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**High resolution IR spectrum of the ν_8 band of CH_2DF :
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**High resolution IR spectrum of the ν_8 band of CH_2DF : analysis of the interactions
with the near vibrational levels**

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Abstract

The IR spectrum of the ν_8 band of monodeutero methyl fluoride has been recorded with a resolution of 0.004 cm^{-1} using a Fourier transform spectrometer. The ν_8 vibration, of A'' symmetry species, gives rise to a pure c -type envelope centred at 1298.58 cm^{-1} , resembling a perpendicular band of a prolate symmetric rotor. From the spectral analysis it has been found that ν_8 is coupled with ν_4 for the low K_a sublevels while for the higher ones other vibrational states, directly or indirectly, are also interacting. For a satisfactory reproduction of the rovibrational data of ν_8 , the Watson's Hamiltonian in the I' representation has been implemented with interaction parameters which included first and second order a -, b -type and c -type Coriolis couplings with ν_4 and ν_9 , respectively. Being these states also interacting with other levels, the effective spectroscopic parameters of the analyzed band up to fourth order have been obtained from a data set formed by the six fundamentals located below 1500 cm^{-1} .

1. Introduction

Accurate spectroscopic constants on methyl fluoride and its isotopologues are of considerable importance for the determination of the equilibrium structure [1], ab initio cubic force field [2] and anharmonic potential energy surface [3]. Among the different isotopologues of this molecule, CH₂DF is the least characterized. Low resolution IR analysis, aimed at the identification of the fundamental bands, was carried out long time ago [4]. In the first high resolution paper [5] the ν_5 and ν_6 fundamentals were analyzed as isolated bands, by fixing the ground state constants to the microwave values obtained by Clark and De Lucia [6]. Subsequently, the ν_3 and ν_4 vibrations were studied using a dyad model [7]; improved ground state parameters were also obtained from a combined fit of infrared **ground state combination differences (GSCDs)** and microwave data of ref. [6]. The anomalous structure of the strongly perturbed ν_9 was then investigated [8] and its interpretation required the re-analysis of the ν_5 and ν_6 states, which are also mutually interacting. The present contribution aims at supplying additional information on CH₂DF through the rovibrational analysis of the ν_8 vibration, located at 1298.58 cm⁻¹. The rotational structure of this perturbed band has been characterized in detail. In order to obtain a satisfactory set of upper state constants, a simultaneous treatment of ν_8 with the near vibrational levels was needed. The model, implemented step by step with interacting terms, required in the final form the employment of experimental data arising from the six lower fundamental bands, located below 1500 cm⁻¹.

2. Experimental

The preparation of CH₂DF was accomplished using the procedure adopted by Edgel and Parts [9] for the synthesis of alkyl and isotopically substituted alkyl fluorides. The precursor was the monodeuterated methyl alcohol (Aldrich, 99%). The purity of the obtained compound, higher than 95%, was tested by gas-chromatography; the gas-phase infrared spectrum showed weak absorptions due to the by-product of synthesis CH₃F. The high resolution (0.004 cm⁻¹, unapodized) spectrum was recorded at the University of Bologna using a Bomen DA3.002 Fourier transform infrared spectrometer. The instrument was equipped with a globar source, a KBr beam splitter and a high-sensitivity mercury-cadmium-tellurium (MCT) semiconductor

as detector. A multipass absorption cell with a base length of 25 cm, by Infrared Analysis, Inc., was used with the optical path length set at 5 m. The spectrum was obtained at room temperature with a sample pressure of about 67 Pa for a negligible pressure broadening ($\cong 0.0004 \text{ cm}^{-1}$). A total of 800 scans were co-added for a satisfactory signal-to-noise ratio. No apodization function was applied. The resulting line width of unblended lines was about 0.005 cm^{-1} . The calculated Doppler line width (FWHM) in the ν_8 band region is 0.0027 cm^{-1} . The weak lines of CH_3F were used as internal standard to calibrate the infrared spectrum [10].

3. Analysis of the spectrum

Monodeutero methyl fluoride is a near prolate asymmetric top of C_s symmetry with the asymmetry parameter $\kappa = -0.978$. The ν_8 mode, described as CD out-plane-bend / CH_2 rock [5], is of A'' symmetry species and gives rise to a c -type band of medium intensity centred at 1298.58 cm^{-1} . The low resolution infrared spectrum of this band, depicted in Figure 1, shows the R branch partially overlapped by the P branch of the weaker ν_4 at 1364.17 cm^{-1} . The prominent clusters in the P and R branches are due to the unresolved ${}^{\text{P,R}}\text{Q}_K(\text{J})$ lines. They were easily identified being the broad and sharp peaks near the band centre typical of the ${}^{\text{P}}\text{Q}_1(\text{J})$ and ${}^{\text{R}}\text{Q}_0(\text{J})$ multiplets respectively. The manifolds corresponding to ${}^{\text{P}}\text{Q}_2(\text{J})$ and ${}^{\text{R}}\text{Q}_1(\text{J})$ are less pronounced since influenced by the most significant effect of asymmetry splitting.

The assignment of the high resolution spectrum proceeded in a straightforward way. The J line sequence belonging to the strong ${}^{\text{R}}\text{Q}_0(\text{J})$ and ${}^{\text{P}}\text{Q}_1(\text{J})$ multiplets was readily checked through GSCDs using the ${}^{\text{P}}\text{P}_{1,2}(\text{J})$ series of transitions. The details of the rotational structure of the sharp ${}^{\text{R}}\text{Q}_0(\text{J})$ group of Figure 1 are depicted in Figure 2, where some unlabelled features belong to the weaker ${}^{\text{R}}\text{P}_K(\text{J})$ and ${}^{\text{P}}\text{R}_K(\text{J})$ subbranches. To be noted that the sequence degrades to lower wavenumbers up to $\text{J}=21$ and then follows an opposite trend. From the assigned transitions a set of preliminary upper state parameters was determined and employed to predict further subbands through a bootstrap procedure. This was done with a semiautomatic program based on correlation plots accounting for wavenumber values and relative intensity of the lines, early proposed by Nakagawa and Overend [11].

In the subbands with $K_a' > 4$ the structure approaches that of a perpendicular band of a symmetric top, as clearly observable in the P-branch region where the interference with lines due to ν_4 is greatly reduced. Concerning the $^P P_K(J)$ and $^R R_K(J)$ subbranches the distinct lines, spreading over a wide range, are regularly spaced by $2\bar{B} \cong 1.56 \text{ cm}^{-1}$. In the $^{P,R} Q_K(J)$ multiplets the J progression degrades towards higher wavenumbers, indicating that the coefficient $(\alpha^A - \alpha^B)$ is positive. A typical example is given in Figure 3, where the details of the $^P Q_7(J)$ manifold are shown. In the same figure it is worth noting the effect of a local interaction splitting the even and odd components of $K_a' = 6$, otherwise degenerate. The crossings occur at $J = 24/25$ even and $J = 25/26$ odd components. The experimental shifts from the predicted line positions, before and after the crossings, suggested that the perturbation arises from a K_a sublevel located at higher energies, as evident in Figure 4 where part of the ν_4 and ν_8 reduced energy level diagram is shown. Therefore the observed irregularities were attributed to second-order a -Coriolis interaction with $K_a = 4$ of ν_4 . Differently, the local crossing identified in the even component of $K_a = 5$ at $J = 20/21$ is due to second-order b -Coriolis mixing with the $K_a = 2$ even component of ν_4 . The effect of this perturbation was already observed in the ν_4 spectral region [7].

The assignment of the subbands with $K_a' > 9$ was performed with care since the involved levels are influenced by stronger interactions. Actually, systematic negative residuals were observed in the transitions with $K_a' \leq 11$ and a third crossing, identified for $K_a = 12$ at $J = 14/15$, was ascribed to first-order b -Coriolis coupling with $K_a = 11$ of ν_4 .

The analysis was carried out between $1180\text{--}1410 \text{ cm}^{-1}$. The assignment, also including the weaker $^P R$ and $^R P$ series, was made up to $K_a' = 12$ with a maximum of $J' = 36$ for the strongest branches.

4. Simultaneous fit of ν_8 with the ν_3 , ν_4 , ν_5 , ν_6 and ν_9 levels.

The ν_8 level was initially analyzed by the Watson A -reduced Hamiltonian in the I' representation [12]. In the preliminary fits, carried out with the ground state constants fixed at the values of [7], the upper state constants showed unsatisfactory values. This is because of interaction with the near vibrational levels. Five fundamentals, besides ν_8 , are located in the

spectral region of CH₂DF below 1500 cm⁻¹: ν_3 (1465 cm⁻¹, A'), ν_4 (1364 cm⁻¹, A'), ν_5 (1055 cm⁻¹, A'), ν_6 (938 cm⁻¹, A') and ν_9 (1133 cm⁻¹, A''). From symmetry considerations it follows that *a*- and *b*-type Coriolis resonances are allowed between vibrations of different symmetry species, whereas *c*-type Coriolis coupling and/or vibrational resonance link those of identical species. Excepting ν_8 , the other bands have already been studied [5, 7, 8].

As mentioned, local crossings showing the typical Coriolis J-dependence were observed in the ν_8 band and ascribed to coupling with ν_4 . Thus, as starting point, the low K_a transitions of ν_8 were simultaneously fitted with the data of the ν_3 / ν_4 dyad, by also including a number of perturbed transitions of ν_4 omitted in the earlier study. At this stage of the analysis, the second order *a*- and *b*-type Coriolis coefficients linking ν_4 and ν_8 were employed. In the calculations, carried out using the SPFIT program written by Pickett [13], an uncertainty of 0.0004 cm⁻¹ was given to lines assigned to single transitions, while for blended or very weak features the uncertainty was 0.0010 cm⁻¹. Next, to perform refinements with higher J and K_a transitions, first-order *a*- and *b*-type Coriolis terms connecting ν_8 with ν_4 were also employed. As expected, their introduction in the fit gave an improvement and most of the spectroscopic parameters of ν_8 displayed suitable values. Only the rotational constant C and the centrifugal distortion parameter Δ_{JK} revealed rather large shifts (0.003 and -0.069×10^{-4} cm⁻¹) from their ground state values. These shifts compared with the corresponding ones obtained for ν_9 [8], of opposite sign (-0.005 and 0.056×10^{-4} cm⁻¹), suggested the presence of a global *c*-type Coriolis resonance. All the assigned transitions of the ν_5 , ν_6 and ν_9 states, mutually interacting as described in ref. [8], were then included in the data set. The preliminary values for the first-order Coriolis couplings, linking ν_8 to the perturbers, were derived from the dimensionless constants of the harmonic force field [14]. Many attempts were performed in this fitting process because the convergence was somewhat difficult to reach. A goal was that the refined upper state parameters should not significantly differ from the ground state values. However, anomalous residuals remained for transitions to $K_a = 12$, especially in the crossing region with ν_4 , and were attributed to mutual interactions also involving ν_5 and ν_9 . Actually, the reduced energy level scheme indicates that $K_a = 12$ of ν_8 crosses $K_a = 11$ of ν_4 and comes close to $K_a = 15$ and $K_a = 14$ of ν_5 and ν_9 , respectively. The lack of experimental data concerning these levels thwarted any attempt to fit the restricted number of transitions of ν_8 with $K'_a = 12$ and so they were omitted from the final refinement. **It should be recalled here that an extension of**

the analysis is mainly dependent on new measurements of the ν_9 band which is very weak and only observed in the spectrum through perturbation-allowed transitions by intensity borrowing from the very strong ν_5 band [8].

The best representation of the interacting level system was finally given by a model involving seven first- and three second-order Coriolis terms together with two second-order vibrational resonance constants, as illustrated in Figure 5. The interaction matrix elements used in the model are summarized, for convenience, in Table 1. The spectroscopic parameters obtained from a fit comprising most of the assigned transitions are given in Tables 2 and 3. The results have been obtained with a standard deviation of $0.107 \times 10^{-2} \text{ cm}^{-1}$ which is higher than the accuracy of the fitted lines but comparable to that obtained in the previous analysis [7, 8]. Table 2 collects the upper state parameters of the $\nu_3 / \nu_4 / \nu_5 / \nu_6 / \nu_8 / \nu_9$ hexade and, for completeness, the ground state constants. On the whole the refined parameters appear well determined. In particular the C and Δ_{JK} terms of ν_9 , that in the previous analysis were deviating from the ground state values, are now more suitable. Where consistent with the available experimental data, the parameters δ_J and δ_K were refined, otherwise they were constrained to the ground state values. No sextic centrifugal distortion constants could be obtained with significance. However, it should be mentioned that some resonance contributions, which the model is unable to account for, still remain.

The Coriolis and Fermi term values are listed in Table 3. To maintain the flexibility of the model, notwithstanding the existence of strong correlations with the molecular parameters, all the coupling coefficients relevant for the analysis were refined. The table also provides, in square brackets, the computed values of the seven first-order Coriolis parameters, derived from the dimensionless constants of the harmonic force field of methyl fluoride [14]. The comparison with the empirical values show generally a satisfactory agreement. The larger difference concerns the smaller coupling coefficient $\xi_{4,8}^b$, probably not too well determined in the refinement.

To test the reliability of the obtained parameters, simulations were performed in different spectral regions of ν_8 . A small portion of the synthetic spectrum is reported in the upper trace of Figure 3. It matches very well with the experimental one (lower trace) and reproduces the anomalous splittings confirming that the employed model satisfactorily represents the system involving the ν_3 , ν_4 , ν_5 , ν_6 , ν_8 , and ν_9 interacting levels.

In conclusion, the high-resolution infrared study of the strongly perturbed ν_8 level of CH_2DF has been reported for the first time. The spectrum has been analyzed in detail and the observed irregularities were attributed to different Coriolis resonances with ν_4 and ν_9 , in turn coupled with other levels. For this reason the previously analyzed systems were included in the data set of ν_8 and, from a combined analysis of six interacting states, a set of accurate spectroscopic constants, which satisfactorily reproduces the experimental features within the measurement accuracy, was determined. With this work all levels of CH_2DF located below 1500 cm^{-1} have been characterized, providing a large amount of data which should give valuable information to test the existing anharmonic potential energy surface and to determine an improved cubic force field of methyl fluoride.

A complete list of the transitions used in the final fit has been deposited as Supplementary online material.

Acknowledgements

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Figure captions

Figure 1. Overview of the low resolution (0.5 cm^{-1}) IR spectrum of the ν_8 *c*-type band of CH_2DF ($P = 24 \text{ kPa}$, path length 10 cm). The R-branch merges with the weaker hybrid ν_4 band. The high resolution spectrum of the central peak marked with * is reproduced in Figure 2.

Figure 2. Spectral portion of the $^R\text{Q}_0(J)$ multiplet of the ν_8 band of CH_2DF ($P = 67 \text{ Pa}$, path length 5 m) showing a turnover in the assignment at $J = 21$. Many weaker unlabelled lines belong to different ^RP and ^PR subbranches with low K_a values.

Figure 3. The $^P\text{Q}_7(J)$ multiplet of the ν_8 band of CH_2DF (lower trace; $P = 67 \text{ Pa}$, path length 5 m) is compared with the simulated spectrum (upper trace). The local resonance observed at $J = 24/25$ and $J = 25/26$, attributed to crossings with $K_a = 4$ of ν_4 , produces split lines with intensity depletion compared to the regular series of the J manifold.

Figure 4. Part of the reduced energy level diagram of ν_4 and ν_8 of CH_2DF . The energies have been calculated from the upper state constants of Table 3. The ν_4 sublevels are indicated by dashed lines, those of ν_8 by full lines. The $\Delta K_a = \pm 2$ (square) and $\Delta K_a = \pm 3$ (circle) crossings are due to *a*- and *b*-type Coriolis resonances.

Figure 5. Vibrational energy levels of CH_2DF below 1500 cm^{-1} . The figure shows the level symmetry and the interaction parameters employed to analyze the $\nu_3 / \nu_4 / \nu_5 / \nu_6 / \nu_8 / \nu_9$ hexade. $\xi_{r,s}^a$ ($a = a, b, c$) denotes first-order Coriolis interaction between ν_r and ν_s ; $\eta_{r,s}^{b,c}$ and $\theta_{r,s}^b$ indicate second order *a*- and *b*-Coriolis coupling while $W_{r,s}$ refers to second order Fermi resonance.

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Table 1. Interaction matrix elements of the Hamiltonian in symmetric top basis

Type Resonance	Interaction Matrix Elements
First order <i>a</i> -Coriolis	$\langle v_r = 1, J, K \mathbf{H} v_s = 1, J, K \rangle = \xi_{r,s}^a \cdot K$
First order <i>b</i> -Coriolis	$\langle v_r = 1, J, K \mathbf{H} v_s = 1, J, K \pm 1 \rangle = \frac{1}{2} \cdot \xi_{r,s}^b \cdot F(J, K)$
First order <i>c</i> -Coriolis	$\langle v_r = 1, J, K \mathbf{H} v_s = 1, J, K \pm 1 \rangle = \pm \frac{1}{2} \cdot \xi_{r,s}^c \cdot F(J, K)$
Second order <i>a</i> -Coriolis	$\langle v_r = 1, J, K \mathbf{H} v_s = 1, J, K \pm 2 \rangle = \mp \frac{1}{2} \cdot \eta_{r,s}^{bc} \cdot F(J, K) \cdot F(J, K \pm 1)$
Second order <i>b</i> -Coriolis	$\langle v_r = 1, J, K \mathbf{H} v_s = 1, J, K \pm 3 \rangle = \pm \frac{1}{8} \cdot \Theta_{r,s}^b \cdot F(J, K) \cdot F(J, K \pm 1) \cdot F(J, K \pm 2)$
Second order Fermi	$\langle v_r = 1, J, K \mathbf{H} v_s = 1, J, K \pm 2 \rangle = \frac{1}{2} \cdot W_{r,s} \cdot F(J, K) \cdot F(J, K \pm 1)$

$$F(J, K) = [J(J+1) - K(K+1)]^{1/2}$$

$$\xi_{r,s}^a = A_e \cdot \zeta_{r,s}^a \cdot (\omega_r + \omega_s) / (\omega_r \cdot \omega_s)^{1/2}$$

$$\xi_{r,s}^b = B_e \cdot \zeta_{r,s}^b \cdot (\omega_r + \omega_s) / (\omega_r \cdot \omega_s)^{1/2}$$

$$\xi_{r,s}^c = C_e \cdot \zeta_{r,s}^c \cdot (\omega_r + \omega_s) / (\omega_r \cdot \omega_s)^{1/2}$$

Table 2. Spectroscopic parameters (cm⁻¹) for the $\nu_3, \nu_4, \nu_5, \nu_6, \nu_8, \nu_9$ band system of CH₂DF ^a

	Ground State ^b	ν_3	ν_4	ν_5	ν_6	ν_8	ν_9
ν_0		1465.477574(26)	1364.168036(32)	1055.544760(37)	938.300406(50)	1298.575644(29)	1132.78084(27)
A	3.99193092	3.9931639(17)	4.001852(97)	3.984741(21)	3.9924941(24)	3.948220(97)	4.002762(23)
B	0.80200182	0.80376852(34)	0.8024725(20)	0.795222(14)	0.79885475(44)	0.7993000(21)	0.797977(15)
C	0.76584315	0.7637980(47)	0.7664034(49)	0.7564945(32)	0.7640131(33)	0.765618(18)	0.764134(17)
$\Delta_J \times 10^5$	0.164497	0.177118(17)	0.157725(40)	0.172092(38)	0.157312(56)	0.166028(17)	0.16673(16)
$\Delta_{JK} \times 10^4$	0.114538	0.127688(99)	0.095506(77)	0.108259(69)	0.116286(74)	0.09643(22)	0.13727(28)
$\Delta_K \times 10^4$	0.45993	0.47221(12)	0.49182(78)	0.44923(15)	0.47274(24)	0.40995(79)	0.49874(84)
$\delta_J \times 10^7$	0.77177	0.5643(15)	1.0934(42)	0.4089(64)	0.77177 ^c	0.6198(16)	0.77177 ^c
$\delta_K \times 10^5$	0.1826	1.4445(55)	0.5656(55)	0.22981(99)	0.1826 ^c	0.16182(99)	0.1826 ^c
^d $\Phi_{JK} \times 10^{10} = 0.587, \Phi_{KJ} \times 10^9 = 0.203, \Phi_K \times 10^8 = 0.237$							
$\sigma \times 10^3$	1.072						
No. of Data	2029	1275	1007	588	1737	210	
$J_{\max}$	37	28	33	27	36	26	
$K_a \max$	11	10	12	10	11	10	

^a Quoted uncertainties are one standard deviation in units of the last significant digit.

^b From [7].

^c Fixed at the ground state value.

^d Ground state sextic coefficients from [7] fixed for the upper levels.

Table 3. Coriolis and Fermi coupling values (cm^{-1}) obtained from the fit of the $\nu_3, \nu_4, \nu_5, \nu_6, \nu_8, \nu_9$ band system of CH_2DF^a

Type Resonance		First Order ^b		Second Order	
<i>a</i> -type Coriolis	$\xi_{4,8}^a$	0.7433(43)	[0.9607]	$\eta_{4,8}^{bc}$	$0.8629(56) \times 10^{-3}$
	$\xi_{5,9}^a$	0.98720(81)	[1.0687]		
<i>b</i> -type Coriolis	$\xi_{4,8}^b$	-0.19524(35)	[0.1386]	$\Theta_{4,8}^b$	$0.11100(19) \times 10^{-1}$
	$\xi_{5,9}^b$	0.72490(76)	[0.6968]	$\Theta_{5,9}^b$	$-0.3126(30) \times 10^{-2}$
<i>c</i> -type Coriolis	$\xi_{3,4}^c$	1.25572(19)	[1.1787]		
	$\xi_{5,6}^c$	0.72747(26)	[0.5653]		
	$\xi_{8,9}^c$	0.7882(19)	[0.8639]		
Fermi				$W_{3,4}$	$-0.856768(33) \times 10^{-3}$
				$W_{5,6}$	$0.174779(33) \times 10^{-3}$

^a Quoted uncertainties are one standard deviation in units of the last significant digit.

^b The parameters in brackets were derived from the harmonic force field [14].

High resolution IR spectrum of the ν_8 band of CH_2DF : analysis of the interactions with the near vibrational levels

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List of the fitted transitions of ν_3 (p. 2–15), ν_4 (p. 15–23), ν_5 (p. 23–30), ν_6 (p. 30–33), ν_8 (p. 33–45) and ν_9 (p. 45–46) bands.

Each column reports:

J' , K_a' , K_c' , J'' , K_a'' , K_c'' , Obs (cm^{-1}), (Obs. - Calc.) $\times 10^4$ (cm^{-1}), Uncertainty $\times 10^4$ (cm^{-1}).

v ₃ Band										14	7	7	15	8	8	1395.6259	-7	4
19	11	8	20	12	9	1364.7098	13	4		19	6	13	20	7	14	1395.6409	4	4
18	11	7	19	12	8	1365.7453	15	4		9	8	1	10	9	2	1395.7217	-10	4
17	11	6	18	12	7	1366.7928	18	4		24	5	19	25	6	20	1395.8643	2	4
22	10	12	23	11	13	1367.5174	1	4		19	11	8	19	12	7	1395.9518	7	4
16	11	5	17	12	6	1367.8524	10	4		20	11	9	20	12	8	1396.4837	3	4
21	10	11	22	11	12	1368.5839	0	4		13	7	6	14	8	7	1396.8186	9	4
15	11	4	16	12	5	1368.9274	13	4		18	6	12	19	7	13	1396.8777	4	4
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