

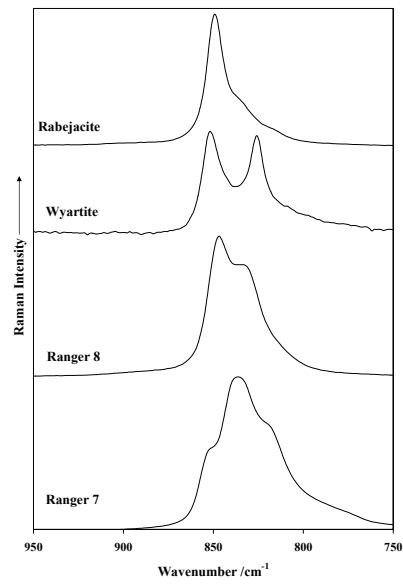
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Raman microscopy has been used to detect the presence of wyartite in the presence of rabejacite. Wyartite is a mineral known for the occurrence of pentavalent  $U^{5+}$ . Two bands at 853 and 837  $cm^{-1}$  are assigned to the  $\nu_1$  stretching modes of the  $UO_2$  units.



**Ray L. Frost, Dermot A. Henry and Kristy Erickson**

*Raman spectroscopic detection of wyartite in the presence of rabejacite*

## Raman spectroscopic detection of wyartite in the presence of rabejacite

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### Abstract

Raman microscopy has been used to affirm the presence of wyartite  $\text{CaU}^{5+}(\text{UO}_2)_2(\text{CO}_3)\text{O}_4(\text{OH})(\text{H}_2\text{O})_7$  in the presence of rabejacite  $(\text{Ca}(\text{UO}_2)_4(\text{SO}_4)_2(\text{OH})_5 \cdot 6\text{H}_2\text{O})$  obtained from the Ranger Mine, Northern Territory, Australia. This occurrence is somewhat unusual in that it means that a uranyl carbonate has been formed under acid conditions. Wyartite is a mineral known for the occurrence of pentavalent  $\text{U}^{5+}$ .<sup>1</sup> A band is observed at  $818 \text{ cm}^{-1}$  in the Raman spectrum of wyartite assigned to the  $\nu_2$  symmetric bending mode of the  $(\text{CO}_3)^{2-}$  units. The presence of carbonate is confirmed by the  $\nu_1$  stretching vibration at  $1071 \text{ cm}^{-1}$  and the  $\nu_3$  stretching vibrations at  $1445$  and  $1345 \text{ cm}^{-1}$ . Two bands are observed at  $853$  and  $837 \text{ cm}^{-1}$  and are assigned to the  $\nu_1$  stretching modes of the  $\text{UO}_2$  units. Raman spectroscopy enables the partial band separation of the  $\nu_2$   $(\text{CO}_3)^{2-}$  and  $\nu_1$  modes of  $\text{UO}_2$ . The Raman spectrum of rabejacite is characterised by an intense sharp band at  $1010 \text{ cm}^{-1}$  assigned to the  $\nu_1$  stretching mode of  $(\text{SO}_4)^{2-}$ . Three bands observed at  $1086$ ,  $1123$  and  $1175 \text{ cm}^{-1}$  are attributed to the  $\nu_3$  antisymmetric stretching modes of  $(\text{SO}_4)^{2-}$ . The mineral rabejacite is also characterised by  $\nu_2$  bending modes at  $457$  and  $394 \text{ cm}^{-1}$  and  $\nu_4$  bending modes at  $666$ ,  $605$ ,  $537$  and  $505 \text{ cm}^{-1}$ . Raman spectroscopy has proven most useful for the detection of wyartite in the presence of other mineral phases.

**Keywords:** rabejacite, wyartite, dehydration, johannite, Raman spectroscopy, infrared spectroscopy

### Introduction

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The mineral wyartite  $\text{CaU}^{5+}(\text{UO}_2)_2(\text{CO}_3)\text{O}_4(\text{OH})(\text{H}_2\text{O})_7$  was first identified in samples from the Shinkolobwe mine (Kasolo mine), Likasi, Shaba (Katanga), Congo (Zaire)<sup>2</sup>. It was found mixed with ianthinite ( $\text{UO}_2 \cdot 5\text{UO}_3 \cdot 10.56\text{H}_2\text{O}$ ) and occurs in crystals flattened on (001) and elongated along (010). The mineral is black to violet black and is orthorhombic with ( $a = 11.25 \text{ \AA}$ ,  $b = 7.08$ , and  $c = 20.98$ ;  $Z = 2$ )<sup>1</sup>. There have been some suggestions that the mineral alters with time to a second wyartite phase<sup>3</sup>. This phase also contains pentavalent U<sup>1</sup>. It is unusual to have a uranyl carbonate with a colour other than yellow. The mineral is unusual in that it contains pentavalent uranium. The mineral has been found with other minerals in the same deposit namely urancalcrite, uraninite, uranophane, and masuyite<sup>4</sup>. The mineral rabejacite ( $\text{Ca}(\text{UO}_2)_4(\text{SO}_5)_2(\text{OH})_5 \cdot 6\text{H}_2\text{O}$ ) is also orthorhombic ( $a = 8.73(1)$ ,  $b = 17.09(2)$ ,  $c = 15.72(2)$ ,  $z = 4$ )<sup>5</sup> and was first described from Rabéjac, Lodève, Hérault, Languedoc-Roussillon, France. Rabejacite was found in two deposits of the Permian basin of Lodeve: in the red Saxonian pelites from Rabejac (7 km S-SE from Lodeve). Rabejacite occurs as flattened tablets (up to 0.1 mm), acicular crystals or rounded nodules (up to 0.3 mm). The infrared spectrum proves the presence of structurally non-equivalent sulfate groups<sup>6</sup>. The mineral is bright to amber yellow and transparent to translucent with a vitreous lustre.

Infrared spectroscopy has been used to study uranium minerals<sup>7</sup>. However, sometime, the infrared spectra are simply used for finger-printing the minerals without structural attribution<sup>8</sup>. Few Raman spectroscopic studies have been undertaken on uranium minerals<sup>9,10</sup>. Nikanovich et al. calculated the vibrational spectrum of calcium uranyl phosphate hexahydrate and compared the calculated results with the experimentally determined values<sup>11</sup>. According to Prins et al. existing data in literature make clear that the wavenumber which belongs to  $\nu_1$  ( $\text{UO}_2$ )<sup>2+</sup> is in most cases found in the region around  $850 \text{ cm}^{-1}$ , whereas the other stretching wavenumber  $\nu_3$  ( $\text{UO}_2$ )<sup>2+</sup> has a distinctly higher wavenumber and is situated in a region near  $950 \text{ cm}^{-1}$ <sup>12,13</sup>. Cejka reported the Raman spectrum of torbernite<sup>14</sup>.

Recently mineral specimens which may contain wyartite and rabejacite were found in the Ranger Mine deposit, Jabiru, Northern Territory, Australia. The mineral contains bright yellow crystals which may be rabejacite. As part of our on-going research into the vibrational spectroscopy of uranium minerals, we report the analysis of rabejacite and wyartite and relate their spectra to their molecular structures.

## Experimental

### *Minerals*

The uranium minerals were obtained from Museum Victoria. Rabejacite and wyartite originated from the Ranger No. 1 deposit, Jabiru, Northern Territory, Australia. A mineral sample M47068 contained predominately rabejacite with some wyartite and is used as a reference sample. The minerals were analysed by X-ray diffraction for phase identification and by electron probe for chemical composition. The following museum samples were used. Chemical analyses for carbon content were obtained.

Rabejacite = M43465 (note this is from the type locality, Rabejac France)

Wyartite = M48554 (Ranger No.1 Deposit XRD identified as wyartite 17 A phase)

or wyartite II)

Ranger 7a = M48552, (Ranger No. 1 XRD identified as rabejacite)

Ranger No. 8 M48553, (Ranger No. 1 XRD identified as mixture of rabejacite and wyartite)

M47068 Ranger No. 1 Deposit identified as mixture of both rabejacite and wyartite.

### ***Raman microprobe spectroscopy***

The crystals of the uranium carbonate or sulphate were placed and orientated 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). Raman spectra were excited by a HeNe laser (633 nm) at a resolution of  $2\text{ cm}^{-1}$  in the range between 100 and  $4000\text{ cm}^{-1}$ . 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 controlled temperatures were obtained using a Linkam thermal stage (Scientific Instruments Ltd, Waterfield, Surrey, England). Details of the technique have been published by the authors<sup>15-20</sup>. Spectral manipulation such as baseline adjustment, smoothing and normalisation was performed using the GRAMS® software package (Galactic Industries Corporation, Salem, NH, USA).

### **Infrared absorption spectroscopy**

Infrared absorption spectra were obtained using the KBr pressed pellet technique using a Perkin-Elmer FT-IR spectrometer 2000 bench using  $4\text{ cm}^{-1}$  resolution with 128 scans. Diffuse Reflectance Fourier Transform Infrared spectroscopic (commonly known as DRIFT) analyses were undertaken using a Bio-Rad 60A spectrometer. 512 scans were obtained at a resolution of  $2\text{ cm}^{-1}$  with a mirror velocity of  $0.3\text{ cm/sec}$ . Spectra were co-added to improve the signal to noise ratio.

### **Results and discussion**

The vibrational spectroscopy of some uranyl carbonates have been published<sup>21,22</sup>. Cejka published the infrared spectrum of wyartite<sup>22</sup>. The spectrum showed a broad band centre around  $3450\text{ cm}^{-1}$ , multiple overlapping bands in the  $1200$  to  $1700\text{ cm}^{-1}$  region, no doubt due to the overlap of the HOH bending mode and the  $\nu_3(\text{CO}_3)^{2-}$  stretching modes. A band was observed around  $900\text{ cm}^{-1}$  and is probably the  $\nu_3$  stretching mode of the uranyl units. Cejka rightly points out the difficulty of the interpretation of the infrared spectra of uranyl carbonates because of the potential overlap of the  $\nu_1$  modes of  $\text{UO}_2$  and the  $\nu_2$  modes of  $(\text{CO}_3)^{2-}$ . Equally well there is the possibility of overlap between the  $\nu_3$  modes of  $(\text{CO}_3)^{2-}$  and the bending modes of water. Whereas the infrared spectra of uranyl sulphates can be strongly influenced by sample preparation<sup>22</sup>, such difficulties are not found with the use of Raman spectroscopy. The  $\nu_1$  symmetric stretching mode of  $(\text{CO}_3)^{2-}$  is observed at around  $1064\text{ cm}^{-1}$  for sodium uranyl tricarbonates ( $\text{Na}_4[\text{UO}_2(\text{CO}_3)_3]$ )<sup>22</sup>. Some variation in

the peak position was observed for synthetic uranyl tricarbonates and a range of 1038 to 1049  $\text{cm}^{-1}$  was found. However a peak position of 1110  $\text{cm}^{-1}$  was observed for blatonite ( $\text{UO}_2\text{CO}_3 \cdot \text{H}_2\text{O}$ ), Wide variation in the peak position of the  $\nu_1$  symmetric stretching mode of  $(\text{CO}_3)^{2-}$  is observed for sharpite ( $(\text{Ca}(\text{UO}_2)_6(\text{CO}_3)_5(\text{OH})_4 \cdot 6\text{H}_2\text{O})^{22}$ ). The  $\nu_3$  of  $(\text{CO}_3)^{2-}$  in the uranyl tricarbonates was found at 1345, 1360, 1562 and 1577  $\text{cm}^{-1}$ . The band as observed at 1367  $\text{cm}^{-1}$  for blatonite and multiple bands for sharpite at 1381, 1418, 1445, 1540  $\text{cm}^{-1}$ . The  $(\text{CO}_3)^{2-}$   $\nu_2$  bending modes were observed for the uranyltricarbonates at 827 and 848  $\text{cm}^{-1}$ , 914  $\text{cm}^{-1}$  for blatonite and at 810 to 850  $\text{cm}^{-1}$  for sharpite; however the latter band position overlaps with the  $\nu_1$  stretching modes of the  $\text{UO}_2$  units. The  $\nu_4$  bending mode of  $(\text{CO}_3)^{2-}$  for the uranyl tricarbonates is observed at 703 and 705  $\text{cm}^{-1}$ , at 690, 705, 760 and 775  $\text{cm}^{-1}$  for sharpite. The  $\nu_1$  stretching modes of the  $(\text{UO}_2)^{2+}$  units is observed between 808 and 831  $\text{cm}^{-1}$  for the uranyl tricarbonates, and between 759 and 780  $\text{cm}^{-1}$  for sharpite.

The infrared spectra of some uranyl compounds containing sulphate<sup>23</sup> such as johannite ( $\text{Cu}(\text{UO}_2)_2(\text{OH})_2(\text{SO}_4)_2 \cdot 8\text{H}_2\text{O}$ )<sup>24</sup>, jachymovite ( $(\text{UO}_2)_8(\text{SO}_4)(\text{OH})_{14} \cdot 13\text{H}_2\text{O}$ )<sup>25</sup>, uranopilite ( $(\text{UO}_2)_6(\text{SO}_4)(\text{OH})_{10} \cdot 12\text{-}13\text{H}_2\text{O}$ )<sup>26</sup>, and zippeites ( $\text{M}^{2+}(\text{UO}_2)_6(\text{SO}_4)_3(\text{OH})_{10} \cdot n\text{H}_2\text{O}$ ) where  $\text{M}^{2+} = \text{Co, Ni, Mg, Zn, Cd}$  and  $n=8$  or 16 have been published<sup>22</sup>. Cejka states that in the case of the  $(\text{SO}_4)^{2-}$  anion,  $C_1$  is the only admissible site symmetry. The vibrations  $\nu_1$  ( $A_1 \rightarrow A$ ) and  $\nu_2$  ( $E \rightarrow 2A$ ) become infrared active; and the doubly degenerate  $\nu_2$  and triply degenerate  $\nu_3$  vibrations and  $\nu_4$  vibrations split. The absorption band for johannite which could only be attributed to the  $\nu_1$  symmetric stretching mode was observed as a very low intensity band at 1040  $\text{cm}^{-1}$ . The band is observed as a very intense band at 1042  $\text{cm}^{-1}$  (R. Frost unpublished data). In the infrared spectra the most intense bands are observed at 1145 and 1096  $\text{cm}^{-1}$  and are assigned to the antisymmetric  $(\text{SO}_4)^{2-}$  stretching vibration. In the Raman spectrum, three intense bands are observed at 1147, 1100 and 1090  $\text{cm}^{-1}$  and are assigned to the antisymmetric  $(\text{SO}_4)^{2-}$  stretching vibration. In the infrared spectrum an absorption band is observed at 619  $\text{cm}^{-1}$  and is attributed to the  $\nu_4$  triply degenerate bending vibration. In the Raman spectrum two bands are observed at 674 and 625  $\text{cm}^{-1}$ . The doubly degenerate  $\nu_2$  bending vibration is observed for johannite at 422 and 384  $\text{cm}^{-1}$ . In the Raman spectrum three bands are observed at 450, 418 and 351  $\text{cm}^{-1}$  and are assigned to this mode. It should also be noted that in the Raman spectrum of johannite three intense bands are observed at 286, 242 and 209  $\text{cm}^{-1}$ . The first two bands are assigned to the doubly degenerate  $\nu_2$  bending modes of the  $(\text{UO}_2)^{2+}$  units. In the infrared spectrum of johannite three weak bands are observed at 832, 821, and 780  $\text{cm}^{-1}$  and are ascribed to the  $\nu_1$  symmetric stretching modes of  $(\text{UO}_2)^{2+}$ . The corresponding  $\nu_3$  antisymmetric stretching modes were observed at 911 and 935  $\text{cm}^{-1}$ . The IR bands at 904  $\text{cm}^{-1}$  (jachymovite) and 930  $\text{cm}^{-1}$  (uranopilite) are also attributed to the  $\nu_3$  antisymmetric stretching modes of  $(\text{UO}_2)^{2+}$ . These bands are not observed in the Raman spectrum of johannite. The symmetric stretching modes of  $(\text{UO}_2)^{2+}$  should occur at 849 and 826  $\text{cm}^{-1}$ .

The Raman spectra in the 750 to 1150  $\text{cm}^{-1}$  region of a selection of samples from the Ranger mine are shown in Figure 1. The 750 to 950  $\text{cm}^{-1}$  region of wyartite is complex (Figure 2). A band is observed at 818  $\text{cm}^{-1}$  and is assigned to the  $\nu_2$  symmetric bending mode of the  $(\text{CO}_3)^{2-}$  units. This assignment is in accordance with the results of previously published data for similar uranyl carbonate minerals. The band is not observed in the spectra of the other samples (Figure 3). The two bands at

853 and 837  $\text{cm}^{-1}$  are assigned to the  $\nu_1$  symmetric stretching modes of the  $(\text{UO}_2)^{2+}$  units. The presence of carbonate is confirmed by the presence of the  $\nu_1$  stretching vibration at 1071  $\text{cm}^{-1}$  and the  $\nu_3$  antisymmetric stretching vibrations at 1445 and 1345  $\text{cm}^{-1}$  (Figure 4). Two bands are observed at 853 and 837  $\text{cm}^{-1}$  and are assigned to the  $\nu_1$  stretching modes of the  $\text{UO}_2$  units. Overlap occurs between the stretching modes of the  $\text{UO}_2$  units and the  $\nu_2$  bending modes of the  $(\text{CO}_3)^{2-}$  units. However Raman spectroscopy (compared with infrared spectroscopic results) does enable the partial band separation of these overlapping bands. In contrast the 950 to 1250  $\text{cm}^{-1}$  region of rabejacite is shown in Figure 5. An intense band is observed at 1010  $\text{cm}^{-1}$  and is attributed to the symmetric stretching mode of sulphate and three bands are observed at 1086, 1123 and 1175  $\text{cm}^{-1}$  and are attributed to the  $\nu_3$  antisymmetric stretching modes of sulphate. A comparison of the spectra in this region can be made with that of johannite. Figure 6 displays the 750 to 950  $\text{cm}^{-1}$  region of the samples from the Ranger mine. Three bands are observed in the  $\nu_1$   $(\text{UO}_2)^{2+}$  stretching region of rabejacite (type mineral: Rabejacite (M43465)) at 849, 837 and 816  $\text{cm}^{-1}$ . Figure 6 shows the variation in the intensity of bands in these positions for the samples from the Ranger 1 deposit. Clearly complexity exists in the  $\text{UO}_2$  stretching region. Band positions of  $\nu_1$   $(\text{UO}_2)^{2+}$  observed in wyartite correspond to the following U-O bonds in uranyl - 853  $\text{cm}^{-1}$  .... 1.759 Å, 837  $\text{cm}^{-1}$  .... 1.774 Å. This is in agreement with the crystal structure analysis<sup>1</sup>. There are three structural distinct uranium positions, two of them are occupied by uranyles. U(1) - O bond lengths are 1.78(2) and 1.87(2) Å, U(2) - O bond lengths are 1.77(2) and 1.80(2) Å.

The Raman spectra of the 300 to 700  $\text{cm}^{-1}$  region of the selected minerals are shown in Figure 7. A low intensity band is observed for wyartite at 667  $\text{cm}^{-1}$  and it is assigned to the  $\nu_4$  bending mode of carbonate. Other bands are observed at 550, 455 and 510  $\text{cm}^{-1}$ . The attribution of these bands is not known. Multiple bands are observed in the bending region of rabejacite. An intense band is observed at 394  $\text{cm}^{-1}$  with another band at 457  $\text{cm}^{-1}$ . These bands are attributed to the  $\nu_2$  bending modes of the  $(\text{SO}_4)^{2-}$ . The doubly degenerate  $\nu_2$  bending vibration is observed for johannite at 422 and 384  $\text{cm}^{-1}$ . In the Raman spectrum of johannite three bands are observed at 450, 418 and 351  $\text{cm}^{-1}$  assigned to the  $\nu_2$  mode. A set of bands are observed at 666, 605, 537 and 505  $\text{cm}^{-1}$ . These bands are assigned to the  $\nu_4$  bending modes of the  $(\text{SO}_4)^{2-}$  of rabejacite. In comparison the Raman spectrum of johannite two bands are observed at 674 and 625  $\text{cm}^{-1}$ . These bands are also found in the other Ranger mineral samples. The mineral sample M47068 is a known sample of rabejacite with some wyartite. The spectra of this sample match that of rabejacite and may contain some wyartite as well. The ranger 8 mineral sample partly matches that of rabejacite.

The Raman spectra of the 150 to 300  $\text{cm}^{-1}$  region are shown in Figure 8. Bands are observed at around 295 and 249  $\text{cm}^{-1}$ . The latter band is intense relative to the 295  $\text{cm}^{-1}$  band. An additional band is observed at around 200  $\text{cm}^{-1}$ . The first two bands are attributed to the  $\nu_2$  bending modes of  $(\text{UO}_2)^{2+}$  units. Some variation in intensity is observed which simply shows the orientation dependence of the sample. The Raman spectra of the hydroxyl stretching region of wyartite and rabejacite are shown in Figure 9. The Raman spectra of this region are inherently of low intensity and the collection of spectra of reasonable quality not easy. Nevertheless the spectra of the two minerals can be distinguished quite readily. The Raman spectrum of wyartite shows bands at 3527, 3416 and 3249  $\text{cm}^{-1}$ . The mineral wyartite contains both hydroxyl units and water. The first two bands at 3527 and 3416  $\text{cm}^{-1}$  are assigned

to the hydroxyl stretching of the OH units and the band at  $3249\text{ cm}^{-1}$  to the hydroxyl stretching of water. The bands at  $2916$  and  $2858\text{ cm}^{-1}$  are ascribed to organic impurities. The Raman spectrum of the hydroxyl stretching region of rabejacite shows two bands at  $3547$  and  $3465\text{ cm}^{-1}$  with a low intensity band at  $3240\text{ cm}^{-1}$ . The attribution of these bands is as for wyartite.

## Conclusions

Raman spectroscopy has been used to affirm the presence of carbonate in uranium minerals obtained from the Ranger Mine, Jabiru, Northern Territory. X-ray diffraction showed the potential presence of the mineral wyartite but because of the detection limits, uncertainty in the analysis occurred. Raman spectroscopy confirms the presence of wyartite in association with rabejacite, the sulphate bearing mineral. This work shows the power of Raman spectroscopy in the determination of minerals containing multiple anions and multiple mineral species. The complex broad overlapping bands found in the infrared spectroscopy of these types of minerals does not occur with Raman spectroscopy where better band separation enables the distinction between the carbonate and sulphate bands.

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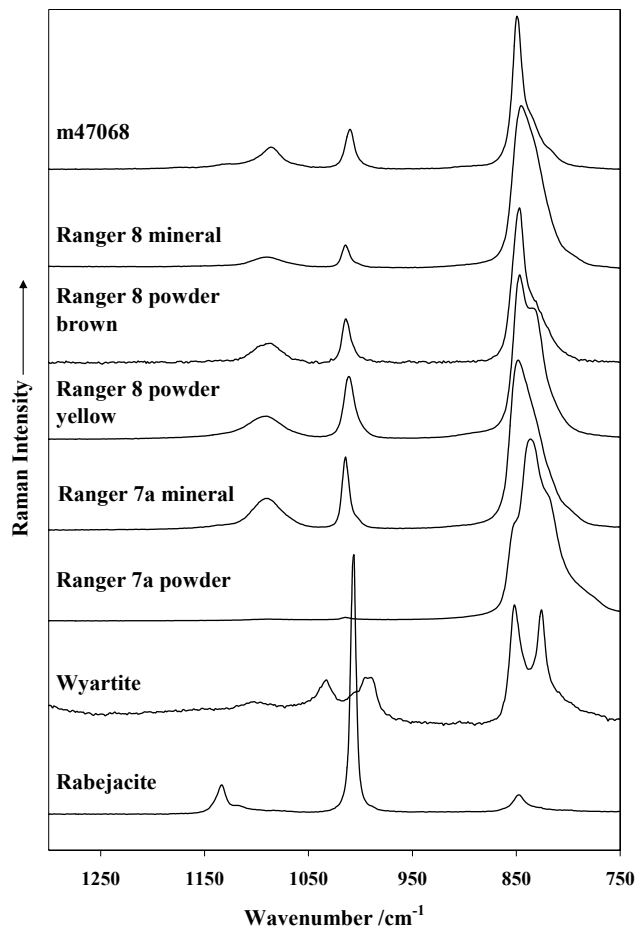
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**Figure 1**

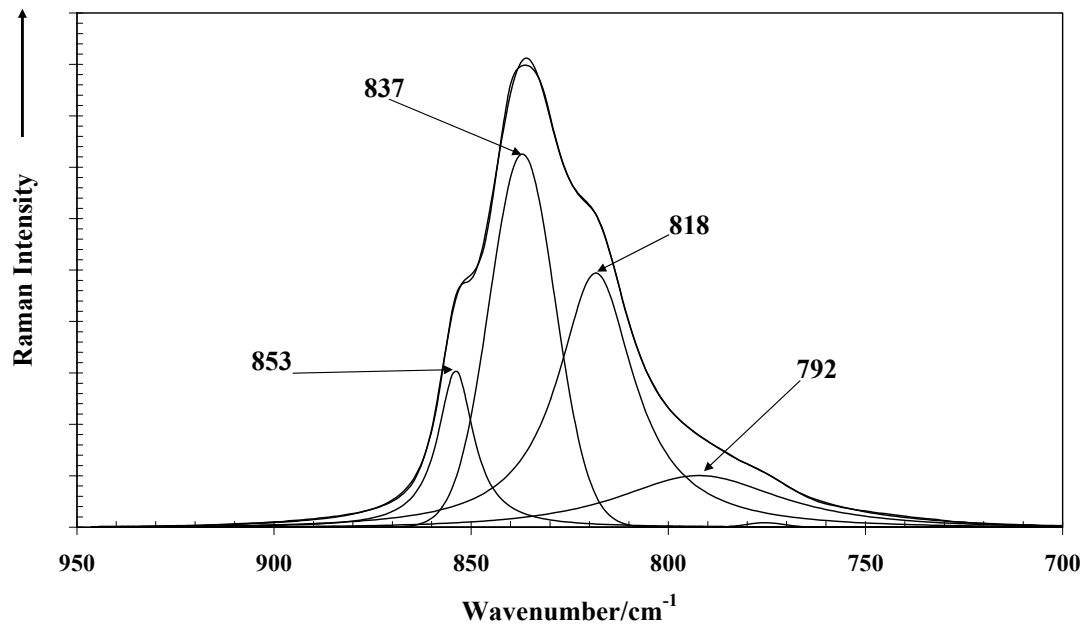


Figure 2

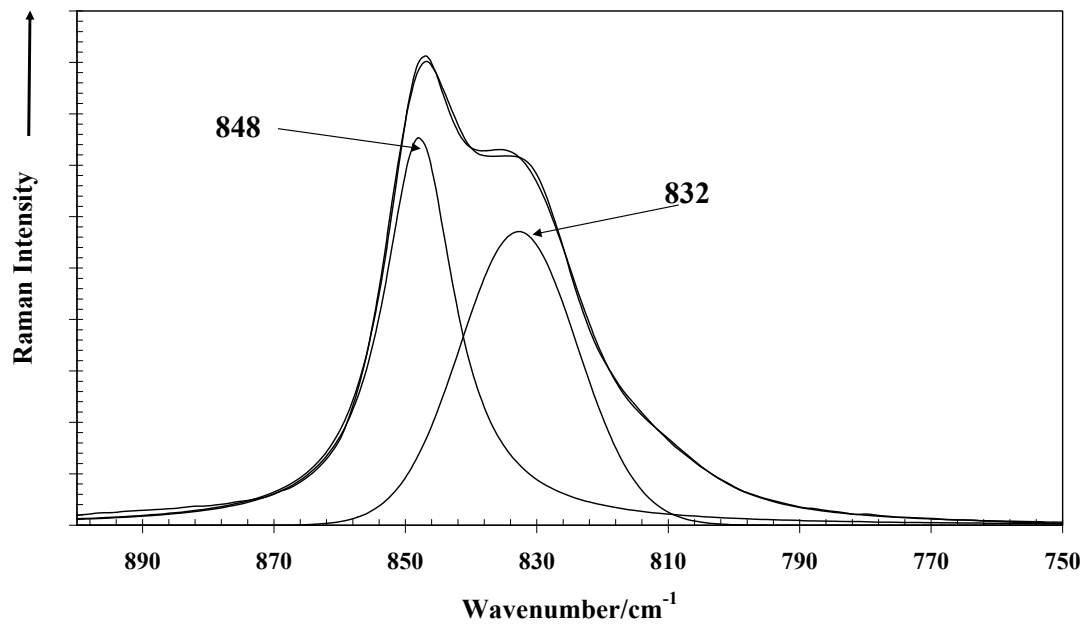


Figure 3

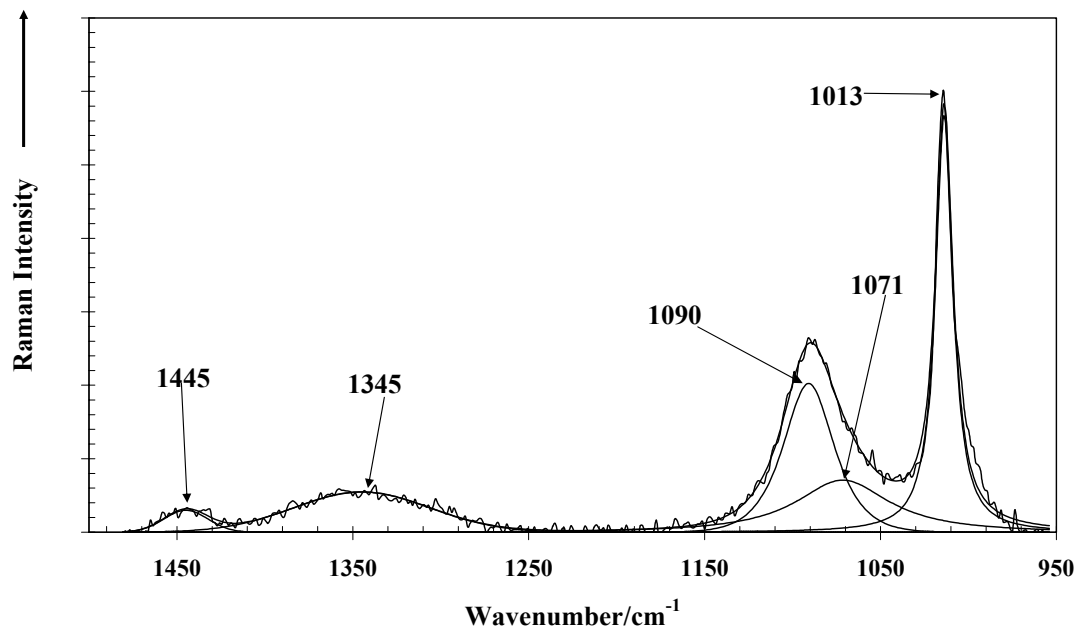


Figure 4

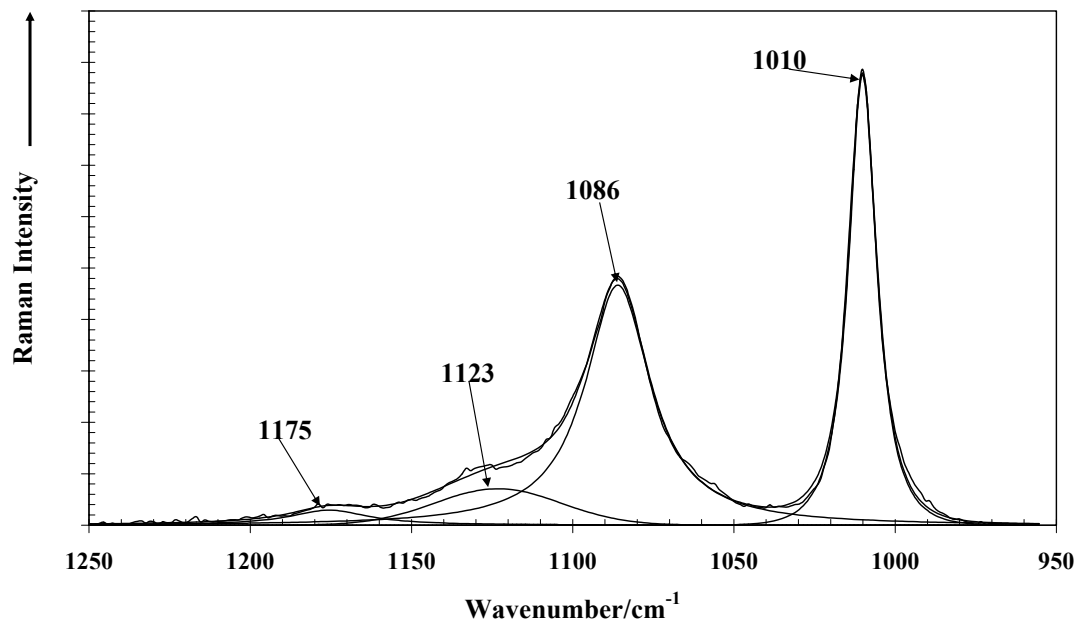
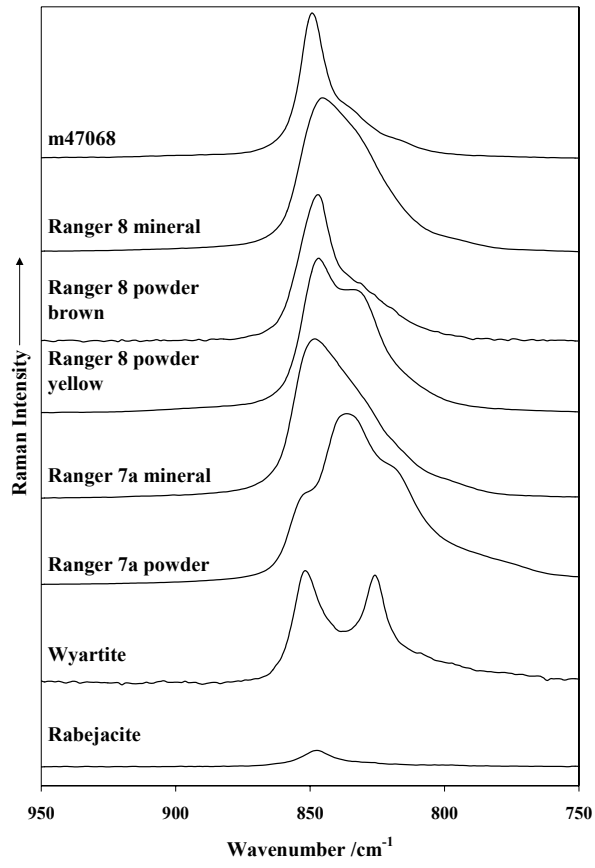
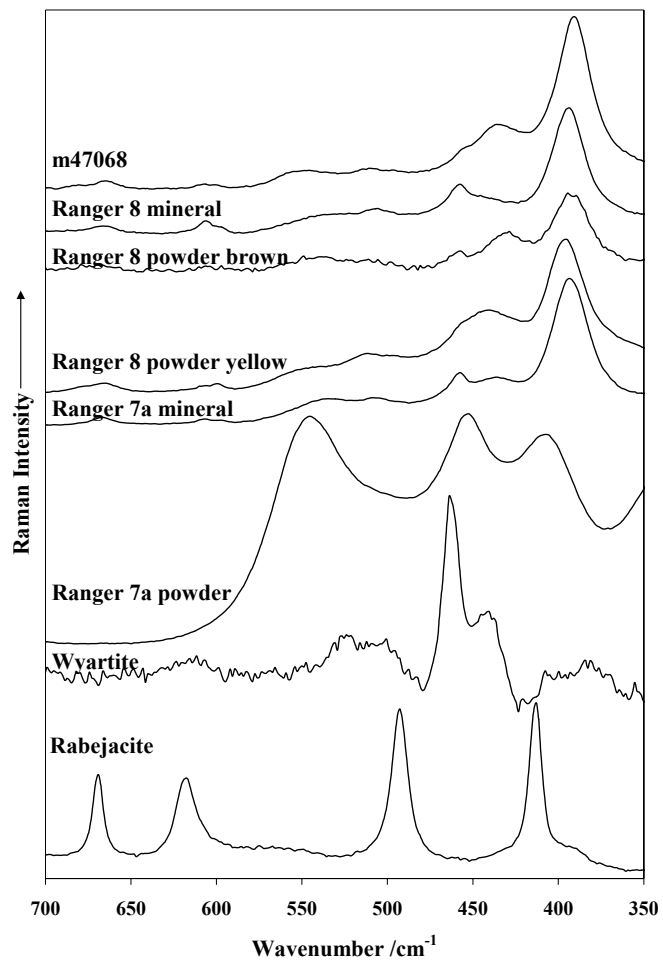


Figure 5

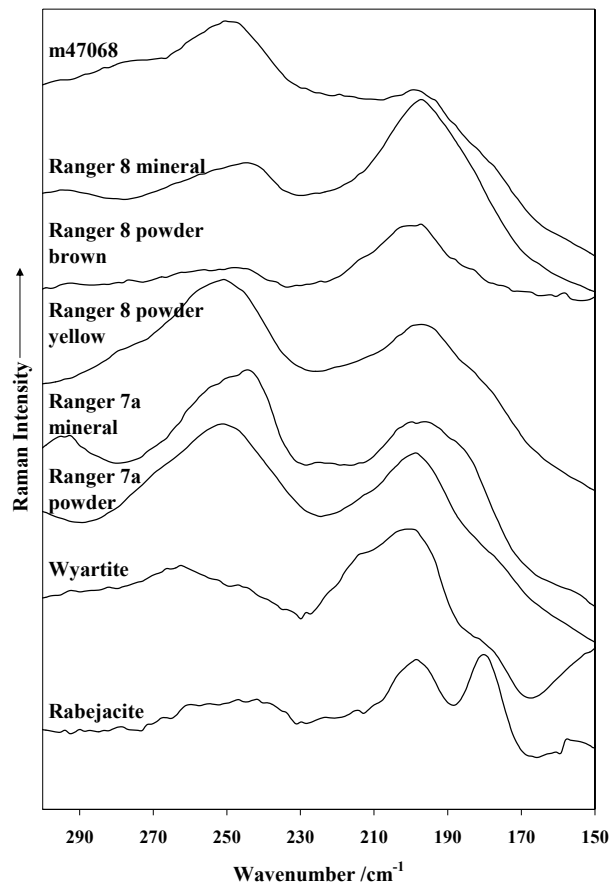


**Figure 6**

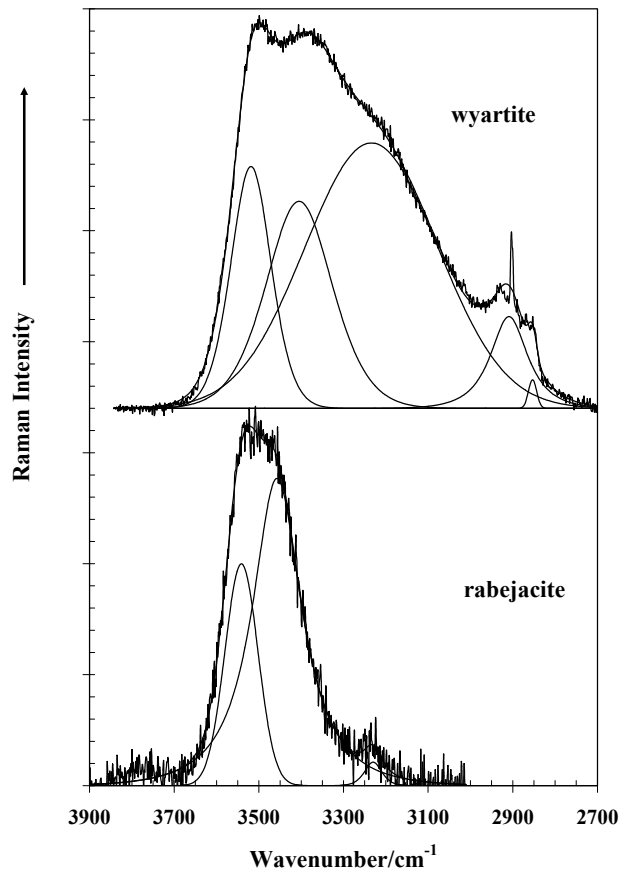




**Figure 7**



**Figure 8**



**Figure 9**

