

Far Infrared Frequency Synthesis with Stabilized CO₂ Lasers: Accurate Measurements of the Water Vapor and Methyl Alcohol Laser Frequencies

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Abstract—A far infrared (FIR) frequency synthesis technique using saturated-absorption stabilized CO₂ lasers and a point-contact diode has been used to measure frequencies of a number of strong CW H₂O, D₂O, and CH₃OH laser lines. The first frequency measurements of the 79- μ m H₂O, the 73- and 108- μ m D₂O, and 11 CO₂-pumped CW ¹²CH₃¹⁶OH laser lines are reported. This measurement is the first demonstration of the general usefulness of CO₂ lasers for accurate synthesis of FIR frequencies.

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DIRECT laser frequency measurements are made by frequency harmonic generation and mixing in nonlinear diodes and crystals [1], [2]. At longer far infrared (FIR) wavelengths, frequency beats between a high harmonic of a klystron and the laser line have been observed. For instance, the 337- μ m HCN line has been beat with the 12th harmonic of a *V* band klystron in a commercially available diode [3], the 118.8- μ m CH₃OH line has been beat with the 33rd harmonic of a *V* band klystron in a Schottky diode [4], and the 84- μ m H₂O line has been beat with the 401st harmonic of a high quality *X* band klystron in a Josephson junction [5].

While this procedure is desirable insofar as it directly connects the unknown laser frequency with an easily measurable microwave frequency (and consequently to excellent frequency standards), there are a number of serious and as yet unresolved limitations. For instance, as the laser frequency increases, the harmonic number of necessity must

increase. Consequently, severe requirements are imposed on the spectral purity of the microwave source [6] as well as on the S/N capability of the harmonic generator and mixing device. While very low noise, spectrally pure oscillators together with detectors such as the Josephson junction may extend the present microwave multiplication limit, there is a current need for accurate frequency measurement in the intermediate and near infrared (IR) spectral region.

Frequencies greater than the FIR have in fact been measured; the current highest measurement is 148 THz [7]. In these experiments a chain of lasers was used to extend frequency measurements from a microwave oscillator to that of the highest laser frequency. In this chain, the n th harmonic of one laser was mixed with the next higher frequency. By using such a chain of lasers it was possible to reduce the mixing order¹ to less than 14 [3]. This procedure was necessary because the S/N decreased rapidly with increasing harmonic number [1].

This technique has been eminently successful, but for wide application, has suffered from a number of drawbacks. First, the limited number of known laser sources makes it difficult to reduce the unknown-laser synthesized-frequency difference to within the range of commonly available microwave oscillators. Second, stability and reproducibility of many laser oscillators are poor; in these cases, highly accurate measurements require continuous frequency monitoring of the lower frequency lasers during the measurement process. And finally, some of the known CW laser oscillators cannot produce, in a compact device, the power required for effective harmonic generation in a point-contact diode.

Many of these problems can be eliminated if two stabilized CO₂ lasers are chosen to synthesize the IR frequencies,² as shown schematically in Fig. 1. The unknown laser frequency is then given by the expression

$$\nu_L = l\nu_1 + m\nu_2 + n\nu_{\mu w} \pm \nu_B.$$

Here ν_L is the unknown laser frequency, ν_1 and ν_2 are frequencies of CO₂ lasers operating in either the 9.4- or 10.4- μm bands, $\nu_{\mu w}$ is a microwave frequency (X band or less), and ν_B is the beat frequency measured with a spectrum analyzer. l , m , and n are small positive or negative integers such that the mixing order is less than about 10. The CO₂ laser is chosen because every line in each of the two laser bands can be stabilized to a narrow saturated-absorption feature very close to the true molecular frequency [9]. These frequencies are reproducible to within 2 parts in 10^{-10} , and the laser stability is good, e.g., [3]

$$\delta\nu/\nu = 3 \times 10^{-11}, \text{ for } \tau = 1 \text{ s.}$$

Absolute frequency measurements of two CO₂ lines, one in each band [3], coupled with a remeasurement of the rotational constants [10] resulted in absolute frequencies for

¹The mixing order is defined as the sum of the absolute values of the harmonic numbers of all the coherent oscillators being mixed in the diode.

²A multiple CO₂ laser frequency synthesis technique has been proposed for relating CO₂ frequencies to the microwave standard without the use of other lasers in the synthesis chain. See [8].

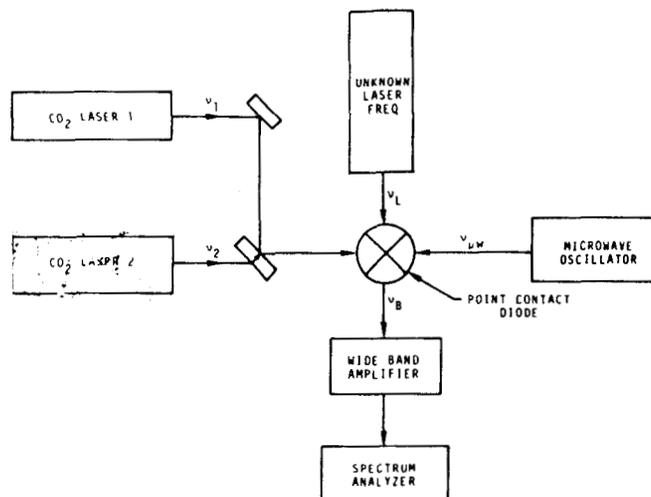


Fig. 1. Schematic diagram of the two-CO₂ laser frequency synthesis experiment.

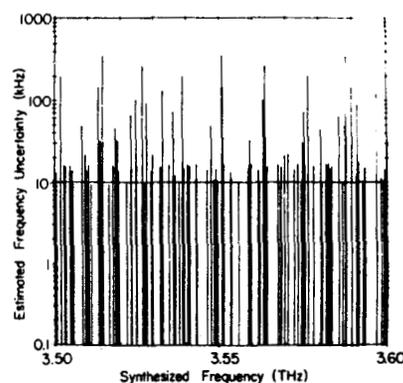


Fig. 2. Difference spectrum from the fundamentals of the first 60 lines in both the 9.4- and 10.4- μm bands of CO₂. The frequency interval taken here, 3.5-3.6 THz, is typical of much of the total frequency range covered. Estimated uncertainty for each synthesized frequency is plotted on the vertical scale.

most of the ¹²C¹⁶O₂ laser lines with a fractional uncertainty of about 1×10^{-9} [11]. Thus, with this technique, the CO₂ lasers can be regarded as secondary frequency standards, and the measured frequency is related to the primary Cs frequency standard through experiments establishing the CO₂ molecular constants. As a result, a comb of over 100 accurately known, reproducible, and highly stable laser frequencies, all with adequate power for harmonic generation, exists in the band from 27 to 33 THz.

If the use of two stabilized ¹²C¹⁶O₂ lasers is considered for frequency synthesis, then many thousands of additional discrete frequencies can be generated. For instance, over 7000 frequencies in the band from 0.025 to 5.8 THz, most with an uncertainty less than 30 kHz, can be produced from differences of the fundamentals of the first 60 lines in both the 9.4- and 10.4- μm bands. As an example, Fig. 2 shows this difference spectrum in the interval from 3.5 to 3.6 THz. If, in addition, sums as well as differences of harmonics from each CO₂ laser are used, then the covered frequency band is greatly extended. In fact, semiuniform frequency coverage from 0.025 to 100 THz can be attained with a mixing order less

than ten. In most cases, the CO₂ combination will produce a frequency that comes within 10 GHz of the one desired. With low mixing orders, *S/N* ratios which are adequate for precision frequency measurements can be expected.

In order to demonstrate the experimental worthiness of this idea, we have used this CO₂ frequency synthesis technique to measure the frequencies of a number of strong CW H₂O, D₂O [12], and CH₃OH [13]-[16] laser lines. Some of these have been measured before by other synthesis techniques [4], [5], [17]-[21] and consequently provide useful comparison with the present measurements. Other motivation stems from the use of laser lines from these molecules for laser magnetic resonance spectroscopy [22], [23], radio astronomy, detector evaluation, and possible standards applications.

Four CO₂ lasers, similar to those described previously [3], were used in the experiment. Two of the lasers were frequency locked to standing-wave saturation dips observed in the 4.3- μ m fluorescent radiation emanating from intracavity absorption cells [9]. Pressure in each absorption cell was 0.035-0.040 torr. No corrections to the CO₂ frequencies were made for pressure, power, or other shifts since these were considered negligible compared to other uncertainties in the experiment. The two ¹²C¹⁶O₂ lasers used to synthesize $\nu_1 - \nu_2$ were offset locked by about 2.5 MHz from the absorption stabilized lasers. This procedure reduced the width of the beat signal caused by the frequency modulation required for line center locking. In each case, the estimated 1-standard-deviation uncertainty in $\nu_1 - \nu_2$ was less than 30 kHz and did not contribute significantly to the uncertainty in the measured frequency.³ Power from each synthesis laser was adjusted to be about 100 mW.

The 8-m-long water vapor laser has also been described previously [3] and in this experiment was filled with H₂ and water vapor to a total pressure of 0.6 torr in a 3:2 ratio. In each measurement, the frequency of the water vapor laser was manually adjusted to the maximum power point of the laser gain curve. Power from the water vapor laser depended on the isotope as well as the transition and ranged from 0.5 to 50 mW.

The other CW FIR laser lines were produced by optically pumping a low-pressure methyl alcohol cell in a waveguide configuration [24] with a 2-m ¹²C¹⁶O₂ laser. The polished-copper cylindrical waveguide cavity, which was also the gas cell, was 14 mm in diameter and 2 m long. Both end mirrors were flat, with one being movable for tuning purposes. CO₂ pump radiation was focused through a 1-mm-diameter hole in the movable input mirror by a 6.4-cm focal length germanium lens. FIR power was coupled out of the other end mirror

through a 2.5-mm-diameter hole. Methyl alcohol vapor pressure ranged from 0.1 to 0.25 torr. The FIR laser had a tuning range of 3-16 MHz, which was both mode and wavelength dependent. Output power ranged from about 0.1 to 10 mW.

All lasers were focused onto the tungsten-nickel point-contact diode with short focal length lenses; microwave power was received from a simple antenna placed close to the diode. The beat frequency between the FIR laser and appropriate harmonic combination of CO₂ lasers and klystron was amplified and subsequently measured with a 1.2-GHz spectrum analyzer.

The CO₂ synthesis combination⁴ together with the measured frequencies of four H₂O and three D₂O laser lines are presented in Table I. Although many CO₂ synthesis combinations often exist for any given FIR frequency, the lowest mixing order was usually chosen. However, in the case of the 28- μ m line, a high mixing order was deliberately chosen for the purpose of measuring the *S/N* ratio. In spite of this high mixing order (9), the *S/N* observed on a spectrum analyzer with a predetection bandwidth of 30 kHz and a video bandwidth of 10 kHz was still greater than 25 dB. The uncertainty in each measured frequency was estimated to be ± 1 MHz, mainly because of difficulty in determining the center of the Doppler and pressure-broadened laser gain curve (10-20 MHz wide). In the third column, measured frequencies have been converted to vacuum wavenumbers with $c = 299\,792\,458$ m/s ($\delta c/c = \pm 4 \times 10^{-9}$), [25].⁵

Two other experiments were performed which did not use the CO₂ laser, but which gave results in good agreement with the results obtained here. In one, the difference frequency between two water vapor lasers oscillating on 78 and 79 μ m, respectively, was beat with a 30-GHz klystron. The measured frequency was 31.298(3) GHz, in good agreement with 31.2968(2) GHz obtained with the CO₂ synthesis technique. In the other a 2-m-long water vapor laser oscillating on 28 μ m which exhibited a Lamb dip was mixed with the 12th harmonic of a 337- μ m HCN laser, of known frequency [3], and a 29-GHz klystron. The frequency of the Lamb dip was found to be within 1 MHz of the top of the gain curve and in good agreement with the results in Table I.

These two results together with previous measurements by other synthesis techniques are listed for comparison at the bottom of Table I. There is general agreement within the estimated experimental errors. The measurements most reproducible and closest to the true molecular frequency are those in which the water vapor laser frequency was set to the center of the Lamb dip. Although the Lamb dip is not necessarily in the center of the gain curve when the laser is operated at maxi-

³Tables of frequencies are given in [11]. Since uncertainties in the rotational constants are correlated, the estimated standard deviation of a frequency difference involves use of the variance-covariance matrix obtained from a least squares fit of the rotational constants to experimental data. The calculation here used experimental data from [10]. A NBS publication giving CO₂ synthesis combinations and estimated errors for the FIR spectral region is in preparation.

⁴Here and again in Table II, the subscripts I and II refer to the CO₂ 10.4- and 9.4- μ m bands, respectively, and are derived from the spectroscopic notation for the lower vibrational level of the laser transition.

⁵This value of c has been recommended by the Consultative Committee for the Definition of the Meter at their meeting in the Bureau Internationale des Poids et Mesures, Sevres, France, and by the International Astronomical Union, Fifteenth General Assembly, Sydney, Australia.

TABLE I
MEASURED FREQUENCIES FOR H₂O AND D₂O LASER LINES

WATER VAPOR LASER LINE (λ-μ)	MEASURED FREQUENCY (MHz) (UNCERTAINTY: ± 1 MHz)	VACUUM WAVENUMBER (cm ⁻¹)	¹² C ¹⁶ O ₂ SYNTHESIS COMBINATION
H ₂ O			
78 ^a	10 718 068.3	357.516 276	3 P ₁₁ (10) - 3 P ₁ (28)
78 ^b	3 821 771.3	127.480 567	P ₁₁ (14) - P ₁ (16)
79 ^c	3 790 474.5	126.736 420	P ₁₁ (10) - P ₁ (18)
119 ^d	2 527 952.0	84.323 402	P ₁₁ (8) - P ₁ (16)
D ₂ O			
72 ^e	4 120 984.2	137.461 240	P ₁₁ (22) - P ₁ (22)
84 ^f	3 557 147.1	118.653 665	P ₁₁ (12) - P ₁ (30)
108 ^g	2 783 066.6	92.833 109	P ₁₁ (10) - P ₁ (24)

Note: Vacuum wavenumbers were calculated from the measured frequencies with $c = 299\,792\,458$ m/s ($\delta c/c = \pm 4 \times 10^{-9}$). The CO₂ synthesis combination with appropriate harmonic numbers is also listed for each measurement. In each case, the estimated 1-standard-deviation uncertainty in the CO₂ synthesized frequency was less than 30 kHz. Microwave frequencies up to 16 GHz were used. A total uncertainty of ± 1.0 MHz has been assigned to each water vapor frequency, mainly because of the difficulty in determining the center of the Doppler and pressure-broadened laser gain curve.

^aEvenson *et al.*, 10 718 073(2) MHz [17]. From HCN laser, 10 718 068.6(5) MHz (Lamb dip). Blaney *et al.*, 10 718 068.71(3) MHz [18] (Lamb dip).

^bEvenson *et al.*, 3 821 775(3) MHz [17]. McDonald *et al.*, 3 821 774.4(3.0) MHz [5].

^cFrom 78-79-μm difference, 3 790 477(3) MHz.

^dFrenkel *et al.*, 2 527 952.8(1) MHz [19] (Lamb dip). Hocker and Javan, 2 527 954.0 MHz [20].

^eThe 72.7-μm line was the only CW line oscillating in our laser near this wavelength. Reference [12] reports CW oscillation at 71.9, but not at 72.7 μm.

^fHocker *et al.*, 3 557 143(2) MHz [21].

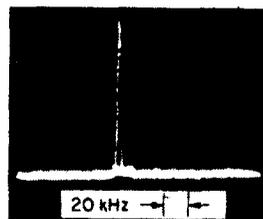


Fig. 3. Beat note at 54 MHz between the 118.8-μm CH₃OH laser line and the synthesized frequency. The spectrum analyzer was set for linear response with RF and video bandwidths of 3 and 10 kHz, respectively. The dc impedance of the diode was 550 Ω.

mum power, the shift between these two points appears to be less than 1 MHz.

A beat signal observed in the measurement of the 118.8-μm methyl alcohol line is shown in Fig. 3. *S/N* ratios varied from 10 to 20 dB. The frequency measurements along with corresponding CO₂ synthesis combinations are presented in Table II. Each CH₃OH line is identified by wavelength and CO₂ pump line. Here wavelengths were calculated from these measured frequencies and in some cases differ slightly from previously published values. In the third column, measured frequencies have been converted to vacuum wavenumbers. Methyl alcohol vapor pressure for each measurement is also given. In each case, the alcohol laser frequency was set to the center of the gain curve. Uncertainty in this procedure resulted in the assignment of a ± 0.5 -MHz error to

each frequency measurement. In comparison, other measurement errors were considered negligible. The CH₃OH frequencies have not been corrected for any type of frequency shift.

The 170.6- and 118.8-μm lines have been measured previously by direct frequency multiplication from the microwave region [4]. The two results 1757.526(6) and 2522.78(1) GHz, are in excellent agreement with the values obtained here.

These measurements demonstrate the feasibility of synthesizing any frequency in the FIR with two ¹²C¹⁶O₂ lasers and a microwave oscillator (X band or less). If, in addition, each ¹²C¹⁶O₂ laser is stabilized to a narrow standing-wave saturated-absorption resonance in ¹²C¹⁶O₂ itself, then the estimated fractional uncertainty in the synthesized frequency is less

TABLE II
MEASURED FREQUENCIES OF 13 METHYL ALCOHOL LASER LINES

METHYL ALCOHOL LASER LINE (μm) (UNCERTAINTY: ± 0.5 MHz)	MEASURED FREQUENCY (MHz)	VACUUM WAVENUMBER (cm^{-1})	ALCOHOL LASER VAPOR PRESSURE (Torr)	CO ₂ PUMP LINE	CO ₂ FREQUENCY SYNTHESIS COMBINATION
70.5	4 251 468.7	141.820 403	0.10	P ₁₁ (34)	R ₁₁ (30) - P ₁ (22)
96.5	3 105 936.6	103.602 901	0.20	R ₁₁ (10)	R ₁₁ (20) - R ₁ (18)
118.6 ^a	2 522 781.0	84.150 935	0.10	P ₁₁ (36)	P ₁₁ (14) - R ₁ (8)
143.0	1 838 839.3	61.337 076	0.10	R ₁ (38)	P ₁₁ (20) - R ₁ (36)
170.6 ^b	1 757 526.3	58.624 765	0.10	P ₁₁ (36)	P ₁₁ (30) - R ₁ (24)
232.9	1 286 999.5	42.929 683	0.20	R ₁₁ (10)	R ₁₁ (32) - P ₁₁ (24)
250.8	1 195 433.9	39.875 383	0.14	R ₁ (34)	R ₁ (34) - P ₁ (20)
251.1	1 193 727.3	39.818 455	0.20	R ₁ (38)	R ₁ (34) - P ₁ (20)
369.1	812 195.4	27.093 924	0.10	P ₁₁ (16)	R ₁ (10) - P ₁ (22)
392.1	764 642.6	25.505 733	0.15	P ₁₁ (36)	R ₁₁ (4) - P ₁₁ (24)
469.0	639 184.6	21.320 902	0.25	R ₁ (38)	P ₁ (8) - P ₁ (32)
570.6	525 427.3	17.326 375	0.15	P ₁₁ (16)	P ₁₁ (18) - P ₁₁ (36)
699.4	428 628.5	14.297 508	0.10	P ₁₁ (36)	R ₁ (38) - R ₁ (14)

Note: Vacuum wavenumbers were calculated from the measured frequencies with $c = 299\,792\,458$ m/s ($\delta c/c = \pm 4 \times 10^{-9}$). The 1-standard-deviation-type uncertainty for each frequency measurement is estimated to be ± 0.5 MHz. The alcohol laser vapor pressure along with the CO₂ pump line are also given. The final column lists the two-laser CO₂ frequency synthesis combination. In each case, the estimated 1-standard-deviation uncertainty in the CO₂ synthesized frequency was less than 30 kHz.

^aFetterman *et al.*, 2522.78(1) GHz [4].

^bFetterman *et al.*, 1757.526(6) GHz [4].

than a few parts in 10^9 . As a result, the accuracy of a measurement is often limited by the reproducibility of the unknown laser frequency. While other combinations and types of lasers can and have been used for frequency synthesis in the past, two stabilized CO₂ lasers by virtue of their large number of lines, high efficiency, simplicity, and excellent frequency reproducibility appear to be the most promising at present.⁶ Since the 2nd and 3rd harmonics of CO₂ have been used to measure CO laser [27] and methane stabilized He-Ne laser frequencies [3], respectively, it is expected that the two-laser CO₂ frequency synthesis technique can be extended to even higher frequencies. Experiments to determine the highest mixing order which will produce usable signals are in progress in anticipation of demonstrating usefulness of this frequency synthesis technique to 100 THz.

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⁶It has been assumed that ¹²C¹⁶O₂ by itself has an adequate number of laser lines for the frequency synthesis process. However, there may be occasions when use of other CO₂ isotopes is advantageous. The frequencies of these isotopes can be measured with the same accuracy as ¹²C¹⁶O₂. See, for example, [26].

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