Notes

Diode laser spectroscopy of methyl chloride overtones at 850–860 nm

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ARTICLE INFO

ABSTRACT

By using a tunable diode laser (TDL) spectrometer 156 CH_3Cl overtone absorption lines have been detected in the range between 11,590 and 11,760 cm^{-1} (8500–8625 Å). Their strengths range around 10^{-26}–10^{-27} cm/molecule and have been measured by utilizing commercial AlGaAs/GaAs laser diodes through the wavelength modulation spectroscopy (WMS) and the 2nd harmonic (2*f*) detection techniques. For one line the self-broadening coefficient has been obtained.

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1. Introduction

Methyl chloride or chloromethane (CH_3Cl) is a symmetric top molecule (C_{3v} symmetry group) and the most abundant natural halocarbon in Earth's atmosphere with the two chlorine isotopes 35 (75.78%) and 37 (24.22%). Methyl chloride is present in the troposphere at a concentration of approximately 1.2 μg/m³ (0.6 ppb); a certain amount of it reaches the stratosphere and there the photodissociation generates chlorine radicals, which contribute to ozone depletion [1]. This is one of the reasons why it has been object of many spectroscopic works especially in the infrared, where the fundamental vibrations are. It has been very much investigated by conventional spectroscopic techniques as well as by the more sophisticated Fourier transform infrared (FTIR) spectrometers [2].

In the gas phase in 1935 Mecke and Vierling [3] identified CH_3Cl overtones at 11,265 cm^{-1} and later also Thompson [4] with a glass Littrow spectrograph observed these overtones at 8880 Å in photographic plates.

Laser absorption spectroscopy has been used by Shang-I Chou et al. [5] for the detection of high-resolution absorption spectra of the first overtone $2\nu_4$ band of CH_3Cl near 1.65 μm by using a DFB InGaAsP diode laser.

The third overtone C–H stretching ($\nu_{CH} = 4$) absorptions in the infrared spectrum of methyl chloride has been identified by Duncan and Law [6].

A systematic study of the overtone spectra of methyl halides has been carried on the 4 and 5 quanta of C–H stretching excitation by Law by using a FTIR spectrometer [7].

In the present work the diode laser absorption spectroscopy technique is utilized in order to observe the rovibrational resonances around 850 nm, presumably overtones ($\nu_{CH} = 4$ polyad) and combinations not yet measured.

The employment of the laser diodes (LDs) enables the use of the wavelength modulation spectroscopy (WMS) technique, which, along with multipass sample cells, is a necessary requirement to increase the signal-to-noise (S/N) ratio and to observe such very weak absorptions lines.

Here we could not distinguish the chlorine 35, 37 isotopes belonging of these bands due to superposition of

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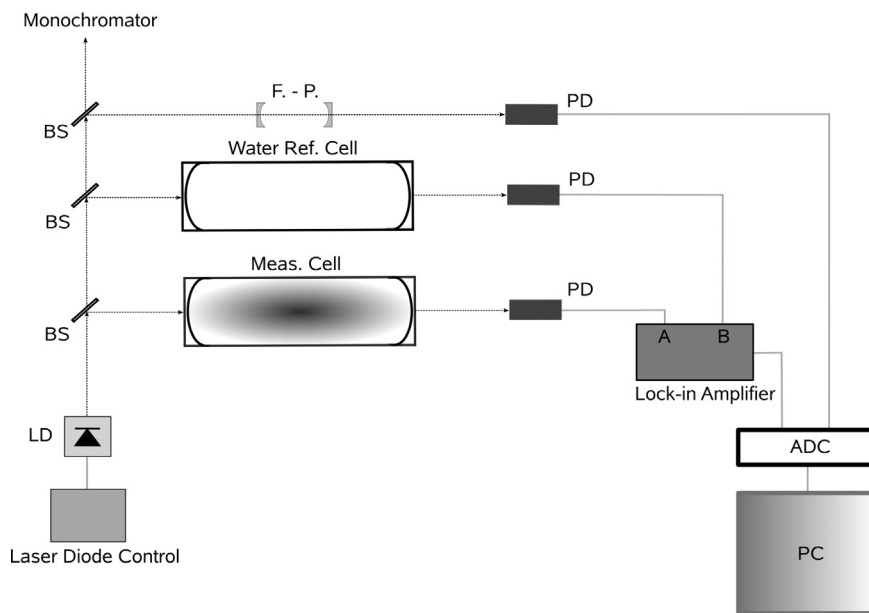 *E-mail address:* lucchesini@ino.it (A. Lucchesini).

Fig. 1. Outline of the experimental apparatus. ADC, analog-to-digital converter; BS, beam splitter; F.–P., Fabry–Perot interferometer; LD, laser diode; M, mirror; PC, desk-top computer; PD, photodiode.

absorption lines at these wavelengths. This is principally caused by the many overlapping bands whose upper states are often coupled by various interactions, such as the Fermi and the Coriolis resonances [7]. For the same reason it was impossible to identify the right quanta of vibration and rotation.

In these conditions more than 150 CH_3Cl absorption lines have been detected and for one of them, at 11,734.00 cm^{-1}, the self-broadening coefficient has been measured at room temperature.

2. Experimental details

The experimental setup for the WMS with the 2nd harmonic detection technique, sketched in Fig. 1, follows partially what used in a previous work [8].

In this case the employed sources have been AlGaAs/GaAs heterostructure LD Roithner RLT85100G and a Thorlabs LP850P030 index-guided multiple quantum well LD. As both of them in sequence were mounted in a "free-running" configuration, their spectral superposition permitted the wavenumbers coverage from 11,590 to 11,760 cm^{-1} with some blank ranges where their mode hops coincided. Their emission has single longitudinal and transverse modes. They were hosted in a small vacuum chamber having a Brewster angle glass exit window. The vacuum was useful when working at low temperature in order to avoid any water vapor condensation and moreover it contributed to maintain the LDs temperature stable. In fact the LDs temperature control is critical as their typical emission wavelength varies as about 0.1 nm/K. In this case the temperature stability and control was guaranteed by a high-stability bipolar temperature controller ($\Delta T = 0.01$ K within 1 h) driving a Peltier junction thermally connected to the LD.

A fine and accurate current control is mandatory (± 10 μA) too, because the LD emission characteristic slope is about 0.01 nm/mA. Therefore a stabilized low-noise ($1 < $ μA rms with 10 MHz bandwidth) Melles Griot 06DLD201 current supply has been adopted for the purpose.

Two 30 m path-length Herriott type multipass cells were used as sample and reference cells. In particular the latter contained water at room temperature vapor pressure ($\simeq 20$ Torr) and was used as the reference for the precise wavenumber measurements. The HITRAN water vapor absorption lines database [9] has been employed for this purpose.

To check the LDs modes and the linearity of their emission frequency a confocal 5 cm Fabry–Perot (F.–P.) interferometer (f.s.r. = 1.5 GHz) was utilized, while a 0.35 m focal length Czerny–Turner monochromator was used for the rough wavelength check (± 0.01 nm).

The methyl chloride was supplied by Matheson Tri Inc.: CP Grade, minimum purity 99.9% ($H_2O \leq 50$ ppm).

The pressure inside the sample cell was measured directly by a Varian 6543-25-045 capacitive pressure gauge (± 0.5 Torr). Here all the experiments were carried on at pressures ranging between 20 and 100 Torr.

Pre-amplified silicon photodiodes (Centronic OSD5-5T, 2.52 mm diameter) have been used as detectors; they did not need to be necessary fast, as the measurements lasted some seconds; the band-width of the lock-in amplifier was set to 10 Hz.

The transmittance $\tau(\nu)$ has been measured following the classical Lambert–Beer expression

$$\tau(\nu) = e^{-\sigma(\nu)z}, \tag{1}$$

where $z = \rho l$ is the product of the absorbing species density ρ (molecule/cm^3) and the optical path l (cm) of the radiation through the sample, that is the column amount

Table 1

Wavenumbers (ν), wavelengths (λ) and maximum absorption cross sections (σ_{max}) at 30 Torr of the measured CH$_3$Cl absorption lines, with maximum error within the second decimal unit. The wavenumbers marked with [a] are probably related to double lines.

ν (cm^{-1})	λ (Å)	$\sigma_{max}\left(10^{-25}\dfrac{cm^2}{molecule}\right)$	ν (cm^{-1})	λ (Å)	$\sigma_{max}\left(10^{-25}\dfrac{cm^2}{molecule}\right)$
11,590.72	8625.27	0.3 ± 0.1	11,638.35	8589.97	3.0 ± 0.5
11,591.20	8624.91	0.2 ± 0.1	11,644.80	8585.21	0.3 ± 0.1
11,591.29	8624.85		11,645.09	8585.00	
11,591.34	8624.81	0.3 ± 0.1	11,645.18	8584.93	0.6 ± 0.1
11,591.57	8624.64		11,645.26	8584.88	0.4 ± 0.1
11,598.12	8619.77	0.3 ± 0.1	11,645.89	8584.41	0.5 ± 0.2
11,598.29	8619.64		11,646.16	8584.21	0.2 ± 0.1
11,598.36	8619.59		11,646.33	8584.09	0.3 ± 0.2
11,609.85	8611.06	1.4 ± 0.4	11,646.65	8583.85	0.5 ± 0.1
11,610.72	8610.41	1.3 ± 0.3	11,653.58	8578.75	
11,612.86	8608.83	0.7 ± 0.1	11,653.68	8578.67	0.3 ± 0.1
11,613.05	8608.69		11,653.87	8578.53	0.3 ± 0.1
11,613.21	8608.57		11,654.06	8578.39	0.5 ± 0.1
11,613.60	8608.28	0.6 ± 0.1	11,654.25	8578.25	0.6 ± 0.1
11,613.74	8608.17	0.3 ± 0.1	11,654.76	8577.88	0.4 ± 0.1
11,616.41	8606.20	0.6 ± 0.3	11,654.85	8577.81	0.5 ± 0.1
11,616.65	8606.02	1.1 ± 0.2	11,655.36	8577.44	0.1 ± 0.1
11,617.42	8605.45		11,655.57	8577.28	
11,617.53	8605.37	1.5 ± 0.7	11,655.64	8577.23	
11,623.59	8600.88	1.0 ± 0.1	11,655.68	8577.20	
11,628.58	8597.19		11,656.73	8576.43	0.3 ± 0.1
11,631.48	8595.04	1.6 ± 0.4	11,657.12	8576.14	0.5 ± 0.2
11,631.88	8594.75	1.2 ± 0.4	11,657.31	8576.00	
11,633.80	8593.33		11,657.40	8575.93	0.3 ± 0.2
11,633.86	8593.29	0.08 ± 0.04	11,661.01	8573.28	1.2 ± 0.2
11,633.94	8593.23		11,661.09	8573.22	
11,633.99	8593.19		11,662.85	8571.93	0.7 ± 0.2
11,634.07	8593.13	1.3 ± 0.1	11,662.95	8571.85	
11,634.27	8592.98		11,663.04	8571.79	
11,634.33	8592.94		11,663.10	8571.74	0.4 ± 0.2
11,634.43[a]	8592.87	0.6 ± 0.1	11,663.42	8571.51	
11,634.50	8592.81		11,663.58	8571.39	2.4 ± 0.6
11,634.59	8592.75		11,664.40	8570.79	1.2 ± 0.6
11,634.64	8592.73		11,664.64	8570.61	
11,634.73	8592.64		11,664.74	8570.54	
11,634.78	8592.61		11,664.89	8570.43	0.4 ± 0.2
11,635.64	8951.97		11,667.22	8567.72	0.5 ± 0.1
11,636.14	8591.60	1.6 ± 0.5	11,669.48	8567.06	0.2 ± 0.1
11,637.34	8590.72	1.2 ± 0.3	11,672.92	8564.53	
11,672.99	8564.48		11,714.64	8534.03	0.9 ± 0.3
11,673.14	8564.37	0.8 ± 0.2	11,717.49	8531.96	
11,677.89	8560.88	0.2 ± 0.1	11,717.64	8531.85	
11,678.02	8560.79	0.3 ± 0.1	11,717.93	8531.63	0.7 ± 0.2
11,678.27	8560.61	0.4 ± 0.1	11,720.09	8530.06	
11,678.38	8560.53	0.2 ± 0.1	11,720.31	8529.90	
11,678.52	8560.42	0.4 ± 0.1	11,725.51	8526.12	0.5 ± 0.3
11,680.90	8558.68	1.3 ± 0.4	11,727.04	8525.01	0.3 ± 0.1
11,681.23[a]	8558.44	0.5 ± 0.2	11,727.12	8524.95	0.6 ± 0.1
11,681.40	8558.31	0.7 ± 0.2	11,727.22	8524.88	0.2 ± 0.1
11,684.29	8556.20	1.0 ± 0.1	11,727.52	8524.66	0.5 ± 0.2
11,684.36	8556.15		11,727.69	8524.53	0.4 ± 0.1
11,684.50[a]	8556.04	1.1 ± 0.1	11,731.42	8521.81	
11,685.07	8555.63		11,731.57	8521.72	0.8 ± 0.2
11,685.20	8555.53	2.0 ± 0.7	11,731.82	8521.53	
11,686.02	8554.93	2.0 ± 0.2	11,734.00	8519.95	0.9 ± 0.2
11,687.38	8553.94	0.5 ± 0.1	11,734.75	8519.41	
11,689.16	8552.63	0.8 ± 0.2	11,734.79	8519.38	0.5 ± 0.1
11,689.40	8552.46	0.2 ± 0.1	11,739.42	8516.02	
11,697.15	8546.79	2.0 ± 0.3	11,739.64	8515.86	
11,698.01	8546.16	0.6 ± 0.2	11,739.80	8515.74	0.5 ± 0.2
11,704.29	8541.58	1.0 ± 0.3	11,739.93	8515.65	0.5 ± 0.1
11,704.50	8541.42		11,740.09	8515.52	0.7 ± 0.1
11,704.67	8541.30		11,740.38	8515.32	0.4 ± 0.2
11,704.87	8541.15		11,740.61	8515.15	1.0 ± 0.3
11,705.12	8540.97	0.8 ± 0.2	11,740.78	8515.03	0.6 ± 0.2
11,705.54	8540.67	0.8 ± 0.2	11,741.03	8514.85	
11,705.88	8540.42		11,741.30	8514.65	0.6 ± 0.2

Table 1 (*continued*)

ν' (cm^{-1})	λ (Å)	$\sigma_{max}\left(10^{-25}\dfrac{cm^2}{molecule}\right)$	ν' (cm^{-1})	λ (Å)	$\sigma_{max}\left(10^{-25}\dfrac{cm^2}{molecule}\right)$
11,706.03	8540.31		11,743.35	8513.17	0.6 ± 0.3
11,706.29	8540.12	0.2 ± 0.1	11,743.52	8513.04	0.5 ± 0.1
11,706.38	8540.05		11743.62	8512.97	0.8 ± 0.2
11,706.45	8540.00	0.5 ± 0.2	11,744.94	8512.01	0.5 ± 0.1
11,706.65	8539.86		11748.57	8509.38	1.0 ± 0.3
11,710.06	8537.37	1.0 ± 0.3	11,751.15	8507.52	
11,710.13	8537.32		11751.29	8507.41	0.9 ± 0.2
11,710.23	8537.24	0.8 ± 0.3	11,756.96	8503.31	0.2 ± 0.1
11,713.20	8535.08	1.0 ± 0.4	11,757.99	8502.57	0.1 ± 0.1
11,713.36	8534.96	0.6 ± 0.3	11,758.61	8502.12	0.3 ± 0.2
11,714.39	8534.21		11,760.67	8500.63	0.5 ± 0.2

(molecule/cm^2). $\sigma(\nu)$ is the absorption cross section (cm^2/molecule).

In this case a small optical depth was verified, that is $\sigma(\nu)z \ll 1$, and Eq. (1) can be approximated by

$$\tau(\nu) \simeq 1 - \sigma(\nu)z. \qquad (2)$$

In order to increase the signal-to-noise ratio (S/N) ratio, the WMS technique described in detail in a previous work [10] has been applied here. This is obtained by modulating the source emission frequency $\bar{\nu}$ at $\nu_m = \omega_m/2\pi$, with amplitude a, that is

$$\nu = \bar{\nu} + a \cos \omega_m t. \qquad (3)$$

In particular the high modulation amplitude regime was necessary for detecting such weak lines, this means a modulation index $m \equiv a/\Gamma > 0.1$, where Γ is the absorption resonance line-width. In this case m ranged mostly between 1 and 2, considering that the CH$_3$Cl Doppler full width at half the maximum (FWHM) at room temperature and 850 nm is 0.61 GHz, and the collisional (Lorentz) FWHM component at 30 Torr is around 0.4 GHz.

Therefore the transmitted intensity depended on the line shape but also on the modulation parameter.

The utilization of the phase detection technique, obtained by a lock-in amplifier, tuned to twice the modulation frequency ($\sim$10 kHz) permitted to get a signal, almost symmetric, the closer to the 2nd derivative of the absorption feature the lower the modulation amplitude was. The symmetry was not perfect for the contemporary amplitude modulation of the source signal that is bound to the variation of the current. Indeed, in high modulation regimes this effect becomes negligible.

A nonlinear least-squares fit procedure was used to extract the line parameters, in particular the line position and its Lorentzian width component used for the pressure broadening measurements.

Even if the 2*f* detection has many advantages as a flat baseline of the signal and final sensitivity limited neither by the detector noise nor by the source noise, it cannot avoid the etalon effect coming principally from the many reflecting surfaces present in the optical path. This was the major drawback of the diode laser frequency modulation spectroscope utilized here and limited its detection sensitivity to absorbance as low as 10^{-6}.

3. Experimental results

One hundred and fifty-six detected absorption lines are listed in Table 1. The maximum wavenumber error (3σ) is within the second decimal unit; this comes out by taking into account the error in the frequency difference between the measured fitted peaks and the reference HITRAN line peaks through the F.–P. interferometer as the frequency marker. The single line peak with its statistical error and the modulation index m were parameters extracted from the non-linear least square fitting procedure that utilized Eq. (13) shown in the Appendix of Ref. [10].

As said previously it was not possible to assign the right ro-vibrational quantum numbers to the single absorption line. Presumably this spectral interval covers part of the 4 quanta of C–H stretching excitation ($4\nu_{CH}$).

When the S/N ratio allowed it, the maximum absorption cross sections (σ_{max}) are reported; they were measured by the direct absorption (DA) technique, because the WMS with high modulation amplitude could bring to big systematic errors in the calculus of the absolute value of the absorbance.

These measurements were performed at room temperature at a pressure around 30 Torr, and 30 m pathlength. The wavelengths have been calculated in air at $T = 294$ K following the work of Edlén [11]. The list cannot pretend to be exhaustive, due to the mode hops suffered by the LDs.

In our knowledge this is the first time these lines have been accurately measured.

An example of the absorption measurement results for CH$_3$Cl at 11,661.01 and 11,661.09 cm^{-1} and $T = 294$ K is shown in Fig. 2, which displays also the H$_2$O reference signal at 11,661.1871 cm^{-1} [9], and the F.–P. interferometer transmission used for the frequency linearization. The lines are broadened by modulation with modulation index $m \simeq 1.8$. The methyl chloride pressure was 30 Torr, while the water vapor pressure in its measurement cell was 19 Torr. The evident etalon effect present in the measurement plots was originated by the reflections inside the multipass cell: the distance between the fringes is $\Delta\nu \simeq 0.006$ cm^{-1}, and the distance between confocal mirrors inside the multipass cell was $l = 42.8$ cm [$\Delta\nu' = 1/(4l)$].

For the 11,734.00 cm^{-1} well isolated absorption line, we extracted the Lorentzian component of the line-width obtained by the fitting procedure of Ref. [10] that takes
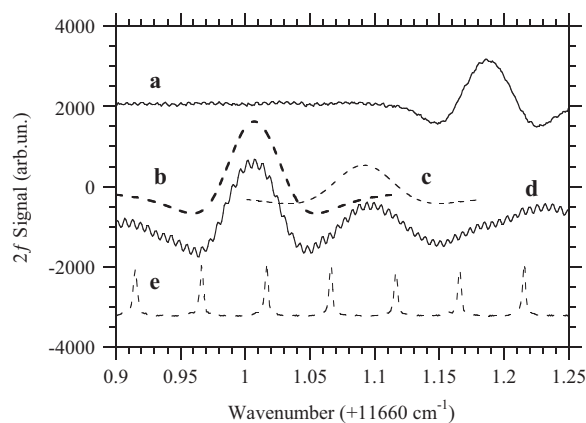
Fig. 2. 2nd harmonic absorption signal of CH_3Cl (d) and H_2O (a) around 857.3 nm obtained by WMS with 10 Hz bandwidth, along with the best peak fits (b and c), and the F.–P. interferometer transmission (f.s. r.=1.5 GHz) (e). p_{CH_3Cl} = 30 Torr, p_{H_2O} = 19 Torr, T=294 K, $m \simeq 1.8$.

Table 2
Lorentzian FWHM (Γ_L) vs. CH_3Cl pressure (p) for the 11734.00 cm^{-1} line.

p (Torr)	Γ_L (GHz)
21	0.3 ± 0.1
30	0.4 ± 0.1
41	0.4 ± 0.1
50	0.6 ± 0.1
60	0.7 ± 0.1
70	1.0 ± 0.1
80	1.0 ± 0.2
89	1.3 ± 0.3

into account the modulation, in this case m=0.75. This component and the Doppler calculated one were put in the Martin–Puerta approximation formula [12] for the Voigt function that well describes the resonance shape. By integrating it in energy we obtained the line-strength with an acceptable error:

$$S = (3.2 \pm 0.7) \times 10^{-27} \text{cm/molecule}.$$

As explained previously, the etalon effect mainly limits the sensitivity of this spectroscopic system, and the minimum detectable line strength is on the order of 1×10^{-27} cm/molecule.

Also a self-broadening measurement at T=293 K has been performed on the same line at 11,734.00 cm^{-1}. In Table 2 the Lorentzian (collisional) FWHMs for different values of the methyl chloride pressure are listed. As said, the pressure error was ± 0.5 Torr.

The corresponding self-broadening coefficient is

$$\gamma_{self} = (11.3 \pm 0.2) \text{ MHz/Torr} = (0.286 \pm 0.005) \text{ cm}^{-1}/\text{atm}.$$

We did not find any other pressure broadening measurement in the literature at these wavenumbers. A comparison can only be tried with what obtained in different spectral regions.

The theoretical work on $CH_3^{35}Cl$ rotational transitions by Dudaryonok et al. [13] has calculated CH_3Cl self-

broadening coefficients: it shows analogous coefficients at high rotational quantum numbers J (upper states 35, 36, 37 for K=0).

Measurements on the ν_5 band by Ramchani et al. [14] show results similar to ours still for high J (J=35, K=1 and J=38, K=4).

The experimental and theoretical self-broadening results for pure rotational transitions in the millimeter wave (186 GHz) obtained by Bray et al. [15] show comparable values for J=37 and $0 < K < 12$ at room temperature.

Also the Bouanic et al. experimental work on the ν_3 fundamental [16] gives self-broadening coefficients, and the values at J=35, K=4 and J=35, K=9 in the ro-vibrational R branch are near the one measured in this work.

4. Conclusion

By using a tunable diode laser spectrometer and the aid of the wavelength modulation and the 2nd harmonic detection techniques 156 methyl chloride overtone absorption lines have been detected at 850–860 nm. Their positions in wavenumbers have been measured within 0.01 cm^{-1} (3σ). Their strengths are on the order of magnitude of 10^{-26}–10^{-27} cm/molecule, which means extinction coefficients of the order of some tens of kilometers in Earth's atmosphere. The self-broadening coefficient has been measured for one of these lines and the results are consistent with what previously measured by using different techniques, even if at different wavelengths. No assignment could be done due to the many overlapping bands whose upper states are often coupled by various interactions (Fermi, Coriolis). An aid could come by working at very low temperature by using the supersonic jet expansion [17]. In this case only the first rotational levels will be populated and the superpositions will be smaller or even absent. Eventually the microwave-optical double resonance [18] could give informations on the rotational quantum numbers associated to the ro-vibrational transitions even in complicated spectra like this one. Hopefully these will be our future projects.

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