2. \tag{EQ 13}$$

2.3.3 HSA 3000 Voltage Ranges

All units provide the detection of electron energies between 0-3000 eV with minimum step widths of 13 meV. The unit can be upgraded by an additional voltage modules Hi-RES or Super Hi-Res allowing 0-300 eV scans with minimum step widths of 1.3 meV / 0.2 meV (Super Hi-RES only within a range 50 eV). This modules based on high resolution EELS technology for fine scans with step widths < 0.2meV and overall ripples below 1 meV for the Super Hi-RES.

| | supplied by | range | resolution | possible step with |
|-----------------------------------|-----------------------|----------|--|--------------------|
| lens potentials, E _{kin} | HSA 3000 standard | 0-3000 V | 18 Bit | <13 mV |
| lens potentials, E _{kin} | HSA 3000 Hi-RES | 0-300 V | 18 Bit | < 1.3 mV |
| lens potentials, E _{kin} | HSA 3000 Super Hi-RES | 0-300 V | 18 Bit within a range of max. 50 eV | < 0.2 mV |
| E _{pass} | HSA 3000 | 0-300 V | 18 Bit | <13 mV |

Table 2: Voltage Ranges Standard, Hi-RES and Super Hi-RES

2.3.4 Slit Orbit Mechanism

In the HSA a Slit Orbit mechanism is fitted as an optional feature for variable choice of slit pairs. The standard analyzer is build with one pair of slits chosen by the customer. The Slit Orbit mechanism is configured for example with following pairs of slits:

Table 3Slit Pairs

| S 1 | S2 |
|------------|-----------|
| 6 x 20 mm | 6 x 20 mm |
| diam. 6 mm | 6 x 20 mm |
| 4 x 15 mm | 4 x 15 mm |
| diam. 4 mm | 4 x 15 mm |
| diam. 1 mm | 1 x 3 mm |

For a given energy resolution and a given tolerated analysis space (area and acceptance angle), the largest possible slit area should be selected. This enabled the highest possible count rate for this parameters and therefore either a short measurement time or a good signal to noise ration in a given time.

The slits are arranged in a row on two slit planes which located in the entrance and exit planes of the hemispheres. When the external rotary dial is turned the slit plates move together across the entrance and exit planes of the hemispheres. The possible range is fixed by mechanically stops.

Although the entrance and exit slits are usually identical the above combination and other combinations may be specified within a pair. The resolution is determined by the mean slit width.

2.3.5 Single Channel Detector (SCD) / Multichannel Detector (MCD)

The detector consists of the following parts:

- arrangement of Channel Electron Multipliers (CEM; 1 for SCD, 5 or 9 for MCD) consisting of discrete collectors, specially screened against external HF - signals for maximum noise rejection;
- multi-pin ceramic high voltage vacuum feedthrough, specially designed for low cross talk;
- SCD / MCD preamplifier;
- high voltage divider for generating the voltages for the assembly.

2.3.5.1 Principles Detection

Due to the spherical symmetry of the HSA, a one - to - one image of the circularly shaped entrance slit with curvature radius R_0 exists in the exit plane, for monochromatic electrons with a nominal pass energy E_{pass} . The images of electrons possessing different energies within the HSA are concentric circles. In a first order approximation, the radial image position R for electrons with kinetic energy E_k is given by

$$\frac{R-R_0}{R_0} = \frac{E_k - E_{pass}}{E_{pass}} \cdot \frac{D}{R_0}, \qquad (EQ \ 14)$$

where D is the HSA dispersion. The theoretical value for D is

$$D = 2 \cdot R_0. \tag{EQ 15}$$

The experimentally determined dispersion value can be slightly different, mainly due to fringing fields at the edges of the analyzer.

Multichannel detection is performed by appropriately arranging 5 or 9 CEM^{\cdot} s as collectors with 5 or 9 exit slits on concentric circles in the exit plane. The radial distance between neighboring exit slits ΔR is selected to meet the requirement of a constant kinetic energy difference between neighboring channels ΔE_k .

The number of particles N_n arriving at each collector C_n is counted separately, and these numbers are stored and preprocessed in the data acquisition unit.

By sweeping the spectrometer voltage U_0 , the spectrometer is moved across each collector channel step by step, and in this way each collector records a complete spectrum, with a fixed energy offset between neighboring channels. Thus, in principle, by sweeping the spectrum once over the detector area, 5 or 9 parallel spectra are recorded simultaneously. As the kinetic energy E_n of the particles arriving at collector C_n is known from equation equation 14, the particle number from each channel, belonging to the same kinetic energy, can simply be added up, resulting in a total particle number for each kinetic energy.

2.3.5.2 Coherence E_{pass} and *Step*

From the analyzer energy dispersion equation, the energy difference ΔE_k between neighboring channels at the distance ΔR one from another is

$$\Delta E_k = \frac{\Delta R}{D} \cdot E_{pass}, \qquad (EQ\ 16)$$

or

$$E_{pass} = \frac{D}{\Delta R} \cdot \Delta E_k, \qquad (EQ \ 17)$$

where D is the analyzer dispersion.

Especially in the CRR mode, where the pass energy changes throughout the spectrum, and so does the energy difference between neighboring channels a calculation of the detected energy of the particle is necessary. Therefore a software routine calculates the particle number N_n in channel C_n at the nominal kinetic energy, by interpolation between the actually measured numbers in channel C_n at the measuring energies nearest below and nearest above the nominal energy.

This algorithm is unequivocal, because there is never more than one nominal energy between two measured energy positions. Due to the interpolation routine, there is no restriction on the energy step due to the analyzer performance. Power supply performance (DAC steps, etc.) limit the possible step widths and range borders. Therefore the software validate the values to the nearest allowed values.

2.3.5.3 Electron Multiplication

A Single Channel Electron Multiplier (channeltron or CEM) is a high gain device for detecting energetic particles such as electron and ions, or radiation. The CEM consist of a small, curved glass tube. The inside wall is coated with a high resistance material. The resistive material becomes a continuous dynode when a potential is applied between the ends of the tube.

By the impact of charged particle, secondary electrons are released from the CEM wall. These electrons are accelerated by the high voltage connected to the CEM and release additional secondary electrons by impact with the wall further along in the CEM. This effect is repeated successively, until finally an "electron cloud" is present at the exit of the CEM.

The average number of electrons leaving the CEM assembly per incident particle is called the gain G. For single particle detection, the gain has to be selected high enough to use the CEM's in "saturated" operation, i.e. each incident particle releases an electron cloud at the exit of the CEM arrangement whose charge is independent of small changes in multiplier voltage. The saturated operation is

necessary for sufficient noise rejection in single particle detection. Usually the minimum gain for saturated operation is about 10^7 , i.e. an electron cloud of more than 10^7 electrons leaves the CEM.

The electron cloud emitted out is finally post accelerated onto the collector electrode of the CEM, and the charge pulse carried by the electron cloud is detected as originating from one incident particle and counted in the preamplifier channel.

One or a set of CEM's, is used in a special arrangement as an electron multiplying component for the PHOIBOS analyzers. The CEMs are all parallel mounted as a unit on a feedtrough flange. The particles passing the exit aperture are post-accelerated to an appropriate kinetic energy onto the CEM.

The operating point for channel electron multiplier (CEM) in the pulse counting mode is usually determined by the point at wich a plateau is reached in the count rate vs voltage characteristic. The plateau occurs when all the signal is being collected at the input of the CEM. Additional increases in voltage raise the gain, but the count rate remains essentially constant. Eventually, a point is reached where ion feedback becomes significant due to the very high gain, and the count rate increase rapidly. This is an undesirable condition since the extra counts are produced within the CEM itself and are not the result of an input. The optimum operating point is about 50 - 100 V beyond the 'knee' of the curve. As the multiplier ages, the knee moves to the right and the voltage must be increased. In general, CEM lifetime will vary as a function of specific application and environment, but is typically on the order of about one year at 40 hours operation per week.



Figure 9

Detector sweep, Count rate vs voltage.

The sensitivity of the preamplifier channels can be varied using a discriminator threshold potentiometer located at the outer face of the MCD preamplifier housing (the value is factory preset, recommended discriminator voltage see section 7.1.5.1).

The pulse output depends largely on the applied voltage and in practice the gain is an increasing function of the applied voltage until the gain reaches about 10^7 : after which point increasing the voltage further will cause the eventual breakdown of the CEM.

(With an proper configured oscilloscope, i.e. impedance 50 Ohm, the necessary pulse height can be checked.)

For the PHOIBOS analyzer an input of one electron the CEM responds by producing an output pulse of charge which contain at least 10^7 electrons and which lasts for approximately 10 nanoseconds.

2.3.5.4 Conversion Voltage

The conversion voltage produced in the HSA 3000 determines the conversion energy

$$E_{conv} = -qU_{conv} \qquad (EQ\ 18)$$

of the charged particles impinging the CEM. The proper conversion voltage has two requirements which must be simultaneously fulfilled:

- the particles energy should be suitable for maximum yield of secondary electron emission at the impact on the CEM wall. This for electrons is roughly in the energy range between 100 and 800 eV.
- For ions, the yield increases with the kinetic energy roughly up to 10 keV.

Standard settings are:

| ٠ | for electrons: | $U_{conv} = +128 V$ | |
|---|----------------|-----------------------------------|--------|
| | | $(U_{conv} - E_{kin} < or equal)$ | 128V). |
| • | for ions: | $U_{conv} = -3 \text{ kV}.$ | |

2.4 Work Function

The basic energetic properties are shown in figure 10 for the example of the measurement of photoelectrons.



Figure 10

Energy scheme in case of photo electron spectroscopy

The spectrometer and the sample are connected to ensure that the Fermi-energies are at the same reference level. The binding energy of the electrons is given by

$$E_{bin} = h\nu - E_{kin} - Wf_{sample} \tag{EQ 19}$$

The energy E'_{kin} (see figure 10) is measured by the spectrometer and after calibrating the work function of the spectrometer, the binding energy of the sample relative to the Fermi level can be measured without knowing its work function because

$$E_{kin} + Wf_{sample} = E'_{kin} + Wf_{spectr}.$$
 (EQ 20)

Chapter3Installation

3.1 Unpacking

Please see "Appendix" on page 45.

3.2 Vacuum Installation

Please see "Appendix" on page 45.

3.3 Electrical Units Installation

The electronic units have to be installed into a 19"-cabinet rack. Good air circulation within the cabinet must be ensured. For wiring of the electronics follow figure 1. Mind the following:

- 1. Connect all units to the same multiple socket.
- 2. The outlet strip must be provided with a protecting line according to the regulations.
- 3. A grounding bar (copper, brass) with a minimum cross section of 6.6 mm^2 has to be installed inside the cabinet. The electronic unit (except for MCD preamplifier) have to be connected to this grounding bar by means of flat braided grounding ribbon, which is connected to the back panels of the electronic unit.
- 4. The grounding bar has to be connected to the screw at the analyzer plug of the spectrometer by means of a flat (minimum width 10 mm) braided grounding ribbon or a cable (black isolation) of minimum cross section of 10 mm².

The connections between analyzer, control unit and computer are described in "Electrical Connections" on page 3.

These connections have to be made before the first operation of the system.

3.4 Preliminary

In case of XPS, the analyzer and X-ray source should be installed to minimize the distance between the X-ray source and the sample. The distance between the sample and the lens T1 must be fixed to the working distance of 40 mm. None of these three parts should have mechanical contact to each other.

Check the resistivity of the pins of the HSA 12-pin (figure 3) and detector feedthroughs (figure 4) to ground and to each other to exclude short circuits. This is especially important to check short circuit between ground plate and cathode. The resistivity has to be infinite for all cases, except between the cathode and anode contacts of the CEM, which should be higher than 20 M Ω internal resistance.

The performance of the MCD is decreasing with the time exposed to air. Try to minimize the time between its mounting and evacuating.

3.5 Baking Out

The vacuum chamber has to be baked out to get good UHV in a reasonable time. The temperature during the bake out should be up to 150° C. A reference thermocouple for the temperature measurement should be attached to the MCD flange (F5 in figure 6)!

Before baking out,

- HSA cable
- preamplifier with connection cables to the flange

must be removed (see figure 1).

The preamplifier can be removed by release one screw. Then the complete unit should be stored for the time of bakeout.

A bakeout time between 12 hours and 24 hours (first time) is recommended.

The interior parts of the PHOIBOS will cool down significantly slower than the housing. The multiplier will degas at first operation after bakeout, so care should be taken to use the detector not at full multiplier voltage and not at full intensity within the first operating hours after bakeout. Recommended is a wait for complete cool down of the detector assembly of approximately 1 day.

We recommend a increasing of the detector voltage over a period of 1.5 hours for the first time and over a period of 10 min for the next times after bake out.

(Use the SpecsLab mode 'Detector Sweep' and set start, end, step and dwelltime parameter for this procedure. Check with 'Validate' the run time for this special 'measurement'.)

Chapter 4 System Operation

4.1 First Operation

If the system is baked (see "Baking Out" on page 22) the vacuum should be controlled. The base pressure should be lower than 10^{-7} mbar (see "Functional Test" on page 24).

Check the electrical connections (see "Electrical Units Installation" on page 21)

4.1.1 Calibration of the Slit Orbit Mechanism

There are different settings available with the PHOIBOS Slit Orbit mechanism (see "Slit Pairs" on page 15). The counter clockwise turn will set to larger slits concerning the possible slit pairs for this analyzer.

The optimum setting is reached, when entrance slits are aligned along the lens axis, i.e. the particle number passing through the lens stages and impinging on the hemispherical capacitor entrance slit S1 is maximum. This is also right the position for the exit slit S2.

In positioning the feedthrough (F2 on figure 6, "Analyzer vacuum housing (PHOIBOS 150)," page 9) to the slit locations, the rotary dial is internally fixed near to the right value by a mechanical rest position.

4.1.2 Detector Operation

For new multiplier (CEM) please read the start-up procedure given for the CEM after bakeout in section 3.5. Normal procedure after bakeout is increasing in small steps (50V) within few minutes over a period of 1 - 2 hours for start-up the detector voltages (see "Electron Multiplication" on page 17 also).

Common:

• A dry-pumped or well-trapped/diffusion-pumped operating environment is desirable. A poor vacuum environment can shorten CEM life or change the operating characteristics.

- A pressure of $1 \cdot 10^{-6}$ mbar or lower is preferred. Higher pressure can result in high background noise due to ion feedback.
- Voltage should be applied to the MCD in small (100 200 V) steps.
- For optimal lifetime, operate the detector at the minimum voltage necessary to obtain an usable signal (see "Electron Multiplication" on page 17).

4.1.3 Functional Test

The detector unit should have been baked out. A silver sample with a size not smaller than 5.14 mm^2 should have been transferred into the vacuum chamber. The sample has to be cleaned by sputtering. The base pressure should be lower than 10^{-7} mbar (10^{-5} Pa) to avoid a damage of the detector by sparks. For special reasons, e.g. for depth profiling with noble gas ions, operation up to $4 \cdot 10^{-7}$ mbar ($4 \cdot 10^{-5}$ Pa) is allowed.

Set the software to XPS $E_{kin} = 800 \text{ eV}$ (start short measure, voltage will stay at the end energy), the pass energy to ~20.0 eV largest slit and the X- ray source to 100 W. Adjust the detector voltage to the value corresponding to the Specification Report of the analyzer (nominal value is 2400 V).

Use the SpecsLab mode 'Detector Sweep' and set start, end, step and dwelltime parameter for this procedure. Check with 'Validate' the run time for this special 'measurement'

Measure a wide energy XPS spectrum scanning the kinetic energy of the particles from 0 to 1.5 keV, e.g. with an energy step of 500meV and pass energy of ~20.0 eV.

In a first rough test without careful adjustment one should get 10^5 cps at the Ag $3d_{5/2}$ peak using 100 W Mg K α X-ray source power and a clean silver sample.

If not, check the photoemission sample current, which should be in the range of 25-40 nA for 100 W Mg K α . Secondly, check the MCD voltage, and the discriminator level of the MCD preamplifier. Check also whether the intensity of the C- and O-peaks are smaller than 2% of the Ag $3d_{5/2}$ peak. Otherwise the sample should be sputtered once more.

4.2 Quick Operation

- 1. Check vacuum conditions.
- 2. Check sample.
- 3. Switch on the analyzer supply HSA 3000.
- 4. Start the acquisition (SpecsLab) and control (source window) software.
- 5. Set detector voltage and other parameter in the source window
- 6. Switch on the excitation source.
- 7. Set the scan parameter for the region.
- 8. Press measure.
- 9. Save the results.
- 10. Switch off the units.

Chapter **5**

Analyzer Test

5.1 Independence of peak position with pass energy

Transfer a silver sample into the system. Warm up your electronic for at least 1 hour. Adjustments:

| Excitation | Mg Kα |
|----------------------|--------------|
| X-ray power | 100 W |
| Slit | 6 x 20 mm |
| Mode | LARGE |
| Binding energy range | 365 - 375 eV |
| Scan | 10 |
| Step time | 100 msec |
| Energy step | 25 - 30 meV |
| Pass energy | ~ 15eV |

Run a spectrum. Set pass energy of about ~5 eV. Repeat the measurement. Compare the spectra. If the voltages fit to the spectrometer, the peak-maxima of both spectra have to point exactly to the same energy. The peaks positions of both spectra should not differ by more than 100 meV. For symmetrical peaks the peak position is independent of E_{pass} . This is not true for Auger electron peaks since the peaks are intrinsically asymmetrical.

If the peak positions differ, a calibration might be needed for the hemisphere voltages U_{GK} (Outer Hemisphere). The voltage set in the HSA 3000. The calibration should be done by vary U_{GK} using the potentiometer P16 in the HSA 3000. To use the potentiometer the rear panel of the HSA 3000 has to be opened.

Usually no calibration should be necessary. For more detailed information please contact SPECS support.

5.2 Kinetic energy scale test

5.2.1 Preparation

Transfer a gold/copper sample into the system. Clean carefully by ion sputtering. With this sample the main peaks are separated far enough for calibration purposes, namely, the Au $4f_{7/2}$ and Cu $2p_{3/2}$ peaks energy difference is 848.7 ± 0.1 eV.

Adjustments:

| Excitation | Mg Kα |
|----------------------|---|
| X-ray power | 100 W |
| Slit | 6 x 20 mm |
| Mode | LARGE |
| Binding energy range | 82 - 86 eV for the Au $4f_{7/2}$ peak |
| | 930 - 937 eV for the Cu $2p_{3/2}$ peak |
| Step time | 100 msec |
| Scan | 10 |
| Energy step | 25 - 30 meV |
| Pass energy | ~8 eV |

5.2.2 Check Peak Position

Check for proper peak positions corresponding to table 4. Note the error. If the peaks offset consistently, check if the proper peak position can be achieved by recalibrating the work function (software WF). If not, a kinetic energy calibration is needed (please contact SPECS).

Table 4Calibration Binding Energies for
non-monochromated Mg Kα X- rays

| Peak | Binding energy, eV |
|----------------------|--------------------|
| Au 4f _{7/2} | 84.00 ± 0.01 |
| Ag 3d _{5/2} | 368.27 ± 0.01 |
| Cu 2p _{3/2} | 932.66 ± 0.02 |

5.3 Specification Check

5.3.1 Survey Spectrum of Silver

The XPS performance of an energy analyzer is usually determined using a silver sample. A cleaned silver sample has to be brought into the vacuum chamber and cleaned by ion sputtering. Make the same adjustments as in the overview spectrum of silver enclosed with the Specification Report on the analyzer. For example:

| Excitation | Mg Ka |
|----------------------|-------------|
| X-ray power | 300 W |
| Slit | 6 x 20 mm |
| Binding energy range | 1000 - 0 eV |
| Energy step | ~300 meV |
| Step time | 100 ms |
| Pass energy | ~20 eV |

a typical XPS overview spectrum taken with an PHOIBOS on silver is shown in figure 11.

5.3.2 Intensity and Resolution

Make the same adjustment as in the Ag 3d doublet spectrum enclosed with the Specification Report on the analyzer.

For example:

| Excitation | Mg Ka |
|-------------------|--------------|
| X-ray power | 300 W |
| Slit | 6 x 20 mm |
| Bin. energy range | 378 - 364 eV |
| Step time | 100 msec |
| No. of scans | 10 |
| Step width | 25 - 30 meV |
| Pass energy | ~6.0 eV |

the well resolved Ag 3d doublet is shown in figure 12. This spectrum is typical for an PHOIBOS. The signal (net intensity, i.e. peak count above background) of the Ag $3d_{5/2}$ peak is about 200 kcps. The background in this case is defined as a straight line between the two neighbor valleys on both sides of the peak. The FWHM of the Ag $3d_{5/2}$ peak is calculated by measuring the peak width at the half height between the peak maximum and the background.

Note:

Because of considerable gain spread of different CEM's the voltage needed for the signal and FWHM specification for the detector unit installed may differ from the nominal value of 2400 V (see section 7.2). Pay attention to the detector voltage value in the Specification Report on the analyzer. With sample and X- ray source positions optimized, the detector voltage, at which FWHM of the Ag $3d_{5/2}$ peak is 0.90 eV, is the working detector voltage.

If measured spectra and achieved values differ essentially from the ones of the Specification Report on the analyzer, it might be that the sample should be sputtered some more, the sample and X- ray source positions should be optimized, or a HSA 3000 calibration might be needed. See although section 6.

In many cases it is helpful to know the intensities, signals, and accompanying FWHMs at different values of pass energy. For such purpose, the measurements described above have to be made at pass energies nearest to the values of 2, 5, 10, 20 and 50eV for slit 6 x 20 mm and up to 200 eV for

diam.1mm slit.

The step time or number of scans should be adapted to the pass energies in such a way that the maximum intensities (in counts not in counts per second) are about the same in every case. Namely, for low pass energies, choose a higher step time than for high pass energies. This gives comparable counting statistics for all measurements

5.4 Typical Spectra

5.4.1 XPS



Figure 11

XPS on silver, wide scan (PHOIBOS 150 SCD)

Parameter for the survey spectrum in figure 11:

| Lens mode: | Large |
|---------------------|-----------|
| Slit: | 6 x 20 mm |
| Sample current: | 128 nA |
| Detector voltage: | 2400 V |
| Conversion voltage: | 128 V |



Figure 12

XPS on silver, Ag 3d (PHOIBOS 150 SCD)

Parameter for the Ag 3d spectrum in figure 12:

| Medium |
|-----------|
| 6 x 20 mm |
| 140 nA |
| 2400 V |
| 128 V |
| |

Chapter 6

Troubleshooting Procedure

In the following a list of possible problems or anomalies and hints for their removal is given. It is assumed that the system was calibrated properly and was working according to the specification before one of the following problems occurred.

6.1 Possible Problems

The following problems may occur during operation of the analyzer system:

- no spectrum
- low intensity
- low resolution
- peaks shifted
- intensity fluctuations
- high background signal
- noisy spectrum
- wrong analyzed area
- Slit Orbit problems

The each statement following ", \rightarrow ", indicates a separate troubleshooting procedure either given in this paper or in another manual.

Table 5No Spectrum

| possible cause | perform check, test, or troubleshooting procedure no. $ ightarrow$ |
|--|--|
| X-rays off | check the X-ray source and the sample current |
| no voltage at detector | check your detector voltages |
| spectrum definition wrong | check spectrum definition |
| cable connection faulty | check cable connections |
| preamplifier box defective | check preamp box \rightarrow section 7.1.5 |
| counter device in control unit defective | contact SPECS |

| Table 5 | No Spectrum |
|---------|-------------|
|---------|-------------|

| possible cause | perform check, test, or troubleshooting procedure no. \rightarrow |
|-----------------------------------|---|
| no energy sweep voltage | check energy sweep \rightarrow section 7.1 and section 7.1.1.1. |
| spectrometer voltages wrong | check spectrometer voltages \rightarrow section 7.1 |
| improper adjustment of slit orbit | check proper adjustment \rightarrow section 4.1.1 |

Table 6Low Intensity

| possible cause | perform check, test, or troubleshooting procedure no. \rightarrow |
|--|--|
| X-ray intensity too low | check whether on Ag sample photo current ≥ 0.2 - 0.3 nA/W |
| sample is dirty | sputter until C and O peak in the spectrum dis- appears |
| sample too rough | remove roughness |
| wrong analyzer - sample distance | adjust distance to 40 mm |
| lens system and HSA out of focus | check lens and HSA electrodes → section 7.1.3 and voltages section 7.1.4 |
| CEM yield to low | measure detector supply voltages → section 7.1.5 |
| conversion voltage too low | check conversion voltage → section 2.3.5.4 |
| MCD preamplifier setting changed | check preamp settings \rightarrow section 2.3.5.3 |
| some channels of the preamplifier unit | check operation of the preamplifier by use of a |
| defective | separated channel mode of the control unit or → section 7.1.5 |
| magnetic fields | Check whether the amount of the deviation depends on the energy of the measured elec- trons and on the pass energy (better by UPS). Check the influence of an external permanent magnet near the vacuum chamber. |
| mechanical wrong adjustments like: | Open analyzer system and check. Do this only |
| grids damaged, lenses misaligned, spheres shifted, MCD shifted. | if all other kinds of faults can be excluded. |
| improper adjustment of Slit Orbit mechanism | check proper adjustment section 4.1.1 |

Table 7Low Energy Resolution

| possible cause | perform check, test, or troubleshooting procedure no. $ ightarrow$ |
|---------------------------------------|--|
| chemical peak broadening | sputter cleaning |
| lateral inhomogeneous charging of the | use charge compensation by means of electron |
| sample | flood gun |

| possible cause | perform check, test, or troubleshooting procedure no. $ ightarrow$ |
|---|--|
| noise or/and ripple on the following | ground sample and check hum and ripple check |
| voltages: | the ground connection of all the power sup- |
| sample, spectrometer voltage U ₀ , | plies \rightarrow section 3.3 |
| detector voltage, | |
| electrode voltages of the analyzer | |
| magnetic fields in the region of the | measure magnetic field |
| spheres | |
| preamplifier setting changed | check preamp setting \rightarrow section 2.3.5.3 |
| lens system and HSA out of focus | check lens and HSA electrodes \rightarrow section 7.1.3 |
| | and voltages section 7.1.4 |
| detector supply voltage wrong | check detector supply voltages \rightarrow section 7.2 |
| carbon coating of HSA spheres damaged | open HSA and check - |
| | do this only if all other checks are negative |
| Humming and ripple on the following | ground sample, check humming and ripple |
| parts: sample, spectrometer voltages, , | |
| multiplier voltage, lens voltage | |

Table 7Low Energy Resolution

Table 8Peaks Shifted Equally

| possible cause | perform check, test, or troubleshooting procedure no. $ ightarrow$ |
|--|---|
| work function setting wrong | Check the work function setting. |
| sample charging | Check the sample ground connection. Use external electron flood gun to compensate for the charging. |
| voltages of the HSA (inner and outer sphere) are wrong | In similar spectra with different pass energies the peaks are shifted. Check HSA voltages → section 7.1.3 |
| zero point drift of the spectrometer volt- age | check zero point of HSA 3000 → section 7.1.1.3 |

Table 9Peaks Shifted Differently

| possible cause | perform check, test, or troubleshooting procedure no. $ ightarrow$ |
|----------------------------|--|
| wrong amplification factor | check U ₀ HSA 3000 \rightarrow section 5.2, section 7.1.1, section 7.1.1.2 |

Table 10Intensity Fluctuations

| possible cause | perform check, test, or troubleshooting procedure no. \rightarrow |
|------------------------------|---|
| malfunction of counter board | \rightarrow control unit manual |

| - | |
|----------------------------------|---|
| possible cause | perform check, test, or troubleshooting procedure no. \rightarrow |
| one lens electrode not connected | Run a spectrum with high speed. |
| | Perform a crosstalk test with all lens tubes. \rightarrow |
| | section 7.1.3.1 |
| malfunction of the energy sweep | check the energy sweep \rightarrow |
| generator | section 7.1.1.1, section 7.1 |

Table 10Intensity Fluctuations

Table 11High Background Signal

| possible cause | perform check, test, or troubleshooting procedure no. \rightarrow |
|---------------------------------------|---|
| field emission at the exit slit of | Background signal independent of Ekin with |
| the HSA | ΔE =const, rises with small E_{pass} - values and |
| | increases in $\Delta E/E$ =const mode. Remove wire- |
| | edge from the mesh at the exit slit. |
| sparks at the anode side | background signal is independent of E_{kin} in $\Delta E/$ |
| of the CEM | E=const mode and background to signal ratio |
| | decreases with increasing of the pass energy. |
| false detector preamplifier threshold | \rightarrow section 2.3.5.3 |

Table 12Noisy Spectrum

| Possible Cause | perform check, test, or troubleshooting procedure no. \rightarrow |
|---------------------------------------|---|
| false detector preamplifier threshold | \rightarrow section 2.3.5.3 |
| high noise of the primary source | on silver sample by different E _{pass} measure signal at the Ag 3d _{5/2} peak (368 eV bin.energy) and noise at the 353 eV bin.energy → signal to noise ratio is independent of E _{pass} |
| electric interference | monitor mains voltage, check ground connec- tions \rightarrow section 3.3 |

 Table 13
 Wrong analyzed area in Small /Medium / Large mode

| possible cause | perform check, test, or troubleshooting procedure no. \rightarrow |
|--------------------------------|---|
| malfunction of lenses | Check tubus voltages. The voltage can be mea- sured either at the 12 pin feedthrough (figure 3 on page 6) |
| income a standard and a fith a | (ligure 5 on page 6): |
| improper adjustment of the | cneck proper adjustment section 4.1.1 |
| Slit Orbit mechanism | |

Chapter

Power Supply Check and Settings

Adjustment and calibration of power supplies have been performed at the factory.

To proof the analyzer, test that the peak position independent of pass energy, kinetic energy scale and check the analyzer specification, see section 5.

Attention!! Before taking off any power supply cover and changing the trimmers set, please consult SPECS. Taking off the cover without a SPECS written consent will void the SPECS warranty!!

Mind the safety hints given on page 2 !!

If no spectrum but a straight line appears after the control unit has been started, either no pulses are arriving at the control unit counter, the counter is defective, or the control voltage from the sweep generator (usually a DAC) is missing. The following checks should be made:

- 1. Check the cable connections (figure 1, figure 3 and figure 4)
 - between counter and preamplifier as well as
 - the connection between HSA 3000 and HSA and
 - the PC and the HSA 3000.
- 2. Control Unit Check, see 7.1
- 3. Preamplifier Check, see 7.1.5
- 4. "Detector settings" on page 40

7.1 Control Unit Check

In this section a connection check for the contacts of the electrodes is made, and the spectrometer voltage U_0 is verified.

For this check a digital voltmeter with a HV probe and capacity measurement ability must be used. The

voltages will drop slightly due to the loading by the voltmeter and HV probe resistance. For these tests this is not important.

7.1.1 Kinetic Energy Check

7.1.1.1 Sweep generator

The sweep generator is usually realized as a DAC board in the HSA 3000. There is normally no adjustment of the DAC necessary. For more detailed information please contact SPECS. To check the DAC please check the spectrometer voltage U_0 refer to section 7.1.1.2.

7.1.1.2 Check the spectrometer voltage U₀

For this check a digital voltmeter with a HV probe should be used.

!! Mind the safety hints given on page 2 **!!**

Set kinetic energy to 0 eV (note WF and start measurement in fixed mode with start energy 0). Connect a digital voltmeter with HV probe to the connector of the pin marked 'KINETIC ENERGY' at the rear panel of the HSA 3000 power supply (see "Connection scheme of the PHOIBOS SCD components" on page 4).

Run a spectrum from 0 to 200 eV and observe the variation of the spectrometer voltage U_0 . If U_0 varies within the chosen energy range, the spectrometer voltage generation is o.k.

7.1.1.3 Zero Check

Set the work function in the software to zero. Set the kinetic energy of 0.000 eV. Set the multiplier voltage to 0 V. Measure with the digital voltmeter (5 1/2 digit type) E_{kin} . Check the output voltage of the HSA 3000.

7.1.2 Check the voltage range of the HSA 3000

7.1.2.1 HSA 3000 range: 3 kV

Preparation

Transfer a gold/copper sample into the system. Clean carefully by ion sputtering. With this sample the main peaks are separated far enough for calibration purposes, namely, the Au $4f_{7/2}$ and Cu $2p_{3/2}$ peaks energy difference is 848.7 ± 0.1 eV. Adjustments:

| agustinents. | |
|-------------------|--|
| Excitation | Mg Kα |
| X-ray power | 100 W |
| Bin. energy range | 81- 87eV for the Au 4f _{7/2} peak |
| | 930 - 936 eV for the Cu $2p_{3/2}$ peak |
| Step time | 50 msec |
| | |

| Energy step | 25 - 30 meV |
|-------------|-------------|
| Pass energy | ~30 eV |

Run spectra of both peaks.

Measure the distance between the peak maxima of gold and copper. If the value found differs from 848.7 eV please inform SPECS. Of course, after every adjustment of the analyzer or replacement of modules HSA 3000 a new spectrum has to be taken to control.

Note: Please taking into account the software set workfunction and possible charges on the sample.

7.1.3 Connection Check for the HSA Electrodes

Check the electrode connection with the electrical feedtrough. For this check a capacity measurement must be used. Best way to check is to measure the capacities between all pins of the feedtrough of the analyzer (see figure 3, "Schematics of the 12-pin HSA feedthrough," page 6.). Switch off the HSA 3000 and remove the connector to the HSA electrodes.

!! Mind the safety hints given on page 2!!

The capacities measured on the HSA and lens electrodes under UHV conditions in table 14, respectively table 15 are only for information. Some differences across the meter should be taken into consideration when checking the values. Comparable absolute values, but correct ratios between the values shows correct connection to the analyzer parts:

| without | PIN | housing | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 |
|---------|---------|---------|----|----|----|----|----|----|----|----|-----|----|-----|----|
| 1 | housing | | 47 | 51 | 52 | 58 | 70 | 51 | 51 | 62 | 201 | 70 | 164 | |
| 0 | 1 | | | 60 | 36 | 34 | 35 | 28 | 29 | 33 | 47 | 30 | 46 | |
| 0 | 2 | | | | 50 | 38 | 39 | 30 | 30 | 33 | 51 | 33 | 47 | |
| 1 | 3 | | | | | 52 | 43 | 33 | 32 | 33 | 51 | 33 | 47 | |
| 1 | 4 | | | | | | 58 | 34 | 33 | 34 | 53 | 33 | 51 | |
| 0 | 5 | | | | | | | 48 | 37 | 38 | 60 | 36 | 56 | |
| 0 | 6 | | | | | | | | 47 | 36 | 51 | 32 | 46 | |
| 1 | 7 | | | | | | | | | 48 | 53 | 33 | 48 | |
| 1 | 8 | | | | | | | | | | 70 | 39 | 60 | |
| 2 | 9 | | | | | | | | | | | 99 | 227 | |
| 1 | 10 | | | | | | | | | | | | 82 | |
| | 11 | | | | | | | | | | | | | |
| | 12 | | | | | | | | | | | | | |

Table 14Capacity measurements (pF), PHOIBOS 100

| without connection | PIN | housing | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 |
|-----------------------|---------|---------|----|----|----|----|----|----|----|----|-----|-----|-----|----|
| 8 | housing | | 52 | 53 | 56 | 56 | 54 | 43 | 45 | 58 | 298 | 117 | 259 | |
| 9 | 1 | | | 70 | 45 | 40 | 40 | 33 | 33 | 37 | 57 | 44 | 54 | |
| 9 | 2 | | | | 57 | 44 | 43 | 34 | 33 | 37 | 57 | 45 | 56 | |
| 8 | 3 | | | | | 58 | 44 | 35 | 35 | 37 | 61 | 46 | 57 | |
| 10 | 4 | | | | | | 59 | 38 | 37 | 39 | 61 | 46 | 57 | |
| 3 | 5 | | | | | | | 42 | 31 | 31 | 51 | 40 | 50 | |
| 3 | 6 | | | | | | | | 45 | 33 | 45 | 36 | 45 | |
| 4 | 7 | | | | | | | | | 45 | 48 | 37 | 45 | |
| 3 | 8 | | | | | | | | | | 64 | 46 | 58 | |
| 2 | 9 | | | | | | | | | | | 169 | 382 | |
| 2 | 10 | | | | | | | | | | | | 132 | |
| | 11 | | | | | | | | | | | | | |
| | 12 | | | | | | | | | | | | | |

Table 15 Capacity measurements (pF), PHOIBOS 150

If the measured capacities differ substantially from the nominal values please contact SPECS. If the capacities have nearly the right values, no short circuit inside the spectrometer is most probably. Proceed according to the troubleshooting procedure given in the manuals.

If the measured capacities have the correct values, a missing contact from the HSA 3000 to the analyzer may be the reason for a faulty spectrum. Missing contacts can be found be the following method.

7.1.3.1 Check the cable contacts

Before this test the correct generation of the spectrometer voltage U₀ should be checked (see section 7.1.1.2).

!! Mind the safety hints given on page 2 **!!**

Check also whether the contacts are in good condition. Since the plug is a movable part which is frequently plugged and unplugged, it is able to become defective. In most cases of contact failures the plug is the reason.

7.1.4 Check HSA voltages

Check whether the electrode voltages are present at the female contacts of the spectrometer plugs. Write down the measured voltages for a given kinetic energy and a given pass energy (note WF in software) in table 16. Send this table to SPECS.

If the measured values are correct, the plug is o.k.

| E _{kin} = | $\mathbf{E}_{\mathbf{paas}} =$ | Slit pair: | WF = | | |
|--------------------|--------------------------------|------------|--------|-------|--|
| voltages | PIN | Large | Medium | Small | |
| T1 | 1 | | | | |
| T2 | 2 | | | | |
| T3 | 3 | | | | |
| T4 | 4 | | | | |
| T5 | 5 | | | | |
| T6 | 5 | | | | |
| T7 | 6 | | | | |
| T8 | 7 | | | | |
| Т9 | 8 | | | | |
| T10 | 9 | | | | |
| inner hemisphere | 10 | | | | |
| outer hemisphere | 11 | | | | |
| not used | 12 | | | | |

Table 16Voltages to ground for all modi

Connect the spectrometer plug and start a spectrum. If no spectrum is obtained, the faulty contact is most probably inside the spectrometer (see "Connection Check for the HSA Electrodes" on page 37). If something like a spectrum is obtained, the missing contact can be found by connecting the electrodes one by one to a different potential (ground or a neighboring electrode potential in the HSA 3000), and taking a spectrum every time. If, after an electrode has been switched, the spectrum does not change its shape, the faulty electrode contact has been found.

7.1.5 Preamplifier Check

7.1.5.1 Discriminator

Check the discriminator threshold using the noise of the signal within one spectrum. The square root of the signal should be equal to the RMS (root mean square) of the noise at this energy.

Use the fixed mode of the acquisition software to estimate the noise at constant kinetic energy for the acquired signal and compare with the square root of the signal (counts not cps). Change the discriminator threshold by the potentiometer at the preamplifier box and check again. If both values similar the discriminator threshold has the right value.

7.1.5.2 Amplifier Check

Set the detector voltage to zero! !! Mind the safety hints given on page 2!!

Disconnect the preamplifier from the detector flange.

Upon light touching with a piece of wire to each pin hole of the preamplifier pin connector, signal must be observed 'number of channels' times on the monitor.

If signal is observed a smaller number of times, not all channels of the MCD preamplifier are o. k..

If no signal or a constant signal is observed, the cable connections between the preamplifier and the HSA 3000, and the preamplifier power must be tested.

7.2 Detector settings

7.2.1 Preamplifier

See section 7.1.5 "Preamplifier Check" on page 39.

7.2.2 Detector Voltage

Because of considerable gain spread of different multipliers the voltage needed for the signal and FWHM specification for the MCP installed may differ from the nominal value of 1700 V.

Pay attention to the detector voltage value in the Specification Report with the analyzer.

Basically, a working detector voltage is the detector voltage, at which FWHM of the Ag $3d_{5/2}$ peak is 0.90 eV (see section 5.3.2), with sample and X- ray source positions optimized.

7.3 WORK FUNCTION

Typical values of the analyzer work function are between 4 eV and 5 eV. The compensation is performed by addition through the software (see software manual).

Chapter **8**

Spare Parts

8.1 Cu Gasket

Cu Gasket DN 350 CF

8.2 Multiplier

8.2.1 SCD multiplier replacement

Single Channel Electron Multiplier for PHOIBOS analyzer

8.2.2 MCD, CEM Array replacement

SPECS order 79150134 5 channels MCD for PHOIBOS analyzer

SPECS order 79150062 9 channels MCD for PHOIBOS analyzer

8.2.3 Channeltron Handling, Storage and Operation

A channeltron or a channeltron array (array of 5 or 9 of single channel multipliers, fused together in a precision matrix) is a high gain device for detecting energetic particles such as electron and ions, or radiation. The channeltron consist of a small, curved glass tube. The inside wall is coated with a high resistance material. The resistive material becomes a continuous dynode when a potential is applied between the ends of the tube. It is fabricated from a lead- doped glass. Proper handling is required and the following precautions must be taken.

8.2.4 Handling

• Shipping containers should be opened only under clean, dust-free conditions.

- No physical object should come in contact with the active area of the detector. The channeltron should be handled by its solid borders using clean, degreased tools fabricated from stainless steel, teflon (PTFE) or other UHV compatible materials.
- The channeltrons should be protected from exposure to particle contamination. Particles which become affixed to the plate can be removed by using a single- hair brush and an ionized dry nitrogen gun.

8.2.4.1 Storage Multiplier

Due to the hygroscopic nature of the doped lead glass, it is important that the channeltrons are stored properly.

Warning: The shipping containers are not suitable for storage periods exceeding the delivery time. Upon delivery to the customer's facility, channeltron must be transferred to a suitable long term storage medium.

- The most effective long term storage condition for the channeltron is a clean (oil free) vacuum.
- A dry box which utilizes an inert gas, such as argon or nitrogen heated above the dew point, is also suitable.
- Desiccator type cabinets which utilize silica gel or other solid desiccants to remove moisture have been proven **unacceptable**.

8.2.5 Change Multiplier

Multiplier loses its gain ability with operation time. It should be changed when a significant degradation in amplification (i.e. intensity) is experienced.

8.2.5.1 Removing the detector flange

- Remove the preamplifier connections.
- Vent the system.
- Open the detector flange, let the detector unit slowly down and put it carefully on a table.

8.2.5.2 Changing the channeltrons

- Note: Use dry nitrogen only in order to remove dust or lint.
- Loose the screws, keeping the channeltrons in place.
- Disconnect all cables.
- Note the orientation and dimension of the used unit.
- Remove the multiplier.
- Put the new multiplier in place.
- Carefully fasten it with the screws.
- Check the channeltrons on being properly mounted.

8.2.5.3 Mounting the detector flange

• Mount the detector flange in the reverse order.

- Check that there is no short circuit for all the pins of the detector supply feedthrough to each other and to ground.
- Pump down.
- Detector must be baked out at a vacuum pressure lower than $1 \cdot 10^{-6}$ mbar. It is bakeable up to 150° C
- Check the detector according section 7.2



Figure 13

Detector unit (9 channel)

Chapter**9**Appendix

9.1 Unpacking

All analyzers and associated electronics are carefully packed before leaving the factory. Please examine packages for damage. If damage is suspected contact SPECS and always retain packing inspection.

After examination, the analyzer should stay in its protective packaging until it can be bolt directly onto the system. Take great care when unpacking to prevent damage. Do not rest the analyzer on ceramic feedtroughs or the viewport. Handle parts on the vacuum side of the flange seals using normal UHV protection, i.e. wear cloves and use clean non-magnetic tools.

9.2 Vacuum Installation

- 1. Open the transport box carefully.
- Check the shock and tilt sensors. If any sensor is discoloured, please inform SPECS immediately and wait for further instructions.
- Carefully lift the analyzer out of the box. Because of the analyzer weight of 80kg SPECS recommend to use a lifting gear. The hooks at the analyzer housing may be of help.
- 4. Keep the analyzer in a horizontal and stable position.
- 5. Remove the lens protection housing from the analyzer mounting flange. Do not touch any vacuum parts without gloves.
- 6. Fix the two delivered stay bolts at the analyzer mounting flange.
- 7. Insert a new DN100CF copper gasket into the vacuum chamber flange.
- 8. Center the analyzer mounting flange above the vacuum chamber flange.
- 9. Introduce the lens system into the vacuum chamber flange very slowly. Do not use any force.
- 10. During the introduction check all other components in the vacuum chamber because of possible mechanical damage.
- 11. Adjust the analyzer at the vacuum chamber flange by the stay bolts.

- 12. Check the working distance of the analyzer.
- 13. Fix the analyzer at the vacuum system flange with the delivered screws, washers and nuts.
- 14. Do not release the lifting gear up to the point where the analyzer is supported by an additional supporting post!
- 15. Check the mechanical stability of the supporting post and the system rack.
- 16. Release the lifting gear.
- 17. Evacuate the chamber to a pressure of below 10^{-5} hPa and bake out (see "Baking Out" on page 22).
- 18. Check the vacuum before and after bakeout.
- 19. After bake out check for no short circuits between all not grounded pins (figure 3 and figure 4 at page 6).
- 20. Connect the analyzer as described in the analyzer manual.
- 21. Before operating the analyzer wait for complete cool down (1 day recommended).

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