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A vacuum ultraviolet filtering monochromator for synchrotron-based spectroscopy

Ireneusz Janik [a],*, Timothy W. Marin [b],**

[a] Notre Dame Radiation Laboratory, University of Notre Dame, Notre Dame, IN 46556, USA
[b] Chemistry Department, Benedictine University, Lisle, IL 60532, USA

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ABSTRACT

We describe the design, characterization, and implementation of a vacuum ultraviolet (VUV) monochromator for use in filtering stray and scattered light from the principal monochromator output of the Stainless Steel Seya VUV synchrotron beam line at the Synchrotron Radiation Center, University of Wisconsin—Madison. We demonstrate a reduction of three orders of magnitude of stray and scattered light over the wavelength range 1400–2000 Å with minimal loss of light intensity, allowing for over six orders of magnitude of dynamic range in light detection. We suggest that a similar filtering scheme can be utilized in any variety of spectroscopic applications where a large dynamic range and low amount of background signal are of import, such as in transmittance experiments with very high optical density.

1. Introduction

We have recently designed an experiment to investigate the vacuum ultraviolet (VUV) spectroscopy of high-temperature, high-pressure water and aqueous samples, with capability of exceeding the water critical point (374 °C, 220 bar). This optical absorbance experiment presents multiple complications due to the high optical density of the sample and the high temperatures and pressures incurred. The details of the experimental setup and preliminary results are to be outlined in a series of papers. In this manuscript, we focus on the optical challenges presented by the experiment. We present the need to implement a secondary filtering monochromator for the VUV synchrotron source to minimize the amount of scattered and stray light. We discuss the monochromator design and demonstrate its capabilities, showing over 6 orders of magnitude in monochromatic light detection sensitivity.

2. Experimental

2.1. Considerations

Liquid water shows an absorption maximum at 1520 Å at 20 °C with an extinction coefficient of $\varepsilon = 1600\ \mathrm{M^{-1}\,cm^{-1}}$ (cross-section $\sigma = 6.1 \times 10^{-18}\ \mathrm{cm^2}$) [1]. The concentration of liquid water under ambient conditions is 55.4 mol $\mathrm{L^{-1}}$. Following the Beer–Lambert Law, these parameters suggest an optical density of ~9 per μm path length of sample. These low light transmittance conditions mandate two constraints to enable enough light to arrive at the detector and achieve a meaningful signal to noise ratio: (1) minimizing the sample path length, and (2) providing a large amount of incident monochromatic light flux. We have tackled the first issue by designing a sample cell with path lengths adjustable down to hundreds of nanometers (details to be discussed in a future publication). The second issue is addressed by using a synchrotron light source. Since an experimental goal is also to investigate the density dependence of the water absorption above its critical temperature, it is further advantageous to access as wide a dynamic light detection range as possible to access a wide range of sample densities without physically changing the sample cell path length.

2.2. Light source and detection

We are making use of the Stainless Steel Seya (Port-051, SS Seya) beam line at the Synchrotron Radiation Center (SRC), University of Wisconsin—Madison, whose beam line VUV monochromator is of a Seya-Namioka design [2–5]. The beam line photon flux approaches 10^{11} photons/sec for a 1-Å passband over the wavelength range needed for these experiments, 1400–2000 Å. This wavelength range is chosen to cover the lowest-lying water absorption band. It would be of interest to probe to lower wavelengths in the future, but we

* Corresponding author. Tel.: +1 574 631 5464; fax: +1 574 631 8068.
** Corresponding author. Tel.: +1 630 829 6579; fax: +1 630 829 6547.
E-mail addresses: ijanik@nd.edu (I. Janik), tmarin@ben.edu (T.W. Marin).

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are currently limited by the transmittance cutoff of sapphire windows used in the sample cell. A measure of the synchrotron beam current and SS Seya beam line flux is continually available to beam line users, allowing later normalization of transmittance signals due to beam drifts. The SS Seya output beam is horizontally polarized, and focuses to a spot that is 2×1 mm with a divergence of 10×3.5 mrad (horizontal $\times$ vertical).

Light detection is achieved using a solar-blind photomultiplier tube (Electron Tubes, Inc., 9403B, -1.65 kV bias) and photon counter (Stanford Research Systems, Inc., SR400, with SR445A preamplifier). With these components, the level of dark counts is in the range of 5 photon/sec. Two counting discriminator voltage thresholds are utilized to improve the dynamic range of our single detector, where thresholds have been selected to fall between the peak voltage distributions of 1 and 2 simultaneous detection events. Under these conditions, detection linearity is observed up to a counting rate of 2.5×10^7 photons/sec, and slits on the beamline monochromator and apertures on the beamline are adjusted to maintain an incident flux below this value. Note that a full statistical treatment such as that suggested by Kissick, et al. [6] was considered for these experiments but was deemed unnecessary considering the fast 4.5-ns rise time of the photomultiplier compared to the average pulse spacing. Considering this upper limit for detection linearity and our dark current level, over six orders of dynamic range are achievable in principle, permitting absorbance measurements of over 6 O.D. However, our diagnostics demonstrate that the extent of scattered and stray light from the SS Seya beam line is on the order of 1 part in 10^3–10^4 throughout the entire wavelength range of interest. Since part of the scattered light occurs at wavelengths not absorbed by the water sample, potential absorption measurements are restricted to 4 O.D. or less.

2.3. Needs and goals

To address the specific issue at hand for this experiment, we have added a secondary monochromator onto the SS Seya beam line to reduce the extent of scattered and stray light, thereby maximizing the dynamic range possible for absorbance measurements. In constructing the monochromator, several goals were in mind: (1) to minimize the volume of the instrument, hence reducing the amount of time needed for bake-out and pumping to achieve ultrahigh vacuum conditions; (2) to force reflections to be as close to normal incidence as possible in order to minimize the number of optical components and reflection losses; (3) to keep reflections in the original plane of the incident beam and lower the level of the output beam; (4) to use optics with reflectivities optimized for the 1400–2000 Å range; (5) to permit proper refocusing of the divergent beam into the sample cell with additional double demagnification; and (6) to use commercially available components wherever possible in order to minimize the costs of machining. Since this monochromator serves only as a filter, the ultimate goal was to maximize flux through the instrument and not be concerned with its resolving power. As such, the primary Seya monochromator is used to dictate wavelength resolution.

3. Design

3.1. Optical design

A long history exists regarding the issues in dealing with the obstacles of instrumental stray and scattered light. It is well-known that undesirable wavelengths reaching the detector may arise from optical imperfections, dust, and grating irregularities. Much of this light can be reduced by careful instrumental design, and we refer the reader elsewhere for general considerations [7,8].

Geometric constraints of our pre-existing apparatus and experimental workspace limited the possible size and shape of the monochromator, necessitating that optical path stray from normal-incidence conditions, vide infra. The commercial availability of the necessary optics further limited our choices for refocusing the beam and forced us to keep the beam from diverging to too large a radius.

With these limitations in mind, ray tracing simulations were performed using Shadow GUI v. 2.3.3 [9] with the parameters of the emerging SS Seya beam from the primary monochromator as the source light. Only two optical elements were used in simulations—a simple plane mirror followed by a concave diffraction grating with a 1.00-m radius. The long focal length of the grating is necessary due to geometrical constraints of the rest of our apparatus, requiring the beam to focus into a separate chamber beyond the filtering monochromator that houses the water sample. An overview of the optical beam path is shown in Fig. 1, where many of the vacuum components have been omitted from the figure to emphasize the optical path. The mirror is placed 1182 mm after the focus of the emerging SS Seya beam, and reflects the center of the beam back upon itself horizontally, but pitches the beam downward at an 18.0° angle. The center of the beam then travels 297 mm before reflecting off the center of the grating, which redirects the beam upward by 18.0°, giving an output beam parallel to the original SS Seya output that focuses at a distance 780 mm past the grating, i.e. 394 mm past the exit flange of the UHV chamber. The distance of the focus from the exit flange is in fact comparable to the distance of SS Seya beamline focus from its exit flange, and hence leaves plenty of working space for setting the experimental chamber containing the sample cell. The rms diameter of the beam at the mirror, grating, and final focus (circle of least confusion) were calculated to be 11.7, 14.5, and 0.45 mm, respectively, and a calculated representation of the final beam focus is shown in Fig. 2. Calculations revealed that < 5 Å resolution should be easily achievable at the sample cell, whose 1.7-mm optical aperture works as a resolving slit. Over the experimental wavelength range, the final focus size is calculated to vary by less than 2%, so translational corrections to the concave grating rotation are unnecessary. Consequently, the grating assembly was designed to simply rotate about the horizontal axis center of curvature for wavelength selection without any translational correction for the wavelength dependence of the focus.

Additional simulations were carried out using parameters that might be applicable to a variety of UV beamlines, where the photon

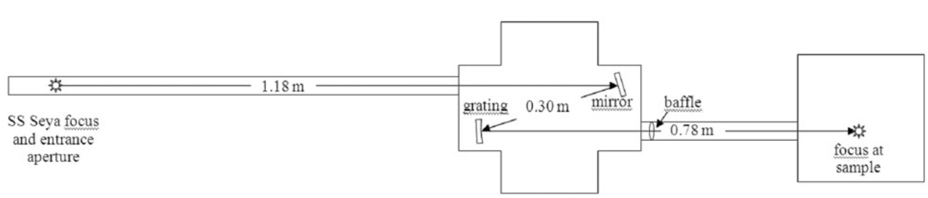

Fig. 1. Overview of optical path through the filtering monochromator.