Nota Técnica / Technical Note

The U-SPEC probe – I+D+i @ ICIQ

Resumen: Esta nota técnica de OPA presenta las principales características de la sonda de inserción U-SPEC desarrollada en el Instituto Catalán de Investigación Química (ICIQ) por el grupo de investigación del Dr. Galan-Mascaros, así como una nota de aplicación describiendo diferentes usos.

Abstract: This OPA technical note presents the main characteristics of the U-SPEC insertion probe developed at the Institute of Chemical Research of Catalonia (ICIQ) by the research froup of Dr. Galan-Mascaros, and a description of potential applications.

ICIQ and novel scientific Instrumentation

For the last 8 years, we have been developing tailor-made physical/chemical instrumentation in the Galan-Mascaros group to solve some of the issues we continuously encounter during our research work in materials sciences. Major instrumentation companies attend routine conditions and problems, but they cannot meet especial conditions, needs and specifications from research laboratories. In most cases, this is due to technical difficulties, complex implementation, and limited size of the marketplace. One of the major motivations for our research team at ICIQ is to develop new scientific instrumentation to cover some specific issues, and along our natural interest in solving scientific challenges, ICIQ and ICREA are going to support the transfer of our advanced instrumentation to a novel spin-off company, ORCHESTRA SCIENTIFIC S.L., with the aim of becoming a unique and exceptional ally to research laboratories and industries with a high I+D+i requirements. As a company, ORCHESTRA Scientific will have a wide experience in research, providing the most adequate solutions to face instant technological challenges, offering commercial products for academics, research and industry. ORCHESTRA Scientific is expected to be fully operative by the end of the year. Meanwhile, its product portfolio is available for testing and evaluation.

The U-SPEC insertion probe

Especially appealing to the readers of Óptica Pura y Aplicada will be the U-SPEC insertion probe. This ICIQ/ICREA patented instrument has been designed to be inserted in a cryostat, allowing the optical pathway to enter (and exit) the cryogenic chamber to collect transmission spectroscopic data from solid state samples (pellets, thin films or single crystals) in the UV-VIS-IR range. U-SPEC is adapted to confined spaces, works in a very large temperature range from 400 K down to 2 K, supports high vacuum, and high magnetic fields (up to 10 T). With a versatile and innovative design, U-SPEC can be adjusted to any commercial or homemade cryostat to perform transmission experiments down to very low temperatures.

This alternative to the most common solution of cryostats equipped with Quartz windows, significantly reduces the free path light, leaving less than 1 mm distance between the sample and the collimating lenses. This is advantageous for several reasons: i) minimizes signal losses; ii) facilitates alignment; iii) allows using small or non-uniform samples and low intensity light sources. In addition, this probe can be combined with magnetic cryostats, where the Quartz windows solution is not appropriate. Indeed, two U-SPEC models are available for the Quantum Design MPMS-XL® and Quantum Design PPMS®. Our long-term strategy is to offer U-SPEC models to transform any scientific equipment with cryogenic capabilities (NRM, EPR, etc.) into a fully-operative spectrometer with an exquisite temperature control.

Its innovative design includes an experimental space sealer, an optic rod and sample holder (Figure 1). The space sealer can be adapted to the inlet mechanism of any cryostat and it contains 4 SMA fibers connectors (other connections may be available upon request). The optic rod brings the optical fibers to the sample holder, at the experimental required position inside the cryostat. It has been tested to perform measurements of transmittance/absorbance and fluorescence in sample pellets, polymer films (PPMA or silice) or single crystals.

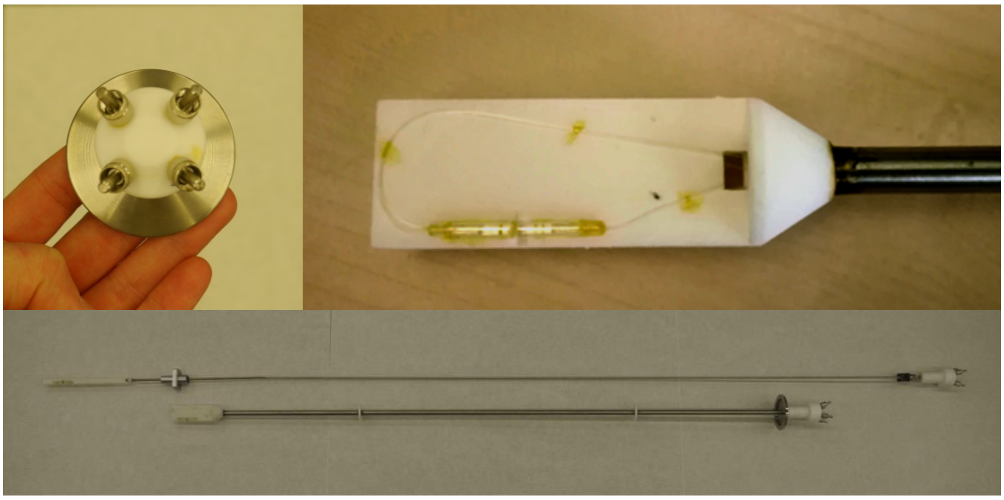

Fig. 1: upper- left corner: space sealer with 4 SMA connector. Upper-right corner: Sample holder. Bottom: two different U-specs adapted to different cryostats.

One interesting feature of U-SPEC is that two optical paths transit the experimental space, one for transmission data and one for background signal. This allows dynamic correction of fluctuations of the intensity power or environmental effects on the fiber optics. The use of this reference extremely minimizes the noise/signal ratio (see figure 2).

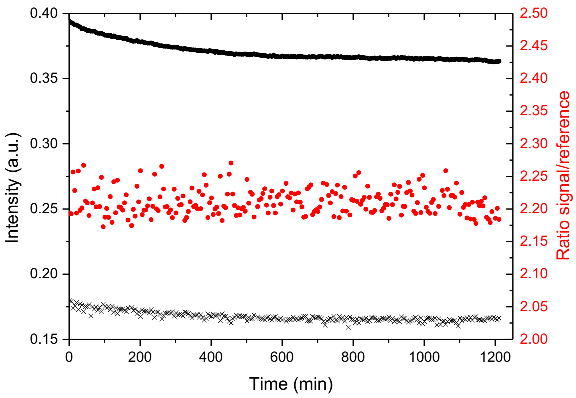

Fig. 2: Time-signal stability (ratio signal/reference in red; transmission and background signals in black: full and empty circles, respectively)

Application Note 1: Absorbance spectra of solids down to very low temperatures.

Collecting UV-vis spectroscopy data at very low temperatures is typically a cumbersome problem. Commercially available spectrometer offer very limited temperature control, typically between –20 and 80°C based either on Peltier technology or on a cryogenic bath. When the user requires lower temperatures, the most common approach is the use of spectroscopic insertion probes which can be inserted into cryogenic baths at fixed temperature, typically down to liquid nitrogen temperature. When precise temperature control or very low temperatures are desired, there is no readily accessible solution to solve this technical problem, except the use of liquid He cryostats with quartz windows from specialized companies. Such approach is justified for large infrastructures, such as synchrotron facilities, but technically and economically very limited for wide applications. Additionally, their use for spectroscopic measurements (when the transmitted light needs to be collected after passing through the sample) is cumbersome due to the long distance between the irradiation source, the sample and the detection system, since the exterior ring of a cryostat is typically over 5 cm wide.

To illustrate the performance of our U-SPEC insertion probe, we present here the temperature-dependent absorbance data for $Co(NO_3)_2.6H_2O$ (Figure 3). This simple salt was dissolved in water, and the solution was jellified with a silicone precursor to form a transparent film. A 2 x 2 mm^2 piece was mounted on the sample space of our U-SPEC model design to fit the

Quantum Design MPMS-XL® cryostat. The absorbance was measured at different temperatures down to a minimum of 2 K with a H9305-04 Hamammatsu detector and a TILL Photonics 150 W Xenon high stability lamp. The spectra show significant variations with temperature due to the thermal depopulation of the energy levels of octahedral Co^{II}.

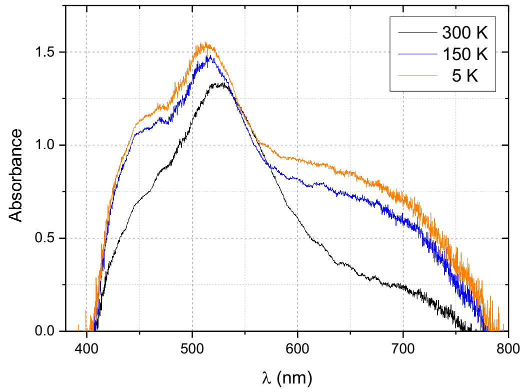

Fig. 3: Absorbance of the salt $Co(NO_3)_2.6H_2O$ as a function of the temperature.

Application Note 2: Absorbance as a function of the magnetic field

Spectroscopy experiments under magnetic fields are even more difficult. The simple solution of Quartz windows is not available in simple magnetic cryostats. Indeed, such measurements are typically associated to large facilities (synchrotron) or specialized research groups. With our U-SPEC probe any magnetometer can be immediately and easily transformed into a magnetospectrometer. U-SPEC is built with non-magnetic parts, and can be adjusted to any insertion mechanism. The stability of the signal under magnetic fields is excellent, as supported by the data in Figure 4. The absolute absorbance of $[Ru(bpy)_3]Cl_2$ as a pressed KBr pellet is robust and reliable under magnetic fields up to 7 T. These measurements were also performed with a Quantum Design MPMS-XL® magnetometer.

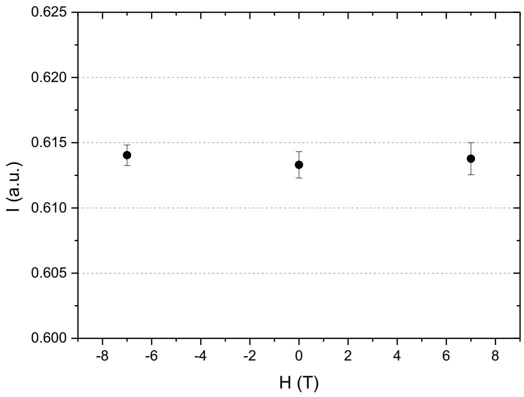

Fig. 4: Stability of the transmission data (λ = 550 nm, T = 70 K.) under magnetic fields: relative error (< 0.2 %).

Acknowledgment

U-SPEC concept and development has received funding from the European Research Council (ERC) through ERC-Stg-279313 (CHEMCOMP) and ERC-PoC-713539 (U-SPEC) grants under the European Union's Horizon 2020 research and innovation program.

