ICTP Winter School, Trieste February 2016

Comb-assisted Spectroscopy

Paolo DE NATALE

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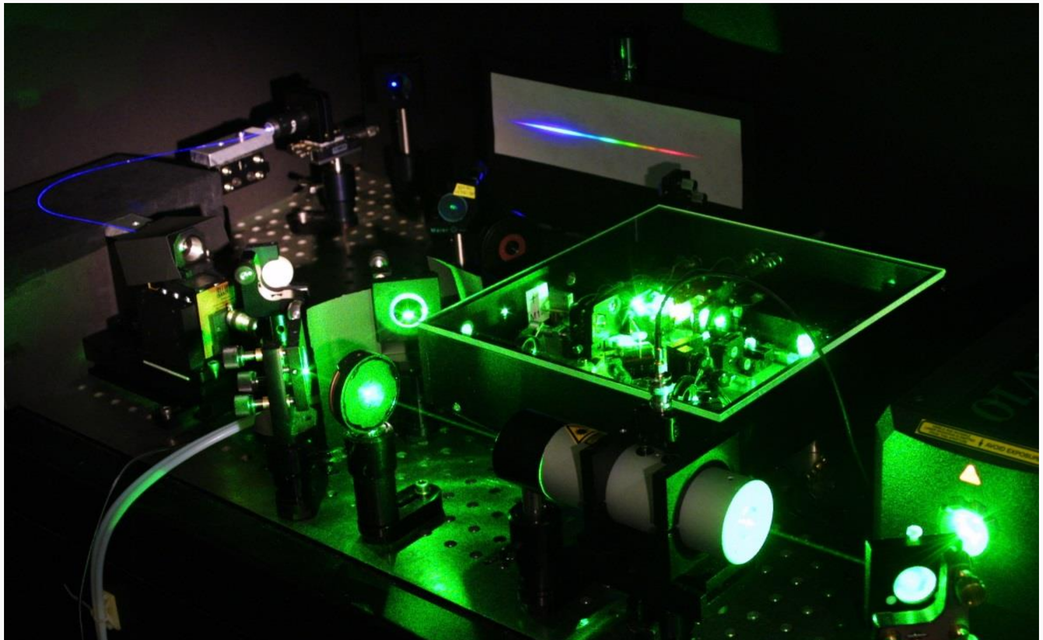
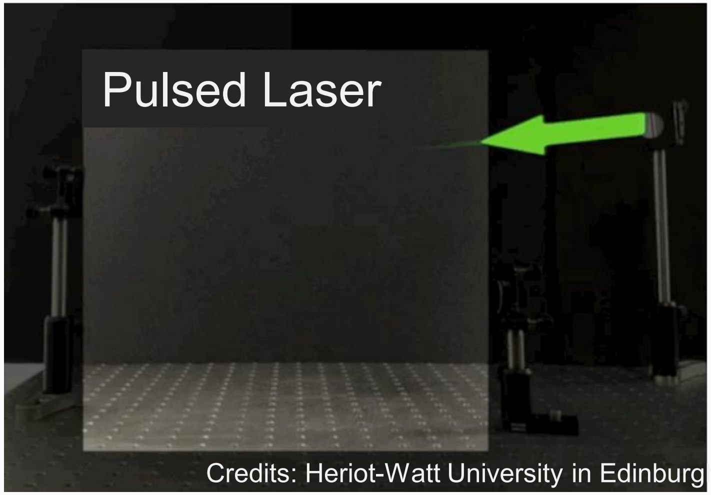
The laser frequency Comb

Theodor W. Haensch
Nobel Prize 2005



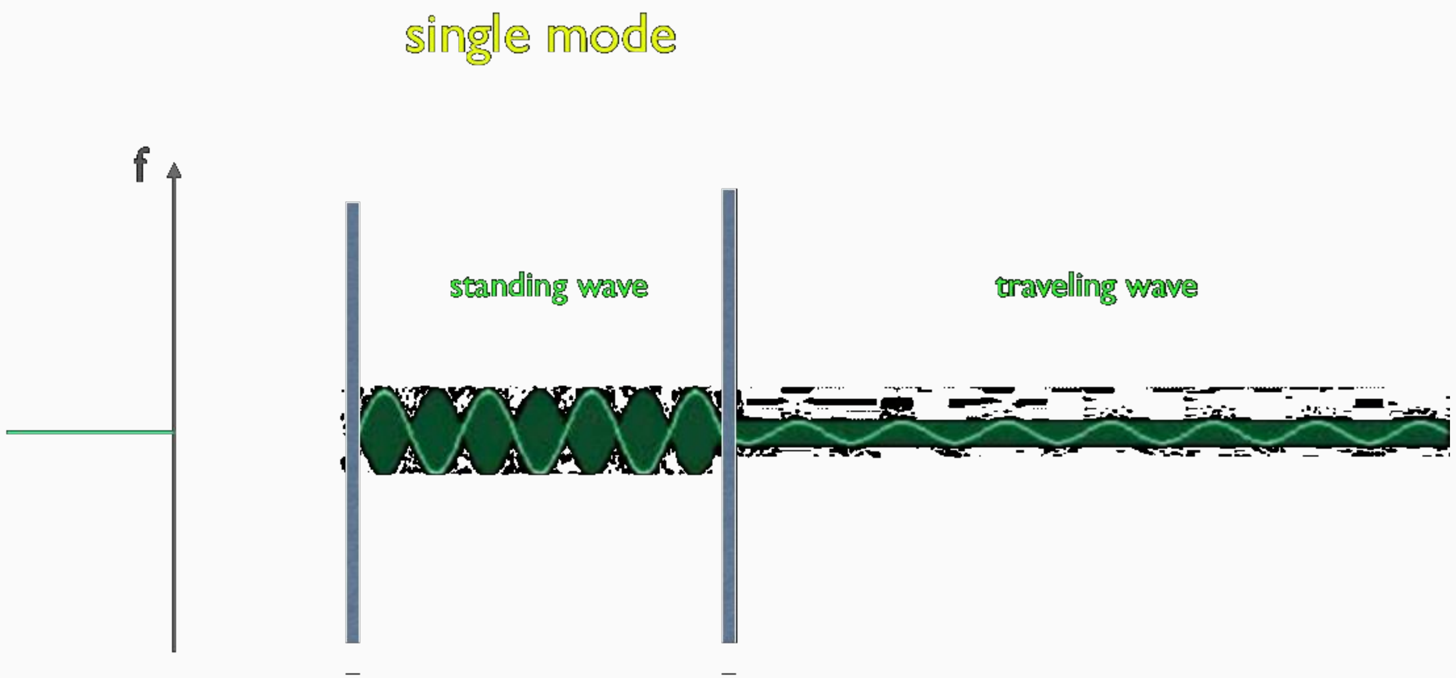
Courtesy of T. W. Haensch – MPQ Institute



Credits: Heriot-Watt University in Edinburg

- A simple instrument to measure frequencies from 0.1 THz to 1000 THz.
- A phase-coherent link between the Optical and RF regions
- A clockwork to transfer the resonant frequency of Atomic Clocks

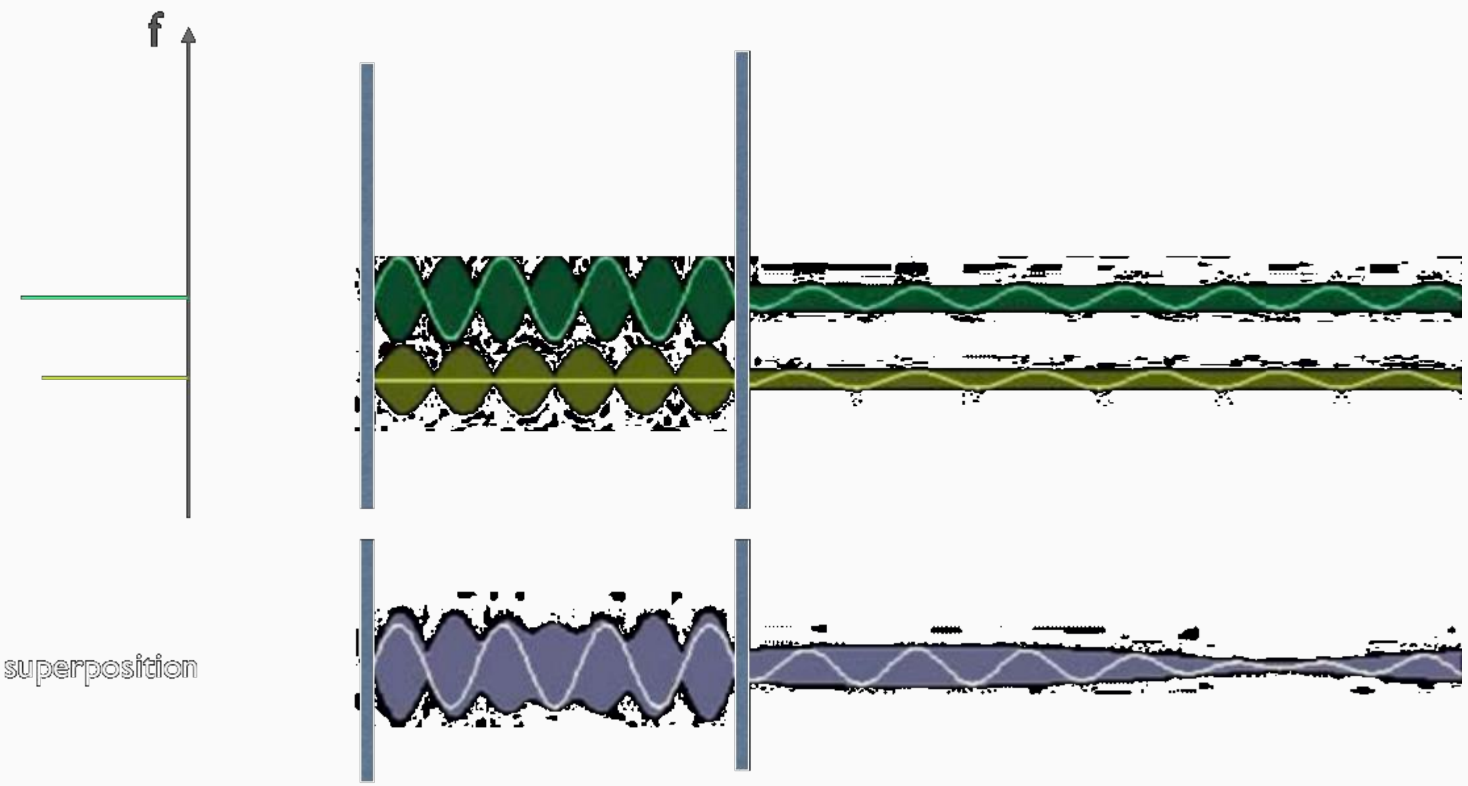
Comb working principle



Courtesy of T. W. Haensch – MPQ Institute

Comb working principle

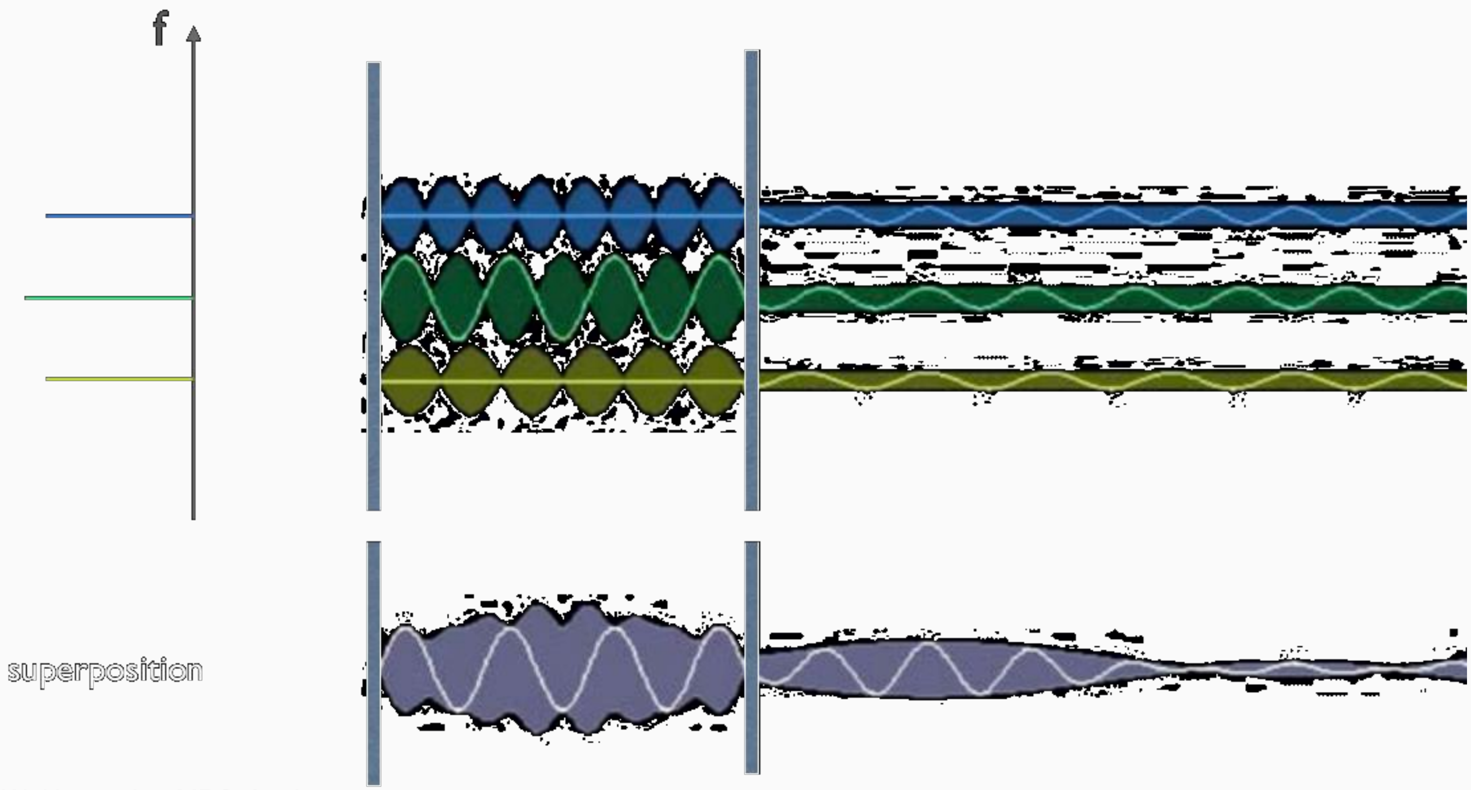
two modes



Courtesy of T. W. Haensch – MPQ Institute

Comb working principle

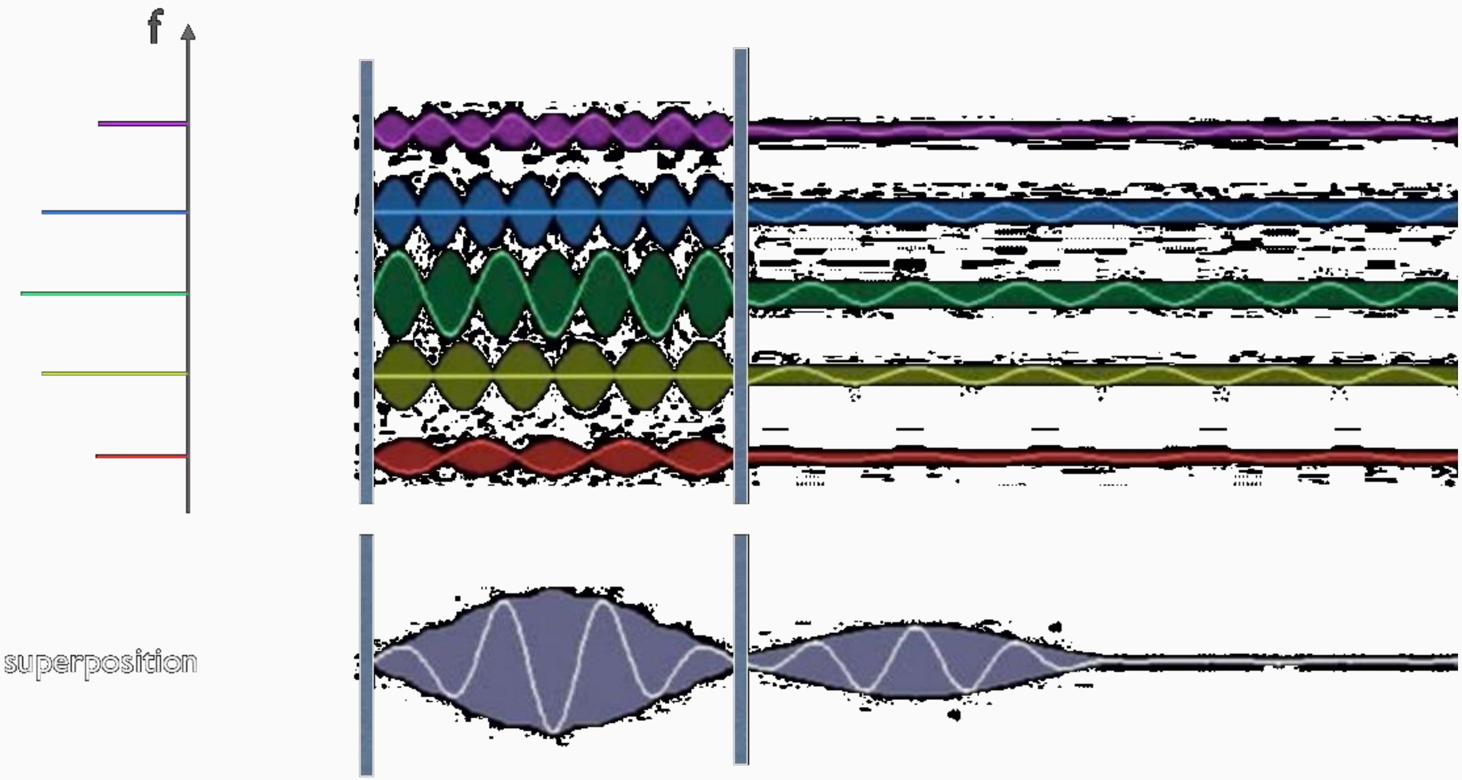
three modes



Courtesy of T. W. Haensch – MPQ Institute

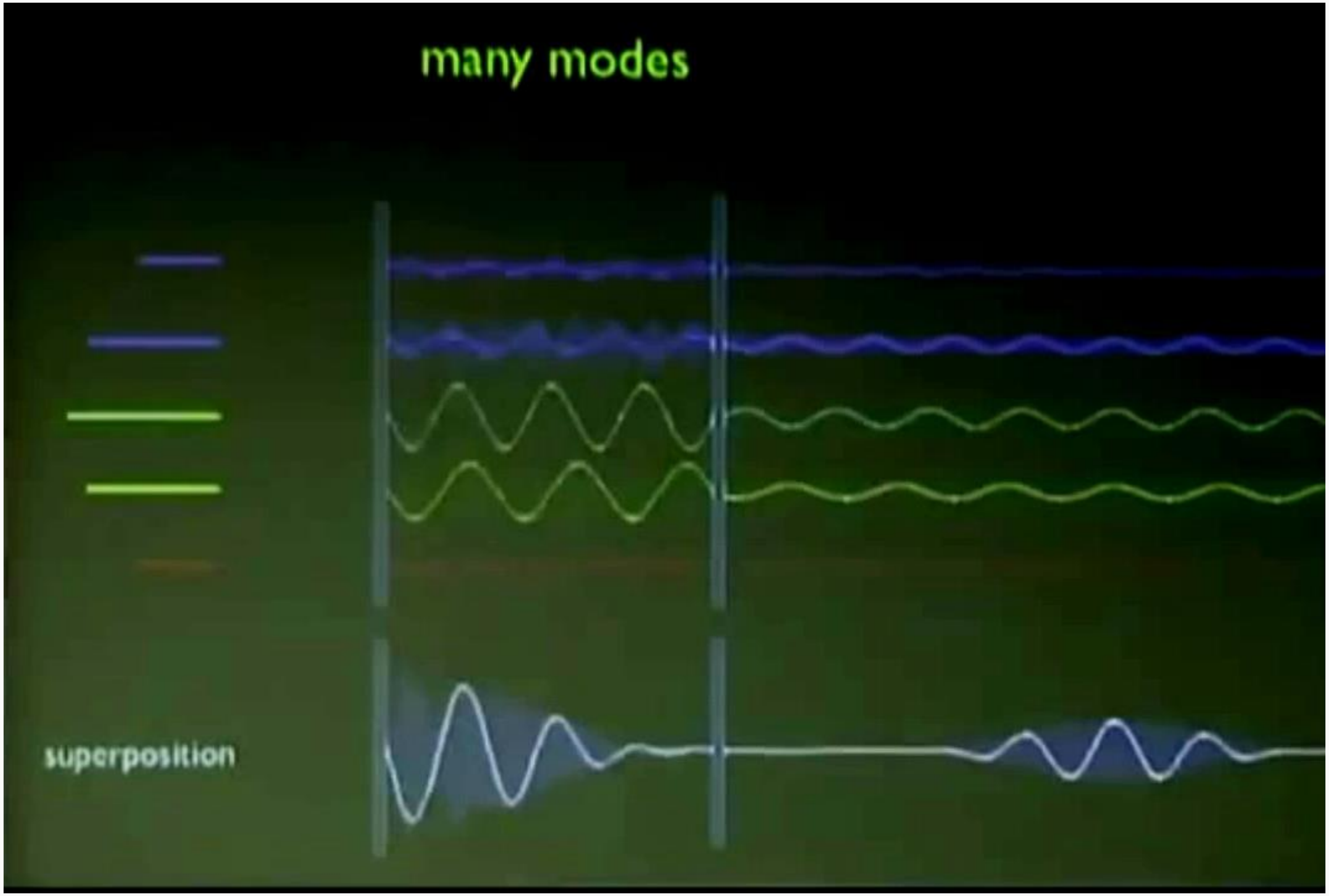
Comb working principle

many modes



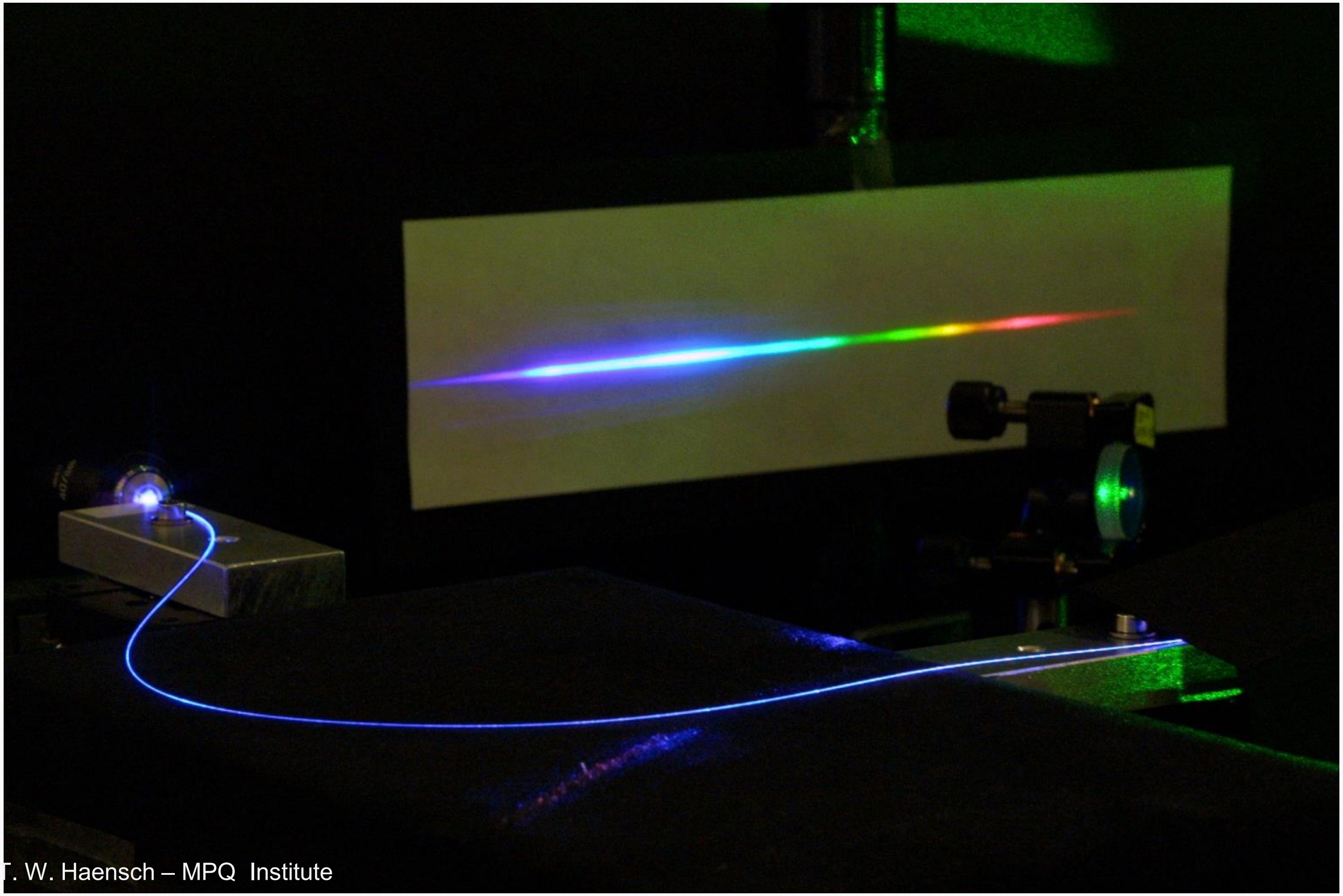
Courtesy of T. W. Haensch – MPQ Institute

Comb working principle



Courtesy of T. W. Haensch – MPQ Institute

Comb working principle



Courtesy of T. W. Haensch – MPQ Institute

Principles of Optical Frequency Combs

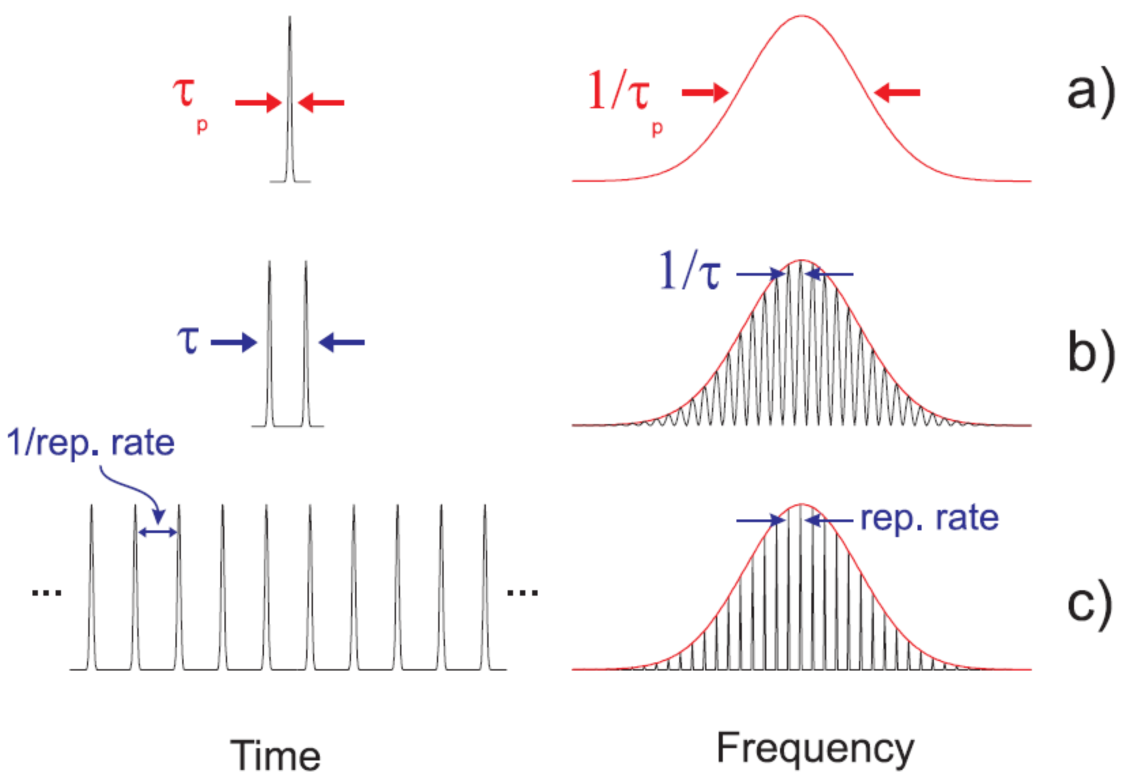


FIGURE 6.1
A single laser pulse has a frequency bandwidth which scales with the inverse of its duration τ_p (a). If one uses a pair of phase-locked pulses, delayed by a time τ, the resulting spectrum maintains the broad envelope of width $1/\tau_p$ but with a sinusoidal modulation of spectral period $1/\tau$ (b). This width sets the new instrumental resolution. If one uses an infinite sequence of pulses, locked in phase and equally delayed in time of a time interval τ, the spectrum breaks up in a *comb* of very narrow lines (the *teeth*) equally spaced by a frequency interval $1/\tau$ (c).

Principles of Optical Frequency Combs

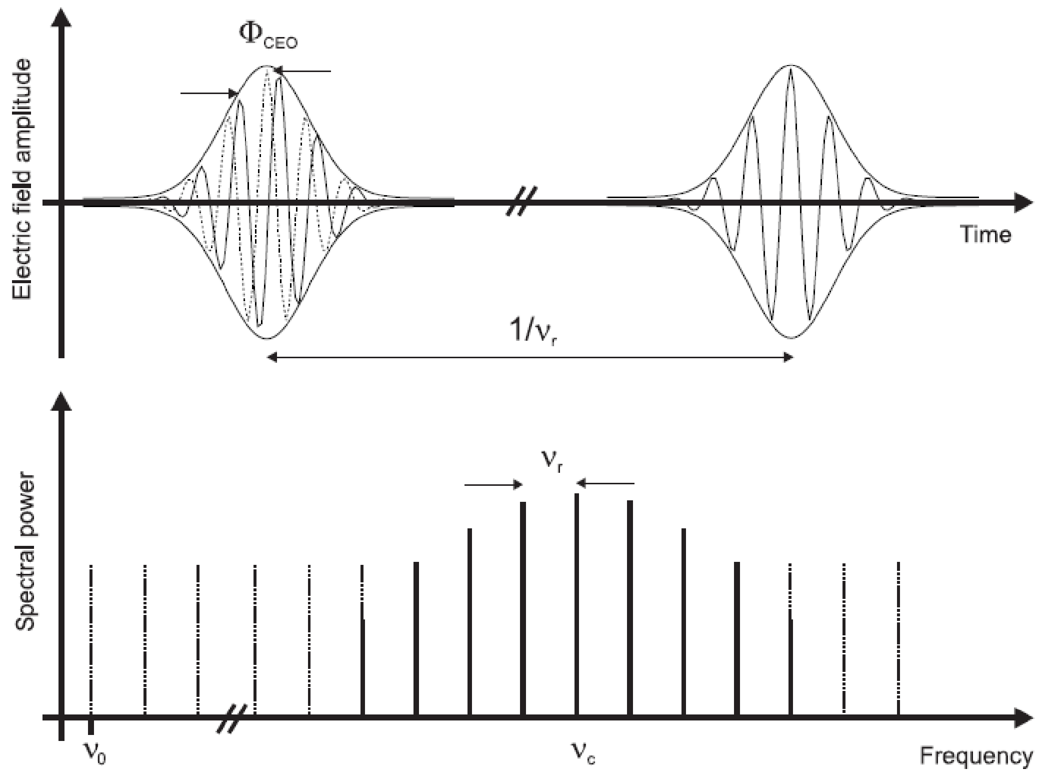
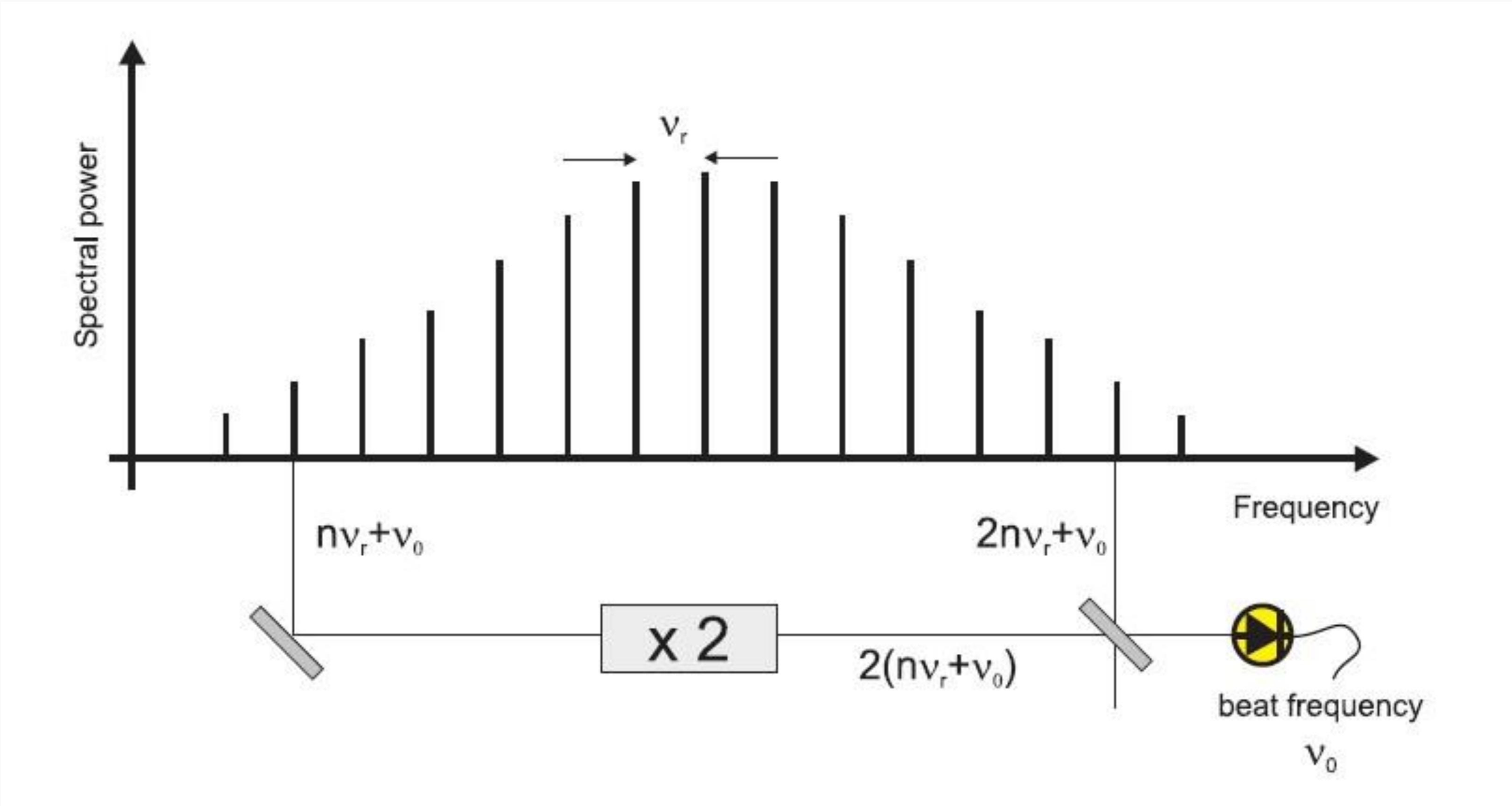


FIGURE 6.6
Scheme of a series of ultrashort laser pulses emitted from a mode-locked laser. Successive pulses are exact replicas of each other apart from a phase factor ϕ_{CEO}. This translates in the frequency domain to a comb-like structure of narrow spectral modes separated by the laser repetition frequency $\nu_r = 1/\tau_r$ and with an offset from zero frequency given by ν_0.

Principles of Optical Frequency Combs

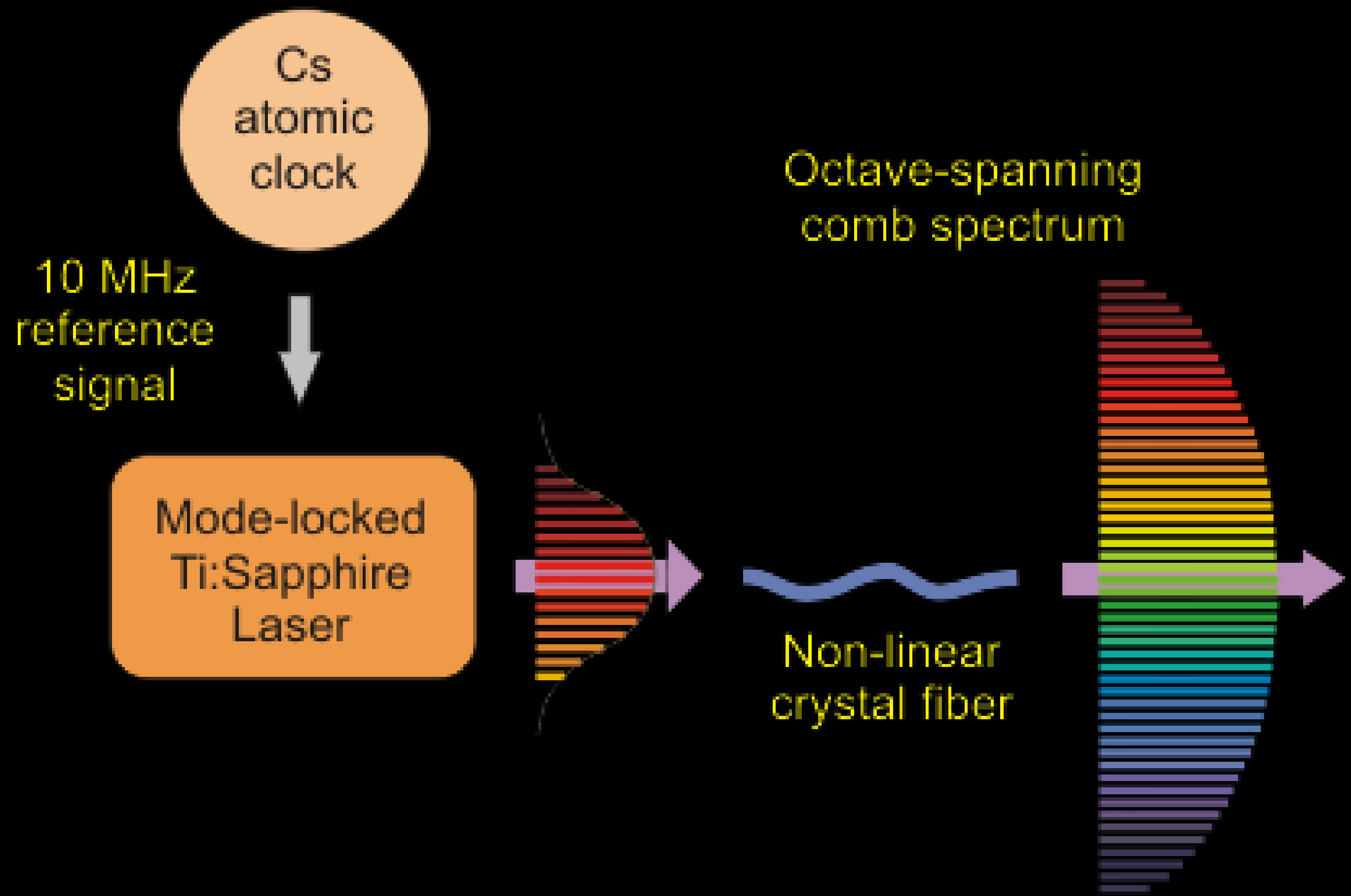


Born for Precise Frequency Measurements

for a review see, e.g.:

Optical comb generators for laser frequency measurement

Maddaloni et al. Meas. Sci. Technol. **20 (2009) 052001**



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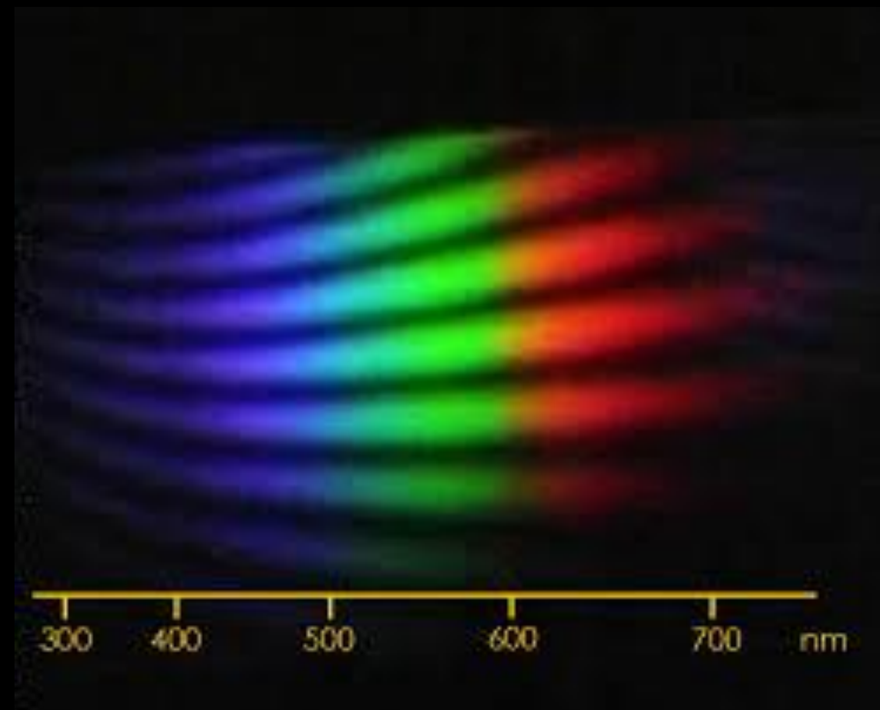
Istituto Nazionale di Ottica

The first evidence of comb behaviour



Experiment performed at LENS, *Firenze, Italy in1998*

July 15, 2000 / Vol. 25, No. 14 / OPTICS LETTERS 1049

Phase-locked white-light continuum pulses: toward a universal optical frequency-comb synthesizer

Marco Bellini

Istituto Nazionale di Ottica Applicata, Largo E. Fermi 6, 50125 Florence, Italy, and European Laboratory for Non Linear Spectroscopy and Istituto Nazionale di Fisica della Materia, Largo E. Fermi 2, 50125 Florence, Italy

Theodor W. Hänsch

European Laboratory for Non Linear Spectroscopy, Istituto Nazionale di Fisica della Materia, and Department of Physics, University of Florence, Largo E. Fermi 2, 50125 Florence, Italy, and Max-Planck-Institut für Quantenoptik, P.O. Box 1513, D-85740 Garching, Germany

Received January 31, 2000

We demonstrate that two white-light continuum pulses that are independently generated by phase-locked ultrashort laser pulses are locked in phase and show surprisingly clear and stable Young interference fringes. The experiment shows that the two generated continua emit essentially in phase and that random phase jitter can remain negligible. This result is not only of interest for studies of nonlinear field–matter interactions but also suggests that such white-light continuum pulses can be used to realize a broad frequency comb for absolute frequency measurements from the IR to the UV.

OCIS codes: 190.0190, 190.2620, 190.5940, 300.6320, 320.7110.

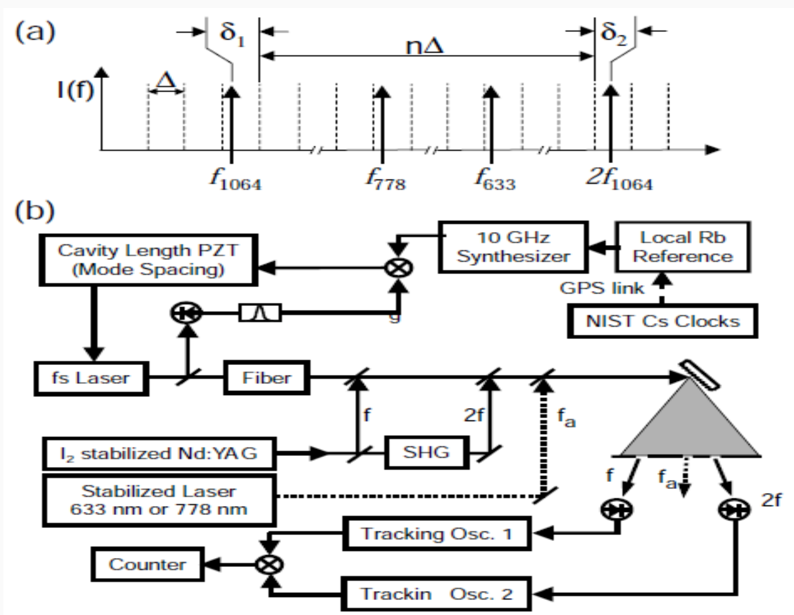
Direct Link between Microwave and Optical Frequencies with a 300 THz Femtosecond Laser Comb

Scott A. Diddams,* David J. Jones, Jun Ye, Steven T. Cundiff, and John L. Hall†
JILA, University of Colorado, and National Institute of Standards and Technology, Boulder, Colorado 80309

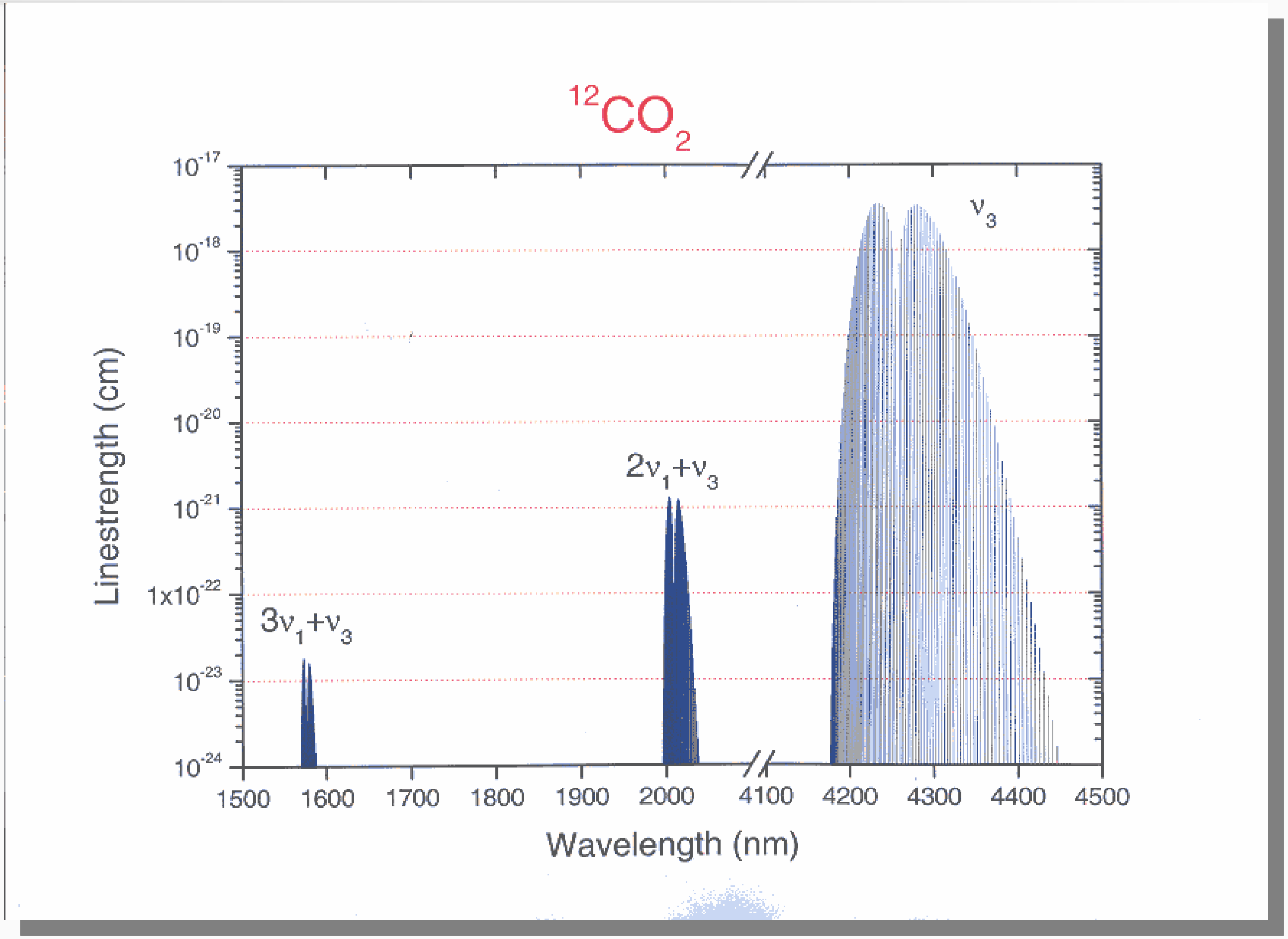
Jinendra K. Ranka and Robert S. Windeler
Bell Laboratories, Lucent Technologies, 700 Mountain Avenue, Murray Hill, New Jersey 070974

Ronald Holzwarth, Thomas Udem, and T. W. Hänsch
Max-Planck-Institut für Quantenoptik, 85748 Garching, Germany
(Received 17 February 2000)

We demonstrate a great simplification in the long-standing problem of measuring optical frequencies in terms of the cesium primary standard. An air-silica microstructure optical fiber broadens the frequency comb of a femtosecond laser to span the optical octave from 1064 to 532 nm, enabling us to measure the 282 THz frequency of an iodine-stabilized Nd:YAG laser directly in terms of the microwave frequency that controls the comb spacing. Additional measurements of established optical frequencies at 633 and 778 nm using the same femtosecond comb confirm the accepted uncertainties for these standards.



Intensity Scaling of Molecular Absorption



…..and natural combs

1256 OPTICS LETTERS / Vol. 27, No. 14 / July 15, 2002

Low-power Lamb-dip spectroscopy of very weak CO2 transitions near 4.25 μm

IEEE JOURNAL OF QUANTUM ELECTRONICS,
VOL. 29, NO. 10, OCTOBER 2693 (1993)

Wide-Span Optical Frequency Comb Generator for Accurate Optical Frequency Difference Measurement

Motonobu Kourogi, Ken'ichi Nakagawa and Motoichi Ohtsu,

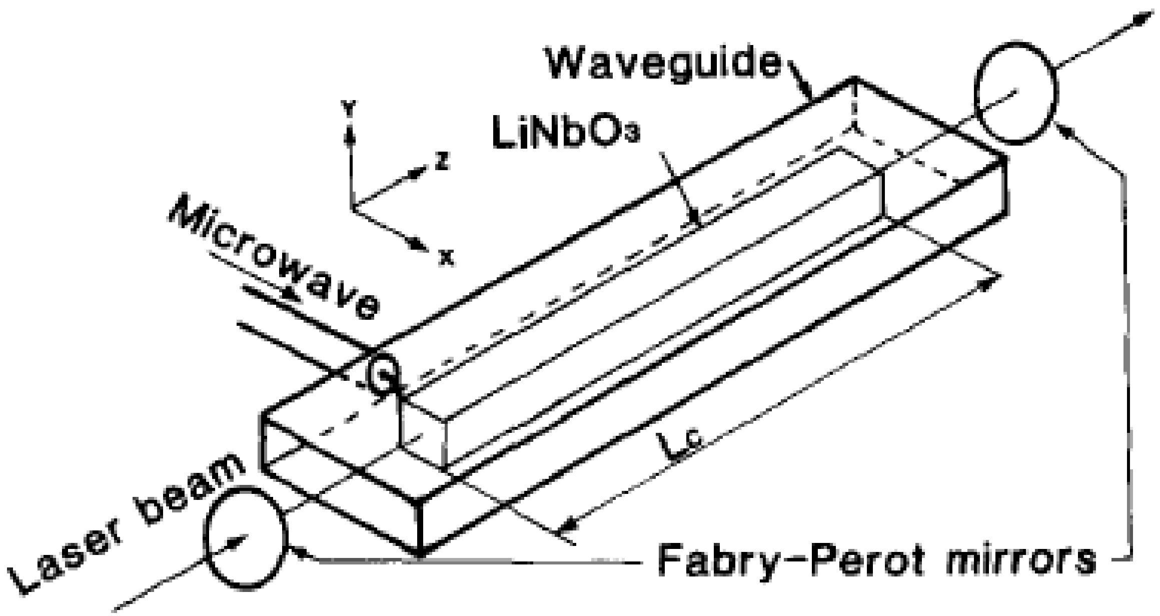
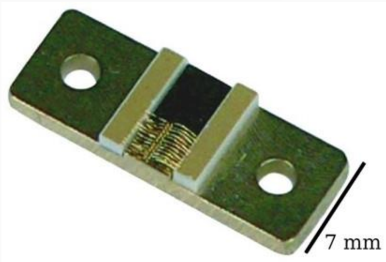


Fig. 2. The construction of the present optical frequency comb generator.

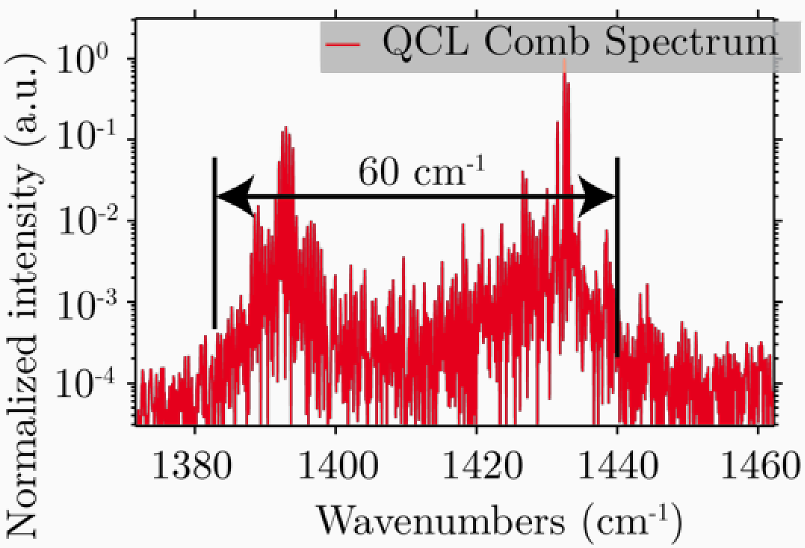
Quantum cascade laser frequency combs

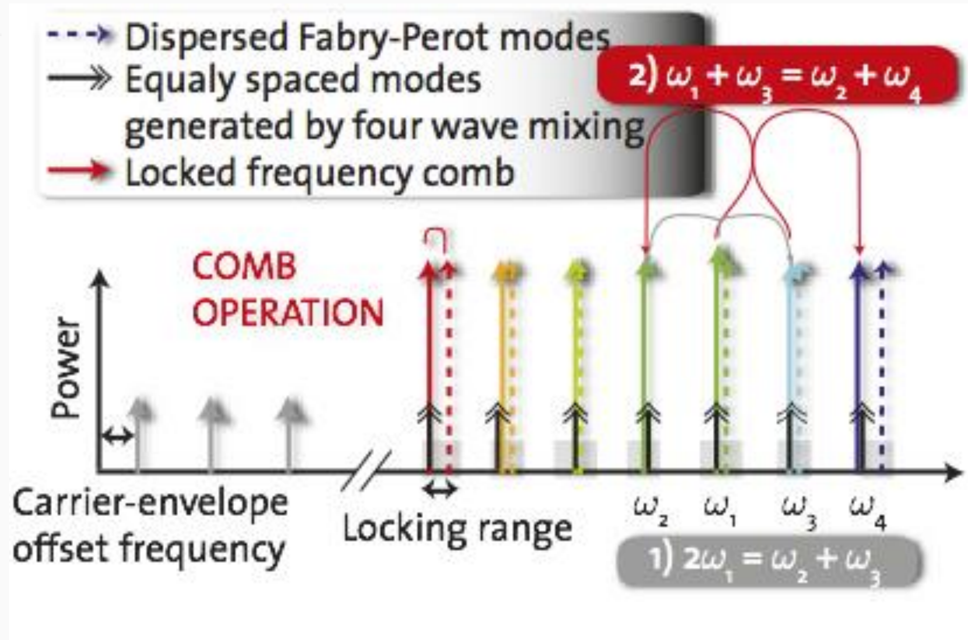


LETTER
doi:10.1038/nature11620

Mid-infrared frequency comb based on a quantum cascade laser

Andreas Hugi[1], Gustavo Villares[1], Stéphane Blaser[2], H. C. Liu[3] & Jérôme Faist[1]





NL $\quad {}^{(3)} E^3$

Hugi et al., Mid-infrared frequency comb based on a quantum cascade laser, Nature 492, 229-233 (2012)

Quantum cascade laser frequency combs

ARTICLES
PUBLISHED ONLINE: 11 MAY 2014 | DOI: 10.1038/NPHOTON.2014.85
nature photonics

Terahertz laser frequency combs

David Burghoff[1]*, Tsung-Yu Kao[1], Ningren Han[1], Chun Wang Ivan Chan[1], Xiaowei Cai[1], Yang Yang[1], Darren J. Hayton[2], Jian-Rong Gao[2,3], John L. Reno[4] and Qing Hu[1]

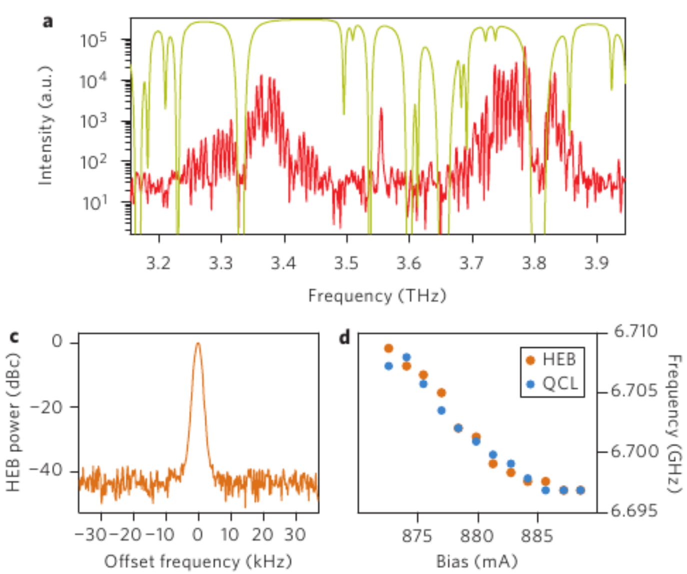
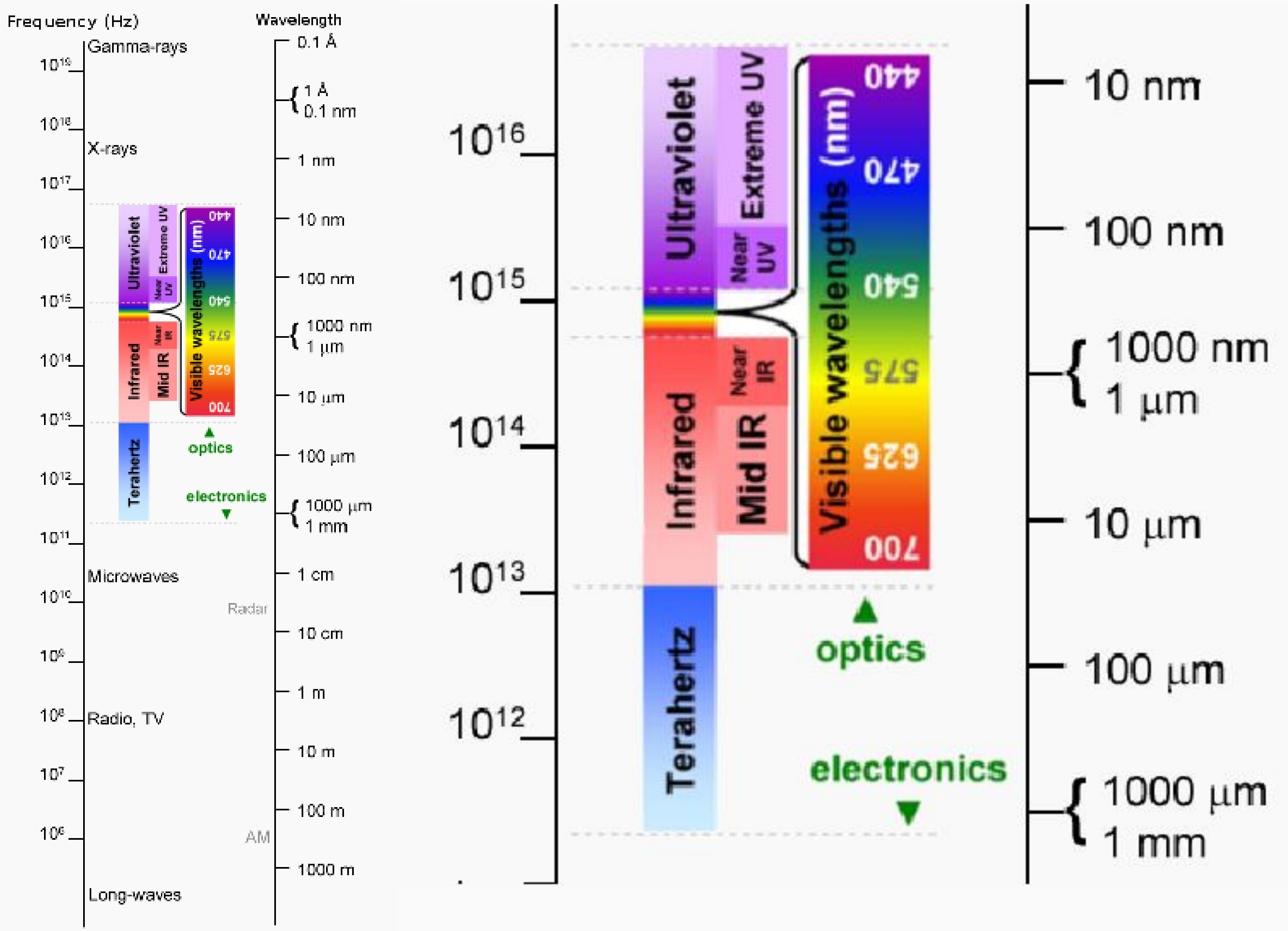


Figure **Continuous-wave spectrum and beat notes. a**, Spectrum of a THz QCL comb biased to 0.9 A at a temperature of 50 K. The device is 20 µm wide, 5 mm long and emits 4.7 mW at 45 K. Atmospheric absorption is shown in yellow. **c**, HEB-detected beat-note offset relative to 6.80 GHz, stabilized. The linewidth of 1.53 kHz is limited by the instrument resolution. **d**, Bias dependence of the beat-note frequency measured by the QCL and by the HEB. A repetition rate tuning of 12 MHz is possible, giving a total frequency shift of 6.80 GHz at 3.8 THz, approximately the mode spacing.

Burghoff et al., Terahertz laser frequency combs, Nature Photon. 8, 462-467 (2014)

Electromagnetic Spectrum

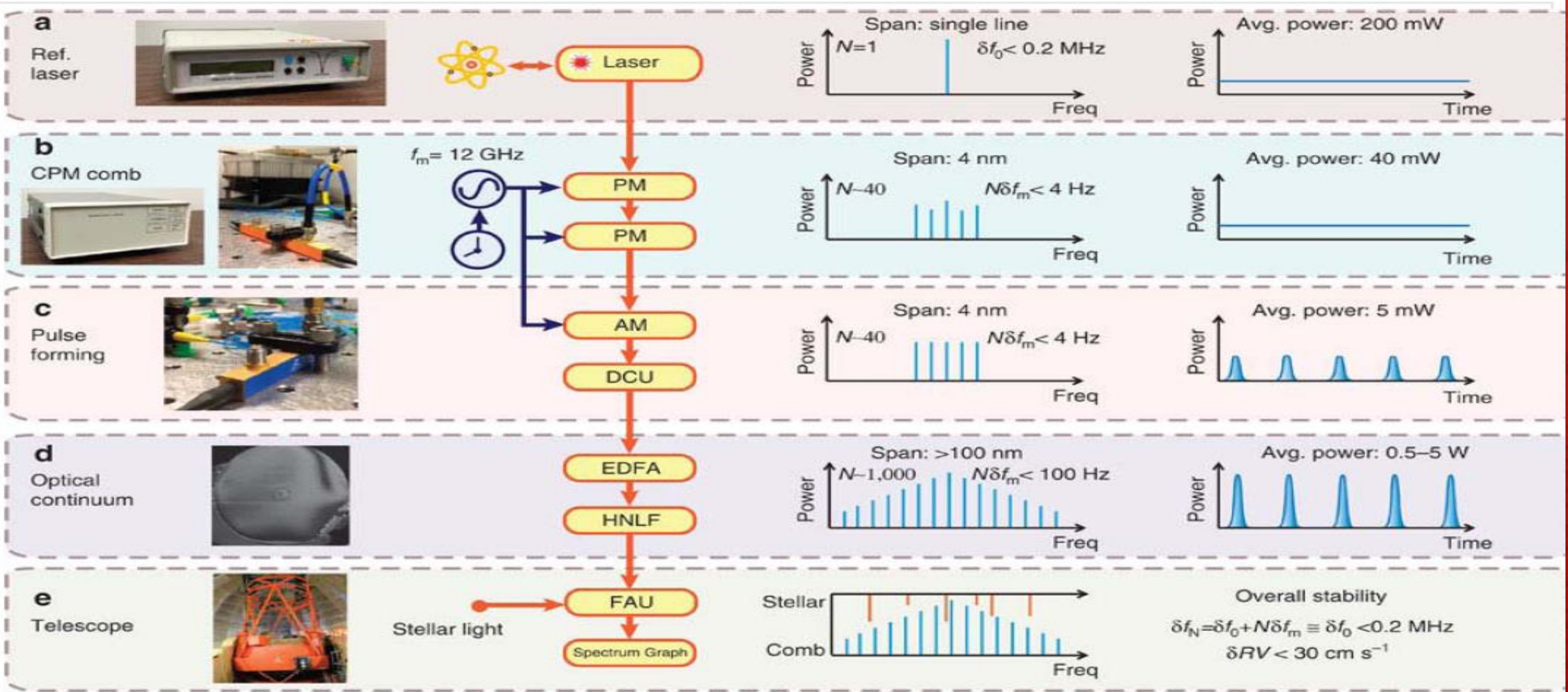


From

Demonstration of a near-IR line-referenced electro-optical laser frequency comb for precision radial velocity measurements in astronomy

X. Yi, K. Vahala, J. Li, S. Diddams, G. Ycas, P. Plavchan, S. Leifer, J. Sandhu, G. Vasisht, P. Chen, P. Gao, J. Gagne, E. Furlan, M. Bottom, E. C. Martin, M. P. Fitzgerald, G. Doppmann & C. Beichman

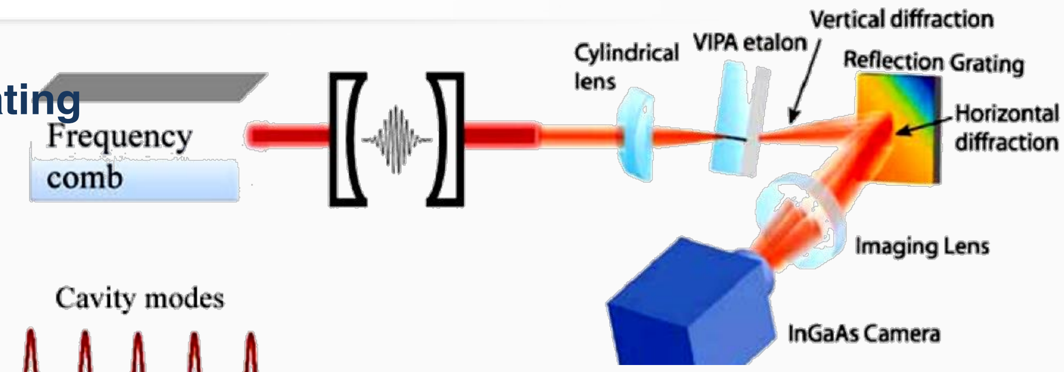
Nature Communications **7**, Article number: 10436 doi:10.1038/ncomms10436

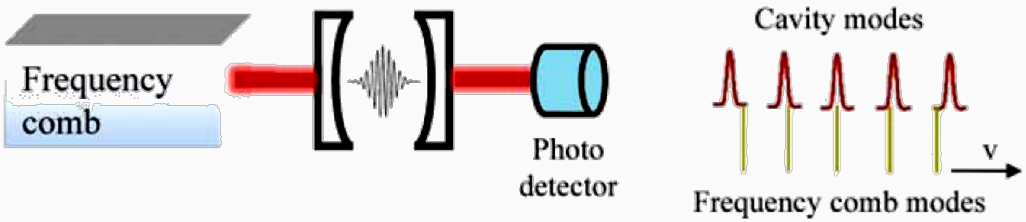


Vertically, the first column contains images of key instruments. (**a–e**) The images are reference laser, Rb clock (left) and phase modulator (right), amplitude modulator, highly nonlinear fibre and telescope. A simplified schematic set-up is in the second column. Third and fourth columns present the comb state in the frequency and temporal domains. The frequency of N-th comb tooth is expressed as $f_N=f_0+N \times f_m$, where f_0 and f_m are the reference laser frequency and modulation frequency, respectively. N is the number of comb lines relative to the reference laser (taken as comb line N=0), RV is radial velocity and δf_N, δf_0 and δf_m are the variance of f_N, f_0 and f_m. (**a**) The reference laser is locked to a molecular transition, acquiring stability of 0.2 MHz, corresponding to 30 cm s^{-1} RV. (**b**) Cascaded phase modulation (CPM) comb: the phase of the reference laser is modulated by two phase modulators (PM), creating several tens of sidebands with spacing equal to the modulation frequency. The RF frequency generator is referenced to a Rb clock, providing stability at the sub-Hz level (δf_m<0.03 Hz at 100 s). (**c**) Pulse forming is then performed by an amplitude modulator (AM) and dispersion compensation unit (DCU), which could be a long single mode fibre (SMF) or chirped fibre Bragg grating (FBG). (**d**) After amplification by an erbium-doped fibre amplifier (EDFA), the pulse undergoes optical continuum broadening in a highly nonlinear fibre (HNLF), extending its bandwidth >100 nm. (**e**) Finally the comb light is combined with stellar light using a fibre acquisition unit (FAU) and is sent into the telescope spectrograph. The overall comb stability is primarily determined by the pump laser.

Direct Comb Spectroscopy

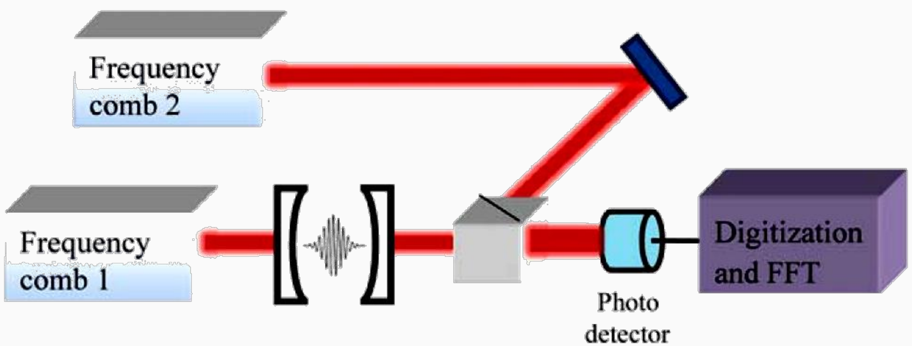
Comb Dispersion by VIPA and Grating





Comb Vernier Spectroscopy

Fourier-Transform Analisys



Dual Comb Spectroscopy

A. Marian, M. C. Stowe, J. R. Lawall, D. Felinto, and J. Ye, Science **306**, 2063 (2004)

S. A. Diddams, L. Hollberg, and V. Mbele, Nature **445**, 627 (2007)

Advanced Optical Fiber-based spectrometers

- Broad Absorptions in Liquids
- Strain (DL/L)
- Temperature variations

Combs for spectroscopy in liquids

201116-2 Avino *et al.* Appl. Phys. Lett. **102**, 201116 (2013)

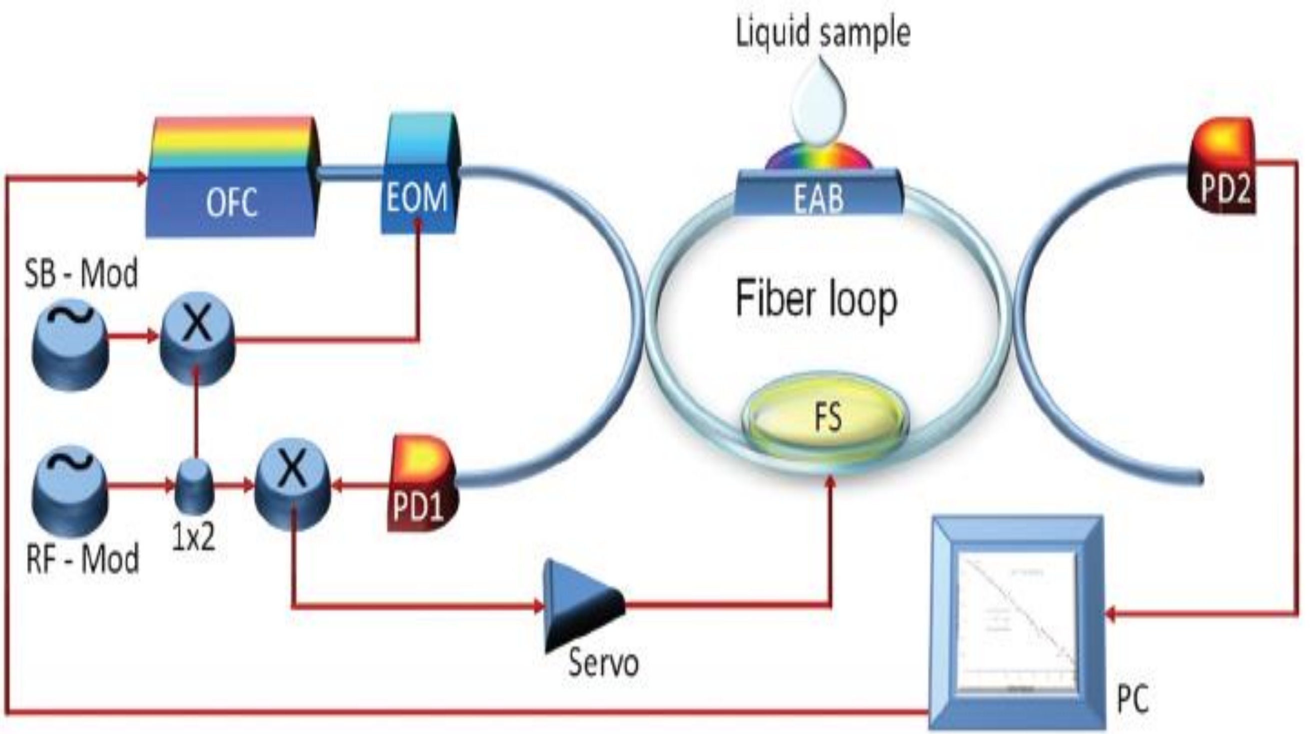
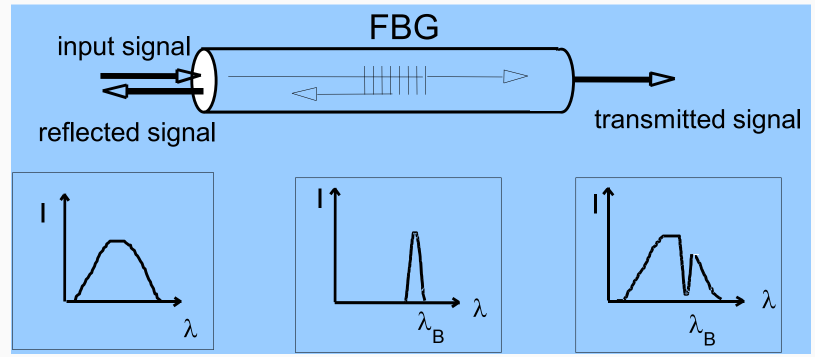


FIG. 1. Experimental layout. OFC: optical frequency comb; EOM: electro-optic phase modulator; SB – Mod: phase modulation to switch the beam off; RF – Mod: phase modulation for PDH locking. PD: photodiode; PC: personal computer; Servo: locking electronics. EAB: evanescent-wave access block; FS: fiber stretcher.

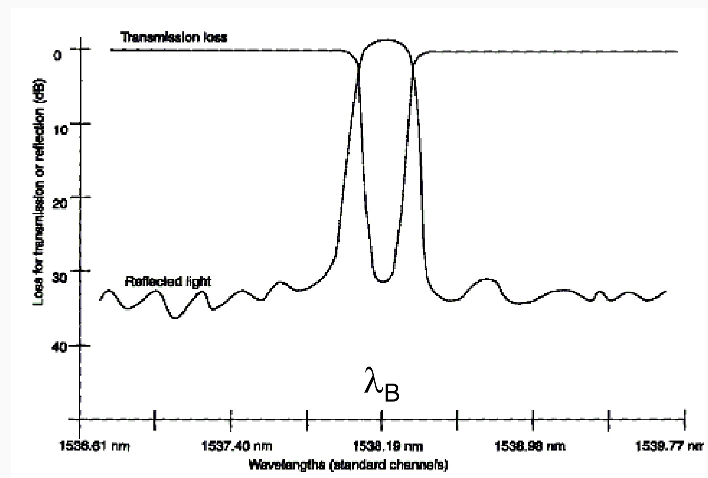
Operating Principle of FBGs

Periodic modulation
of the refractive index
in the core of an optical fiber



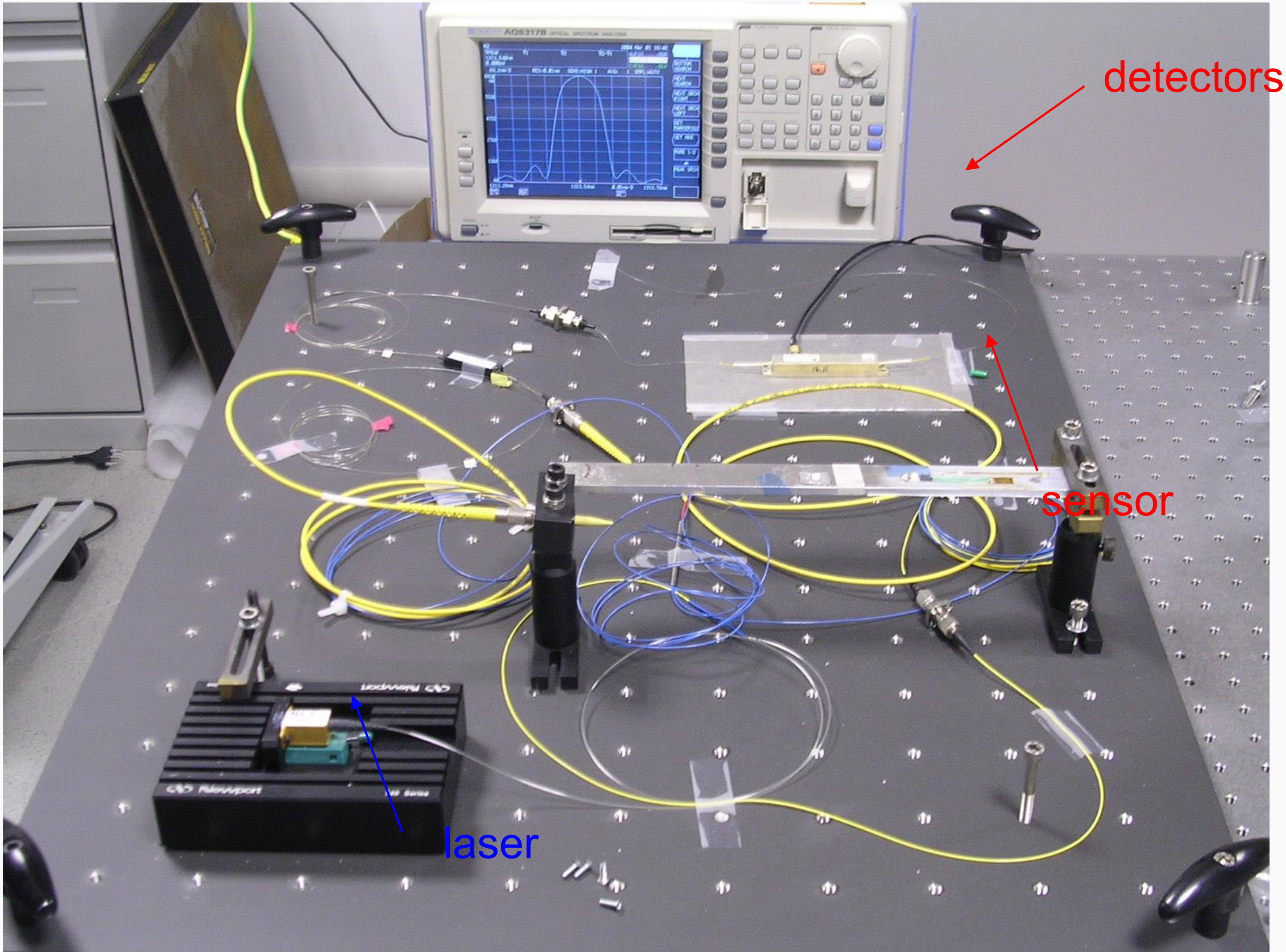
Bragg
max reflectivity

$$\lambda_B = 2n_{eff}\Lambda$$



Any change in the grating pitch and/or refractive index results in ($\Delta L/L=10^{-6} \Rightarrow 1\ \mu\varepsilon$) **a shift of λ_B ~ 10^{-3} nm/$\mu\varepsilon$**

Set-up for Deformation Sensor



FBG-based sensors

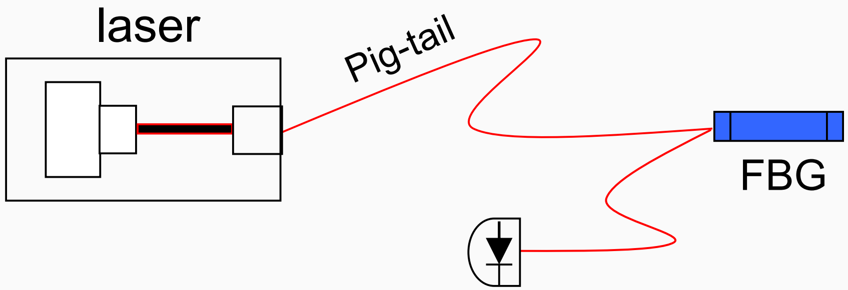
Capabilities:

- static and dynamic point strain measurement;
- field and remote operation;
- immunity to EMI;
- embedding in materials;
- monitoring networks;
- low weight and cost;
- application as field sensors in several areas.

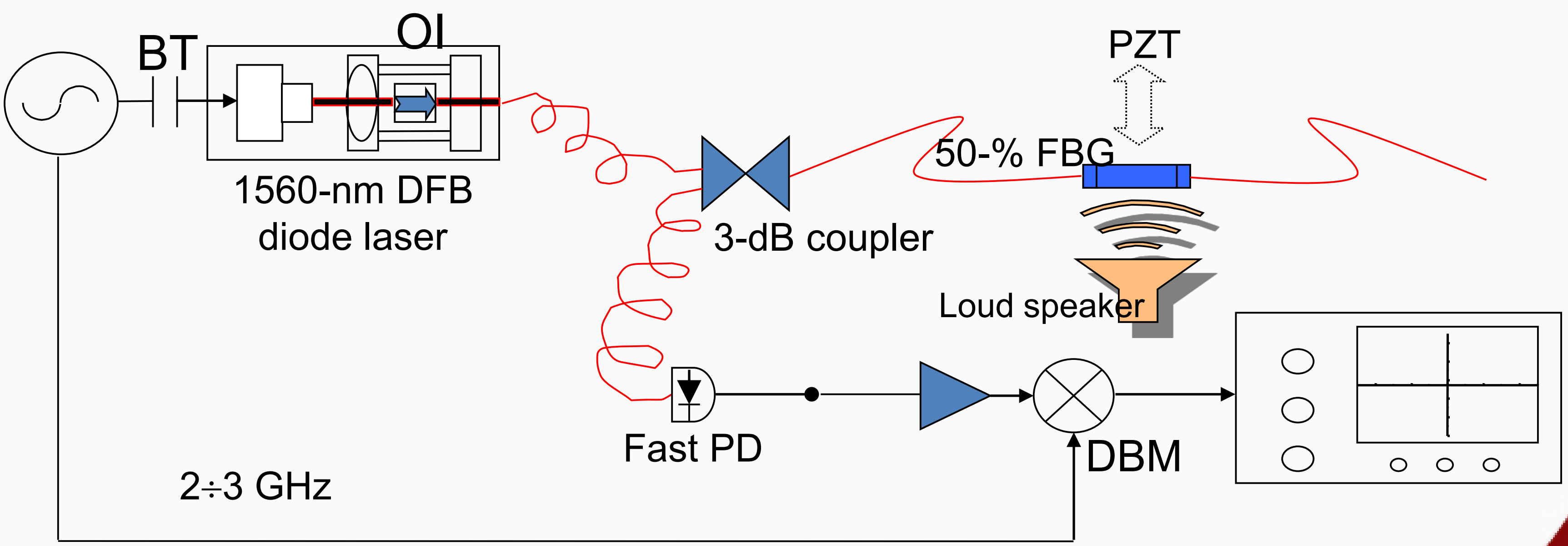
Conventional interrogation techniques:

- broad-band sources combined to passive or active filters;
- limited sensitivity;
- frequency response.

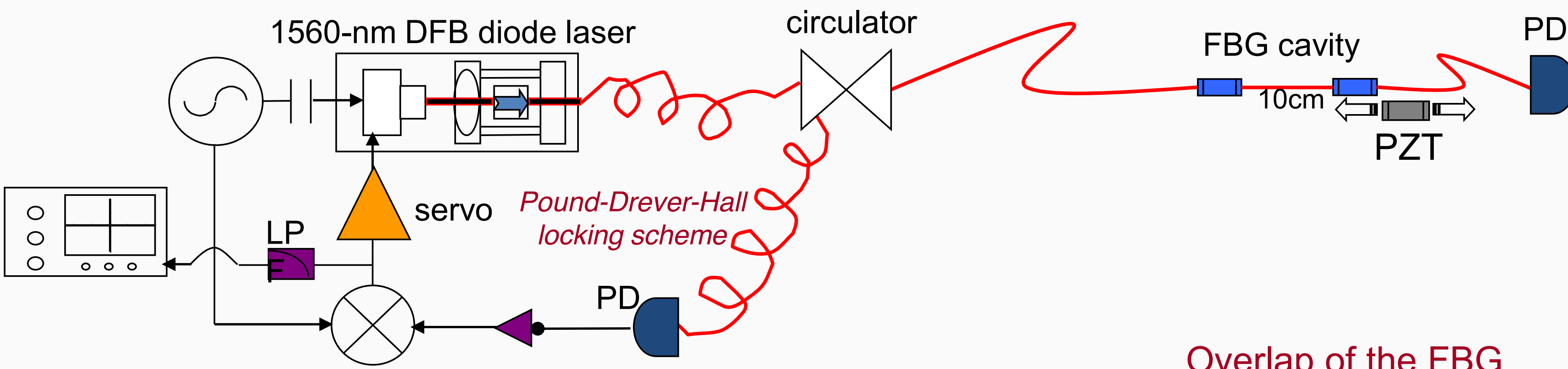
Interrogation of FBGs using a laser source

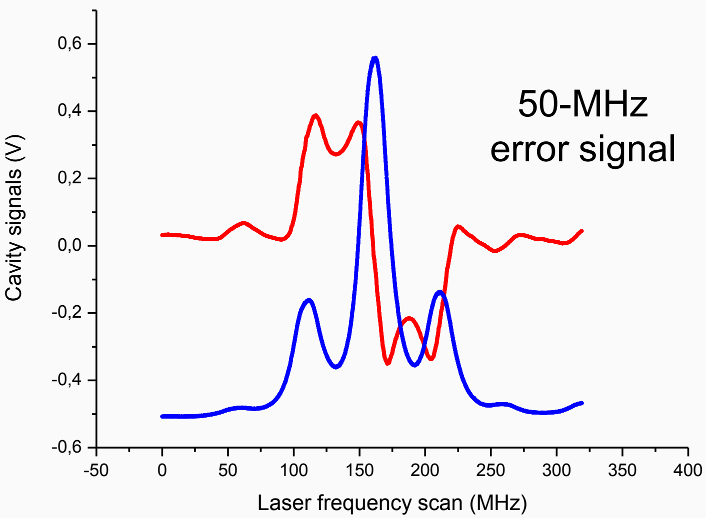


FM-based Bragg-grating set-up



FBG-based Fabry-Pérot strain sensor

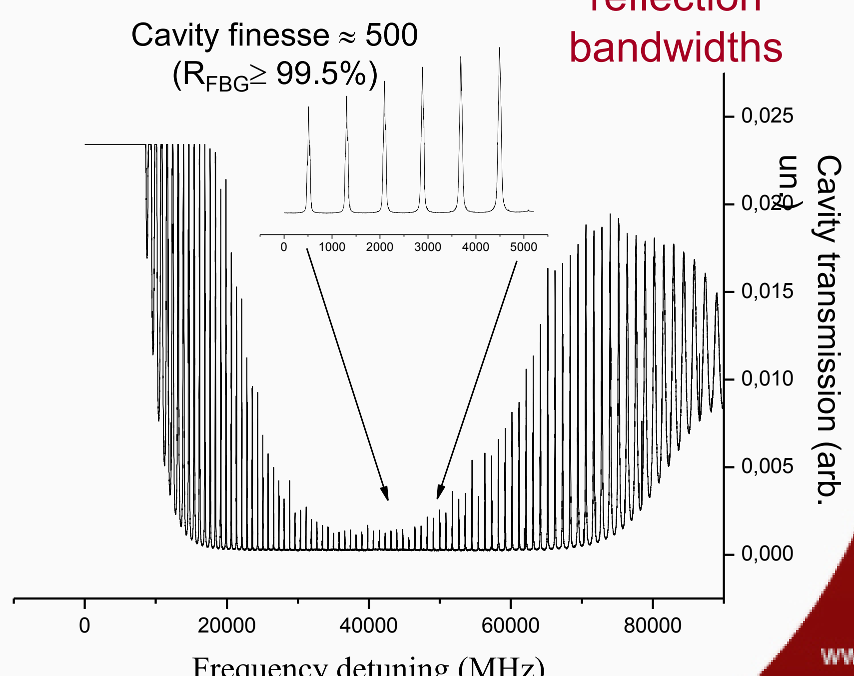




50-MHz error signal

Strain monitor

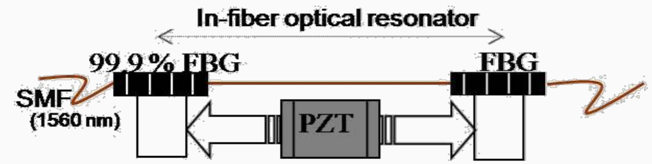
Overlap of the FBG reflection bandwidths



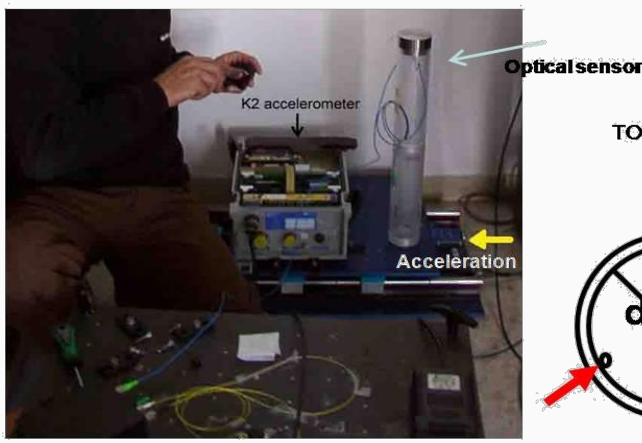
Fiber Optical Sensors

High-sensitivity strain sensing apparatus

•High-sensitivity laser-based strain and temperature sensing using "fiber Bragg grating" (FBG) resonators: interrogation of fiber optic sensors by highly-stabilized lasers

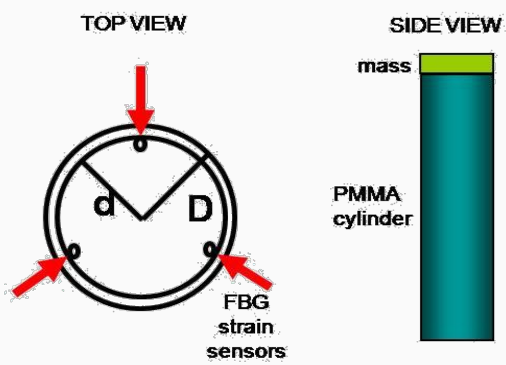


Optical-frequency combs for laser frequency stabilization → Strain meas towards the 10^{-14} level

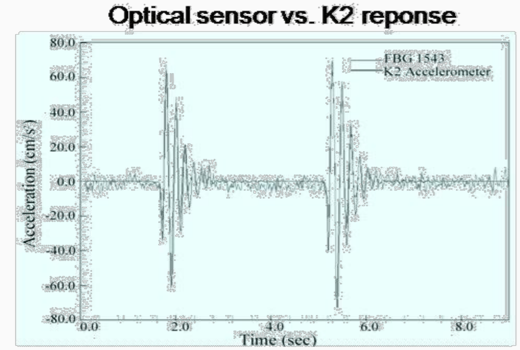


FBG flexural beam accelerometer: comparison with a commercial K2 seismometer on a shaking table

•Seismometers based on FBG technology: spectroscopic interrogation by frequency-modulated telecom diode lasers



Optical sensor vs. K2 reponse



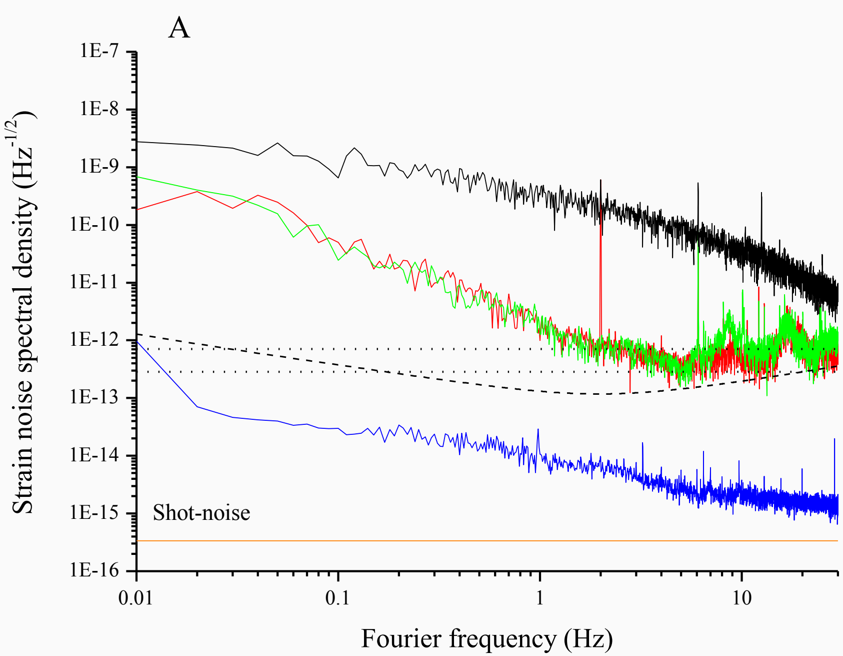
Italian patent : No. RM2007A000589 (2007).

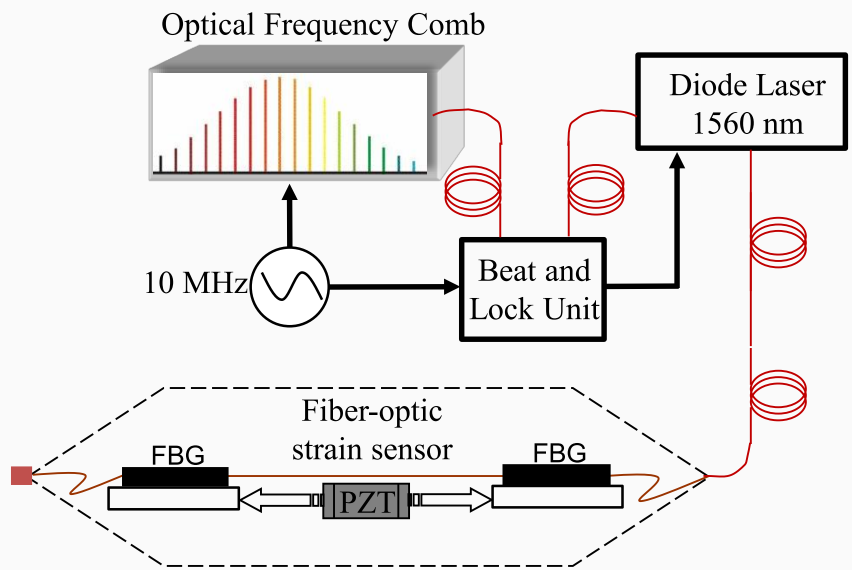
Comb-assisted strain sensing

Probing the Ultimate Limit of Fiber-Optic Strain Sensing

G. Gagliardi,[1*] M. Salza,[1] S. Avino,[1] P. Ferraro,[1] P. De Natale[2]

Science AAAS
SCIENCE VOL 330 19 NOVEMBER 2010



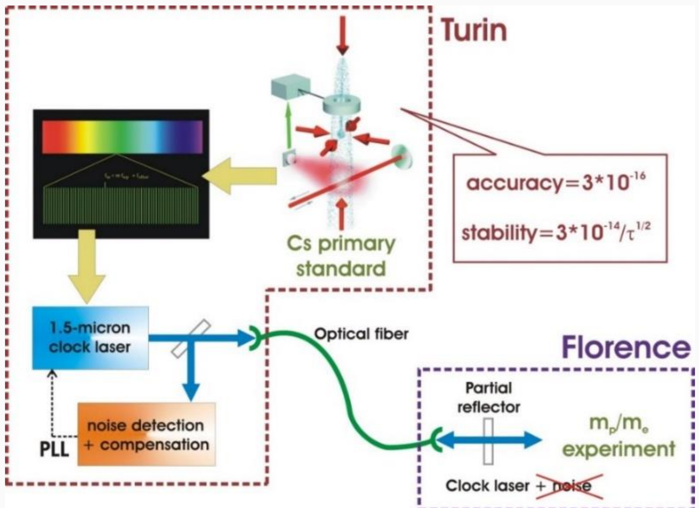


Thermodynamic noise limited strain resolution ~ 900-350 fε/√Hz in the infrasonic range

NATIONAL OPTICAL FIBER LINK

GPS limits the comb accuracy to 5x10^{-12}

□ improved primary standard



Istituto Nazionale di Ricerca Metrologica

A 1.5-micron laser, continuosly referenced to the primary Cs fountain via an OFC, is disseminated through a long optical fiber. Part of the laser radiation is reflected back and used to cancel the phase noise accumulated during the fiber propagation

Accuracy=3·10^{-16} **Stability=3·10^{-14} /τ $^{1/2}$**

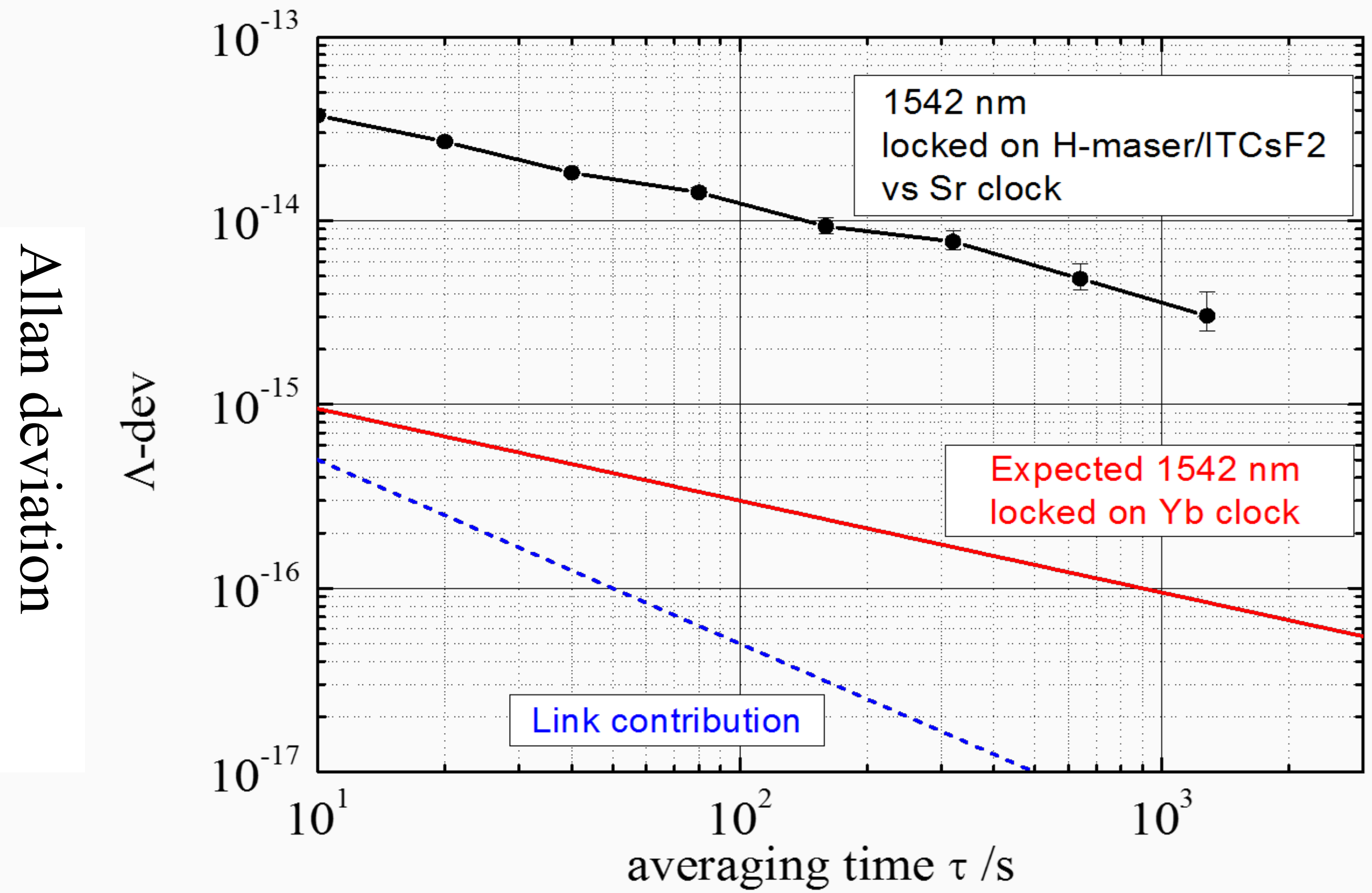
📖 D. Calonico et al., Appl. Phys. B **117**, 979 (2014)

Pushing the ultimate resolution in the spectroscopic frequency measurement down to 10^{-18} by stabilizing the frequency comb, to which the probe laser is referenced, against an optical atomic standard

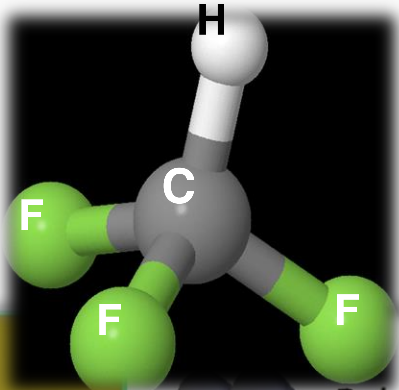
□ Yb lattice clock under construction at INRIM

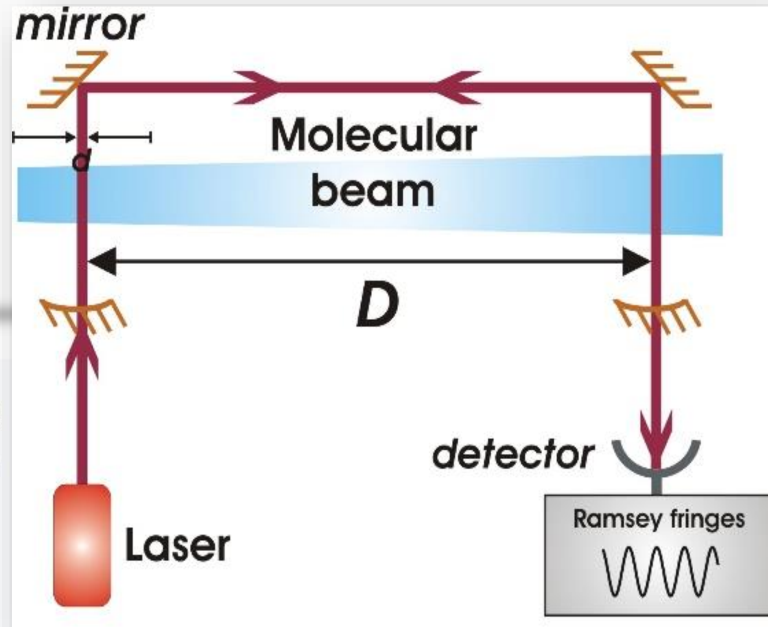
$$\frac{1}{\frac{\nu_{vib}(M)}{\nu_{el}(Yb)}}\frac{\partial\left[\frac{\nu_{vib}(M)}{\nu_{el}(Yb)}\right]}{\partial t} = -0.5\frac{\dot{\beta}}{\beta} - N_{Yb}\frac{\dot{\alpha}}{\alpha}$$

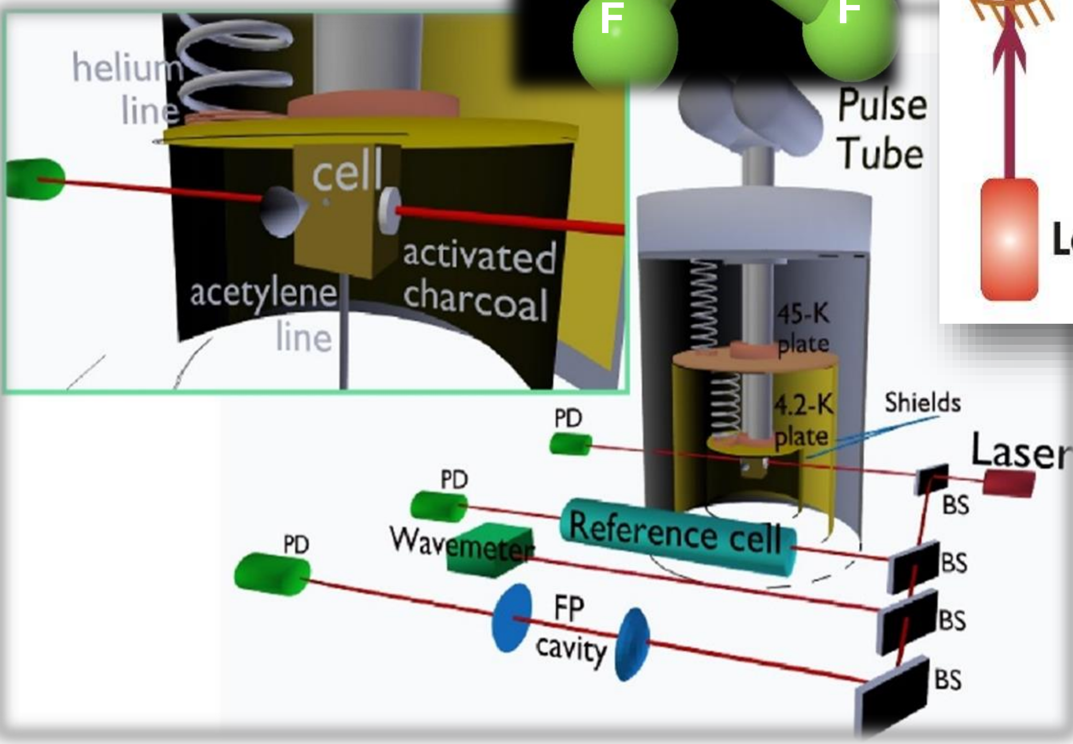
Stability transferred by the link (at 642 km distance, Turin-Florence, Italy)



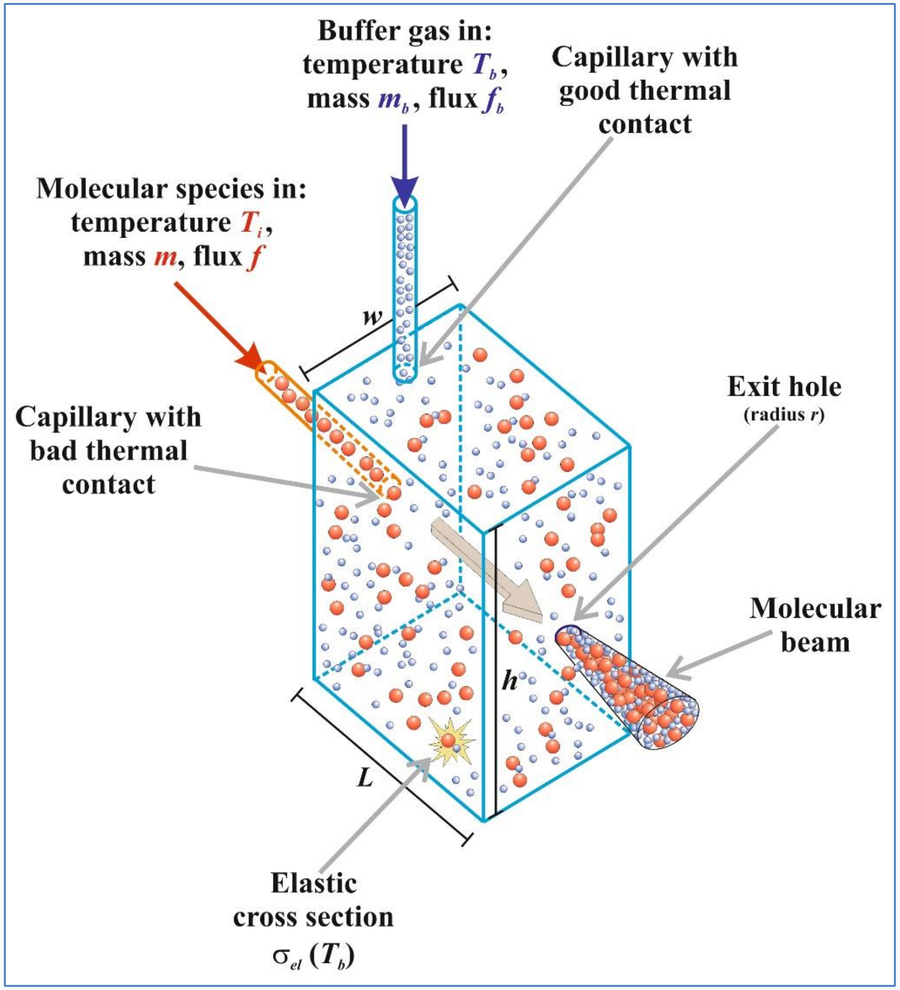
Cold Molecules







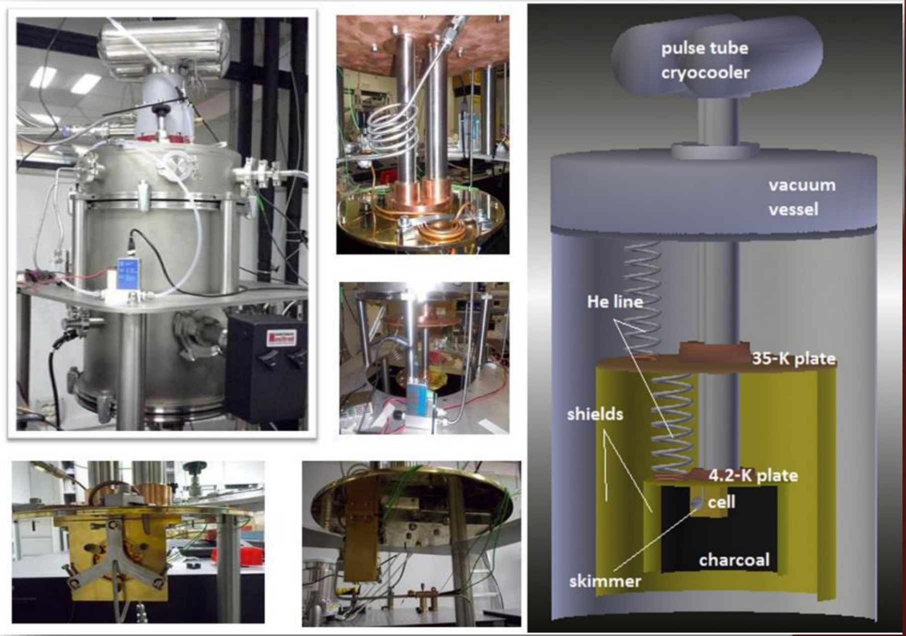
BUFFER GAS COOLING



Both translational and rotational (internal) degrees of freedom of the desired molecular species are cooled via collisions with a thermal bath of helium in a cryogenic cell

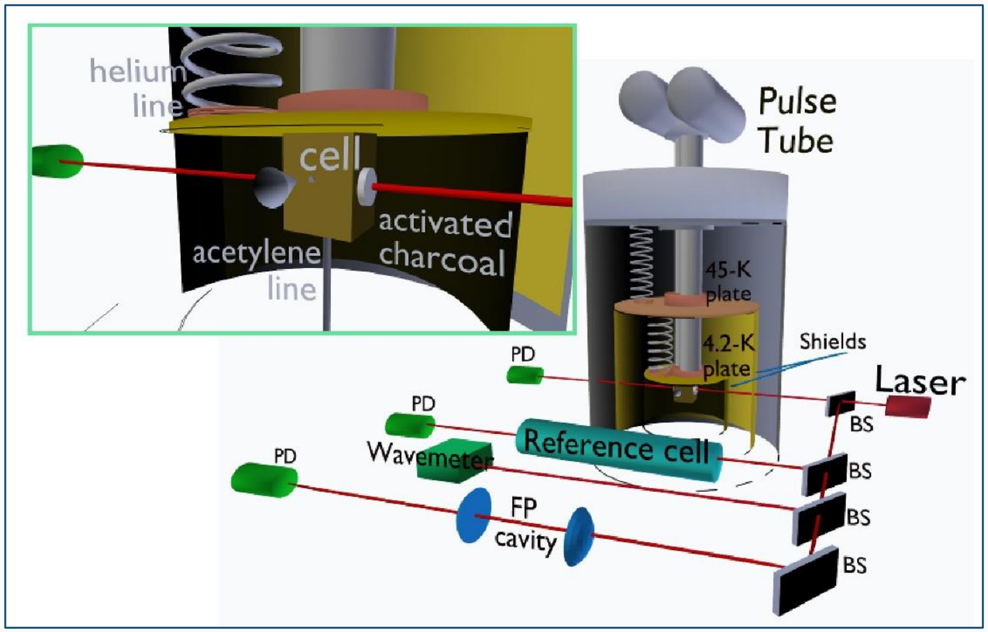
📖 S.E. Maxwell et al., Phys. Rev. Lett. **95**, 173201 (2005)

📖 N.R. Hutzler et al., Chem. Rev. **112**, 4803 (2012)



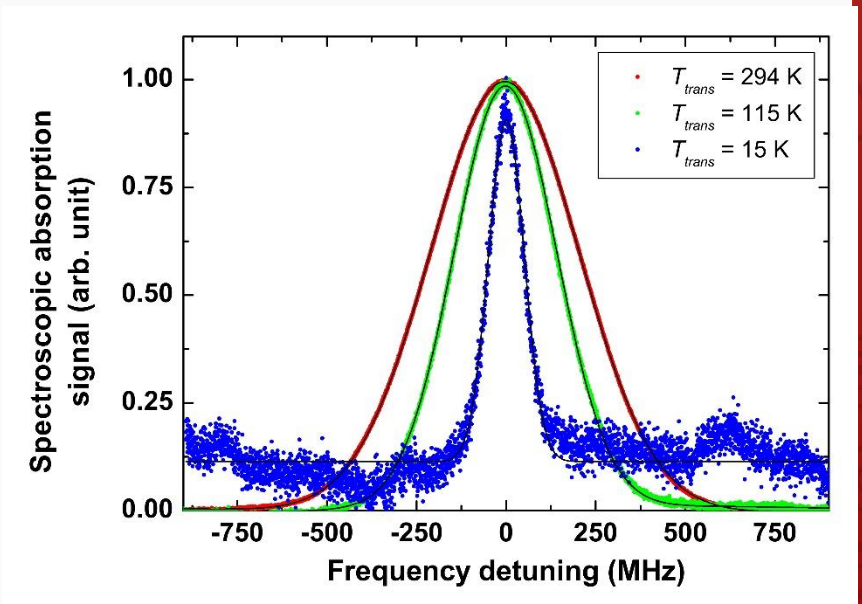
CHARACTERIZING THE COLLISIONAL COOLING PROCESS

Laser-absorption ro-vibrational spectroscopy of the $^{12}C_2H_2$ $(\upsilon_1 + \upsilon_3)$ band is performed



Due to a non perfect thermal exchange between the copper pipe and the two PT plates, a temperature of about 15 K is measured on the He line just before the entrance into the BGC cell (at 4.2 K); to bridge this gap, an improved setup for better cooling of the He line is under construction

Translational temperatures are extracted by Doppler thermometry



$$G(\nu) = G_0 \exp\left[-\frac{4\ln 2\,(\nu-\nu_0)^2}{\sigma_D^2}\right]$$

$$\sigma_D = \frac{\nu_0}{c}\sqrt{\frac{8\ln 2\, k_B T_{trans}}{m}}$$

📖 L. Santamaria et al., The Astrophysical Journal **801**:50 (2015)

MEASUREMENT OF TIME VARIATION OF CONSTANTS: A PROPOSAL

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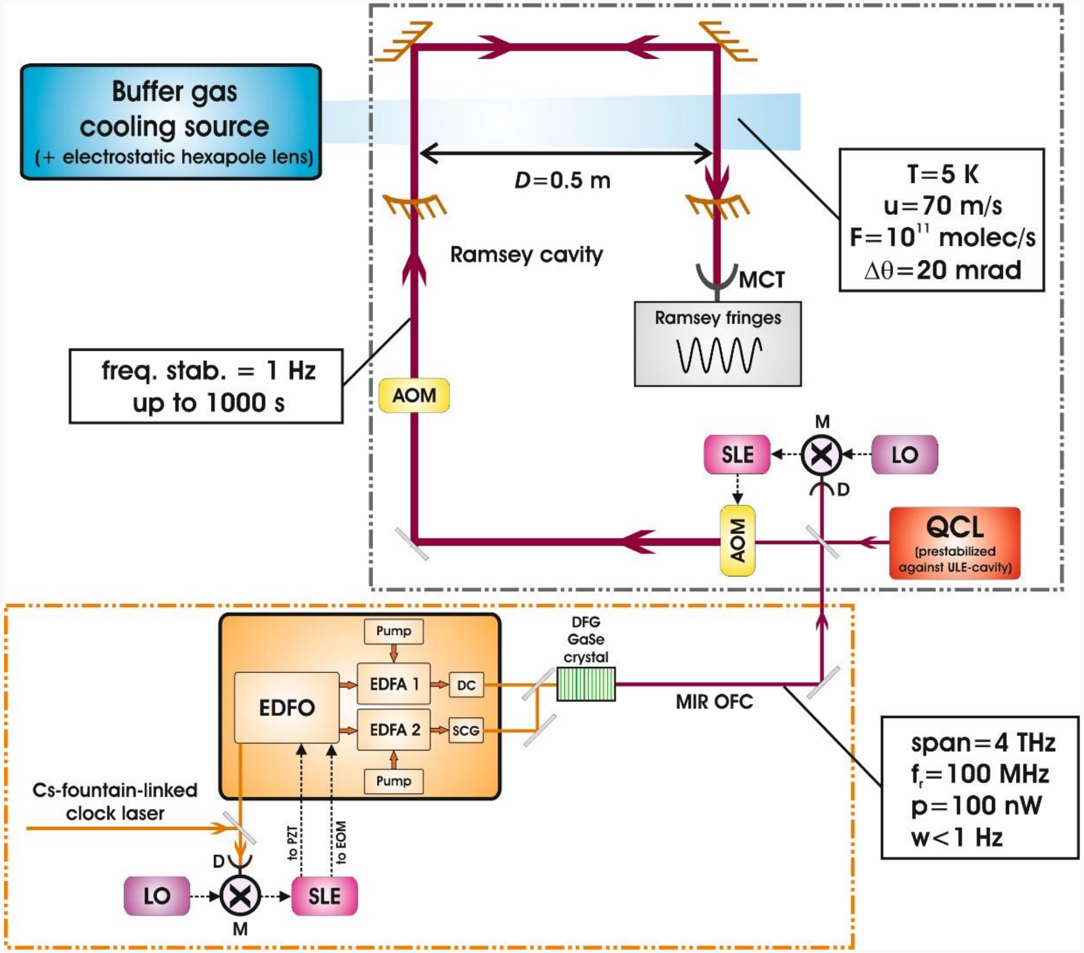


The cold fluoroform (CF_3H) beam is extracted from a buffer-gas-cooling source (and then collimated by means of an electrostatic hexapole lens). Employed in a 2-photon Ramsey-fringes interrogation, the probe source is a mid-infrared quantum cascade laser at 8.63 micron, phase-locked to a specially-developed optical frequency comb (OFC) that is ultimately referenced to the cesium primary standard via the National optical fiber link.

$$\mathcal{P} = \frac{u}{2D} = 35\ \text{Hz} \qquad SNR = 25,\ N_{avg} = 100$$

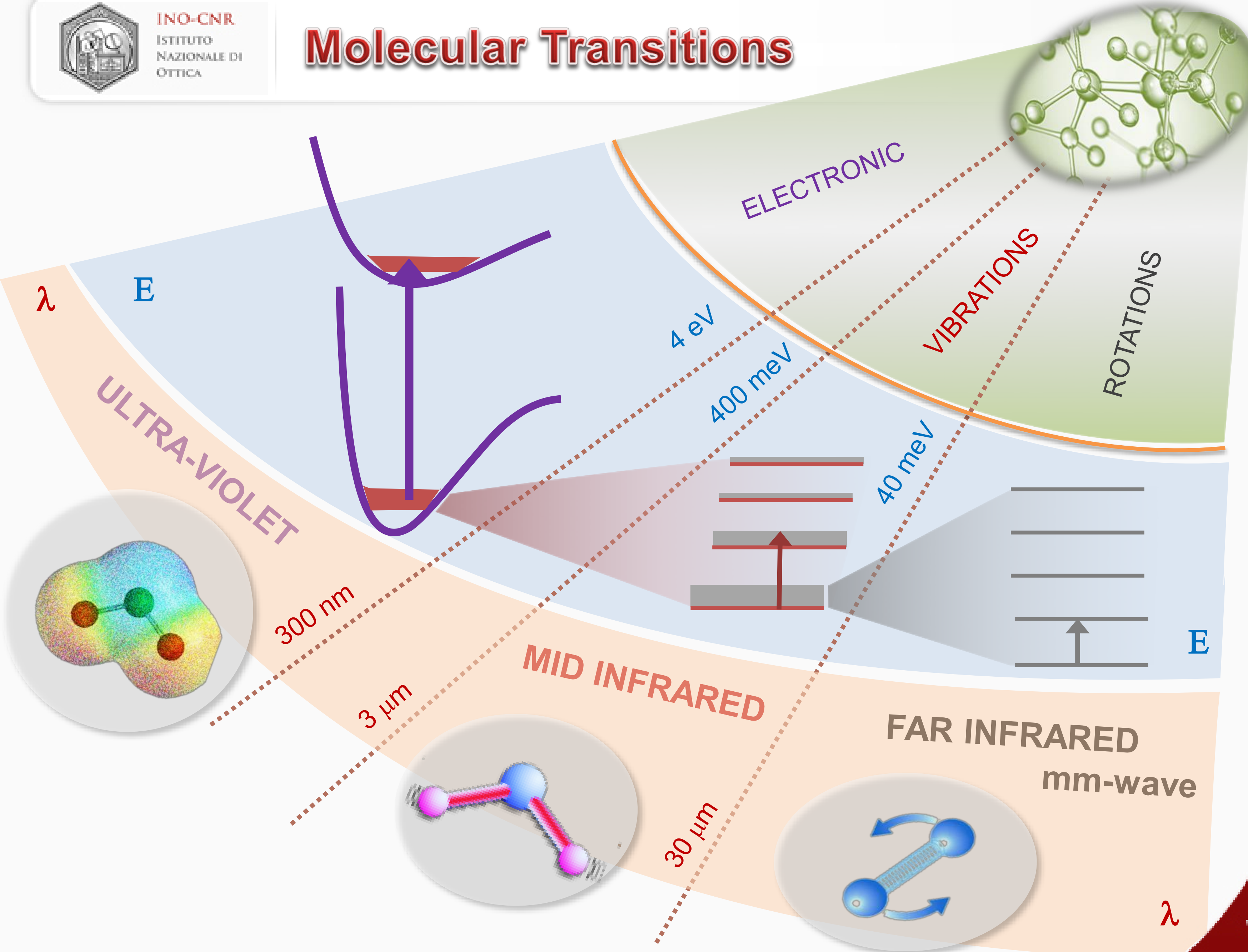
$$\mathcal{R} \sim \frac{\mathcal{P}}{SNR \cdot \sqrt{N_{avg}}} = 140\ \text{mHz}$$

⇩

$$\mathbf{4 \cdot 10^{-15}}$$

📖 L. Santamaria et al., J. Mol. Spectrosc. **300**, 116 (2014)

Molecular Transitions



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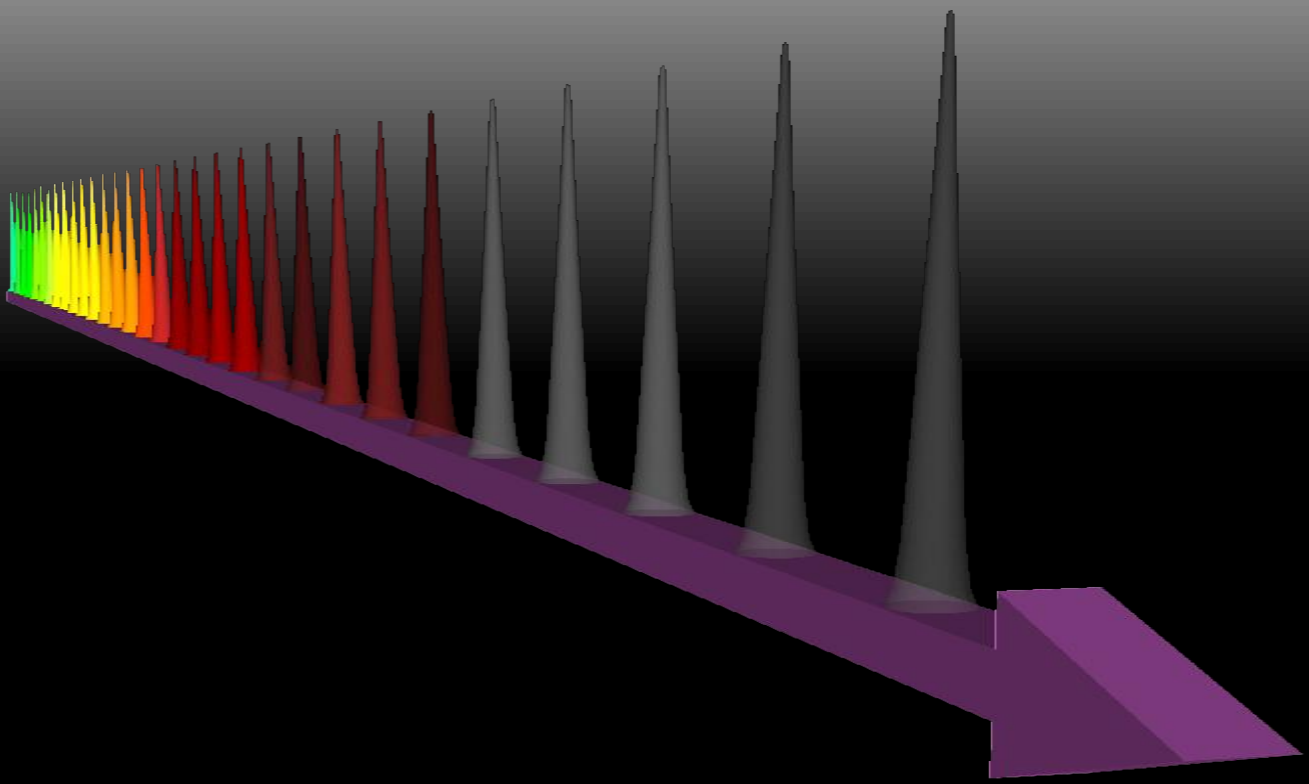
Moving deep into the Infrared

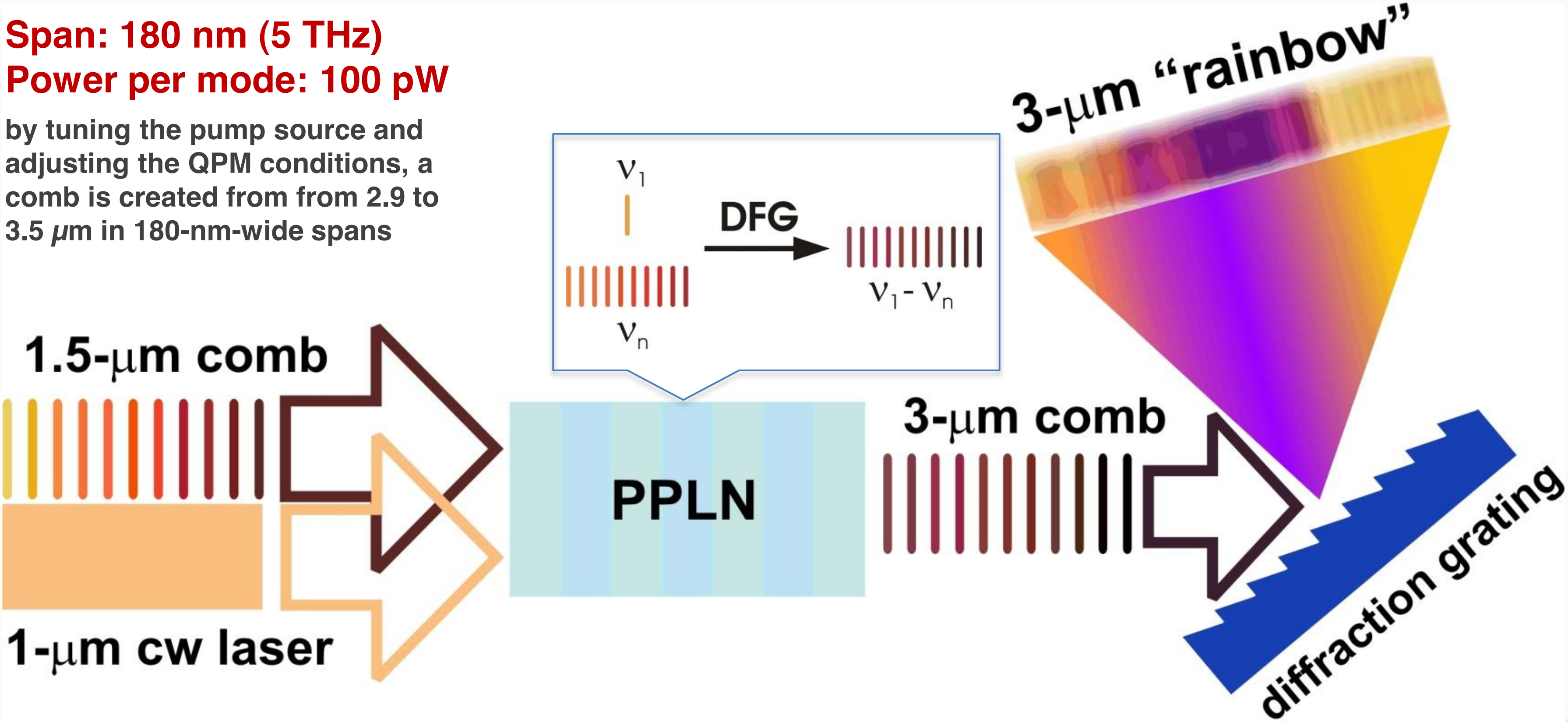


EXTENDING THE COMB TO THE MIR

only vis/NIR combs are commercially available □ linking direct IR sources to OFCs

Span: 180 nm (5 THz)
Power per mode: 100 pW

by tuning the pump source and adjusting the QPM conditions, a comb is created from from 2.9 to 3.5 *μ*m in 180-nm-wide spans



P. Maddaloni et al., New Journal of Physics **8**, 262 (2006)

MIR-comb generation

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OPTICS LETTERS / Vol. 37, No. 12 / June 15, 2012

Widely-tunable mid-infrared frequency comb source based on difference frequency generation

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[2]Physics Department of Politecnico di Milano – Polo di Lecco, via Ghislanzoni 24, 23900 Lecco, Italy

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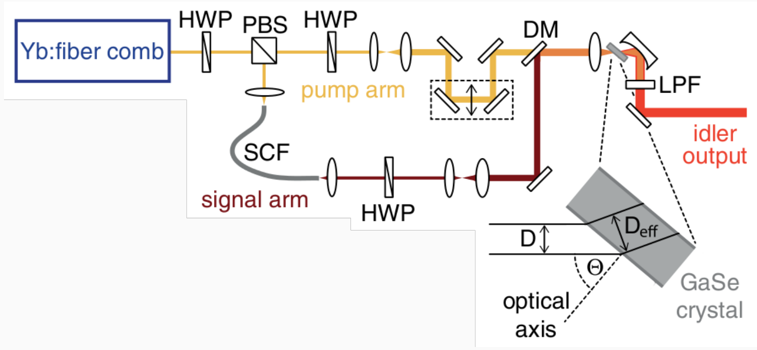
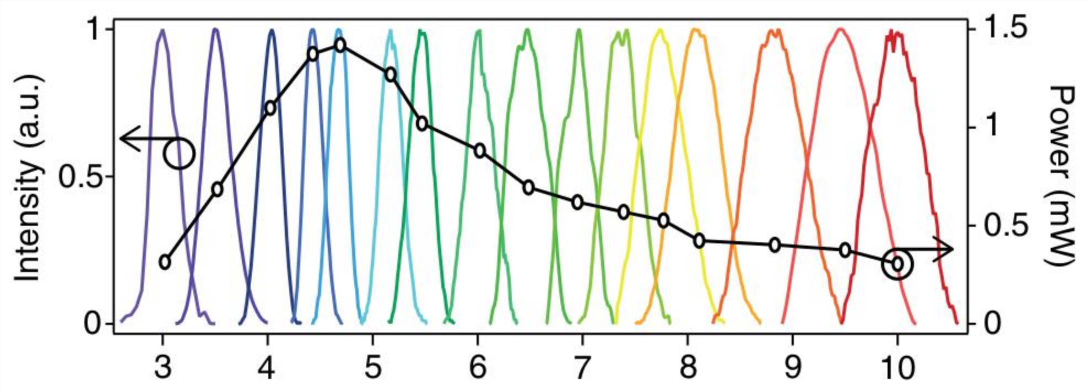
Received March 12, 2012; accepted April 23, 2012;
posted April 25, 2012 (Doc. ID 164602); published June 6, 2012

We report on a mid-IR frequency comb source of unprecedented tunability covering the entire 3–10 μm molecular fingerprint region. The system is based on difference frequency generation in a GaSe crystal pumped by a 151 MHz Yb:fiber frequency comb. The process was seeded with Raman-shifted solitons generated in a highly nonlinear suspended-core fiber with the same source. Average powers up to 1.5 mW were achieved at the 4.7 μm wavelength.

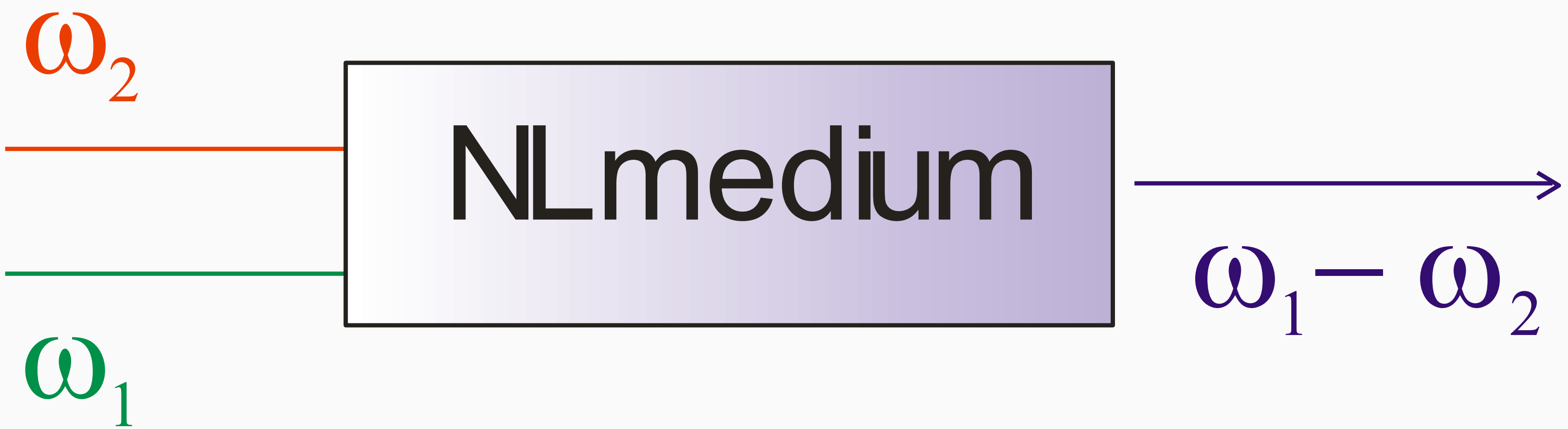
OCIS codes: 140.3070, 190.4410, 190.7110, 300.6340.





With a **GaSe** non-linear crystal they obtain a very wide tunability range (3-10 μm) and an overall optical power up to 1.5 mW.

Difference Frequency Generation - DFG

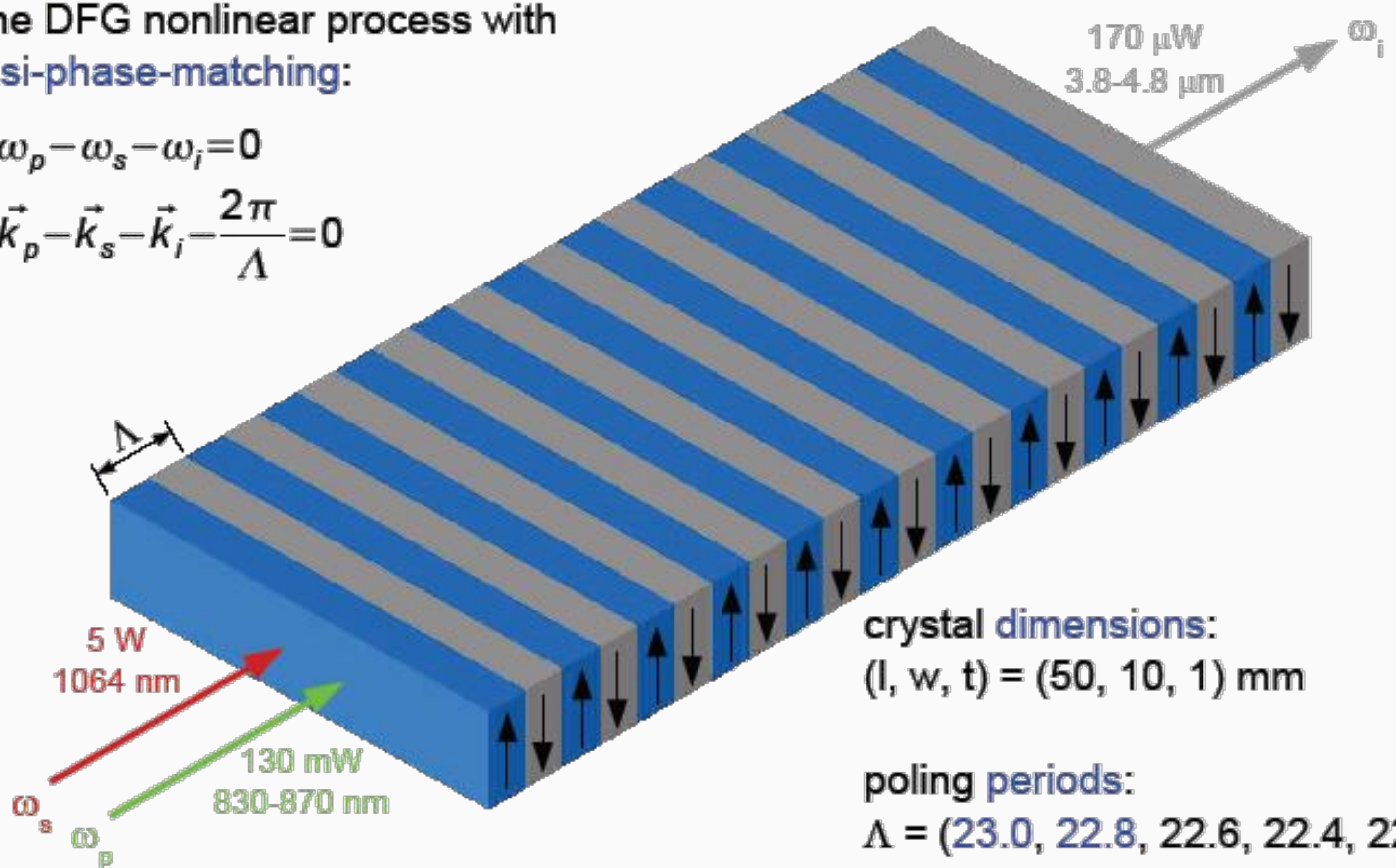


The Source: DFG in PP-Crystals

Energy and momentum conservation in the DFG nonlinear process with quasi-phase-matching:

$$\omega_p - \omega_s - \omega_i = 0$$

$$\vec{k}_p - \vec{k}_s - \vec{k}_i - \frac{2\pi}{\Lambda} = 0$$

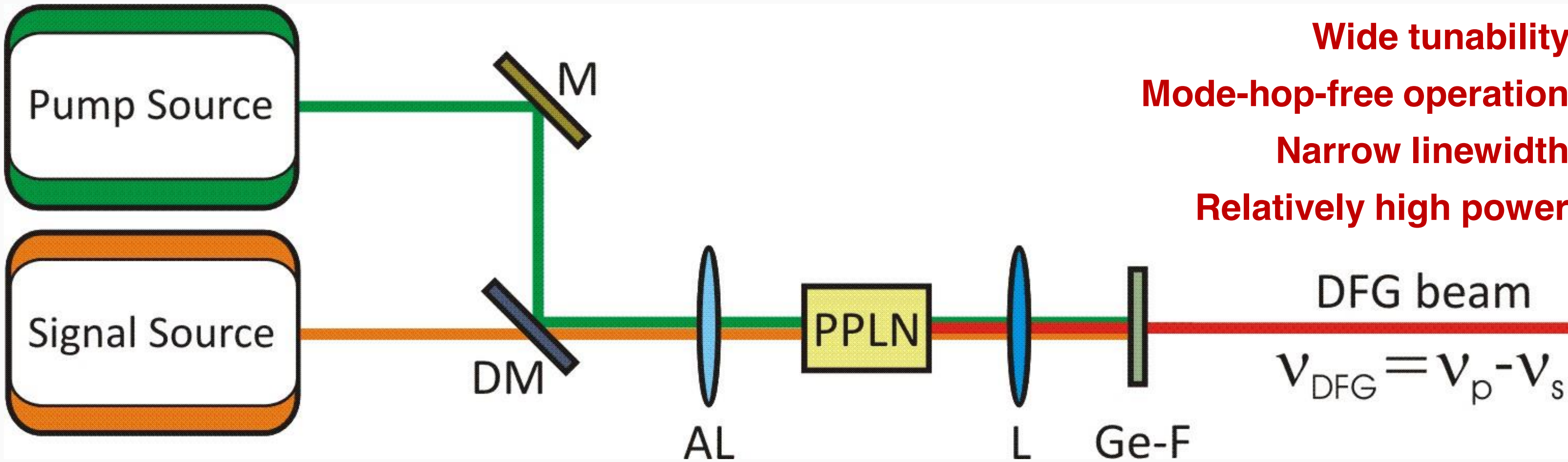


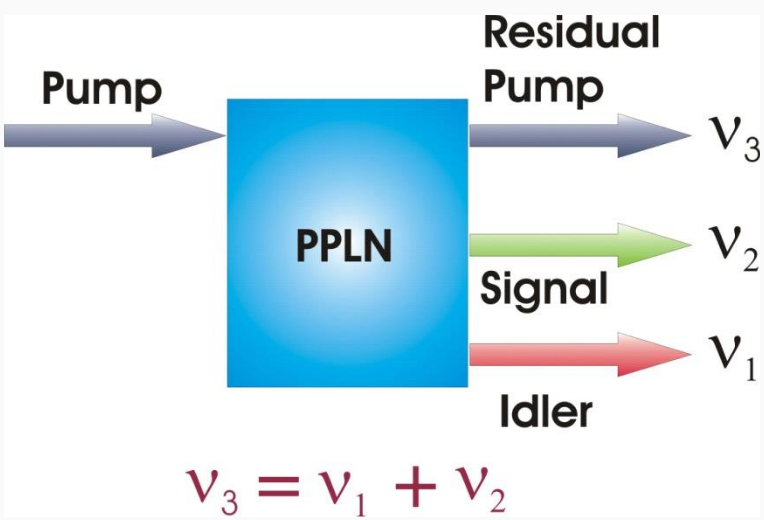
crystal dimensions:
(l, w, t) = (50, 10, 1) mm

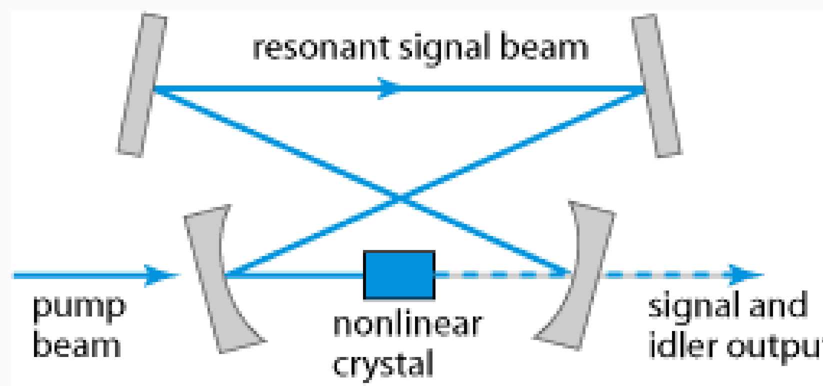
poling periods:
Λ = (23.0, 22.8, 22.6, 22.4, 22.2, 22.0, 21.8, 21.6, 21.4) µm

Optical Frequency-Down Conversion

- **Wide tunability**
- **Mode-hop-free operation**
- **Narrow linewidth**
- **Relatively high power**







Much higher power, but cavity is needed to be frequency-stable and with single-mode behaviour

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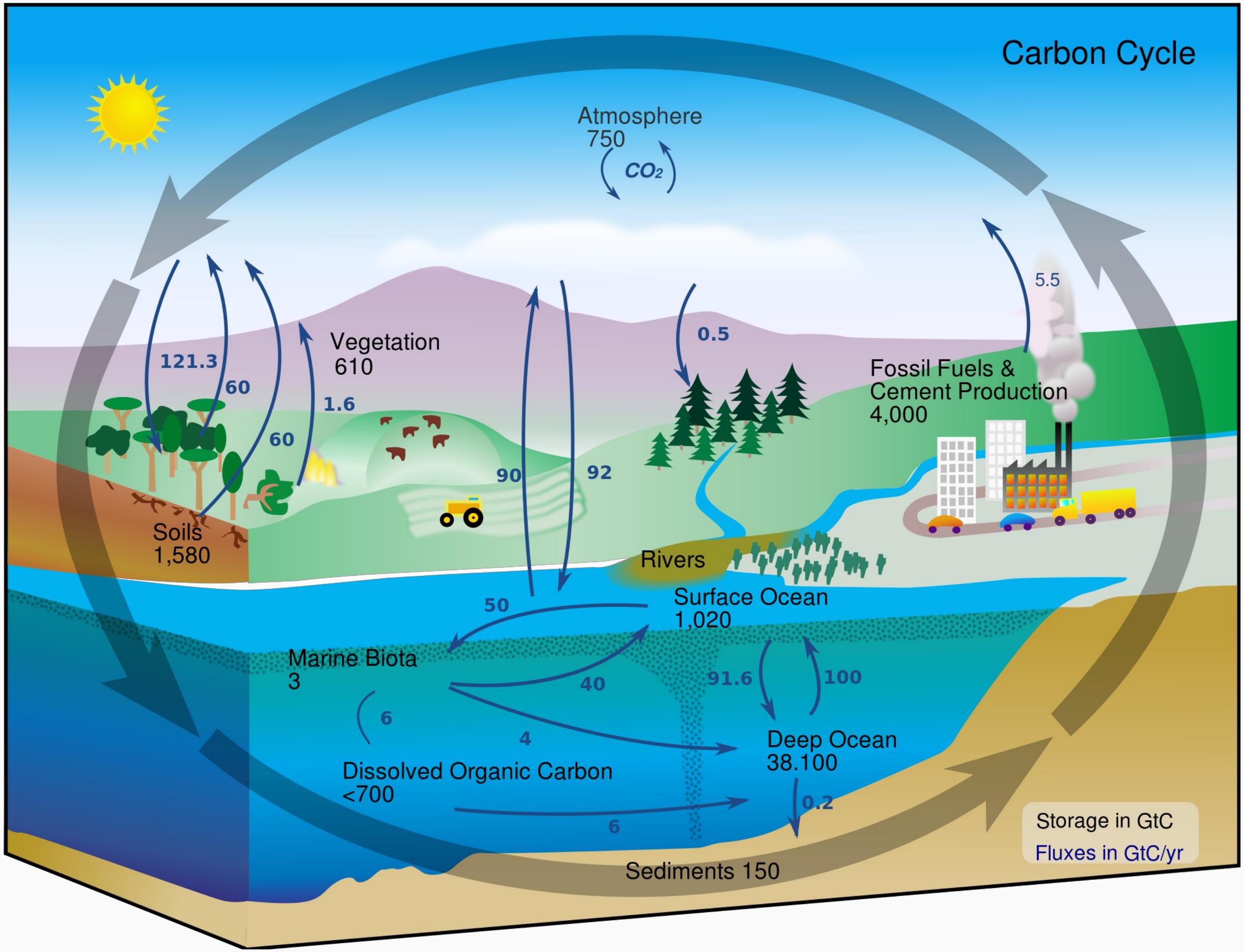
Carbon cycle...



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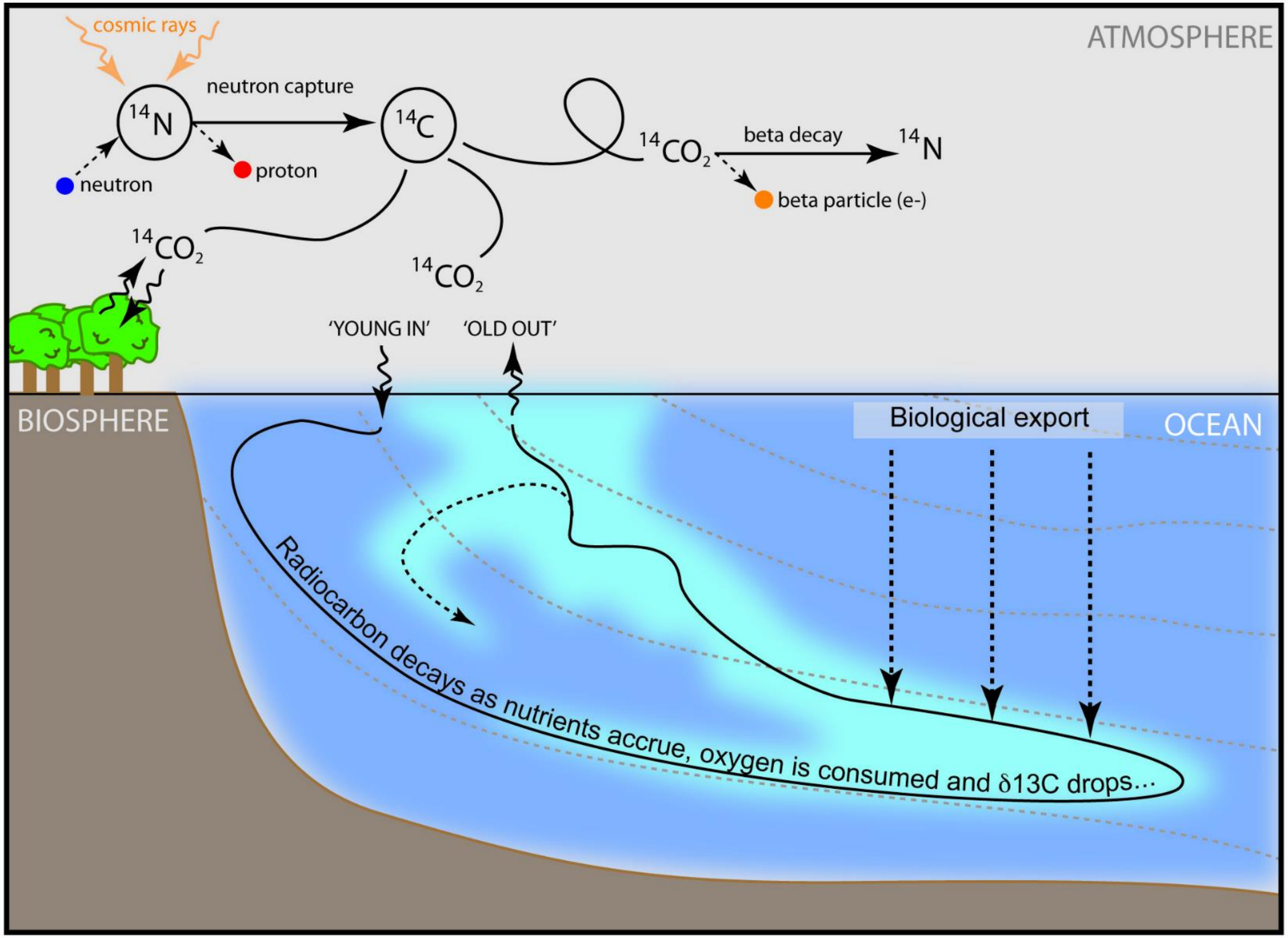
...and radiocarbon cycle



The New York Times

ASIA PACIFIC | NEWS ANALYSIS 179 COMMENTS

Deal on Carbon Emissions by Obama and Xi Jinping Raises Hopes for Upcoming Paris Climate Talks

By CORAL DAVENPORT NOV. 12, 2014

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Nazionale di
Ottica

ENVIRONMENT

Energy and T… for Managin…

Aristides A. N. Patrinos[1]* and Rich…

Despite some uncertai… scientific and politic… is that the level of glo… of greenhouse gases (GHGs) … to atmospheric concentration… between 450 and 500 parts per … (1) to avoid serious, if not catast… on life and property. Achiev… poses some formidable challe… inertia in the climate system (… for generations), as well as in … capital investment. Furthermo… nomic sector and country em… these challenges, a broad range … be required.

Despite some uncertainties, today's scientific and political consensus is that the level of global emissions of greenhouse gases (GHGs) needs to lead to atmospheric concentrations somewhere between 450 and 500 parts per million (ppm) (*1*) to avoid serious, if not catastrophic, effects on life and property. Achieving this goal poses some formidable challenges. There is inertia in the climate system (GHGs survive for generations), as well as in GHG-emitting capital investment. Furthermore, every economic sector and country emits. To meet these challenges, a broad range of actions will be required.

www.sciencemag.org

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The Challenge

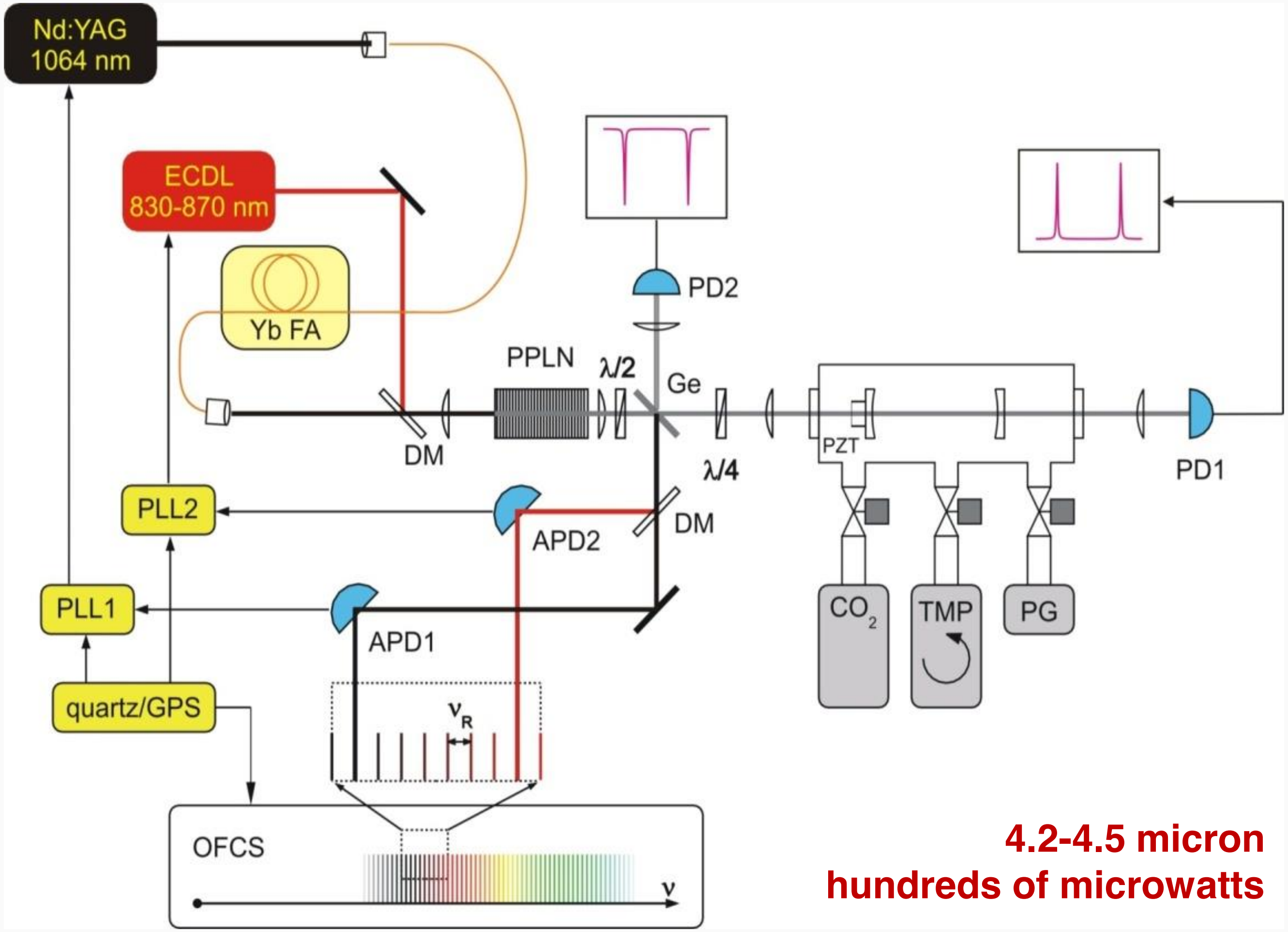
To meet these formidable challenges, sensors are needed with:

- High sensitivity, for trace gas sensing
- High resolution, for a high degree of discrimination
- Infrared spectral coverage, to match strong molecular bands
- Wide tunability, to interrogate a large number of molecules
- High precision, to reduce databases statistical uncertainties
- High accuracy, to reduce systematic effects getting high reproducibility

↓

Laser-based Infrared Spectrometers

A “simple” Comb-Assisted DFG Setup



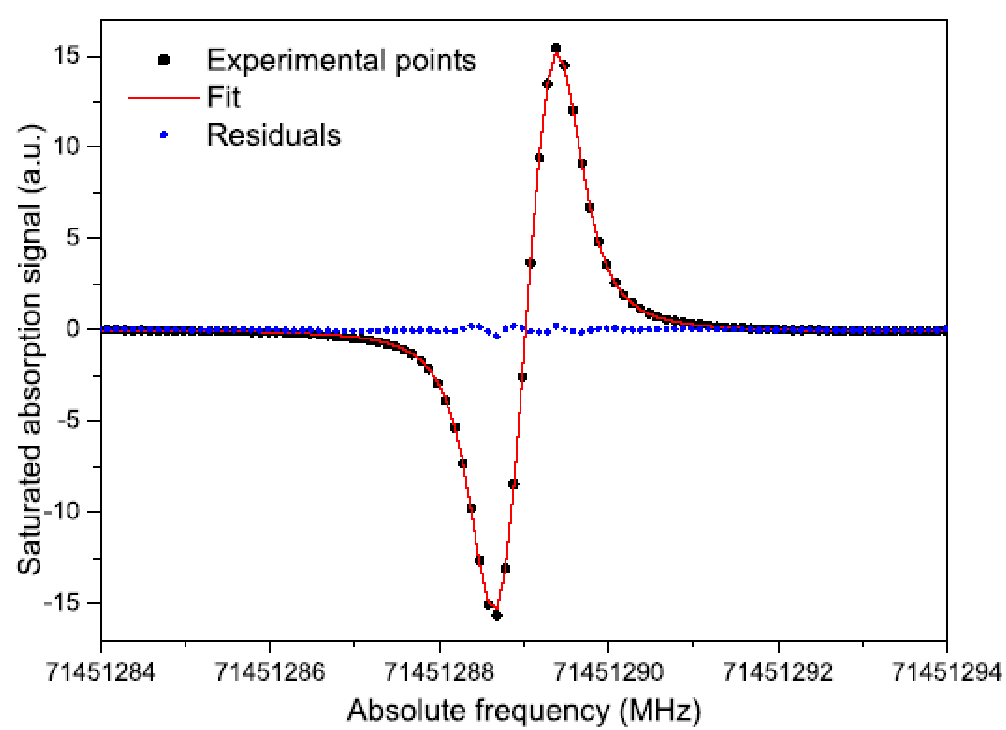
4.2-4.5 micron
hundreds of microwatts

$$f_p = n_p f_r + f_0 + f_{beat}^p$$

$$f_s = n_s f_r + f_0 + f_{beat}^s$$

$$f_{DFG} \equiv f_p - f_s = (n_p - n_s) f_r + (f_{beat}^p - f_{beat}^s)$$

Cavity-Enhanced Saturation Spectroscopy



Source	Expression[a]	Actual Value
Natural lifetime	$k^3\mu^2/3\pi\epsilon_o\hbar$	0.2 kHz
Collisions	$c_P P$	90 kHz
Transit time	$\sqrt{\ln 2/\pi}\sqrt{k_B T/m}\,1/w$	400 kHz
IR jitter	$[1-(\nu_s/\nu_p)]\Gamma_c$	<50 kHz
Power	$\sqrt{1+(I/I_s)}$	$\times 1.4\ [(I/I_s)=1]$
Modulation		$\times 1.3\ (\beta=1)$

CO_2 (00^01-00^00) R(56) saturated-absorption line at 2383.359 cm^{-1}

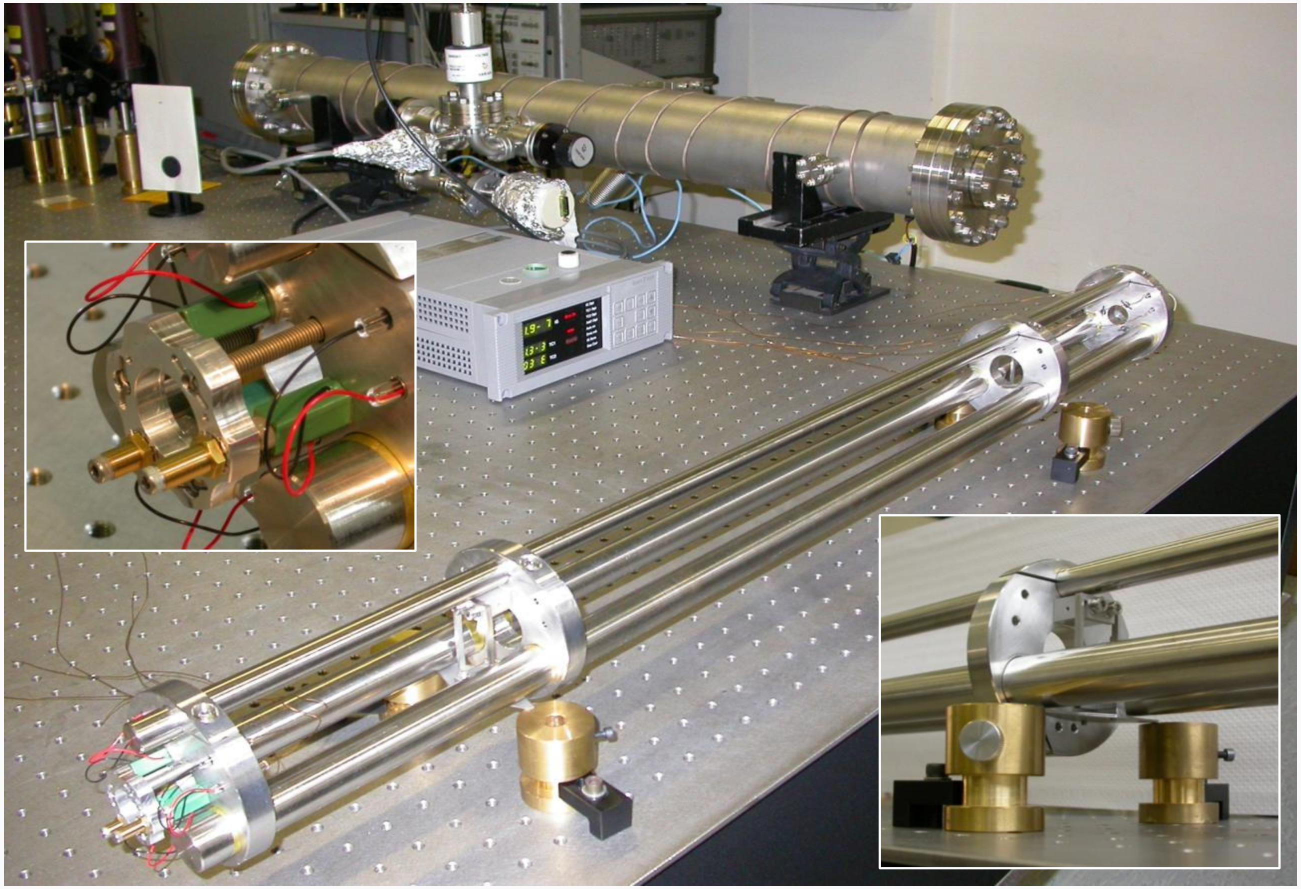
D. Mazzotti, et al., Opt. Lett. **30** , 997 (2005)

uncertainty of 800 Hz (10^{-11})

in the absolute frequency

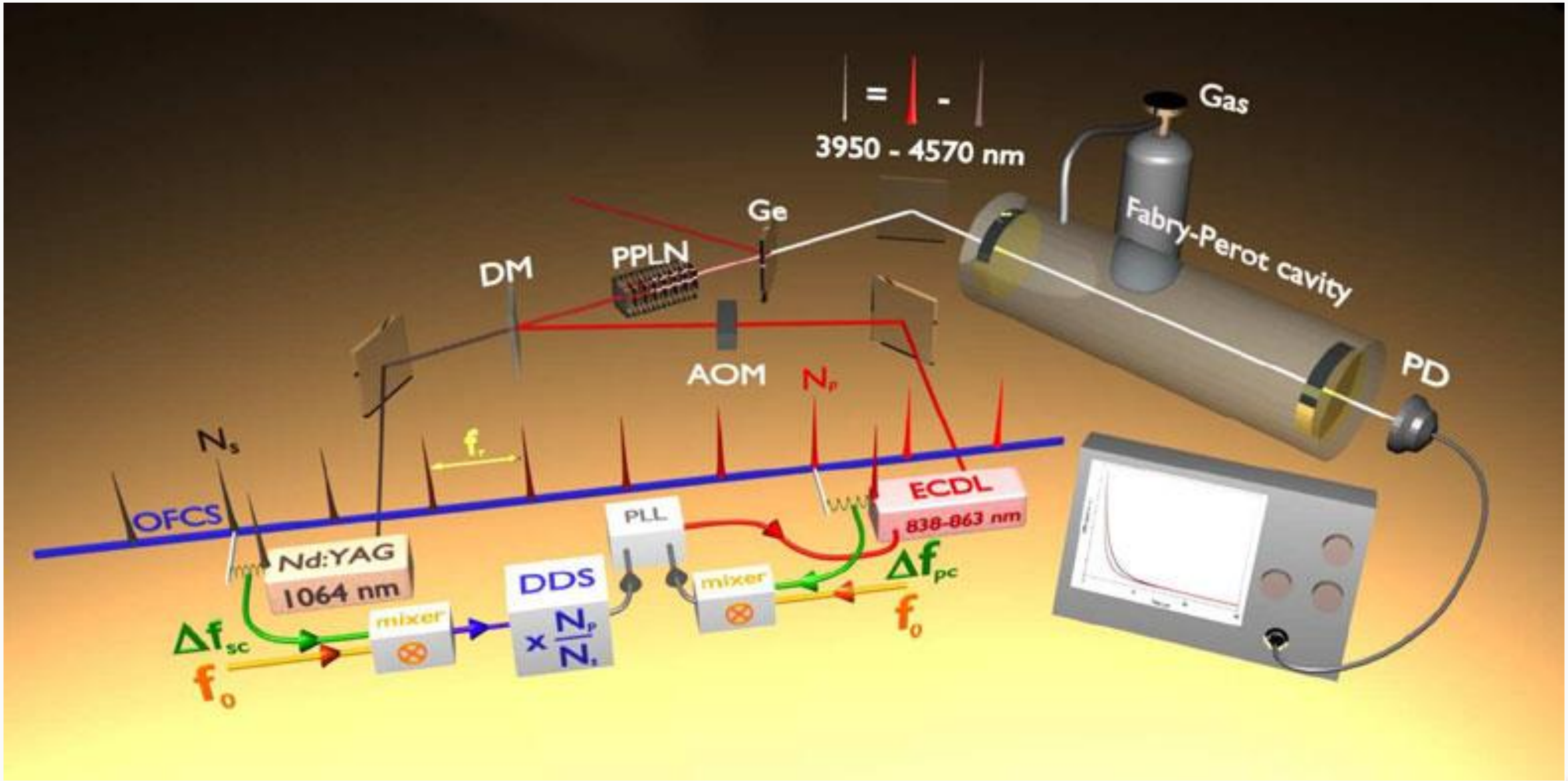
Main limitation comes from transit time

Pushing up Sensitivity by Cavity Enhancement



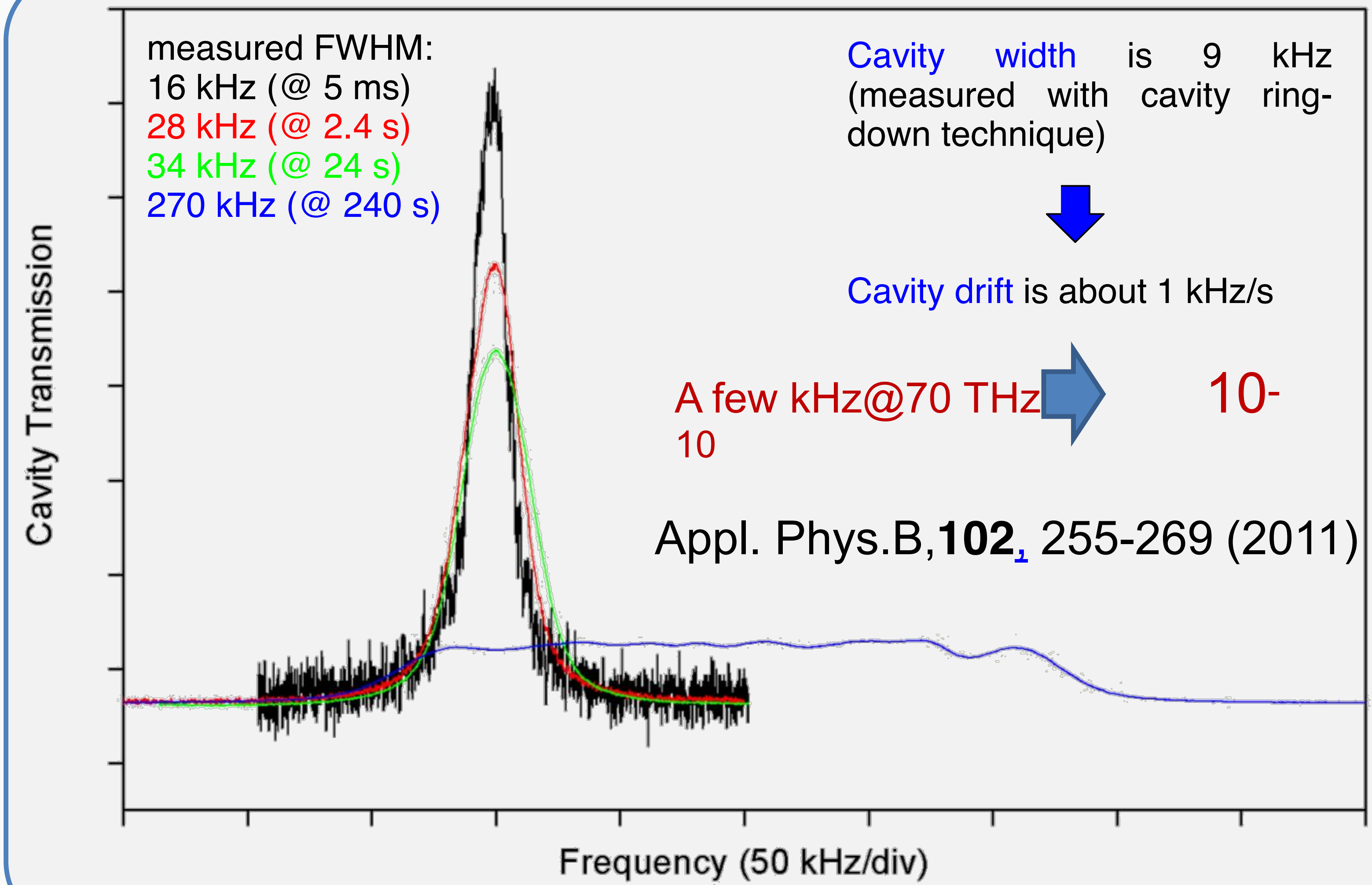
11000<F<25000 @ 4.5 μm
FSR = 150 MHz

Metrological spectroscopy: a smart DFG link to an Optical Frequency Comb Synthesizer



***I. Galli et al.*, Optics Express 17, 9582 (2009)**
original scheme proposed by Telle et al., Appl. Phys. B (2002))

Coupling IR radiation into an enhancement cavity



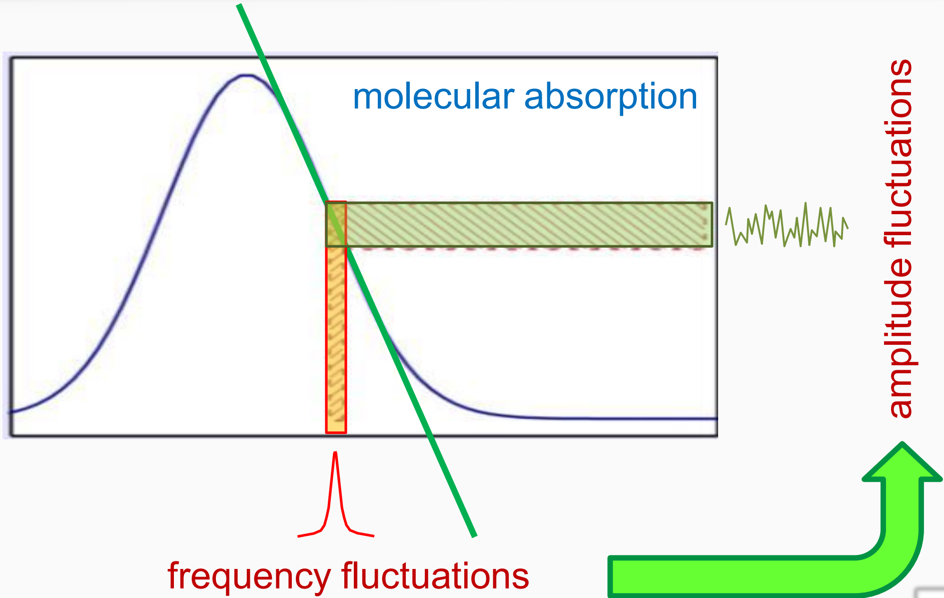
Cavity width is 9 kHz (measured with cavity ring-down technique)

Cavity drift is about 1 kHz/s

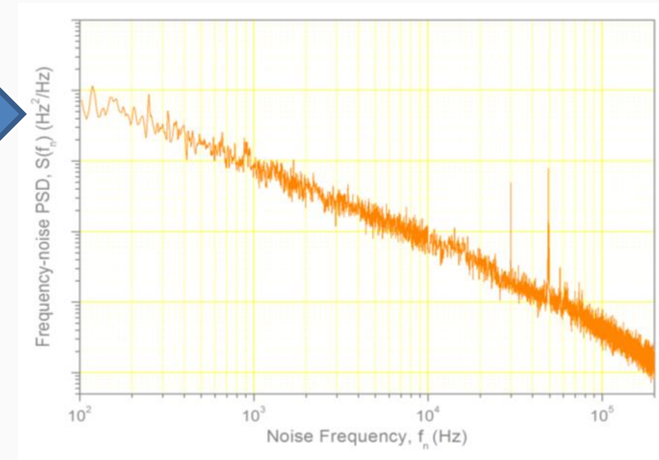
A few kHz@70 THz 10-
10

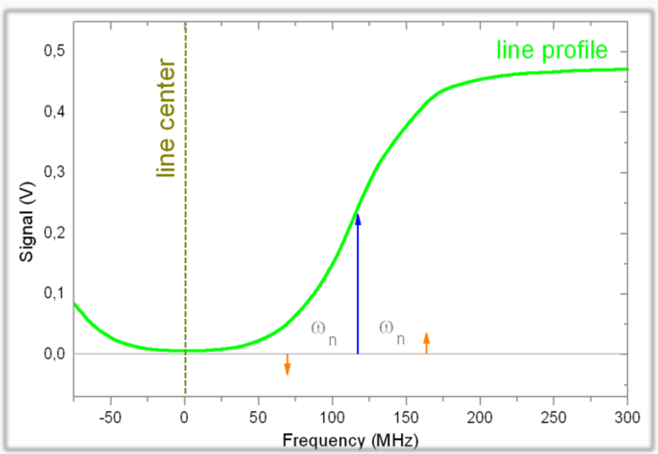
Appl. Phys.B,**102**, 255-269 (2011)

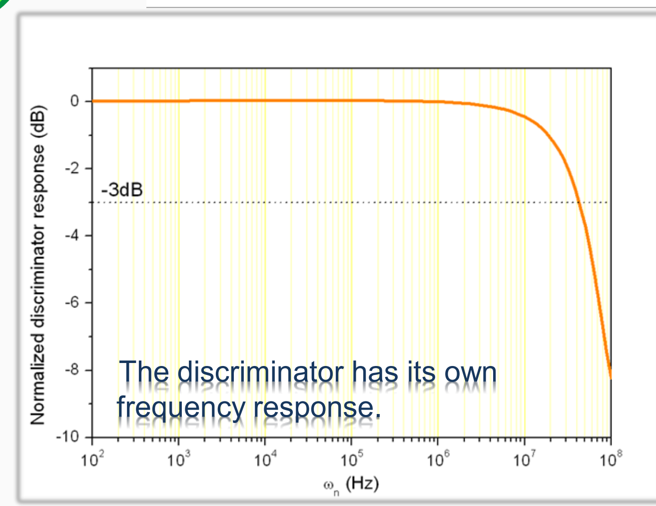
Measuring the Laser frequency-noise: principle



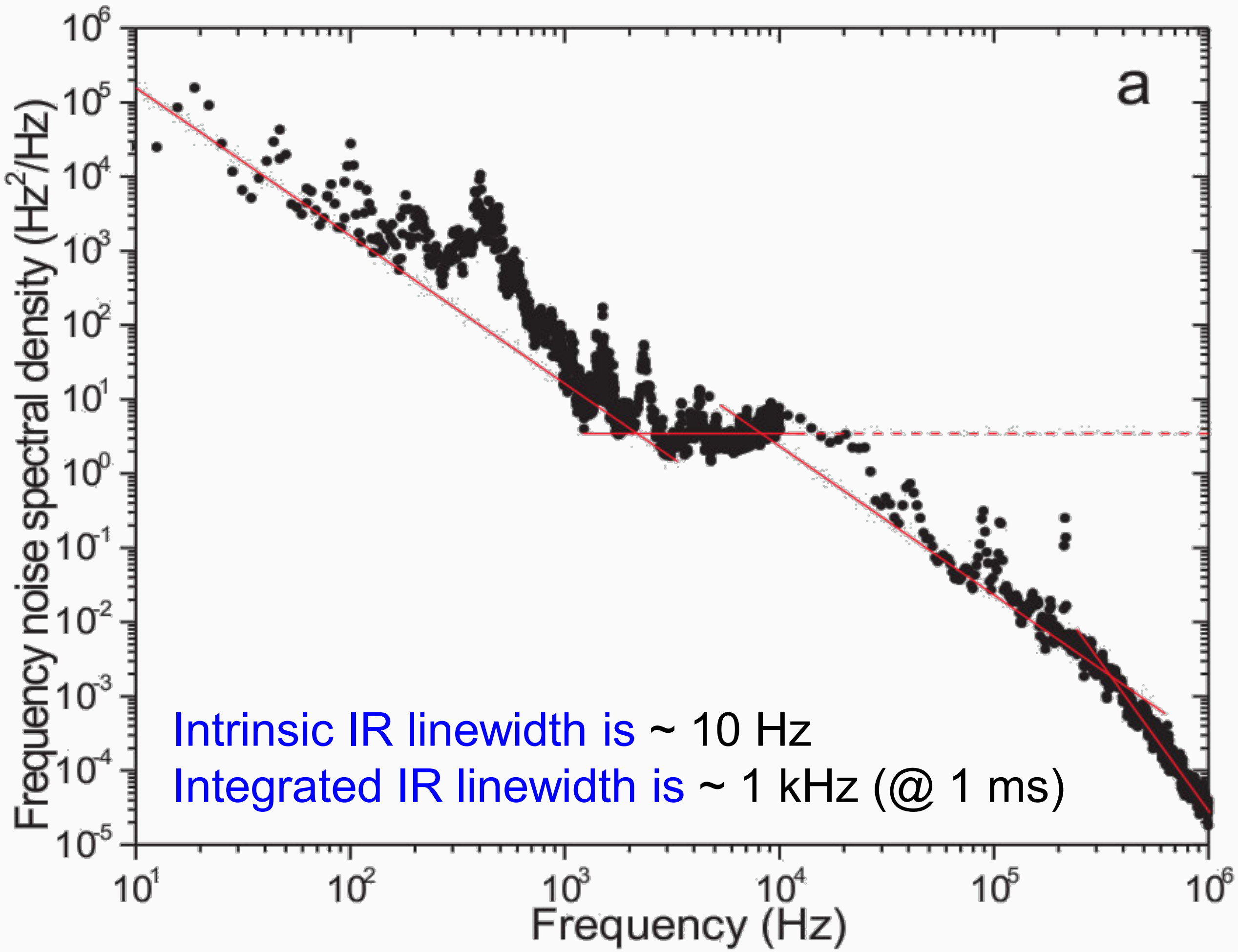
Frequency-noise power spectral density





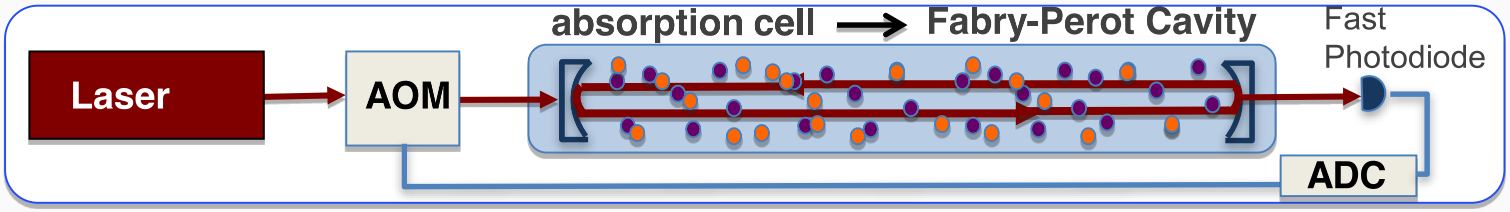


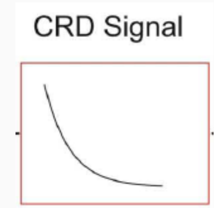
Narrowing the IR linewidth



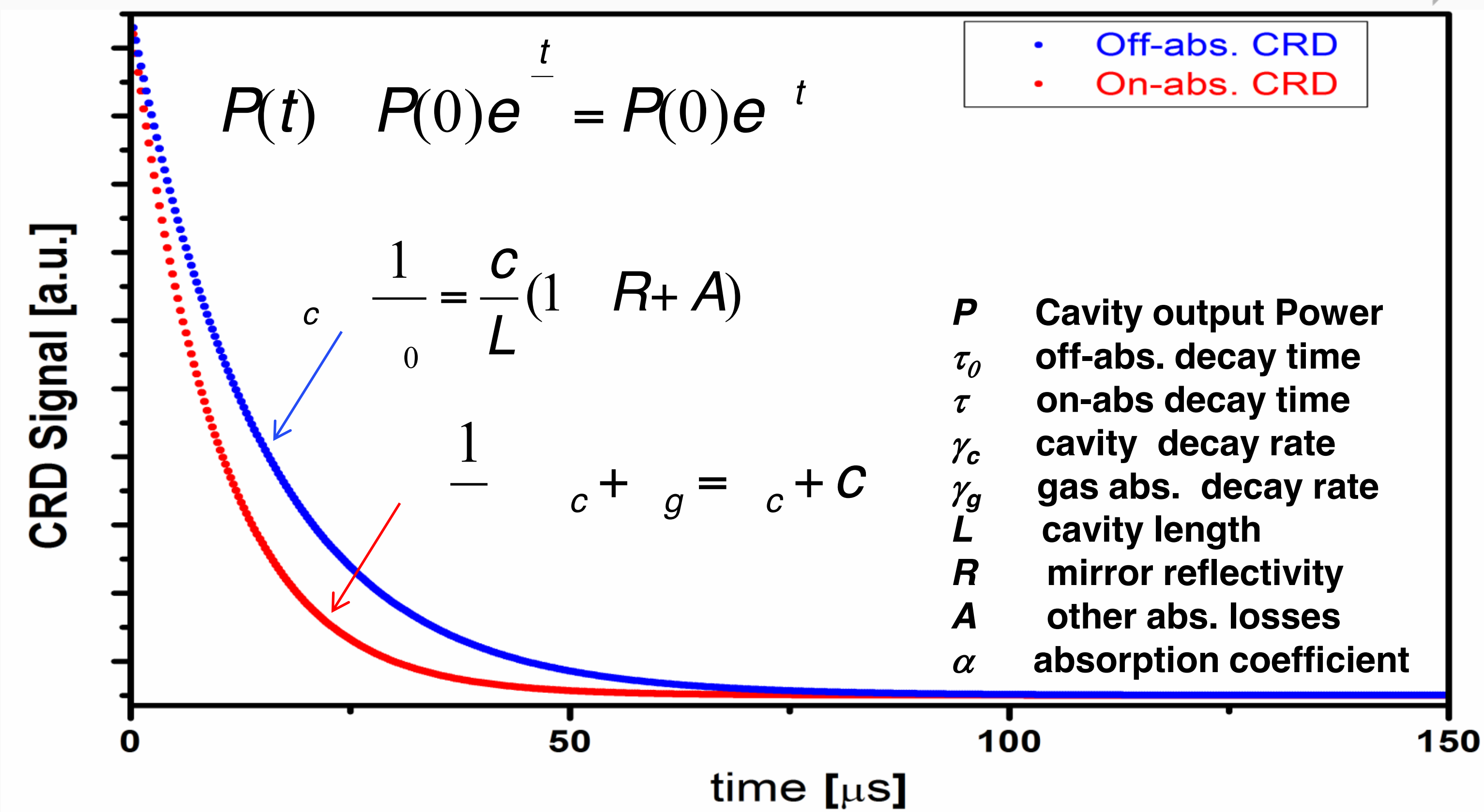
Ultra-stable, widely tunable and absolutely linked mid-IR coherent source
I. Galli et al., Optics Express **17**, 9582 (2009)

Linear Cavity Ringdown vs….

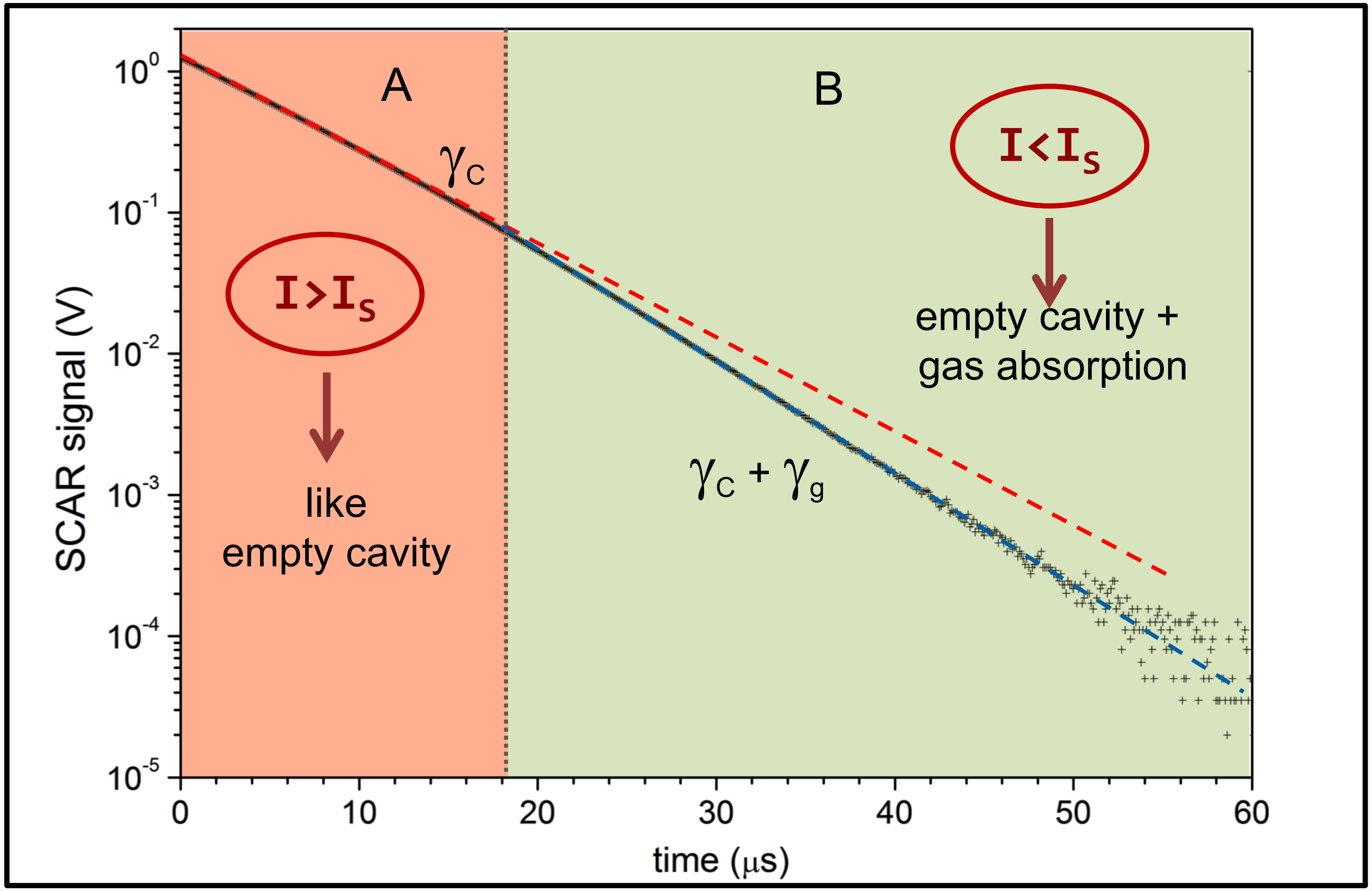




Measurement of the Cavity Ring Down time variations → $c\alpha$



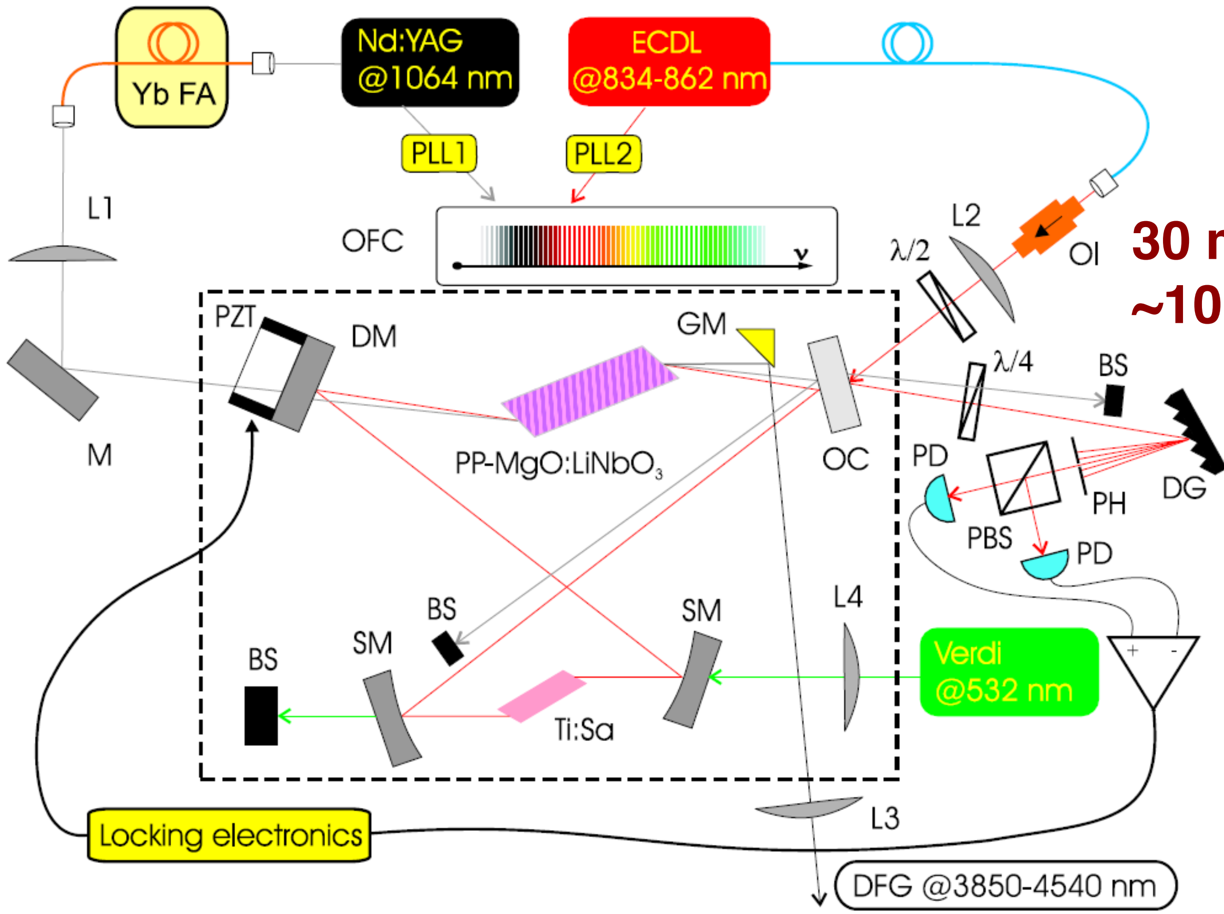
….Nonlinear Cavity Ringdown: Saturated absorption Cavity Ringdown-SCaR



Slope increase from region A to B is proportional to the absorption coefficient and hence to the gas concentration

G. Giusfredi et al. Phys. Rev. Lett. **104**, 110801 (2010)

Nonlinear absorption phenomena require power: Intra-cavity DFG



30 mW cw power @ 4.5 μm
~10Hz linewidth

Opt. Lett. ***35****, 3616 (2010)*

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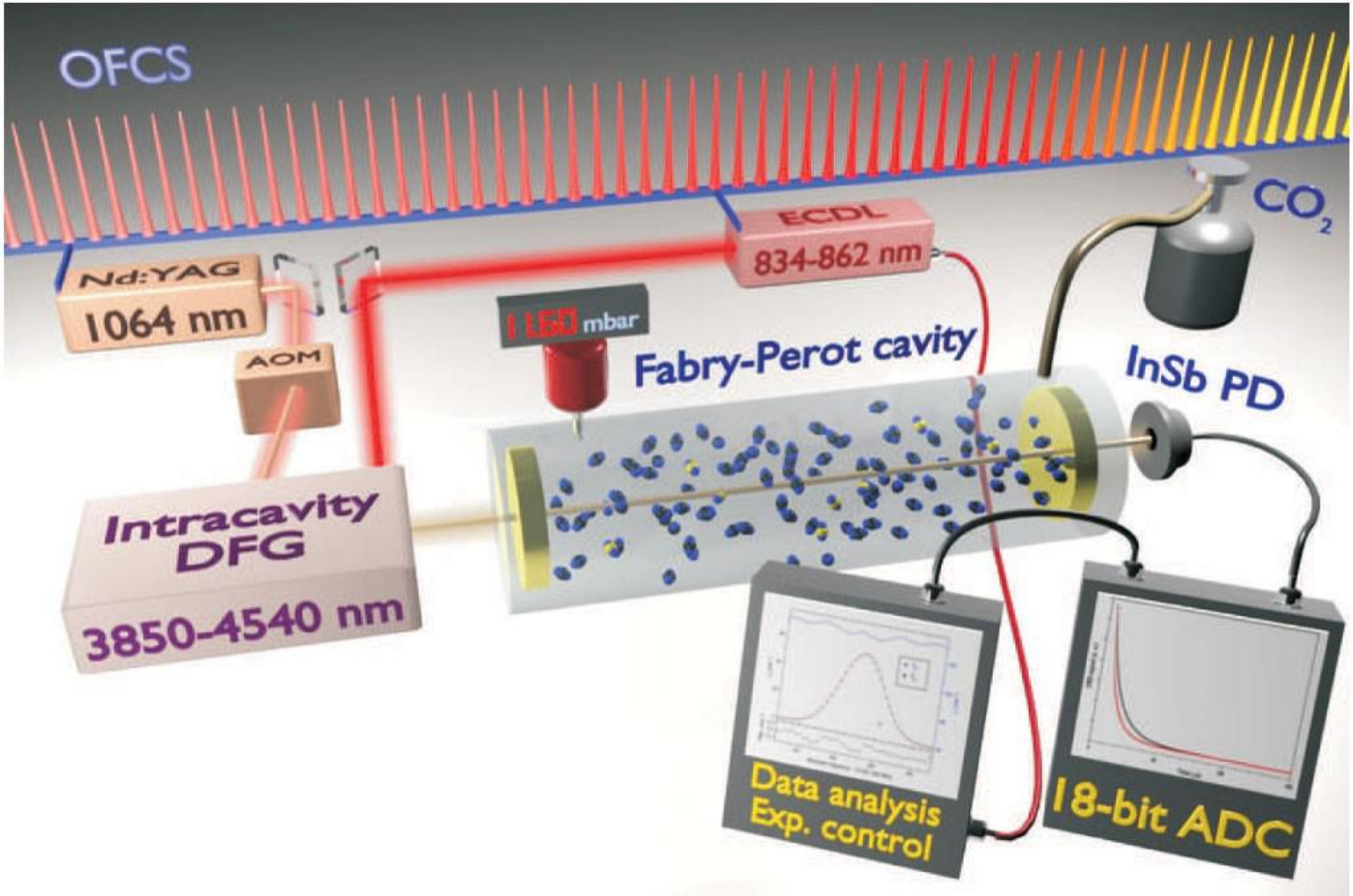
Combining Precision and Sensitivity: The SCAR set-up



Phys. Rev. Lett. **107**, 270802 (2011)

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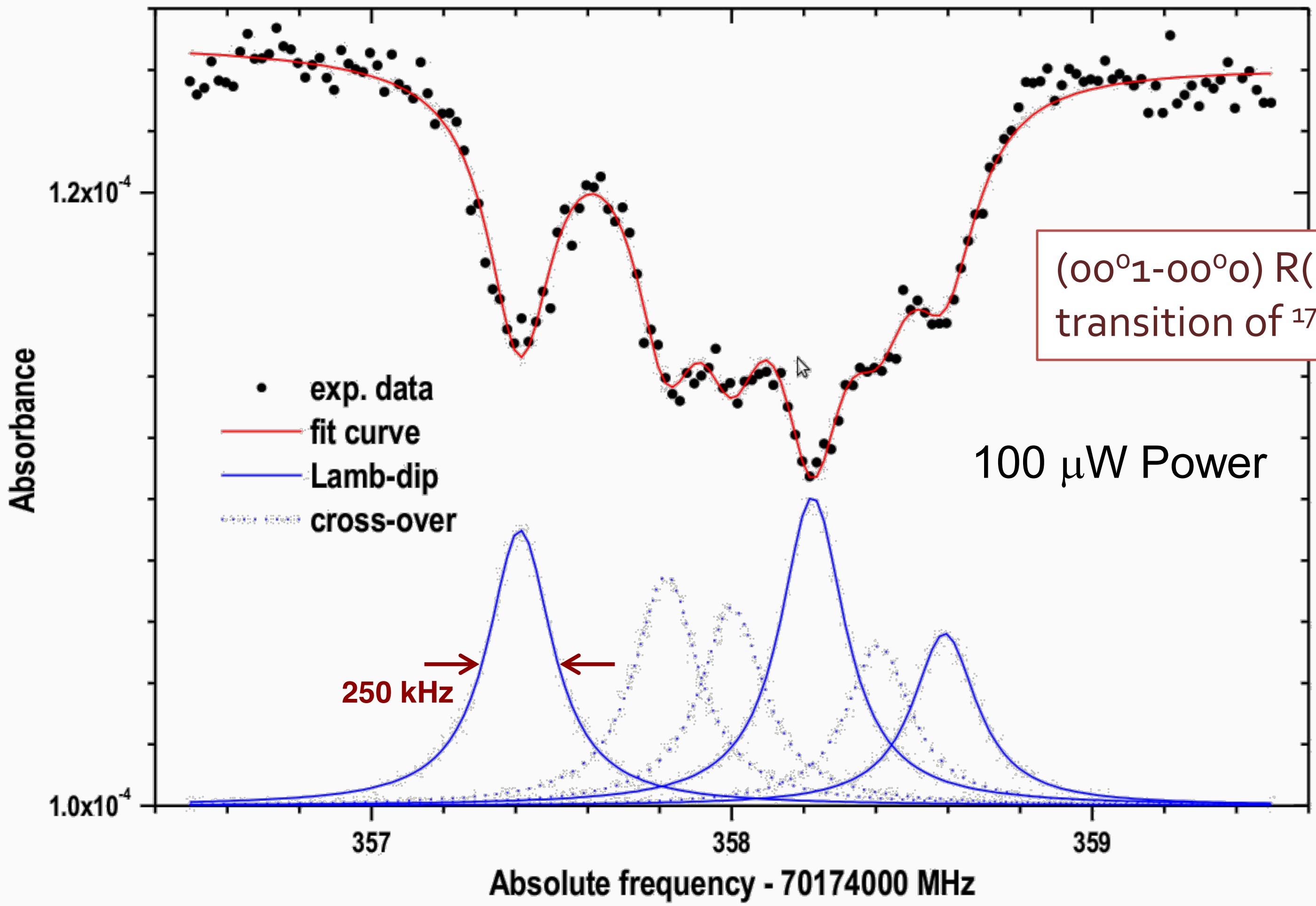
Hunting $^{14}CO_2$: Background and motivations

- ✓ Radiocarbon (^{14}C) is a key isotopic species for analysis in many different sectors worldwide. It is contained in $^{14}CO_2$ BUT it is a VERY elusive species (about 1 ppt concentration in natural abundance).

- ✓ To date, two main techniques are widely used to measure radiocarbon concentration, both with some disadvantages:
  - Liquid Scintillation Counting (LSC) has very long measurement times (hours-days), requires sample with complex preparation and big mass (1-10 g of C);
  - Accelerator Mass Spectrometry (AMS) requires large (7-200 m^2), expensive (0.3-3 M€) and high-maintenance facilities, with slow turnaround time (1-4 weeks) and high costs (400-1000 €) for each measured sample.
- ✓ High-sensitivity mid-IR laser spectroscopy has recently approached the performance of these two techniques, while reducing many of their disadvantages.

SCAR: the resolution



(00°1-00°0) R(0) transition of ^{17}OCO

100 µW Power

Phys. Rev. Lett. ***104***, *110801 (2010)*

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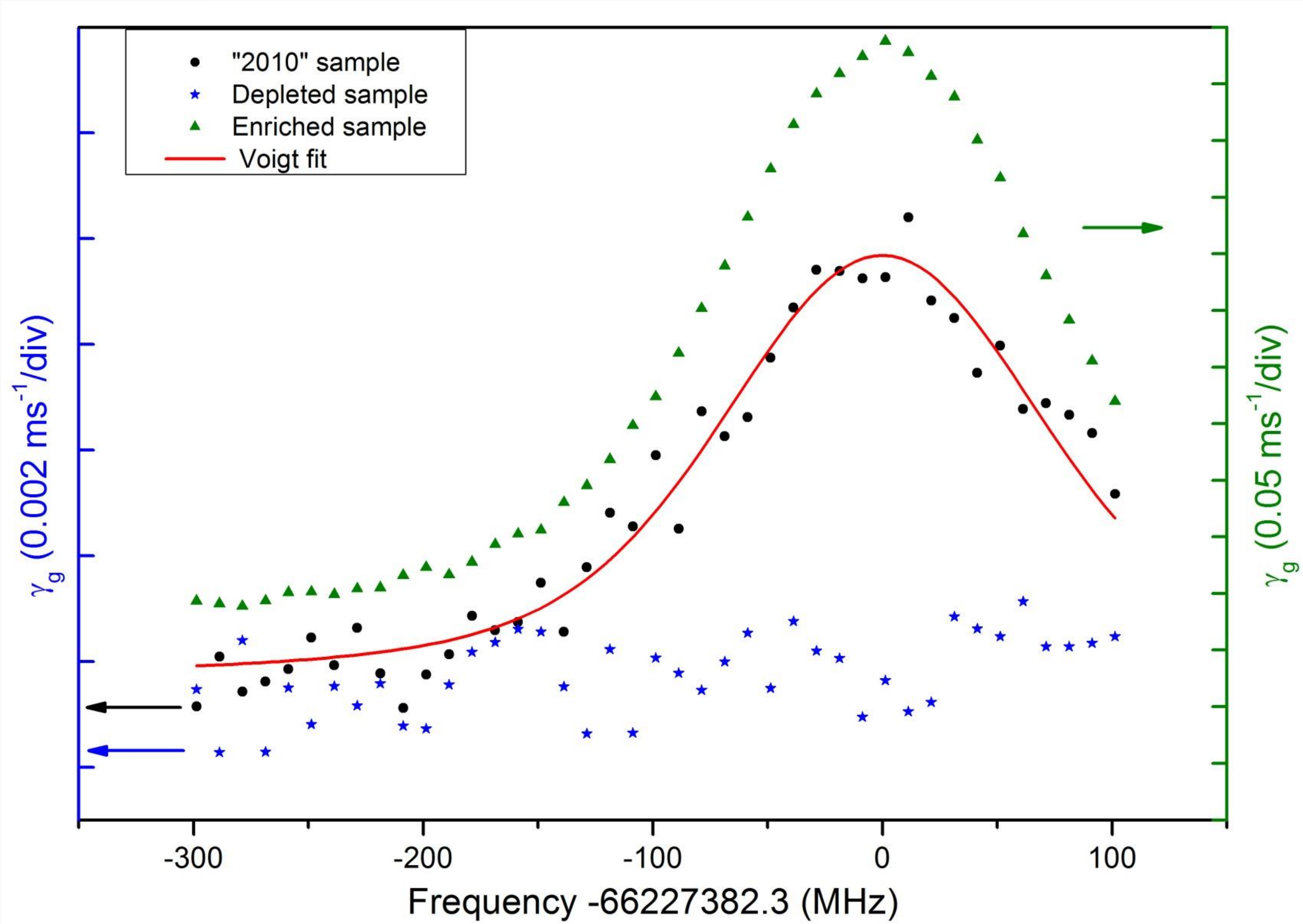
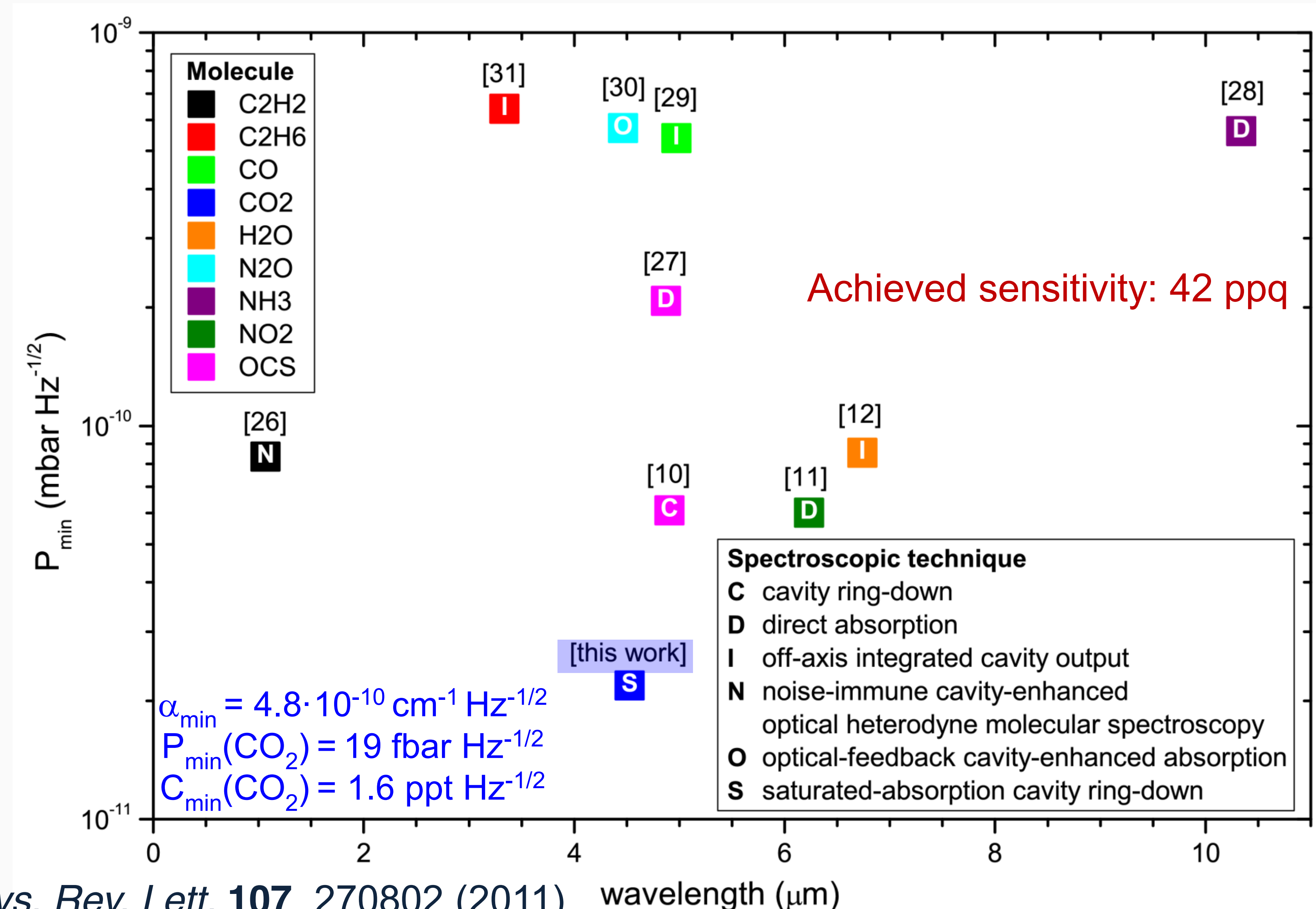
Natural abundance (1×10^{-12}) detection of $^{14}C^{16}O_2$



Phys. Rev. Lett. **107**, 270802 (2011)

SCAR: the sensitivity



Phys. Rev. Lett. **107**, 270802 (2011)

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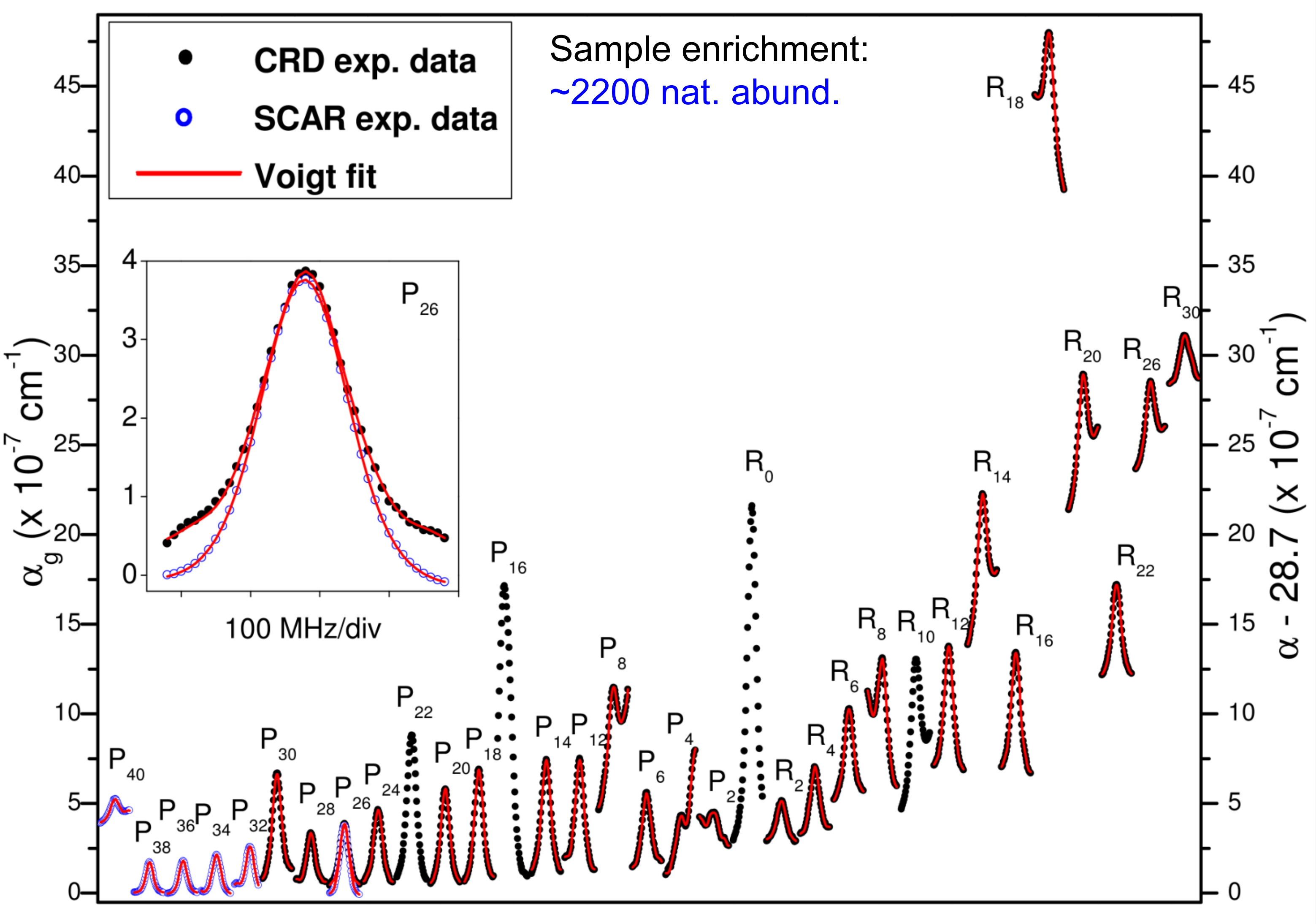
Spectroscopy of the $^{14}C^{16}O_2$ ν_3 band



Mol. Phys. **109**, 2267 (2011)

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Retrieved spectroscopic parameters

Table 1. Transitions frequencies, uncertainties and residuals from the fit of the lines measured for the ν_3 fundamental band of $^{14}C^{16}O_2$. All numbers are in MHz.

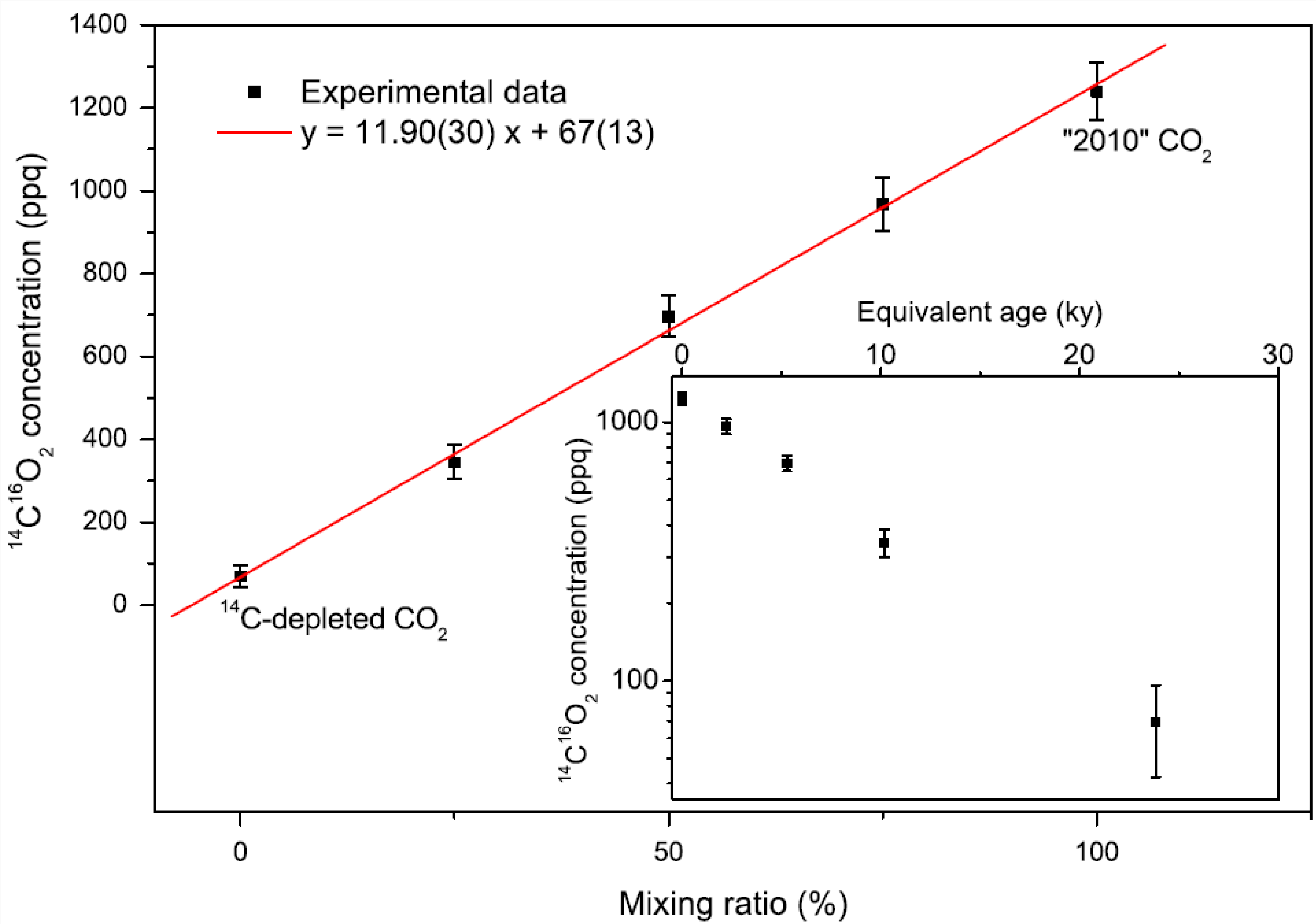
Transition	Obs. Freq.	Uncertainty[a]	Obs.-Calc.[b]
R(30)	67367614.181	4.795	1.759
R(26)	67294430.381	0.825	2.317 *
R(22)	67218457.563	0.875	0.047
R(20)	67179428.939	0.743	−0.260
R(18)	67139706.163	0.731	−0.314
R(16)	67099289.625	1.017	−0.450
R(14)	67058180.523	0.877	−0.197
R(12)	67016379.675	0.828	0.529
R(8)	66930706.117	1.178	3.834 *
R(6)	66886832.920	1.111	4.442 *
R(4)	66842264.775	1.112	−0.645
R(2)	66797014.526	2.003	0.666
P(2)	66680873.788	2.765	−3.952
P(4)	66633216.481	3.176	−5.929
P(6)	66584878.904	1.132	−3.113 *
P(8)	66535859.047	1.443	1.710
P(12)	66435758.242	0.996	−0.005
P(14)	66384685.331	2.458	−0.078
P(18)	66280498.427	0.801	1.310
P(20)	66227383.132	0.865	−0.128
P(24)	66119121.905	1.715	1.758
P(26)	66063972.706	0.755	0.194
P(28)	66008145.242	1.749	−3.339
P(32)	65894474.448	0.747	−0.680
P(34)	65836627.418	0.742	0.147
P(36)	65778106.638	0.729	0.186
P(38)	65718914.039	0.752	0.510
P(40)	65659047.695	1.795	−1.677

Table 2. Spectroscopic parameters (in cm^{-1}) obtained for the ν_3 fundamental band of $^{14}C^{16}O_2$.

Parameter	This work[a]	Ref. [6]
ν_0	2225.801399(17)	2225.80239(16)
B_0	0.390253082(58)	0.39025488(18)
$D_0 \times 10^{-7}$	1.33144(37)	1.3372(20)
B_1	0.38739025628[b]	
$D_1 \times 10^{-7}$	1.327523[b]	
$H_1 \times 10^{-14}$	−3.08[b]	
$L_1 \times 10^{-17}$	1.244[b]	
$\sigma_{FIT} \times 10^{-5}$	3.1 → 930 kHz	

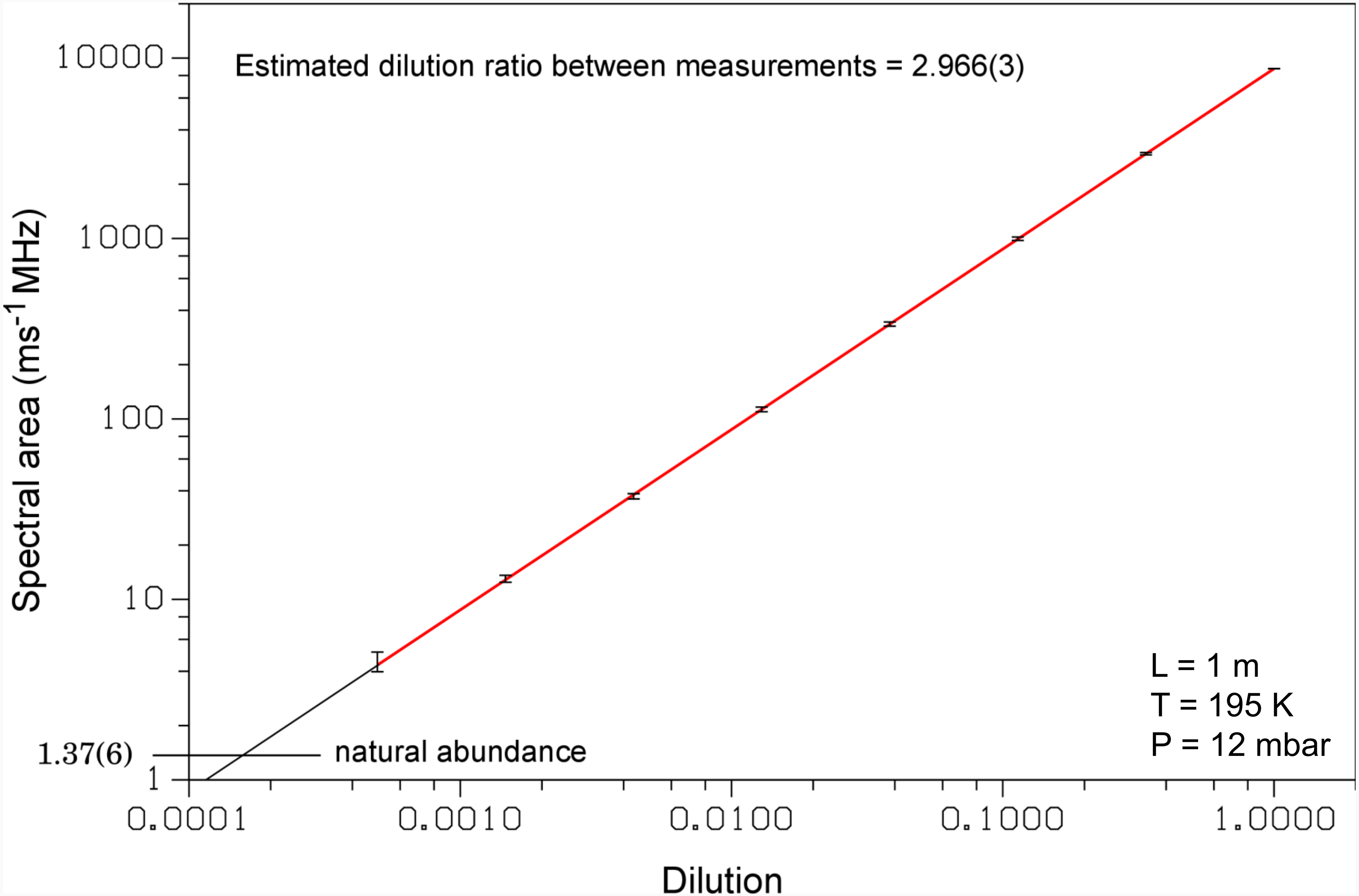
Mol. Phys. **109**, 2267 (2011)

Detection linearity

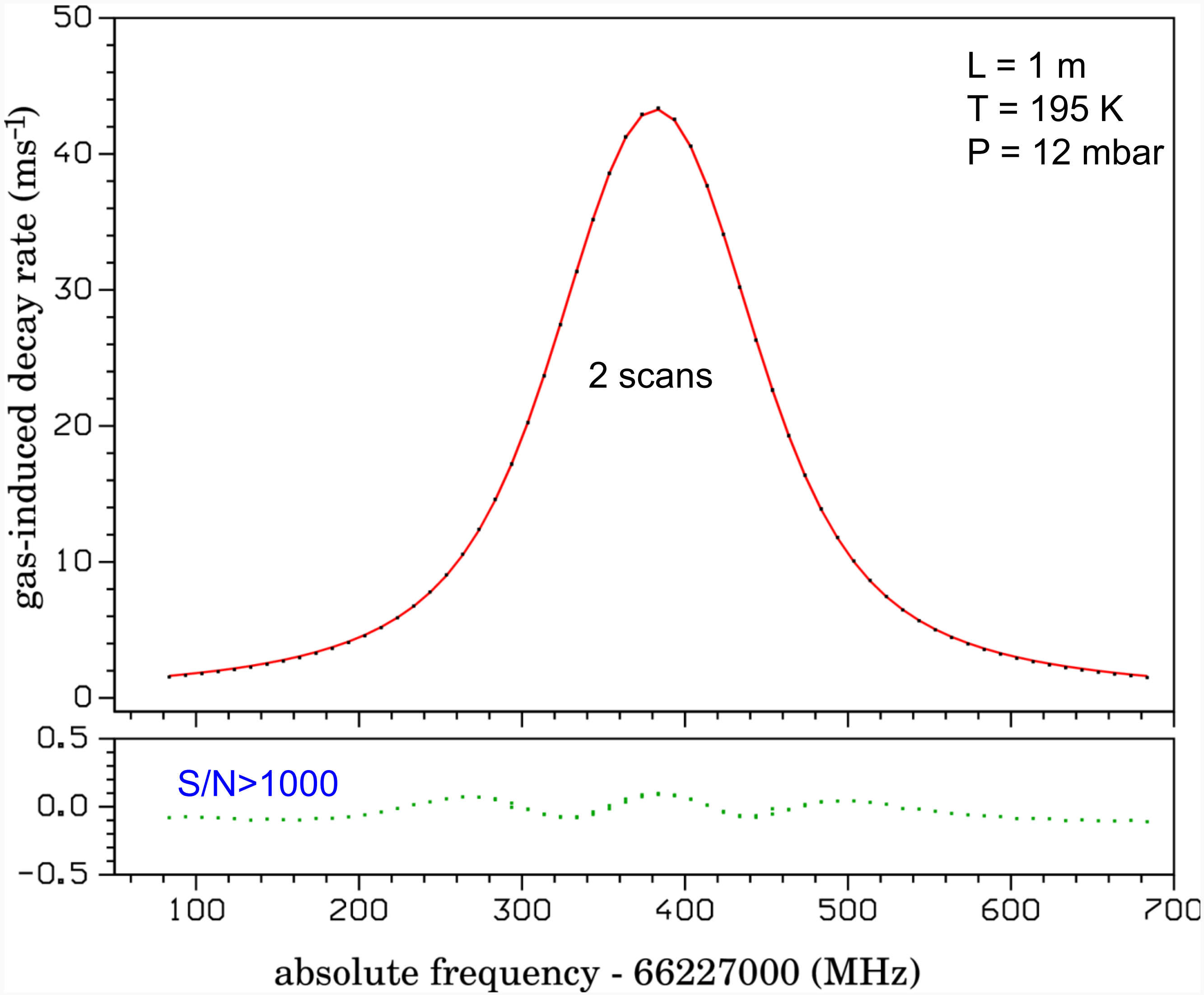


Phys. Rev. Lett. **107**, 270802 (2011)

Detection linearity (1-6400 nat. abund.)



Voigt lineshape fitting (6400 nat. abund.)



Preliminary AMS-SCAR intercalibration

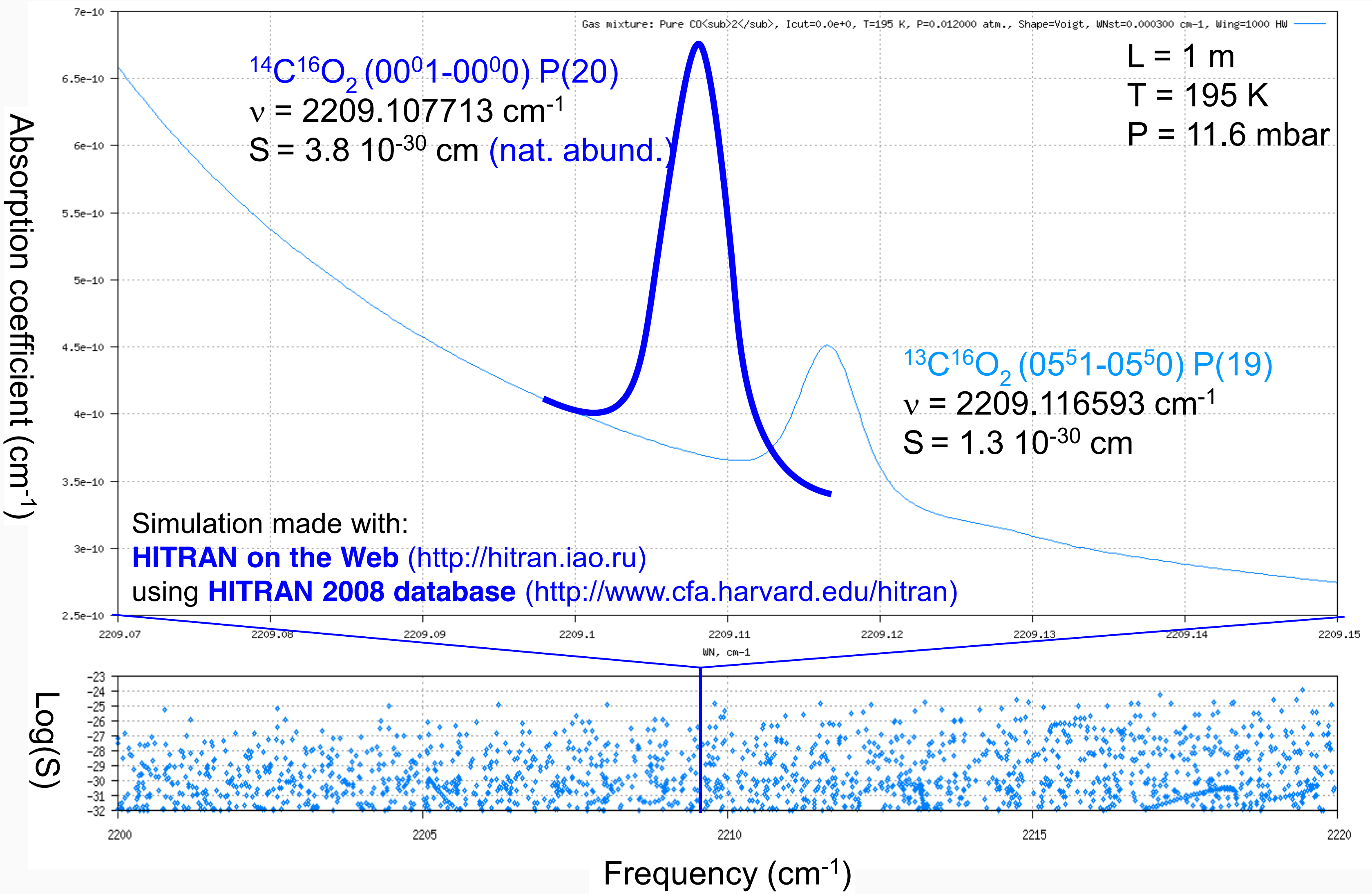
	AMS		SCAR	
	pMC	ppq	ms^{-1} MHz	ppq
modern sample 	106.68 ± 0.37	1253.3 ± 7.8	1.491 ± 0.026	1248 ± 22
fossil sample 	0.26 ± 0.05	3.1 ± 0.6	-0.004 ± 0.036	-4 ± 30

assuming no uncertainty
on linestrength!

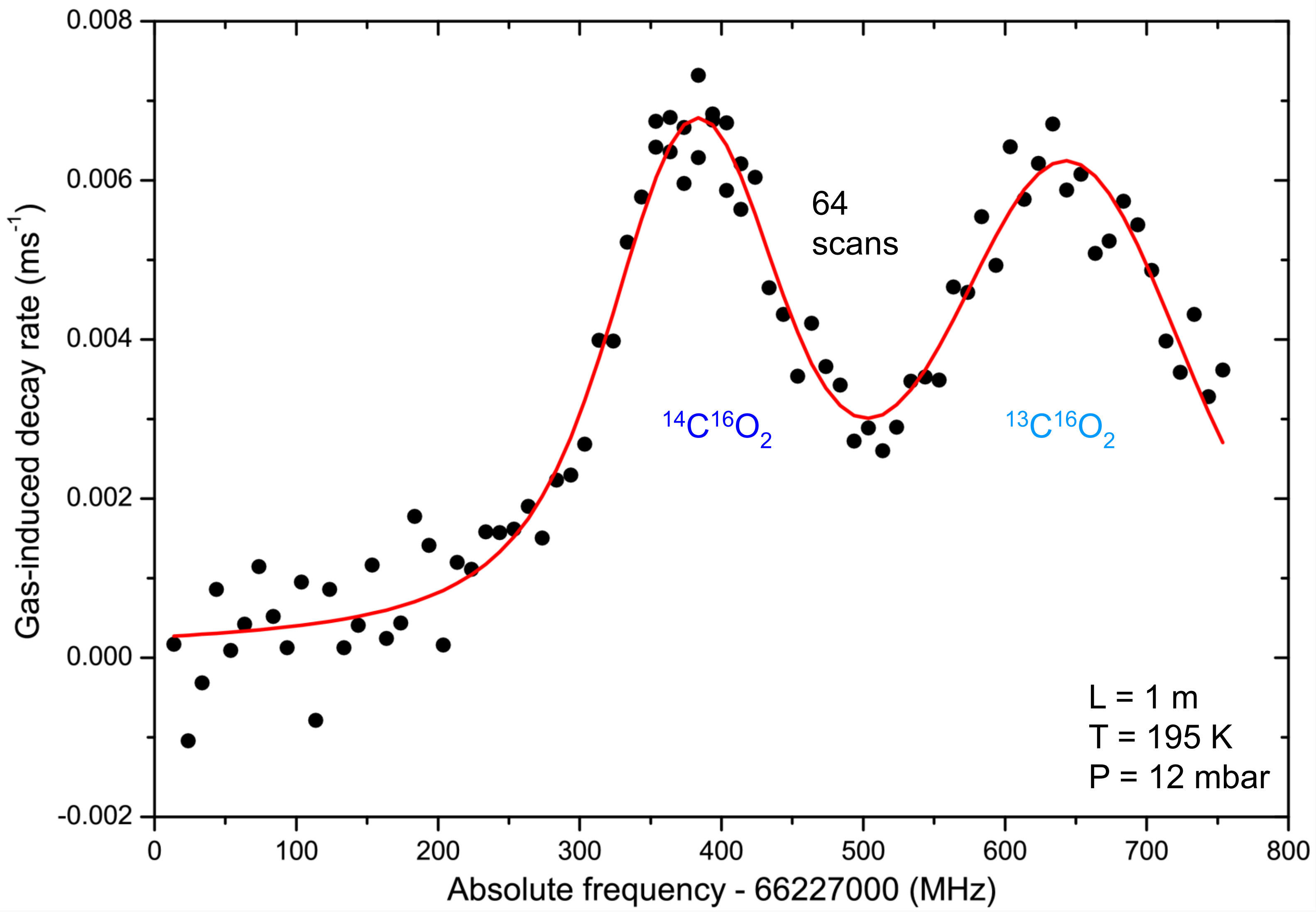
Comparison with competitor techniques

	LSC	AMS	SCAR
^{14}C detection method	β-decay count	^{14}C ion count	absorbed photons
C mass (mg)	~4000	<1	~70
Sample material	C_6H_6	C+Fe	CO_2
Measurement repeatability	restricted to same technique	restricted to same technique	allowed by any technique
Background and/or interferences	cosmic rays	^{13}CH $^{12}CH_2$	$^{13}CO_2$ (T>170 K) N_2O (>100 ppt) O_3 (>10 ppm)
Measurement time (h)	~17	~1	~3
Precision for modern samples (pMC)	~0.5	~0.3	~2
Limiting factor	Poisson statistics	Poisson statistics	optical S/N ratio
Footprint (m^2)	~1	~100	~2
Cost (k€)	~120	~3000	~300

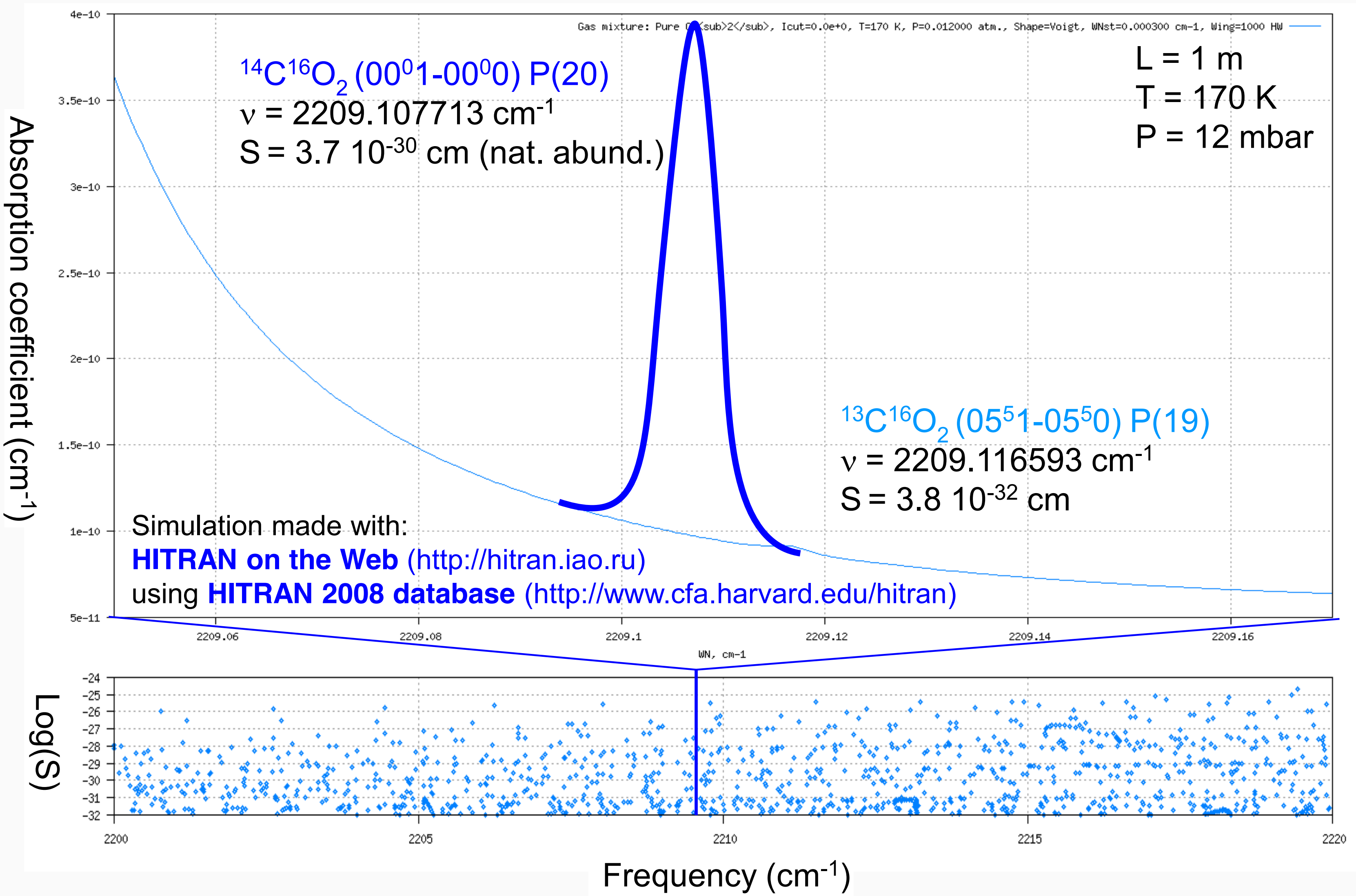
Target line for $^{14}C^{16}O_2$ detection



Interference from $^{13}C^{16}O_2$ at 195 K



Towards lower temperature



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Relative measurement precision

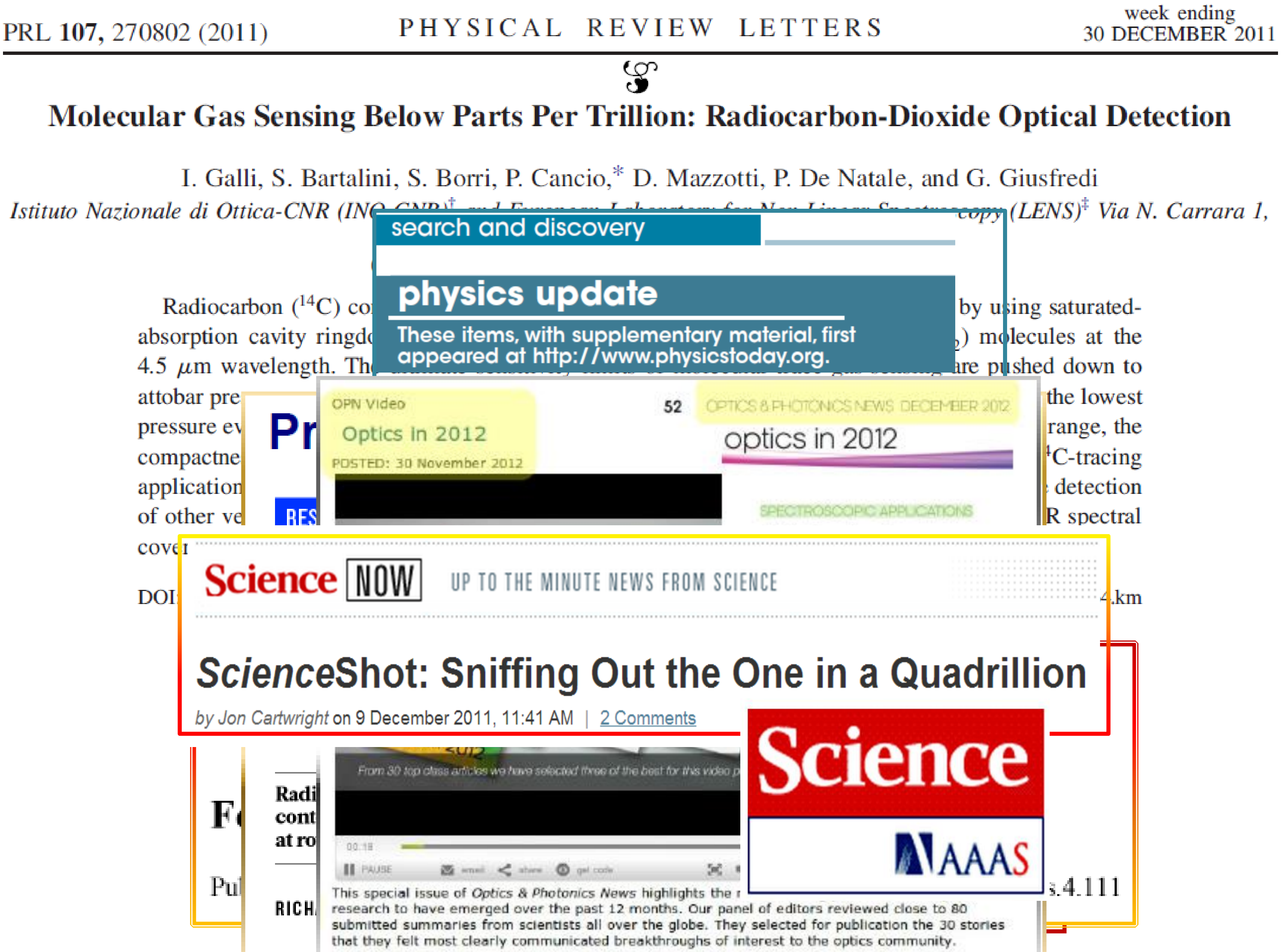
$$\boxed{\frac{P_0}{P}} = \frac{1}{n_s}\frac{P_s}{P}\frac{T}{T_s}\frac{1}{S_{mn}}\int_{-\infty}^{+\infty}\alpha(\nu;P_i,T)d\nu$$

Pressure can be measured/stabilized to better than **0.1%**

Temperature can be measured/stabilized to better than **0.3%**

Linestrength can be stabilized to better than **0.1%**

Spectral area can be measured at **2%**



http://www.osa-opn.org/home/multimedia/optics_in_2012/

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Perspectives: work in progress

	Present setup	Future setup
Cell material	stainless steel	quartz
Volume (L)	~8	~0.7
Optical finesse	~11000	~15000
C mass (mg)	~70	~6
Laser source	intra-cavity DFG	tunable CW DFB-QCL1
Absolute freq. reference	OFCS referenced to quartz/Rb/GPS clock	$^{14}C^{16}O_2$-locked/narrowed CW DFB-QCL2
Temperature (K)	195	170
Cooling method	dry ice	closed-cycle Stirling cryocooler
Footprint (m^2)	~2	~1
Cost (k€)	~300	<100

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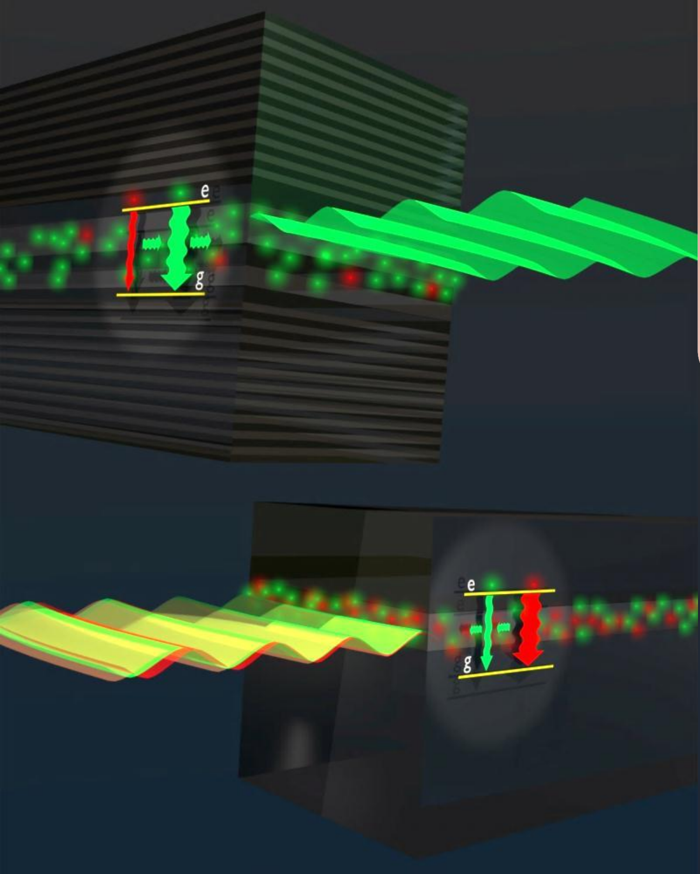
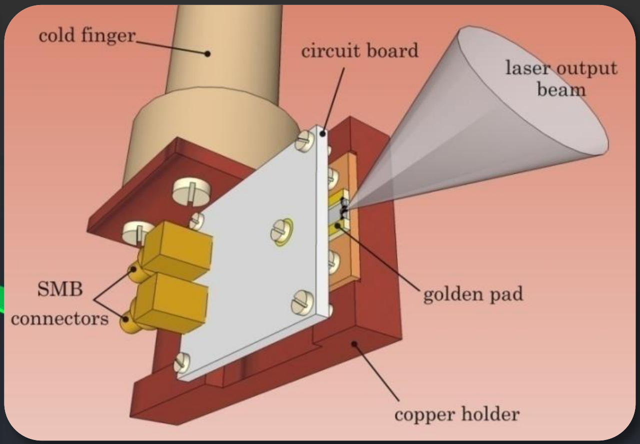
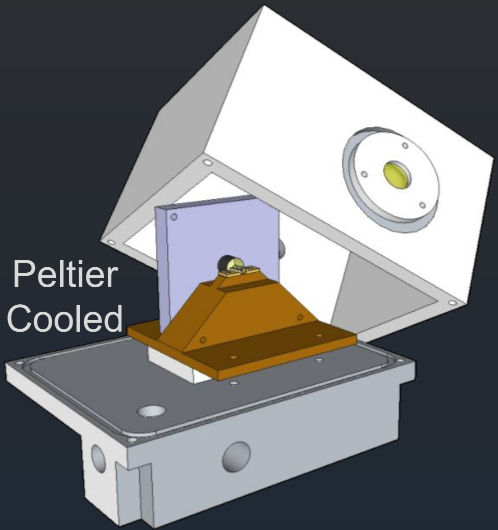
Quantum Cascade Lasers





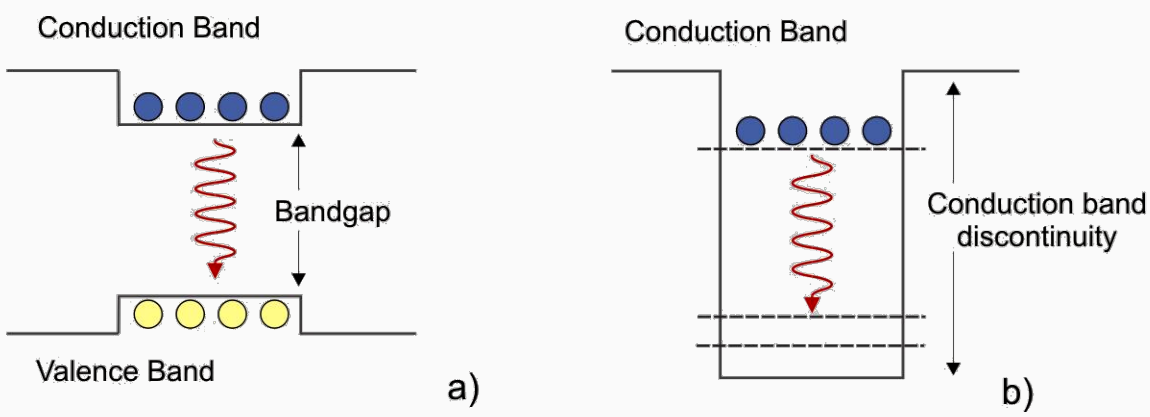


Peltier Cooled

QCLs are emerging as the main sources in the mid and far infrared:

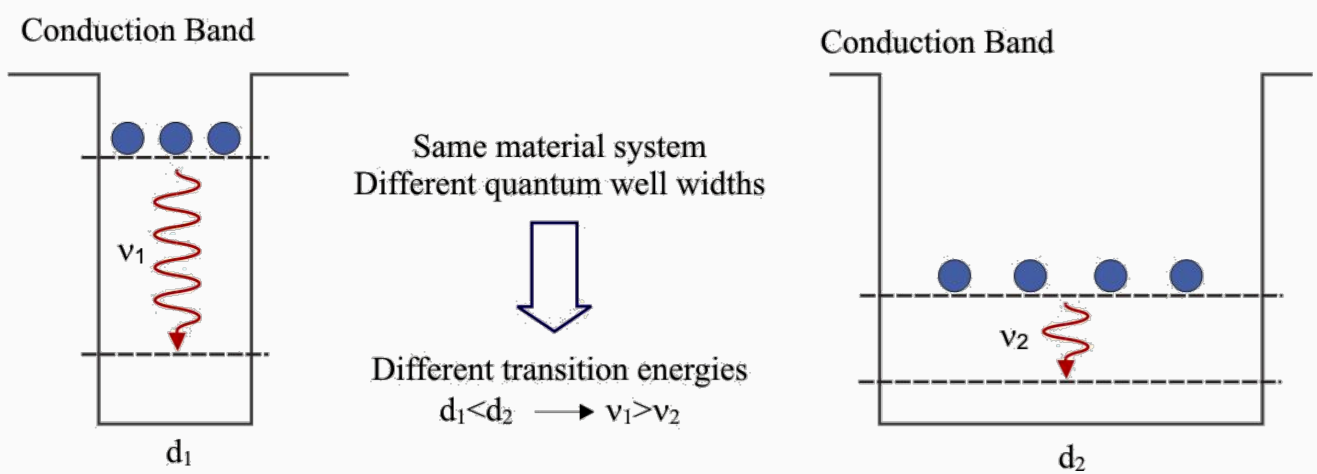
- ✓ Compactness
- ✓ Custom wavelength
- ✓ High power output
- ✓ Single mode emission

QCL vs. NIR Laser diodes

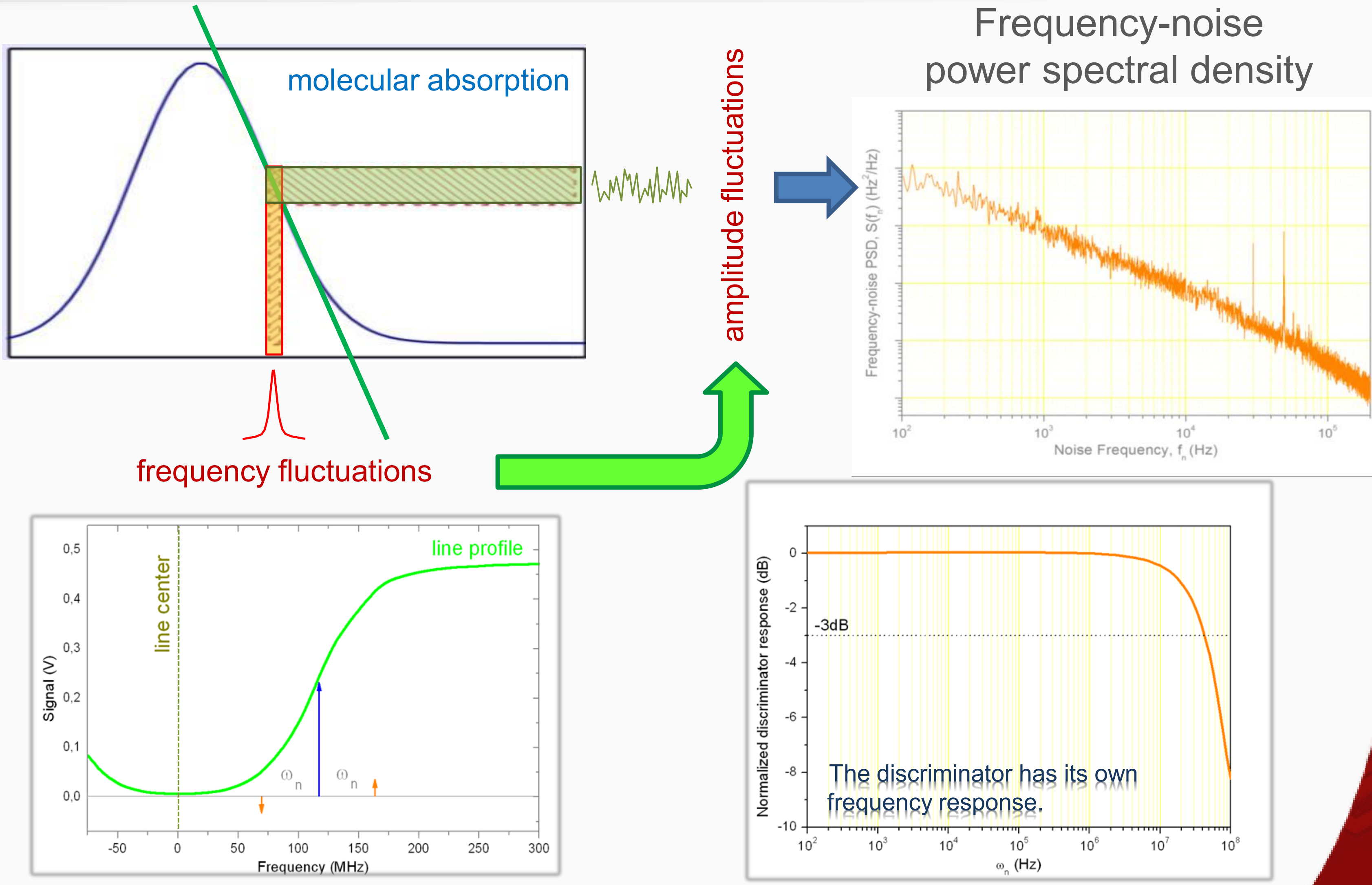


Diode laser: emission for electron-hole recombination (a).

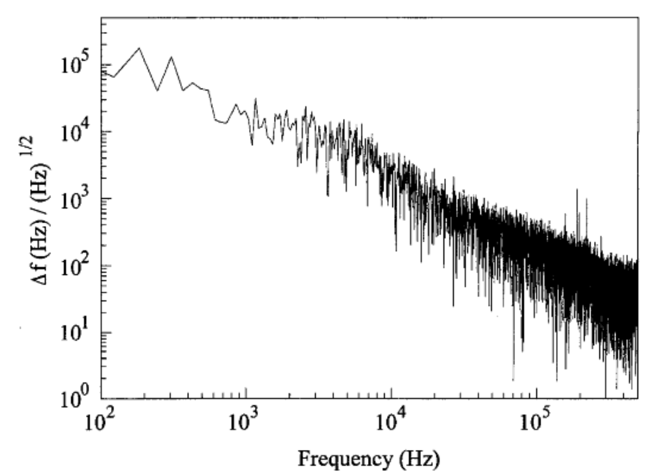
QCL: only conduction band involved (b).



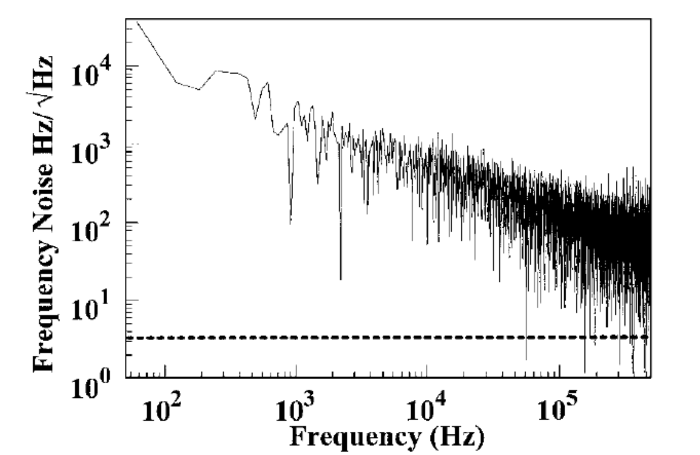
Measuring the QCL's frequency-noise: principle



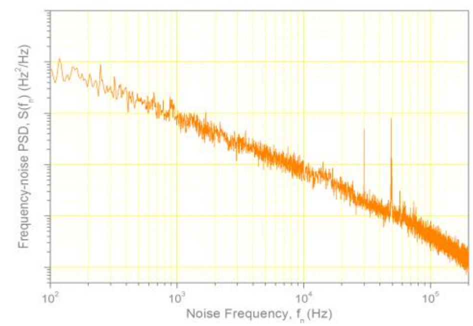
Observing QCL Intrinsic Linewidth



Williams *et al.*, Opt. Lett. **24**, 1844 (1999)



Myers *et al.*, Opt. Lett. **27**, 170 (2002)



"Observing the Intrinsic Linewidth of a Quantum-Cascade Laser: Beyond the Schawlow-Townes Limit",
S. Bartalini et al., *Phys. Rev. Lett.* **104** , 083904 (2010)
"The intrinsic limits of Quantum-Cascade Lasers"
Physics Today , p.20, (May 2010)

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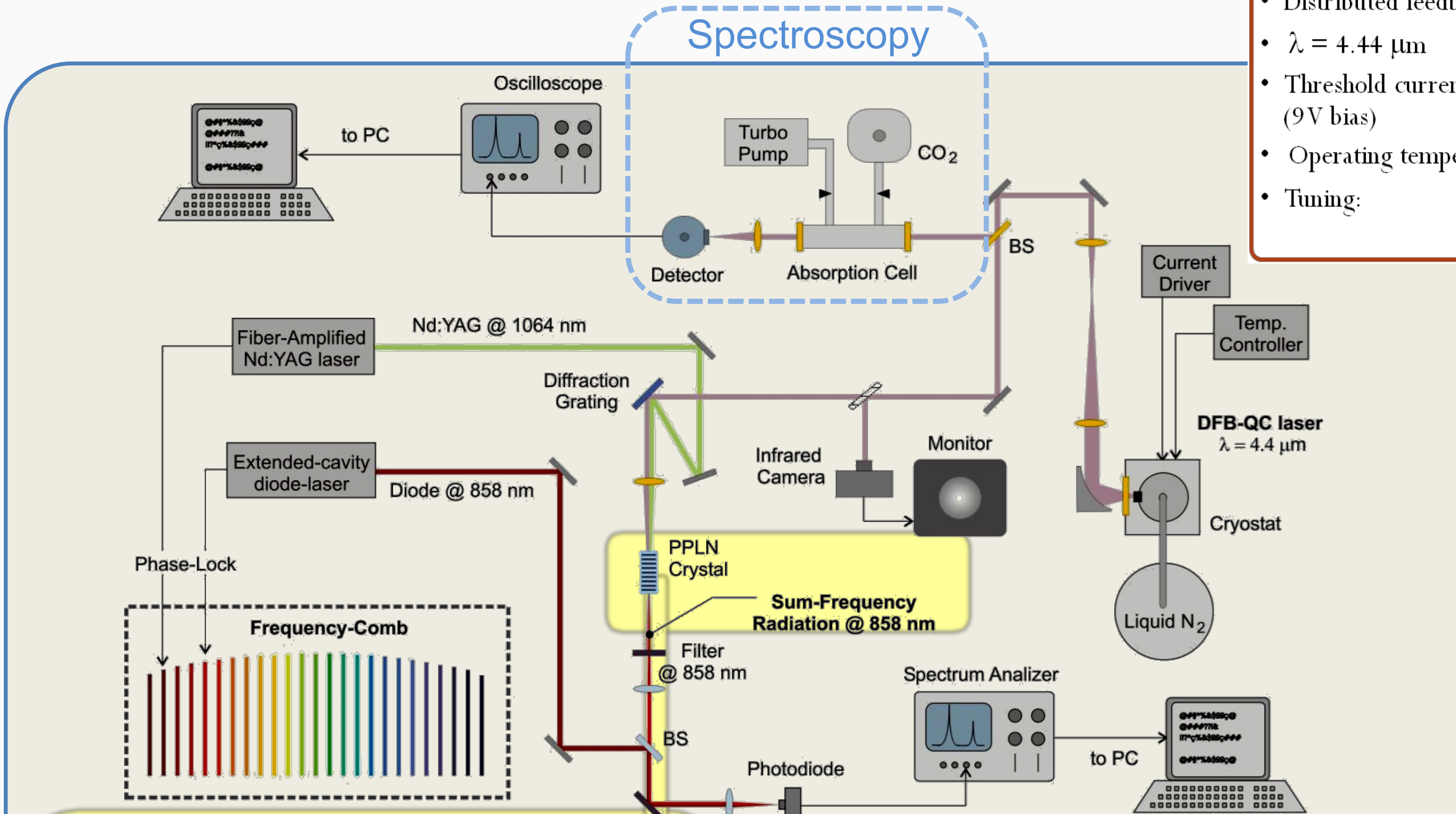
Basic QCL-Comb set-up

QCL specs:

- Distributed feedback
- $\lambda = 4.44\ \mu m$
- Threshold current: 240 mA (9V bias)
- Operating temperature: 84 K
- Tuning: ~ 840 MHz/µA
  ~ 2 GHz/K



Sum frequency generation:

- Nd:YAG power on PPLN Crystal: 1.2 W
- QC Laser power on PPLN Crystal: 2.0 mW
- Generated power @ 858 nm: 7 µW

Beat-note detection:

- Power @ 858 nm: 7 µW
- Laser Diode power: 100 uW
- Beat-note amplitude: -35 dBm; SNR = 45 dB

988 OPTICS LETTERS / Vol. 32, No. 8 / April 15, 2007

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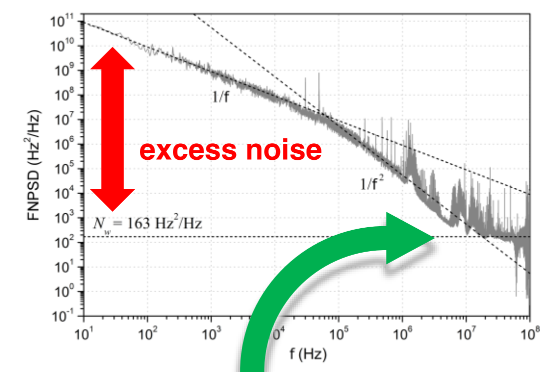
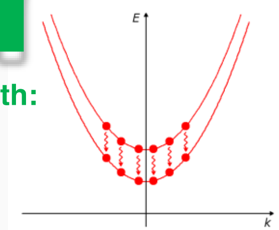
QCLs: noise suppression

Bartalini et al., PRL 104, 083904 (2010)



Intrinsic linewidth: inter-subband transition limit



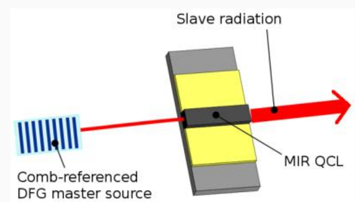
Three techniques:

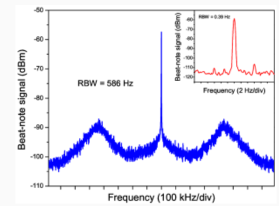
Opt. Lett. **37**, 1011, (2012)

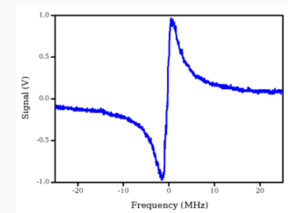
Opt. Expr. **37**, 4811 (2012)

	Optical injection locking	Phase lock	Frequency lock
Reference	Comb-referenced DFG source	Comb-referenced DFG source	Molecular absorption line
Linewidth	48 kHz	500 Hz	760 Hz

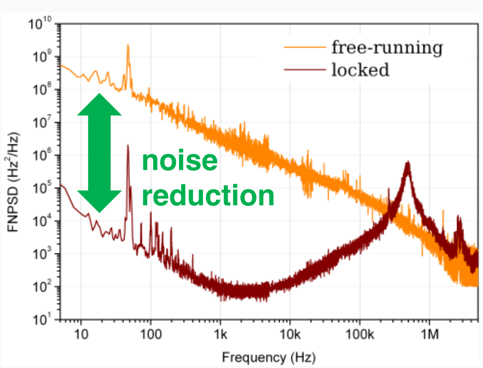
Appl. Phys. Lett. **102**, *121117* (2013)

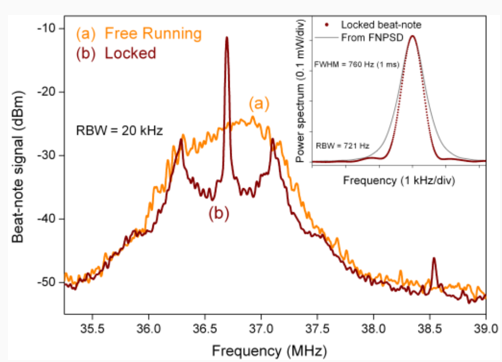




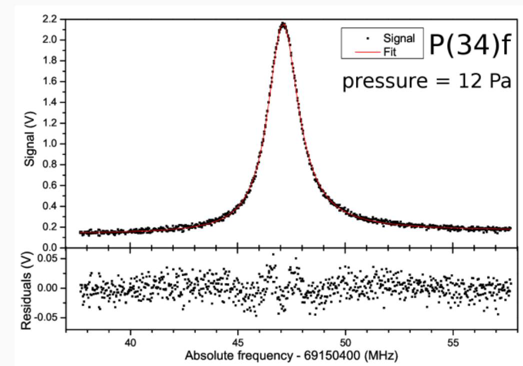


The results





CO_2 high resolution spectroscopy



Molecular Physics, **111**, 2041 (2013)

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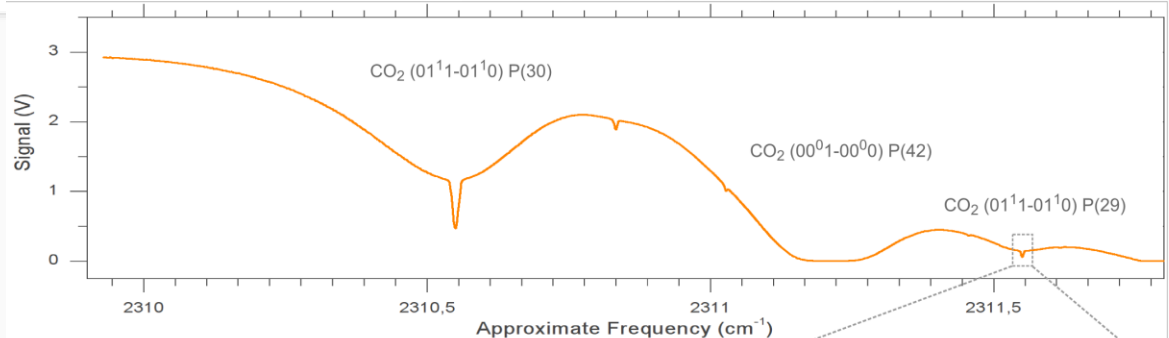
Accuracy improvement
of absolute frequency measurements



From

Doppler-limited direct-absorption spectroscopy

with a **free running** QCL

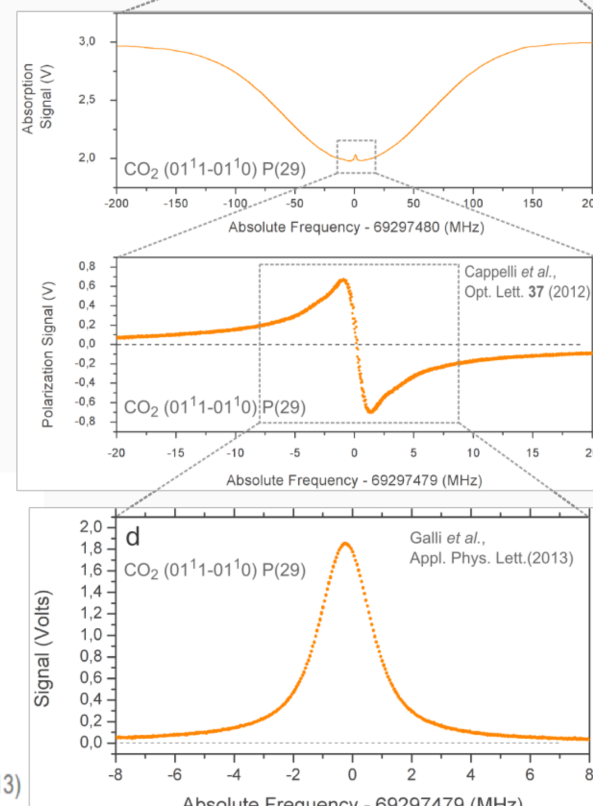
To

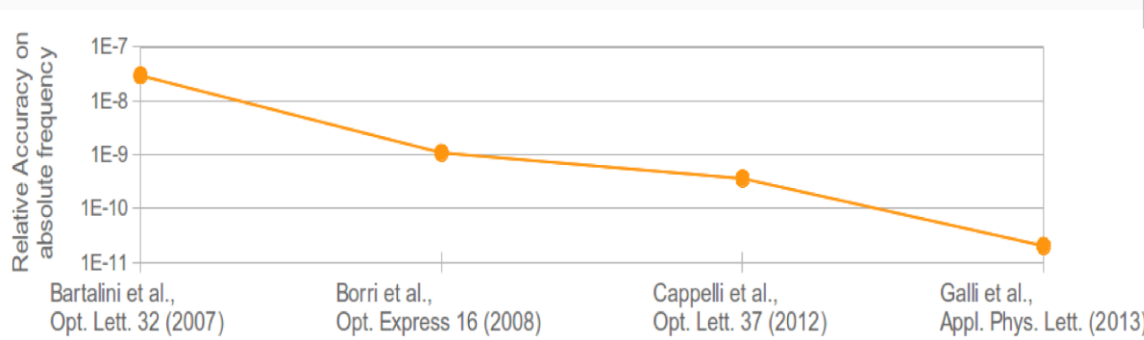
Sub-Doppler saturated-absorption spectroscopy

with a **comb-assisted** QCL

accuracy improved by

more than **3 orders of magnitude**





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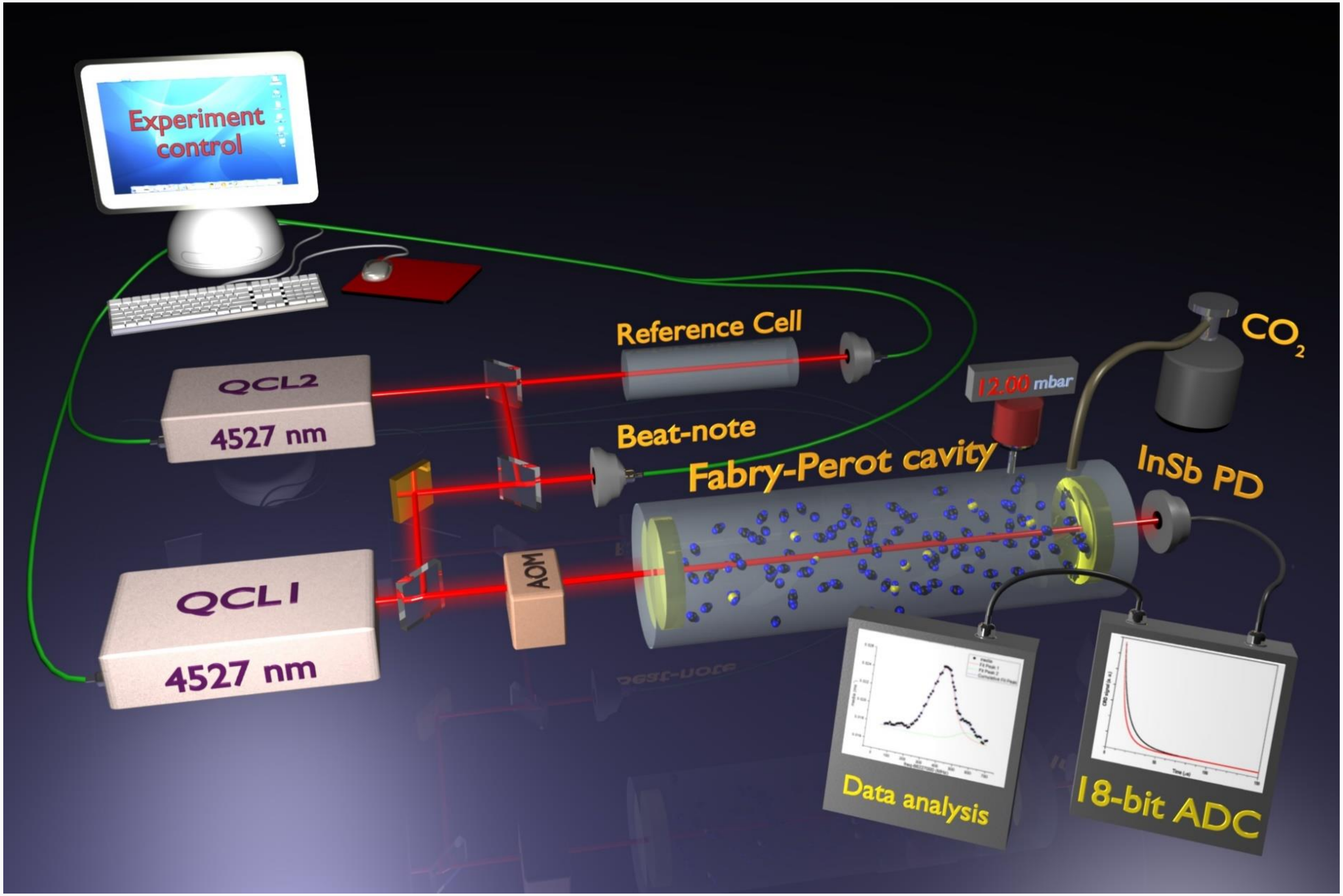
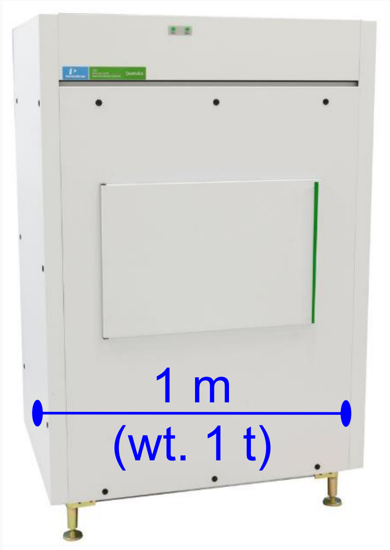
SCAR2 setup



unpublished

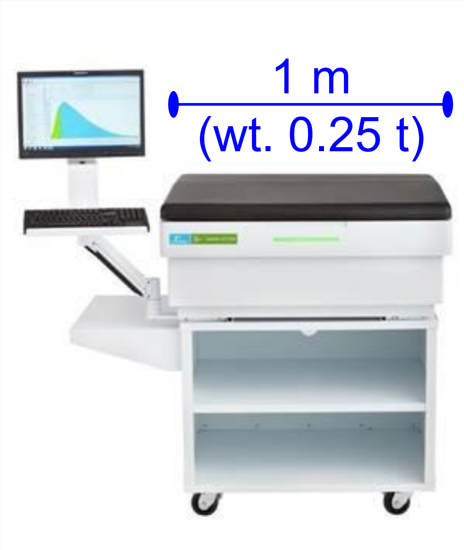
Examples of commercial apparatuses

LSC



PerkinElmer

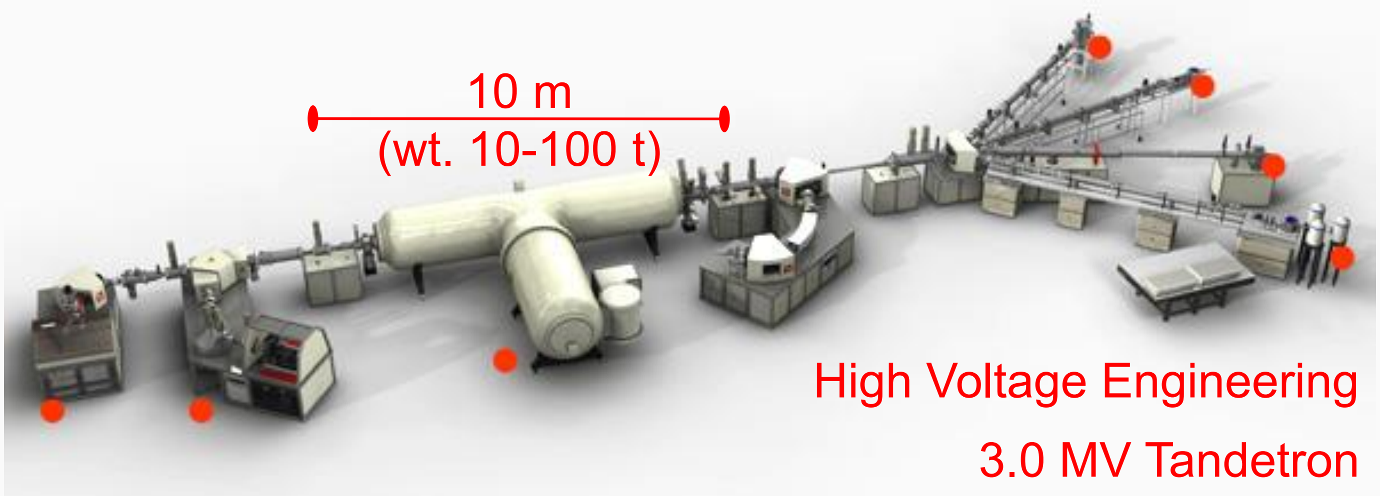
Quantulus 1220



PerkinElmer

Quantulus GCT 6220

AMS



High Voltage Engineering

3.0 MV Tandetron

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Saturated-absorption cavity ring-down (SCAR)

- ✓ Time-separated measurements in differential schemes (full/empty cavity, on/off-resonant gas) have intrinsic technical fluctuations.
- ✓ Recording CRD spectra in the saturation-absorption regime has a double advantage:
  - sub-Doppler resolution for free;
  - gas absorption can be discriminated against cavity losses in a single ring-down event.
- ✓ Due to a varying saturation parameter during the ring-down process, the decay curves are not truly exponential.
- ✓ An accurate theoretical model must be adopted to fit the experimental data, taking into account both the transverse beam shape and all homogeneous/inhomogeneous broadening mechanisms.

→ *J. Opt. Soc. Am. B* **32**, 2223 (2015)

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Technical upgrades

	SCAR1 →	SCAR2
Cell material	stainless steel	quartz
Volume (L)	~8	~0.7
Optical finesse	~11 000	~18 000
C mass (mg)	~70	~6
Laser source	intra-cavity DFG	tunable CW DFB-QCL1
Absolute freq. reference	OFCS referenced to quartz/Rb/GPS clock	N_2O-locked CW DFB-QCL2
Temperature (K)	195	170
Cooling method	dry ice	acoustic-Stirling cryocooler
Footprint (m^2)	~2	~1
Cost (k€)	~300	~150

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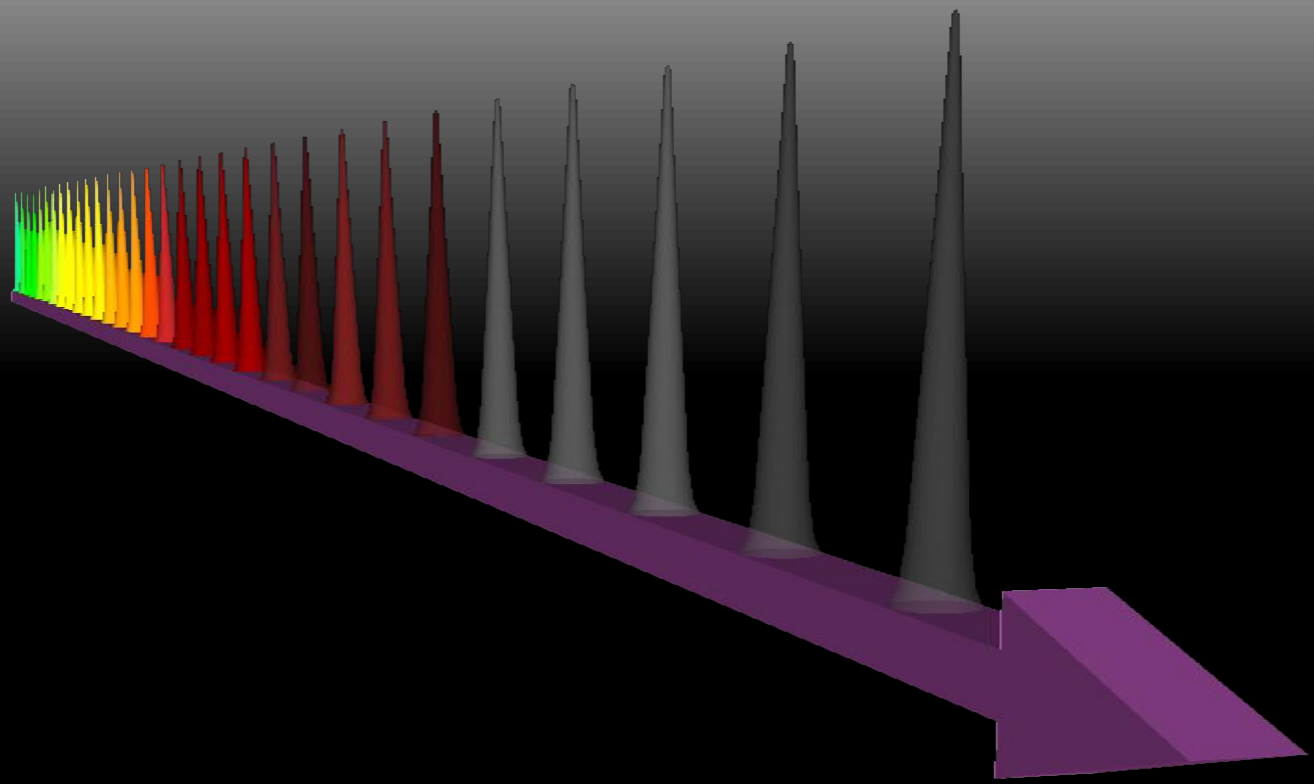
Upcoming results for SCAR2

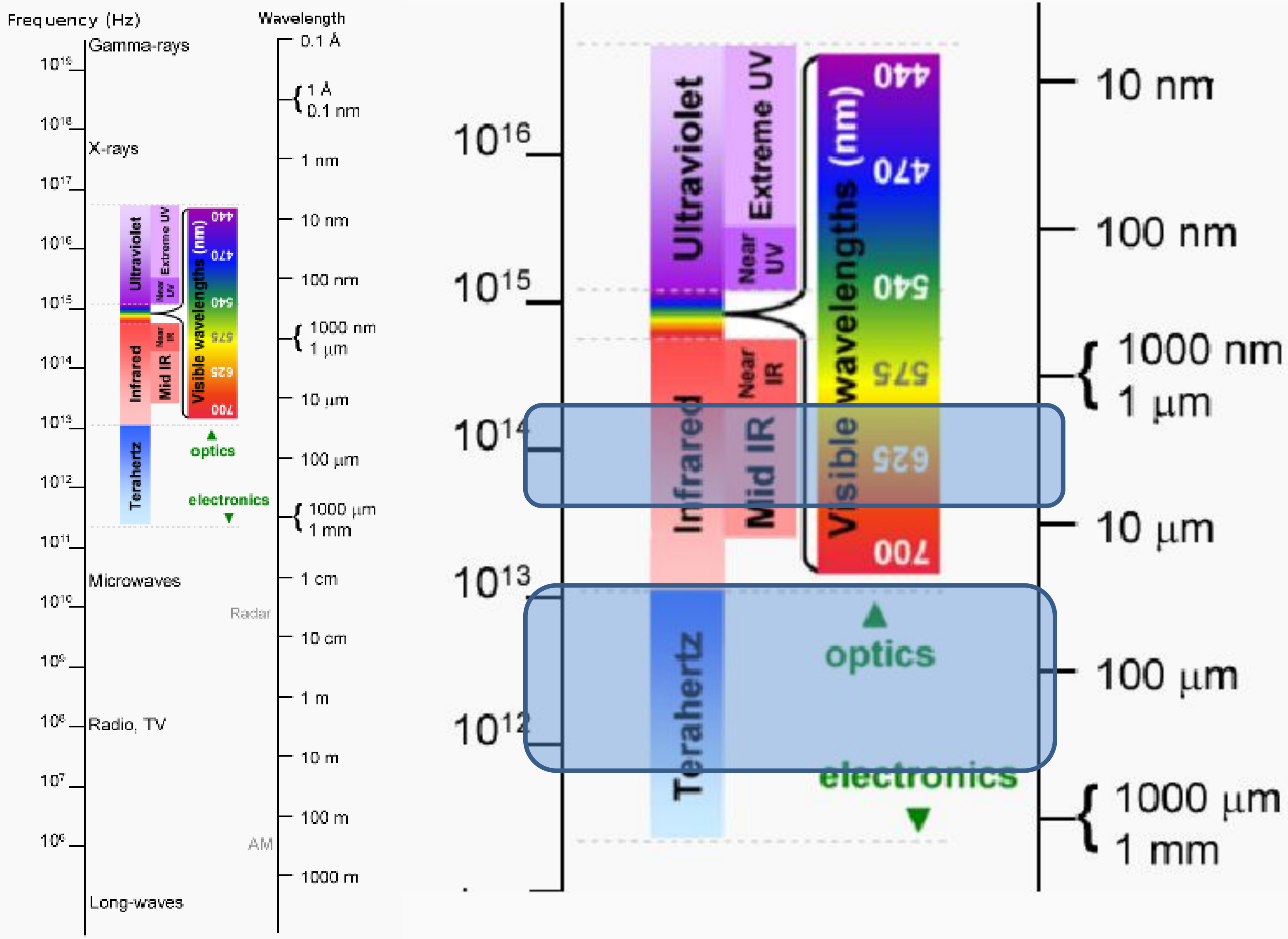
New results, demonstrating few ppq sensitivity of SCAR spectroscopy, will be published in:

Galli et al. Optica, (2016)

Moving deeper into the (Far) Infrared



Electromagnetic Spectrum



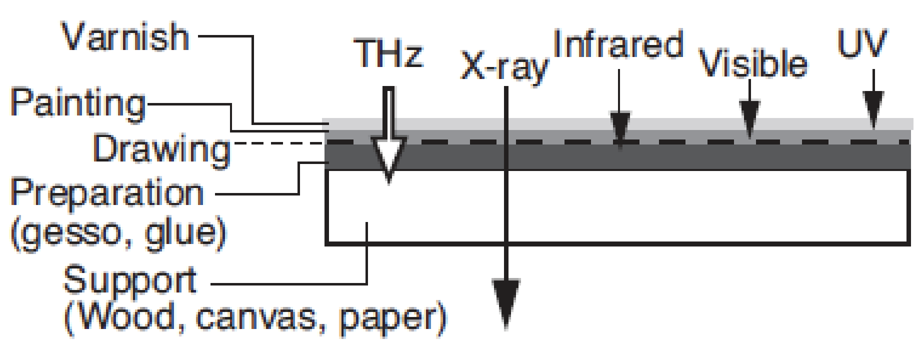
Basics

Properties & advantages

- Non-invasive, non-ionizing, non-contact and non-destructive examination method
- Can penetrate opaque materials
- Complementary to traditional methods such as X-ray, UV, VIS and IR imaging/spectroscopy
- Radiation frequency can tune the depth of penetration

State of the art

- Rapid instrumental progress and development in last decades
- No commercial spectral libraries in spite of a range of commercial instruments
- Currently a database with 500 spectra of art materials (pigments, dyes, binders)



Comb-assisted QCL-based THz Spectroscopy

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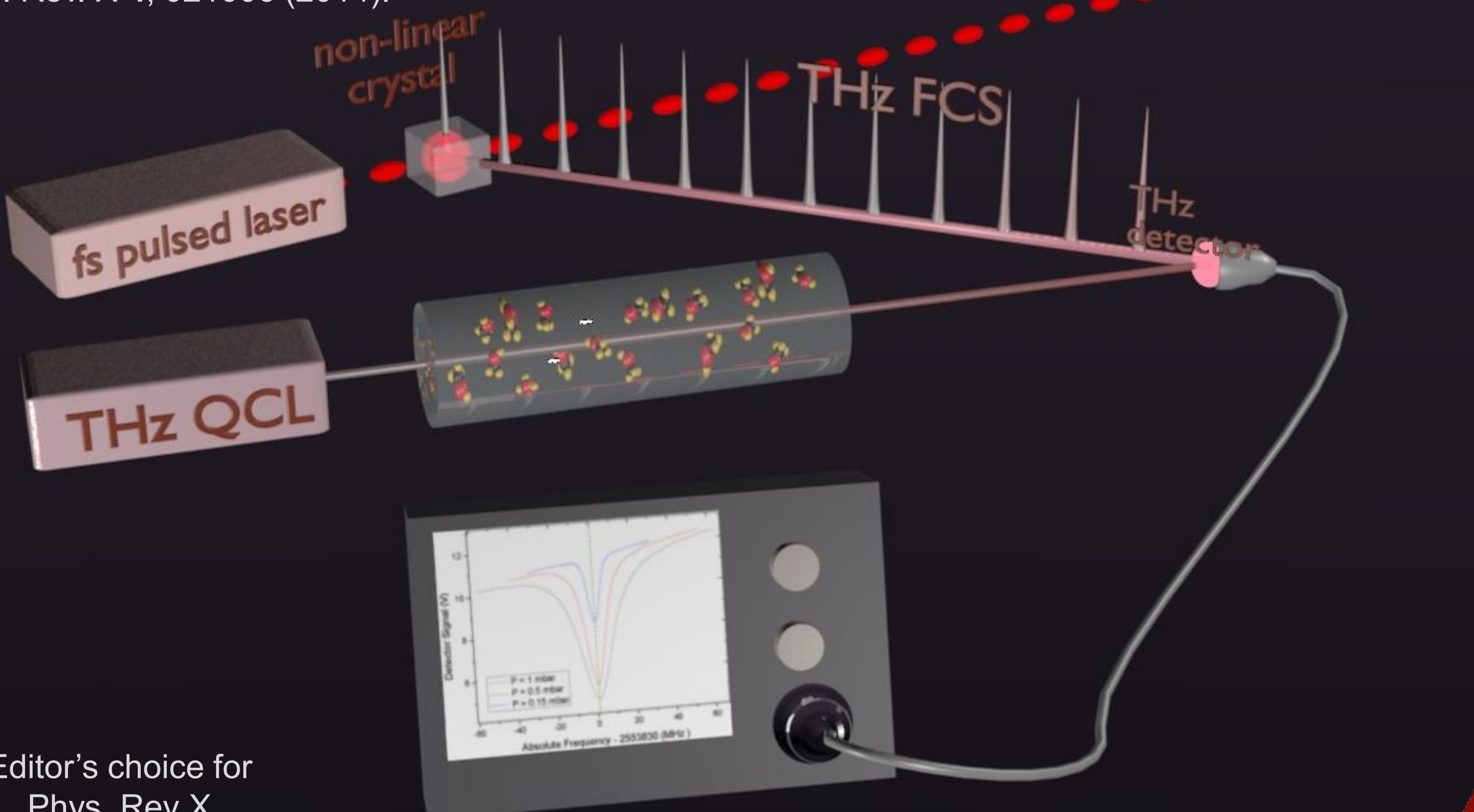
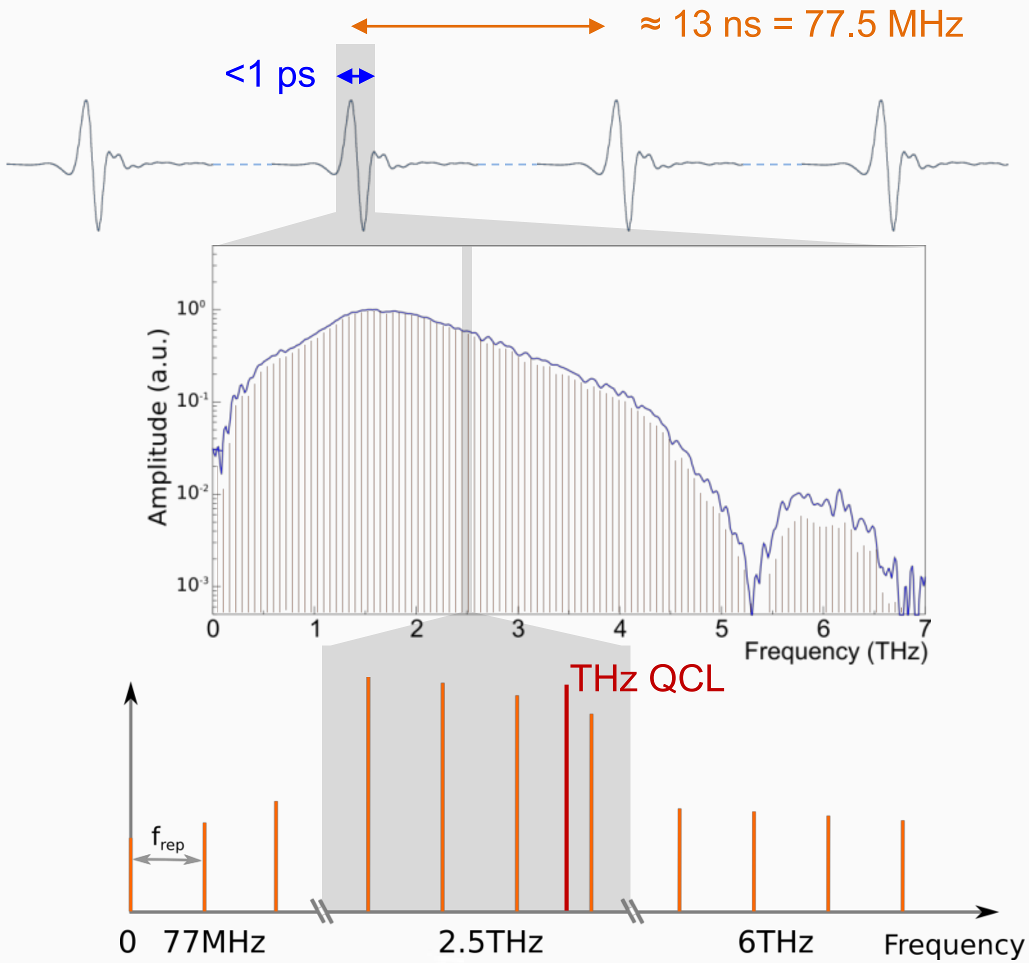
S. Bartalini et al,
Frequency Comb Assisted Terahertz Quantum Cascade Laser Spectroscopy,
Phys. Rev. X **4**, 021006 (2014).



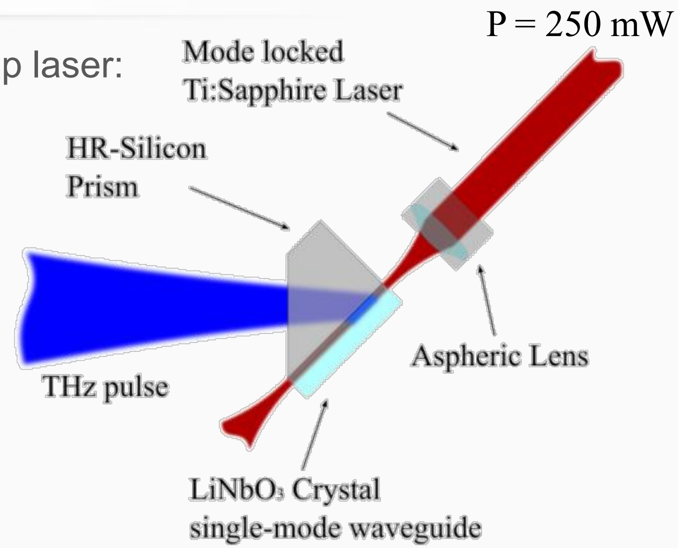
Editor's choice for
Phys. Rev X
Highlights

THz pulsed radiation: THz comb

Usually employed for THz TDS, but…



Pump laser:



Total power = 1 μW
Power per tooth = 30 pW

zero-CEO frequency
6-octaves-spanning COMB
(more than 60000 teeth)

S. Yokoyama, *Opt. Express* **16**, 13052(2008).
L. Consolino *et al.*, *Nat. Comm.* **3**, 1040 (2012).

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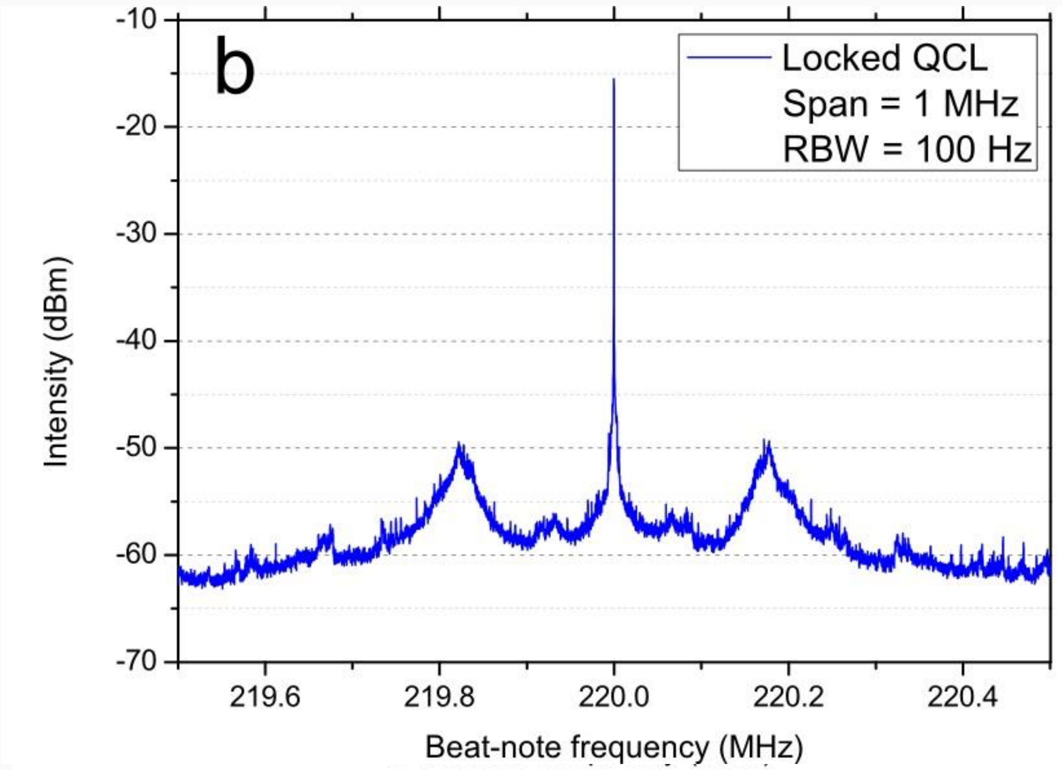
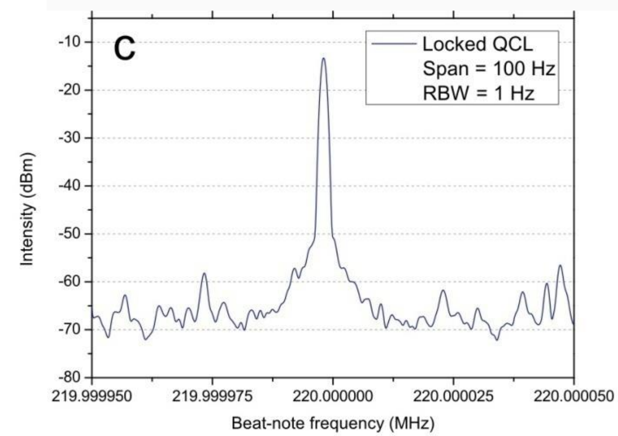
Phase-lock of a THz QCL to a THz COMB



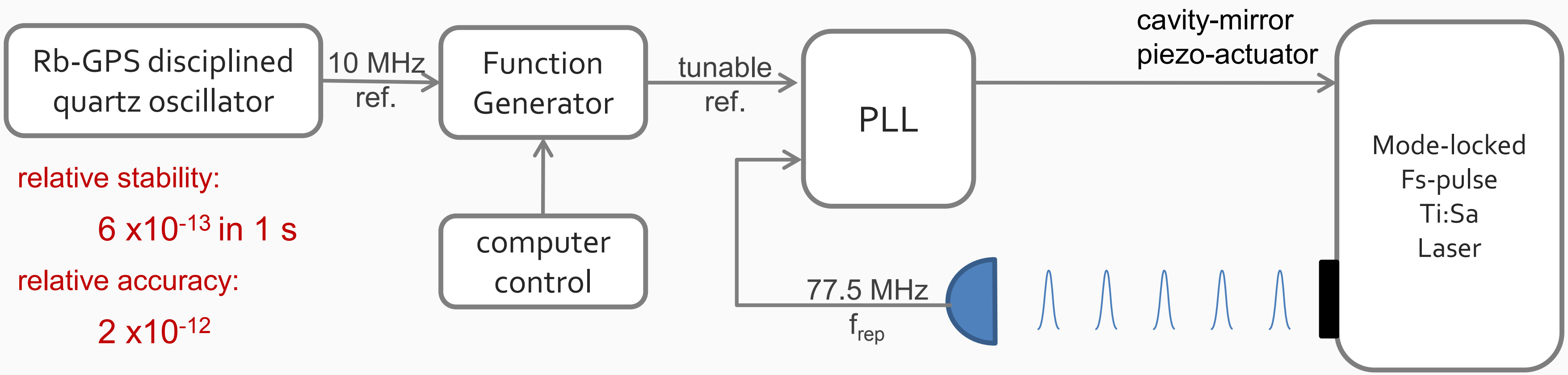
About 70-80% of the QCL power effectively phase-locked

S/N = 50 dBm (1 Hz – RBW)

Electronic Bandwidth = 200 kHz



Stabilization of the COMB repetition rate

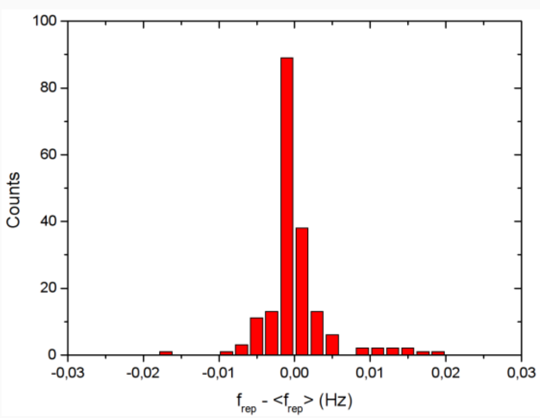


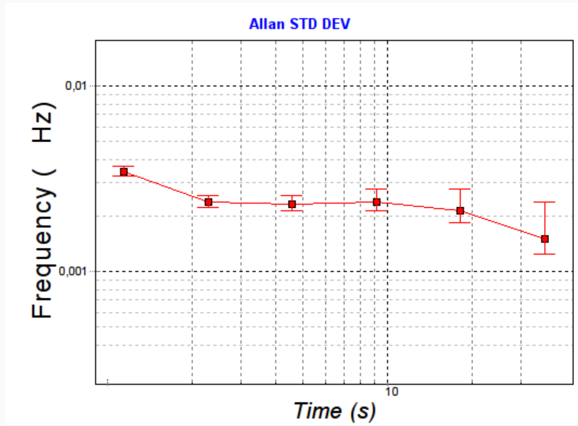
relative stability:

6×10^{-13} in 1 s

relative accuracy:

2×10^{-12}





For the THz comb:

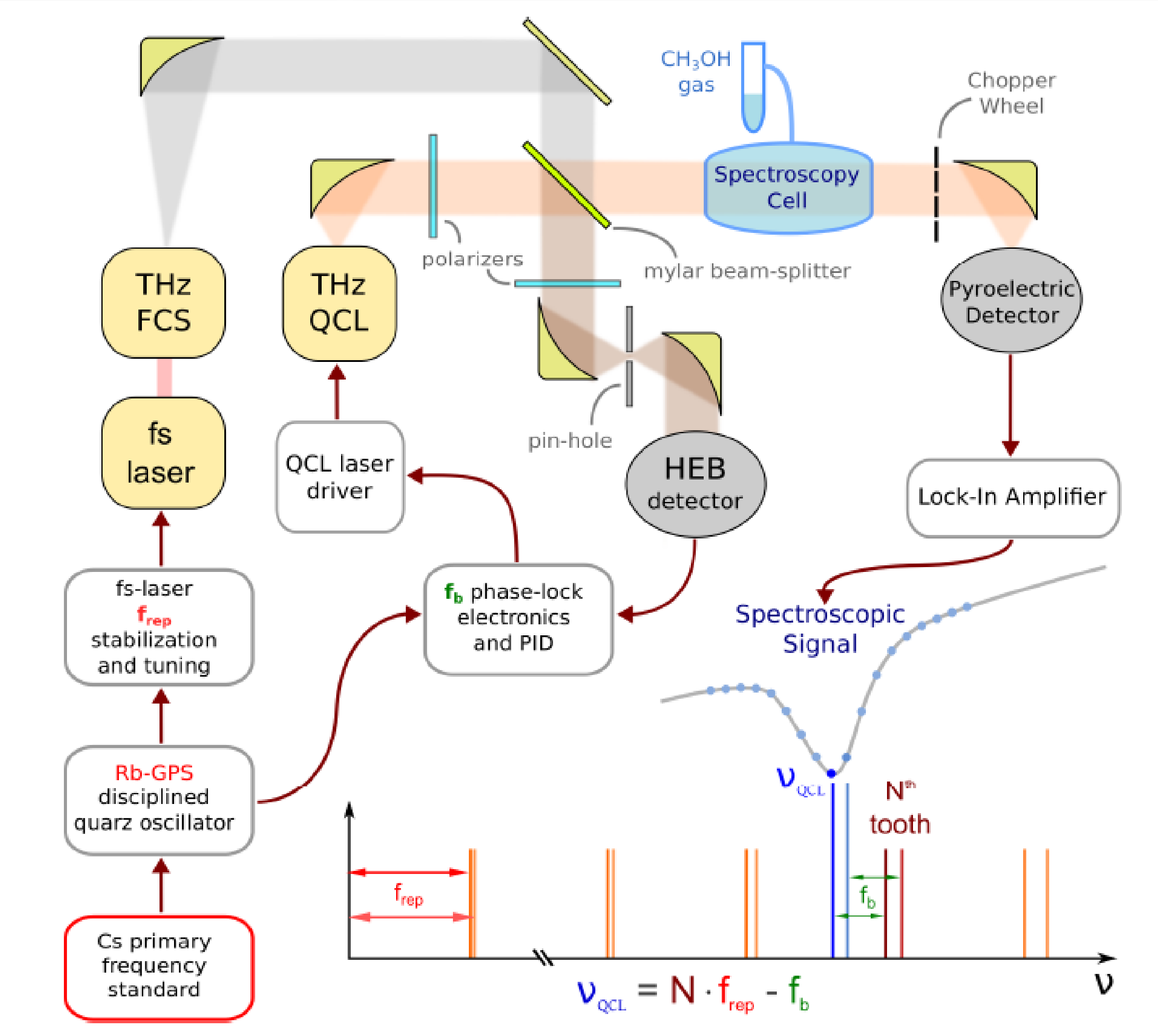
- offset instabilities cancel out
- only f_{rep} instabilies contribute to the tooth linewidth

Allan variance < 4mHz

Measured f_{rep} relative stability better than 5×10^{-11}

~100Hz level @ 2.5 THz

Spectroscopic Setup



THz-comb-assisted Spectroscopy

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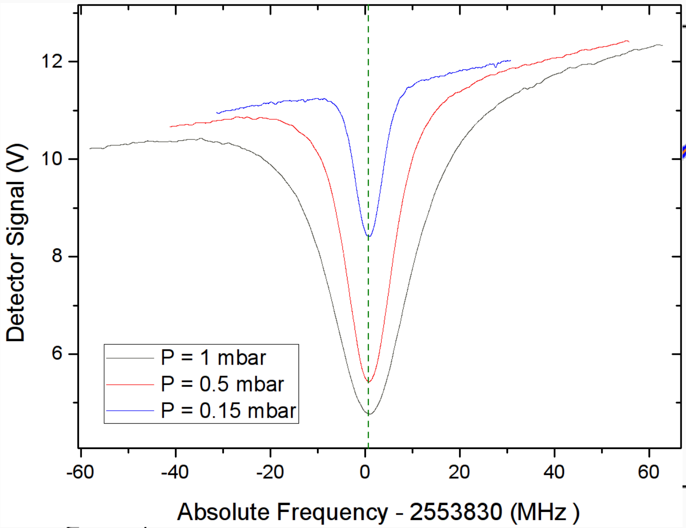
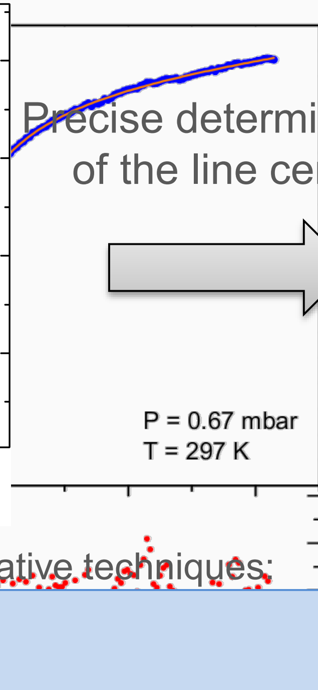
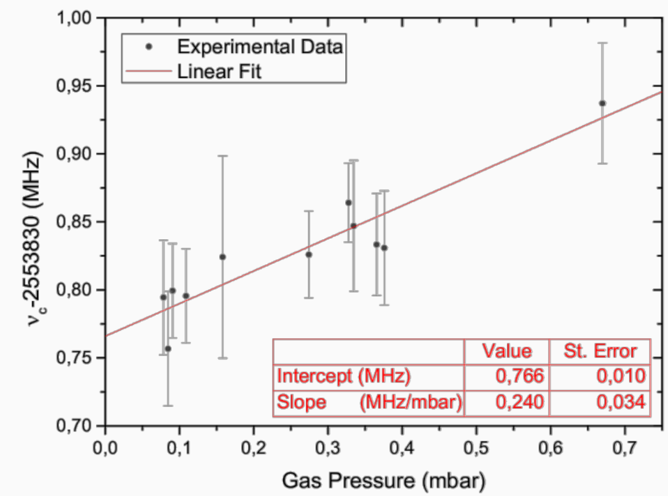
ν_0 = 2. 553 830 766 (10) THz





Precise determination of the line center



Comparison with state-of-the-art alternative techniques:

Transition linewidth ~ MHz (FWHM)

S/N > 100

expected error on ν_c: ~ 10 kHz

Doppler limited spectroscopy

QCL based metrology

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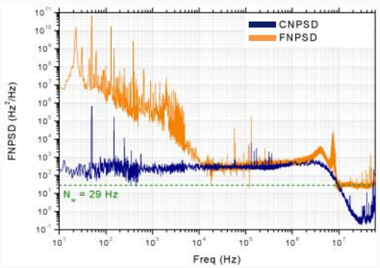
SOURCE: QCL



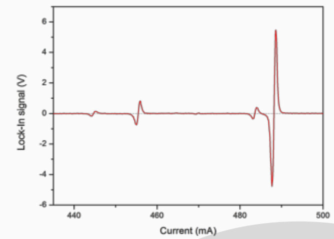
M.S. Vitiello *et al.*
Nat. Photon. **6**, 525 (2012).

THz QCLs are suitable for metrological-grade experiments

$\Delta\nu = \pi N_w \sim 90$ Hz

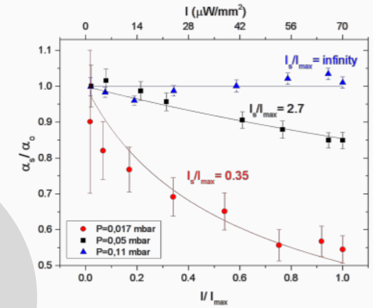
SPECTROSCOPY

High-sensitivity techniques

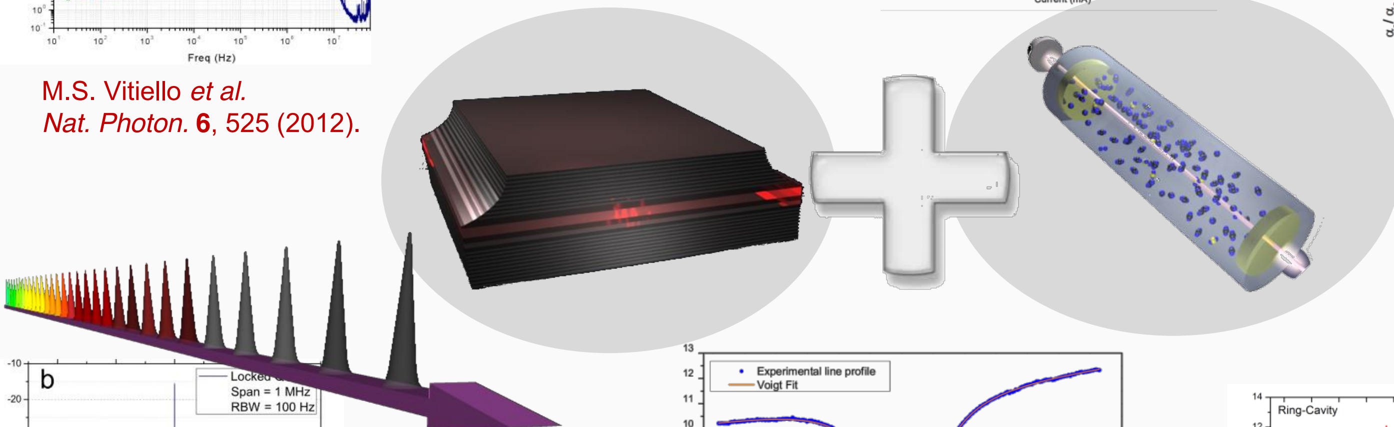


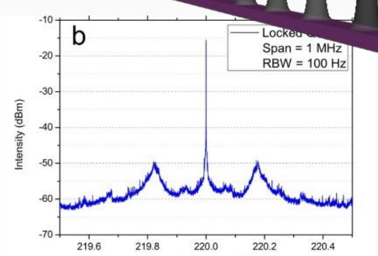
L. Consolino *et al.*, Sensors **13,** 3331 (2013).

Saturation



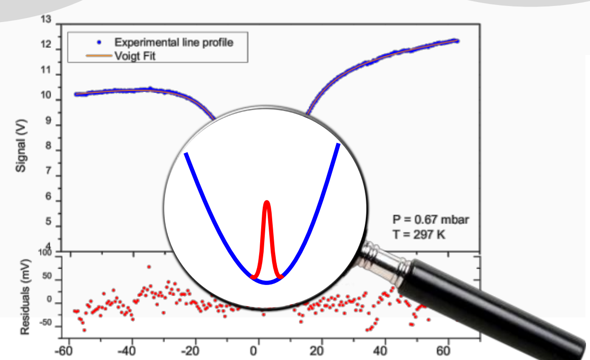
L. Consolino *et al.*
Appl. Phys. Lett.
106, 021108 (2015)

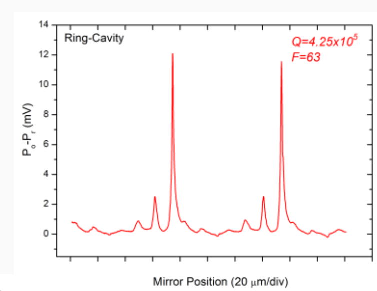




L. Consolino *et al.*,
Nat. Comm. **3**, 1040 (2012).

Cavity enhancement





A. Campa *et al.*
Optics Express,
23, 3751 (2015).

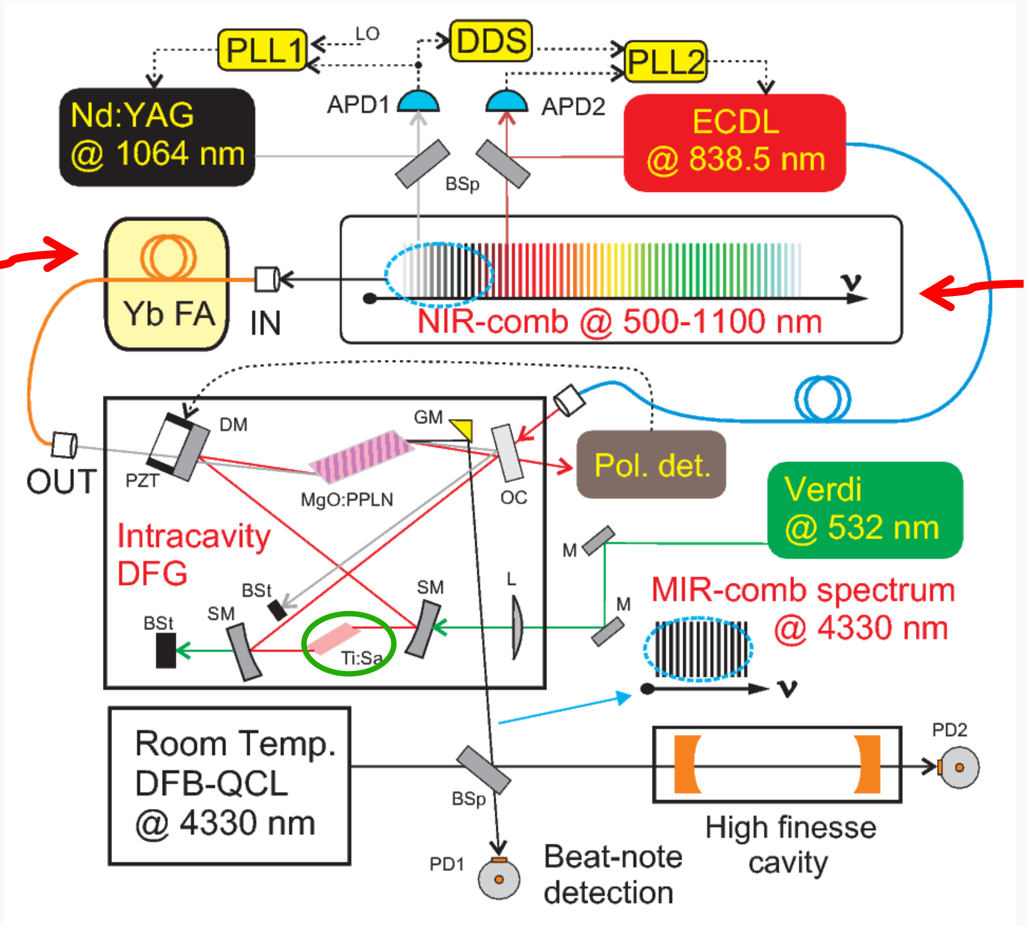
Absolute Frequency

S. Bartalini *et al*, *Phys. Rev. X* **4**, 021006 (2014).

IC-DFG-comb generation: the setup

First approach: **intracavity DFG generation**.

Yb FA output (*signal*): 1032 – 1045 nm

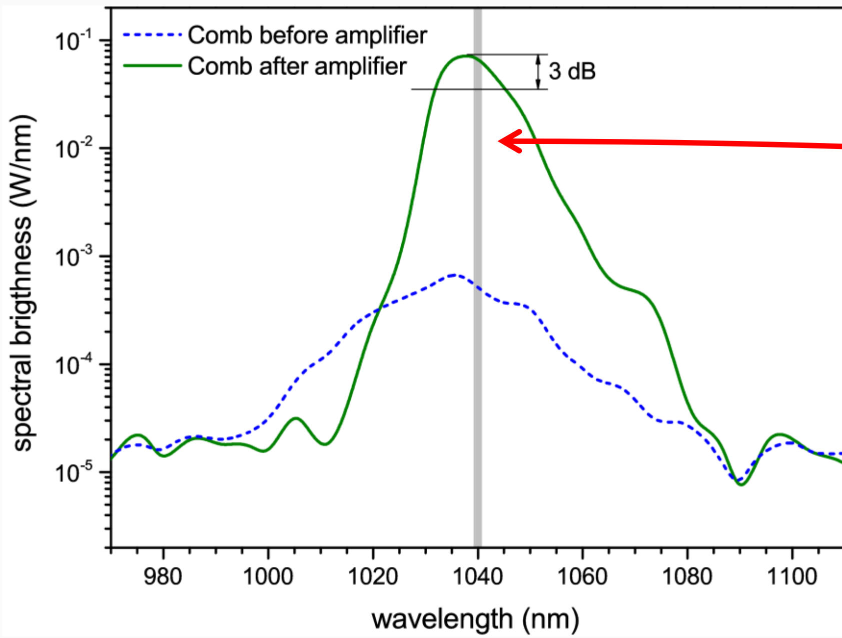


NIR-comb: mode-locked Ti:Sa with $f_r \approx 1$ GHz

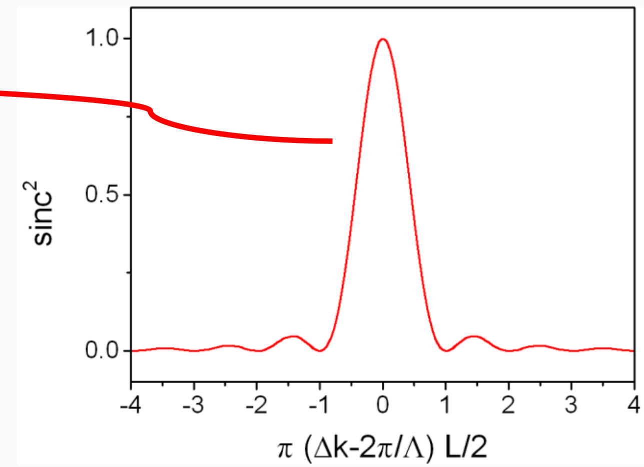
Galli et al., Opt. Express 21, 28877-28885 (2013).

IC-DFG-comb : the characteristics

NIR-comb spectrum



IC-DFG-comb envelope
(phase-matching bandwidth)



FWHM = 27 nm

	tunability	power
ECDL/Ti:Sa	838 – 863 nm	30 W
NIR-comb	1032 – 1045 nm *	5 W (ampl.)
IC-DFG-comb	4.2 – 5.0 µm	0.5 mW

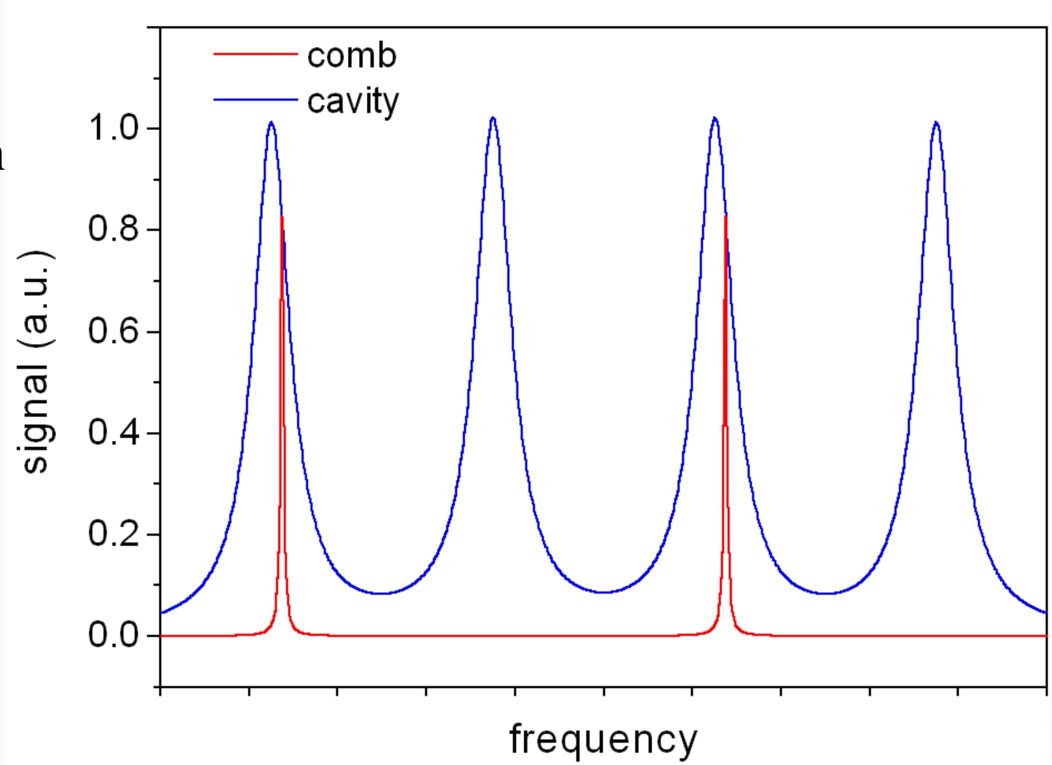
* amplified NIR-comb FWHM

IC-DFG-comb : the high-finesse cavity

Characteristics:

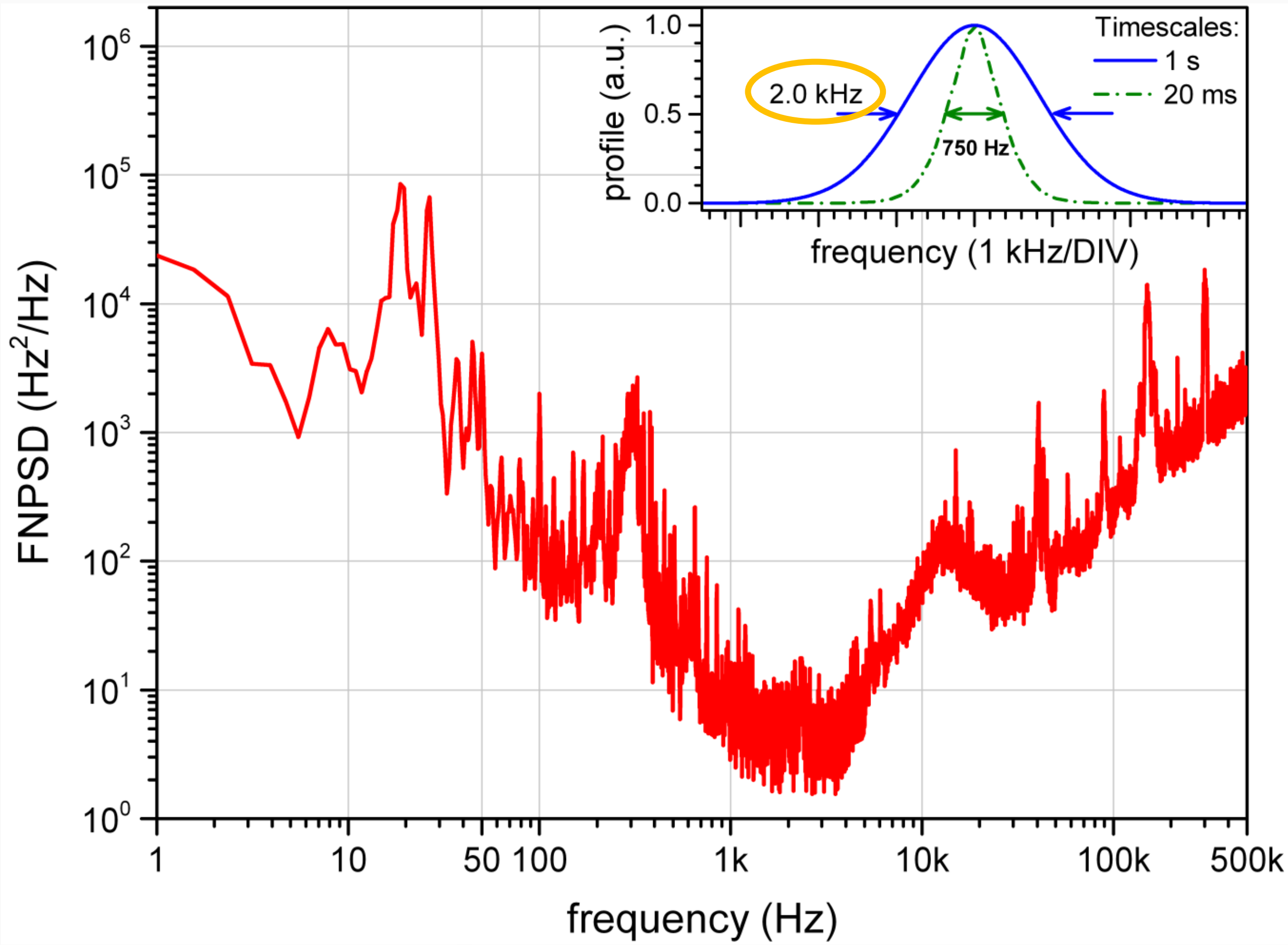
Length = 1 m Finesse = 8000

To **maximize** the transmission we have matched the repetition rate of the comb and the free spectral range of the cavity.



IC-DFG-comb : the teeth linewidth

The FNPSD of the comb in matching condition:



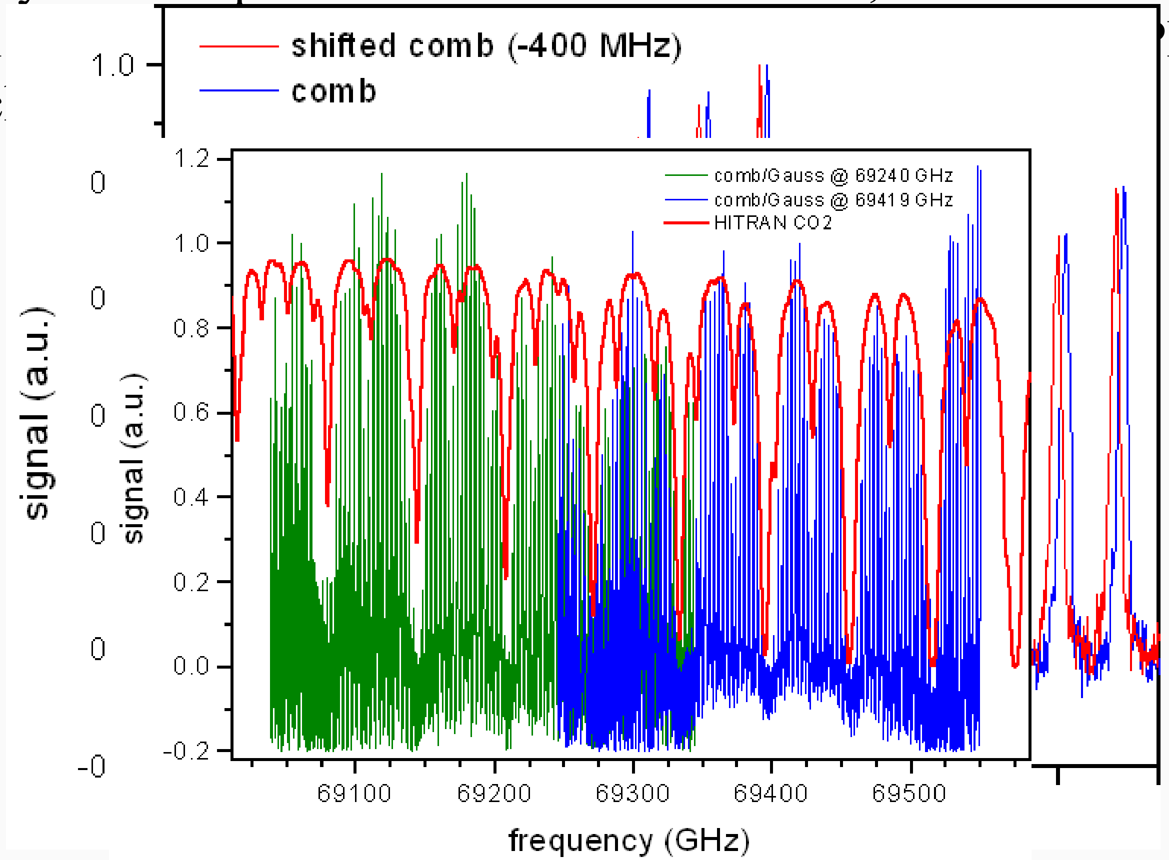
2 kHz linewidth
instead of
100 kHz
thanks to the **DDS**

Galli et al., Opt. Express 21, 28877-28885 (2013).

IC-DFG-comb : CO_2 spectroscopy

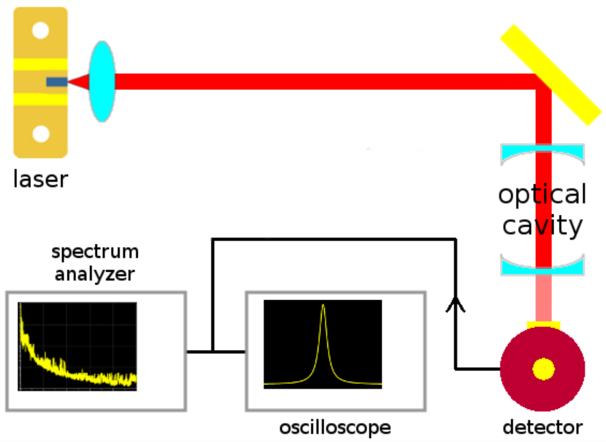
It is also possible to shift the comb:

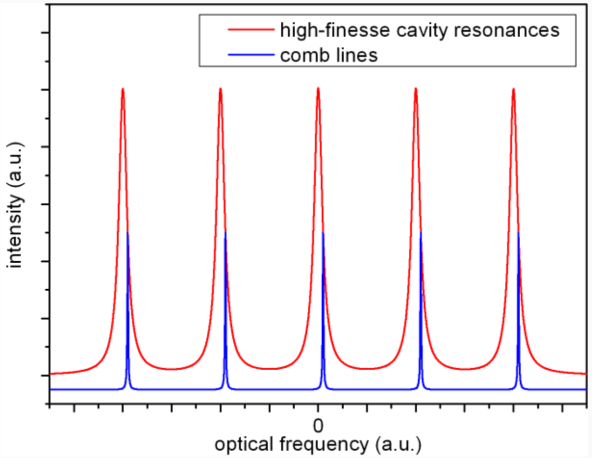
- **fine shift** in order to cover the frequencies between the teeth, either by tuning the pump frequency or the repetition rate of the NIR-comb;
- **wide shift** in ... PPLN crystal temperature (c...

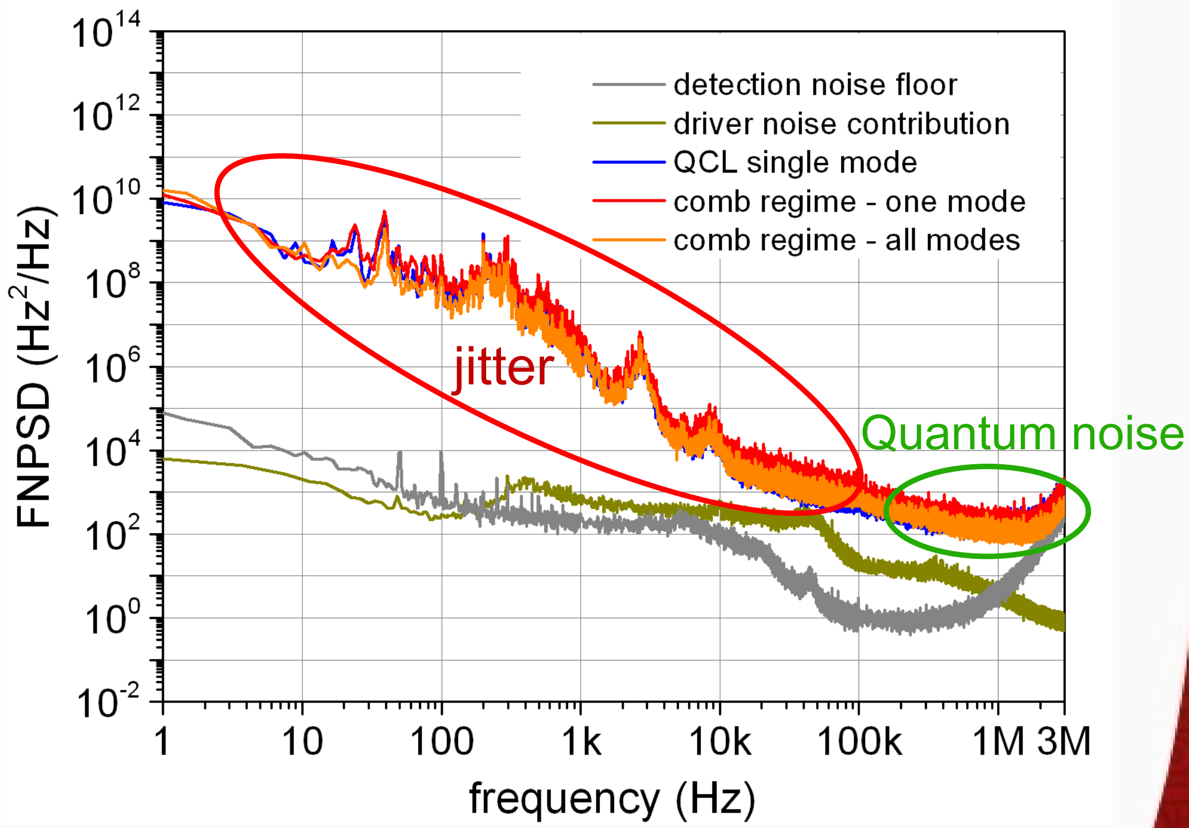


Galli et al., Mid-infrared frequency comb for broadband high precision and sensitivity molecular spectroscopy, Opt. Lett. 39, 5050-5053 (2014)

QCL-comb: the frequency noise



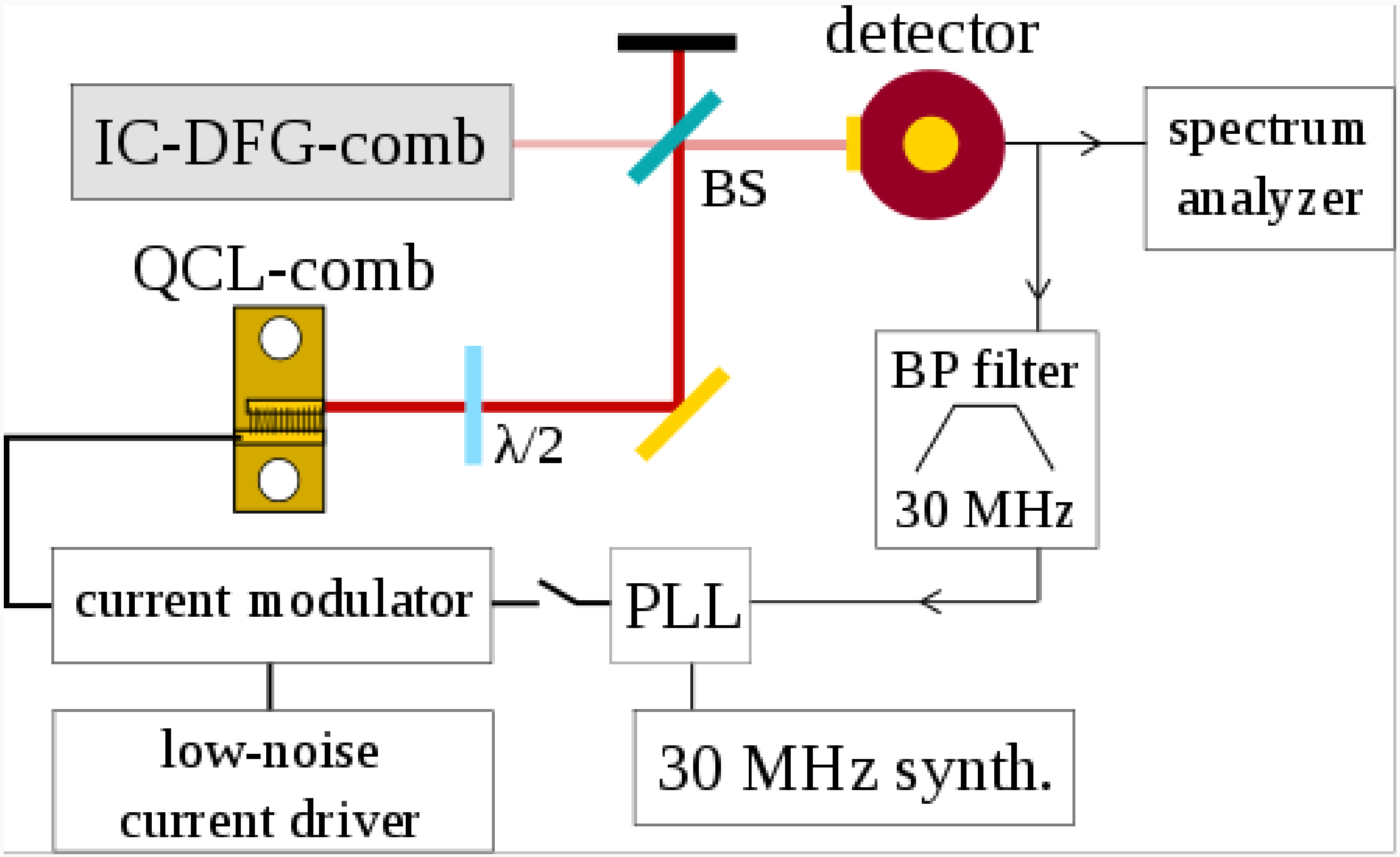




The modes are coherent among themselves, but the overall emission has to be stabilized.

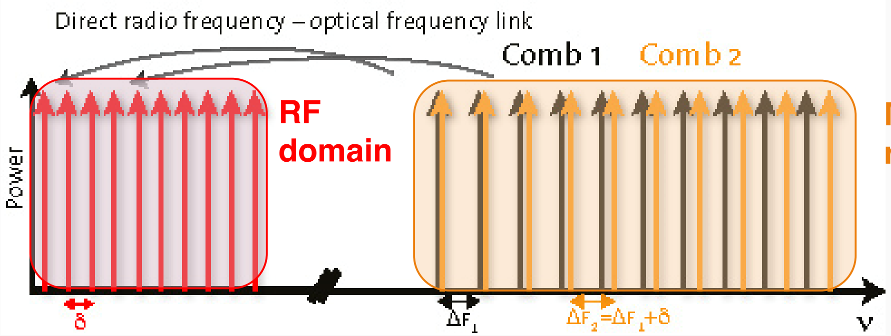
Cappelli et al., Intrinsic linewidth of quantum cascade laser frequency combs, Optica 2, 836-840 (2015)

QCL-comb: the phase-locking of a mode to an IC-DFG-comb mode

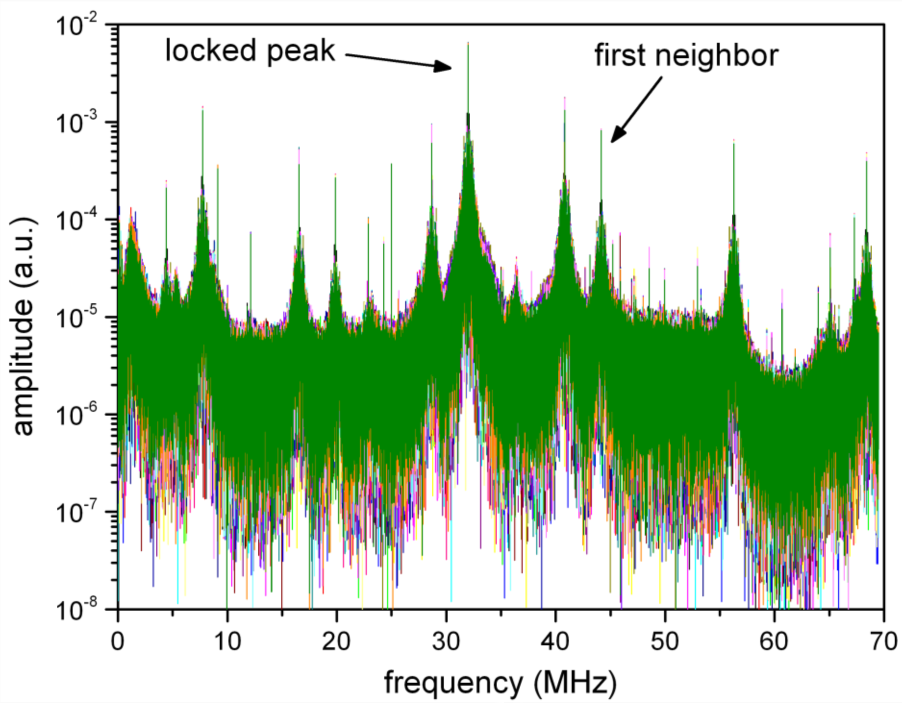


QCL provided by ETH Zürich, Switzerland.

The dual comb technique



Dual comb technique: down-conversion of the IR spectra down to the RF.

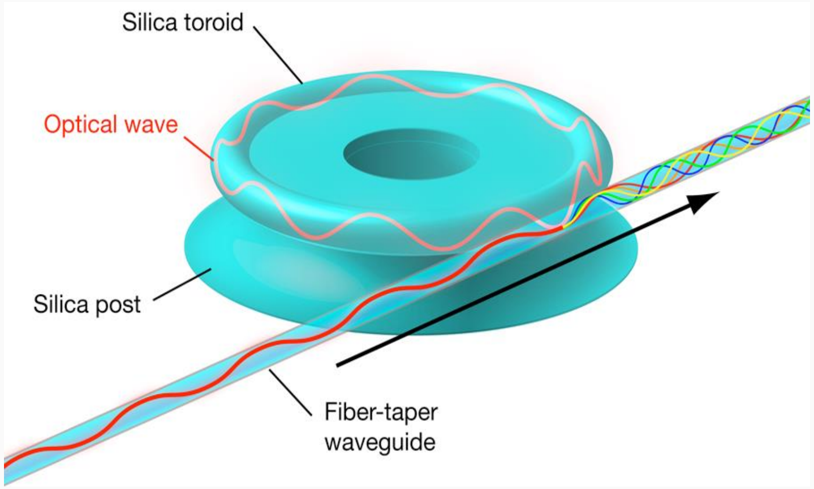


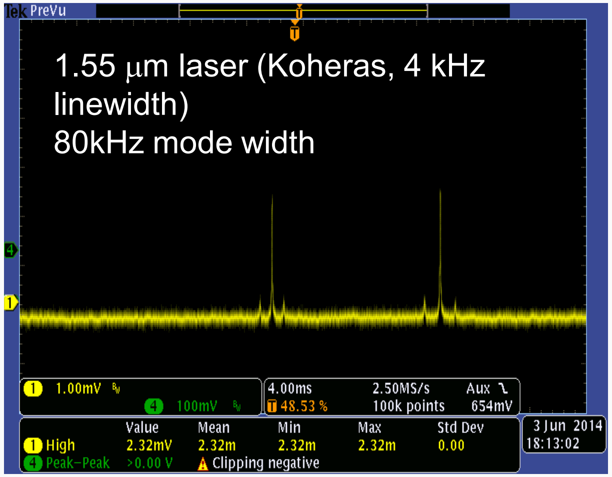
← 20 frames 2-ms long

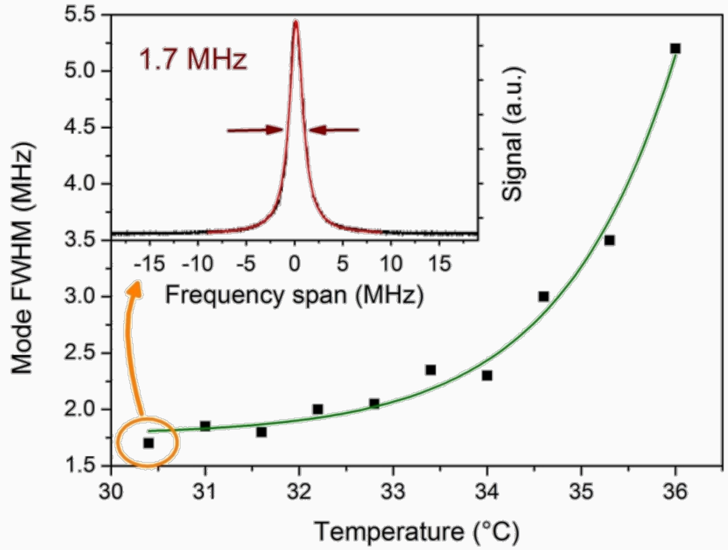
The locking effectively narrows the peaks from 500 kHz to **less than 20 kHz** on a 1-s time scale.

Whispering Gallery Mode micro-Resonators

- coupling through evanescent waves
- compact and robust devices
- transparent from near- to mid-IR (CaF_2, MgF_2)
- suitable for laser stabilization and comb generation

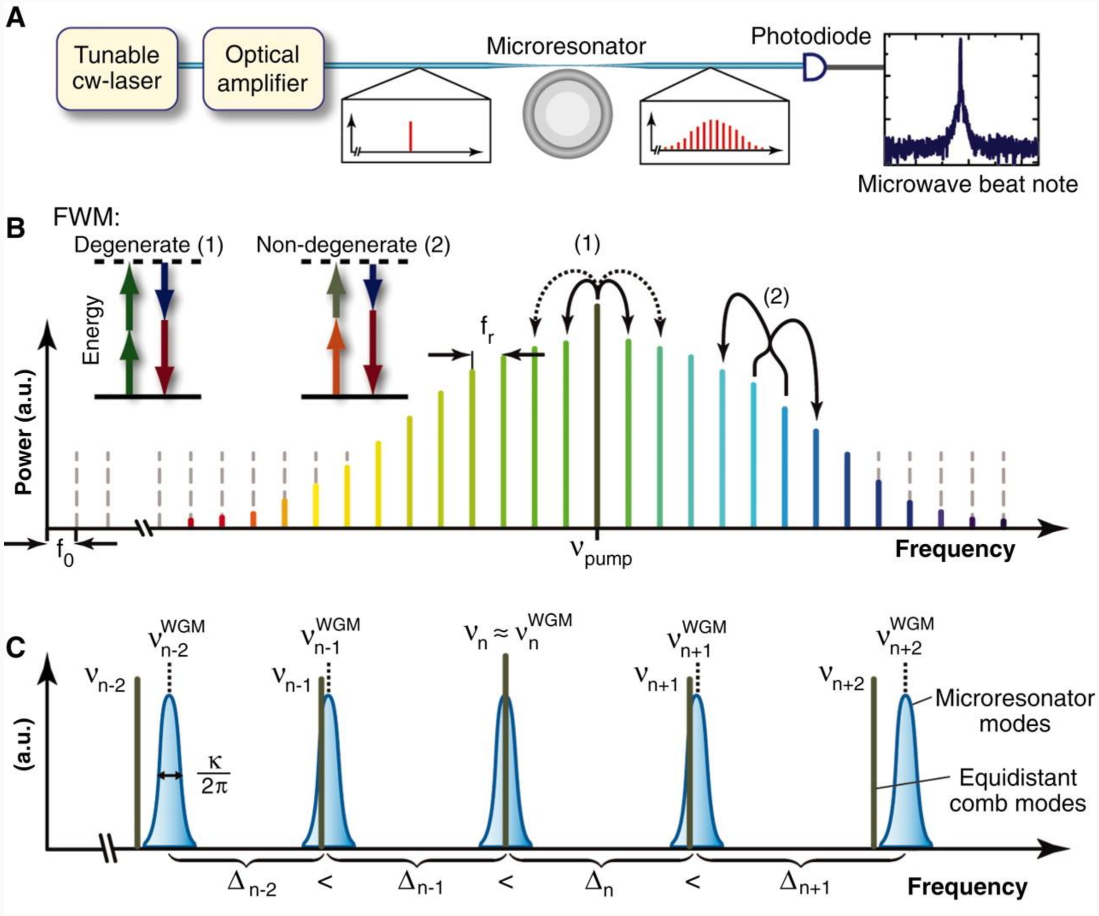


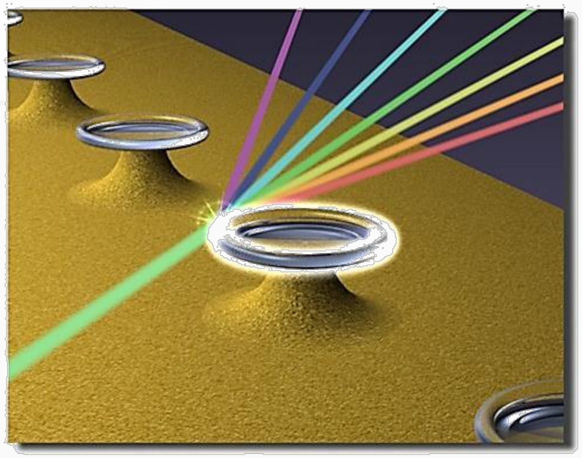




- modes as narrow as <10 kHz in the near-IR ($Q \sim 10^{10}$)
- $\sim$ 1 MHz width in the mid-IR ($Q \sim 10^7 \div 10^8$)

Kerr-comb generation





Kerr-comb generation by four wave mixing

from Kippenberg et al., Science **332**, 555-559 (2011)

high Q
small size

nonlinear optical phenomena can be generated at **very low coupled power levels**

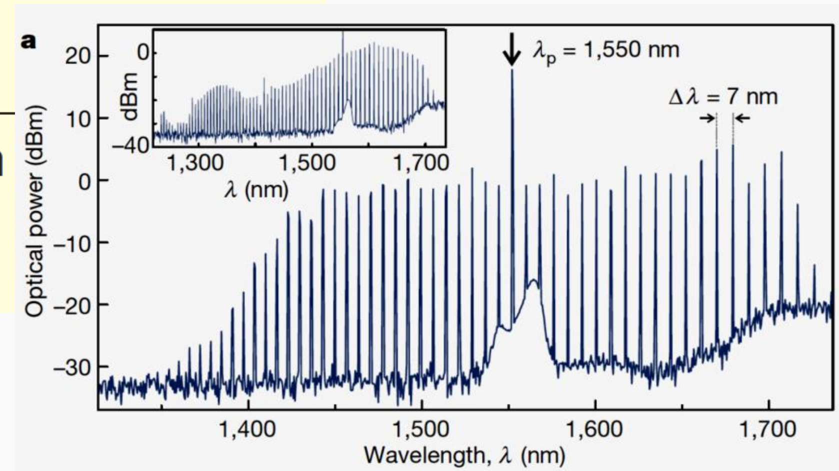
Kerr-comb generation using a silica microresonator

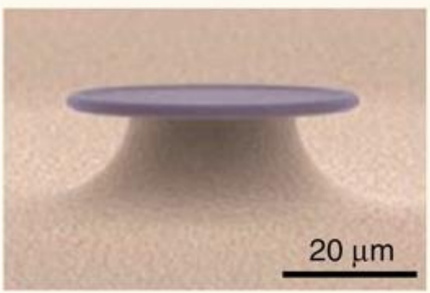

nature Vol 450|20/27 December 2007|doi:10.1038/nature06401

LETTERS

Optical frequency comb generation from a microresonator

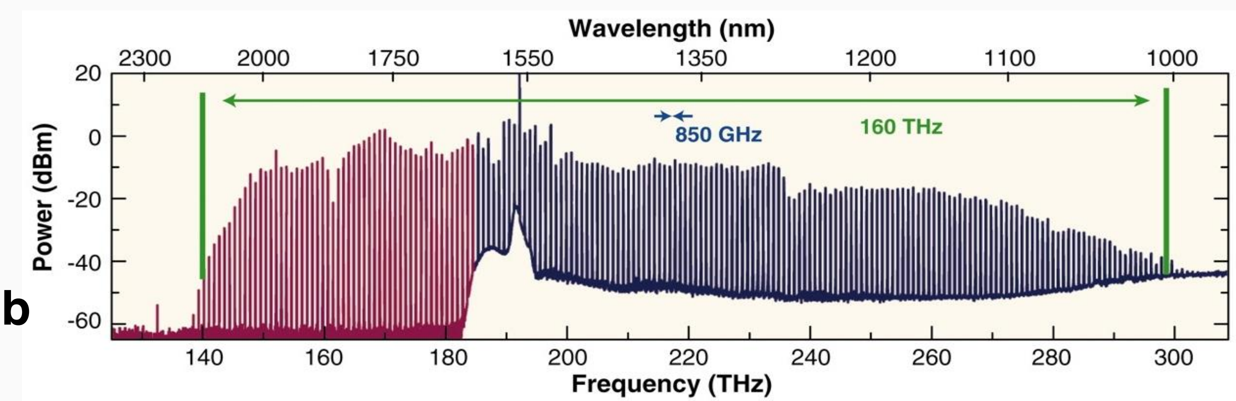
P. Del'Haye[1], A. Schliesser[1], O. Arcizet[1], T. Wilken[1], R. Holzwarth[1] & T. J. Kippenberg[1]





Silica

Octave-spanning comb

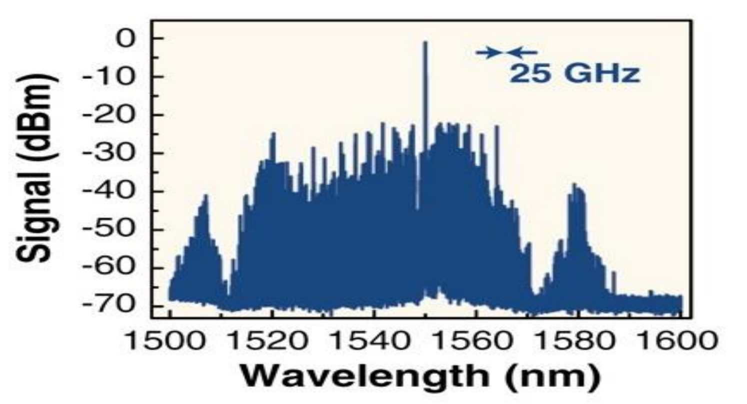


Del'Haye, Herr, Gavartin, Gorodetsky, Holzwarth, and Kippenberg, Phys. Rev. Lett. **107**, 063901 (2011)

Kerr-comb generation

Tunable Comb with a Crystalline CaF2 Whispering Gallery Mode Resonator

Crystalline

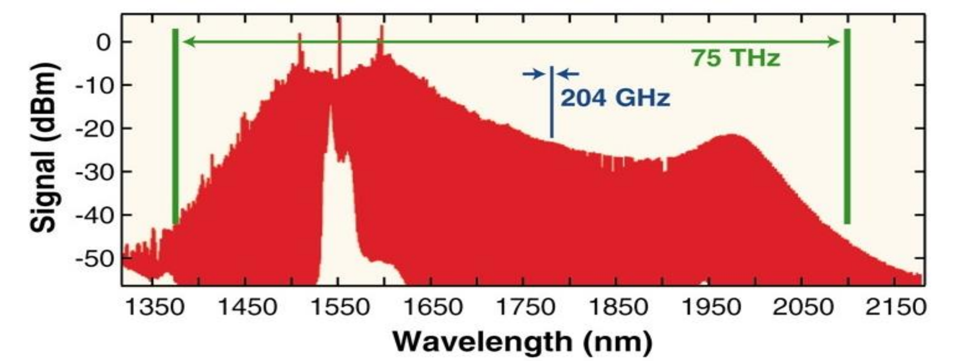


controllable tuning of the comb repetition frequency by changing the frequency of the pump laser

Savchenkov, Matsko, Ilchenko, Solomatine, Seidel, and Maleki, Phys. Rev. Lett. **101**, 093902 (2008)

Comb generated in integrated SiN resonator

Silicon nitride



Foster, Levy, Kuzucu, Saha, Lipson, and Gaeta, Opt. Express **19**, 14233-14239 (2011)

Kerr-comb generation in the mid infrared

nature COMMUNICATIONS

ARTICLE

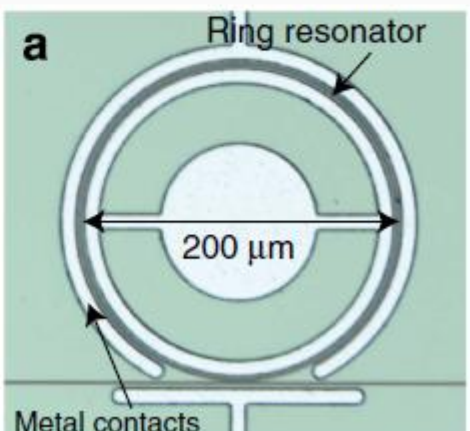
Received 10 Aug 2014 | Accepted 15 Jan 2015 | Published 24 Feb 2015

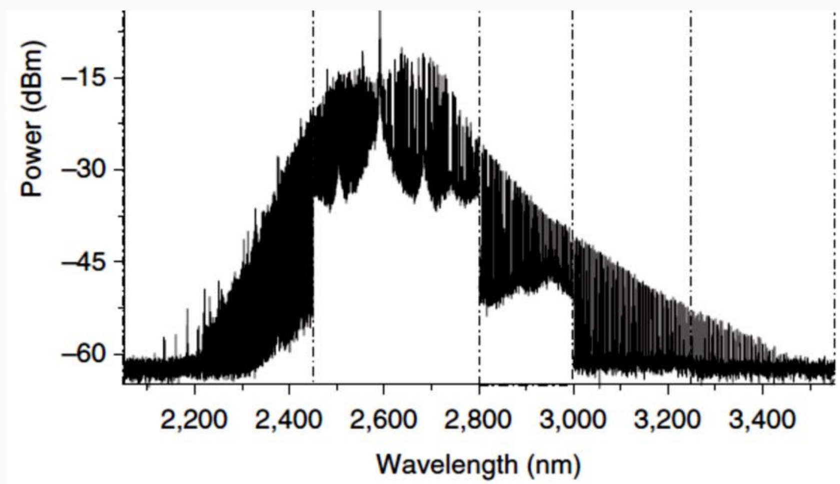
DOI: 10.1038/ncomms7299

Silicon-chip mid-infrared frequency comb generation

Austin G. Griffith[1], Ryan K.W. Lau[2], Jaime Cardenas[1], Yoshitomo Okawachi[2], Aseema Mohanty[1], Romy Fain[1], Yoon Ho Daniel Lee[1], Mengjie Yu[2], Christopher T. Phare[1], Carl B. Poitras[1], Alexander L. Gaeta[2,3] & Michal Lipson[1,3]

- SiN resonator pumped with an OPO (2.5 to 3.2 µm, 100-kHz linewidth)
- broadband frequency comb spanning from 2.1 to 3.5 µm





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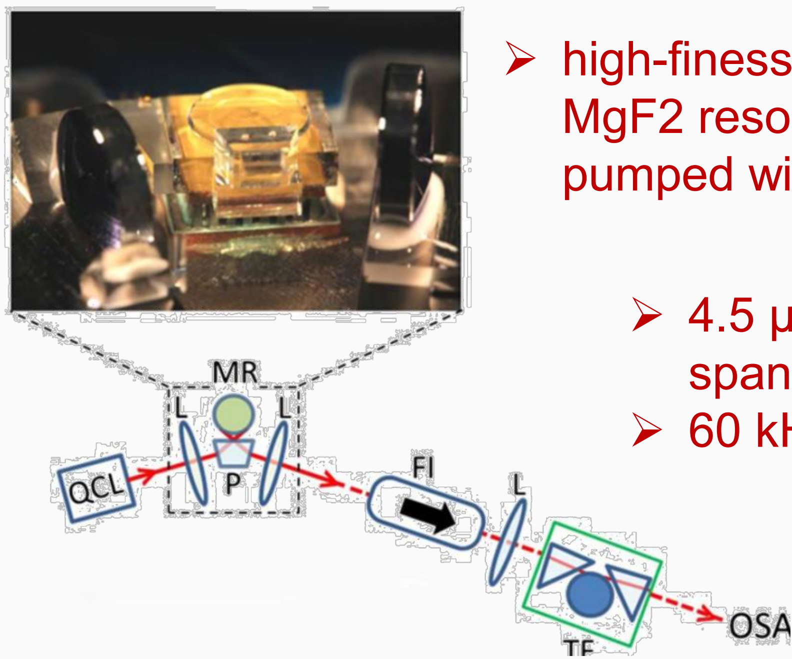
Kerr-comb generation in the mid infrared

3468 Vol. 40, No. 15 / August 1 2015 / *Optics Letters* Letter

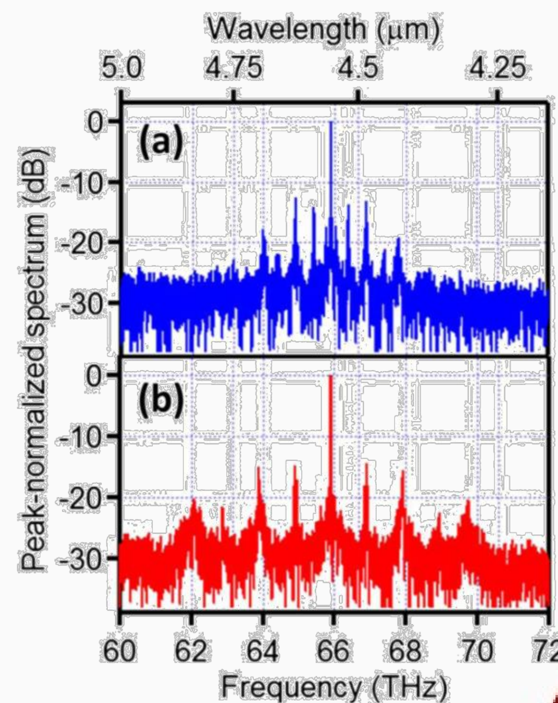
Optics Letters

Generation of Kerr combs centered at 4.5 μm in crystalline microresonators pumped with quantum-cascade lasers

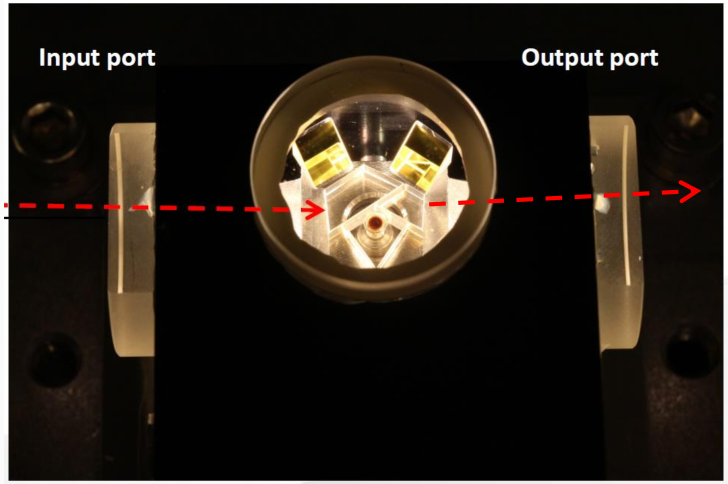
Anatoliy A. Savchenkov,[1] Vladimir S. Ilchenko,[1] Fabio Di Teodoro,[2,3] Paul M. Belden,[2] William T. Lotshaw,[2] Andrey B. Matsko,[1,*] and Lute Maleki[1]



- high-finesse CaF_2 and MgF_2 resonators pumped with QCLs
- 4.5 μm half-octave spanning comb
- 60 kHz linewidth



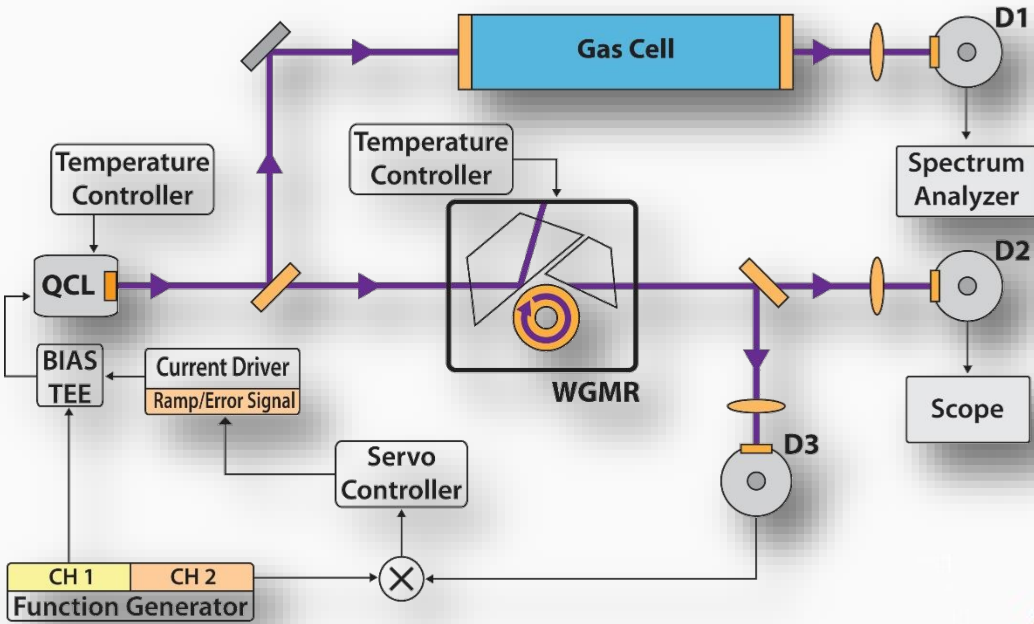
CaF_2 micro-resonator for mid-IR laser stabilization



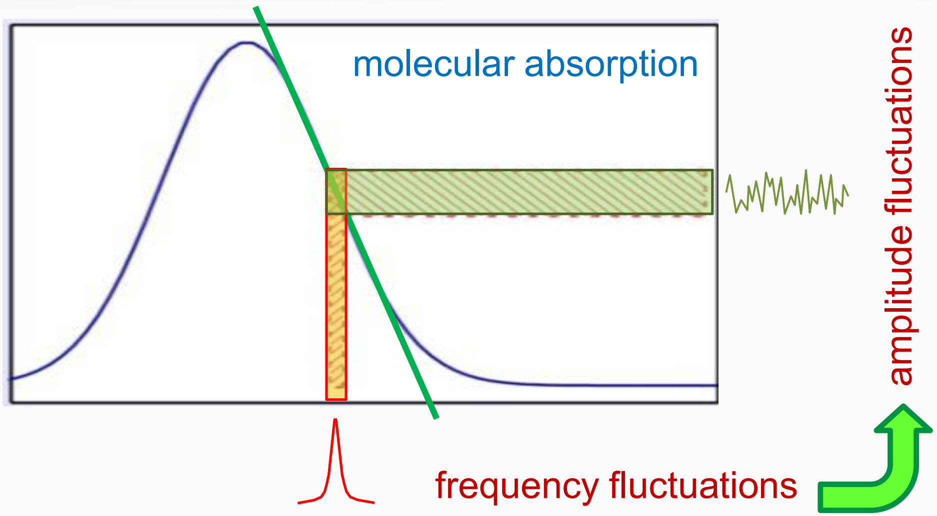
CaF2 toroidal WGMR
3.6 mm diameter --> FSR=18.9 GHz @ 4.3 μm
Coupling: Al2O3 (sapphire) prisms

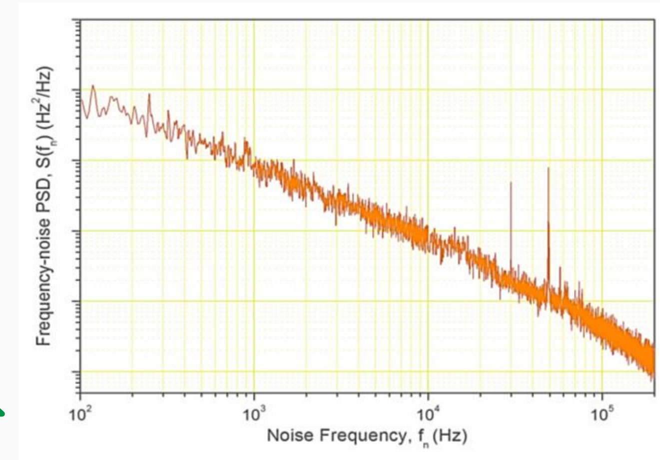
Q-factor ~2 x 10^9 @ 1.5 μm (*F*=250k)
~4 x 10^7 @ 4.3 μm (*F*=11k)

- DFB QCL @ 4.3 μm (Hamamatsu) 700 mA thr., 10 mW
- low-noise current driver
- Reference cell with pure CO2



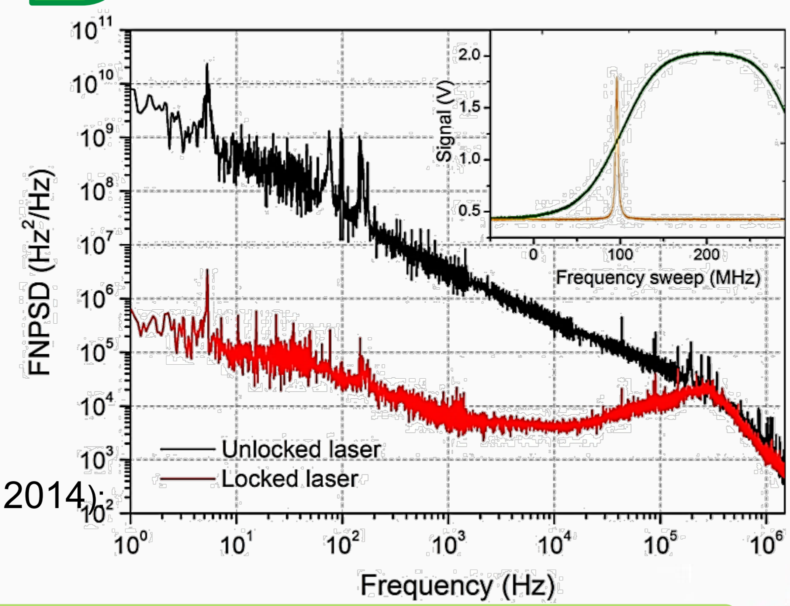
Measuring the laser frequency noise





Free-running QCL:
700 kHz FWHM (1 s timescale)

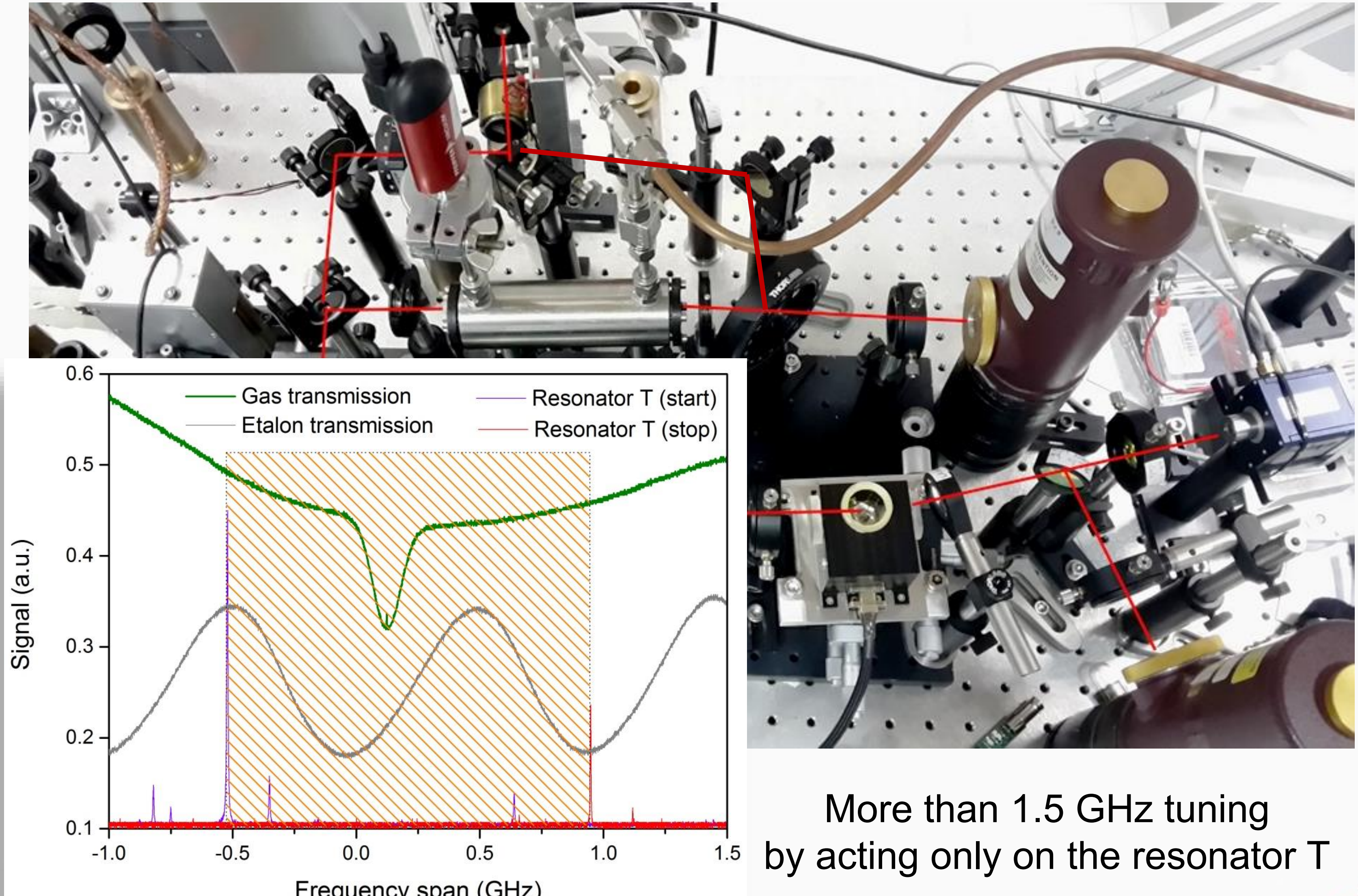
Locked QCL:
15 kHz FWHM (1 s timescale)
10 kHz (1 ms)



Locking to V-shaped cavity (Fasci et al. OL 39, 4946, 2014):
4 kHz (1 ms); 1 MHz (1 s)

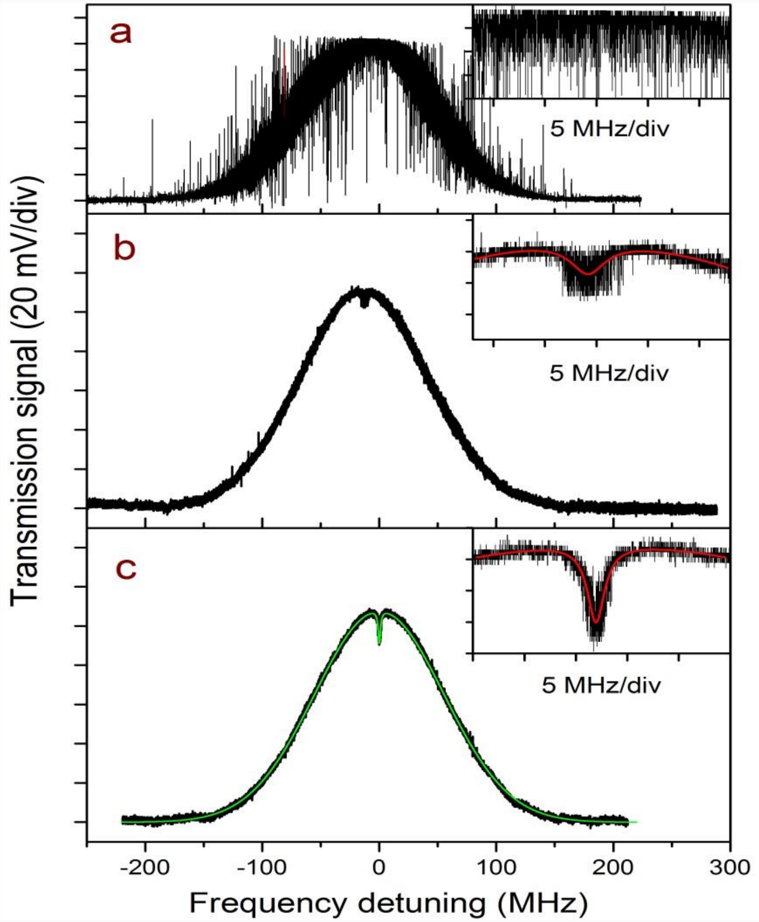
Siciliani et al., Laser and Photonics Reviews **10**, 153–157 (2016)

Doppler-free spectroscopy – electronic lock



More than 1.5 GHz tuning
by acting only on the resonator T

Doppler-free spectroscopy – electronic lock



Commercial vs home-made low-noise driver
free-running vs locked laser

- Pump power: about 5 mW
- 30 seconds span time

Improved precision on center frequency determination by more than a factor 3

WORK IN PROGRESS...

Borri et al., *Tunable microcavity-stabilized quantum cascade laser for mid-IR high-resolution spectroscopy and sensing*, Sensors 2016 (in press)

Physics

SERIES IN OPTICS AND OPTOELECTRONICS

SERIES EDITORS: E ROY PIKE AND ROBERT G W BROWN

Laser-Based Measurements for Time and Frequency Domain Applications

A Handbook

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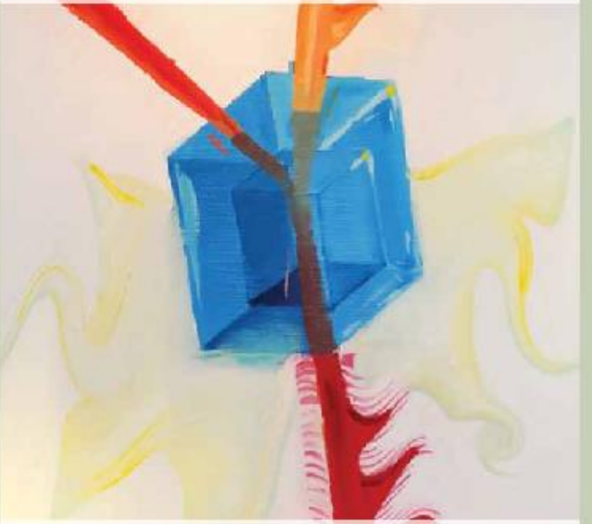
A Handbook



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