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C^{2}SIRS: the Canadian Consortium for Synchrotron Infrared Spectroscopy

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Principal Contacts

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General Specifications for the IR Beamlines

Status	Approved and funded as of December 31, 2001
Source	Bending magnet
Monochromator	Michelson interferometer
Spectral range:	
IR-1 (far IR)	10–4000 cm^{-1}
IR-2 and IR-3	450–6000 cm^{-1}
Flux at 1000 cm^{-1}	10^{13} photons/mm^2/100 mA
Brilliance	10^{16} photons/mm^2/mrad^2/ 0.1% bandwidth
Resolution	0.001 cm^{-1}
Focused spot size	3 µm

Overview

Three infrared beamlines have been approved at the CLS. A single design team is designing them, and a single beamline team will eventually manage them. These three beamlines will form the core of C^{2}SIRS: the Canadian Consortium for Synchrotron Infrared Spectroscopy. The goal is to create a national resource within the CLS, where Canadian researchers will have access to the most advanced instrumentation available. Canada has a long tradition of excellence in IR spectroscopy, and thus we are able to draw on a large pool of expertise from the academic, government and industrial communities.

Two beamlines (IR-1 and a combined IR-2/3) are currently being built for the initial suite of CLS beamlines. After studying a number of options, it was decided that the three beamlines would have identical designs for the collection optics and vacuum chambers, with optimum performance for both the mid- and far IR.

Each beamline will have an endstation chosen for the scientific mission of the beamline. In the case of IR-1, it will require a spectrometer with the highest possible spectral resolution, as well as optics for the far IR. In the case of IR-2/3, the endstation will require a high-quality microscope, a photoacoustic cell, and a wide range of sample configurations.

Science

Far IR Spectroscopy: IR-1

The IR-1 beamline will be a facility for high-resolution far infrared spectroscopy.

High resolution. The emphasis on high resolution spectroscopy relates directly to the high brightness available from a synchrotron. Brightness (or “brilliance”) can be expressed as infrared flux through a given focal spot (aperture) for a given solid angle. The relation between brightness and high *spatial* resolution is self-evident. The relation between brightness and *spectral* resolution is connected with the way a Fourier transform spectrometer (FTS) works.

For a particular wavelength, spectral resolution, and collimator focal length, there is a maximum aperture size that ensures that the incoming IR radiation is sufficiently parallel to give interference fringes in the Michelson interferometer, which is the heart of the FTS. For example, to give Doppler-limited resolution (approximately 0.0008 cm^{-1}) of CO_2 molecules at 200 K and 500 cm^{-1} (λ = 20 μm), the required aperture diameter is only 0.8 mm for an f/4 FTS. A synchrotron can deliver far more IR photons through this small aperture than can a thermal source like the globar normally used in IR spectrometers. In contrast, for "medium" spectral resolution (such as 0.2 cm^{-1}) at the same wavelength, the maximum aperture is 13 mm, and the globar, being an extended source, can deliver more photons than the synchrotron. High resolution is valuable not just for its own sake, but also because it is clearly desirable to work at the "intrinsic" resolution of a physical system (such as at the Doppler width for a gas sample). Otherwise, one loses very valuable characteristic information about that system.

Far infrared region. We are especially interested in the far infrared region precisely because it is presently so difficult to obtain good results using conventional sources. This region includes the rotational spectra of important light molecules, and the low-frequency vibrational modes of many interesting systems undergoing large amplitude motion. Far IR has also recently become accessible for astrophysical studies using satellite-borne spectrometers. Thus, the interest is high from the molecular spectroscopy point of view in spite of many difficulties.

Superior far infrared (FIR) capability will also be important for studying condensed matter at high *spatial* resolution, where source brightness is a major experimental limitation. Since the energies of phonons in highly correlated electronic materials and intermolecular vibrations in solids are in the far IR region, the IR-1 beamline will enable advanced study of many systems. One example area is the study of matter under high pressure using diamond anvil cells, where sample volumes are by necessity very small and the cell windows have a high refractive index.

IR Spectromicroscopy: IR 2/3

High source brightness is the key requirement of point-by-point mapping in IR spectromicroscopy. Only synchrotron sources make it possible to reach the diffraction limit of spatial resolution, namely 3–20 μm in the mid-IR.

Shannon Mould, Curtis Mullin, Chad Sewell and John Swirsky checking diagrams.

Biological Emphasis: IR-2

For complex biological samples such as human tissues, the wavelengths of infrared light absorbed by a sample (its infrared spectrum) provide a direct indication of sample biochemistry. In essence, the IR spectrum is a biochemical fingerprint. With the correct choice of sampling methodology—such as an infrared microscope—information on the biochemical nature of disease states can be obtained from tissue samples. This information can often be useful diagnostically: variations in spectral signatures arising from nucleic acids, proteins and lipids can provide important information about a number of disease states.

With traditional instrumentation, samples of 20 µm or greater must be analysed, meaning that spectra are acquired from groups of cells within tissues, rather than individual cells. Spectra may therefore represent an average of normal and abnormal cells. Using a synchrotron as the source of the infrared light, spectra may be acquired from sample regions as small as 5 µm, allowing studies of tissues on a cell-by-cell basis. The potential applications of this high resolution IR spectromicroscopy are enormous. For example, it will allow the molecular nature of a number of forms of cancer and Alzheimer’s disease to be probed. It is expected that synchrotron-based IR microscopy will form an important part of a unique new programme in Imaging in Infectious and Endemic Diseases being developed jointly by the NRC (the Institute for Biodiagnostics and the Biotechnology Research Institute), Health Canada, Agriculture Canada/ Canadian Food Inspection Agency, and the Department of National Defence.

Of equal importance is that synchrotron-based IR microscopy will enable studies of the interaction of new drugs with individual human cells, providing a new method for assessing the effectiveness of these agents. Such studies will be very useful to the Canadian biotechnology industry.

Industrial Emphasis: IR-3

The infrared spectrum of a material is one of its most highly characteristic properties. In fact, because samples in virtually any form can be analysed, infrared spectroscopy has become one of the most powerful analytical techniques available.

The practical advantages of being able to analyse very small samples (or small areas of larger samples) are many and varied. Applications range from semiconductor analysis to art conservation. For example, various small impurities on the surfaces of semiconductors can be detected, which is useful in the quality control of microelectronics. Difficult materials such as clay and carbonaceous solids can also be studied. In the field of art conservation, tiny fragments of important artifacts (such as documents, books, paintings, or statues) can be examined to assess the state of the artifact before, during and after cleaning.

The proposed beamline will be used by a wide variety of industrial, government and academic scientists in applications that will have significant effects upon the lives of many Canadians.

Instrumentation

Far IR Spectroscopy: IR-1

The spectrometer must meet exacting specifications.

Spectral resolution. The spectrometer must have a maximum optical path difference (MOPD) of 7 metres or greater, corresponding to a resolution limit of 0.00086 cm^{-1} or better (as given by the full width at half maximum of the unapodized instrumental line shape, approximately 0.60/MOPD).

Wavenumber range. The spectrometer must provide coverage from less than 50 cm^{-1} to greater than 10 000 cm^{-1}. The primary range for the CLS FIR beamline will be 50–1000 cm^{-1}.

Vacuum. The spectrometer must be fully evacuable to a pressure of less than 0.1 torr.

Operational considerations. The spectrometer should be capable of efficient operation at low (greater than 1 cm^{-1}) and medium (0.1–1 cm^{-1}) resolution, as well as at higher resolution (less than 0.1 cm^{-1}). The spectrometer should be a standard production item, not a “one-off” or prototype instrument. The spectrometer should utilize a well-supported standard software package for instrument operation, data collection, and data manipulation. Since the CLS is a user facility, the software should be actively supported by the manufacturer and should be compatible with industry standards.

All of these requirements can be met by the Bruker Model IFS 120 HR spectrometer. The IFS 120 HR is a modular instrument, and is available with a resolution limit from 0.005 cm^{-1} to less than 0.001 cm^{-1}. Increased MOPD is achieved by adding extension modules that provide longer mirror travel. A number of labs operate versions of the IFS 120 with MOPD = 7.14 m.

An application headed by Bob McKellar was made in October of 2001 to the NSERC Major Installation Grant program for partial funding of the spectrometer. The decision is expected in March, 2002.

IR Spectromicroscopy: IR 2/3

The endstation will be a research-level commercial FTIR spectrometer coupled to a synchrotron IR microscope. It must initially satisfy the requirements of both the biological and industrial research communities, so it will be equipped with the widest possible range of sampling accessories in order to satisfy their diverse needs. Among these needs, photoacoustic FTIR spectroscopy is particularly important. Requirements for the spectrometer include the following:

- Step-scan capability for depth profiling studies
- Rapid scan capability
- Wide spectral range (10 cm^{-1}–12000 cm^{-1})
- Dual-source capability
- Far IR and mid-IR detectors
- A wide range of sampling accessories, including
  - —a photoacoustic cell
  - —diffuse reflectance capability
  - —ATR accessories
  - —mid-IR polarizers
- An IR microscope with
  - —a mapping stage and associated software
  - —a high-sensitivity, long-hold MCT detector
  - —interchangeable ATR and grazing-angle objectives
  - —a diamond anvil cell
  - —an adapter to allow upgrading to Focal Plane Array operation
  - —a fluorescence attachment.

Layout

All three beamlines will have essentially the same layout, which is shown in Figure 7·1. The primary differences between the beamlines will be in the designs and the capabilities of their FTIR spectrometers.

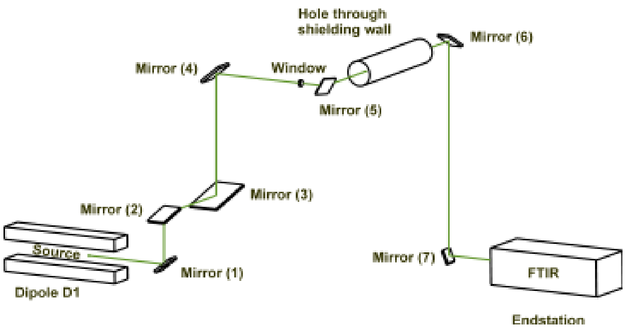

Figure 7·1 *Simplified diagram of a generic IR beamline.*

Performance

Locating and sizing the infrared ports was a large part of the initial design process, as these factors strongly affect the output available. Re-imaging the source, extracting photons from the UHV environment, and relaying the beam to the spectrometer are also important. The expected flux and brightness curves reflect the available output for the design of 58-milliradian vertical acceptance. This size allows an optimum match for a wavelength of 170 μm (70 cm^{-1}). Figures 7·2 and 7·3 compare the brightness and flux of various sources with those of the CLS at an operating current of 100 mA.

The current design will use the radiation from a standard bending magnet. The heat load issue is being addressed by using a slotted mirror design that will allow the high-energy photons to pass through and not be absorbed. The use of diamond for the window material will allow the passage of visible to long-wave IR radiation, and provide full spectral output for both beamlines.

The beam paths are being kept to the shortest distances possible to make the collimated beams less susceptible to mechanical vibration. This entails having the beam-path go through the sidewall of the storage ring tunnel, and removes the need for lead shielding at the experiment station. The evacuated beam path will minimize interference from atmospheric absorption. The floor areas for the experimental endstations will be enclosed to provide a controlled environment for the researcher and to comply with Biosafety Level 2 requirements.

The preliminary design report was completed in October of 2001. Detailed design is in process.

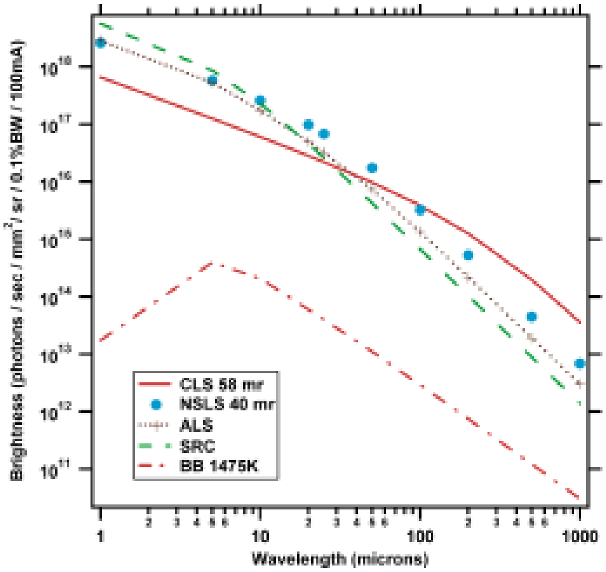

Figure 7·2 *Brightness expected for the CLS operating at 100 mA current, compared to IR beamlines at other synchrotrons and to black body radiation.*

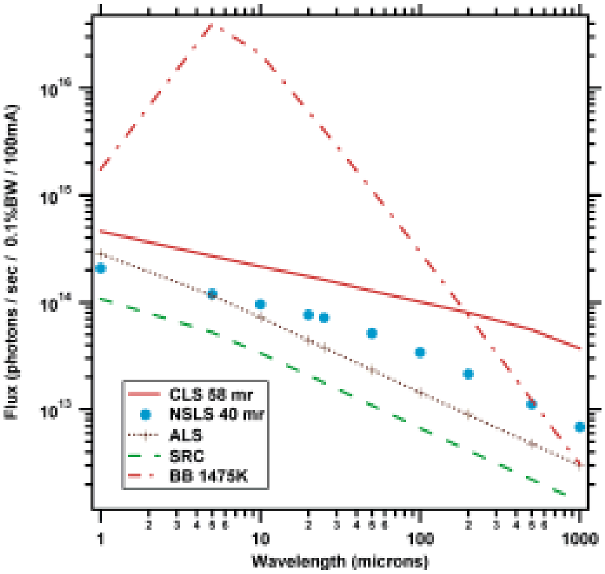

Figure 7·3 *Flux expected for the CLS operating at 100 mA current, compared to IR beamlines at other synchrotrons and to black body radiation.*

Beam Team Research Activities

IR-0: Beamline U10B at the NSLS

A Memorandum of Understanding (MOU) has been signed by G. M. Bancroft on behalf of CLSI, by which the CLS became part of a Participating Research Team (PRT) on the U10B Beamline at NSLS at the Brookhaven National Laboratories. Under the terms of the MOU, Canadian scientists were given access to 20% of the available beam time on this endstation. In return, CLSI has made arrangements to provide on-site support for Canadian and general users of the beamline. The MOU came into effect on September 1 of 2001, with the appointment of Tim May at the CLS. While his main responsibility was to design the IR beamlines at the CLS, Dr. May also assisted CLS users at U10B during allotted time slots, and provided some general user support during the first 10 months of the PRT. Dr. Nebojsa Marinkovic, a beamline scientist with the IR line at U2B (NSLS), took over the responsibilities for the half-time position at NSLS on June 1, 2001.

The U10B line is equipped with a Nicolet 860 FTIR and a Continuum microscope with independent fluorescence imaging capabilities. IR mapping can be achieved with spatial resolution down to a few microns, depending on the nature of the sample and the spectral regions of

Kathy Gough (University of Manitoba) working at the NSLS U10B synchrotron FTIR microscope.

interest. Plans are underway for some collaborative technical developments.

Canadian scientists who have either traveled to NSLS or sent samples for analysis in the past year include Farid Benesebaa (ICPET-NRC), Marc Del Bigio (Pathology, U. of Manitoba), Ian Dixon (Institute for Cardiovascular Sciences, U. Manitoba), Kathleen Gough (Chemistry, U. of Manitoba), Bernhard Juurlink (College of Medicine, U. of Saskatchewan), Kirk Michaelian (NRCan), Eric Pellerin (IMI-NRC), Keith Robinson and Ken Schmidt (Alberta Synchrotron Institute, on behalf of several Alberta scientists), and David Westaway (Centre for Research in Neurodegenerative Diseases, U. of Toronto).

Further information on the U10B beamline can be found at http://nslsweb.nsls.bnl.gov/infrared/u10b. While the CLS membership in the PRT guarantees access for 20% of U10B beamtime, proposed experiments must still meet normal requirements: they must require synchrotron capability, be non-proprietary, and (if successful) ultimately be published in the open literature. To obtain access, interested persons should contact Neb Marinkovic and Kathy Gough:

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IR-1: Far IR Spectroscopy

The far IR research program comprises initiatives that will tackle important problems now outstanding. It includes a balance of specific and generic projects. Some of these are well-defined projects that are sure of success, such as internal rotor molecule studies at ultrahigh resolution and research using high-pressure diamond anvils. Others carry higher risk and greater uncertainties: examples include carbon chain molecules in supersonic jets and far IR probing of individual quantum dots.

Table 7·1 IR design team

Emil Hallin	Canadian Light Source
Tim May	Canadian Light Source
Mike Jackson	IBD, NRC (Winnipeg)
Farid Bensebaa	ICPET, NRC (Ottawa)
Bob McKellar	SIMS, NRC (Ottawa)
Kirk Michaelian	NRCan (Devon, AB)
Thomas Ellis	Université de Montréal
Wolfgang Jäger	University of Alberta
Kathy Gough	University of Manitoba
Peter Bernath	University of Waterloo

Principal researchers in each area are listed below:

- *Gas phase spectroscopy studies*
  —R. M. Lees, N. Moazzen-Ahmadi, I. Ozier, L.-H. Xu (internal rotation and intramolecular energy transfer)
  —W. Jäger, A. R. W. McKellar, N. Moazzen-Ahmadi, I. Ozier, G. C. Tabisz (weakly bound complexes and intermolecular forces)
  —A. Adam, R. L. Brooks, P. F. Bernath, D. W. Tokaryk (molecular ions and radicals)
  —P. F. Bernath, W. Jäger, N. Moazzen-Ahmadi, A. R. W. McKellar, D. W. Tokaryk (carbon chain molecules and laboratory astrophysics)
- *Very high pressure studies*
  —S. Desgreniers, D. Klug, J. Tse
- *Surface/interface studies*
  —T. Ellis, G. Lopinski, D. Roy (adsorbates and ultrathin organic films)
  —H. C. Liu, N. Rowell (characterization of microstructures and devices)

IR 2/3: Spectromicroscopy

The following paragraph is the full abstract of a paper published in the *Review of Scientific Instruments* in December of 2001. It describes recent research of Kirk Michaelian (NRCan) demonstrating that synchrotron sources offer significant advantages for photoacoustic IR experiments with medium to high spatial resolution.

The use of synchrotron radiation (SR) as a far infrared and mid-infrared source in the measurement of photoacoustic FTIR spectra of solids is demonstrated for the

Table 7·2 Far IR beamline team

On-site development team	
Emil Hallin	Canadian Light Source
Tim May	Canadian Light Source
Condensed phase spectroscopy	
H. C. Liu	IMS, NRC (Ottawa)
Nelson Rowell	INMS, NRC (Ottawa)
Dennis D. Klug	SIMS, NRC (Ottawa)
Greg P. Lopinski	SIMS, NRC (Ottawa)
John S. Tse	SIMS, NRC (Ottawa)
Thomas Ellis	Chimie, Université de Montréal
Gas phase spectroscopy	
A. Robert W. McKellar	SIMS, NRC (Ottawa)
Wolfgang Jäger	Chemistry, U. of Alberta
Irving Ozier	Physics and Astronomy, U. of British Columbia
Nasser Moazzen-Ahmadi	Physics and Astronomy, U. of Calgary
Robert L. Brooks	Physics, U. of Guelph
George C. Tabisz	Physics, U. of Manitoba
Allan G. Adam	Chemistry, U. of New Brunswick
Li-Hong Xu	Physics, U. of New Brunswick
Dennis W. Tokaryk	Physics, U. of New Brunswick
Ronald M. Lees	Physics, U. of New Brunswick
Peter F. Bernath	Chemistry, U. of Waterloo

first time in this work. Initial experiments were performed at beamline U10A at the National Synchrotron Light Source, Brookhaven National Laboratory. For synchrotron photoacoustic spectroscopy to be feasible, it must yield results superior to those obtained with a conventional thermal (globar) source; accordingly, SR and globar photoacoustic spectra recorded under similar conditions were compared in detail. The intensities of SR far infrared photoacoustic spectra were found to be consistently greater than the corresponding globar spectra. At shorter wavelengths, SR always underfills the effective aperture (or, alternatively, the sample size); SR is a superior source in a spectral region that is a function of this aperture. The high wave number limit of this region exhibits a power-law dependence on aperture size. This investigation also showed that the entire mid-infrared photoacoustic spectrum is more intense using SR and apertures smaller than approximately 0.5 mm.
[K. H. Michaelian; R. S. Jackson; C. C. Homes. Synchrotron infrared photoacoustic spectroscopy. *Rev. Sci. Instrum.* **2001,** *72*(12), 4331-4336.]

Table 7·3 Spectromicroscopy beamline team

On-site development team	
Emil Hallin	Canadian Light Source
Tim May	Canadian Light Source
Biological emphasis	
Mike Jackson	IBD, NRC (Winnipeg)
Thomas Ellis	Chimie, Université de Montréal
Kathy Gough	Chemistry, U. of Manitoba
Industrial emphasis	
Ken Schmidt	ASI, U. of Alberta
Kirk Michaelian	NRCan (Devon, AB)
Denis Masson	Nortel Networks (Ottawa)
Eric Pellerin	IMI, NRC (Boucherville, QC)
Farid Bensebaa	ICPET, NRC (Ottawa)
Ron Cavell	Chemistry, U. of Alberta

Table 7·4 Milestones in 2000 and 2001

February 2, 2000	Beamline design team meeting, SIMS, NRC (Ottawa)
March 13, 2000	Start of MOU; CLS becomes part of PRT at U10B (NSLS)
April 18, 2000	IR Beamlines reviewed by FAC
October 1, 2000	Beamline design team meeting, NRCan (Ottawa)
November 17, 2000	Infrared Spectroscopy and Microscopy Workshop, Saskatoon
May 14, 2001	Beamline design team meeting, IBD (Winnipeg)
May 22–24, 2001	NSLS Users' Meeting and Infrared Workshop, Brookhaven, NY
May 30, 2001	Symposium: Advances in Spectromicroscopy, 84th CSC Conference, Montreal

Design Team and Beamline Teams

The IR design team, the far IR beamline team (BT), the spectromicroscopy BT, and the beamline milestones are listed in Tables 7·1 through 7·4. The BT lists are not complete, but rather highlight those who have contributed in the past year. The goal is to make the teams as inclusive as possible.