

Atmospheric pressure photoionization using tunable VUV synchrotron radiation

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ABSTRACT

We report here the first coupling of an atmospheric pressure photoionization (APPI) source with a synchrotron radiation beamline in the vacuum ultra-violet (VUV). A commercial APPI source of a QStar Pulsar *i* from AB Sciex was modified to receive photons from the DISCO beamline at the SOLEIL synchrotron radiation facility. Photons are delivered at atmospheric pressure in the 4–20 eV range. The advantages of this new set up, termed SR-APPI, over classical APPI are spectral purity and continuous tunability. The technique may also be used to perform tunable photoionization mass spectrometry on fragile compounds difficult to vaporize by classical methods.

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1. Introduction

Atmospheric pressure photoionization (APPI) is the latest of the atmospheric pressure ionization techniques to appear, which may be coupled to liquid chromatography. It was developed independently and simultaneously by Bruins and co-workers [1] and by Syage and coworkers [2]. The development of this ionization source was driven by the need for an ionization method to study hydrophobic compounds. The technique is based on VUV irradiation of an atmospheric spray of the solution containing the analyte. Fig. 1 shows the principle of the AB Sciex PhotosprayTM ion source.

The nebulizer is made of a quartz tube resistively heated in which the liquid flux from the liquid chromatography is sprayed. The gas phase formed this way is submitted to irradiation by VUV photons coming from a discharge lamp in rare gases. The most widely used source is the krypton lamp, producing photons of 10.6 eV in the DC mode, with a minor contamination of 10 eV [3]. Xenon (8.4 and 9.6 eV) and argon (11.8 eV) lamps may be also found commercially and are occasionally used [4]. Ions generated through the interaction of VUV light with the spray are attracted by a voltage difference between the lamp holder and the entrance plate of the mass spectrometer [1].

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In the case the analyte (M) can be ionized directly by the photons but not the solvents, a radical cation M^+ is formed in the vapor phase, which might react further [5]. This mode of operation, referred to as direct photoionization, as opposite to mediated (or assisted) photoionization, sometimes lacks of sensitivity. When the solvent itself is ionized, cascades of reactions lead to chemical ionization, which assists in the ionization of the analyte and increases the sensitivity [6]. Often, the solvent system coming from the liquid chromatography pumps cannot be ionized by the photons of the lamp. In such cases, the assistance effect is obtained by the adjunction of an organic molecule, called dopant, which is easily ionized by the lamp. Toluene and acetone are among the most commonly used dopants [7].

Since its release on the market in the beginning of the century, APPI has attracted a growing interest [8–12]. Indeed, any kind of solvent may be used with APPI, even the most non-polar one [13–15].

We report here the concept and the first results of the coupling of an APPI source from AB Sciex with DISCO, a VUV beamline at the SOLEIL synchrotron radiation facility [16]. First of all, the use of monochromator ensures spectral purity, which was not granted by the line sources of classical devices. In addition, photon wavelength tunability allows for a better control of the ionization and fragmentation process. Because the wavelength range available is extended as compared to the lamps, some solvents are now accessible to ionization, thus avoiding the necessity of the addition of dopant molecules.

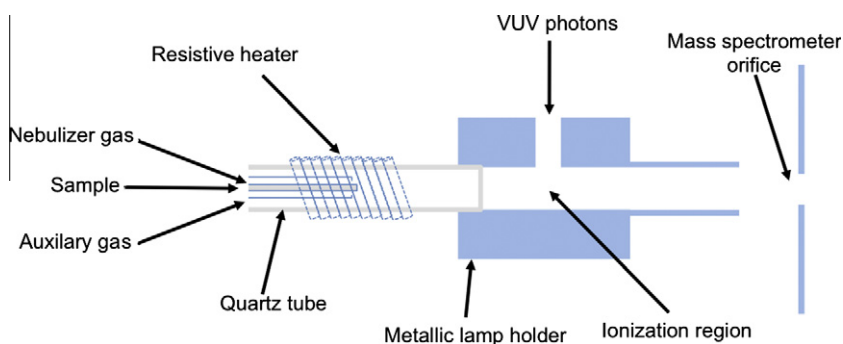


Fig. 1. Schematic of the APPI ion source.

2. Technical description and design

The DISCO beamline has been described in details elsewhere [16]. Briefly, it is a bending magnet based beamline, which delivers UV and VUV photons to three end-stations. One of those, referred to as the Atmospheric Pressure EXperiment (APEX), receives the atmospheric pressure photoionization set up. The energy domain covered by APEX ranges from 4 to 20 eV, using a 250 lines mm^{-1} variable line spacing grating blazed at 10 eV. Typical resolving power goes from 875 (550 μm slits) at 60 nm (20.66 eV) to 2700 (800 μm slits) at 180 nm (6.89 eV). This energy domain includes and extends all the commercially available APPI line sources. The photon energy scale is calibrated using the photoabsorption lines of xenon, argon and nitrogen.

The beamline has been fitted with a differential pumping system to deliver photons of energy higher than the transparency limit of the vacuum windows (such as MgF_2 or CaF_2), whose detailed description has been given elsewhere [17]. Table 1 lists the ionization energy of some of the most common solvents used in liquid chromatography, as well as that of nitrogen. Feeding the differential pumping with a well-chosen gas leads to define a spectral region in which VUV photons will be transmitted. Neon, argon and nitrogen cut off photons below 60, 94 and 99 nm, respectively [17]. The valve that isolates the differential pumping from the beamline bears a MgF_2 window. The latter is transparent down to 115 nm [17].

It appears immediately from Table 1 that, thanks to the tunability of synchrotron radiation, some very common and useful solvents, such as methanol, acetonitrile, methylene chloride or

water, may be ionized by APPI. This clearly suppresses the need for addition of acetone, toluene or anisole.

One of the extra advantages of the differential pumping system is that it also acts as a gas filter, suppressing higher orders from the monochromator [18]. This specificity guarantees spectral purity.

A commercial ion source has been modified so that the photons could be delivered by the beamline. The nebulizer and housing have been preserved in order to maintain conditions inside the source similar to those of the commercial set up with the lamp. Fig. 2 shows a panoramic photograph of the APEX branch line, showing the four-stage differential pumping along with the ion source and mass spectrometer. The mass spectrometer is a QStar Pulsar *i* from AB Sciex.

The photon beam height is 1400 mm from the ground [16], which does not match the height of the ion source of the QStar, measured well-at 1155 mm. In order to properly align the source and mass spectrometer to the photon beam, a frame has been designed to support the spectrometer and to allow fine alignment. This frame is depicted at Fig. 3. Three high torque stepper motors (KML062F07 from SLO-SYN) ensure translation of the spectrometer along vertical (z) and horizontal (x) axis and in the direction of the beam (s). The stroke for each of those movements is ± 50 mm for x , $-20/+120$ mm for z and ± 50 mm for s axis. The vertical z movement is ensured by four ball screw jacks (from Power Jacks). The alignment is carried out first by sending visually the white beam (monochromator at zero order) into the ionization region. Then, a finer alignment may be realized on the magnitude of the total ion current recorded by the mass spectrometer.

Table 1

List of the main organic solvents and dopants used in APPI along with their ionization energy (from the NIST database) [21]. The nature of the gas fed into the differential pumping system is also given. The shaded part of the table indicates when use of the differential pumping is mandatory.

Compound	Ionization energy (eV)	Gas	Configuration
N_2	15.58	Neon	Differential pumping
Water	12.63	Neon	Differential pumping
Acetonitrile	12.20	Neon–Argon	Differential pumping
Chloroform	11.37	Neon– N_2	Differential pumping
Dichloromethane	11.32	Neon– N_2	Differential pumping
Methanol	10.84	Neon–Ar– N_2	Differential pumping
Ethanol	10.48	Neon–Ar– N_2	MgF_2 window valve
Isopropanol	10.17	Ar– N_2	MgF_2 window valve
<i>n</i> -Hexane	10.13	Ar– N_2	MgF_2 window valve
Heptane	9.89	Ar– N_2	MgF_2 window valve
Acetone	9.70	Ar– N_2	MgF_2 window valve
Tetrahydrofurane	9.40	Ar– N_2	MgF_2 window valve
Toluene	8.83	Ar– N_2	MgF_2 window valve
Anisole	8.20	Ar– N_2	MgF_2 window valve

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