

# Mid Infrared Spectromicroscopy Beamline 01B1-1

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## Beamline Overview

Status	Operational – accepting proposals
Source	Bending Magnet
Spectrometer	Bruker IFS66v/S FTIR
Spectral range	6000 - 750 $\text{cm}^{-1}$ (< 0.5 eV)
Flux	$10^{13}$ Photons/s/0.1%BW
Brilliance	$10^{16}$ Photons/s/0.1%BW / $\text{mm}^2/\text{mrad}^2$
Resolution	16 – 0.12 $\text{cm}^{-1}$
Spot size	Diffraction limited

## Introduction

The Mid-Infrared Spectromicroscopy beamline was conceived as a laboratory for high-spatial resolution spectromicroscopy. It takes advantage of the brightness of synchrotron IR emission to overcome throughput limitations of IR microscopes when operated close to the diffraction limit. As such, it is an effective tool for performing spectromicroscopy and mapping experiments on small samples in the micrometre size range. The beamline has been operational for users since July 2007. Table 1 shows a chronology of the milestones for the beamline.

**Table 1:** Beamline 01B1-1 Milestones

2005	Beamline construction, first light and spectra
2006	Commissioning begins
2007	First open to users
2008	Full user operations
2009	Hyperion 3000 microscope with FPA detector acquired

## Science

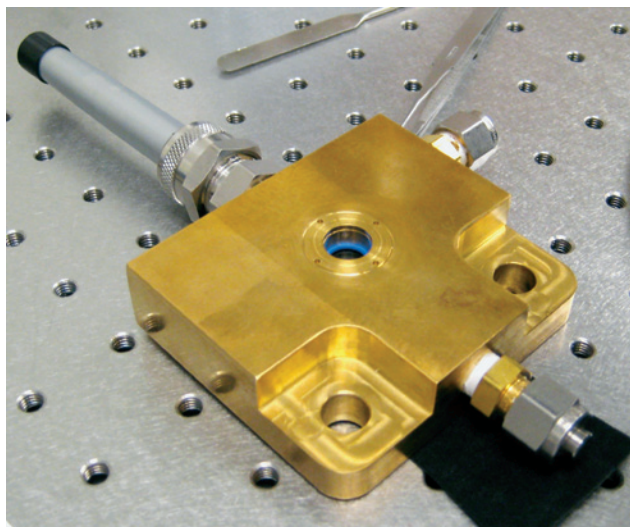
The beamline is currently being used for diffraction limited spectromicroscopy and mapping in the mid-infrared region and for photoacoustic spectroscopy in the mid- to far-infrared regions. The user community has a variety of research interests, mostly pertaining to the life sciences. These include research in plant freeze-resistance, environmental chemistry, pathology, fungal biology, animal science, agriculture, food science and cellular biochemistry. Interest has also been expressed by groups working in material and surface science, chemistry and geology, and applications are expected to expand in this direction in the future.

A research-level Bruker Optics IFS66v/S FTIR spectrometer and a Hyperion 2000 IR microscope operate as the beamline's endstation. Light extracted from port 01B1-1 is relayed using a combination of four planar and three ellipsoidal gold-

coated mirrors. A diamond window separates the Ultra High Vacuum of the storage ring from the rough vacuum section of the beamline vacuum system. A KRS-5 window is between the spectrometer and beamline.

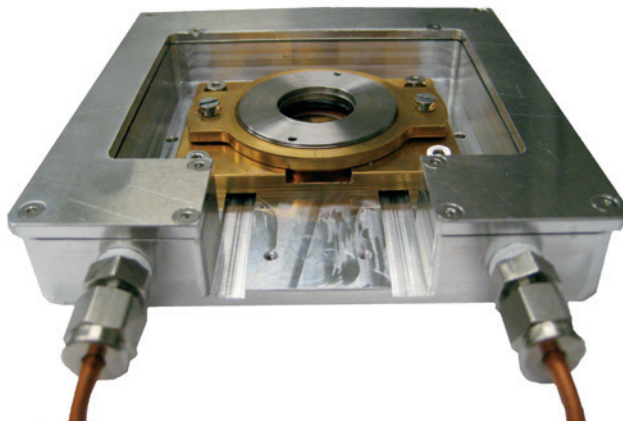
Features include wide spectral coverage, step-scan operation, vacuum operation, and low noise level ( $\sim 10^{-6}$  AU). The Hyperion 2000 IR microscope uses 15x, 36x, Attenuated Total Reflection and Grazing Incidence Reflection objectives. The microscope performs reflection or transmission experiments and mapping areas of interest on the sample.

An MTEC photoacoustic cell is available for use in the sample compartment of the spectrometer, and a cell is under development for photoacoustic spectroscopy of microscopic samples. The cell consists of a small sealable volume connected to a highly sensitive microphone (see Figure 1). The cell mounts on the microscope stage allowing sample visualization and beam focus using the microscope objective. The unit will be used to perform photoacoustic studies of individual particles, grains and surfaces.



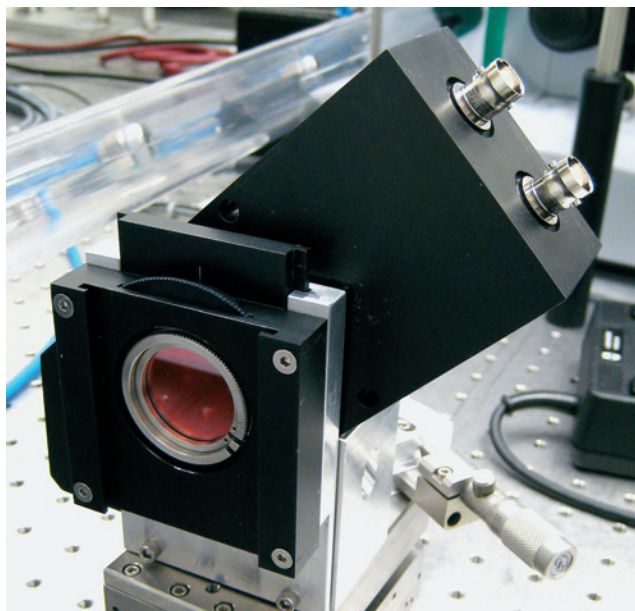
**Figure 1:** Sample cell for photo-acoustic spectromicroscopy. A window and spacer are above the cell volume, and the detector microphone is on the left side.

A sample cell is in development to allow temperature controlled spectromicroscopy measurements on samples in liquid (see Figure 2). Operating temperature ranges from  $-15$  to  $+100^\circ\text{C}$ . An INSTEC stage for measurements needing a greater temperature range is available for solid samples.



**Figure 2:** Thermostatic cell for liquid samples. The cell sits at the focus on the sample stage of the Hyperion microscope.

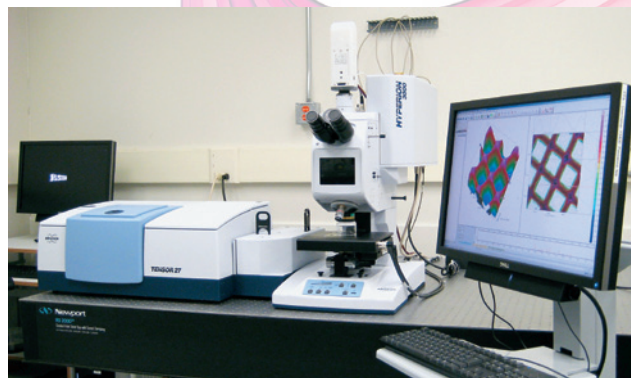
A Hinds ZnSe photo-elastic modulator, shown in Figure 3, has been used in-line with the Hyperion microscope to allow rapid vibrational linear dichroism (VLD) measurements on microscopic samples [1]. The unit operates in double modulation experiments, in conjunction with a GWC demodulator. Step scanning using a lock-in amplifier is being evaluated as another capability for users.



**Figure 3:** Photo-elastic modulator for VLD experiments on microscope samples. Analysis of oriented bonds is possible.

The facility now has a new Bruker Tensor 27 FTIR spectrometer, with a resolution better than  $1\text{ cm}^{-1}$ , and a Hyperion 3000 microscope. It was installed in August as an off-line tool for large area mapping (see Figure 4). This microscope is available for use during scheduled beamtime, and is located in the same room as the mid-IR endstation. The new microscope has a standard  $250\text{ }\mu\text{m}$  single element MCT detector, and a  $64 \times 64$  element focal plane array (FPA) detector. The FPA makes possible very rapid scanning of 4096 spectra over an area  $170\text{ }\mu\text{m} \times 170\text{ }\mu\text{m}$ , using the 15x

objective. By concatenating FPA images into a mosaic a very large area is quickly mapped. This allows users to scan large areas of samples or tissues to determine regions of interest for the higher spatial resolution scans using the beamline Hyperion 2000.



**Figure 4:** Bruker Tensor 27 and Hyperion 3000 microscope with focal plane array detector obtains  $3\text{ }\mu\text{m}$  pixel size using a 15x objective.

The installation of an active optics unit allows for compensation of synchrotron beam drift and stabilization of alignment, with remarkable effects on beamline performance [2]. Commissioning experiments show the following noise levels for a  $3\text{ }\mu\text{m}$  pinhole in transmission:  $1000\text{-}1200\text{ cm}^{-1}$  region: 0.03 (%T) RMS.  $2800\text{-}3000\text{ cm}^{-1}$  region: 0.008 (%T) RMS.

The mid-IR beamline is fully operational, with excellent signal-to-noise levels and diffraction limited performance obtained. Development has now shifted to accessories and ancillary equipment optimized for specific user-driven experiments.

## References

1. May, T., Ellis, T. and Reininger, R. 2007. Mid-infrared spectromicroscopy beamline at the Canadian Light Source. Proceedings of 14<sup>th</sup> National Conference on Synchrotron Radiation Instrumentation. N.I.M.A. 582, 111-113.
2. Quaroni, L., Zlateva, T. et al. 2008. Measurement of Molecular Orientation in a Subcellular Compartment by Synchrotron Infrared Spectromicroscopy. ChemPhysChem, 9, 1380 – 1382, DOI: 10.1002/cphc.200800211.