Absolute radiometric calibration of a VUV spectrometer in the wavelength range 46-300 nm

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Abstract

Vacuum ultraviolet (VUV) radiation corresponds to photon energies exceeding 6.2 eV and provides access to atomic and molecular resonant transitions in various plasmas. Determining population densities of electronic levels directly connected to the ground state requires an absolute intensity calibration of the VUV spectrometer. For this purpose, synchrotron radiation from electron storage rings is the typical primary standard source associated with transport effort and operating costs. Alternatively, a complex procedure using several secondary standards and discharges directly at the setup in use can be applied. In this work, an absolute intensity calibration avoiding synchrotron radiation to cover the wavelength range 46-300 nm accessible with two detectors—a photomultiplier tube and a channel electron multiplier—is presented. A combination of different standard sources—two deuterium arc lamps, branching-ratios of a nitrogen plasma and a high current hollow cathode—is applied for the relative calibration. The absolute calibration is based on simultaneous measurements of atomic lines in a low pressure helium discharge with the VUV spectrometer and an absolutely calibrated optical emission spectrometer. Special focus is laid on the uncertainties of the relative and absolute calibration depending on the wavelength range. The applicability of the intensity calibration is demonstrated at a hydrogen plasma in the wavelength range 80-300 nm. Using the emissivity of the Lyman series, population densities of the first six excited atomic states are calculated considering opacity effects. The excellent agreement with results from the Balmer series by optical emission spectroscopy manifests the high validity of the performed absolute intensity calibration.

Keywords: VUV spectroscopy, absolute intensity calibration, low pressure hydrogen plasma

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1. Introduction

Vacuum ultraviolet (VUV) radiation gives access to photon energies exceeding 6.2 eV and is essentially connected to resonant atomic and ionic lines as well as resonant molecular transitions of various substances [1]. The absolute intensity calibration of a VUV spectrometer requires either synchrotron radiation or a complex procedure using a compilation of secondary standards together with plasma radiation. The latter provide continuous emission only in a limited range and are mainly based on line radiation. Due to a high effort regarding the technical setup and big challenges of quantitative measurements, absolute spectroscopy in the VUV range has been applied only to a limited extent for many decades.

However, the recent years show a growing interest in VUV spectroscopy especially for plasma diagnostic purposes in diverse contexts. VUV spectrometers allow monitoring of relevant impurities present in magnetically confined plasmas [2, 3] and VUV photon fluxes were identified to be able to play a significant role in plasma surface treatment processes [4, 5, 6]. Quantification of VUV radiation is necessary for the control of arising VUV photon fluxes and hereinafter for an optimization of the specific application. This in turn requires absolutely calibrated VUV spectroscopic systems. Different approaches regarding a radiometric calibration in the VUV range can be found in [7, 8, 9].

Beyond, VUV emission spectroscopy offers access to the population densities of atomic and molecular states directly connected to the ground state. For instance, in hydrogen the resonsant atomic Lyman series ending on the ground level is located in the VUV range with the most intense L_α line at 121.6 nm. Since self-absorption might play a role, the absolute emissivity of the Lyman lines together with corresponding transitions in the optical range can give information about the opacity of the plasma.

Hence, to exploit the full potential of VUV emission spectroscopy an absolute intensity calibration of the applied VUV spectrometer is indispensable. For this purpose VUV radiation sources with a known intensity distribution are required. The typical primary source standard for the VUV region is given by electron storage rings where the acceleration of electrons produce a photon emission extending over the entire electromagnetic spectral range [10]. The intensity of the synchrotron radiation can be directly calculated from the operating parameters of the storage ring. Due to the related huge efforts, an inhouse calibration of the VUV spectrometer is desirable. Moreover, a routine calibration procedure available in the laboratory allows to monitor the temporal stability of the VUV spectrometer which is necessary since components might alter with time due to VUV photons.

A combination of a number of different sources and methods for an intensity calibration of a VUV monochromator is being presented in this work. The relative calibration is obtained using different secondary radiation standards. Depending on the individual emission, each of them enables a stepwise calibration in specific wavelength ranges. The standard approach applying a deuterium arc lamp and/or using branching ratios in nitrogen [11] gives a relative calibra-

tion down to approximately 116 nm. However, the latter implies a 'gap' in the range 116.4-127.3 nm which needs to be interpolated. The range below 116 nm is accessible with a high current hollow cathode [12, 13, 14]. The partly overlap among the sources allows a continuous calibration between 46 nm and 300 nm accessible with two detectors. The absolute radiometric calibration of the VUV spectrometer is based on scaling of the relative calibration curve to an absolute value and requires an absolutely intensity calibrated optical emission spectrometer. Simultaneous measurements with both spectrometers at a helium discharge are performed. The crucial prerequisite for this approach is that VUV and optical spectrometer detect the identical emission region of the plasma. Therefore, a detailed characterization of the plasma is essential. Gradients within the discharge or reflexions at components should be avoided. Alternatively, successive measurements at the same line of sight with changing the spectrometer is possible whereas the reproducibility of the discharge must be guaranteed.

As solid angle limiting optics are not present, an aperture in front of the VUV spectrometer is applied to define the viewing volume. The inverse sensitivity of the VUV spectrometer crucially depends on the emitting solid angle of the light source as well as on the collecting solid angle of the instrument itself and the calibration has to be performed at the specific setup in use. Furthermore, several steps of the calibration procedure are based on the emission from a plasma, for which the setup in use is applied. The absolute radiometric calibration of the spectrometer as well as the presented measurements in hydrogen are performed at the experimental setup *PlanICE*.

2. Experimental setup

2.1. Inductively coupled plasma (ICP) experiment

Discharges at the planar ICP experiment *PlanICE* are generated with a radiofrequency of 2 MHz and a power of up to 2 kW which is applied to a planar solenoid. The cylindrical stainless steel plasma vessel ($\varnothing 0.15$ m, height 0.1 m, base pressure below several 10^{-4} Pa) has a volume of l.77 liters and is separated from the solenoid by a quartz plate. Gas feeding takes place via mass flow controllers and operational pressures in the range of 0.3 Pa to 10 Pa are accessible. Several ports and quartz windows within the vessel wall allow diagnostic access to the plasma. A horizontal cut through the plasma vessel as well as the lines of sight of the applied diagnostics are depicted in figure 1. For the VUV and optical emission spectrometer the corresponding viewing volumes are indicated. During the calibration procedure of the VUV spectrometer different calibration standards are used (see section 3). The deuterium arc lamps and the high current hollow cathode (HCHC) are positioned in front of or mounted on the flange opposite of the VUV spectrometer. In the presented measurements in hydrogen absolute densities of the first six excited atomic states are obtained both with the VUV spectrometer and with optical emission spectroscopy from the atomic Lyman and Balmer series, respectively. Together with a moveable Langmuir probe the latter was additionally used to obtain the gas temperature as well

as the atomic ground state density. Both parameter are necessary to consider reabsorption effects on the measured line radiation. Moreover, monitoring of the temporal stability and reproducibility of the plasma takes place with an optical survey spectrometer. The rotational symmetry of the discharge has been investigated with radial profiles obtained with the moveable Langmuir probe and measurements with optical emission spectroscopy. The results at different rotation-symmetric lines of sight lie within the limits of the measurement errors (i.e. below $\pm 10\,\%$).

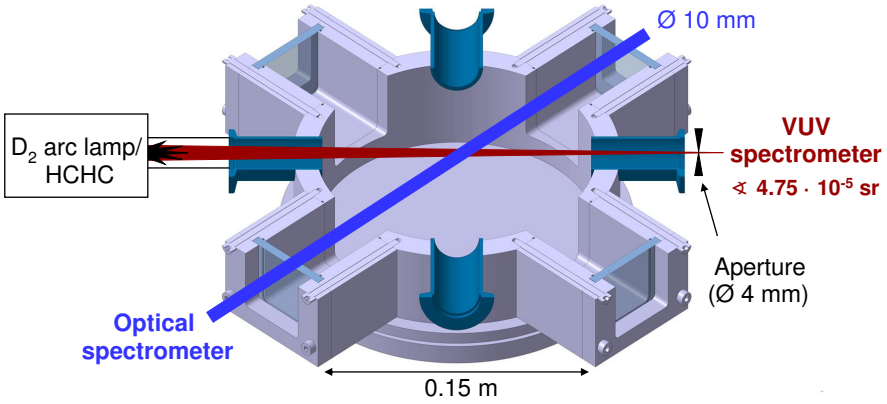

Figure 1: Setup of the ICP experiment *PlanICE*. The lines of sight of the applied diagnostics including the corresponding viewing volumes for optical and VUV spectroscopy are indicated. The calibration standards D_2 arc lamp and high current hollow cathode (HCHC) are positioned opposite of the VUV spectrometer.

2.2. VUV spectrometer

The VUV spectrometer is a scanning monochromator *Model 225* by *McPherson* with a focal length of 1 m. It is designed in normal incidence configuration enclosing a total angle of 15° and is based on single reflection. Incoming light hits a concave reflective diffraction grating with 1200 grooves per mm and a blaze wavelength at 120 nm. Due to the spectrometer's mechanics the accessible wavelength ranges up to 300 nm. The lower threshold is determined by the photon detection method and is given in the manual to be in the range of 30-50 nm [15]. The scanning speed is set to $10\,\frac{\mathrm{nm}}{\mathrm{min}}$ with a wavelength reproducibility better than $\pm 1\,\%$. The width of entrance and exit slit is set to $50\,\mu$m resulting in a Gaussian line profile with a FWHM (full width half maximum) ranging from 30 pm to rougly 37 pm over the accessible wavelength region. The applied standard grating consists of a borosilicate crown glass substrate coated with an Al/MgF_2 layer. The VUV monochromator can be supplied either with a solar-blind photomultiplier tube (PMT, *EMR 51F-08-18*, 18 stages, 2800 V

operating voltage) or a channel electron multiplier (CEM, *Model 425, McPherson*, 1600 V operating voltage). The PMT is equipped with a semitransparent cesium-telluride photocathode applied on a lithium fluoride window. The cut-on wavelength of LiF around 104.5 nm [10] determines the lower wavelength limit while the photocathode material characterizes the higher limit at roughly 400 nm [16]. The CEM is windowless and sensitive in the range 30-180 nm due to the MgF_2 coating of the inner surfaces. In both cases the diffracted radiation is detected and converted into a voltage signal measured by a digital multimeter (*DMM 5017, PREMA*) with an integration time of 100 ms. For measurements above 200 nm a quartz filter in a manual filter wheel in front of the entrance slit is inserted into the line of sight in order to avoid the impact of higher diffraction orders. Between two measurements a shutter is used. Apertures in front of the entrance slit limit the solid angle which is roughly estimated to be $4.75 \cdot 10^{-5}$ sr. Subsequently, the diameter of the viewing volume ranges from 4.3 mm at the entrance into the plasma vessel up to 5.5 mm at the opposite wall of the experiment. The VUV spectrometer is directly connected to the plasma chamber and differentially evacuated to a background pressure around several 10^{-5} Pa with a combination of oilfree roughing and turbolmolecular pumps. During plasma operation at a pressure typically in the range 0.3-10 Pa, inside the VUV spectrometer the pressure does not exceed 10^{-3} Pa. The wavelength calibration was performed with a mercury lamp using the emission line at 253.6 nm in zeroth order.

3. Calibration standards

The idea of intensity calibration (which is not restricted to the field of spectroscopy) is based on the principle of comparing the intensity distribution measured by the spectrometer with the known and reproducible spectral intensity $I_{\mathrm{stand}}(\lambda)$ of a radiation standard in units of photons/s/m^2/nm. With the prerequisite of a wavelength calibrated system the inverse spectral sensitivity (or calibration curve) $\rho^{-1}(\lambda)$ is then determined via

$$\rho^{-1}(\lambda) = \frac{I_{\mathrm{stand}}(\lambda)}{I_{\mathrm{meas}}(\lambda)}, \tag{1}$$

where $I_{\mathrm{meas}}(\lambda)$ is the measured signal, e.g. in units of counts/s or volts. The inverse spectral sensitivity of the VUV spectrometer is given in units of photons/s/m^2/nm/V. In the following ρ^{-1} at a given wavelength will be also referred to as calibration factor in the sense that multiplying measured values by the calibration factor results in spectroscopic quantities. A relative calibration considers the variation of the sensitivity depending on the wavelength while an absolute calibration gives absolute photon numbers.

Special attention has to be paid regarding the emitted or collected solid angle both of the radiation source and the spectroscopic system. Those are necessary to derive a correlation between the number of photons emitted by the source and collected by the spectrometer leading to the corresponding output

signal. A calculation in turn depends on the mutual alignment of standard source and the spectrometer. Several apertures and vacuum components along the corresponding lines of sight may lead to a complex system. Care has to be taken that the source radiation completely fills the acceptance volume of the spectrometer [17]. It is crucial to achieve an identical illumination of the grating during the calibration procedure and the measurements [18]. Using point like standard sources complicates the situation since even slight reciprocal shifts that might occur during the evacuation process or an imprecise adjustment could have a crucial and not assessable influence on the solid angle. Moreover, the conservation of the illuminated grating area might not be given with a point like source since the plasma during application provides a spatially extended emission. In order to avoid resulting uncertainties, wherever possible extended radiation sources should be preferred for an absolute intensity calibration. Point like sources are, however, applicable for a relative calibration.

In the following, the applied calibration standards are introduced with regard to their application during the calibration of the VUV spectrometer. Since they only allow for a relative calibration, section 4 will describe the techniques for the final absolute radiometric calibration.

3.1. Calibration standard sources

3.1.1. Deuterium arc lamp I: 190.0-400.0 nm

The deuterium lamp I (*Model 6316, L.O.T. Oriel GmbH*) is used for the relative calibration of the VUV spectrometer. It is an external light source and consists of a glass bulb that is equipped with a fused silica window. Inside, the filament together with a power supply (*Model 68840, L.O.T. Oriel GmbH*) generates a deuterium discharge at a pressure around several hundreds of Pascal. An apperture with a diameter of 1 mm between the discharge and the window defines the size of the plasma discharge that can be measured. The source was absolutely calibrated against a secondary standard [19], which has been in turn absolutely intensity calibrated at the *Physikalisch-Technische Bundesanstalt Braunschweig und Berlin (PTB)*, in units of $\mu W/cm^2/nm$. The wavelength range dependent relative uncertainties are $\pm 16\,\%$ (190-195 nm), $\pm 10\,\%$ (195-240 nm) and $\pm 9\,\%$ (240-350 nm), respectively. The setup included an optical focussing system using an aperture and quartz lenses leading to a dependency of the calibrated radiation on the distance of 500 mm as well as on the exact focal point in the discharge arc. The emission spectrum shows the D_2 dissociation continuum below 400 nm whereas absorption of radiation in air is observed below 190 nm. During the calibration of the VUV spectrometer the deuterium lamp was positioned in front of the opened flange across from the spectrometer (figure 1). Hence, the whole system was vented. Differently from the calibration setup of the lamp, no focussing optics were used and the calibration distance of 500 mm could not be met due to the size of the plasma vessel. A minimal distance of 750 mm between deuterium discharge and entrance slit of the VUV spectrometer could be achieved. The deuterium lamp I can be considered as a point-shaped radiation source and was therefore used only for the relative calibration of the VUV spectrometer equipped with the PMT.

For optical emission spectroscopy such a lamp is often applied as relative standard source extending the absolute radiometric calibration performed with an Ulbricht sphere below 400 nm. It is reasonable to use the identical calibration source for the corresponding wavelength ranges of both the optical and the VUV spectrometer.

3.1.2. Deuterium arc lamp II: 116.0-400.0 nm

The deuterium lamp II (*L9841, Hamamatsu*) is applied in order to perform a relative calibration of the VUV spectrometer equipped with the CEM and the PMT. It can be directly attached to the vacuum vessel which allows operation under vacuum conditions. The arc is housed in a glass bulb with a MgF_2 window and is driven by a current-stabilized power supply (*C9559, Hamamatsu*). An aperture with a diameter of 0.5 mm sets the emitting solid angle. The absolute radiometric calibration of the source was performed with the electron storage ring BESSY II at the *Physikalisch-Technische Bundesanstalt Braunschweig und Berlin* in units of $\mu W/mm^2/sr/nm$ and is thus not limited to a fixed calibration distance. The corresponding uncertainty is 7 % in the range 116.0-120.4 nm, 18 % in the interval 120.6-122.6 nm, 7 % in the range 122.8-170.0 nm and 3.5 % from 172.0 nm up to 400.0 nm [20]. The gaps result from the wavelength resolution given in [20]. In addition to the dissociation continuum, the spectrum of the source consists of molecular deuterium bands below 165 nm and includes the D_α line at 121.53 nm. The transmission of the MgF_2 window limits the emitted radiation to above the cut-on wavelength ($\approx$113 nm [10]). For the calibration of the VUV spectrometer the lamp was mounted on the flange opposite of the VUV spectrometer (figure 1). The vacuum system was evacuated eliminating absorption effects in air. The issues of a point like source also apply for the deuterium lamp II which was consequently only used to extend the relative calibration of the VUV spectrometer down to 116.5 nm.

3.1.3. High current hollow cathode (HCHC): 46.1-123.6 nm

A high current hollow cathode represents the possibility for an intensity calibration down to the extreme ultraviolet region (XUV) and is used for the relative calibration of the VUV spectrometer below the cut-on wavelength of MgF_2. The applied model *DKK 042* is based on a glow-discharge for which a detailed description of the setup can be found in [12, 13, 14]. The hollow-cathode is operated with the rare gases helium, neon, argon, krypton or xenon at pressures around 20-120 Pa. The gas flow in the range of 60 sccm is continuously readjusted with a needle valve to keep the discharge at the working point of 500 V and 1 A [21]. An aperture with a diameter of 1.2 mm limits the solid angle for the emitted radiation. The hollow-cathode serves as secondary standard with the unique feature that an individual exemplar being identical in construction to an original prototype does not need an individual intensity calibration. The intensities of this prototype have been absolutely calibrated with the PTB beam line at the electron storage ring BESSY in units of $\mu W/sr$. Calibrated intensities of the source arising from atomic and ionic transitions of the working gas range from 123.6 nm (Kr I) down to 46.1 nm (Ne II) and can be found in [13]. They

are valid within ±5 % [22] for the replica used in this work. Alike the deuterium lamp II, the HCHC is directly flanged to the plasma vessel on the opposite side of the VUV spectrometer (figure 1) and applied for a relative intensity calibration of the VUV spectrometer equipped with the CEM and PMT.

3.1.4. Branching ratios of nitrogen: 116.4-212.7 nm

Atomic and molecular branching-ratios in nitrogen are used for relatively calibrating VUV spectrometers [11]. Considering two transitions arising from the same upper level, the ratio of their intensities is independent of the actual population density of the upper state and is equal to the ratio of the transition probabilities [23] as long as opacity effects can be neglected. The branching-ratio technique applies both to atomic lines and the integrated radiation emitted by vibrational bands in molecules. For the calibration of the VUV spectrometer equipped with the PMT a nitrogen discharge (3 Pa, 600 W) is ignited in the plasma vessel and the molecular Lyman-Birge-Hopfield system (100-260 nm [24]) as well as two atomic transitions at 116.4 nm and 131.1 nm (with the same upper state $2s^2 2p^2 3d\ ^2\mathrm{D}$ ending on the metastables states $2s^2 2p^3\ ^2\mathrm{D^o}$ and $^2\mathrm{P^o}$, respectively) were observed. Within the Lyman-Birge-Hopfield system vibrational transitions starting from the same upper state with vibrational quantum numbers 0-6 to lower states were recorded. Regarding the atomic emission lines, transition probabilities are found in the *NIST* database [25]. In the case of vibrational transitions in nitrogen molecules relative emission intensities are applied which were measured in [26]. The overlap of the molecular transitions leads to a nearly continuous relative calibration between 127.3 nm up to 212.7 nm which can be extended down to 116.4 nm with the atomic lines. However due to the lack of data, the calibration curve is not accessible between 116.4 nm and 127.3 nm but is interpolated by default [11]. One should keep in mind that reabsorption effects of the atomic transitions might occur and might contribute to the uncertainty of the calibration procedure. On the other hand, the molecular band are are uneffected by opacity.

4. Calibration technique

In the following section the absolute intensity calibration of the VUV spectrometer using the described standards is discussed for the two detectors. It is pointed out that the presented calibration includes the spectral sensitivity of the applied detector (PMT or CEM), in the case of existing windows their transmission, the wavelength dependent grating efficiency as well as apertures within the line of sight. Changing the detector does not influence the other components but has an effect on the over-all sensitivity of the VUV spectrometer. For convenience, the calibration of the VUV spectrometer equipped either with the PMT or the CEM including windows, gratings and apertures will be often stated shortly as 'calibration of the PMT/CEM' in the following. It is stressed that the calibration procedures using the PMT and the CEM are interlinked and partially depend on each other.

4.1. Photomultiplier tube (PMT)

A scan over the whole accessible wavelength range is divided into two separate measurements, one below 200 nm without any filter and one in the range 200-300 nm with the quartz filter installed.

(a) First, a relative calibration curve is determined using the deuterium lamp I in the wavelength range 200-300 nm with the quartz filter and from 190 nm to 300 nm without the filter, respectively. The latter is necessary to be able to scale the calibration curve for measurements below 200 nm without the quartz filter to an absolute value in step (c). In general, the directly mountable deuterium arc lamp II could also be applied.

(b) Afterwards, the relative calibration curve without the quartz filter is extended with the branching-ratio technique down to 116.4 nm using a nitrogen discharge (3 Pa, 500 W).

(c) Subsequently, absolute scaling factors are obtained by simultaneous measurements with the VUV spectrometer and an absolutely calibrated optical spectrometer (*Acton SP2750*, grating with 1800 lines/mm, CCD camera) at a helium discharge (5.2 Pa, 400 W) at rotation-symmetric lines of sight. The symmetry of the plasma has been determined to be within $\pm 10\%$ for different rotation-symmetric lines of sight. The light-collecting system of the optical spectrometer consists of an achromatic mirror that focuses the radiation wavelength-independently into a UV enhanced fibre. The optical components like mirror, fiber aperture and the solid angle limiting aperture stop are adjusted in a way that the viewing cone is nearly cylindrical with a diameter of 1 cm. An absolutely calibrated Ulbricht sphere (*K-300WH/20 / SLP 120-80, L.O.T.-Oriel*) served for an intensity calibration in the wavelength range 380-800 nm which was extended to the lower limit of 200 nm with the deuterium arc lamp I described above in section 3.1.1. For the absolute intensity calibration of the VUV spectrometer the five helium lines at 269.61 nm ($1s9p\ ^3\mathrm{P^o}$), 272.32 nm ($1s8p\ ^3\mathrm{P^o}$), 276.38 nm ($1s7p\ ^3\mathrm{P^o}$), 282.91 nm ($1s6p\ ^3\mathrm{P^o}$) and 294.51 nm ($1s5p\ ^3\mathrm{P^o}$) are recorded and compared to the corresponding emissivity that is measured at the same time with the absolutely calibrated optical system. The electronic configuration and the corresponding spectroscopic notation of the upper states are given in brackets whereas the symbol o indicates an odd parity. The investigated transitions end on the common level $1s2s\ ^3\mathrm{S}$. As a result, an absolute radiometric calibration factor is obtained at these wavelengths and the already obtained relative calibration curve is scaled to match with these values.

(d) The result from the previous step is relatively continued with the directly attachable deuterium arc lamp II down to 116.5 nm. First, a relative curve is obtained from comparing the standard's emission with the measured spectrum. Afterwards, it is scaled to the absolute radiometric calibration curve whereas the scaling takes place at 183 nm. Instead of simply dividing

the emission of the lamp by the measured spectrum, wavelength intervals with a fixed length of 1 nm are integrated both in the absolute spectral intensity of the standard and in the measured spectrum. This is of great importance in the range below 165 nm where the deuterium spectrum mostly consists of lines [27].

(e) The absolute radiometric calibration in the wavelength region below 116.5 nm is performed with measurements using the PMT as well as the absolutely calibrated CEM (see section 4.2) at same plasma discharges. Covering the range 107.4-120.1 nm, xenon, nitrogen and oxygen plasmas are ignited in the experiment's vessel and atomic and ionic lines were recorded with both detectors. The discharges are operated at various pressures (0.3-10 Pa) and generator powers (300-600 W) in order to observe a multitude of different lines and to reduce the influence of the operating parameters on the calibration. The plasma emission cannot be measured simultaneously since changing the detectors is not possible without venting the spectrometer. Therefore, attention has to be paid regarding the stability and reproducibility of the dicharges which was done by plasma monitoring with the optical emission survey spectroscopy. A reproducibility of the plasma emission better than $\pm 10\,\%$ was oberseved. Following the same approach and operating the high current hollow cathode with argon or xenon as radiation source absolute radiometric calibration factors at 104.8 nm (Ar I), 106.7 nm (Ar I), 116.4 nm (Xe I) and 123.6 nm (Xe I) are obtained.

The presented intensity calibration procedure of the VUV spectrometer equipped with the PMT includes several overlap regions between the different calibration standards which is shown in figure 2 (a). The colours represent the applied standards and techniques.

Full symbols indicate that the corresponding calibration standard was considered for the final calibration curve, open values are shown to give an impression of the overlap regions. Table 1 summarizes the calibration standards and gives two wavelength ranges, namely the ones which are accessible due to the calibration of the sources including overlap regions and the intervals that were finally considered for the calibration curve of the VUV spectrometer.

Finally, a fit function is determined from the calibration factors below 200 nm with the objective of a smoothed inverse sensitivity of the VUV spectrometer. Since the inverse sensitivity ranges over several orders of magnitude and shows highly varying slopes, a combination of several fit functions is necessary to cover the whole wavelength range. For this procedure the curve fitting tool *Datafit* (Oakdale Engineering) was used and the results are depicted as red line in figure 2 (a). In the range 200-300 nm the calibration curve is already available originating from the deuterium continuum, hence no fit function is applied.

4.2. Channel electron multiplier (CEM)

The absolute radiometric calibration of the VUV spectrometer equipped with the CEM is achieved via the following steps:

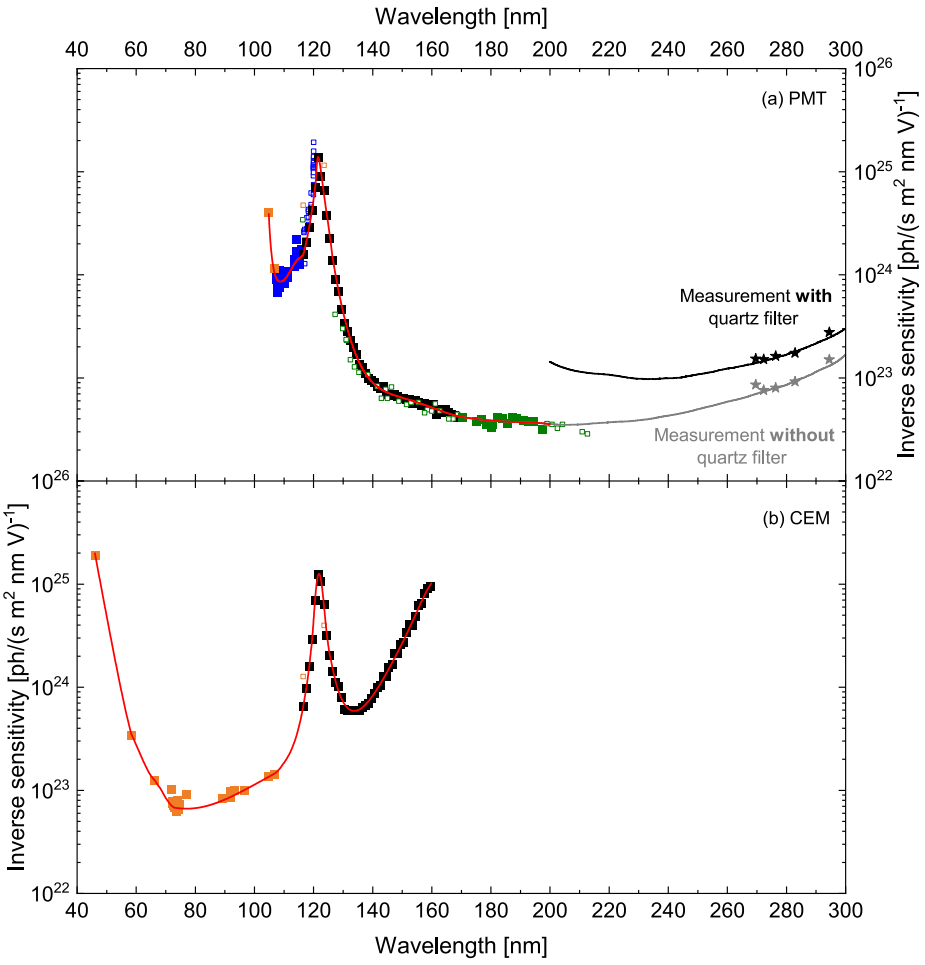

Figure 2: Inverse spectral sensitivity of the VUV spectrometer equipped with **(a) PMT** and **(b) CEM**. During the radiometric calibration procedure a high current hollow cathode (orange squares), Xe, N_2 and O_2 discharges (blue squares), several branching ratios in nitrogen (green squares) and two deuterium arc lamps (with fused silica window, black/grey line, or with MgF_2 window, black squares) as well as a helium discharge (black/grey stars corresponding to the calibration with and without quartz filter) were applied. Full symbols state that the corresponding source was considered for the final radiometric calibration curve, open symbols indicate overlap regions. The red lines represent a combination of fit functions obtained with *DataFit*.

Standard source	Accessible wavelength range	Considered wavelength range	Figure 2 (a)
HCHC	104.8 - 123.6 nm	104.8 - 106.7 nm	orange squares
Xe, N_2 and O_2 discharges	107.4 - 120.1 nm	107.4 - 116.5 nm	blue squares
Deuterium arc lamp II	116.0 - 400.0 nm	116.5 - 169.5 nm	black squares
Branching ratios in nitrogen	116.4 - 210.0 nm	169.5 - 198.2 nm	green squares
Deuterium arc lamp I	190.0 - 400.0 nm	198.2 - 300.0 nm	grey line
		200.0 - 300.0 nm (with quartz filter)	black line
He discharge	269.6 - 294.5 nm		black/gray stars

Table 1: Applied standard sources for the radiometric calibration of the VUV spectrometer equipped with the PMT. The two deuterium arc lamps and the nitrogen branching ratios are used for a relative calibration which is scaled to an absolute value via simultaneous detection of the helium lines with VUV and optical spectroscopy. In contrast, the measurements with the PMT and CEM using the HCHC and the discharges in Xe, N_2 and O_2 give directly the absolute radiometric calibration with the prerequiste of an absolute intensity calibration of the CEM below 116.5 nm.

(a) First, the directly attachable deuterium arc lamp II is applied to perform a relative calibration in the wavelength range 116.5 nm to 160 nm. The upper limit is given due to the decreasing sensitivity of the detector in combination with the low intensity of the deuterium lamp in this spectral range. Above 160 nm the calculated inverse sensitivity would thus start to oscillate due to a poor signal to noise ratio.

(b) Subsequently, the relative calibration curve is scaled to an absolute value for which the absolute intensity calibration of the PMT above 140 nm is required. The PMT is used to obtain the fraction of the emitted radiation of the deuterium arc lamp II that is entering into the VUV spectrometer by comparing the measured spectrum to the calibrated emission of the radiation standard. The fraction is determined separately for each 1 nm interval in the range 140-198 nm where the PMT's absolute radiometric calibration is independent from the CEM (see section 4.1) and is finally averaged. It includes the solid angle between the source and the VUV spectrometer and is therefore dependent on the mutual adjustment but independent of the detector.

(c) In the last step, successively operating the high current hollow cathode with helium, neon, argon, krypton and xenon relative calibration factors

are obtained between 46.1 nm and 123.6 nm. Using the overlap with the results from the first step, a scaling factor is obtained to extend the absolute radiometric calibration down to 46.1 nm.

Figure 2 (b) depicts the inverse sensitivity of the VUV spectrometer equipped with the CEM. Full symbols are included in the final calibration curve, open symbols show the overlap between the two standard sources. Table 2 summarizes the described calibration procedure with the corresponding wavelength intervals of the applied sources.

Standard source	Accessible wavelength range	Considered wavelength range	Figure 2 (b)
HCHC	46.1 - 123.6 nm	46.1 - 116.5 nm	orange squares
Deuterium arc lamp II	116.0 - 400.0 nm	116.5 - 160.0 nm	black squares

Table 2: Applied standard sources for the relative calibration of the VUV spectrometer equipped with the CEM. The absolute radiometric calibration additionally requires the absolute radiometric calibration of the PMT in the range above 140 nm.

Analogously to the procedure with the PMT, a combination of fit functions is determined with *DataFit* which is plotted as a red line in figure 2 (b).

4.3. Discussion of inverse sensitivity

With the combination of photomultiplier tube and channel electron multiplier the VUV spectrometer covers an over-all wavelength range from 46 nm to 300 nm. For both detectors the calibration curves show a maximum around 120 nm surrounded by two minima and increase towards the edges of the accessible wavelength region. For the PMT the useable range is 104.8-300 nm while the CEM is applicable between 46.1 nm and 160 nm.

Regarding the PMT, the steep increase below 108 nm of the calibration curve in figure 2 (a) is due to the LiF window in front. Its transmission strongly decreases towards the cut-on wavelength which is roughly at 104.5 nm depending on the ambient temperature, processing and handling [23, 10]. From 150 nm to 230 nm a broad global minimum represents the highest sensitivity range of the VUV spectrometer equipped with the PMT. Inserting the quartz filter for measurements above 200 nm shifts the calibration curve upwards with the minimum moved to 235 nm. The ratio of the corresponding black and grey curves is wavelength dependent due to the transmission of the quartz filter and increases towards to its cut-on wavelength around ≈160 nm. The trend of the curves approaching the upper wavelength limit is independent of the filter. The applied model uses a semitransparent cesiumtelluride cathode whose response drops significantly above ≈260-270 nm [23, 16] leading to a rise of the inverse sensitivity.

With the CEM (see figure 2 (b)) the VUV spectrometer is most sensitive around 80 nm. Below 60 nm and in the wavelength range above 140 nm the inverse sensitivity rapidly increases.

In general, the blaze wavelength of the grating determines the sensitivity maximum of the spectrometer. Therefore, regarding the fact that the applied grating is blazed at 120 nm, the occurence of a peak in this wavelength range in both calibration curves is particularly striking. This increase of more than one order of magnitude can be traced back to the applied standard grating. A comparative measurement with a replacement grating (platinum coating, blaze wavelength at 45 nm) using the deuterium arc lamp II and the photomultiplier tube as detector was performed. The resulting inverse sensitivities shown in figure 3 (normalized in the region with highest sensitivity, arbitrarily at 169.5 nm) confirm the peak as a characteristic of the standard grating. The spectrometer together with the standard grating has been operated for about fourty years with oil-lubricated vacuum pumps before they were replaced by oil-free systems in recent years. Regarding oil-lubricated pumps, a back-diffusion of oil vapour into the recipient cannot be totally avoided. Irridiation of adsorbed hydrocarbons (e.g. on the grating area) with intense VUV radiation can lead to a formation of carbon films reducing drastically the reflective properties [28]. This degradation might also occur wavelength dependently. Suffering of the grating from the impact of VUV radiation in combination with hydrocarbon contamination is likely. The curves in figure 2 therefore might result from a superposition of the effect due to the blazing at 120 nm and the sensitivity degradation due carbon films. Moreover, an influence of air humidity during storage of the grating or venting of the spectrometer can not be excluded. Hence, for longterm reproducibility of measurements with the VUV spectrometer in this range, it is indispensable to monitor the degradation effects. In addition, it becomes clear that the standard calibration procedure accordingly to [11] is inadequate in the present case. Using only the interpolation of the nitrogen branching ratios in the range 116.4-127.3 nm would have let to an underestimation of the inverse sensitivity by one order of magnitude for the L_α line.

4.4. Uncertainty sources

Since the calibration procedure includes a number of standard sources and techniques, the error—which is not straightforwardly assessable—will be investigated for individual wavelength ranges depending on the applied detector. The uncertainty of the relative calibration is based on the error inherent to the relative calibration of the specific standard. In general, the absolute radiometric calibration of the VUV spectrometer was obtained following two approaches: On the one hand, relative calibration curves were scaled to an absolute value. In this case, the uncertainty is determined by the uncertainties of the standard source used for the relative calibration and of the absolute calibration factor as well as the uncertainty arising from the scaling process. On the other hand regarding the PMT in the wavelength interval 107.4-116.5 nm, measurements with the PMT and the absolutely calibrated CEM were performed at different plasma dicharges and using the high current hollow cathode. Here, only the

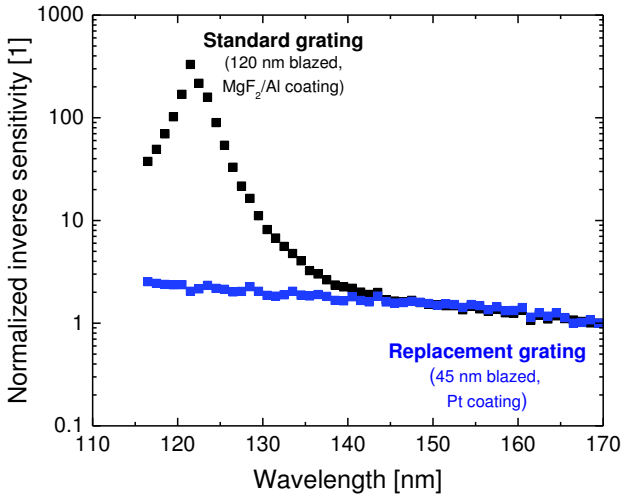

Figure 3: Comparison of the inverse sensitivity of the VUV spectrometer equipped with the PMT between different gratings determined with the deuterium arc lamp II. The black data points refer to the standard grating (Al/MgF_2 coating), the blue curve is obtained with the replacement grating (Pt coating, blaze wavelength 45 nm).

uncertainties of the radiation source and the calibration of the CEM is considered. The resulting total relative uncertainties are displayed in table 3 and table 4 applying a Gaussian error propagation. No additional error due to the corresponding fit functions is considered. Uncertainty sources for the performed calibration will be discussed separately in the following.

Independently from the standard sources the uncertainty of the calibration procedure is further determined by the measurement of the voltage output produced by the detectors which is done by a digital multimeter within error limits below $\pm 1\,\%$ [29]. This values is taken as uncertainty of the VUV spectrometer in the following consideration. The absolute radiometric calibration of the VUV spectrometer equipped with the PMT relies on the measurement of emission lines in helium at the same time with the optical spectrometer. Hence, it strongly depends on the error of the measured emissivity by optical emission spectroscopy. The typical error of the applied spectrometer is in the range of $\pm 10\,\%$ due to its calibration and reproducibility. Further aspects including the rotational symmetry (see figure 1) of the helium discharge and slightly different viewing volumes of optical and VUV spectrometer are assumed to be negligible. The shift of the relative calibration curve to the absolute helium emissivities in step (c) is performed with an estimated error of $\pm 14\,\%$ with quartz filter and $\pm 12\,\%$ without quartz filter. These two contributions have to be considered for the uncertainty of the absolute radiometric calibration in addition to the uncertainties of the relative calibration obtained from the deuterium arc lamps

and the branching-ratios. Regarding both deuterium arc lamps, relative uncertainties of their absolute intensity calibration are given in the corresponding manuals and are taken as an upper limit for the uncertainty of their relative calibration (see section 3.1.1 and 3.1.2). Due to a distance dependent calibration of the deuterium lamp I and the fact that the calibrated distance could not be met in the presented calibration procedure of the VUV spectrometer, an additional error of ±10 % is considered in the uncertainty of the relative calibration. The reproducibility due to aging of the lamps over the operation period has not been further investigated and is neglected in the analysis. The branching-ratios in nitrogen serve as an overlap between the two deuterium lamps and an error indication is rather difficult. The uncertainty of the determined calibration factors depends on the accuracy of the relative intensities of the investigated vibrational bands or atomic lines taken from literature and strongly on the measurement. Partly, molecular nitrogen bands are overlapping each other and atomic lines or suffer from low intensity leading to an overall estimated uncertainty of ±20 % for the relative calibration. Finally, the results are scaled leading to an uncertainty of the absolute radiometric calibration of 25 % including the uncertainty of the relative calibration (20 %) and the VUV spectrometer itself (1 %) as well as the two contributions stated above resulting from the reproducibility of the optical spectrometer (10 %) and the shift of the relative curves to the helium emissivities (12 %) in step (c). The relative extension below 116.5 nm is performed via measurements both with the PMT and the absolutely calibrated CEM using different plasmas in the experiment's vessel for the wavelength range 107.4-116.5 nm or the emission of the high current hollow cathode in the interval 104.8-106.7 nm (step (e) in section 4.1). In both cases the uncertainty of the absolute radiometric calibration of the CEM in the corresponding wavelength interval (60 %, see table 4) as well as the uncertainties of the discharges have to be taken into account. In the range 107.4-116.5 nm the reproducibility of the plasmas is of crucial importance since changing the detectors requires venting the vacuum system. For this reason, the corresponding measurements have been performed at several operating parameters for each gas. The maximum standard deviation of the obtained relative calibration factors at a fixed wavelength is ±18 % and is taken as the uncertainty of the relative calibration. As a results one obtains ±63 % for the absolute radiometric calibration of the PMT. Regarding the HCHC for the range 104.8-106.7 nm and its relative uncertainty, several error sources may play a role like an uncertainty of the calibrated line intensities (±13 %), the long term stability of the source (±10 % [13]), the accuracy of the voltage drop over the discharge (±5 %) as well as the integration of the measured lines (±10 %) [2]. As already mentioned above the applied copy of the hollow cathode was not directly calibrated against an electron storage ring. Therefore, the given validity of ±5 % of the tabulated line intensities [22] for a replica of the originally calibrated source is considered. The overall uncertainty of the HCHC and hence of the relative calibration of the PMT in the range 104.8-106.7 nm accounts to ±20 % and ±63 % for the absolute radiometric calibration, respectively.

The absolute radiometric calibration of the CEM in the wavelength range

Wavelength range (PMT)	Quartz filter	Relative uncertainty of relative calibration	Relative uncertainty of absolute calibration
104.8 - 107.4 nm	w/o	20 %	63 %
107.4 - 116.5 nm	w/o	18 %	63 %
116.5 - 120.4 nm	w/o	7 %	17 %
120.4 - 122.8 nm	w/o	18 %	24 %
122.8 - 169.5 nm	w/o	7 %	17 %
169.5 - 198.4 nm	w/o	20 %	25 %
198.4 - 200.0 nm	w/o	14 %	21 %
200.0 - 240.0 nm	w/	14 %	22 %
240.0 - 300.0 nm	w/	13 %	22 %

Table 3: Relative uncertainty of the relative and absolute radiometric calibration of VUV spectrometer equipped with the PMT (without and with quartz filter in line of sight).

above 116.5 nm is based on the deuterium arc lamp II and the absolute radiometric calibration of the PMT in the wavelength range 140-198 nm. As discussed, the uncertainty of the relative calibration of the CEM is given by the wavelength dependent uncertainty of the deuterium lamp (see section 3.1). The PMT was used to determine the solid angle dependent radiation that is entering the VUV spectrometer. As result, a scaling factor is obtained to transform the relative calibration curve obtained with the deuterium lamp II onto an absolute scale. The standard deviation of the scaling factor amounts to ±5 % and has to be included in the uncertainty of the absolute radiometric calibration of the CEM. Moreover, the uncertainties of the deuterium lamp II as well as of the calibration of the PMT in the range 140-198 nm are considered. Since several calibration standards were applied for the absolute radiometric calibration of the PMT to cover the interval 140-198 nm, different uncertainty values were determined. As an upper limit the maximal value of ±25 % from the branching-ratios is used. Uncertainties of ±26 % (116.5-120.4 nm; 122.8-160.0 nm) and ±31 % (120.4-122.8 nm) follow for the absolute radiometric calibration of the CEM. Below 116.5 nm the high current cathode was applied. The uncertainty of the relative calibration was determined to be ±20 %. Using an overlap region with the deuterium lamp II the relative calibration is absolutely scaled. However, the overlap includes only two krypton lines at the wavelength positions 116.5 nm and 123.6 nm and is therefore poor with an estimated error of ±50 %. This value is considered in addition to the uncertainties of the relative calibration (±20 %) as well as of the absolute radiometric calibration of the CEM with the deuterium lamp (±27 %) in the overlap range to obtain an uncertainty of ±60% for the absolute radiometric calibration below 116.5 nm.

Figure 4 depicts the absolute radiometric calibration curves of the VUV spectrometer equipped with the PMT or the CEM (black lines) together with the corresponding uncertainty ranges taken from table 3 and 4 (blue tubes). For the PMT above 200 nm only the curve for the measurements with the quartz

Wavelength range (CEM)	Relative uncertainty of relative calibration	absolute calibration
$< 116.5\,\mathrm{nm}$	20 %	60 %
116.5 - 120.4 nm	7 %	27 %
120.4 - 122.8 nm	18 %	31 %
122.8 - 160.0 nm	7 %	27 %

Table 4: Relative uncertainty of relative and absolute radiometric calibration of VUV spectrometer equipped with CEM (without quartz filter in line of sight).

filter is shown. In the wavelength region above 116.5 nm the uncertainty of the CEM is higher by 7-10 % compared to the PMT. In order to minimize the uncertainty of the absolute spectra taken with the VUV spectrometer, specific measurement intervals are defined for the different detectors: The wavelength range scanned with the PMT is set to 116.5-300 nm while the CEM will be installed for measurements below 116.5 nm.

5. Application to a hydrogen plasma

The applicability of the obtained absolute radiometric calibration of the VUV spectrometer is demonstrated for a hydrogen plasma. Figure 5 illustrates an absolutely calibrated intensity spectrum of a hydrogen discharge at 1 Pa and a generator power of 1100 W taken at *PlanICE* with the VUV spectrometer. The CEM was used in the wavelength region below 116.5 nm, the PMT in the range 116.5-300 nm. It is assumed that changing the detectors does not affect the reproducibility of the measurements. The stability of the discharges themselves during the measurements with PMT and CEM, respectively, was monitored with optical emission spectroscopy. Additionally, the gas temperature T_{gas} and the global atomic ground state density $n_{\mathrm{H(n''=1)}}$ are derived from optical emission spectroscopy together with a collisional radiative model [30, 31, 32]. The electron temperature of $10.2\,\mathrm{eV}\ \pm 1\,\mathrm{eV}$ and electron density of $(3.0 \pm 1.2) \cdot 10^{16}\mathrm{m}^{-3}$ are measured with a Langmuir probe.

The most prominent emission features in the VUV are the resonant atomic Lyman series and molecular transitions represented by the Werner band ($C\ ^1\Pi_u \rightarrow X\ ^1\Sigma_g^+$, ≈80-130 nm), the Lyman band ($B\ ^1\Sigma_u^+ \rightarrow X\ ^1\Sigma_g^+$, ≈130-190 nm) as well as the Continuum ($a\ ^3\Sigma_g^+ \rightarrow b\ ^3\Sigma_u^+$, ≈190-280 nm). The molecular bands partly overlap, hence, the given wavelength intervals are assigned to the particular transition corresponding to their main emission ranges. The emission lines of the Lyman series correspond to atomic transitions from excited states n' into the ground state $\mathrm{n}'' = 1$. In the present setup the Lyman series can be detected from the L_α line ($n' = 2$ at 121.6 nm) down to the L_ζ line ($n' = 7$ at 93.1 nm) with the VUV spectrometer.

In the following the population densities of the first six excited states of atomic hydrogen are determined both from the Lyman series in the VUV range

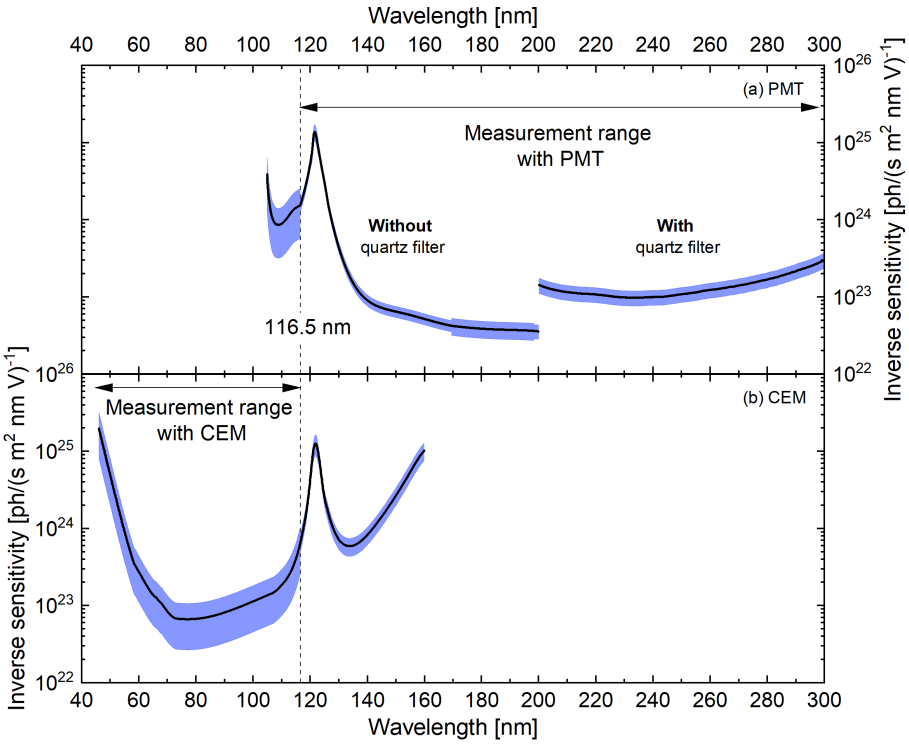

Figure 4: Absolute radiometric calibration curves of the VUV spectrometer (black lines) equipped with the (a) PMT or (b) CEM with wavelength dependent uncertainty ranges (blue areas) from table 3 and 4.

as well as from the Balmer series in the optical region (see figure 5(c)). However, the first excited state is only accessible via the L_α line.

Due to a high density of atomic hydrogen in the ground state the Lyman series is affected by reabsorption. Emitted radiation is trapped along the line of sight leading to a reduced measured intensity. This opacity effect might play an important role [33] and can be taken into account with the line escape factor method [34]. Depending on the plasma geometry and emission profiles as well as the atomic ground state density and T_{gas} so-called line escape factors Θ_{line} can be calculated for each Lyman line. The emissivity measured outside the plasma can thus be expressed with

$$I = n_{n'} A_{n'n''} \Theta_{\mathrm{line}}, \tag{2}$$

where $n_{n'}$ denotes the population density of the upper state n' of the transition and $A_{n'n''}$ is the corresponding Einstein coefficient. The measured emissivity of the L_α line can be corrected with the line escape factor $\Theta_{\mathrm{line}} = 9.7 \cdot 10^{-3}$ [35].

Analogously to the Lyman series in the VUV range, radiative transitions into the first excited state $n'' = 2$ of atomic hydrogen are summarized to the Balmer

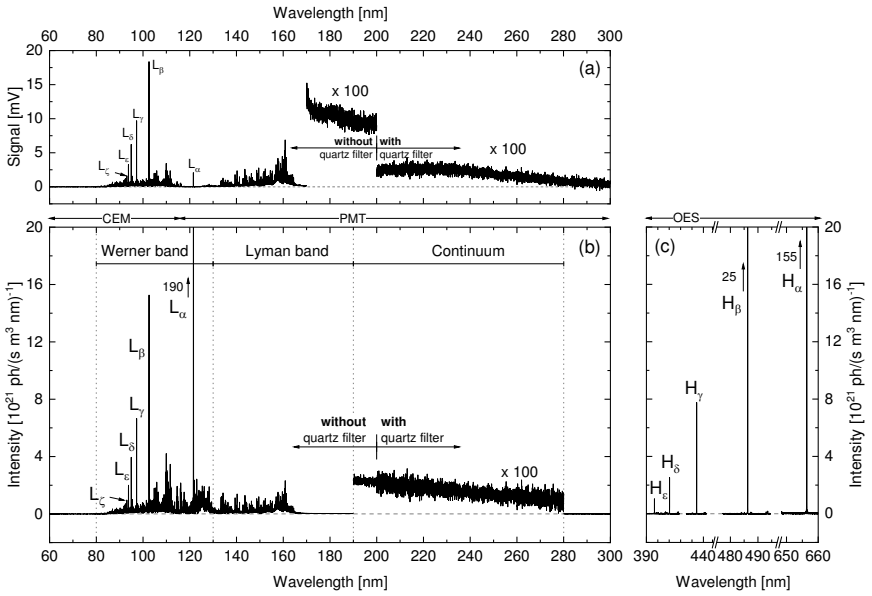

Figure 5: Emission spectra of a hydrogen discharge at 1 Pa and 1100 W generator power obtained with the VUV spectrometer (a, b) and with the optical high resolution spectrometer (c). Both spectrometer are absolutely intensity calibrated whereas (a) shows the measured signal without taking into account the inverse sensitivity of the VUV spectrometer. In the wavelength range 170-300 nm the signal is multiplied by a factor of 100. The lower part (b) depicts the absolute emission spectrum including the radiometric calibration. The Lyman and Balmer series of atomic hydrogen as well as several molecular bands are indicated. The continuum is multiplied by a factor of 100.

series which is located in the optical range. For the Balmer series reabsorption can be neglected and the corresponding Θ_{line} is set to 1. Atomic levels with $n' \geq 3$ represent a common upper level of a Lyman line and a Balmer line. For the Lyman series the line escape factors from [35] were taken for an atomic density of $n_{\mathrm{H(n''=1)}} = 2.8 \cdot 10^{19}\,\mathrm{m}^{-3}$ (with a relative error of 5 %) and a gas temperature of 600 K $\pm$ 25 K. Both values are obtained with optical emission spectroscopy together with collisional radiative modelling. The result is shown in figure 6. The emission of L_α leads to a density of $1.5 \cdot 10^{15}\,\mathrm{m}^{-3}$ in the first excited state $n' = 2$. With increasing n' the population densities evaluated via the Lyman series first show a drop to $1.3 \cdot 10^{14}\,\mathrm{m}^{-3}$ in the second excited state $n' = 3$ and then decreases slowly down to $5.4 \cdot 10^{13}\,\mathrm{m}^{-3}$ in level $n' = 7$.

It can be seen that an excellent agreement is obtained between the results from the Lyman series with the values calculated from the Balmer emission. The uncertainty range which might be estimated in a very conservative way clearly exceeds the deviation between the Lyman and the Balmer series. The comparison demonstrates impressively the validity of the absolute intensity calibration of the VUV spectrometer in the VUV range.

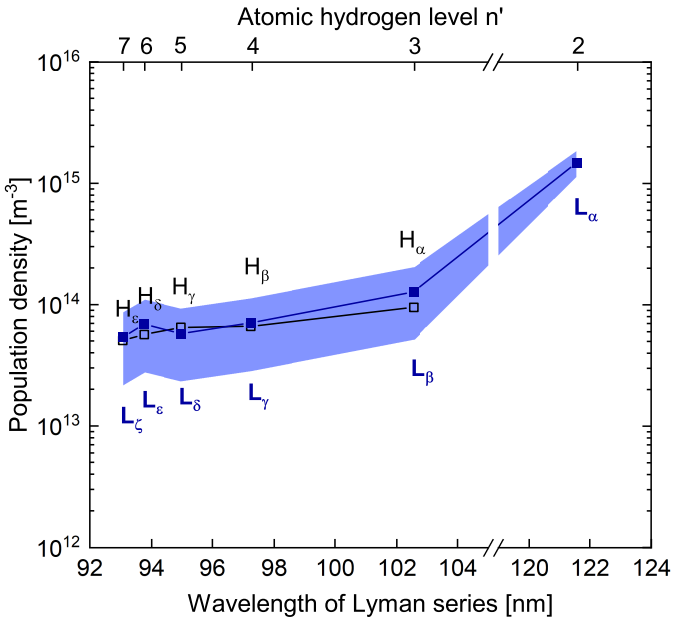

Figure 6: Population densities of the atomic states $n' = 2 - 7$ derived from emissivities of the six Lyman lines $L_\alpha, L_\beta, L_\gamma, L_\delta, L_\epsilon$ and L_ζ (blue squares) and of the five Balmer lines $H_\alpha, H_\beta, H_\gamma, H_\delta$ and H_ϵ (black squares) with corresponding common upper level n'. The x axis corresponds to the wavelength of the Lyman series. For the results obtained from the Lyman series the uncertainty range is given as blue shaded area resulting from the wavelength dependent uncertainty of the absolute intensity calibration of the VUV spectrometer (see tables 3 and 4). The relative uncertainty of $\pm 10\,\%$ of the optical spectrometer lies within the symbols of the Balmer series.

6. Conclusion

VUV photons in plasmas arise from atomic or molecular states directly connected to the ground state. Typically, synchrotron radiation provided by electron storage rings is the main primary standard source for absolute intensity calibration purposes in the VUV range. However, it is inseparably linked with high efforts. This work presents an alternative and more easily accessible—but nevertheless complex—absolute radiometric calibration procedure. The relative calibration was performed by applying a combination of different available calibration source standards. The prerequisite of the absolute radiometric calibration is an absolutely intensity calibrated optical emission spectrometer. It was used for simultaneous measurements of atomic lines in a helium discharge at the setup in use. The procedure also allows to monitor aging effects due to VUV photons. A VUV spectrometer was relatively and absolutely intensity calibrated between 46 nm and 300 nm using two detectors—a CEM and a PMT. The former is used for measurements below 116.5 nm while the latter is applied in the range 116.5-300 nm. In the future, the calibrated VUV spectrometer

will be applied for the investigation of absolute VUV photon fluxes in various plasmas (e.g. noble gases).

The uncertainty of the absolute radiometric calibration using the PMT in the range 116.5-300 nm is estimated to be between 17 % and 25 %, the uncertainty of the relative calibration is estimated to be below 20 %. For the CEM the estimated uncertainty of the absolute radiometric calibration amounts to 60 % whereas the uncertainty of the relative calibration is estimated to be 20 % in the applied wavelength range 46-116.5 nm.

A crucial influence of the installed diffraction grating on the inverse sensitivity of the VUV spectrometer between 110 nm and 130 nm was identified which is assigned to degradation effects due to VUV radiation in combination with contaminating adsorbates. The standard procedure based on the interpolation of the nitrogen branching ratios would have led to an underestimation up to one order of magnitude in this wavelength range. This observation stresses the need for preferably continuous radiation standards and the necessity of routine calibrations to monitor further degradation.

With the absolutely calibrated VUV spectrometer an emission spectrum of atomic and molecular transitions of a low pressure hydrogen discharge was taken in the calibrated wavelength range. Population densities of the first six excited atomic states were calculated from the Lyman series and an excellent agreement with results using the corresponding Balmer emission lines in the optical region was obtained. This demonstrates the validity of the presented absolute intensity calibration of the VUV spectrometer.

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