Mid-infrared laser phase-locking to a remote near-infrared frequency reference for high-precision molecular spectroscopy

B Chanteau$^1$, O Lopez$^1$, W Zhang$^2$, D Nicolodi$^2$, B Argence$^1$, F Auguste$^1$, M Abgrall$^2$, C Chardonnet$^1$, G Santarelli$^{2,3}$, B Darquié$^1$, Y Le Coq$^2$ and A Amy-Klein$^{1,4}$

$^1$Laboratoire de Physique des Lasers, Université Paris 13, Sorbonne Paris Cité, CNRS, 99 Avenue Jean-Baptiste Clément, F-93430 Villetaneuse, France
$^2$LNE-SYRTE, Observatoire de Paris, CNRS, UPMC, 61 Avenue de l’Observatoire, F-75014 Paris, France
$^3$Laboratoire Photonique, Numérique et Nanosciences, Université de Bordeaux 1, Institut d’Optique and CNRS, 351 cours de la Libération, F-33405 Talence, France

E-mail: amy@univ-paris13.fr

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Abstract. We present a method for accurate mid-infrared frequency measurements and stabilization to a near-infrared ultra-stable frequency reference, transmitted with a long-distance fibre link and continuously monitored against state-of-the-art atomic fountain clocks. As a first application, we measure the frequency of an OsO$_4$ rovibrational molecular line around 10 $\mu$m with an uncertainty of 8 $\times$ 10$^{-13}$. We also demonstrate the frequency stabilization of a mid-infrared laser with fractional stability better than 4 $\times$ 10$^{-14}$ at 1 s averaging time and a linewidth below 17 Hz. This new stabilization scheme gives us the ability to transfer frequency stability in the range of 10$^{-15}$ or even better, currently accessible in the near infrared or in the visible, to mid-infrared lasers in a wide frequency range.

$^4$Author to whom any correspondence should be addressed.

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1. Introduction

With their rich internal structure, molecules can play a decisive role in precision tests of fundamental physics. They are, for example, now being used to test fundamental symmetries [1–3] and to measure either absolute values of fundamental constants [4] or their temporal variation [5–6]. Most of those experiments can be cast as the measurement of molecular frequencies. Ultra-stable and accurate sources in the mid-infrared (MIR) spectral region, the so-called molecular fingerprint region that hosts many intense rovibrational signatures, are thus highly desirable. MIR laser frequency stabilization has been performed for a long time using molecular references such as CH$_4$ or OsO$_4$ (see for instance [7–10]). However obtained stability is at least one order of magnitude below those of visible or near-infrared lasers stabilized to an ultra-stable cavity. Moreover, only a few molecular lines can be used when ultra-high accuracy is needed.

In this paper we present a method for accurate MIR laser frequency stabilization. The frequency reference is a near-infrared cavity-stabilized laser continuously monitored against primary standards, and the coherent frequency link between near-infrared and MIR frequencies is obtained by using an optical frequency comb. Moreover, we demonstrate this stabilization scheme with a remote near-infrared frequency reference transferred via an optical fibre link from a national metrological institute (NMI). This technique is thus accessible to any laboratory that can be connected to such an NMI with a fibre optical link [11].

Optical frequency combs have proven to be essential for laser frequency measurement and stabilization from the infrared to ultraviolet domain (see for instance [12]). Fractional accuracy and stability (at 1 day averaging time) down to a few $10^{-16}$ are potentially reachable when the frequency reference is provided by advanced primary standards. Extension to the MIR spectral domain has been demonstrated by comparing the MIR laser frequency with a very high harmonic of the comb repetition rate using sum-frequency generation (SFG) or difference-frequency generation [13–20]. Efforts have also been made towards the development of MIR frequency combs [10, 21–26].

In this paper we first describe the setup for coherent frequency stability transfer between near-infrared and MIR frequencies around 10 $\mu$m. Then we demonstrate absolute frequency measurement of a MIR frequency with a fractional resolution of at least $4 \times 10^{-14}$. We also report a first application to high-resolution molecular spectroscopy with a fractional uncertainty of $8 \times 10^{-13}$ on the line centre. Finally, we present the MIR laser frequency stabilization against the near-infrared frequency reference.
2. Experimental setup

The experimental setup is shown in figure 1. The ultra-stable optical reference located at LNE-SYRTE is a 1.54 µm fibre laser locked to a high-finesse cavity. Its fractional frequency instability was measured to be lower than $2 \times 10^{-15}$ at 1 s and $10^{-14}$ at 100 s (after a 0.3 Hz s$^{-1}$ drift was removed) [27]. Its frequency is measured using a fibre fs laser centred around 1.55 µm. The laser repetition rate is phase-locked to the optical reference frequency after removal of the comb frequency offset $f_0$. Fast corrections are applied to an intra-cavity electro-optic modulator (bandwidth $>$400 kHz) and slower corrections to a piezo-electric transducer (PZT) controlling the laser cavity length (bandwidth $\sim$10 kHz) [28]. The absolute frequency of the comb repetition rate 36th harmonic (9 GHz) is continuously measured against the LNE-SYRTE frequency references, which includes an H-maser, a cryogenic oscillator and Cs fountains [29–30]. It enables real-time measurement of the ultra-stable laser frequency drift and its correction by applying to the driving frequency of an acousto-optic modulator an opposite linear drift (with a step every ms) updated every 100 s. This makes up an ultra-stable near-infrared reference, the frequency of which is currently traceable to primary standards with a $10^{-14}$ uncertainty after 100 s.

This optical reference signal is transmitted to Laboratoire de Physique des Lasers (LPL) through a 43 km long optical link [27]. The free-running link exhibits a propagation instability of $2 \times 10^{-14}$ at 1 s and $10^{-15}$ between 100 s and 1 day. When compensated, the link instability has been measured to be roughly $10^{-15}$ s$^{-1}$ and to reach around $10^{-18}$ after 10$^3$ s (see figure 3) [27]. The frequency stability and accuracy of the reference signal are thus preserved at the LPL optical link end.

At LPL, a low-noise laser diode (free-running linewidth below 10 kHz) is phase-locked to the incoming signal with a bandwidth of 100 kHz and constitutes the local optical frequency

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**Figure 1.** Experimental setup. The MIR laser frequency can be controlled with either the beat-note $\Delta_2$ or the OsO$_4$ absorption signal. PLL: phase-lock loop; PDH: Pound–Drever–Hall stabilization; SFG: sum-frequency generation; AOM: acousto-optic modulator; USC: ultra-stable cavity.
Figure 2. Sum-frequency of a comb output centred at 1850 nm (purple comb), of mode frequencies \( q \, f_{\text{rep}} + f_0 \) with \( q \) an integer, and the MIR laser (of frequency \( \nu_{\text{MIR}} \) around 10 \( \mu \)m) results in a shifted comb (brown comb) centred at 1550 nm of mode frequencies \( q \, f_{\text{rep}} + f_0 + \nu_{\text{MIR}} \). The beat-note of this shifted comb with the comb main output centred at 1550 nm (red comb), of mode frequencies \( p \, f_{\text{rep}} + f_0 \) with \( p \) an integer, can be written as

\[
\Delta \equiv \pm (q \, f_{\text{rep}} + f_0 + \nu_{\text{MIR}}) - (p \, f_{\text{rep}} + f_0),
\]

which results in \( \Delta = \pm (\nu_{\text{MIR}} - (p - q) \, f_{\text{rep}}) \). SFG: sum-frequency generation.

reference \( \nu_{\text{ref}} \). The repetition rate \( f_{\text{rep}} \) of a 1.55 \( \mu \)m fibre fs laser is phase-locked to \( \nu_{\text{ref}} \). To that purpose, the beat-note \( \Delta_1 \) between \( \nu_{\text{ref}} \) and the \( N \)th comb mode (\( N \sim 780 000 \)) is used, after removal of the comb frequency offset \( f_0 \):

\[
\nu_{\text{ref}} - N \, f_{\text{rep}} = \pm \Delta_1. \tag{1}
\]

Fast and slow corrections are applied to an intra-cavity electro-optic modulator and a PZT, respectively, as performed at LNE-SYRTE [28]. A second beat-note \( \Delta_2 \) compares the MIR laser frequency \( \nu_{\text{MIR}} \) around 10 \( \mu \)m and the \( n \)th harmonic of the repetition rate with \( n \approx 120 000 \):

\[
\nu_{\text{MIR}} - n \, f_{\text{rep}} = \pm \Delta_2. \tag{2}
\]

This signal is generated using SFG of the MIR light and an additional comb output centred on 1.85 \( \mu \)m, generated in a nonlinear fibre (figure 2) [14]. This comb output (\( \sim 25 \) mW) and the MIR laser beam (\( \sim 100 \) mW) are focused in a 10 mm long crystal of AgGaSe$_2$ for type-I SFG. The measured efficiency is around 0.4 mW/W$^2$ and the phase-matching bandwidth (for the 1.85 \( \mu \)m comb) is about 30 nm (\( \sim 3 \) THz). The resulting shifted comb, centred on 1.55 \( \mu \)m, is combined with the 1.55 \( \mu \)m fs laser output. An adjustable delay line enables us to control the overlapping of the pulses in the time domain. About \( 10^4 \) mode pairs generate the beat-note \( \Delta_2 \) which shows a signal-to-noise ratio of about 30 dB in a 100 kHz bandwidth. An RF tracking oscillator is phase-locked to this beat-note. As a result of the frequency difference between two modes of the same comb, \( \Delta_2 \) is independent of the comb offset \( f_0 \).

Combining (1) and (2), the MIR laser frequency \( \nu_{\text{MIR}} \) is finally obtained as

\[
\nu_{\text{MIR}} = \pm \Delta_2 + \frac{n}{N} (\nu_{\text{ref}} \mp \Delta_1) \tag{3}
\]

with \( n/N \) roughly equal to 0.15. The MIR frequency is thus directly linked to the near-infrared frequency reference, once the integers \( n \) and \( N \) and the signs have been determined.
3. Mid-infrared frequency measurement and stabilization

To characterize the phase-coherent link between the near-infrared frequency reference and the MIR frequency, we used this setup to measure the absolute frequency of a CO$_2$ laser stabilized onto an OsO$_4$-saturated absorption line. Such an OsO$_4$-stabilized CO$_2$ laser constitutes the current state-of-the-art MIR secondary reference standard [7, 8]. In this work, the CO$_2$ laser was locked either to the P(55) line of $^{190}$OsO$_4$ near the 10.55 $\mu$m P(16) CO$_2$ laser line (red circles ) or to the R(67) line of $^{192}$OsO$_4$ near the 10.25 $\mu$m R(20) CO$_2$ laser line (blue squares ). The propagation instability of the compensated LPL-SYRTE fibre link (green up-triangles ▲) and the stability of the SYRTE frequency reference that is made of a combination of an H-maser, a cryogenic oscillator and Cs fountains [29] (dashed line) are shown for comparison.

The relationship of equation (3) between the frequency of the 1.54 $\mu$m LNE-SYRTE reference laser and that of the MIR laser is ensured by means of coherent phase-lock loops.
Thus the accuracy of the MIR frequency measurement only depends on the uncertainty of 
the near-infrared frequency reference. The latter is known with an uncertainty of about $10^{-14}$ 
after 100 s averaging time, when only steered with the H-maser which is sufficient for this 
experiment. The $3 \times 10^{-16}$ Cs fountain accuracy [30] can ultimately be reached and then 
transferred from the optical reference to the MIR frequency.

As a first application to high-precision spectroscopy, we determined the absolute frequency 
of the P(55) line of $^{190}$OsO$_4$ by measuring the OsO$_4$-stabilized CO$_2$ laser frequency. Eleven 
measurements were performed between December 2011 and April 2012. The beat-note 
$\Delta_2$, the repetition rate $f_{\text{rep}}$ and the frequency $\nu_{\text{ref}}$ were counted with a gate time of 1 s. 
$\Delta_2$ and $\nu_{\text{ref}}$ were combined using equation (3) to calculate the CO$_2$/OsO$_4$ frequency. Signs 
in equation (3) and values of $n$ and $N$ were unambiguously deduced from $f_{\text{rep}}$ and the value $\nu_{\text{OsO}_4/1999} = 28\,412\,648\,819\,596$ (45) Hz of the CO$_2$/OsO$_4$ frequency obtained by combining 
two independent measurements reported in the literature [31, 32]. The mean value over 600 
measurements of 1 s of the CO$_2$/OsO$_4$ frequency gives one data point. For each data point, 
we correct the frequency of the LNE-SYRTE H-maser using the data published by Bureau 
International des Poids et Mesures\(^5\). Between each measurement the OsO$_4$ absorption cell 
was pumped and filled again or the whole experiment was switched off and on. We obtained $\nu_{\text{OsO}_4/2012} = 28\,412\,648\,819\,588$ (24) Hz where the uncertainty is the weighted $1 - \sigma$ deviation 
of the data points. It is $-8$ Hz from the value $\nu_{\text{OsO}_4/1999}$ and $+8$ Hz from another measurement 
performed in 2004 with a microwave-referenced Ti:Sa frequency comb with an uncertainty of 
58 Hz [13]. Within $1 - \sigma$ error bars the present result agrees with the previous measurements 
and confirms the very high accuracy of the measurement setup. The factor of 2 improvement 
of the uncertainty obtained in the measurement reported here, still limited by the molecular 
reference, is due to a better control of the OsO$_4$ pressure and optimization of the CO$_2$ laser 
locking parameters.

From the previous results, we conclude that the coherent frequency chain is a viable and 
potentially much better alternative to an OsO$_4$ molecular transition for frequency stabilization 
of a MIR source. This was investigated by locking the CO$_2$ laser frequency to the optical comb: 
the beat-note of the free-running CO$_2$ laser with the comb repetition rate $n$th harmonic, $\Delta_2$, 
was phase-locked onto a stable frequency synthesizer with a 400 Hz bandwidth. The obtained 
CO$_2$ laser frequency stability is characterized by measuring the beat-note $\Delta_3$ with a second 
independent CO$_2$ laser stabilized onto OsO$_4$. Figure 4 displays this beat-note signal, fitted with 
a Lorentzian of linewidth 17 Hz (full-width at half-maximum). In the case of a Lorentzian 
lineshape, the contribution of each laser linewidth adds [33] and we deduce a linewidth between 
8.5 and 17 Hz for each laser, the state of the art for a CO$_2$ laser [7]. Figure 5 displays $\Delta_3$’s 
frequency noise power spectral density (PSD) (red trace). Using the beat-note $\Delta_2$ with the 
local frequency comb, the frequency noise PSD of the OsO$_4$-stabilized CO$_2$ laser was also 
measured (figure 5, blue trace). The two PSDs almost perfectly overlap as expected from 
efficient phase stabilization. Together with the above results, it shows that the comb-stabilized 
MIR laser frequency noise is at least as low as that of the OsO$_4$-stabilized laser. The former 
is most probably much lower, potentially compatible with the frequency noise of the optical 
frequency reference of which the inferred PSD (including noise added by the link) is displayed 
in figure 5 (dotted black line). This noise level is the lowest reachable with our stabilization 
scheme.

\(^5\) See publication of the clocks’ rates relative to TAI on ftp://ftp2.bipm.org/pub/tai/publication/.

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Figure 4. Down-converted beat-note of two independent CO₂ lasers, one stabilized on an OsO₄ resonance line, the other phase-locked to a comb repetition rate high-harmonic; the red line is a Lorentzian fit of linewidth 17 Hz.

Figure 5. Frequency noise PSD of (a) the free-running CO₂ laser (green trace), (b) the beat-note between the CO₂ laser stabilized onto the frequency comb and an independent OsO₄-stabilized CO₂ laser (red trace), (c) the OsO₄-stabilized CO₂ laser measured with the comb (blue trace) and (d) the optical reference (dotted black line). The free-running CO₂ laser PSD has been measured using the beat-note $\Delta_2$ with the local frequency comb and is given for comparison.

4. Conclusion

We have demonstrated a coherent frequency chain linking a remote ultra-stable 1.54 $\mu$m frequency reference and a MIR source, leading to the control of the absolute MIR frequency.
It uses reliable commercially available fibre-based frequency combs and an optical reference potentially available to any laboratory connected to a fibre network [11]. Stability below $4 \times 10^{-14}$ at 1 s was demonstrated, and we expect it to be in the $10^{-15}$ range. Using a state-of-the-art near-infrared ultra-stable laser [34] may reduce this value even further. The $3 \times 10^{-16}$ accuracy of the LNE-SYRTE Cs fountains is potentially within reach.

Frequency tuning of such a stabilized MIR laser source, required for high-resolution spectroscopy, is achievable by scanning the near-infrared frequency referencing the comb. Tuning the frequency offset between the LNE-SYRTE optical reference and the LPL laser diode of frequency $\nu_{\text{ref}}$ (see equation (3)) would result in a tuning range of a few GHz.

This setup enables us to stabilize MIR laser sources in a much wider spectral range than is currently possible using the OsO$_4$ molecular standard. With the present setup the 9–11 $\mu$m range is accessible, limited by the nonlinear crystal and the central frequency of the auxiliary 1.85 $\mu$m comb output used in the SFG. Nevertheless, it can easily be extended to the whole 5–20 $\mu$m range with the proper comb spectrum and crystal optimization. Orientation-patterned GaAs would for instance ensure a wide tunability [35]. Our stabilization technique is thus particularly well suited to quantum cascade lasers that have achievable wavelengths covering the whole MIR region [36]. Moreover, the ongoing work on dissemination of optical reference through internet fibre networks over a continental scale [11] will eventually enable many laboratories to access an ultra-stable optical reference. Thus such ultra-stable and accurate MIR sources could benefit a very wide molecular spectroscopy community.

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