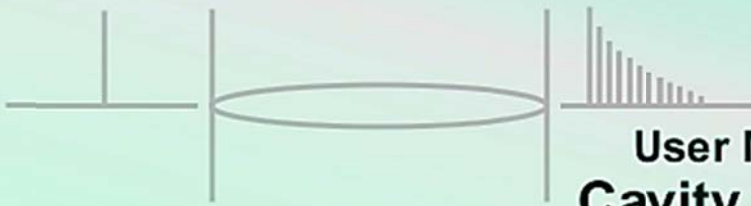


## Summer School and User Meeting Schedule

	Sun 26/6/2011	Mon 27/6/2011	Tue 28/6/2011	Wed 29/6/2011
9:00 AM		IS 1 Brown	IS 5 Axner	IS 8 Engeln
9:20 AM	first lecture			
9:40 AM	Kevin Lehmann	IS 2	IS 6	IS 9
10:00 AM	Chernoff Hall 202	Osthoff	Gagliardi	Ruth
10:20 AM	Break Chernoff Hall Atrium			
10:40 AM	Break	CS Nahler	CS Harren	CS Zhu
11:00 AM	second lecture	CS Grilli	CS Zeuner	CS Baharini
11:20 AM	Richard Engeln	IS3	IS 7	IS 10
11:40 AM	Chernoff Hall 202	Miller	Linnartz	Foltynowicz-Matyba
12:00 PM	Lunch	Lunch	Lunch	Lunch
12:20 PM	University Club	University Club	University Club	University Club
12:40 PM				
1:00 PM				
1:20 PM	third lecture	IS 4	Excursion	IS 11
1:40 PM	Gianluca Gagliardi	Mackenzie		Chow
2:00 PM	Chernoff Hall 202	CS Rushworth		IS 12
2:20 PM	Break	CS Didriche		Lehmann
2:40 PM	Break Chernoff Hall Atrium			Break
3:00 PM	forth lecture	CS McKeever		CS Aziz
3:20 PM	Hans-Peter Look	CS Chen		CS Munzke
3:40 PM	Chernoff Hall 202	CS Siller		CS St-Gelais
4:00 PM		Tiger Optics		
4:20 PM		Poster session		
4:40 PM		4th floor lounge		
5:00 PM		at Chernoff Hall		
5:20 PM				
5:40 PM				
6:00 PM	Reception at the	Banquet	All presentations are in room 117 in Chernoff Hall, 90 Bader Lane unless indicated otherwise (updated June 21 <sup>st</sup> , 2011)	
6:20 PM	Agnes Etherington	at Senior Staff Mess		
6:40 PM	Art Gallery	Royal Military College		
7:00 PM				
7:20 PM				
7:40 PM				



**User Meeting and Summer School  
Cavity Enhanced Spectroscopy  
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The logo for ICIP-É CIPIS, featuring a stylized red 'S' shape and a cluster of five colored dots (red, yellow, green, blue, purple) above the text.

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## 1.) Applications of Visible Cavity Enhanced Spectroscopy to Atmospheric Measurements

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Cavity enhanced absorption (or extinction) spectroscopy has seen increasing application as an analytical method in atmospheric science in recent years. This presentation will survey recent development of field instrumentation from our laboratories based on this technique, with an emphasis on the evolution of the technology and lessons learned from deployment on platforms such as ships, aircraft and a tall (300 m) tower. The first example is an instrument for the detection of a series of nitrogen oxides (NO, NO<sub>2</sub>, NO<sub>3</sub> and N<sub>2</sub>O<sub>5</sub>) and ozone (O<sub>3</sub>) by cavity ring-down spectroscopy. The nitrogen oxides, particularly NO<sub>3</sub> and N<sub>2</sub>O<sub>5</sub>, are trace species whose measurement has presented a significant analytical challenge in the past, while ozone represents an alternative to a standard measurement that may offer some advantages for precision and simplicity. This instrument originally utilized pulsed lasers, but has been demonstrated more recently with diode lasers for greater versatility in field environments and in the variety of species that it can detect. The second example is the measurement of glyoxal (CHOCHO) and nitrous acid (HONO) using broadband methods. These are trace species that have normally measured by spectroscopic methods over fixed, long open paths, but that are now accessible to detection using smaller, more mobile cavity enhanced instruments. The instrument was originally demonstrated using a Xe arc lamp source, but it currently uses light emitting diodes (LEDs) for greater versatility in the field.

## 2.) Quantification of nitrogen oxide reservoir species by blue diode laser cavity ring-down spectroscopy

**Hans D. Osthoff**<sup>\*</sup>, Dipayan Paul, Amanda Furgeson, Levi H. Mielke, and Robert D. Thaler

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The oxides of nitrogen are important tropospheric trace gases. For example, NO<sub>x</sub> (=NO+NO<sub>2</sub>) catalyzes the rate of photochemical ozone (O<sub>3</sub>) production, which is of interest to regional air quality and human health. In the troposphere, NO<sub>x</sub> has a lifetime of only a few days; its main sink is conversion to nitric acid (HNO<sub>3</sub>). NO<sub>x</sub> can be stored in so-called reservoir species, whose lifetimes are longer than that of NO<sub>x</sub> and whose decomposition, usually after long-range transport to remote regions, regenerates NO<sub>x</sub>. Important NO<sub>x</sub> reservoir species include peroxy-carboxylic nitric anhydrides (PANs, RC(O)O<sub>2</sub>NO<sub>2</sub>) and alkyl nitrates (ANs, RONO<sub>2</sub>), which are formed mainly during daytime, and the predominantly nocturnal species dinitrogen pentoxide (N<sub>2</sub>O<sub>5</sub>) and nitryl chloride (ClNO<sub>2</sub>). The atmospheric chemistry of PANs and N<sub>2</sub>O<sub>5</sub> in ambient air is relatively well established; in contrast, considerable uncertainty remains about the role of ANs and ClNO<sub>2</sub>, mainly due to a lack of analytical techniques to measure these species in ambient abundance.

Here, a novel method, thermal dissociation cavity ring-down spectroscopy (TD-CRDS), to quantify the mixing ratios of PANs, ANs, or ClNO<sub>2</sub> with tens of parts-per-trillion by volume (pptv) sensitivity is presented. Mixing ratios of the NO<sub>x</sub> reservoir species are determined by blue diode laser CRDS of NO<sub>2</sub>, produced from quantitative and selective thermal dissociation in a heated inlet, relative to NO<sub>2</sub> observed in an unheated reference channel. The TD-CRDS response was evaluated using parallel measurements of PANs and ClNO<sub>2</sub> by chemical ionization mass spectrometry (CIMS) and by NO<sub>y</sub> (= NO+NO<sub>2</sub>+HNO<sub>3</sub>+ΣPAN+ΣAN+2N<sub>2</sub>O<sub>5</sub>+ClNO<sub>2</sub>+...) chemiluminescence (CL). The PANs are fully dissociated at an inlet temperature of 250 °C and the response is noticeably non-linear at mixing ratios above 10 parts-per-billion by volume (ppbv) due to recombination of the TD fragments. For ClNO<sub>2</sub> and ANs, the linear dynamic range of the TD-CRDS extends from the detection limit of 20 pptv (1 σ, 1 min) to 30 ppbv, the highest mixing ratio tested. The TD profiles of ClNO<sub>2</sub> and ANs overlap, which has implications for measurements of ΣAN by thermal dissociation (with detection as NO<sub>2</sub>) in ambient air. Advantages, limitations, and applications of the TD-CRDS method are discussed.

### 3.) Cavity-enhanced Laser-Induced Fluorescence: CeLIF

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We have combined cavity ring-down spectroscopy (CRDS) and laser-induced fluorescence (LIF) in a single-beam experiment such that their respective advantages create a very powerful spectroscopic technique. The background-free LIF measurement delivers the ultra-high sensitivity and extends the dynamic range available to a stand-alone CRDS setup whereas the ring-down measurement provides the quantitative calibration of the LIF signal.

With CeLIF the signal-to-detection limit increases up to a factor of 100 in comparison to CRDS, demonstrated in fig. 1 for an absorbance  $\alpha$  in the range  $1\text{--}40 \cdot 10^{-5} \text{ m}^{-1}$ . With the improved detection sensitivity we successfully recorded gas-phase absorption spectra of 1,4-bis(phenylethynyl)benzene (BPEB) and 2,5-bis(phenylethynyl)thiophene seeded in molecular beams. The simultaneously recorded CeLIF and CRDS spectra of BPEB are shown in fig. 2; cf. [Greaves *et al.*, JPCA **110**, 2114 (2006)]. CeLIF greatly increases detection sensitivity over CRDS or LIF for fast fluorescing molecules (many organic molecules) in a confined absorption volume (molecular beam, surface).

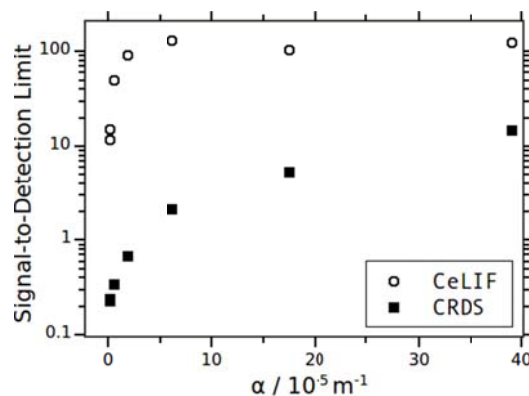


Fig 1: CeLIF increases signal-to-detection limit over CRDS.

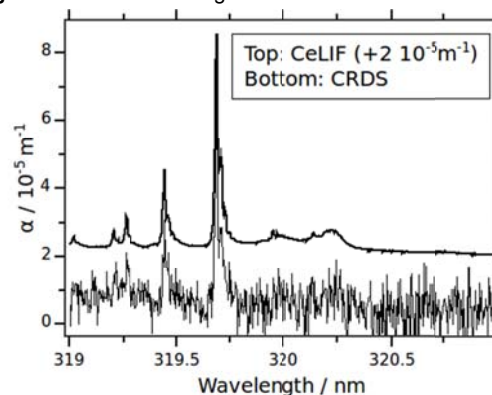


Fig 2: CeLIF and CRDS spectra of BPEB.

### 4.) Transportable ML-CEAS spectrometer for *in situ* measurement of BrO and IO radicals

Grilli Roberto, Chadi Abd-Alrahman, Samir Kassi, Guillaume Méjean, Irene Ventrillard, Daniele Romanini\*

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The detection of highly chemical reactive halogenated species, such as BrO and IO represents a challenge for analytical techniques due to the low lifetime and the low abundance of these molecules. Their ability to take part to oxidative processes, such as the oxidation of dimethyl sulphide (DMS), increases the interest of the atmospheric community to better understand the role of halogenated molecules, mainly in the Antarctica boundary layer where information from the polar ice cores, unique archive of climate proxies, could be retrieved.<sup>1</sup>

Detection of part per trillion (pptv,  $1:10^{12}$ ) levels of BrO and IO radicals can be performed by probing rotationally structured electronic transitions in the near-UV employing mode-locked cavity-enhanced absorption spectroscopy (ML-CEAS).<sup>2</sup> The spectrometer is composed by a broadly tunable, broadband frequency-doubled Ti:Sa mode-locked frequency comb laser injected into a high-finesse optical cavity and a high resolution spectrometer based on a high-order diffraction grating and a back-thinned CCD camera. The robust and compact spectrometer is contained in a  $120 \times 60$  cm breadboard, and by injecting alternatively two parallel high-finesse cavities detection of pptv levels of bromine and iodine oxide can be performed.

The performance of the spectrometer achieved so far corresponds to minimum detectable absorption coefficients of  $8 \times 10^{-10} \text{ cm}^{-1}$  in 10 s acquisition and  $1 \times 10^{-9} \text{ cm}^{-1}$  in 40 s of acquisition for IO and BrO, respectively, leading to a sensitivity of 1 and 1.7 pptv of IO and BrO at atmospheric pressure.<sup>3</sup>

<sup>1</sup> S. Preunkert, B. Jourdain, M. Legrand, R. Udisti, S. Becagli, O. Cerri, "Seasonality of sulfur species (dimethyl sulfide, sulfate, and methanesulfonate) in Antarctica: Inland versus coastal regions" J. Geophys. Res. 113, D15302 (2008).

<sup>2</sup> T. Gherman, D. Romanini, "Mode-locked cavity-enhanced absorption spectroscopy", Opt. Express 10, 1033 (2002).

<sup>3</sup> G. Méjean, S. Kassi, and D. Romanini, "Cavity enhanced absorption spectroscopy with an optical comb: detection of atmospheric radicals in the near UV", Optics Letters 33, 1231 (2008).

## 5.) Using Cavity Ringdown Spectroscopy to Obtain the Spectra of Free Radicals That Affect the Quality of Your Life

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Few things affect your quality of life more than the air you breathe and the temperature of your immediate environment. Since more than 80% of the energy used in North America today is still derived from fossil fuels, these two quantities are not unrelated. Annually more than 100 Tg of organic molecules are injected into the troposphere and mostly degraded via oxidative processes involving free radical intermediates. Many of those intermediates are the same as the ones involved in the combustion of fossil fuels. Two of the key oxidizing intermediates are hydroxyl, OH, (day) and nitrate, NO<sub>3</sub>, (night) radicals and early oxidation intermediates of organic compounds include the alkoxy (RO) and peroxy (RO<sub>2</sub>) families of radicals. The spectroscopy of OH is well known, but there is still a lot to be learned about the spectroscopy of RO, RO<sub>2</sub>, and NO<sub>3</sub> radicals both for diagnostic purposes and in terms of characterizing their molecular properties and benchmarking quantum chemistry calculations.

Cavity ringdown spectroscopy (CRDS) of these free radical intermediates provides diagnostics for the detection and monitoring of these molecules, allowing the determination of species concentrations, reaction mechanisms, and rate constants in the laboratory and also in real-world chemistry. To obtain such spectra, we produce the radicals within the optical cavity of a CRDS apparatus. Currently we have three separate CRDS apparatuses, all of which typically operate in the IR. One is an ambient temperature, moderate resolution ( $\approx 0.1 \text{ cm}^{-1}$ ) system which provides survey scans of vibronic spectra. A second system provides free-jet cooling with high resolution ( $\leq 100 \text{ MHz}$ ) and yields rotationally resolved spectra. A third system is ambient temperature but has two optical cavities allowing dual wavelength (2 $\lambda$ -CRDS) spectroscopy. A few of the spectra obtained with these CRDS systems will be discussed.

**Monday, June 27<sup>th</sup>, 1:20 pm**

## 6.) Applications of broadband cavity-based techniques to kinetic studies in the condensed phase

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I will report on recent work both within our group and in collaboration with others, in which we explore the possibilities and limitations of cavity-based techniques at interfaces and in the bulk liquid phase. To date, much of our work has used simple evanescent wave variants of cavity ring-down spectroscopy (CRDS) and cavity enhanced absorption spectroscopy (CEAS) to study dynamical interfacial phenomena such as adsorption, redox dissolution of immobilized nanoparticles, nanostructured material growth, ordering at liquid|air interfaces and DNA-induced desorption.<sup>1</sup> In these studies we have typically sacrificed absolute sensitivity and spectral information (*i.e.*, tuneability) for high repetition rates which we achieve by the use of cheap, modulated broadband diode lasers in the visible region of the spectrum. In solution this permits the study of diffusion-controlled reactions in real time.

Recently, we have begun to incorporate supercontinuum sources into our CEAS experiments. Together with broadband (400-800 nm) coated cavity mirrors, this provides ultrabroadband spectral coverage with high sensitivity and fast acquisition times. In this way it is possible to record the entire visible spectrum of an evanescent field region in  $< 10 \mu\text{s}$ .<sup>2</sup> One major focus of our current work is the application of CRDS and supercontinuum CEAS techniques to the study of magnetic field effects in the reaction of spin-correlated radical pairs in solution.<sup>3</sup> The aim is to identify the chemical basis underlying the magneto-reception mechanism in animals, especially birds, and is part of a wider program to illustrate inherently quantum effects in biological systems.

<sup>1</sup> M. Schnippering, S. R. T. Neil, S. R. Mackenzie, and P. R. Unwin, *Chemical Society Reviews* **40**, 207 (2011).

<sup>2</sup> L. van der Sneppen, G. Hancock, C. Kaminski, T. Laurila, S. R. Mackenzie, S. R. T. Neil, R. Peverall, G. A. D. Ritchie, M. Schnippering, and P. R. Unwin, *Analyst* **135**, 133 (2010).

<sup>3</sup> S. R. T. Neil, K. Maeda, K. B. Henbest, M. Goez, R. Hemmens, C. R. Timmel, and S. R. Mackenzie, *Mol. Phys.* **108**, 993 (2010).

## 7.) Microfluidic applications of two-mirror and fibre-loop cavity ring-down spectroscopy

**Cathy Rushworth, Dean James, Bobby Oag and Claire Vallance\***

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There is a growing need for the sensitive analysis of small amounts of liquids, especially in the area of microfluidics, for example in lab-on-a-chip or micro-total-analysis-systems ( $\mu$ TAS). Cavity ringdown spectroscopy (CRDS) holds considerable promise as a technique for the analysis of such samples. We are currently investigating the integration of microfluidic chips into both two-mirror and fibre-loop based cavities, enabling CRDS measurements on pL – nL liquid sample volumes.

Microfluidic chips with channel thicknesses ranging from 30 to 100  $\mu$ m have been fabricated in-house and inserted directly into a two-mirror cavity at Brewster's angle. Measurements on aqueous Rhodamine 6G dye indicate a detection limit of around 2  $\mu$ mol dm<sup>-3</sup>, corresponding to a detection sensitivity of 0.2 cm<sup>-1</sup>. The same experimental arrangement has also been used to demonstrate pH sensing, through measurements on millimolar concentrations of phenolphthalein in a series of buffered solutions, and reaction kinetics monitoring, using the iodine clock reaction as a test system.

In the fibre-loop cavity approach, both 50  $\mu$ m core and 105  $\mu$ m core diameter fibres have been characterised. We have investigated the absorption of Rhodamine 6G in both direct absorption mode, in which the sample is introduced between the two separated fibre ends, and evanescent wave absorption mode, in which the sample is introduced to an area of optical fibre from which the cladding has been removed. Currently, the evanescent wave approach has a slightly better detection limit than direct absorption (10  $\mu$ mol dm<sup>-3</sup> compared with 50  $\mu$ mol dm<sup>-3</sup>) owing to the restricted pathlengths achievable when separating small-core optical fibre ends.

While the sensitivity of two-mirror CRDS is currently higher than that achievable in fibre-loop CRDS, we have recently developed a novel, high-efficiency, low-loss light coupler for fibre loop CRDS, which should allow us to improve the sensitivity of our fibre-loop CRDS by up to two orders of magnitude. The new coupling scheme allows virtually 100% of the excitation light to enter the cavity, while maintaining a low loss on each pass.

## 8.) FANTASIO+: a versatile experimental set-up to investigate molecules and molecular complexes of atmospheric and astrophysical relevance

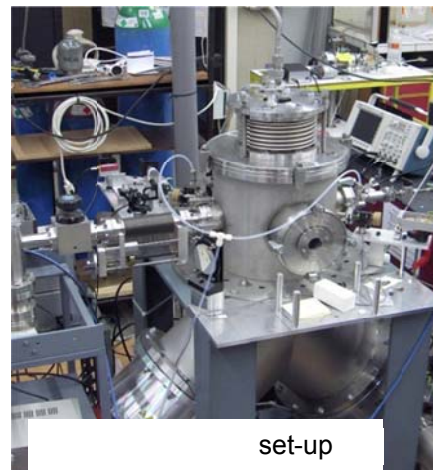
**K. Didriche\*, C. Lauzin, T. Földes, X. de Ghellinck and M. Herman**

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We have built the FANTASIO+ set-up<sup>a</sup> (for “Fourier trANsform, Tunable diode and quadrupole mAss spectrometers interfaced to a Supersonic expansIOn”). The supersonic expansion is produced by two large turbomolecular pumps allowing the gaseous samples to be cooled down to a temperature of 3 K and molecular van der Waals complexes to be formed. They are probed around 1.5  $\mu$ m by a sub-Doppler resolution CW-CRDS with a 130  $\mu$ s ring down time. The performances will be illustrated (including the first observation of the van der Waals complex: C<sub>2</sub>H<sub>2</sub>-Kr) and current updates (including pulsed and liquid samples injectors) will be presented.

A complementary probe technique is being developed, tunable from 3000 to 9000 cm<sup>-1</sup>, coupling an optical parametric oscillator (OPO), pumped by a Ti:Sa femtosecond laser to a high finesse cavity and, to a high resolution continuous scan Fourier transform interferometer. The first CEAS results will be presented.



<sup>a</sup>Didriche *et al.*, **Mol. Phys.** 2010, 108, 2158-2164

## 9.) High Sensitivity and Noise Rejection for Trace Gas Sensing via Sideband-Locked Dual-Cavity Detection

**Jason McKeever\***, Anthony E. Miller, C. Ricardo Viteri, John K. Stockton, Ari K. Tuchman

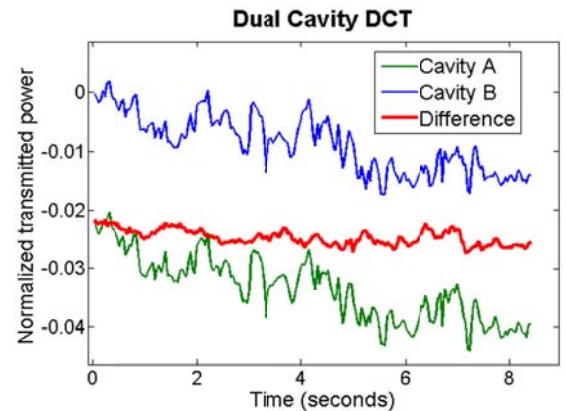
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[\\*jmckeever@entanglementtech.com](mailto:*jmckeever@entanglementtech.com)

Motivated by the need for robust, selective and sensitive trace gas detection capable of field-deployment, we report on development of a compact sensor consisting of two optical cavities built into the same spacer block. We have made use of a versatile, high-bandwidth locking scheme using a fiber-coupled electro-optic modulator. We use first-order sidebands as the cavity probe fields, and we feed-back to the frequency of these sidebands to maintain resonance. The high speed and low required drive voltages of this waveguide-based modulator could be exploited to stabilize any laser with fast frequency fluctuations without relying on direct laser feedback.

Our sensor, operating at a wavelength of 1064 nm, has been characterized in two configurations – cavity ring down (CRD) and direct cavity transmission (DCT). In CRD mode, we have achieved an empty cavity sensitivity of  $1.4 \times 10^{-12} \text{ cm}^{-1} \text{ Hz}^{-1/2}$  for a single cavity of length 9.5 cm, finesse  $1.8 \times 10^5$ , peak transmitted power of 0.1 mW and repetition rate of 5 kHz. For single-cavity DCT, we have reached sensitivities below  $10^{-12} \text{ cm}^{-1} \text{ Hz}^{-1/2}$  at frequencies higher than 60 Hz. For longer averaging times, by exploiting correlations between intensity fluctuations at the two cavity outputs, we have achieved noise rejection of 12 dB, bringing us to the  $10^{-10} \text{ cm}^{-1}$  level with 20 Hz bandwidth (see Fig. 1).

We also discuss our progress in developing a second generation system at a wavelength of 2.3 microns where molecular transitions are much stronger for a range of potential analytes.



## 10.) Advances in Trace Gas Analysis in Industrial Applications Using Continuous-wave Cavity Ring-Down Spectroscopy

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Following the successful and pioneering development of commercial analyzers for sub parts-per-billion (ppb) level detection of moisture in inert gases, Tiger Optics has extended the advantages of Continuous-Wave Cavity Ring-Down Spectroscopy (CW-CRDS) to a series of challenging, real-world industrial applications. With particular emphasis on field data, we will demonstrate the strong capability of CW-CRDS for trace species detection in such important gas monitoring applications as moisture analysis in challenging hydride gas matrices, as well as oxygen monitoring for ultra-high-purity (UHP) bulk gases. In other industrial applications, species such as ammonia, hydrogen fluoride, and hydrogen chloride, some with very “tricky” surface chemistry, serve as critical indicators for process analytical control under widely varying process conditions. In addition, Tiger’s CW-CRDS has also been adopted by over a dozen major national metrology institutes, where it often serves as a transfer standard. With a variety of flexible configurations, CW-CRDS-based instruments address these various applications with a self-verifying measurement solution that is fast and sensitive, yet extremely robust and simple to operate.

## 11.) Cavity Enhanced Velocity Modulation Spectroscopy

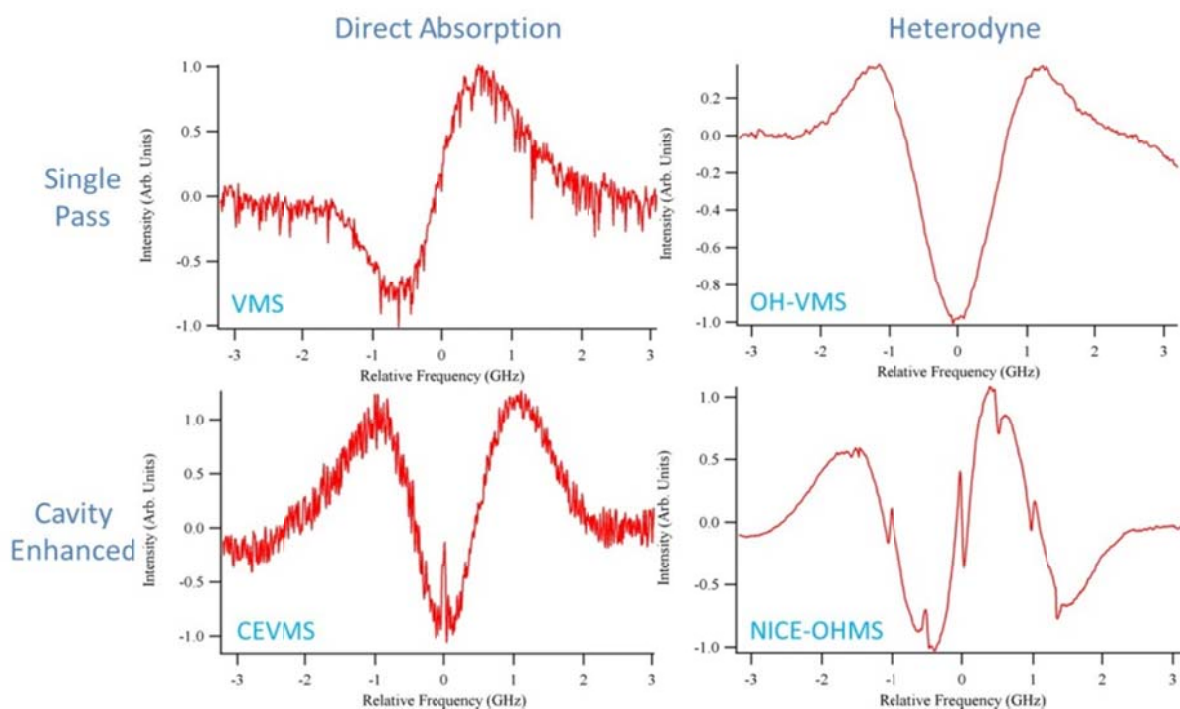
**Brian Siller, Andrew Mills, Michael Porambo and Benjamin McCall\***

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Spectroscopy of molecular ions is of interest to a variety of fields, but laboratory studies tend to be difficult because in the plasmas used to study ions, interfering neutral molecules tend to be orders of magnitude more abundant. The technique of velocity modulation has been used successfully to study dozens of molecular ions over the past several decades because it allows the isolation of ionic absorption signals from those of neutral molecules. When a voltage is applied across a discharge cell, cations in the resulting plasma are accelerated toward the cathode, inducing a Doppler shift in their absorption profiles. By applying an AC voltage and demodulating the absorption signal with a lock-in amplifier, the signals from ions whose velocity is being modulated can be detected independent of any neutral absorption signals that are not modulated. Until recently, the most sensitive velocity modulation instruments have used unidirectional multipass cells with up to ~8 passes, combined with heterodyne detection for noise suppression.

We have improved upon this technique by combining it with cavity enhanced absorption spectroscopy. This allows for greatly increased path length of the laser through the plasma, while simultaneously enabling sub-Doppler spectroscopy through the observation of Lamb dips. Even further improvement in sensitivity has been attained by combining velocity modulation with NICE-OHMS. This allows for the simultaneous advantages of long path length, low noise level, and sub-Doppler resolution, while still retaining ion-neutral discrimination. Initial proof of concept spectroscopy was done on  $N_2^+$  with a Titanium Sapphire laser and cavity finesse ~300.



## 12.) Noise-Immune Cavity-Enhanced Optical Heterodyne Molecular Spectroscopy (NICE-OHMS): A Laser-based Cavity Enhanced Spectroscopic Technique for Sensitive Detection of Gases

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Noise-Immune Cavity-Enhanced Optical Heterodyne Molecular Spectroscopy (NICE-OHMS) is a laser-based spectroscopic technique developed for high precision frequency standard applications at JILA in Boulder, CO, USA. The technique provides ultrahigh absorption sensitivity by combining an external cavity for increased interaction length with the sample with frequency modulation for reduced influence of noise. In addition, both Doppler-broadened and Doppler-free signals can be measured. In its first realization, based on a well stabilized solid state laser, an impressive absorption sensitivity of  $10^{-14} \text{ cm}^{-1}$  was demonstrated for molecular species detection.<sup>1</sup> This is several orders of magnitude better than any other optical detection technique can achieve, which gives NICE-OHMS a large potential for sensitive trace gas analysis.

Our research group is developing the NICE-OHMS technique further, with the main aim of reducing the complexity of the instrumentation and thereby making the technique more attractive for trace gas detection.<sup>2</sup> The systems developed so far are based upon narrowband fiber lasers, as well as a narrowband DFB laser, and have, whenever possible, utilized fiber-coupled components for a more compact realization. Using  $\text{C}_2\text{H}_2$  as the pilot species, we have scrutinized the dependence of the technique on a variety of parameters for optimum sensitivity, selectivity, and thereby its applicability. Using a cavity with a finesse of 5700, we have presently demonstrated an absorption sensitivity of  $2 \times 10^{-11} \text{ cm}^{-1} \text{ Hz}^{-1/2}$ , which allows detection of low ppt concentrations of acetylene in atmospheric samples. Means to improve on this figure of merit into the  $10^{-12} \text{ cm}^{-1} \text{ Hz}^{-1/2}$  range is under way. The talk will provide a short description of the basic features of the technique and some of the most important results.

<sup>1</sup>J. Ye, L. S. Ma, and J. Hall, "Ultrasensitive detection in atomic and molecular physics: demonstration in...", J. Opt. Soc. Am. B **15**, 6-15 (1998).

<sup>2</sup>A. Foltynowicz, F. M. Schmidt, W. Ma, and O. Axner, "NICE-OHMS: Current status and future potential", Appl. Phys. B **92**, 313-326 (2008).

## 13.) Dielectric optical resonators for mechanical and chemical sensing

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Over the past several years, fiber-optic resonators have been used as mechanical probes by virtue of their intrinsic sensitivity to length changes<sup>1</sup> as well as chemical sensors<sup>2</sup> based on direct absorption or evanescent-wave spectroscopy. In a recent work, we devised a diode-laser system for strain interrogation of a high-finesse fiber Bragg-grating cavity, achieving a  $10^{-13}$  resolution in the infrasonic and acoustic frequency ranges, thanks to the exceptional stability of a fiber-based optical frequency comb (OFC)<sup>3</sup>. OFC synthesizers are also very unique as coherent-light sources for cavity-enhanced spectroscopy<sup>4</sup>. Their emission spectrum covers a wide wavelength range that is well suited to broad-band absorption or Fourier-transform spectroscopy<sup>5</sup>. Here we report our recently-developed methods for liquid chemical sensing in dielectric optical resonators. Different systems, relying either on high-finesse fiber-optic cavities or spherical resonators are illustrated. Pound-Drever-Hall (PDH) frequency locking techniques are implemented for low-noise and wide dynamic range readout of the sensors. Cavity ring-down spectroscopy in an optical-fiber ring is performed by an OFC synthesizer. The OFC teeth spacing and the cavity free-spectral-range are frequency matched by a PDH loop acting on the comb repetition rate and the fiber cavity length. The interaction with chemical species occurs when the evanescent field is exposed along the cavity-medium interface, thereby reducing the internal photon lifetime. Fast amplitude modulation and phase-shift measurements are carried out to provide the absorption-induced cavity loss. Preliminary results about detection of amine-group compounds are shown. Also, we describe experiments in progress on liquid-drop

spherical microresonators. Free-space laser beam excitation of whispering-gallery modes in oil droplets with characterization of the observed spectrum is presented. Future developments on cavity-enhanced spectroscopy in the near-infrared are discussed.

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#### **14.) Real-time breath monitoring using OPO-based Off-Axis Integrated Cavity Output Spectroscopy**

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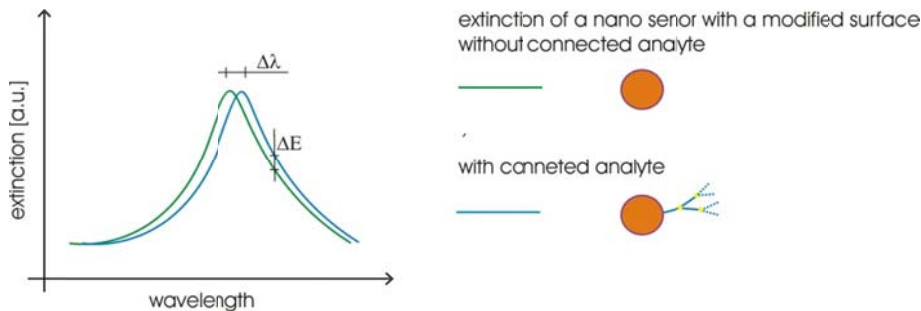
Trace gases are distinctive indicators of biological processes occurring in plants, animals and humans. An excellent method for highly sensitive and selective detection of these volatile organic compounds is mid-infrared laser spectroscopy, where many molecules have strong distinctive absorption patterns. To capture the dynamics of the gas concentrations and detect multi-component gas samples we use a fast scanning near infrared diode laser with a mode-hop-free wavelength coverage tuning range (up to  $5 \text{ cm}^{-1}$ ) at a speed of up to 100 THz/s. This tunable diode laser light (amplified in a fiber amplifier) is converted to the mid-infrared wavelength region with a single resonant Optical Parametric Oscillator (OPO). The OPO is used as a passive converter and has, as such, several advantages in comparison other sources of coherent radiation in the mid-IR region, such as continuous tuning coverage between 2.5 - 4  $\mu\text{m}$ , a narrow linewidth (100 MHz) and a cw power of 1.2 W. In combination with Off Axis Integrated Cavity Output Spectroscopy, these advantages are used to build a rapid and sensitive trace gas detector for gases such as ethane, methane, acetone at the sub-ppbv level concentrations at sub-second time scale. The fast subsecond response time of the system is able to show quantitatively methane, water,  $\text{CO}_2$  and ethane ( $\text{C}_2\text{H}_6$ , at the low part per billion levels) in single exhalations of human breath.

#### **15.) Read-out of a nanoparticle based sensor system by Cavity Ring-Down spectroscopy**

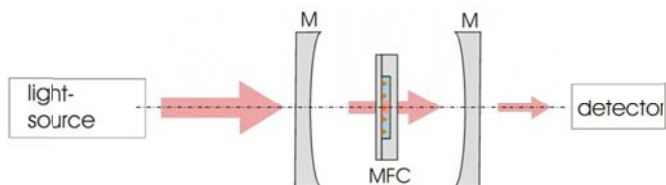
**Thomas Zeuner, Wolfgang Paa, Christian Mühlig, Gabriele Schmidl, Thomas Henkel, Andrea Csaki**

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Metallic sensor particles with a dimension of a few tenth nanometers could be used for selective detection of analytes in fluids. The metallic nanoparticles show typical optic properties caused by their plasmon resonances. This leads to a characteristic extinction spectrum which is determined by material, size, shape and the local environment (especially the refractive index) of the particle. The surfaces of the particles can be modified to exhibit a high and selective affinity for special analyte molecules. The binding of an analyte molecule to a modified nanoparticle leads to a local change of its environment and an altered extinction spectrum of the nanoparticle sensor (figure 1). For nano sensors in fluids with a high concentration of analyte molecules this effect can be observed by a commercial two-beam spectrometer. In our setup less than a monolayer of the nanoparticles are located on a surface which is included in a microfluidic chip. This microfluidic chip with the located nano sensors is placed in a Cavity Ring-Down (CRD) setup to increase the measurement sensitivity for the small extinction changes in the case of only tiny concentrations (figure 2). Two different kinds of CRD setups were realized: One setup uses a diode laser and a photo multiplier for detection the extinction changes at only a single wavelength. The second one incorporates a super continuum laser as an excitation source and a spectrometer for parallel detection of changes (amplitude and/or shift) in the extinction spectrum of the nanosensors. In first measurements we optimized the setup and the sensor concentration to the maximum of sensitivity of the setup. We show that is possible to measure the extinction spectrum of a microfluidic chip with a super continuum CRD setup. First results which demonstrate the application as a sensor using a test analyte will be shown.



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## 16.) Cavity Enhanced Techniques: Applications in Astrochemical Laboratory Research

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The conditions in space are extreme and definitely not in favor of chemical processes; temperatures are low, radiation fields are intense and particle densities are exceedingly small. Nevertheless, more than 150 different molecular species have been identified in star-forming regions. These comprise both small and complex species as well as stable and transient molecules and are the result of a largely unexplored chemical evolution. Today, astrochemists explain the chemical complexity as the cumulative outcome of gas, grain and gas-grain interactions. Gas phase models explain the observed abundance of exotic molecules such as the linear carbon chain radical  $\text{HC}_{13}\text{N}$ , but it is not possible to explain the presence of e.g. acetonitrile, a precursor molecule for the simplest amino-acid glycine. Exciting new evidence has now been found that icy dust grains act as catalytic sites for molecule formation. Thermal and vacuum-ultraviolet processing as well as atom-bombardment of inter- and circumstellar ices trigger a fascinating solid state chemistry.

This talk will review how cavity enhanced techniques are used to identify molecules and molecular reaction networks in space, covering different frequency regimes and using both regular cavity ring-down techniques and broadband methods. Applications vary from simulated interstellar clouds, using supersonic plasma expansions to study radical species, to circumstellar ice analogues, reflecting reactive processes on icy grains at low temperatures.

## 17.) Plasma-surface interactions studied with CRD spectroscopy

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Plasma is used in industrial applications ranging from the deposition of thin layers for solar cells to diamond-like layers for the hardening of tools, and from cleaning of air to etching of wafers in IC-technology. All applications of these man-made plasma have one effect in common: the interaction of plasma with a surface.

During my presentation I will discuss examples of the study of the interaction of a plasma created from mixtures of nitrogen and hydrogen with a surface leading to ammonia production and the deposition of amorphous silicon by means of a SiH<sub>4</sub>-containing Ar/H<sub>2</sub> plasma. In the first study a cw-CRD technique was used, which allowed for fast parameter studies of the ammonia production. For example, the NH<sub>3</sub> production was determined as function of the ratio of N<sub>2</sub> and H<sub>2</sub> in the gas mixture and for different pressures in the plasma vessel. It turned out that indeed ammonia is produced at the surface of the vessel and that, independent of pressure (between 10 and 200 Pa), at optimal condition 12 % of the gasses inside the plasma vessel is ammonia [1]. In the second study the pulsed nature of the CRD technique was explicitly used to investigate the growth processes during the plasma deposition of a thin layer [2]. Often the surface reaction probability  $\beta$  of the species has been obtained indirectly or under process conditions different from the actual plasma deposition process. With time-resolved cavity ring down ( $\tau$ -CRD)  $\beta$  is obtained *during* plasma deposition: the CRD method is used to map an increased radical density due to a pulsed rf bias to the substrate in addition to a continuously operated remote SiH<sub>4</sub> plasma [2]. This yields simultaneously information on the surface reaction probability  $\beta$  and the density of the radicals under specific plasma conditions.

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## 18.) Incoherent broadband cavity-enhanced evanescent wave spectroscopy of surface-adsorbed biomolecules

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An improvement of conventional attenuated total reflection spectroscopy is demonstrated by applying an incoherent broadband light source (short-arc Xe-lamp or supercontinuum source) in a cavity-enhanced evanescent-wave absorption setup. Two different experimental designs enable the measurement of time dependent absorption spectra of either solution films or surface species (or the transition between both). The measurement principle will be illustrated on basis of the evaporation process of a solvent on a fused silica surface.

With this approach the time dependent absorption spectra of several biomolecules have been measured, the following examples will be presented:

(A) Palladium- (PdOEP), Platinum- (PtOEP) and Zinc-octaethyl porphyrins (ZnOEP) were studied in thin acetone solution films and on fused silica between 390 and 625 nm. The maximum sensitivity of the corresponding setup was estimated at approximately  $2 \times 10^{-5}$  per pass, which translates into a minimal detectable surface density of less than  $2 \times 10^{-3}$  monolayers for the porphyrins studied (based on absorption in the strong Soret bands around 400 nm). Changes of surface and solution spectra are characterised and discussed on basis of observed band broadenings and spectral shifts.

(B) Chlorophyll A and B were studied between 550 and 700 nm with regard to their photo degradation properties. Time dependent spectra will be shown and potential interpretations briefly discussed.

## 19.) Investigation of the Photolysis of Nitric Acid on Various Surfaces by Using Cavity Ring-Down Spectroscopy and Its Novel Variant

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Nitric acid ( $\text{HNO}_3$ ) is a major atmospheric oxidation product of  $\text{NO}_x$ . Although its gas-phase photolysis in the troposphere is slow, the photolysis rate for  $\text{HNO}_3$  deposited on ground and vegetation surfaces to form HONO and  $\text{NO}_x$  has been reported to be 1-2 orders of magnitude faster than that in the gas phase. The photolysis of  $\text{HNO}_3$  adsorbed on ground surfaces has been proposed as a major daytime source of HONO in low- $\text{NO}_x$  environments. To understand the difference between the nitric acid photolysis rate in the gas phase and the rate of photolysis on surfaces, my group have determined the UV absorption cross sections of  $\text{HNO}_3$  adsorbed on fused silica surfaces in the 290-365 nm region with a novel variant of cavity ring-down technique. Our study showed that the surface absorption cross sections of  $\text{HNO}_3$  are at least two orders of magnitude higher than the cross section values of the nitric acid vapor, in the wavelength region studied. We also directly measured the 308 nm absorption cross sections of  $\text{HNO}_3$  on Al surfaces and on ice films with a complementary technique, and compared the  $\text{HNO}_3$  cross section values on various surfaces. My group have investigated the photolysis of nitric acid in the gas phase, on aluminum surfaces, on ice films, and on fused silica surfaces, by using excimer laser photolysis combined with cavity ring-down spectroscopy or with its novel variant. We monitored both the ground state  $\text{NO}_2$  and the electronically-excited  $\text{NO}_2$ ,  $\text{NO}_2^*$ , produced from the  $\text{HNO}_3$  photolysis.  $\text{NO}_2^* + \text{OH}$  is a predominant photolysis pathway (if not the only photolysis pathway) from the gas phase photolysis of  $\text{HNO}_3$  at 308 nm. We have determined the  $\text{NO}_2^*$  quantum yields from the  $\text{HNO}_3$  photolysis on aluminum surfaces, on ice films, and on fused silica surfaces, and examined the dependence of the photolysis quantum yields on surface types.

## 20.) Using cw-CRDS to probe some products obtained during hydrocarbon oxidation in a jet-stirred reactor (JSR)

**Bahrini Chiheb\*, Herbinet Olivier, Alexandre Pierre, Battin-Leclerc Frédérique**

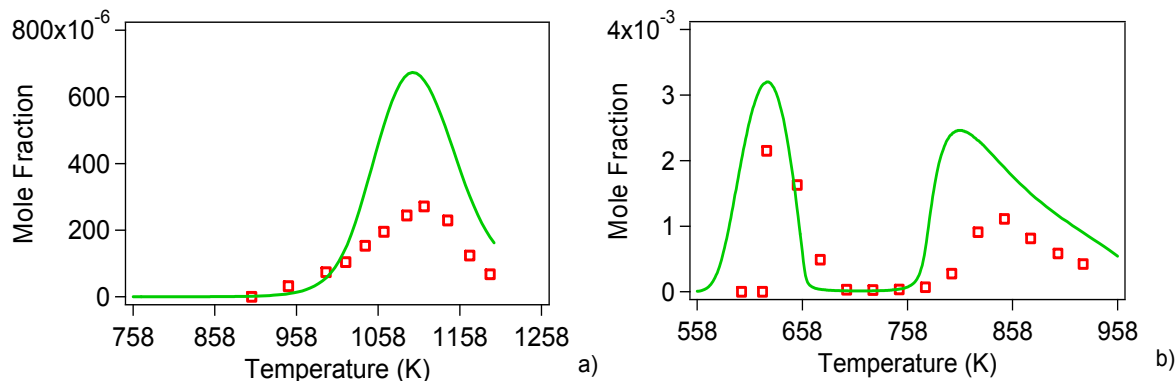
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Experimental studies of the oxidation of hydrocarbons in apparatuses such as jet-stirred reactors (JSR) provide useful data for the validation of detailed kinetic models and for the quantification of the impact of transport on the atmosphere. These data are useful for the development of more efficient and cleaner motors. Different techniques have already been used to analyze the oxidation products: i) gas chromatography, ii) mass spectrometry with electron impact ionization (EI-MS), iii) Reflectron time-of-flight mass spectrometry (RTOF-MS) with photoionization by tunable VUV radiation obtained using a synchrotron. The latter technique allowed the quantification of unstable species such as alkylhydroperoxides and ketohydroperoxides [1,2].

We present here the preliminary results obtained during the oxidation of methane and n-butane in a JSR using, for the first time, a cw-CRDS setup coupled to the reactor to detect oxidation products. The high sensitivity of this absorption technique allowed the quantification of some stable species:  $\text{H}_2\text{O}$ ,  $\text{CH}_4$  and  $\text{CH}_2\text{O}$ , whose IR spectra around 1506 nm are well structured, and are well known, particularly for  $\text{CH}_4$ , for which the region 1.48 – 1.63  $\mu\text{m}$  was re-investigated recently by the same cw-CRDS technique [3], and  $\text{CH}_2\text{O}$ , for which the region 1.53 – 1.47  $\mu\text{m}$  was recorded with the Cavity-Enhanced Absorption Spectroscopy technique (CEAS) with a resolution of  $0.001 \text{ cm}^{-1}$  [4].

The next step of our project will be dedicated to the detection, using the same cw-CRD technique, of radical species such as  $\text{HO}_2$ , which also has a well-structured IR spectrum in the region 1.49 – 1.51  $\mu\text{m}$  [5]. The radical  $\text{HO}_2$  is a very important intermediate in the combustion chemistry of hydrocarbons. The quantification of this radical will provide useful information for a better understanding of the chemistry occurring in combustion systems. These data will be used to test and refine kinetic models.

The detection limit of the cw-CRDS technique is about  $10^9 \sim 10^{10}$  molecules. $\text{cm}^{-3}$  for  $\text{HO}_2$ . Kinetic models predict a concentration of about  $10^{12}$  molecules. $\text{cm}^{-3}$  (few ppm) of  $\text{HO}_2$  in the reactor at a temperature of  $\sim 1100$  K. The lifetime of radicals in the reactor is very sensitive to recombination reactions at the wall of the reactor, leading to radical loss. To reduce this loss, better suited reactor with a surface treatment by a thin layer of inert and hydrophobic Si will be used later in this study.



Experimental (symbols) and computed (line) mole fraction profiles of formaldehyde. a) Oxidation of methane. b) Oxidation of n-butane.

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## 21.) Cavity-enhanced optical frequency comb spectroscopy

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Optical frequency combs are produced by mode-locked lasers, whose spectrum spans hundreds of terahertz of bandwidth and consists of a series of equidistant narrow lines with frequencies given by  $\nu_n = n f_{rep} + f_{ceo}$ , where  $f_{rep}$  is the repetition rate of the laser and  $f_{ceo}$  is the carrier envelope offset frequency. The two radio frequencies can be directly measured and adjusted, providing full control over each comb line and making the frequency combs an excellent tool for precision metrology. In spectroscopy, the frequency combs can be used as frequency reference for measurements with continuous wave lasers, or, more powerfully, to perform the measurements directly. Direct frequency comb spectroscopy (DFCS), which is virtually equivalent to a simultaneous measurement with tens of thousands of narrow laser lines, combines the large spectral bandwidth of the pulsed laser with the high resolution provided by individual comb lines, allowing simultaneous and unambiguous detection of many molecular trace species. Moreover, the equidistant spectrum of the comb allows it to be efficiently coupled into an external high-finesse optical cavity, providing significant enhancement of the interaction length with the sample. Due to the dispersion in the cavity mirrors, the free spectral range of the cavity varies as a function of optical frequency, which usually prevents simultaneous coupling of the entire comb spectrum into the cavity. Another issue is the laser frequency-to-amplitude noise conversion by the narrow cavity modes, which plagues all cavity-enhanced techniques. Various detection methods and comb-to-cavity coupling schemes have been developed to overcome these two limitations, allowing cavity-enhanced (CE-) DFCS to reach sensitivities in the  $10^{-10}$   $\text{cm}^{-1} \text{Hz}^{-1/2}$  range<sup>1</sup>. With the combined advantages of high sensitivity, broad spectral bandwidth, high resolution, and fast acquisition times, CE-DFCS has the potential to become the ultimate tool for applications such as precision spectroscopy and trace gas detection for breath analysis or atmospheric research<sup>2</sup>.

<sup>1</sup> M. J. Thorpe and J. Ye, *Appl. Phys. B* 91 (3-4), 397 (2008).

<sup>2</sup> F. Adler, M. J. Thorpe, K. C. Cossel, and J. Ye, in *Annual Review Of Analytical Chemistry*, Vol 3, pp. 175-205 (2010).

22.) To be Announced

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23.) Sensitivity Limits for Cavity Ring-down and Related Spectroscopic Techniques.

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In this talk, we shall discuss theoretical sensitivity limits for different forms of Cavity Ring-Down Spectroscopy and related techniques, such as phase-shift CRDS, swept frequency CRDS, and Cavity Enhanced Absorption Spectroscopy. We will also discuss our experimental work to improve cw-CRDS sensitivity by attention to a number of issues, such as the methods use to fit data and eliminate outliers, control of the cavity Fresnel number, and the extinction coefficient of the intensity modulator used to extinguish the field into the cavity.

24.) Development and application of an automated near IR cavity ring-down spectrometer (CRDS) combined with sample preconcentration, for continuous measurement of ethene (C<sub>2</sub>H<sub>4</sub>) in Bristol air

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CRDS in the near IR region is greatly facilitated by the availability of lasers, relevant optics and detectors. The need to use weaker ro-vibrational spectroscopic lines in this region, however, means it is still challenging to achieve adequate sensitivity to reach the detection limits required to monitor the very low abundance of molecules such as ethene in ambient air. As an associated technique to improve the detection limit of CRDS, sample pre-concentration has therefore been developed and used in Bristol in recent years.

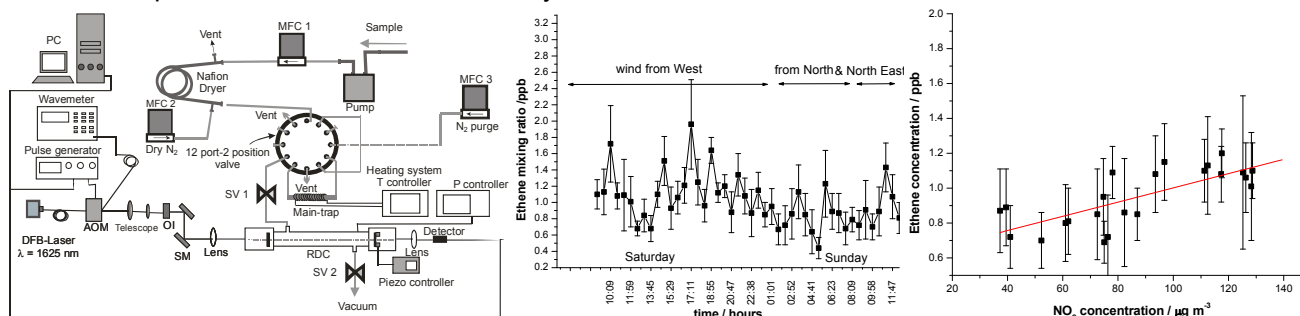


Figure 1: Schematic diagram of the CRDS setup (left); Variation of ethene in Bristol air on the weekend of Saturday 11/12/2010 and Sunday 12/12/2010 (middle); Correlation between ethene (C<sub>2</sub>H<sub>4</sub>) and nitrogen dioxide (NO<sub>2</sub>) in Bristol air (right).

Here, improvements are reported to the preconcentration method, with use of a different adsorbent material to our prior work that achieves a higher preconcentration factor. With a fully automated system, continuous measurements of ethene in Bristol air were performed over representative weekdays and weekends, and diurnal variations recorded for 24 hours at time intervals of 30 minutes. The data have been analysed in the context of meteorological conditions and probable pollution sources and sinks.

## 25.) Multiple wavelength fiber-optical absorption spectroscopy on nanoliter volumes

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Many applications in analytical spectroscopy require extremely sensitive measurements on small liquid sample volumes at low micro-molar concentrations. Here, phase-shift cavity ring-down (CRD) spectroscopy is used to determine the optical loss of a sample from the phase lag between intensity modulated light entering and exiting the cavity. Dual-wavelength detection is performed by using two different quasi-continuous light sources modulated at a separate frequency and frequency-division multiplexing of the detector signal. A single broadband detector records the total signal which is demodulated by lock-in amplifiers at the corresponding two frequencies thus avoiding a dispersive element or interferometer. Applying an optical waveguide loop as the cavity medium permits multiwavelength absorption detection in a 100 nL liquid volume.

Concentration measurements of two dyes were performed simultaneously at two wavelengths (405 nm and 810 nm) in either pure samples or in mixtures. The respective concentrations could be quantified regardless of the composition of the sample. A detection limit of  $0.02 \text{ cm}^{-1}$  at 405 nm was achieved and the dynamic range was determined to  $1 \text{ }\mu\text{M} - 500 \text{ }\mu\text{M}$ . No crosstalk between the two channels was observed. Implementing light emitting diodes also allows multiwavelength CRD spectroscopy in the UV-wavelength range for absorption of biological samples, such as proteins, RNA and DNA.

## 26.) Functionalized silicon Fabry-Perot microcavities for chemical vapor sensing

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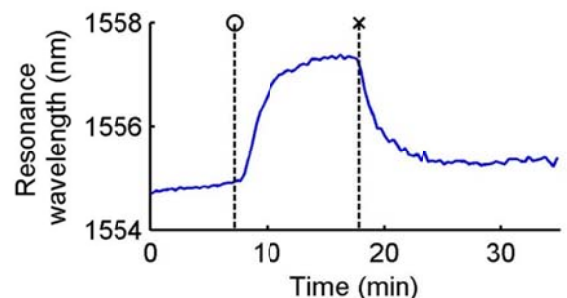
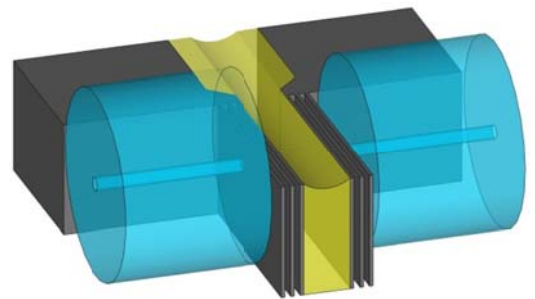
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Fabry-Perot microcavities based on two Bragg mirrors vertically etched in silicon are filled with gas permeable polymers (Figure 1). As these polymers absorb gas, their refractive index changes, which induces a shift of the resonance wavelength of the cavity. The amplitude of this shift is a function of the concentration of the targeted gas in the vicinity of the cavity and of the refractive index difference between the polymer and the gas.

The main advantage the Fabry-Perot resonator is its ability to perform volume refractive index sensing, in contrast to evanescent wave sensors such as tapered optical fibers or on-chip waveguided resonators. This greatly simplifies the coating of the device since surface effects occurring at the interface of the polymer and the resonator become negligible, and since the initial refractive index of the polymer does not need to be matched to that of the waveguiding structure.

Preliminary results for a cavity filled with a poly (diphenyl siloxane) - poly(dimethyl siloxane) copolymer and exposed to xylene vapour yield a linear sensitivity of  $0.015 \text{ nm/ppm}$  for concentrations between 120 (Figure 2) and 1300 ppm. Future work will involve testing of other gas, other polymers and broader ranges of concentrations.



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## Poster Presentations

### 27.) Vernier effect of an optical frequency comb coupled to a high-finesse cavity: a novel laser spectroscopy tool

**Chadi Abd Alrahman, Roberto Grilli, Samir Kassi, Guillaume Méjean, Irene Ventrillard, Daniele Romanini**

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We will present recent results in our development efforts of new techniques of femtosecond laser absorption spectroscopy in a high finesse cavity. These follow the development of the technique of ML-CEAS (Mode-Locked Cavity-Enhanced Absorption Spectroscopy)<sup>4</sup> previously demonstrated in our laboratory and which we are currently applying to the detection of reactive molecules in field campaigns<sup>5</sup>. As in ML-CEAS we exploit the frequency comb properties of femtosecond laser oscillators and of optical cavities. However, while in ML-CEAS these are closely matched over the whole laser spectrum, we are now looking into situations where the combs are mismatched such as the one leading to the Vernier effect, where only a small fraction of the laser spectrum is matched to the cavity comb, leading to frequency selection without the need of a spectrograph (indispensable for ML-CEAS).

### 28.) Studies on the heterogeneous photosensitized transformation of model HULIS by iron(III) halides under humid conditions

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Heterogeneous photochemical reactivity of atmospheric aerosols causes degradation of their organic content, formation of secondary organic aerosols (SOA), and enhancement in the uptake and release of gas phase species. While bulk aqueous photochemistry of transition metal cations and chromophores relevant to aqueous aerosols have been the subject of a number of reviews and book chapters, extrapolating results from these bulk studies to photochemistry occurring at the surface of atmospheric aerosols is *not a straightforward task*. This is mainly because the molecular environment of the liquid/air and solid/air interfaces is different from that in the bulk.

In this poster, we show results on the photosensitized transformation of solid gallic acid externally mixed with  $\text{FeX}_3$  ( $\text{X}=\text{Cl}$  and  $\text{Br}$  as photosensitizer) under humid conditions. Gallic acid is a hydrolysis product of tannic acid, a model macromolecule for humic-like substances in aerosols and aged polyaromatic hydrocarbons (PAH). These studies were conducted using diffuse reflectance infrared Fourier transform spectroscopy (DRIFTS) under dry and humid condition (<1, 30 and 60% RH). Changes to surface functional groups were monitored as a function of time, % $\text{FeX}_3$  and intensity of simulated solar light.

Difference absorbance spectra show positive and negative features suggesting the formation of organohalide compounds in the condensed phase and changes to GA functional groups. Apparent first order rate constants were derived from the kinetic analysis of these features. Acetone was one of the gas phase products detected. Potential release of gaseous  $\text{X}_2$  and other VOCs was suggested from a proposed mechanism. DRIFTS experiments were complemented with water uptake measurements on thin films of GA and a GA/ $\text{FeX}_3$  mixture using a quartz crystal microbalance (QCM) as a function of RH (<1-60%). Results indicate continuous multilayer formation of adsorbed water with monolayer forming at 30 and 12%, respectively. The implication of our studies on the aging of multicomponent aerosols due to heterogeneous photochemistry and its incorporation into cloud chemistry models will be discussed.

<sup>4</sup> T. Gherman, D. Romanini, "Mode-locked cavity-enhanced absorption spectroscopy", *Opt. Express* 10, 1033 (2002).

<sup>5</sup> G. Méjean, S. Kassi, D. Romanini, "Cavity enhanced absorption spectroscopy with an optical comb: detection of atmospheric radicals in the near UV", *Optics Letters* 33, 1231 (2008).

## 29.) The near-IR absorption spectra of gaseous trace constituents using Fourier-transform broadband cavity-enhanced absorption spectroscopy

**Sophie Dixneuf<sup>1,2</sup>, Albert A. Ruth<sup>1,\*</sup>, Deirdre M. O'Leary<sup>1</sup>, Ravi M.K. Varma<sup>3</sup>, Johannes Orphal<sup>4</sup>**

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By combining incoherent broadband cavity-enhanced absorption spectroscopy (IBBCEAS) with a Fourier transform (FT) detection scheme high spectral resolutions can be achieved over a wide spectral range, which is essentially limited by the high reflectivity range of dielectric cavity mirrors. A high spectral resolution ( $<0.2 \text{ cm}^{-1}$ ) over a wide spectral range enables the trace detection of atmospheric constituents in the near infrared with high selectivity, however, at the expense of acquisition time.

In this work the methyl cyanide ( $\text{CH}_3\text{CN}$ ) absorption spectrum was measured between  $\sim 6000$  and  $8000 \text{ cm}^{-1}$  with a resolution of  $0.12 \text{ cm}^{-1}$  using a supercontinuum light source in conjunction with a cavity of  $644 \text{ cm}$  length. Methyl cyanide (=acetonitrile) is a molecule of considerable atmospheric importance which for instance is generated in fires and occurs in the atmospheres of the giant planets. The FT-IBBCEAS setup was validated in comparison with a high-resolution diode laser based off-axis cavity enhanced absorption setup within specific certain spectral ranges.

Two different configurations of the setup (i) a long cavity employing a supercontinuum source, and (ii) a short cavity employing a Xe arc lamp, will be discussed in the context of absorption measurements involving other compounds. The absorption of methyl iodide ( $\text{CH}_3\text{I}$ ), a molecule present in the marine boundary layer (due to the biogenic emission of marine organisms) whose radioactive isotopes need to be closely monitored in the environment of nuclear power stations, was measured with a resolution of  $0.09 \text{ cm}^{-1}$  at 3 Torr. Absorption measurements of  $\text{NO}_2$  at 10 Torr will also feature in this presentation.

## 30.) Gas correlation cavity phase shift spectrometer

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We have developed and tested a cavity enhanced spectrometer that uses a superluminescent diode (SLD) light source and a gas correlation filter to measure gas concentration. The filter is a cell containing the gas to be measured, in this case acetylene, which is inserted in front of the cavity. The column density of the filter is chosen to give significant bleaching of the SLD spectrum at the target gas absorption wavelengths. By applying a square wave current to the SLD and measuring the phase shift of the light transmitted through the cavity, the cavity losses can be determined. These can be compared to the losses without the filter. In another implementation, an electro-optic phase modulator is placed between the filter and the cavity. When the modulator is on, the sidebands of nearby wavelengths fill in the holes bleached into the SLD spectrum by the filter. The phase shift is compared with and without the EOM. The use of a broad band light source ensures that the entire spectral band is probed, reducing some effects of temperature on the measurement, while the gas filter provides spectral selectivity.

The bandwidth of the SLD (6.4 THz) spans thousands of cavity modes. Emitted light from the SLD was aligned with the cavity axis using a two-lens telescope. An off-axis parabolic mirror focused the light transmitted through the cavity onto an InGaAs photodiode, and the resulting signal was conditioned with a low-noise preamplifier before detection with a lock-in. Based on the minimum detectable change in phase shift derived from the Allan variance, a minimum ( $2\sigma$ ) detectable absorption for the cavity enhanced detection was determined to be  $1 \times 10^{-10} \text{ cm}^{-1}$ . Using the EOM to modulate the filtered light gave a signal that was more spectrally selective for acetylene. However, due to the sparse nature of the acetylene spectrum and the relatively low modulation index, this translated into a modest detection limit of about 5 ppm.

## 31.) Evanescent-wave optical cavity-based techniques for probing of molecular thin films

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The study of molecular properties and chemical dynamics at the interfaces is of fundamental importance in chemistry and physics. Due to the surface-confined nature of the evanescent field, evanescent-wave cavity ring-down spectroscopy (EW-CRDS) is particularly suitable for probing interfacial phenomena.<sup>1-3</sup> Here, we report on studies of thin molecular films at the silica|air interface. Polarization-dependent EW-CRDS has been used to study adsorption, molecular orientation<sup>4</sup> and photo-degradation of a range of dyes at the fused silica|air interface. In particular, EW-CRDS has been used to study *in situ* photobleaching of thin films of methylene blue under both O<sub>2</sub> and N<sub>2</sub> atmospheres. Under similar excitation conditions, photobleaching occurs much faster in N<sub>2</sub> than in O<sub>2</sub> an observation which we interpret as due to the efficient quenching of excited triplet state intermediates by oxygen. No orientational dependence to the photo-degradation is observed and for sub monolayer films the kinetics in both O<sub>2</sub> and N<sub>2</sub> is apparently dominated by second order kinetics. We propose a model accounting for these observations and report rate constants for the key steps.

<sup>1</sup> A. C. R. Pipino, J. W. Hudgens, and R. E. Huie, *Review of Scientific Instruments* **68**, 2978 (1997).

<sup>2</sup> A. C. R. Pipino, *Physical Review Letters* **83**, 3093 (1999).

<sup>3</sup> M. Schnippering, S. R. T. Neil, S. R. Mackenzie, and P. R. Unwin, *Chemical Society Reviews* **40**, 207 (2011).

<sup>4</sup> F. P. Li and R. N. Zare, *Journal of Physical Chemistry B* **109**, 3330 (2005).

## 32.) Applications of cavity ring-down aerosol extinction spectrometer to laboratory and field studies

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<sup>2</sup> *National Institute for Environmental Studies, 16-2, Onogawa, Tsukuba 305-8506, Japan*

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Aerosol particles have an important role in radiation balance in the atmosphere by scattering and absorbing incident light. Therefore, accurate determination of the optical properties of atmospheric aerosols is essential. We developed a pulsed cavity ring-down aerosol extinction spectrometer (CRD-AES) to measure extinction coefficients at 355 and 532 nm and its relative humidity (RH) dependence and applied to laboratory and field studies on optical properties of aerosols. In the laboratory studies, the optical properties of secondary organic aerosols (SOAs) generated during the photooxidation of toluene in the presence of NO<sub>x</sub> and the ozonolysis of  $\alpha$ -pinene have been investigated using the CRD-AES and a 3 $\lambda$  nephelometer. As a result, significant light absorption was found at 355 nm for the toluene SOAs. In contrast, no significant absorption was found either at 355 or 532 nm for the  $\alpha$ -pinene SOAs. Our results indicate that light absorption by the SOAs formed from the photooxidation of aromatic hydrocarbons have a potential to influence the total aerosol light absorption, especially at UV wavelengths. In the field studies, the RH dependence of extinction coefficients has been measured in central Tokyo during summertime. The relationship between RH dependence of extinction coefficients and aerosol chemical compositions, which were measured by a time-of-flight aerosol mass spectrometer and an EC/OC analyzer (thermal-optical analysis), have been investigated.

### 33.) Optical cavity-based techniques as probes of magnetic field effects in biological systems

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We report on progress in developing optical cavity-based spectroscopic techniques to study photo-induced solution-phase reactions in biological systems proceeding via spin-correlated radical pairs. In particular we are interested in how the kinetics and/or yields of such reactions might be affected by applied magnetic fields. The physical origin of these magnetic field effects (MFEs) is usually discussed within the framework of the radical pair mechanism (RPM) based on the magnetosensitivity of the singlet-triplet mixing efficiency in spin-selective reactions.

MFEs have been studied in a variety of contexts<sup>1</sup> including the magneto-reception mechanism in migratory birds.<sup>2</sup> Experiments investigating MFEs are typically based on optical spectroscopy and have traditionally used flash-photolysis transient absorption techniques to detect short-lived radical species. Such methods are hampered in application to real biological systems by their requirement for high concentrations, long averaging times and large sample volumes to minimize the effects of photo-degradation. Real biological samples of interest can typically be produced only in small ( $\mu\text{L}$ ) volumes and at low (nM) concentrations.

In an attempt to provide the step-change in sensitivity required, we have developed a variety of complementary cavity-based techniques, including variants of cavity ring-down spectroscopy, cavity enhanced absorption spectroscopy (CEAS) and supercontinuum broadband CEAS, which have been tested and optimized for MFE-measurement in solution.<sup>3,4</sup>

<sup>1</sup> U. E. Steiner and T. Ulrich, *Chemical Reviews* **89**, 51 (1989).

<sup>2</sup> T. Ritz, S. Adem, and K. Schulten, *Biophysical Journal* **78**, 707 (2000).

<sup>3</sup> S. R. T. Neil, K. Maeda, K. B. Henbest, M. Goetz, R. Hemmens, C. R. Timmel, and S. R. Mackenzie, *Mol. Phys.* **108**, 993 (2010).

<sup>4</sup> M. Schnippering, S. R. T. Neil, S. R. Mackenzie, and P. R. Unwin, *Chemical Society Reviews* **40**, 207 (2011).

### 34.) A rack-mounted diode laser cavity ring-down spectrometer for measurement of NO<sub>3</sub> and N<sub>2</sub>O<sub>5</sub> with automated zeroing and calibration

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The oxides of nitrogen are important trace gases in the troposphere, the lowest layer of Earth's atmosphere, as they play key roles in the photochemical formation of ozone (O<sub>3</sub>). At night, a considerable amount of NO<sub>x</sub> (= NO+NO<sub>2</sub>) is converted to the nitrate radical (NO<sub>3</sub>), produced from oxidation of NO<sub>2</sub> by O<sub>3</sub>, and dinitrogen pentoxide (N<sub>2</sub>O<sub>5</sub>), which in ambient air is in equilibrium with NO<sub>2</sub> and NO<sub>3</sub>. Accurate measurements of NO<sub>3</sub> and N<sub>2</sub>O<sub>5</sub> are needed to gain further insights into nocturnal nitrogen oxide chemistry. A commonly used measurement technique is cavity ring-down spectroscopy (CRDS) utilizing the strong absorption line of NO<sub>3</sub> at 662 nm and quantifying N<sub>2</sub>O<sub>5</sub> indirectly by conversion to NO<sub>3</sub> in a heated inlet. However, since NO<sub>3</sub> is a highly reactive radical and N<sub>2</sub>O<sub>5</sub> readily reacts on surfaces, the accuracy of these measurements is limited by knowledge of the NO<sub>3</sub> and N<sub>2</sub>O<sub>5</sub> inlet transmission efficiencies. Further, a limitation of current instruments is that they are rather large and cannot be rack-mounted.

In this presentation, a recently constructed compact, rack-mountable red diode laser CRDS for simultaneous measurements of NO<sub>3</sub> and N<sub>2</sub>O<sub>5</sub> is described. The instrument is equipped with an automated zeroing and calibration scheme. For calibration, the effluent of a diffusion source containing solid N<sub>2</sub>O<sub>5</sub> was added to the tip of the instrument's inlet. A heater placed between the diffusion source and the instrument inlet allowed delivery of either NO<sub>3</sub> or N<sub>2</sub>O<sub>5</sub>. Addition of excess NO was used to titrate NO<sub>3</sub> to NO<sub>2</sub>, which (unlike NO<sub>3</sub> and N<sub>2</sub>O<sub>5</sub>) is transmitted with 100% efficiency and was quantified using blue diode laser CRDS. The instrument response (for N<sub>2</sub>O<sub>5</sub>) was typically ~40% of the theoretical maximum, limited by reduced inlet transmission efficiencies and a slight mismatch of the laser diode output with the narrow NO<sub>3</sub> absorption line. The current CRDS has a precision (1 $\sigma$ , 10s) of 5 parts-per-trillion by volume (pptv). The CRDS was used to quantify N<sub>2</sub>O<sub>5</sub> on a rooftop on the University of Calgary campus over a 3-day period in August, 2010. A maximum of 135 pptv of N<sub>2</sub>O<sub>5</sub> was observed. Advantages and limitations of the current red diode laser CRDS are discussed.

### 35.) Off-axis cavity ring down spectroscopy based on a continuous-wave optical parametric oscillator

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Continuous-wave cavity ring down spectroscopy (cw-CRDS) is a sensitive absorption technique for trace gas analysis. Although it is highly sensitive and relatively fast, ring down repetition rate and spectral resolution are limited by the cavity free spectral range (FSR). Normally, the injected beam is mode matched to the lowest transverse electro-magnetic mode (TEM<sub>00</sub>) of the cavity. Light is coupled into the cavity only when standing wave condition is fulfilled. Scanning of the laser without variation of the cavity length leads to transmission comb where recorded ring down times are separated in frequency by the FSR.

Recently Romanini [1] *et. al.* reported an off-axis (OA) CRDS spectrometer operating in the 766 nm region where the FSR of the cavity is reduced by  $N = 4$  times from the original. In this re-entrant condition, the cavity length is chosen to provide degeneracy of transverse modes. If the injection is adequately off-axis, the beam returns to the starting point after  $N$  round trips. This divides the FSR to  $N$  group of degenerated modes which are equally frequency-spaced. We present an off-axis (OA) CRDS spectrometer based on a home-built continuous-wave optical parametric oscillator (cw-OPO) operating in the mid-infrared region (2.75 - 3.45  $\mu\text{m}$ ). We have also studied how variations of coupling and cavity parameters affect the performance of the spectrometer. The measurement of formaldehyde (H<sub>2</sub>CO) using an OA-CRDS spectrometer will be presented.

[1] J. Courtois, A. K. Mohamed and D. Romanini, *Opt. Express* **18**(5), 1 March 2010

### 36.) DNA and RNA characterizations in nanoliter volumes by fiber-loop cavity ring-down spectroscopy

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In biochemistry labs around the world deoxyribonucleic acid (DNA) and ribonucleic acid (RNA) is distinguished and quantified using mainly optical detection methods. Optical absorption measurements at 260 nm and 280 nm can be used to quantify nucleic acid contamination of protein samples and protein contamination of nucleic acids. For pure DNA samples the ratio is typically  $A_{260}/A_{280} = 1.7$  to 2.0, and  $A_{260}/A_{280} = 1.5$  to 2.0 for RNA. If the sample also contains contaminations of proteins or phenols, the  $A_{260}/A_{280}$  ratio is considerably smaller. The state of the art in DNA/RNA quantification and protein contamination involves a UV-spectrometer. In most absorption spectrometers the sample is placed in a cuvette, which requires large sample volumes of, typically, several hundreds of microliters. But in many applications only a few microliter of sample are available and often the part of the sample used for measurements cannot be further processed.

Fiber-loop cavity ring-down spectroscopy (CRDS) permits absorption detection of dilute liquid samples in detection volumes as small as a few nanoliters. In phase-shift CRDS the light intensity is modulated sinusoidally and the ring-down time is obtained from the phase-shift between the light entering and exiting the cavity. We achieved simultaneous dual-wavelength detection of two light sources by modulating each light source at a different frequency and a broadband detector detects the combined signal. By demodulating the signal with lock-in amplifiers at the corresponding frequencies, the phase-shift of each light source can be extracted. To measure DNA and RNA samples UV-wavelengths are required, but diode lasers are commercially available only for wavelength longer than 350 nm. Therefore we use UV-LEDs at wavelength of 260 nm and 280 nm. We can quantify the absorbance at both wavelengths simultaneously and the ratio  $A_{260}/A_{280}$  of the sample can be determined. In a preliminary measurement a detection limit of 10  $\mu\text{g}/\text{mL}$  was achieved at a wavelength of 260 nm in a detection volume of only 100 nL. The  $A_{260}/A_{280}$  ratio could be determined with a standard deviation of 0.1 (sample: ketoprofen 15  $\mu\text{g}/\text{mL}$ ).

### 37.) The LabVIEW application for off-axis cavity enhanced absorption spectroscopy

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Abstract—This paper presents the graphical program design based on Laboratory Virtual Instrumentation Engineering Workbench (LabVIEW) for off-axis cavity enhanced absorption spectroscopy (OA-CEAS). The program with friendly user interface has the abilities of instrument controlling, data acquiring, processing, displaying and storing in real time. By using this virtual instrumentation (VI), laser, oscilloscope, wavemeter and data acquisition board can be easily controlled through personal computer (PC), and the absorption spectrum will be recorded and displayed conveniently after graph fitting or filtering. The experimental result proves that the design of multiplex function satisfies the experimental requirements well. All subVIs making up this software can be shared by other similar experimental programs with a little variation. Finally this program is made independent of LabVIEW environment and can be applied separately.

Keywords-OA-CEAS; Data processing; Instrument control; LabVIEW

### 38.) Using Liquid Core Waveguides and Micro Optical Devices for Cavity Ring-Down spectroscopy

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Absorption losses of small liquid samples, as needed in modern analytical industry, can be measured by cavity ring-down spectroscopy when the optical cavity is entirely built out of optical fibres. In fibre-loop ring-down spectroscopy (FLRDS) the sample is injected into a small gap in the loop cavity. To decrease the detection limit of the system one can extend the path through the sample by increasing the gap width. However, this will also decrease the transmission through the gap and the gap width is practically limited to a distance similar to the diameter of the fiber core. Liquid core waveguides (LCW) can be used to extend the sample gap without a significant decrease in the transmission between the fibre ends. A cavity entirely made of a LCW would maximize the sample path, though coupling light into the optical cavity has its challenges. We present here first results of CRD spectroscopy using a liquid core waveguide loop and discuss the coupling of light into a pure LCW-cavity through fluorescence emission.

Detection of small liquid samples can also be performed by refractive index sensing using micro-ring resonators. In collaboration with the National Research Council in Ottawa, sub-micron silicon waveguides (260 nm high by 450 nm wide) were patterned on a silica substrate. Light traveling through the silicon waveguides exhibits a strong evanescent field due to the compact dimensions and the high refractive index contrast with the surrounding media. This evanescent wave is highly sensitive to changes in the surrounding refractive index, which is our method for chemical detection. Microring resonators have been coated with siloxane based solid phase microextraction matrices for the detection of volatile organic compounds in air including the BTEX class of environmental contaminants. Additionally, periodic mesoporous silicate materials have been applied for the detection of heavy metals in water. The micro-resonator platform combined with highly parallelized detection presents a versatile system for the development of sensors for a broad range of environmental and chemical contaminants.

### 39.) Musical Instrument Pickup based on a Laser locked to an Optical Fiber Resonator

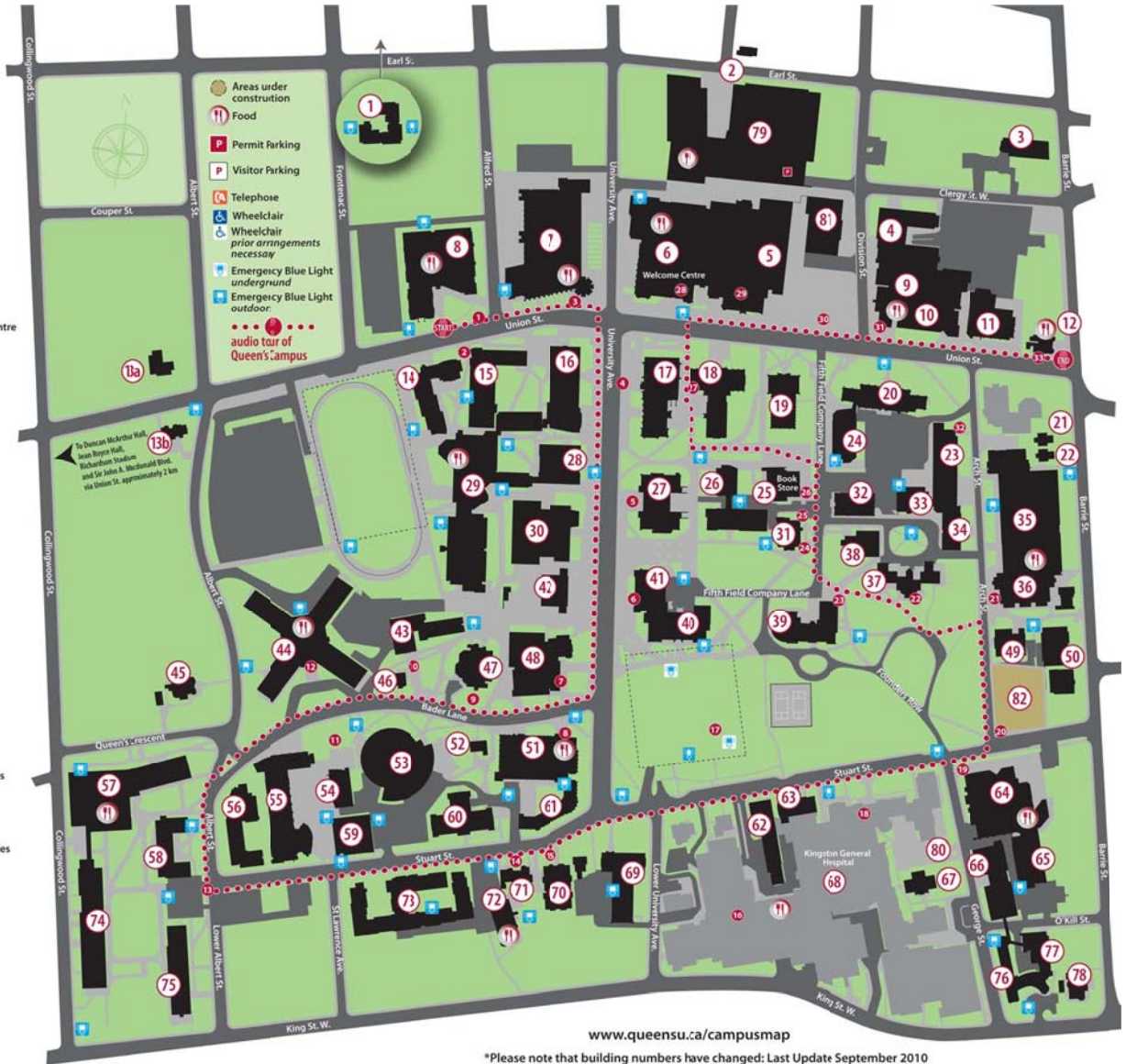
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Fiber Fabry-Perot (FFP) optical cavities, consisting of pairs of high reflectivity fiber Bragg gratings, can be used as sensitive vibration and strain sensors. Any mechanical disturbance of the cavity causes a shift in the resonance frequencies. When attached to musical instruments, these devices make very effective acoustic transducers. A distributed feedback (DFB) laser was locked to a 25 MHz-wide fringe of a FFP cavity, mounted on an acoustic guitar, using the Pound-Drever-Hall method. The correction signal contained acoustic frequency components up to 25 kHz and could be used to accurately reproduce the instrument sound. The pickup system is largely immune to optical noise sources and exhibits a flat frequency response with a distortion free range of about 50 dB.

# Queen's University Campus Map

- 49 Abramsky Hall
- 61 Adelaide Hall
- 48 Agnes Etherington Art Centre
- 52 Ban Righ Centre
- 51 Ban Righ Hall
- 9 Beamish-Munro Hall
- 36 Biosciences Complex
- 64 Botherrell Hall
- 24 Bruce Yrting
- 65 Cancer Research Institute
- 50 Catarqui Building
- 31 Carruthers Hall
- 54 Chernoff Auditorium
- 55 Chernoff Hall
- 60 Chowr Hall
- 25 Clark Hall
- 2 Community Outreach Centre
- 34 Craine Building
- 17 Douglas Library
- 16 Dunning Hall
- 4 Dupuis Hall
- 35 Earl Hall
- 30 Ellis Hall
- 62 Etherington Hall
- 71 Film and Media
- 26 Fleming Hall
- 21 Four Directions Aboriginal Student centre
- 80 GIDRUWing
- 8 Goodes Hall
- 10 Goodwin Hall
- 74 Gordon-Brockington Hall
- 18 Gordon Hall
- 12 Grad Club
- 41 Grant Hall
- 46 Grey House
- 1 Harkness International Hall
- 47 Harrison-LeCaine Hall
- 23 Humphrey Hall
- 32 Jackson Hall
- 42 Jeffery Hall
- 6 John Deutsch University Centre
- 33 Kathleen Ryan Hall
- 40 Kingston Hall
- 68 Kingston General Hospital
- 70 LaSalle Building
- 57 Leonard Hall
- 73 Leggett Hall
- 66 Louise D Acton Building
- 3 MacGillivray Brown Hall
- 29 Mackintosh-Corry Hall
- 78 Macklem House
- 69 McLaughlin Hall
- 58 McNeill House
- 20 Miller Hall
- 75 Morris Hall
- 67 Museum of Health Care
- 19 Nicol Hall
- 38 Old Medical Building
- 27 Ontario Hall
- 5 Physical Education Centre
- 14 Policy Studies Building
- 79 Queens Centre
- 13 Queens Daycare (13a, 13b)
- 22 Queens Quaterly/McGill-Queen's Pass
- 77 Quinte Thousand Island Lodge
- 59 Rideau Building
- 28 Richardson Hall
- 63 Richardson Laboratory
- 45 School of English
- 81 School of Kinesiology and Health Studies
- 82 School of Medical Building
- 15 Sir John A Macdonald Hall
- 53 Stirling Hall
- 7 Stauffer Library
- 37 Summerhill
- 39 Theological Hall
- 72 University Club
- 44 Victoria Hall
- 76 Waldron Tower
- 11 Walter Light Hall
- 43 Watson Hall
- 56 Watts Hall



User Meeting and Summer School:  
 Reception:  
 Lunch Breaks:

Chernoff Hall (55) (the only open entrance on Sunday is on Bader lane)  
 Agnes Etherington Art Gallery (48)  
 University Club (72)

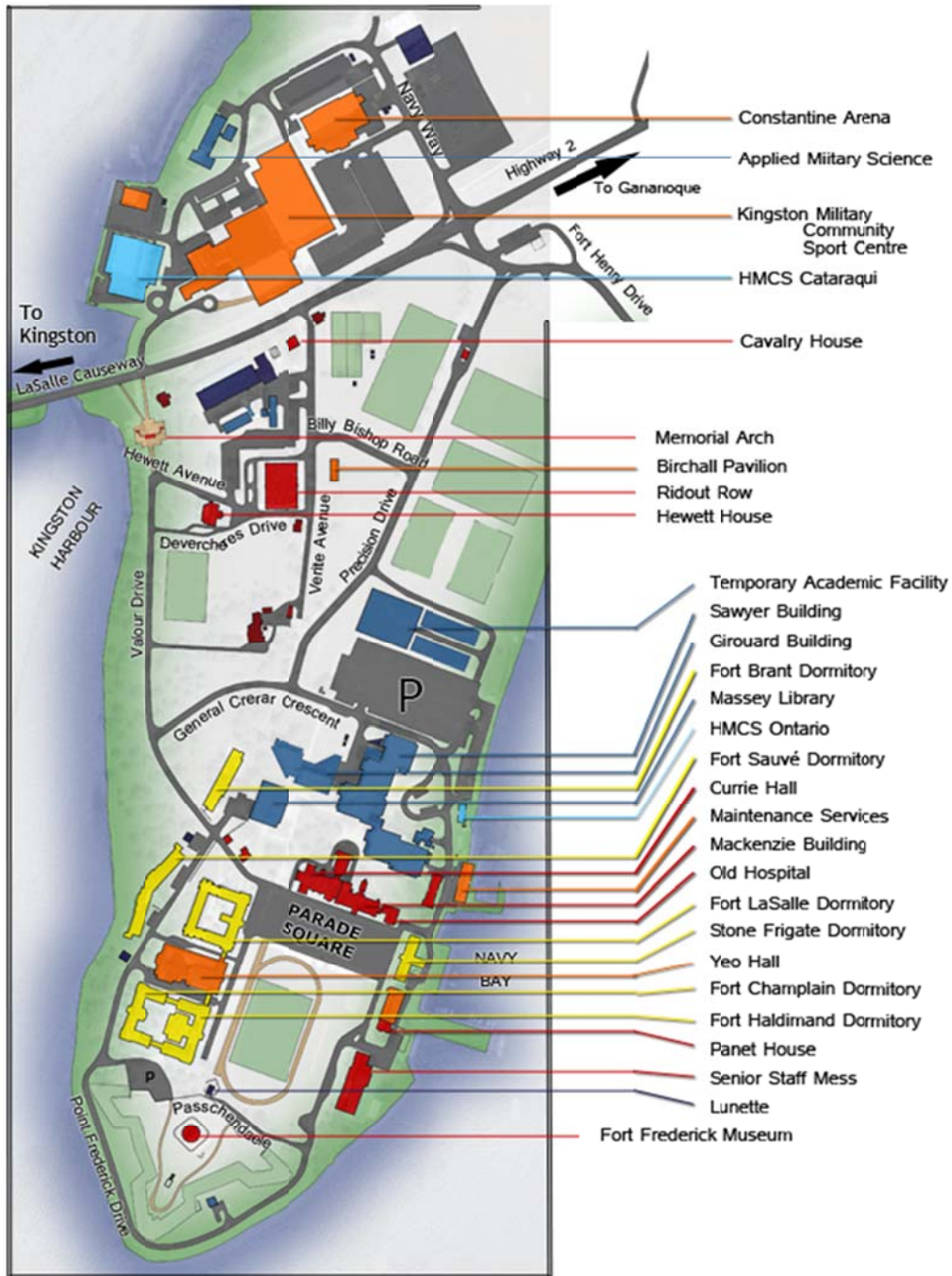
On Campus Parking:

Underground Parkade – entrance on Stuart St, opposite lower University Ave.

Residences:

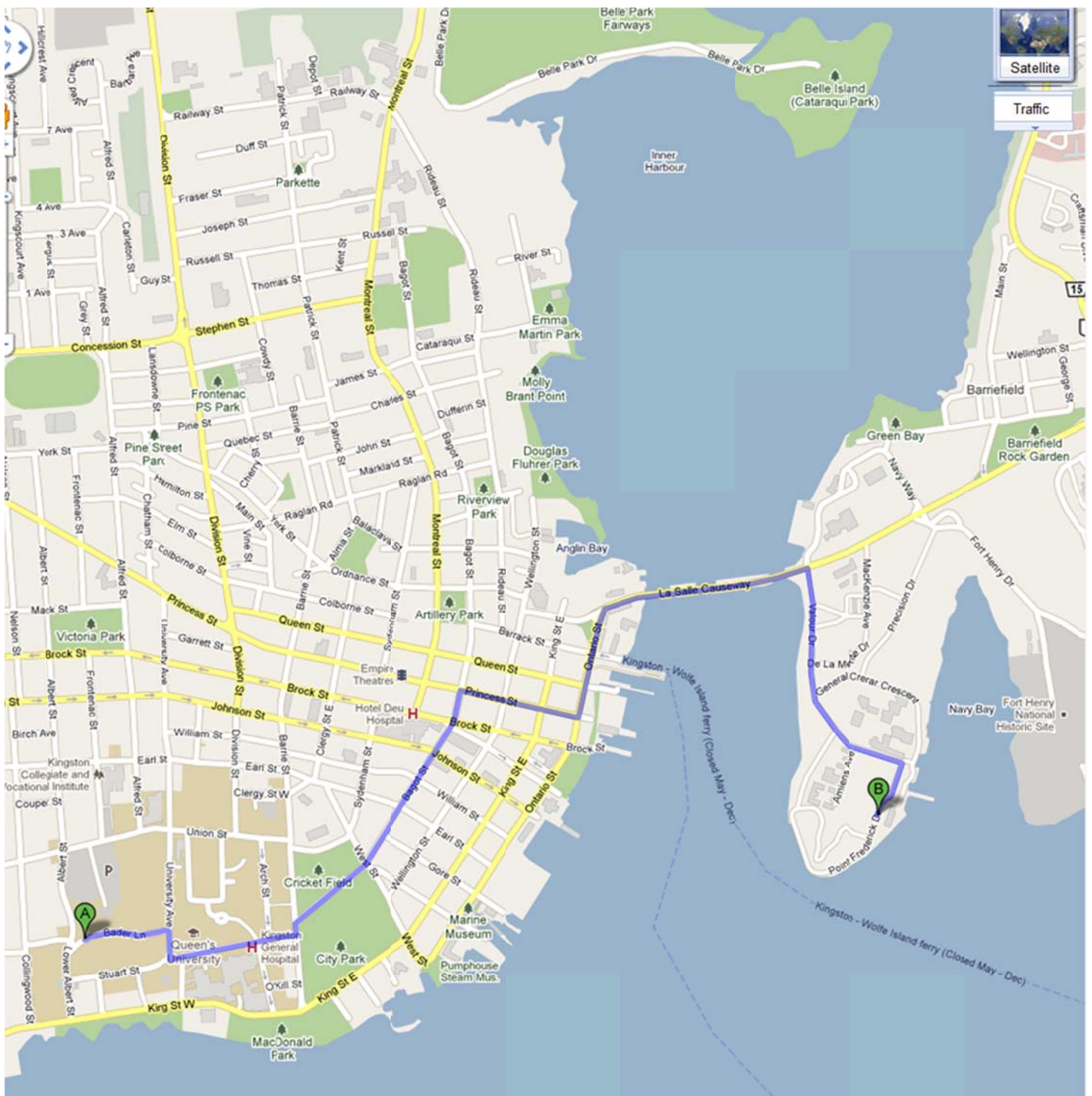
Leggett Hall (73)  
 McNeill House (58)  
 Leonard Hall (57)

# Royal Military College Campus Map



Banquet: Senior Staff Mess

# Walking Route to Senior Staff Mess at Royal Military College



A: Chernoff Hall Queen's University

B: Senior Staff Mess, Royal Military College

Distance: about 4 km (50 min)

# Excursions



## 1000 Island Boat Cruise:

- Departure by Bus (McCoy Charters) from Chernoff Hall at 1:00 pm
- Departure by Boat from Gananoque dock at 2:00 pm.
- Return around 4:30 to Gananoque
- Bus Departure at Gananoque at 5:00 pm



## Fort Henry Tour:

- Departure from Chernoff Hall in private vehicles at around 1:00 pm
- Return by about 4:30 pm



## Hiking Trip at Rock Dunder

- Departure from Chernoff Hall in private vehicles at around 1:00 pm
- Arrival at Rock Dunder by 1:40 pm
- Hike until about 4:00 pm – this is 4 km hike to a lookout point (126 m high).
- Return to Kingston by about 4:40 pm

***At about 5 pm: BBQ dinner in the backyard of the Loock Residence, 674 Rogers Sideroad (off Hwy #2). Please RSVP by Monday evening using the sheets at the Poster Session and Banquet.***

# Contacts

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