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### **Raman spectroscopy of uranyl rare earth carbonate kamotoite(Y)**

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#### **Abstract**

Raman spectroscopy at 298 and 77K has been used to study the mineral kamotoite-(Y), a uranyl rare earth carbonate mineral of formula  $Y_2(UO_2)_4(CO_3)_3(OH)_8 \cdot 10-11H_2O$ . The mineral is characterised by two Raman bands at  $1130.9\text{ cm}^{-1}$  and  $1124.6\text{ cm}^{-1}$  assigned to the  $\nu_1$  symmetric stretching mode of the  $(CO_3)^{2-}$  units, while those at  $1170.4$  and  $862.3\text{ cm}^{-1}$  (77 K) to the  $\delta$  U-OH bending vibrations. The assignment of the two bands at  $814.7$  and  $809.6\text{ cm}^{-1}$  is difficult because of the potential overlap between the symmetric stretching modes of the  $(UO_2)^{2+}$  units and the  $\nu_2$  bending modes of the  $(CO_3)^{2-}$  units. Only a single band is observed in the 77 K spectrum at  $811.6\text{ cm}^{-1}$ . One possible assignment is that the band at  $814.7\text{ cm}^{-1}$  is attributable to the  $\nu_1$  symmetric stretching mode of the  $(UO_2)^{2+}$  units and the second band at  $809.6\text{ cm}^{-1}$  is due to the  $\nu_2$  bending modes of the  $(CO_3)^{2-}$  units. Bands observed at  $584$  and  $547.3\text{ cm}^{-1}$  are attributed to water librational modes. An intense band at  $417.7\text{ cm}^{-1}$  resolved into two components at  $422.0$  and  $416.6\text{ cm}^{-1}$  in the 77 K spectrum is assigned to an  $Y_2O_2$  stretching vibration. Bands at  $336.3$ ,  $286.4$  and  $231.6\text{ cm}^{-1}$  are assigned to the  $\nu_2$   $(UO_2)^{2+}$  bending modes. U-O bond lengths in uranyl are calculated from the wavenumbers of the uranyl symmetric stretching vibrations. The presence of symmetrically distinct uranyl and carbonate units in the crystal structure of kamotoite-(Y) is assumed. Hydrogen-bonding network related to the presence of water molecules and hydroxyls is shortly discussed.

Keywords: kamotoite, mineral, uranyl, rare earth elements, carbonate, Raman spectroscopy

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## Introduction

Uranyl carbonates are common minerals which precipitate from carbonate-bearing solutions in the oxidized zones of uranium deposits. Several of them may contain yttrium and rare-earth elements which are minor constituents of uraninite [1]. Complex uranyl carbonates are important products of the uraninite dissolution and also formation of secondary, i.e. uranyl minerals. Uranyl carbonates may be also formed during hydration-oxidation alteration of spent nuclear fuel and are therefore important for the disposal of the nuclear waste. A knowledge of crystal structures and crystal chemistry, geochemistry and paragenesis of uranyl carbonates is therefore very necessary for the environmental chemistry and geochemistry of radionuclides [1].

Kamotoite is the monoclinic uranyl rare earth carbonate mineral of formula  $Y(\text{REE})_2(\text{UO}_2)_4(\text{CO}_3)_3 \cdot 10\text{-}11\text{H}_2\text{O}$  [1-3]. The mineral is related to the minerals astrocyanite-(Ce)  $[\text{Cu}_2(\text{Ce},\text{Nd},\text{La})_2(\text{UO}_2)(\text{CO}_3)_5(\text{OH})_2 \cdot 1.5\text{H}_2\text{O}]$  [4], shabaite-(Nd)  $[\text{Ca}(\text{Nd},\text{Sm},\text{Y})_2(\text{UO}_2)(\text{CO}_3)_4(\text{OH})_2 \cdot 6\text{H}_2\text{O}]$  [5] and bijvoetite-(Y)  $[(\text{Y},\text{Dy})_2(\text{UO}_2)_4(\text{CO}_3)_4(\text{OH})_6 \cdot 11\text{H}_2\text{O}]$  [6] or  $[\text{M}^{3+}_8(\text{H}_2\text{O})_{25}(\text{UO}_2)_{16}\text{O}_8(\text{OH})_8(\text{CO}_3)_{16}](\text{H}_2\text{O})_{14}$ ,  $\text{M} = \text{Y}, \text{REE}$  [1]. Kamotoite-(Y) is formed in the oxidized zone above a uranium-bearing Cu-Co deposit at Kamoto mine, Shaba, Democratic Republic of Congo [1, 2, 7]. It occurs as crusts of elongated yellow blades on uraninite matrix [8]. The mineral is commonly associated with shabaite-(Nd) and uranophane, less frequently with the rare uranyl and rare earth phosphate françoisite-(Nd) [3]. Kamotoite-(Y) is monoclinic, space group  $P2_1/a$ ,  $a$  21.22(1),  $b$  12.93(1),  $c$  12.39(1) Å,  $Z = 4$  [1]. No other structural data on kamotoite-(Y) are available. Some crystallochemical relations may be probably found between bijvoetite-(Y), the crystal structure of which is known [1], and kamotoite-(Y). Crystal structures of shabaite-(Nd) and astrocyanite-(Ce) are yet not known.

The infrared spectrum has been published [9]. Jones and Jackson [10] published infrared spectrum (graph and wavenumbers of bands) without any assignment. All available infrared wavenumbers of kamotoite-(Y) are included in Table 1. A low intensity band at  $1120\text{ cm}^{-1}$  was assigned to the  $\nu_1$  symmetric stretching mode of the  $(\text{CO}_3)^{2-}$  units. Čejka published both the infrared absorbance spectrum and a DRIFT spectrum of kamotoite and reported this low intensity band at  $1123\text{ cm}^{-1}$  [11]. Botto et al. reported the  $(\text{CO}_3)^{2-}$  antisymmetric stretching modes as intense infrared bands at  $1540$  and  $1333\text{ cm}^{-1}$  [9]. Čejka reported the splitting of the first band into bands at  $1553$  and  $1536\text{ cm}^{-1}$  [11]. Botto et al. proposed that the splitting of the antisymmetric stretching mode indicated a bidentate bonding of the carbonate units to the uranyl groups [9]. Botto et al. also assigned the bands at  $505$ ,  $470$  and  $352\text{ cm}^{-1}$  to the uranyl group [9]. The position of these bands are partly not in agreement with the published data (DRIFT  $504$ ,  $496$ ,  $443$  and  $403\text{ cm}^{-1}$  of Čejka [11]) and  $509$ ,  $366$  and  $280\text{ cm}^{-1}$  by Jones and Jackson [10]. Further the assignment of bands in these positions to the uranyl group is open to question. Botto et al. ascribed bands at  $270$  and  $215$  to uranyl-carbonate stretching and carbonate bending vibrations [9]. In the light of more recent studies this assignment would not appear to be correct. Anderson et al. [12] attributed Raman and infrared bands observed in the spectra of  $\text{K}_4[(\text{UO}_2)(\text{CO}_3)_3]$  in the regions  $48\text{-}96\text{ cm}^{-1}$  to translations (including K),  $106\text{-}132\text{ cm}^{-1}$

to  $(\text{UO}_2)^{2+}$  librations, 152-211  $\text{cm}^{-1}$  to  $(\text{CO}_3)^{2-}$  librations, and 241-307  $\text{cm}^{-1}$  to the  $\nu_2$  ( $\delta$ )  $(\text{UO}_2)^{2+}$  bending vibrations. Volod'ko et al. [13] assigned bands in the region 70-165  $\text{cm}^{-1}$  to the  $\delta$  ( $\text{OUO}_{\text{ligand}}$ ) and  $\delta$  ( $\text{O}_{\text{ligand}}\text{UO}_{\text{ligand}}$ ) vibrations, 200-225  $\text{cm}^{-1}$  to the  $\nu$  ( $\text{U-O}_{\text{ligand}}$ ) vibrations, and close to 255 and 280  $\text{cm}^{-1}$  to the  $\nu_2$  ( $\delta$ )  $(\text{UO}_2)^{2+}$  vibrations.

One of the difficulties in studying the infrared (and Raman) spectra of uranyl carbonates is the potential overlap of bands associated with  $(\text{UO}_2)^{2+}$  and the  $(\text{CO}_3)^{2-}$  units. The region for the symmetric stretching vibration of the  $(\text{CO}_3)^{2-}$  units is a spectral window free from bands ascribed to the  $(\text{UO}_2)^{2+}$  units. One potential overlap is between the antisymmetric stretching vibrations of the  $(\text{CO}_3)^{2-}$  units and the  $\delta$  water modes. Another major difficulty is the possible overlap of the symmetric stretching modes of the  $(\text{UO}_2)^{2+}$  units and the bending modes of the  $(\text{CO}_3)^{2-}$  units. There is another consideration caused by the presence or absence of water in the structure. The presence of water may cause significant shifts in the bands associated with both  $(\text{UO}_2)^{2+}$  units and  $(\text{CO}_3)^{2-}$  units. In this work we report the Raman spectra of kamotoite at 298 and 77 K and relate the spectra to the structure and composition of the minerals.

## Experimental

### *Mineral*

The mineral kamotoite was obtained on loan from Museum Victoria and originated from Kamoto East Open cut, Kamoto, Kolwezi, Western area, Shaba Cu belt, Shaba (Katanga), Congo (Zaire) nowadays Democratic Republic of Congo [14-16].

### *Raman spectroscopy*

The crystals of kamotoite were placed on the stage of an Olympus BHSM microscope, equipped with 10x and 50x objectives and part of a Renishaw 1000 Raman microscope system, which also includes a monochromator, a filter system and a Charge Coupled Device (CCD). The crystals are homogenous. The spatial resolution of the instrument is slightly less than 1 micron. This determines the size of the crystals to be measured. Raman spectra were excited by a HeNe laser (633 nm) at a resolution of 2  $\text{cm}^{-1}$  in the range between 100 and 4000  $\text{cm}^{-1}$ . The nominal spectral resolution of the instrument is 2  $\text{cm}^{-1}$ . Repeated acquisition using the highest magnification was accumulated to improve the signal to noise ratio. Spectra were calibrated using the 520.5  $\text{cm}^{-1}$  line of a silicon wafer. In order to ensure that the correct spectra are obtained, the incident excitation radiation was scrambled. Previous studies by the authors provide more details of the experimental technique. Spectra at liquid nitrogen temperature were obtained using a Linkam thermal stage (Scientific Instruments Ltd, Waterfield, Surrey, England). Details of the technique have been published by the authors [17-20].

Spectroscopic manipulation such as baseline adjustment, smoothing and normalisation were performed using the Spectracalc software package GRAMS (Galactic Industries Corporation, NH, USA). Band component analysis was undertaken using the Jandel 'Peakfit' software package, which enabled the type of fitting function to be selected and allows specific parameters to be fixed or varied

accordingly. Band fitting was done using a Gauss-Lorentz cross-product function with the minimum number of component bands used for the fitting process. The Gauss-Lorentz ratio was maintained at values greater than 0.7 and fitting was undertaken until reproducible results were obtained with squared correlations of  $r^2$  greater than 0.995.

## Results and Discussion

The Raman spectra of kamotoite in the 650 to 1150  $\text{cm}^{-1}$  region at 298 and 77 K are shown in Figure 1. The results of the Raman spectral analysis are reported in Table 1 together with the published infrared data. The Raman spectra in this region show three features (a) bands around 1130  $\text{cm}^{-1}$  (b) bands centred upon 915  $\text{cm}^{-1}$  and (c) a band at 747  $\text{cm}^{-1}$ . The band at 1130.9  $\text{cm}^{-1}$  is strongly asymmetric and a second band may be resolved at 1124.6  $\text{cm}^{-1}$ . These two bands are observed at 1133.3 and 1124.0  $\text{cm}^{-1}$  in the 77 K spectrum. These bands are assigned to the  $\nu_1$  symmetric stretching mode of the  $(\text{CO}_3)^{2-}$  units. Botto et al. reported a low intensity band at 1120  $\text{cm}^{-1}$  [9] which corresponds with the Raman bands observed in this work. Čejka [11] reported bands at 1123  $\text{cm}^{-1}$  which corresponds precisely with the second of the Raman bands. The splitting of the symmetric stretching mode implies two different symmetrically distinct  $(\text{CO}_3)^{2-}$  units in the kamotoite structure. Band at 1170.4 and 862.3  $\text{cm}^{-1}$  (77 K) may be connected with  $\delta$  U-OH bending vibration.

In the Raman spectrum of kamotoite a band is observed at 814.7  $\text{cm}^{-1}$  which may be resolved into two components at 814.7 and 809.6  $\text{cm}^{-1}$ . The band shifts to 811.6  $\text{cm}^{-1}$  in the 77 K spectrum. This spectral region is where there is potential overlap between the symmetric stretching modes of the  $(\text{UO}_2)^{2+}$  units and the  $\nu_2$  bending modes of the  $(\text{CO}_3)^{2-}$  units. The problem of band overlap between the symmetric stretching modes of the  $(\text{UO}_2)^{2+}$  units and the  $\nu_2$  bending modes of the  $(\text{CO}_3)^{2-}$  units is exacerbated by the non-equivalence of the  $(\text{CO}_3)^{2-}$  units in the structure of kamotoite. No bands were observed in the infrared spectra as published by Čejka in the 814  $\text{cm}^{-1}$  region although a, low intensity band at 835  $\text{cm}^{-1}$  as observed in the DRIFT spectrum of kamotoite. One possible assignment is that the band at 814.7  $\text{cm}^{-1}$  is attributable to the  $\nu_1$  symmetric stretching mode of the  $(\text{UO}_2)^{2+}$  units and the second band at 809.6  $\text{cm}^{-1}$  is due to the  $\nu_2$  bending modes of the  $(\text{CO}_3)^{2-}$  units. It is interesting that the two bands shift in opposite directions in the 77 K spectrum and only one band centred at 811.6  $\text{cm}^{-1}$  is observed in the 77 K spectrum. An empirical relation  $R_{\text{U-O}} = 106.50[\nu_1(\text{UO}_2)^{2+}]^{-2/3} + 0.575$  Å by Bartlett and Cooney [21] enables the calculation of U-O bond lengths in uranyles. Wavenumbers of all bands which may be assigned to the  $\nu_1(\text{UO}_2)^{2+}$  are used for these calculations: 1.796/814.7 and 1.801/809.6 Å (298 K), 1.798/812.7, 1.799/811.6 and 1.819/792.0 Å (77 K). Botto et al. [9] inferred from the wavenumber of the  $\nu_3(\text{UO}_2)^{2+}$  observed in the infrared spectrum the U-O bond length in uranyl 1.77 Å. Obtained results are close to the U-O bond lengths  $\sim 1.80$  Å in uranyles of bijvoetite-(Y) [1] and agree also with the average U-O bond lengths in natural and synthetic uranyl compounds [22-24]

A very low intensity band is observed in the 298 K spectrum at  $920\text{ cm}^{-1}$  which is attributed to the antisymmetric stretching modes of the  $(\text{UO}_2)^{2+}$  units. Čejka reported a band at  $911\text{ cm}^{-1}$  which was assigned to this vibration [11]. A band is observed at  $746.1\text{ cm}^{-1}$  in the 298 K Raman spectrum which shifts to  $747.2\text{ cm}^{-1}$  in the 77 K spectrum and represents the  $\nu_4$  in-plane bending region of the  $(\text{CO}_3)^{2-}$  units.

The low wavenumber region of the Raman spectra of kamotoite at 298 and 77 K are shown in Figure 2. Two broad bands are observed at  $584$  and  $547.3\text{ cm}^{-1}$  in the 298 K spectrum which shift to  $528.3$  and  $544.6\text{ cm}^{-1}$  in the 77 K spectrum. One possible assignment is that these bands may be attributed to water librational modes. An intense band is observed in the 298 K spectrum at  $417.7\text{ cm}^{-1}$  which may be resolved into two components at  $422.0$  and  $416.6\text{ cm}^{-1}$  in the 77 K spectrum. The intensity and sharpness of the band suggests that the band is due to a symmetric stretching vibration and one possibility is that the band is due to an  $\text{Y}_2\text{O}_2$  stretching vibration. A band is found at  $336.3\text{ cm}^{-1}$  in the 298 K spectrum which shifts to  $338.4\text{ cm}^{-1}$  in the 77 K spectrum. Two other bands are observed in the 77 K spectrum at  $286.4$  and  $231.6\text{ cm}^{-1}$ . These bands are assigned to the  $\nu_2$   $(\text{UO}_2)^{2+}$  bending modes. The results of the Raman spectra lead to the conclusions that not only are the UO bond lengths different but that some of the  $(\text{UO}_2)^{2+}$  units are symmetrically distinct and therefore not equivalent. Čejka suggested that a possible coincidence of  $\nu_2$   $\delta$   $(\text{UO}_2)^{2+}$  and U-O ligand vibrations [11].

The spectral region between  $1200$  and  $1800\text{ cm}^{-1}$  is shown in Figure 3. This spectral region shows poor signal to noise. The reason for the low intensity of the bands in this region is that the bands are strongly infrared active and yet are poor Raman scatterers. The band at  $1634.4\text{ cm}^{-1}$  is due to the water bending mode. Čejka reported the band in the infrared spectrum at  $1610\text{ cm}^{-1}$  in the KBr absorption spectrum and at  $1645\text{ cm}^{-1}$  in the DRIFT spectrum [11]. The bands in the Raman spectrum at  $1551.0$ ,  $1382.0$  and  $1337.7\text{ cm}^{-1}$  are assigned to the antisymmetric stretching vibrations of the  $(\text{CO}_3)^{2-}$  units. Botto et al. found infrared bands at  $1540$  and  $1355\text{ cm}^{-1}$  which is in reasonable agreement with the position of the Raman bands reported in this work [9]. Botto et al. suggested that the large splitting of this vibrational mode was due to the bidentate bonding of the  $(\text{CO}_3)^{2-}$  units to the uranyl groups [9].

The Raman spectrum of the OH stretching region is shown in Figure 4. Two bands are observed at  $3516.0$  and  $3361.0\text{ cm}^{-1}$ . Čejka gave infrared bands in the DRIFT spectrum as  $3564$  and  $3496\text{ cm}^{-1}$  although only a single band at  $3420\text{ cm}^{-1}$  was observed in the KBr absorption spectrum [11]. From the spectra may be inferred that free or weakly hydrogen-bonded  $(\text{OH})^-$  units and more strongly hydrogen bonded water molecules are present in the crystal structure of kamotoite-(Y). This is supported by the corresponding bands observed in the infrared spectra of this mineral [3, 9, 10] and general conclusions by Libowitzky [25]. The arrangement of water molecules and hydroxyls may be important for the stability of the crystal structure of kamotoite-(Y) Hawthorne [26, 27].

## Conclusions

Raman spectra of the uranyl containing secondary mineral kamotoite-(Y) are presented, interpreted, and compared with published infrared spectra of this mineral.

Bands attributed to the uranyl and carbonate units stretching and bending vibrations, the stretching and bending vibrations and libration modes of water molecules and the stretching and bending vibrations of hydroxyls are discussed. U-O bond lengths in uranyl are calculated from the uranyl stretching vibrations. Short comment to the hydrogen-bonding network in kamotoite-(Y) crystal structure is given.

This study serves to show the usefulness of Raman spectroscopy for the study of the uranium minerals. The Raman spectra are well resolved with spectroscopic windows for each of the regions for the vibrating units of the mineral, kamotoite. The bands are well resolved and are narrow when compared with the infrared spectra. The infrared spectra are inherently broad with overlapping bands superimposed on non linear backgrounds of significant intensity. This separation of the bands enables a better and more detailed assignment of the Raman bands, a task which is not so easy in the infrared spectra. This work serves to show the application of Raman spectroscopy for the in-situ analysis of a uranyl carbonate mineral known as kamotoite. The use of the microscope and associated Raman spectrometer allows single crystals to be selected for the analysis. It should be noted there is almost no sample preparation apart from the alignment of the crystals in the incident beam. Raman spectroscopy may be used with a thermal stage allowing spectra to be obtained at any temperature. The collection of Raman data at liquid nitrogen temperature enables significantly improved band separation. Raman spectroscopy has by its very nature normally narrow bands as compared with infrared spectroscopy, and by obtaining data at 77 K, improved signal to noise is achieved.

### **Acknowledgements**

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<b>Raman</b>	<b>Raman</b>	<b>infrared</b>	<b>infrared</b>	<b>infrared</b>	<b>infrared</b>
		KBr	DRIFT	KBr	KBr
298 K Centre (cm <sup>-1</sup> )	77 K Centre (cm <sup>-1</sup> )	Published [11]	Published [11]	Published [9]	Published [10]
3516.0 3361.0		3420	3564 3496	3540 3370	3423 (2929) (2857) (2457) (1865) 1731
1634.4 1551.2		1610 1553	1645	1620	1606
1337.6		1536 1352	1539 1356	1540 1355	1537 1364
1130.9 1124.6	1170.4 1133.3 1124.0	1123 999 911	1121	1120	1195 1155 1122
	862.3		904 835	910 865 830	911
814.7 809.6	812.7 811.6 792.0				
745	747.2 732.3	744	738	745	742
	617.3	682	629		660
584.0 547.3	582.3 544.6	504	583 496	470	509
	422.0		443		
417.7 336.3	416.6 338.4		403		
	286.4 274.3 251.9 231.6			352 310 270 255 215	366 280

**Table 1 Results of the Raman spectral analysis of kamotoite and comparison with published infrared data.**

### *List of Tables*

Table 1 Results of the Raman spectral analysis of kamotoite and comparison with published infrared data.

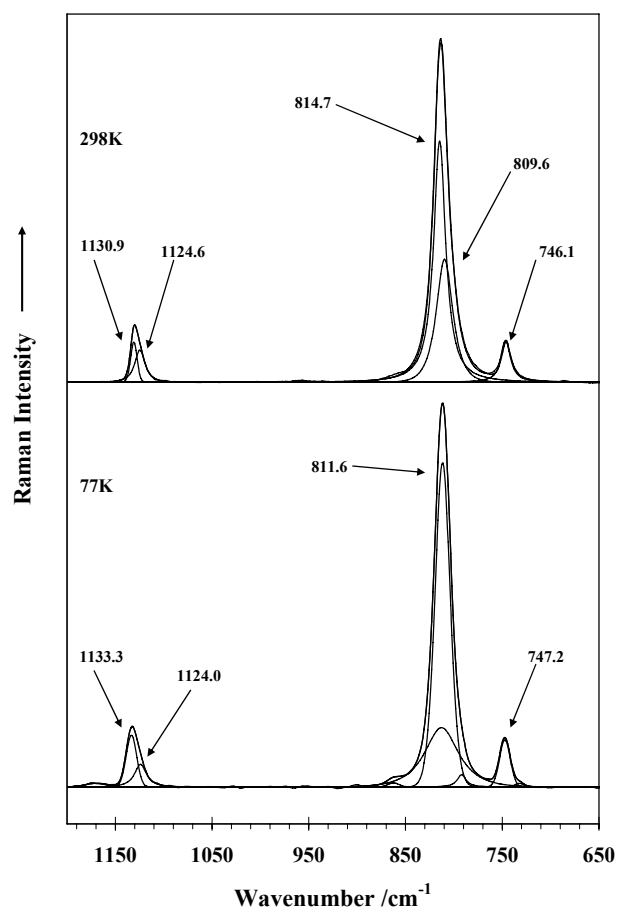
### *List of Figures*

Figure 1 Raman spectra of kamotoite-(Y) at 298 and 77 K between 650 and 1200  $\text{cm}^{-1}$ .

Figure 2 Raman spectra of kamotoite-(Y) at 298 and 77 K between 200 and 600  $\text{cm}^{-1}$ .

Figure 3 Raman spectra of kamotoite-(Y) at 298 between 1200 and 1800  $\text{cm}^{-1}$ .

Figure 4 Raman spectra of kamotoite-(Y) at 298 between 2900 and 3900  $\text{cm}^{-1}$ .



**Figure 1**

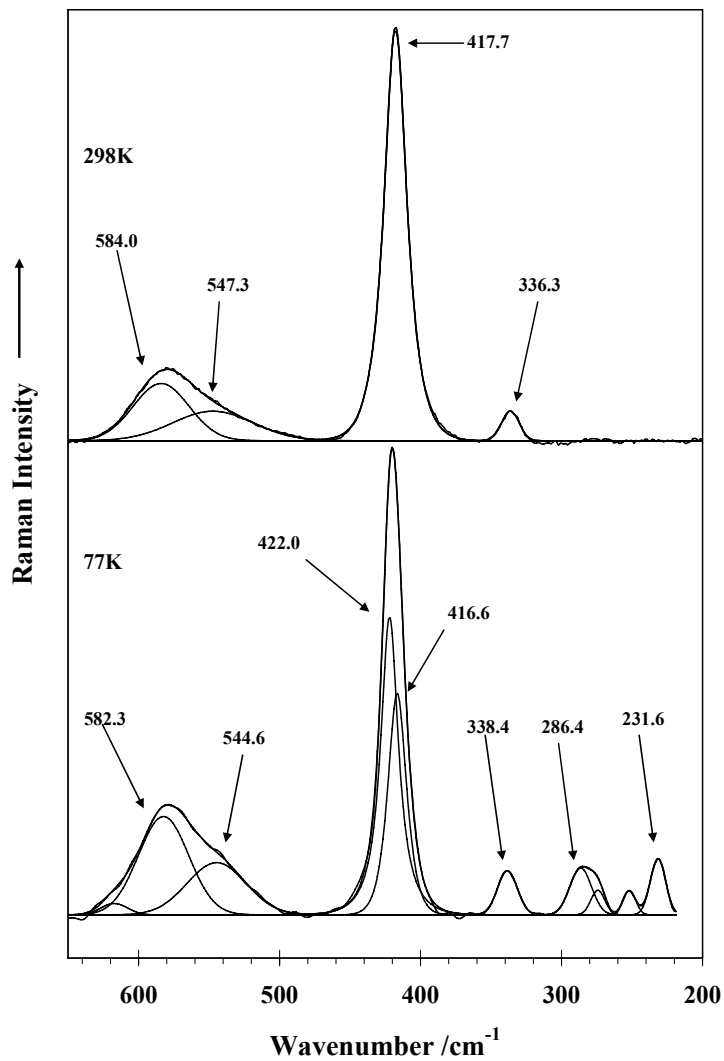


Figure 2

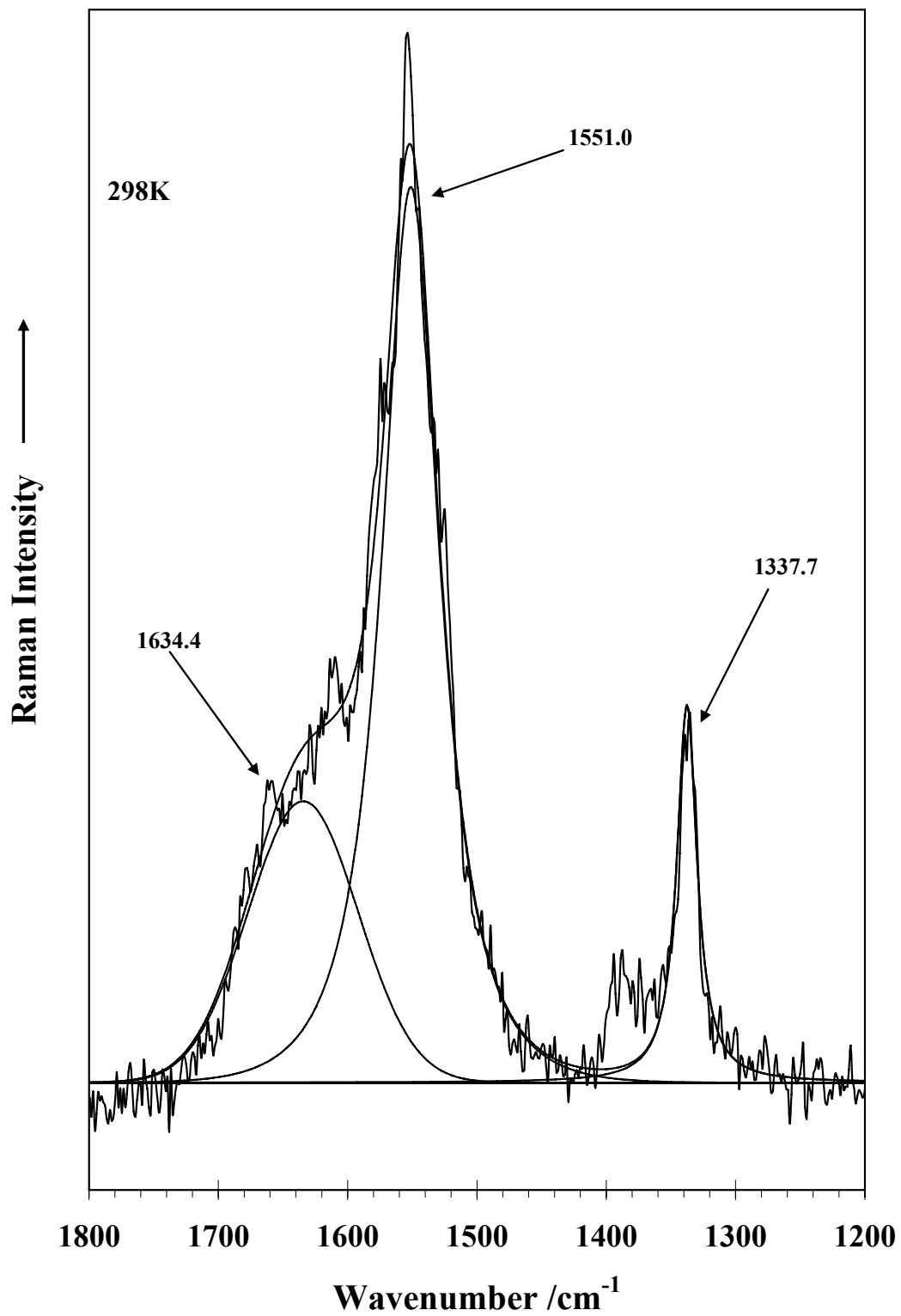
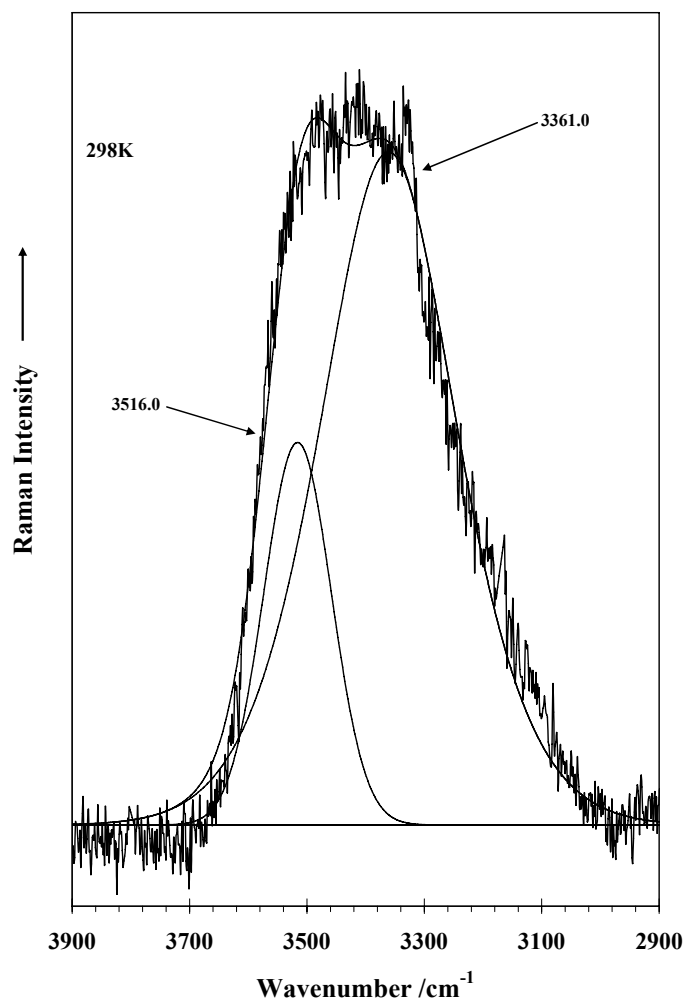


Figure 3



**Figure 4**