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# Absolute frequency measurement of acetylene transitions in the region of 1540 nm

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Received: 9 December 2003/

Revised version: 28 February 2004

Published online: 28 April 2004 • © Springer-Verlag 2004

**ABSTRACT** An optical frequency comb has been used to measure the frequency of a diode laser system, locked to the P(10), P(11), P(15), P(16), P(20), and P(21) transitions in the  $\nu_1 + \nu_3$  overtone band of  $^{13}\text{C}_2\text{H}_2$ . When locked to any of these transitions, the laser frequency showed a stability of  $4 \times 10^{-12}$  in 1 s and a reproducibility of better than 1 kHz. The frequency of the reference P(16) transition was found to be  $P(16) = 194\,369\,569\,383.83$  kHz with an uncertainty of 0.32 kHz, based on measurements of a single system, and 2.5 kHz, based on the reproducibility of independent systems.

PACS 06.30.Ft; 33.20.Ea; 42.62.Fi

## 1 Introduction

Accurate and practical frequency standards in the region of 1500 nm are important for basic atomic physics, dimensional metrology, and, in particular, the calibration of channel frequencies in wavelength-division-multiplexed (WDM) systems. Although uncertainties in the WDM carrier frequencies as large as 100 MHz can be tolerated for channel spacings of 50 GHz, laboratory standards must have much lower uncertainty. Each stage in a calibration link between the devices in the field and the laboratory standard typically requires a reduction in the uncertainty of one or two orders of magnitude. Therefore, high quality standards, with uncertainties below  $10^{-9}$ , have been developed in recent years in the region of 1540 nm or 195 THz. Probably the most widely studied are standards based on the ro-vibrational transitions in acetylene, since the frequencies of these transitions form a regular grid spanning much of the optical telecommunication C-band [1–18].

Although the fundamental C–H vibrational transitions for acetylene are in the region of 3–3.5  $\mu\text{m}$ , weak anharmonic overtone transitions, in which the vibrational energy increases by two quanta, can be driven by radiation in the region of 1500–1550 nm [19]. Since  $^{12}\text{C}_2\text{H}_2$  and  $^{13}\text{C}_2\text{H}_2$  are symmetric molecules with no permanent dipole moment,

standards based on these molecules are expected to be relatively immune to Stark and pressure shifts [11]. For  $^{12}\text{C}_2\text{H}_2$ , the  $\nu_1 + \nu_3$  overtone band of rovibrational transitions consists of a series of over 40 lines, spaced by 60–80 GHz, extending from approximately 1515 nm to 1540 nm, while for  $^{13}\text{C}_2\text{H}_2$  the same band is shifted to longer wavelengths by approximately 8 nm [2, 14]. A number of laser systems have been developed that are frequency-locked to either the Doppler-broadened or the Doppler-free saturated absorption overtone transitions in either  $^{12}\text{C}_2\text{H}_2$  or  $^{13}\text{C}_2\text{H}_2$ . Since the Doppler-broadened linewidth at room temperature is approximately 500 MHz, standards locked to these features have shown relatively poor reproducibility and an instability in the range of  $10^{-10}$  to  $10^{-9}$  in 1 s [2, 4, 8]. Saturated absorption by these weak transitions [10] was first seen by Labacheleterie et al. [3] through the use of a Fabry–Pérot enhancement cavity. The saturated absorption feature has a linewidth below 1 MHz, mainly limited by pressure broadening and transit-time effects. Laser systems locked to this feature have shown excellent performance with a reproducibility of 1 to 10 kHz ( $< 5 \times 10^{-11}$ ) [5, 6, 14, 15], an instability of  $10^{-12}$  for an averaging time of 1 s and a instability floor of less than  $4 \times 10^{-13}$  after several hundred seconds of averaging [16–18].

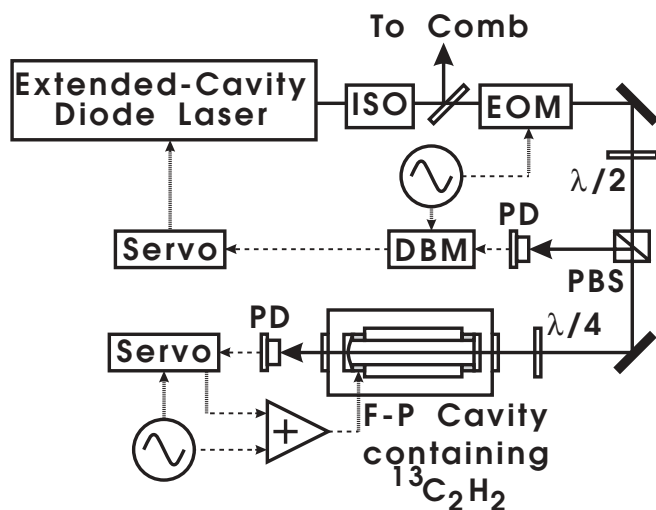
Interest in the acetylene transitions for WDM applications led to absolute frequency measurements. Latrasse et al. [4] used a calibrated Fourier-transform spectrometer to measure the frequencies of many transition in  $\text{C}_2\text{HD}$  and  $^{13}\text{C}_2\text{H}_2$  with an uncertainty of 50 MHz. More accurate measurements were soon carried out at the National Metrology Institute of Japan (NMIJ) where either an electro-optic modulator (EOM) based comb generator or a two-color fiber laser was used to bridge the frequency gap between a rubidium-based optical frequency standard at 778 nm and a laser locked to the acetylene transitions [7, 12, 14]. These measurements have resulted in a knowledge of the absolute frequencies of over ninety transitions in  $^{12}\text{C}_2\text{H}_2$  and  $^{13}\text{C}_2\text{H}_2$  to an uncertainty of 150 kHz. The absolute frequency of the P(16)  $\nu_1 + \nu_3$  transition in  $^{13}\text{C}_2\text{H}_2$  was measured by the same workers to an accuracy of 12 kHz [14]. Based on these measurements, the Comité International des Poids et Mesures (CIPM) in 2001 adopted a value of 194 369 569.4 MHz with an uncertainty of 0.1 MHz or  $5.2 \times 10^{-10}$  for the P(16) ( $\nu_1 + \nu_3$ ) transition in  $^{13}\text{C}_2\text{H}_2$  [20].

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The revolution in optical frequency measurements that has occurred as a result of the introduction of frequency combs based on femtosecond mode-locked lasers [21, 22], has made it possible to measure the frequency of almost any optical source with an accuracy limited only by the local frequency/time standard. Recently such combs have been used at the National Institute of Advanced Industrial Science and Technology in Japan (AIST) [16] and the National Physical Laboratory in the UK (NPL) [17] to measure the frequency of the P(16) line in  $^{13}\text{C}_2\text{H}_2$  to uncertainties of 0.076 kHz and 1.3 kHz, respectively. We have recently developed a diode laser system which is locked to saturated transitions in acetylene [15, 18]. Here, we report optical comb measurements, with uncertainties of less than 1 kHz, of the frequencies of six transitions in  $^{13}\text{C}_2\text{H}_2$  with wavelengths ranging from 1538.8 nm to 1545.5 nm.

## 2 Acetylene-stabilized laser system

The acetylene-stabilized laser system is shown in Fig. 1. It has been described in detail in [18]. The output from an extended-cavity diode laser (ECDL) (New Focus model 6328H) was phase modulated at a frequency of 10 MHz and locked to a resonance in a Fabry–Pérot (F–P) cavity through the Pound–Drever–Hall technique [23]. When locked to the F–P cavity, the laser linewidth was reduced to approximately 70 kHz (50 ms observation time) from a free-running linewidth of 500 kHz. The F–P cavity consisted of two mirrors of equal reflectivity, one flat and one with a radius of 1 m, mounted on piezo tubes at the ends of an invar spacer. The length of the cavity was 30 cm and the finesse was 360. It was placed in a vacuum tank which was filled with research-grade, isotopically pure  $^{13}\text{C}_2\text{H}_2$  at a pressure of  $20 \pm 1$  mTorr ( $2.67 \pm 0.13$  Pa). For locking to the saturated absorption feature, a 1.137 kHz modulation was applied to one of the cavity piezos and the transmitted signal was demodulated at the third harmonic ( $3f$ ) of the modulation frequency with a commercial lock-in amplifier. The resulting signed error signal



**FIGURE 1** Schematic diagram of the acetylene-stabilized laser system. ISO – Faraday isolator; EOM – electro-optic modulator;  $\lambda/2$ ,  $\lambda/4$  – waveplates; PBS – polarizing beamsplitter; DBM – double-balanced mixer; PD – photodiode

was sent to a homemade integrating servo system (open-loop unity gain at 100 Hz) that controlled the same cavity piezo. The saturated absorption feature had a depth of 7% of the Doppler-broadened line and a FWHM of approximately 800 kHz, primarily due to transit-time and pressure broadening. A signal-to-noise ratio (S/N) of approximately 40 in a 25-Hz bandwidth was obtained for the  $3f$  demodulated signal. In practice, the laser system could remain locked to the acetylene transition for periods of several hours, limited by thermal drifts in the F–P cavity.

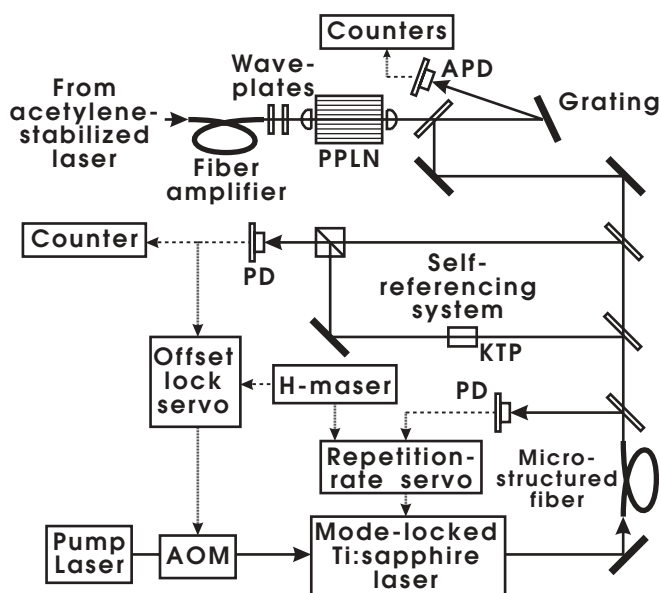
A second laser system was constructed and a study made of the system stability and sensitivities to various parameters [18]. For these measurements, the standard operating conditions of both systems were set as follows: intracavity one-way power = 120 mW ( $35 \text{ W/cm}^2$  one-way intracavity intensity near the input mirror); acetylene pressure = 20 mTorr (2.67 Pa); and modulation amplitude = 1.0 MHz, peak-to-peak. All studies were carried out with the lasers locked to the P(16) line in  $^{13}\text{C}_2\text{H}_2$ .

Heterodyne beat measurements between the two systems showed that each laser had a 1 s-instability of  $7 \times 10^{-12}$  and an instability floor of  $4 \times 10^{-13}$  at 100 s. The relative frequency of the two lasers varied by  $\pm 5$  kHz corresponding to a frequency reproducibility of  $\pm 2.5$  kHz for each system. The instability floor and reproducibility of the particular system used in the work reported here were likely better than these values. A shift in the Pound–Drever–Hall lockpoint, due to polarization changes in a coupling fiber, which was used only in the second system, was seen and is believed to be the primary cause of the observed relative drifts and offsets between the two systems.

Systematic shifts due to changes in the operating conditions have been studied with both lasers locked to the P(16) transition. For the system used in the work reported here, the modulation-dependent shift was  $+4700 \pm 300 \text{ Hz/MHz}_{\text{pp}}$ , the intracavity power shift was  $-11.4 \pm 0.6 \text{ Hz/mW}$ , and the pressure shift was  $-230 \pm 20 \text{ Hz/mTorr}$  [18].

## 3 Frequency comb and measurement system

The optical comb and the system for measuring the frequency of the acetylene-stabilized laser are shown in Fig. 2. The comb used in this work has been described elsewhere [24]. A modelocked Ti:sapphire laser (GigaOptics Gigajet 20), pumped by 5 to 6 W at 532 nm (Coherent Verdi V-8), produced a regular train of 30 to 50 fs-pulses at a repetition rate of approximately 700 MHz. The output was focussed into a 20 to 30 cm-long piece of microstructure fiber [25] in which the spectrum was broadened to over an octave from approximately 500 nm to 1100 nm. The spectrum consisted of a regular comb of optical frequencies separated by the pulse repetition frequency,  $f_{\text{rep}}$  and with an extrapolated frequency offset from 0 Hz of  $f_0$ . A servo with open-loop unity-gain at 2 kHz was used to phase lock the repetition frequency to a high quality synthesizer (Agilent 4423B) through piezo control of the laser cavity length. The synthesizer was referenced to a 10 MHz-signal provided by the NRC hydrogen maser H4, which in turn was monitored through the NRC cesium clock ensemble. A self-referencing system [21] employing a 5 mm-long KTP doubling crystal, was used to measure  $f_0$ . The  $f_0$



**FIGURE 2** Schematic diagram of the optical frequency comb and the frequency doubling system. PPLN – periodically-poled lithium niobate crystal; APD – avalanche photodiode; PD – photodiode; AOM – acousto-optic modulator

signal was amplified and filtered before being digitally divided by 256 and phase locked, through pump-power modulation, to the output from a second maser-referenced synthesizer (SRS DS345). A servo with open-loop unity-gain at 70 kHz was used for this lock. The comb from 500 nm to 950 nm was combined on a beamsplitter with the frequency-doubled light from the acetylene-referenced laser system. A 1200-line/mm grating was used to reflect the spectral region of interest onto an avalanche photodiode that detected the heterodyne beat frequencies between the light from the acetylene-stabilized laser and the nearest comb elements. A bandpass filter was used to select one of these beat frequencies,  $f_B$ , for counting.

Since the comb cannot measure frequencies in the region of 195 THz (1540 nm) directly, the output from the acetylene-stabilized laser was frequency doubled in periodically-poled lithium niobate (PPLN). The PPLN crystal was 1.5 cm long and contained a number of waveguides with differing widths and an effective poling period of approximately 14  $\mu\text{m}$ . The output from the laser was sent by an optical fiber to the room containing the comb and amplified to 32 mW in an erbium fiber amplifier. Half-wave and quarter-wave waveplates were used to adjust the polarization of the light from the amplifier before it was focussed by a 20 $\times$  microscope objective into the PPLN. The crystal was mounted on a thermoelectric cooling element to permit wavelength tuning of the doubling process. However, it was necessary to use several waveguides to cover all the wavelengths of interest. The frequency doubled output from the PPLN had an approximately Gaussian cross section and a power of 10 to 30  $\mu\text{W}$ .

Accurate absolute frequency measurements with an optical frequency comb require high fidelity in the phase-lock servos and counters. Several precautions were taken to ensure the accuracy of the comb measurements in this work.

The S/N ratio of the self-referencing ( $f_o$ ) beat was approximately 40 dB in a 100 kHz bandwidth. The short-term width of this signal was found to vary from less than 100 kHz

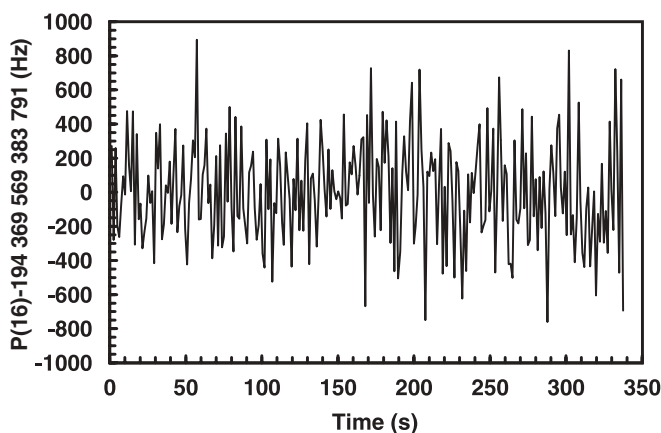
to almost 1 MHz and depended critically on the settings of the mode-locked laser. While the divide-by-256 circuit was not required for the more stable signals, it guaranteed stable phase locking of the  $f_o$  signal when the free-running frequency stability was poor. Since the power and S/N of the  $f_o$  signal could vary with changes in the coupling through the microstructure fiber, this signal was counted directly to monitor the integrity of the phase lock. Data samples for which the counted value differed from the synthesizer setting ( $\times 256$ ) by more than a few hertz, were discarded.

The power and S/N ratio of the  $f_B$  signal was also strongly dependent on the coupling through the microstructure fiber. The S/N ratio of this signal was 30 to 35 dB in a 100 kHz bandwidth. If the S/N dropped below approximately 27 dB, counting errors occurred. These were detected by splitting the  $f_B$  signal and counting it simultaneously with two counters (HP5342A and HP5385A). Any data samples for which the counters differed significantly were discarded.

The  $f_{\text{rep}}$  heterodyne beat had a S/N of over 60 dB in a 100 kHz bandwidth. Therefore, the repetition-rate lock was robust and no phase-lock cycle slips were detected. Such slips are easily detected since they result in errors in the measured optical frequency of over 500 kHz. Since the comb was in an acoustically noisy environment, the comb elements were broad and only partly narrowed by the slow repetition-rate lock. This acoustic noise resulted in excess noise in the comb over what would be expected from the hydrogen maser alone. In other measurements with a quiet laser source, the 1-s instability was measured to be  $4 \times 10^{-13}$ , approximately twice that of the maser [24].

#### 4 Results and discussion

The doubled frequency of the acetylene-stabilized laser was measured on several days with the comb. For these measurements, the laser was locked to the saturated absorption features of the transitions P(10), P(11), P(15), P(16), P(20), and P(21) in the  $\nu_1 + \nu_3$  overtone band of  $^{13}\text{C}_2\text{H}_2$ . The laser was operated at our standard operating conditions – an intracavity, one-way power of 120 mW (35 W/cm<sup>2</sup> inside the cavity near the input mirror), an acetylene pressure of 20 mTorr (2.67 Pa) and a modulation amplitude of 1.0 MHz,



**FIGURE 3** A record of the 1-s comb measurements of the frequency of the laser locked to the P(16) transition in  $^{13}\text{C}_2\text{H}_2$

peak-to-peak. The F–P cavity was filled with fresh acetylene at the start of each day and allowed to stabilize for several hours. Prior to each measurement run, the laser system servos were unlocked and zeroed before they were relocked.

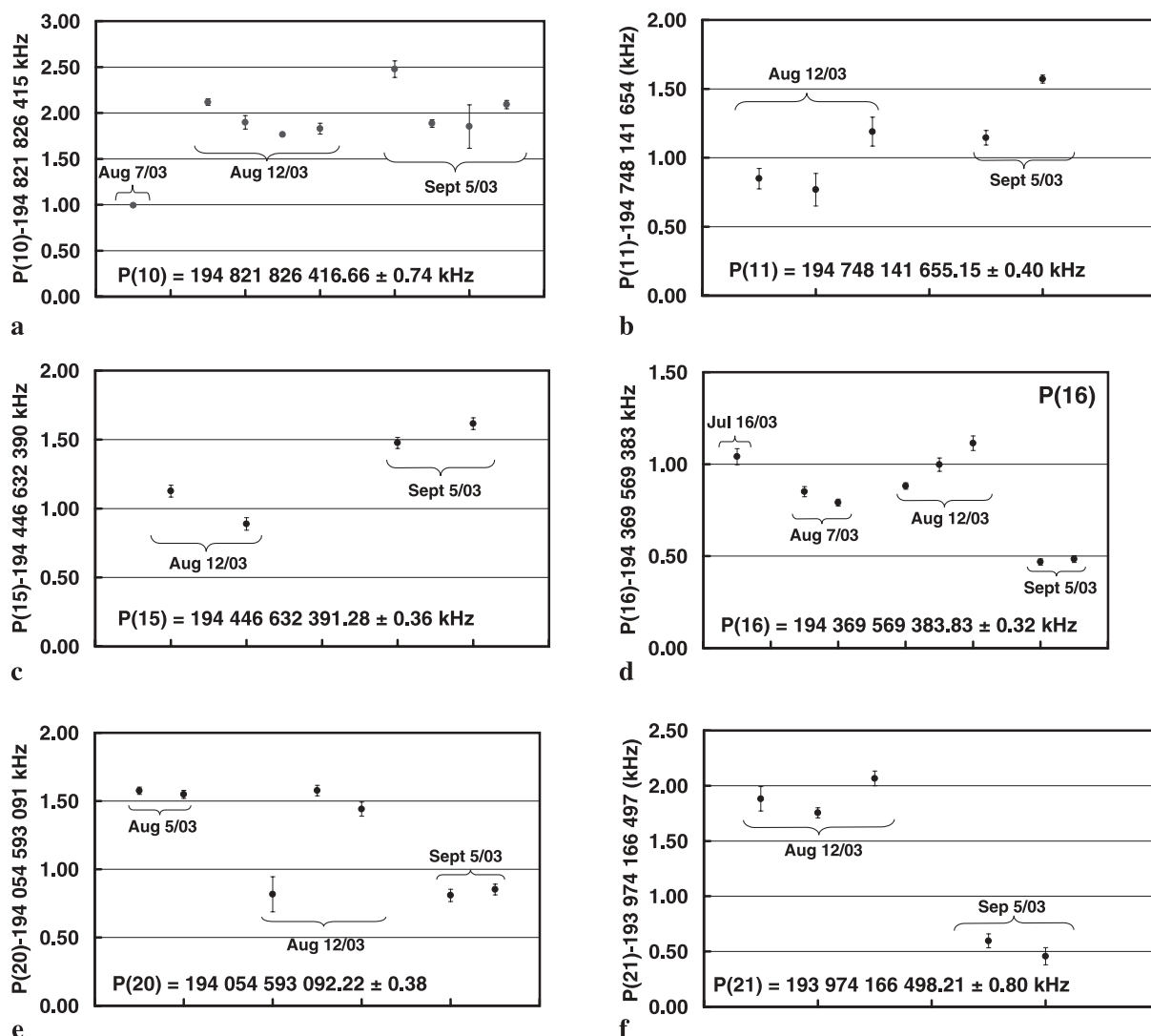
The two counter readings of the heterodyne beat between a comb element and the frequency-doubled acetylene-stabilized laser,  $f_B$ , and the counter reading of the offset frequency,  $f_o$ , were recorded by a computer during each measurement run. Counter gate times of 1 s, with a dead time between readings of approximately 0.3 s, were used throughout the measurements. Most runs were three to five minutes in duration and had no dropouts due to discarded data. Figure 3 shows a sample measurement with the laser locked onto P(16). The laser frequency,  $f_{\text{laser}}$ , was calculated from,

$$2 \times f_{\text{laser}} = n f_{\text{rep}} \pm f_o \pm f_B, \quad (1)$$

where,  $f_{\text{rep}}$  was equal to the synthesizer setting, and  $f_o$  and  $f_B$  were measured. The value of  $n$  and the proper  $\pm$  signs

were chosen through knowledge of the approximate value of  $f_{\text{laser}}$  [7]. The standard deviation of the 1-s measurements of the laser frequency for the data shown in Fig. 3 was 300 Hz and the drift rate was  $-0.22$  Hz/s. Most runs showed a standard deviation for the 1 s-measurements of less than 750 Hz and a drift rate below 1 Hz/s. The corresponding 1 s-instability was  $4 \times 10^{-12}$ , much larger than the comb instability of  $4 \times 10^{-13}$  [24], and, therefore, representative of the instability of the acetylene-stabilized laser. This value is lower than the value of  $7 \times 10^{-12}$  determined from our previous measurements with two similar systems [18]. Although the pressure, power, and modulation settings had been optimized for the P(16) line, the measured stability was similar for the other transitions.

The average laser frequency and an error bar equal to the standard deviation of the mean are plotted in Figs. 4a to f for each run. For each transition, the average frequency for each day is calculated. The unweighted average of these values is listed on each figure along with an uncertainty equal to the



**FIGURE 4** Summary of the comb-based measurements of the frequency of the laser locked to the  $^{13}\text{C}_2\text{H}_2$   $\nu_1 + \nu_3$  overtone transitions: **a** P(10); **b** P(11); **c** P(15); **d** P(16); **e** P(20); and **f** P(21). The average frequency, calculated from an unweighted average of each experimental day's results, is listed on each graph

scatter of the plotted points. These averages are equal to the center frequencies of the transitions at our operating conditions. During these measurements, the hydrogen maser offset from TAI was less than  $8 \times 10^{-14}$ , corresponding to a frequency offset at 194 THz of less than 16 Hz. Except for P(10) and P(21), the scatter of the plotted values shown in Fig. 4 was less than 800 Hz. This level of resetability for a single laser system is much better than the frequency reproduceability between similar systems [16–18] and, therefore not representative of the reproduceability of the standard. A more realistic value was obtained in our previous intercomparison of two similar, but completely independent, systems [18], where it was found that the frequency reproduceability was approximately 2.5 kHz.

The absolute frequencies found in this work are, to our knowledge, the most accurate values determined for the lines P(10), P(11), P(15), P(20), and P(21). Previously, the frequencies of many of the overtone transitions in  $^{12}\text{C}_2\text{H}_2$  and  $^{13}\text{C}_2\text{H}_2$  were measured to an accuracy of 0.120 MHz using an EOM-based optical frequency comb generator and a rubidium standard at 778 nm [7]. The frequencies found in our measurements are in good agreement with those found earlier. The values are identical for P(10), while our value is higher by 0.12 MHz, 0.07 MHz, 0.05 MHz, and 0.10 MHz for P(11), P(15), P(20), and P(21), respectively.

In October 2003, the CIPM adopted a revised value of  $P(16) = 194\,369\,569\,385 \pm 10$  kHz for the P(16) transition frequency at recommended operating conditions of 1.3 to 5.3 Pa pressure,  $1.5 \pm 1.0$  MHz<sub>p-p</sub> modulation amplitude, and  $25 \pm 13$  W/cm<sup>2</sup> [26]. For our laser system at our standard operating conditions, we previously reported a modulation shift of  $4700 \pm 300$  Hz/MHz<sub>pp</sub>, an intracavity power shift of  $-11.4 \pm 0.6$  Hz/mW, and a pressure shift of  $-230 \pm 20$  Hz/mTorr [18]. Using these sensitivities and assuming that the extrapolations are linear, the center frequency of the P(16) transition at the middle values of the CIPM-recommended operating conditions is shifted by  $+1.7 \pm 0.2$  kHz to a value of  $P(16)$  (CIPM conditions) =  $194\,369\,569\,385.5 \pm 0.4$  kHz. Again, considering the reproducibilities of independent laser systems and the possibly incorrect assumption of linear extrapolation of the shift sensitivities, an uncertainty value of 2.5 kHz is more appropriate.

The frequency of the laser locked to P(16), as determined from the comb measurements, was

$$P(16)_{\text{NRC}} = 194\,369\,569\,383.83 \pm 0.32 \text{ kHz}.$$

It is of interest to compare our measurements of the P(16) transition frequency to recent results from AIST in Japan [16] and the NPL in the UK [17]. Comb measurements of two P(16) systems at AIST found frequencies of,

$$P(16)_{\text{AIST1}} = 194\,369\,569\,383.6 \pm 1.3 \text{ kHz},$$

and

$$P(16)_{\text{AIST2}} = 194\,369\,569\,386.5 \pm 1.6 \text{ kHz},$$

while comb measurements at NPL found,

$$P(16)_{\text{NPL1}} = 194\,369\,569\,387.616 \pm 0.076 \text{ kHz},$$

for one system and offset measurements of another system gave,

$$P(16)_{\text{NPL2}} = 194\,369\,569\,384.2 \text{ kHz}.$$

The laser systems used at NRC, AIST, and NPL, were similar in design although the operating parameters varied slightly. The F–P enhancement cavities at AIST and NPL had sealed-off, Brewster-windowed acetylene cells filled to pressures of 4 Pa and 3 Pa, respectively, while the cavity at NRC could be refilled and had a pressure 2.67 Pa. The NPL system used a one-way intracavity intensity of 28 W/cm<sup>2</sup> and a modulation amplitude of 2 MHz<sub>pp</sub>, compared to respective values at NRC of 35 W/cm<sup>2</sup> and 1 MHz<sub>pp</sub>. Using our measured shift sensitivities, it is expected that the frequency of the NRC laser operated at NPL conditions would be shifted higher by 4.5 kHz to  $194\,369\,569\,388.3 \pm 0.4$  kHz, a value somewhat higher than either of NPL results. However, considering that the operating frequencies of systems built in the same laboratory are reproducible to only 2.5 kHz, the agreement between the results obtained from different laboratories is very good.

## 5 Conclusions

We have presented optical comb measurements of the frequency of a diode laser system locked to six transitions in the  $\nu_1 + \nu_3$  overtone band of  $^{13}\text{C}_2\text{H}_2$ . The following values, based on measurements performed over several days, were obtained:

$$P(10) = 194\,821\,826\,416.66 \pm 0.74 \text{ kHz},$$

$$P(11) = 194\,748\,141\,655.15 \pm 0.40 \text{ kHz},$$

$$P(15) = 194\,446\,632\,391.28 \pm 0.36 \text{ kHz},$$

$$P(16) = 194\,369\,569\,383.83 \pm 0.32 \text{ kHz},$$

$$P(20) = 194\,054\,593\,092.22 \pm 0.38 \text{ kHz},$$

$$P(21) = 193\,974\,166\,498.21 \pm 0.80 \text{ kHz}.$$

These values, which span the spectral region from 1538.8 nm to 1545.5 nm should serve as useful calibration frequencies in WDM and other applications. Based on an intercomparison of two completely independent laser systems, the reproducibility of similar lasers locked to any of these transitions is approximately 2.5 kHz.

Our results for the P(16) transition are in excellent agreement with recent comb measurements of similar laser systems developed at AIST in Japan and NPL in the UK. The unweighted average of the NRC, AIST, and NPL results for lasers locked to the P(16) transition is,

$$P(16)_{\text{average}} = 194\,369\,569\,385.1 \pm 1.8 \text{ kHz},$$

in agreement with the recent CIPM-recommended value of this frequency of  $194\,369\,569\,385 \pm 10$  kHz [26]. The excellent reproducibility of this laser system, now at the level of 1 part in  $10^{11}$ , supports its application as a useful and accurate infrared frequency standard.

**ACKNOWLEDGEMENTS** We would like to acknowledge J.-S. Boulanger and S. Cundy for providing and monitoring the Cs-based frequency signal, and R. Pelletier and W. Hoger for assistance with the electronics. The authors would like to thank C.S. Edwards (NPL) and A. Onae

(AIST) for providing us with their groups' results prior to publication. We would like to thank G. Baldenberger of the Institut National d'Optique (INO) for collaboration in the production of the waveguide-based PPLN doubling crystals used in this work. We would also like to acknowledge the support provided by the Canadian Institute for Photonics Innovation (CIPI).

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